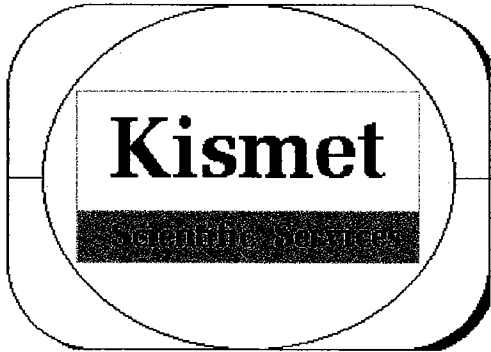


## **Appendix C**

### **Data Quality Review Report**



## Chemical Data Quality Review

### Haines Fairbanks Pipeline Herbicides Survey, AK

Work Order NO: 03-043

Prepared For: US Army Corps of Engineer, Alaska District

Project Received: 10/20/2003 and 11/28/03

Project Completed: 12/28/2003

Approved By: Ajmal M. Ilias

Dr. Ajmal M. Ilias  
President

<b>Kismet Scientific Services</b>	Project: Haines Fairbanks Pipeline Herbicides Survey, AK (03-043)	Version: 1.0
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## **Acronyms, Definition of Data Flags and Abbreviations:**

QC:	Quality Control
LCS:	Laboratory Control Samples
LCSD:	Laboratory Control Sample Duplicate
MS:	Matrix Spike
MSD:	Matrix Spike Duplicate
RPD:	Relative Percent Difference
N/R:	Not Reported
ND or --:	Not detected at MDL/MRL
PQL:	Practical Quantitation Limit
MRL:	Method Reporting Limit
MDL:	Method Detection Limit
J:	Estimated data detected below MRL/PQL
E:	Estimated data because of RPD failure or one or more other internal QC failure
B:	Found in the method blank as well as in the associated samples
Bu:	Qualified as undetected because of laboratory contamination
ADEC:	Alaska Department of Environmental Conservation
EMPC:	Estimated maximum possible concentration
µg:	Microgram
EPA:	Environmental Protection Agency
USACE:	United States Army Corps of Engineers
DQO:	Data Quality Objectives
CQAR:	Chemical Quality Assurance Report
HT:	Holding Time
ICV:	Initial Calibration Verification
CCV:	Continuing Calibration Verification
LE QC:	Laboratory Established Quality Control
TIC:	Tentatively Identified Compounds
WO:	Work Order
SPCC:	System Performance Check Compounds
CCC:	Calibration Check Compounds
Dup:	Duplicate analysis
NC:	NC = Can't be calculated, data of one or both duplicates are below the MRL
Q	Lower estimates due to quantitative interference
EMPC	Estimated maximum possible concentration

## 1. SUMMARY:

- 1.1 Positive results of the primary laboratories' data are summarized in Table I. The dioxin/furan method blanks were contaminated with 0.084 through 17.2 ppt (parts per trillion). Analytes detected within a factor of five to the method blank contamination are considered detected due to laboratory contamination and are flagged with "Bu" in Table I. Dioxin/furan analytes, when greater than five times the blank contamination are considered estimated data and are flagged with "J, B" in Table I. Analytes detected above ten times the blank contamination are not considered affected due to laboratory contamination. Caltest of California (CAL-CA) did not report method detection limits (MDL) and used high method reporting limits (MRL) of 1-2 ppm for carbamate pesticides, fenuron. Fenuron was not detected in any of the associated samples. If fenuron was present below the MRL of 1-2 ppm, it may not have been reported. Overall, data precision and accuracy of dioxin/furan, herbicides and fenuron are admissible based on the majority of passing internal quality control (QC) and blind duplicate data agreements.
- 1.2 The primary and quality assurance (QA) laboratories' data comparisons are shown in Tables IIA, IIB, IIIA and IIIB. All data agree, except for a minor disagreement of total tetrachlorodibenzofuran (Total TCDF) in Table IIA because of the QA laboratory's incorrect MRL reporting and possible overestimation of Total TCDF data by the primary laboratory, which is considered insignificant. For details, refer to Section 9 and comments of the respective tables.
- 1.3 Completeness: The completeness score is 93.6 % for dioxin/furan, 86.4 % for fenuron and 100 % for herbicides. The overall completeness score is 93.3 %, which meets the project completeness limits of 90 % and is admissible. For details, refer to section 11 of this report.

## 2. BACKGROUND:

The project (03-043) samples were collected from the Haines Fairbanks Pipeline Herbicides Survey, AK, site on August 30 through October 22, 2003. The primary samples were shipped to three laboratories, Paradigm Analytical Laboratories Inc. of North Carolina (PAL-NC), Severn Trent Laboratories Inc. of Seattle, Washington (STL-WA) and Caltest Analytical Laboratory of Napa, California (CAL-CA), on September 2 through October 23, 2003. The primary samples were received by these laboratories on September 3 through October 25, 2003. The quality assurance (QA) samples were shipped to Severn Trent Laboratories Inc. of Sacramento, California (STL-CA) and STL of Vermont (STL-VT) on September 2 and October 23, 2003. These samples were received at the QA Laboratories on September 3, 9 and October 25, 2003.

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### 3. OBJECTIVES:

- 3.1 Thirty two soil samples with three blind duplicates were collected to determine the extent of chemical contamination on the Haines Fairbanks Pipeline Herbicides Survey site of Alaska.
- 3.2 Two QA soil samples were submitted to evaluate the primary laboratories' partial data.

### 4. PROJECT ORGANIZATION:

- 4.1 The U.S. Army Corps of Engineers (USACE), Pacific Ocean Division, Alaska District, collected the project samples.
- 4.2 Paradigm Analytical Laboratories Inc. of North Carolina (PAL-NC), Severn Trent Laboratories Inc. of Seattle, Washington (STL-WA) and Caltest Analytical Laboratory of Napa, California (CAL-CA) analyzed the primary samples.
- 4.3 Severn Trent Laboratory Inc. of Sacramento, California (STL-CA) and STL of Colchester, Vermont (STL-VT) analyzed the QA samples.

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## 5. PROJECT FINDINGS:

All detected analytes are summarized in Table I. No chlorinated herbicides (herbicides) and carbamate pesticides (fenuron) were detected in any of the 32 soil samples above the MDL/MRL.

**TABLE I.** Dioxin/Furan reported by PAL-NC

Prefix: 03HFPH-  
Units: ng/Kg (ppt)

Matrix : Soil	Samples					
	Report: G379-6					
Analytes Detected	-01SO	-02SO	-03SO	-04SO	-05SO	-06SO
2,3,7,8-TCDD	--	--	0.196* J	0.113* J	0.117* J	0.173* J
1,2,3,7,8-PeCDD	--	--	0.283* J	0.119* J	0.0771* J	0.119* J
1,2,3,4,7,8-HxCDD	--	--	0.334* J	0.0877* J	0.114* J	0.139 J
1,2,3,6,7,8-HxCDD	--	--	0.931 J	0.124 J	0.066 J	0.377 J
1,2,3,7,8,9-HxCDD	--	--	0.624 J	0.168 J	0.0771* J	0.339 J
1,2,3,4,6,7,8-HpCDD	0.513 J	0.460 J	18.0	0.562 J	0.401 J	9.34 J
OCDD	2.26 J, Bu	1.75 J, Bu	117 B	2.95 J, B	1.76 J, Bu	81.2 B
2,3,7,8-TCDF	--	0.0676 J	0.177* J	0.0877 J	0.0877 J	0.146 J
1,2,3,7,8-PeCDF	--	--	0.196 J	0.126 J	0.0638* J	0.100 J
2,3,4,7,8-PeCDF	--	--	0.196 J, Bu	0.101 J, Bu	0.585 J, B	0.139 J, Bu
1,2,3,4,7,8-HxCDF	--	--	0.730 J	0.0954 J	0.0345 J	0.246 J
1,2,3,6,7,8-HxCDF	--	--	0.448 J, B	0.119 J, Bu	0.0638 J, Bu	0.169 J, Bu
2,3,4,6,7,8-HxCDF	--	--	0.483 J	0.0954 J	--	0.169 J
1,2,3,7,8,9-HxCDF	--	--	0.322 J	0.193 J	--	--
1,2,3,4,6,7,8-HpCDF	0.416 J,Bu	0.230 J,Bu	12.2 B	0.330 J, Bu	0.178 J, Bu	4.89 B
1,2,3,4,7,8,9-HpCDF	--	--	1.03 J	0.150* J	--	0.327 J
OCDF	--	--	24.9	0.706 J	0.157	17.2
Total TCDDs	--	0.228	1.58	--	0.0904	1.33
Total PeCDDs	--	--	2.47 B	--	--	0.116 Bu
Total HxCDDs	--	-	4.24	0.356	0.167	1.84
Total HpCDDs	0.808	0.676	27.7	0.810	0.401	14.7
Total TCDFs	0.732	0.295	4.97	0.598	0.749	6.82
Total PeCDFs	--	--	1.87 B	0.227 Bu	0.101 Bu	0.997 B
Total HxCDFs	--	--	14.7	0.534	0.162	6.39
Total HpCDFs	0.416	0.230 Bu	40.4 B	0.560 Bu	0.178 Bu	19.2 B

