



**Quality Assurance Summary Report
for Synthetic Organic Compounds**

for the

Coastal Fish Contaminant Project Year 1, 1998-1999

Prepared for:

**Del Rasmussen
State Water Resources Control Board**

**Bob Brodberg & Margy Gassel
OEHHA**

Prepared by:

**David Crane
Kathleen Regalado
Laurie Smith**

June 16, 2000

CFCP 1998 YEAR 1 QA SUMMARY – PESTICIDES AND PCBs

Samples were received in three batches:

L-385-99 (42 samples) Received on 11/02/99
L-009-00 (39 samples) Received on 01/11/00
L-041-00 (18 samples) Received on 02/09/00

The samples were analyzed in three batches:

L-385-99 Set 1 (13 samples) Bench Sheet 71
L-385-99 Set 2 (29 samples) Bench Sheets 75, 76
L-009/041-00 Set 3 (56 samples) Bench Sheets 84, 85, 86

Summary of Analysis Sets:

Set 1 (L-385-99), Bench Sheet 71

13 samples were extracted and one sample was lost (99-549) during extraction (extract was spilled). Sample 99-549 was re-analyzed with Set 2.

Results from sample 99-550 were not reported because of low surrogate recovery. Sample 99-550 is currently being re-analyzed.

Corrective Action (Qualified Data)

✓ Heptachlor

All ND and \leq RL should be qualified "UJ". No results $>$ RL were reported.

✓ Methyl parathion

All ND and \leq RL should be qualified "UJ". No results $>$ RL were reported.

Congeners #8, #29, #56, and #60

Analyses failed due to 0% recovery in the MS, MSD or both. These analytes will be designated as not analyzed (NA).

Congeners #18, #27, and #128

These congeners were low for either the SRM or the MS/MSD or both. Results for congeners #18, #27 and #128 should be qualified as follows:

Qualify all $<$ RL and ND	"UJ"
Qualify all reported concentrations \geq RL	"J"

99-0356 - Results are in spreadsheet but not listed on Dave's lists of samples

Set 2 (L-385-99), Bench Sheets 75 and 76

29 samples were extracted and analyzed successfully.

Corrective Action (Qualified Data)

✓ Heptachlor epoxide

All ND and \leq RL should be qualified "UJ". No results $>$ RL were reported.

✓ Heptachlor

All ND and \leq RL for bench sheet 75 samples should be qualified "UJ". No results $>$ RL were reported.

✓ Cis-nonachlor

All ND and \leq RL should be qualified "UJ" and reported concentrations should be qualified "J".

✓ Ethion

Qualify all ND results "UJ".

✓ Delta-HCH

Analysis failed, all data entries are NA (not analyzed).

Congener #66

All data entries $>$ RL are qualified "J".

Congener #29

For samples on bench sheet 75 only: All data entries $>$ RL are qualified "J". Qualify all $<$ RL and ND "UJ".

Congener #56

For samples on bench sheet 75 only: All data entries $>$ RL are qualified "J". Qualify all $<$ RL and ND "UJ".

Set 3 (L-009-00 and L-041-00) Bench Sheets 84, 85, 86

56 samples were extracted and three samples were lost during extraction (extract was spilled). Sample 99-764, 99-885 and 99-963 are currently being re-analyzed.

Corrective Action (Qualified Data)

✓ Ethion

Qualify all ND results "UJ".

✓ Delta-HCH

Analysis failed, all data entries are NA (not analyzed).

✓ Heptachlor

All ND and \leq RL for bench sheet 75 samples should be qualified "UJ". No results $>$ RL were reported.

✓ Methyl parathion

All ND and \leq RL should be qualified "UJ".

EXTRACTION BENCH SHEET

WPCL LAB # L-385-99
 PROJECT NAME: 1998 CECS
 PROJECT CODE:
 CLIENT NAME:

NUMBER OF SAMPLES: 13 + QC
 SAMPLE TYPE: FISH TISSUE
 ANALYSIS TYPE: SYNTHETIC ORGANIC
 STORAGE LOCATION: TSI FH

EXTRACTION START: 12/15/99
 EXTRACTION FINISHED: 2/14/2000
 EXTRACT STORAGE:
 PROJECT DUE DATE:

SAMPLE I.D.	ASE SAMPLE SIZE	MOISTURE				ASE EXTRACT DATE & INIT 12/15/99 ASE CELL ID	K-D/u K-D DATE&INIT FV = 5 mL	LIPID ALICUOT = 1 mL			GPC 4 mL DL	FLORISIL				PAHs DATE&INIT AL/SIL FV=0.1 ml
		PLANCHET WEIGHT	WET SAMPLE WEIGHT	DRY SAMPLE +PLANCHET	% H2O			PLANCHET WEIGHT	PLANCHET + LIPID	% LIPID		F1	F2	F3	F4	
1. METHOD BLANK	1.000	1.300	N.A.	1.300	0.00	38008	12/29/99	1.28381	1.28384	0.0000	12/29/99	2-4-00	LDG			
2. 99-409-t	10.023	1.289	3.715	2.024	80.2	38391	1/11/2000	1.30138	1.31295	.5772						
3. 99-742-t	10.008	1.290	3.161	1.957	78.9	38004		1.29830	1.31374	.7468						
4. 99-923-t	10.015	1.282	1.244	1.525	80.5	38238		1.29518	1.30728	.6041						
5. 99-757-t	10.196	1.293	3.046	1.912	79.7	38339		1.29499	1.30575	.5277		2-8-00	LDG			
6. 99-882-t	10.432	1.277	3.018	1.942	78.0	38029		1.28074	1.31706	1.741						
7. 99-889-t	10.506	1.267	3.285	1.922	80.1	38278		1.27983	1.28829	.4026						
8. 99-892-t	10.410	1.269	3.535	1.980	79.9	31459		1.28127	1.29400	.6079	✓	✓	✓	✓		
9. 99-758-t	10.780	1.287	3.307	2.034	77.4	38019		1.28184	1.31111	1.358	1/28/2000	2-9-2000	LDG			
10. 99-759-t	10.410	1.262	3.734	2.052	78.8	31356		1.27876	1.28943	.5125						
11. 99-760-t	10.174	1.264	3.039	1.838	81.1	31401		1.28916	1.30074	.5691						
12. 99-550-t	10.201	1.280	3.381	1.929	80.8	38338	1-3-2000 G.H.	1.28596	1.28842	.1206		✓	✓	✓	✓	
13. 99-549-t	10.320	1.302	3.589	2.087	78.1	38342		1.29193	1.30546	.6410		N/A			✓	
14. 99-946-t	6.824	1.297	1.033	1.540	76.5	38273		1.29759	1.31325	1.147		2-11-2000	LDG			
15. 99-409-t MS	10.130	1.292	3.077	1.902	80.2	31261		1.28731	1.29783	.5192						
16. 99-409-t MS	10.257	1.290	3.099	1.905	80.2	38364		1.29943	1.30904	.4685		✓	✓	✓	✓	
17. LCS-50 PCB	N.A.	N.A.				35330	✓	1.30333	1.30333	0.0000		2-11-2000	LDG			
18. SEM1588a	1.006	N.A.				38272	1-12-2000	1.28236	1.48169	99.07						
19. 99-742-t Dup.	10.844	1.295	3.367	2.013	78.7	38341		1.29356	1.30547	.7829						
20. 99-760-t Dup.	10.032	1.295	3.042	1.873	81.0	38368	✓	1.28445	1.29516	.5338	✓	✓	✓	✓	✓	
21.																
22.																
23.																

SPIKING SOLUTION NAME: S.O. SPELE-SOIL; COMPLEX AGC; ppDCBP
 SPIKING SOLUTION CODE: P120299A; P062299B; P072799B
 SPIKING SOLUTION CONC. (ug/mL): 40 PPB; 20 PPB; 20 PPB
 SPIKING SOLUTION VOL. (mL): 1.00 mL; 1.00 mL; 1.00 mL
 SPIKED DATE/BY: G.M. 12/15/99; G.M. 12/15/99; G.M. 12/15/99
 WITNESSED DATE/BY: MC121599 MC121599 MC121599

SURROGATE SOLUTION NAME: PESTICIDE SURROGATE
 SURROGATE SOLUTION CODE: P113099A
 SURROGATE SOLUTION CONC. (ug/mL): 40 PPB
 SURROGATE SOLUTION VOL. (mL): 1.00 mL
 SPIKED DATE/BY: G.M. 12/15/99
 WITNESSED DATE/BY: MC121599

COMMENTS:

* SAMPLES 2 THROUGH 8 ARE PRIORITY.
 ✓ SAMPLE (14) SMALL SAMPLE SIZE DUE TO NSS.
 SAMPLE (B) SPLITTED - LOST 1/28/2000 G.M.
 □ SAMPLES 1-4: P120299A; P113099A; P062299B; P072799B

EXTRACTION BENCH SHEET

WPCL LAB # L-009-00
 PROJECT NAME: 1998 CFCs
 PROJECT CODE:
 CLIENT NAME:

NUMBER OF SAMPLES: 19 + QC
 SAMPLE TYPE: FISH TISSUE
 ANALYSIS TYPE: S.O. & PCB
 STORAGE LOCATION: TSMF3

EXTRACTION START: 4/1/2000
 EXTRACTION FINISHED: 4/27/2000
 EXTRACT STORAGE:
 PROJECT DUE DATE:

