Report No. 809

PCBs in Oil Proficiency Testing

Round 6

June 2013

Acknowledgments

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Mr G Ienco of Transformer Fitness Pty Ltd.

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CONTENTS

1. FOREWORD

This report summarises the results of a proficiency testing program on the determination of polychlorinated biphenyls (PCBs) in oil. It constitutes the sixth round of an ongoing series of programs.

The program was conducted in April 2013 by Proficiency Testing Australia (PTA). The aim of the program was to assess laboratories' abilities to competently perform the prescribed analyses.

The Program Coordinator was Ms L Galbraith and the Technical Advisor was Mr G Ienco from Transformer Fitness Pty Ltd. This report was authorised by Ms W Fajloun, PTA Quality Coordinator.

2. FEATURES OF THE PROGRAM

(a) Participants were provided with 3 samples from the labelled PTA Sample 1, PTA Sample 2, PTA Sample 3 and PTA Sample 4, with each consisting of approximately 4ml of oil containing PCBs.

(b) A total of 11 laboratories received samples, all of which were Australian participants. All laboratories submitted results by the due date.

(c) Laboratories were provided with the *Instructions to Participants* and *Results Sheet* (see Appendix C). Laboratories were requested to perform the tests according to their routine methods and to record their results on the *Results Sheet*.

(d) Prior to sample distribution, a number of randomly selected samples were analysed for homogeneity. Based on the results of this testing (see Appendix B), the homogeneity of the samples was established.

(e) Each laboratory was randomly allocated a unique code number for the program to ensure confidentiality of results. Reference to each laboratory in this report is by code number only.

(f) Results (as reported by participants) with corresponding summary statistics (i.e. number of results, median, uncertainty of the median, normalised interquartile range, robust coefficient of variation, minimum, maximum and range) are presented in Appendix A (for each sample and for each of the analyses performed). Measurement Uncertainty (MU) is also presented where supplied by participants. Please note that this information is presented for information purposes only and has not been used for the formal evaluation of results.

(g) A robust statistical approach, using z-scores, was utilised to assess laboratories' testing performance (see Section 4). Robust z-scores and z-score charts relevant to each test are presented in Appendix A.

(h) The document entitled *Guide to Proficiency Testing Australia,* 2012 (reference [1]) defines the statistical terms and details the statistical procedures referred to in this report.

3. FORMAT OF THE APPENDICES

(a) Appendix A contains the analysis of results reported by laboratories for the samples. This section contains the following for each determinant, where appropriate:

- a table of results and calculated z-scores;
- a list of summary statistics; and
- ordered z-score charts.

(b) Appendix B contains details of the homogeneity testing.

(c) Appendix C contains copies of the *Instructions to Participants* and *Results Sheet*.

4. STATISTICAL DESIGN OF THE PROGRAM

(a) Outlier Results and Z-scores

In order to assess laboratories' testing performance, a robust statistical approach, using z-scores, was utilised. Z-scores give a measure of how far a result is from the consensus value (i.e. the median), and gives a "score" to each result relative to the other results in the group.

A z-score close to zero indicates that the result agrees well with those from other laboratories. Whereas, a z-score with an absolute value greater than or equal to 3.0 is considered to be an outlier and is marked by the symbol "**§**".

(b) Results Tables and Summary Statistics

Each of these tables contains the results returned by each laboratory, including the code number for the method used, and the robust z-score calculated for each result.

Results have been entered exactly as reported by participants. That is, laboratories which did not report results to the precision (i.e. number of

significant figures) requested on the Results Sheet have **not** been rounded to the requested precision before being included in the statistical analysis.

A list of summary statistics appears at the bottom of each of the tables of results and consists of:

- the number of results for that test/sample (*No. of Results*);
- the median of these results, i.e. the middle value (*Median*);
- the uncertainty of the median;
- the normalised interquartile range of the results (*Normalised IQR*);
- the robust coefficient of variation, expressed as a percentage (*Robust CV*) - i.e. 100 x *Normalised IQR* / *Median*;
- the minimum and maximum laboratory results; and
- the range (*Maximum* - *Minimum*).

The median is a measure of the centre of the data.

The normalised IQR is a measure of the spread of the results. It is calculated by multiplying the interquartile range (IQR) by a correction factor which converts the IQR to an estimate of the standard deviation. The IQR is the difference between the upper and lower quartiles (i.e. the values above and below which a quarter of the results lie, respectively).

Please see reference [1] for further details on these robust summary statistics.

(c) Ordered Z-Score Charts

On these charts each laboratory's robust z-score is shown, in order of magnitude, and is marked with its code number. From these charts, each laboratory can readily compare its performance relative to the other laboratories.

These charts contain solid lines at +3.0 and -3.0, so that outliers are clearly identifiable as those laboratories whose "bar" extends beyond these "cut-off" lines. The y-axis of these charts has been limited, so very large z-scores appear to extend beyond the chart boundary.

The following table summarises the results submitted by participants for the program.

TABLE A: SUMMARY STATISTICS

Aroclor Type	PTA Sample	No. of Results	Median	Normalised IQR
1242	1	5	N/A	N/A
	2	4	N/A	N/A
	3	5	N/A	N/A
	4	5	N/A	N/A
1248	1	4	N/A	N/A
	2	4	N/A	N/A
	3	4	N/A	N/A
	4	4	N/A	N/A
1254	1	8	35.15	12.76
	2	6	N/A	N/A
	3	5	N/A	N/A
	4	8	4.35	1.94
1260	1	5	N/A	N/A
	2	7	51.00	17.13
	3	7	N/A	N/A
	4	8	2.69	0.33
Other	1	2	N/A	N/A
	2	1	N/A	N/A
	3	2	N/A	N/A
	4	3	N/A	N/A
Total PCBs	1	8	36.90	12.76
	2	8	83.08	15.75
	3	6	N/A	N/A
	4	8	6.95	2.34

5. PTA AND TECHNICAL ADVISOR'S COMMENTS

5.1 Metrological Traceability and Measurement Uncertainty of Assigned Values

Consensus values (median) derived from 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 in corresponding tables in Appendix A.

