



BARR

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EPA Region 5 Records Ctr.



204738

September 2, 2003

Sonia Vega
On-Scene Coordinator
U.S. Environmental Protection Agency, Region 5
Emergency Response Branch
520 Lafayette Road North
St. Paul, MN 55155-4194

Re: Preliminary Analytical Data – UNVALIDATED
Laboratory Batch Number – E2300499
Docket No. V-W-'03-748
St. Regis Paper Company Site – Cass Lake, Minnesota

Dear Ms. Vega:

In accordance with your request and in an effort to provide analytical data as as received, enclosed are the most recently received data sheets from Columbia Analytical Services for samples collected in August 2003 in compliance with the UAO issued to International Paper for the St. Regis Paper Company Superfund Site in Cass Lake, MN. It is important to note that these data sheets contain unvalidated data that has not been subjected to quality assurance review and, as such, is not suitable for use in support of any decision making process. Validated data will be provided at a later date in accordance with the schedule outlined in the Order.

If you have any questions concerning this information, please contact me at (952)832-2876 or Tom Ross of International Paper at (901)419-3899.

Sincerely



Thomas D. Mattison
Project Coordinator

cc: Tim Drexler, U.S. EPA – RPM (w/ attachment) ✓
Tom Ross, IP (w/ attachment)

Form 1

CLIENT ID.

PCDD/PCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

H25-26 0-4

Lab Name: Columbia Analytical Services Episode No.:

Lab Code: CAS SDG No.: Method: 8290 Lab Sample ID: E2300499-001A

Client Name: BARR ENGINEERING Sample Wt/Vol: 13.255 g or mL: g

Matrix (Aqueous/Solid/Ash): Solid Initial Calibration Date: 08/05/03

Sample Receipt Date: 08/08/03 Instrument ID: 70S

Ext. Date: 08/13/03 GC Column: DB-5

Ext. Vol (ul): 20.0 Inj. Vol (ul): 1.0 Sample Data Filename: B15550#9

Analysis Date: 18-AUG-03 Time: 17:39:40 Blank Data Filename: B15550#2

Dilution Factor: 1 Cal. Ver. Data Filename: B15549#1

Concentration Units (pg/L or ng/Kg dry weight): ng/Kg. % Moisture/Lipid: 10.13

ANALYTE	CONCENTRATION FOUND	DETECTION LIMIT	Qual. (1)	ION ABUND. RATIO (2)	RRT (2)	MEAN RRF
2,3,7,8-TCDD	0.554	0.130	I	0.85	1.000	0.97
1,2,3,7,8-PeCDD	7.988	0.133		1.58	1.000	0.93
1,2,3,4,7,8-HxCDD	24.097	0.155		1.16	0.998	1.00
1,2,3,6,7,8-HxCDD	107.760	0.133		1.24	1.000	1.16
1,2,3,7,8,9-HxCDD	49.055	0.148		1.21	1.009	1.04
1,2,3,4,6,7,8-HpCDD	3557.550	5.598	E	1.05	1.079	0.93
OCDD	35056.413	0.518	E	0.87	1.172	1.00
2,3,7,8-TCDF	1.652	0.114	C	0.75	1.000	0.91
1,2,3,7,8-PeCDF	8.211	1.039		1.62	1.001	0.89
2,3,4,7,8-PeCDF	9.694	1.065		1.60	1.025	0.87
1,2,3,4,7,8-HxCDF	143.957	5.641		1.26	1.000	1.14
1,2,3,6,7,8-HxCDF	32.185	5.244		1.31	1.004	1.22
1,2,3,7,8,9-HxCDF	31.771	7.590		1.26	1.041	0.85
2,3,4,6,7,8-HxCDF	56.259	6.416		1.28	1.017	1.00
1,2,3,4,6,7,8-HpCDF	849.206	3.605	E	1.04	1.000	1.40
1,2,3,4,7,8,9-HpCDF	79.605	5.396		1.04	1.039	0.93
OCDF	3135.163	0.617	E	0.89	1.004	1.14
Total Tetra-Dioxins	1.735	0.130				
Total Penta-Dioxins	38.663	0.133				
Total Hexa-Dioxins	548.324	0.133				
Total Hepta-Dioxins	6976.685	5.598				
Total Tetra-Furans	9.510	0.114				
Total Penta-Furans	229.318	1.065				
Total Hexa-Furans	1909.050	5.244				
Total Hepta-Furans	3557.684	3.605				

(1) Qualifiers: See flag definitions.

(2) RRTs and ion ratios are specified in Tables 11 and 8, Method 8290. 8290F1

PCDD/PCDF TOXICITY EQUIVALENCE (TEQ) SUMMARY
Use for Sample and Blank Results with Values < Detection Limit (DL)

Lab Name: Columbia Analytical Services

Client ID:

H25-2004

Lab Code: CAS

8290

Lab Sample ID:

E2300499-001A

Client Name:

Barr Engineering

Sample Wt/Vol:

3.25g

Matrix (Solid/Aqueous/Waste/Ash/Tissue):

Solid

Initial Calibration Date:

8/5/2003

Sample Receipt Date:

8/8/2003

Instrument ID:

70S

Ext. Date:

8/13/2003

GC Column ID:

db5

Ext. Vol (uL 20.0)

Inj. Vol (uL 1.0)

Analysis Date:

8/18/2003

Sample Filename:

B15550#9

Analysis Time:

17:39:40

Blank Data Filename:

B15550#2

Dilution Factor:

1

Calibration Verification Filename:

B15549#1

Concentration Units (pg/L or ng/Kg dry weight):

ng/Kg

Solids/Lipids, %:

10.13

PARAMETER	Detection Limit (DL)	DL/2	CONCENTRATION	TEF (1)	TEF-ADJUSTED CONCENTRATION
2,3,7,8-TCDD			0.55	1.0	0.55
1,2,3,7,8-PeCDD			7.99	1.0	7.988
1,2,3,4,7,8-HxCDD			24.40	0.1	2.41
1,2,3,6,7,8-HxCDD			107.76	0.1	10.78
1,2,3,7,8,9-HxCDD			49.06	0.1	4.91
1,2,3,4,6,7,8-HpCDD			3557.55	0.01	35.58
OCDD			35056.41	0.0001	3.51
2,3,7,8-TCDF			1.36	0.1	0.14
1,2,3,7,8-PeCDF			8.21	0.05	0.41
2,3,4,7,8-PeCDF			9.69	0.5	4.85
1,2,3,4,7,8-HxCDF			143.96	0.1	14.40
1,2,3,6,7,8-HxCDF			32.19	0.1	3.22
1,2,3,7,8,9-HxCDF			31.77	0.1	3.18
2,3,4,6,7,8-HxCDF			56.26	0.1	5.63
1,2,3,4,6,7,8-HpCDF			849.21	0.01	8.49
1,2,3,4,7,8,9-HpCDF			79.61	0.01	0.80
OCDF			3335.16	0.0001	0.33
				Total TEQ:	107.15

(1) World Health Organization (WHO) adopted Toxicity Equivalence Factors (TEFs) taken from: Van der Berg, et.al; Toxicity Equivalence Factors (TEFs) for PCBs, PCDDs/PCDFs for Humans and Wildlife, Environmental Health Perspective 106:775-792 (1998.)

Note: Non-detected values are reported as one-half the detection limit (DL/2.)

The 2,3,7,8-TCDF value is reported from the confirmed, db-225, column.

Form 1

CLIENT ID

PCDD/PCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

F27-29 0-4

Lab Name: Columbia Analytical Services Episode No.

Lab Code: CAS SDG No..

Method. 8290 Lab Sample ID: E2300499-002A

Client Name: BARR ENGINEERING

Sample Wt/Vol: 11.119 g or mL: g

Matrix (Aqueous/Solid/Ash): Solid

Initial Calibration Date: 08/05/03

Sample Receipt Date: 08/08/03

Instrument ID: 7051

Ext. Date: 08/13/03

GC Column: DB-5

Ext. Vol (ul): 20.0 Inj. Vol (ul): 1.0

Sample Data Filename: B15552#3

Analysis Date: 18-AUG-03 Time: 23:36:23

Blank Data Filename: B15552#2

Dilution Factor: 1

Cal. Ver. Data Filename: B15550#12

Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture/Lipid: 4.27

ANALYTE	CONCENTRATION FOUND	DETECTION LIMIT	Qual. ION ABUND. (1)	RATIO (2)	RRT (2)	MEAN RRF
2,3,7,8-TCDD	2.751	0.263		0.72	1.001	1.00
1,2,3,7,8-PeCDD	68.132	0.506		1.65	1.001	0.97
1,2,3,4,7,8-HxCDD	259.848	0.472		1.23	0.998	1.04
1,2,3,6,7,8-HxCDD	2127.183	0.425	E	1.24	1.000	1.15
1,2,3,7,8,9-HxCDD	635.314	0.432	E	1.24	1.008	1.13
1,2,3,4,6,7,8-HpCDD	40753.677	34.586	E	1.00	1.079	1.00
OCDD	190847.234	4.883	E	0.93	1.172	1.04
2,3,7,8-TCDF	32.439	0.252	C	0.78	1.001	0.92
1,2,3,7,8-PeCDF	110.641	7.007		1.62	1.000	0.94
2,3,4,7,8-PeCDF	143.354	6.841		1.57	1.025	0.96
1,2,3,4,7,8-HxCDF	995.229	17.991	E	1.30	1.000	1.33
1,2,3,6,7,8-HxCDF	232.647	17.614		1.27	1.003	1.36
1,2,3,7,8,9-HxCDF	140.684	24.881		1.37	1.039	0.96
2,3,4,6,7,8-HxCDF	366.171	20.052		1.27	1.017	1.20
1,2,3,4,6,7,8-HpCDF	9833.653	20.803	E	1.05	1.000	1.45
1,2,3,4,7,8,9-HpCDF	864.082	29.243	E	1.05	1.038	1.03
OCDF	59940.553	2.172	E	0.88	1.004	1.35
Total Tetra-Dioxins	15.869	0.263				
Total Penta-Dioxins	278.673	0.506				
Total Hexa-Dioxins	7753.017	0.425				
Total Hepta-Dioxins	75413.649	34.586				
Total Tetra-Furans	143.243	0.252				
Total Penta-Furans	2648.473	6.841				
Total Hexa-Furans	7250.670	17.614				
Total Hepta-Furans	34820.480	20.803				

(1) Qualifiers: See flag definitions.

(2) RRTs and ion ratios are specified in Tables 11 and 8, Method 8290. 8290F1

PCDD/PCDF TOXICITY EQUIVALENCE (TEQ) SUMMARY
Use for Sample and Blank Results with Values < Detection Limit (DL)

Lab Name: Columbia Analytical Services

Client ID:

F2720-4

Lab Code: CAS

8290

Lab Sample ID:

E2300399-002A

Client Name:

Barr Engineering

Sample Wt/Vol:

301.11g

Matrix (Solid/Aqueous/Waste/Ash/Tissue):

Solid

Initial Calibration Date:

8/5/2003

Sample Receipt Date:

8/8/2003

Instrument ID:

70S

Ext. Date:

8/13/2003

GC Column ID:

db5

Ext. Vol (uL 20.0)

Inj. Vol (uL 1.0)

Analysis Date:

8/18/2003

Sample Filename:

B15552#3

Analysis Time:

23:26:23

Blank Data Filename:

B15552#2

Dilution Factor:

1

Calibration Verification Filename:

B15550#12

Concentration Units (pg/L or ng/Kg dry weight):

ng/Kg

Solids/Lipids, %:

4.27

PARAMETER	Detection Limit (DL)	DL/2	CONCENTRATION	TEF (1)	TEF-ADJUSTED CONCENTRATION
2,3,7,8-TCDD			2.75	1.0	2.75
1,2,3,7,8-PeCDD			68.13	1.0	68.132
1,2,3,4,7,8-HxCDD			259.85	0.1	25.98
1,2,3,6,7,8-HxCDD			2127.18	0.1	212.72
1,2,3,7,8,9-HxCDD			635.31	0.1	63.53
1,2,3,4,6,7,8-HpCDD			40753.68	0.01	407.54
OCDD			190847.23	0.0001	19.08
2,3,7,8-TCDF			26.00	0.1	2.60
1,2,3,7,8-PeCDF			110.64	0.05	5.53
2,3,4,7,8-PeCDF			113.35	0.5	56.68
1,2,3,4,7,8-HxCDF			995.23	0.1	99.52
1,2,3,6,7,8-HxCDF			232.65	0.1	23.26
1,2,3,7,8,9-HxCDF			40.68	0.1	4.07
2,3,4,6,7,8-HxCDF			366.17	0.1	36.62
1,2,3,4,6,7,8-HpCDF			9833.65	0.01	98.34
1,2,3,4,7,8,9-HpCDF			864.08	0.01	8.64
OCDF			59940.55	0.0001	5.99
				Total TEQ:	1140.99

(1) World Health Organization (WHO) adopted Toxicity Equivalence Factors (TEFs) taken from: Van den Berg, et.al; Toxicity Equivalence Factors (TEFs) for PCBs, PCDDs/PCDFs for Humans and Wildlife, Environmental Health Perspective 106:775-792 (1998.)

Note: Non-detected values are reported as one-half the detection limit (DL/2.)

The 2,3,7,8-TCDF value is reported from the confirmed, db-225, column.

Form 1

CLIENT ID

PCDD/PCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

F27-29 4-12

Lab Name: Columbia Analytical Services Episode No.:

Lab Code: CAS SDG No.:

Method: 8290 Lab Sample ID: E2300499-003A

Client Name: BARR ENGINEERING

Sample Wt/Vol: 10.669 g or mL: g

Matrix (Aqueous/Solid/Ash): Solid

Initial Calibration Date: 08/05/03

Sample Receipt Date: 08/08/03

Instrument ID: 70S

Ext. Date: 08/13/03

GC Column: DB-5

Ext. Vol (ul): 20.0 Inj. Vol (ul): 1.0

Sample Data Filename: B15552#7

Analysis Date: 19-AUG-03 Time: 03:00:08

Blank Data Filename: B15552#2

Dilution Factor: 1

Cal. Ver. Data Filename: B15550#12

Concentration Units (pg/L or ng/Kg dry weight) ng/Kg % Moisture/Lipid: 14.20

ANALYTE	CONCENTRATION FOUND	DETECTION LIMIT	Qual. (1)	ION ABUND. RATIO (2)	RRT (2)	MEAN RRF
2,3,7,8-TCDD	1.621	0.263		0.70	1.001	1.00
1,2,3,7,8-PeCDD	31.990	0.728		1.57	1.001	0.97
1,2,3,4,7,8-HxCDD	123.327	0.962		1.21	0.998	1.04
1,2,3,6,7,8-HxCDD	752.528	0.867	E	1.26	1.000	1.15
1,2,3,7,8,9-HxCDD	242.369	0.881		1.26	1.009	1.13
1,2,3,4,6,7,8-HpCDD	25142.433	21.015	E	1.02	1.079	1.00
OCDD	157051.836	3.082	E	0.93	1.172	1.04
2,3,7,8-TCDF	7.652	0.300	C	0.80	1.001	0.92
1,2,3,7,8-PeCDF	63.597	6.061		1.57	1.000	0.94
2,3,4,7,8-PeCDF	4.835	5.917		1.73	1.025	0.96
1,2,3,4,7,8-HxCDF	453.990	24.951		1.29	1.000	1.33
1,2,3,6,7,8-HxCDF	123.298	24.430		1.30	1.003	1.36
1,2,3,7,8,9-HxCDF	*	34.507	U	*	*	0.96
2,3,4,6,7,8-HxCDF	229.307	27.810		1.28	1.016	1.20
1,2,3,4,6,7,8-HpCDF	6308.981	19.986	E	1.06	1.000	1.45
1,2,3,4,7,8,9-HpCDF	447.453	28.095		1.15	1.038	1.03
OCDF	36377.517	5.866	E	0.89	1.004	1.35
Total Tetra-Dioxins	4.306	0.263				
Total Penta-Dioxins	111.481	0.728				
Total Hexa-Dioxins	2746.821	0.867				
Total Hepta-Dioxins	46495.754	21.015				
Total Tetra-Furans	51.629	0.300				
Total Penta-Furans	1685.717	5.917				
Total Hexa-Furans	4151.581	24.430				
Total Hepta-Furans	30589.640	19.986				

(1) Qualifiers: See flag definitions.

(2) RRTs and ion ratios are specified in Tables 11 and 8, Method 8290. 8290F1

Form 3

PCDD/PCDF TOXICITY EQUIVALENCE (TEQ) SUMMARY
Use for Sample and Blank Results with Values < Detection Limit (DL)

Lab Name Columbia Analytical Services

Client ID
F27029412

Lab Code CAS 8290
Client Name Barr Engineering

Lab Sample ID E2300499-003A
Sample Wt/Vol 10.669

Matrix (Solid/Aqueous/Waste/Ash/Tissue) Solid
Sample Receipt Date 8/8/2003
Ext Date 8/13/2003
Ext. Vol (uL 20 0 Inj Vol (uL 1 0
Analysis Date 8/19/2003
Analysis Time 3:00:08

Initial Calibration Date: 8/5/2003
Instrument ID 70S
GC Column ID db5

Dilution Factor 1

Sample Filename B15552#7
Blank Data Filename B15552#2
Calibration Verification Filename B15550#12

Concentration Units (pg/L or ng/Kg dry weight) ng/Kg Solids/Lipids, % 14.20

PARAMETER	Detection Limit (DL)	DL/2	CONCENTRATION	TEF (1)	TEF-ADJUSTED CONCENTRATION
2,3,7,8-TCDD			1.62	1.0	1.62
1,2,3,7,8-PeCDD			31.99	1.0	31.99
1,2,3,4,7,8-HxCDD			123.33	0.1	12.33
1,2,3,6,7,8-HxCDD			75.25	0.1	7.525
1,2,3,7,8,9-HxCDD			24.24	0.1	2.424
1,2,3,4,6,7,8-HpCDD			251.42	0.01	2.5142
OCDD			157051.84	0.0001	15.71
2,3,7,8-TCDF			0.56	0.1	0.056
1,2,3,7,8-PeCDF			63.60	0.05	3.18
2,3,4,7,8-PeCDF			64.84	0.5	32.42
1,2,3,4,7,8-HxCDF			459.99	0.1	46.00
1,2,3,6,7,8-HxCDF			128.30	0.1	12.83
1,2,3,7,8,9-HxCDF	34.507	17.254	17.25	0.1	1.73
2,3,4,6,7,8-HxCDF			229.31	0.1	22.93
1,2,3,4,6,7,8-HpCDF			6308.98	0.01	63.09
1,2,3,4,7,8,9-HpCDF			447.45	0.01	4.47
OCDF			36377.52	0.0001	3.64
				Total TEQ:	603.41

(1) World Health Organization (WHO) adopted Toxicity Equivalence Factors (TEFs) taken from Van der Berg, et al, Toxicity Equivalence Factors (TEFs) for PCBs, PCDDs/PCDFs for Humans and Wildlife, Environmental Health Perspective 106 775-792 (1998)

Note Non-detected values are reported as one-half the detection limit (DL/2)

The 2,3,7,8-TCDF value is reported from the confirmed, db-225, column

Form 1

PCDD/PCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

CLIENT ID

J29-30 0-4

Lab Name: Columbia Analytical Services Episode No..

Lab Code: CAS SDG No.: Method: 8290 Lab Sample ID: E2300499-004A

Client Name: BARR ENGINEERING Sample Wt/Vol: 17.682 g or mL: g

Matrix (Aqueous/Solid/Ash): Solid Initial Calibration Date: 08/05/03

Sample Receipt Date: 08/08/03 Instrument ID: 709

Ext. Date: 08/13/03 GC Column: DB-5

Ext. Vol (ul): 20.0 Inj. Vol (ul): 1.0 Sample Data Filename: B15552#4

Analysis Date: 19-AUG-03 Time: 00:27:21 Blank Data Filename: B15552#2

Dilution Factor: 1 Cal. Ver. Data Filename: B15550#12

Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture/Lipid: 27.75

ANALYTE	CONCENTRATION FOUND	DETECTION LIMIT	Qual. ION (1)	ABUND. RATIO (2)	RRT (2)	MEAN RRF
2,3,7,8-TCDD	1.696	0.554	K	0.92	1.001	1.00
1,2,3,7,8-PeCDD	46.175	0.752		1.55	1.000	0.97
1,2,3,4,7,8-HxCDD	166.244	0.929		1.21	0.998	1.04
1,2,3,6,7,8-HxCDD	1593.983	0.838	E	1.23	1.000	1.15
1,2,3,7,8,9-HxCDD	388.579	0.851		1.23	1.008	1.13
1,2,3,4,6,7,8-HpCDD	44776.835	25.109	E	1.02	1.079	1.00
OCDD	203283.378	3.486	E	1.00	1.173	1.04
2,3,7,8-TCDF	16.925	0.412	C	0.81	1.001	0.92
1,2,3,7,8-PeCDF	423.408	3.877		1.52	1.001	0.94
2,3,4,7,8-PeCDF	140.406	3.785		1.56	1.025	0.96
1,2,3,4,7,8-HxCDF	1372.929	28.853	E	1.28	1.000	1.33
1,2,3,6,7,8-HxCDF	261.935	28.249		1.32	1.003	1.36
1,2,3,7,8,9-HxCDF	63.38	39.902		1.08	1.039	0.96
2,3,4,6,7,8-HxCDF	459.973	32.158	E	1.27	1.017	1.20
1,2,3,4,6,7,8-HpCDF	13422.956	25.148	E	1.06	1.000	1.45
1,2,3,4,7,8,9-HpCDF	1181.881	35.352	E	1.06	1.038	1.03
OCDF	70629.737	2.641	E	0.88	1.004	1.35

Total Tetra-Dioxins 9.900 0.554
 Total Penta-Dioxins 182.718 0.752
 Total Hexa-Dioxins 5044.637 0.838
 Total Hepta-Dioxins 82564.666 25.109
 Total Tetra-Furans 116.211 0.412
 Total Penta-Furans 2490.251 3.785
 Total Hexa-Furans 8468.187 28.249
 Total Hepta-Furans 53717.586 25.148

(1) Qualifiers: See flag definitions.

(2) RRTs and ion ratios are specified in Tables 11 and 8, Method 8290. 8290F1

PCDD/PCDF TOXICITY EQUIVALENCE (TEQ) SUMMARY
 Use for Sample and Blank Results with Values < Detection Limit (DL)

Lab Name: Columbia Analytical Services

Client ID
 J29-310-4

Lab Code: CAS 8290
 Client Name: Barr Engineering

Lab Sample ID: E2300499-004A
 Sample Wt/Vol: 7.68g

Matrix (Solid/Aqueous/Waste/Ash/Tissue): Solid
 Sample Receipt Date: 8/8/2003
 Ext Date: 8/13/2003
 Ext Vol (uL 20 0): Inj Vol (uL 1 0)
 Analysis Date: 8/19/2003
 Analysis Time: 0:27:21

Initial Calibration Date: 8/5/2003
 Instrument ID: 70S
 GC Column ID: db5

Sample Filename: B15552#4
 Blank Data Filename: B15552#2

Dilution Factor: 1

Calibration Verification Filename: B15550#12

Concentration Units (pg/L or ng/Kg dry weight): ng/Kg
 Solids/Lipids, %: 27.75

PARAMETER	Detection Limit (DL)	DL/2	CONCENTRATION	TEF (1)	TEF-ADJUSTED CONCENTRATION
2,3,7,8-TCDD			1.70	1.0	1.70
1,2,3,7,8-PeCDD			46.18	1.0	46.175
1,2,3,4,7,8-HxCDD			166.24	0.1	16.62
1,2,3,6,7,8-HxCDD			1593.98	0.1	159.40
1,2,3,7,8,9-HxCDD			38.58	0.1	3.86
1,2,3,4,6,7,8-HpCDD			4476.84	0.01	44.77
OCDD			203283.38	0.0001	20.33
2,3,7,8-TCDF			13.76	0.1	1.38
1,2,3,7,8-PeCDF			123.41	0.05	6.17
2,3,4,7,8-PeCDF			149.41	0.5	70.20
1,2,3,4,7,8-HxCDF			1372.93	0.1	137.29
1,2,3,6,7,8-HxCDF			261.94	0.1	26.19
1,2,3,7,8,9-HxCDF			63.29	0.1	6.33
2,3,4,6,7,8-HxCDF			459.97	0.1	46.00
1,2,3,4,6,7,8-HpCDF			13422.96	0.01	134.23
1,2,3,4,7,8,9-HpCDF			1181.88	0.01	11.82
OCDF			70629.74	0.0001	7.06
Total TEQ:					1177.52

(1) World Health Organization (WHO) adopted Toxicity Equivalence Factors (TEFs) taken from Van der Berg, et al, Toxicity Equivalence Factors (TEFs) for PCBs, PCDDs/PCDFs for Humans and Wildlife, Environmental Health Perspective 106 775-792 (1998)

Note Non-detected values are reported as one-half the detection limit (DL/2)

The 2,3,7,8-TCDF value is reported from the confirmed, db-225, column

Form 1

CLIENT ID.

PCDD/PCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

I27-29 0-4

Lab Name: Columbia Analytical Services Episode No.:

Lab Code: CAS SDG No.:

Method: 8290 Lab Sample ID: E2300499-005A

Client Name: BARR ENGINEERING

Sample Wt/Vol: 11.316 g or mL: g

Matrix (Aqueous/Solid/Ash): Solid

Initial Calibration Date: 08/05/03

Sample Receipt Date: 08/08/03

Instrument ID: 70S

Ext. Date: 08/13/03

GC Column:DB-5

Ext. Vol(ul):20.0 Inj. Vol(ul):1.0

Sample Data Filename: B15552#5

Analysis Date: 19-AUG-03 Time: 01:18:16

Blank Data Filename: B15552#2

Dilution Factor: 1

Cal. Ver. Data Filename: B15550#12

Concentration Units (pg/L or ng/Kg dry weight) ng/Kg % Moisture/Lipid: 15.06

ANALYTE	CONCENTRATION FOUND	DETECTION LIMIT	Qual. (1)	ION ABUND. RATIO (2)	RRT (2)	MEAN RRF
2,3,7,8-TCDD	3.564	0.569		0.81	1.001	1.00
1,2,3,7,8-PeCDD	54.371	0.838		1.59	1.000	0.97
1,2,3,4,7,8-HxCDD	178.610	0.970		1.24	0.998	1.04
1,2,3,6,7,8-HxCDD	1058.343	0.874	E	1.26	1.000	1.15
1,2,3,7,8,9-HxCDD	354.764	0.888		1.26	1.009	1.13
1,2,3,4,6,7,8-HpCDD	31453.667	24.837	E	1.03	1.079	1.00
OCDD	189089.520	4.704	E	0.93	1.173	1.04
2,3,7,8-TCDF	11.527	0.321	C	0.80	1.001	0.92
1,2,3,7,8-PeCDF	95.503	3.212		1.63	1.000	0.94
2,3,4,7,8-PeCDF	56.931	3.136		1.60	1.025	0.96
1,2,3,4,7,8-HxCDF	766.538	31.784	E	1.32	1.000	1.33
1,2,3,6,7,8-HxCDF	219.845	31.119		1.29	1.003	1.36
1,2,3,7,8,9-HxCDF	41.879	43.956		1.24	1.039	0.96
2,3,4,6,7,8-HxCDF	351.808	35.425		1.28	1.017	1.20
1,2,3,4,6,7,8-HpCDF	7918.921	24.316	E	1.05	1.000	1.45
1,2,3,4,7,8,9-HpCDF	639.858	34.182	E	1.06	1.038	1.03
OCDF	663.478	2.516	E	0.89	1.004	1.35
Total Tetra-Dioxins	13.017	0.569				
Total Penta-Dioxins	209.519	0.838				
Total Hexa-Dioxins	4036.637	0.874				
Total Hepta-Dioxins	57097.080	24.837				
Total Tetra-Furans	80.749	0.321				
Total Penta-Furans	1586.209	3.136				
Total Hexa-Furans	6518.802	31.119				
Total Hepta-Furans	36831.749	24.316				

(1) Qualifiers: See flag definitions.

(2) RRTs and ion ratios are specified in Tables 11 and 8, Method 8290. 8290F1

PCDD/PCDF TOXICITY EQUIVALENCE (TEQ) SUMMARY
Use for Sample and Blank Results with Values < Detection Limit (DL)

Lab Name Columbia Analytical Services

Client ID

129-220-4

Lab Code CAS 8290

Lab Sample ID

E2300199-005A1

Client Name Barr Engineering

Sample Wt/Vol

31.316

Matrix (Solid/Aqueous/Waste/Ash/Tissue) Solid

Initial Calibration Date 8/5/2003

Sample Receipt Date 8/8/2003

Instrument ID 70S

Ext Date 8/13/2003

GC Column ID db5

Ext. Vol (uL 20 0 Inj Vol (uL 1 0

Analysis Date 8/19/2003

Sample Filename B15552#5

Analysis Time 1:18:16

Blank Data Filename B15552#2

Dilution Factor 1

Calibration Verification Filename B15550#12

Concentration Units (pg/L or ng/Kg dry weight) ng/Kg Solids/Lipids, % 15.06

PARAMETER	Detection Limit (DL)	DL/2	CONCENTRATION	TEF (1)	TEF-ADJUSTED CONCENTRATION
2,3,7,8-TCDD			5.56	1.0	3.56
1,2,3,7,8-PeCDD			54.37	1.0	54.371
1,2,3,4,7,8-HxCDD			178.61	0.1	17.86
1,2,3,6,7,8-HxCDD			105.834	0.1	105.83
1,2,3,7,8,9-HxCDD			35.48	0.1	35.48
1,2,3,4,6,7,8-HpCDD			314.54	0.01	314.54
OCDD			189089.52	0.0001	18.91
2,3,7,8-TCDF			8.93	0.1	0.89
1,2,3,7,8-PeCDF			95.50	0.05	4.78
2,3,4,7,8-PeCDF			96.93	0.5	48.47
1,2,3,4,7,8-HxCDF			76.654	0.1	76.65
1,2,3,6,7,8-HxCDF			21.985	0.1	21.98
1,2,3,7,8,9-HxCDF			41.88	0.1	4.19
2,3,4,6,7,8-HxCDF			351.81	0.1	35.18
1,2,3,4,6,7,8-HpCDF			7918.92	0.01	79.19
1,2,3,4,7,8,9-HpCDF			639.86	0.01	6.40
OCDF			43463.48	0.0001	4.35
				Total TEQ:	832.63

(1) World Health Organization (WHO) adopted Toxicity Equivalence Factors (TEFs) taken from Van der Berg, et al, Toxicity Equivalence Factors (TEFs) for PCBs, PCDDs/PCDFs for Humans and Wildlife, Environmental Health Perspective 106 775-792 (1998)

Note Non-detected values are reported as one-half the detection limit (DL/2)

The 2,3,7,8-TCDF value is reported from the confirmed, db-225, column.

Form 1

CLIENT ID.

PCDD/PCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

I26-27 0-4

Lab Name: Columbia Analytical Services Episode No.:

Lab Code: CAS SDG No.:

Method: 8290 Lab Sample ID: E2300499-006A

Client Name: BARR ENGINEERING

Sample Wt/Vol: 10.774 g or mL: g

Matrix (Aqueous/Solid/Ash): Solid

Initial Calibration Date: 08/05/03

Sample Receipt Date: 08/08/03

Instrument ID: 705

Ext. Date: 08/13/03

GC Column: DB-5

Ext. Vol (ul): 20.0 Inj. Vol (ul): 1.0

Sample Data Filename: B15552#6

Analysis Date: 19-AUG-03 Time: 02:09:13

Blank Data Filename: B15552#2

Dilution Factor: 1

Cal. Ver. Data Filename: B15550#12

Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture/Lipid: 18.39

ANALYTE	CONCENTRATION FOUND	DETECTION LIMIT	Qual. (1)	ION ABUND. RATIO (2)	RRT (2)	MEAN RRF
2,3,7,8-TCDD	*	0.459	U	*	*	1.00
1,2,3,7,8-PeCDD	9.682	0.644		1.66	1.001	0.97
1,2,3,4,7,8-HxCDD	33.454	0.673		1.42	0.998	1.04
1,2,3,6,7,8-HxCDD	186.024	0.607		1.23	1.000	1.15
1,2,3,7,8,9-HxCDD	60.261	0.617		1.26	1.009	1.13
1,2,3,4,6,7,8-HpCDD	5493.547	6.145	E	1.03	1.078	1.00
OCDD	49994.599	2.166	E	0.87	1.171	1.04
2,3,7,8-TCDF	2.286	0.241	C	0.76	1.000	0.92
1,2,3,7,8-PeCDF	19.506	2.024		1.63	1.001	0.94
2,3,4,7,8-PeCDF	17.275	1.976		1.73	1.025	0.96
1,2,3,4,7,8-HxCDF	152.673	10.443		1.28	1.000	1.33
1,2,3,6,7,8-HxCDF	412.100	10.225		1.30	1.003	1.36
1,2,3,7,8,9-HxCDF	*	14.443	U	*	*	0.96
2,3,4,6,7,8-HxCDF	62.760	11.640		1.25	1.017	1.20
1,2,3,4,6,7,8-HpCDF	1239.497	4.582	E	1.05	1.000	1.45
1,2,3,4,7,8,9-HpCDF	100.831	6.441		1.08	1.038	1.03
OCDF	4542.115	6.317	E	0.89	1.004	1.35
Total Tetra-Dioxins	*	0.459	U			
Total Penta-Dioxins	41.510	0.644				
Total Hexa-Dioxins	732.167	0.607				
Total Hepta-Dioxins	10318.979	6.145				
Total Tetra-Furans	9.937	0.241				
Total Penta-Furans	412.219	1.976				
Total Hexa-Furans	2430.140	10.225				
Total Hepta-Furans	5425.383	4.582				

(1) Qualifiers: See flag definitions.

(2) RRTs and ion ratios are specified in Tables 11 and 8, Method 8290. 8290F1

PCDD/PCDF TOXICITY EQUIVALENCE (TEQ) SUMMARY
Use for Sample and Blank Results with Values < Detection Limit (DL)

Lab Name Columbia Analytical Services

Client ID

126-270-4

Lab Code CAS 8290

Lab Sample ID

E2300499-006A

Client Name Barr Engineering

Sample Wt/Vol

0.77g

Matrix (Solid/Aqueous/Waste/Ash/Tissue) Solid

Initial Calibration Date: 8/5/2003

Sample Receipt Date 8/8/2003

Instrument ID 70S

Ext Date 8/13/2003

GC Column ID db5

Ext Vol (uL 20 0 Inj Vol (uL 1 0

Analysis Date 8/19/2003

Sample Filename B15552#6

Analysis Time 2:09:13

Blank Data Filename B15552#2

Dilution Factor 1

Calibration Verification Filename B15550#12

Concentration Units (pg/L or ng/Kg dry weight) ng/Kg

Solids/Lipids, % 18.39

PARAMETER	Detection Limit (DL)	DL/2	CONCENTRATION	TEF (1)	TEF-ADJUSTED CONCENTRATION
2,3,7,8-TCDD	0.459	0.230	0.23	1.0	0.23
1,2,3,7,8-PeCDD			9.68	1.0	9.68
1,2,3,4,7,8-HxCDD			33.45	0.1	3.35
1,2,3,6,7,8-HxCDD			186.02	0.1	18.60
1,2,3,7,8,9-HxCDD			60.26	0.1	6.03
1,2,3,4,6,7,8-HpCDD			5493.55	0.01	54.94
OCDD			49994.90	0.0001	5.00
2,3,7,8-TCDF			1.91	0.1	0.19
1,2,3,7,8-PeCDF			19.51	0.05	0.98
2,3,4,7,8-PeCDF			17.28	0.5	8.64
1,2,3,4,7,8-HxCDF			157.67	0.1	15.77
1,2,3,6,7,8-HxCDF			41.10	0.1	4.11
1,2,3,7,8,9-HxCDF	14.443	7.222	7.22	0.1	0.72
2,3,4,6,7,8-HxCDF			62.76	0.1	6.28
1,2,3,4,6,7,8-HpCDF			1239.50	0.01	12.39
1,2,3,4,7,8,9-HpCDF			100.83	0.01	1.01
OCDF			4542.12	0.0001	0.45
				Total TEQ:	148.36

(1) World Health Organization (WHO) adopted Toxicity Equivalence Factors (TEFs) taken from: Van der Berg, et al, Toxicity Equivalence Factors (TEFs) for PCBs, PCDDs/PCDFs for Humans and Wildlife, Environmental Health Perspective 106 775-792 (1998)

Note: Non-detected values are reported as one-half the detection limit (DL/2)

The 2,3,7,8-TCDF value is reported from the confirmed, db-225, column

Form 3

CLIENT ID.

PCDD/PCDF TOXICITY EQUIVALENCE SUMMARY

Use for Sample and Blank Results

I26-27 0-4

Lab Name: Columbia Analytical Services Episode No.:

Lab Code: CAS Method: 8290 Lab Sample ID: E2300499-006A

Client Name: BARR ENGINEERING Sample Wt/Vol: 10.774 g or mL: g

Matrix (aqueous/solid/leachate): Solid Initial Calibration Date: 08/05/03

Sample Receipt Date: 08/08/03 Instrument ID: 70S

Ext. Date: 08/13/03 GC Column ID: DB-5

Ext. Vol(ul):20.0 Inj. Vol(ul):1.0 Sample Data Filename: B15552#6

Analysis Date: 19-AUG-03 Time: 02:09:13 Blank Data Filename: B15552#2

Dilution Factor: 1 Cal. Ver. Data Filename: B15550#12

Concentration Units (pg/L or ng/Kg dry weight) ng/Kg Moisture: 18.39

	CONCENTRATION	TEF (1)	TEF-ADJUSTED CONCENTRATION
2,3,7,8-TCDD	*	X 1.0	*
1,2,3,7,8-PeCDD	9.68	X 0.5	4.84e+00
1,2,3,4,7,8-HxCDD	33.45	X 0.1	3.35e+00
1,2,3,6,7,8-HxCDD	186.02	X 0.1	1.86e+01
1,2,3,7,8,9-HxCDD	60.26	X 0.1	6.03e+00
1,2,3,4,6,7,8-HpCDD	5493.55	X 0.01	5.49e+01
OCDD	49994.90	X 0.001	5.00e+01
2,3,7,8-TCDF	2.21	X 0.1	2.21e-01
1,2,3,7,8-PeCDF	19.54	X 0.05	9.75e-01
2,3,4,7,8-PeCDF	17.27	X 0.5	8.64e+00
1,2,3,4,7,8-HxCDF	157.67	X 0.1	1.58e+01
1,2,3,6,7,8-HxCDF	41.10	X 0.1	4.11e+00
1,2,3,7,8,9-HxCDF	*	X 0.1	*
2,3,4,6,7,8-HxCDF	62.76	X 0.1	6.28e+00
1,2,3,4,6,7,8-HpCDF	1239.50	X 0.01	1.24e+01
1,2,3,4,7,8,9-HpCDF	100.83	X 0.01	1.01e+00
OCDF	4542.11	X 0.001	4.54e+00

Total: 1.917e+02

(1) Taken from Interim Procedures for Estimating Risks Associated with Exposure to Mixtures of Chlorinated Dibenzo-p-Dioxin and -Dibenzofurans (CDDs and CDFs) and 1989 Update (EPA/625/3-89/016, March 1989.)



Confirmation Data

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CAS, INC.
2378-TCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

CLIENT ID.
H25-26-0-4

Lab Name: Columbia Analytical Services Episode No.:

Client Name: BARR ENGINEERING Lab Sample ID: E2300499-001A

Matrix (aqueous/solid/leachate): Solid Sample Wt/Vol: 13.255g or mL: g

Sample Receipt Date: 08/08/03 Initial Calibration Date: 08/06/03

Ext. Date: 08/13/03 Instrument ID: 70S

Analysis Date: 18-AUG-03 Time: 18:40:14 GC Column ID: DB-225

Ext. Vol(ul): 20.0 Sample Data Filename: A19525#9

Inj. Vol(ul): 1.0 Blank Data Filename: A19525#3

Dilution Factor: 1 Cal Ver. Data Filename: A19525#2

Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture: 10.13

ANALYTE	CONCENTRATION FOUND(1)	DETECTION LIMIT	ION ABUND. RATIO	RRT	TEF
2,3,7,8-TCDF	1.357	0.509	0.86	1.001	0.136

INT. STANDARD	SPIKE CONCENT. (pg)	CONCENT. FOUND (pg)	RECOV. %	ION ABUND. RATIO	RRT
13C-2,3,7,8-TCDF	1000	593.20	59.32	0.78	1.06
CLEANUP STANDARD					
37C1-2,3,7,8-TCDD	800	670.68	83.83		0.99

(1) '**' indicates non-detected.

