Report No. 859

SOILS (CHEMICAL)
ROUND 9

PROFICIENCY TESTING PROGRAM

June 2014

ACKNOWLEDGMENTS

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Mr M Blades from Environmental Resource Associates (ERA), USA. This assistance included input into the design of the program and technical advice. PTA also wishes to acknowledge Environmental Resource Associates (ERA), USA for sample supply and conducting homogeneity and stability testing.

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CONTENTS

1. FOREWORD

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

Proficiency Testing Australia (PTA) conducted the program in April 2014. The Program Coordinator was Dr M Li. The Technical Adviser was Mr M Blades from Environmental Resource Associates (ERA), USA. This report was authorised by Mrs F Watton, PTA Quality - Business Development Manager. 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, normalised interquartile range (IQR), uncertainty of the median, robust coefficient of variation (CV), minimum, maximum and range.

3. FEATURES OF THE PROGRAM

(a) A total of nine laboratories received samples. All participating 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 Sheet”. 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, normalised interquartile range (IQR), uncertainty of the median, 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 Sheet”.

5. SUMMARY OF RESULTS

TABLE A: SUMMARY OF RESULTS

Test	No. of Results	Median (µg/kg)	Normalised IQR
Aldrin	9	36.95	7.23
alpha-BHC	9	29.40	3.53
beta-BHC	9	153.0	29.7
delta-BHC	8	38.97	6.08
gamma-BHC (Lindane)	9	n/a	n/a
alpha-Chlordane	8	236.0	57.4
gamma-Chlordane	9	161.2	33.2
4,4'-DDD	9	196.0	45.5
4,4'-DDE	9	207.2	24.9
4,4'-DDT	9	59.40	8.02
Dieldrin	9	128.0	16.2
Endrin	9	78.95	18.07
Endrin aldehyde	7	100.05	42.95
Endrin ketone	5	n/a	n/a
Endosulfan I	9	n/a	n/a
Endosulfan II	9	n/a	n/a
Endosulfan sulphate	9	97.00	24.26
Heptachlor	9	76.35	10.51
Heptachlor epoxide	8	49.30	14.13
Methoxychlor	8	256.5	39.0

NOTE: Statistical analysis was not applied for gamma-BHC (Lindane), 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 AND TECHNICAL ADVISER'S 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 19.7% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 3.23 µg/kg.

(b) alpha-BHC

A robust CV of 12.0% was obtained. One outlier result was identified for laboratory 2. The uncertainty of the median is ± 1.67µg/kg.

(c) beta-BHC

A robust CV of 19.4% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 12.4 µg/kg.

(d) delta-BHC

A robust CV of 16.0% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 2.94 µg/kg.

(e) gamma-BHC (Lindane)

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

(f) alpha-Chlordane

A robust CV of 24.3% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 25.4 µg/kg.

(g) gamma-Chlordane

A robust CV of 20.6% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 13.9 µg/kg.

(h) 4,4'-DDD

A robust CV of 23.2% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 19.0 µg/kg.

(i) 4,4'-DDE

A robust CV of 12.0% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 10.4 µg/kg.

(j) 4,4'-DDT

A robust CV of 13.6% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 3.58 µg/kg.

(k) Dieldrin

A robust CV of 12.6% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 6.8 µg/kg.

(l) Endrin

A robust CV of 23.1% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 8.07 µg/kg.

(m) Endrin aldehyde

A robust CV of 44.8% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 25.11 µg/kg.

(n) Endrin ketone

Statistical analysis was not applied as there were only five results. PTA requires a minimum of six results in order to perform statistical analysis.

(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 25.2% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 10.83 µg/kg.

(r) Heptachlor

A robust CV of 13.9% was obtained. No result was identified as an outlier. The uncertainty of the median is ± 4.69 µg/kg.

(s) Heptachlor epoxide

A robust CV of 28.7% was obtained. One outlier result was identified for laboratory 1. The uncertainty of the median is ± 6.26 µg/kg.

(t) Methoxychlor

A robust CV of 15.2% was obtained. One outlier result was identified for laboratory 1. The uncertainty of the median is ± 17.3 µg/kg.

(iv) Overall Performance

The overall performance of the participants in this study was within expectations. The median values for eleven of the twenty analytes were within 30% of the expected values, with four other analytes being within 35% of the expected values. Only one analyte was considerably outside of expectations:

- The Aldrin median value was 32% of the expected value.

Measurement uncertainties (MUs) do not account for the low recoveries of this analyte.

One analyte, Endrin Ketone, did not have enough data reported to conduct a full statistical evaluation, although the median of the data reported was at 76% of the expected value.

Statistical analysis was not applied to gamma-BHC (Lindane) as the results did not follow a normal distribution. The median of the data reported was at 64% of the expected value. Statistical analysis was not applied to Endosulfan I and Endosulfan II as there were insufficient numerical results reported. Although certified at <5 µg/kg, two false positive results were reported for Endosulfan I.

Based on the tight accuracy of much of the data reported, the MUs appear to be more than adequate.

TABLE B: STATISTICAL OUTLIER RESULTS (By Laboratory Code)

Test	Laboratory Codes
alpha-BHC	**2**
Heptachlor epoxide	**1**
Methoxychlor	**1**

Two 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.

In general, the precision of this study was consistent with historically based expectations. Although most results had a z-score of less than 2.0, two laboratories had instances of z-scores of 3.0 or greater.

With the information provided for the methods used, there is no discernible pattern with respect to variability between methods. Some laboratories only reported their extraction methods, and some laboratories only reported their analytical methods.

Most reported MUs are in the range of 10-30%. Some laboratories reported MU values significantly outside of this range, and some laboratories reported MU values within the 50-100% range.

This study provides a valid assessment of laboratories' performance for the analysis of organochlorine pesticides in soil. Those results with a z-score of 3.0 or greater should be treated as non-conforming results and appropriate corrective action should be taken.

At the conclusion of the corrective action process, 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 closely assessed 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.

Further work may be needed in the area of MU calculation due to the range of MUs reported by participants.