J = Estimated data reported below MRL/PQL

Bu = Qualified as undetected because of laboratory contamination

B = Found in the method blank as well as in the associated samples

-- = Not detected at MDL/MRL

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**TABLE I. Dioxin/Furan reported by PAL-NC  
Continued...**

Prefix: 03HFPH-  
Units: ng/Kg (ppt)

Matrix : Soil	Samples					
	Report: G379-6 Continued...			Report: G379-7		
Analytes Detected	-07SO	-09SO	-10SO	-11SO	-12SO	-13SO
2,3,7,8-TCDD	0.158* J	--	0.382* J	--	--	--
1,2,3,7,8-PeCDD	0.141 J	0.0766 J	1.71* J	0.0411* J	--	0.477* J
1,2,3,4,7,8-HxCDD	0.204* J	0.0893* J	3.52 J	0.135* J	--	--
1,2,3,6,7,8-HxCDD	0.506 J	0.120 J	12.6	--	--	--
1,2,3,7,8,9-HxCDD	0.404 J	0.112 J	8.42	--	--	--
1,2,3,4,6,7,8-HpCDD	11.2	1.17 J	251	0.803 J, Bu	0.708 J, Bu	0.458 J, Bu
OCDD	92.0 B	6.37 B	1870 B	5.19 J, Bu	4.55 J, Bu	1.67 J, Bu
2,3,7,8-TCDF	0.141* J	0.0919 J	0.394 J	0.0895 J	0.119 J	0.172 J
1,2,3,7,8-PeCDF	0.184 J	0.0638 J	1.29 J	0.0532* J	0.0732 J	--
2,3,4,7,8-PeCDF	0.174 J, Bu	0.069 J,Bu	3.4 J, Bu	0.0532	0.0732 J	0.763 J
1,2,3,4,7,8-HxCDF	0.401 J	0.0485* J	8.59	--	--	--
1,2,3,6,7,8-HxCDF	0.256* J,Bu	0.082 J,Bu	3.45 J, Bu	0.0508 J	0.763 J	--
2,3,4,6,7,8-HxCDF	0.293 J	0.0485 J	4.36 J	--	--	--
1,2,3,7,8,9-HxCDF	0.181 J	0.0434 J	2.94 J	--	--	--
1,2,3,4,6,7,8-HpCDF	5.78 B	0.311 J,Bu	87.1 B	0.220 J	0.186 J	0.210 J
1,2,3,4,7,8,9-HpCDF	0.440 J	--	4.04 J	--	--	--
OCDF	18.7	0.426 J	187	0.327* J, Bu	--	--
Total TCDDs	1.06	0.105	0.287	--	--	0.870
Total PeCDDs	0.283 Bu	0.138 Bu	5.17 B	--	--	0.446
Total HxCDDs	2.0	0.281	58.8	0.121	--	--
Total HpCDDs	17.7	2.0	412	2.02 J, Bu	1.18 Bu	0.458 Bu
Total TCDFs	3.81	0.511	8.64	0.581	0.375	4.39
Total PeCDFs	1.25 B	0.508 B	28.0 B	0.119	0.146	0.118
Total HxCDFs	7.9	0.454	188 B	0.186	0.0702	--
Total HpCDFs	21.0 B	0.605 Bu	324	0.220	0.317	--

J = Estimated data reported below MRL/PQL

Bu = Qualified as undetected because of laboratory contamination

B = Found in the method blank as well as in the associated samples

-- = Not detected at MDL/MRL

\* = Estimated maximum possible concentration

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**TABLE I. Dioxin/Furan reported by PAL-NC**

**Continued...**

Prefix: 03HFPH-  
Units: ng/Kg (ppt)

Matrix : Soil	Samples				
	Report:G379-7 Continued...	Report: G379-8			
Analytes Detected	-14SO	-15SO	-16SO	-17SO	-18SO
2,3,7,8-TCDD	--	--	--	--	--
1,2,3,7,8-PeCDD	--	0.0807 J, Bu	--	--	--
1,2,3,4,7,8-HxCDD	--	--	--	--	--
1,2,3,6,7,8-HxCDD	--	--	--	--	--
1,2,3,7,8,9-HxCDD	--	--	--	--	--
1,2,3,4,6,7,8-HpCDD	0.419 J, Bu	0.884 J, Bu	1.62 J, B	0.971 J, Bu	1.05 J, B
OCDD	1.49 J, B	5.71 J, B	9.48 J	85.91 J, B	6.84 B
2,3,7,8-TCDF	0.127 J	0.0138 J	0.236 J	0.117 J	0.106 J
1,2,3,7,8-PeCDF	0.088 J, Bu	--	--	--	--
2,3,4,7,8-PeCDF	0.0810 J, Bu	0.0773* J, Bu	0.162 J, Bu	0.0665 J, Bu	--
1,2,3,4,7,8-HxCDF	--	--	--	--	--
1,2,3,6,7,8-HxCDF	--	--	--	--	--
2,3,4,6,7,8-HxCDF	--	--	--	--	--
1,2,3,7,8,9-HxCDF	--	--	--	--	--
1,2,3,4,6,7,8-HpCDF	0.183 J	0.498* J	0.540 J	0.194 J	0.234 J
1,2,3,4,7,8,9-HpCDF	--	--	--	--	--
OCDF	--	0.477	1.01 J	0.367 J	0.282 J
Total TCDDs	--	0.356	0.462	0.336	0.326
Total PeCDDs	--	0.202	--	--	--
Total HxCDDs	--	--	--	--	--
Total HpCDDs	0.630 Bu	1.51 B	2.72 B	1.50 B	1.84 B
Total TCDFs	1.98	1.68	12.2	0.800	0.870
Total PeCDFs	0.711	--	0.804 Bu	0.0663 Bu	--
Total HxCDFs	--	--	0.254 Bu	--	--
Total HpCDFs	0.183	--	1.01	0.194	0.234

J = Estimated data reported below MRL/PQL  
 Bu = Qualified as undetected because of laboratory contamination  
 B = Found in the method blank as well as in the associated samples  
 -- = Not detected at MDL/MRL  
 \* = Estimated maximum possible concentration



**TABLE I. Dioxin/Furan reported by PAL-NC**

Prefix: 03HFPH-  
Units: ng/Kg (ppt)

Continued...

Matrix : Soil	Samples				
	Report:G379-8 Continued...				
Analytes Detected	-20SO	-21SO	-22SO	-23SO	-24SO
2,3,7,8-TCDD	--	--	0.199* J	--	--
1,2,3,7,8-PeCDD	--	--	1.37 J	0.196* J, Bu	--
1,2,3,4,7,8-HxCDD	--	--	5.51	--	--
1,2,3,6,7,8-HxCDD	--	--	18.7	0.23* J	--
1,2,3,7,8,9-HxCDD	--	--	10.5 B	0.187 J, Bu	--
1,2,3,4,6,7,8-HpCDD	0.940 J, Bu	1.04 J, B	912 B	3.82 B	2.22 J, B
OCDD	6.0 J, B	6.12 B	12200 E, B	26.1 B	14.3 B
2,3,7,8-TCDF	0.118* J	0.086 J	0.111 J	0.130 J	0.092 J
1,2,3,7,8-PeCDF	--	--	0.177 J, Bu	--	--
2,3,4,7,8-PeCDF	--	--	0.265 J, Bu	0.0663 J, Bu	0.0741 J, Bu
1,2,3,4,7,8-HxCDF	--	--	5.36 B	--	0.0628* J, Bu
1,2,3,6,7,8-HxCDF	--	0.0816 J, Bu	2.07 J, B	--	0.0853 J, Bu
2,3,4,6,7,8-HxCDF	--	--	4.96	--	--
1,2,3,7,8,9-HxCDF	--	--	1.08 J, B	--	--
1,2,3,4,6,7,8-HpCDF	0.268 J	0.342 J	268	0.891 J	0.485 J
1,2,3,4,7,8,9-HpCDF	--	--	19.1	--	--
OCDF	0.252 J	0.527 J	2700 E	3.26 J	1.40 J
Total TCDDs	0.147	0.086	0.147	0.152	--
Total PeCDDs	--	0.168	7.33	0.154	0.0920
Total HxCDDs	--	0.196	86.5	0.73	--
Total HpCDDs	1.28 B	2.47 B	1360 B	6.21 B	3.86 B
Total TCDFs	0.179	0.432	2.04	0.751	0.0920
Total PeCDFs	--	0.135 Bu	6.97 B	--	0.195 Bu
Total HxCDFs	--	0.238 Bu	170 B	0.706 Bu	0.483 Bu
Total HpCDFs	0.268	0.626	1550	2.32	1.28