UNRECORDED

CAS, INC.
 2378-TCDF ANALYSIS DATA SHEET
 Use for Sample and Blank Results

CLIENT ID:
 F27-290-4

Lab Name: Columbia Analytical Services Episode No.:

Client Name: BARR ENGINEERING Lab Sample ID: E2300499-002A

Matrix (aqueous/solid/leachate): Solid Sample Wt/Vol. 11.119g or mL: g

Sample Receipt Date: 08/08/03 Initial Calibration Date: 08/06/03

Ext. Date: 08/13/03 Instrument ID: 70S

Analysis Date: 20-AUG-03 Time: 12:47:54 GC Column ID: DB-225

Ext. Vol(ul): 20.0 Sample Data Filename: A19528#4

Inj. Vol(ul): 1.0 Blank Data Filename: A19528#3

Dilution Factor: 1 Cal. Ver. Data Filename: A19528#2

Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture: 95.73

ANALYTE	CONCENTRATION FOUND (1)	DETECTION LIMIT	ION ABUND. RATIO	RRT	TEF
2,3,7,8-TCDF	26.003	1.417	0.76	1.001	2.600

INT. STANDARD	SPIKE CONCENT. (pg)	CONCENT. FOUND (pg)	RECOV. %	ION ABUND. RATIO	RRT
13C-2,3,7,8-TCDF	1000	347.92	34.79 Y	0.78	1.06
CLEANUP STANDARD					
37Cl-2,3,7,8-TCDD	800	512.46	64.06		0.99

(1) '*' indicates non-detected

CAS, INC
 2378-TCDF ANALYSIS DATA SHEET
 Use for Sample and Blank Results

CLIENT ID
 F27-29 4-12

Lab Name: Columbia Analytical Services Episode No..
 Client Name: BARR ENGINEERING Lab Sample ID: E2300499-003A
 Matrix (aqueous/solid/leachate): Solid Sample Wt/Vol: 10.669g or mL: g
 Sample Receipt Date: 08/08/03 Initial Calibration Date: 08/06/03
 Ext. Date: 08/13/03 Instrument ID: 70S
 Analysis Date: 20-AUG-03 Time: 13.22:50 GC Column ID: DB-225
 Ext. Vol(ul): 20.0 Sample Data Filename: A19528#5
 Inj. Vol(ul): 1.0 Blank Data Filename: A19528#3
 Dilution Factor: 1 Cal. Ver. Data Filename: A19528#2
 Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture: 85.80

ANALYTE	CONCENTRATION FOUND(1)	DETECTION LIMIT	ION ABUND. RATIO	RRT	TEF
2,3,7,8-TCDF	5.596	1.240	0.70	1.001	0.560

INT. STANDARD	SPIKE CONCENT. (pg)	CONCENT. FOUND (pg)	RECOV. %	ION ABUND. RATIO	RRT
13C-2,3,7,8-TCDF	1000	483.46	48.35	0.78	1.06
CLEANUP STANDARD					
37C1-2,3,7,8-TCDD	800	641.89	80.24		0.99

(1) '**' indicates non-detected.

USEPA, ITD

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CAS, INC.
 2378-TCDF ANALYSIS DATA SHEET
 Use for Sample and Blank Results

CLIENT ID: J29-30-0-4

Lab Name: Columbia Analytical Services Episode No.:
 Client Name: BARR ENGINEERING Lab Sample ID: E2300499-004A
 Matrix (aqueous/solid/leachate): Solid Sample Wt/Vol: 17.682g or mL: g
 Sample Receipt Date: 08/08/03 Initial Calibration Date: 08/06/03
 Ext. Date: 08/13/03 Instrument ID: 70S
 Analysis Date: 20-AUG-03 Time: 13:57:45 GC Column ID: DB-225
 Ext. Vol(ul): 20.0 Sample Data Filename: A19528#6
 Inj. Vol(ul): 1.0 Blank Data Filename: A19528#3
 Dilution Factor: 1 Cal. Ver. Data Filename: A19528#2
 Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture: 72.25

ANALYTE	CONCENTRATION FOUND(1)	DETECTION LIMIT	ION ABUND. RATIO	RRT	TEF
2,3,7,8-TCDF	13.764	1.356	0.75	1.002	1.376

INT. STANDARD	SPIKE CONCENT. (pg)	CONCENT. FOUND (pg)	RECOV. %	ION ABUND. RATIO	RRT
13C-2,3,7,8-TCDF	1000	218.96	21.90 Y	0.79	1.06
CLEANUP STANDARD					
37C1-2,3,7,8-TCDD	800	360.72	45.09		0.99

(1) '**' indicates non-detected.

USEPA, ITD

CAS, INC.
2378-TCDF ANALYSIS DATA SHEET
Use for Sample and Blank Results

CLIENT ID
I27-2970-4

Lab Name: Columbia Analytical Services Episode No.:

Client Name: BARR ENGINEERING Lab Sample ID: E2300499-005A

Matrix (aqueous/solid/leachate): Solid Sample Wt/Vol: 11.316g or mL: g

Sample Receipt Date: 08/08/03 Initial Calibration Date: 08/06/03

Ext. Date: 08/13/03 Instrument ID: 70S

Analysis Date: 20-AUG-03 Time: 14:32:40 GC Column ID: DB-225

Ext. Vol(ul): 20.0 Sample Data Filename: A19528#7

Inj. Vol(ul): 1.0 Blank Data Filename: A19528#3

Dilution Factor: 1 Cal. Ver. Data Filename: A19528#2

Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture: 84.94

ANALYTE	CONCENTRATION FOUND(1)	DETECTION LIMIT	ION ABUND. RATIO	RRT	TEF
2,3,7,8-TCDF	8.926	1.930	0.70	1.001	0.893

INT. STANDARD	SPIKE CONCENT. (pg)	CONCENT. FOUND (pg)	RECOV. %	ION ABUND. RATIO	RRT
13C-2,3,7,8-TCDF	1000	334.76	33.48 Y	0.78	1.06
CLEANUP STANDARD					
37C1-2,3,7,8-TCDD	800	621.01	77.63		0.99

(1) '**' indicates non-detected.

UNAVAILABLE

USEPA, ITD

CAS, INC.
 2378-TCDF ANALYSIS DATA SHEET
 Use for Sample and Blank Results

CLIENT ID
 I26-27-0-4

Lab Name: Columbia Analytical Services Episode No.:
 Client Name: BARR ENGINEERING Lab Sample ID: E2300499-006A
 Matrix (aqueous/solid/leachate): Solid Sample Wt/Vol: 10.374g or ml/g
 Sample Receipt Date: 08/08/03 Initial Calibration Date: 08/06/03
 Ext Date: 08/13/03 Instrument ID: 70S
 Analysis Date: 20-AUG-03 Time: 15:07:35 GC Column ID: DB-225
 Ext. Vol(ul): 20.0 Sample Data Filename: A19528#8
 Inj. Vol(ul): 1.0 Blank Data Filename: A19528#3
 Dilution Factor: 1 Cal. Ver. Data Filename: A19528#2
 Concentration Units (pg/L or ng/Kg dry weight): ng/Kg % Moisture: 81.61

ANALYTE	CONCENTRATION FOUND(1)	DETECTION LIMIT	ION ABUND. RATIO	RRT	TEF
2,3,7,8-TCDF	1.909	0.813	0.75	1.001	0.191

INT. STANDARD	SPIKE CONCENT. (pg)	CONCENT. FOUND (pg)	RECOV. %	ION ABUND. RATIO	RRT
13C-2,3,7,8-TCDF	1000	592.47	59.25	0.78	1.06
CLEANUP STANDARD					
37Cl-2,3,7,8-TCDD	800	695.91	86.99		0.99

(1) '*' indicates non-detected.

BARR

Chain of Custody

4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 837-1000

FD-20517

Project Number

23/11 005257 310

No 16027

Sample Identification	Collection		Matrix			Type		IOC
	Date	Time	Water	Soil	Other	Grab	Comp.	
1.H25-26 0-4	8-5-03	1740		X				
2.F27-29 0-4	8-0-3	1620						
3.F27-29 4-12		1620						
4.J29-30 0-4		840						
5.F27-29 0-4		1210						
6.F26-27 0-4	V	1445		V				
7.								
8.								
9.								
10.								
11.								
12.								
13.								
14.								
15.								
16.								

Number of Containers/Preservative														Total No. Of Containers				
Volatile Organic (Unpres.)	Volatile Organic (Pres.)	Semivolatile Organic	Total Metals (HNO ₃)	Dissolved Metals (HNO ₃)	General (Unpreserved)	Cyanide (NaOH, Asc. Acid)	Nutrients (H ₂ SO ₄)	Oil and Grease (H ₂ SO ₄)	TOC (H ₂ SO ₄)	Sulfide (Zn Acetate)	Dioxin	Whirlpak	Total Phenol (H ₂ SO ₄)		Methane	(HCL)/DRO, 1L Glass	Lugols, Glass, Amber	Formalin, Glass
																		402
																		2
																		2

Project Manager:
TDM

Project Contact:
M. Nelson

Laboratory:
CAS

Remarks/
Analysis Required:

PAH, PCB, PCDD/F

↓ ↓ ↓

PCDD/F

↓

Sampled By: EPG, MTA

Remarks:

Relinquished By: [Signature]

Relinquished By:

Samples Shipped VIA Air Freight Fed. Exp Sampler Other

Date 8/7/03 Time 9:00

Date Time

Received by: [Signature]

Received by:

Air Bill Number:

Date 8/4/03 Time 10:00

Date Time

H R L G I S T D F O R M S C H A I N C S T C D R



Barr Engineering Company
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Phone 952-832-2600 • Fax 952-832-2601 • www.barr.com

Minneapolis MN • Hibbing MN • Duluth MN • Ann Arbor MI • Jefferson City MO

September 25, 2003

Sonia Vega
On-Scene Coordinator
U S Environmental Protection Agency, Region 5
Emergency Response Branch
520 Lafayette Road North
St Paul, MN 55155-4194

Re: Validated Analytical Data
Laboratory Batch Number – E2300499
Docket No. V-W-'03-C-748
St. Regis Paper Company Site – Cass Lake, Minnesota

Dear Ms Vega:

Barr has validated the analytical data contained in the laboratory batch number(s) identified above. Enclosed are the following documents related to these analytical data:

- Data Summary Table(s) {Attachment A}
- Validation Summary Report(s) {Attachment B}
- Complete analytical data package(s) from Columbia Analytical Services {Attachment C}

The data summary tables include the TEQ_{DF}-WHO₉₈ values calculated using the validated data. The TEQ calculation used a value of ½ the detection limit for any congener that was not detected.

All data met the data quality objectives and are useable as reported. In accordance with the Unilateral Administrative Order, on September 25, 2003 International Paper authorized Columbia Analytical Services to analyze the archived sample J29-30 4-12 for PCDD/F since sample J29-30 0-4 exceeded 1 ppb TEQ_{DF}-WHO₉₈.

If you have any questions concerning this information, please contact me at (952)832-2876 or Tom Ross of International Paper at (901)419-3899.

Sincerely,

Thomas D. Mattison
Project Coordinator

cc: Tim Drexler, U.S. EPA – RPM (w/ Attachments A, B & C)
Mony Chabria and Tom Turner, U S. EPA (w/ Attachments A & B)
Tom Ross, IP (w/ Attachment A, B & C)
Steve Ginski, IP (w/ Attachments A & B)
Rick Rothman, Bingham (w/ Attachments A & B)

Attachment A

Table 1
Validated Dioxin/Furan Concentration in Surface Soil
St. Regis Paper Company Site
Docket No: V-W-'03-C-748

(concentrations in µg/kg (ppb))

Location	F27-29 0-4	F27-29 4-12	H25-26 0-4	I26-27 0-4	I27-29 0-4	J29-30 0-4
Date	8/6/2003	8/6/2003	8/5/2003	8/6/2003	8/6/2003	8/6/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	0.003	0.002	0.00055 j	<0.00034	0.00079 j	0.0017 EMPC
1,2,3,7,8-PeCDD	0.068	0.032	0.008	0.004	0.021	0.046
1,2,3,4,7,8-HxCDD	0.260	0.123	0.024	0.010	0.054	0.166
1,2,3,6,7,8-HxCDD	0.963	0.274 j	0.108	0.064	0.320	3.072
1,2,3,7,8,9-HxCDD	0.295 j	0.242	0.049	0.022	0.112	0.389
1,2,3,4,6,7,8-HpCDD	30.852	11.364	2.344	2.496	11.694	186.701
OCDD	249.540 e	133.473 e	22.122 e	24.152	124.075	2182.551 e
2,3,7,8-TCDF	0.026	0.006	0.001	<0.00098	0.004	0.014
1,2,3,7,8-PeCDF	0.111	0.064	0.008	0.007	0.030	0.123
2,3,4,7,8-PeCDF	0.113	0.065	0.010	0.009	0.035	0.140
1,2,3,4,7,8-HxCDF	0.723	0.460	0.144	0.061	0.308	2.625
1,2,3,6,7,8-HxCDF	0.233	0.128	0.032	0.016	0.076	0.262
1,2,3,7,8,9-HxCDF	0.041	<0.035	0.032	0.021	0.084 EMPC	0.063
2,3,4,6,7,8-HxCDF	0.366	0.229	0.056	0.026	0.129	0.887
1,2,3,4,6,7,8-HpCDF	7.167	2.489	0.530	0.450	2.713	28.274
1,2,3,4,7,8,9-HpCDF	0.639	0.447	0.080	0.045	0.207	2.125
OCDF	29.433	14.042	1.686	1.682	12.664	132.899
TCDD, Total	0.016	0.004	0.002	<0.00034	0.003	0.010
PeCDD, Total	0.279	0.111	0.039	0.014	0.082	0.183
HxCDD, Total	7.753	2.747	0.548	0.257	1.388	5.045
HpCDD, Total	75.414	46.496	6.977	4.105	23.525	82.565
TCDF, Total	0.143	0.052	0.010	0.004	0.047	0.116
PeCDF, Total	2.648	1.686	0.229	0.154	0.759	2.490
HxCDF, Total	7.251	4.152	1.909	0.926	5.721	8.468
HpCDF, Total	34.820	30.590	3.558	2.094	13.912	53.718
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.840	0.370	0.120	0.060	0.310	3.300

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Attachment B

**Data Validation Report – Dioxin/Furan
Laboratory Report / Batch E2300499
Received September 11, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Report: September 23, 2003

The data validation of the Columbia Analytical Services, Inc. (CAS) laboratory data for the dioxin and furan (CDD/CDF) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil and/or groundwater samples contained in the aforementioned report is complete and detailed below.

The analytical data were reviewed in accordance with the U.S. EPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, Draft Final dated March, 2002 (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8290 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Mass calibration and mass spectrometer resolution
- Window defining mix
- Instrument stability
- Initial calibration and ongoing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Second column confirmation

Data Validation Report
Laboratory Report / Batch E2300499
Received September 11, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report: September 23, 2003

Six soil sample results are contained in this laboratory report. They are as follows:

H25-26 0-4'

F27-29 0-4'

F27-29 4-12'

J29-30 0-4'

J27-29 0-4'

J26-27 0-4'

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report E2300499 as a result of data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected August 5 and 6, 2003, cooled to 4^oC and sent to the laboratory. Per the chain-of-custody and subsequent laboratory acknowledgement receipt forms, the laboratory received the samples August 8, 2003 in acceptable condition. It should be noted that discrepancies between method 8290 and the Guidelines exist for the technical extraction and analysis holding times. The Guidelines recommend that soil samples be extracted within 10 days of receipt and analyzed within 30 days. Alternately, Method 8290 and the QAPP for the project indicate that a 30 day extraction/45 day analysis holding times apply. Method 8290 also states that these holding times are only recommendations as dioxins and furans are very stable in a variety of matrices. All samples were extracted and analyzed within 30 days. No qualifiers are applied.

Mass Calibration and Mass Spectrometer Resolution

Mass calibration and mass spectrometer resolution (MC/MSR) checks are required to ensure that the mass calibration and mass spectrometer resolution is set at a $\geq 10,000$ resolving power. This resolution is confirmed at the beginning and end of every 12-hour analysis period.

It should be noted that the Guidelines contain language that refers to evaluation techniques that due to upgrades in analytical systems software, are obsolete at CAS's Houston facility. Specifically, the

Data Validation Report
Laboratory Report / Batch E2300499
Received September 11, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report: September 23, 2003

evaluation of the resolution by the measure of deviation between the exact m/z and the theoretical m/z at less than 5 ppm has been replaced with a instrumentation function that implicitly sets the error to zero at all calibration points so there is no longer any need to use peak matching conditions to verify the exact mass. Therefore, resolving power is evaluated by close review of the resolution of PFK peak profiles where high mass ion (380 9760) and low mass ion (304 9824) are reported

CAS performed the MC/MSR at the appropriate frequency and obtained acceptable results for the PFK calibrant confirming MC/MSR at a resolving power of 10,000

Window Defining Mix

The window defining mix (WDM) is necessary to establish the appropriate switching times for the selection ion group descriptors. As stated in the Guidelines, the frequency of the WDM is once every 12 hours prior to calibration verification. However, Method 8290, Section 8.3.2.2.2. allows the laboratory, if running consecutive 12-hour shifts, to use the ending calibration verification (if all acceptance criteria are met) of the first 12-hour analysis period as the beginning calibration verification of the second 12-hour analysis period. In these cases, the WDM was analyzed after the calibration verification but still falls within the 12-hour analytical period. While the Guidelines indicate the WDM must be analyzed prior to the calibration verification sample, CAS's order of analysis for the system performance check samples did not appear to affect the overall system instrumentation and no qualifiers have been applied

CAS performed the WDM analysis for every 12-hour sample analysis period and presented the acceptable switching times for each homologue group on the corresponding Form 5s.

Chromatographic Resolution

This check is performed to ascertain the separation of closely eluting dioxin/furan isomers. This is performed using SICP (selected ion current profile) of each isomer. The criteria require that the 2,3,7,8-TCDD peak and the 1,2,3,8-TCDD peak be resolved with a valley of $\leq 25\%$ for the DB-5 column.

CAS provided summary peak to valley results as specified above on each Form 5. In each case, the $\leq 25\%$ criterion was met. The raw chromatograms were also reviewed to confirm this information.

Data Validation Report
Laboratory Report / Batch E2300499
Received September 11, 2003

St Regis Paper Company Superfund Site – Cass Lake Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report September 23, 2003

Instrument Stability

Instrument stability is an important aspect of this analytical system. Ongoing calibration verification using a CS-3 standard is performed once every 12-hour period. This standard is used to evaluate the isomer retention times, ion abundance criteria, sensitivity and ongoing calibration criteria.

CAS met the requirements of the absolute retention times (RT) for the first internal standard (13C-1,2,3,4-TCDD) of ± 15 seconds (of the initial calibration standard) and the associated >25.0 minute RT requirement for each of the 12-hour CS3 calibration verification standards. The relative retention time and ion abundance criteria were also met for each of the native and label CDD/CDFs in the ongoing CS-3 standard results.

Signal-to-noise (S/N) ratios were summarized for the native and labeled CDD/CDFs were acceptable at >10:1.

The relative response factor (RRF) criteria of <25% difference between CS-3 native compounds as compared to the initial calibration data was acceptable for the August 14, August 18, September 5, and September 8 analytical runs. For August 23, September 1, and August 19 analytical runs, exceedance of the 25% ending calibration verification standard criterion required the laboratory to provide the mean RRFs from the beginning and ending calibration verification analysis and those factors were then employed for quantitation, as specified in Method 8290, Section 8.3.2.4 and 7.7.4.4.

While the Guidelines indicate review of the relative responses (RR), Method 8290 has no such requirement, as RR's are not used in final quantitation of sample results, therefore, no review was performed.

Review of the above elements indicates a high level of instrument stability and no qualifiers are required.

Initial Calibration and Calibration Verification

Satisfactory instrument calibration is crucial to ensuring the accurate qualitative and quantitative results for each of the CDD/CDF compounds. Initial calibration procedures define the linear range and mean relative response factors that will be used for sample quantitation.

Data Validation Report
Laboratory Report / Batch E2300499
Received September 11, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report: September 23, 2003

CAS's initial calibration summary information met all the relevant acceptance criteria including the relative responses (RRFs) of <25% for native compounds and <35% for the labeled compounds, ion abundance ratios (IARs) within +/- 15%, absolute retention times within the WDM windows, and signal-to-noise ratios of >10:1.

Calibration verification (or continuing calibration) summary information also met all relevant acceptance criteria including the frequency of the continuing calibration verifications at both the beginning and ending of each 12-hour analytical run, the RRFs %D within <25% for native compounds and <35% for labeled compounds of initial calibration, the IARs within the +/-15%, and signal-to-noise of >10:1.

Initial calibration data were inadvertently omitted from the original data package received September 11, 2003 for the 2,3,7,8-TCDF second column confirmation instrument for the initial calibration date of August 5, 2003. The laboratory compiled the missing information and it is included in the original report, however, the additional pages are not paginated. The initial calibration data met all acceptance criteria and are included in the Second Column Confirmation section of this report.

Method Blank Analyses

Laboratory method blank analyses are used to determine the existence and magnitude of contamination introduced at the laboratory.

CAS's analysis of method blank samples met all acceptance criteria. No positive concentrations were reported in the method blanks above the CRQL. Trace concentrations of OCDD and HpCDD were detected in one or more of the method blanks associated with the analytical batch. However, associated sample concentrations are far greater than the blank concentration, therefore, no data require qualification. All method blank samples were prepared and analyzed at the appropriate frequency.

Laboratory Control Samples (Ongoing Precision/Accuracy)

The laboratory is required to prepare and analyze a sample of spiked reference matrix to measure the accuracy of the analytical process/system/method.

Data Validation Report
Laboratory Report / Batch E2300499
Received September 11, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report: September 23, 2003

CAS performed laboratory control sample (LCS) analyses at the appropriate frequency for the analytical batch. LCS results met associated acceptance criteria for percent recovery of the spiked concentrations as presented in Table 6 of Exhibit D of the Guidelines. All RRT and IARs were also acceptable in the LCS samples associated with the analytical batch indicating an in-control analytical system.

Labeled Compound Recovery (Surrogate Standard Recovery)

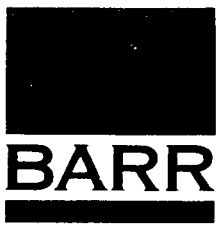
Because the introduced labeled compounds (CDD/CDFs) serve as the isotopic quantitative mechanism for this method, recoveries should be closely monitored for laboratory and method effectiveness.

CAS met the minimum acceptance criteria for the labeled compound recoveries as presented in Table 7 of Exhibit D of the Guidelines. These recovery windows are wider than CAS's internally generated acceptance criteria. Labeled compound recoveries qualified with a "Y" in the original data package indicate the recovery falls outside the laboratory-generated acceptance limits. None of these minor failures exceeded the Guidelines limits, therefore, no data require qualification.

Second Column Confirmation

A second column confirmation of the 2,3,7,8-TCDF isomer is required by both Methods 1613 and 8290 due to a known lack of isomer specificity for this isomer on the DB-5 or equivalent column. All operating conditions of the second column analytical system must be identical to those of the primary system.

CAS performed the second column confirmational analysis when the original analytical run had positive 2,3,7,8-TCDF detections. All performance criteria (as previously detailed above) were met prior to and during the confirmational analytical runs.



Barr Engineering Company
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September 29, 2003

Sonia Vega
On-Scene Coordinator
U.S. Environmental Protection Agency, Region 5
Emergency Response Branch
520 Lafayette Road North
St. Paul, MN 55155-4194

Re: Validated Analytical Data
Laboratory Batch Number – K2305902
Docket No. V-W-'03-C-748
St. Regis Paper Company Site – Cass Lake, Minnesota

Dear Ms. Vega:

Barr has validated the analytical data contained in the laboratory batch number(s) identified above. Enclosed are the following documents related to these analytical data:

- Data Summary Table(s) {Attachment A}
- Validation Summary Report(s) {Attachment B}
- Complete analytical data package(s) from Columbia Analytical Services {Attachment C}

All data met the data quality objectives and are useable as reported. If you have any questions concerning this information, please contact me at (952)832-2876 or Tom Ross of International Paper at (901)419-3899.

Sincerely,

Thomas D. Mattison
Project Coordinator

cc: Tim Drexler, U.S. EPA – RPM (w/ Attachments A, B & C)
Mony Chabria and Tom Turner, U.S. EPA (w/ Attachments A & B)
Tom Ross, IP (w/ Attachment A & B)
Steve Ginski, IP (w/ Attachments A & B)
Rick Rothman, Bingham (w/ Attachments A & B)

Attachment A

Table 2
Validated PAH/PCP Concentrations in Surface Soil
St. Regis Paper Company Site
Docket No: V-W-'03-C-748

(concentrations in µg/kg)

Location	F27-29 0-4	F27-29 4-12	H25-26 0-4
Date	8/6/2003	8/6/2003	8/5/2003
Lab	CAS	CAS	CAS
Solids, total, %	95.4	82.3	92.3
<u>Carcinogenic PAHs</u>			
Benzo(a)anthracene	280	170	95
Chrysene	620	260	130
Benzo(b)fluoranthene	680	610	120
Benzo(k)fluoranthene	500	380	120
Benzo(a)pyrene	330	380	120
Indeno(1,2,3-cd)pyrene	650	560	130
Dibenz(a,h)anthracene	88	120	22
<u>Non-Carcinogenic PAHs</u>			
Naphthalene	22	40	5.7
2-Methylnaphthalene	19	28	<5.1
Acenaphthylene	45	61	20
Acenaphthene	<5.1	8.3	<5.1
Fluorene	6.0	8.3	<5.1
Phenanthrene	79	74	46
Anthracene	210	330	35
Fluoranthene	430	290	190
Pyrene	460	320	200
Benzo(g,h,i)perylene	310	500	110
Pentachlorophenol	1400	2000	<210

Attachment B

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2305902
Received September 12, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: September 24, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete as detailed below

The analytical data were reviewed in accordance with the U.S EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

Three soil sample results are contained in this laboratory report. They are as follows:

F27-29 4-12

F27-29 0-4

H25-26 0-4

Data Validation Report
Laboratory Report / Batch K2305902
Received September 12, 2003

St Regis Paper Company Superfund Site - Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report September 24, 2003

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2305902 as a result of the data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected on August 5 and 6, 2003 and received at the CAS Houston laboratory on August 8, 2003 with an accompanying chain-of-custody (COC) form. All samples were received intact and were properly chilled (within 2-6 °C). The samples were properly stored until shipped overnight to the CAS Kelso laboratory for PAH and PCP analysis. The samples were received on August 9, 2003 in Kelso intact and at 4 ± 1 °C (temperature blank). No sample identification discrepancies are noted.

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibrations (ICALs) for PAHs and PCP instrument using selected ion monitoring (SIM) were performed on August 23 and August 29 using 8-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05. All target analytes met the <30 %RSD data validation criteria.

Data Validation Report
Laboratory Report / Batch K2305902
Received September 12, 2003

St Regis Paper Company Superfund Site – Cass Lake Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report September 24, 2003

However, the average RSD of the RRFs for benzo(b)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene and dibenz(a,h)anthracene fell outside the method criteria of <15%RSD. Following their SOP the laboratory utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being <20%. The mean RSD for this initial calibration event was calculated as 7.7%, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard, all target analytes met the laboratory's criteria of < 20% difference. There are no data validation criteria for second source standards.

Continuing Calibration Verification

All three continuing calibration verifications (analyzed August 27, 28 and September 2) met the data validation criteria for frequency and the <+25% difference RRFs from ICAL as well as the minimum RRFs of > 0.05 for all target analytes except PCP. Data from the August 28, 2003 continuing calibration show that PCP fell beyond the <+25% criteria at 26%. Following the Guidelines, up to four compounds can fall beyond continuing calibration verification %D if their corresponding RRFs are >0.01 and the %D is still <40%. Therefore, no qualifiers are required.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Surrogate Standard Recovery

All surrogate spike recoveries from the project samples met the data validation acceptance criteria. Dilution of samples F27-29 0-4' and F27-29 4-12' were required for PCP, therefore, recoveries of

Data Validation Report
Laboratory Report / Batch K2305902
Received September 12, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report September 24, 2003

2,4,6-tribromophenol surrogate are not applicable. However, associated recoveries did fall within the acceptance window.

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project samples and associated quality control samples met the method and data validation criteria.

Matrix Spike Recovery

Sample H25-26 0-4' was utilized as the matrix spike and matrix spike duplicate sample. With the exception of PCP, all percent recoveries and relative percent differences (RPDs) were within the laboratory's internal control limits. However, due to concentrations of PCP just below the laboratory method reporting limit for this compound, calculated PCP percent recoveries appear to fall above the criteria in both the MS/MSD samples. If, however, the recovery calculations are performed using the estimated PCP concentration of the sample instead of the ND, percent recoveries improve to fall within acceptance limits. No analytical error is associated with this anomaly; therefore, no qualifiers are applied.



Barr Engineering Company
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Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO

October 1, 2003

Sonia Vega
On-Scene Coordinator
U.S. Environmental Protection Agency, Region 5
Emergency Response Branch
520 Lafayette Road North
St. Paul, MN 55155-4194

Re: Validated Analytical Data
Laboratory Batch Number – K2306086, K2306177, K2306184, K2306188, K2306189,
K2306181
Docket No. V-W-'03-C-748
St. Regis Paper Company Site – Cass Lake, Minnesota

Dear Ms. Vega:

Barr has validated the analytical data contained in the laboratory batch number(s) identified above. Enclosed are the following documents related to these analytical data:

- Data Summary Table(s) {Attachment A}
- Validation Summary Report(s) {Attachment B}
- Complete analytical data package(s) from Columbia Analytical Services {Attachment C}

All data met the data quality objectives and are useable as reported. If you have any questions concerning this information, please contact me at (952)832-2876 or Tom Ross of International Paper at (901)419-3899.

Sincerely,

Thomas D. Mattison
Project Coordinator

cc: Tim Drexler, U.S. EPA – RPM (w/ Attachments A, B & C)
Mony Chabria and Tom Turner, U.S. EPA (w/ Attachments A & B)
Tom Ross, IP (w/ Attachment A & B)
Steve Ginski, IP (w/ Attachments A & B)
Rick Rothman, Bingham (w/ Attachments A & B)

Attachment A

Table 2
Validated PAH/PCP Concentrations in Surface Soil
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/kg)

Location	D25-26 0-4	D27-29 0-4	DF 8-9 0-4	DE 8 9 4-12	F 18-19 0-4	F 18-19 4-12	F24-25 0-4	F24 25 4-12	F24-25 4 12D	F29-30 0 4	F20 21 0 4	F 22 23 0-4	F 27 29 0 4	F 27 29 4 12
Date	8/8/2003	8/13/2003	8/8/2003	8/8/2003	8/13/2003	8/13/2003	8/13/2003	8/13/2003	8/13/2003	8/8/2003	8/15/2003	8/8/2003	8/6/2003	8 6 2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS
Dup														
Carcinogenic PAHs														
Benzo(a)anthracene	62	30	6100	5800	55	120	91	120	200	280	520	260	280	170
Chrysene	110	56	14000	8800	110	210	140	340	540	530	770	340	620	260
Benzo(b)fluoranthene	150	69	18000	9500	180	530	210	460	740	800	800	440	680	610
Benzo(k)fluoranthene	160	67	15000	8200	140	370	190	360	560	600	710	400	500	380
Benzo(a)pyrene	90	29	7700	4000	55	280	150	140	140	360	550	380	330	380
Indeno(1,2,3-cd)pyrene	170	88	4800	2300	130	410	300	330	420	730	720	700	650	500
Dibenz(a,h)anthracene	24	12	930	510	21	63	43	62	93	110	120	87	88	120
Non-Carcinogenic PAHs														
Naphthalene	9.5	6.7	140	23	5.8	18	17	19	15	45	12	14	22	40
2-Methylnaphthalene	7.2	6.0	42	10	5.7	14	16	15	13	34	9.9	10	19	28
Acenaphthylene	11	6.5	550	470	9.6	29	18	17	22	72	52	75	45	61
Acenaphthene	<5.1	<5.0	28	11	<5.1	<5.0	<5.0	<5.1	<5.1	7.2	<5.0	<5.1	5.1	8.3
Fluorene	<5.1	<5.0	30	19	<5.1	<5.0	<5.0	7.8	7.1	9.0	<5.0	7.1	6.0	8.3
Phenanthrene	26	19	180	68	12	23	54	56	110	130	68	190	79	74
Anthracene	33	22	1400	520	29	79	78	120	160	350	63	59	210	330
Fluoranthene	130	71	2000	2900	89	150	190	200	450	570	740	660	430	290
Pyrene	120	57	6300	6900	86	170	140	140	260	530	490	460	460	370
Benzo(g,h,i)perylene	120	56	2700	1300	74	220	180	170	200	490	400	530	310	800
Pentachlorophenol	430	<200	<410	210	360	270	940	1400	1900	1100	210	310	1400	2000

Table 2
Validated PAH/PCP Concentrations in Surface Soil
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/kg)

Location	FOA-02-1-0-4	FOA-02-2-0-4	FOA-02-2-4-12	FOA-02-3-0-4	FOA-02-4-0-4	FOA-02-5-0-4	FOA-02-6-0-4	FOA-02-6-4-12	GS-1	GS-1D	H25-26 0-4	J27-29 0 4
Date	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/13/2003	8/13/2003	8/5/2003	8/8/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS
Dup												
<u>Carcinogenic PAHs</u>												
Benzo(a)anthracene	850	73	24	11	83	27	40	18	250	280	95	260
Chrysene	1400	110	40	16	110	39	66	24	320	360	130	710
Benzo(b)fluoranthene	930	120	39	15	92	30	52	18	290	310	120	2600
Benzo(k)fluoranthene	1200	120	35	16	92	33	48	21	290	310	120	1700
Benzo(a)pyrene	870	86	27	11	85	25	38	21	310	340	120	1900
Indeno(1,2,3-cd)pyrene	790	120	39	11	95	32	52	23	330	380	130	1500
Dibenz(a,h)anthracene	190	19	5.6	<5.1	13	<5.0	7.2	<5.1	48	52	22	490
<u>Non-Carcinogenic PAHs</u>												
Naphthalene	34	7.5	<5.0	<5.1	<5.0	<5.0	10	<5.1	12	11	5.7	40
2-Methylnaphthalene	28	6.6	<5.0	<5.1	<5.0	<5.0	6.5	<5.1	9.4	8.8	5.1	25
Acenaphthylene	86	9.7	<5.0	<5.1	<5.0	<5.0	<5.1	<5.1	12	9.7	20	120
Acenaphthene	190	<5.1	<5.0	<5.1	<5.0	<5.0	<5.1	<5.1	9.3	20	5.1	9.7
Fluorene	96	<5.1	<5.0	<5.1	<5.0	<5.0	<5.1	<5.1	12	20	5.1	16
Phenanthrene	780	35	15	<5.1	49	29	32	11	240	310	16	100
Anthracene	410	22	8.7	<5.1	19	5.7	6.7	<5.1	35	47	35	490
Fluoranthene	3900	160	54	12	180	61	98	39	670	700	190	700
Pyrene	2300	130	43	12	140	55	80	34	400	520	200	540
Benzo(g,h,i)perylene	610	83	27	7.0	67	22	37	18	210	240	110	1000
Pentachlorophenol	3500	240	200	<210	<200	<200	240	210	210	200	210	100

Table 2
Validated PAH/PCP Concentrations in Surface Soil
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/kg)

Location	J27-29 12-24	J27-29 4-12	NWWD-02 4-12	RES24 4-12	SW-44 0-4
Date	8/8/2003	8/8/2003	8/15/2003	8/14/2003	8/11/2003
Lab	CAS	CAS	CAS	CAS	CAS
Dup					
<u>Carcinogenic PAHs</u>					
Benzo(a)anthracene	<5 0	<5 1	220	12	670
Chrysene	<5 0	6 4	240	24	950
Benzo(b)fluoranthene	<5 0	9 7	210	18	850
Benzo(k)fluoranthene	<5 0	8 1	220	15	740
Benzo(a)pyrene	<5 0	<5 1	240	10	720
Indeno(1,2,3-cd)pyrene	<5 0	7 6	220	14	740
Dibenz(a,h)anthracene	<5 0	<5 1	36	<5 0	110
<u>Non-Carcinogenic PAHs</u>					
Naphthalene	<5 0	<5 1	11	6 1	19
2-Methylnaphthalene	<5 0	<5 1	7 0	<5 0	8 8
Acenaphthylene	<5 0	<5 1	25	<5 0	69
Acenaphthene	<5 0	<5 1	<5 0	<5 0	10
Fluorene	<5 0	<5 1	<5 0	<5 0	15
Phenanthrene	<5 0	<5 1	66	25	350
Anthracene	<5 0	<5 1	19	<5 0	110
Fluoranthene	<5 0	5 7	380	57	1500
Pyrene	<5 0	5 3	310	33	1200
Benzo(g,h,i)perylene	<5 0	<5 1	140	11	440
Pentachlorophenol	<200	<210	<200	<200	850

Attachment B

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306086
Received September 19, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: September 29, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete and is detailed below

The analytical data were reviewed in accordance with the U S EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003) In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

Seven soil sample results are contained in this laboratory report They are as follows:

**J27-29 0-4”
F22-23 0-4”**

**J27-29 12-24”
DE8-9 0-4”**

**E29-30 0-4”
DE8-9 4-12”**

D25-26 0-4”

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2306086 as a result of data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected on August 8, 2003 and received at the CAS Houston laboratory on August 12, 2003 with an accompanying chain-of-custody (COC) form. The samples were received intact and were properly chilled (within 2-6 °C). The samples were properly stored until shipped overnight to the CAS Kelso laboratory for PAH and PCP analysis. The samples were received on August 15, 2003 in Kelso intact and at 4 ± 3°C (temperature blank). The sample for J27-29 4-12' was inadvertently not included with this shipment, it was shipped to Kelso on August 19, 2003 and is reported under separate cover (K2306188).

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project sample met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS Form 5s and associated quantitation and tune reports. Note that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs and PCP instrument using selected ion monitoring (SIM) was performed on August 23, 2003 and September 3, 2003 using 8-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes

Data Validation Report
Laboratory Report / Batch K2306086
Received September 19, 2003

St. Regis Paper Company Superfund Site - Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: September 29, 2003

met the data validation requirement of >0.05 . All target analytes met the $<30\%$ RSD data validation criteria with the exception of PCP on September 3, 2003. The average RSD of the RRFs for PCP (26.0%) did exceed the method criteria of $<15\%$ RSD, however the laboratory utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being $<20\%$. The mean RSD for this initial calibration event was calculated as 7.4% thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard, all target analytes met the laboratory's criteria of $<20\%$ difference. There are no data validation criteria for second source calibration verification standards.

Continuing Calibration Verification

The August 27, 2003 continuing calibration verification met the data validation criteria for frequency and the $<+25\%$ difference RRFs from the ICAL and the minimum RRF of >0.05 for all targets except dibenz(a,h)anthracene. The percent difference was -26% for this compound. This calibration verification summary is associated with a diluted analytical run of which dibenz(a,h)anthracene was not a target compound, therefore, no qualification is necessary. The September 3 and 4, 2003 continuing calibration verification met the data validation criteria for frequency and the $<+25\%$ difference RRFs from ICAL as well as the minimum RRFs of >0.05 for all target analytes.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Data Validation Report
Laboratory Report / Batch K2306086
Received September 19, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report September 29, 2003

Surrogate Standard Recovery

All surrogate spike recoveries from the project samples met the data validation acceptance criteria except for the sample J27-29 12-24'. The laboratory re-extracted and re-analyzed the sample with similar results. The fluorene-d10 surrogate recovery fell just below the acceptance window of 43-98% at 37%. The biphenyl-d10 surrogate recovery was 15%. Because the recoveries were >10% and the fluorene-d10 recovery was nominally outside the lower acceptance criteria window of 43% and the laboratory produced similar results on the second analysis of the sample, no qualification is applied.

Internal Standard Recovery

All internal standards were reviewed for area counts and retention time criteria. All internal standards from the project sample and associated quality control samples met the method and data validation criteria except for the original analysis of sample DE8-9 0-4'. The sample was diluted and all internal standard criteria were met. The reported results are associated with the acceptable diluted analysis, therefore, no qualification is required.

