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SUMMARY FICP REPORT
Interlaboratory Study on the Analysis of Organochlorinated Insecticides in Natural Waters

by

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Management Perspective

This is a summary report for the 1988 FICP Interlaboratory Study on the analysis of organochlorinated insecticides in water. Ninety-three laboratories in the FICP Check Sample Program were contacted concerning the above study, thirty-five agreed to participate, and twenty-three laboratories provided results.

This study showed that there are a great variety of methods and analytical techniques currently in use for the analysis of organochlorinated insecticides in water. However, despite this variation, most laboratories proved capable of providing accurate results on the samples provided. The interlaboratory medians agreed well with the design values of the samples and for most parameters, the range of results was quite reasonable.

Dr. J. Lawrence
Director, Research and Applications Branch

Perspective-gestion

Ce document est un rapport sommaire sur l'étude interlaboratoire de 1988 du CFIP portant sur l'analyse d'échantillons d'eau contenant des insecticides organochlorés. Parmi les 93 laboratoires participant au programme d'assurance de la qualité interlaboratoire auxquels on a demandé de participer, 35 laboratoires ont accepté et 23 d'entre eux ont fourni des résultats.

Cette étude a montré qu'il existe une grande variété de méthodes et de techniques analytiques utilisées pour doser les insecticides organochlorés dans l'eau. Malgré cette variété, la plupart des laboratoires ont dosé avec précision les substances contenues dans les échantillons qui leur ont été fournis. Les médianes interlaboratoires coïncidaient avec les valeurs théoriques et la variation des résultats étaient acceptables pour la plupart des paramètres.

Docteur J. Lawrence
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Abstract

An intercomparison study on the determination of eighteen organochlorinated insecticides in water is described. 23 Canadian laboratories participated by analyzing three water samples fortified by the participants themselves at the time of analysis with the spiking solutions provided. The concentration of these pesticides in the water samples ranged from 5 to 2500 ng/L. In addition, three direct-injection samples containing the same compounds were analyzed by each participant. Analytical data were assessed by various statistical treatments to identify outlying results and to evaluate laboratory performance. Most laboratories performed extremely well, whereas the performance of a few others on certain parameters indicated a need to reevaluate their internal quality control practices and the quality of their in-house standards.

Résumé

Une étude comparative portant sur le dosage de 18 insecticides organochlorés présents dans des échantillons d'eau est décrite. Vingt-trois laboratoires canadiens ont analysé chacun trois échantillons d'eau enrichis par les participants eux-mêmes au moment de l'analyse avec des solutions d'enrichissement qui leur ont été fournies. La concentration des pesticides dans les échantillons d'eau variait entre 5 et 2500 ng/L. De plus, chaque laboratoire a reçu trois échantillons contenant les mêmes composés et destinés à être analysés par injection directe. Les résultats des analyses ont été évalués à l'aide de différents traitements statistiques permettant de repérer les données aberrantes et de juger de la qualité de ces résultats. La plupart des laboratoires ont obtenu de très bons résultats tandis que quelques-uns d'entre eux ont montré des faiblesses relativement à certains paramètres et devraient donc revoir leurs procédures de contrôle de la qualité de même que leurs normes internes.

SUMMARY FICP REPORT

Interlaboratory Study on the Analysis of
Organochlorinated Insecticides in Natural Waters

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Yvonne D. Stokker and A.S.Y. Chau

Introduction

The following is a summary of the above study which is now closed.

In April, 1988, 93 participants in the FICP Check Sample Program were invited to participate in a round-robin study on the analysis of organochlorinated insecticides in water. In early June, 34 sets of samples were sent to those laboratories who had indicated an interest in participating. A few months later, a 35th laboratory joined the study. From the 35 sets of samples sent out, 23 sets of results were received. A list of the participating laboratories is given in Table 1.

Study Design

The participants in this study were requested to analyze for 18 organochlorinated insecticides (OCs) in six test samples. Each sample set was comprised of three 1.0 L water samples and six sealed glass ampules, each containing different mixtures of the 18 OCs in either acetone or iso-octane. The three water samples, (#1 and #2 from Lake Ontario and distilled water for sample #3), were to be fortified with the eighteen OCs using spiking solutions #1, #2 and #3, respectively. These water samples had previously been shown to be clean of the OCs under study. The resultant OC concentrations in the water samples are listed as the 'Design Values' on the data summaries in Tables 3 to 5. Following fortification, each sample was to be extracted using the laboratory's own routine method of analysis. Ampules #4, #5 and #6 were injection-ready 'standard' solutions of the same parameters in iso-octane. The participants were requested to determine the concentrations of the OCs using their own in-house standards and calibration procedures. Samples #2 (Lake Ontario water sample) and #5 (direct-injection ampule) were designed to contain the same concentration levels of each OC so that participants could estimate losses of OCs or contamination problems from the extraction procedure itself.

The wide range of concentration levels was selected in order to accommodate the specified detection limits or lowest routine concentration levels for each laboratory that had expressed an interest in participating.

Methodologies

The analytical procedures used by the participants in this study are presented in Table 2. Several different techniques were used for the extraction of the OCs from the water samples as well as for their analytical measurement.

The most commonly used method of extraction was that of liquid-liquid extraction using either dichloromethane or hexane. Two laboratories used benzene to extract the OCs from the water while one participant acidified their water samples to pH 2 with phosphoric acid before extraction. Only one laboratory extracted the OCs by passing the water samples through an XAD-7 resin column, using ethyl acetate to elute the compounds of interest.