J = Estimated data reported below MRL/PQL

Bu = Qualified as undetected because of laboratory contamination

B = Found in the method blank as well as in the associated samples

-- = Not detected at MDL/MRL

\* = Estimated maximum possible concentration

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**TABLE I. Dioxin/Furan reported by PAL-NC  
Continued...**

Prefix: 03HFPH-  
Units: ng/Kg (ppt)

Matrix : Soil	Samples				
	Report:G379-8 Continued...				
Analytes Detected	-25SO	-26SO	-27SO	-28SO	-29SO
2,3,7,8-TCDD	--	--	--	0.326* J	--
1,2,3,7,8-PeCDD	0.167* J, Bu	0.127* J, Bu	--	--	0.294* J, Bu
1,2,3,4,7,8-HxCDD	--	--	--	--	0.345 J, Bu
1,2,3,6,7,8-HxCDD	0.23 J	--	--	0.102 J, Bu	4.61 B
1,2,3,7,8,9-HxCDD	0.190* J, Bu	--	--	--	1.15 J, Bu
1,2,3,4,6,7,8-HpCDD	2.53 J, B	0.941 J, Bu	0.465 J, Bu	0.948 J, Bu	77.9 B
OCDD	17.8 B	5.01 J, Bu	1.94 Bu	5.11 J, B	480 B
2,3,7,8-TCDF	0.115 J	0.084 J	--	0.162 J, Bu	0.255 J, Bu
1,2,3,7,8-PeCDF	0.0658 J, Bu	0.054* J, Bu	--	0.074* J, Bu	0.260 J, Bu
2,3,4,7,8-PeCDF	0.0894 J, Bu	0.063* J, Bu	--	--	0.541 J, Bu
1,2,3,4,7,8-HxCDF	--	0.054* J, Bu	--	--	0.620 J, Bu
1,2,3,6,7,8-HxCDF	0.101 J, Bu	0.0775 J, Bu	--	0.067 J, Bu	0.543 J, Bu
2,3,4,6,7,8-HxCDF	--	--	--	--	1.01 J, B
1,2,3,7,8,9-HxCDF	--	--	--	--	0.504 J, Bu
1,2,3,4,6,7,8-HpCDF	0.844 J	0.241 J	0.343 J, Bu	0.305 J, Bu	11.7 B
1,2,3,4,7,8,9-HpCDF	--	--	--	--	0.467* J, Bu
OCDF	2.30 J	0.291 J	0.594* J, Bu	0.548 J, Bu	9.10 B
Total TCDDs	0.202	0.112	--	--	--
Total PeCDDs	0.350	0.187	--	--	0.507 Bu
Total HxCDDs	0.748	--	--	0.277 Bu	14.7 B
Total HpCDDs	5.23 B	0.941 Bu	0.693 Bu	1.87 B	124 B
Total TCDFs	0.623	0.532	--	0.569	1.19
Total PeCDFs	0.292 Bu	0.230 Bu	--	0.118 Bu	3.54 B
Total HxCDFs	0.623 Bu	0.261 Bu	--	0.067 Bu	29.4 B
Total HpCDFs	1.96	0.383	0.724 Bu	0.702 Bu	33 B

J = Estimated data reported below MRL/PQL  
 Bu = Qualified as undetected because of laboratory contamination  
 B = Found in the method blank as well as in the associated samples  
 -- = Not detected at MDL/MRL  
 \* = Estimated maximum possible concentration  
 E = Estimated data Exceeded calibration limits

**TABLE I. Dioxin/Furan reported by PAL-NC  
Continued...**

Prefix: 03HFPH-  
Units: ng/Kg (ppt)

Matrix : Soil	Samples				
	Report:G379-8 Continued...				
Analytes Detected	-30SO	-31SO	-32SO	-33SO	-34SO
2,3,7,8-TCDD	--	--	--	--	--
1,2,3,7,8-PeCDD	0.664 J, Bu	--	0.318* J, Bu	1.1 J, Bu	--
1,2,3,4,7,8-HxCDD	0.785 J, Bu	--	--	2.37 J, B	--
1,2,3,6,7,8-HxCDD	7.78 B	--	0.453* J, Bu	10.9 B	--
1,2,3,7,8,9-HxCDD	2.12 J, B	--	0.479 J, Bu	7.51 B	0.204 J, Bu
1,2,3,4,6,7,8-HpCDD	143 B	0.650 J, Bu	5.57 J, B	270 B	1.04 J, Bu
OCDD	994 B	2.82 J, Bu	35.9 B	1860 B	4.94 J, B
2,3,7,8-TCDF	0.389* J, Bu	--	0.475 J, B	0.248 J, Bu	--
1,2,3,7,8-PeCDF	0.583 J, Bu	0.115 J, Bu	0.345 J, Bu	0.378 J, Bu	--
2,3,4,7,8-PeCDF	1.06 J, Bu	--	0.264 J, Bu	0.692 J, Bu	0.105 J, Bu
1,2,3,4,7,8-HxCDF	1.48 J, Bu	--	0.246 J, Bu	2.34 J, B	--
1,2,3,6,7,8-HxCDF	1.06 J, B	0.127 J, Bu	0.282 J, Bu	1.43 J, B	0.130 J, Bu
2,3,4,6,7,8-HxCDF	1.99 J, B	--	0.170 J, Bu	1.83 J, B	--
1,2,3,7,8,9-HxCDF	1.19 J, B	--	--	0.378 J, Bu	--
1,2,3,4,6,7,8-HpCDF	22.3 B	0.184 J, Bu	1.41 J, B	54.3 B	0.582 J, Bu
1,2,3,4,7,8,9-HpCDF	1.02 J, B	--	--	4.54 B	--
OCDF	16.8 B	--	4.06 B	360 B	--
Total TCDDs	--	--	0.533	3.85	--
Total PeCDDs	2.59 B	--	1.78 B	5.54 B	--
Total HxCDDs	25.5 B	--	3.39 B	61.0 B	0.359 Bu
Total HpCDDs	231 B	0.977 Bu	10.5 B	448 B	1.88 B
Total TCDFs	2.18	--	12.2	8.96	--
Total PeCDFs	6.53 B	0.115 Bu	2.50 B	8.59 B	0.189 Bu
Total HxCDFs	53.2 B	0.127 Bu	2.55 B	50.2 B	0.130 Bu
Total HpCDFs	66.8 B	0.471 Bu	4.84 B	225 B	0.582 Bu

**Summary:** 0.0771 through 12200 ppt of dioxin and 0.0345 through 2700 ppt of furan were detected in this tier of analysis. Analytes detected within a factor of five to the method blank contamination were considered detected due to laboratory contamination and are flagged with "Bu". Analytes detected within a factor of 5 to 10 of the method blank contamination are considered estimated data and are flagged with "J, B". Analytes detected above ten times the method blank contamination are not considered affected due to laboratory contamination. Data flagged with "E" should be viewed estimated data because analytes concentration exceeded the calibration range.

J = Estimated data reported below MRL/PQL

-- = Not detected at MDL/MRL

B = Found in the method blank as well as in the associated samples

Bu = Qualified as undetected because of laboratory contamination

\* = Estimated maximum possible concentration

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## 6. ANALYTICAL REFERENCES:

Number	Title	Date
EPA SW-846, Third Edition	Test Methods for Evaluating Solid Waste	12/96
EPA-600/4-79-020	Methods for Chemical Analysis of Water and Waste	3/86
ADEC UST Procedure Manual, 18AAC78, Appendices D, E, & F	Guidance for treatment of Petroleum Contaminated Soil and Water at UST Site	11/02
USACE EM200-1-6	USACE Chemical Quality Assurance Report	10/97
DOD	Quality System Manual for Environmental Laboratories	06/02
USACE Shell Document	QA/QC Criteria Development	11/98

## 7. EVALUATION OF THE PRIMARY LABORATORIES' DATA:

7.1 Primary Laboratories' Methods: The following is a listing of preparations and analytical methods as reported in the laboratories' data deliverable.