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

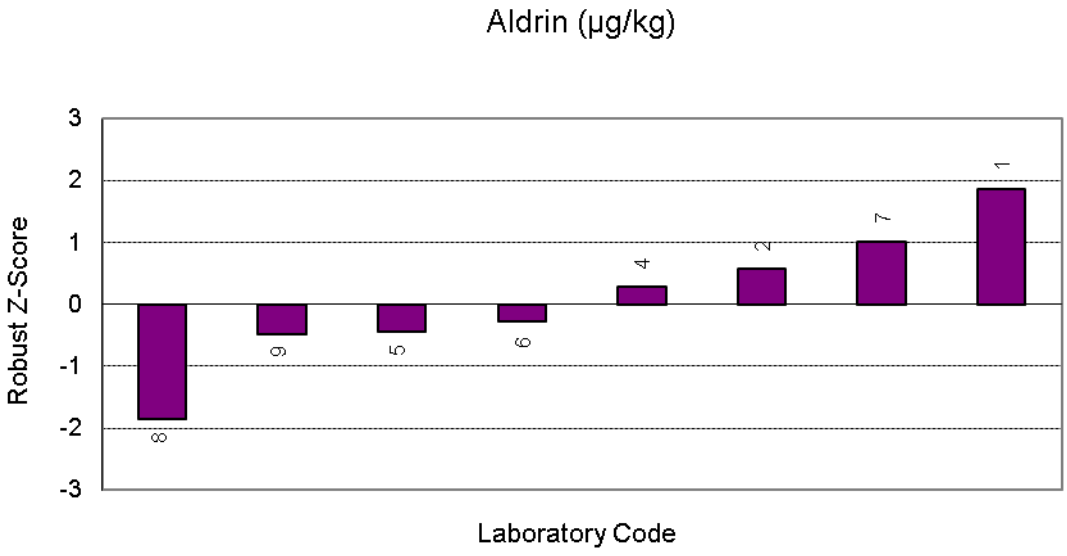
Appendix	A1	Aldrin
	A2	alpha-BHC
	A3	beta-BHC
	A4	delta-BHC
	A5	gamma-BHC (Lindane)
	A6	alpha-Chlordane
	A7	gamma-Chlordane
	A8	4,4'-DDD
	A9	4,4'-DDE
	A10	4,4'-DDT
	A11	Dieldrin
	A12	Endrin
	A13	Endrin aldehyde
	A14	Endrin ketone
	A15	Endosulfan I
	A16	Endosulfan II
	A17	Endosulfan sulphate
	A18	Heptachlor
	A19	Heptachlor epoxide
	A20	Methoxychlor

Aldrin (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	50.5	10.1	1.86	USEPA 3550C; USEPA8081B
2	41.1	19	0.57	FSO-02
3	<100	*	n/a	In-house based on USEPA 3550C & USEPA 8081B
4	39	16	0.28	RLS-OM-05
5	33.7	11	-0.45	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	34.9	10	-0.28	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	44.4	21.7	1.02	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	23.5	25.9	-1.85	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	33.5	6.70	-0.47	REF USEPA 8082

No. of results	9
Median	36.95
Normalised IQR	7.29
Robust CV	19.7%
Minimum	23.5
Maximum	50.5
Range	27.0
Uncertainty (Median)	3.23

NOTE: n/a - indicates not applicable.

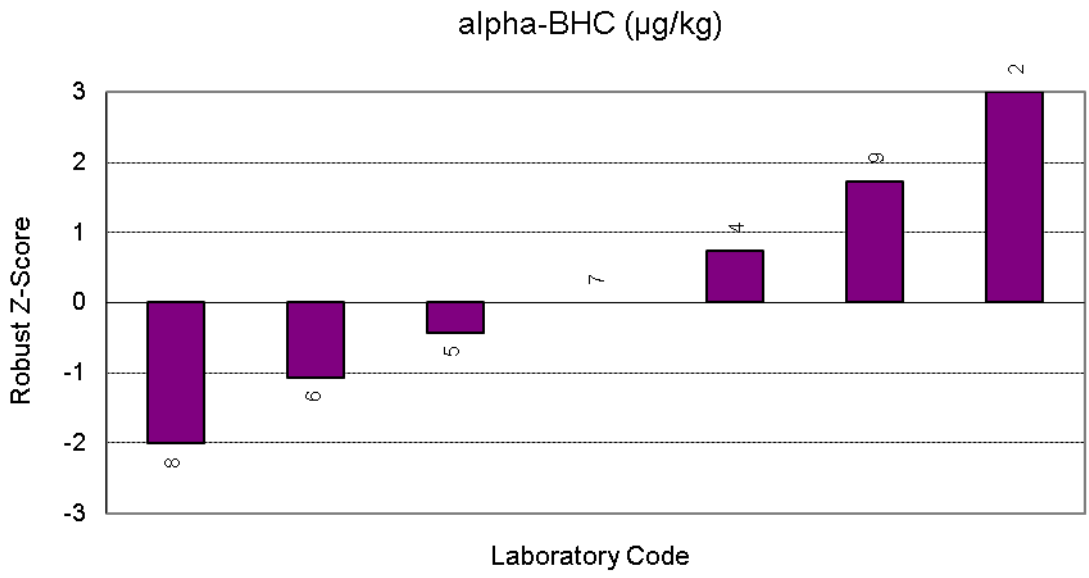
* - indicates no result returned for this test.



alpha-BHC (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	<50.0	10.0	n/a	USEPA 3550C; USEPA8081B
2	40.4	18	3.12 §	FSO-02
3	<100	*	n/a	In-house based on USEPA 3550C & USEPA 8081B
4	32	13	0.74	RLS-OM-05
5	27.9	8.4	-0.43	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	25.6	10	-1.08	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	29.4	8.45	0.00	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	22.3	9.37	-2.01	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	35.5	7.10	1.73	REF USEPA 8082