SAMPLE I.D.	ASE SAMPLE SIZE	MOISTURE				ASE EXTRACT DATE & INIT 4/1/00 BT ASE CELL ID	K-D/u K-D DATE & INIT FV = 5 mL	LIPID ALICUOT = 1 mL			GPC 4 mL S/L	FLORISIL				PAHs DATE & INIT AL/SIL FV=0.1 mL
		PLANCHET WEIGHT	WET SAMPLE WEIGHT	DRY SAMPLE + PLANCHET	% H2O			PLANCHET WEIGHT	PLANCHET + LIPID	% LIPID		F1	F2	F3	F4	
✓ 1. Method Blank	1.000	1.295	N/A	1.295	0.00	3/381	04/12/00	1.28279	1.28279	0.0000	04/17/00					
✓ 2. 99-532-E	10.425	1.291	3.014	1.916	79.3	38321	ms	1.28287	1.24320	0.4954	ms					
✓ 3. 99-949-E	10.278	1.288	3.195	1.940	79.4	38381		1.27501	1.28742	0.6037						
✓ 4. 99-774-E	10.330	1.285	3.354	2.070	76.6	38439		1.27581	1.28183	0.2914						
✓ 5. 99-950-E	10.337	1.291	3.310	2.016	78.1	38278		1.27124	1.30964	1.858						
✓ 6. 99-951-E	10.257	1.286	3.309	1.960	79.4	38019	04/13/00	1.28013	1.29322	0.6381	ms					
✓ 7. 99-952-E	10.020	1.291	3.120	1.912	80.1	38273	ms	1.28439	1.29338	0.4186						
✓ 8. 99-764-E	10.878	1.290	3.170	1.998	77.7	38364		1.27164	1.33153	2.753						
✓ 9. 99-773-E	10.142	1.286	3.057	1.930	78.9	31459		1.27997	1.28425	0.3096						
✓ 10. 99-730-E	10.190	1.284	3.555	2.049	78.5	38013		1.29154	1.30086	0.4573						
✓ 11. 99-729-E	10.824	1.301	2.298	1.728	81.4	38370		1.27767	1.28223	0.2097						
✓ 12. 99-725-E	10.443	1.294	3.027	1.911	79.4	38368		1.29200	1.29882	0.3245						
✓ 13. 99-749-E	10.340	1.290	3.081	1.954	78.4	35330		1.29094	1.30875	0.8612						
✓ 14. 99-765-E	10.160	1.293	3.173	1.976	78.5	31261		1.28616	1.29134	0.2549						
✓ 15. 99-341-E	10.678	1.286	3.025	1.892	80.0	38338		1.28959	1.29726	0.3324	10.00					
✓ 16. 99-747-E	10.107	1.300	3.220	2.004	78.1	38391		1.28894	1.31212	1.147	ms					
✓ 17. 99-728-E	10.200	1.299	3.610	2.110	77.5	38339		1.29103	1.30511	0.6902						
✓ 18. 99-727-E	10.836	1.293	3.155	1.949	79.2	38356		1.29385	1.30254	0.4010						
✓ 19. 99-755-E	10.510	1.294	3.308	2.013	78.3	38392		1.29076	1.33273	1.997						
✓ 20. 99-711-E	10.272	1.290	3.157	2.021	76.8	38004		1.28488	1.33416	2.399						
✓ 21. 99-532-E Dup	10.957	1.296	3.521	2.063	78.2	38238		1.28886	1.29649	0.3482						
✓ 22. 99-747-E M.S.	10.804	1.295	3.492	2.065	77.9	38272		1.29126	1.32304	1.471						
✓ 23. LCS-50 & PCB	N/A	N/A			N/A	31401		1.28779	1.28774	N/A						
✓ 24. SRM 1588a	1.014	N/A			N/A	38303		1.29392	1.50671	104.9						

SPIKING SOLUTION NAME: S.O. SPIKE SOLN. COMPLEX A+C, ppDCBP
 SPIKING SOLUTION CODE: P120299A - P062299B - P072799B
 SPIKING SOLUTION CONC. (ug/mL): 40 PPB - 20 PPB - 20 PPB
 SPIKING SOLUTION VOL. (mL): 1.00mL; 1.00mL; 1.00mL
 SPIKED DATE/BY: GN 4-1-2000; GN 4-1-2000; GN 4-1-2000
 WITNESSED DATE/BY: ms 04/01/00; ms 04/01/00; ms 04/01/00

COMMENTS:

21-15% AND 22-15% WERE BROUGHT TO FV 5 mL MISTAKENLY

SURROGATE SOLUTION NAME: PESTICIDE SURROGATE
 SURROGATE SOLUTION CODE: P032500A
 SURROGATE SOLUTION CONC. (ug/mL): 40 PPB
 SURROGATE SOLUTION VOL. (mL): 1.00mL
 SPIKED DATE/BY: GN 4-1-2000
 WITNESSED DATE/BY: ms 04/01/00

EXTRACTION BENCH SHEET

WPCL LAB # L-009-00
 PROJECT NAME: 1998 CFCs
 PROJECT CODE:
 CLIENT NAME:

NUMBER OF SAMPLES: 19 + QC
 SAMPLE TYPE: FISH TISSUE
 ANALYSIS TYPE: SO AND PCB
 STORAGE LOCATION: TSMF3

EXTRACTION START: 4/7/2000
 EXTRACTION FINISHED: 5/2/2000
 EXTRACT STORAGE:
 PROJECT DUE DATE:

SAMPLE I.D.	ASE SAMPLE SIZE	MOISTURE				ASE EXTRACT DATE & INIT 4/10/00 ASE CELL ID	K-D/u K-D DATE&INIT FV = 5 mL	LIPID			GPC 4 mL DL	FLORISIL				PAHs DATE&INIT AL/SIL FV=0.1 ml
		PLANCHET WEIGHT	WET SAMPLE WEIGHT	DRY SAMPLE +PLANCHET	% H2O			ALICUOT = 1 mL				F1	F2	F3	F4	
								PLANCHET WEIGHT	PLANCHET + LIPID	% LIPID						
1. METHADOL	1.000	1.276	N/A	1.276	0.00	31215	04/17/00	1.28130	1.28130	0.0000	4/10/00	4-2	4-00	-1	06	
2. 99-383-t	10.159	1.273	3.159	1.937	79.0	31356		1.28800	1.30035	0.6078						
3. 99-384-t	10.643	1.278	3.171	1.992	77.5	38341		1.29337	1.30513	0.5525						
4. 99-385-t	10.514	1.275	3.260	2.002	77.7	38028		1.29129	1.30800	0.7945				*		
5. 99-356-t	10.390	1.287	3.330	2.044	77.3	38342		1.27731	1.29575	0.8874						
6. 99-357-t	10.213	1.289	3.188	1.989	78.0	38273		1.29030	1.30758	0.8460						
7. 99-754-t	10.204	1.280	3.281	1.919	80.5	35330		1.27805	1.29070	0.6227						
8. 99-352-t	10.546	1.280	3.817	2.080	79.0	31459		1.27360	1.28420	0.5017						
9. 99-531-t	10.114	1.276	3.109	1.932	78.9	31381		1.27679	1.29066	0.9823						
10. 99-533-t	10.228	1.282	3.865	2.116	78.4	38391		1.27972	1.30049	1.015						
11. 99-732-t	10.352	1.281	3.439	2.030	78.2	38004		1.29703	1.30911	0.5835						
12. 99-726-t	10.042	1.289	3.207	1.982	78.4	38321		1.28272	1.29490	0.6064	04/19/00					
13. 99-746-t	10.984	1.283	3.513	1.980	80.2	38370		1.28859	1.29849	0.4507						
14. 99-457-t	10.168	1.282	3.406	2.044	77.6	38303		1.28115	1.29034	0.7470						
15. 99-751-t	10.122	1.276	3.232	1.960	78.8	31401		1.28526	1.29380	0.4218						
16. 99-885-t	10.194	1.282	1.556	1.653	76.2	38278		1.29513	1.38598	4.456						
17. 99-855-t	6.564	N/A				38368		1.28919	1.29662	0.5600						
18. 99-351-t	10.174	1.277	3.055	1.980	77.0	38013		1.27067	1.32295	2.274						
19. 99-387-t	10.664	1.277	3.133	1.931	79.1	38392		1.28054	1.29630	0.4516						
20. 99-389-t	10.300	1.282	3.213	1.990	78.0	38238		1.29340	1.33640	2.094						
21. 99-383-t	10.110	1.287	3.080	1.937		38019		1.29373	1.30844	N/A						
22. 99-385-t	10.409	1.277	3.186	1.923	77.5	38339		1.28848	N/A	0.7046						
23. LCS-SO & PCB	N/A	N/A			N/A	38029		1.28709	N/A	N/A						
24. SRM1588a	1.014	N/A			N/A	38381		1.28489	1.49298	101.4						
SPIKING SOLUTION NAME																

SPIKING SOLUTION NAME: S.O. SPIKE SOLN; COMPLEX AC; PPDCBP
 SPIKING SOLUTION CODE: 720299A; P02299B; P072799B
 SPIKING SOLUTION CONC. (ug/mL): 40 PPB; 20 PPB; 20 PPB
 SPIKING SOLUTION VOL. (mL): 1.00 mL; 1.00 mL; 1.00 mL
 SPIKED DATE/BY: G11 4-7-00; G17 4-7-00; G17 4-7-00
 WITNESSED DATE/BY: MS 040700; MS 040700; MS 040700

SURROGATE SOLUTION NAME: PESTICIDE SURROGATE
 SURROGATE SOLUTION CODE: P302500A
 SURROGATE SOLUTION CONC. (ug/mL): 40 PPB
 SURROGATE SOLUTION VOL. (mL): 1.00 mL
 SPIKED DATE/BY: G17 4-1-00
 WITNESSED DATE/BY: MS 040700