Primary Laboratory	Parameters	Preparation Methods	Analytical Methods	COC
PAL-North Carolina	Dioxin/Furan	3540C	EPA 8290	8290 Dioxin
STL-WA	Herbicides	--	EPA 8151GC/MS	EPA8151Herbicides
CAL-CA	Fenuron	EPA 3540C	EPA 8321	8321 Fenuron

COC = Chain of Custody Requested Methods

7.1.1 Method Deviations: The analytical methods employed for analyses were compatible to the COC requested test methods and are admissible. STL-WA did not report the sample preparation method for herbicides. STL-WA used the EPA8151-GC/MS modified method instead of EPA method 8151A but, Overall met the COC requested test methods.

7.1.2 Proof of Data Review: The primary laboratories data were reviewed by at least two other staff in addition to the analyst.

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7.2 Chain of Custody Records and Sample Cooler Receipt Forms: All chain of custody (COC) records, sample shipping and preservation conditions, as documented on the sample cooler receipt (SCR) form, were evaluated according to the EPA, and the USACE ER1110-1-263 regulations. The following additional notations were made.

PAL-NC Report: G379-6 through G379-9	The sample coolers were received at cooler temperatures of 4.1 C°, 0.3 C°, 4.1 C°, and 1.3 C°. Two of the four cooler temperature readings did not meet the EPA recommended cooling preservation range of 4 ± 2 C°. Since the soil samples were not frozen, low cooling preservation did not adversely impact the soil dioxin/furan samples.
STL-WA Reports: 115863,115980, 115894 and 117173	Four sample coolers were received at cooler temperatures of 3.0 C° through 4.7 C° with temperature blank readings of 1 C° through 4.0 C°, which met the EPA recommended cooling preservation range of 4 ± 2 C°.
CAL-CA Reports: D090095, D090219, D090304 and D100846	Four coolers were received at cooler temperatures of 1.6 C° through 5.4 C°. Three of the four cooler temperatures met the EPA recommended sample cooling preservation range of 4 ± 2 C°. The other cooler (CAL Report D090304) was 1.6 C° at arrival. Since these samples were not frozen, the analysis of fenuron (carbamate pesticide) soil samples were not affected.

7.3 Sample Holding Times (HT), Method Detection Limits (MDL)/Practical Quantitation Limits (PQL), Method Blanks, Extraction Efficiency, Accuracy and Precision: Sample HT, MDL/PQL or method reporting limits (MRL), initial calibration verification (ICV), and continuing calibration verification (CCV), where applicable, were evaluated using the ADEC and EPA method criteria. The laboratory's blanks were evaluated for the absence of targeted analytes. The extraction efficiencies (EE), accuracy and precision of the data were represented by surrogates recovery, matrix spike (MS), matrix spikes duplicates (MSD), laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) recoveries, and relative percent difference (RPD) results. The laboratory's reported surrogate, MS/MSD, LCS/LCSD recoveries and RPDs were compared to the EPA, applicable Corps of Engineers QC criteria and/or laboratory established Quality Control (LE QC) acceptance limits for out of control results. Field blind duplicates (Tables IIA through IVC) were evaluated to meet the partial audit requirements of the primary laboratory.

7.3.1 Dioxin/Furan (EPA Method 8290) reported by PAL-NC:

7.3.1.1 Holding Time: Dioxin and furan samples were collected on August 30 through September 6, October 21 and 22, 2003. These samples were extracted on September 4, 8, 11 and November 6 and were analyzed on September 8, 11, 14, 15 and November 8 and 9, 2003. All samples were extracted within 16 days and analyzed within 19 days of sample

collection, which met the EPA specified extraction holding time of 30 days and analysis holding time of 40 days.

**TABLE 7.3.1.1: Dioxin/Furan Holding Times**

Project: Haines Fairbanks Pipeline Herbicides Survey  
 Matrix: Soil  
 Prefix: 03HFPH-

Sample ID	Date Collected	Extraction Date	Date Analyzed	ADEC/EPA H.T.	
				EX	AN
-01SO through -07SO -09SO and -10SO	8/30 & 31/03 and 9/1/03	9/4/03	9/8 through 9/11/03	30 days	40 days
-11SO through -14SO	9/2 & 3/03	9/8/03	9/15/03	"	"
-15SO through -18SO -20SO through -26SO	9/4 & 5/03 and 9/6/03	9/11/03	9/14 & 15/03	"	"
-27SO through -34SO	10/21 & 22/03	11/6/03	10/8 & 9/03	"	"

EX = EPA extraction holding time; AN = EPA analysis holding time

7.3.1.2 Method Blanks: Four sets of method blanks were analyzed. Four to 22 analytes including total dioxin and furan ranging in concentration from 0.08 through 17.2 ppt were detected in the method blanks. Analytes detected within a factor of five to the method blank contamination were considered detected because of laboratory contamination and are flagged with "Bu" in Table I. Analytes detected between a factor of five to ten to the method blanks are viewed as estimated data and flagged with "J, B" in Table I. Analytes detected above a factor of ten to the method blank were not considered affected by laboratory contamination.

**TABLE 7.3.1.2: Dioxin/Furan Method Blanks**

Project: Haines Fairbanks Pipeline Herbicides Survey  
 Matrix: Soil  
 Unit: ng/kg (ppt)

Analytes Detected	PAL Reports and Associated Samples	Method Blank Levels
OCDD	<b>Report G379-6:</b> ( 03HFPH01SO through -07SO, -09SO and -10SO)	0.542 J
2,3,4,7,8 PeCDF	"	0.09 J
1,2,3,6,7,8-HxCDF	"	0.0840 J
1,2,3,4,5,7,8-HpCDF	"	0.122 J
Total PeCDDs	"	0.342 J
Total PeCDFs	"	0.0900 J
Total HpCDFs	"	0.122
1,2,3,4,6,7,8-HpCDD	<b>Report G379-7:</b> ( 03HFPH11SO through -14SO)	1.59 J
OCDD	"	17.2
OCDF	"	1.64 J
Total HpCDDs	"	1.59
1,2,3,7,8-PeCDD	<b>Report G379-8:</b> ( 03HFPH15SO through -18SO, -20SO through -26SO)	0.120* J
1,2,3,7,8,9-HxCDD	"	0.142* J
1,2,3,4,7,8-HxCDD	"	0.196 J
OCDD	"	0.946 J
1,2,3,7,8-PeCDF	"	0.124* J
2,3,4,7,8-PeCDF	"	0.126 J
1,2,3,4,7,8-HxCDF	"	0.110 J
1,2,3,6,7,8-HxCDF	"	0.126 J
1,2,3,7,8,9-HxCDF	"	0.166 J
Total HpCDDs	"	0.196
Total PeCDFs	"	0.126
Total HxCDFs	"	0.292

J = Estimated data reported below MRL/PQL

\* = Estimated maximum possible concentration

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**TABLE 7.3.1.2: Dioxin/Furan Method Blanks Continued...**

Project: Haines Fairbanks Pipeline Herbicides Survey  
 Matrix: Soil  
 Unit: ng/Kg (ppt)

Analytes Detected	PAL Reports and Associated Samples	Method Blank Levels
1,2,3,7,8-PeCDD	<b>Report G379-9: ( 03HFPH27SO through -34SO)</b>	0.176* J
1,2,3,4,7,8-HxCDD	"	0.130 J
1,2,3,6,7,8-HxCDD	"	0.184* J
1,2,3,7,8,9-HxCDD	"	0.248 J
1,2,3,4,6,7,8-HpCDD	"	0.228 J
OCDD	"	0.818 J
2,3,7,8-TCDF	"	0.0860* J
1,2,3,7,8-PeCDF	"	0.174 J
2,3,4,7,8-PeCDF	"	0.164 J
1,2,3,4,7,8-HxCDF	"	0.140 J
1,2,3,6,7,8-HxCDF	"	0.148 J
2,3,4,6,7,8-HxCDF	"	0.112 J
1,2,3,7,8,9-HxCDF	"	0.162 J
1,2,3,4,6,7,8-HpCDF	"	0.146 J
1,2,3,4,7,8,9-HpCDF	"	0.168 J
OCDF	"	0.418 J
Total PeCDDs	"	0.176
Total HxCDDs	"	0.378
Total HpCDDs	"	0.228
Total PeCDF	"	0.338
Total HxCDFs	"	0.400
Total HpCDF	"	0.314

J = Estimated data reported below MRL/PQL

\* = Estimated maximum possible concentration

7.3.1.3 **MDL/MRL:** A MDL of 0.121 through 0.954 ppt and a MRL of 0.5 through 5.0ppt were used for dioxin/furan sample analysis. The MDLs and MRLs used for dioxin/furan analysis met method specified PQLs.