Matrix Spike Recovery

The sample D25-26 0-4' served as the Matrix Spike (MS) and Matrix Spike Duplicate (MSD) sample. Matrix spike percent recoveries were greater than expected for PCP, indeno(1,2,3-cd)pyrene and benzo(g,h,i)perylene at 210%, 144%, and 126%, respectively. The associated matrix spike duplicate percent recoveries for indeno(1,2,3-cd)pyrene and benzo(g,h,i)perylene were 117% and 104%, respectively and fell within acceptance limits as did the associated RPDs. The PCP percent recovery in the matrix spike duplicate sample was 145%, still slightly above acceptance criteria. The RPD for PCP MS/MSD was acceptable as 18%. The associated LCS and LCSD sample results were acceptable indicating a slight sample matrix effect. Because this would equate to a potential high bias within the sample and because positive PCP detections were quantified, based on professional judgment no data qualifiers were applied due to the MS/MSD results.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306177
Received September 18, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Report: September 23, 2003

The data validation process for the Columbia Analytical Services, Inc. (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete and detailed below.

The analytical data were reviewed in accordance with the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery
- Overall assessment

Eight soil sample results are contained in this laboratory report. They are as follows:

D27-29 0-4"	E24-25 4-12"	E18-19 4-12"	GS-1
E18-19 0-4"	E24-25 4-12"D	E24-25 0-4"	GS-1D

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2306177 as a result of data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected on August 13, 2003 and received at the CAS Houston laboratory on August 15, 2003 with an accompanying chain-of-custody (COC) form. All samples were received intact and were properly chilled (within 2-6 °C). The samples were properly stored until shipped overnight to the CAS Kelso laboratory for PAH and PCP analysis. The samples were received on August 19, 2003 in Kelso intact and at 5.7 °C (temperature blank). Some of the labels on the sample containers had field sample identifiers that did not match the identifiers listed on the COC. The identifiers written on the sample container lids did match the identifiers written on the COC form with the exception of sample GS-1 and GS-1D. These two samples had 0-4 and 0-4D respectively written on the sample container lids.

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3 1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs and PCP instrument using selected ion monitoring (SIM) was performed on September 3, 2003 using 8-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05 . All target analytes met the $<30\%$ RSD data validation criteria. The average RSD of the RRFs for PCP (26.0%) did exceed the method criteria of $<15\%$ RSD however the laboratory utilized an alternative calibration evaluation as specified in section 5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being $<20\%$. The mean RSD for this initial calibration event was calculated as 7.4%, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard, all target analytes met the laboratory's criteria of $<20\%$ difference. There are no data validation criteria for second source standards.

Continuing Calibration Verification

The September 4, 2003 continuing calibration verifications met the data validation criteria for frequency and the $<+25\%$ difference RRFs from ICAL as well as the minimum RRFs of >0.05 for all target analytes.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Surrogate Standard Recovery

All surrogate spike recoveries from the project samples met the data validation acceptance criteria.

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project samples and associated quality control samples met the method and data validation criteria.

Matrix Spike Recovery

A non-project sample was utilized for the Matrix Spike (MS) and Matrix Spike Duplicate (MSD). Since the source of the sample that was spiked was a non-project, the data has limited applicability to the project data. All percent recoveries and RPDs from the MS and MSD were within the laboratory's internal control limits.

Field Duplicate Results

Field duplicate results evaluate overall sampling and laboratory precision. Samples GS-1 and E24-25 4-12" served as the field duplicates for this submittal. The average Relative Percent Difference (RPD) for the detected compounds in samples GS-1 and GS-1D was approximately 19%. The average RPD for the detected compounds in samples E24-25 4-12" and E24-25 4-12"D was approximately 35%. Both average RPD results displayed an acceptable level of precision for the low level nature of the analytical method.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306184
Received September 19, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: September 29, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete and detailed below

The analytical data were reviewed in accordance with the U S EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

Two soil sample results are contained in this laboratory report. They are as follows:

NWWD-02 4-12”

F20-21 0-4”

Data Validation Report
Laboratory Report / Batch K2306184
Received September 19 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: September 29 2003

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2306184 as a result of data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected on August 15, 2003 and received at the CAS Houston laboratory on August 16, 2003 with an accompanying chain-of-custody (COC) form. The samples were received intact and were properly chilled (within 2-6 °C). The samples were properly stored until shipped overnight to the CAS Kelso laboratory for PAH and PCP analysis. The samples were received on August 19, 2003 in Kelso intact and at 5.7°C (temperature blank).

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project sample met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS Form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs and PCP instrument using selected ion monitoring (SIM) was performed on September 3, 2003 using 8-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05. All target analytes met the <30 %RSD data validation criteria with the exception of PCP on September 3, 2003. The average RSD of the RRFs for PCP (26.0%) did exceed

Data Validation Report
Laboratory Report / Batch K2306184
Received September 19, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report September 29, 2003

the method criteria of $<15\%$ RSD, however the laboratory utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes, average RSDs, and subsequent comparison to the requirement of the mean RSD being $<20\%$. The mean RSD for this initial calibration event was calculated as 7.4%, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard, all target analytes met the laboratory's criteria of $<20\%$ difference. There are no data validation criteria for second source calibration verification standards.

Continuing Calibration Verification

The September 4, 5, and 6, 2003 continuing calibration verifications met the data validation criteria for frequency and the $<+25\%$ difference RRFs from the ICAL and the minimum RRF of >0.05 for all target compounds. The September 5, 2003 continuing calibration percent difference did exceed the internal laboratory criterion of 20% for dibenz(a,h)anthracene. The percent difference was 25% for this compound. This calibration verification summary is associated with a diluted analytical run of which dibenz(a,h)anthracene was not a target compound, in addition the 25% does meet the Guideline criterion, therefore, no qualification is necessary. The September 4 and 6, 2003 continuing calibration verification met the data validation criteria for frequency and the $<-25\%$ difference RRFs from ICAL as well as the minimum RRFs of >0.05 for all target analytes.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Data Validation Report
Laboratory Report / Batch K2306184
Received September 19, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report September 29, 2003

Surrogate Standard Recovery

All surrogate spike recoveries from the project samples met the data validation acceptance criteria for the project samples. Percent recoveries of the terphenyl-d14 surrogate fell just above the acceptance window of 61-122 at 128%, 126% and 124% in the MSD, LCS and LCSD samples respectively. No qualification is applied to the project samples.

Internal Standard Recovery

All internal standards were reviewed for area counts and retention time criteria. All internal standards from the project sample and associated quality control samples met the method and data validation criteria.

Matrix Spike Recovery

The sample F20-21 0-4' served as the Matrix Spike (MS) and Matrix Spike Duplicate (MSD) sample for the analytical batch. Matrix spike percent recoveries were acceptable. The MSD recovery for PCP was slightly above the expected range of 70-130 at 147%. Because the LCS and LCSD sample recoveries were acceptable and the RPDs between the MS and MSD are acceptable, no qualifiers are applied.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306188
Received September 18, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: September 29, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete and is detailed below

The analytical data were reviewed in accordance with the U S EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003) In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

One soil sample result set is contained in this laboratory report. It is as follows.

J27-29 4-12'

Data Validation Report
Laboratory Report / Batch K2306188
Received September 18 2003

St Regis Paper Company Superfund Site - Cass Lake Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report September 29 2003

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2306188 as a result of data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The sample was collected on August 8, 2003 and received at the CAS Houston laboratory on August 12, 2003 with an accompanying chain-of-custody (COC) form. The sample was received intact and was properly chilled (within 2-6 °C). The sample was inadvertently not included with the first shipment to Kelso with laboratory batch number K2306086, however, it was properly stored until shipped overnight to the CAS Kelso laboratory for PAH and PCP analysis. The sample was received on August 19, 2003 in Kelso intact and at 5-7°C (temperature blank).

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project sample met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS Form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs and PCP instrument using selected ion monitoring (SIM) was performed on September 3, 2003 using 8-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05. All target analytes met the <30 %RSD data validation criteria. The average

Data Validation Report
Laboratory Report / Batch K2306188
Received September 18, 2003

St. Regis Paper Company Superfund Site - Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: September 29, 2003

RSD of the RRFs for PCP (26.0%) did exceed the method criteria of <15% RSD, however the laboratory utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being <20%. The mean RSD for this initial calibration event was calculated as 7.4%, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard, all target analytes met the laboratory's criteria of <20% difference. There are no data validation criteria for second source calibration verification standards.

Continuing Calibration Verification

The September 4, 2003 continuing calibration verification met the data validation criteria for frequency and the <+25% difference RRFs from ICAL as well as the minimum RRFs of >0.05 for all target analytes.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process system method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Surrogate Standard Recovery

All surrogate spike recoveries from the project samples met the data validation acceptance criteria.

Internal Standard Recovery

All internal standards were reviewed for area counts and retention time criteria. All internal standards from the project sample and associated quality control samples met the method and data validation criteria.

Data Validation Report
Laboratory Report / Batch K2306188
Received September 18 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: September 29, 2003

Matrix Spike Recovery

A non-project sample was utilized for the Matrix Spike (MS) and Matrix Spike Duplicate (MSD). Since the source of the sample that was spiked was a non-project, the data has limited applicability to the project data. All percent recoveries and RPDs from the MS and MSD were within the laboratory's internal control limits.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306189
Received September 18, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: September 29, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete and is detailed below

The analytical data were reviewed in accordance with the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

One soil sample result set is contained in this laboratory report. It is for sample:

RES 24 4-12”

Data Validation Report
Laboratory Report / Batch K2306189
Received September 18, 2003

St. Regis Paper Company Superfund Site - Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: September 29, 2003

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2306189 as a result of data validation process. All data met the data quality objective (DQO) and are useable as reported.

Holding Times, Preservation and Storage

The sample was collected on August 14, 2003 and received at the CAS Houston laboratory on August 16, 2003 with an accompanying chain-of-custody (COC) form. All samples were received intact and were properly chilled (within 2-6 °C). The sample was properly stored until shipped overnight to the CAS Kelso laboratory for PAH and PCP analysis. The sample was received on August 19, 2003 in Kelso intact and at 5.7 °C (temperature blank).

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs and PCP instrument using selected ion monitoring (SIM) was performed on September 3, 2003 using 8-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05. All target analytes met the <30 %RSD data validation criteria. The average RSD of the RRFs for PCP (26.0%) did exceed the method criteria of <15%RSD, however the

Data Validation Report
Laboratory Report / Batch K2306189
Received September 18, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: September 29, 2003

laboratory utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes, average RSDs, and subsequent comparison to the requirement of the mean RSD being <20%. The mean RSD for this initial calibration event was calculated as 7.4%, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard. All target analytes met the laboratory's criteria of < 20% difference. There are no data validation criteria for second source standards.

Continuing Calibration Verification

The September 4, 2003 continuing calibration verifications met the data validation criteria for frequency and the <+25% difference RRFs from ICAL, as well as the minimum RRFs of > 0.05 for all target analytes.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Surrogate Standard Recovery

All surrogate spike recoveries from the project sample met the data validation acceptance criteria.

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project samples and associated quality control samples met the method and data validation criteria.

Data Validation Report
Laboratory Report / Batch K2306189
Received September 18, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report September 29, 2003

Matrix Spike Recovery

A non-project sample was utilized for the Matrix Spike (MS) and Matrix Spike Duplicate (MSD). Since the source of the sample that was spiked was a non-project, the data has limited applicability to the project data. All percent recoveries and RPDs from the MS and MSD were within the laboratory's internal control limits.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306181
Received September 19, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: September 30, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete and is detailed below.

The analytical data were reviewed in accordance with the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

Nine soil sample results are contained in this laboratory report. They are as follows:

SW44 0-4"	FOA-02-1 0-4"	FOA-02-2 0-4"	FOA-02-2 4-12"
FOA-02-3 0-4"	FOA-02-4 0-4"	FOA-02-5 0-4"	FOA-02-6 0-4"
FOA-02-6 4-12"			

Data Validation Report
Laboratory Report / Batch K2306181
Received September 19, 2003

St. Regis Paper Company Superfund Site - Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: September 30, 2003

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2306181 as a result of data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected on August 11th and 12th, 2003 and received at the CAS Houston laboratory on August 14, 2003 with an accompanying chain-of-custody (COC) form. All samples were received intact and were properly chilled (within 2-6 °C). The samples were properly stored until shipped overnight to the CAS Kelso laboratory for PAH and PCP analysis. The samples were received on August 19, 2003 in Kelso intact and at 5-7 °C (temperature blank).

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs and PCP instrument using selected ion monitoring (SIM) was performed on September 3, 2003 using 8-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05 . All target analytes met the $<30\%$ RSD data validation criteria. The average RSD of the RRFs for PCP (26.0%) did exceed the method criteria of $<15\%$ RSD, however the

Data Validation Report
Laboratory Report / Batch K2306181
Received September 19, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report September 30, 2003

laboratory utilized an alternative calibration evaluation as specified in section 7.5 1 2 1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being <20%. The mean RSD for this initial calibration event was calculated as 7.4%, thus meeting the method criteria

The laboratory report also included results from the analysis of a second source calibration check standard, all target analytes met the laboratory's criteria of < 20% difference. There are no data validation criteria for second source standards

Continuing Calibration Verification

The September 4th and 5th, 2003 continuing calibration verifications met the data validation criteria for frequency and the <+25% difference RRFs from ICAL as well as the minimum RRFs of > 0.05 for all target analytes. The September 5th, 2003 continuing calibration verification for dibenz(a,h) anthracene fell outside the laboratory control limit of 15% D, but met the Guideline criteria of <+25% as stated above. Additionally, this continuing calibration verification run was only used to as a diluted analytical run for select samples. Dibenz(a,h) anthracene was not a target compound from this analytical run.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Surrogate Standard Recovery

All surrogate spike recoveries from the project samples met the data validation acceptance criteria with the exception of biphenyl-d10 in sample FOA-01-2 0-4". Recovery of this surrogate was

Data Validation Report
Laboratory Report / Batch K2306181
Received September 19, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report September 30, 2003

nominally outside limits of 39-97% at 31%. Following the Guidelines, one surrogate can fall outside acceptance criteria if greater than 10% therefore, no qualifiers are required.

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project samples and associated quality control samples met the method and data validation criteria.

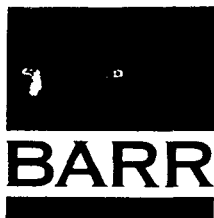
Matrix Spike Recovery

Sample FOA-02-6 4-12" served as the Matrix Spike (MS) and Matrix Spike Duplicate (MSD) for the analytical batch. All percent recoveries and RPDs from the MS and MSD were within the laboratory's internal control limits.

Field Duplicate Results

No field duplicates were included in this analytical batch.

Attachment C



Barr Engineering Company
4700 West 77th Street • Minneapolis, MN 55435-4803
Phone 952-832-2600 • Fax 952-832-2601 • www.barr.com

Minneapolis MN • Hibbing MN • Duluth MN • Ann Arbor MI • Jefferson City MO

October 3, 2003

Sonia Vega
On-Scene Coordinator
U.S. Environmental Protection Agency, Region 5
Emergency Response Branch
520 Lafayette Road North
St. Paul, MN 55155-4194

Re: Validated Analytical Data
Laboratory Batch Number – E2300509
Docket No. V-W-'03-C-748
St. Regis Paper Company Site – Cass Lake, Minnesota

Dear Ms. Vega:

Barr has validated the analytical data contained in the laboratory batch number(s) identified above. Enclosed are the following documents related to these analytical data:

- Data Summary Table(s) {Attachment A}
- Validation Summary Report(s) {Attachment B}
- Complete analytical data package(s) from Columbia Analytical Services {Attachment C}

The data summary tables include the TEQ_{DF}-WHO₉₈ values calculated using the validated data. The TEQ calculation used a value of ½ the detection limit for any congener that was not detected.

All data met the data quality objectives and are useable as reported. In accordance with the Unilateral Administrative Order, on October 3, 2003 International Paper authorized Columbia Analytical Services to analyze the archived sample J25-26 0-4 for PCDD/F since the adjacent sample J26-27 0-4 exceeds 1 ppb TEQ_{DF}-WHO₉₈.

If you have any questions concerning this information, please contact me at (952)832-2876 or Tom Ross of International Paper at (901)419-3899.

Sincerely,

Thomas D. Mattison
Project Coordinator

cc: Tim Drexler, U.S. EPA – RPM (w/ Attachments A, B & C)
Mony Chabria and Tom Turner, U.S. EPA (w/ Attachments A & B)

Attachment A

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 Attorney-Client Communication
 Attorney Work Product

Table 1

Validated Dioxin/Furan Concentration in Soil
 St. Regis Paper Company Site
 Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	D25-26 0-4 8/8/2003 CAS	DE8-9 0-4 8/8/2003 CAS	DE8-9 4-12 8/8/2003 CAS	E29-30 0-4 8/8/2003 CAS	F22-23 0-4 8/8/2003 CAS	F27-29 0-4 8/6/2003 CAS
2,3,7,8-TCDD	<0.0003	<0.0006	<0.00052	0.006	0.002	0.003
1,2,3,7,8-PeCDD	0.021	0.017	0.011	0.143	0.050	0.068
1,2,3,4,7,8-HxCDD	0.049	0.067	0.046	0.438	0.143	0.260
1,2,3,6,7,8-HxCDD	0.404	0.206	0.132	1.602	0.402	0.963
1,2,3,7,8,9-HxCDD	0.135	0.130	0.090	0.748	0.313	0.295 j
1,2,3,4,6,7,8-HpCDD	12.855	8.516	6.274	49.437	13.533	30.852
OCDD	133.391	55.358 e	45.773 e	428.350	133.542	249.540 e
2,3,7,8-TCDF	0.004	0.001	0.0009 EMPC	0.014	0.003	0.026
1,2,3,7,8-PeCDF	0.038	0.006	0.004	0.086	0.017	0.111
2,3,4,7,8-PeCDF	0.033	0.007	0.005	0.094	0.019	0.113
1,2,3,4,7,8-HxCDF	0.372	0.068	0.053	0.617 EMPC	0.228	0.723
1,2,3,6,7,8-HxCDF	0.112	0.023	0.017	0.309	0.080	0.233
1,2,3,7,8,9-HxCDF	<0.0269	0.002	0.0016 j EMPC	0.030	0.029	0.041
2,3,4,6,7,8-HxCDF	0.176	0.045	0.036	0.566	0.151	0.366
1,2,3,4,6,7,8-HpCDF	3.413	1.131	0.949	12.178	2.411	7.167
1,2,3,4,7,8,9-HpCDF	0.360	0.106	0.075	0.806	0.276	0.639
OCDF	12.861	5.449	4.530	58.002	11.891	29.433
TCDD, Total	0.002	<0.0006	<0.00052	0.031	0.006	0.016
PeCDD, Total	0.082	0.074	0.046	0.547	0.175	0.279
HxCDD, Total	1.766	2.062	1.151	8.027	2.704	7.753
HpCDD, Total	28.397	22.841	13.136	92.152	33.768	75.414
TCDF, Total	0.030	0.009	0.006	0.251	0.053	0.143
PeCDF, Total	0.753	0.190	0.156	3.336	0.764	2.648
HxCDF, Total	3.317	1.336	1.020	9.664	4.802	7.251
HpCDF, Total	17.592	5.412	3.816	45.124	15.592	34.820
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.346	0.179	0.130	1.305	0.374	0.840

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

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Attorney Work Product

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location	F27-29 4-12	H25-26 0-4	I26-27 0-4	I27-29 0-4	J26-27 0-4	J26-27 4-12
Date	8/6/2003	8/5/2003	8/6/2003	8/6/2003	8/8/2003	8/8/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	0.002	0.00055 _j	<0.00034	0.00079 _j	0.002	0.000554 _j
1,2,3,7,8-PeCDD	0.032	0.008	0.004	0.021	0.083	0.026
1,2,3,4,7,8-HxCDD	0.123	0.024	0.010	0.054	0.313	0.089
1,2,3,6,7,8-HxCDD	0.274 _j	0.108	0.064	0.320	5.251	3.090
1,2,3,7,8,9-HxCDD	0.242	0.049	0.022	0.112	0.830	0.255
1,2,3,4,6,7,8-HpCDD	11.364	2.344	2.496	11.694	158.645	112.241
OCDD	133.473 _e	22.122 _e	24.152	124.075	1504.395 _e	1406.612 _e
2,3,7,8-TCDF	0.006	0.001	<0.00098	0.004	0.065	0.037
1,2,3,7,8-PeCDF	0.064	0.008	0.007	0.030	0.412	0.209
2,3,4,7,8-PeCDF	0.065	0.010	0.009	0.035	0.755	0.308
1,2,3,4,7,8-HxCDF	0.460	0.144	0.061	0.308	9.161	4.746
1,2,3,6,7,8-HxCDF	0.128	0.032	0.016	0.076	1.489	0.757
1,2,3,7,8,9-HxCDF	<0.035	0.032	0.021	0.084 EMPC	0.079	0.049
2,3,4,6,7,8-HxCDF	0.229	0.056	0.026	0.129	2.451	1.382
1,2,3,4,6,7,8-HpCDF	2.489	0.530	0.450	2.713	50.506	30.098
1,2,3,4,7,8,9-HpCDF	0.447	0.080	0.045	0.207	4.332	2.502
OCDF	14.042	1.686	1.682	12.664	209.140 _e	171.091 _e
TCDD, Total	0.004	0.002	<0.00034	0.003	0.027	0.009
PeCDD, Total	0.111	0.039	0.014	0.082	0.253	0.070
HxCDD, Total	2.747	0.548	0.257	1.388	12.278	5.265
HpCDD, Total	46.496	6.977	4.105	23.525	70.667	71.507
TCDF, Total	0.052	0.010	0.004	0.047	0.338	0.144
PeCDF, Total	1.686	0.229	0.154	0.759	9.483	3.986
HxCDF, Total	4.152	1.909	0.926	5.721	24.238	13.173
HpCDF, Total	30.590	3.558	2.094	13.912	47.376	42.756
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.370	0.120	0.060	0.310	4.753	2.837

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	J26-27 12-24 8/8/2003 CAS	J27-29 0-4 8/8/2003 CAS	J27-29 4-12 8/8/2003 CAS	J27-29 12-24 8/8/2003 CAS	J29-30 0-4 8/6/2003 CAS
2,3,7,8-TCDD	<0.00016	0.002	<0.000162	<0.00013	0.0017 EMPC
1,2,3,7,8-PeCDD	0.0019 j	0.033	<0.00017	0.00055 j	0.046
1,2,3,4,7,8-HxCDD	0.0086 EMPC	0.071	0.00076 j	0.0014 j	0.166
1,2,3,6,7,8-HxCDD	0.189	2.426	0.009	0.043	3.072
1,2,3,7,8,9-HxCDD	0.023	0.243	0.0011 j	0.003	0.389
1,2,3,4,6,7,8-HpCDD	5.335	79.840 e	0.282	1.096	186.701
OCDD	52.529 e	747.571 e	2.208 e	8.740	2182.551 e
2,3,7,8-TCDF	0.004	0.010	<0.000163	<0.0012	0.014
1,2,3,7,8-PeCDF	0.023	0.127	<0.00016	0.0016 j	0.123
2,3,4,7,8-PeCDF	0.029	0.181	0.00054 j	0.003	0.140
1,2,3,4,7,8-HxCDF	0.252	2.798	0.008	0.036	2.625
1,2,3,6,7,8-HxCDF	0.048	0.386	0.0013 j	0.006	0.262
1,2,3,7,8,9-HxCDF	<0.0103	0.033 EMPC	<0.00061	<0.00061	0.063
2,3,4,6,7,8-HxCDF	0.070	0.844	0.003	0.012	0.887
1,2,3,4,6,7,8-HpCDF	1.515	28.143	0.116	0.490	28.274
1,2,3,4,7,8,9-HpCDF	0.089	2.203	0.012	0.062	2.125
OCDF	9.036	154.944 e	0.624	2.748	132.899
TCDD, Total	0.001	0.018	<0.00016	<0.00013	0.010
PeCDD, Total	0.003	0.138	<0.00017	0.001	0.183
HxCDD, Total	0.487	5.660	0.021	0.095	5.045
HpCDD, Total	9.989	66.753	0.461	2.229	82.565
TCDF, Total	0.015	0.113	<0.00016	0.004	0.116
PeCDF, Total	0.420	2.748	0.006	0.033	2.490
HxCDF, Total	2.976	11.552	0.124	0.608	8.468
HpCDF, Total	8.349	39.244	0.572	3.440	53.718
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.153	2.005	0.007	0.029	3.300

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range.

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Attachment B

**Data Validation Report – Dioxin/Furan
Laboratory Report / Batch E2300509
Received September 22, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Data Validation Report: October 2, 2003

The data validation of the Columbia Analytical Services, Inc. (CAS) laboratory data for the dioxin and furan (CDD/CDF) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete as detailed below

The analytical data were reviewed in accordance with the U.S. EPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, Draft Final dated March, 2002 (Guidelines) as specified in the project-specific Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8290 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Mass calibration and mass spectrometer resolution
- Window defining mix
- Initial calibration
- Instrument stability and continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Second column confirmation

Data Validation Report
Laboratory Report / Batch E2300509
Received September 22, 2003

St. Regis Paper Company Superfund Site - Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report October 2, 2003

Eleven soil sample and one field blank results are contained in this laboratory report. They are as follows:

J26-27 0-4"	J26-27 4-12"	J26-27 12-24"	J27-29 0-4"
J27-29 4-10"	J27-29 12-24"	E29-30 0-4"	D25-26 0-4"
F22-23 0-4"	DE8-9 0-4"	DE8-9 4-12"	FB-2

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report E2300509 as a result of the data validation process. All data met the data quality objectives (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected August 8, 2003, cooled to 4°C and sent to the laboratory. Per the chain-of-custody and subsequent laboratory acknowledgement receipt forms, the laboratory received the samples August 12, 2003 in acceptable condition and at 4°C. It should be noted that discrepancies between Method 8290 and the Guidelines exist for the technical extraction and analysis holding times. The Guidelines recommend that soil samples be extracted within 10 days of receipt and analyzed within 30 days. Alternately, Method 8290 and the QAPP for the project indicate that 30-day extraction/45 day analysis holding times apply. Method 8290 also states that these holding times are only recommendations as dioxins and furans are very stable in a variety of matrices. All samples were extracted and analyzed within 30 days. No qualifiers are applied.

Mass Calibration and Mass Spectrometer Resolution

Mass calibration and mass spectrometer resolution (MC/MSR) checks are required to ensure that the mass calibration and mass spectrometer resolution is set at a $\geq 10,000$ resolving power. This resolution is confirmed at the beginning and end of every 12-hour analysis period.

It should be noted that the Guidelines contain language that refers to evaluation techniques that due to upgrades in analytical systems software, are obsolete at CASs Houston facility. Specifically, the evaluation of the resolution by the measure of deviation between the exact m/z and the theoretical m/z at less than 5 ppm has been replaced with an instrumentation function that implicitly sets the

error to zero at all calibration points so there is no longer any need to use peak matching conditions to verify the exact mass. Therefore, resolving power is evaluated by close review of the resolution of PFK peak profiles where high mass ion (380 9760) and low mass ion (304 9824) are reported.

CAS performed the MC/MSRs at the appropriate frequency and obtained acceptable results for the PFK calibrant confirming MC/MSRs at a resolving power of 10,000. Note: CAS used three instrument systems to analyze the project samples and provided appropriate documentation from the three systems in the raw data package. All system's documentation were evaluated during the data validation process.

Window Defining Mix

The window defining mix (WDM) is necessary to establish the appropriate switching times for the selection ion group descriptors. As stated in the Guidelines, the frequency of the WDM is once every 12 hours prior to calibration verification. However, Method 8290, Section 8.3.2.2.2 allows the laboratory, if running consecutive 12-hour shifts, to use the ending calibration verification (if all acceptance criteria are met) of the first 12-hour analysis period as the beginning calibration verification of the second 12-hour analysis period. In these cases, the WDM was analyzed after the calibration verification but still falls within the 12-hour analytical period. While the Guidelines indicate the WDM must be analyzed prior to the calibration verification sample, CAS's order of analysis for the system performance check samples did not appear to affect the overall system instrumentation and no qualifiers have been applied.

CAS performed the WDM analysis for every 12-hour sample analysis period and presented the acceptable switching times for each homologue group on the corresponding Form 5.

Chromatographic Resolution

This check is performed to ascertain the separation of closely eluting dioxin/furan isomers. This is performed using SICP (selected ion current profile) of each isomer. The criteria requires that the 2,3,7,8-TCDD and the 1,2,3,8-TCDD peaks (for the DB-5 column) and the 2,3,7,8-TCDF and the 2,3,4,7-TCDF peaks (for the DB-225 column) be resolved with a valley of $\leq 25\%$.

CAS provided summary peak to valley results as specified above on each Form 5. In each case, the $\leq 25\%$ criterion was met. The raw chromatograms were also reviewed to confirm this information.

Initial Calibration

Satisfactory instrument calibration is crucial to ensuring the accurate qualitative and quantitative results for each of the CDD/CDF compounds. Initial calibration procedures define the linear range and mean relative response factors that will be used for sample quantitation.

All of the initial calibration summary information met all the relevant acceptance criteria including the relative responses (RRFs) of $<25\%$ for native compounds and $<35\%$ for the labeled compounds, ion abundance ratios (IARs) within $\pm 15\%$, absolute retention times within the WDM windows, and signal-to-noise ratios of $>10:1$.

Initial calibration for the 2,3,7,8-TCDF second column confirmation instrument occurred on August 6, 2003. The initial calibration data met all acceptance criteria and is included in the Second Column Confirmation section of this report.

Instrument Stability and Continuing Calibration Verification

Instrument stability is an important aspect of this analytical system. Ongoing calibration verification using a CS-3 standard is performed once every 12-hour period. This standard is used to evaluate the isomer retention times, ion abundance criteria, sensitivity and ongoing calibration criteria. The continuing calibration verification summary information met all relevant acceptance criteria including the frequency of the continuing calibration verifications at the both beginning and ending of each 12-hour analytical run, the RRFs $\pm 25\%$ within $<25\%$ for native compounds and $<35\%$ for labeled compounds of initial calibration, the IARs within the $\pm 15\%$, and signal-to-noise of $>10:1$.

CAS met the requirements of the absolute retention times (RT) for the first internal standard (13C-1,2,3,4-TCDD) of ± 15 seconds (of the initial calibration standard) and the associated >25.0 minute RT requirement for each of the 12-hour CS3 calibration verification standards. The relative retention time and ion abundance criteria were also met for each of the native and labeled CDD/CDFs in the ongoing CS-3 standard results.

Data Validation Report
Laboratory Report / Batch E2300509
Received September 22, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report October 2 2003

The relative response factor (RRF) criterion of <25% difference between CS-3 native compounds as compared to the initial calibration data was acceptable for instrument A (August 23 and August 25), for instrument C (August 22 and September 15), and for instrument B (August 17, August 18, August 22, and August 23) analytical runs. For August 23-24, (instrument B), the exceedence of the 25% ending calibration verification standard criterion required the laboratory to provide the mean RRFs (using Form 6) from the beginning and ending calibration verification analysis and the mean factors were then employed for quantitation, as specified in Method 8290, Section 8.3.2.4 and 7.7.4.4. For the calibration verification of instrument B performed on September 15, the %D RRF criteria was exceeded for 1,2,3,7,8,9-HxCDF. However the data for 1,2,3,7,8,9-HxCDF from the September 15 run on instrument B was not used, since the samples were previously run and the 1,2,3,7,8,9-HxCDF data from the original run was used for the TEF calculations. No data qualifiers were assigned to the project data.

While the Guidelines indicate review of the relative responses (RR), Method 8290 has no such requirement, as RR's are not used in final quantitation of sample results, therefore, no review was performed.

Review of the above elements indicates a high level of instrument stability and no qualifiers are required.

Method Blank Analyses

Laboratory method blank analyses are used to determine the existence and magnitude of contamination introduced at the laboratory.

CAS's analysis of method blank samples met all acceptance criteria. No positive concentrations were reported in the method blanks above the CRQL. Trace concentrations of OCDD and HpCDD were detected in one or more of the method blanks associated with the analytical batch. However, associated sample concentrations are far greater than the blank concentration, therefore, no data requires qualification. All method blank samples were prepared and analyzed at the appropriate frequency.

Laboratory Control Samples (Ongoing Precision/Accuracy)

The laboratory is required to prepare and analyze a sample of spiked reference matrix to measure the accuracy of the analytical process/system method.

CAS performed laboratory control sample (LCS) analyses at the appropriate frequency for the analytical batch. All LCS results met associated acceptance criteria for percent recovery of the spiked concentrations as presented in Table 6 of Exhibit D of the Guidelines. All RRTs and IARs were also acceptable in the LCS samples associated with the analytical batch, indicating an in-control analytical system.

Labeled Compound Recovery (Surrogate Standard Recovery)

Because the introduced labeled compounds (CDD/CDFs) serve as the isotopic quantitative mechanism for this method, recoveries should be closely monitored for laboratory and method effectiveness.

CAS met the minimum acceptance criteria for the labeled compound recoveries as presented in Table 7 of Exhibit D of the Guidelines. These recovery windows are wider than CAS's internally generated acceptance criteria. Labeled compound recoveries qualified with a "Y" in the original data package indicate the recovery falls outside the laboratory-generated acceptance limits. None of these minor failures exceed the Guidelines limits; therefore, no data requires qualification.

Second Column Confirmation

A second column confirmation of the 2,3,7,8-TCDF isomer is required by both Methods 1613 and 8290 due to a known lack of isomer specificity for this isomer on the DB-5 or equivalent column. All operating conditions of the second column analytical system must be identical to those of the primary system.

CAS performed the second column confirmational analysis when the original analytical run had positive 2,3,7,8-TCDF detections. All performance criteria (as previously detailed above) were met prior to and during the confirmational analytical runs.



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Minneapolis MN • Hibbing MN • Duluth MN • Ann Arbor MI • Jefferson City MO

October 9, 2003

Sonia Vega
On-Scene Coordinator
U S Environmental Protection Agency, Region 5
Emergency Response Branch
520 Lafayette Road North
St Paul, MN 55155-4194

Re: Validated Analytical Data
Laboratory Batch Number – E2300519, K2306052, K2305943, K2306160
Docket No. V-W-'03-C-748
St. Regis Paper Company Site – Cass Lake, Minnesota

Dear Ms Vega:

Barr has validated the analytical data contained in the laboratory batch number(s) identified above. Enclosed are the following documents related to these analytical data

- Data Summary Table(s) {Attachment A}
- Validation Summary Report(s) {Attachment B}
- Complete analytical data package(s) from Columbia Analytical Services {Attachment C}

The data summary tables include the TEQ_{DF}-WHO₀₈ values calculated using the validated data. The TEQ calculation used a value of ½ the detection limit for any congener that was not detected.

All data met the data quality objectives and are useable as reported. If you have any questions concerning this information, please contact me at (952)832-2876 or Tom Ross of International Paper at (901)419-3899.

Sincerely,

Thomas D Mattison
Project Coordinator

cc: Tim Drexler, U.S. EPA – RPM (w/ Attachments A, B & C)
Mony Chabria and Tom Turner, U.S. EPA (w/ Attachments A & B)

Attachment A

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location	F27-29 0-4	F27-29 4-12	H25-26 0-4	I26-27 0-4	I27-29 0-4	J29-30 0-4
Date	8/6/2003	8/6/2003	8/5/2003	8/6/2003	8/6/2003	8/6/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	0.003	0.002	0.00055 j	<0.00034	0.00079 j	0.0017 EMPC
1,2,3,7,8-PeCDD	0.068	0.032	0.008	0.004	0.021	0.046
1,2,3,4,7,8-HxCDD	0.260	0.123	0.024	0.010	0.054	0.166
1,2,3,6,7,8-HxCDD	0.963	0.274 j	0.108	0.064	0.320	3.072
1,2,3,7,8,9-HxCDD	0.295 j	0.242	0.049	0.022	0.112	0.389
1,2,3,4,6,7,8-HpCDD	30.852	11.364	2.344	2.496	11.694	186.701
OCDD	249.540 e	133.473 e	22.122 e	24.152	124.075	2182.551 e
2,3,7,8-TCDF	0.026	0.006	0.001	<0.00098	0.004	0.014
1,2,3,7,8-PeCDF	0.111	0.064	0.008	0.007	0.030	0.123
2,3,4,7,8-PeCDF	0.113	0.065	0.010	0.009	0.035	0.140
1,2,3,4,7,8-HxCDF	0.723	0.460	0.144	0.061	0.308	2.625
1,2,3,6,7,8-HxCDF	0.233	0.128	0.032	0.016	0.076	0.262
1,2,3,7,8,9-HxCDF	0.041	<0.035	0.032	0.021	0.084 EMPC	0.063
2,3,4,6,7,8-HxCDF	0.366	0.229	0.056	0.026	0.129	0.887
1,2,3,4,6,7,8-HpCDF	7.167	2.489	0.530	0.450	2.713	28.274
1,2,3,4,7,8,9-HpCDF	0.639	0.447	0.080	0.045	0.207	2.125
OCDF	29.433	14.042	1.686	1.682	12.664	132.899
TCDD, Total	0.016	0.004	0.002	<0.00034	0.003	0.010
PeCDD, Total	0.279	0.111	0.039	0.014	0.082	0.183
HxCDD, Total	7.753	2.747	0.548	0.257	1.388	5.045
HpCDD, Total	75.414	46.496	6.977	4.105	23.525	82.565
TCDF, Total	0.143	0.052	0.010	0.004	0.047	0.116
PeCDF, Total	2.648	1.686	0.229	0.154	0.759	2.490
HxCDF, Total	7.251	4.152	1.909	0.926	5.721	8.468
HpCDF, Total	34.820	30.590	3.558	2.094	13.912	53.718
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.840	0.370	0.120	0.060	0.310	3.300

Data qualifiers and footnotes

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location	D25-26 0-4	DE8-9 0-4	DE8-9 4-12	E29-30 0-4	F22-23 0-4	J26-27 0-4
Date	8/8/2003	8/8/2003	8/8/2003	8/8/2003	8/8/2003	8/8/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	<0.0003	<0.0006	<0.00052	0.006	0.002	0.002
1,2,3,7,8-PeCDD	0.021	0.017	0.011	0.143	0.050	0.083
1,2,3,4,7,8-HxCDD	0.049	0.067	0.046	0.438	0.143	0.313
1,2,3,6,7,8-HxCDD	0.404	0.206	0.132	1.602	0.402	5.251
1,2,3,7,8,9-HxCDD	0.135	0.130	0.090	0.748	0.313	0.830
1,2,3,4,6,7,8-HpCDD	12.855	8.516	6.274	49.437	13.533	158.645
OCDD	133.391	55.358 e	45.773 e	428.350	133.542	1504.395 e
2,3,7,8-TCDF	0.004	0.001	0.0009 EMPC	0.014	0.003	0.065
1,2,3,7,8-PeCDF	0.038	0.006	0.004	0.086	0.017	0.412
2,3,4,7,8-PeCDF	0.033	0.007	0.005	0.094	0.019	0.755
1,2,3,4,7,8-HxCDF	0.372	0.068	0.053	0.617 EMPC	0.228	9.161
1,2,3,6,7,8-HxCDF	0.112	0.023	0.017	0.309	0.080	1.489
1,2,3,7,8,9-HxCDF	<0.0269	0.002	0.0016 j EMPC	0.030	0.029	0.079
2,3,4,6,7,8-HxCDF	0.176	0.045	0.036	0.566	0.151	2.451
1,2,3,4,6,7,8-HpCDF	3.413	1.131	0.949	12.178	2.411	50.506
1,2,3,4,7,8,9-HpCDF	0.360	0.106	0.075	0.806	0.276	4.332
OCDF	12.861	5.449	4.530	58.002	11.891	209.140 e
TCDD, Total	0.002	<0.0006	<0.00052	0.031	0.006	0.027
PeCDD, Total	0.082	0.074	0.046	0.547	0.175	0.253
HxCDD, Total	1.766	2.062	1.151	8.027	2.704	12.278
HpCDD, Total	28.397	22.841	13.136	92.152	33.768	70.667
TCDF, Total	0.030	0.009	0.006	0.251	0.053	0.338
PeCDF, Total	0.753	0.190	0.156	3.336	0.764	9.483
HxCDF, Total	3.317	1.336	1.020	9.664	4.802	24.238
HpCDF, Total	17.592	5.412	3.816	45.124	15.592	47.376
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.346	0.179	0.130	1.305	0.374	4.753