COMMENTS:

* ACCORDING TO C.O.C 99-885-t AND 99-854-t ARE SAMPLE, BOTTLE LABELED 99-885-t
 NO MORE (17) 99-855-t - INSUFFICIENT SAMPLE
 (*) 21. 99-383-t LOST 041700 MS
 LIPID DATA FOR LOT 21 0.00 - 0.00 - 0.00
 * Half of Sample 4 (F3) lost on final Transfer
 [X] Sample 16 (F1) lost

EXTRACTION BENCH SHEET

WPCL LAB # L-041-00
 PROJECT NAME: 1998 CFCs
 PROJECT CODE:
 CLIENT NAME:

NUMBER OF SAMPLES: 18 + QC
 SAMPLE TYPE: FISH TISSUE, Muscle
 ANALYSIS TYPE: S.O. & PCB CLAN
 STORAGE LOCATION: TS/IFI

EXTRACTION START: 4/12/2000
 EXTRACTION FINISHED: 05/02/2000
 EXTRACT STORAGE:
 PROJECT DUE DATE:

SAMPLE I.D.	ASE SAMPLE SIZE	MOISTURE				ASE EXTRACT DATE & INIT 4/12/00 G7 ASE CELL ID	K-D/u K-D DATE&INIT FV = 5 mL	LIPID			GPC 4 mL (5L)	FLORISIL				PAHs DATE&INIT AL/SIL FV=0.1 mL
		PLANCHET WEIGHT	WET SAMPLE WEIGHT	DRY SAMPLE +PLANCHET	% H2O			ALICUOT = 1 mL				F1	F2	F3	F4	
								PLANCHET WEIGHT	PLANCHET + LIPID	% LIPID						
1. MET. B.L.	1.000	1.285	N/A	1.284	0.00	38029	04/10/00	1.27145	1.27145	0.0000	04/20/00					
2. 99-0939-t	10.327	1.284	3.280	2.000	78.2	31381		1.28921	1.30568	0.7974						
3. 99-0940-t	10.109	1.290	3.179	1.974	78.5	38381		1.27343	1.28519	0.5817						
4. 99-0941-t	10.180	1.291	3.211	1.978	78.4	38342		1.29000	1.30420	0.6414						
5. 99-0943-t	10.425	1.268	3.138	1.815	82.6	38303		1.28421	1.31403	1.430						
6. 99-0956-t	10.485	1.283	3.314	1.883	81.9	38321		1.28720	1.29186	0.2222						
7. 99-0957-t	10.931	1.284	3.054	1.924	79.1	31459		1.28448	1.31748	1.510						
8. 99-0958-t	10.601	1.270	3.222	2.027	76.5	38391		1.28214	1.32416	1.982						
9. 99-0967-t	10.046	1.286	3.733	1.887	83.9	31215		1.28420	1.28921	0.2489						
10. 99-0960-t	10.157	1.284	3.438	2.095	77.7	38028		1.29797	1.33744	1.944						
11. 99-0959-t	10.312	1.274	3.496	2.078	77.1	38341		1.29224	1.30260	0.5023						
12. 99-0966-t	10.405	1.298	3.414	1.998	79.5	38004		1.28508	1.33174	2.242						
13. 99-0963-t	10.270	1.284	3.188	2.155	72.7	38339		1.29097	1.31955	N/A						
14. 99-0962-t	10.274	1.275	3.244	2.063	75.7	31401		1.27240	1.27890	0.3163						
15. 99-0964-t	10.592	1.290	3.200	1.960	79.1	38273		1.28145	1.31884	1.764						
16. 99-1017-t	10.094	1.297	3.028	1.904	80.0	38392		1.28948	1.30355	0.6870						
17. 99-1018-t	10.152	1.283	3.215	1.764	85.0	38368		1.28403	1.28767	0.7193						
18. 99-1016-t	10.145	1.282	3.544	1.925	81.9	38238		1.28822	1.32251	1.690						
19. 99-866-t	10.464	1.282	3.269	1.973	78.9	35330		1.29297	1.31039	0.8324						
20. 99-0963-t Dup	10.730	1.291	3.807	2.350	72.2	38278		1.28044	1.31714	1.711						
21. 99-1017-t MS	10.315	1.284	3.375	1.970	79.7	38370		1.27192	1.28737	0.7489						
22. LCS-508 PCB	N/A	N/A				31354		1.29583	1.29575	N/A						
23. SR11588e	N/A	N/A				38019		1.28858	1.49605	100.0						
24. 99-866-t Dup	10.123	1.280	3.002	1.918	78.7	38013		1.28864	1.30622	0.8673						

SPIKING SOLUTION NAME: 501 8201-5

501 8201-5

SPIKING SOLUTION NAME: S.O. SPEKLE SURR.; COMPLEX AC; ppDCB
 SPIKING SOLUTION CODE: P120299A; P042299B; P022199B
 SPIKING SOLUTION CONC. (ug/mL): 40PPB; 20PPB; 20PPB
 SPIKING SOLUTION VOL. (mL): 1.00mL; 1.00mL; 1.00mL
 SPIKED DATE/BY: G.M. 04/12/00; G7 04/12/00; G7 04/12/00
 WITNESSED DATE/BY: MC 04/12/00; MC 04/12/00; MC 04/12/00

SURROGATE SOLUTION NAME: PESTICIDE SURROGATE
 SURROGATE SOLUTION CODE: P041200A
 SURROGATE SOLUTION CONC. (ug/mL): 40PPB
 SURROGATE SOLUTION VOL. (mL): 1.00mL
 SPIKED DATE/BY: G7 04/12/00
 WITNESSED DATE/BY: MC 04/12/00

COMMENTS:

SAMPLE (2) 99-0956-t - MOSTLY SKEL.
 SAMP (3) SR11588e - 1.007g
 (X) Sample #13 99-0963-t 10.730 04/20/00 MC

⑦

Sampler Gary Ichikawa		Ph # 831-633-6032		Send Results To same				Lab Number L-009-00			
Address PO Box 747				Address				Field Number			
				City CA				Lab Storage TSM F3			
City Moss Landing		Zip 95039		Copies To				Spill Title			
Date Required/Reason 12/31/1999				Address				Suspect			
Shipped Via Courier				City Zip				Index-PCA			
<input type="checkbox"/> Fish & Wildlife Loss Date: _____ Region: _____				Water Temp: _____ F or C				pH: _____ DO: _____ mg/L Conductivity: _____ umhos/cm			
<input type="checkbox"/> DFG Code Violation: _____											
<input type="checkbox"/> Suspected or Potential Problem											
<input type="checkbox"/> Routine Analysis				Analysis Requested							
Sample Identification/Location (Draw map on separate sheet if necessary)				Collection Date _____ Time _____							
99-747-t 9999033BRS0101 Oceanside Pier				9							
99-728-t 99990445OKB0101 La Jolla Kelp Beds				9							
99-727-t 99990445CSH0201 La Jolla Kelp Beds				9							
99-755-t 9999051BYC0101 Mission Bay Jetty (south)				9							
99-711-t 99990611QUF0101 Ocean Beach Pier				9							
99-383-t 99990745BLS0101 Pt. Loma Kelp Bed				9							
99-384-t 99990745CSH0201 Pt. Loma Kelp Bed				9							
99-385-t 99990745OKB0301 Pt. Loma Kelp Bed				9							
99-356-t 99990811SSB0101 Shelter Island Pier				9							
99-357-t 99990811BSB0201 Shelter Island Pier				9							
99-754-t 99990816BLS0001 Shelter Island Pier				9							
99-352-t 99990911SSB0201 5th Avenue Marina Pier				9							
99-531-t 99991011WSP0101 Imperial Beach Pier				9							
99-533-t 99991001BRS0301 Imperial Beach Pier				9							
Problem Description 1999 Coastal Fish Contamination Study								Pollution Action Kit: Yes <input type="checkbox"/> No <input type="checkbox"/>			
Suspect/Incident Location								Glove Size: Large <input type="checkbox"/> Medium <input type="checkbox"/>			
Comments/Special Instructions Full Organics Scan on all samples								Hazmat Shipper Requested: Yes <input type="checkbox"/> No <input type="checkbox"/>			
Samples Relinquished By (Signature) John Negrey				Print Name John Negrey				Date 1-11-00			
								Received By (Signature) D.B. Crane			
								Print Name D.B. CRANE			

Sampler Gary Ichikawa	Ph # 831-633-6032	Send Results To same	Lab Number L-007-00
Address PO Box 747		Address	Field Number
City Moss Landing	Zip 95039	City CA	Lab Storage TSM F3
Date Required/Reason 12/31/1999		Copies To	Spill Title
Shipped Via Courier		Address	Suspect
		City	Index-PCA

<input type="checkbox"/> Fish & Wildlife Loss Date: _____ Region: _____		Water Temp: _____ F or C _____ pH: _____ DO: _____ mg/L Conductivity: _____ umhos/cm _____	
<input type="checkbox"/> DFG Code Violation: _____			
<input type="checkbox"/> Suspected or Potential Problem			
<input type="checkbox"/> Routine Analysis			
Analysis Requested			
Sample Identification/Location (Draw map on separate sheet if necessary)		Collection	
Date		Time	
99-732-1 99991168SSB0101 Agua Medionda Lagoon		9	
99-726-1 99991168ODT0201 Agua Hedionda Lagoon		9	
99-746-1 99991233OWC0301 Crystal Pier		9	
99-457-1 99991311SSB0101 Coronado Pier Ferry Landing		9	
99-751-1 99991318DT0701 Coronado Pier		9	
99-854-1 99330118SHS0201 Santa Cruz Wharf		3	
99-885-1 99330118SSD0301 Santa Cruz Wharf		3	
99-855-1 99330118SSD0301 Santa Cruz Wharf		3	
99-351-1 99330711SSP0101 Pismo Pier		3	
99-387-1 99330711BRS0301 Pismo Pier		3	
99-389-1 99330711WSP0401 Pismo Pier		3	