5.2 Analysis of Results by Method Groups

All laboratories reported the same method for all tests on all samples in Round 6 PCBs in Oil Proficiency Testing Program, therefore results generated using these methods have been pooled for analysis and can be found in Appendix A.

5.3 Legislation-Environmental

In 2000, the Victorian Environment Protection Act 1970 was varied to include PCBs as a notifiable chemical. As stated in this order - "polychlorinated biphenyls" are any material or waste containing "polychlorinated biphenyls" at a concentration of more than 2 mg/kg.

The Victorian EPA publication 693b (2001) states that wastes containing greater than 50mg/kg is a prescribed waste (schedule PCB) and must undergo treatment. A concentration of less than 50mg/kg but greater than 2mg/kg is classified as containing 'non-schedule PCBs'. If the concentration is less than 2mg/kg then it is classified as 'PCB-free' as described in the National Management Plan (ANZEC). Therefore we have three levels in which critical decisions are made about a waste material and how it will be disposed of and potentially treated. These levels are greater than 50mg/kg, less than 50mg/kg but greater than 2mg/kg, and less than 2mg/kg. For this assessment, it is critical that the PCB values reported at these three critical levels are accurate and precise.

5.4 False Negative / Positive Results and Misidentification

Of the 11 laboratories submitting results for Round 6 PCBs in Oil, 4 laboratories reported false positives or false negatives. These laboratories must investigate their analytical procedures related to standards preparation, staff training in correct identification and quantification of Aroclors, and contamination control of laboratory equipment that is used for these analyses.

Two laboratories reported Aroclor 1242 in PTA Sample 2 when none of this analyte was present in any of the PTA Samples. This indicates a problem in correct identification of Aroclors, or the possibility of chronic contamination of analytical equipment.

Laboratory 22 reported a false positive for Aroclor 1260 in PTA Sample 1 and Aroclor 1242 in PTA Sample 2. Laboratory 26 also reported a false positive for Aroclor 1242 in PTA Sample 2 and Laboratory 8 reported a false positive for Aroclor 1254 in PTA Sample 3. The PCB misidentification typically leads to a false positive of a specific Aroclor type but does not lead to a false positive as such, as the samples do contain PCBs. The current Australian regulations state a total PCB amount and do not distinguish between PCB types.

Laboratory 15 failed to detect Aroclor 1254 in PTA Sample 2 and Laboratory 10 received a false negative for Aroclor 1254 in PTA Sample 2. Laboratory 8 reported higher detection limits than the critically required regulatory value of 2mg/kg for some Aroclors, as indicated in the results tables in Appendix A.

As the electron capture detectors (ECDs) and to some degree the Mass Spectrometer respond to the amount of chlorine present in a PCB molecule, they will give very different responses to different PCB types. The problem with PCB misidentification is that if the wrong standard is used, this can lead to large errors in quantitation.

5.5 PCB Mixtures

Mixtures of PCBs are very difficult to identify and quantitate using standard techniques for profiling. PTA Sample 2 and PTA Sample 4 contain both Aroclor 1254 and Aroclor 1260. Seven out of eight laboratories were able to detect Aroclor 1260 in PTA Sample 2 whilst only five out of the eight laboratories reporting results for PTA Sample 2 were able to detect Aroclor 1254. Laboratory 8 did not record results for any Aroclors in PTA Sample 2 however correctly identified the total PCBs. All laboratories were able to identify Aroclors 1254 and 1260 in PTA Sample 4.

5.6 General Comments

Overall, the performance of participants in this round was good, with no results being identified as a statistical outlier. A couple of laboratories did not perform the analysis for one or more individual Aroclors requested in this trial. Laboratories should have use of the different PCBs standards. If a laboratory is performing analysis of environmental samples there is concern that they could misidentify PCB type (which leads to errors in quantitation) if they do not include in analysis common Aroclors. All laboratories carrying out environmental testing of Aroclors should obtain these commonly available standards.

All laboratories reported using the method Gas Chromatography/Electron Capture Detection. Due to the misidentification by some laboratories it appears that they are not conducting extensive clean-ups during the preparative steps. This may add variation to the overall results.

6. REFERENCE

[1] *Guide to Proficiency Testing Australia*, 2012 (This document can be found on the PTA website, www.pta.asn.au)

APPENDIX A

Results and Data Analysis

Aroclor Type 1242							
Laboratory Code	PTA Sample 1			PTA Sample 2			Method
	Result		MU	Result		MU	
7	<2			NA			Gas Chromatography / ECD
8	NA			<20^			Gas Chromatography / ECD
9	NA			NR			Gas Chromatography / ECD
10	<1			<1			Gas Chromatography / ECD
15	NA			ND			Gas Chromatography / ECD
21	ND			NA			Gas Chromatography / ECD
22	<2	±	0.5	3.9‡	±	0.78	Gas Chromatography / ECD
26	<2	±	0.5	4.50‡	±	0.5	Gas Chromatography / ECD
27	ND			ND			Gas Chromatography / ECD
28	<1	±	<1	NA			Gas Chromatography / ECD
29	ND			ND			Gas Chromatography / ECD

No of Results:	5	4

Notes: ND indicates not detected.
NA indicates not applicable.
NR indicates not recorded.
‡ denotes a false positive result.
^ Indicates a higher detection limit than the critically required regulatory value of 2mg/kg was reported.
No statistical analysis was conducted for Aroclor 1242 as there were not enough results to conduct a viable statistical analysis.