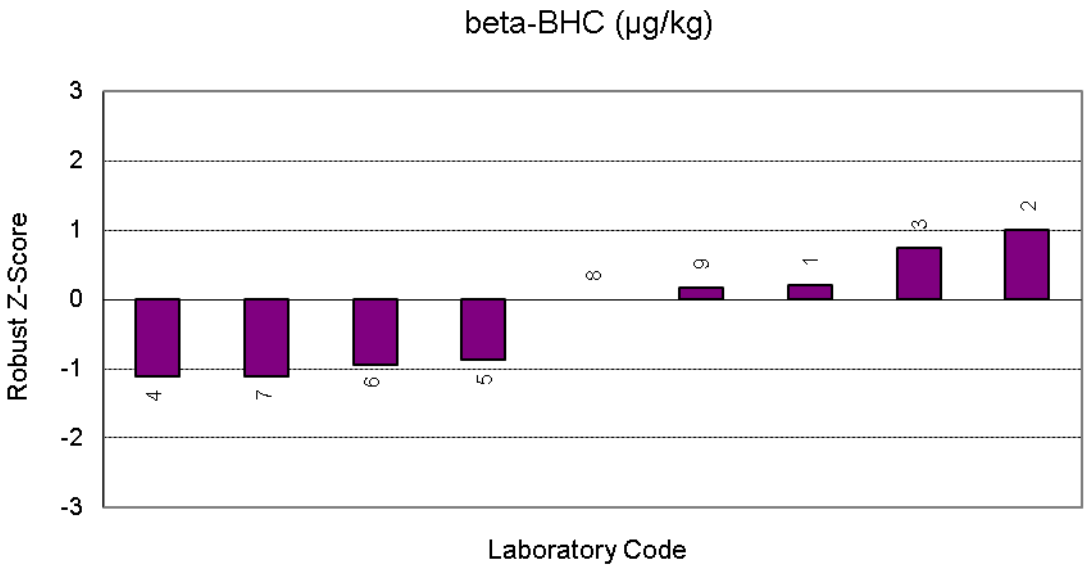
No. of results	9
Median	29.40
Normalised IQR	3.53
Robust CV	12.0%
Minimum	22.3
Maximum	40.4
Range	18.1
Uncertainty (Median)	1.67

NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).
n/a - indicates not applicable.
* - indicates no result returned for this test.



beta-BHC (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	159	32	0.20	USEPA 3550C; USEPA8081B
2	183	21	1.01	FSO-02
3	175	70	0.74	Extraction with 20:80 Acetone:Hexane
4	120	48	-1.11	RLS-OM-05
5	127.4	49	-0.86	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	125	10	-0.94	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	120	24.1	-1.11	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	153	84.2	0.00	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	158	31.5	0.17	REF USEPA 8082

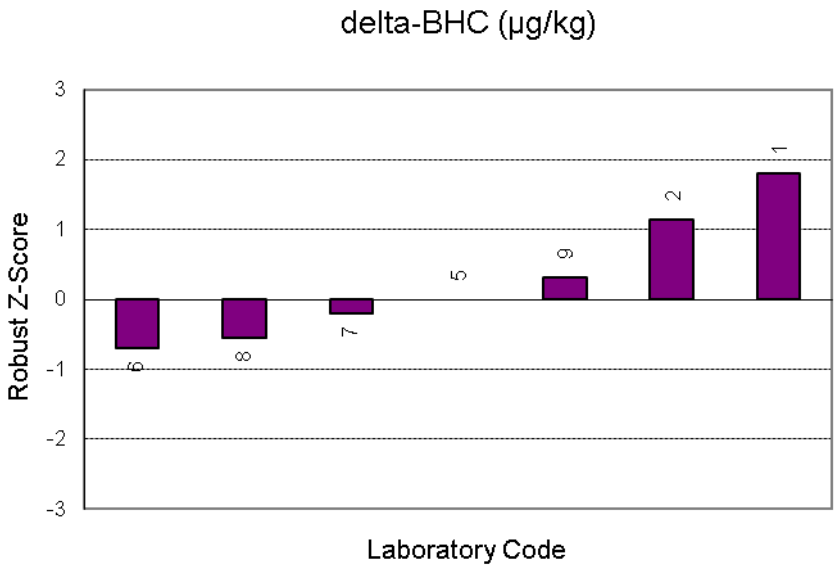
No. of results	9
Median	153.0
Normalised IQR	29.7
Robust CV	19.4%
Minimum	120
Maximum	183
Range	63
Uncertainty (Median)	12.4



delta-BHC (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	50.2	10.0	1.81	USEPA 3550C; USEPA8081B
2	46.1	17	1.15	FSO-02
3	<100	*	n/a	Analysed by GC-ECD with dual column
5	38.97	14	0.00	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	34.6	25	-0.70	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	37.7	18.0	-0.20	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	35.5	8.88	-0.56	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	40.9	8.18	0.31	REF USEPA 8082

No. of results	8
Median	38.97
Normalised IQR	6.22
Robust CV	16.0%
Minimum	34.6
Maximum	50.2
Range	15.6
Uncertainty (Median)	2.94

NOTE: n/a - indicates not applicable.
* - indicates no result returned for this test.



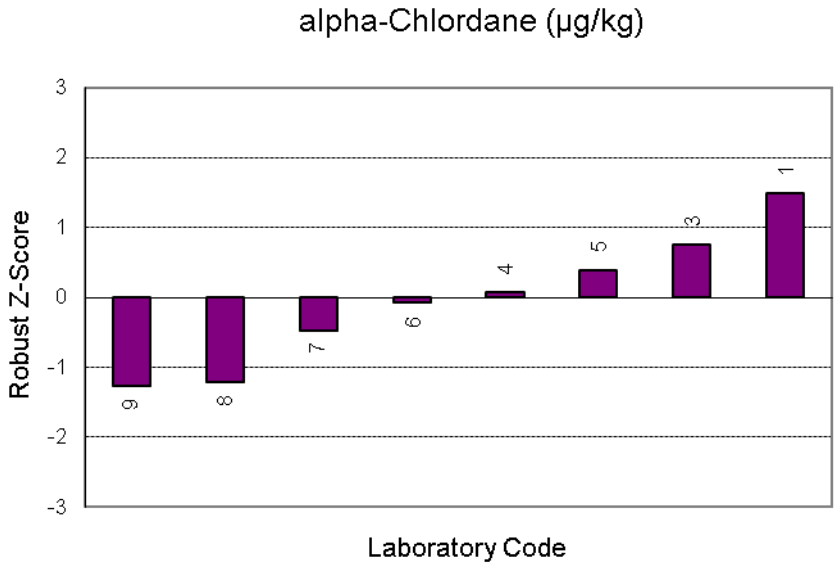
gamma-BHC (Lindane) (µg/kg)			
Lab Code	**Result**	**MU**	**Method**
1	164	33	USEPA 3550C; USEPA8081B
2	154	21	FSO-02
3	146	58.4	Analysed by GC-ECD with dual column
4	110	44	RLS-OM-05
5	100.1	29	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	88.8	15	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	107	27.4	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	104	66.6	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	143	28.6	REF USEPA 8082