Problem Description 1999 Coastal Fish Contamination Study	Pollution Action Kit: Yes <input type="checkbox"/> No <input type="checkbox"/>
Suspect/Incident Location * SAMPLE 99-854 NOT IN BOXES NOT SHIPPED PER AUTUMN 1-12-00	Glove Size: Large <input type="checkbox"/> Medium <input type="checkbox"/>
Comments/Special Instructions Full Organics Scan on all samples AND 99-885 ARE SAME SAMPLE USE NO 99-854	Hazmat Shipper Requested: Yes <input type="checkbox"/> No <input type="checkbox"/>

Samples Relinquished By (Signature) John Negrey	Print Name John Negrey	Date 1-11-00	Received By (Signature) D.B. Crane	Print Name D.B. Crane

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

Laboratory No.: L-385-99

Page 1

Project Title: CFCS 1998 Year 1 Run 1

CALIBRATION

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

QAQC VERIFICATION

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

SAMPLE ANALYSIS

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

LIMS

- Results were entered into LIMS correctly
- The prepared and analytical dates was correct
- The correct MB/DCS/LCS data were entered
- NA The correct footnotes were used
- The data sheets are complete and included
- Method blanks are included with correct prep and analyzed dates
- Anomalies are written and entered

SIGNATURES BELOW INDICATE THE ABOVE CRITERIA HAVE BEEN MET

CHEMIST Kathleen Regalado

DATE 7/24/00

REVIEWER Bob Crane

DATE 7/24/00

SEE ELECTRONIC ANOMALY: _____

NO ANOMALIES: _____

COMMENTS

See 22. (page 10) Corrective Action - Pesticides
See 22. (page 18) Corrective Action - PCBs

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

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Summary Information

Name of Reviewer: <u>D. Crane</u>		Title: <u>Lab Director</u>	
Bench Sheet Numbers: <u>71</u>			
Required Samples		Sample Results Provided	
Sample Location or Sample ID	Analyte(s)	Sample Location or Sample ID	Analyte(s)
99-409	SO and PCB	99-409	SO and PCB
99-742		99-742	
99-923		99-923	
99-757		99-757	
99-882		99-882	
99-889		99-889	
99-892		99-892	
99-758		99-758	
99-759		99-759	
99-760		99-760	
99-550*		99-946	
99-549*			
99-946			
		<p>* 99-550 is currently being re-analyzed because of low surrogate recovery.</p> <p>*99-549 was lost (spilled) during extraction and was re-analyzed with L-385-99 set 2.</p>	

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Pesticide Data Inspection Checklist

1. Method Used / Extraction Completion Date: SO-TISSUE-PREP.SOPv5 / February 14, 2000
2. Number of Samples Analyzed: 12
3. Number of concentrations levels used for instrument calibration: 7

- | | |
|--|---|
| 4. Total No. of CCVs Required: <u>2</u>
(One for each 10 samples after the first 10 samples on each instrument) | Total No. of CCVs Reported: <u>2</u> |
| 5. Total No. of CCBs Required: <u>2</u>
(One for each CCV) | Total No. of CCBs Reported: <u>2</u> |
| 6. Total No. of Field Blanks Required: <u>NA</u>
(One per site or per 10 samples, whichever is more frequent) | Total No. of Field Blanks Reported: <u>NA</u> |
| 7. Total No. of Method Blanks Required: <u>1</u>
(One per batch) | Total No. of Method Blanks Reported: <u>1</u> |
| 8. Total No. of SRM analyses Required: <u>1</u>
(One per batch) | Total No. of SRM Analyses Reported: <u>1</u> |
| 9. Total No. of MS/MSD samples Required: <u>1</u>
(One MS/MSD per batch) | Total No. of MS/MSD samples Reported: <u>1</u> |
| 10. Total No. sample Duplicates Required: <u>1</u>
(One per 20 samples) | Total No. of sample Duplicates Reported: <u>2</u> |

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DATA QUALITY ASSURANCE REPORT**

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Pesticide Data Inspection Checklist

11. Initial Calibration

- a. Was a multiple point initial calibration performed*? ☒ Yes ☐ No
- b. Were all sample concentrations reported within the calibration range? ☒ Yes ☐ No
- c. 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 linearity criteria? $R^2 \geq 0.995$ ☒ Yes ☐ No
- e. If no, was a calculation curve used to calculate sample concentrations? ☐ Yes ☐ No

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

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

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

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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 followed 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:

<u>Analyte</u>	<u>Failed ICV Recovery</u>	<u>Concentration Detected in ICB</u>	<u>Affected Samples</u>
----------------	----------------------------	--------------------------------------	-------------------------

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

- a. Were CCVs run prior to each batch of 10 samples on each instrument? ☒ Yes ☐ No
- b. Were all CCV results within the specified windows" RPD \leq 25% (75-125% Rec) ☐ Yes ☒ No
- c. Was each CCV followed 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:

<u>Analyte</u>	<u>Affected Samples</u>	<u>Shifting Missing CCV/CCB</u>	<u>Failed CCV/CCB ID</u>
			RPD
Tedion	BS 71		CCV 2 (33%, 36%)
DBCE (surrogate)	BS 71		CCV 2 (33%, 42%)
Endrin	BS 71		CCV 2 (OK, 27%)
DBCE	BS71		CCV 3 (OK, 31%)

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DATA QUALITY ASSURANCE REPORT**

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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? ☒ 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

16. Field Blanks

- 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

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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:

<u>Analyte</u>	<u>SRM % R</u>	<u>SRM % R</u>	<u>Affected Samples</u>
gamma-HCH	56%		BS 71
Heptachlor epoxide	58%		BS 71
cis-nonachlor	56%		BS 71

18. MS/MSD Results

- a. Were appropriate number of MS/MSD pairs analyzed? ☒ Yes ☐ No
- b. Were all MS/MSD recoveries within specified windows? ($\geq 50\%$ Rec) ☐ Yes ☒ No
- c. Were all RPDs within the specified window? ($RPD \leq 50\%$) ☐ 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:

<u>Analyte</u>	<u>MS % R</u>	<u>MSD % R</u>	<u>MS/MSD RPD</u>	<u>LCS</u>	<u>Affected Samples</u>
p,p'-DDE	107*	40.0*	7.8	98.3	BS 71
heptachlor	14.8	30.7	69.7	31.5	BS 71
methyl parathion	45.3	44.9	0.8	55.7	BS 71

*p,p'-DDE concentration in unspiked sample was too high for concentration of spiking solution.

MS = 70.7 ppb DDE
MSD = 65.4 ppb DDE
unspiked = 62.2 ppb DDE

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Pesticide Data Inspection Checklist

19. Surrogate Recoveries

- a. Were appropriate surrogates analyzed? ☒ Yes ☐ No
- b. Were all surrogate recoveries within specified windows? ($\geq 50\%$ 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:

Analyte

Surrogate % R

Affected Samples

20. Duplicate Sample Precision

- a. Did duplicate sample analyses demonstrate acceptable precision? $RPD \leq 50\%$ ☒ 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:

Analyte

Sample

Sample Dup.

RPD

Affected Samples

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Pesticide Data Inspection Checklist

21.	Narrative	Corrective Action Taken?												
Section 13.	<p>Continuing Calibration Verification</p> <p>F3 CCV 2 (mid-sequence CCV) recoveries were low for tedion, DBCE (surrogate) and endrin (DB17 column only). CCV 2 is a mid-sequence CCV, the low recoveries for tedion and endrin were not found in the CCV 3 (the end of the run CCV). DBCE was also low in CCV 3 on one GC column (DB17). DBCE recovery on the DB5 was acceptable. DB5 data will be used for F3 surrogate correction.</p>	NO												
Section 17.	<p>SRM Results</p> <p>Three target analytes did not meet the SRM recovery criteria of 70-130% of the 95% CI. Gamma-HCH (56%), heptachlor epoxide (58%) and cis-nonachlor (56%). MS/MSD results for all three of these target analytes were acceptable. A possible possible explanation for the low recoveries in the SRM is that the F2 surrogate recovery (p,p'DDD*) was high in this sample (SRM). The recovery of the F2 surrogate was 118%. The average F2 surrogate recovery for all of the other samples in this set was 91.2%. Since the F2 analytes are corrected for surrogate recovery using this surrogate the surrogate corrected results for the F2 analytes could be low. If the F2 analytes in this sample are corrected using the average F2 surrogate recovery factor all of the F2 analyte concentrations are acceptable. The high DDD* recovery in the SRM is probably due to a coeluting peak in the F2 chromatogram. This interference was not found in the samples. No corrective action is necessary.</p>	NO												
Section 18.	<p>MS/MSD Results</p> <p><u>p,p'-DDE</u></p> <p>Recovery problems with p,p'-DDE are due to the relatively high concentration of DDE in the unspiked sample relative to the amount spiked:</p> <table><tr><td></td><td>DDE (ppb)</td><td></td></tr><tr><td>MS</td><td>70.7</td><td></td></tr><tr><td>MSD</td><td>65.4</td><td>MS/MSD RPD = 7.8%</td></tr><tr><td>unspiked</td><td>62.2</td><td></td></tr></table> <p>SRM and LCS recovery for p,p'-DDE were both acceptable. RPD for MS/MSD acceptable. No corrective action necessary.</p> <p><u>Heptachlor</u></p> <p>Low recovery of heptachlor is a continuing problem. The current method does not accurately or precisely quantify heptachlor. All ND, ≤RL should be qualified "UJ". Heptachlor was not reported at concentrations ≥RL in any of the samples.</p> <p><u>Methyl parathion</u></p> <p>Low recovery of methyl parathion is not unusual. The recovery of methyl parathion was marginally low at 45.3 and 44.9%. The precision was good at 0.8% RPD. The LCS recovery was slightly higher and acceptable at 55.7%. All ND, ≤RL should be qualified "UJ". Methyl parathion was not reported at concentrations ≥RL in any of the samples.</p>		DDE (ppb)		MS	70.7		MSD	65.4	MS/MSD RPD = 7.8%	unspiked	62.2		<p>NO</p> <p>YES</p> <p>YES</p>
	DDE (ppb)													
MS	70.7													
MSD	65.4	MS/MSD RPD = 7.8%												
unspiked	62.2													

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DATA QUALITY ASSURANCE REPORT**

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Pesticide Data Inspection Checklist

22. Corrective Action Taken

Heptachlor

All ND and \leq RL should be qualified "UJ" and reported concentrations should be qualified "J".