7.3.1.4 **Internal Standards/Surrogates:** Nine labeled internal standard/surrogates and five labeled clean-up standard/surrogates were used in the sample extraction process to determine the extraction efficiencies of compounds of interest. All internal standard/surrogate recoveries met the method QC limit of 40-135%.

7.3.1.5 **LCS Recoveries:** LCS recoveries were 80 through 115%, which met the method and LE QC limit of 80-137% and are admissible.



7.3.1.6 ICV/CCV: All calibration related data met method specifications. The %D between the mean relative response factor (MRRF) and continuing calibration response factor (CCRRF) were between 0.82 and 15.5 which met the method and the LE QC limits of 20.

7.3.1.7 MS/MSD Recoveries and RPD: MS/MSD recoveries are performed to check the matrix effect and to determine the accuracy of analyte recovery from the matrix. Associated RPD are calculated to measure the precision between MS and MSD data. Two sets of MS/MSD recoveries were performed. All MS/MSD recoveries met the LE QC limit of 75-125% and RPD limits of 20, except for the RPD of OCDD in PAL-NC Report G379-6 which was 26.7%. Data precision of OCDD were admitted based on other passing RPDs (Report G379-6, spike sample 03HFPH06SO)

**TABLE 7.3.1.7: Dioxin/Furan MS/MSD Recoveries**

Analytes	Associated Samples	% Recoveries			LE QC Limits	
		MS	MSD	RPD	% Recovery	RPD
<b>PAL-NC Report: G379-9 OCDD</b>	03HFPH29SO	85.9	112	<b>26.7</b>	75-125	20

7.3.1.8 Blind Duplicates: Dioxin/Furan blind duplicate data are shown in Tables IIA, IIIA and IVA. All data agree.

7.3.1.9 Overall Evaluation of Dioxin/Furan Data: Dioxin and furan data are admissible based on the majority of passing internal QC data and blind duplicate data agreements, except for low levels of 22 analytes detected in the method blank at 0.08 through 17.2 ppt. Analytes detected in the associated samples within a factor of five to the blank contamination are qualified with "Bu" as undetected because of laboratory contamination (Table I). Analytes detected within a factor of five to ten of the method blank contamination are considered estimated data and flagged with "J, B" (Table I).

7.3.2 Solvent Extractable Carbamate Pesticide (Fenuron) Analyzed by EPA Method 8321:

7.3.2.1 Holding Time: All samples (preserved) were extracted within 14 days. Samples were collected from August 30 through September 5, October 21 and 22, extracted on September 10, 17 and November 4, and analyzed on September 24 and November 7, 2003, which met the EPA extraction holding times of 14 days and analysis holding time of 40 days.

**TABLE 7.3.2.1: Carbamate Pesticide (Fenuron) Holding Time**

Project: Haines Fairbanks Pipeline Herbicides Survey  
 Matrix: Soil  
 Prefix: 03HFPH-

Sample ID	Date Collected	Extraction Date	Date Analyzed	ADEC/EPA H.T.	
				EX	AN
-01SO through -07SO -09SO and -10SO	8/30/03 through 9/1/03	9/10/03	9/24/03	14 days	40 days
-11SO through -14SO	9/2 and 3/03	9/10/03	9/24/03	"	"
-15SO through -18SO -20SO through -26SO	9/4 and 5/03	9/17/03	9/24/03	"	"
-27SO through -34SO	10/21 and 22/03	11/4/03	11/7/03	"	"

EX = EPA extraction holding time; AN = EPA analysis holding time

7.3.2.2 Method Blanks: Fenuron was not detected in any of the method blanks.

7.3.2.3 MDL/MRL: The MDL was not reported. The laboratory used a MRL of 1-2 ppm for fenuron which is about 100 times higher than the method specified MDL of 0.025 µg/g (0.025 ppm) (EPA Method 8321A-41 of SW846). Fenuron present below 1 ppm, may not have been reported.

7.3.2.4 Surrogate Recoveries: Two surrogates, tebuthiuron and chloroxuran, were used in the fenuran analysis to determine extraction efficiencies of analytes from the sample matrix. All surrogate recoveries were within the LE QC limit of 30-140%.

7.3.2.5 LCS/LCSD Recoveries and RPD: Two sets of LCS recoveries were performed. Both met the LE QC limits of 50-150%.

7.3.2.6 ICV/CCV: Seven point calibration was used. The correlation coefficients were greater than 0.9999 and are admissible. The ICV/CCV %D were below 20.

7.3.2.7 MS/MSD Recoveries and RPD: Two sets of MS/MSD recoveries were performed. The MS/MSD recoveries and RPD of fenuron were within the LE QC limit of 50-150%.

7.3.2.8 Blind Duplicates: Fenuron blind duplicate data are shown in Tables IIC, IIIC and IVC. All data agree.

7.3.2.9 Overall Evaluation of Fenuron Data: The laboratory did not report MDL and used a high MRL of 1-2 ppm. Analytes present below 1 ppm may not have been reported. Data precision and accuracy are admissible based on passing MS/MSD recoveries, associated RPD, LCS/LCSD recoveries and surrogate recoveries.

7.3.3 Chlorinated Herbicides (EPA Method 8151 GC/MS Modified):

7.3.3.1 Holding Time: Herbicide samples were collected from August 30 through September 5 and October 21 and 22, 2003. Samples were extracted on September 8, 15 and October 30, 2003 and were analyzed on September 12, 16 and November 6, 2003. All soil samples were extracted within 11 days and analyzed within 15 days of sample collection. Both the extraction and analysis holding time of these samples met the EPA specified extraction holding time of 14 days and analysis holding time of 40 days.

**TABLE 7.3.3.1: Chlorinated Herbicides**

Project: Haines Fairbanks Pipeline Herbicides Survey  
 Matrix: Soil  
 Prefix: 03HFPH-

Sample ID	Date Collected	Extraction Date	Date Analyzed	ADEC/ EPA H.T.	
				EX	AN
-01SO through -07SO, -09SO and -10SO	8/30 & 31/03 and 9/1/03	9/10/03	9/24/03	14 days	40 days
-11SO through -14SO	9/1 & 2/03	9/10/03	9/24/03	"	"
-15SO through -18SO, -20SO through -26SO	9/4 & 5/03	9/17/03	9/24/03	"	"
-27SO through -34SO	10/21/03 and 10/23/03	11/04/03	11/07/03	"	"

EX = EPA extraction holding time; AN = EPA analysis holding time

7.3.3.2 Method Blank: No herbicides were detected in the method blank.

7.3.3.3 MDL/MRL: A MDL of 1.65 through 11.6 ppb and a MRL of 9.1 through 26.2 ppb were used for herbicides soil sample analysis. The MDL and MRL used for soil sample analysis met the method required practical quantitation limits (PQLs).

- 7.3.3.4 Surrogate Recoveries: One surrogate, 1,2-Dichlorophenylacetic acid was used in the pesticides analysis to determine extraction efficiencies of analytes from the sample matrix. All surrogate recoveries were within the EPA method and LE QC limit of 53-135%.
- 7.3.3.5 LCS/LCSD (BS/BSD) Recoveries: LCS/LCSD recoveries and associated RPDs were within the method and LE QC limit, except for recoveries of picloram in one of four reports (Report 117173) where the LCS (31.7%) and LCSD (34.2%) were below 50-150%. Since no LE QC limits are established for picloram, the low recoveries are admissible.
- 7.3.3.6 Calibration and ICV/CCV: GC/MS tuning with decafluorotriphenylphosphine (DFTPP) met method specification. Six point calibration were performed. The % difference (%D) between average relative response factor (AgRRF) and continuing calibration response factor (CCRF) were less than 10, which is admissible.
- 7.3.3.7 MS/MSD and associated RPD: MS/MSD recoveries are performed to check the matrix effect and to determine the accuracy of analyte recovery from the matrix. Associated RPD are calculated to measure the precision between MS and MSD data. Three sets of MS/MSD recoveries were performed using targeted analytes. All MS/MSD recoveries met the QC limits except for the MS/MSD recoveries of 2,4-D in two of three sets and picloram MS/MSD in one of three sets. MS/MSD recoveries of 2,4-D were above the QC limits. Since no 2,4-D were detected in any of the associated sample, high recoveries did not adversely affect the 2,4-D data. Picloram MS/MSD (10.5/28.7 %) recoveries in one of the three sets were poor. Picloram data were admitted based on the other two sets of passing MS/MSD recoveries. MSD recovery of 2,4,5-T was above the LE QC limits in STL-WA Report 115980, but the MS and RPD met the QC limits. RPD of picloram in STL-WA was above the QC limit. Since picloram was not detected in any of the associated samples, high RPD did not adversely affect the data.