The number of different procedures for cleaning up the sample extracts was even more varied than the number of different extraction techniques. Two laboratories used 3% deactivated Silica Gel columns, one participant applied their extracts to Neutral Alumina columns and nine used Florisil columns with varying degrees of activation. Ten laboratories did not use any cleanup columns. Only one laboratory further cleaned the extracts with mercury.

All participants in this study used electron capture gas chromatography for their OC analyses. Two laboratories used packed column GC, four used megabore columns and eight of the remaining laboratories analyzed for the OCs on capillary columns using dual ECD.

Only one laboratory in this study reported spike recoveries for the extraction of OCs from water.

Results and Discussion

Tables 3 to 8 list all sample results reported by the participants. While most laboratories provided data for all 18 OCs, a few omitted several parameters for which they do not routinely analyze. Only three laboratories were unable to measure all parameters in the water sample at the lowest concentration level (Sample #3). Four laboratories found some of the OCs to be below their detection limits in the lowest concentration injection-ready sample (Ampule #6). Because only three of the twenty-three laboratories participating in this study were able to detect any a-BHC in Ampule #4, it is believed that this particular compound may have been overlooked in the preparation of the sample.

The interlaboratory medians agreed quite well with the design values for all OCs except p,p'-methoxychlor. Recovery of this particular compound from both the water samples and the ampules ranged from 38 to 43%. It is believed, therefore, that the error may have occurred in the preparation of the methoxychlor stock solution that was used to prepare each of the OC mixtures.

Outliers were not rejected when calculating the interlaboratory medians since 98% of the results were well within five-fold of the design values for each parameter in both the water samples and the injection-ready samples. Except for the 'detection limit' samples (#3 and #6), each interlaboratory median was within 20% of its design value. In these two less concentrated samples, however, many laboratories were still capable of achieving good results for many of the OCs of interest.

Samples #2 (water sample) and #5 (direct-injection ampule) were designed to contain the same concentration levels of each OC so that participants could evaluate losses of OCs or contamination problems due to the extraction procedure itself. While most laboratories proved capable of achieving less than 20% difference between results for the majority of the OCs in these two samples, those who did suffer losses in their extraction or cleanup procedures saw this reflected primarily in the recoveries of aldrin, a-BHC, a-chlordane, b-endosulfan, and/or mirex.

Conclusion

The results of this study indicate that most laboratories, while using considerably different analytical methodologies, have the capability of performing sensitive, accurate analyses for organochlorinated insecticides in water. The majority of participants generated comparable and satisfactory results for all 18 OCs under study and demonstrated minimal losses due to the methodology itself. However, as seen by some of the extreme outlying results, a few laboratories should review their in-house quality control practices and the quality of their calibration standards.

FICP INTERLABORATORY QC STUDY ON ORGANOCHLORINATED INSECTICIDES IN WATER

Table 1. List of Participating Laboratories

Federal Government:

1. Agriculture Canada
   Laboratory Services Division
   Western and Northern Region
   Calgary, Alberta

2. Environment Canada
   Atlantic Region Water Quality Laboratory
   Moncton, New Brunswick

3. Environment Canada
   C&P (EPS) Laboratory Services
   Wastewater Technology Centre
   Burlington, Ontario

4. Environment Canada
   National Water Quality Laboratory
   Burlington, Ontario

5. Environment Canada
   C&P (EPS) Laboratory Services
   West Vancouver, B.C.

6. Health and Welfare Canada
   Health Protection Branch
   Halifax, Nova Scotia

Provincial Governments:

7. Agriculture et Alimentation Québec
   Complêxe Scientifique
   Ste-Foy, Quebec

8. Alberta Environmental Centre
   Pesticide Analysis and Research Station
   Vegreville, Alberta

9. Manitoba Environment and Workplace Safety and Health
   W.M. Ward Technical Services Lab.
   Winnipeg, Manitoba

List of Participating Laboratories (continued)

10. Ontario Ministry of Agriculture and Food
    Agriculture Laboratory Services
    Guelph, Ontario

11. Ontario Ministry of the Environment
    Drinking Water Organics Section
    Rexdale, Ontario

12. Ministère de l'Environnement du Québec
    Laboratoire de Montréal
    St. Vincent-de-Paul (Laval), Quebec

13. Ontario Ministry of the Environment
    Pesticide Laboratory
    Rexdale, Ontario

14. Ministère de l'Environnement du Quebec
    Complexe Scientifique
    Ste-Foy, Quebec

Private Laboratories:

15. Barringer Laboratories
    Mississauga, Ontario

16. Cantest Ltd.
    Vancouver, B.C.

17. Chemex Labs Alberta Ltd.
    Calbary, Alberta

18. Chemlab Inc.
    Saint John, N.B.

19. Enviroclean
    London, Ontario

20. Enviro-test Laboratories
    Edmonton, Alberta

21. Mann Testing Laboratories ltd.
    Mississauga, Ontario

22. New Brunswick Research and Productivity Council
    Chemical and Biotechnical Services
    Fredricton, N.B.

23. Novalab Ltee
    Lachine, P.Q.

Table 2. Summary of Analytical Methodologies for the Analysis of OCs in Water Samples