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location	J26-27 4-12	J26-27 12-24	J27-29 0-4	J27-29 4-12	J27-29 12-24	SW-44 0-4
Date	8/8/2003	8/8/2003	8/8/2003	8/8/2003	8/8/2003	8/12/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	0.000554 _j	<0.00016	0.002	<0.000162	<0.00013	<0.00081
1,2,3,7,8-PeCDD	0.026	0.0019 _j	0.033	<0.00017	0.00055 _j	0.017
1,2,3,4,7,8-HxCDD	0.089	0.0086 EMPC	0.071	0.00076 _j	0.0014 _j	0.026
1,2,3,6,7,8-HxCDD	3.090	0.189	2.426	0.009	0.043	0.216
1,2,3,7,8,9-HxCDD	0.255	0.023	0.243	0.0011 _j	0.003	0.105
1,2,3,4,6,7,8-HpCDD	112.241	5.335	79.840 e	0.282	1.096	8.119
OCDD	1406.612 e	52.529 e	747.571 e	2.208 e	8.740	72.045
2,3,7,8-TCDF	0.037	0.004	0.010	<0.000163	<0.0012	<0.0010
1,2,3,7,8-PeCDF	0.209	0.023	0.127	<0.00016	0.0016 _j	0.008
2,3,4,7,8-PeCDF	0.308	0.029	0.181	0.00054 _j	0.003	0.008
1,2,3,4,7,8-HxCDF	4.746	0.252	2.798	0.008	0.036	0.081
1,2,3,6,7,8-HxCDF	0.757	0.048	0.386	0.0013 _j	0.006	0.026
1,2,3,7,8,9-HxCDF	0.049	<0.0103	0.033 EMPC	<0.00061	<0.00061	<0.016
2,3,4,6,7,8-HxCDF	1.382	0.070	0.844	0.003	0.012	0.054
1,2,3,4,6,7,8-HpCDF	30.098	1.515	28.143	0.116	0.490	0.967
1,2,3,4,7,8,9-HpCDF	2.502	0.089	2.203	0.012	0.062	0.184
OCDF	171.091 e	9.036	154.944 e	0.624	2.748	6.763
TCDD, Total	0.009	0.001	0.018	<0.00016	<0.00013	<0.00081
PeCDD, Total	0.070	0.003	0.138	<0.00017	0.001	0.050
HxCDD, Total	5.265	0.487	5.660	0.021	0.095	0.923
HpCDD, Total	71.507	9.989	66.753	0.461	2.229	14.968
TCDF, Total	0.144	0.015	0.113	<0.00016	0.004	0.010
PeCDF, Total	3.986	0.420	2.748	0.006	0.033	0.195
HxCDF, Total	13.173	2.976	11.552	0.124	0.608	1.545
HpCDF, Total	42.756	8.349	39.244	0.572	3.440	6.775
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	2.837	0.153	2.005	0.007	0.029	0.177

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location	SW-7 0-4	SW-7 4-12	SW-41 0-4	SW-42 0-4	FOA-02-1-0-4	FOA-02-2-0-4
Date	8/11/2003	8/11/2003	8/11/2003	8/12/2003	8/12/2003	8/12/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	<0.00081	<0.000528	0.002	<0.000468	0.004	0.001
1,2,3,7,8-PeCDD	0.041	0.017	0.041	0.021	0.089	0.013
1,2,3,4,7,8-HxCDD	0.127	0.054	0.138	0.068	0.288	0.035
1,2,3,6,7,8-HxCDD	1.006 _j	0.774 _j	1.019 _j	0.467	0.774	0.289
1,2,3,7,8,9-HxCDD	0.334	0.165	0.348	0.192	0.352 _j	0.093
1,2,3,4,6,7,8-HpCDD	51.621	45.229	55.172	12.112	27.598	4.794
OCDD	459.098	439.818	517.194 _e	115.280	238.156	45.6 _b
2,3,7,8-TCDF	0.007	0.005	0.013	0.005	0.008	0.006
1,2,3,7,8-PeCDF	0.023	0.018	0.041	0.019	0.049	0.026
2,3,4,7,8-PeCDF	0.034	0.026	0.064	0.024	0.060	0.031
1,2,3,4,7,8-HxCDF	0.279	0.226	0.426	0.134	0.323 _j EMPC	0.300
1,2,3,6,7,8-HxCDF	0.063	0.036	0.079	0.036	0.202	0.071
1,2,3,7,8,9-HxCDF	<0.0216	<0.022	0.010	0.004	0.106 EMPC	0.007 EMPC
2,3,4,6,7,8-HxCDF	0.142	0.082	0.183	0.065	0.342	0.118
1,2,3,4,6,7,8-HpCDF	5.202	4.306	6.214	1.506	4.924	1.158
1,2,3,4,7,8,9-HpCDF	0.329 _j EMPC	<0.664	<1.040	0.187	<0.850	0.191
OCDF	38.912	36.458	45.538	8.344	26.012	4.099
TCDD, Total	0.007	0.007	0.022	0.005	0.016	0.002
PeCDD, Total	0.208	0.094	0.223	0.114	0.269	0.049
HxCDD, Total	4.712	3.459	5.779	2.600	5.161	1.043
HpCDD, Total	81.045	69.661	99.057	32.082	33.783	12.457
TCDF, Total	0.078	0.053	0.164	0.046	0.077	0.031
PeCDF, Total	0.733	0.489	1.127	0.511	1.462	0.560
HxCDF, Total	5.757	4.649	3.168	2.720	5.208	3.822
HpCDF, Total	35.423	32.188	47.660	10.232	19.113	7.067
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.878	0.713	0.974	0.282	0.721	0.189

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (mutual or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	FOA-02-2-4-12 8/12/2003 CAS	FOA-02-3-0-4 8/12/2003 CAS	FOA-02-4-0-4 8/12/2003 CAS	FOA-02-5-0-4 8/12/2003 CAS	FOA-02-6-0-4 8/12/2003 CAS	FOA-02-6-4-12 8/12/2003 CAS
					0 00083 j EMPC	<0 000111
2,3,7,8-TCDD	<0 000201	<0 000131	<0 000193	0 001		
1,2,3,7,8-PeCDD	0 006	0 003	0 008	0 014	0 010	0 000995 j
1,2,3,4,7,8-HxCDD	0 018	0 012	0 029	0 031	0 029	0 003
1,2,3,6,7,8-HxCDD	0 094	0 041	0 204	0 093	0 086	0 010
1,2,3,7,8,9-HxCDD	0 045	0 021	0 062	0 074	0 080	0 008
1,2,3,4,6,7,8-HpCDD	4 349	1 881	6 506	3 709	3 315	0 408
OCDD	41.2 b	15 875	58 9 e	32 69 b	27 0 eb	3 31 e
2,3,7,8-TCDF	<0 00066 j	0 001	0 001	<0 00035	<0 00044 j	<0 000098
1,2,3,7,8-PeCDF	0 006	0 0010 j	0 009	0 0015 j	0 0021 j	0 00024 j EMPC
2,3,4,7,8-PeCDF	0 006	0 0013 j	0 010	0 0013 j	0 0020 j	0 00030 j EMPC
1,2,3,4,7,8-HxCDF	0 072	0 017	0 115	0 018	0 022	0 003
1,2,3,6,7,8-HxCDF	0 021	0 006	0 028	0 011	0 012	0 0015 j
1,2,3,7,8,9-HxCDF	0 002 j	0 002 j	<0 00011	0 0032	<0 0013	<0 00034
2,3,4,6,7,8-HxCDF	0 035	0 011	0 050	0 025	0 022	0 0029 j
1,2,3,4,6,7,8-HpCDF	0 960	0 274	1 227	0 417	0 440	0 066
1,2,3,4,7,8,9-HpCDF	0 062	0 026	0 112	0 050	0 046	0 007
OCDF	4 237	1 563	7 276	2 550	2 750	0 319
TCDD, Total	0 001	<0 00013	<0 00019	0 006	0 021	<0 000111
PeCDD, Total	0 021	0 007	0 024	0 055	0 114	0 007
HxCDD, Total	0 402	0 181	0 694	0 488	0 698	0 063
HpCDD, Total	5 318	2 694	11 401	5 759	5 276	0 655
TCDF, Total	0 007	0 003	0 005	0 007	0 013	0 001
PeCDF, Total	0 142	0 040	0 178	0 061	0 081	0 009
HxCDF, Total	1 048	0 290	0 746	0 422	0 465	0 067
HpCDF, Total	2 834	1 174	6 015	1 745	1 788	0 265
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 097	0 038	0 147	0 087	0 078	0 009

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 3
Validated PAH/PCP Concentrations in Groundwater
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/L)

Location	102 Basswood	104 Norway	127 1st	15611 61st	218 Elm	233 2nd St.	514 1st St.	514 1st St.	521 1st
Date	8/9/2003	8/9/2003	8/9/2003	8/9/2003	8/9/2003	8/15/2003	8/12/2003	8/12/2003	8/9/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS
Dup								DUP	
Benzo(a)anthracene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Chrysene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Benzo(b)fluoranthene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Benzo(k)fluoranthene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Benzo(a)pyrene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Indeno(1,2,3-cd)pyrene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Dibenz(a,h)anthracene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Naphthalene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
2-Methylnaphthalene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Acenaphthylene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Acenaphthene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Fluorene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Phenanthrene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Anthracene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Fluoranthene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Pyrene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Benzo(g,h,i)perylene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Pentachlorophenol	<0.60	<0.60	<0.60	<0.60	<0.60 *	<0.60	<0.60	<0.60	<0.60

* - estimated value QA/QC value not met

Attachment B

**Data Validation Report – Dioxin/Furan
Laboratory Report / Batch E2300519
Received September 25, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Report: October 7, 2003

The data validation of the Columbia Analytical Services, Inc (CAS) laboratory data for the dioxin and furan (CDD/CDF) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete as detailed below

The analytical data were reviewed in accordance with the U S EPA Analytical Operations Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, Draft Final dated March, 2002 (Guidelines) as specified in the project-specific Quality Assurance Project Plan (QAPP, Barr, June 2003) In addition to the Guidelines, specific SW-846 Method 8290 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include

- Overall assessment
- Holding times, preservation and storage
- Mass calibration and mass spectrometer resolution
- *Window defining mix*
- Instrument stability
- Initial calibration and ongoing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Second column confirmation

Data Validation Report
Laboratory Report / Batch E2300519
Received September 25, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report October 7, 2003

Fourteen soil sample results are contained in this laboratory report. They are as follows

SW-7 0-4"	SW-7 4-12"	SW-41 0-4"	SW-42 0-4"	SW 44 0-4"
SW-7 4-12"D	FOA-02-1 0-4"	FOA-02-2 0-4"	FOA 02-2 4-12"	FOA-02-3 0-4"
FOA-02-4 0-4"	FOA-02-5 0-4"	FOA-02-6 0-4"	FOA-02-6 4-12"	

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report E2300519 as a result of the data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected August 11th and 12th, 2003, cooled to 4°C and sent to the laboratory. Per the chain-of-custody and subsequent laboratory acknowledgement receipt forms, the laboratory received the samples were received in acceptable condition. It should be noted that discrepancies between Method 8290 and the Guidelines exist for the technical extraction and analysis holding times. The Guidelines recommend that soil samples be extracted within 10 days of receipt and analyzed within 30 days. Alternately, Method 8290 and the QAPP for the project indicate that 30 day extraction/45 day analysis holding times apply. Method 8290 also states that these holding times are only recommendations as dioxins and furans are very stable in a variety of matrices. All samples were extracted and analyzed within 30 days. No qualifiers are applied.

Mass Calibration and Mass Spectrometer Resolution

Mass calibration and mass spectrometer resolution (MC/MSR) checks are required to ensure that the mass calibration and mass spectrometer resolution is set at a $\geq 10,000$ resolving power. This resolution is confirmed at the beginning and end of every 12-hour analysis period.

It should be noted that the Guidelines contain language that refers to evaluation techniques that due to upgrades in analytical systems software, are obsolete at CASs Houston facility. Specifically, the evaluation of the resolution by the measure of deviation between the exact m/z and the theoretical

m/z at less than 5 ppm has been replaced with an instrumentation function that implicitly sets the error to zero at all calibration points so there is no longer any need to use peak matching conditions to verify the exact mass. Therefore, resolving power is evaluated by close review of the resolution of PFK peak profiles where high mass ion (380 9760) and low mass ion (304 9824) are reported.

CAS performed the MC/MSR at the appropriate frequency and obtained acceptable results for the PFK calibrant confirming MC/MSR at a resolving power of 10,000.

Window Defining Mix

The window defining mix (WDM) is necessary to establish the appropriate switching times for the selection ion group descriptors. As stated in the Guidelines, the frequency of the WDM is once every 12 hours prior to calibration verification. However, Method 8290, Section 8.3.2.2.2 allows the laboratory, if running consecutive 12-hour shifts, to use the ending calibration verification (if all acceptance criteria are met) of the first 12-hour analysis period as the beginning calibration verification of the second 12-hour analysis period. In these cases, the WDM was analyzed after the calibration verification but still falls within the 12-hour analytical period. While the Guidelines indicate the WDM must be analyzed prior to the calibration verification sample, CAS's order of analysis for the system performance check samples did not appear to affect the overall system instrumentation and no qualifiers have been applied.

CAS performed the WDM analysis for every 12-hour sample analysis period and presented the acceptable switching times for each homologue group on the corresponding Form 5s.

Chromatographic Resolution

This check is performed to ascertain the separation of closely eluting dioxin/furan isomers. This is performed using SICP (selected ion current profile) of each isomer. The criteria requires that the 2,3,7,8-TCDD and the 1,2,3,8-TCDD peaks (for the DB-5 column) and the 2,3,7,8-TCDF and the 2,3,4,7-TCDF peaks (for the DB-225 column) be resolved with a valley of $\leq 25\%$.

CAS provided summary peak to valley results as specified above on each of Form 5. In each case, the $\leq 25\%$ criterion was met. The raw chromatograms were also reviewed to confirm this information.

Instrument Stability

Instrument stability is an important aspect of this analytical system. Ongoing calibration verification using a CS-3 standard is performed once every 12-hour period. This standard is used to evaluate the isomer retention times, ion abundance criteria, sensitivity and ongoing calibration criteria.

CAS met the requirements of the absolute retention times (RT) for the first internal standard (13C-1,2,3,4-TCDD) of ± 15 seconds (of the initial calibration standard) and the associated >25.0 minute RT requirement for each of the 12-hour CS3 calibration verification standards. The relative retention time and ion abundance criteria were also met for each of the native and labeled CDD/CDFs in the ongoing CS-3 standard results.

Signal-to-noise (S/N) ratios were summarized for the native and labeled CDD/CDFs were acceptable at >10:1.

The relative response factor (RRF) criterion of <25% difference between CS-3 native compounds as compared to the initial calibration data was acceptable for the August 22, August 27, August 28, and September 9, analytical runs. For August 27-28 (second 12 hour run), September 3, September 4, and September 11-12 (second 12 hour run), August 23 and August 22 analytical runs, exceedence of the 25% ending calibration verification standard criterion required the laboratory to provide the mean RRFs from the beginning and ending calibration verification analysis and those factors were then employed for quantitation, as specified in Method 8290, Section 8.3.2.4 and 7.7.4.4.

While the Guidelines indicate review of the relative responses (RR), Method 8290 has no such requirement, as RR's are not used in final quantitation of sample results, therefore, no review was performed.

Review of the above elements indicates a high level of instrument stability and no qualifiers are required.

Initial Calibration and Calibration Verification

Satisfactory instrument calibration is crucial to ensuring the accurate qualitative and quantitative results for each of the CDD/CDF compounds. Initial calibration procedures define the linear range and mean relative response factors that will be used for sample quantitation.

Data Validation Report
Laboratory Report / Batch E2300519
Received September 25, 2003

St. Regis Paper Company Superfund Site - Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report: October 7, 2003

CAS's initial calibration summary information met all the relevant acceptance criteria including the relative responses (RRFs) of <25% for native compounds and <35% for the labeled compounds, ion abundance ratios (IARs) within +/- 15%, absolute retention times within the WDM windows, and signal-to-noise ratios of >10:1.

Calibration verification (or continuing calibration) summary information also met all relevant acceptance criteria including the frequency of the continuing calibration verifications at both the beginning and ending of each 12-hour analytical run, the RRFs %D within <25% for native compounds and <35% for labeled compounds of initial calibration (or alternate Form 5 used for average RRF as in Instrument Stability section), the IARs within the +/-15%, and signal-to-noise ratios of >10:1.

Method Blank Analyses

Laboratory method blank analyses are used to determine the existence and magnitude of contamination introduced at the laboratory.

CAS's analysis of method blank samples met all acceptance criteria. No positive concentrations were reported in the method blanks above the CRQL. Trace concentrations of OCDD and HpCDD were detected in one or more of the method blanks associated with the analytical batch. However, associated sample concentrations are far greater than the blank concentration, therefore, no data requires qualification. All method blank samples were prepared and analyzed at the appropriate frequency.

Laboratory Control Samples (Ongoing Precision/Accuracy)

The laboratory is required to prepare and analyze a sample of spiked reference matrix to measure the accuracy of the analytical process/system/method.

CAS performed laboratory control sample (LCS) analyses at the appropriate frequency for the analytical batch. LCS results met associated acceptance criteria for percent recovery of the spiked concentrations as presented in Table 6 of Exhibit D of the Guidelines. All RRTs and IARs were also acceptable in the LCS samples associated with the analytical batch indicating an in-control analytical system.

Labeled Compound Recovery (Surrogate Standard Recovery)

Because the introduced labeled compounds (CDD/CDFs) serve as the isotopic quantitative mechanism for this method, recoveries should be closely monitored for laboratory and method effectiveness.

CAS met the minimum acceptance criteria for the labeled compound recoveries as presented in Table 7 of Exhibit D of the Guidelines. These recovery windows are wider than CAS's internally generated acceptance criteria. Labeled compound recoveries qualified with a "Y" in the original data package indicate the recovery falls outside the laboratory-generated acceptance limits. None of these minor failures exceed the Guidelines limits, therefore, no data requires qualification.

Second Column Confirmation

A second column confirmation of the 2,3,7,8-TCDF isomer is required by both Methods 1613 and 8290 due to a known lack of isomer specificity for this isomer on the DB-5 or equivalent column. All operating conditions of the second column analytical system must be identical to those of the primary system.

CAS performed the second column confirmational analysis when the original analytical run had positive 2,3,7,8-TCDF detections. All performance criteria (as previously detailed above) were met prior to and during the confirmational analytical runs.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2305943
Received September 24, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: October 8, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment groundwater samples contained in the aforementioned report is complete as detailed below

The analytical data were reviewed in accordance with the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

Six water sample results are contained in this laboratory report. They are as follows:

**15611 61st
127 1st**

**218 Elm
102 Basswood**

521 1st

104 Norway

Data Validation Report
Laboratory Report / Batch K2305943
Received September 24, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: October 8, 2003

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2305943 as a result of data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected on August 11, 2003 and received at the CAS Kelso laboratory on August 12, 2003 with an accompanying chain-of-custody (COC) form. All samples were received intact and were properly chilled (within 2-6 °C).

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs and PCP instrument using selected ion monitoring (SIM) was performed on August 15, 2003 using 8-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05. All target analytes met the <30 %RSD data validation criteria. The average RSD of the RRFs for PCP (21.0%) did exceed the method criteria of <15%RSD, however it meets the Guideline criteria of 25%. Following their SOP CAS utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value

Data Validation Report
Laboratory Report / Batch K2305943
Received September 24, 2003

St. Regis Paper Company Superfund Site - Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: October 8, 2003

of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being <20%. The mean RSD for this initial calibration event was calculated as 6.0%, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard, all target analytes met the laboratory's criteria of < 20% difference. There are no data validation criteria for second source standards.

Continuing Calibration Verification

The August 20, 2003 continuing calibration verifications met the data validation criteria for frequency and the <+25% difference RRFs from ICAL as well as the minimum RRFs of > 0.05 for all target analytes except indeno(1,2,3-cd)pyrene. The laboratory reanalyzed the samples later under continuing calibration conditions that did meet acceptance criteria for indeno(1,2,3-cd)pyrene. The indeno(1,2,3-cd)pyrene sample results are quantitated from the analytical run associated with acceptable continuing calibration verification. Similarly, control criterion was exceeded in subsequent continuing calibration verification standards; however, the project samples analyzed in the sequence did not contain these target analytes (pentachlorophenol and pyrene) and the error equates to a potential high bias, no action/qualifiers are applied.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Surrogate Standard Recovery

Slightly higher than expected surrogate recoveries were reported for the matrix spike and the laboratory control sample for the fluorene-d10 surrogate (102%, and 103%, respectively). The

Data Validation Report
Laboratory Report / Batch K2305943
Received September 24, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report October 8, 2003

Guidelines indicate that one surrogate can fall outside acceptance criteria if $>10\%$. Because surrogate recoveries were $>10\%$ and that the error equates to a potential high bias and the nominal degree in which the recoveries were out, no qualifiers are required. All other surrogate spike recoveries from the project samples met the data validation acceptance criteria.

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project samples and associated quality control samples met the method and data validation criteria.

Matrix Spike Recovery

A sample from laboratory batch K2306052 served as the Matrix Spike (MS) and Matrix Spike Duplicate (MSD) for this analytical batch. All the spike and spike duplicate recoveries and RPDs met the laboratory generated acceptance criteria.

Field Duplicate Results

No field duplicate samples are associated with this analytical batch.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306052
Received September 24, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: October 8, 2003

The data validation process for the Columbia Analytical Services, Inc. (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment groundwater samples contained in the aforementioned report is complete as detailed below.

The analytical data were reviewed in accordance with the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

Three water sample results are contained in this laboratory report. They are as follows:

514 1st St. M-1 (514 1st Duplicate) FB-1

Data Validation Report
Laboratory Report / Batch K2306052
Received September 24 2003

St Regis Paper Company Superfund Site - Cass Lake Minnesota
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Date of Validation Report October 8 2003

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2306052 as a result of data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected on August 12-13, 2003 and received at the CAS Kelso laboratory on August 14, 2003 with an accompanying chain-of-custody (COC) form. All samples were received intact and were properly chilled (within 2-6 °C).

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs and PCP instrument using selected ion monitoring (SIM) was performed on August 15, 2003 using 8-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05. All target analytes met the <30 %RSD data validation criteria. The average RSD of the RRFs for PCP (21.0%) did exceed the method criteria of <15%RSD, however it meets the Guideline criteria of 25%. Following their SOP CAS utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value

Data Validation Report
Laboratory Report / Batch K2306052
Received September 24 2003

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of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being <20%. The mean RSD for this initial calibration event was calculated as 6.0%, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard. All target analytes met the laboratory's criteria of < 20% difference. There are no data validation criteria for second source standards.

Continuing Calibration Verification

The August 20, 2003 continuing calibration verifications met the data validation criteria for frequency and the <+25% difference RRFs from ICAL as well as the minimum RRFs of > 0.05 for all target analytes except indeno(1,2,3-cd)pyrene. The laboratory reanalyzed the samples later under continuing calibration conditions that did meet acceptance criteria for indeno(1,2,3-cd)pyrene. The indeno(1,2,3-cd)pyrene sample results are quantitated from the analytical run associated with acceptable continuing calibration verification. Similarly, control criterion was exceeded in subsequent continuing calibration verification standards; however, the project samples analyzed in the sequence did not contain these target analytes (pentachlorophenol and pyrene) and the error equates to a potential high bias. No action/qualifiers are applied.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Surrogate Standard Recovery

Slightly higher than expected surrogate recoveries were reported for samples FB-1, the matrix spike and the laboratory control sample for the fluorene-d10 surrogate (99%, 102%, and 103%,

Data Validation Report
Laboratory Report / Batch K2306052
Received September 24, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

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respectively) The Guidelines indicate that one surrogate can fall outside acceptance criteria if >10%. Because surrogate recoveries were >10% and that the error equates to a potential high bias and the nominal degree in which the recoveries were out, no qualifiers are required. All other surrogate spike recoveries from the project samples met the data validation acceptance criteria.

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project samples and associated quality control samples met the method and data validation criteria.

Matrix Spike Recovery

The sample 514 1st St served as the Matrix Spike (MS) and Matrix Spike Duplicate (MSD) for this analytical batch. All the spike and spike duplicate recoveries and RPDs met the laboratory generated acceptance criteria.

Field Duplicate Results

Field duplicate results evaluate overall sampling and laboratory precision. Sample 514 1st St was collected in duplicate (labeled M-1). Both the native and duplicate sample concentrations were non-detect therefore, no RPD calculations were performed. These results displayed an acceptable level of precision for the low level nature of the analytical method and overall sampling procedures.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306160
Received September 24, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: October 8, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment groundwater samples contained in the aforementioned report is complete as detailed below.

The analytical data were reviewed in accordance with the U S EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003) In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

One water sample results are contained in this laboratory report. It is as follows

233 2nd St.

Data Validation Report
Laboratory Report / Batch K2306160
Received September 24, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: October 8, 2003

Overall Assessment

Due to low matrix spike and matrix spike duplicate and laboratory control sample percent recoveries, the result for 233 2nd St pentachlorophenol has been qualified with an "*" indicating an estimated value, QA/QC criteria not met. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The sample was collected on August 15, 2003 and received at the CAS Kelso laboratory on August 19, 2003 with an accompanying chain-of-custody (COC) form. The sample was received intact and was properly chilled (within 2-6 °C). The sample was properly stored until shipment.

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs and PCP instrument using selected ion monitoring (SIM) was performed on September 3, 2003 using 8-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05. All target analytes met the <30 %RSD data validation criteria. The average RSD of the RRFs for PCP (26.0%) did exceed the method criteria of <15%RSD, however the laboratory utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA

Data Validation Report
Laboratory Report / Batch K2306160
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Date of Validation Report October 8, 2003

8000B. This option allows for the calculation of the mean value of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being <20%. The mean RSD for this initial calibration event was calculated as 7.4%, thus meeting the method criteria

The laboratory report also included results from the analysis of a second source calibration check standard, all target analytes met the laboratory's criteria of < 20% difference. There are no data validation criteria for second source standards.

Continuing Calibration Verification

The September 4, 2003 continuing calibration verifications met the data validation criteria for frequency and the <+25% difference RRFs from ICAL as well as the minimum RRFs of > 0.05 for all target analytes.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria with the exception of pentachlorophenol. The percent recovery was lower than expected at 48%. The laboratory's narrative indicates that the acceptance window of 70-130% is temporary and default. CAS noted that they have insufficient data points available to generate final calculated statistical control limits and these percent recoveries were consistent and within the historical range expected for the procedure. Following Guidelines, the data are qualified accordingly in the data tables.

Surrogate Standard Recovery

All surrogate spike recoveries from the project samples met the data validation acceptance criteria.

Data Validation Report
Laboratory Report / Batch K2306160
Received September 24, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report October 8, 2003

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project sample and associated quality control samples met the method and data validation criteria.

Matrix Spike Recovery

The sample 233 2nd St served as the Matrix Spike (MS) and Matrix Spike Duplicate (MSD). The percent recoveries were lower than expected at 52% and 55%. The RPD was acceptable at 4%. However, because the same variability exists in the laboratory control sample, the corresponding sample result is qualified as “*” indicating an estimated value as QA/QC criteria was not met. All percent recoveries and RPDs from the MS and MSD were within the laboratory’s internal control limits.

Field Duplicate Results

No field duplicate samples were associated with this analytical batch.



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October 22, 2003

Sonia Vega
On-Scene Coordinator
U.S. Environmental Protection Agency, Region 5
Emergency Response Branch
520 Lafayette Road North
St. Paul, MN 55155-4194

Re: Validated Analytical Data
Laboratory Batch Number – E2300523
Docket No. V-W-'03-C-748
St. Regis Paper Company Site – Cass Lake, Minnesota

Dear Ms. Vega:

Barr has validated the analytical data contained in the laboratory batch number(s) identified above. Enclosed are the following documents related to these analytical data:

- Data Summary Table(s) {Attachment A}
- Validation Summary Report(s) {Attachment B}
- Complete analytical data package(s) from Columbia Analytical Services {Attachment C}

The data summary tables include the TEQ_{DF}-WHO₉₈ values calculated using the validated data. The TEQ calculation used a value of ½ the detection limit for any congener that was not detected.

All data met the data quality objectives and are useable as reported.

If you have any questions concerning this information, please contact me at (952)832-2876 or Tom Ross of International Paper at (901)419-3899.

Sincerely,

Thomas D. Mattison
Project Coordinator

cc: Tim Drexler, U.S. EPA – RPM (w/ Attachments A, B & C)
Mony Chabria and Tom Turner, U.S. EPA (w/ Attachments A & B)

Attachment A

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	F27-29 0-4 8/6/2003 CAS	F27-29 4-12 8/6/2003 CAS	H25-26 0-4 8/5/2003 CAS	I26-27 0-4 8/6/2003 CAS	I27-29 0-4 8/6/2003 CAS	J29-30 0-4 8/6/2003 CAS
2,3,7,8-TCDD	0 003	0 002	0 00055 j	<0 00034	0 00079 j	0 0017 EMPC
1,2,3,7,8-PeCDD	0 068	0 032	0 008	0 004	0 021	0 046
1,2,3,4,7,8-HxCDD	0 260	0 123	0 024	0 010	0 054	0 166
1,2,3,6,7,8-HxCDD	0 963	0 274 j	0 108	0 064	0 320	3 072
1,2,3,7,8,9-HxCDD	0 295 j	0 242	0 049	0 022	0 112	0 389
1,2,3,4,6,7,8-HpCDD	30 852	11 364	2 344	2 496	11 694	186 701
OCDD	249 540 e	133 473 e	22 122 e	24 152	124 075	2182 551 e
2,3,7,8-TCDF	0 026	0 006	0 001	<0 00098	0 004	0 014
1,2,3,7,8-PeCDF	0 111	0 064	0 008	0 007	0 030	0 123
2,3,4,7,8-PeCDF	0 113	0 065	0 010	0 009	0 035	0 140
1,2,3,4,7,8-HxCDF	0 723	0 460	0 144	0 061	0 308	2 625
1,2,3,6,7,8-HxCDF	0 233	0 128	0 032	0 016	0 076	0 262
1,2,3,7,8,9-HxCDF	0 041	<0 035	0 032	0 021	0 084 EMPC	0 063
2,3,4,6,7,8-HxCDF	0 366	0 229	0 056	0 026	0 129	0 887
1,2,3,4,6,7,8-HpCDF	7 167	2 489	0 530	0 450	2 713	28 274
1,2,3,4,7,8,9-HpCDF	0 639	0 447	0 080	0 045	0 207	2 125
OCDF	29 433	14 042	1 686	1 682	12 664	132 899
TCDD, Total	0 016	0 004	0 002	<0 00034	0 003	0 010
PeCDD, Total	0 279	0 111	0 039	0 014	0 082	0 183
HxCDD, Total	7 753	2 747	0 548	0 257	1 388	5 045
HpCDD, Total	75 414	46 496	6 977	4 105	23 525	82 565
TCDF, Total	0 143	0 052	0 010	0 004	0 047	0 116
PeCDF, Total	2 648	1 686	0 229	0 154	0 759	2 490
HxCDF, Total	7 251	4 152	1 909	0 926	5 721	8 468
HpCDF, Total	34 820	30 590	3 558	2 094	13 912	53 718
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 840	0 370	0 120	0 060	0 310	3 300

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	D25-26 0-4 8/8/2003 CAS	DE8-9 0-4 8/8/2003 CAS	DE8-9 4-12 8/8/2003 CAS	E29-30 0-4 8/8/2003 CAS	F22-23 0-4 8/8/2003 CAS	J26-27 0-4 8/8/2003 CAS
2,3,7,8-TCDD	<0 0003	<0 0006	<0 00052	0 006	0 002	0 002
1,2,3,7,8-PeCDD	0 021	0 017	0 011	0 143	0 050	0 083
1,2,3,4,7,8-HxCDD	0 049	0 067	0 046	0 438	0 143	0 313
1,2,3,6,7,8-HxCDD	0 404	0 206	0 132	1 602	0 402	5 251
1,2,3,7,8,9-HxCDD	0 135	0 130	0 090	0 748	0 313	0 830
1,2,3,4,6,7,8-HpCDD	12 855	8 516	6 274	49 437	13 533	158 645
OCDD	133 391	55 358 e	45 773 e	428 350	133 542	1504 395 e
2,3,7,8-TCDF	0 004	0 001	0 0009 EMPC	0 014	0 003	0 065
1,2,3,7,8-PeCDF	0 038	0 006	0 004	0 086	0 017	0 412
2,3,4,7,8-PeCDF	0 033	0 007	0 005	0 094	0 019	0 755
1,2,3,4,7,8-HxCDF	0 372	0 068	0 053	0 617 EMPC	0 228	9 161
1,2,3,6,7,8-HxCDF	0 112	0 023	0 017	0 309	0 080	1 489
1,2,3,7,8,9-HxCDF	<0 0269	0 002	0 0016 j EMPC	0 030	0 029	0 079
2,3,4,6,7,8-HxCDF	0 176	0 045	0 036	0 566	0 151	2 451
1,2,3,4,6,7,8-HpCDF	3 413	1 131	0 949	12 178	2 411	50 506
1,2,3,4,7,8,9-HpCDF	0 360	0 106	0 075	0 806	0 276	4 332
OCDF	12 861	5 449	4 530	58 002	11 891	209 140 e
TCDD, Total	0 002	<0 0006	<0 00052	0 031	0 006	0 027
PeCDD, Total	0 082	0 074	0 046	0 547	0 175	0 253
HxCDD, Total	1 766	2 062	1 151	8 027	2 704	12 278
HpCDD, Total	28 397	22 841	13 136	92 152	33 768	70 667
TCDF, Total	0 030	0 009	0 006	0 251	0 053	0 338
PeCDF, Total	0 753	0 190	0 156	3 336	0 764	9 483
HxCDF, Total	3 317	1 336	1 020	9 664	4 802	24 238
HpCDF, Total	17 592	5 412	3 816	45 124	15 592	47 376
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 346	0 179	0 130	1 305	0 374	4 753

Data qualifiers and footnotes

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	J26-27 4-12 8/8/2003 CAS	J26-27 12-24 8/8/2003 CAS	J27-29 0-4 8/8/2003 CAS	J27-29 4-12 8/8/2003 CAS	J27-29 12-24 8/8/2003 CAS	SW-44 0-4 8/12/2003 CAS
2,3,7,8-TCDD	0 000554 j	<0 00016	0 002	<0 000162	<0 00013	<0 00081
1,2,3,7,8-PeCDD	0 026	0 0019 j	0 033	<0 00017	0 00055 j	0 017
1,2,3,4,7,8-HxCDD	0 089	0 0086 EMPC	0 071	0 00076 j	0 0014 j	0 056
1,2,3,6,7,8-HxCDD	3 090	0 189	2 426	0 009	0 043	0 216
1,2,3,7,8,9-HxCDD	0 255	0 023	0 243	0 0011 j	0 003	0 105
1,2,3,4,6,7,8-HpCDD	112 241	5 335	79 840 e	0 282	1 096	8 119
OCDD	1406 612 e	52 529 e	747 571 e	2 208 e	8 740	72 045
2,3,7,8-TCDF	0 037	0 004	0 010	<0 000163	<0 0012	<0 0010
1,2,3,7,8-PeCDF	0 209	0 023	0 127	<0 00016	0 0016 j	0 008
2,3,4,7,8-PeCDF	0 308	0 029	0 181	0 00054 j	0 003	0 008
1,2,3,4,7,8-HxCDF	4 746	0 252	2 798	0 008	0 036	0 081
1,2,3,6,7,8-HxCDF	0 757	0 048	0 386	0 0013 j	0 006	0 026
1,2,3,7,8,9-HxCDF	0 049	<0 0103	0 033 EMPC	<0 00061	<0 00061	<0 016
2,3,4,6,7,8-HxCDF	1 382	0 070	0 844	0 003	0 012	0 054
1,2,3,4,6,7,8-HpCDF	30 098	1 515	28 143	0 116	0 490	0 967
1,2,3,4,7,8,9-HpCDF	2 502	0 089	2 203	0 012	0 062	0 184
OCDF	171 091 e	9 036	154 944 e	0 624	2 748	6 763
TCDD, Total	0 009	0 001	0 018	<0 00016	<0 00013	<0 00081
PeCDD, Total	0 070	0 003	0 138	<0 00017	0 001	0 050
HxCDD, Total	5 265	0 487	5 660	0 021	0 095	0 923
HpCDD, Total	71 507	9 989	66 753	0 461	2 229	14 968
TCDF, Total	0 144	0 015	0 113	<0 00016	0 004	0 010
PeCDF, Total	3 986	0 420	2 748	0 006	0 033	0 195
HxCDF, Total	13 173	2 976	11 552	0 124	0 608	1 545
HpCDF, Total	42 756	8 349	39 244	0 572	3 440	6 775
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	2 837	0 153	2 005	0 007	0 029	0 177

Data qualifiers and footnotes

e - estimated value Analyte exceeds upper end of the linear calibration range

j - estimated value Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	SW-7 0-4 8/11/2003 CAS	SW-7 4-12 8/11/2003 CAS	SW-41 0-4 8/11/2003 CAS	SW-42 0-4 8/12/2003 CAS	FOA-02-1-0-4 8/12/2003 CAS	FOA-02-2-0-4 8/12/2003 CAS
2,3,7,8-TCDD	<0 00081	<0 000528	0 002	<0 000468	0 004	0 001
1,2,3,7,8-PeCDD	0 041	0 017	0 041	0 021	0 089	0 013
1,2,3,4,7,8-HxCDD	0 127	0 054	0 138	0 068	0 288	0 035
1,2,3,6,7,8-HxCDD	1 006 j	0 774 j	1 019 j	0 467	0 774	0 289
1,2,3,7,8,9-HxCDD	0 334	0 165	0 348	0 192	0 352 j	0 093
1,2,3,4,6,7,8-HpCDD	51 621	45 229	55 172	12 112	27 598	4 794
OCDD	459 098	439 818	517 194 e	115 280	238 156	45 6
2,3,7,8-TCDF	0 007	0 005	0 013	0 005	0 008	0 006
1,2,3,7,8-PeCDF	0 023	0 018	0 041	0 019	0 049	0 026
2,3,4,7,8-PeCDF	0 034	0 026	0 064	0 024	0 060	0 031
1,2,3,4,7,8-HxCDF	0 279	0 226	0 426	0 134	0 323 j EMPC	0 300
1,2,3,6,7,8-HxCDF	0 063	0 036	0 079	0 036	0 202	0 071
1,2,3,7,8,9-HxCDF	<0 0216	<0 022	0 010	0 004	0 106 EMPC	0 007 EMPC
2,3,4,6,7,8-HxCDF	0 142	0 082	0 183	0 065	0 342	0 118
1,2,3,4,6,7,8-HpCDF	5 202	4 306	6 214	1 506	4 924	1 158
1,2,3,4,7,8,9-HpCDF	0 329 j EMPC	<0 664	<1 040	0 187	<0 850	0 191
OCDF	38 912	36 458	45 538	8 344	26 012	4 099
TCDD, Total	0 007	0 007	0 022	0 005	0 016	0 002
PeCDD, Total	0 208	0 094	0 223	0 114	0 269	0 049
HxCDD, Total	4 712	3 459	5 779	2 600	5 161	1 043
HpCDD, Total	81 045	69 661	99 057	32 082	33 783	12 457
TCDF, Total	0 078	0 053	0 164	0 046	0 077	0 031
PeCDF, Total	0 733	0 489	1 127	0 511	1 462	0 560
HxCDF, Total	5 757	4 649	3 168	2 720	5 208	3 822
HpCDF, Total	35 423	32 188	47 660	10 232	19 113	7 067
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 878	0 713	0 974	0 282	0 721	0 189

Data qualifiers and footnotes

e - estimated value Analyte exceeds upper end of the linear calibration range

j - estimated value Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location	FOA-02-2-4-12	FOA-02-3-0-4	FOA-02-4-0-4	FOA-02-5-0-4	FOA-02-6-0-4	FOA-02-6-4-12
Date	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/12/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	<0 000201	<0 000131	<0 000193	0 001	0 00083 _j EMPC	<0 000111
1,2,3,7,8-PeCDD	0 006	0 003	0 008	0 014	0 010	0 000995 _j
1,2,3,4,7,8-HxCDD	0 018	0 012	0 029	0 031	0 029	0 003
1,2,3,6,7,8-HxCDD	0 094	0 041	0 204	0 093	0 086	0 010
1,2,3,7,8,9-HxCDD	0 045	0 021	0 062	0 074	0 080	0 008
1,2,3,4,6,7,8-HpCDD	4 349	1 881	6 506	3 709	3 315	0 408
OCDD	41 2	15 875	58 9 e	32 69	27 0 e	3 31 e
2,3,7,8-TCDF	<0 00066 _j	0 001	0 001	<0 00035	<0 00044 _j	<0 000098
1,2,3,7,8-PeCDF	0 006	0 0010 _j	0 009	0 0015 _j	0 0021 _j	0 00024 _j EMPC
2,3,4,7,8-PeCDF	0 006	0 0013 _j	0 010	0 0013 _j	0 0020 _j	0 00030 _j EMPC
1,2,3,4,7,8-HxCDF	0 072	0 017	0 115	0 018	0 022	0 003
1,2,3,6,7,8-HxCDF	0 021	0 006	0 028	0 011	0 012	0 0015 _j
1,2,3,7,8,9-HxCDF	0 002 _j	0 002 _j	<0 00011	0 0032	<0 0013	<0 00034
2,3,4,6,7,8-HxCDF	0 035	0 011	0 050	0 025	0 022	0 0029 _j
1,2,3,4,6,7,8-HpCDF	0 960	0 274	1 227	0 417	0 440	0 066
1,2,3,4,7,8,9-HpCDF	0 062	0 026	0 112	0 050	0 046	0 007
OCDF	4 237	1 563	7 276	2 550	2 750	0 319
TCDD, Total	0 001	<0 00013	<0 00019	0 006	0 021	<0 000111
PeCDD, Total	0 021	0 007	0 024	0 055	0 114	0 007
HxCDD, Total	0 402	0 181	0 694	0 488	0 698	0 063
HpCDD, Total	5 318	2 694	11 401	5 759	5 276	0 655
TCDF, Total	0 007	0 003	0 005	0 007	0 013	0 001
PeCDF, Total	0 142	0 040	0 178	0 061	0 081	0 009
HxCDF, Total	1 048	0 290	0 746	0 422	0 465	0 067
HpCDF, Total	2 834	1 174	6 015	1 745	1 788	0 265
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 097	0 038	0 147	0 087	0 078	0 009