Aroclor Type 1242							
Laboratory Code	PTA Sample 3			PTA Sample 4			Method
	Result		MU	Result		MU	
7	<2			<2			Gas Chromatography / ECD
8	<0.1			<0.1			Gas Chromatography / ECD
9	<1			<1			Gas Chromatography / ECD
10	<1			NA			Gas Chromatography / ECD
15	ND			ND			Gas Chromatography / ECD
21	ND			ND			Gas Chromatography / ECD
22	NA			<2	±	0.5	Gas Chromatography / ECD
26	NA			<2	±	0.5	Gas Chromatography / ECD
27	ND			NA			Gas Chromatography / ECD
28	<1	±	<1	ND	±	ND	Gas Chromatography / ECD
29	ND			NA			Gas Chromatography / ECD

No of Results:	5	5

Notes: ND indicates not detected.
NA indicates not applicable.
No statistical analysis was conducted for Aroclor 1242 as there were not enough results to conduct a viable statistical analysis.

Aroclor Type 1248							
Laboratory Code	PTA Sample 1			PTA Sample 2			Method
	Result		MU	Result		MU	
7	<2			NA			Gas Chromatography / ECD
8	NA			<20^			Gas Chromatography / ECD
9	NA			NR			Gas Chromatography / ECD
10	<1			<1			Gas Chromatography / ECD
15	NA			NT			Gas Chromatography / ECD
21	ND			NA			Gas Chromatography / ECD
22	ND			ND			Gas Chromatography / ECD
26	<2	±	0.5	<2	±	0.5	Gas Chromatography / ECD
27	<1.0			<1.0			Gas Chromatography / ECD
28	NT	±	NT	NA			Gas Chromatography / ECD
29	ND			ND			Gas Chromatography / ECD

No of Results:	4	4

Notes: ND indicates not detected.
NA indicates not applicable.
NT indicates not tested.
NR indicates not recorded.
^ Indicates a higher detection limit than the critically required regulatory value of 2mg/kg was reported.
No statistical analysis was conducted for Aroclor 1248 as there were not enough results to conduct a viable statistical analysis.

Aroclor Type 1248							
Laboratory Code	PTA Sample 3			PTA Sample 4			Method
	Result		MU	Result		MU	
7	<2			<2			Gas Chromatography / ECD
8	<0.1			<0.1			Gas Chromatography / ECD
9	<1			<1			Gas Chromatography / ECD
10	<1			NA			Gas Chromatography / ECD
15	NT			NT			Gas Chromatography / ECD
21	ND			ND			Gas Chromatography / ECD
22	NA			ND			Gas Chromatography / ECD
26	NA			<2	±	0.5	Gas Chromatography / ECD
27	ND			NA			Gas Chromatography / ECD
28	NT	±	NT	NT	±	NT	Gas Chromatography / ECD
29	ND			NA			Gas Chromatography / ECD

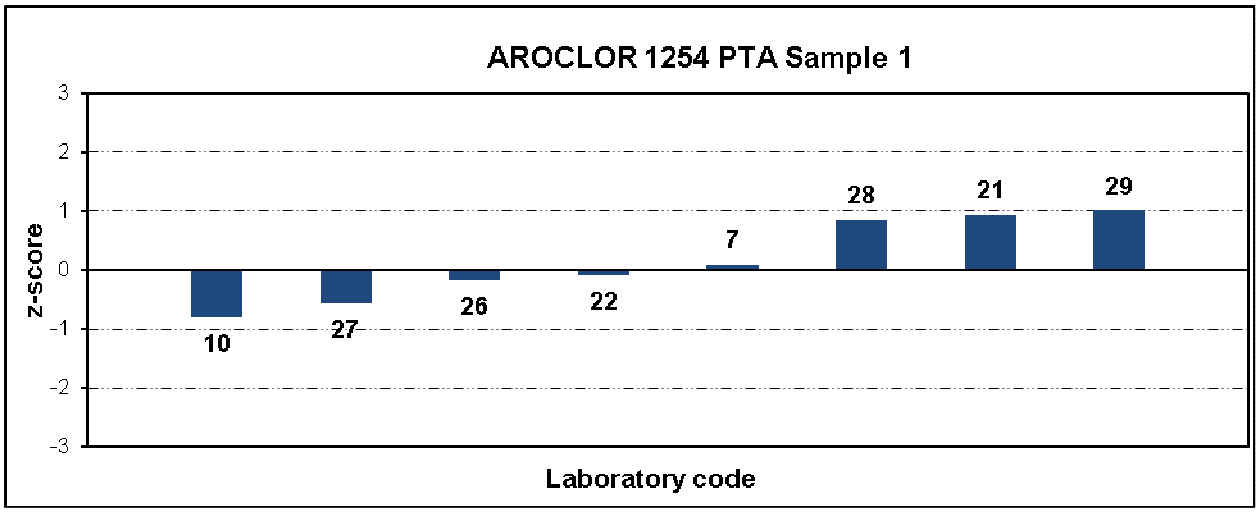
No of Results:	4	4

Notes: ND indicates not detected.
NA indicates not applicable.
NT indicates not tested.
No statistical analysis was conducted for Aroclor 1248 as there were not enough results to conduct a viable statistical analysis.