No. of results 9

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

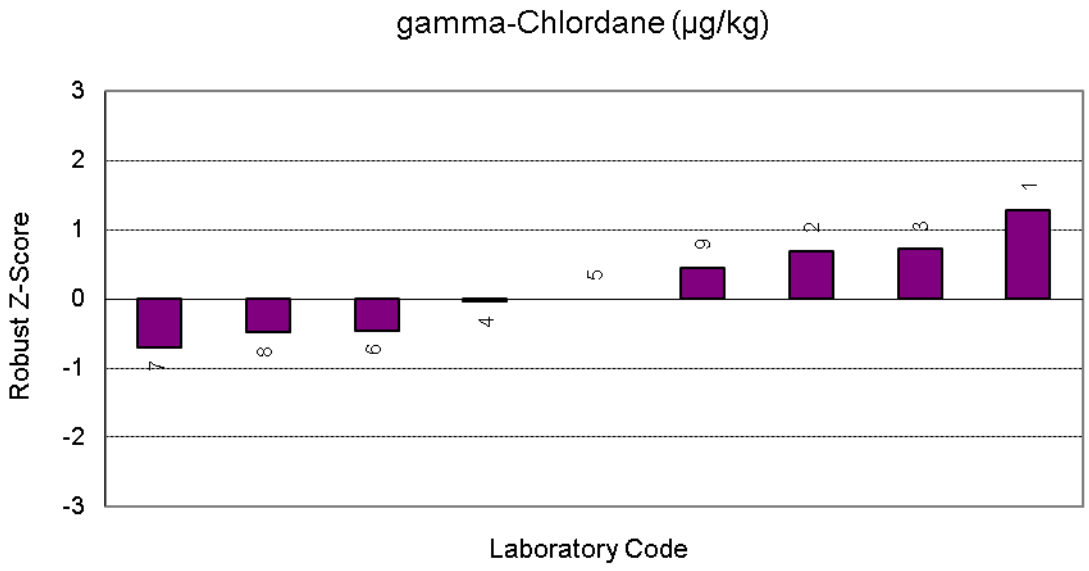
alpha-Chlordane (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	321	64	1.48	USEPA 3550C; USEPA8081B
3	279	112	0.75	Analysed by GC-ECD with dual column
4	240	96	0.07	RLS-OM-05
5	258.2	83	0.39	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	232	20	-0.07	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	209	98	-0.47	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	166	101	-1.22	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	163	32.5	-1.27	REF USEPA 8082

No. of results	8
Median	236.0
Normalised IQR	57.4
Robust CV	24.3%
Minimum	163
Maximum	321
Range	158
Uncertainty (Median)	25.4



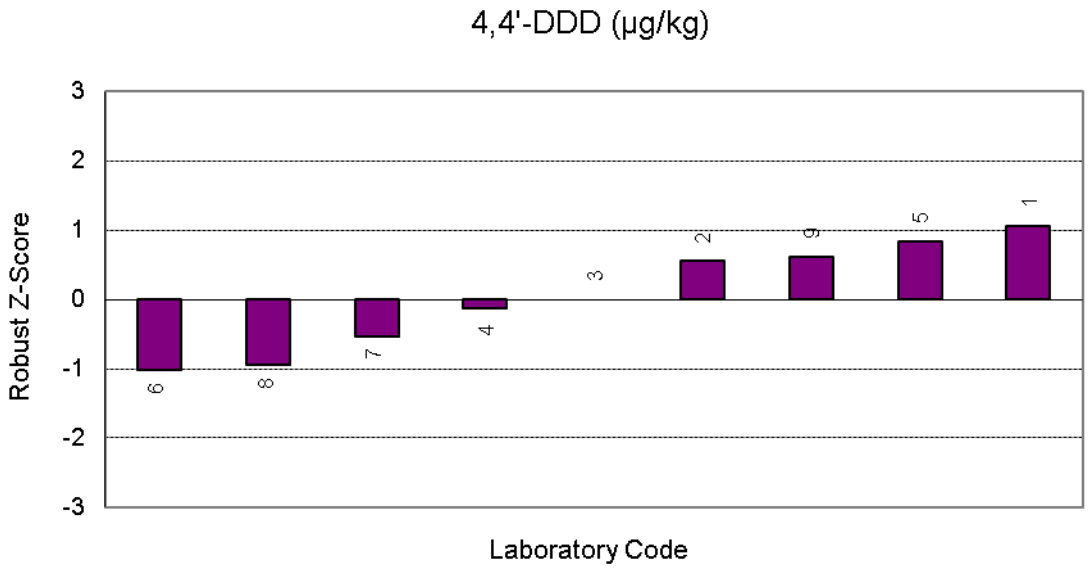
gamma-Chlordane (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	204	41	1.29	USEPA 3550C; USEPA 8081B
2	184	26	0.69	FSO-02
3	185	74	0.72	Analysed by GC-ECD with dual column
4	160	64	-0.04	RLS-OM-05
5	161.2	49	0.00	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	146	14	-0.46	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	138	20	-0.70	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	145	98.6	-0.49	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	176	35.2	0.45	REF USEPA 8082

No. of results	9
Median	161.2
Normalised IQR	33.2
Robust CV	20.6%
Minimum	138
Maximum	204
Range	66
Uncertainty (Median)	13.9



