CDFG FISH AND WILDLIFE WATER POLLUTION CONTROL LABORATORY
DATA QUALITY ASSURANCE REPORT

Laboratory No.: L-150, 180, 195, 202, 231-00 **Page 1**
Project Title: 1999 Coastal Fish Contamination Program (CFCP Year 2)

CALIBRATION

Y ICAL or ICAL Summary & ICV/CCV included
Y ICAL, ICV/CCV criteria met
Y Standards labeled or correctly identified by data system
NA Tune criteria met and copy included (GCMS only)

QAQC VERIFICATION

Y Method blank and LCS frequencies were met
Y LCS and MB copies are included if applicable
Y LCS and Mb data are within control limits
Y SRM data complete
N SRM data within control limits
Y MS/MSD data complete if applicable
N MS/MSD data within control limits
N Precision results within control limits
Y Holding times were met
NA All samples within tune time (GCMS only)
Y If the batch QC data did not meet criteria, appropriate comments were made

SAMPLE ANALYSIS

Y Logbooks/Prep bench sheets are properly filled out
Y Manual integrations are reviewed
Y All raw data is included
Y All analytes are reported correctly
Y Correct reporting limits were used
Y Surrogate recovery data complete
N Surrogate recovery data within control limits
NA Spectra are present for all positive analytes (GCMS only)

Y - YES
N - NO
NA - Not Applicable

CHEMIST Kathleen Regalado DATE 12-5-2000

REVIEWER David Crane DATE 12-5-2000

PROJECT QA SUMMARY

Pesticides: Total no. of data points = 6,493
Total no. of data points rejected = 151 (2.3%)
Total no. of data points qualified = 151 (2.3%)
PCB Congeners: Total no. of data points = 7,248
Total no. of data points rejected = 0
Total no. of data points qualified = 0
Overall total no. of data points = 13,741
Overall total no. of data points rejected = 1.1% / Overall total no. of data points qualified = 1.1%

Summary Information

Name of Reviewer: D. Crane Title: Lab Director

Bench Sheet Numbers: 94, 95, 96, 97, 102, 103, 104, 106, 107, 108, 116 Samples Received: See Below

Required Samples		Required Samples	
Sample ID	Sample ID	Sample ID	Sample ID
BS 94 **(1998 CFCP RR)** 99-383 RR 99-0963 RR **(1999 CFCP L-150-00)** 99-1132 99-1135 99-1136 99-1137 99-1138 99-1140 99-1142 99-1206 99-1207 99-1208 **BS 95** **(1999 CFCP L-150-00)** 99-1059 99-1058 99-1064 99-1062 99-1077 99-1078 99-1080 99-1081 99-1083 99-1085 99-1088 **BS 96** **(1999 CFCP L-180-00)** 99-1464 99-1465 99-1098 99-1097 99-1091 99-1092 00-0373 00-0375 99-1462 99-1463 99-1093 99-1094 99-1096 99-1095 99-1759	**BS 97** **(1999 CFCP L-180-00)** 99-1760 99-1255 99-1256 99-1257 99-2525 99-2526 99-2527 99-1758 99-1989 99-1990 99-1991 99-1992 99-1466 99-1030 99-1093B **BS 102** **(1999 CFCP L-195-00)** 99-1250 99-1253(off) 99-1253(on) 99-1467 99-1468 99-1993 99-1994 99-1995 99-1996 00-0449 00-0453 99-1269 **BS 103** **(1999 CFCP L-195-00)** 99-1268 99-1266 99-1265 99-1263 99-1264 99-1214 99-1215 99-1217 99-1259 99-1260 99-1262 00-0673 00-0672	**BS 104** **(1998 CFCP RR)** 99-550 99-885 **BS 106** **(1999 CFCP L-202-00)** 00-0409 00-0411 99-1495 99-1497(skin on) 00-0670 99-1794 00-0667 00-0668 00-0450 00-0451 99-1497(skin off) 99-1498(skin on) 99-1498(skin off) 99-1499 99-1791 99-1792 00-0414 00-0406 **BS 107** **(1999 CFCP L-202-00)** 00-0407 99-1272 99-1099 99-1458 00-0752 00-0753(skin on) 00-0753(skin off) 99-1027 00-0617 00-0616 99-1248 99-1246 99-1247 00-0354 99-1089 99-1090 99-1190 00-0353	**BS 108** **(1999 CFCP L-231-00)** 00-0649 00-0712 00-0657 00-0457 00-0611 00-0458 00-0775 00-0664 00-0663 00-0661 00-0654 00-0609 00-0714 00-0713 00-0711 00-0659 00-0660 **BS 116** **(1999 CFCP L-202,195-00)** 99-1029 00.0333 00-0675 00-0725 99-1497(skin off) RR 99-1998 RR 99-1250 RR