Lab. No.	Extraction	Cleanup Steps	Analysis	GC columns, etc.
F02	-extract 500mL water with 3 x 100mL CH_2Cl_2 -dry through Na_2SO_4		dual ECD	30m x .32mm DB-1 30m x .32mm DB-210 -spike recoveries 90-112%
F07	-extract with 3 x 60mL CH_2Cl_2 -dry through Na_2SO_4	-8cm x 3% deact. Silica Gel: elute A (25mL hexane); B (30mL benzene)	ECD	30m x .25mm DB-5
F19	-extract with 2 x 100mL hexane		ECD	?
F28	-500mL water through XAD-7 column -vacuum-dry column -elute with 100mL ethyl acetate		ECD	30m x .53mm DB-17
F33	-extract with 3 x 100mL CH_2Cl_2	-8cm x 3% deact. Silica Gel: elute A (30mL hexane); B (60mL CH_2Cl_2/hexane [1+1])	dual ECD	30m x .25mm DB-1 30m x .25mm DB-5
F35	-add NaCl; acidify to pH 2 with phosphoric acid -extract with 3 x CH_2Cl_2		ECD	30m SPB-608
F38	-extract with hexane	-activated Florisil cleanup	ECD	1.8m x 2mm packed column [1.5% OV-17/2.0% OV-210]
F40	-extract with CH_2Cl_2 -dry with Na_2SO_4	-Florisil cleanup column, elute with CH_2Cl_2	dual ECD	??
F43	-extract with (100+50+50)mL CH_2Cl_2	-10g Florisil column; elute with petroleum ether/ethyl ether	ECD (not dual)	30m x .518mm DB-608 30m x .329mm DB-5
F47	-extract 300mL water with NaCl + 3 x CH_2Cl_2; dry through Na_2SO_4		ECD	30m x .25mm DB-5
F56	(analyzed 'standards' only)		?	?
F58	-extract 800mL water with 3 x hexane		dual ECD	30m x .32mm DB-1701 30m x .32mm DB-5
F59	-extract with 3 x 50mL CH_2Cl_2/hexane [15+85]; dry through Na_2SO_4	-mini 4% deact. Florisil; elute with hexane to ethyl ether/hexane (4 fractions)	ECD	megabore DB-608

(Table 2 continues next page)

Table 2 cont'd. Summary of Analytical Methodologies for the Analysis of OCs in Water Samples

Lab. No.	Extraction	Cleanup Steps	Analysis	GC columns, etc.
F61	-extract with NaCl + 3 x 100mL CH_2Cl_2 -dry through Na_2SO_4		ECD (not dual)	30m DB-1701 30m DB-5
F63	-extract with 3 x 60mL CH_2Cl_2 -dry through Na_2SO_4	-midi Florisil column; elute two fractions: CH_2Cl_2/hexane and CH_2Cl_2	dual ECD	30m x .25mm DB-5 30m x .25mm DB-1701
F64	-extract with CH_2Cl_2	-Florisil column	dual ECD	DB-17, DB-1701
F65	-extract with CH_2Cl_2	-Florisil cleanup; elute with ether/hexane	dual ECD	30m x .25mm DB-5 30m x .25mm DB-1701
F77	-extract with 100mL hexane		ECD	30m x .25mm DB-5
F79	-extract with (100+50+50)mL hexane -dry through Na_2SO_4	-Florisil cleanup; elute with ether/hexane	ECD	30m DB-5
F83	-extract with 3 x 60mL CH_2Cl_2 -pass through Na_2SO_4		ECD	25m x .2mm Ultra-2
F85	-extract with (80+40+40)mL CH_2Cl_2 -dry with Na_2SO_4	-Florisil/CH_2Cl_2 column; elute with CH_2Cl_2 -remove S-compounds with Hg	dual ECD	30m x .25mm SPB-1 30m x .25mm DB-1701
F86	-extract with 3 x 25mL benzene -dry through Na_2SO_4	-micro 5% deact. Neutral Alumina column; elute with benzene/hexane [25+75]	ECD-^{3}H	packed 3% SP-2401
F93	-extract with 4 x 25mL benzene		ECD	30m x .5mm DB-608

1988 FICP INTERLABORATORY QC STUDY

on

ORGANOCHLORINATED INSECTICIDES IN WATER

Table 3. Results for Sample 1 (water) ng/L

Laboratory No.	Parameter								
	Aldrin	a-BHC	b-BHC	g-BHC (lindane)	a-Chlordane (cis)	g-Chlordane (trans)	p,p'-DDD (TDE)	p,p'-DDE	o,p'-DDT
F02	66	91	188	95	419	337	130	144	128
F07	65.4	74.5	-	80.5	405	367	-	202	298
F19	93.1	109	-	106	463	432	142	197	142.1
F28	77	61	180	72	398	417	-	180	66
F33	83.6	114	-	110	506	418	186	224	217
F35	137	87	247	91	-	236	308	286	-
F38	87	79	-	79	420	370	170	170	180
F40	85.0	67.5	200	66	460	395	253	264	245
F43	100	100	250	120	590	450	400	200	-
F47	-	70	172	79	-	-	151	147	-
F58	123	81	278	92	455	394	314	185	166
F61	68	91	210	80	-	260	200	410	420
F63	96.70	96.92	194.68	99.88	-	-	199.46	184.53	210.30
F64	109	101	160	110	204	317	216	106	-
F65	57.6	60.3	204.4	64.4	379.7	256.0	224.0	187.7	231.5
F77	73.9	78.2	155.7	86.9	419.2	357.5	183.8	184.6	145.0
F79	-	97	190	94	-	-	200	210	-
F83	165	193	363	206	-	-	581	503	85.5
F85	78.0	70.0	186.0	97.0	395.0	320.0	218.0	85.0	230.0
F86	-	-	-	-	-	-	-	5.06	581
F93	0.67	110	190	110	-	-	-	200	-
Interlab Median	84.3	89.0	192.3	93.0	419.2	367.0	200.0	187.7	210.3
Design Value	96.7	98.4	176.4	97.5	495.3	433.1	191.9	201.0	194.0