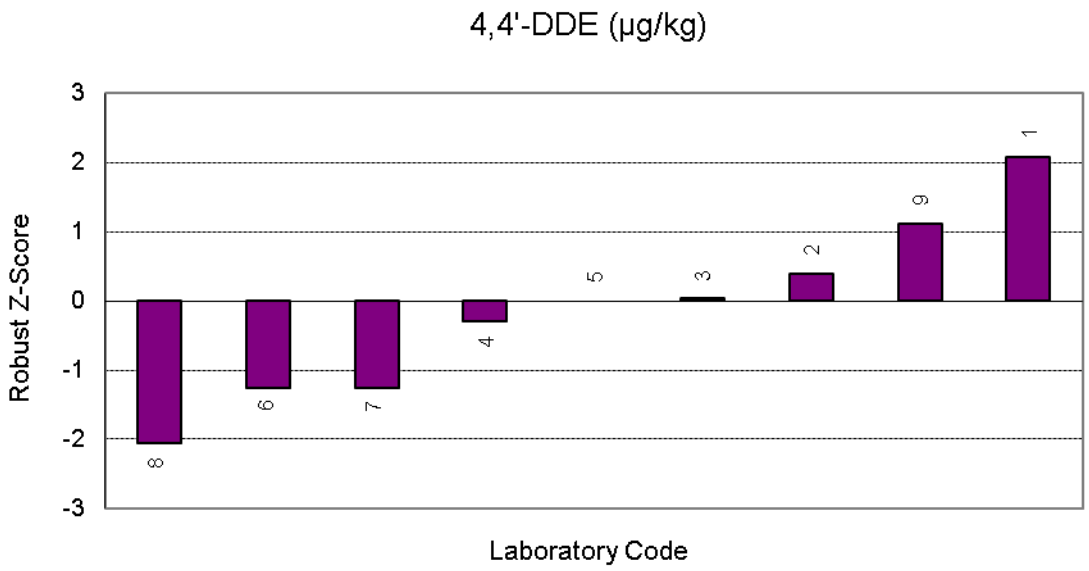
4,4'-DDD (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	244	49	1.06	USEPA 3550C; USEPA 8081B
2	221	55	0.55	FSO-02
3	196	78.4	0.00	Analysed by GC-ECD with dual column
4	190	76	-0.13	RLS-OM-05
5	234.3	120	0.84	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	150	12	-1.01	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	172	31	-0.53	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	153	103	-0.95	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	224	44.7	0.62	REF USEPA 8082

No. of results	9
Median	196.0
Normalised IQR	45.5
Robust CV	23.2%
Minimum	150
Maximum	244
Range	94
Uncertainty (Median)	19.0



4,4'-DDE (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	259	52	2.08	USEPA 3550C; USEPA 8081B
2	217	57	0.39	FSO-02
3	208	83.2	0.03	Analysed by GC-ECD with dual column
4	200	80	-0.29	RLS-OM-05
5	207.2	110	0.00	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	176	16	-1.25	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	176	92	-1.25	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	156	87.4	-2.06	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	235	47.0	1.12	REF USEPA 8082

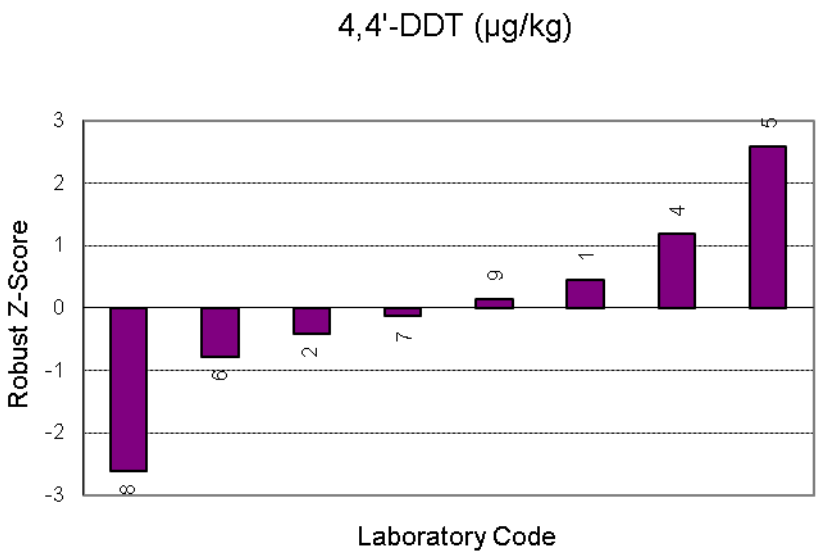
No. of results	9
Median	207.2
Normalised IQR	24.9
Robust CV	12.0%
Minimum	156
Maximum	259
Range	103
Uncertainty (Median)	10.4



4,4'-DDT (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	63.0	12.6	0.45	USEPA 3550C; USEPA 8081B
2	56.1	107	-0.41	FSO-02
3	<100	*	n/a	Analysed by GC-ECD with dual column
4	69	28	1.19	RLS-OM-05
5	80.3	47	2.59	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	53	26	-0.79	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	58.3	23	-0.14	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	38.3	22.2	-2.61	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	60.5	12.1	0.14	REF USEPA 8082

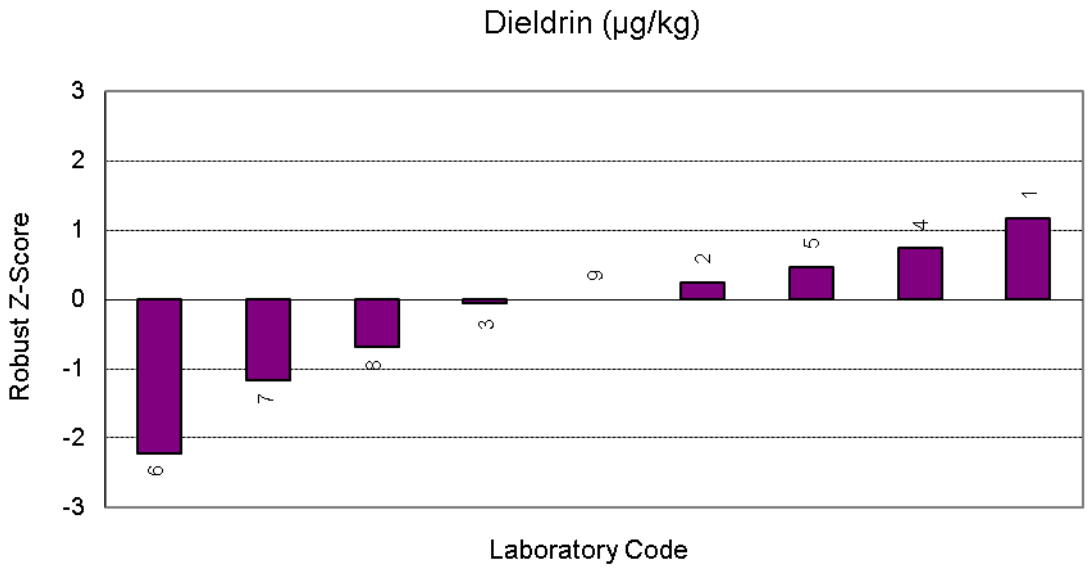
No. of results	9
Median	59.40
Normalised IQR	8.08
Robust CV	13.6%
Minimum	38.3
Maximum	80.3
Range	42.0
Uncertainty (Median)	3.58

NOTE: n/a - indicates not applicable.
* - indicates no result returned for this test.