Aroclor Type 1254					
Laboratory Code	PTA Sample 1				Method
	Result		MU	Robust z-score	
7	36.3	±	6.5	0.09	Gas Chromatography / ECD
8	NA			NA	Gas Chromatography / ECD
9	NA			NA	Gas Chromatography / ECD
10	24.9	±	7.50	-0.80	Gas Chromatography / ECD
15	NA			NA	Gas Chromatography / ECD
21	47	±	3	0.93	Gas Chromatography / ECD
22	34.0	±	6.8	-0.09	Gas Chromatography / ECD
26	33.02	±	0.5	-0.17	Gas Chromatography / ECD
27	28	±	5.6	-0.56	Gas Chromatography / ECD
28	46	±	4.6	0.85	Gas Chromatography / ECD
29	48	±	12	1.01	Gas Chromatography / ECD

No of Results:	8
Median:	35.15
Normalised IQR:	12.76
Robust CV:	36.3%
Uncertainty of median:	5.65
Minimum:	24.9
Maximum:	48.0
Range:	23.1

Note: NA indicates not applicable.



Aroclor Type 1254							
Laboratory Code	PTA Sample 2			PTA Sample 3			Method
	Result		MU	Result		MU	
7	NA			<2			Gas Chromatography / ECD
8	41	±	16	0.11‡	±	0.04	Gas Chromatography / ECD
9	NR			<1			Gas Chromatography / ECD
10	<1†			<1			Gas Chromatography / ECD
15	ND			ND			Gas Chromatography / ECD
21	NA			ND			Gas Chromatography / ECD
22	39.6	±	7.9	NA			Gas Chromatography / ECD
26	31.18	±	0.5	NA			Gas Chromatography / ECD
27	34	±	6.8	ND			Gas Chromatography / ECD
28	NA			<1	±	<1	Gas Chromatography / ECD
29	30	±	8	ND			Gas Chromatography / ECD

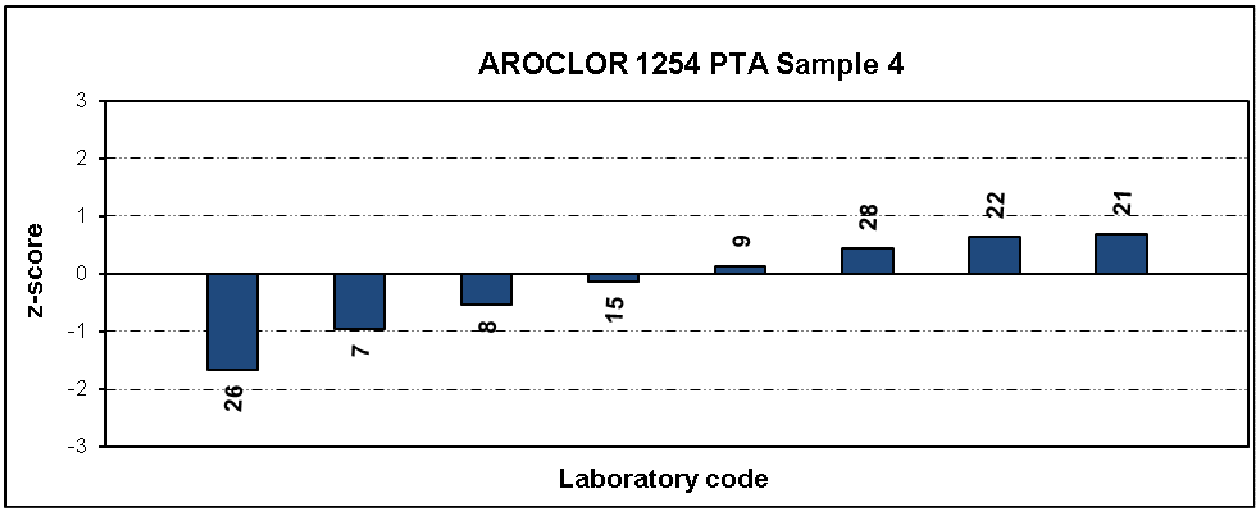
No of Results:	6	5

Notes: ND indicates not detected.
NA indicates not applicable.
NR indicates not recorded.
† denotes a false negative result.
‡ denotes a false positive result.
No statistical analysis was conducted for Aroclor 1254 PTA Sample 2 and PTA Sample 3 as there were not enough results to conduct a viable statistical analysis.

Aroclor Type 1254				
Laboratory Code	PTA Sample 4			Method
	Result	MU	Robust z-score	
7	2.49	0.5	-0.96	Gas Chromatography / ECD
8	3.3	1.3	-0.54	Gas Chromatography / ECD
9	4.6	1.38	0.13	Gas Chromatography / ECD
10	NA		NA	Gas Chromatography / ECD
15	4.1 ±	0.5	-0.13	Gas Chromatography / ECD
21	5.7	0.6	0.70	Gas Chromatography / ECD
22	5.6	1.4	0.64	Gas Chromatography / ECD
26	1.14	0.5	-1.65	Gas Chromatography / ECD
27	NA		NA	Gas Chromatography / ECD
28	5.2	0.52	0.44	Gas Chromatography / ECD
29	NA		NA	Gas Chromatography / ECD

No of Results:	8
Median:	4.35
Normalised IQR:	1.94
Robust CV:	44.6%
Uncertainty of median:	0.86
Minimum:	1.1
Maximum:	5.7
Range:	4.6