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**TABLE 7.3.3.7: Chlorinated Herbicides MS/MSD Recoveries and associated RPD**

Analytes	Associated Samples	% Recoveries			LE QC Limits	
		MS	MSD	RPD	% Recovery	RPD
<b>STL-WA Report:115863:</b> <b>SET: 1</b>						
2,4-D	03HFPH06SO	137	144	5	58-121	30
2,4,5-T	"	99.2	97.2	2	67-131	30
Picloram	"	75	87.4	15	50-150	30
<b>STL-WA Report:115980</b> <b>SET: 2</b>						
2,4-D	03HFPH26SO	161	200	22	58-121	30
2,4,5-T	"	131	140	6.6	67-131	30
Picloram	"	106	93.3	13	50-150	30
<b>STL-WA Report:117173</b> <b>Set: 3</b>						
2,4-D	03HFPH29SO	120	85	34	58-121	30
2,4,5-T	"	87.9	89.4	1.7	67-131	30
Picloram	"	10.5	28.7	93	50-150	30

7.3.3.8 Blind Duplicates: Blind duplicate data are shown in Tables IIB, IIIB and IVB. All data agree.

7.3.3.9 Overall Evaluation of Herbicides Data: Herbicides data are admissible based on the majority of passing internal QC data and blind duplicate data agreements. MS/MSD recoveries of 2,4-D were high in two of the three sets of MS/MSD recoveries. Since 2,4-D was not detected in any samples, data are not affected. Picloram MS/MSD recoveries were low in one of the three sets, but data were admitted based on other passing MS/MSD recoveries and surrogate recoveries.

## 8. EVALUATION OF THE QA LABORATORIES' DATA

8.1 QA Laboratories' Methods: The following is a listing of preparations and analytical methods as reported in the laboratories' data deliverable.

QA Laboratories	Parameters	Preparation Methods	Analytical Methods	COC
STL-CA	Dioxin/Furan	--	EPA 8290	8290 Dioxin/Furan
STL-VT	Herbicides	--	EPA 8151	Herbicides 8151A

-- = Not reported

8.1.1 Method Deviations. The laboratory used the methods listed on the COC. However, the laboratory did not list the sample preparation methods for dioxin/furan and herbicides.

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8.1.2 Proof of Data Review: Data generated by the QA laboratory was reviewed by the analyst and a second staff member.

8.2 COC Records and Sample Cooler Receipt Forms (SCR): All COC records and SCR forms were evaluated according to the EPA, and USACE ER110-1-263 regulations. The following additional notations were made.

STL-CA Report G31040235 and G31090334: The coolers' temperature recorded on the receipt at the laboratory were 2.0 and 4.0 C°, with a temperature blank of 3 C°, which met the EPA recommended temperature range of  $4 \pm 2$  C°.

STL-VT Report 95671 and 95790: The coolers' temperature recorded on the receipt at the laboratory were 2.0 and 3.0 C°, which met the EPA recommended temperature range of  $4 \pm 2$  C°.

8.3 Sample Holding Times (HT), Method Detection Limits (MDL)/Method Reporting Limits (MRL), Method Blanks, Extraction Efficiency, Accuracy and Precision: Sample HT, MDL/MRL, initial calibration verification (ICV), and continuing calibration verification (CCV), where applicable, were evaluated using the EPA method criteria. The laboratory's blanks were evaluated for the absence of targeted analytes. The extraction efficiencies (EE), accuracy and precision of the data were represented by surrogates recovery, matrix spike (MS), matrix spikes duplicates (MSD), laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) recoveries, and relative percent difference (RPD) results. The laboratory's reported surrogate, MS/MSD, LCS/LCSD recoveries and RPD were compared to the EPA and/or laboratory established Quality Control (LE QC) acceptance limits for out of control results.

8.3.1 Dioxin/Furan (EPA Method 8290) reported by STL-CA:

8.3.1.1 Holding Times: Dioxin/Furan samples were collected on September 1 and 4, extracted on September 8 and 10 and analyzed on September 11 and 12, 2003. Both soil samples were extracted within seven days and analyzed within 10 days of sample collection. Both extraction and analysis holding times of these samples met the EPA specified extraction holding time of 30 days and analysis holding time of 40 days.

8.3.1.2 Method Blanks: Two sets of method blanks were analyzed. No targeted analytes were detected.

8.3.1.3 MDL/MRL: A MDL of 0.29 through 10 ppt and a MRL of 0.46 through 21 ppt were used for dioxin/furan sample analysis. The MDLs and MRLs used for dioxin/furan analysis met method specified PQLs. The QA laboratory reported slightly higher values for MDL than MRL for some

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analytes (see Tables IIA and IIIA). MRLs are generally three to ten times higher than MDLs.

- 8.3.1.4 Internal Standard/Surrogate Recoveries: Nine labeled surrogates were used in the sample extraction process to determine the extraction efficiencies of compounds of interest. All internal standard/surrogate recoveries met the method and QC limits of 40-135%.
- 8.3.1.5 LCS Recoveries: LCS recoveries were 87.3 through 122, which met the method and LE QC limit of 59-150 and are admissible.
- 8.3.1.6 ICV/CCV: All calibration related data met method specifications. The %D between the mean relative response factor (MRRF) and continuing calibration response factor (CCRRF) were less than or equal to 10.8 and are admissible.
- 8.3.1.7 MS/MSD Recoveries and RPD: MS/MSD recoveries are performed to check the matrix effect and to determine the accuracy of analyte recovery from the matrix. Associated RPD are calculated to measure the precision between MS and MSD data. One set of MS/MSD recoveries were performed which met the LE QC limit of 59-150%. The RPD was below 25, which is admissible.
- 8.3.1.8 Overall Evaluation of Dioxin/Furan Data: Dioxin and furan data are admissible based on the majority of passing internal QC data, except for incorrect reporting of MRLs for some analytes.

8.3.2 Chlorinated Herbicides (EPA Method 8151) Analyzed by STL-VT:

- 8.3.2.1 Holding Time: Herbicide samples were collected from September 1 and 4, extracted on September 8 and 10 and analyzed on September 11 and 12, 2003. Both soil samples were extracted within 12 days and analyzed within 15 days of sample collection. Both extraction and analysis holding time of these samples met the EPA specified extraction holding time of 14 days and analysis holding time of 40 days.
- 8.3.2.2 Method Blank: No targeted analytes were detected in any of the method blanks.
- 8.3.2.3 MDL/MRL: The laboratory did not report MDL and used 30-300 ppb of MRL for herbicide analysis, which is above the method estimated detection limit of 0.1 through 66 ppb. Low levels of herbicides, if present below the MRL, may not have been reported.

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- 8.3.2.4 **Surrogate Recoveries:** Surrogates are used in the sample extraction process to determine the extraction efficiencies of compounds of interest. One surrogate, dichloroacetic acid, was used for herbicides analysis. All surrogate recoveries in the associated samples, 03HFPH08SO and 03HFPH19SO, were 84 and 95%, respectively, which met the LE QC limit of 10-202%.
- 8.3.2.5 **MS/MSD and Associated RPD:** MS/MSD recoveries are performed to check the matrix effect and to determine the accuracy of analyte recovery from the matrix. Associated RPDs are calculated to measure the precision between MS and MSD data. All MS/MSD recoveries (39-87%) met the LE QC limit of 10-146% and RPD ranged from 10 to 29, which also met the LE QC limit of 40.
- 8.3.2.6 **LCS/LCSD Recoveries:** Two sets of LCS/LCSD recoveries were performed. All LCS/LCSD recoveries were within the LE QC limit of 10-140%, except for the recoveries of picloram. The picloram LCS recoveries was zero percent, which did not meet the broad LE QC limit 10-146%. Since picloram was not detected in the sample and LCSD recoveries (85%) was within the QC limit, high LCS recovery did not adversely impact the data.

**TABLE 8.3.2.7: Chlorinated Herbicides LCS/LCSD Recoveries and associated RPD**

Analytes	Associated Samples	% Recoveries			LE QC Limits	
		LCS	LCSD	RPD	% Recovery	RPD
2,4-D	03HFPH19SO	70	90	25	10-146	40
2,4,5-T	"	70	85	19	10-146	40
Picloram	"	0	85	200	10-148	40

- 8.3.2.7 **Calibration and ICV/CCV:** The correlation coefficient was 0.994 or better. The percent difference (%D) between the average response factor and continuing calibration response factors were below 20, except for one CCV where %D for picloram as 23.7, which was admitted.
- 8.3.2.8 **Overall Evaluation of Herbicides Data:** The laboratory did not report MDL data and used a high MRL of 30-300 ppb. Low levels of targeted herbicides, if present below 30 ppb, may not have been reported. The laboratory used a broader LE QC limit of 10-202% for surrogate recoveries and 10-146% for LCS and matrix spike recoveries. Since no targeted analytes were detected in the sample, the extended QC limit did not adversely affect the QA herbicides data.