Dieldrin (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	147	29	1.17	USEPA 3550C; USEPA8081B
2	132	39	0.25	FSO-02
3	127	50.8	-0.06	Analysed by GC-ECD with dual column
4	140	56	0.74	RLS-OM-05
5	135.5	60	0.46	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	92	13	-2.23	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	109	46	-1.17	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	117	50.3	-0.68	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	128	25.6	0.00	REF USEPA 8082

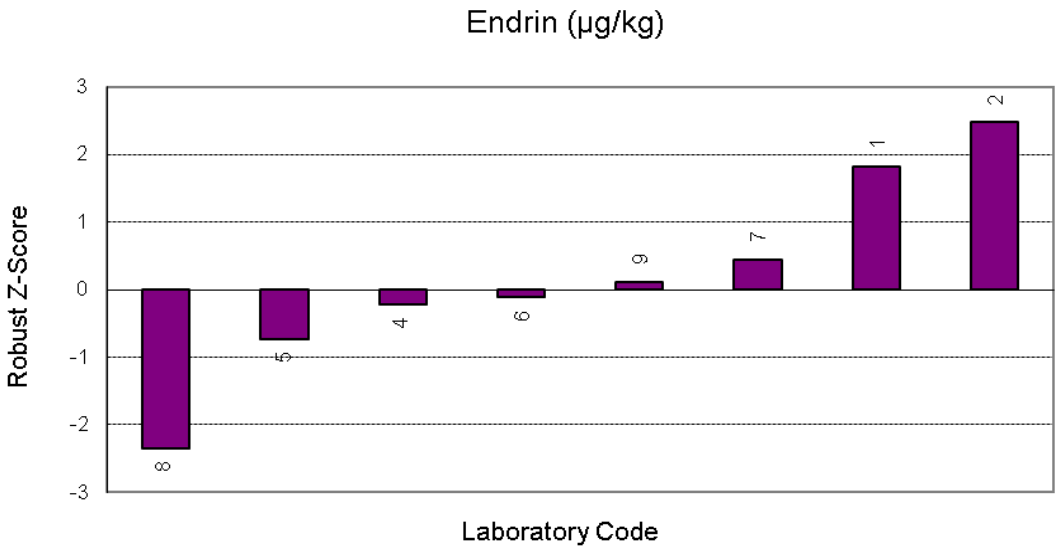
No. of results	9
Median	128.0
Normalised IQR	16.2
Robust CV	12.6%
Minimum	92
Maximum	147
Range	55
Uncertainty (Median)	6.8



Endrin (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	112	22	1.82	USEPA 3550C; USEPA8081B
2	124	34	2.47	FSO-02
3	<100	*	n/a	Analysed by GC-ECD with dual column
4	75	30	-0.22	RLS-OM-05
5	65.6	44	-0.73	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	77	13	-0.11	Pre-wet with Phosphoric acid, Sonication with Hexane / Acetone; GC-ECD analysis.
7	87.1	26.1	0.45	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	36.1	39.7	-2.35	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	80.9	16.2	0.11	REF USEPA 8082

No. of results	9
Median	78.95
Normalised IQR	18.21
Robust CV	23.1%
Minimum	36.1
Maximum	124
Range	87.9
Uncertainty (Median)	8.07

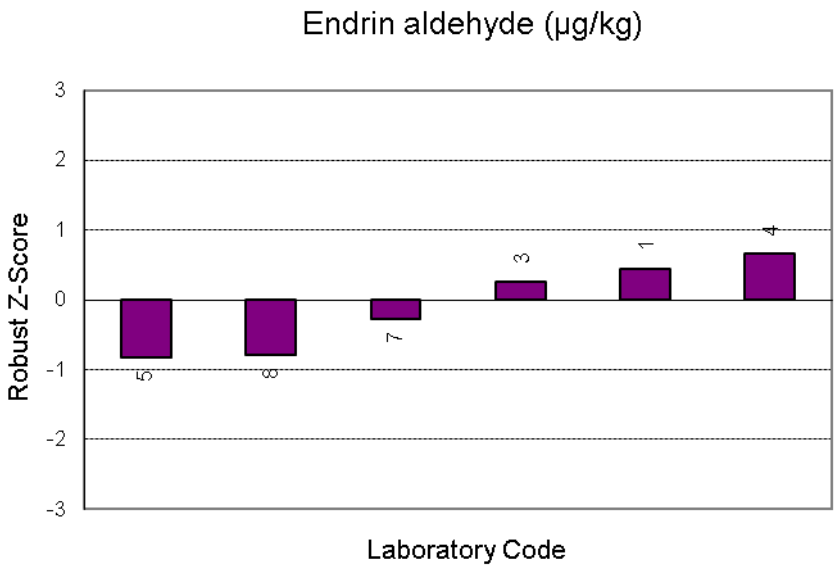
NOTE: n/a - indicates not applicable.
* - indicates no result returned for this test.



Endrin aldehyde (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	120	24	0.45	USEPA 3550C; USEPA8081B
3	112	44.8	0.27	Analysed by GC-ECD with dual column
4	130	52	0.67	RLS-OM-05
5	62.8	36	-0.83	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
7	88.1	41.9	-0.27	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	64.4	148	-0.80	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	<10	*	n/a	

No. of results	7
Median	100.05
Normalised IQR	44.81
Robust CV	44.8%
Minimum	62.8
Maximum	130
Range	67.2
Uncertainty (Median)	25.11

NOTE: n/a - indicates not applicable.
* - indicates no result returned for this test.



Endrin ketone (µg/kg)			
Lab Code	**Result**	**MU**	**Method**
1	133	27	USEPA 3550C; USEPA8081B
3	101	40.4	Analysed by GC-ECD with dual column
4	120	48	RLS-OM-05
5	122.9	60	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
9	104	20.7	REF USEPA 8082

No. of results 5

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

Endosulfan I (µg/kg)			
Lab Code	**Result**	**MU**	**Method**
1	<50.0	10.0	USEPA 3550C; USEPA8081B
2	<50	*	FSO-02
3	<100	*	Analysed by GC-ECD with dual column
4	34	14	RLS-OM-05
5	<1	0.46	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	<10	*	USEPA 3540C/8081A
7	37.3	11.2	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	<3.3	*	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	<10	*	REF USEPA 8082

No. of results 9

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

* - indicates no result returned for this test.