nd not detected

\- not analyzed

1988 FICP INTERLABORATORY QC STUDY

on

ORGANOCHLORINATED INSECTICIDES IN WATER

Table 3 cont'd. Results for Sample 1 (water) ng/L

Laboratory No.	Parameter								
	p,p'-DDT	Dieldrin (HEOD)	a-Endo-sulfan	b-Endo-sulfan	Endrin	Heptachlor	Heptachlor Epoxide	p,p'-Methoxy-chlor	Mirex
F02	150	171	910	860	158	77	205	626	116
F07	143	102	848	765	92.0	65.0	163	472	113
F19	641	179	1000	974	285	114	201	2180	191
F28	201	162	1036	1022	140	79	200	1016	-
F33	225	200	1080	1100	182	95.0	210	883	189
F35	361	240	-	912	802	119	-	2095	251
F38	180	120	670	500	100	170	130	2300	190
F40	280	180	626	570	186	62.5	187	1510	264
F43	250	220	1500	-	220	120	250	1100	220
F47	158	-	-	-	-	-	173	892	168
F58	160	192	1058	886	173	83	215	846	208
F61	190	200	-	1200	300	90	220	1100	190
F63	225.44	190.78	1171.59	985.66	161.30	93.27	194.75	875.41	182.94
F64	114	75	338	726	81	91	62	90	115
F65	304.6	225.1	251.7	562.0	255.2	60.2	224.5	1301.0	75.4
F77	168.2	155.9	941.0	592.0	159.3	79.2	204.3	811.2	1604
F79	230	-	-	-	-	97	190	910	160
F83	436	478	2860	2360	522	192	478	-	-
F85	215.0	140.0	540.0	500.0	170.0	74.0	160.0	1400.0	245.0
F86	924	-	-	-	-	-	-	-	-
F93	220	190	-	590	190	130	110	840	160
Interlab Median	220.0	185.0	941.0	860.0	177.5	91.0	200.0	910.0	189.5
Design Value	193.8	186.8	912.6	1024.2	207.2	96.4	209.2	2410.6	195.7

nd not detected

\- not analyzed

1988 FICP INTERLABORATORY QC STUDY

on

ORGANOCHLORINATED INSECTICIDES IN WATER

Table 4. Results for Sample 2 (water) ng/L

Laboratory No.	Parameter								
	Aldrin	a-BHC	b-BHC	g-BHC (lindane)	a-Chlordane (cis)	g-Chlordane (trans)	p,p'-DDD (TDE)	p,p'-DDE	o,p'-DDT
F02	14	16	30	19	72	60	23	31	28
F07	13.1	13.2	-	15.6	64.6	58.3	-	20.8	79.1
F19	20.8	25.8	-	21.3	101	79.9	31.8	41.6	95.3
F28	14	7	35	10	70	78	-	36	11
F33	13.1	21.5	-	22.0	124	98.9	38.8	41.4	35.1
F35	29	20	55	19	-	56	59	61	-
F38	16	14	-	13	75	65	29	30	31
F40	17.5	12.0	30.0	11.5	65.0	77.0	38.0	38.0	30.0
F43	15	<20	45	<20	100	70	65	40	-
F47	-	11	39	20	-	-	27	25	-
F58	18	19	42	17	83	70	55	33	33
F59	14.9	9.67	25.6	10.0	67.3	71.1	-	30.4	-
F61	12	26	59	16	-	99	42	180	220
F63	17.90	17.42	34.12	17.85	-	-	35.34	36.14	37.41
F64	16	14	28	18	42	66	34	19	-
F65	12.5	9.4	43.2	11.5	54.5	89.8	25.0	33.8	52.1
F77	15.2	14.7	32.0	16.7	74.4	65.4	32.3	34.5	26.5
F79	-	14	38	15	-	-	34	47	-
F83	47.4	47.4	90.6	45.0	-	-	120	115	278
F85	18.0	19.0	42.0	17.0	91.0	72.0	46.0	44.0	40.0
F86	-	-	-	-	-	-	-	108	146
F93	10	8.9	22	9.4	-	-	-	20	-
Interlab Median	15.2	14.4	38.0	16.9	73.2	70.6	34.0	36.1	37.4
Design Value	19.3	19.7	35.3	19.5	99.1	86.6	38.4	40.2	38.8

nd not detected

\- not analyzed

1988 FICP INTERLABORATORY QC STUDY

on

ORGANOCHLORINATED INSECTICIDES IN WATER

Table 4 cont'd. Results for Sample 2 (water) ng/L

Laboratory No.	Parameter								
	p,p'-DDT	Dieldrin (HEOD)	a-Endo-sulfan	b-Endo-sulfan	Endrin	Heptachlor	Heptachlor Epoxide	p,p'-Methoxy-chlor	Mirex
F02	36	28	145	132	28	18	34	125	21
F07	29.3	33.2	150	145	30.2	11.4	35.4	179	23.2
F19	173	39.3	201	218	62.3	25.0	43.4	491	45.0
F28	34	31	202	212	29	15	38	227	-
F33	45.9	42.4	303	319	40.0	14.9	53.0	211	50.9
F35	nd	50	-	229	187	24	-	356	66
F38	31	28	160	120	24	30	34	550	32
F40	30.0	28.2	105	83	33.5	12.0	34.0	155.0	37.0
F43	35	40	260	-	40	20	40	200	35
F47	29	-	-	-	-	-	31	175	33
F58	31	37	197	146	33	15	40	163	31
F59	29.4	28.5	167	168	-	13.7	30.5	124	413
F61	38	41	-	240	150	14	46	220	39
F63	37.32	36.32	233.99	187.10	30.1	16.8	36.25	167.6	32.78
F64	13	14	65	159	19	16	13	18	25
F65	60.2	39.1	240.5	260.0	58.2	13.4	39.6	260.1	15.5
F77	33.7	29.2	156.6	64.1	26.1	17.2	36.9	111.9	195.5
F79	46	-	-	-	-	19	39	190	29
F83	103	100	665	578	121	55.0	104	-	-
F85	39.0	43.0	124.0	147.0	33.0	18.0	42.0	190.0	40.0
F86	232	-	-	-	-	-	-	-	-
F93	30	20	-	98	21	7.3	23	90	15
Interlab Median	35.0	36.3	182.0	163.5	33.0	16.4	37.5	184.5	33.0
Design Value	38.8	37.4	182.5	204.8	41.4	19.3	41.8	482.1	39.1