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location	NWWD-04 0-4	NWWD-05 0-4	RES16A 0-4	RES16B 0-4	RES24 0-4	RES 24 4-12
Date	8/14/2003	8/14/2003	8/14/2003	8/14/2003	8/14/2003	8/14/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	<0 00029	<0 0002	0 0007 j EMPC	<0 0004	<0 0009	<0 0003
1,2,3,7,8-PeCDD	0 002 j	0 0012 j EMPC	0 017	0 005	0 004	0 001 j
1,2,3,4,7,8-HxCDD	0 006 EMPC	0 0031 EMPC	0 052	0 013	0 008	0 004
1,2,3,6,7,8-HxCDD	0 035	0 017	0 344	0 061	0 028	0 014
1,2,3,7,8,9-HxCDD	0 017	0 0091	0 125	0 034	0 019	0 008
1,2,3,4,6,7,8-HpCDD	0 929	0 550	10 281	1 511	0 975	0 447
OCDD	8 519	4 652 e	101 918 e	14 372	7 553	3 657 e
2,3,7,8-TCDF	<0 00097	<0 0004	0 003	<0 0009	<0 0009	<0 0003
1,2,3,7,8-PeCDF	0 002 j EMPC	0 0006 j EMPC	0 025	0 003 j	<0 0009	<0 0006
2,3,4,7,8-PeCDF	0 002 j	0 0010 j	0 028	0 004	<0 0008	<0 0006
1,2,3,4,7,8-HxCDF	0 027	0 010	0 292	0 039	0 010	0 007
1,2,3,6,7,8-HxCDF	0 008	0 0040	0 081	0 015	0 004	<0 005
1,2,3,7,8,9-HxCDF	0 005	0 002 j	0 057	0 010	<0 003	<0 007
2,3,4,6,7,8-HxCDF	0 014	0 0061	0 130	0 025	<0 003	<0 006
1,2,3,4,6,7,8-HpCDF	0 296	0 128	2 972	0 449	0 187	0 110
1,2,3,4,7,8,9-HpCDF	0 022	0 009	0 229	0 033	0 013	<0 004
OCDF	1 131	0 457	10 816 e	1 556 e	0 733	0 385
TCDD, Total	0 012	0 0004	0 003	<0 0004	<0 0009	<0 0003
PeCDD, Total	0 024	0 009	0 066	0 018	0 015	0 006
HxCDD, Total	0 178	0 097	1 340	0 338	0 180	0 088
HpCDD, Total	2 085	1 059	19 424	3 353	1 650	0 889
TCDF, Total	0 006	0 003	0 034	0 022	0 002	0 002
PeCDF, Total	0 065	0 029	0 613	0 166	0 033	0 034
HxCDF, Total	0 431	0 176	4 797	0 703	0 206	0 172
HpCDF, Total	1 217	0 488	12 789	1 704	0 750	0 393
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 0283	0 0143	0 287	0 0482	0 0243	0 0121

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
 St. Regis Paper Company Site
 Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	RES 28 0-4 8/14/2003 CAS	RES28 0-4D 8/14/2003 CAS	SWD-01 0-4 8/14/2003 CAS	SWD-02 0-4 8/14/2003 CAS	SWD-03 0-4 8/14/2003 CAS	SWD-04 0-4 8/14/2003 CAS
2,3,7,8-TCDD	<0 0002	<0 0008	0 0008 j	<0 0004	<0 0004	<0 0005
1,2,3,7,8-PeCDD	0 002 j	0 003 j	0 003	0 004	0 008	0 004
1,2,3,4,7,8-HxCDD	0 006	0 008	0 006	0 010	0 011	0 009
1,2,3,6,7,8-HxCDD	0 032	0 029	0 019	0 035	0 043	0 027
1,2,3,7,8,9-HxCDD	0 016	0 016	0 016	0 026	0 032	0 024
1,2,3,4,6,7,8-HpCDD	1 145	0 990	0 471	1 175	0 891	0 736
OCDD	9 748 e	9 166	3 169 e	9 804	7 074	5 350
2,3,7,8-TCDF	<0 0004	<0 0009	0 0006 j	0 0007 j	0 004	<0 0007
1,2,3,7,8-PeCDF	0 002 j	<0 0009	0 0009 j	0 002 j EMPC	0 0017 j EMPC	0 001 j
2,3,4,7,8-PeCDF	0 002 j	<0 0008	0 002 j	0 003	0 015	0 002 j
1,2,3,4,7,8-HxCDF	0 021	0 020	0 008	0 015	0 024	0 010
1,2,3,6,7,8-HxCDF	0 006 EMPC	0 006 EMPC	0 006	0 008	0 030	0 005
1,2,3,7,8,9-HxCDF	0 006	<0 006	0 002 j	0 003 j	0 009	0 002 j
2,3,4,6,7,8-HxCDF	0 013	0 009	0 011	0 016	0 080	0 009
1,2,3,4,6,7,8-HpCDF	0 262	0 270	0 115	0 211	0 220	0 156
1,2,3,4,7,8,9-HpCDF	0 017	0 021	0 007	0 015	0 017	0 011
OCDF	0 969	0 948	0 303	0 899	0 754	0 573
TCDD, Total	<0 0002	<0 0008	0 001	0 004	0 004	<0 0005
PeCDD, Total	0 011	0 006	0 012	0 022	0 052	0 025
HxCDD, Total	0 174	0 180	0 127	0 219	0 346	0 218
HpCDD, Total	1 970	1 904	0 885	2 058	1 766	1 498
TCDF, Total	0 007	<0 0009	0 026	0 053	0 266	0 016
PeCDF, Total	0 064	0 058	0 148	0 211	1 571	0 071
HxCDF, Total	0 355	0 344	0 223	0 344	1 064	0 205
HpCDF, Total	1 010	1 091	0 321	0 825	0 780	0 542
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 0289	0 0265	0 0182	0 0324	0 0508	0 0234

Data qualifiers and footnotes:

e - estimated value Analyte exceeds upper end of the linear calibration range

j - estimated value Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	SWD-05 0-4 8/14/2003 CAS	D20-21 0-4 8/13/2003 CAS	D27-29 0-4 8/13/2003 CAS	E11-13 0-4 8/13/2003 CAS	E13-15 0-4 8/13/2003 CAS
2,3,7,8-TCDD	<0 0003	0 005	0 001	0 003	<0 0002
1,2,3,7,8-PeCDD	0 002 j	0 105	0 026	0 061	0 010
1,2,3,4,7,8-HxCDD	0 004	0 334	0 083	0 170	0 025
1,2,3,6,7,8-HxCDD	0 020	2 314	0 406	1 368	0 274
1,2,3,7,8,9-HxCDD	0 014	0 756	0 186	0 401	0 067
1,2,3,4,6,7,8-HpCDD	0 908	72 773	13 044	51 460	9 581
OCDD	5 516	519 390 e	115 856	493 923 e	98 013 e
2,3,7,8-TCDF	0 0007 j	0 020	0 007	0 005	<0 0007
1,2,3,7,8-PeCDF	0 001 j	0 081	0 040	0 039	0 005
2,3,4,7,8-PeCDF	0 001 j	0 113	0 050	0 041	0 005
1,2,3,4,7,8-HxCDF	0 007	1 034	0 343	1 323	0 290
1,2,3,6,7,8-HxCDF	0 003	0 252	0 096	0 200	0 040
1,2,3,7,8,9-HxCDF	<0 002	0 020	<0 007	<0 000856	<0 001
2,3,4,6,7,8-HxCDF	0 005	0 432	0 160	0 386	0 072
1,2,3,4,6,7,8-HpCDF	0 108	12 966	3 285	12 807	2 720
1,2,3,4,7,8,9-HpCDF	0 006	0 981	0 250	1 514	0 455
OCDF	0 430	62 855	11 740	58 715	13 455
TCDD, Total	0 002	0 023	0 013	0 017	0 001
PeCDD, Total	0 017	0 340	0 094	0 208	0 039
HxCDD, Total	0 148	7 190	1 739	4 413	0 886
HpCDD, Total	1 844	34 277	21 470	37 461	17 735
TCDF, Total	0 019	0 123	0 034	0 086	0 016
PeCDF, Total	0 050	2 180	0 950	1 040	0 161
HxCDF, Total	0 136	6 584	5 123	5 772	4 504
HpCDF, Total	0 356	10 551	12 616	26 249	15 195
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 0193	1 610	0 361	1 180	0 229

Data qualifiers and footnotes:

e - estimated value Analyte exceeds upper end of the linear calibration range

j - estimated value Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location	E18-19 0-4	E18-19 4-12	E24-25 0-4	E24-25 4-12	E24-25 4-12D	GS-1
Date	8/13/2003	8/13/2003	8/13/2003	8/13/2003	8/13/2003	8/13/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	0.001	0.0007 _j	0.0007 _j	0.0004 _j	0.0006 _j	<0.0002
1,2,3,7,8-PeCDD	0.036	0.014	0.015	0.010	0.011	0.0009 _j
1,2,3,4,7,8-HxCDD	0.100	0.046	0.050	0.039	0.040	0.002 _j EMPC
1,2,3,6,7,8-HxCDD	0.517	0.303	0.489	0.625	0.516	0.008
1,2,3,7,8,9-HxCDD	0.230	0.099	0.097	0.094	0.101	0.006
1,2,3,4,6,7,8-HpCDD	17.327	9.884	15.946	18.757	15.029	0.236
OCDD	131.712	96.380 _e	173.869	196.286	153.060 _e	1.760
2,3,7,8-TCDF	0.002	0.001	0.006	0.005	0.006	<0.0002
1,2,3,7,8-PeCDF	0.033	0.008	0.057	0.092	0.076	<0.0002
2,3,4,7,8-PeCDF	0.013	0.008	0.046	0.051	0.054	<0.0002
1,2,3,4,7,8-HxCDF	0.168	0.094	0.488	0.629	0.510	<0.005
1,2,3,6,7,8-HxCDF	0.061	0.030	0.115	0.164	0.179	0.002 _j EMPC
1,2,3,7,8,9-HxCDF	<0.0003	<0.0005	0.015	0.038	0.036	<0.005
2,3,4,6,7,8-HxCDF	0.113	0.062	0.182	0.246	0.274	0.003 _j
1,2,3,4,6,7,8-HpCDF	3.061	1.418	4.221	5.503	4.360	0.047
1,2,3,4,7,8,9-HpCDF	0.253	0.133	0.338	0.555	0.379	0.003 _j
OCDF	15.518	7.294	13.069	14.100	11.844	0.125
TCDD, Total	0.005	0.011	0.015	0.003	0.003	<0.0002
PeCDD, Total	0.096	0.039	0.069	0.025	0.030	0.005
HxCDD, Total	2.011	1.138	1.615	1.720	1.879	0.043
HpCDD, Total	23.016	18.291	27.647	25.001	25.404	0.414
TCDF, Total	0.026	0.012	0.037	0.026	0.030	0.001
PeCDF, Total	0.438	0.204	0.800	0.890	0.927	0.030
HxCDF, Total	3.181	1.816	8.491	4.449	10.762	0.046
HpCDF, Total	11.554	7.110	16.143	17.161	17.821	0.153
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.386	0.208	0.409	0.494	0.423	0.006

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location	GS-1D	RES39 0-4	RES40 0-4
Date	8/13/2003	8/13/2003	8/13/2003
Lab	CAS	CAS	CAS
Dup			
2,3,7,8-TCDD	<0 0001	0 0004 j	0 0005 j EMPC
1,2,3,7,8-PeCDD	0 0006 j	0 004	0 009
1,2,3,4,7,8-HxCDD	0 001 j	0 009	0 027
1,2,3,6,7,8-HxCDD	0 005	0 028	0 101
1,2,3,7,8,9-HxCDD	0 004	0 023	0 068
1,2,3,4,6,7,8-HpCDD	0 134	0 671	2 958
OCDD	0 971	5 292	25 606
2,3,7,8-TCDF	<0 0006	<0 001	0 001
1,2,3,7,8-PeCDF	<0 0002	0 0009 j	0 003
2,3,4,7,8-PeCDF	<0 0002	0 001 j	0 004
1,2,3,4,7,8-HxCDF	<0 003	0 009	0 036
1,2,3,6,7,8-HxCDF	<0 003	0 005	0 015
1,2,3,7,8,9-HxCDF	<0 003	<0 0003	0 005
2,3,4,6,7,8-HxCDF	<0 003	0 008	0 024
1,2,3,4,6,7,8-HpCDF	0 028	0 160	0 632
1,2,3,4,7,8,9-HpCDF	<0 001	0 011	0 036
OCDF	0 100	0 612	3 174
TCDD, Total	0 001	0 000	0 004
PeCDD, Total	0 003	0 018	0 051
HxCDD, Total	0 033	0 177	0 601
HpCDD, Total	0 247	1 515	6 737
TCDF, Total	0 001	0 009	0 026
PeCDF, Total	0 007	0 049	0 165
HxCDF, Total	0 024	0 192	0 865
HpCDF, Total	0 099	0 595	2 483
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 004	0 022	0 079

e - estimated value Analyte exceeds upper end of the linear calibration range

EMPC - estimated maximum possible concentration

Attachment B

**Data Validation Report – Dioxin/Furan
Laboratory Report / Batch: E2300523
Received: October 3, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Data Validation Report: October 16, 2003

The data validation of the Columbia Analytical Services, Inc. (CAS) laboratory data for the dioxin and furan (CDD/CDF) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete and is detailed below.

The analytical data were reviewed in accordance with the U.S. EPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, Draft Final dated March, 2002 (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8290 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Mass calibration and mass spectrometer resolution
- Window defining mix
- Initial calibration
- Instrument stability and continuing calibration verification
- Method blank analysis
- Laboratory control samples
- Second column confirmation
- Matrix Spikes

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Laboratory Report / Batch: E2300523
Received: October 3, 2003

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Thirteen soil sample and two water sample results are contained in this laboratory report. They are as follows:

D27-29 0-4"	D20-21 0-4"	E18-19 0-4"	E18-19 4-12"
E13-15 0-4"	E11-13 0-4"	E24-25 0-4"	E24-25 4-12"
E24-25 4-12D"	E24-25-ER	GS-1	GS-1D
RES39 0-4"	RES40 0-4"	RES39-ER	

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report E2300523 as a result of data validation process. All data met the data quality objectives (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected August 13, 2003, cooled to 4°C and sent to the laboratory. Per the chain-of-custody and subsequent laboratory acknowledgement receipt forms, the laboratory received the samples August 15, 2003 in acceptable condition and at 4°C. It should be noted that discrepancies between method 8290 and the Guidelines exist for the technical extraction and analysis holding times. The Guidelines recommend that soil samples be extracted within 10 days of receipt and analyzed within 30 days. Alternately, Method 8290 and the QAPP for the project indicate that a 30-day extraction/45 day analysis holding times apply. Method 8290 also states that these holding times are only recommendations as dioxins and furans are very stable in a variety of matrices. All samples were extracted and analyzed within 30 days. No qualifiers are applied.

Mass Calibration and Mass Spectrometer Resolution

Mass calibration and mass spectrometer resolution (MC/MSR) checks are required to ensure that the mass calibration and mass spectrometer resolution is set at a $\geq 10,000$ resolving power. This resolution is confirmed at the beginning and end every 12-hour analysis period.

It should be noted that the Guidelines contain language that refer to evaluation techniques that due to upgrades in analytical systems software, are obsolete at CASs Houston facility. Specifically, the evaluation of the resolution by the measure of deviation between the exact m/z and the theoretical

m/z at less than 5 ppm has been replaced with a function of instrumentation that implicitly sets the error to zero at all calibration points so there is no longer any need to use peak matching conditions to verify the exact mass. Therefore, resolving power is evaluated by close review of the resolution of PFK peak profiles where high mass ion (380.9760) and low mass ion (304.9824) are reported.

CAS performed the MC/MSRs at the appropriate frequency and obtained acceptable results for the PFK calibrant confirming MC/MSRs at a resolving power of 10,000. Note: CAS used three instrument systems to analyze the project samples and provided appropriate documentation from the three systems in the raw data package. All system's documentation were evaluated during the data validation process.

Window Defining Mix

The window defining mix (WDM) is necessary to establish the appropriate switching times for the selection ion group descriptors. As stated in the Guidelines, the frequency of the WDM is every 12 hours prior calibration verification. However, Method 8290, Section 8.3.2.2.2. allows the laboratory, if running consecutive 12-hour shifts, to use the ending calibration verification (if all acceptance criteria is met) of the first 12-hour analysis period as the beginning calibration verification of the second 12-hour analysis period. In these cases, the WDM was analyzed after the calibration verification but still falls within the 12-hour analytical period. While the Guidelines indicate the WDM must be analyzed prior to the calibration verification sample, CAS's order of analysis for the system performance check samples did not appear to affect the overall system instrumentation and no qualifiers have been applied.

CAS performed the WDM analysis for every 12-hour sample analysis period and presented the acceptable switching times for each homologue group on the corresponding Form 5s.

Chromatographic Resolution

This check is performed to ascertain the separation of closely eluting dioxin/furan isomers. This is performed using SICP (selected ion current profile) of each isomer. The criteria require that the 2,3,7,8-TCDD peak and the 1,2,3,8-TCDD peak be resolved with a valley of $\leq 25\%$ for the DB-5 column.

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CAS provided summary peak to valley results as specified above on each of the Form 5s. In each case, the $\leq 25\%$ criterion was met. The raw chromatograms were also reviewed to confirm this information.

Initial Calibration

Satisfactory instrument calibration is crucial to ensuring the accurate qualitative and quantitative results for each of the CDD/CDF compounds. Initial calibration procedures define the linear range and mean relative response factors that will be used for sample quantitation.

For the primary analytical systems (Instrument B and C), the initial calibration summary information met all the relevant acceptance criteria including the relative responses (RRFs) of $<25\%$ for native compounds and $<35\%$ for the labeled compounds, ion abundance ratios (IARs) within $\pm 15\%$, absolute retention times within the WDM windows, and signal-to-noise ratios of $>10:1$.

Initial calibration for the 2,3,7,8-TCDF second column confirmation instrument (Instrument A) occurred on August 6, 2003. The initial calibration data met all acceptance criteria and is included in the Second Column Confirmation section of this report.

Instrument Stability and Continuing Calibration Verification

Instrument stability is an important aspect of this analytical system. Ongoing calibration verification using a CS-3 standard is performed for every 12-hour period. This standard is used to evaluate the isomer retention times, ion abundance criteria, sensitivity and ongoing calibration criteria. The continuing calibration verification summary information met all relevant acceptance criteria including the frequency of the continuing calibration verifications at the both beginning and ending of each 12-hour analytical run, the RRFs $\%D$ within $<25\%$ for native compounds and $<35\%$ for labeled compounds of initial calibration, the IARs within the $\pm 15\%$, and signal-to-noise of $>10:1$.

CAS met the requirements of the absolute retention times (RT) for the first internal standard (^{13}C -1,2,3,4-TCDD) of ± 15 seconds (of the initial calibration standard) and the associated >25.0 minute RT requirement for most of the 12-hour CS3 calibration verification standards. However, occasionally, the RT for the first internal standard falls outside this 15 second criteria. It should be noted that the 15 second criteria is not specific to Method 8290. All the RTs for internal standards in

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each CS-3 standards fell within the switching time window as defined by the WDM and each CS-3 standard recorded acceptable percent recoveries therefore, using professional judgment as stated in the Guidelines, no data are qualified based on these RT deviations. The relative retention time and ion abundance criteria were also met for each of the native and label CDD/CDFs in the ongoing CS-3 standard results.

The relative response factor (RRF) criteria of <25% difference between CS-3 native compounds as compared to the initial calibration data was acceptable for all instrument A runs. For the instrument B runs on August 20, 21, the criteria were met. For the instrument B run on September 13, the criteria were not met. For the instrument C runs on August 28, 31, September 12, 19, the criteria were met. For the instrument C runs on August 23, 27, 29, September 13, the criteria were not met. For the runs where the criteria were exceeded, the data package contained the appropriate documentation (form 6a & 6b), which was then validated. Note: The exceedances of the 25% ending calibration verification standard criteria required the laboratory to provide the mean RRFs (using form 6) from the beginning and ending calibration verification analysis and the mean factors were then employed for quantitation, as specified in Method 8290, Section 8.3.2.4 and 7.7.4.4. No data qualifiers were assigned to the project data.

While the Guidelines indicate review of the relative responses (RR), method 8290 has no such criteria, as RR's are not used in final quantitation of sample results, therefore, no review was performed.

Review of the above elements indicates a high level of instrument stability and no qualifiers are required.

Method Blank Analysis

Laboratory method blank analyses are used to determine the existence and magnitude of contamination introduced at the laboratory.

CAS prepared 6 method blank samples during the analysis of the project samples. Three method blanks were non-detect for all target analytes. Three method blanks had a detection of OCDD (1.6 – 2.5 ng/kg) - but below the project required quantitation limit of 5.0 ng/kg. The trace concentrations

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of OCDD were within common laboratory practice levels of OCDD. All method blank samples were prepared and analyzed at the appropriate frequency.

Laboratory Control Samples (Ongoing Precision/Accuracy)

The laboratory is required to prepare and analyze a sample of spiked reference matrix for measures of accuracy of the analytical process/system/method.

CAS performed laboratory control sample (LCS) analyses at the appropriate frequency for the analytical batch. All seven sets of LCS results met associated acceptance criteria for percent recovery of the spiked concentrations as presented in Table 6 of Exhibit D of the Guidelines. All RRT and IARs were also acceptable in the LCS samples associated with the analytical batch indicating an in-control analytical system.

Labeled Compound Recovery (Surrogate Standard Recovery)

Because the introduced labeled compounds (CDD/CDFs) serve as the isotopic quantitative mechanism for this method, recoveries should be closely monitored for laboratory and method effectiveness.

CAS met the minimum acceptance criteria for the labeled compound recoveries as presented in Table 7 of Exhibit D of the Guidelines. However, these recovery windows are wider than CAS's internally generated acceptance criteria. Labeled compound recoveries qualified with a "Y" in the original data package indicate the recovery falls outside the laboratory-generated acceptance limits. None of these minor failures exceeded the Guidelines limits; therefore, no data requires qualification.

Second Column Confirmation

A second column confirmation of the 2,3,7,8-TCDF isomer is required by both Methods 1613 and 8290 due to a known lack of isomer specificity for this isomer on the DB-5 or equivalent column. All operating conditions of the second column analytical system must be identical to those of the primary system.

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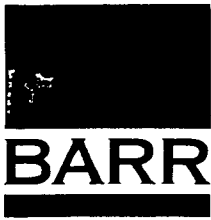
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CAS performed the second column confirmational analysis when the original analytical run had positive 2,3,7,8-TCDF detections. All performance criteria (as previously detailed above) were met prior to and during the confirmational analytical runs.

Matrix Spike Recoveries

Sample RES40 0-4" was used for a MS and MSD spike sample. Due to the high native concentration of OCDD, OCDF, and 1,2,3,4,6,7,8-HpCDD in the sample relative to the spiked level, the matrix spike recovery could not be accurately determined. All other spike recoveries were within the laboratory control limits of 50-150%.



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October 23, 2003

Sonia Vega
On-Scene Coordinator
U S Environmental Protection Agency, Region 5
Emergency Response Branch
520 Lafayette Road North
St Paul, MN 55155-4194

Re: Validated Analytical Data
Laboratory Batch Numbers – E2300560, E2300584, E2300528, E2300570, E2300569,
K2306717, K2306923, K2306727, K 2306721
Docket No. V-W-'03-C-748
St. Regis Paper Company Site – Cass Lake, Minnesota

Dear Ms. Vega:

Barr has validated the analytical data contained in the laboratory batch number(s) identified above. Enclosed are the following documents related to these analytical data:

- Data Summary Table(s) {Attachment A}
- Validation Summary Report(s) {Attachment B}
- Complete analytical data package(s) from Columbia Analytical Services {Attachment C}

The data summary tables include the $TEQ_{DF-WHO_{98}}$ values calculated using the validated data. The TEQ calculation used a value of $\frac{1}{2}$ the detection limit for any congener that was not detected.

All data met the data quality objectives and are useable as reported. In accordance with the Unilateral Administrative Order, on October 3, 2003 International Paper authorized Columbia Analytical Services to analyze the archived samples C11-12 4-12, A17-19 4-12, A19-20 4-12, AB3-4 4-12, A5-6 4-12, C4-5 4-12, A6-7 0-4, A6-7 4-12 and D10-11 0-4 for PCDD/F since the adjacent or overlying sample exceeds 1 ppb $TEQ_{DF-WHO_{98}}$.

If you have any questions concerning this information, please contact me at (952)832-2876 or Tom Ross of International Paper at (901)419-3899

Sincerely,

Thomas D. Mattison
Project Coordinator

cc: Tim Drexler, U.S. EPA – RPM (w/ Attachments A, B & C)
Mony Chabria and Tom Turner, U.S. EPA (w/ Attachments A & B)

Attachment A

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	F27-29 0-4 8/6/2003 CAS	F27-29 4-12 8/6/2003 CAS	H25-26 0-4 8/5/2003 CAS	I26-27 0-4 8/6/2003 CAS	I27-29 0-4 8/6/2003 CAS	J29-30 0-4 8/6/2003 CAS
2,3,7,8-TCDD	0 003	0 002	0 00055 j	<0 00034	0 00079 j	0 0017 EMPC
1,2,3,7,8-PeCDD	0 068	0 032	0 008	0 004	0 021	0 046
1,2,3,4,7,8-HxCDD	0 260	0 123	0 024	0 010	0 054	0 166
1,2,3,6,7,8-HxCDD	0 963	0 274 j	0 108	0 064	0 320	3 072
1,2,3,7,8,9-HxCDD	0 295 j	0 242	0 049	0 022	0 112	0 389
1,2,3,4,6,7,8-HpCDD	30 852	11 364	2 344	2 496	11 694	186 701
OCDD	249 540 e	133 473 e	22 122 e	24 152	124 075	2182 551 e
2,3,7,8-TCDF	0 026	0 006	0 001	<0 00098	0 004	0 014
1,2,3,7,8-PeCDF	0 111	0 064	0 008	0 007	0 030	0 123
2,3,4,7,8-PeCDF	0 113	0 065	0 010	0 009	0 035	0 140
1,2,3,4,7,8-HxCDF	0 723	0 460	0 144	0 061	0 308	2 625
1,2,3,6,7,8-HxCDF	0 233	0 128	0 032	0 016	0 076	0 262
1,2,3,7,8,9-HxCDF	0 041	<0 035	0 032	0 021	0 084 EMPC	0 063
2,3,4,6,7,8-HxCDF	0 366	0 229	0 056	0 026	0 129	0 887
1,2,3,4,6,7,8-HpCDF	7 167	2 489	0 530	0 450	2 713	28 274
1,2,3,4,7,8,9-HpCDF	0 639	0 447	0 080	0 045	0 207	2 125
OCDF	29 433	14 042	1 686	1 682	12 664	132 899
TCDD, Total	0 016	0 004	0 002	<0 00034	0 003	0 010
PeCDD, Total	0 279	0 111	0 039	0 014	0 082	0 183
HxCDD, Total	7 753	2 747	0 548	0 257	1 388	5 045
HpCDD, Total	75 414	46 496	6 977	4 105	23 525	82 565
TCDF, Total	0 143	0 052	0 010	0 004	0 047	0 116
PeCDF, Total	2 648	1 686	0 229	0 154	0 759	2 490
HxCDF, Total	7 251	4 152	1 909	0 926	5 721	8 468
HpCDF, Total	34 820	30 590	3 558	2 094	13 912	53 718
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 840	0 370	0 120	0 060	0 310	3 300

Data qualifiers and footnotes

e - estimated value Analyte exceeds upper end of the linear calibration range

j - estimated value Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location	D25-26 0-4	DE8-9 0-4	DE8-9 4-12	E29-30 0-4	F22-23 0-4	J26-27 0-4
Date	8/8/2003	8/8/2003	8/8/2003	8/8/2003	8/8/2003	8/8/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	<0.0003	<0.0006	<0.00052	0.006	0.002	0.002
1,2,3,7,8-PeCDD	0.021	0.017	0.011	0.143	0.050	0.083
1,2,3,4,7,8-HxCDD	0.049	0.067	0.046	0.438	0.143	0.313
1,2,3,6,7,8-HxCDD	0.404	0.206	0.132	1.602	0.402	5.251
1,2,3,7,8,9-HxCDD	0.135	0.130	0.090	0.748	0.313	0.830
1,2,3,4,6,7,8-HpCDD	12.855	8.516	6.274	49.437	13.533	158.645
OCDD	133.391	55.358 e	45.773 e	428.350	133.542	1504.395 e
2,3,7,8-TCDF	0.004	0.001	0.0009 EMPC	0.014	0.003	0.065
1,2,3,7,8-PeCDF	0.038	0.006	0.004	0.086	0.017	0.412
2,3,4,7,8-PeCDF	0.033	0.007	0.005	0.094	0.019	0.755
1,2,3,4,7,8-HxCDF	0.372	0.068	0.053	0.617 EMPC	0.228	9.161
1,2,3,6,7,8-HxCDF	0.112	0.023	0.017	0.309	0.080	1.489
1,2,3,7,8,9-HxCDF	<0.0269	0.002	0.0016 j EMPC	0.030	0.029	0.079
2,3,4,6,7,8-HxCDF	0.176	0.045	0.036	0.566	0.151	2.451
1,2,3,4,6,7,8-HpCDF	3.413	1.131	0.949	12.178	2.411	50.506
1,2,3,4,7,8,9-HpCDF	0.360	0.106	0.075	0.806	0.276	4.332
OCDF	12.861	5.449	4.530	58.002	11.891	209.140 e
TCDD, Total	0.002	<0.0006	<0.00052	0.031	0.006	0.027
PeCDD, Total	0.082	0.074	0.046	0.547	0.175	0.253
HxCDD, Total	1.766	2.062	1.151	8.027	2.704	12.278
HpCDD, Total	28.397	22.841	13.136	92.152	33.768	70.667
TCDF, Total	0.030	0.009	0.006	0.251	0.053	0.338
PeCDF, Total	0.753	0.190	0.156	3.336	0.764	9.483
HxCDF, Total	3.317	1.336	1.020	9.664	4.802	24.238
HpCDF, Total	17.592	5.412	3.816	45.124	15.592	47.376
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.346	0.179	0.130	1.305	0.374	4.753

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	J26-27 4-12 8/8/2003 CAS	J26-27 12-24 8/8/2003 CAS	J27-29 0-4 8/8/2003 CAS	J27-29 4-12 8/8/2003 CAS	J27-29 12-24 8/8/2003 CAS	SW-44 0-4 8/12/2003 CAS
2,3,7,8-TCDD	0.000554 j	<0.00016	0.002	<0.000162	<0.00013	<0.00081
1,2,3,7,8-PeCDD	0.026	0.0019 j	0.033	<0.00017	0.00055 j	0.017
1,2,3,4,7,8-HxCDD	0.089	0.0086 EMPC	0.071	0.00076 j	0.0014 j	0.056
1,2,3,6,7,8-HxCDD	3.090	0.189	2.426	0.009	0.043	0.216
1,2,3,7,8,9-HxCDD	0.255	0.023	0.243	0.0011 j	0.003	0.105
1,2,3,4,6,7,8-HpCDD	112.241	5.335	79.840 e	0.282	1.096	8.119
OCDD	1406.612 e	52.529 e	747.571 e	2.208 e	8.740	72.045
2,3,7,8-TCDF	0.037	0.004	0.010	<0.000163	<0.00012	<0.0010
1,2,3,7,8-PeCDF	0.209	0.023	0.127	<0.00016	0.0016 j	0.008
2,3,4,7,8-HxCDF	0.308	0.029	0.181	0.00054 j	0.003	0.008
1,2,3,4,7,8-HxCDF	4.746	0.252	2.798	0.008	0.036	0.081
1,2,3,6,7,8-HxCDF	0.757	0.048	0.386	0.0013 j	0.006	0.026
1,2,3,7,8,9-HxCDF	0.049	<0.0103	0.033 EMPC	<0.00061	<0.00061	<0.016
2,3,4,6,7,8-HxCDF	1.382	0.070	0.844	0.003	0.012	0.054
1,2,3,4,6,7,8-HpCDF	30.098	1.515	28.143	0.116	0.490	0.967
1,2,3,4,7,8,9-HpCDF	2.502	0.089	2.203	0.012	0.062	0.184
OCDF	171.091 e	9.036	154.944 e	0.624	2.748	6.763
TCDD, Total	0.009	0.001	0.018	<0.00016	<0.00013	<0.00081
PeCDD, Total	0.070	0.003	0.138	<0.00017	0.001	0.050
HxCDD, Total	5.265	0.487	5.660	0.021	0.095	0.923
HpCDD, Total	71.507	9.989	66.753	0.461	2.229	14.968
TCDF, Total	0.144	0.015	0.113	<0.00016	0.004	0.010
PeCDF, Total	3.986	0.420	2.748	0.006	0.033	0.195
HxCDF, Total	13.173	2.976	11.552	0.124	0.608	1.545
HpCDF, Total	42.756	8.349	39.244	0.572	3.440	6.775
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	2.837	0.153	2.005	0.007	0.029	0.177

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location	SW-7 0-4	SW-7 4-12	SW-41 0-4	SW-42 0-4	FOA-02-1-0-4	FOA-02-2-0-4
Date	8/11/2003	8/11/2003	8/11/2003	8/12/2003	8/12/2003	8/12/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	<0.00081	<0.000528	0.002	<0.000468	0.004	0.001
1,2,3,7,8-PeCDD	0.041	0.017	0.041	0.021	0.089	0.013
1,2,3,4,7,8-HxCDD	0.127	0.054	0.138	0.068	0.288	0.035
1,2,3,6,7,8-HxCDD	1.006 _j	0.774 _j	1.019 _j	0.467	0.774	0.289
1,2,3,7,8,9-HxCDD	0.334	0.165	0.348	0.192	0.352 _j	0.093
1,2,3,4,6,7,8-HpCDD	51.621	45.229	55.172	12.112	27.598	4.794
OCDD	459.098	439.818	517.194 _e	115.280	238.156	45.6
2,3,7,8-TCDF	0.007	0.005	0.013	0.005	0.008	0.006
1,2,3,7,8-PeCDF	0.023	0.018	0.041	0.019	0.049	0.026
2,3,4,7,8-PeCDF	0.034	0.026	0.064	0.024	0.060	0.031
1,2,3,4,7,8-HxCDF	0.279	0.226	0.426	0.134	0.323 _j EMPC	0.300
1,2,3,6,7,8-HxCDF	0.063	0.036	0.079	0.036	0.202	0.071
1,2,3,7,8,9-HxCDF	<0.0216	<0.022	0.010	0.004	0.106 EMPC	0.007 EMPC
2,3,4,6,7,8-HxCDF	0.142	0.082	0.183	0.065	0.342	0.118
1,2,3,4,6,7,8-HpCDF	5.202	4.306	6.214	1.506	4.924	1.158
1,2,3,4,7,8,9-HpCDF	0.329 _j EMPC	<0.664	<1.040	0.187	<0.850	0.191
OCDF	38.912	36.458	45.538	8.344	26.012	4.099
TCDD, Total	0.007	0.007	0.022	0.005	0.016	0.002
PeCDD, Total	0.208	0.094	0.223	0.114	0.269	0.049
HxCDD, Total	4.712	3.459	5.779	2.600	5.161	1.043
HpCDD, Total	81.045	69.661	99.057	32.082	33.783	12.457
TCDF, Total	0.078	0.053	0.164	0.046	0.077	0.031
PeCDF, Total	0.733	0.489	1.127	0.511	1.462	0.560
HxCDF, Total	5.757	4.649	3.168	2.720	5.208	3.822
HpCDF, Total	35.423	32.188	47.660	10.232	19.113	7.067
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.878	0.713	0.974	0.282	0.721	0.189

Data qualifiers and footnotes

e - estimated value. Analyte exceeds upper end of the linear calibration range.

