



Report No. 810

**SOILS (CHEMICAL)
(ROUND 7)**

PROFICIENCY TESTING PROGRAM

June 2013

ACKNOWLEDGMENTS

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1. **FOREWORD**

This report summarises the results of the seventh round of a proficiency testing program for the chemical analysis of soil samples.

Proficiency Testing Australia (PTA) conducted the program in April 2013. The Program Coordinator was Dr M Li. The Technical Adviser was Mr C Wood from Environmental Resource Associates (ERA), USA. This report was authorised by Ms W Fajloun, PTA Quality Coordinator. The aim of the program was to assess laboratories' ability to competently perform the prescribed analyses.

2. **STATISTICAL DESIGN OF THE PROGRAM**

For each statistically analysed test, robust statistical procedures were used to generate the z-scores and summary statistics for each test - number of results, median, uncertainty of the median, normalised interquartile range (IQR), robust coefficient of variation (CV), minimum, maximum and range.

3. **FEATURES OF THE PROGRAM**

- (a) A total of nine laboratories received samples, and all laboratories returned results for inclusion in the final report.
- (b) Participating laboratories were each supplied with one flame-sealed ampoule, each containing 30.0 ± 0.2 grams of soil.
- (c) The following determinations were to be performed on the samples:
 - Aldrin
 - alpha-BHC
 - beta-BHC
 - delta-BHC
 - gamma-BHC (Lindane)
 - alpha-Chlordane
 - gamma-Chlordane
 - 4,4'-DDD
 - 4,4'-DDE
 - 4,4'-DDT
 - Dieldrin
 - Endrin
 - Endrin aldehyde
 - Endrin ketone
 - Endosulfan I
 - Endosulfan II
 - Endosulfan sulphate

- Heptachlor
 - Heptachlor epoxide
 - Methoxychlor
- (d) Homogeneity and stability were analysed for randomly selected samples. Based on this testing, it was concluded that the samples were sufficiently homogeneous and stable. Therefore any results identified as outliers could not be attributed to sample variability (Appendix B).
- (e) Participating laboratories were requested to perform their tests according to the "Instructions to Participants" and to record their results on the accompanying "Results Sheets". They were distributed to participants with the samples (Appendix C).
- (f) Each laboratory was randomly allocated a unique code number for the program to enable confidentiality of results. Reference to each laboratory in this report is made by its code number.

4. **FORMAT OF APPENDICES**

Appendix A

For each test, where appropriate, the following information is given:

- (i) The results reported by laboratories, and calculated z-scores.
- (ii) A table of robust statistics - number of results, median, uncertainty of the median, normalised interquartile range (IQR), robust coefficient of variation (CV), minimum, maximum and range.
- (iii) Z-score charts.

Appendix B

- (i) Homogeneity Testing and Stability Testing.

Appendix C

- (i) "Instructions to Participants".
- (ii) "Results Sheets".

5. SUMMARY OF RESULTS

TABLE A: SUMMARY OF RESULTS

Test	No. of Results	Median (µg/kg)	Normalised IQR
Aldrin	9	57.70	10.31
alpha-BHC	8	155.8	18.7
beta-BHC	8	n/a	n/a
delta-BHC	8	90.4	24.6
gamma-BHC (Lindane)	9	n/a	n/a
alpha-Chlordane	9	48.30	9.06
gamma-Chlordane	7	n/a	n/a
4,4'-DDD	9	259.0	28.0
4,4'-DDE	9	251.0	30.1
4,4'-DDT	9	76.90	12.33
Dieldrin	9	148.0	22.0
Endrin	9	129.0	17.3
Endrin aldehyde	5	n/a	n/a
Endrin ketone	5	n/a	n/a
Endosulfan I	2	n/a	n/a
Endosulfan II	2	n/a	n/a
Endosulfan sulphate	9	92.70	18.97
Heptachlor	9	240.0	49.0
Heptachlor epoxide	9	130.0	14.0
Methoxychlor	8	166.0	38.3

NOTE: Statistical analysis was not applied for beta-BHC, gamma-BHC (Lindane), gamma-Chlordane, Endrin aldehyde, Endrin ketone, Endosulfan I and Endosulfan II.

6. STATISTICAL OUTLIER RESULTS

In order to achieve the program's aim of assessing laboratories' testing performance, a robust statistical approach, which uses z-scores has been utilised. The z-score is a measure of how far the result(s) is from the consensus value - a normalised value which gives a "score" to each result relative to the other results in the group. Therefore a z-score close to zero means that the result agrees well with those from other laboratories. An outlier will be any result(s) which has an absolute z-score value greater than or equal to 3.0.

For each laboratory, a single robust z-score was calculated. A single robust z-score (denoted by Z) for a laboratory's sample A would then be:

$$Z = \frac{A - \text{Median (A)}}{\text{NormIQR (A)}}$$

For further information on the calculation and interpretation of z-scores, please see the *Guide to Proficiency Testing Australia, 2012* (reference [1]).

7. **PTA COMMENTS**

(i) Metrological Traceability and Measurement Uncertainty (MU) of Assigned Values

Consensus values (median) derived from the participants' results are used in this program. These values are not metrologically traceable to an external reference.

As the assigned value for this program is the median of the results submitted by the participants, the uncertainty of the median has been calculated and is presented below.

(ii) Analysis of Results by Method Groups

In order for methods to be grouped for analysis, PTA requires at least 11 sets of results from the same method group. As there were less than 11 results submitted for each method, reliable conclusions cannot be drawn from analysing grouped methods on this occasion. Therefore, results from all method groups have been pooled for analysis.

(iii) Chemical Testing

(a) Aldrin

A robust CV of 17.9% was obtained. No result was identified as an outlier. The uncertainty of the median is $\pm 4.31 \mu\text{g}/\text{kg}$.

(b) alpha-BHC

A target CV of 12.0% was used. Outlier results were identified for laboratories 1 and 6. The uncertainty of the median is $\pm 8.3 \mu\text{g}/\text{kg}$.

(c) beta-BHC

Statistical analysis was not applied as the results are not normally distributed.

(d) gamma-BHC (Lindane)

Statistical analysis was not applied as the results are not normally distributed.

(e) gamma-Chlordane

Statistical analysis was not applied as the results are not normally distributed.

(f) *delta-BHC*

A robust CV of 27.2% was obtained. No result was identified as an outlier. The uncertainty of the median is $\pm 10.9 \mu\text{g/kg}$.

(g) *alpha-Chlordane*

A robust CV of 18.7% was obtained. No result was identified as an outlier. The uncertainty of the median is $\pm 3.78 \mu\text{g/kg}$.