nd not detected

not analyzed

1988 FICP INTERLABORATORY QC STUDY
on
ORGANOCHLORINATED INSECTICIDES IN WATER

Table 5. Results for Sample 3 (water) ng/L

Laboratory No.	Parameter								
	Aldrin	a-BHC	b-BHC	g-BHC (lindane)	a-Chlordane (cis)	g-Chlordane (trans)	p,p'-DDD (TDE)	p,p'-DDE	o,p'-DDT
F02	nd	6	nd	6	17	13	nd	nd	nd
F07	6.80	0.192	-	4.96	17.5	15.7	-	4.34	16.5
F19	5.00	<1.2	-	5.15	26.7	20.9	4.40	5.30	22.8
F28	43	0.8	5.0	4.0	22	21	-	5.0	5.0
F33	3.01	2.46	-	5.46	22.3	19.1	3.24	3.62	3.53
F35	5.8	2.7	8.1	6.2	-	14.3	7.2	7.7	-
F38	4.0	2.1	-	3.9	19	16	3.6	3.7	3.7
F40	6.0	4.0	-	5.5	26.0	22.0	10.0	7.3	7.0
F43	<10	<20	<30	<20	23	20	<30	<10	-
F47	-	nd	nd	nd	-	-	nd	nd	-
F58	7	4	20	8	29	23	14	10	14
F59	2.98	2.36	3.32	2.67	13.1	13.8	-	3.06	-
F61	4.6	nd	15	3.2	-	35	3.7	33	29
F63	4.80	nd	5.28	5.02	-	-	5.68	5.30	10.02
F64	4	2	3	6	14	21	3	2	-
F65	1.6	nd	nd	1.8	15.5	10.0	nd	2.3	6.2
F77	3.9	2.8	5.6	5.4	20.4	7.3	4.4	4.8	4.0
F79	-	2.9	2.5	1.7	-	-	2.0	2.7	-
F83	15.4	12.7	20.3	14.0	-	-	17.8	22.9	12.9
F85	4.0	2.0	7.0	6.0	22.0	19.0	7.0	3.5	5.0
F86	-	-	-	-	-	-	-	23	27
F93	28	3.6	5.0	4.7	-	-	-	3.9	-
Interlab Median	4.80	2.58	5.44	5.15	21.2	19.1	4.40	4.80	8.51
Design Value	4.83	4.92	4.41	4.88	24.8	21.7	4.80	5.02	4.85

nd not detected
- not analyzed

1988 FICP INTERLABORATORY QC STUDY
on
ORGANOCHLORINATED INSECTICIDES IN WATER

Table 5 cont'd. Results for Sample 3 (water) ng/L

Laboratory No.	Parameter								
	p,p'-DDT	Dieldrin (HEOD)	a-Endo-sulfan	b-Endo-sulfan	Endrin	Heptachlor	Heptachlor Epoxide	p,p'-Methoxy-chlor	Mirex
F02	nd	nd	22	22	nd	nd	6	18	3
F07	4.06	4.18	20.9	20.9	7.32	3.73	4.88	24.0	7.49
F19	43.6	5.30	28.6	29.5	16.25	6.10	7.65	64.4	15.4
F28	4.0	5.0	23	26	8.0	5.0	6.0	22	-
F33	3.48	4.38	22.9	20.3	7.88	3.69	5.27	25.7	8.32
F35	nd	7.5	-	32.8	53.9	5.9	-	59.1	17.3
F38	4.1	3.2	18	12	5.5	5.6	5.7	96	8.4
F40	8.1	6.2	25.0	34.0	12.0	5.0	6.0	30.0	15.0
F43	<30	<20	32	-	<20	<20	<10	<40	20
F47	nd	-	-	-	-	-	nd	31	nd
F58	10	12	24	25	17	6	8	45	21
F59	nd	3.36	14.1	16.8	-	2.55	3.13	17.1	103
F61	4.3	5.3	-	31	31	3.0	3.9	62	10
F63	6.1	5.22	29.8	26.08	8.30	4.8	5.33	26.22	9.87
F64	2	2	12	27	nd	4	2	nd	8
F65	2.7	3.2	14.3	30.0	5.8	1.8	3.3	16.0	3.3
F77	4.9	4.7	20.4	13.5	7.8	4.3	5.1	17.3	78.6
F79	5.4	-	-	-	-	1.5	2.1	10	2.2
F83	24.6	19.4	95.5	89.9	38.6	14.3	17.2	-	-
F85	6.0	5.0	22.0	23.0	8.0	5.7	6.0	23.0	15.0
F86	26	-	-	-	-	-	-	-	-
F93	5.3	8.0	-	16	12	5.1	4.7	33	7.1
Interlab Median	5.30	5.00	22.5	25.5	8.30	4.90	5.30	26.0	9.94
Design Value	4.85	4.67	22.8	25.6	10.36	4.82	5.23	60.3	9.78