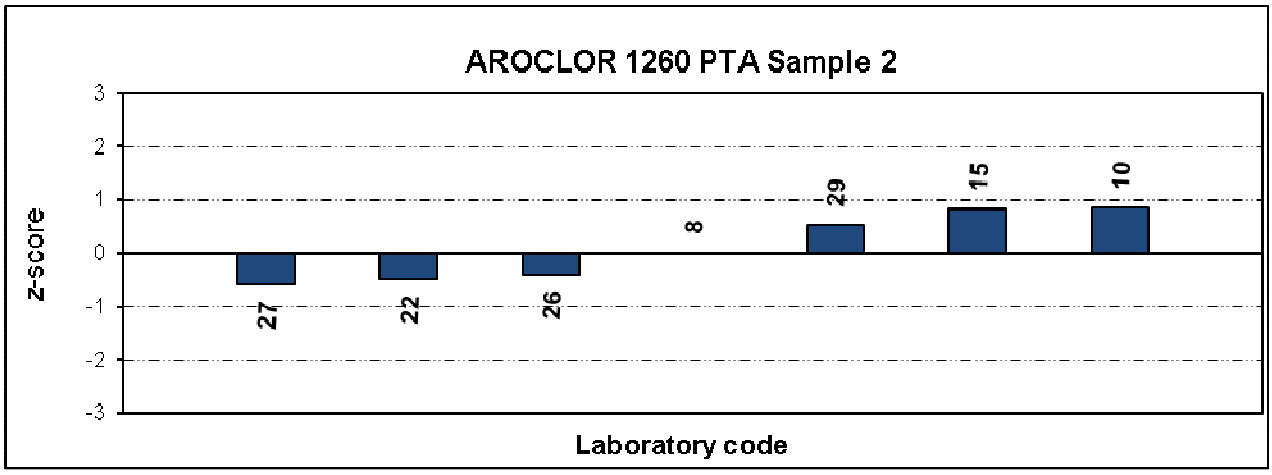
Note: NA indicates not applicable.



Aroclor Type 1260								
Laboratory Code	PTA Sample 1			PTA Sample 2				Method
	Result		MU	Result		MU	Robust z-score	
7	<2			NA			NA	Gas Chromatography / ECD
8	NA			51	±	20	0.00	Gas Chromatography / ECD
9	NA			NA			NA	Gas Chromatography / ECD
10	<1			65.5	±	19.7	0.85	Gas Chromatography / ECD
15	NA			65.0	±	5	0.82	Gas Chromatography / ECD
21	ND			NA			NA	Gas Chromatography / ECD
22	2.12‡	±	0.54	42.8	±	8.6	-0.48	Gas Chromatography / ECD
26	<2	±	0.5	44.17	±	0.5	-0.40	Gas Chromatography / ECD
27	ND			41	±	8.2	-0.58	Gas Chromatography / ECD
28	<1	±	<1	NA			NA	Gas Chromatography / ECD
29	ND			60	±	15	0.53	Gas Chromatography / ECD

No of Results:	5	7
Median:		51.00
Normalised IQR:		17.13
Robust CV:		33.6%
Uncertainty of median:		8.12
Minimum:		41.0
Maximum:		65.5
Range:		24.5

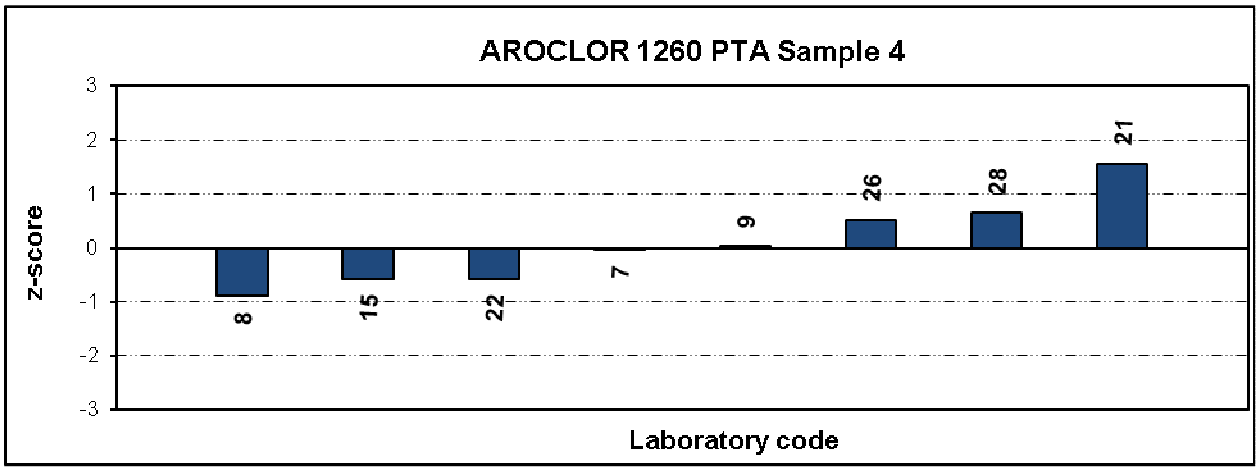
Notes: ND indicates not detected.
NA indicates not applicable.
‡ denotes a false positive result.
No statistical analysis was conducted for Aroclor 1260 PTA Sample 1 as there were not enough results to conduct a viable statistical analysis.



Aroclor Type 1260							
Laboratory Code	PTA Sample 3			PTA Sample 4			Method
	Result		MU	Result		MU	Robust z-score	
7	<2			2.68	±	0.5	-0.03	Gas Chromatography / ECD
8	0.44	±	0.18	2.4	±	0.96	-0.89	Gas Chromatography / ECD
9	<1			2.7	±	0.81	0.03	Gas Chromatography / ECD
10	<1			NA			NA	Gas Chromatography / ECD
15	0.6	±	0.1	2.5	±	0.5	-0.58	Gas Chromatography / ECD
21	0.84	±	0.06	3.2	±	0.6	1.56	Gas Chromatography / ECD
22	NA			2.5	±	0.62	-0.58	Gas Chromatography / ECD
26	NA			2.86	±	0.5	0.52	Gas Chromatography / ECD
27	ND			NA			NA	Gas Chromatography / ECD
28	<1	±	<1	2.9	±	0.29	0.64	Gas Chromatography / ECD
29	ND			NA			NA	Gas Chromatography / ECD

No of Results:	7	8
Median:		2.69
Normalised IQR:		0.33
Robust CV:		12.1%
Uncertainty of median:		0.14
Minimum:		2.4
Maximum:		3.2
Range:		0.8

Notes: ND indicates not detected.
NA indicates not applicable.
No statistical analysis was conducted for Aroclor 1260 PTA Sample 3 as the majority of results were reported as "less than" values.