(h) *4,4'-DDD*

A robust CV of 10.8% was obtained. One result was identified as an outlier for laboratory 1. The uncertainty of the median is $\pm 11.7 \mu\text{g/kg}$.

(i) *4,4'-DDE*

A target CV of 12.0% was used. One result was identified as an outlier for laboratory 1. The uncertainty of the median is $\pm 12.6 \mu\text{g/kg}$.

(j) *4,4'-DDT*

A robust CV of 16.0% was obtained. One result was identified as an outlier for laboratory 4. The uncertainty of the median is $\pm 5.15 \mu\text{g/kg}$.

(k) *Dieldrin*

A robust CV of 14.9% was obtained. No result was identified as an outlier. The uncertainty of the median is $\pm 9.2 \mu\text{g/kg}$.

(l) *Endrin*

A robust CV of 13.4% was obtained. No result was identified as an outlier. The uncertainty of the median is $\pm 7.2 \mu\text{g/kg}$.

(m) *Endrin aldehyde*

Statistical analysis was not applied as there were only five results.

(n) *Endrin ketone*

Statistical analysis was not applied as there were only five results.

(o) *Endosulfan I*

Statistical analysis was not applied as there were insufficient numerical results.

(p) *Endosulfan II*

Statistical analysis was not applied as there were insufficient numerical results.

(q) *Endosulfan sulphate*

A robust CV of 20.5% was obtained. One result was identified as an outlier for laboratory 3. The uncertainty of the median is $\pm 7.92 \mu\text{g}/\text{kg}$.

(r) *Heptachlor*

A robust CV of 20.4% was obtained. No result was identified as an outlier. The uncertainty of the median is $\pm 20.5 \mu\text{g}/\text{kg}$.

(s) *Heptachlor epoxide*

A robust CV of 10.8% was obtained. No result was identified as an outlier. The uncertainty of the median is $\pm 5.8 \mu\text{g}/\text{kg}$.

(t) *Methoxychlor*

A robust CV of 23.1% was obtained. No result was identified as an outlier. The uncertainty of the median is $\pm 17.0 \mu\text{g}/\text{kg}$.

(iv) Overall Performance

The overall performance of the participants is within expectations.

Four laboratories have outliers as shown in Table B. The cause of the outliers should be investigated by the participating laboratories. Potential causes for the outliers could be interferences from the matrix, instrument conditions, transcription and calculation errors, sample preparation errors and human errors.

MUs do not account for the low recoveries of these analytes. Based on the tight accuracy of much of the data reported, the MUs appear to be more than adequate. Most reported MUs are in the range of 10-30%.

In general, the precision of this study is consistent with historically based expectations. Most results were considered satisfactory with a z-score of less than 2.0.

TABLE B: STATISTICAL OUTLIER RESULTS (By Laboratory Code)

Test	Laboratory Codes
alpha-BHC	1 , 6
4,4'-DDD	1
4,4'-DDE	1
4,4'-DDT	4
Endosulfan sulphate	3

This study provides a valid assessment of laboratory performance for the analysis of organochlorine pesticides in soil. Those results with an absolute z-score of 3.0 or greater should be treated as non-conforming results and should be subject to root cause analysis and corrective action. At the conclusion of the corrective action process, it is suggested that a remedial proficiency testing sample should be analysed to ensure the effectiveness of the corrective action. Any values with z-scores between 2.0 and 3.0 should be reviewed closely to determine if they are indicative of potential analytical problems. Laboratories should also look at all of their z-scores and conduct further investigation if their scores were consistently toward the high or low end of all of the reported data.

On the whole, the study should provide valuable information to the participants on the performance of the methods and equipment used.

8. **REFERENCE**

[1] *Guide to Proficiency Testing Australia, 2012.*

This document can be found on the PTA website at www.pta.asn.au.

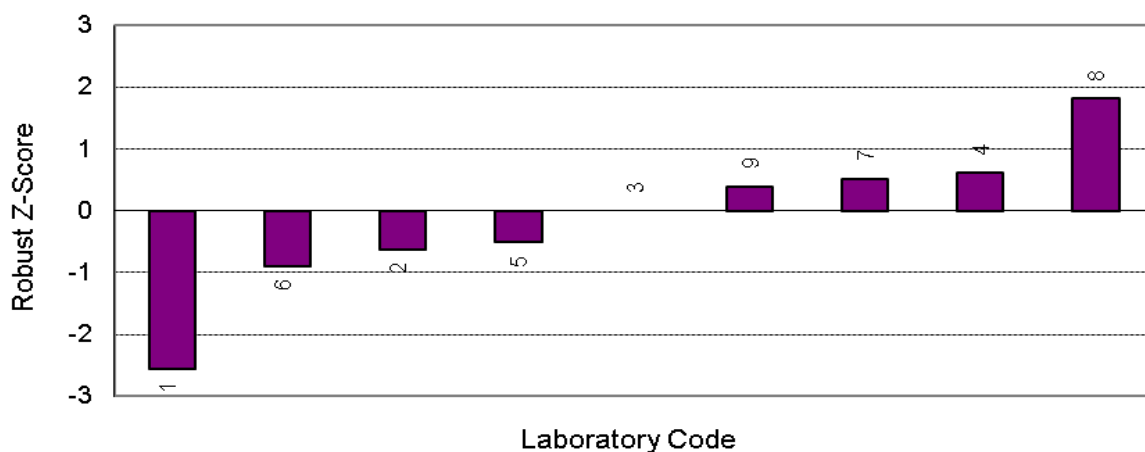
APPENDIX A

Summary of Results and Z-Score Charts

Appendix	A1	Aldrin
	A2	alpha-BHC
	A3	beta-BHC
	A3	gamma-BHC (Lindane)
	A3	gamma-Chlordane
	A4	delta-BHC
	A5	alpha-Chlordane
	A6	4,4'-DDD
	A7	4,4'-DDE
	A8	4,4'-DDT
	A9	Dieldrin
	A10	Endrin
	A11	Endrin aldehyde
	A11	Endrin ketone
	A12	Endosulfan I
	A12	Endosulfan II
	A13	Endosulfan sulphate
	A14	Heptachlor
	A15	Heptachlor epoxide
	A16	Methoxychlor

Aldrin ($\mu\text{g}/\text{kg}$)				
Lab Code	Result	MU	Robust Z-Score	Method
1	31.4	9.3	-2.55	SM6630/US EPA 8081A
2	51.2	15.4	-0.63	GC-ECD
3	57.7	10.4	0.00	GC-ECD
4	64.03	16.01	0.61	USEPA 8081
5	52.5	29	-0.50	FSO-02
6	48.4	15	-0.90	Pre-w et with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	63.0	25.0	0.51	GC-ECD
8	76.4	15.3	1.81	USEPA3550C,8081B
9	61.7	30.9	0.39	