2. Extraction Method Used / Extraction Completion Date(s): **SO-TISSUE-PREP.SOPv5 / May 2, 2000**

3. Number of Samples Analyzed: **135**

4. Number of concentrations levels used for instrument calibration: **7**

4.	Total No. of CCVs Required: (One for each 10-15 analyses)	30	Total No. of CCVs Reported:	30
5.	Total No. of CCBs Required: (One for each CCV)	30	Total No. of CCBs Reported:	30
6.	Total No. of Field Blanks Required: (One per site or per 10 samples, whichever is more frequent)	NA	Total No. of Field Blanks Reported:	NA
7.	Total No. of Method Blanks Required: (One per batch)	10	Total No. of Method Blanks Reported:	9
8.	Total No. of SRM analyses Required: (One per batch)	10	Total No. of SRM Analyses Reported:	9
9.	Total No. of MS/MSD samples Required: (One MS/MSD per batch)	10	Total No. of MS/MSD samples Reported:	9+1MSD
			Total No. of sample Duplicates Reported:	13
10.	Total No. sample Duplicates Required (One per 20 samples)	7		

Pesticide Data Inspection Checklist

11. Initial Calibration

1. Was a multiple point initial calibration performed*? ☒Yes ☐No
2. Were all sample concentrations reported within the calibration range? ☐Yes ☒No
3. If no, list bench sheet, sample number, analyte and dilution.

Bench Sheet	Sample	Analyte	Dilution Used
97	99-1255	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	99-1256	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	99-1257	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	99-1989	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	99-1466	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
102	99-1995	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	99-1996	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	99-1269	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	99-1250 dup	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
103	99-1265	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	99-1215	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	99-1262	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	00-0673	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	00-0672	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
107	00-0407	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
116	00-0725	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	00-0725 dup	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)
	99-1250 RR	p,p-DDE	1 - 11 (0.1 mL + 1.0 mL)

d. Did the initial calibration meet acceptance criteria? $R^2 \geq 0.995$ ☒Yes ☐No

*A three point (minimum) initial calibration should be performed for each Analyte; the RSD of the RFs of calibration standards ≤ 20%.

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12. Method Detection Limit (MDL)/Minimum Level (ML)

1. Did the laboratory demonstrate their ability to achieve the required MDL? ☒Yes ☐No
2. Did the initial calibration range encompass the ML? ☒Yes ☐No
3. Were all field samples detected below the ML reported as non-detects? ☒Yes ☐No
4. If the answer to item a, b, or c above was "no", describe problem:

Pesticide Data Inspection Checklist

13. Initial Calibration Verification (ICV) Initial Calibration Blanks (ICB):

a. Was an ICV run prior to field samples? ☒Yes ☐No

b. Were ICV results within the specified windows? (75-125% Rec) ☒Yes ☐No

c. Was the ICV preceded by an ICB? ☒Yes ☐No

d. Was the ICB free from contamination? ☒Yes ☐No

e. If any item in a-d above was answered "no", list problems below:

Analyte	Failed ICV Recovery	Concentration Detected in ICB	Affected Samples

14. Continuing Calibration Verification (CCV)/Continuing Calibration Blank (CCB)

a. Were CCVs run prior to each batch of 10-15 analyses on each instrument? ☒Yes ☐No

b. Were all CCV results within the specified windows" (75-125% Rec) ☐Yes ☒No

c. Was each CCV preceded by a CCB? ☒Yes ☐No

d. Was each CCB free from contamination? ☒Yes ☐No

e. If any item in a-d above was answered "no", list problems below:

Analyte	Affected Samples	Shifting Missing CCV/CCB		Failed CCV/CCB ID	
					DB5/DB17
Tedion	L-150-00		vial 36	70/74	
			vial 43	74/78	
"	L-180-00		vial 57	71/84	
			vial 64	64/75	
			vial 71	61/72	
			vial 76	59/74	
"	L-195-00 All CCVs Acceptable				
"	L-202-00		vial 36	71/71	
			vial 43	71/64	
			vial 50	61/64	
			vial 57	54/56	
"	L-231-00 All CCVs Acceptable				

Pesticide Data Inspection Checklist

15. Laboratory (Method) Blanks

a. Was a method blank analyzed for each instrument & sample batch? ☐ Yes ☒ No

b. Was each method blank demonstrated to be free from contamination? (<RL) ☐ Yes ☒ No

c. Were equipment blanks demonstrated to be free from contamination? **NA** ☐ Yes ☐ No

d. If the answer to item a or b was "no", document problems below:

Analyte	Affected Samples	Blank Concentration Reported	Shift Missing MB
All	BS 94		MB Lost During Extraction
Methoxychlor	BS 97	5.84 ppb	
Methoxychlor	BS 106	9.16 ppb	

16. Field Blanks **NOT APPLICABLE**

a. Was a field blank analyzed for each 10 samples per site? ☐ Yes ☐ No

b. Was each field blank demonstrated to be free from contamination? <RL ☐ Yes ☐ No

c. If the answer to item a or b was "no", document problems below:

Analyte	Affected Samples	Blank Concentration Reported	Shift Missing FB

Pesticide Data Inspection Checklist

17. SRM Results

a. Was appropriate SRM analyzed? ☒Yes ☐No

b. Were SRM recoveries within specified windows? (70-130% of 95% CI) ☐Yes ☒No

c. Was appropriate corrective action employed on affected samples? ☒Yes ☐No

d. If the answer was "no" to items a-d above, document affected samples:

Analyte	SRM % R	SRM % R	Affected Samples
All	SRM Lost During GPC		BS 104
t-chlordane	68.9		BS 102
c-nonachlor	67.6		BS 103
t-chlordane	67.0		BS 116
c-nonachlor	63.0		BS 116

18. MS/MSD Results

a. Were appropriate number of MS/MSD pairs analyzed? ☒Yes ☐No

b. Were all MS/MSD recoveries within specified windows? (≥50% Rec) ☐Yes ☒No

c. Were all RPDs within the specified window? (RPD ≤ 50%) ☐Yes ☒No

d. Was appropriate corrective action employed on affected samples? ☒Yes ☐No

e. If the answer was "no" to items a-d above, document affected samples:

Analyte	MS % R	MSD % R	MS/MSD RPD	Affected Samples
All	MS Lost	MSD is OK	---	BS 107
delta-HCH	NR	NR	—	All
m-parathion	45.1	58.1	OK	BS 96
heptachlor	57.8	41.4	OK	BS 97
m-parathion	54.8	42.3	OK	BS 97
diazinon	67.4	47.0	OK	BS 102
endosulfan I	40.2	39.2	OK	BS 102
ethyl parathion	51.2	39.3	OK	BS 102
heptachlor	30.1	30.1	OK	BS 103
ethyl parathion	39.7	118	Failed	BS 103
ethion	42.9	45.4	OK	BS 106
ethion	35.2	33.9	OK	BS 108
diazinon	43.7	54.8	OK	BS 116
ethion	45.2	37.0	OK	BS 116

Pesticide Data Inspection Checklist

19. Surrogate Recoveries

a. Were appropriate surrogates analyzed? ☒Yes ☐No

b. Were all surrogate recoveries within specified windows? (≥ 50% Rec) ☐Yes ☒No

c. Were all target analyte concentrations corrected for surrogate recovery? ☒Yes ☐No

d. Was appropriate corrective action employed on affected samples? ☒Yes ☐No

e. If the answer was "no" to items a-d above, document affected samples:

Surrogate	Surrogate % R	Affected Samples
DBCE	39.5	99-1093B (BS 97)
DBCE	40.0	99-2525 dup (BS 97)

20. Duplicate Sample Precision

a. Did duplicate sample analyses demonstrate acceptable precision? RPD ≤ 50% ☐Yes ☒No

b. Did field duplicate demonstrate acceptable precision? **NOT APPLICABLE** ☐Yes ☐No

c. If the answer was "no" to items a-d above, document affected samples:

Sample No.	Analyte	Sample	Sample Dup.	RPD	Affected Samples
99-1246	ethyl parathion	0.996 ppb	2.46 ppb	84.7	BS 107
00-0725	oxadiazon	3.34 ppb	6.42 ppb	63.1	BS 116
00-0667	% Lipid	0.533%	0.148%	113	BS 106

21. **Narrative**	**Corrective Action Taken?**

Page 5. Section 14. Continuing Calibration Verification (CCV) for Tedion

Several CCVs for Tedion were not within the 75-125% recovery acceptance limits. This was an ongoing problem throughout the GC analyses. All of the recoveries were greater than 50% and most were close to the acceptance limits. Tedion is an F3 Florisil fraction analyte and CCVs for all of the other F3 analytes were acceptable. Normally, failure of a CCV would require re-calibration of the GC and re-analysis of the sample extracts following the last acceptable CCV and prior to the next acceptable CCV in the analysis sequence. Since tedion was the only problem analyte in the F3 CCVs, we made the decision not to repeatedly try to re-calibrate for the F3 analytes. Instead, all of the tedion results affected by the bad CCVs will be qualified. The F3 CCVs are analyzed every 5 samples (rather than every 10 samples) because of the difficulty at maintaining the calibration throughout the analysis sequence for the F3 extracts which are typically high in lipid content.

Page 6. Section 15. Laboratory Method Blanks

The Method Blank for **Bench Sheet 94** was lost during extraction. One Method blank is required for each batch of samples. Normally, each bench sheet to be a batch of samples. A total of 10 bench sheets were used for this project and nine Method Blanks were reported. All of the pesticide analytes except for methoxychlor were ND or <RL in all of the remaining Method Blanks. None of the samples from BS 94 contained reportable concentrations of methoxychlor. **NO**

The Method Blank for **Bench Sheet 97 contained 5.84 ppb of methoxychlor.** None of the samples from BS 97 contained reportable concentrations of methoxychlor. **NO**

The Method Blank for **Bench Sheet 106 contained 9.16 ppb of methoxychlor**. None of the samples from BS 106 contained reportable concentrations of methoxychlor. **NO**

Page 7. Section 17. SRM Results

The SRM analyzed with **BS 104** was lost during GPC cleanup. Bench Sheet 104 contain

ed **NO**

only 2 samples (re-runs) from the CFCP (sample 99-550-t and 99-885-t). Since a MS/MSD and LCS were extracted with this bench sheet and the overall SRM analysis rate for the 99 CFCP is 6.7%, no corrective action is necessary.

SRM recoveries for **trans-chlordane** were slightly low (68.9% and 67.0%) for **BS 102** and **NO**

BS 116 respectively and recoveries for **cis-nonachlor** were low (67.6% and 63.0%) for **BS 103** and **BS 116** respectively. Since the MS and MSD recoveries were acceptable for all of the sample batches or bench sheets, no corrective action is necessary.

Page 7. Section 18. M S/MSD Results

BS 107 - The MS for BS 107 was lost during extraction/GPC. The MSD was OK and will **NO**

be used with the LCS to provide recovery information.

Delta-HCH - was not recovered or the recoveries were low and inconsistent. All delta-HCH **YES**

results will be reported as not analyzed due to the analyte recovery failure.

Methyl parathion - The recovery of methyl parathion was low in the MS for **BS 96** (45.1%) **NO**

and was low in the MSD for **BS 97** (42.3%). Recoveries of methyl parathion were acceptable (>50%) in the MSD for BS 96 and in the MS for BS 97. The MS/MSD RPDs were also acceptable for both bench sheets. Laboratory control spike (LCS) recovery for methyl parathion were acceptable for both BS 96 and BS 97.

Heptachlor -

The recovery of heptachlor was low for the MSD for **BS 97** (41.4%). Recovery for the MS **NO**

was acceptable and the MS/MSD RPD was acceptable. The recovery of heptachlor was also low (48.0%) in the LCS for BS 97.

The recovery of heptachlor was low for both the MS (30.1%) and MSD (30.1%) for BS 103. **YES**

The recovery for heptachlor was also low for the LCS (19.3%) for BS 103.