Aroclor Type Other							
Laboratory Code	PTA Sample 1			PTA Sample 2			Method
	Result		MU	Result		MU	
7	<2			NA			Gas Chromatography / ECD
8	NA			NA			Gas Chromatography / ECD
9	NA			NR			Gas Chromatography / ECD
10	ND			ND			Gas Chromatography / ECD
15	NA			NT			Gas Chromatography / ECD
21	ND			NA			Gas Chromatography / ECD
22	ND			ND			Gas Chromatography / ECD
26	<2	±	0.5	<2			Gas Chromatography / ECD
27	ND			ND			Gas Chromatography / ECD
28	NT	±	NT	NA			Gas Chromatography / ECD
29	ND			ND			Gas Chromatography / ECD

No of Results:	2	1

Notes: ND indicates not detected.
NA indicates not applicable.
NT indicates not tested.
NR indicates not recorded.
No statistical analyses were conducted for this Aroclor as there were not enough results to conduct a viable statistical analysis.

Aroclor Type Other							
Laboratory Code	PTA Sample 3			PTA Sample 4			Method
	Result		MU	Result		MU	
7	<2			<2			Gas Chromatography / ECD
8	NA			NA			Gas Chromatography / ECD
9	<1			<1			Gas Chromatography / ECD
10	ND			NA			Gas Chromatography / ECD
15	NT			NT			Gas Chromatography / ECD
21	ND			ND			Gas Chromatography / ECD
22	NA			ND			Gas Chromatography / ECD
26	NA			<2			Gas Chromatography / ECD
27	ND			NA			Gas Chromatography / ECD
28	NT	±	NT	NT	±	NT	Gas Chromatography / ECD
29	ND			NA			Gas Chromatography / ECD

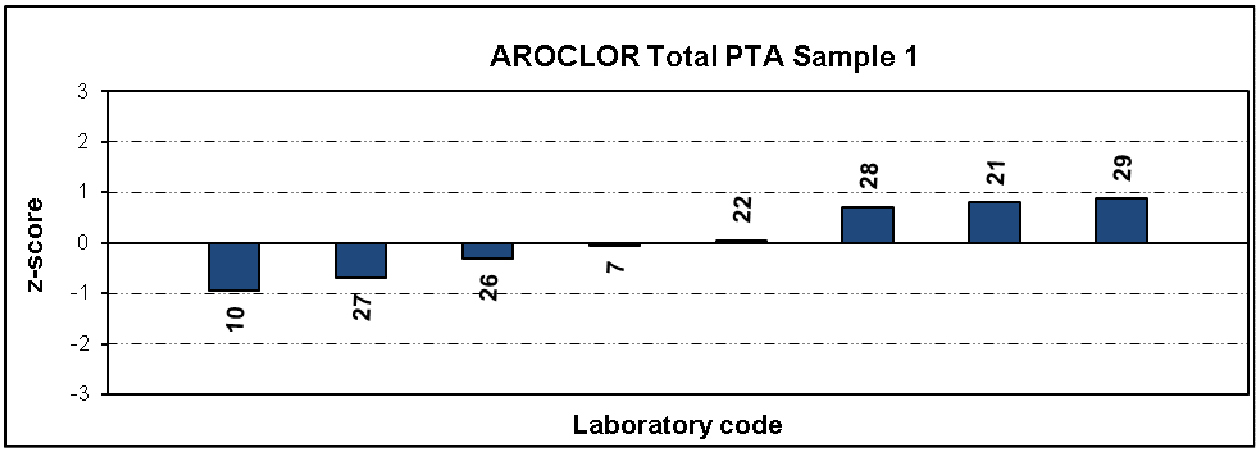
No of Results:	2	3

Notes: ND indicates not detected.
NA indicates not applicable.
NT indicates not tested.
No statistical analyses were conducted for this Aroclor as there were not enough results to conduct a viable statistical analysis.

Aroclor Type Total					
Laboratory Code	PTA Sample 1				Method
	Result		MU	Robust z-score	
7	36.3	±	6.5	-0.05	Gas Chromatography / ECD
8	NA			NA	Gas Chromatography / ECD
9	NA			NA	Gas Chromatography / ECD
10	24.9	±		-0.94	Gas Chromatography / ECD
15	NA			NA	Gas Chromatography / ECD
21	47	±	3	0.79	Gas Chromatography / ECD
22	37.5	±	9.4	0.05	Gas Chromatography / ECD
26	33.02	±	0.5	-0.30	Gas Chromatography / ECD
27	28	±	5.6	-0.70	Gas Chromatography / ECD
28	46	±	4.6	0.71	Gas Chromatography / ECD
29	48	±	12	0.87	Gas Chromatography / ECD

No of Results:	8
Median:	36.90
Normalised IQR:	12.76
Robust CV:	34.6%
Uncertainty of median:	5.65
Minimum:	24.9
Maximum:	48.0
Range:	23.1