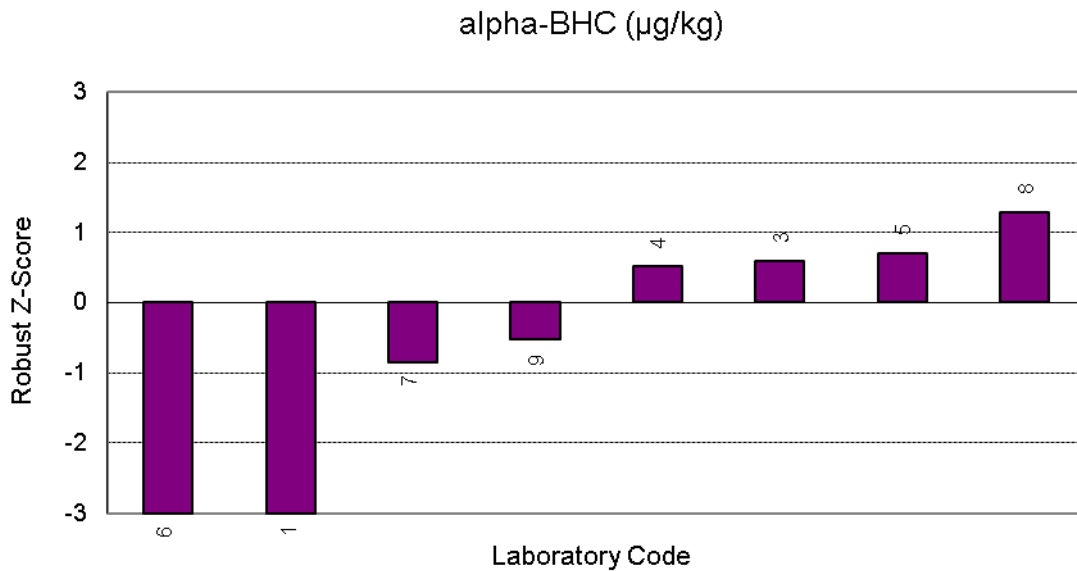
No. of results	9
Median	57.70
Normalised IQR	10.31
Robust CV	17.9%
Minimum	31.4
Maximum	76.4
Range	45.0
Uncertainty (Median)	4.31

Aldrin ($\mu\text{g}/\text{kg}$)

alpha-BHC (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	97.8	8.2	-3.10 §	SM6630/US EPA 8081A GC-ECD USEPA 8081 FSO-02 Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis. GC-ECD USEPA3550C,8081B
3	167	30.1	0.60	
4	165.6	41.41	0.52	
5	169	63	0.71	
6	89.7	27	-3.54 §	
7	140	56	-0.85	
8	180	36	1.29	
9	146	72.9	-0.52	

No. of results 8
 Median 155.8
 Normalised IQR 18.7
 Target CV 12.0%
 Minimum 89.7
 Maximum 180
 Range 90.3
 Uncertainty (Median) 8.3

NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).



beta-BHC ($\mu\text{g}/\text{kg}$)			
Lab Code	Result	MU	Method
1	84.3	8.0	SM6630/US EPA 8081A
3	145	26.1	GC-ECD
4	139.7	34.93	USEPA 8081
5	117	46	FSO-02
6	82.9	32	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	101	40	GC-ECD
8	127	25	USEPA3550C,8081B
9	126	63.2	

No. of results 8

NOTE: Statistical analysis was not applied as the results are not normally distributed.

gamma-BHC (Lindane) ($\mu\text{g}/\text{kg}$)			
Lab Code	Result	MU	Method
1	138	16.5	SM6630/US EPA 8081A
2	210	63	GC-ECD
3	227	40.8	GC-ECD
4	221.8	55.44	USEPA 8081
5	211	44	FSO-02
6	146.9	42	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	188	75	GC-ECD
8	274	55	USEPA3550C,8081B
9	213	108	

No. of results 9

NOTE: Statistical analysis was not applied as the results are not normally distributed.

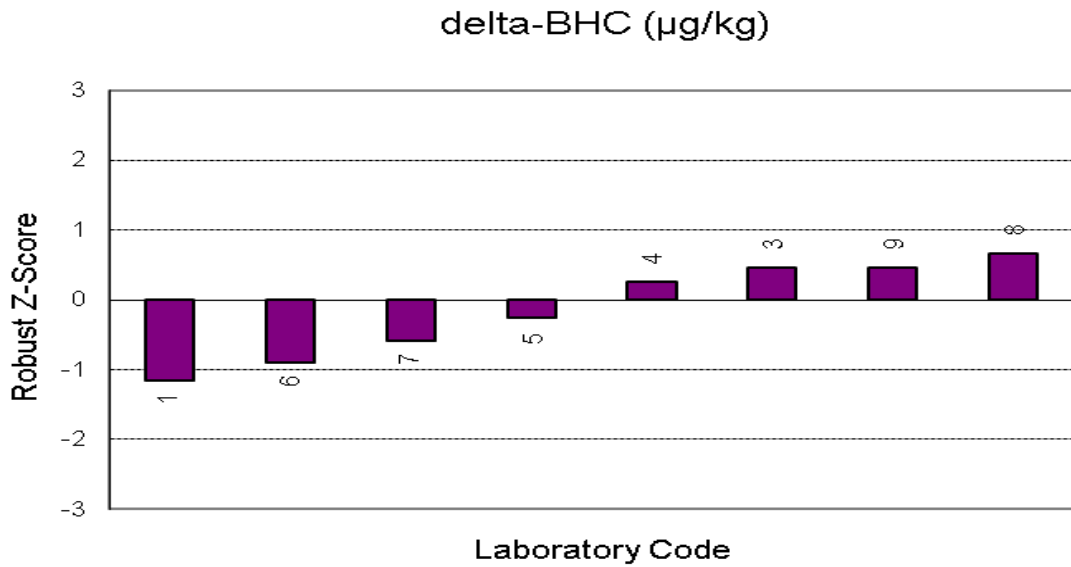
gamma-Chlordane ($\mu\text{g}/\text{kg}$)			
Lab Code	Result	MU	Method
1	181	15.6	SM6630/US EPA 8081A
3	258	46.4	GC-ECD
5	256	121	FSO-02
6	210.4	64	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	210	84	GC-ECD
8	277	55	USEPA3550C,8081B
9	275	137	

No. of results 7

NOTE: Statistical analysis was not applied as the results are not normally distributed.