Endosulfan II (µg/kg)			
Lab Code	**Result**	**MU**	**Method**
1	<50.0	10.0	USEPA 3550C; USEPA8081B
2	<100	*	FSO-02
3	<100	*	Analysed by GC-ECD with dual column
4	<10	*	RLS-OM-05
5	<1	0.52	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	<10	*	USEPA 3540C/8081A
7	<10	*	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	<3.3	*	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	<10	*	REF USEPA 8082

No. of results 9

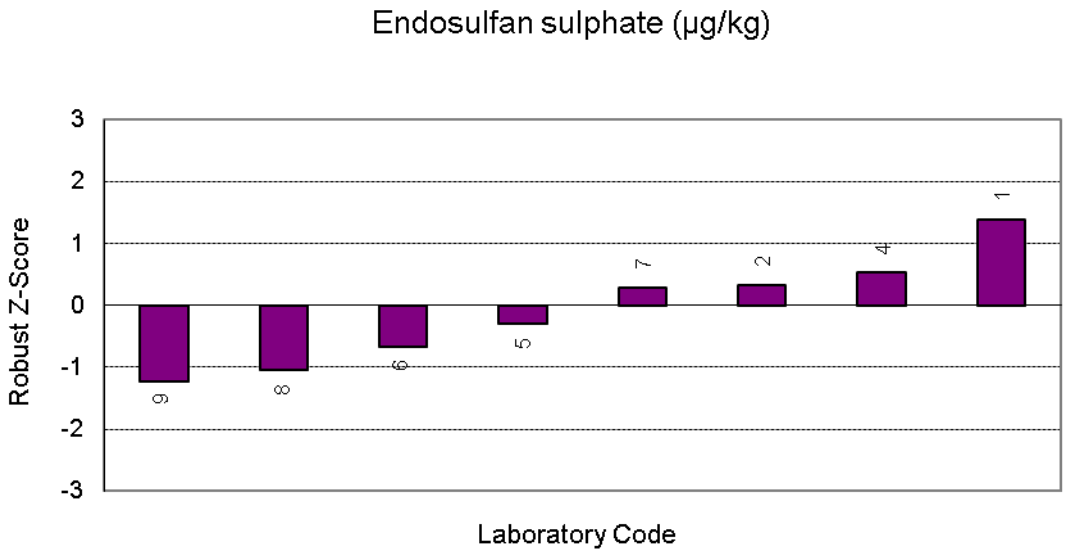
NOTE: Statistical analysis was not applied as there were insufficient numerical results

* - indicates no result returned for this test.

Endosulfan sulphate (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	131	26	1.39	USEPA 3550C; USEPA8081B
2	105	29	0.33	FSO-02
3	<100	*	n/a	Analysed by GC-ECD with dual column
4	110	44	0.53	RLS-OM-05
5	90.0	56	-0.29	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	80.9	18	-0.66	USEPA 3540C/8081A
7	104	20	0.29	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	71.3	78.4	-1.05	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	67.0	13.4	-1.23	REF USEPA 8082

No. of results	9
Median	97.00
Normalised IQR	24.44
Robust CV	25.2%
Minimum	67.0
Maximum	131
Range	64.0
Uncertainty (Median)	10.83

NOTE: n/a - indicates not applicable.
* - indicates no result returned for this test.

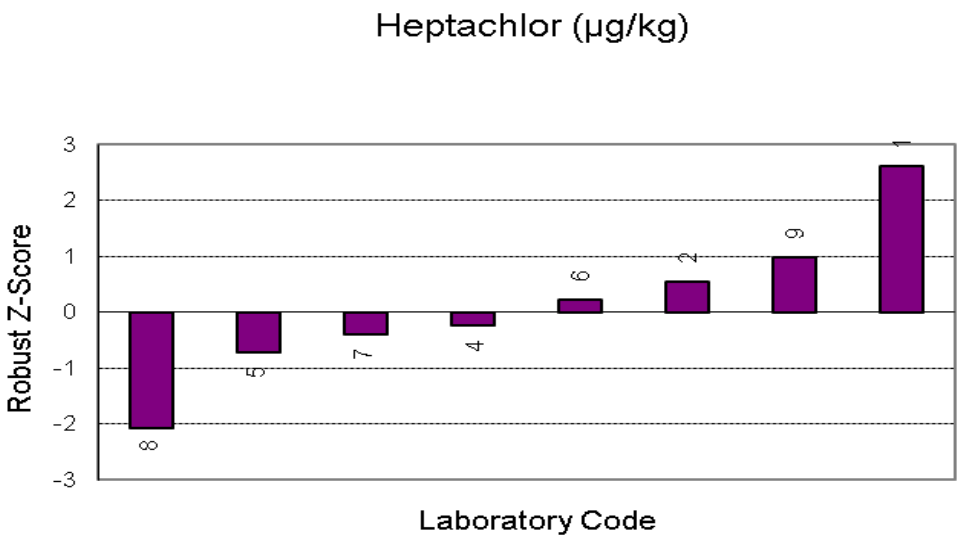


Heptachlor (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	104	21	2.61	USEPA 3550C; USEPA 8081B
2	82.1	38	0.54	FSO-02
3	<100	*	n/a	Analysed by GC-ECD with dual column
4	74	30	-0.22	RLS-OM-05
5	68.7	24	-0.72	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	78.7	10	0.22	USEPA 3540C/8081A
7	72.1	29.2	-0.40	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	54.5	30.5	-2.06	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	86.8	17.4	0.99	REF USEPA 8082

No. of results	9
Median	76.35
Normalised IQR	10.59
Robust CV	13.9%
Minimum	54.5
Maximum	104
Range	49.5
Uncertainty (Median)	4.69

NOTE: n/a - indicates not applicable.