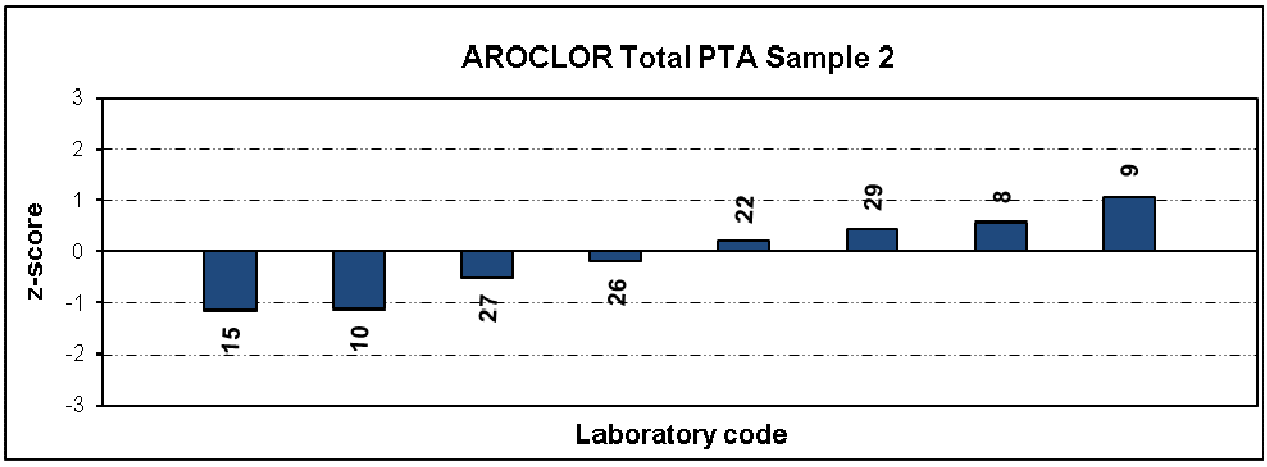
Note: NA indicates not applicable.



Aroclor Type Total					
Laboratory Code	PTA Sample 2				Method
	Result		MU	Robust z-score	
7	NA			NA	Gas Chromatography / ECD
8	92	±	36	0.57	Gas Chromatography / ECD
9	100	±	30	1.07	Gas Chromatography / ECD
10	65.5			-1.12	Gas Chromatography / ECD
15	65.0	±	5	-1.15	Gas Chromatography / ECD
21	NA			NA	Gas Chromatography / ECD
22	86.3	±	21.6	0.20	Gas Chromatography / ECD
26	79.85	±	0.5	-0.20	Gas Chromatography / ECD
27	75	±	15	-0.51	Gas Chromatography / ECD
28	NA			NA	Gas Chromatography / ECD
29	90	±	23	0.44	Gas Chromatography / ECD

No of Results:	8
Median:	83.08
Normalised IQR:	15.75
Robust CV:	19.0%
Uncertainty of median:	6.98
Minimum:	65.0
Maximum:	100.0
Range:	35.0

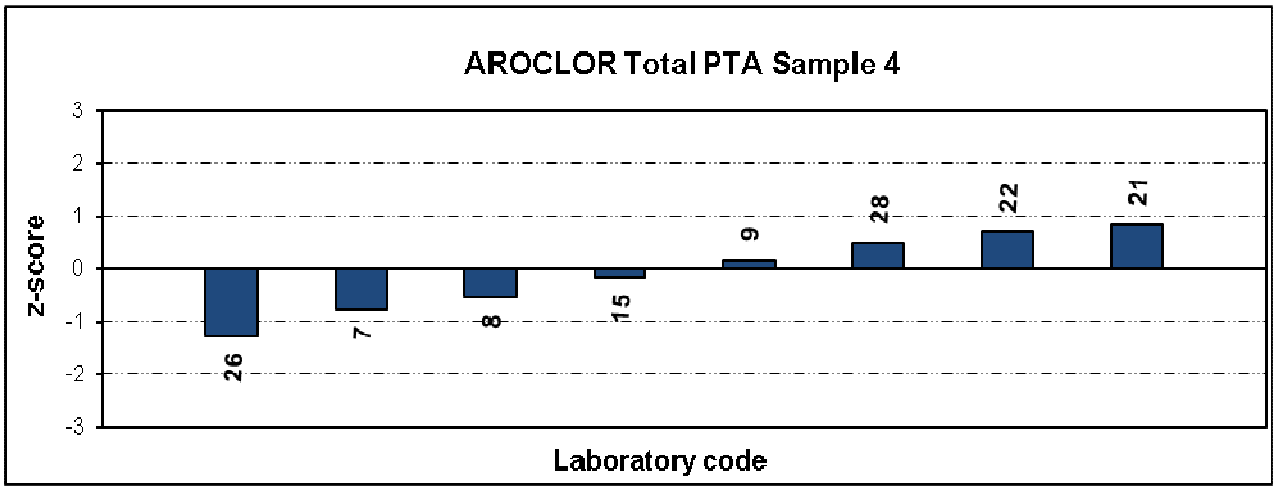
Note: NA indicates not applicable.