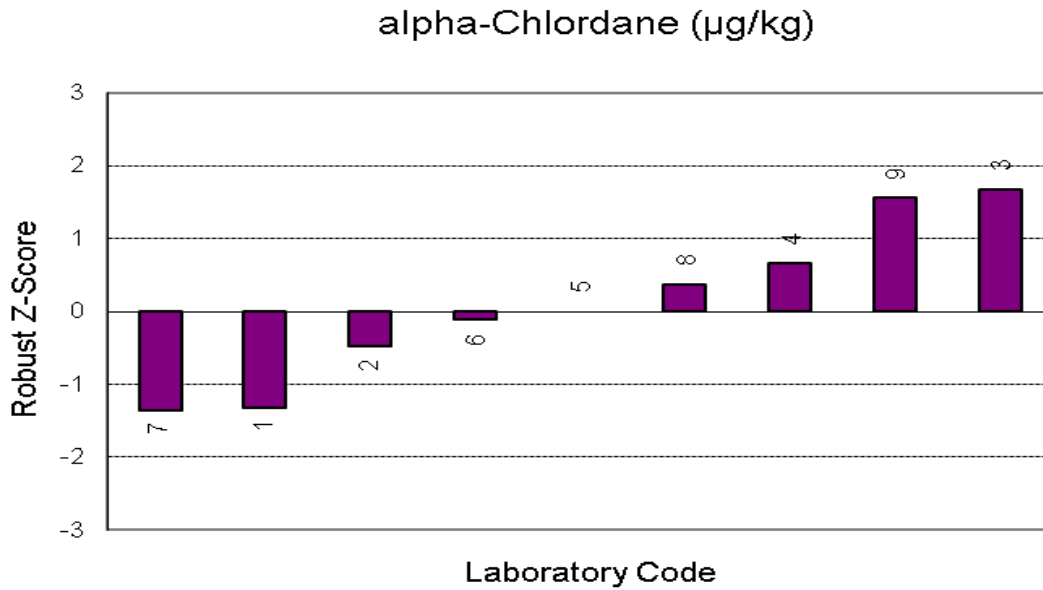
delta-BHC (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	61.9	25.3	-1.16	SM6630/US EPA 8081A GC-ECD USEPA 8081 FSO-02 Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis. GC-ECD USEPA3550C,8081B
3	102	18.4	0.47	
4	96.79	24.20	0.26	
5	84.1	31	-0.26	
6	68.1	24	-0.91	
7	76.0	30	-0.59	
8	107	21	0.67	
9	102	51.2	0.47	

No. of results 8
 Median 90.4
 Normalised IQR 24.6
 Robust CV 27.2%
 Minimum 61.9
 Maximum 107
 Range 45.1
 Uncertainty (Median) 10.9



alpha-Chlordane (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	36.4	10.0	-1.31	SM6630/US EPA 8081A
2	44.0	13.2	-0.47	GC-ECD
3	63.5	11.43	1.68	GC-ECD
4	54.36	13.59	0.67	USEPA 8081
5	48.3	24	0.00	FSO-02
6	47.4	16	-0.10	Pre-w et w ith Phosphoric acid, Sonication w ith Hexane / Acetone; GC-ECD analysis.
7	36.0	14	-1.36	GC-ECD
8	51.7	10.3	0.38	USEPA3550C,8081B
9	62.4	31.2	1.56	

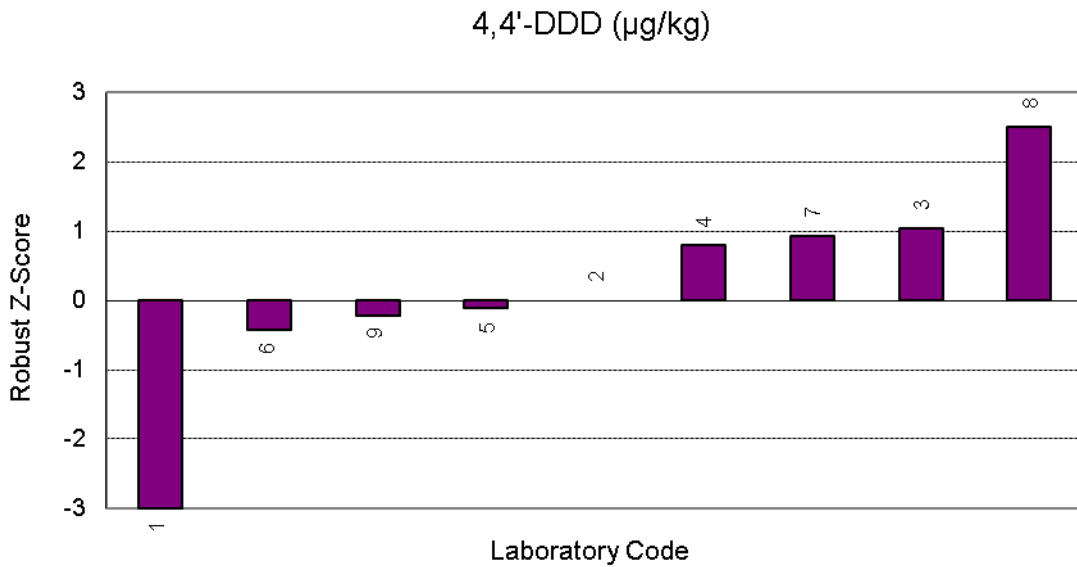
No. of results 9
 Median 48.30
 Normalised IQR 9.06
 Robust CV 18.7%
 Minimum 36.0
 Maximum 63.5
 Range 27.5
 Uncertainty (Median) 3.78



4,4'-DDD (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	171	13.3	-3.15 §	SM6630/US EPA 8081A
2	259	77.7	0.00	GC-ECD
3	288	51.8	1.04	GC-ECD
4	281.2	70.29	0.79	USEPA 8081
5	256	169	-0.11	FSO-02
6	247.2	120	-0.42	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	285	112	0.93	GC-ECD
8	329	66	2.50	USEPA3550C,8081B
9	253	126	-0.21	

No. of results 9
 Median 259.0
 Normalised IQR 28.0
 Robust CV 10.8%
 Minimum 171
 Maximum 329
 Range 158
 Uncertainty (Median) 11.7