nd not detected

1988 FICP INTERLABORATORY QC STUDY
on
ORGANOCHLORINATED INSECTICIDES IN WATER

Table 6. Results for Sample 4 (ampule) pg/uL

Laboratory No.	Parameter								
	Aldrin	a-BHC	b-BHC	g-BHC (lindane)	a-Chlordane (cis)	g-Chlordane (trans)	p,p'-DDD (TDE)	p,p'-DDE	o,p'-DDT
F02	8.5	nd	8.8	8.5	45.7	38.0	7.8	8.5	8.0
F07	8.81	nd	-	9.16	32.0	29.3	-	8.59	39.5
F19	9.87	<0.25	-	10.6	46.6	38.6	5.49	9.87	24.3
F28	10.6	0.49	12.9	11.6	46.3	42.4	-	11.2	5.78
F33	5.64	nd	-	12.4	41.3	40.4	5.07	6.26	6.56
F35	17.9	nd	15.6	12.1	-	32.8	13.6	17.6	-
F38	8.4	nd	-	7.0	38	33	7.6	8.1	8.5
F40	10.0	nd	10.7	10.0	45.5	42.0	11.0	12.0	11.0
F43	10	nd	14	12	58	40	20	10	-
F47	-	nd	6.2	5.7	-	-	5.2	5.6	-
F56	10	nd	10	8	38	24	nd	11	15
F58	10	nd	11	10	53	43	27	11	13
F59	22.7	nd	11.0	10.1	42.8	43.1	-	13.6	-
F61	7.2	nd	9.8	7.2	-	44	8.4	39	61
F63	9.682	nd	9.588	9.88	-	-	13.22	10.55	9.30
F64	10.624	nd	5.097	8.910	44.419	33.207	7.745	7.479	-
F65	10.1	nd	9.0	10.0	54.2	33.8	8.5	10.4	12.5
F77	9.4	nd	8.7	8.9	42.2	36.9	9.4	9.8	7.5
F79	-	1	10	7.3	-	-	6.2	11	-
F83	36.1	7.7	34.0	31.9	-	-	40.6	44.5	14.5
F85	8.5	nd	10.0	8.0	39.0	31.0	12.5	5.0	9.0
F86	-	-	-	-	-	-	-	51	54
F93	0	0	10	10	-	-	-	11	-
Interlab Median	10.0	nd	10.0	9.94	44.4	38.0	8.50	10.55	11.75
Design Value	9.67	9.84	8.82	9.75	49.5	43.3	9.59	10.05	9.70

nd not detected

1988 FICP INTERLABORATORY QC STUDY

on

ORGANOCHLORINATED INSECTICIDES IN WATER

Table 6 cont'd. Results for Sample 4 (ampule) pg/uL

Laboratory No.	Parameter								
	p,p'-DDT	Dieldrin (HEOD)	a-Endo-sulfan	b-Endo-sulfan	Endrin	Heptachlor	Heptachlor Epoxide	p,p'-Methoxy-chlor	Mirex
F02	9.6	8.4	46.3	49.3	14.0	8.2	9.5	42.2	17.4
F07	10.1	8.68	36.3	33.6	12.8	9.52	9.70	41.0	16.8
F19	11.0	9.59	50.0	52.1	23.7	10.6	11.0	59.3	20.8
F28	16.1	11.1	46.9	48.7	16.9	10.0	11.6	45.6	-
F33	6.73	9.47	40.2	41.6	18.3	6.72	11.2	48.8	14.8
F35	7.41	15.6	-	73.8	124.0	11.2	-	33.8	35.4
F38	14	6.8	40	35	13	12	8.0	280	31
F40	8.0	9.0	55.0	55.0	20.0	10.0	12.0	35.0	20.0
F43	14	12	80	-	24	14	12	60	30
F47	5.9	-	-	-	-	-	7.2	28.4	15.3
F56	nd	9	53	56	15	8	7	52	28
F58	13	11	52	46	17	9	11	42	23
F59	15.6	10.6	47.2	80.7	-	9.04	10.9	78.2	264
F61	8.7	9.3	-	65	41	9.6	11	53	22
F63	12.25	11.29	63.57	60.95	17.26	9.89	11.53	50.52	21.5
F64	nd	8.203	32.170	49.265	27.555	7.586	4.716	97.183	16.570
F65	13.3	10.8	24.4	25.0	26.7	10.8	11.7	39.0	10.8
F77	9.7	9.3	44.4	40.8	16.0	9.2	11.1	38.0	210.6
F79	8.5	-	-	-	-	18.6	15.6	62	24
F83	27.0	31.8	181	113	47.0	34.5	34.4	-	-
F85	10.5	9.0	35.0	42.0	13.5	8.0	9.0	47.5	19.5
F86	112	-	-	-	-	-	-	-	-
F93	20	10	-	50	22	12	11	33	20
Interlab Median	11.0	9.53	46.9	49.3	18.3	9.89	11.0	47.5	21.2
Design Value	9.69	9.34	45.6	51.2	20.7	9.64	10.46	120.5	19.6