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location	FOA-02-2-4-12	FOA-02-3-0-4	FOA-02-4-0-4	FOA-02-5-0-4	FOA-02-6-0-4	FOA-02-6-4-12
Date	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/12/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	<0.000201	<0.000131	<0.000193	0.001	0.00083 _j EMPC	<0.000111
1,2,3,7,8-PeCDD	0.006	0.003	0.008	0.014	0.010	0.000995 _j
1,2,3,4,7,8-HxCDD	0.018	0.012	0.029	0.031	0.029	0.003
1,2,3,6,7,8-HxCDD	0.094	0.041	0.204	0.093	0.086	0.010
1,2,3,7,8,9-HxCDD	0.045	0.021	0.062	0.074	0.080	0.008
1,2,3,4,6,7,8-HpCDD	4.349	1.881	6.506	3.709	3.315	0.408
OCDD	41.2	15.875	58.9 _e	32.69	27.0 _e	3.31 _e
2,3,7,8-TCDF	<0.00066 _j	0.001	0.001	<0.00035	<0.00044 _j	<0.000098
1,2,3,7,8-PeCDF	0.006	0.0010 _j	0.009	0.0015 _j	0.0021 _j	0.00024 _j EMPC
2,3,4,7,8-PeCDF	0.006	0.0013 _j	0.010	0.0013 _j	0.0020 _j	0.00030 _j EMPC
1,2,3,4,7,8-HxCDF	0.072	0.017	0.115	0.018	0.022	0.003
1,2,3,6,7,8-HxCDF	0.021	0.006	0.028	0.011	0.012	0.0015 _j
1,2,3,7,8,9-HxCDF	0.002 _j	0.002 _j	<0.00011	0.0032	<0.0013	<0.00034
2,3,4,6,7,8-HxCDF	0.035	0.011	0.050	0.025	0.022	0.0029 _j
1,2,3,4,6,7,8-HpCDF	0.960	0.274	1.227	0.417	0.440	0.066
1,2,3,4,7,8,9-HpCDF	0.062	0.026	0.112	0.050	0.046	0.007
OCDF	4.237	1.563	7.276	2.550	2.750	0.319
TCDD, Total	0.001	<0.00013	<0.00019	0.006	0.021	<0.000111
PeCDD, Total	0.021	0.007	0.024	0.055	0.114	0.007
HxCDD, Total	0.402	0.181	0.694	0.488	0.698	0.063
HpCDD, Total	5.318	2.694	11.401	5.759	5.276	0.655
TCDF, Total	0.007	0.003	0.005	0.007	0.013	0.001
PeCDF, Total	0.142	0.040	0.178	0.061	0.081	0.009
HxCDF, Total	1.048	0.290	0.746	0.422	0.465	0.067
HpCDF, Total	2.834	1.174	6.015	1.745	1.788	0.265
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.097	0.038	0.147	0.087	0.078	0.009

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	NWWD-04 0-4 8/14/2003 CAS	NWWD-05 0-4 8/14/2003 CAS	RES16A 0-4 8/14/2003 CAS	RES16B 0-4 8/14/2003 CAS	RES24 0-4 8/14/2003 CAS	RES 24 4-12 8/14/2003 CAS
2,3,7,8-TCDD	<0 00029	<0 0002	0 0007 _j EMPC	<0 0004	<0 0009	<0 0003
1,2,3,7,8-PeCDD	0 002 _j	0 0012 _j EMPC	0 017	0 005	0 004	0 001 _j
1,2,3,4,7,8-HxCDD	0 006 EMPC	0 0031 EMPC	0 052	0 013	0 008	0 004
1,2,3,6,7,8-HxCDD	0 035	0 017	0 344	0 061	0 028	0 014
1,2,3,7,8,9-HxCDD	0 017	0 0091	0 125	0 034	0 019	0 008
1,2,3,4,6,7,8-HpCDD	0 929	0 550	10 281	1 511	0 975	0 447
OCDD	8 519	4 652 _e	101 918 _e	14 372	7 553	3 657 _e
2,3,7,8-TCDF	<0 00097	<0 0004	0 003	<0 0009	<0 0009	<0 0003
1,2,3,7,8-PeCDF	0 002 _j EMPC	0 0006 _j EMPC	0 025	0 003 _j	<0 0009	<0 0006
2,3,4,7,8-PeCDF	0 002 _j	0 0010 _j	0 028	0 004	<0 0008	<0 0006
1,2,3,4,7,8-HxCDF	0 027	0 010	0 292	0 039	0 010	0 007
1,2,3,6,7,8-HxCDF	0 008	0 0040	0 081	0 015	0 004	<0 005
1,2,3,7,8,9-HxCDF	0 005	0 002 _j	0 057	0 010	<0 003	<0 007
2,3,4,6,7,8-HxCDF	0 014	0 0061	0 130	0 025	<0 003	<0 006
1,2,3,4,6,7,8-HpCDF	0 296	0 128	2 972	0 449	0 187	0 110
1,2,3,4,7,8,9-HpCDF	0 022	0 009	0 229	0 033	0 013	<0 004
OCDF	1 131	0 457	10 816 _e	1 556 _e	0 733	0 385
TCDD, Total	0 012	0 0004	0 003	<0 0004	<0 0009	<0 0003
PeCDD, Total	0 024	0 009	0 066	0 018	0 015	0 006
HxCDD, Total	0 178	0 097	1 340	0 338	0 180	0 088
HpCDD, Total	2 085	1 059	19 424	3 353	1 650	0 889
TCDF, Total	0 006	0 003	0 034	0 022	0 002	0 002
PeCDF, Total	0 065	0 029	0 613	0 166	0 033	0 034
HxCDF, Total	0 431	0 176	4 797	0 703	0 206	0 172
HpCDF, Total	1.217	0 488	12 789	1.704	0 750	0.393
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 0283	0 0143	0 287	0 0482	0 0243	0 0121

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	RES 28 0-4 8/14/2003 CAS	RES28 0-4D 8/14/2003 CAS	SWD-01 0-4 8/14/2003 CAS	SWD-02 0-4 8/14/2003 CAS	SWD-03 0-4 8/14/2003 CAS	SWD-04 0-4 8/14/2003 CAS
2,3,7,8-TCDD	<0 0002	<0 0008	0 0008 j	<0 0004	<0 0004	<0 0005
1,2,3,7,8-PeCDD	0 002 j	0 003 j	0 003	0 004	0 008	0 004
1,2,3,4,7,8-HxCDD	0 006	0 008	0 006	0 010	0 011	0 009
1,2,3,6,7,8-HxCDD	0 032	0 029	0 019	0 035	0 043	0 027
1,2,3,7,8,9-HxCDD	0 016	0 016	0 016	0 026	0 032	0 024
1,2,3,4,6,7,8-HpCDD	1 145	0 990	0 471	1 175	0 891	0 736
OCDD	9 748 e	9 166	3 169 e	9 804	7 074	5 350
2,3,7,8-TCDF	<0 0004	<0 0009	0 0006 j	0 0007 j	0 004	<0 0007
1,2,3,7,8-PeCDF	0 002 j	<0 0009	0 0009 j	0 002 j EMPC	0 0017 j EMPC	0 001 j
2,3,4,7,8-PeCDF	0 002 j	<0 0008	0 002 j	0 003	0 015	0 002 j
1,2,3,4,7,8-HxCDF	0 021	0 020	0 008	0 015	0 024	0 010
1,2,3,6,7,8-HxCDF	0 006 EMPC	0 006 EMPC	0 006	0 008	0 030	0 005
1,2,3,7,8,9-HxCDF	0 006	<0 006	0 002 j	0 003 j	0 009	0 002 j
2,3,4,6,7,8-HxCDF	0 013	0 009	0 011	0 016	0 080	0 009
1,2,3,4,6,7,8-HpCDF	0 262	0 270	0 115	0 211	0 220	0 156
1,2,3,4,7,8,9-HpCDF	0 017	0 021	0 007	0 015	0 017	0 011
OCDF	0 969	0 948	0 303	0 899	0 754	0 573
TCDD, Total	<0 0002	<0 0008	0 001	0 004	0 004	<0 0005
PeCDD, Total	0 011	0 006	0 012	0 022	0 052	0 025
HxCDD, Total	0 174	0 180	0 127	0 219	0 346	0 218
HpCDD, Total	1 970	1 904	0 885	2 058	1 766	1 498
TCDF, Total	0 007	<0 0009	0 026	0 053	0 266	0 016
PeCDF, Total	0 064	0 058	0 148	0 211	1 571	0 071
HxCDF, Total	0 355	0 344	0 223	0 344	1 064	0 205
HpCDF, Total	1 010	1 091	0 321	0 825	0 780	0 542
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 0289	0 0265	0 0182	0 0324	0 0508	0 0234

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-03-C-748**

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	SWD-05 0-4 8/14/2003 CAS	D20-21 0-4 8/13/2003 CAS	D27-29 0-4 8/13/2003 CAS	E11-13 0-4 8/13/2003 CAS	E13-15 0-4 8/13/2003 CAS	E18-19 0-4 8/13/2003 CAS
2,3,7,8-TCDD	<0.0003	0.005	0.001	0.003	<0.0002	0.001
1,2,3,7,8-PeCDD	0.002 j	0.105	0.026	0.061	0.010	0.036
1,2,3,4,7,8-HxCDD	0.004	0.334	0.083	0.170	0.025	0.100
1,2,3,6,7,8-HxCDD	0.020	2.314	0.406	1.368	0.274	0.517
1,2,3,7,8,9-HxCDD	0.014	0.756	0.186	0.401	0.067	0.230
1,2,3,4,6,7,8-HpCDD	0.908	72.773	13.044	51.460	9.581	17.327
OCDD	5.516	519.390 e	115.856	493.923 e	98.013 e	131.712
2,3,7,8-TCDF	0.0007 j	0.020	0.007	0.005	<0.0007	0.002
1,2,3,7,8-PeCDF	0.001 j	0.081	0.040	0.039	0.005	0.033
2,3,4,7,8-PeCDF	0.001 j	0.113	0.050	0.041	0.005	0.013
1,2,3,4,7,8-HxCDF	0.007	1.034	0.343	1.323	0.290	0.168
1,2,3,6,7,8-HxCDF	0.003	0.252	0.096	0.200	0.040	0.061
1,2,3,7,8,9-HxCDF	<0.002	0.020	<0.007	<0.000856	<0.001	<0.0003
2,3,4,6,7,8-HxCDF	0.005	0.432	0.160	0.386	0.072	0.113
1,2,3,4,6,7,8-HpCDF	0.108	12.966	3.285	12.807	2.720	3.061
1,2,3,4,7,8,9-HpCDF	0.006	0.981	0.250	1.514	0.455	0.253
OCDF	0.430	62.855	11.740	58.715	13.455	15.518
TCDD, Total	0.002	0.023	0.013	0.017	0.001	0.005
PeCDD, Total	0.017	0.340	0.094	0.208	0.039	0.096
HxCDD, Total	0.148	7.190	1.739	4.413	0.886	2.011
HpCDD, Total	1.844	34.277	21.470	37.461	17.735	23.016
TCDF, Total	0.019	0.123	0.034	0.086	0.016	0.026
PeCDF, Total	0.050	2.180	0.950	1.040	0.161	0.438
HxCDF, Total	0.136	6.584	5.123	5.772	4.504	3.181
HpCDF, Total	0.356	10.551	12.616	26.249	15.195	11.554
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.0193	1.610	0.361	1.180	0.229	0.386

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	E18-19 4-12 8/13/2003 CAS	E24-25 0-4 8/13/2003 CAS	E24-25 4-12 8/13/2003 CAS	E24-25 4-12D 8/13/2003 CAS	GS-1 8/13/2003 CAS	GS-1D 8/13/2003 CAS
2,3,7,8-TCDD	0 0007 j	0 0007 j	0 0004 j	0 0006 i	<0 0002	<0 0001
1,2,3,7,8-PeCDD	0 014	0 015	0 010	0 011	0 0009 j	0 0006 j
1,2,3,4,7,8-HxCDD	0 046	0 050	0 039	0 040	0 002 j EMPC	0 001 j
1,2,3,6,7,8-HxCDD	0 303	0 489	0 625	0 516	0 008	0 005
1,2,3,7,8,9-HxCDD	0 099	0 097	0 094	0 101	0 006	0 004
1,2,3,4,6,7,8-HpCDD	9 884	15 946	18 757	15 029	0 236	0 134
OCDD	96 380 e	173 869	196 286	153 060 e	1 760	0 971
2,3,7,8-TCDF	0 001	0 006	0 005	0 006	<0 0002	<0 0006
1,2,3,7,8-PeCDF	0 008	0 057	0 092	0 076	<0 0002	<0 0002
2,3,4,7,8-PeCDF	0 008	0 046	0 051	0 054	<0 0002	<0 0002
1,2,3,4,7,8-HxCDF	0 094	0 488	0 629	0 510	<0 005	<0 003
1,2,3,6,7,8-HxCDF	0 030	0 115	0 164	0 179	0 002 j EMPC	<0 003
1,2,3,7,8,9-HxCDF	<0 0005	0 015	0 038	0 036	<0 005	<0 003
2,3,4,6,7,8-HxCDF	0 062	0 182	0 246	0 274	0 003 j	<0 003
1,2,3,4,6,7,8-HpCDF	1 418	4 221	5 503	4 360	0 047	0 028
1,2,3,4,7,8,9-HpCDF	0.133	0 338	0 555	0 379	0 003 j	<0 001
OCDF	7 294	13 069	14 100	11 844	0 125	0 100
TCDD, Total	0 011	0 015	0 003	0 003	<0 0002	0 001
PeCDD, Total	0 039	0 069	0 025	0 030	0 005	0 003
HxCDD, Total	1 138	1 615	1 720	1 879	0 043	0 033
HpCDD, Total	18 291	27 647	25 001	25 404	0 414	0 247
TCDF, Total	0 012	0 037	0 026	0 030	0 001	0 001
PeCDF, Total	0 204	0 800	0 890	0 927	0 030	0 007
HxCDF, Total	1 816	8 491	4 449	10 762	0 046	0 024
HpCDF, Total	7 110	16 143	17 161	17 821	0 153	0 099
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 208	0 409	0 494	0 423	0 006	0 004

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-03-C-748**

[concentration in µg/Kg (ppb)]

Location	RES39 0-4	RES40 0-4	\NWD-01 0-4	\NWD-02 0-4	\NWD-02 4-12	\NWD-03 0-4
Date	8/13/2003	8/13/2003	8/15/2003	8/15/2003	8 15 2003	8/15/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	0 0004 j	0 0005 j EMPC	<0 0003	0 0004 j EMPC	<0 0002	<0 00009
1,2,3,7,8-PeCDD	0 004	0 009	0 001 j	0 007	0 001 j	0 0008 j
1,2,3,4,7,8-HxCDD	0 009	0 027	0 003 EMPC	0 017	0 002 j	0 003
1,2,3,6,7,8-HxCDD	0 028	0 101	0 011	0 051	0 009	0 012
1,2,3,7,8,9-HxCDD	0 023	0 068	0 007	0 041	0 006	0 005
1,2,3,4,6,7,8-HpCDD	0 671	2 958	0 349	1 390	0 269	0 381
OCDD	5 292	25 606	2 651 e	9 956 e	2 066 e	3 356 e
2,3,7,8-TCDF	<0 001	0 001	<0 0009	<0 0005	<0 0006	<0 0006
1,2,3,7,8-PeCDF	0 0009 j	0 003	<0 0003	0 001 j	0 0007 j	0 0009 j
2,3,4,7,8-PeCDF	0 001 j	0 004	0 0008 j EMPC	0 001 j	0 0005 j EMPC	0 001 j
1,2,3,4,7,8-HxCDF	0 009	0 036	0 006 EMPC	0 013	0 005	0 011
1,2,3,6,7,8-HxCDF	0 005	0 015	0 002 j EMPC	0 006	0 002 j	0 003
1,2,3,7,8,9-HxCDF	<0 0003	0 005	0 0007 j EMPC	0 001 j	0 002 j	0 003
2,3,4,6,7,8-HxCDF	0 008	0 024	0 004	0 010	0 004	0 004
1,2,3,4,6,7,8-HpCDF	0 160	0 632	0 069	0 299	0 059	0 110
1,2,3,4,7,8,9-HpCDF	0 011	0 036	0 004	0 017	0 006	0 008
OCDF	0 612	3 174	0 233	1 182	0 228	0 400
TCDD, Total	0 000	0 004	0 001	0 001	<0 0002	0 000
PeCDD, Total	0 018	0 051	0 008	0 025	0 003	0 005
HxCDD, Total	0 177	0 601	0 077	0 273	0 047	0 058
HpCDD, Total	1 515	6 737	0 688	2 696	0 476	0 682
TCDF, Total	0 009	0 026	0 008	0 007	0 003	0 003
PeCDF, Total	0 049	0 165	0 029	0 055	0 018	0 022
HxCDF, Total	0 192	0 865	0 090	0 301	0 090	0 144
HpCDF, Total	0 595	2 483	0 230	1 086	0 226	0 456
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 022	0 079	0 010	0 040	0 008	0 011

Data qualifiers and footnotes

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-03-C-748

[concentration in µg/Kg (ppb)]

Location	NWWD-06 0-4	NWWD-07 0-4	RES38 0-4	I20-21 0-4	B2-3-0-4	AB3-4-0-4
Date	8/15/2003	8/15/2003	8/15/2003	8/15/2003	8/26/2003	8/26/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	<0.0003	<0.0003	<0.0003	0.001	0.003	0.010
1,2,3,7,8-PeCDD	0.001 j	0.0009 j EMPC	0.002 j	0.024	0.025	0.162
1,2,3,4,7,8-HxCDD	0.003 j	0.002 j EMPC	0.004 EMPC	0.070	0.150	0.525
1,2,3,6,7,8-HxCDD	0.015	0.010	0.019	0.292	0.619	2.558
1,2,3,7,8,9-HxCDD	0.006	0.006	0.012	0.183	0.380	0.904 j
1,2,3,4,6,7,8-HpCDD	0.401	0.284	0.504	11.694	24.965	77.805
OCDD	2.960 e	2.148 e	3.55 e	79.825 e	212.795	765.367 e
2,3,7,8-TCDF	<0.0005	<0.0009	<0.0007	0.002	<0.0009	0.006
1,2,3,7,8-PeCDF	0.002 j	0.0006 j EMPC	0.001 j EMPC	0.010	0.011	0.067
2,3,4,7,8-PeCDF	0.002 j	0.0008 j EMPC	0.002 j	0.010	0.014	0.076
1,2,3,4,7,8-HxCDF	0.015	0.006	0.016	0.096	0.145	1.368
1,2,3,6,7,8-HxCDF	0.004 EMPC	0.002 j	0.008	0.043	0.066	0.373
1,2,3,7,8,9-HxCDF	0.003 j EMPC	0.001 j	0.004	0.018	0.020	0.138 EMPC
2,3,4,6,7,8-HxCDF	0.006	0.004	0.012	0.077	0.081	0.610
1,2,3,4,6,7,8-HpCDF	0.082	0.064	0.158	2.182	3.910	18.777
1,2,3,4,7,8,9-HpCDF	0.005	0.004	0.014	0.136	0.193	1.823
OCDF	0.152	0.187	0.321	7.118	20.694	93.716
TCDD, Total	<0.0003	0.001	0.000	0.005	0.015	0.064
PeCDD, Total	0.004	0.003	0.010	0.088	0.254	0.571
HxCDD, Total	0.077	0.060	0.104	1.469	3.354	9.284
HpCDD, Total	0.778	0.562	0.905	15.922	38.273	83.990
TCDF, Total	0.004	0.004	0.013	0.022	0.069	0.213
PeCDF, Total	0.041	0.021	0.082	0.322	0.533	1.962
HxCDF, Total	0.179	0.090	0.286	1.932	3.525	9.338
HpCDF, Total	0.279	0.206	0.528	5.378	14.299	44.082
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.013	0.008	0.018	0.257	0.526	1.93

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	AB4-5-0-4 8/26/2003 CAS	AB4-5-4-12 8/26/2003 CAS	C3-4 0-4 8/26/2003 CAS	C3-4 4-12 8/26/2003 CAS	TH-1-0-4 8/26/2003 CAS	A5-6-0-4 8/26/2003 CAS
2,3,7,8-TCDD	0.024	0.002	0.005	0.001 _j	<0.0003 _j	0.004 EMPC
1,2,3,7,8-PeCDD	0.317	0.024	0.092	0.033	<0.0003	0.078
1,2,3,4,7,8-HxCDD	1.377	0.077	0.235	0.116	<0.0004	0.248
1,2,3,6,7,8-HxCDD	7.616	0.610	1.869	0.464	0.002 _j	1.259
1,2,3,7,8,9-HxCDD	2.309	0.200	0.555	0.228	0.0007 _j EMPC	0.527
1,2,3,4,6,7,8-HpCDD	262.189 _e	23.993	53.631	16.405	0.086	50.122
OCDD	2728.565 _e	238.989	461.755	125.080	0.832	454.858
2,3,7,8-TCDF	0.035	0.004	0.011	0.004	<0.0002	0.006
1,2,3,7,8-PeCDF	0.236	0.020	0.049	0.022	<0.0003	0.040
2,3,4,7,8-PeCDF	0.224	0.019	0.043	0.018	<0.0003	0.039
1,2,3,4,7,8-HxCDF	3.066	0.206	0.448	0.137	0.0007 _j	0.443
1,2,3,6,7,8-HxCDF	0.778	0.048	0.151	0.055	0.0005 _j EMPC	0.132
1,2,3,7,8,9-HxCDF	0.618 EMPC	0.037	0.080	<0.059	<0.0004	0.076
2,3,4,6,7,8-HxCDF	1.647	0.096	0.273	0.109	0.001 _j EMPC	0.247
1,2,3,4,6,7,8-HpCDF	54.610	3.932	9.074	2.657	0.014	8.513
1,2,3,4,7,8,9-HpCDF	5.443	0.276	0.569	0.215	<0.0009	0.612
OCDF	351.915	26.731	44.226	13.720	0.081	48.655
TCDD, Total	0.133	0.009	0.050	0.004	<0.0003	0.018
PeCDD, Total	1.053	0.072	0.361	0.147	<0.0003	0.267
HxCDD, Total	23.219	2.106	5.675	1.994	0.008	4.357
HpCDD, Total	170.123	38.090	64.496	24.311	0.153	55.495
TCDF, Total	0.468	0.028	0.090	0.043	<0.0002	0.075
PeCDF, Total	4.844	0.339	1.115	0.548	<0.0003	0.751
HxCDF, Total	20.466	1.435	4.319	3.370	0.023	3.599
HpCDF, Total	88.689	17.479	27.110	12.562	0.062	25.998
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	5.74	0.473	1.17	0.365	0.002	1.04

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
 St. Regis Paper Company Site
 Docket No.: V-W-'03-C-748

{concentration in µg/Kg (ppb)}

Location Date Lab Dup	A5-6-4-12 8/26/2003 CAS	C4-5-0-4 8/26/2003 CAS	C5-6-0-4 8/26/2003 CAS	A7-8-0-4 8/26/2003 CAS	AC8-9-0-4 8/26/2003 CAS	AC9-10-0-4 8/26/2003 CAS
2,3,7,8-TCDD	0.002	0.006	0.003	0.003	0.003	0.003
1,2,3,7,8-PeCDD	0.042	0.093	0.053	0.046	0.057	0.061
1,2,3,4,7,8-HxCDD	0.130	0.246	0.156	0.155	0.172	0.199
1,2,3,6,7,8-HxCDD	1.709	1.755	1.019 _j	0.826	0.609	0.612
1,2,3,7,8,9-HxCDD	0.437	0.535	0.379	0.323	0.301	0.330
1,2,3,4,6,7,8-HpCDD	65.723	69.309	38.464	29.459	21.457	25.202
OCDD	583.647 _e	681.493 _e	333.568	267.751 _e	190.148	199.105
2,3,7,8-TCDF	0.014	0.021	0.008	0.007	0.003	0.004
1,2,3,7,8-PeCDF	0.061	0.135	0.041	0.034	0.021	0.021
2,3,4,7,8-PeCDF	0.065	0.104	0.038	0.037	0.022	0.023
1,2,3,4,7,8-HxCDF	0.544	0.829	0.323	0.516	0.254	0.236
1,2,3,6,7,8-HxCDF	0.125	0.259	0.100	0.114	0.074	0.073
1,2,3,7,8,9-HxCDF	0.121 EMPC	0.234	0.079	0.109	<0.075	<0.065
2,3,4,6,7,8-HxCDF	0.256 EMPC	0.453 EMPC	0.199 EMPC	0.206	0.139 EMPC	0.167
1,2,3,4,6,7,8-HpCDF	9.238	13.570	6.217	5.996	3.781	4.352
1,2,3,4,7,8,9-HpCDF	0.704	0.941	0.351	0.496 _j	0.334	0.351
OCDF	65.537	75.893	36.028	30.233	21.819	26.545
TCDD, Total	0.003	0.030	0.013	0.011	0.013	0.023
PeCDD, Total	0.123	0.367	0.186	0.149	0.198	0.215
HxCDD, Total	5.547	5.467	3.602	3.122	2.822	2.769
HpCDD, Total	80.623	66.563	56.113	48.004	41.192	39.236
TCDF, Total	0.054	0.131	0.043	0.044	0.046	0.062
PeCDF, Total	1.119	1.876	0.639	0.807	0.674	0.743
HxCDF, Total	3.987	7.392	2.782	7.324	2.174	2.417
HpCDF, Total	35.981	12.860	25.395	25.523	19.549	18.306
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	1.23	1.51	0.790	0.684	0.508	0.563

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748**

[concentration in µg/Kg (ppb)]

Location	BNSF-1-0-4	BNSF-2-0-4	BNSF-3-0-4	BNSF-4-0-4	BNSF-5-0-4	BNSF-6-0-4
Date	9/5/2003	9/5/2003	9/5/2003	9/5/2003	9/5/2003	9/5/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	0.0009 j	0.002	0.002	0.0003 j	0.0004, EMPC	0.0002 j
1,2,3,7,8-PeCDD	0.002 j EMPC	0.016	0.009	0.004	0.006	0.0008 j EMPC
1,2,3,4,7,8-HxCDD	0.004	0.033	0.015	0.009	0.015	0.0006 j
1,2,3,6,7,8-HxCDD	0.011	0.077	0.048	0.032	0.054	0.003
1,2,3,7,8,9-HxCDD	0.009	0.067	0.034	0.018	0.029	0.002 j
1,2,3,4,6,7,8-HpCDD	0.271	2.303	1.321	0.972	1.946	0.063
OCDD	2.2278 e	14.799	9.989	7.409	16.279	0.551
2,3,7,8-TCDF	<0.0001	<0.0008	0.001	<0.0007	<0.0008	<0.001
1,2,3,7,8-PeCDF	0.0003 j	0.002 j	0.002 j	0.001 j	0.002 j	<0.00007
2,3,4,7,8-PeCDF	0.0003 j EMPC	0.003 j	0.004	0.003 j	0.003 j	0.0008 j
1,2,3,4,7,8-HxCDF	0.004	0.024	0.022	0.013	0.022	0.002 j
1,2,3,6,7,8-HxCDF	0.002 j	0.012	0.008	0.008	0.009	0.0006 j
1,2,3,7,8,9-HxCDF	0.0004 j	0.002 j	0.004	0.004 EMPC	<0.0005	<0.0001
2,3,4,6,7,8-HxCDF	0.003	0.021	0.014	0.015	0.017	0.001 j
1,2,3,4,6,7,8-HpCDF	0.054	0.391	0.259	0.228	0.358	0.014
1,2,3,4,7,8,9-HpCDF	0.003	0.027	0.019	0.013	0.025	0.001 j EMPC
OCDF	0.193	1.411	0.896	0.734	1.534	0.049
TCDD, Total	0.001	0.005	0.027	0.007	0.003	0.010
PeCDD, Total	0.010	0.080	0.088	0.021	0.035	0.013
HxCDD, Total	0.090	0.577	0.307	0.199	0.284	0.018
HpCDD, Total	0.533	3.499	2.291	1.888	3.360	0.133
TCDF, Total	0.003	0.025	0.029	0.068	0.032	0.005
PeCDF, Total	0.020	0.162	0.118	0.243	0.144	0.013
HxCDF, Total	0.077	0.589	0.397	0.337	0.443	0.023
HpCDF, Total	0.183	1.296	0.928	0.768	1.412	0.047
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.010	0.072	0.045	0.029	0.047	0.003

Data qualifiers and footnotes

e - estimated value Analyte exceeds upper end of the linear calibration range

j - estimated value Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

**Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-03-C-748**

[concentration in µg/Kg (ppb)]

Location	A10-11-0-4	A10-11-4-12	A11-12-0-4	A11-12-4-12	A12-14-0-4	A12-14-4-12
Date	8/27/2003	8/27/2003	8/27/2003	8/27/2003	8/27/2003	8/27/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	0.003	0.002	0.004 EMPC	0.003	0.004	0.002 EMPC
1,2,3,7,8-PeCDD	0.060	0.018	0.074	0.042	0.085	0.048
1,2,3,4,7,8-HxCDD	0.209	<0.0005	0.276	0.165	0.325	0.191
1,2,3,6,7,8-HxCDD	0.891 _j EMPC	0.511	1.186 _j	1.214	1.222 _j	0.737
1,2,3,7,8,9-HxCDD	0.389	0.145	0.354 _j EMPC	0.288	0.513	0.339
1,2,3,4,6,7,8-HpCDD	28.075 EMPC	21.501	38.511	47.267	37.526	27.216
OCDD	310.616	222.669	329.111	421.893 _e	328.469	227.847
2,3,7,8-TCDF	0.007	0.002	0.004	0.003	0.005	0.003
1,2,3,7,8-PeCDF	0.033	0.011	0.031	0.021	0.036	0.021
2,3,4,7,8-PeCDF	0.038	0.012	0.033	0.021	0.038	0.023
1,2,3,4,7,8-HxCDF	0.433	0.239	0.415	0.344	0.444	0.331
1,2,3,6,7,8-HxCDF	0.112	0.044	0.119	0.083	0.138	0.087
1,2,3,7,8,9-HxCDF	<0.003	<0.002	<0.005	<0.003	<0.005	<0.012
2,3,4,6,7,8-HxCDF	0.193	0.098	0.426	0.121	0.266	0.182
1,2,3,4,6,7,8-HpCDF	7.583	5.007	6.957	8.639	6.991	4.960
1,2,3,4,7,8,9-HpCDF	0.491	0.396	0.399 _j EMPC	0.526	0.594	0.507
OCDF	22.534 EMPC	28.480	35.373	45.752	32.917	23.863
TCDD, Total	0.020	0.002	0.009	0.016	0.022	0.020
PeCDD, Total	0.230	0.056	0.212	0.131	0.278	0.139
HxCDD, Total	4.239	1.772	3.100	3.131	4.609	3.116
HpCDD, Total	60.929	34.191	55.329	47.336	56.427	48.335
TCDF, Total	0.064	0.023	0.070	0.046	0.076	0.042
PeCDF, Total	0.882	0.277	0.917	0.535	1.008	0.597
HxCDF, Total	3.172	1.645	3.410	2.429	3.785	2.522
HpCDF, Total	25.231	23.655	25.960	22.973	24.648	24.292
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0.702	0.425	0.869	0.889	0.888	0.602

Data qualifiers and footnotes

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect

EMPC - estimated maximum possible concentration

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location	A17-19-0-4	A19-20-0-4	A20-22-0-4	A20-22-4-12	A27-28-0-4	C11-12-0-4
Date	8/27/2003	8/27/2003	8/27/2003	8/27/2003	8/27/2003	8/27/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS
Dup						
2,3,7,8-TCDD	0 003	0 006 EMPC	0 007	0 003	0 005	0 004
1,2,3,7,8-PeCDD	0 086	0 161	0 130	0 068	0 113	0 071
1,2,3,4,7,8-HxCDD	0 245	0 484	0 406	0 235	0 404	0 279
1,2,3,6,7,8-HxCDD	2 576	2 330	1 083 j	1 621	1 565	1 621
1,2,3,7,8,9-HxCDD	0 554	0 936 j	0 382 j EMPC	0 567	0 459 j	0 519
1,2,3,4,6,7,8-HpCDD	95 976	71 289	33 180	61 901	50 051	60 551
OCDD	769 523 e	527 177 e	264 190	489 042	441 714	536 045 e
2,3,7,8-TCDF	0 005	0 010	0 008	0 011	0 012	0 008
1,2,3,7,8-PeCDF	0 029	0 050	0 049	0 049	0 060	0 048
2,3,4,7,8-PeCDF	0 033	0 058	0 053	0 070	0 059	0 054
1,2,3,4,7,8-HxCDF	0 356	0 474	0 544 j	0 653 j	0 481	0 515
1,2,3,6,7,8-HxCDF	0 104	0 194	0 138 j EMPC	0 150	0 186	0 128
1,2,3,7,8,9-HxCDF	0 047 EMPC	0 023	<0 150	<0 121	<0 102	<0 005
2,3,4,6,7,8-HxCDF	0 264	0 399	0 430	0 311 EMPC	0 485	0 267
1,2,3,4,6,7,8-HpCDF	12 120	11 104	6 072	9 525	6 704	9 889
1,2,3,4,7,8,9-HpCDF	0 631	1 119	0 464 j	0 715 j	0 572 j	0 832
OCDF	86 279	59 734	26 901	60 364	35 866	64 587
TCDD, Total	0 018	0 038	0 049	0 052	0 039	0 022
PeCDD, Total	0 254	0 506	0 428	0 220	0 440	0 253
HxCDD, Total	7 367	8 524	6 280	6 578	8 789	5 220
HpCDD, Total	85 726	79 750	54 913	64 726	65 223	59 701
TCDF, Total	0 061	0 128	0 115	0 092	0 107	0 078
PeCDF, Total	0 910	1 996	1 741	1 528	1 916	1 083
HxCDF, Total	3 507	6 273	6 163	5 003	5 925	3 811
HpCDF, Total	35 925	40 408	9 759	12 118	10 563	8 954
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	1 70	1 578	0 899	1 250	1 136	1 21

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	C12-14-0-4 8/27/2003 CAS	C23-24-0-4 8/27/2003 CAS	FOA1-Comp1 0-4 8/27/2003 CAS	FOA1-Comp2 0-4 8/27/2003 CAS	FOA1-Comp2 4-12 8/27/2003 CAS	FOA1-Comp3 0-4 8/27/2003 CAS
2,3,7,8-TCDD	0 002 EMPC	0 0009 j EMPC	0 006	0 003	0 0006 i EMPC	0 001
1,2,3,7,8-PeCDD	0 064	0 024	0 090	0 053	0 025	0 019
1,2,3,4,7,8-HxCDD	0 247	0 078	0 283	0 161	0 082	0 059
1,2,3,6,7,8-HxCDD	0 843 j	0 273	0 804	0 467	0 301	0 234
1,2,3,7,8,9-HxCDD	0 443	0 164	0 540	0 339	0 180	0 105
1,2,3,4,6,7,8-HpCDD	29 690	6 065	25 518	11 585	8 456	7 295
OCDD	246 315	52 209	232 814	107 461	83 484	69 005
2,3,7,8-TCDF	0 005	0 001	0 005	0 004	0 003	0 003
1,2,3,7,8-PeCDF	0 028	<0 011	0 020	0 021	0 019	0 014
2,3,4,7,8-PeCDF	0 030	<0 011	0 024	0 017	0 016	0 017
1,2,3,4,7,8-HxCDF	0 308	0 096	0 230	0 207	0 294	0 205
1,2,3,6,7,8-HxCDF	0 094	0 038	0 097	0 088	0 089	0 051
1,2,3,7,8,9-HxCDF	<0 006	<0 002	<0 078	<0 086	<0 002	0 004 EMPC
2,3,4,6,7,8-HxCDF	0 205	0 069	0 228	0 180	0 151	0 095
1,2,3,4,6,7,8-HpCDF	4 973	1 250	3 601	2 205	1 873	1 703
1,2,3,4,7,8,9-HpCDF	0 471	0 165	0 513	0 383	0 322	0 168
OCDF	25 663	4 946	16 893	9 305	6 957	6 207
TCDD, Total	0 010	0 005	0 053	0 023	0 001	0 004
PeCDD, Total	0 227	0 095	0 435	0 251	0 091	0 084
HxCDD, Total	3 828	1 374	5 144	2 729	1 555	1 238
HpCDD, Total	51 472	17 098	44 669	33 883	21 195	16 466
TCDF, Total	0 057	0 028	0 077	0 059	0 034	0 025
PeCDF, Total	0 749	0 411	0 947	0 948	0 678	0 443
HxCDF, Total	2 690	2 769	3 002	4 938	4 256	3 062
HpCDF, Total	21 778	8 274	4 932	14 836	10 458	7 750
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 676	0 181	0 653	0 368	0 260	0 204

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 1

Validated Dioxin/Furan Concentration in Soil
St. Regis Paper Company Site
Docket No.: V-W-'03-C-748

[concentration in µg/Kg (ppb)]

Location Date Lab Dup	FOA1- Comp4 0-4 8/27/2003 CAS	FOA1- Comp5 0-4 8/27/2003 CAS	FOA1- Comp6 0-4 8/27/2003 CAS
2,3,7,8-TCDD	<0 0008	<0 0009	<0 0007
1,2,3,7,8-PeCDD	0 010	0 010	0 012
1,2,3,4,7,8-HxCDD	0 030	0 031	0 037
1,2,3,6,7,8-HxCDD	0 204	0 331	0 220
1,2,3,7,8,9-HxCDD	0 066	0 076	0 073
1,2,3,4,6,7,8-HpCDD	3 770	5 977	4 856
OCDD	41 396	59 337	49 678
2,3,7,8-TCDF	<0 0008	0 005	0 003
1,2,3,7,8-PeCDF	0 014	0 032	0 015
2,3,4,7,8-PeCDF	0 020	0 034	0 015
1,2,3,4,7,8-HxCDF	0 240	0 241	0 157
1,2,3,6,7,8-HxCDF	0 055	0 065	0 045
1,2,3,7,8,9-HxCDF	0 035	<0 008	<0 004
2,3,4,6,7,8-HxCDF	0 095	0 090	0 077
1,2,3,4,6,7,8-HpCDF	1 013	1 534	1 131
1,2,3,4,7,8,9-HpCDF	0 158	0 217	0 156
OCDF	3 523	10 052	5 355
TCDD, Total	<0 0009	<0 0009	<0 0007
PeCDD, Total	0 030	0 027	0 036
HxCDD, Total	0 755	1 012	0 834
HpCDD, Total	11 858	18 445	13 463
TCDF, Total	0 004	0 014	0 005
PeCDF, Total	0 346	0 545	0 309
HxCDF, Total	2 833	3 995	2 663
HpCDF, Total	7 417	16 179	8 854
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1)	0 147	0 197	0 149

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 2
Validated Dioxin/Furan Concentration in Groundwater
St. Regis Paper Company Site
Docket No: V-W-'03-C-748

(concentrations in pg/L)

Location	316 GRANT UTLEY
Date	8/26/2003
Lab	CAS
Dup	
Exceedance Key	
2,3,7,8-TCDD	<3 153
1,2,3,7,8-Dioxin penta	<3 333
1,2,3,4,7,8-Dioxin, hexa	<2 667
1,2,3,6,7,8-Dioxin, hexa	<2 253
1,2,3,7,8,9-Dioxin, hexa	<2 413
1,2,3,4,6,7,8-Dioxin, hepta	<2 870
Dioxin octa	<9 400
2,3,7,8-TCDF	<4 086
1,2,3,7,8-Dibenzofuran, penta	<1 958
2,3,4,7,8-Dibenzofuran, penta	5 031 j EMPC
1,2,3,4,7,8-Dibenzofuran, hexa	<1.825
1,2,3,6,7,8-Dibenzofuran, hexa	<1 691
1,2,3,7,8,9-Dibenzofuran, hexa	<2 290
2,3,4,6,7,8-Dibenzofuran, hexa	<1 993
1,2,3,4,6,7,8-Dibenzofuran, hepta	<1 801
1,2,3,4,7,8,9-Dibenzofuran, hepta	<2.557
Dibenzofuran octa	<3 712
Dioxin tetra, Total	36 961
Dioxin penta, Total	59 171
Dioxin, hexa, Total	<2.253
Dioxin, hepta, Total	<2 870
Dibenzofuran tetra, Total	<2 554
Dibenzofuran penta, Total	5 499
Dibenzofuran, hexa, Total	5.193
Dibenzofuran, hepta, Total	<1.801
TEQ _{DF} - WHO ₉₈ (ND = 1/2 DL) (1000000)	6 81

Data qualifiers and footnotes:

e - estimated value. Analyte exceeds upper end of the linear calibration range

j - estimated value. Analyte concentration is below method reporting limit and above non-detect.

EMPC - estimated maximum possible concentration.

(1) TEQ_{DF}-WHO₉₈ values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range.