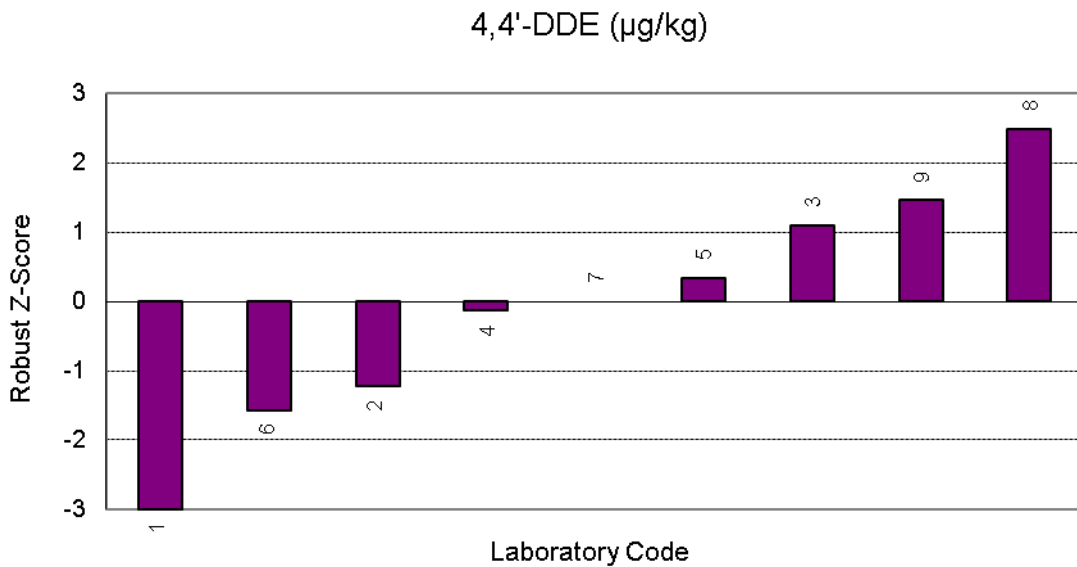
NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).



4,4'-DDE (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	153	14.8	-3.25 §	SM6630/US EPA 8081A
2	214	64.2	-1.23	GC-ECD
3	284	51.1	1.10	GC-ECD
4	247.3	61.83	-0.12	USEPA 8081
5	261	164	0.33	FSO-02
6	203.4	110	-1.58	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	251	100	0.00	GC-ECD
8	326	65	2.49	USEPA.3550C,8081B
9	295	148	1.46	

No. of results 9
 Median 251.0
 Normalised IQR 30.1
 Target CV 12.0%
 Minimum 153
 Maximum 326
 Range 173
 Uncertainty (Median) 12.6

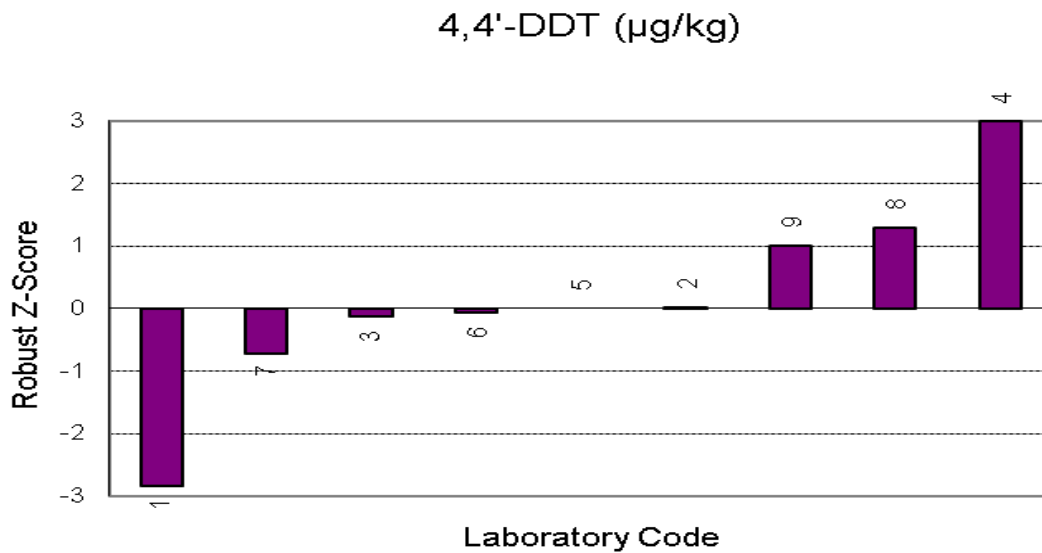
NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).



4,4'-DDT (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	41.9	25.6	-2.84	SM6630/US EPA 8081A
2	77.1	23.1	0.02	GC-ECD
3	75.2	13.5	-0.14	GC-ECD
4	123.9	30.97	3.81	USEPA 8081
5	76.9	43	0.00	FSO-02
6	76.1	45	-0.06	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	68.1	27	-0.71	GC-ECD
8	92.8	18.6	1.29	USEPA3550C,8081B
9	89.3	44.6	1.01	

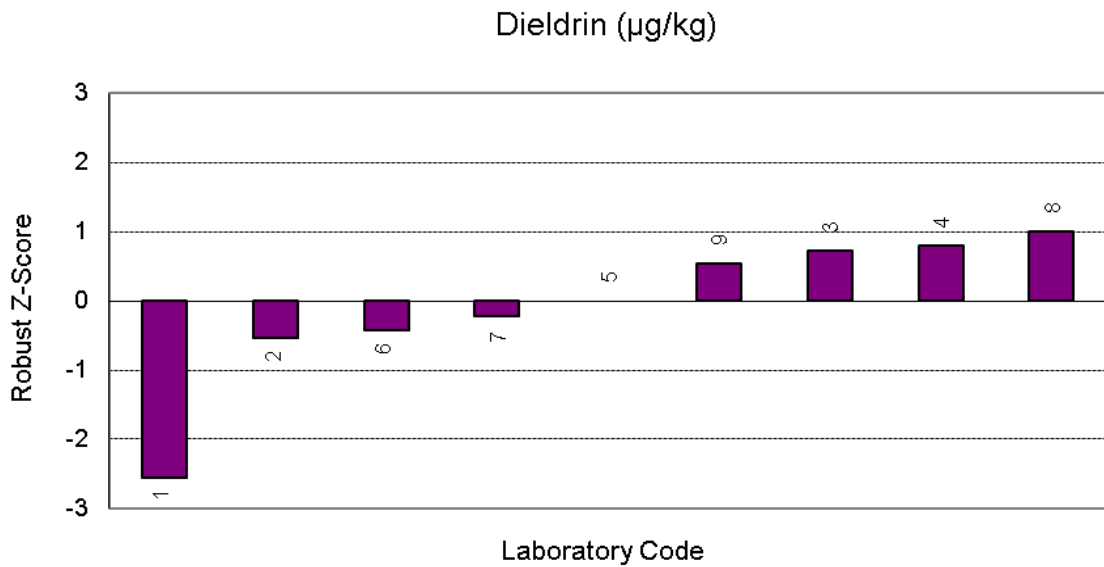
No. of results 9
 Median 76.90
 Normalised IQR 12.33
 Robust CV 16.0%
 Minimum 41.9
 Maximum 123.9
 Range 82.0
 Uncertainty (Median) 5.15

NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).