Pesticide Data Inspection Checklist

21. **Narrative (cont.)**	**Corrective Action Taken?**
Page 7. Section 18. M S/MSD Results (continued)	
Diazinon - The recovery of diazinon was low (47.0%) for the MSD for **BS 102.** Recovery for the MS was acceptable and the MS/MSD RPD was acceptable. Recovery of diazinon for the LCS was also low (31.1%) for BS 102..	**NO**
The recovery of diazinon was low (43.7%) for the MS for **BS 116.** Recovery for the MSD was acceptable and the MS/MSD RPD was acceptable. Recovery of diazinon for the LCS was also low (25.6%) for BS 116.	**NO**
Endosulfan I - The recovery of endosulfan I was low for both the MS (40.2%) and the MSD (39.2%) for **BS 102.** The recovery of endosulfan I was aslo low for the LCS (46.3%).	**YES**
Ethyl parathion - The recovery of ethyl parathion was low (39.3%) for the MSD for **BS 102.** Recovery for the MS was acceptable and the MS/MSD RPD was acceptable. Recovery of diazinon for the LCS was acceptable (58.3%) for BS 102.	**NO**
The recovery of ethyl parathion was low (39.7%) for the MSD for **BS 103.** Recovery for the MS was acceptable. The MS/MSD RPD was not acceptable. Recovery of diazinon for the LCS was acceptable (58.3%) for BS 103.	**YES**
Ethion - The recoveries of ethion were low for both the MS and MSD for BS 106, BS 108, and BS 116. The recoveries of ethion were also low for the LCS for these bench sheets.	**YES**
The matrix spike was lost for BS 107. MSD and LCS recoveries of all pesticides, with the exception of delta-HCH were acceptable.	**NO**
Page 8. Section 19. Surrogate Recoveries	
DBCE recoveries were low for samples **99-1093B** (39.5%) and **99-2525 dup** (40.0%). DBCE is the F3 surrogate and is used to correct the F3 target analyte concentrations in the samples. All F3 target analyte results for these two samples will be qualified.	**YES**
Page 8. Section 20. Duplicate Sample Precision	
Sample 99-1246 - The RPD for ethyl parathion was 84.7% which exceeds the acceptance limit of 50%.. However, the reported concentrations of this analyte was reported at the reporting limit of 2 ppb in one sample and <2 ppb in the other sample. Since these results are at the reporting limit for the analyte, no corrective action will be taken.	**NO**
Sample 00-0725 - The RPD for oxadiazon was 63.1% which exceeds the acceptance limit of 50%.. However, the reported concentrations of this analyte was reported at the reporting limit of 3 ppb in one sample and 6 ppb in the other sample. Since these results are near the reporting limit for the analyte, no corrective action will be taken.	**NO**

Sample 00-0667 - The RPD for the %lipid analysis was 113% which exceeds the acceptance limit. The calculation were double checked and no calculation errors were found. Precision for the target analyte and surrogate duplicates were acceptable. The percent lipid results for this sample will be qualified.	**YES**

Pesticide Data Inspection Checklist

22. **Corrective Action Taken**

Tedion: For samples affected by the CCV failure -
All tedion results reported at concentrations above the reporting limit will be qualified "**J**".
All tedion results reported less than the reporting limit of 2 ppb (-2) will be qualified "**UJ**".

L-150-00 - samples: 99-0383, 99-0963, 99-1132, 99-1135, 99-1136, 99-1137, 99-1138, 99-1140
L-180-00 - samples: 99-1990, 99-1991, 99-1992, 99-1466, 99-1030, 99-1093B, 99-2525dup
L-202-00 - samples: 99-1497(on), 00-0670, 99-1794, 00-0667, 00-0668, 00-0450, 00-0451, 99-1497(off), 99-1498(on), 99-1498(off), 99-1499, 99-1791, 99-1792, 00-0414, 00-0406, 00-0667dup, 00-0407, 99-1272, 99-1099, 99-1458, 00-0752, 00-0753(on), 00-0753(off), 99-1027, 00-0617, 00-0616, 99-1248, 99-1246, 99-1247, 00-0354, 99-1089, 99-1090, 99-1190, 99-1246dup, 00-0353

Delta-HCH: All delta-HCH results will be reported as Not Analyzed (**NA**).

Heptachlor: For all samples on **BS 103** heptachlor results will be qualified due to MS/MSD recovery failure as follows:
All heptachlor results reported at concentrations above the reporting limit will be qualified "**J**".
All heptachlor results reported less than the reporting limit of 2 ppb (-2) will be qualified "**UJ**".