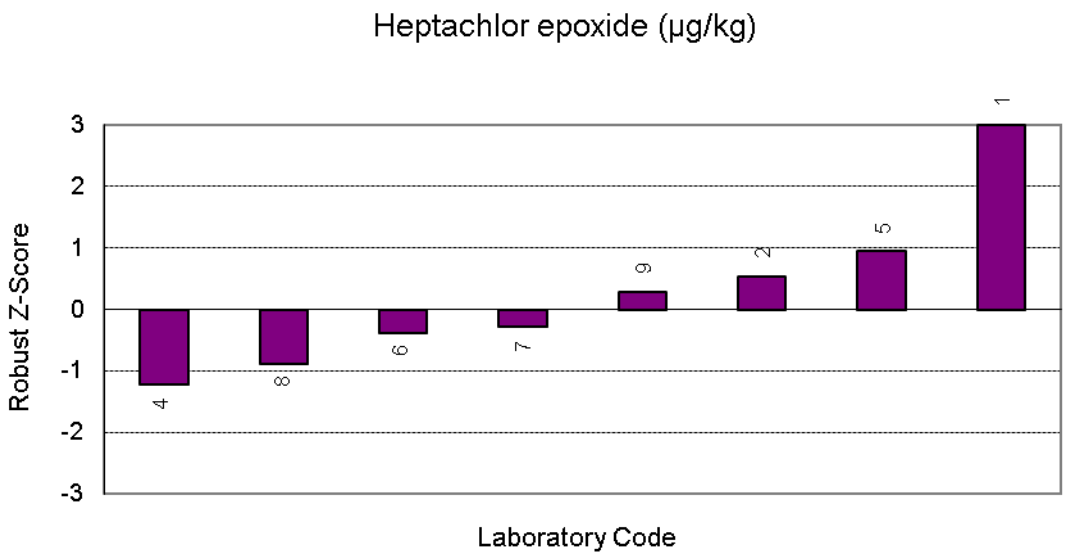
* - indicates no result returned for this test.



Heptachlor epoxide (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	92.3	18.5	3.04 §	USEPA 3550C; USEPA8081B
2	56.8	35	0.53	FSO-02
4	32	13	-1.22	RLS-OM-05
5	62.6	18	0.94	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	44.0	11	-0.37	USEPA 3540C/8081A
7	45.4	11.4	-0.28	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	36.8	22.1	-0.88	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	53.2	10.6	0.28	REF USEPA 8082

No. of results	8
Median	49.30
Normalised IQR	14.13
Robust CV	28.7%
Minimum	32
Maximum	92.3
Range	60.3
Uncertainty (Median)	6.26

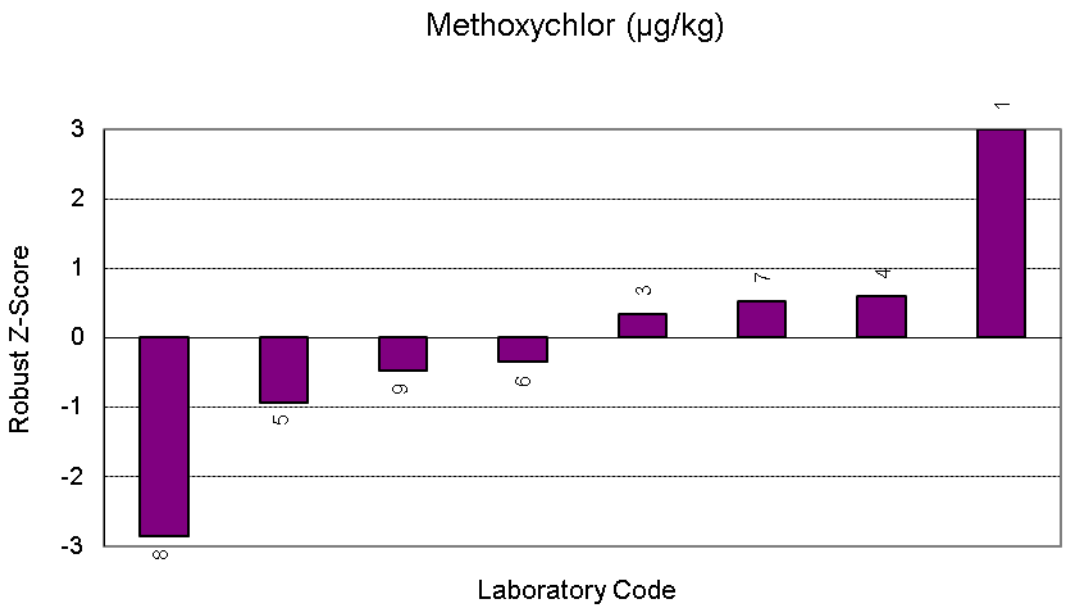
NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).



Methoxychlor (µg/kg)				
Lab Code	**Result**	**MU**	**Robust Z-Score**	**Method**
1	389	78	3.40 §	USEPA 3550C; USEPA8081B
3	270	108	0.35	Analysed by GC-ECD with dual column
4	280	112	0.60	RLS-OM-05
5	220	150	-0.94	Sonication Extraction in Hexane/Acetone. Dual Column ECD/ECD ac analysis.
6	243	35	-0.35	USEPA 3540C/8081A
7	277	87	0.53	End-over-end tubling + sonication with 1:1 DCM acetone GCMS analysis
8	145	69.6	-2.86	Solvent extraction followed by GPC & florisil clean-up. Analysed by GCMS.
9	238	47.5	-0.47	REF USEPA 8082

No. of results	8
Median	256.5
Normalised IQR	39.0
Robust CV	15.2%
Minimum	145
Maximum	389
Range	244
Uncertainty (Median)	17.3

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



APPENDIX B

Homogeneity and Stability Testing

HOMOGENEITY TESTING

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

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 Sheet

SOILS PROFICIENCY TESTING PROGRAM

ROUND 9

APRIL 2014

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 ± 2℃. 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 homogenised 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 analyse this standard as 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**

- The sample is for laboratory use only.
- All required safety procedures should be followed.

5. **Reporting**

- Please submit results on the Results Sheet provided.
- In addition to reporting the results, record the method of extraction and analysis.
- Please report each analyte to the units (µg/kg) indicated on the Results Sheet.
- 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 ≈ 2) and reported as ± in (µg/kg).

7. Testing should commence as soon as possible after sample receipt, and results reported **NO LATER THAN 18 April 2014** 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 9
APRIL 2014

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 9
APRIL 2014

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 18 APRIL 2014.**

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-