nd not detected

1988 FICP INTERLABORATORY QC STUDY
on
ORGANOCHLORINATED INSECTICIDES IN WATER

Table 7. Results for Sample 5 (ampule) pg/uL

Laboratory No.	Parameter								
	Aldrin	a-BHC	b-BHC	g-BHC (lindane)	a-Chlordane (cis)	g-Chlordane (trans)	p,p'-DDD (TDE)	p,p'-DDE	o,p'-DDT
F02	20.3	20.4	40.6	21.4	104	86.3	38.3	42.0	39.9
F07	12.6	14.2	-	15.9	64.0	58.5	-	19.4	83.0
F19	20.0	18.2	-	22.1	94.3	77.7	25.2	40.5	74.7
F28	17.8	15.1	38.1	17.2	85.4	90.9	-	39.2	15.7
F33	9.22	21.4	-	21.5	116	102	22.6	32.5	22.3
F35	37.3	29.9	64.8	27.2	-	61.2	75.6	49.8	-
F38	18	18	-	16	79	69	32	33	34
F40	15.0	14.0	30.0	14.0	77.0	65.5	30.0	40.0	45.0
F43	20	25	50	25	110	85	75	40	-
F47	-	19.5	38	19	-	-	35	35	-
F56	21	6	44	18	73	49	30	36	27
F58	29	27	60	29	196	156	94	62	57
F59	20.4	21.5	40.7	18.4	93.3	98.5	-	47.8	-
F61	14	23	38	12	-	59	31	91	130
F63	17.54	18.938	32.83	18.22	-	-	50.82	34.17	28.53
F64	10.384	13.266	12.264	13.223	44.550	48.916	20.717	23.878	-
F65	16.7	15.5	6.8	17.3	78.2	137.0	33.5	27.6	78.0
F77	18.8	25.7	38.2	19.3	86.7	73.0	36.2	37.8	28.2
F79	-	21	36	20	-	-	38	65	-
F83	31.8	28.9	47.7	27.6	-	-	59.5	66.6	20.4
F85	15.5	15.5	30.0	15.0	71.0	57.0	37.5	15.5	33.5
F86	-	-	-	-	-	-	-	127	157
F93	20	20	40	20	-	-	-	42	-
Interlab Median	18.4	19.8	38.2	18.7	85.4	73.0	35.6	40.0	37.0
Design Value	19.3	19.7	35.3	19.5	99.1	86.6	38.4	40.2	38.8

nd not detected
- not analyzed

1988 FICP INTERLABORATORY QC STUDY

on

ORGANOCHLORINATED INSECTICIDES IN WATER

Table 7 cont'd. Results for Sample 5 (ampule) pg/uL

Laboratory No.	Parameter								
	p,p'-DDT	Dieldrin (HEOD)	a-Endo-sulfan	b-Endo-sulfan	Endrin	Heptachlor	Heptachlor Epoxide	p,p'-Methoxy-chlor	Mirex
F02	45.6	38.6	210	234	36.0	20.4	43.5	214	38.6
F07	24.2	21.5	182	171	16.8	13.3	31.7	104	23.1
F19	85.9	36.0	192	196	48.3	23.3	51.7	230	38.0
F28	49.1	36.4	205.9	190.7	30.6	17.8	42.8	179.2	-
F33	23.8	39.1	303	323	36.5	15.6	49.2	204	47.1
F35	61.9	60.3	-	245.0	231.0	28.4	-	346.0	66.7
F38	36	30	180	180	25	34	33	660	37
F40	32.0	30.0	165.0	190.0	35.0	14.0	33.0	125.0	30.0
F43	50	45	280	-	45	25	55	240	57
F47	38	-	-	-	-	-	45	192	41
F56	57	39	228	247	16	16	16	188	53
F58	57	62	435	231	52	27	65	349	66
F59	55.1	37.1	213	248	-	17.9	41.7	218	596
F61	26	34	-	240	63	18	41	190	39
F63	38.05	36.65	224.4	213.7	30.83	18.19	36.06	170.03	37.18
F64	4.433	25.909	87.832	140.658	42.743	nd	14.176	153.709	23.540
F65	25.6	30.0	103.0	120.0	18.6	16.5	36.4	153.3	22.4
F77	37.4	35.0	170.1	171.1	32.6	19.5	41.0	161.8	410.3
F79	50	-	-	-	-	5.0	84	440	66
F83	38.9	55.5	378	322	56.8	33.0	62.7	-	-
F85	34.5	35.0	116.5	140.0	27.0	15.5	14.5	144.0	33.5
F86	245	-	-	-	-	-	-	-	-
F93	60	38	-	200	38	24	44	180	33
Interlab Median	38.9	36.5	205.9	200.0	36.0	18.1	41.7	190.0	38.8
Design Value	38.8	37.4	182.5	204.8	41.4	19.3	41.8	482.1	39.1

nd not detected

not analyzed

1988 FICP INTERLABORATORY QC STUDY

on

ORGANOCHLORINATED INSECTICIDES IN WATER

Table 8. Results for Sample 6 (ampule) pg/uL

Laboratory No.	Parameter								
	Aldrin	a-BHC	b-BHC	g-BHC (lindane)	a-Chlordane (cis)	g-Chlordane (trans)	p,p'-DDD (TDE)	p,p'-DDE	o,p'-DDT
F02	1.06	0.41	0.51	0.65	0.95	0.90	1.79	1.86	2.58
F07	0.863	0.504	-	0.740	1.03	1.27	-	2.47	9.00
F19	1.09	0.54	-	0.72	1.11	1.03	2.50	1.18	<2
F28	1.20	0.50	1.12	0.83	1.60	1.70	-	2.30	3.56
F33	0.81	0.40	-	0.57	0.98	0.95	1.73	1.74	3.05
F35	2.96	0.66	nd	1.56	-	1.05	4.37	nd	-
F38	nd	nd	-	nd	nd	nd	1.7	1.5	2.4
F40	1.0	0.5	0.8	0.6	1.0	1.0	3.0	2.1	3.0
F43	1.3	0.54	0.74	0.78	1.2	1.1	5.4	2.2	-
F47	-	nd	nd	0.3	-	-	1.3	1.1	-
F56	1.5	nd	nd	trace	1.1	0.7	6.7	3.0	16.0
F58	1	1	2	1	3	3	13	3	4
F59	0.95	0.40	nd	0.52	0.86	1.04	-	2.53	-
F61	0.78	4.3	0.89	0.66	-	7.3	2.3	62	81
F63	0.948	0.419	0.429	0.79	-	-	2.63	2.16	2.93
F64	0.905	0.295	nd	0.445	0.982	0.795	0.578	1.158	-
F65	1.1	nd	nd	nd	1.5	2.3	nd	nd	nd
F77	1.2	0.5	nd	1.0	nd	1.1	2.6	2.3	2.2
F79	-	0.35	0.48	0.50	-	-	1.9	5.4	-
F83	5.6	5.2	8.2	4.6	-	-	8.8	10.6	6.1
F85	1.0	0.5	0.75	0.7	1.1	1.0	1.9	2.3	2.6
F86	-	-	-	-	-	-	-	6	16
F93	0.7	0.6	0.7	0.6	-	-	-	1.7	-
Interlab Median	1.00	0.50	0.75	0.68	1.10	1.045	2.50	2.30	3.05
Design Value	0.967	0.394	0.441	0.585	0.991	0.866	1.92	2.01	2.91