Methyl parathion

All ND and \leq RL should be qualified "UJ" and reported concentrations should be qualified "J".

Data Qualifiers:

- 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.

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PCB Data Inspection Checklist

19. Method Used: SO-TISSUE-PREP.SOPv5

20. Number of Samples Analyzed:

21. Number of concentrations levels used for instrument calibration: 8

4. Total No. of CCVs Required: (One for each 10 samples after the first 10 samples on each instrument)	<u>2</u>	Total No. of CCVs Reported:	<u>2</u>
5. Total No. of CCBs Required: (One for each CCV)	<u>2</u>	Total No. of CCBs Reported:	<u>2</u>
6. Total No. of Field Blanks Required: (One per site or per 10 samples, whichever is more frequent)	<u>NA</u>	Total No. of Field Blanks Reported:	<u>NA</u>
7. Total No. of Method Blanks Required: (One per batch)	<u>1</u>	Total No. of Method Blanks Reported:	<u>1</u>
8. Total No. of SRM analyses Required: (One per batch)	<u>1</u>	Total No. of SRM Analyses Reported:	<u>1</u>
9. Total No. of MS/MSD samples Required: (One MS/MSD per batch)	<u>1</u>	Total No. of MS/MSD samples Reported:	<u>1</u>
10. Total No. sample Duplicates Required (One per 20 samples)	<u>1</u>	Total No. of sample Duplicates Reported:	<u>2</u>

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PCB Data Inspection Checklist

11. Initial Calibration

- a. Was a multiple point initial calibration performed*? ☒ Yes ☐ No
- b. Were all sample concentrations reported within the calibration range? ☒ Yes ☐ No
- c. 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 linearity criteria? $R^2 \geq 0.995$ ☒ Yes ☐ No
- e. If no, was a calculation curve used to calculate sample concentrations? ☐ Yes ☐ No

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

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

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

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PCB Data Inspection Checklist

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

- | | | | |
|----|--|---|-----------------------------|
| a. | Was an ICV run prior to field samples? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| b. | Were ICV results within the specified windows? (75-125% Rec) | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| c. | Was the ICV followed by an ICB? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| d. | Was the ICB free from contamination? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |

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

<u>Analyte</u>	<u>Failed ICV Recovery</u>	<u>Concentration Detected in ICB</u>	<u>Affected Samples</u>
----------------	----------------------------	--------------------------------------	-------------------------

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

- | | | | |
|----|---|---|-----------------------------|
| a. | Were CCVs run prior to each batch of 10 samples on each instrument? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| b. | Were all CCV results within the specified windows" (75-125% Rec) | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| c. | Was each CCV followed by a CCB? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| d. | Was each CCB free from contamination? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |

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

<u>Analyte</u>	<u>Affected Samples</u>	<u>Shifting Missing CCV/CCB</u>	<u>Failed CCV/CCB ID</u>
----------------	-------------------------	---------------------------------	--------------------------

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PCB 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:

<u>Analyte</u>	<u>Affected Samples</u>	<u>Blank Concentration Reported</u>	<u>Shift Missing MB</u>
----------------	-------------------------	-------------------------------------	-------------------------

16. Field Blanks

- a. Was a field blank analyzed for each 10 samples per site? **NA** ☐ 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:

<u>Analyte</u>	<u>Affected Samples</u>	<u>Blank Concentration Reported</u>	<u>Shift Missing FB</u>
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CDFG FISH AND WILDLIFE WATER POLLUTION CONTROL LABORATORY DATA QUALITY ASSURANCE REPORT

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Project Title: CFCS 1998 Year 1 Run 1

PCB 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:

<u>Analyte</u>	<u>SRM % R</u>	<u>SRM % R</u>	<u>Affected Samples</u>
#87	63.6		BS 71
#105	45.4		BS 71
#128	50.0		BS 71
#156	45.9		BS 71

18. MS/MSD Results

- a. Were appropriate number of MS/MSD pairs analyzed? ☒ Yes ☐ No
- b. Were all MS/MSD recoveries within specified windows? ($\geq 50\%$ Rec) ☐ Yes ☒ No
- c. Were all RPDs within the specified window? ($RPD \leq 50\%$) ☒ 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:

<u>Analyte</u>	<u>MS % R</u>	<u>MSD % R</u>	<u>MS/MSD RPD</u>	<u>LCS</u>	<u>Affected Samples</u>
#8	0.0	13.2	—	59.7	BS 71
#18	30.1	43.5	36.6	71.0	BS 71
#27	31.4	39.6	23.0	76.8	BS 71
#29	0.0	0.0	—	22.9	BS 71
#128	44.2	52.3	16.9	85.0	BS 71
#110	43.3	52.4	19.1	93.3	BS 71
#56	0.0	0.0	—	35.3	BS 71
#60	0.0	0.0	—	48.5	BS 71

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

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PCB Data Inspection Checklist

19. Surrogate Recoveries

- a. Were appropriate surrogates analyzed? ☒ Yes ☐ No
- b. Were all surrogate recoveries within specified windows? ($\geq 50\%$ 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: **NA**

Surrogate

Surrogate % R

Affected Samples

20. Duplicate Sample Precision

- a. Did duplicate sample analyses demonstrate acceptable precision? $RPD \leq 50\%$ ☐ 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:

Analyte
#105

Sample
0.714

Sample Dup.
0.304

RPD
80.6

Affected Samples
BS 71

**CDFG FISH AND WILDLIFE WATER POLLUTION CONTROL LABORATORY
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PCB Data Inspection Checklist

21.	Narrative	Corrective Action Taken?
17.0	SRM Results	
	Low SRM recovery for congeners #87, 105, 128, and 156. These congeners did not meet SRM recovery criteria of 70-130% of the 95% CI.	
	<u>#87, 105 and 156 -</u>	NO
	These three congeners had acceptable MS/MSD recoveries. No corrective action taken.	
	<u>#128</u>	YES
	MS recovery for #128 was marginally low at 44.2%, MSD recovery was acceptable but low at 52.3%. The LCS recovery was acceptable at 85.0. All ND, ≤RL should be qualified "UJ" and reported concentrations should be qualified "J".	
18.0	MS/MSD Results	YES
	Congeners #8, #29, #56, and #60 were not recovered in either the MS, MSD or both. Congeners #18, #27, #128, and #110 had low recoveries (<50%) in the MS and #18 and #27 also had low recoveries in the MSD. The LCS had low recoveries for #29, #56, and #60. The MS exhibited a rolling baseline, which is unusual in the F1 fractions. Surrogate recoveries for the MS, MSD and LCS were all good (81-87%). Data for congener #110 is acceptable and will not be flagged because of acceptable SRM and MSD recoveries.	
20.0	Duplicate Sample Precision	NO
	The RPD for congener #105 between sample 99-742 and its duplicate was 80.6%. Precision criteria requires RPD ≤50% however, the reported concentration of congener #105 is near the reporting limit of 0.2 ppb and the precision criterion applies only to concentrations ≥ 10xRPD which for PCB congeners would be 2.0 ppb.	

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PCB Data Inspection Checklist

22. Corrective Action Taken

Congeners #8, #29, #56, and #60 analyses failed due to 0% recovery in the MS, MSD or both. These analytes will be designated as not analyzed (NA).

Results for congeners #18, #27, and #128 were low for either the SRM or the MS/MSD or both. Results for congeners #18, #27 and #128 should be qualified as follows:

Qualify all <RL and ND	"UJ"
Qualify all reported concentrations \geq RL	"J"

Data Qualifiers:

- 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.