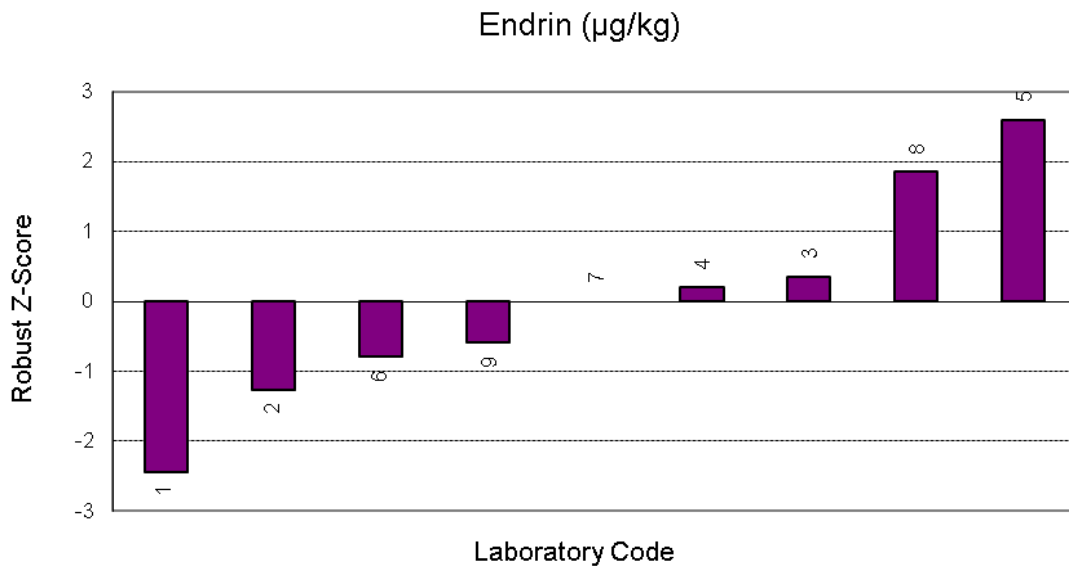
Dieldrin (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	91.6	13.2	-2.56	SM6630/US EPA 8081A
2	136	40.8	-0.54	GC-ECD
3	164	29.5	0.73	GC-ECD
4	165.5	41.38	0.79	USEPA 8081
5	148	67	0.00	FSO-02
6	138.8	62	-0.42	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	143	56	-0.23	GC-ECD
8	170	34	1.00	USEPA3550C,8081B
9	160	80.1	0.54	

No. of results 9
 Median 148.0
 Normalised IQR 22.0
 Robust CV 14.9%
 Minimum 91.6
 Maximum 170
 Range 78.4
 Uncertainty (Median) 9.2



Endrin (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	86.7	25.8	-2.44	SM6630/US EPA 8081A
2	107	32.1	-1.27	GC-ECD
3	135	24.3	0.35	GC-ECD
4	132.5	33.13	0.20	USEPA 8081
5	174	109	2.60	FSO-02
6	115.2	77	-0.80	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	129	48	0.00	GC-ECD
8	161	32	1.85	USEPA3550C,8081B
9	119	59.7	-0.58	

No. of results 9
 Median 129.0
 Normalised IQR 17.3
 Robust CV 13.4%
 Minimum 86.7
 Maximum 174
 Range 87.3
 Uncertainty (Median) 7.2



Endrin aldehyde ($\mu\text{g}/\text{kg}$)			
Lab Code	Result	MU	Method
3	153	27.5	GC-ECD
4	167.4	41.85	USEPA 8081
6	126.7	71	Pre-w et with Phosphoric acid, Sonication w ith Hexane / Acetone; GC-ECD analysis.
7	<10		
8	176	35	USEPA3550C,8081B

No. of results 5

NOTE: Statistical analysis was not applied as there are only five results.

Endrin ketone ($\mu\text{g}/\text{kg}$)			
Lab Code	Result	MU	Method
3	149	26.8	GC-ECD
4	171.0	42.74	USEPA 8081
6	135.4	66	Pre-w et w ith Phosphoric acid, Sonication w ith Hexane / Acetone; GC-ECD analysis.
7	109	44	0
8	165	33	USEPA3550C,8081B

No. of results 5

NOTE: Statistical analysis was not applied as there are only five results.

Endosulfan I ($\mu\text{g}/\text{kg}$)			
Lab Code	Result	MU	Method
1	<10		SM6630/US EPA 8081A
2	<10		GC-ECD
3	<50		
4	<50	12.50	USEPA 8081
5	<50		FSO-02
6	5.35	2	Pre-w et w ith Phosphoric acid, Sonication w ith Hexane / Acetone; GC-ECD analysis.
7	<10		
8	<50.0	10.0	USEPA3550C,8081B
9	7.84	3.92	

No. of results 2

NOTE: Statistical analysis was not applied as there were insufficient numerical results.

Endosulfan II ($\mu\text{g}/\text{kg}$)			
Lab Code	Result	MU	Method
1	<10		SM6630/US EPA 8081A
2	<10		GC-ECD
3	<50		
4	<50	12.5	USEPA 8081
5	<100		FSO-02
6	9.69	4.3	Pre-w et w ith Phosphoric acid, Sonication w ith Hexane / Acetone; GC-ECD analysis.
7	<10		
8	<50.0	10.0	USEPA3550C,8081B
9	9.84	4.92	

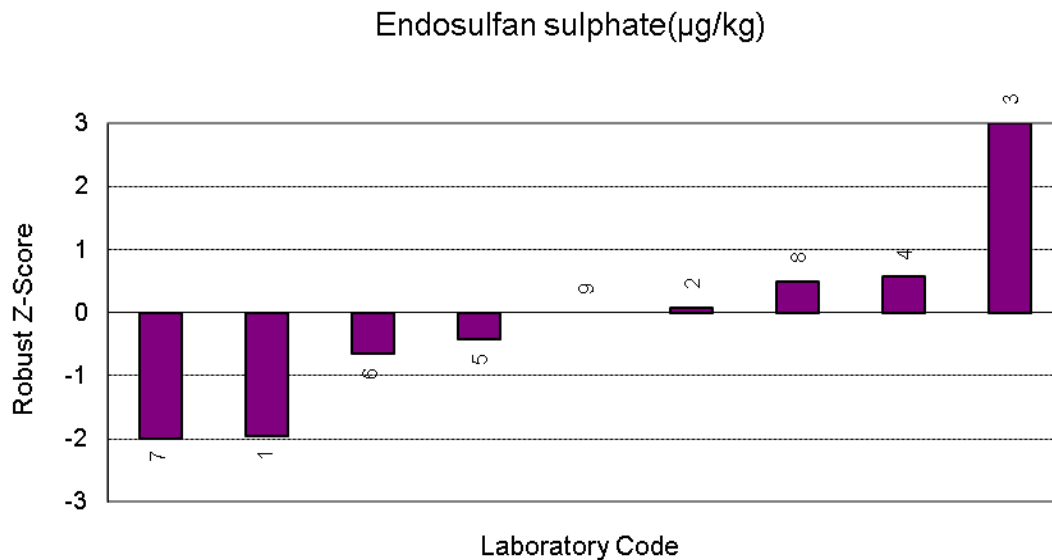
No. of results 2

NOTE: Statistical analysis was not applied as there were insufficient numerical results.