Table 3
Validated PAH/PCP Concentrations in Surface Soil
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/kg)

Location	A12-14 0-4	A20-22 0-4	A20-22 4-12	A27-28 0-4	A5-6 0-4	A5-6 4-12	BNSF-1-0-4	BNSF-2-0-4	BNSF-3-0-4	BNSF-4-0-4	BNSF-5-0-4	BNSF 6-0-4	C 23-24 0-4	C3-4 0-4	C 3 4 4-12
Date	8/27/2003	8/27/2003	8/27/2003	8/27/2003	8/26/2003	8/26/2003	9/5/2003	9/5/2003	9/5/2003	9/5/2003	9/5/2003	9/5/2003	8/27/2003	8/26/2003	8/26/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS
Solids, %	93.9	94.6	94.9	94.0	95.0	93.8	97.2	94.4	92.5	95.4	92.7	97.1	94.3	85.3	91.6
Carcinogenic PAHs															
Benzo(a)anthracene	380	240	93	310	110	99	--	--	--	--	--	--	1100	480	320
Chrysene	700	640	210	740	230	170	--	--	--	--	--	--	1200	720	440
Benzo(b)fluoranthene	700	720	380	1100	260	230	--	--	--	--	--	--	1300	750	370
Benzo(k)fluoranthene	540	530	230	1200	200	150	--	--	--	--	--	--	1300	660	370
Benzo(a)pyrene	300	290	110	540	110	120	--	--	--	--	--	--	1300	620	400
Indeno(1,2,3-cd)pyrene	420	580	320	950	190	220	--	--	--	--	--	--	1300	840	370
Dibenz(a,h)anthracene	95	110	71	260	33	35	--	--	--	--	--	--	230	130	56
Non-Carcinogenic PAHs															
Naphthalene	78	190	140	63	64	19	--	--	--	--	--	--	23	74	23
2-Methylnaphthalene	89	200	190	75	59	19	--	--	--	--	--	--	17	58	17
Acenaphthylene	57	62	26	130	22	23	--	--	--	--	--	--	160	110	38
Acenaphthene	<5.0	8.0	7.5	10	<4.3	<4.3	--	--	--	--	--	--	41	6.1	4.4
Fluorene	5.1	11	10	9.4	<4.3	<4.3	--	--	--	--	--	--	61	9.6	4.5
Phenanthrene	120	220	170	170	130	54	--	--	--	--	--	--	800	290	200
Anthracene	170	220	140	380	61	73	--	--	--	--	--	--	260	200	58
Fluoranthene	800	530	190	410	320	190	--	--	--	--	--	--	2500	950	710
Pyrene	610	450	210	430	230	170	--	--	--	--	--	--	2100	850	680
Benzo(g,h,i)perylene	300	480	290	730	120	150	--	--	--	--	--	--	1200	620	280
Pentachlorophenol	1200	1500	7300	2600	2500	5400	<200 *	<200 *	<200 *	<200 *	<200 *	<200 *	200	2800	930

Table 3
Validated PAH/PCP Concentrations in Surface Soil
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/kg)

Location Date Lab	D25-26 0-4 8/8/2003 CAS	D27-29 0-4 8/13/2003 CAS	DE8-9 0-4 8/8/2003 CAS	DE8-9 4-12 8/8/2003 CAS	E18 19 0-4 8/13/2003 CAS	E18-19 4-12 8/13/2003 CAS	F 24-25 0 4 8/13/2003 CAS	E24-25 4-12 8/13/2003 CAS	E29-30 0-4 8/8/2003 CAS	F22-23 0 4 8/8/2003 CAS	F27-29 0 4 8/6/2003 CAS	F27-29 4-12 8/6/2003 CAS	FOA 02-1-0-4 8/12/2003 CAS	FOA 02 2 0 4 8/12/2003 CAS
Solids, %	98	98	97.2	96.4	97.3	95.5	98.9	86.5	97.7	97.4	95.4	82.3	93.8	98.6
Carcinogenic PAHs														
Benzo(a)anthracene	62	30	6100	5800	55	120	91	120	280	260	280	170	850	73
Chrysene	110	56	14000	8800	110	210	140	340	530	340	620	260	1400	110
Benzo(b)fluoranthene	150	69	18000	9500	180	530	210	460	800	440	680	610	930	120
Benzo(k)fluoranthene	160	67	15000	8200	140	370	190	360	600	400	500	380	1200	120
Benzo(a)pyrene	90	29	7700	4000	55	280	150	140	360	380	330	380	870	86
Indeno(1,2,3-cd)pyrene	170	88	4800	2300	130	410	300	330	730	700	650	560	790	120
Dibenz(a,h)anthracene	24	12	930	510	21	63	43	62	110	87	88	120	190	19
Non-Carcinogenic PAHs														
Naphthalene	9.5	6.7	140	23	5.8	18	17	19	45	14	22	40	34	7.5
2-Methylnaphthalene	7.2	6.0	42	10	5.7	14	16	15	34	10	19	28	28	6.6
Acenaphthylene	11	6.5	550	470	9.6	29	18	17	72	75	45	61	86	9.7
Acenaphthene	<5.1	<5.0	28	11	<5.1	<5.0	<5.0	<5.1	7.2	<5.1	<5.1	8.3	190	5.1
Fluorene	<5.1	<5.0	30	19	<5.1	<5.0	<5.0	7.8	9.0	7.1	6.0	8.3	96	<5.1
Phenanthrene	26	19	180	68	12	23	54	56	130	190	79	74	780	35
Anthracene	33	22	1400	520	29	79	78	120	350	59	210	330	410	22
Fluoranthene	130	71	2000	2900	89	150	190	200	570	660	430	290	3900	160
Pyrene	120	57	6300	6900	86	170	140	140	530	460	460	320	2300	130
Benzo(g,h,i)perylene	120	56	2700	1300	74	220	180	170	490	530	310	500	610	83
Pentachlorophenol	430	<200	<410	210	360	270	940	1400	1100	310	1400	2000	3500	240

Table 3
Validated PAH/PCP Concentrations in Surface Soil
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/kg)

Location	FOA-02-2-4-12	FOA-02-3-0-4	FOA-02-4-0-4	FOA-02-5-0-4	FOA-02-6-0-4	FOA-02-6-4-12	FOA1-Comp1 0-4	FOA1-Comp2 0 4	FOA1-Comp2 4-12	FOA1-Comp3 0 4
Date	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/12/2003	8/27/2003	8/27/2003	8/27/2003	8/27/2003
Lab	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS	CAS
Solids, %	96.5	85.2	99.2	98.8	97.7	97.5	94.8	95.9	96.0	93.9
Carcinogenic PAHs										
Benzo(a)anthracene	24	11	83	27	40	18	380	190	12	460
Chrysene	40	16	110	39	66	24	590	310	20	610
Benzo(b)fluoranthene	39	15	92	30	52	18	670	680	28	2000
Benzo(k)fluoranthene	35	16	92	33	48	21	550	530	24	1500
Benzo(a)pyrene	27	11	85	25	38	21	500	440	11	1400
Indeno(1,2,3-cd)pyrene	39	11	95	32	52	23	560	500	22	1400
Dibenz(a,h)anthracene	5.6	<5.1	13	<5.0	7.2	<5.1	100	96	<5.1	550
Non-Carcinogenic PAHs										
Naphthalene	<5.0	<5.1	<5.0	<5.0	10	<5.1	44	26	9.5	44
2-Methylnaphthalene	<5.0	<5.1	<5.0	<5.0	6.5	<5.1	41	24	6.7	38
Acenaphthylene	<5.0	<5.1	<5.0	<5.0	<5.1	<5.1	64	39	<5.1	170
Acenaphthene	<5.0	<5.1	<5.0	<5.0	<5.1	<5.1	15	<5.0	<5.1	12
Fluorene	<5.0	<5.1	<5.0	<5.0	<5.1	<5.1	12	<5.0	<5.1	10
Phenanthrene	15	<5.1	49	29	32	11	180	44	11	86
Anthracene	8.7	<5.1	19	5.7	6.7	<5.1	220	110	14	390
Fluoranthene	54	12	180	61	98	39	650	180	20	410
Pyrene	43	12	140	55	80	34	540	260	21	420
Benzo(g,h,i)perylene	27	7.0	67	22	37	18	480	310	17	1000
Pentachlorophenol	<200	<210	<200	<200	240	<210	2800	540	<210	460

Table 3
Validated PAH/PCP Concentrations in Surface Soil
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/kg)

Location Date Lab	FOA1-Comp4 0-4 8/27/2003 CAS	FOA1-Comp5 4-12 8/27/2003 CAS	FOA1-Comp6 0-4 8/27/2003 CAS	GS-1 8/13/2003 CAS	H25-26 0-4 8/5/2003 CAS	J27-29 0-4 8/8/2003 CAS	J27-29 12-24 8/8/2003 CAS	J27-29 4-12 8/8/2003 CAS	NWWD 02 4 12 8/15/2003 CAS	RI S24 4 12 8/14/2003 CAS	SW-44 0-4 8/11/2003 CAS	TH 1 0 4 8/26/2003 CAS
Solids, %	94.9	94.6	94.8	97.9	92.3	96.6	85.9	96.2	96	98.7	84	91.2
Carcinogenic PAHs												
Benzo(a)anthracene	70	77	32	250	95	260	<5.0	<5.1	220	12	670	
Chrysene	100	190	72	320	130	710	<5.0	6.4	240	24	950	
Benzo(b)fluoranthene	130	65	96	290	120	2600	<5.0	9.7	210	18	850	
Benzo(k)fluoranthene	130	61	74	290	120	1700	<5.0	8.1	220	15	740	
Benzo(a)pyrene	88	5.9	38	310	120	1900	<5.0	<5.1	240	10	720	
Indeno(1,2,3-cd)pyrene	120	27	78	330	130	1500	<5.0	7.6	220	14	740	
Dibenz(a,h)anthracene	35	7.7	21	48	22	490	<5.0	<5.1	36	<5.0	110	
Non-Carcinogenic PAHs												
Naphthalene	21	<5.0	36	12	5.7	40	<5.0	<5.1	11	6.1	19	
2-Methylnaphthalene	22	<5.0	38	9.4	<5.1	25	<5.0	<5.1	7.0	<5.0	8.8	
Acenaphthylene	18	6.0	11	12	20	120	<5.0	<5.1	25	<5.0	6.9	
Acenaphthene	<5.0	5.5	<5.0	9.3	<5.1	9.7	<5.0	<5.1	<5.0	<5.0	10	
Fluorene	<5.0	11	<5.0	12	<5.1	16	<5.0	<5.1	<5.0	<5.0	15	
Phenanthrene	44	<5.0	32	240	46	100	<5.0	<5.1	66	25	350	
Anthracene	47	33	35	35	35	490	<5.0	<5.1	19	<5.0	110	
Fluoranthene	140	910	66	670	190	700	<5.0	5.7	380	57	1500	
Pyrene	130	410	61	400	200	540	<5.0	5.3	310	33	1200	
Benzo(g,h,i)perylene	110	22	66	210	110	1000	<5.0	<5.1	140	11	440	
Pentachlorophenol	380	7000	450	<210	210	7400	<200	<210	200	200	850	180

-- Not analyzed

* Estimated value, QA/QC criteria not met

Table 3
(concentrations in ug/kg)

- Not analyzed
- * Estimated value, QA/QC criteria not met

Table 4
Validated PAH/PCP/PCB Concentrations in Groundwater
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/L)

Location Date Lab	102 Basswood 8/9/2003 CAS	104 Norway 8/9/2003 CAS	127 1st 8/9/2003 CAS	15611 61st 8/9/2003 CAS	218 Elm 8/9/2003 CAS	233 2nd St. 8/15/2003 CAS	316 GRANT UTLEY 8/26/2003 CAS	514 1st St. 8/12/2003 CAS	521 1st 8/9/2003 CAS
<u>Carcinogenic PAHs</u>									
Benzo(a)anthracene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Chrysene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Benzo(b)fluoranthene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Benzo(k)fluoranthene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Benzo(a)pyrene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Indeno(1,2,3-cd)pyrene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Dibenzo(a,h)anthracene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
<u>Non-Carcinogenic PAHs</u>									
Naphthalene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
2-Methylnaphthalene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Acenaphthylene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Acenaphthene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Fluorene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Phenanthrene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Anthracene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Fluoranthene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Pyrene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Benzo(g,h,i)perylene	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020 h	<0.020	<0.020
Pentachlorophenol	<0.60	<0.60	<0.60	<0.60	<0.60	<0.60 *	<0.99 h*	<0.60	<0.60
PCB-1016	--	--	--	--	--	--	<0.20 h	--	--
PCB-1221	--	--	--	--	--	--	<0.39 h	--	--
PCB-1232	--	--	--	--	--	--	<0.20 h	--	--
PCB-1242	--	--	--	--	--	--	<0.20 h	--	--
PCB-1248	--	--	--	--	--	--	<0.20 h	--	--
PCB-1254	--	--	--	--	--	--	<0.20 h	--	--
PCB-1260	--	--	--	--	--	--	<0.20 h	--	--

-- Not analyzed

* Estimated value, QA/QC criteria not met

h Holding time not met

Table 5
Validated PAH/PCP/PCB Concentrations in IDW Water
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/L)

Location	RINSE WATER IDW
Date	9/5/2003
Lab	CAS
2,3,7,8-TCDD	<5.142
1,2,3,7,8-Dioxin penta	<5.486
1,2,3,4,7,8-Dioxin, hexa	19.812 j
1,2,3,6,7,8-Dioxin, hexa	108.929
1,2,3,7,8,9-Dioxin, hexa	30.376
1,2,3,4,6,7,8-Dioxin, hepta	2040.515
Dioxin octa	14346.009 e
2,3,7,8-TCDF	<5.520
1,2,3,7,8-Dibenzofuran, penta	9.586 j
2,3,4,7,8-Dibenzofuran, penta	10.345 j
1,2,3,4,7,8-Dibenzofuran, hexa	55.411
1,2,3,6,7,8-Dibenzofuran, hexa	11.689 j
1,2,3,7,8,9-Dibenzofuran, hexa	19.332 j EMPC
2,3,4,6,7,8-Dibenzofuran, hexa	19.910 j EMPC
1,2,3,4,6,7,8-Dibenzofuran, hepta	302.639
1,2,3,4,7,8,9-Dibenzofuran, hepta	41.210
Dibenzofuran octa	1749.133
Dioxin tetra, Total	<5.142
Dioxin penta, Total	<5.486
Dioxin, hexa, Total	374.427
Dioxin, hepta, Total	3504.988
Dibenzofuran tetra, Total	<5.520
Dibenzofuran penta, Total	160.980
Dibenzofuran, hexa, Total	628.791
Dibenzofuran, hepta, Total	1469.843
TEQ_{DF} - WHO₉₈ (ND = 1/2 DL) (1000000)	63.2
<u>Carcinogenic PAHs</u>	
Benzo(a)anthracene	0.041
Chrysene	0.60
Benzo(b)fluoranthene	<0.20
Benzo(k)fluoranthene	<0.20
Benzo(a)pyrene	<0.20
Indeno(1,2,3-cd)pyrene	<0.20
Dibenz(a,h)anthracene	<0.20
<u>Non-Carcinogenic PAHs</u>	
Naphthalene	0.24
2-Methylnaphthalene	0.15
Acenaphthylene	0.14
Acenaphthene	0.17
Fluorene	0.058
Phenanthrene	0.30
Anthracene	0.33
Fluoranthene	0.36
Pyrene	0.36
Benzo(g,h,i)perylene	<0.20
Pentachlorophenol	51 *

Table 5
Validated PAH/PCP/PCB Concentrations in IDW Water
St. Regis Company Site
Docket No: V-W-'03-C-748

(concentrations in ug/L)

Location	RINSE WATER IDW
Date	9/5/2003
Lab	CAS

Data qualifiers and footnotes:

e - estimated value Analyte exceeds upper end of the linear calibration range

j - estimated value Analyte concentration is below method reporting limit and above non-detect

* Estimated value, QA/QC criteria not met

EMPC - estimated maximum possible concentration

(1) TEQ_{DF-WHO₉₈} values shown above are calculated by multiplying the validated congener concentrations by their respective toxicity equivalency factors (TEF) and summing across all congeners. The results may differ from the TEQ concentrations reported in the laboratory data package, which are based on unvalidated data from each independent sample analysis (initial or dilution), and may include congener concentrations that exceeded the specified calibration range

Attachment B

**Data Validation Report – Dioxin/Furan
Laboratory Report / Batch: E2300560
Received: October 15, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Data Validation Report: October 23, 2003

The data validation of the Columbia Analytical Services, Inc (CAS) laboratory data for the dioxin and furan (CDD/CDF) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete as detailed below

The analytical data were reviewed in accordance with the U S EPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, Draft Final dated March, 2002 (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003) In addition to the Guidelines, specific SW-846 Method 8290 criteria were also considered as slight differences in some of the performance aspects exist between the documents In general, the areas covered by the validation process include

- Overall assessment
- Holding times, preservation and storage
- Mass calibration and mass spectrometer resolution
- Window defining mix
- Initial calibration
- Instrument stability and continuing calibration verification
- Method blank analysis
- Laboratory control samples
- Second column confirmation
- Matrix Spikes

Data Validation Report
Laboratory Report / Batch E2300560
Received October 15 2003

St. Regis Paper Company Superfund Site - Cass Lake Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report October 23 2003

Fifteen soil and one water sample results are contained in this laboratory report. They are as follows:

B2-3 0-4"	AB3-4 0-4"	AB4-5 0-4"	AB4-5 4-12"	C3-4 0-4"
C3-4 4-12"	TH-1 0-4"	A5-6 0-4"	A5-6 4-12"	A5-6 4-12D"
A5-6 0-4 ER	C5-6 0-4"	C4-5 0-4"	A7-8 0-4"	AC8-9 0-4"
AC9-10 0-4"				

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report E2300560 as a result of data validation process. All data met the data quality objectives (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected August 26, 2003, cooled to 4°C and sent to the laboratory. Per the chain-of-custody and subsequent laboratory acknowledgement receipt forms, the laboratory received the samples August 28, 2003 in acceptable condition and at 4°C. It should be noted that discrepancies between method 8290 and the Guidelines exist for the technical extraction and analysis holding times. The Guidelines recommend that soil samples be extracted within 10 days of receipt and analyzed within 30 days. Alternately, Method 8290 and the QAPP for the project indicate that a 30-day extraction/45 day analysis holding times apply. Method 8290 also states that these holding times are only recommendations as dioxins and furans are very stable in a variety of matrices. All samples were extracted and analyzed within 30 days. No qualifiers are applied.

Mass Calibration and Mass Spectrometer Resolution

Mass calibration and mass spectrometer resolution (MC/MSR) checks are required to ensure that the mass calibration and mass spectrometer resolution is set at a $\geq 10,000$ resolving power. This resolution is confirmed at the beginning and end of every 12-hour analysis period.

It should be noted that the Guidelines contain language that refer to evaluation techniques that due to upgrades in analytical systems software, are obsolete at CASs Houston facility. Specifically, the evaluation of the resolution by the measure of deviation between the exact m/z and the theoretical m/z at less than 5 ppm has been replaced with a function of instrumentation that implicitly sets the

Data Validation Report
Laboratory Report / Batch: E2300560
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Removal Site Evaluation and Supplemental Assessment

Date of Report: October 23, 2003

error to zero at all calibration points so there is no longer any need to use peak matching conditions to verify the exact mass. Therefore, resolving power is evaluated by close review of the resolution of PFK peak profiles where high mass ion (380.9760) and low mass ion (304.9824) are reported.

CAS performed the MC/MSRs at the appropriate frequency and obtained acceptable results for the PFK calibrant confirming MC/MSRs at a resolving power of 10,000. Note: CAS used three instrument systems to analyze the project samples and provided appropriate documentation from the three systems in the raw data package. All system's documentation were evaluated during the data validation process.

Window Defining Mix

The window defining mix (WDM) is necessary to establish the appropriate switching times for the selection ion group descriptors. As stated in the Guidelines, the frequency of the WDM is every 12 hours prior to calibration verification. However, Method 8290, Section 8.3.2.2.2. allows the laboratory, if running consecutive 12-hour shifts, to use the ending calibration verification (if all acceptance criteria are met) of the first 12-hour analysis period as the beginning calibration verification of the second 12-hour analysis period. In these cases, the WDM was analyzed after the calibration verification but still falls within the 12-hour analytical period. While the Guidelines indicate the WDM must be analyzed prior to the calibration verification sample, CAS's order of analysis for the system performance check samples did not appear to affect the overall system instrumentation and no qualifiers have been applied.

CAS performed the WDM analysis for every 12-hour sample analysis period and presented the acceptable switching times for each homologue group on the corresponding Form 5s.

Chromatographic Resolution

This check is performed to ascertain the separation of closely eluting dioxin/furan isomers. This is performed using SICP (selected ion current profile) of each isomer. The criteria requires that the 2,3,7,8-TCDD peak and the 1,2,3,8-TCDD peak be resolved with a valley of $\leq 25\%$ for the DB-5 column.

Data Validation Report
Laboratory Report / Batch: E2300560
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St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report: October 23, 2003

CAS provided summary peak to valley results as specified above on each of the Form 5s. In each case, the $\leq 25\%$ criterion was met. The raw chromatograms were also reviewed to confirm this information.

Initial Calibration

Satisfactory instrument calibration is crucial to ensuring the accurate qualitative and quantitative results for each of the CDD/CDF compounds. Initial calibration procedures define the linear range and mean relative response factors that will be used for sample quantitation.

For the primary analytical systems (Instrument B and C), the initial calibration summary information met all the relevant acceptance criteria including the relative responses (RRFs) of $<25\%$ for native compounds and $<35\%$ for the labeled compounds, ion abundance ratios (IARs) within $\pm 15\%$, absolute retention times within the WDM windows, and signal-to-noise ratios of $>10:1$.

Initial calibration for the 2,3,7,8-TCDF second column confirmation instrument (Instrument A) occurred on August 6, 2003. The initial calibration data met all acceptance criteria and is included in the Second Column Confirmation section of this report.

Instrument Stability and Continuing Calibration Verification

Instrument stability is an important aspect of this analytical system. Ongoing calibration verification using a CS-3 standard is performed for every 12-hour period. This standard is used to evaluate the isomer retention times, ion abundance criteria, sensitivity and ongoing calibration criteria. The continuing calibration verification summary information met all relevant acceptance criteria including the frequency of the continuing calibration verifications at the both beginning and ending of each 12-hour analytical run, the RRFs $\%D$ within $<25\%$ for native compounds and $<35\%$ for labeled compounds of initial calibration, the IARs within the $\pm 15\%$, and signal-to-noise of $>10:1$.

CAS met the requirements of the absolute retention times (RT) for the first internal standard (^{13}C -1,2,3,4-TCDD) of ± 15 seconds (of the initial calibration standard) and the associated >25.0 minute RT requirement for most of the 12-hour CS3 calibration verification standards. However, occasionally, the RT for the first internal standard falls outside this 15 second criteria. It should be noted that the 15 second criteria is not specific to Method 8290. All the RTs for internal standards in

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each CS-3 standards fell within the switching time window as defined by the WDM and each CS-3 standard recorded acceptable results all other quality control aspects (i.e., ion abundance, analyte response, instrument sensitivity) therefore, using professional judgment as stated in the Guidelines, no data is qualified based on these RT deviations. The relative retention time and ion abundance criteria were also met for each of the native and label CDD/CDFs in the ongoing CS-3 standard results.

The relative response factor (RRF) criteria of <25% difference between CS-3 native compounds as compared to the initial calibration data was acceptable for all instrument A runs. For the instrument B runs on September 8, 11, and 22 the criteria were not met. For the instrument C runs on September 24 the criteria was not met. All other ongoing calibration verification data yielded acceptable results. For the runs where the criteria were exceeded, the data package contained the appropriate documentation (Form 6a & 6b), which was then validated. Note: The exceedances of the 25% ending calibration verification standard criteria required the laboratory to provide the mean RRFs (using Form 6) from the beginning and ending calibration verification analysis and the mean factors were then employed for quantitation, as specified in Method 8290, Section 8.3.2.4 and 7.7.4.4. No data qualifiers were assigned to the project data.

While the Guidelines indicate review of the relative responses (RR), method 8290 has no such criteria, as RR's are not used in final quantitation of sample results, therefore, no review was performed.

Review of the above elements indicates a high level of instrument stability and no qualifiers are required.

Method Blank Analysis

Laboratory method blank analyses are used to determine the existence and magnitude of contamination introduced at the laboratory.

CAS prepared 6 method blank samples during the analysis of the project samples. Three method blanks were non-detect for all target analytes. Three method blanks had a detection of OCDD but below the project required quantitation limit of 5.0 ng/kg. The trace concentrations of OCDD were

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Laboratory Report / Batch E2300560
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St Regis Paper Company Superfund Site – Cass Lake Minnesota
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within common laboratory practice levels of OCDD. All method blank samples were prepared and analyzed at the appropriate frequency.

Laboratory Control Samples (Ongoing Precision/Accuracy)

The laboratory is required to prepare and analyze a sample of spiked reference matrix for measures of accuracy of the analytical process/system/method.

CAS performed laboratory control sample (LCS) analyses at the appropriate frequency for the analytical batch. All seven sets of LCS results met associated acceptance criteria for percent recovery of the spiked concentrations as presented in Table 6 of Exhibit D of the Guidelines. All RRT and IARs were also acceptable in the LCS samples associated with the analytical batch indicating an in-control analytical system.

Labeled Compound Recovery (Surrogate Standard Recovery)

Because the introduced labeled compounds (CDD/CDFs) serve as the isotopic quantitative mechanism for this method, recoveries should be closely monitored for laboratory and method effectiveness.

CAS met the minimum acceptance criteria for the labeled compound recoveries as presented in Table 7 of Exhibit D of the Guidelines. However, these recovery windows are wider than CAS's internally generated acceptance criteria. Labeled compound recoveries qualified with a "Y" in the original data package indicate the recovery falls outside the laboratory-generated acceptance limits. None of these minor failures exceeded the Guidelines limits, therefore, no data requires qualification.

Second Column Confirmation

A second column confirmation of the 2,3,7,8-TCDF isomer is required by both Methods 1613 and 8290 due to a known lack of isomer specificity for this isomer on the DB-5 or equivalent column. All operating conditions of the second column analytical system must be identical to those of the primary system.

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CAS performed the second column confirmational analysis when the original analytical run had positive 2,3,7,8-TCDF detections. All performance criteria (as previously detailed above) were met prior to and during the confirmational analytical runs

Matrix Spike Recoveries

Sample C3-4 4-12" was used for a MS and MSD spike sample. Due to the high native concentration of some of the target analytes in the sample relative to the spiked level, the matrix spike recovery could not be accurately determined. In cases where the native concentration is not as high spike recoveries were within the laboratory control limits of 50-150%.

**Data Validation Report – Dioxin/Furan
Laboratory Report / Batch: E2300584
Received: October 16, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Data Validation Report: October 23, 2003

The data validation of the Columbia Analytical Services, Inc (CAS) laboratory data for the dioxin and furan (CDD/CDF) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete as detailed below

The analytical data were reviewed in accordance with the U S. EPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, Draft Final dated March, 2002 (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8290 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Mass calibration and mass spectrometer resolution
- Window defining mix
- Initial calibration
- Instrument stability and continuing calibration verification
- Method blank analysis
- Laboratory control samples
- Second column confirmation
- Matrix Spikes

Data Validation Report
Laboratory Report / Batch E2300584
Received October 16, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report: October 23, 2003

Seven soil sample and two water sample results are contained in this laboratory report. They are as follows:

BNSF-6 0-4"	BNSF-6 0-4D"	BNSF-6 0-4"ER	BNSF-5 0-4"
BNSF-4 0-4"	BNSF-3 0-4"	BNSF-2 0-4"	BNSF-1 0-4"

Rinsate Water

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report E2300584 as a result of the data validation process. All data met the data quality objectives (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected September 5, 2003, cooled to 4°C and sent to the laboratory. Per the chain-of-custody and subsequent laboratory acknowledgement receipt forms, the laboratory received the samples September 9, 2003 in acceptable condition and at 4°C. It should be noted that discrepancies between method 8290 and the Guidelines exist for the technical extraction and analysis holding times. The Guidelines recommend that soil samples be extracted within 10 days of receipt and analyzed within 30 days. Alternately, Method 8290 and the QAPP for the project indicate that a 30-day extraction/45 day analysis holding times apply. Method 8290 also states that these holding times are only recommendations as dioxins and furans are very stable in a variety of matrices. All samples were extracted and analyzed within 30 days. No qualifiers are applied.

Mass Calibration and Mass Spectrometer Resolution

Mass calibration and mass spectrometer resolution (MC/MSR) checks are required to ensure that the mass calibration and mass spectrometer resolution is set at a $\geq 10,000$ resolving power. This resolution is confirmed at the beginning and end of every 12-hour analysis period.

It should be noted that the Guidelines contain language that refer to evaluation techniques that, due to upgrades in analytical systems software, are obsolete at CAS's Houston facility. Specifically, the evaluation of the resolution by the measure of deviation between the exact m/z and the theoretical m/z at less than 5 ppm has been replaced with a function of instrumentation that implicitly sets the

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error to zero at all calibration points so there is no longer any need to use peak matching conditions to verify the exact mass. Therefore, resolving power is evaluated by close review of the resolution of PFK peak profiles where high mass ion (380 9760) and low mass ion (304 9824) are reported.

CAS performed the MC/MSRs at the appropriate frequency and obtained acceptable results for the PFK calibrant confirming MC/MSRs at a resolving power of 10,000. Note CAS used three instrument systems to analyze the project samples and provided appropriate documentation from the three systems in the raw data package. All system's documentation were evaluated during the data validation process.

Window Defining Mix

The window defining mix (WDM) is necessary to establish the appropriate switching times for the selection ion group descriptors. As stated in the Guidelines, the frequency of the WDM is every 12 hours prior to calibration verification. However, Method 8290, Section 8.3.2.2.2 allows the laboratory, if running consecutive 12-hour shifts, to use the ending calibration verification (if all acceptance criteria are met) of the first 12-hour analysis period as the beginning calibration verification of the second 12-hour analysis period. In these cases, the WDM was analyzed after the calibration verification but still falls within the 12-hour analytical period. While the Guidelines indicate the WDM must be analyzed prior to the calibration verification sample, CAS's order of analysis for the system performance check samples did not appear to affect the overall system instrumentation and no qualifiers have been applied.

CAS performed the WDM analysis for every 12-hour sample analysis period and presented the acceptable switching times for each homologue group on the corresponding Form 5s.

Chromatographic Resolution

This check is performed to ascertain the separation of closely eluting dioxin/furan isomers. This is performed using SICP (selected ion current profile) of each isomer. The criteria requires that the 2,3,7,8-TCDD peak and the 1,2,3,8-TCDD peak be resolved with a valley of $\leq 25\%$ for the DB-5 column.

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St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
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CAS provided summary peak to valley results as specified above on each of the Form 5s. In each case, the $\leq 25\%$ criterion was met. The raw chromatograms were also reviewed to confirm this information.

Initial Calibration

Satisfactory instrument calibration is crucial to ensuring the accurate qualitative and quantitative results for each of the CDD/CDF compounds. Initial calibration procedures define the linear range and mean relative response factors that will be used for sample quantitation.

For the primary analytical systems (Instrument B and C), the initial calibration summary information met all the relevant acceptance criteria including the relative responses (RRFs) of $<25\%$ for native compounds and $<35\%$ for the labeled compounds, ion abundance ratios (IARs) within $\pm 15\%$, absolute retention times within the WDM windows, and signal-to-noise ratios of $>10:1$.

Initial calibration for the 2,3,7,8-TCDF second column confirmation instrument (Instrument A) occurred on August 6, 2003. The initial calibration data met all acceptance criteria and is included in the Second Column Confirmation section of this report.

Instrument Stability and Continuing Calibration Verification

Instrument stability is an important aspect of this analytical system. Ongoing calibration verification using a CS-3 standard is performed for every 12-hour period. This standard is used to evaluate the isomer retention times, ion abundance criteria, sensitivity and ongoing calibration criteria. The continuing calibration verification summary information met all relevant acceptance criteria including the frequency of the continuing calibration verifications at the both beginning and ending of each 12-hour analytical run, the RRFs $\%D$ within $<25\%$ for native compounds and $<35\%$ for labeled compounds of initial calibration, the IARs within the $\pm 15\%$, and signal-to-noise of $>10:1$.

CAS met the requirements of the absolute retention times (RT) for the first internal standard (^{13}C -1,2,3,4-TCDD) of ± 15 seconds (of the initial calibration standard) and the associated >25.0 minute RT requirement for most of the 12-hour CS3 calibration verification standards. However, occasionally, the RT for the first internal standard falls outside this 15 second criteria. It should be noted that the 15 second criteria is not specific to Method 8290. All the RTs for the internal

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standards in each CS-3 standard fell within the switching time window as defined by the WDM and each CS-3 standard recorded acceptable results all other quality control aspects (i.e., ion abundance, analyte response, instrument sensitivity) therefore using professional judgment as stated in the Guidelines, no data is qualified based on these RT deviations. The relative retention time and ion abundance criteria were also met for each of the native and label CDD/CDFs in the ongoing CS-3 standard results.

The relative response factor (RRF) criteria of <25% difference between CS-3 native compounds as compared to the initial calibration data was acceptable for all instrument A runs. For the instrument B run on August 18, the criteria were not met, a form 6 was provided. For the instrument C run on September 19, the criteria were not met, a form 6 was provided. For the instrument C runs on August 22, and October 2, the criteria were met. For the runs where the criteria were exceeded, the data package contained the appropriate documentation (form 6a & 6b), which was then validated. Note: The exceedances of the 25% ending calibration verification standard criteria required the laboratory to provide the mean RRFs (using form 6) from the beginning and ending calibration verification analysis and the mean factors were then employed for quantitation, as specified in Method 8290, Section 8.3.2.4 and 7.7.4.4. No data qualifiers were assigned to the project data.

While the Guidelines indicate review of the relative responses (RR), method 8290 has no such criteria, as RR's are not used in final quantitation of sample results, therefore, no review was performed.

Review of the above elements indicates a high level of instrument stability and no qualifiers are required.

Method Blank Analysis

Laboratory method blank analyses are used to determine the existence and magnitude of contamination introduced at the laboratory.

CAS prepared 3 method blank samples during the analysis of the project samples. One of the method blanks (aqueous) was non-detect for all target analytes. Two method blanks (solid) had a detection of OCDD (2.324 – 3.411 ng/kg) - but below the project required quantitation limit of 5.0 ng/kg. One method blank (solid) had a detection of OCDF at 0.719 ng/kg. The trace concentrations of OCDD

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Received: October 16, 2003

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and OCDF were within common laboratory practice levels of OCDD/OCDF. All method blank samples were prepared and analyzed at the appropriate frequency.

Laboratory Control Samples (Ongoing Precision/Accuracy)

The laboratory is required to prepare and analyze a sample of spiked reference matrix for measures of accuracy of the analytical process/system/method.

CAS performed laboratory control sample (LCS) analyses at the appropriate frequency for the analytical batch. All five sets of LCS results met associated acceptance criteria for percent recovery of the spiked concentrations as presented in Table 6 of Exhibit D of the Guidelines. All RRT and IARs were also acceptable in the LCS samples associated with the analytical batch indicating an in-control analytical system.

Labeled Compound Recovery (Surrogate Standard Recovery)

Because the introduced labeled compounds (CDD/CDFs) serve as the isotopic quantitative mechanism for this method, recoveries should be closely monitored for laboratory and method effectiveness.

CAS met the minimum acceptance criteria for the labeled compound recoveries as presented in Table 7 of Exhibit D of the Guidelines. However, these recovery windows are wider than CAS's internally generated acceptance criteria. Labeled compound recoveries qualified with a "Y" in the original data package indicate the recovery falls outside the laboratory-generated acceptance limits. None of these minor failures exceeded the Guidelines limits; therefore, no data requires qualification.

Second Column Confirmation

A second column confirmation of the 2,3,7,8-TCDF isomer is required by both Methods 1613 and 8290 due to a known lack of isomer specificity for this isomer on the DB-5 or equivalent column. All operating conditions of the second column analytical system must be identical to those of the primary system.

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CAS performed the second column confirmational analysis when the original analytical run had positive 2,3,7,8-TCDF detections. All performance criteria (as previously detailed above) were met prior to and during the confirmational analytical runs.

Matrix Spike Recoveries

No matrix spike data was provided in the report. However, the report case narrative indicated that the matrix spike associated with the analytical samples had high native concentrations of dioxins/furans therefore many recoveries were outside the acceptance criteria.

**Data Validation Report – Dioxin/Furan
Laboratory Report / Batch: E2300528
Received: October 3, 2003 (Revision rec'd 10/7/03)**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Data Validation Report: October 23, 2003

The data validation of the Columbia Analytical Services, Inc (CAS) laboratory data for the dioxin and furan (CDD/CDF) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete as detailed below

The analytical data were reviewed in accordance with the U S EPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, Draft Final dated March, 2002 (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8290 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Mass calibration and mass spectrometer resolution
- Window defining mix
- Initial calibration
- Instrument stability and continuing calibration verification
- Method blank analysis
- Laboratory control samples
- Second column confirmation
- Matrix Spikes

Data Validation Report
Laboratory Report / Batch E2300528
Received October 3, 2003

St Regis Paper Company Superfund Site – Cass Lake Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report October 23, 2003

Nine soil and one water sample results are contained in this laboratory report. They are as follows:

RES38 0-4" NWWD-06 0-4" NWWD-01 0-4" NWWD-07 0-4"
NWWD-02 0-4" NWWD-02 4-12" I20-21 0-4" NWWD-03 0-4"
NWWD-06 0-4"D NWWD-06 ER

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report E2300528 as a result of the data validation process. All data met the data quality objectives (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected August 15, 2003, cooled to 4°C and sent to the laboratory. Per the chain-of-custody and subsequent laboratory acknowledgement receipt forms, the laboratory received the samples August 16, 2003 in acceptable condition and at 4°C. The laboratory misinterpreted one of the samples I20-21 0-4" as F20-21 0-4" upon log in. This has been corrected in the data tables and documented in the original report. It should be noted that discrepancies between method 8290 and the Guidelines exist for the technical extraction and analysis holding times. The Guidelines recommend that soil samples be extracted within 10 days of receipt and analyzed within 30 days. Alternately, Method 8290 and the QAPP for the project indicate that a 30-day extraction/45 day analysis holding times apply. Method 8290 also states that these holding times are only recommendations as dioxins and furans are very stable in a variety of matrices. All samples were extracted and analyzed within 30 days. No qualifiers are applied.

Mass Calibration and Mass Spectrometer Resolution

Mass calibration and mass spectrometer resolution (MC/MSR) checks are required to ensure that the mass calibration and mass spectrometer resolution is set at a $\geq 10,000$ resolving power. This resolution is confirmed at the beginning and end of every 12-hour analysis period.

It should be noted that the Guidelines contain language that refer to evaluation techniques that due to upgrades in analytical systems software, are obsolete at CASs Houston facility. Specifically, the evaluation of the resolution by the measure of deviation between the exact m/z and the theoretical

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m/z at less than 5 ppm has been replaced with a function of instrumentation that implicitly sets the error to zero at all calibration points so there is no longer any need to use peak matching conditions to verify the exact mass. Therefore, resolving power is evaluated by close review of the resolution of PFK peak profiles where high mass ion (380 9760) and low mass ion (304 9824) are reported.

CAS performed the MC/MSRs at the appropriate frequency and obtained acceptable results for the PFK calibrant confirming MC/MSRs at a resolving power of 10,000. Note: CAS used three instrument systems to analyze the project samples and provided appropriate documentation from the three systems in the raw data package. All system's documentation were evaluated during the data validation process.

Window Defining Mix

The window defining mix (WDM) is necessary to establish the appropriate switching times for the selection ion group descriptors. As stated in the Guidelines, the frequency of the WDM is every 12 hours prior to calibration verification. However, Method 8290, Section 8.3.2.2.2, allows the laboratory, if running consecutive 12-hour shifts, to use the ending calibration verification (if all acceptance criteria are met) of the first 12-hour analysis period as the beginning calibration verification of the second 12-hour analysis period. In these cases, the WDM was analyzed after the calibration verification but still falls within the 12-hour analytical period. While the Guidelines indicate the WDM must be analyzed prior to the calibration verification sample, CAS's order of analysis for the system performance check samples did not appear to affect the overall system instrumentation and no qualifiers have been applied.

CAS performed the WDM analysis for every 12-hour sample analysis period and presented the acceptable switching times for each homologue group on the corresponding Form 5s.

Chromatographic Resolution

This check is performed to ascertain the separation of closely eluting dioxin/furan isomers. This is performed using SICP (selected ion current profile) of each isomer. The criteria requires that the 2,3,7,8-TCDD peak and the 1,2,3,8-TCDD peak be resolved with a valley of $\leq 25\%$ for the DB-5 column.

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CAS provided summary peak to valley results as specified above on each of the Form 5s. In each case, the $\leq 25\%$ criterion was met. The raw chromatograms were also reviewed to confirm this information.

Initial Calibration

Satisfactory instrument calibration is crucial to ensuring the accurate qualitative and quantitative results for each of the CDD/CDF compounds. Initial calibration procedures define the linear range and mean relative response factors that will be used for sample quantitation.

For the primary analytical systems (Instrument B and C), the initial calibration summary information met all the relevant acceptance criteria including the relative responses (RRFs) of $<25\%$ for native compounds and $<35\%$ for the labeled compounds, ion abundance ratios (IARs) within $\pm 15\%$, absolute retention times within the WDM windows, and signal-to-noise ratios of $>10:1$.

Initial calibration for the 2,3,7,8-TCDF second column confirmation instrument (Instrument A) occurred on August 6, 2003. The initial calibration data met all acceptance criteria and is included in the Second Column Confirmation section of this report.

Instrument Stability and Continuing Calibration Verification

Instrument stability is an important aspect of this analytical system. Ongoing calibration verification using a CS-3 standard is performed for every 12-hour period. This standard is used to evaluate the isomer retention times, ion abundance criteria, sensitivity and ongoing calibration criteria. The continuing calibration verification summary information met all relevant acceptance criteria including the frequency of the continuing calibration verifications at the both beginning and ending of each 12-hour analytical run, the RRFs $\pm 25\%$ within $<25\%$ for native compounds and $<35\%$ for labeled compounds of initial calibration, the IARs within the $\pm 15\%$, and signal-to-noise of $>10:1$.

CAS met the requirements of the absolute retention times (RT) for the first internal standard (^{13}C -1,2,3,4-TCDD) of ± 15 seconds (of the initial calibration standard) and the associated >250 minute RT requirement for most of the 12-hour CS3 calibration verification standards. However, occasionally, the RT for the first internal standard falls outside this 15 second criteria. It should be noted that the 15 second criteria is not specific to Method 8290. All the RTs for internal standards in

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each CS-3 standards fell within the switching time window as defined by the WDM and each CS-3 standard recorded acceptable results all other quality control aspects (i.e., ion abundance, analyte response, instrument sensitivity) therefore, using professional judgment as stated in the Guidelines, no data is qualified based on these RT deviations. The relative retention time and ion abundance criteria were also met for each of the native and label CDD/CDFs in the ongoing CS-3 standard results.

The relative response factor (RRF) criteria of <25% difference between CS-3 native compounds as compared to the initial calibration data was acceptable for all instrument A runs. For the instrument B runs on September 3 and 13, the criteria were not met. For the instrument C runs on September 3, and 13, the criteria were not met. For the instrument C runs on August 30, 31, September 1, and 9 the criteria were met. For the runs where the criteria were exceeded, the data package contained the appropriate documentation (Form 6a & 6b), which was then validated. Note: The exceedances of the 25% ending calibration verification standard criteria required the laboratory to provide the mean RRFs (using Form 6) from the beginning and ending calibration verification analysis and the mean factors were then employed for quantitation, as specified in Method 8290, Section 8.3.2.4 and 7.7.4.4. No data qualifiers were assigned to the project data.

While the Guidelines indicate review of the relative responses (RR), method 8290 has no such criteria, as RR's are not used in final quantitation of sample results, therefore, no review was performed.

Review of the above elements indicates a high level of instrument stability and no qualifiers are required.