See 20. Corrective Action (Pesticides)

See 20. Corrective Action (PCBs)

CDFG Water Pollution Control Laboratory
Data Inspection Checklist

Summary Information

Name of Reviewer: D. CRANE Title: LAB DIRECTOR

Required Samples		Sample Results Provided	
Sample Location or Sample ID	Analyte(s)	Sample Location or Sample ID	Analyte(s)
✓ 99-664 - 99-1032		All 29 results provided	
✓ 99-665 - 99-1031			
✓ 99-666 - 99-743			
✓ 99-842 - 99-821			
✓ 99-843 - 99-948			
✓ 99-673			
✓ 99-558			
✓ 99-560			
✓ 99-884			
✓ 99-867			
✓ 99-883			
✓ 99-873			
✓ 99-541			
✓ 99-543			
✓ 99-549			
99-545			
✓ 99-406			
✓ 99-825			
✓ 99-879			
✓ 99-880			
✓ 99-894			
✓ 99-896			
✓ 99-539			
✓ 99-858			
✓ 99-823			

Data Inspection Checklist

- | | | | |
|----|--|------------------------|--|
| 1. | Method Used: | SO-TISSUE-PREP. SOP v5 | |
| 2. | Number of concentrations levels used for instrument calibration: | 8 | |
| 3. | Total No. of CCVs Required:
(One for each 10 samples after the first 10 samples on each instrument) | 4 | Total No. of CCVs Reported: 7 |
| 4. | Total No. of CCBs Required:
(One for each CCV) | 4 | Total No. of CCBs Reported: 7 |
| 5. | 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 |
| 6. | Total No. of Method Blanks Required:
(One per batch) | 2 | Total No. of Method Blanks Reported: 2 |
| 7. | Total No. of SRM analyses Required:
(One per batch) | 2 | Total No. of SRM Analyses Reported: 2 |
| 8. | Total No. of MS/MSD samples Required:
(One MS/MSD per batch) | 2 | Total No. of MS/MSD samples Reported: 2 |
| 9. | Total No. sample Duplicates Required
(One per 20 samples) | 2 | Total No. of sample Duplicates Reported: 3 |

Data Inspection Checklist

10. Initial Calibration

1. Was a multiple point initial calibration performed*? ☒ Yes ☐ No
2. Were all sample concentrations reported within the calibration range? ☒ Yes ☐ No

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

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

- d. Did the initial calibration meet linearity criteria? ☒ Yes ☐ No
- e. If no, was a calculation curve used to calculate sample concentrations? ☐ Yes ☐ No

70.995

*A three point (minimum) initial calibration should be performed for each Analyte; if the RSD of the mean RRF is less than 15%, or if the RSD of the mean RF is less than 25%, then the averaged RRF or RF, respectively, may be used for that Analyte.

11. 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:

Data Inspection Checklist

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

- a. Was an ICV run prior to field samples? ☒ Yes ☐ No 85-115%
- b. Were ICV results within the specified windows? ☒ Yes ☐ No
- c. Was the ICV followed 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:

<u>Analyte</u>	<u>Failed ICV Recovery</u>	<u>Concentration Detected in ICB</u>	<u>Affected Samples</u>
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13. Continuing Calibration Verification (CCV)/Continuing Calibration Blank (CCB)

- a. Were CCVs run prior to each batch of 10 samples on each instrument? ☒ Yes ☐ No
- b. Were all CCV results within the specified windows? ☒ Yes ☐ No 75-125%
- c. Was each CCV followed 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:

<u>Analyte</u>	<u>Affected Samples</u>	<u>Shifting Missing CCV/CCB</u>	<u>Failed CCV/CCB ID</u>
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Data Inspection Checklist

14. **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? ☒ Yes ☐ No
- c. If the answer to item a or b was "no", document problems below:

AnalyteAffected SamplesBlank Concentration ReportedShift Missing MB15. **Field Blanks**

- 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? ☐ Yes ☐ No
- c. If the answer to item a or b was "no", document problems below:

AnalyteAffected SamplesBlank Concentration ReportedShift Missing FB

NA

Data Inspection Checklist

16. SRM Results

- a. Was appropriate SRM analyzed? ☒ Yes ☐ No
- b. Were SRM recoveries within specified windows? ☐ 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:

BS 75 76
Analyte SRM % RPD SRM % RPD

Affected Samples

low o,p'DDE	75 >30%	OK	BS 75
high dieldrin	>30%	OK	BS 75
low alpha HCH	OK	>30%	BS 76
low gamma HCH	>30%	OK	BS 75
low hept epoxide	>30%	>30%	BS 75, 76
low cis nonachlor	>30%	>30%	BS 75, 76

17. MS/MSD Results

- a. Were appropriate number of MS/MSD pairs analyzed? ☒ Yes ☐ No
- b. Were all MS/MSD recoveries within specified windows? ☐ Yes ☒ No >50%
- c. Were all RPDs within the specified window? ☐ Yes ☒ No <35%
- d. Was appropriate corrective action (e.g., MSA for GFAA, serial dilution for ICP) employed on affected samples? ☒ Yes ☐ No
- e. If the answer was "no" to items a-d above, document affected samples:

BS 75 76 n24 RSD
Analyte MS % R MSD % R MS/MSD RPD

Affected Samples

ethion	35.9/30.0	42.2/61.7	32.4	BS 75, 76
delta-HCH	0/6	27.4/61.0	156	BS 75, 76
heptachlor	31.7/40.1	OK		BS 75

Data Inspection Checklist

18. Additional Information

- a. Were Instrument Tune Data Provided? (GC/MS only) ☐ Yes ☐ No
- b. Were equipment blanks demonstrated to be free from contamination? ☐ Yes ☐ No
- c. Were statements of data quality provided? ☐ Yes ☐ No
- d. Did field duplicate demonstrate acceptable precision? ☐ Yes ☐ No

19. Narrative

Corrective
Action
Taken

SPM - Recoveries (USBA Cod Liver Oil) ppb
Low o,p'-DDE BS 75 samples Acceptable (14.7-29.9) No
Actual ~~9.2~~ 10.1
Acceptable ~~Rec~~ for BS 76 (16.7%)

dieldrin - high recovery BS 75 Acceptable (106-208) No
Actual % Rec = 276%
Acceptable ~~Rec~~ for BS 76 (177%)

alpha-HCH - low recovery BS 76 Acceptable (57.3-115%) No
Actual % Rec = 51.1%
Acceptable for BS 75 = 62.9

gamma-HCH low for BS 75 Acceptable = (16.2-34.6) No
Actual = 14.4
Acceptable for BS 76 = 19.5

hept epoxide low for both BS 75 & 76
Acceptable = (21.1-43.0) Yes
Actual BS 75 = 18.1
BS 76 = 16.9

Cis-norachlor low for both BS 75, 76
Acceptable = (64.4-127) Yes
Actual BS 75 = 59.0
BS 76 = 58.6

Corrective
Action
Taken?

MS/MSD

Qthion

<50% recovery for all but BS 76 MSD = 61.7% Yes
problem analyte — poor recovery/precision

Delta-HCH

<50% recovery — all
problem Analyte — poor recovery/precision Yes

heptachlor

low recovery BS 75 31.7% / 40.1 % No.
Precision — OK
BS 76 Acceptable recovery/precision

20.

Corrective Action Taken

heptachlor epoxide - Qualify < RL and ND "UT"
No data > RL reported

Cis-nonachlor - Qualify < RL and ND "UT"
Qualify data > RL "J"

ethion - Qualify < ND "UT"

delta-HCH - Analysis Failed - All entries NA

~~heptachlor~~ -

Data Inspection Checklist

- | | | | |
|----|--|--------------------------|--|
| 1. | Method Used: | SO - TISSUE PREP. SOP V5 | |
| 2. | Number of concentrations levels used for instrument calibration: | 8 | |
| 3. | Total No. of CCVs Required:
(One for each 10 samples after the first 10 samples on each instrument) | 4 | Total No. of CCVs Reported: 7 |
| 4. | Total No. of CCBs Required:
(One for each CCV) | 4 | Total No. of CCBs Reported: 7 |
| 5. | 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 |
| 6. | Total No. of Method Blanks Required:
(One per batch) | 2 | Total No. of Method Blanks Reported: 2 |
| 7. | Total No. of SRM analyses Required:
(One per batch) | 2 | Total No. of SRM Analyses Reported: 2 |
| 8. | Total No. of MS/MSD samples Required:
(One MS/MSD per batch) | 2 | Total No. of MS/MSD samples Reported: 2 |
| 9. | Total No. sample Duplicates Required
(One per 20 samples) | 2 | Total No. of sample Duplicates Reported: 3 |

Data Inspection Checklist

10. Initial Calibration

1. Was a multiple point initial calibration performed*? ☒ Yes ☐ No
2. Were all sample concentrations reported within the calibration range? ☒ Yes ☐ No

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

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

- d. Did the initial calibration meet linearity criteria? ☒ Yes ☐ No $R^2 > 0.995$
- e. If no, was a calculation curve used to calculate sample concentrations? ☐ Yes ☐ No

*A three point (minimum) initial calibration should be performed for each Analyte; if the RSD of the mean RRF is less than 15%, or if the RSD of the mean RF is less than 25%, then the averaged RRF or RF, respectively, may be used for that Analyte.