nd not detected

\- not analyzed

1988 FICP INTERLABORATORY QC STUDY

on

ORGANOCHLORINATED INSECTICIDES IN WATER

Table 8 cont'd. Results for Sample 6 (ampule) pg/uL

Laboratory No.	Parameter								
	p,p'-DDT	Dieldrin (HEOD)	a-Endo-sulfan	b-Endo-sulfan	Endrin	Heptachlor	Heptachlor Epoxide	p,p'-Methoxy-chlor	Mirex
F02	4.77	4.31	0.98	1.07	3.76	2.57	3.05	4.21	8.30
F07	4.47	4.21	1.04	1.09	10.7	3.57	3.18	4.74	8.49
F19	<2	5.50	1.36	1.93	5.43	2.97	3.36	<25	10.9
F28	6.27	5.11	2.42	5.98	4.21	2.96	3.64	12.4	-
F33	4.97	4.00	1.00	1.03	3.60	2.80	2.97	3.39	9.82
F35	10.40	3.95	-	4.69	35.50	4.74	-	nd	19.20
F38	3.4	3.6	nd	nd	3.0	nd	2.4	84	3.6
F40	3.0	4.5	1.0	1.6	9.5	2.8	3.5	3.7	10.0
F43	5.4	5.1	1.7	-	4.8	3.3	3.6	6.2	10
F47	2.5	-	-	-	-	-	1.9	3.2	7.4
F56	nd	4.7	4.1	nd	12.2	2.9	4.9	23.6	nd
F58	6	6	4	8	5	3	5	10	12
F59	6.86	5.03	1.00	1.37	-	2.43	3.19	6.09	96.8
F61	4.3	3.9	-	3.2	41	3.1	3.6	5.0	12
F63	5.44	3.65	1.97	3.00	3.60	2.41	2.79	2.39	9.10
F64	nd	2.718	1.035	nd	5.965	1.845	1.470	28.569	9.828
F65	nd	3.3	1.7	1.9	nd	3.3	3.4	nd	5.0
F77	5.1	5.0	nd	nd	4.4	3.1	3.7	4.9	54.4
F79	4.7	-	-	-	-	6.0	5.0	7.4	13.0
F83	14.7	17.3	88.0	7.4	19.3	11.6	11.9	-	-
F85	5.0	4.4	1.4	1.9	3.6	2.7	3.0	5.4	10.9
F86	10	-	-	-	-	-	-	-	-
F93	4.7	5.0	-	2	4.2	3.1	3.2	4.0	8.1
Interlab Median	4.99	4.45	1.40	1.93	4.90	2.99	3.36	5.20	10.00
Design Value	4.85	4.67	0.913	1.024	5.18	2.89	3.14	12.05	9.78

nd not detected

1988 FICP Interlaboratory QC Study on Organochlorinated Insecticides in Water

APPENDIX

The following results were received from another participant after the data summary was prepared and released for distribution. These data are reproduced below for information only as they are not included and evaluated in this report.

March 1, 1989

Results for Laboratory F066

Parameter	Water Samples (ng/L)			Ampules (pg/uL)		
	1	2	3	4	5	6
Aldrin	209	36.7	10.8	9.41	16.6	0.78*
a-BHC	165	8.7	<3	<1	19.1	0.35*
b-BHC	343	68.4	16.0	11.8	45.0	<2
g-BHC	172	12.4	13.9	9.03	18.2	0.56*
a-Chlordane	1056	123	59.1	45.9	86.2	0.96*
g-Chlordane	872	125	50.9	39	74.0	0.95*
p,p'-DDD	369	52.3	9.9	8.34	31.9	1.77*
p,p'-DDE	370	103	11.7	9.5	35.5	1.95*
o,p'-DDT	366	71	11.5	8.85	34.7	2.69*
p,p'-DDT	364	59.9	12.6	13.5	40.3	5.08*
Dieldrin	352	58	16.6	9.19	35.1	4.48*
a-Endosulfan	1963	365	54.3	45	178	1.02*
b-Endosulfan	2264	403	40.1	51.9	206	1.06*
Endrin	328	58	16.6	16.8	30.5	4.05*
Heptachlor	167	33.6	11.2	8.43	17.0	2.61*
Hept. Epoxide	274	50.7	9.8	6.93	26.3	2.13*
Methoxychlor	1750	374	44.0	44.1	176	5.26*
Mirex	350	62.4	22.6	20	38.8	9.43*

* Concentrations below which this laboratory would not normally quantify.