Endosulfan sulphate (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	55.6	18.1	-1.96	SM6630/US EPA 8081A
2	94.1	28.2	0.07	GC-ECD
3	184	33.1	4.81 §	GC-ECD
4	103.5	25.87	0.57	USEPA 8081
5	84.9	62	-0.41	FSO-02
6	80.3	50	-0.65	Pre-wet w ith Phosphoric acid, Sonication w ith Hexane / Acetone; GC-ECD analysis.
7	55.0	21	-1.99	
8	102	20	0.49	USEPA3550C,8081B
9	92.7	46.4	0.00	

No. of results 9
 Median 92.70
 Normalised IQR 18.97
 Robust CV 20.5%
 Minimum 55.0
 Maximum 184
 Range 129.0
 Uncertainty (Median) 7.92

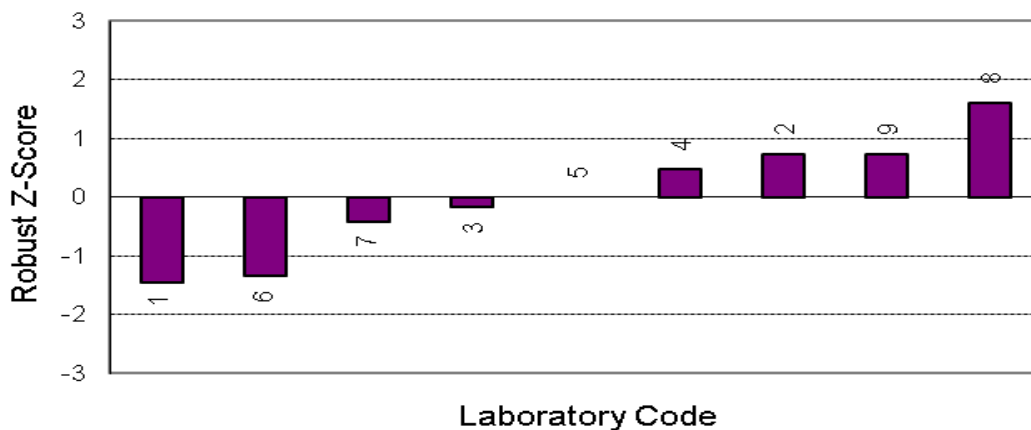
NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).



Heptachlor (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	169	12.4	-1.45	SM6630/US EPA 8081A
2	276	82.8	0.74	GC-ECD
3	232	41.7	-0.16	GC-ECD
4	263.4	65.86	0.48	USEPA 8081
5	240	91	0.00	FSO-02
6	174.6	60	-1.34	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	220	88	-0.41	
8	319	64	1.61	USEPA3550C,8081B
9	276	138	0.74	

No. of results 9
 Median 240.0
 Normalised IQR 49.0
 Robust CV 20.4%
 Minimum 169
 Maximum 319
 Range 150
 Uncertainty (Median) 20.5

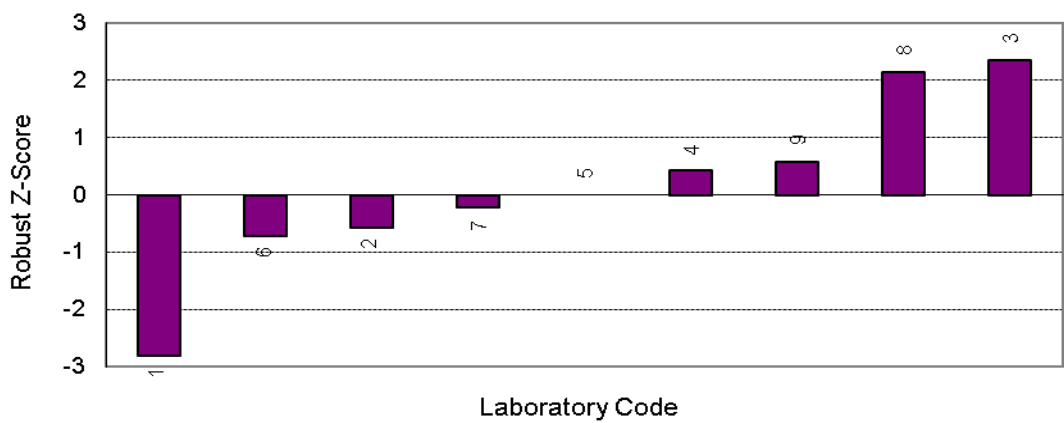
Heptachlor (µg/kg)



Heptachlor epoxide (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	90.7	12.1	-2.81	SM6630/US EPA 8081A
2	122	36.6	-0.57	GC-ECD
3	163	29.3	2.36	GC-ECD
4	135.9	33.97	0.42	USEPA 8081
5	130	46	0.00	FSO-02
6	120	34	-0.72	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	127	51	-0.21	
8	160	32	2.15	
9	138	68.8	0.57	USEPA3550C,8081B

No. of results 9
 Median 130.0
 Normalised IQR 14.0
 Robust CV 10.8%
 Minimum 90.7
 Maximum 163
 Range 72.3
 Uncertainty (Median) 5.8