Method Blank Analysis

Laboratory method blank analyses are used to determine the existence and magnitude of contamination introduced at the laboratory.

CAS prepared 6 method blank samples during the analysis of the project samples. Three method blanks were non-detect for all target analytes. Three method blanks had a detection of OCDD but below the project required quantitation limit of 5.0 ng/kg. The trace concentrations of OCDD were

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within common laboratory practice levels of OCDD. All method blank samples were prepared and analyzed at the appropriate frequency.

Laboratory Control Samples (Ongoing Precision/Accuracy)

The laboratory is required to prepare and analyze a sample of spiked reference matrix for measures of accuracy of the analytical process/system/method.

CAS performed laboratory control sample (LCS) analyses at the appropriate frequency for the analytical batch. All seven sets of LCS results met associated acceptance criteria for percent recovery of the spiked concentrations as presented in Table 6 of Exhibit D of the Guidelines. All RRT and IARs were also acceptable in the LCS samples associated with the analytical batch, indicating an in-control analytical system.

Labeled Compound Recovery (Surrogate Standard Recovery)

Because the introduced labeled compounds (CDD/CDFs) serve as the isotopic quantitative mechanism for this method, recoveries should be closely monitored for laboratory and method effectiveness.

CAS met the minimum acceptance criteria for the labeled compound recoveries as presented in Table 7 of Exhibit D of the Guidelines. However, these recovery windows are wider than CAS's internally generated acceptance criteria. Labeled compound recoveries qualified with a "Y" in the original data package indicate the recovery falls outside the laboratory-generated acceptance limits. None of these minor failures exceeded the Guidelines limits; therefore, no data requires qualification.

Second Column Confirmation

A second column confirmation of the 2,3,7,8-TCDF isomer is required by both Methods 1613 and 8290 due to a known lack of isomer specificity for this isomer on the DB-5 or equivalent column. All operating conditions of the second column analytical system must be identical to those of the primary system.

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CAS performed the second column confirmational analysis when the original analytical run had positive 2,3,7,8-TCDF detections. All performance criteria (as previously detailed above) were met prior to and during the confirmational analytical runs.

Matrix Spike Recoveries

Sample NWWD-03 0-4" was used for a MS and MSD spike sample. Due to the high native concentration of OCDD, OCDF, and 1,2,3,4,6,7,8-HpCDD in the sample relative to the spiked level, the matrix spike recovery could not be accurately determined. All other spike recoveries were within the laboratory control limits of 50-150%.

**Data Validation Report – Dioxin/Furan
Laboratory Report / Batch: E2300570
Received: October 18, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Data Validation Report: October 23, 2003

The data validation of the Columbia Analytical Services, Inc (CAS) laboratory data for the dioxin and furan (CDD/CDF) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete as detailed below

The analytical data were reviewed in accordance with the U S EPA Analytical Operations Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, Draft Final dated March, 2002 (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003) In addition to the Guidelines, specific SW-846 Method 8290 criteria were also considered as slight differences in some of the performance aspects exist between the documents In general, the areas covered by the validation process include

- Overall assessment
- Holding times, preservation and storage
- Mass calibration and mass spectrometer resolution
- Window defining mix
- Initial calibration
- Instrument stability and continuing calibration verification
- Method blank analysis
- Laboratory control samples
- Second column confirmation
- Matrix Spikes

Data Validation Report
Laboratory Report / Batch: E2300570
Received: October 18, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Report: October 23, 2003

Twenty-three soil and one water sample results are contained in this laboratory report. They are as follows:

A10-11 0-4"	A10-11 4-12"	A11-12 0-4"	A11-12 4-12"	C11-12 0-4"	
A12-14 0-4"	A12-14 4-12"	C12-13 0-4"	A17-19 0-4"	A17-19 4-12"	C23-24 0-4"
A17-19 0-4D	A17-19 0-4ER	A19-20 0-4"	A20-22 0-4"	A20-22 0-4"	A27-28 0-4"
FOA1COMP1 0-4"		FOA1COMP2 0-4"		FOA1COMP3 0-4"	
FOA1COMP4 0-4"		FOA1COMP5 0-4"		FOA1COMP5 4-12"	
FOA1COMP6 0-4"					

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report E2300560 as a result of data validation process. With the exception of sample FOA1-COMP5 4-12", all data met the data quality objective (DQOs) and are useable as reported. Data reported for FOA1-COMP5 4-12" is invalid due to an error during sample check in. One jar of sample FOA1-COMP5 0-4" was incorrectly bar-coded as FOA1-COMP5 4-12" and used for analysis. This data will be used as another field duplicate sample and will be evaluated with the overall quality control samples during detailed quality control sample data assessment. The correct sample for FOA1-COMP5 4-12" is being analyzed and will be reported under separate cover.

Holding Times, Preservation and Storage

The samples were collected August 27, 2003, cooled to 4°C and sent to the laboratory. Per the chain-of-custody and subsequent laboratory acknowledgement receipt forms, the laboratory received the samples August 29, 2003 in acceptable condition and at 4°C. It should be noted that discrepancies between method 8290 and the Guidelines exist for the technical extraction and analysis holding times. The Guidelines recommend that soil samples be extracted within 10 days of receipt and analyzed within 30 days. Alternately, Method 8290 and the QAPP for the project indicate that a 30-day extraction/45 day analysis holding times apply. Method 8290 also states that these holding times are only recommendations as dioxins and furans are very stable in a variety of matrices. All samples were extracted and analyzed within 30 days. No qualifiers are applied.

Mass Calibration and Mass Spectrometer Resolution

Mass calibration and mass spectrometer resolution (MC/MSR) checks are required to ensure that the mass calibration and mass spectrometer resolution is set at a $\geq 10,000$ resolving power. This resolution is confirmed at the beginning and end of every 12-hour analysis period.

It should be noted that the Guidelines contain language that refer to evaluation techniques that due to upgrades in analytical systems software, are obsolete at CASs Houston facility. Specifically, the evaluation of the resolution by the measure of deviation between the exact m/z and the theoretical m/z at less than 5 ppm has been replaced with a function of instrumentation that implicitly sets the error to zero at all calibration points so there is no longer any need to use peak matching conditions to verify the exact mass. Therefore, resolving power is evaluated by close review of the resolution of PFK peak profiles where high mass ion (380 9760) and low mass ion (304 9824) are reported.

CAS performed the MC/MSRs at the appropriate frequency and obtained acceptable results for the PFK calibrant confirming MC/MSRs at a resolving power of 10,000. Note CAS used three instrument systems to analyze the project samples and provided appropriate documentation from the three systems in the raw data package. All system's documentation were evaluated during the data validation process.

Window Defining Mix

The window defining mix (WDM) is necessary to establish the appropriate switching times for the selection ion group descriptors. As stated in the Guidelines, the frequency of the WDM is every 12 hours prior to calibration verification. However, Method 8290, Section 8.3.2.2.2. allows the laboratory, if running consecutive 12-hour shifts, to use the ending calibration verification (if all acceptance criteria are met) of the first 12-hour analysis period as the beginning calibration verification of the second 12-hour analysis period. In these cases, the WDM was analyzed after the calibration verification but still falls within the 12-hour analytical period. While the Guidelines indicate the WDM must be analyzed prior to the calibration verification sample, CAS's order of analysis for the system performance check samples did not appear to affect the overall system instrumentation and no qualifiers have been applied.

CAS performed the WDM analysis for every 12-hour sample analysis period and presented the acceptable switching times for each homologue group on the corresponding Form 5s

Chromatographic Resolution

This check is performed to ascertain the separation of closely eluting dioxin/furan isomers. This is performed using SICP (selected ion current profile) of each isomer. The criteria requires that the 2,3,7,8-TCDD peak and the 1,2,3,8-TCDD peak be resolved with a valley of $\leq 25\%$ for the DB-5 column.

CAS provided summary peak to valley results as specified above on each of the Form 5s. In each case, the $\leq 25\%$ criterion was met. The raw chromatograms were also reviewed to confirm this information.

Initial Calibration

Satisfactory instrument calibration is crucial to ensuring the accurate qualitative and quantitative results for each of the CDD/CDF compounds. Initial calibration procedures define the linear range and mean relative response factors that will be used for sample quantitation.

For the primary analytical systems (Instrument B and C), the initial calibration summary information met all the relevant acceptance criteria including the relative responses (RRFs) of $<25\%$ for native compounds and $<35\%$ for the labeled compounds, ion abundance ratios (IARs) within $\pm 15\%$, absolute retention times within the WDM windows, and signal-to-noise ratios of $>10:1$.

Initial calibration for the 2,3,7,8-TCDF second column confirmation instrument (Instrument A) occurred on August 6, 2003. The initial calibration data met all acceptance criteria and is included in the Second Column Confirmation section of this report.

Instrument Stability and Continuing Calibration Verification

Instrument stability is an important aspect of this analytical system. Ongoing calibration verification using a CS-3 standard is performed for every 12-hour period. This standard is used to evaluate the isomer retention times, ion abundance criteria, sensitivity and ongoing calibration criteria. The continuing calibration verification summary information met all relevant acceptance criteria.

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including the frequency of the continuing calibration verifications at the both beginning and ending of each 12-hour analytical run, the RRFs %D within <25% for native compounds and <35% for labeled compounds of initial calibration, the IARs within the +/-15%, and signal-to-noise of >10 1 CAS met the requirements of the absolute retention times (RT) for the first internal standard (¹³C-1,2,3,4-TCDD) of \pm 15 seconds (of the initial calibration standard) and the associated >25 0 minute RT requirement for most of the 12-hour CS3 calibration verification standards. However, occasionally, the RT for the first internal standard falls outside this 15 second criteria. It should be noted that the 15 second criteria is not specific to Method 8290. All the RTs for internal standards in each CS-3 standards fell within the switching time window as defined by the WDM and each CS-3 standard recorded acceptable results all other quality control aspects (i.e., ion abundance, analyte response, instrument sensitivity) therefore, using professional judgment as stated in the Guidelines, no data are qualified based on these RT deviations. The relative retention time and ion abundance criteria were also met for each of the native and label CDD/CDFs in the ongoing CS-3 standard results.

The relative response factor (RRF) criteria of <25% difference between CS-3 native compounds as compared to the initial calibration data was acceptable for all instrument A runs. For the instrument B runs on September 11, 16, 15, and 29 the criteria were not met. For the instrument C run on October 1 the criteria was not met. All other ongoing calibration verification data yielded acceptable results. For the runs where the criteria were exceeded, the data package contained the appropriate documentation (Form 6a & 6b), which was then validated. Note: The exceedances of the 25% ending calibration verification standard criteria required the laboratory to provide the mean RRFs (using Form 6) from the beginning and ending calibration verification analysis and the mean factors were then employed for quantitation, as specified in Method 8290, Section 8.3.2.4 and 7.7.4.4. No data qualifiers were assigned to the project data.

While the Guidelines indicate review of the relative responses (RR), method 8290 has no such criteria, as RR's are not used in final quantitation of sample results, therefore, no review was performed.

Review of the above elements indicates a high level of instrument stability and no qualifiers are required.

Method Blank Analysis

Laboratory method blank analyses are used to determine the existence and magnitude of contamination introduced at the laboratory.

CAS prepared 11 method blank samples during the analysis of the project samples. Four method blanks were non-detect for all target analytes. Six of the method blanks had detections of OCDD but all were below the project required quantitation limit of 5.0 ng/kg. One method blank sample had a trace detection of OCDF as well. The trace concentrations of were within common laboratory practice levels. None of the positive concentrations adversely affect the sample data as sample concentrations are far greater than any positive blank detections. All method blank samples were prepared and analyzed at the appropriate frequency.

Laboratory Control Samples (Ongoing Precision/Accuracy)

The laboratory is required to prepare and analyze a sample of spiked reference matrix for measures of accuracy of the analytical process/system/method.

CAS performed laboratory control sample (LCS) analyses at the appropriate frequency for the analytical batch. All five sets of LCS results met associated acceptance criteria for percent recovery of the spiked concentrations as presented in Table 6 of Exhibit D of the Guidelines. All RRT and IARs were also acceptable in the LCS samples associated with the analytical batch indicating an in-control analytical system.

Labeled Compound Recovery (Surrogate Standard Recovery)

Because the introduced labeled compounds (CDD/CDFs) serve as the isotopic quantitative mechanism for this method, recoveries should be closely monitored for laboratory and method effectiveness.

CAS met the minimum acceptance criteria for the labeled compound recoveries as presented in Table 7 of Exhibit D of the Guidelines. However, these recovery windows are wider than CAS's internally generated acceptance criteria. Labeled compound recoveries qualified with a "Y" in the original data package indicate the recovery falls outside the laboratory-generated acceptance limits. None of these minor failures exceeded the Guidelines limits; therefore, no data requires qualification

Second Column Confirmation

A second column confirmation of the 2,3,7,8-TCDF isomer is required by both Methods 1613 and 8290 due to a known lack of isomer specificity for this isomer on the DB-5 or equivalent column. All operating conditions of the second column analytical system must be identical to those of the primary system.

CAS performed the second column confirmational analysis when the original analytical run had positive 2,3,7,8-TCDF detections. All performance criteria (as previously detailed above) were met prior to and during the confirmational analytical runs.

Matrix Spike Recoveries

Sample FOA1-COMP2 0-4" was used for a MS and MSD spike sample. Due to the high native concentration of some of the target analytes in the sample relative to the spiked level, the matrix spike recovery could not be accurately determined. In cases where the native concentration is not as high, spike recoveries were within the laboratory control limits of 50-150%.

**Data Validation Report – Dioxin/Furan
Laboratory Report / Batch E2300569
Received September 26, 2003 (rev. October 7, 2003)**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Report: October 23, 2003

The data validation of the Columbia Analytical Services, Inc (CAS) laboratory data for the dioxin and furan (CDD/CDF) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment groundwater sample contained in the aforementioned report is complete as detailed below.

The analytical data were reviewed in accordance with the U S EPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, Draft Final dated March 2002 (Guidelines) as specified in the project-specific Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8290 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Mass calibration and mass spectrometer resolution
- Window defining mix
- Instrument stability
- Initial calibration and ongoing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Second column confirmation

One groundwater sample result is contained in this laboratory report. It is as follows:

316 Grant Utley

Overall Assessment

Due to OCDD concentrations in the corresponding laboratory method blank sample, the 316 Grant Utley OCDD sample concentration is considered false positive and has been adjusted accordingly in the data summary table. No qualifiers were assigned to the sample results contained in laboratory report E2300569 as a result of the data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The sample was collected August 26, cooled to 4°C and sent to the laboratory. Per the chain-of-custody and subsequent laboratory acknowledgement receipt forms, the laboratory received the samples August 29, 2003 in acceptable condition. It should be noted that discrepancies between Method 8290 and the Guidelines exist for the technical extraction and analysis holding times. The Guidelines recommend that soil samples be extracted within 10 days of receipt and analyzed within 30 days. Alternately, Method 8290 and the QAPP for the project indicate that 30 day extraction/45 day analysis holding times apply. Method 8290 also states that these holding times are only recommendations as dioxins and furans are very stable in a variety of matrices. All samples were extracted and analyzed within 30 days. No qualifiers are applied.

Mass Calibration and Mass Spectrometer Resolution

Mass calibration and mass spectrometer resolution (MC/MSR) checks are required to ensure that the mass calibration and mass spectrometer resolution is set at a $\geq 10,000$ resolving power. This resolution is confirmed at the beginning and end of every 12-hour analysis period.

It should be noted that the Guidelines contain language that refers to evaluation techniques that due to upgrades in analytical systems software, are obsolete at CASs Houston facility. Specifically, the evaluation of the resolution by the measure of deviation between the exact m/z and the theoretical m/z at less than 5 ppm has been replaced with an instrumentation function that implicitly sets the error to zero at all calibration points so there is no longer any need to use peak matching conditions to verify the exact mass. Therefore, resolving power is evaluated by close review of the resolution of PFK peak profiles where high mass ion (380.9760) and low mass ion (304.9824) are reported.

CAS performed the MC/MSR at the appropriate frequency and obtained acceptable results for the PFK calibrant confirming MC/MSR at a resolving power of 10,000.

Window Defining Mix

The window defining mix (WDM) is necessary to establish the appropriate switching times for the selection ion group descriptors. As stated in the Guidelines, the frequency of the WDM is once every 12 hours prior to calibration verification. However, Method 8290, Section 8.3.2.2.2. allows the laboratory, if running consecutive 12-hour shifts, to use the ending calibration verification (if all acceptance criteria are met) of the first 12-hour analysis period as the beginning calibration verification of the second 12-hour analysis period. In these cases, the WDM was analyzed after the calibration verification but still falls within the 12-hour analytical period. While the Guidelines indicate the WDM must be analyzed prior to the calibration verification sample, CAS's order of analysis for the system performance check samples did not appear to affect the overall system instrumentation and no qualifiers have been applied.

CAS performed the WDM analysis for every 12-hour sample analysis period and presented the acceptable switching times for each homologue group on the corresponding Form 5.

Chromatographic Resolution

This check is performed to ascertain the separation of closely eluting dioxin/furan isomers. This is performed using SICP (selected ion current profile) of each isomer. The criteria requires that the 2,3,7,8-TCDD and the 1,2,3,8-TCDD peaks (for the DB-5 column) and the 2,3,7,8-TCDF and the 2,3,4,7-TCDF peaks (for the DB-225 column) be resolved with a valley of $\leq 25\%$.

CAS provided summary peak to valley results as specified above on each of Form 5. In each case, the $\leq 25\%$ criterion was met. The raw chromatograms were also reviewed to confirm this information.

Initial Calibration

Satisfactory instrument calibration is crucial to ensuring the accurate qualitative and quantitative results for each of the CDD/CDF compounds. Initial calibration procedures define the linear range and mean relative response factors that will be used for sample quantitation.

For the primary analytical system (instrument C) CAS's initial calibration summary information (performed May 22, 2003) met all the relevant acceptance criteria including the relative responses (RRFs) of <25% for native compounds and <35% for the labeled compounds, ion abundance ratios (IARs) within +/- 15%, absolute retention times within the WDM windows, and signal-to-noise ratios of >10.1.

Initial calibration for the 2,3,7,8-TCDF second column confirmation instrument (instrument A) occurred on August 6, 2003. The initial calibration data met all acceptance criteria and is included in the Second Column Confirmation section of this report.

Instrument Stability and Continuing Calibration Verification

Instrument stability is an important aspect of this analytical system. Ongoing calibration verification using a CS-3 standard is performed once every 12-hour period. This standard is used to evaluate the isomer retention times, ion abundance criteria, sensitivity and ongoing calibration criteria.

CAS met the requirements of the absolute retention times (RT) for the first internal standard (13C-1,2,3,4-TCDD) of ± 15 seconds (of the initial calibration standard) and the associated >25.0 minute RT requirement for each of the 12-hour CS3 calibration verification standards. The relative retention time and ion abundance criteria were also met for each of the native and labeled CDD/CDFs in the ongoing CS-3 standard results.

Signal-to-noise (S/N) ratios were summarized for the native and labeled CDD/CDFs were acceptable at >10:1.

The relative response factor (RRF) criterion of <25% difference between CS-3 native compounds as compared to the initial calibration data was acceptable for instrument C (September 10) and for instrument A (September 11) analytical runs. No qualifiers were assigned to the project data.

While the Guidelines indicate review of the relative responses (RR), Method 8290 has no such requirement, as RR's are not used in final quantitation of sample results, therefore, no review was performed.

Review of the above elements indicates a high level of instrument stability and no qualifiers are required.

Method Blank Analyses

Laboratory method blank analyses are used to determine the existence and magnitude of contamination introduced at the laboratory.

The method blank sample was prepared and analyzed at the appropriate frequency. The method blank sample associated with this project sample had a positive OCDD concentration of 45.010 pg/L. This concentration of OCDD is below the CRQL of 50 pg/L. The associated sample concentration of 9.3 pg/L would indicate that it is likely a false positive result due to the ubiquitous nature of OCDD and the high blank concentration (as compared to the sample result). The sample result has been adjusted accordingly in the data summary table. It is represented as <9.4 pg/L.

Laboratory Control Samples (Ongoing Precision/Accuracy)

The laboratory is required to prepare and analyze a sample of spiked reference matrix to measure the accuracy of the analytical process/system/method.

CAS performed laboratory control sample (LCS) analyses at the appropriate frequency for the analytical batch. The LCS results met associated acceptance criteria for percent recovery of the spiked concentrations as presented in Table 6 of Exhibit D of the Guidelines. All RRTs and IARs were also acceptable in the LCS samples associated with the analytical batch indicating an in-control analytical system.

Labeled Compound Recovery (Surrogate Standard Recovery)

Because the introduced labeled compounds (CDD/CDFs) serve as the isotopic quantitative mechanism for this method, recoveries should be closely monitored for laboratory and method effectiveness.

CAS met the minimum acceptance criteria for the labeled compound recoveries as presented in Table 7 of Exhibit D of the Guidelines as well as CAS's own, more stringent recovery windows. No data requires qualification due to labeled compound recoveries.

Second Column Confirmation

A second column confirmation of the 2,3,7,8-TCDF isomer is required by both Methods 1613 and 8290 due to a known lack of isomer specificity for this isomer on the DB-5 or equivalent column. All operating conditions of the second column analytical system must be identical to those of the primary system.

CAS performed the second column confirmational analysis when the original analytical run had positive 2,3,7,8-TCDF detections. All performance criteria (as previously detailed above) were met prior to and during the confirmational analytical runs.

Matrix Spike Recoveries

No matrix spike summary information was reported for this data package. It was likely a non-project sample that was utilized, therefore, would have limited applicability to the project results.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306717
Received: October 3, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: October 23, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete as detailed below

The analytical data were reviewed in accordance with the U S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

Thirteen soil sample results are contained in this laboratory report. They are as follows:

A12-14 0-4”	A20-22 0-4”	A20-22 4-12”	C23-24 0-4”
A27-28 0-4”	FOA1-COMP1 0-4”	FOA1-COMP2 0-4”	FOA1-COMP2 4-12”
FOA1-COMP3 0-4”	FOA1-COMP4 0-4”	FOA1-COMP5 0-4”	FOA1-COMP5 4-12”
FOA1-COMP6 0-4”			

Data Validation Report
Laboratory Report / Batch K2306727
Received October 3, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: October 23, 2003

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2306717 as a result of data validation process. With the exception of sample FOA1-COMP5 0-4", all data met the data quality objective (DQOs) and are useable as reported. Data reported for FOA1-COMP5 0-4" is invalid due to an error during sample check in. One jar of sample FOA1-COMP5 4-12" was incorrectly bar-coded as FOA1-COMP5 0-4" and sent to Kelso from Houston for analysis. This data will be used as another field duplicate sample and will be evaluated with the overall quality control samples during detailed quality control sample data assessment.

Holding Times, Preservation and Storage

The samples were collected on August 27, 2003 and received at the CAS Houston laboratory on August 29, 2003 with an accompanying Chain-of-Custody (COC) form. All samples were received intact and were properly chilled (within 2-6 C). The samples were then shipped overnight to the CAS Kelso laboratory for semivolatile analysis (PAHs and PCP) and were received on September 4, 2003 intact and at 2.9 C (temperature blank). A discrepancy between one of the samples received and the chain of custody record was noted on the cooler receipt and preservation form. The laboratory was contacted to resolve the discrepancy. The laboratory discovered that upon sample check in, one jar for sample FOA1-COMP5 0-4" was mislabeled FOA1-COMP5 4-12 and sent to Kelso. Kelso noted that while both sample containers read FOA1-COMP5- 4-12" and none read FOA1-COMP5 0-4", they defaulted to Houston's numeric bar-code and proceeded with the analysis. Data reported as FOA1-COMP5-0-4" incorrect/invalid and is being removed from the final data set. The correct fraction of sample is being reanalyzed by both Kelso and Houston and will be reported under separate cover.

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the USEPA CLP National Functional Guidelines for Organic Data Review (NFG) and the SW-846 8270 method. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass

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Laboratory Report / Batch K2306727
Received October 3, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report October 23, 2003

spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the new tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The Initial Calibration (ICAL) for PAHs and PCP by Selected Ion Monitoring (SIM) was performed on September 13 and 19, 2003 using 7-10 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05 . All target analytes met the $<30\%$ RSD data validation criteria. The average RSD of the RRFs for PCP (22.7 and 19.6%) did exceed the method criteria of $<15\%$ RSD, however the laboratory utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being $<20\%$. The mean RSD for this initial calibration event was calculated as 7.4%, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard; all target analytes met the laboratory's criteria of $<20\%$ difference. There are no data validation criteria for second source standards.

Continuing Calibration Verification

The September 19, 20, and 22, 2003 continuing calibration verifications met the data validation criteria of $<+25\%$ difference and RRFs (>0.05) for all target analytes. The September 17 and 18, 2003 continuing calibration verifications met the data validation criteria of $<+25\%$ difference and RRFs (>0.05) for all target analytes except Indeno(1,2,3-cd)pyrene. However all data reported for this compound came from the September 19, 20, or 22, 2003 analytical runs.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Data Validation Report
Laboratory Report / Batch K2306727
Received October 3, 2003

St Regis Paper Company Superfund Site - Cass Lake Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report October 23, 2003

Laboratory Control Samples

For the verification of the analytical process/system method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Surrogate Standard Recovery

The matrix spike associated with the project samples had two surrogate standards and the laboratory control sample had one surrogate standard exceed laboratory criteria. Since the majority of matrix spike values and all laboratory control sample spike values met all percent recovery criteria, it does not appear these exceedances adversely affected the data. All remaining surrogate spike recoveries from the project samples met the data validation acceptance criteria.

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project samples and associated quality control samples met the method and data validation criteria.

Matrix Spike Recovery

A project sample (FOA1-Comp2 4-12") was utilized for the Matrix Spike (MS) and Matrix Spike Duplicate (MSD). All percent recoveries and RPDs from the MS and MSD were within the laboratory's internal control limits.

Field Duplicate Results

No field duplicate samples were contained in this submittal.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306923
Received October 14, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: October 23, 2003

The data validation process for the Columbia Analytical Services, Inc. (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete as detailed below.

The analytical data were reviewed in accordance with the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

Six soil and one water sample results are contained in this laboratory report. They are as follows:

**BNSF-6 0-4”
BNSF-2 0-4”**

**BNSF-6 0-4”D
BNSF-1 0-4”**

**BNSF-5 0-4”
RINSATE WATER**

BNSF-3 0-4”

Data Validation Report
Laboratory Report / Batch K2306923
Received October 14, 2003

St Regis Paper Company Superfund Site - Cass Lake Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report October 23, 2003

Overall Assessment

Qualifiers indicated PCP estimated concentrations, were assigned to the sample results contained in laboratory report K2306923 as a result of data validation process. However, the data still meets the data quality objective (DQOs) and are useable as qualified.

Holding Times, Preservation and Storage

The samples were collected on September 5, 2003 and received at the CAS Houston laboratory on September 6, 2003 with an accompanying chain-of-custody (COC) form. All samples were received intact and were properly chilled (within 2-6 °C). The samples were properly stored until shipped overnight to the CAS Kelso laboratory for PAH and PCP analysis. The samples were received on September 10, 2003 in Kelso intact and at 4-5 °C (temperature blank). All samples were received in good condition and consistent with the COC.

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs (water only) and PCP instrument using selected ion monitoring (SIM) was performed on September 13, 2003 and September 19, 2003 and September 29, 2003. The individual and average relative response factors (RRFs) for all target analytes met the

Data Validation Report
Laboratory Report / Batch K2306923
Received October 14, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report: October 23, 2003

data validation requirement of >0.05 . All target analytes met the $<30\%$ RSD data validation criteria. The average RSD of the RRFs for PCP did exceed the method criteria of $<15\%$ RSD for each calibration. However, the laboratory utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being $<20\%$. The mean RSD for this initial calibration event was calculated as 5.7% for the September 19, 2003 calibration, 5.6% for the September 13, 2003 calibration, and 6.3% for the September 29, 2003 calibration, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard, all target analytes met the laboratory's criteria of $<20\%$ difference. There are no data validation criteria for second source standards.

Continuing Calibration Verification

The September 19, 20, 22 and 29, 2003 continuing calibration verifications (CCV) met the data validation criteria for frequency and the $<+25\%$ difference RRFs from ICAL as well as the minimum RRFs of >0.05 for all target analytes. The indeno (1,2,3- cd) pyrene results for the CCV on September 17, 2003 exceeded the % RSD criteria (33%). This was not a factor for the project samples as only PCP was analyzed on September 17, 2003. The PCP CCV for September 27, 2003 exceeded % RSD criteria (27%). All associated samples were qualified and should be considered estimated.

Method Blank Analysis

Both method blanks (soil and water) were non-detect for all target analytes. The method blanks samples were prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared four LCS samples, two corresponding to the soil analysis and two corresponding to the water analysis. PCP fell below laboratory criteria for LCS sample percent recovery in both the LCS and LCS duplicate associated with the water sample and the LCS associated with the soil samples. All

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Laboratory Report / Batch K2306923
Received October 14, 2003

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associated data has been qualified as estimated. All remaining spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Surrogate Standard Recovery

All surrogate spike recoveries from the project samples met the data validation acceptance criteria.

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project samples and associated quality control samples met the method and data validation criteria.

Matrix Spike Recovery

A non-project sample was utilized for the Matrix Spike (MS) and Matrix Spike Duplicate (MSD). Since the source of the sample that was spiked was a non-project, the data has limited applicability to the project data. All percent recoveries for PCP from the MS and MSD fell below the laboratory's internal control limits. All PCP results for the project samples have been qualified. Several PAH MS/MSD results fell outside laboratory acceptance criteria, however the results for acenaphthene, fluorene, phenanthrene and fluoranthene are not applicable as the native concentration was greater than four times the spiked amount. In addition to these compounds, anthracene and pyrene also fell below laboratory acceptance criteria. Since the LCS data and calibration data for the PAHs fell within laboratory acceptance criteria, no PAH data was qualified based on the MS/MSD results.

Field Duplicate Results

Field duplicate results evaluate overall sampling and laboratory precision. Samples BNSF-6 0-4" and BNSF-6 0-4"D served as the field duplicates for this submittal. Both the sample and the duplicate were non-detect for the target parameters.

**Data Validation Report – PAHs and PCP
Laboratory Report / Batch K2306727
Received: October 2, 2003 (Revision rec'd 10/7/03)**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: October 23, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment soil samples contained in the aforementioned report is complete as detailed below.

The analytical data were reviewed in accordance with the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

Six soil sample results are contained in this laboratory report. They are as follows:

C3-4 0-4"	C3-4 4-12"	TH-1 0-4"	A5-6 0-4"
A5-6 4-12"	A5-6 0-4"D		

Data Validation Report
Laboratory Report / Batch K2306727
Received October 2, 2003

St Regis Paper Company Superfund Site – Cass Lake Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report October 23 2003

Overall Assessment

No qualifiers were assigned to the sample results contained in laboratory report K2306727 as a result of data validation process. All data met the data quality objective (DQOs) and are useable as reported.

Holding Times, Preservation and Storage

The samples were collected on August 26, 2003 and received at the CAS Houston laboratory on September 4, 2003 with an accompanying chain-of-custody (COC) form. All samples were received intact and were properly chilled (within 2-6 °C). The samples were properly stored until shipped overnight to the CAS Kelso laboratory for PAH and PCP analysis. The samples were received on September 4, 2003 in Kelso intact and at 2.9 °C (temperature blank). Some of the labels on the sample containers had field sample identifiers that did not match the identifiers listed on the COC. The identifiers written on the sample container lids did match the identifiers written on the COC.

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for PAHs and PCP instrument using selected ion monitoring (SIM) was performed on September 13, 2003 using 5 concentrations levels of the target analytes. The individual and average relative response factors (RRFs) for all target analytes met the data validation

Data Validation Report
Laboratory Report / Batch K2306727
Received October 2, 2003

St Regis Paper Company Superfund Site - Cass Lake Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report October 23, 2003

requirement of >0.05 . All target analytes met the $<30\%$ RSD data validation criteria. The average RSD of the RRFs for PCP did exceed the method criteria of $<15\%$ RSD, however the laboratory utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being $<20\%$. The mean RSD for this initial calibration event was calculated as 5.7%, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard; all target analytes met the laboratory's criteria of $<20\%$ difference. There are no data validation criteria for second source standards.

Continuing Calibration Verification

The September 15 and 16, 2003 continuing calibration verifications met the data validation criteria for frequency and the $<+25\%$ difference RRFs from ICAL as well as the minimum RRFs of >0.05 for all target analytes. The September 17, 2003 continuing calibration verification met the data validation criteria with the exception of the %D for indeno (1, 2, 3-cd) pyrene. The %D exceeded the $<25\%$ criteria (33%) for this compound. Two dilutions of project samples were analyzed on September 17, only for final quantitation of fluoroanthene. Since indeno (1, 2, 3-cd) pyrene was not quantified with the analytical batch in question, the data is usable as reported.

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared two LCS samples. All spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

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Laboratory Report / Batch K2306727
Received October 2 2003

St Regis Paper Company Superfund Site - Cass Lake Minnesota
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Date of Validation Report October 23, 2003

Surrogate Standard Recovery

The matrix spike associated with the project samples had two surrogate standards and the laboratory control sample had one surrogate standard exceed laboratory criteria. Since the majority of matrix spike values and all laboratory control sample spike values met all percent recovery criteria, it does not appear these exceedances adversely affected the data. All remaining surrogate spike recoveries from the project samples met the data validation acceptance criteria.

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project samples and associated quality control samples met the method and data validation criteria.

Matrix Spike Recovery

All percent recoveries and RPDs from the MS and MSD were within the laboratory's internal control limits, with the exception of the matrix spike recovery for indeno (1, 2, 3-cd) pyrene and pentachlorophenol. The recovery for indeno(1,2,3-cd) pyrene was slightly above the laboratory criteria of 33-133% at 135%. Since the matrix spike duplicate and laboratory control sample percent recoveries met established criteria for this compound, it is unlikely that this affected the project samples and therefore, no data are qualified. The pentachlorophenol concentrations in the native sample (A5-6-4-12) was greater than four times the spiked concentration, therefore the pentachlorophenol spike results are not applicable.

Field Duplicate Results

Field duplicate results evaluate overall sampling and laboratory precision. Samples A5-06-0-4 and A5-06-0-4D served as the field duplicates for this submittal. The average Relative Percent Difference (RPD) for the detected compounds was approximately 15.2%. These average RPD results displayed an acceptable level of precision for the low level nature of the analytical method.

**Data Validation Report – PAHs, PCP and PCB
Laboratory Report / Batch K2306721
Received: October 18, 2003**

**St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment**

Date of Validation Report: October 23, 2003

The data validation process for the Columbia Analytical Services, Inc (CAS) laboratory data for the polynuclear aromatic hydrocarbons (PAHs), pentachlorophenol (PCP) and polychlorinated biphenyls (PCB) analysis of the Cass Lake Removal Site Evaluation and Supplemental Assessment groundwater sample contained in the aforementioned report is complete as detailed below.

The analytical data were reviewed in accordance with the U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (1999) (Guidelines) as specified in the Quality Assurance Project Plan (QAPP, Barr, June 2003). In addition to the Guidelines, specific SW-846 Method 8270, 8082 and 8000 criteria were also considered as slight differences in some of the performance aspects exist between the documents. In general, the areas covered by the validation process include:

- Overall assessment
- Holding times, preservation and storage
- Instrument performance (tuning)
- Initial calibration
- Continuing calibration verification
- Method blank analysis
- Laboratory control samples (ongoing precision/accuracy)
- Surrogate recovery
- Internal standard recovery
- Matrix spike recovery

One groundwater sample results are contained in this laboratory report. It is as follows:

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Data Validation Report
Laboratory Report / Batch K2306721
Received October 14, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
Removal Site Evaluation and Supplemental Assessment

Date of Validation Report October 23 2003

Overall Assessment

Data qualifiers were assigned to the sample results contained in laboratory report K2306721 as a result of data validation process. All samples were extracted past their analytical holding times to do laboratory shipping errors. In addition, PCP initial calibration, laboratory control sample and matrix spike/matrix spike results did not meet laboratory criteria. The PCB analysis was performed following EPA Method 8082 and met all the method requirements. Since the Guidelines are based on a combination of Methods 8081 and Method 8082, several review items included in the Guidelines are not applicable to PCB analysis.

All data met the data quality objective (DQOs) and are useable with qualification.

Holding Times, Preservation and Storage

The samples were collected on August 26, 2003 and received at the CAS Houston laboratory on September 2, 2003 with an accompanying chain-of-custody (COC) form. All samples were received intact and were properly chilled (within 2-6 °C). The samples were properly stored until shipped overnight to the CAS Kelso laboratory for PAH/PCP and PCB analysis. The samples were received on September 10, 2003 in Kelso intact and at 2.9 °C (temperature blank). Some of the labels on the sample containers had field sample identifiers that did not match the identifiers listed on the COC. The identifiers written on the sample container lids did match the identifiers written on the COC. As the samples were received at the Kelso laboratory past the analytical holding times for extraction, the holding times were not met. All analytical data were qualified and should be considered potentially biased low.

GCMS Instrument Performance Check

All instrument performance checks (tunes) run during the analysis of the project samples met the acceptance criteria for frequency, mass abundance, and mass ratios as listed in the CAS form 5s and associated quantitation and tune reports. Note, that the CAS tune criteria are somewhat different than those listed in the Guidelines and follow guidance from SW-846 8270. The CAS tune criteria reflect the mass spectrometer manufacturer's (Agilent) recommendations for tuning requirements for the latest generation of analytical mass spectral-based instrumentation. Section 7.3.1.2 and Table 3 of SW-846 8270C state that alternate tuning criteria may be used as long as they do not result in

Data Validation Report
Laboratory Report / Batch K2306721
Received October 14, 2003

St. Regis Paper Company Superfund Site – Cass Lake, Minnesota
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Date of Validation Report: October 23, 2003

adversely affected method performance. The 8270-method performance criteria were not adversely affected by the use of the alternate tuning criteria. The CAS tuning criteria limits were used as the analytical instrument system tuning performance criteria for this project. The tune frequency met the method and data validation criteria.

Initial Calibration

The initial calibration (ICAL) for the PAH and PCP instrument using selected ion monitoring (SIM) was performed on September 13, 2003 and the PCB instrument was calibrated on September 8, 2003. The individual and average relative response factors (RRFs) for all target analytes met the data validation requirement of >0.05 . All target analytes met the $<30\%$ RSD data validation criteria with the exception of PCP. The average RSD of the RRFs for PCP (26.0%) did exceed the method criteria of $<15\%$ RSD, however the laboratory utilized an alternative calibration evaluation as specified in section 7.5.1.2.1 of EPA 8000B. This option allows for the calculation of the mean value of all the target analytes average RSDs and subsequent comparison to the requirement of the mean RSD being $<20\%$. The mean RSD for this initial calibration event was calculated as 5.6%, thus meeting the method criteria.

The laboratory report also included results from the analysis of a second source calibration check standard; all target analytes met the laboratory's criteria of $<20\%$ difference with the exception of PCBs. The second PCB column (used for confirmation purposes) failed this 20% criterion. All data were quantified with the original column. There are no data validation criteria for second source standards.

Continuing Calibration Verification

The September 19, 2003 continuing calibration verification %RSD was above acceptance criteria for PCP. All associated data were qualified. All remaining CCVs met the data validation criteria for frequency and the $<+25\%$ difference RRFs from ICAL as well as the minimum RRFs of >0.05 for all target analytes.

Data Validation Report
Laboratory Report / Batch K2306721
Received October 14, 2003

St Regis Paper Company Superfund Site – Cass Lake, Minnesota
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Date of Validation Report: October 23, 2003

Method Blank Analysis

The method blank was non-detect for all target analytes. The method blank sample was prepared and analyzed at the appropriate frequency.

Laboratory Control Samples

For the verification of the analytical process/system/method accuracy, the laboratory prepared one LCS sample. The LCS results for PCP fell below laboratory acceptance criteria. All associated data were qualified. All remaining spiked analyte percent recoveries met the data validation and the laboratory's internal acceptance criteria.

Surrogate Standard Recovery

All surrogate spike recoveries from the project samples met the data validation acceptance criteria.

Internal Standard Recovery

All internal standards were reviewed for area and retention time criteria. All internal standards from the project samples and associated quality control samples met the method and data validation criteria.

Matrix Spike Recovery

A non-project sample was utilized for the Matrix Spike (MS) and Matrix Spike Duplicate (MSD). Since the source of the sample that was spiked was a non-project, the data has limited applicability to the project data. The percent recoveries for PCP fell below laboratory acceptance criteria. All associated data were qualified. All remaining percent recoveries and RPDs from the MS and MSD were within the laboratory's internal control limits.

Field Duplicate Results

No field duplicates were included with this data package.