Endosulfan I: For all samples on **BS 102** endosulfan I results will be qualified due to MS/MSD recovery failure as follows:
All endosulfan I results reported at concentrations above the reporting limit will be qualified "**J**".
All endosulfan I results reported less than the reporting limit of 2 ppb (-2) will be qualified "**UJ**".

Ethyl parathion: For all samples on **BS 103** ethyl parathion results will be qualified due to MS/MSD recovery and precision failure as follows:
All ethyl parathion results reported at concentrations above the reporting limit will be qualified "**J**".
All ethyl parathion results reported less than the reporting limit of 2 ppb (-2) will be qualified "**UJ**".

Ethion: For all samples on **BS 106**, **BS 108**, and **BS 116** ethion results will be qualified due to MS/MSD recovery failure as follows:
All ethion results reported at concentrations above the reporting limit will be qualified "**J**".
All ethion results reported less than the reporting limit of 6 ppb (-6) will be qualified "**UJ**".

Samples 99-1093B and 99-2525 dup: All F3 analytes for these two samples will be qualified due to low surrogate recovery as follows:
All F3 results reported at concentrations above the reporting limit will be qualified "**J**".
All F3 results reported less than the reporting limit of 6 ppb (-6) will be qualified "**UJ**".

Sample 00-0667 and 00-0667 dup: Percent lipid results for these two samples will be reported as not analyzed (NA) due to the high RPD between the two analyses.

DATA QUALIFIER DEFINITIONS:

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

NA - Not Analyzed

CDFG FISH AND WILDLIFE WATER POLLUTION CONTROL LABORATORY
DATA QUALITY ASSURANCE REPORT

Laboratory No.: L-150, 180, 195, 202, 231-00 **Page 15**

Project Title: 1999 Coastal Fish Contamination Program (CFCP Year 2)

PCB Data Inspection Checklist

1. Method Used / Extraction Completion Date: **SO-TISSUE-PREP.SOPv5 / May 2, 2000**

5. Number of Samples Analyzed: **135**

6. Number of concentrations levels used for instrument calibration: **7**

4. Total No. of CCVs Required: 30 (One for each 10-15 samplest) Total No. of CCVs Reported: 30

8. Total No. of CCBs Required: 30 (One for each CCV) Total No. of CCBs Reported: 30

9. Total No. of Field Blanks Required: NA (One per site or per 10 samples, whichever is more frequent) Total No. of Field Blanks Reported: NA

10. Total No. of Method Blanks Required: 10 (One per batch) Total No. of Method Blanks Reported: 9

11. Total No. of SRM analyses Required: 10 (One per batch) Total No. of SRM Analyses Reported: 9

12. Total No. of MS/MSD samples Required: 10 (One MS/MSD per batch) Total No. of MS/MSD samples Reported: 9+MSD

Total No. of sample Duplicates Reported: 11

13. Total No. sample Duplicates Required 7 (One per 20 samples)

PCB Data Inspection Checklist

12. Initial Calibration

4. Was a multiple point initial calibration performed*? ☒Yes ☐No

5. Were all sample concentrations reported within the calibration range? ☒Yes ☐No

6. If no, list method and analytes for which initial calibration was not performed or which exceeded the calibration range.

Analyte	No ICAL (Y/N)	Exceeded ICAL Range (Y/N)

d. Did the initial calibration meet acceptance criteria? $R^2 \geq 0.995$ ☒Yes ☐No

*A three point (minimum) initial calibration should be performed for each Analyte; the RSD of the RFs of calibration standards ≤ 20%.

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19. Method Detection Limit (MDL)/Minimum Level (ML)

1. Did the laboratory demonstrate their ability to achieve the required MDL? ☒Yes ☐No

2. Did the initial calibration range encompass the ML? ☒Yes ☐No

3. Were all field samples detected below the ML reported as non-detects? ☒Yes ☐No

4. If the answer to item a, b, or c above was “no”, describe problem:

PCB Data Inspection Checklist

20. Initial Calibration Verification (ICV) Initial Calibration Blanks (ICB):

a. Was an ICV run prior to field samples? ☒Yes ☐No

b. Were ICV results within the specified windows? (75-125% Rec) ☒Yes ☐No

c. Was the ICV preceded by an ICB? ☒Yes ☐No

d. Was the ICB free from contamination? ☒Yes ☐No

e. If any item in a-d above was answered "no", list problems below:

Analyte	Failed ICV Recovery	Concentration Detected in ICB	Affected Samples

21. Continuing Calibration Verification (CCV)/Continuing Calibration Blank (CCB)

a. Were CCVs run prior to each batch of 10-15 samples on each instrument? ☒Yes ☐No

b. Were all CCV results within the specified windows" (75-125% Rec) ☒Yes ☐No

c. Was each CCV preceded by a CCB? ☒Yes ☐No

d. Was each CCB free from contamination? ☒Yes ☐No

e. If any item in a-d above was answered "no", list problems below:

Analyte	Affected Samples	Shifting Missing CCV/CCB	Failed CCV/CCB ID

PCB Data Inspection Checklist

22. Laboratory (Method) Blanks

a. Was a method blank analyzed for each instrument & sample batch? ☐Yes ☒No

b. Was each method blank demonstrated to be free from contamination? (<RL) ☐Yes ☒No

c. Were equipment blanks demonstrated to be free from contamination? **NA** ☐Yes ☐No

d. If the answer to item a or b was "no", document problems below:

Analyte	Affected Samples	Blank Concentration Reported	Shift Missing MB
All	BS 94		MB Lost During Extraction
PCB 70	BS 95	0.268 ppb	
PCB 95	"	0.240	
PCB 101	"	0.268	
PCB 110	"	0.322	
PCB 118	"	0.345	
PCB 118	BS 97	0.206	
PCB 118	BS 103	0.206	
PCB 52	BS 107	0.229	
PCB 70	"	0.293	
PCB 101	"	0.280	
PCB 110	"	0.290	
PCB 118	"	0.365	
PCB 95	BS 116	0.203	
PCB 101	"	0.247	
PCB 110	"	0.294	
PCB 118	"	0.237	

23. Field Blanks NOT APPLICABLE

a. Was a field blank analyzed for each 10 samples per site? ☐Yes ☐No

b. Was each field blank demonstrated to be free from contamination? <RL ☐Yes ☐No

c. If the answer to item a or b was "no", document problems below:

Analyte	Affected Samples	Blank Concentration Reported	Shift Missing FB

PCB Data Inspection Checklist

24. SRM Results

a. Was appropriate SRM analyzed? ☒Yes ☐No

b. Were SRM recoveries within specified windows? (70-130% of 95% CI) ☐Yes ☒No

c. Was appropriate corrective action employed on affected samples? ☒Yes ☐No

d. If the answer was "no" to items a-d above, document affected samples:

Analyte	SRM % R	SRM % R	Affected Samples
All	SRM Lost During GPC		BS 104
PCB 170	54.1 (Average)		All

25. MS/MSD Results

a. Were appropriate number of MS/MSD pairs analyzed? ☒Yes ☐No

b. Were all MS/MSD recoveries within specified windows? (≥50% Rec) ☒Yes ☐No

c. Were all RPDs within the specified window? (RPD ≤ 50%) ☒Yes ☐No

d. Was appropriate corrective action employed on affected samples? **NA** ☐Yes ☐No

e. If the answer was "no" to items a-d above, document affected samples:

Analyte	MS % R	MSD % R	MS/MSD RPD	Affected Samples
All	MS Lost	MSD is OK	---	BS 107

PCB Data Inspection Checklist

21. Surrogate Recoveries

a. Were appropriate surrogates analyzed? ☒Yes ☐No

b. Were all surrogate recoveries within specified windows? (≥ 50% Rec) ☒Yes ☐No

c. Were all target analyte concentrations corrected for surrogate recovery? ☒Yes ☐No

d. Was appropriate corrective action employed on affected samples? **NA** ☐Yes ☐No

e. If the answer was "no" to items a-d above, document affected samples:

Surrogate	Surrogate % R	Affected Samples

22. Duplicate Sample Precision

a. Did duplicate sample analyses demonstrate acceptable precision? RPD ≤ 50% ☐Yes ☒No

b. Did field duplicate demonstrate acceptable precision? **NA** ☐Yes ☐No

c. If the answer was "no" to items a-d above, document affected samples:

Sample #	Analyte	Sample	Sample Dup.	RPD	Affected Samples
99-1138	PCB 28	0.138	0.255	59.5	BS 94
99-1140	PCB 28	0.149	0.250	50.5	BS 94
99-2525	PCB 105	0.147	0.246	50.6	BS 97
99-1214	PCB 31	0.175	0.317	57.7	BS 103
99-1214	PCB 33	0.110	0.271	84.8	BS 103
99-1250	PCB 56	0	0.256	—	BS 102/116

PCB Data Inspection Checklist

		Corrective Action Taken?
22.	**Narrative**	

Page 15. Section 15. Laboratory Method Blanks

The Method Blank for **Bench Sheet 94** was lost during extraction. One Method blank is required for each batch of samples. Normally, each bench sheet to be a batch of samples. A total of 10 bench sheets were used for this project and nine Method Blanks were reported. Concentrations of PCB congeners reported in the remaining 9 Method Blanks ranged from 0.206 ppb to 0.365 ppb. Since the Method Blank was for BS 94 was lost, the reporting limits for any congeners reported in the other 9 Method Blanks will be increased to 0.4 ppb to account for the possibility of low level contamination of the samples. **YES**

Page 16. Section 17. SRM Results

The SRM analyzed with **BS 104** was lost during GPC cleanup. Bench Sheet 104 contained only 2 samples (re-runs) from the CFCP (sample 99-550-t and 99-885-t). Since a MS/MSD and LCS were extracted with this bench sheet and the overall SRM analysis rate for the 99 CFCP is 6.7%, no corrective action is necessary. **NO**

Page 16. Section 18. MS/MSD Results

BS 107 - The MS for BS 107 was lost during extraction/GPC. The MSD was OK and will be used with the LCS to provide recovery information. **NO**

Page 17. Section 20. Duplicate Sample Precision

RPDs exceeded the 50% criteria for 5 congeners in 6 duplicate analyses. The concentations of the congeners for these duplicates were near or slightly below the reporting limit (RL) of 0.2 ppb. **NO**

No corrective action is necessary since all of the congener concentrations were near the RL.

PCB Data Inspection Checklist

21. **Narrative (cont.)** **Corrective Action Taken?**

PCB Data Inspection Checklist

22. **Corrective Action Taken**

Samples from BS 94 -
PCB congeners reported at low concentrations in the 9 Method Blanks include: **#52, #70, #95, #101, #110, and #118.** All of the PCB congener concentrations reported in the Method Blanks were < 2 x RL. Because the Method Blank for BS 94 was lost, the reporting limits for congeners noted above will be increased to **0.4 ppb** for all of the samples from BS 94.

Samples from BS 95 -
PCB congeners reported at low concentrations in the Method Blank for BS 95 include: **#70** (0.268 ppb), **#95** (0.240 ppb), **#101** (0.268 ppb), **#110** (0.322 ppb), and **#118** (0.345 ppb). The reporting limits for the five congeners noted above will be increased to **0.4 ppb** for all of the samples from BS 95 because of the low level PCB contamination in the Method Blank.

Samples from BS 97 -
PCB congener **#118** was reported at a concentration of 0.206 ppb in the Method Bland for BS 97. The reporting limit

for PCB congener #118 will be increased to **0.3 ppb** for all of the samples from BS 97.

Samples from BS 103 -
PCB congener **#118** was reported at a concentration of 0.206 ppb in the Method Bland for BS 103. The reporting limit for PCB congener #118 will be increased to **0.3 ppb** for all of the samples from BS 103.

Samples from BS 107 -
PCB congeners reported at low concentrations in the Method Blank for BS 107 include: **#52** (0.229 ppb), **#70** (0.293 ppb), **#101** (0.280 ppb), **#110** (0.290 ppb), and **#118** (0.365 ppb). The reporting limits for the five congeners noted above will be increased to **0.4 ppb** for all of the samples from BS 107 because of the low level PCB contamination in the Method Blank.

Samples form BS 116 -
PCB congeners reported at low concentrations in the Method Blank for BS 116 include: **#95** (0.203 ppb), **#101** (0.247 ppb), **#110** (0.294 ppb), and **#118** (0.237 ppb). The reporting limits for the five congeners noted above will be increased to **0.3 ppb** for all of the samples from BS 116 because of the low level PCB contamination in the Method Blank.