## 9. COMPARISON OF PRIMARY AND QA LABORATORIES' DATA

The primary blind duplicate and QA data comparisons are shown in Tables IIA, IIB, IIIA and IIIB. Dioxin/furan data comparisons are shown in Tables IIA and IIIA. Herbicides data comparisons are shown in Tables IIB and IIIB. All data agree, except for total TCDFs data in Table IIA due in part to the QA laboratory's incorrect MRL reporting. Individual tetrachlorodibenzofuran data agree. Therefore the disparity in total tetrachlorodibenzofuran data is likely due to estimation of Total TCDFs. The QA laboratory did not detect any TCDF above 0.29 ppt, but the primary laboratory reported individual TCDF at 0.141 and 0.146 ppt, below the MRL. Since the primary laboratory's individual TCDF data agree within a factor of three to the QA laboratory's MDL and data comparisons below the MRLs are not considered significant, total TCDF data comparisons are not considered significant for data comparison purposes. The intra and inter laboratory data for sample must be within a factor of five for soil to be considered in agreement. The primary and QA laboratories' MDL/MRL or PQL must be within a factor of 10 to be considered comparable. Estimated data (results which have been quantitated below the method reporting limits and qualified with a "J" flag) should not be considered significant for the purpose of data comparison. All primary and QA samples data agree, except for minor disagreement in total TCDF in Table IIA.

## 10. LESSONS LEARNED AND PROBLEMS ENCOUNTERED:

- 10.1 The QA laboratory, STL-VT, did not report MDL for herbicides and reported high MRL of 30 -300 ppb. Analytes present below the MRLs may not have been reported.
- 10.2 The primary laboratory, CAL-CA, did not report fenuron data of Sample 03HFPH14SO (Report D090095).
- 10.3 The primary laboratory, STL-WA, reported herbicides data by method EPA8151A in the raw data, but reported final results with EPA method 8151-GC/MS modified. It is not clear which method was actually used for the herbicides analysis.
- 10.4 The QA laboratory, STL-CA, reported higher or the same MDL values than MRL for certain dioxin and furon analytes (Tables IIA and IIIA)

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## 11. COMPLETENESS:

- 11.1 Dioxin/Furan: All dioxin/furan method blanks were contaminated with targeted analytes. The MDL was not reported for all targeted analytes. Therefore, the completeness score is 93.6%, which meets the minimum acceptability range of 90%.

Score Parameter	Score Assigned	Score Received for (Dioxin/furan)
Sample Preservation (COC and SCR)	10 Points	10 Points
MDL	10 Points	8 Points
MRL/PQL	10 Points	10 Points
ICV/CCV	10 Points	10 Points
Method Blank	10 Points	5 Points
Surrogate Recoveries	10 Points	10 Points
LCS/LCSD	10 Points	10 Points
MS/MSD Recoveries and RPD	10 Points	10 Points
Blind Duplicates	10 Points	10 Points
Holding Time	10 Points	10 Points
Correct Method Use	10 Points	10 Points
<b>Total Points:</b>	<b>110</b>	<b>103</b>

- 11.2 Carbamate Pesticides (Fenuron): The primary laboratory did not report MDL and used high MRLs. The completeness score is 86.4%, which did not meet the minimum acceptability range of 90%.

Score Parameter	Score Assigned	Score Received for (Fenuron)
Sample Preservation (COC and SCR)	10 Points	10 Points
MDL	10 Points	0 Points
MRL/PQL	10 Points	5 Points
ICV/CCV	10 Points	10 Points
Method Blank	10 Points	10 Points
Surrogate Recoveries	10 Points	10 Points
LCS/LCSD	10 Points	10 Points
MS/MSD Recoveries and RPD	10 Points	10 Points
Blind Duplicates	10 Points	10 Points
Holding Time	10 Points	10 Points
Correct Method Use	10 Points	10 Points
<b>Total Points:</b>	<b>110</b>	<b>95</b>

11.3 Chlorinated Herbicides: No data have been rejected. All data are useable. Therefore, the completeness score is 100%.

11.4 Overall Data Completeness: The total completeness score is 93.6% for dioxin/furan, 86.4% for fenuron and 100% for herbicides. Therefore, the overall completeness score is as follows:

Completeness = (Validation and acceptable data obtained)/(Total Data Planned) x 100

Completeness = 308/330 x 100 = 93.3%.

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**TABLE IIA.** Comparison of Primary and QA Dioxin and Furan Data

Prefix: 03HFPH-  
Units: ng/Kg (ppt)

PAL-NC (primary) Laboratory Methods: Dioxin/furan (EPA 5040C/EPA 8290)			STL-CA (QA) Laboratory Methods: Dioxin/furan (NR/EPA 8290)				
Matrix : Soil	Primary Laboratory				QA Laboratory		
Analytes Detected	Samples		Detection Limits		Sample	Detection Limits	
	-06SO	-07SO	MDL	MRL	-08SO	MDL	MRL
2,3,7,8-TCDD	0.173* J	0.158* J	0.164	0.5	ND	0.42	0.73
1,2,3,7,8-PeCDD	0119* J	0.141 J	0.481	2.5	ND	1.1	1.1
1,2,3,4,7,8-HxCDD	0.139 J	0.204	0.411	2.5	ND	1.0	0.96
1,2,3,6,7,8-HxCDD	0.377 J	0.536 J	0.25	2.5	ND	1.3	1.0
1,2,3,7,8,9-HxCDD	0.339 J	0.404 J	0.25	2.5	ND	1.1	0.94
1,2,3,4,6,7,8-HpCDD	9.34	11.2	0.30	2.5	13	1.0	10.0
OCDD	81.2	92.0	NR	5.0	96	10	21.0
2,3,7,8-TCDF	0.146 J	0.141* J	0.1	0.5	ND	0.29	0.46
1,2,3,7,8-PeCDF	0.100 J	0.184 J	0.25	2.5	ND	1.0	0.86
2,3,4,7,8-PeCDF	0.139 J, Bu	0.174 J, Bu	NR	2.5	ND	1.0	0.86
1,2,3,4,7,8-HxCDF	0.246 J	0.401 J	0.25	2.5	ND	1.0	1.1
1,2,3,6,7,8-HxCDF	0.169 J, Bu	0.256 J, Bu	0.25	2.5	ND	1.0	1.1
2,3,4,6,7,8-HxCDF	0.169 J	0.293 J	0.25	2.5	ND	1.0	1.1
1,2,3,7,8,9-HxCDF	ND	0.181 J	0.25	2.5	ND	1.0	1.4
1,2,3,4,6,7,8-HpCDF	4.89 B	5.72 B	NR	2.5	6.5 J	1.0	10.0
1,2,3,4,7,8,9-HpCDF	0.327 J	0.44 J	0.25	2.5	ND	1.0	0.96
OCDF	17.2	18.7	0.50	5.0	19 J	2.8	21
Total TCDDs	1.33	1.06	NR	NR	ND	0.84	0.84
Total PeCDDs	0.116 Bu	0.283 Bu	NR	NR	ND	1.1	1.1
Total HxCDDs	1.84	2.0	NR	NR	ND	1.0	1.0
Total HpCDDs	14.7	17.7	NR	NR	19	10.0	10.0
Total TCDFs	6.82	3.81	NR	NR	ND	0.67	0.67
Total PeCDFs	0.997 B	1.25 B	NR	NR	ND	0.99	0.99
Total HxCDFs	6.39	7.90	NR	NR	ND	2.9	2.9
Total HpCDFs	19.2 B	21.0 B	NR	NR	22.0	10.0	10.0

**Comments:** The primary blind duplicate and the QA dioxin/furan data agree within a factor of three to each other or their MDL/MRL, except for the data of QA TCDFs due to their incorrect MRL reporting. The QA laboratory reported the same MDL and MRL for total TCDFs and for many other analytes. For some analytes the MDL was higher than the MRL (see above table). Normally MRLs are three to ten times higher than MDL. Data comparison below the MRL are not considered significant.