Aroclor Type Total							
Laboratory Code	PTA Sample 3			PTA Sample 4			Method
	Result		MU	Result		MU	Robust z-score	
7	<2			5.17	±	1.0	-0.76	Gas Chromatography / ECD
8	0.55	±	0.22	5.7	±	2.3	-0.53	Gas Chromatography / ECD
9	<1			7.3	±	2.19	0.15	Gas Chromatography / ECD
10	ND			NA			NA	Gas Chromatography / ECD
15	0.6	±	0.1	6.6	±	0.5	-0.15	Gas Chromatography / ECD
21	0.84	±	0.06	8.9	±	0.6	0.83	Gas Chromatography / ECD
22	NA			8.6	±	2.2	0.70	Gas Chromatography / ECD
26	NA			4.0	±	0.5	-1.26	Gas Chromatography / ECD
27	ND			NA			NA	Gas Chromatography / ECD
28	<2	±	<2	8.1	±	0.81	0.49	Gas Chromatography / ECD
29	ND			NA			NA	Gas Chromatography / ECD

No of Results:	6	8
Median:		6.95
Normalised IQR:		2.34
Robust CV:		33.7%
Uncertainty of median:		1.04
Minimum:		4.0
Maximum:		8.9
Range:		4.9

Notes: ND indicates not detected.
NA indicates not applicable.
No statistical analysis was conducted for Aroclor Total PTA Sample 3 as the majority of results were reported as "less than" values.



APPENDIX B

Homogeneity Testing

Homogeneity Testing

Sample preparation and homogeneity testing were conducted by BHP. Statistical analyses showed that all samples were sufficiently homogeneous so that any results later identified as outliers should not be attributed to any significant sample variability.

1. Sample Identification: Aroclor 1242

Test		Bottle number							
		1	2	3	4	5	6	7	8
1242	PTA 1 (54)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	PTA 2 (15)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	PTA 3 (1087)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	PTA 4 (742)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

2. Sample Identification: Aroclor 1248

Test		Bottle number							
		1	2	3	4	5	6	7	8
1248	PTA 1 (54)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	PTA 2 (15)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	PTA 3 (1087)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	PTA 4 (742)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

3. Sample Identification: Aroclor 1254

Test		Bottle number							
		1	2	3	4	5	6	7	8
1254	PTA 1 (54)	16.2	17.0	16.8	17.6	17.1	18.0	17.6	16.1
	PTA 2 (15)	23.5	14.1	21.0	18.6	27.6	21.7	17.8	20.6
	PTA 3 (1087)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	PTA 4 (742)	1.48	1.85	2.29	1.99	2.02	1.95	2.66	3.61

4. Sample Identification: Aroclor 1260

Test		Bottle number							
		1	2	3	4	5	6	7	8
1260	PTA 1 (54)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	PTA 2 (15)	75.3	53.0	61.1	61.0	55.7	65.0	63.6	62.2
	PTA 3 (1087)	0.73	0.85	0.91	1.10	0.80	1.01	0.83	0.87
	PTA 4 (742)	3.20	2.86	2.99	2.95	3.05	3.04	3.00	3.36

APPENDIX C

Documentation

Proficiency Testing Australia
Proficiency Testing Program

PCBs in Oil – Round 6
INSTRUCTIONS TO PARTICIPANTS

To ensure that all results from this program are analysed appropriately, participants are asked to read and adhere carefully to the following instructions:

1. Three oil samples containing Polychlorinated Biphenyls (PCBs) are supplied.

2. The samples are labelled:

   PTA Sample 1
   PTA Sample 2
   PTA Sample 3
   PTA Sample 4

Note: Due to shortage in sample numbers participants will receive only 3 samples. This could be any combination of PTA Sample 1, 2, 3 or 4.

3. The samples are to be tested for the Aroclor types listed on the results sheet, and the amounts of each Aroclor type quantified in mg/kg.

4. Tests are to be conducted using your routine method.

5. Results are to be recorded to the accuracy and reporting basis indicated on the *Results Sheet:*

Please do not leave empty boxes on the results sheet.

   i) results are to be reported in mg/kg, to at least two significant figures.
   ii) levels less than your level of detection may be reported as a "<" value.
   iii) for the AROCLOR types tested and not detected, report "Not Det".
   iv) for the AROCLOR types not tested, report "Not tested".

6. Laboratories are also requested to calculate and report an estimate of measurement uncertainty for each reported result. All estimates of uncertainty of measurement must be provided as a 95% confidence interval (coverage factor $k \approx 2$) and reported in mg/kg.

7. On the *Results Sheet* record the appropriate method code number:

   1. Gas Chromatography/Electron Capture Detection
   2. Gas Chromatography/Mass Spectrometric Detection
   3. Other (please specify)

6. Testing may commence as soon as samples are received. Please return results, **NO LATER THAN 29 April 2013,** to:

Laura Galbraith
Proficiency Testing Australia
PO Box 7507
SILVERWATER NSW 2128
AUSTRALIA
Phone: +612 9736 8397
Fax: +612 9743 6664
Email: laura.galbraith@pta.asn.au

PROFICIENCY TESTING AUSTRALIA
PCBs in Oil Round 6
Results Sheet

Lab Code: «Code»

RESULTS (mg/kg)	PTA Sample 1	±MU	PTA Sample 2	±MU	PTA Sample 3	±MU	PTA Sample 4	±MU
AROCLOR TYPE 1242								
AROCLOR TYPE 1248								
AROCLOR TYPE 1254								
AROCLOR TYPE 1260								
AROCLOR TYPE Other								
TOTAL PCBs								

Please do not leave empty boxes on the results sheet.

i) results are to be reported in mg/kg, to at least two significant figures.
ii) levels less than your level of detection may be reported as a "<" value.
iii) for the AROCLOR types tested and not detected, report "Not Det".
iv) for the AROCLOR types not tested, report "Not tested".

METHOD (Please tick):

□ Gas Chromatography/Electron Capture Detection □ Gas Chromatography/Mass Spectrometric Detection □ Other (please specify)

Print name: _____ Signature: _____ Date: _____

Please return by 29 April 201 to: Laura Galbraith Fax: 02 9743 6664 Email: laura.galbraith@pta.asn.au

- End of Report -