11. 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:

Data Inspection Checklist

12. 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? ☒ Yes ☐ No
- c. Was the ICV followed 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:

<u>Analyte</u>	<u>Failed ICV Recovery</u>	<u>Concentration Detected in ICB</u>	<u>Affected Samples</u>
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13. Continuing Calibration Verification (CCV)/Continuing Calibration Blank (CCB)

- a. Were CCVs run prior to each batch of 10 samples on each instrument? ☒ Yes ☐ No
- b. Were all CCV results within the specified windows? ☒ Yes ☐ No
- c. Was each CCV followed 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:

<u>Analyte</u>	<u>Affected Samples</u>	<u>Shifting Missing CCV/CCB</u>	<u>Failed CCV/CCB ID</u>
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Data Inspection Checklist

14. 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? ☒ Yes ☒ No
- c. If the answer to item a or b was "no", document problems below:

Analyte	Affected Samples	Blank Concentration Reported	Shift Missing MB
BS 75 PCB 110	BS 75	0.239 ppb	
PCB 118	"	0.244 ppb	

Samples ~~with~~ results not qualified
 RL = 0.2 ppb

15. Field Blanks

- 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? ☐ 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
NA			

Data Inspection Checklist

16. SRM Results

- a. Was appropriate SRM analyzed? ☒ Yes ☐ No
- b. Were SRM recoveries within specified windows? ☐ 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:

<u>Analyte</u>	<u>SRM % R</u>	<u>SRM % R</u>	<u>Affected Samples</u>
# 66	high >30%	high >30%	BS 75, 76
# 87	OK	low-borderline	BS 76
# 118	high >30% OK	OK	BS 75 OK
# 156	OK	low-borderline	BS 76
# 31	high >30%	OK	BS 75

17. MS/MSD Results

- a. Were appropriate number of MS/MSD pairs analyzed? ☒ Yes ☐ No
- b. Were all MS/MSD recoveries within specified windows? ☐ Yes ☒ No
- c. Were all RPDs within the specified window? ☒ Yes ☐ No
- d. Was appropriate corrective action (e.g., MSA for GFAA, serial dilution for ICP) employed on affected samples? ☒ Yes ☐ No
- e. If the answer was "no" to items a-d above, document affected samples:

<u>Analyte</u>	<u>MS % R</u>	<u>MSD % R</u>	<u>MS/MSD RPD</u>	<u>Affected Samples</u>
# 29	31.1	32.3	OK	BS 75
# 56	43.8	41.4	OK	BS 75

Data Inspection Checklist

18. Additional Information

- a. Were Instrument Tune Data Provided? (GC/MS only) ☐ Yes ☐ No
- b. Were equipment blanks demonstrated to be free from contamination? ☒ Yes ☐ No
- c. Were statements of data quality provided? ☒ Yes ☐ No
- d. Did field duplicate demonstrate acceptable precision? ☒ Yes ☐ No

19. Narrative

Method Blanks - low level contamination for #110, #118
contamination is at the reporting limit - No
No corrective action necessary

Corrective
Action?SRM Results -PPb

#66 high Acceptable = (37.2-73.1)
Actual BS 75 = 151
BS 76 = 134

Yes

#87 low Acceptable = (38.6-74.6)
BS 75 OK = 45.8
76 = 37.9

No

#156 low Acceptable = (17.9-37.8)
BS 75 OK
BS 76 17.8

No

#31 high Acceptable = (5.64-11.2)
BS 75 = 12.0
76 = OK

No

MS/MSD

#29 low BS 75 31.1 / 32.3%

Yes

BS 76 OK

#56 low BS 75 43.8 / 41.4%

Yes

BS 76 OK

20.

Corrective Action Taken

66 All entries > RL Qualify "J"

29 Bench Sheet 75 only Qualify > RL "J"
" < RL & ND "UJ"# 56 Bench Sheet 75 only Qualify > RL "J"
" < RL & ND "UJ"

See 21. page 10-11 (Pesticides)

See 21. page 18 (PCBs)

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

Laboratory No.: L-009/041-2000

Page 2

Project Title: CFCS 1998 Year 1 Run 3

Summary Information

Name of Reviewer: <u>D. Crane</u>		Title: <u>Lab Director</u>	
Bench Sheet Numbers: <u>84, 85, 86</u>		Samples Received: <u>1/11/00 (L-009), 2/09/00 (L-041)</u>	
Required Samples		Sample Results Provided	
<p>Sample Location or Sample ID</p> <p>L-009-00 BS 84 and 85</p> <p>99-532</p> <p>99-949</p> <p>99-744 → 774?</p> <p>99-950</p> <p>99-951</p> <p>99-952</p> <p>99-764</p> <p>99-773</p> <p>99-730</p> <p>99-729</p> <p>99-725</p> <p>99-749</p> <p>99-765</p> <p>99-341</p> <p>99-747</p> <p>99-728</p> <p>99-727</p> <p>99-755</p> <p>99-711</p> <p>99-383</p> <p>99-384</p> <p>99-385 ← 356?</p> <p>99-386 ← Not in Brod. spreadsheet</p> <p>99-357</p> <p>99-754</p> <p>99-352</p> <p>99-531</p> <p>99-533</p> <p>99-732</p> <p>99-726</p> <p>99-746</p> <p>99-457</p> <p>99-751</p> <p>99-885 — Not on Brod spreadsheet</p> <p>99-855</p> <p>99-351</p> <p>99-387</p> <p>99-389</p> <p>L-041-00 BS 86</p> <p>99-939 99-962</p> <p>99-940 ✓ 99-963</p> <p>99-941 99-964</p> <p>99-943 99-1017</p> <p>99-956 99-1018</p> <p>99-957 99-1016</p> <p>99-958 99-866</p> <p>99-967</p> <p>99-960</p> <p>99-959</p> <p>99-966</p>	<p>Analyte(s)</p> <p>SO and PCB</p>	<p>Sample Location or Sample ID</p> <p>All Samples Results Provided except</p> <p>99-764 (F3)</p> <p>99-885 (F1)</p> <p>99-963</p> <p>Lost</p> <p>The lost samples are currently being Re-analyzed.</p>	<p>Analyte(s)</p> <p>SO and PCB</p>

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

Laboratory No.: L-009/041-2000
Project Title: CFCS 1998 Year 1 Run 3

Page 3

Pesticide Data Inspection Checklist

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

2. Number of Samples Analyzed: 56

3. Number of concentrations levels used for instrument calibration: ⁷(F1 & F3), 8 (F2)

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

6

Total No. of CCVs Reported:

6

5. Total No. of CCBs Required:
(One for each CCV)

6

Total No. of CCBs Reported:

6

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)

3

Total No. of Method Blanks Reported:

3

8. Total No. of SRM analyses Required:
(One per batch)

3

Total No. of SRM Analyses Reported:

3

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

3*

Total No. of MS/~~MSD~~ samples Reported:

3

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

3

Total No. of sample Duplicates Reported:

2**

* * Sample 99-0963-t was lost during extraction
duplicate analysis frequency = 3.6%

* 3 MS were analyzed with BS 84, 85, 86 which
were analyzed as a single batch of samples.
MS frequency = 5.4% Precision will be determined
as %RSD on the 3 MS.

**CDFG FISH AND WILDLIFE WATER POLLUTION CONTROL LABORATORY
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11. Initial Calibration

- a. Was a multiple point initial calibration performed*? ☒ Yes ☐ No
- b. Were all sample concentrations reported within the calibration range? ☒ Yes ☐ No
- c. 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 $\leq 20\%$.

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

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

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

- | | | | |
|----|--|---|-----------------------------|
| a. | Was an ICV run prior to field samples? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| b. | Were ICV results within the specified windows? (75-125% Rec) | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| c. | Was the ICV followed by an ICB? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| d. | Was the ICB free from contamination? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| e. | If any item in a-d above was answered "no", list problems below: | | |

<u>Analyte</u>	<u>Failed ICV Recovery</u>	<u>Concentration Detected in ICB</u>	<u>Affected Samples</u>
----------------	----------------------------	--------------------------------------	-------------------------

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

- | | | | |
|----|---|---|--|
| a. | Were CCVs run prior to each batch of 10-15 analyses on each instrument? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| b. | Were all CCV results within the specified windows" (75-125% Rec) | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> No |
| c. | Was each CCV followed by a CCB? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| d. | Was each CCB free from contamination? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| e. | If any item in a-d above was answered "no", list problems below: | | |

<u>Analyte</u>	<u>Affected Samples</u>	<u>Shifting Missing CCV/CCB</u>	<u>Failed CCV/CCB ID</u>
----------------	-------------------------	---------------------------------	--------------------------

b. All FI Pst ~~777~~
~~1-1000~~

Vial 41

All CCV's prior to and following Vial 41 were acceptable.
Determined bad injection - Vial 41. Samples not affected, no
corrective action.

~~P2 Pst Ethion~~

~~Vial 74~~

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

Analyte

Affected Samples

Blank Concentration Reported

Shift Missing MB

16. Field Blanks

- a. Was a field blank analyzed for each 10 samples per site? ☐ Yes ☐ No **NA**
- b. Was each field blank demonstrated to be free from contamination? <RL ☐ Yes ☐ No **NA**
- c. If the answer to item a or b was "no", document problems below:

Analyte

Affected Samples

Blank Concentration Reported

Shift Missing FB

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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:

<u>Analyte</u>	<u>SRM % R</u>	<u>SRM % R</u>	<u>Affected Samples</u>
Ca-chloride	51%		BS 86
o,p'-DDE	65%	52%	BS 85, 86
α-HCH	55%		BS 85
* γ-HCH	ND	65%	BS 84, 86
hept epox	51.5%	56.5% 59.5%	BS 84, 85, 86
HEP cis-Monochloro	60.6%	62.2% 56.4%	BS 84, 85, 86
dieldrin	61.9%		BS 85

18. MS/MSD Results

- a. Were appropriate number of MS/MSD pairs analyzed? ☒ Yes ☐ No
- b. Were all MS/MSD recoveries within specified windows? ($\geq 50\%$ Rec) ☐ Yes ☒ No
- c. Were all RPDs within the specified window? ($RPD \leq 50\%$) **RSD $\leq 50\%$** ☒ 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:

<u>Analyte</u>	<u>MS % R</u>	<u>MSD % R</u>	<u>MS ^{70-130% Rec} MSD RPD</u>	<u>RSD</u>	<u>Affected Samples</u>
* dithion	46.8	37.8	29.2	23.2	All
* delta-HCH	6.2	1.9	0.0		All
* heptachlor	66.5	46.2	89.9	32.4	All
* methyl parathion	42.5	42.8	61.4	22.2	All

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19. Surrogate Recoveries

- a. Were appropriate surrogates analyzed? ☒ Yes ☐ No
- b. Were all surrogate recoveries within specified windows? ($\geq 50\%$ 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:

<u>Surrogate</u>	<u>Surrogate % R</u>	<u>Affected Samples</u>
DBCE	37.0	SRM 1588a BS 84
DBCE	30.4	SRM 1588a BS 86

20. Duplicate Sample Precision

- a. Did duplicate sample analyses demonstrate acceptable precision? $RPD \leq 50\%$ ☒ Yes ☐ No
- b. Did field duplicate demonstrate acceptable precision? ☒ Yes ☐ No
- c. If the answer was "no" to items a-d above, document affected samples:

<u>Analyte</u>	<u>Sample</u>	<u>Sample Dup.</u>	<u>RPD</u>	<u>Affected Samples</u>
----------------	---------------	--------------------	------------	-------------------------

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21. Narrative

Corrective Action
Taken?