J = Estimated data reported below MRL/PQL; \* = Estimated maximum possible concentration (EMPC)  
 ND = Not detected at MDL/MRL; B = Found in the method blank as well as in the associated samples  
 NR = Not reported  
 Bu = Qualified as undetected because of laboratory contamination

**TABLE IIB. Comparison of Primary and QA Herbicides Data**

STL-WA (primary) Laboratory Methods: Herbicides (NR/EPA 8151-GC/MS Mod.)					Project: HFP Herbicides Survey, AK		
					Prefix: 03HFPH- Units: µg/Kg		
					STL-VT (QA) Laboratory Methods: Herbicides (NR/EPA 8151)		
Matrix : Soil	Primary Laboratory				QA Laboratory		
Analytes Detected	Samples		Detection Limits		Sample	Detection Limits	
None	-06SO	-07SO	MDL	MRL	-08SO	MDL	MRL
	ND	ND	1.65-6.78	10.1-26.5	ND	NR	30-300

**Comments:** The primary blind duplicate and the QA herbicides data agree.

**TABLE IIC. Comparison of Primary Laboratory's Blind Duplicate Pesticides (fenuron) Data**

CAL-CA (Primary) Laboratory Methods: Fenuron(EPA 3540/EPA 8321)				Project: HFP Herbicides Survey, AK	
				Units: mg/Kg	
Matrix : Soil	Primary Laboratory's Data				
Analytes Detected	Sample		Detection Limits		
None	03HFPH06SO	03HFPH07SO	MDL	MRL	
	ND	ND	NR	1-2	

**Comments:** The primary laboratory's blind duplicate data agree.

J = Estimated data reported below MRL/PQL  
 ND = Not detected at MDL/MRL  
 N/A = Not applicable  
 NR = Not reported

**TABLE IIIA. Comparison of Primary and QA Dioxin and Furan Data**

PAL-NC (primary) Laboratory Methods: Dioxin/furan (EPA 5040C/EPA 8290)		Prefix: 03HFPH-						
		Units: ng/Kg (ppt)						
Matrix : Soil		Primary Laboratory				QA Laboratory		
		Samples		Detection Limits		Sample	Detection Limits	
Analytes Detected		-17SO	-18SO	MDL	MRL	-19SO	MDL	MRL
1,2,3,4,6,7,8-HpCDD		0.971 J, Bu	1.05 J, Bu	0.30	2.5	ND	0.59	0.90
OCDD		5.91 J, B	6.84 B	NR	5.0	ND	5.7	3.6
2,3,7,8-TCDF		0.117 J	0.106 J	NR	0.5	ND	0.17	0.2
2,3,4,7,8-PeCDF		0.066 J, Bu	ND	0.301	2.5	ND	0.59	0.3
1,2,3,4,7,8,9-HpCDF		0.194 J, Bu	0.234 J, Bu	0.25	2.5	ND	0.59	0.27
OCDF		0.367 J	0.282 J	0.50	5.0	ND	1.6	0.71
Total TCDDs		0.336	0.326	0.286	NR	ND	0.33	0.33
Total HpCDDs		1.50 B	1.84 B	NR	NR	ND	0.91	0.91
Total TCDFs		0.800 Bu	0.87 Bu	0.1	NR	ND	0.44	0.44
Total PeCDFs		0.0.063	ND	NR	NR	ND	0.34	0.34
Total HpCDFs		0.194	0.234	0.25	NR	ND	0.53	0.53

**Comments:** The primary blind duplicate and the QA dioxin/furan data agree within a factor of five to each other or their MDL/MRL. Data comparisons below the MRL are not considered significant.

J = Estimated data reported below MRL/PQL

\* = Estimated maximum possible concentration (EMPC)

ND = Not detected at MDL/MRL

B = Found in the method blank as well as in the associated samples

NR = Not reported

Bu = Qualified as undetected because of laboratory contamination

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**TABLE IIIB.** Comparison of Primary and QA Chlorinated Herbicides Data

					Project: HFP Herbicides Survey, AK		
					Prefix: 03FHPPH-		
					Units: µg/Kg		
STL-WA (primary) Laboratory Methods: Herbicides (NR/EPA 8151-GC/MS Mod.)				STL-VT (QA) Laboratory Methods: Herbicides(NR/EPA 8151)			
Matrix : Soil	Primary Laboratory				QA Laboratory		
Analytes Detected	Samples		Detection Limits		Sample	Detection Limits	
	-17SO	-18SO	MDL	MRL	-19SO	MDL	MRL
None	ND	ND	1.65-6.78	10.1-26.5	ND	NR	30-300

**Comments:** The primary blind duplicate and the QA laboratory's data agree.

**TABLE IIIC.** Comparison of Primary Laboratory's Blind Duplicate Pesticides (fenuron) Data

				Project: HFP-herbicides Survey, AK	
				Units: mg/kg	
CAL-CA (Primary) Laboratory Methods: Fenurate(EPA 3540/EPA 8321)					
Matrix : Soil	Primary Laboratory's Data				
Analytes Detected	Sample		Detection Limits		
	03HFPPH17SO	03HFPPH18SO	MDL	MRL	
None	ND	ND	NR	1-2	

**Comments:** The primary laboratory's blind duplicate data agree.

J = Estimated data reported below MRL/PQL

ND = Not detected at MDL/MRL

B = Found in the method blank as well as in the associated samples

NR = Not reported

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**TABLE IVA. Comparison of Primary and QA Dioxin and Furan Data**

Project: HFP Herbicides Survey, AK  
Units: ng/Kg (ppt)

PAL-NC (primary) Laboratory Methods: Dioxin/furan (EPA 3540C/EPA 8290)				
Matrix : Soil	Primary Laboratory			
Analytes Detected	Samples		Detection Limits	
	03HFPH29SO	03HFPH30SO	MDL	MRL
1,2,3,7,8-PeCDD	0.294 J	0.664 J	0.354	2.5
1,2,3,4,7,8-HxCDD	0.345 J	0.785 J	0.411	2.5
1,2,3,6,7,8-HxCDD	4.61	7.78	0.25	2.5
1,2,3,7,8,9-HxCDD	1.15 J	2.12 J	0.25	2.5
1,2,3,4,6,7,8-HpCDD	77.9	143	0.30	2.5
OCDD	480	994	NR	5.0
2,3,7,8-TCDF	0.255 J	0.389* J	0.218	2.5
1,2,3,7,8-PeCDF	0.260 J	0.583 J	0.25	2.5
2,3,4,7,8-PeCDF	0.541 J	1.06 J	NR	2.5
1,2,3,4,7,8-HxCDF	0.620 J	1.48 J	0.25	2.5
1,2,3,6,7,8-HxCDF	0.543 J	1.06 J	0.25	2.5
2,3,4,6,7,8-HxCDF	1.01 J	1.99 J	0.25	2.5
1,2,3,7,8,9-HxCDF	0.504 J	1.19 J	0.25	2.5
1,2,3,4,6,7,8-HpCDF	11.7	22.3	NR	2.5
1,2,3,4,7,8,9-HpCDF	0.467* J	1.02 J	0.365	2.5
OCDF	9.10	16.8	0.5	5.0
Total PeCDDs	0.507	2.59	NR	NR
Total HxCDDs	14.7	25.5	NR	NR
Total HpCDDs	124	231	NR	NR
Total TCDFs	1.19	2.18	NR	NR
Total PeCDFs	3.54	6.53	NR	NR
Total HxCDFs	29.4	53.2	NR	NR
Total HpCDFs	33.0	66.8	NR	NR

**Comments:** The primary blind duplicate data agree close to a factor of five to each other or their MDL/MRL. Data comparison below the MRL are not considered significant.

J = Estimated data reported below MRL/PQL  
 \* = Estimated maximum possible concentration (EMPC)  
 ND = Not detected at MDL/MRL  
 B = Found in the method blank as well as in the associated samples  
 NR = Not reported  
 Bu = Qualified as undetected because of laboratory contamination

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**TABLE IVB.** Comparison of Primary Laboratory's Chlorinated Herbicides Blind Duplicate Data

Project: HFP Herbicides Survey, AK
Prefix: 03HFPH-
Units: µg/Kg
STL-WA (primary) Laboratory Methods: Herbicides (NR/EPA 8151-GC/MS modified)
Matrix : Soil
Primary Laboratory's Data
Analytes Detected
Samples
Detection Limits
None
03HFPH29SO
03HFPH30SO
MDL
MRL
ND
ND
1.65-6.78
10.1-26.5

**Comments:** The primary laboratory's blind duplicate data agree.

**TABLE IVC.** Comparison of Primary Laboratory's Blind Duplicate Pesticides (fenuron) Data

Project: HFP Herbicides Survey, AK
Units: mg/kg
CAL-CA (Primary) Laboratory Methods: Fenuron(EPA 3540/EPA 8321)
Matrix : Soil
Primary Laboratory's Data
Analytes Detected
Sample
Detection Limits
None
03HFPH29SO
03HFPH30SO
MDL
MRL
ND
ND
NR
1-2

**Comments:** The primary laboratory's blind duplicate data agree.

J = Estimated data reported below MRL/PQL  
 ND = Not detected at MDL/MRL  
 B = Found in the method blank as well as in the associated samples