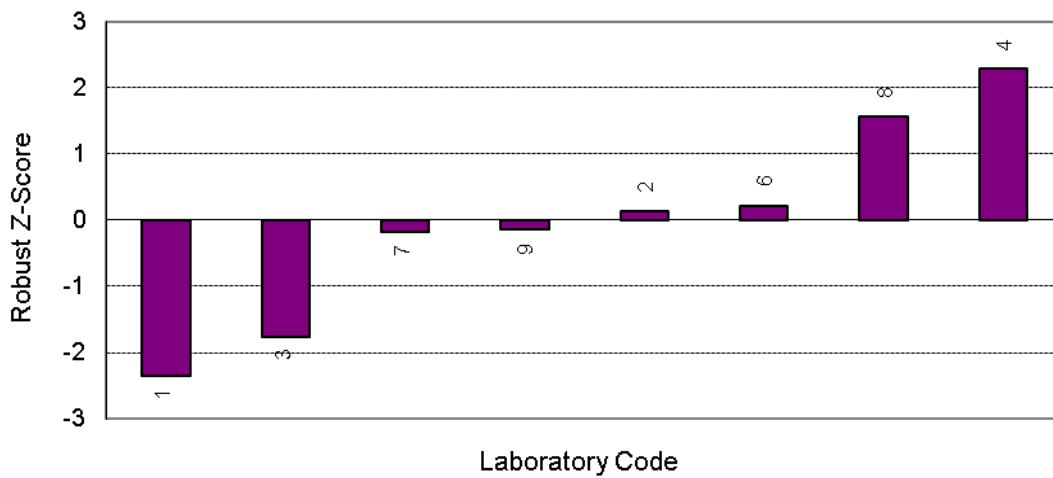
Heptachlor epoxide (µg/kg)



Methoxychlor (µg/kg)				
Lab Code	Result	MU	Robust Z-Score	Method
1	76.0	25.7	-2.35	SM6630/US EPA 8081A
2	171	51.3	0.13	GC-ECD
3	98.6	17.7	-1.76	GC-ECD
4	253.8	63.45	2.29	USEPA 8081
6	174.5	120	0.22	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	159	64	-0.18	
8	226	45	1.57	USEPA3550C,8081B
9	161	80.4	-0.13	

No. of results 8
 Median 166.0
 Normalised IQR 38.3
 Robust CV 23.1%
 Minimum 76.0
 Maximum 253.8
 Range 177.8
 Uncertainty (Median) 17.0

Methoxychlor (µg/kg)



APPENDIX B

Homogeneity and Stability Testing

HOMOGENEITY TESTING

The samples utilised in this program were supplied by Environmental Resource Associates (ERA).

Testing material was packaged in one flame-sealed ampoule containing 30.0 ± 0.2 grams of soil.

For this program, homogeneity testing was performed by ERA. Statistical analysis showed that the samples were sufficiently homogeneous, so that any results identified as outliers could not be attributed to sample variability.

STABILITY TESTING

Stability testing was also undertaken by ERA. The analysis of the stability testing results showed that the samples were sufficiently stable for testing during the program.

APPENDIX C

Instructions to Participants

and

Results Sheets

SOILS PROFICIENCY TESTING PROGRAM

APRIL 2013

INSTRUCTIONS TO PARTICIPANTS

1. **Sample**

This standard is packaged in one flame-sealed ampoule containing 30.0 ± 0.2 grams of soil. It is not preserved, and it should be stored at $4 \pm 2^\circ\text{C}$. This product is intended to be used as a quality control check of the entire analytical process for the analytes/matrix included in the standard. Although the soil standard has been thoroughly blended prior to shipping, the standard should be homogenized prior to opening the ampoule due to settling which may occur during shipping.

2. **Analysis**

Carefully snap the top off the Organochlorine Pesticides ampoule in a fume hood to avoid inhalation of dust.

Transfer the entire contents of the ampoule to the extraction vessel.

Use a weight of 30 grams for calculation purposes.

Prepare and analyze this standard per your normal analytical procedures.

3. **Tests Requested**

The following tests are to be conducted on the sample:

Aldrin, alpha-BHC, beta-BHC, delta-BHC, gamma-BHC(Lindane), alpha-Chlordane, gamma-Chlordane, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Dieldrin, Endrin, Endrin aldehyde, Endrin ketone, Endosulfan I, Endosulfan II, Endosulfan sulphate, Heptachlor, Heptachlor epoxide and Methoxychlor

All results are to be reported on the attached Results Sheet. Please ensure that the method used is entered on the Results Sheet for each analyte. If the method is not a standard method, please provide a precise description.

4. **Safety**

- (i) Sample is for laboratory use only.
- (ii) All required safety procedures should be followed

5. **Reporting**

- (a) Please submit results on the Results Sheets provided.
- (b) In addition to reporting the results, record the method of extraction and analysis.
- (c) Please report each analyte to the units ($\mu\text{g}/\text{kg}$) indicated on the Results Sheets.
- (d) Please report results to three significant figures. We acknowledge that this may not be your laboratory's normal reporting procedure, however you are requested to follow these instructions for statistical purposes.

6. **Measurement Uncertainty**

Laboratories are requested to calculate and report an estimate of measurement uncertainty (MU) for each reported measurement result. All estimates of MU must be given as a 95% confidence interval (coverage factor $k \approx 2$) and reported as \pm in ($\mu\text{g}/\text{kg}$).

7. Testing should commence as soon as possible after sample receipt, and results reported

NO LATER THAN 30 April 2013 to:

Mail: Dr Michael Li
Senior Scientific Officer
Proficiency Testing Australia
PO Box 7507
SILVERWATER NSW 2138 AUSTRALIA

Phone: +61 2 9736 8397
Fax: +61 2 9743 6664
Email: michael.li@pta.asn.au

8. For this program your laboratory has been allocated the code number shown on the attached Results Sheet. All reference to your laboratory in reports associated with the program will be through this code number, thus ensuring the confidentiality of your results.

C3

PROFICIENCY TESTING AUSTRALIA

SOILS PROFICIENCY PROGRAM - ROUND 7

**APRIL 2013
RESULTS SHEET**

Laboratory Code

Analyte	Result (µg/kg)	±MU (µg/kg)	Method
Aldrin			
alpha-BHC			
beta-BHC			
delta-BHC			
gamma-BHC(Lindane)			
alpha-Chlordane			
gamma-Chlordane			
4,4'-DDD			
4,4'-DDE			
4,4'-DDT			
Dieldrin			
Endrin			

PROFICIENCY TESTING AUSTRALIA
SOILS PROFICIENCY PROGRAM - ROUND 7
APRIL 2013

RESULTS SHEET

Laboratory Code:

Analyte	Result (µg/kg)	±MU (µg/kg)	Method
Endrin aldehyde			
Endrin ketone			
Endosulfan I			
Endosulfan II			
Endosulfan sulphate			
Heptachlor			
Heptachlor epoxide			
Methoxychlor			

Method description (if not standard method): _____

Date of Testing: _____ Signature: _____

Please return results **NO LATER THAN 30 APRIL 2013**.

Dr Michael Li
 Senior Scientific Officer
 Proficiency Testing Australia
 PO Box 7507 SILVERWATER NSW 2128
 Phone: +61 2 9736 8397 Fax: +61 2 9743 6664
 Email: michael.li@pta.asn.au

- END OF REPORT -