FI Dillutions:

No

Undiluted samples exceeded calibration
and were diluted and re-analyzed -

<u>AS VIAL</u>	<u>SAMPLE</u>	<u>DILUTION</u>
7	99-532	0.2 + 1.0
13	99-764	0.2 + 1.0
18	99-749	0.2 + 1.0
28	99-755	0.2 + 1.0
29	99-711	0.2 + 1.0
30	99-532 dup	0.2 + 1.0
33	SRM 1588a	0.2 + 1.0
64	↓	↓
90		

Duplicate Analysis Frequency (page 3): NO

Sample 99-0963-t was lost during extraction resulting in duplicate analysis frequency of 3.6% for this set. For the entire year 1 project we analyzed 7 duplicate samples out of 101 analyses resulting in overall duplicate frequency of 6.9%.

Continuing Calibration Verification (page 5): NO

The FI Pesticide CCV (Vial 41) failed for all target analytes. All CCV's prior to and following Vial 41 were acceptable. We conclude that the GC Injection was bad. Samples were not affected and no corrective action taken.

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21. Narrative (cont.)

Corrective Action
Taken?

SRM Results (page 7)

YES - delta HCH

Low SRM 1588a target analyte recoveries were observed for the following analytes:

Cis-chlordane

heptachlor epoxide

op-DDT

cis-nonachlor

α -HCH

dieldrin

γ -HCH

The high lipid content of the SRM and the relatively high contamination of the F2 fraction makes this SRM especially difficult.

All of the SRM recoveries were >50% except gamma-HCH recovery for the SRM from BS 84 which was ND.

MS/MSD Results (page 7)

Yes

Four target compounds exhibited low MS recovery on one or more of the Matrix Spikes

Ethion

Yes

% Rec ranged from 29.2 to 46.8% - low for all BS.

Delta-HCH

Yes

% Recovery ranged from ND to 6.2% - low for all BS.

heptachlor

Yes

Heptachlor results from BS 85 only (46.2%).

methyl parathion

Yes

Low recovery for BS 84 (42.5%) and 85 (42.8%)

Surrogate Recovery (page 8)

No

Low recovery for DBCP (F3 analytes) in SRM 1588a BS 84 and 86

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22. Corrective Action Taken

ND or <RL Data Qualifies
Reported Conc.

Ethion - Low MS ~~test~~ Recovery
Qualify Results for all samples
in BS 84, 85, 86. All results
are ND. ~~or~~

UT

~~UT~~

Delta-HCH - Method failure to recover
this analyte. All results for
this compound should be reported
NA - not analyzed

Heptachlor - Slightly low recovery MS
recovery for BS 85. Qualify
results for samples on BS 85 only.
All results are ND.

UT

~~UT~~

Methyl Parathion -

low recovery for ^{MS} ~~samples~~ on
BS 84 and 85 but good precision.
All results ND or <RL.

UT

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1. Method Used / Extraction Completion Date: SO-TISSUE-PREP.SOPv5 / May 2, 2000
2. Number of Samples Analyzed: 56
3. Number of concentrations levels used for instrument calibration: 8

4. Total No. of CCVs Required: (One for each 10-15 samplest)	<u>6</u>	Total No. of CCVs Reported:	<u>6</u>
5. Total No. of CCBs Required: (One for each CCV)	<u>6</u>	Total No. of CCBs Reported:	<u>6</u>
6. Total No. of Field Blanks Required: (One per site or per 10 samples, whichever is more frequent)	<u>NA</u>	Total No. of Field Blanks Reported:	<u>NA</u>
7. Total No. of Method Blanks Required: (One per batch)	<u>3</u>	Total No. of Method Blanks Reported:	<u>3</u>
8. Total No. of SRM analyses Required: (One per batch)	<u>3</u>	Total No. of SRM Analyses Reported:	<u>3</u>
9. Total No. of MS/MSD samples Required: (One MS/MSD per batch)	<u>3*</u>	Total No. of MS/MSD samples Reported:	<u>3</u>
10. Total No. sample Duplicates Required (One per 20 samples)	<u>3</u>	Total No. of sample Duplicates Reported:	<u>2**</u>

* See page 3
** See page 3

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PCB Data Inspection Checklist

11. Initial Calibration

- a. Was a multiple point initial calibration performed*? ☒ Yes ☐ No
- b. Were all sample concentrations reported within the calibration range? ☒ Yes ☐ No
- c. 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 $\leq 20\%$.

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

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

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PCB 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 followed 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:

<u>Analyte</u>	<u>Failed ICV Recovery</u>	<u>Concentration Detected in ICB</u>	<u>Affected Samples</u>
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14. 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 followed 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:

<u>Analyte</u>	<u>Affected Samples</u>	<u>Shifting Missing CCV/CCB</u>	<u>Failed CCV/CCB ID</u>
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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. If the answer to item a or b was "no", document problems below:

<u>Analyte</u>	<u>Affected Samples</u>	<u>Blank Concentration Reported</u>	<u>Shift Missing MB</u>
----------------	-------------------------	-------------------------------------	-------------------------

16. Field Blanks

- 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:

<u>Analyte</u>	<u>Affected Samples</u>	<u>Blank Concentration Reported</u>	<u>Shift Missing FB</u>
----------------	-------------------------	-------------------------------------	-------------------------

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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	SRM % R	Affected Samples
PCB 66	250	260	285	BS 84, 85, 86
105	OK	OK	194	BS 86
128	68.9	65.2	66.1	BS 84, 85, 86
118	OK	OK	168	BS 86
156	69.4	67.4	67.8	BS 84, 85, 86
170	OK	68.9	OK	BS 85

18. MS/MSD Results

- a. Were appropriate number of MS/MSD pairs analyzed? ☒ Yes ☐ No
- b. Were all MS/MSD recoveries within specified windows? ($\geq 50\%$ Rec) ☐ Yes ☒ No
- c. Were all RPDs within the specified window? ($RPD \leq 50\%$) ☐ 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:

MS analyzed in triplicate - 3 bench sheets

Analyte	MS % R	MS % R	MS % R	MS/MSD RPD	Affected Samples
#5*	77.5	19.0	67.0	57.9	BS 84, 85, 86
#15*	ND	ND	24	—	BS 84, 85, 86

* We do not report Conger #5 or #15 because of analysis difficulty.

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19. Surrogate Recoveries

- | | | |
|---|---|-----------------------------|
| a. Were appropriate surrogates analyzed? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| b. Were all surrogate recoveries within specified windows? ($\geq 50\%$ Rec) | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| c. Were all target analyte concentrations corrected for surrogate recovery? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| d. Was appropriate corrective action employed on affected samples? | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| e. If the answer was "no" to items a-d above, document affected samples: | | |

Surrogate

Surrogate % R

Affected Samples

20. Duplicate Sample Precision

- | | | |
|--|---|-----------------------------|
| a. Did duplicate sample analyses demonstrate acceptable precision? $RPD \leq 50\%$ | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| b. Did field duplicate demonstrate acceptable precision? | <i>NA</i> <input type="checkbox"/> Yes | <input type="checkbox"/> No |
| c. If the answer was "no" to items a-d above, document affected samples: | | |

Analyte

Sample

Sample Dup.

RPD

Affected Samples

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PCB Data Inspection Checklist

21. Narrative

Corrective Action
Taken?

Duplicate Analysis Frequency (Page 12)
(See page 9)

No

Item 17b. (page 16) - SRM Results

PCB 66 High ~~sample~~ recovery (250-285%)
~~PCB 105~~ MS and LCS recoveries were all
Excellent. Matrix interference with this SRM.

~~Yes~~ No

PCB 105 High recovery BS - 86 only
MS and LCS recovery were good. Matrix
interference with this SRM

No

PCB 128 Borderline low recovery (65-68%)
MS and LCS recoveries good

No

PCB 118 High recovery BS 86 only
MS and LCS recoveries excellent

No

PCB 156 Borderline low recoveries BS 84, 85, 86
(67.4-69.4%). MS recoveries excellent,
LCS recoveries good.

No

PCB 170 Borderline low recoveries for BS 85
(68.9%). MS and LCS recoveries are
good.

No

Item 18 b,c. (page 16) MS/MSD Results

MS analyzed in triplicate (1 per Bench sheet)
Precision based on % RSD rather than RPD.
PCB #5, 15 were the only congeners not meeting all
QA criteria. PCB 5 and 15 are not reported.

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PCB Data Inspection Checklist

21. Narrative (cont.)

Corrective Action
Taken? _____

All congener data reported without
qualification.

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.

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22. Corrective Action Taken

None