Report No. 1056

Waters Proficiency Testing Program

Round No. 220

- Total Recoverable Oil and Grease -

February 2018

Acknowledgments

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Dr M Buckley-Smith, Global Proficiency Ltd (New Zealand). Also our thanks go to Global Proficiency Pty Ltd (Australia) for the supply and distribution of the samples.

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CONTENTS

1. Foreword

This report summarises the results of a proficiency testing program on the determination of Total Recoverable Oil and Grease in waters. This is round 220 in a planned series of programs involving the analysis of chemical and physical parameters of waters.

The round was conducted in October/November 2017 by Proficiency Testing Australia (PTA). The main aim of the program was to assess laboratories' abilities to competently perform the prescribed analyses.

The Program Coordinator was Mrs K Cividin and the Technical Adviser was Dr M Buckley-Smith, Global Proficiency Ltd (New Zealand). This report was authorised by Mr P Briggs, General Manager.

2. Program Features and Design

2.1 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. Please note that a number of laboratories reported more than one set of results and, therefore, their code numbers (with letter) could appear several times in the same data set.

2.2 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.

2.3 Participants were provided with two glass vials (labelled PTA 1 and PTA 2) for analysis of Total Recoverable Oil and Grease.

2.4 A total of 31 laboratories received samples, comprising:

- 24 Australian participants; and
- 7 overseas participants, including:
    - Indonesia (1), Malaysia (4), New Zealand (1), Papua New Guinea (1).

Of these 31 laboratories, 5 were unable to submit results by the due date.

2.5 Results (as reported by participants) with corresponding summary statistics (i.e. number of results, median, normalised interquartile range, uncertainty of the median, robust coefficient of variation, minimum, maximum and range) are presented in Appendix A (for each sample and for each of the analyses performed).

2.6 A robust statistical approach, using z-scores, was utilised to assess laboratories' testing performance (see Section 3). Robust z-scores and ordered z-score charts relevant to each test are presented in Appendix A.

The document entitled *Guide to Proficiency Testing Australia,* 2016 (reference [1]) defines the statistical terms and details the statistical procedures referred to in this report.

2.7 A tabulated listing of laboratories (by code number) identified as having outlier results can be found on page 10.

2.8 Based on the quality control weight checks (see Appendix B) it was considered that the samples utilised for this program were homogeneous. As such, any results later identified as outliers could not be attributed to any notable sample variability.

3. Statistical Format

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

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

3.1 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 with an absolute value less than or equal to 2.0 is considered to be satisfactory, 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 "**§**". Laboratories are also encouraged to review results which have an absolute z-score value between 2.0 and 3.0 (i.e. $2.0 < |z\text{-score}| < 3.0$). These results are considered to be questionable results.

Each determination was examined for outliers with all methods pooled. The table on page 10 summarises the outlier results detected.

3.2 Results Tables and Summary Statistics

The tables in Appendix A contain 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/decimal places) 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 results tables and consists of:

- *No. of Results*: the total number of results for that test/sample;
- *Median*: the middle value of the results;
- *Normalised IQR*: the normalised interquartile range of the results;
- *Uncertainty of the Median*: a robust estimate of the standard deviation of the *Median*;
- *Robust CV*: the robust coefficient of variation expressed as a percentage, i.e. 100 x *Normalised IQR* / *Median*;
- *Minimum*: the lowest laboratory result;
- *Maximum*: the highest laboratory result; and
- *Range*: the difference between the *Maximum* and *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).

For normally distributed data, the uncertainty of the median is approximated by:

$$\sqrt{\frac{\pi}{2}} \times \frac{normIQR}{\sqrt{n}}$$

n = number of results

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

3.3 Ordered Z-score Charts

The charts in Appendix A indicate each laboratory's robust z-score, in order of magnitude, marked with its laboratory 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.

4. PTA and Technical Adviser's Comments

4.1 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. Please note that target CV's have been used to calculate the z-scores.

Sample preparation was undertaken according to Global Proficiency Ltd's Standard Operating Procedures to ensure samples were fit-for-purpose, homogeneous and stable. Weight checks were undertaken on all samples to ensure that the variability on doping concentrations was less than 3%. Dope concentrations presented in Table 1 illustrate the average oil and grease sample weight and the 95% confidence interval for weight variation at manufacturing, expressed as ± 2SD (standard deviations).

Sample PTA 1 was prepared from high purity mineral oil and stearic acid which has a low viscosity in distilled water. Sample PTA 2 was prepared from a mixture of vegetable oil, anhydrous milk fat (AMF) and stearic acid in distilled water. Samples were prepared as ready-to-test (500 mL) samples, packaged in Teflon capped amber glass bottles and preserved with acid to pH 2.

Solutions were stable and homogeneous, and medians obtained from this proficiency round indicated average recoveries of 75.2% for PTA 1 and 84.8% for PTA 2 compared to the expected levels (dope concentration), as shown in Table 1. These were much lower than published precision data, which indicated that laboratories should be able to yield an average recovery of 93% with the Liquid-Liquid Partition Gravimetric method (APHA 5520 B), recovery of 87.1% - 92.5% with the Solid Phase Partition Gravimetric method (APHA 5520 B), and recovery of 98.7% for Soxhlet Extraction method (APHA 5520 D).

Table 1. Comparison of expected levels (dope concentration) and proficiency medians. The values of the calculated uncertainty of the median are also presented.

Analysis	Sample	Dope Concentration ± 2SD (mg/L)	Median (mg/L)	Uncertainty of the median (mg/L)
Total Recoverable Oil and Grease	PTA 1	87.91 ± 1.25	66.10	2.73
	PTA 2	60.60 ± 1.81	51.40	2.02

As the assigned value for each sample in this program is the median of the results submitted by the participants, the uncertainty of the median for each sample has been calculated and is presented in the Table 1.

4.2 Analysis of Round 220 Results

Table 2 compares the Total Recoverable Oil and Grease medians and robust CVs from this round to those obtained in previous PTA rounds. Both samples contained sufficient oil and grease (>10 mg/sample) to enable testing using the gravimetric methods. CV's used in this study agreed well with published precision data from APHA 5520 G Solid Phase Partition Gravimetric method which indicated that CVs ranging between 17%-38% could be expected for various wastewater types. APHA indicated labs should achieve closer to 8.7% for Liquid-Liquid Partition Gravimetric method (APHA 5520 B) or 1.86% for Soxhlet Extraction method (APHA 5520 D).

Please note that all z-scores were calculated using a target *coefficient of variation* (CV).

Table 2. Comparison of current round variability and proficiency medians of Total Recoverable Oil and Grease testing with the results of the previous three rounds.

Round	Sample	Median (mg/L)	Robust CV (%)	Participants
This Round	PTA 1	66.10	17.1*	27
	PTA 2	51.40	15.7*	25
Report 1002	PTA 1	39.40	18.4	25
	PTA 2	76.30	9.9	25
Report 955	PTA 1	24.80	37.4	38
	PTA 2	77.50	9.9	37

*Indicates a target CV was used.

Bias / Accuracy

The Total Recoverable Oil and Grease testing was successfully performed, with satisfactory results (|z-score| ≤ 2.0) ranging between 45.5 – 83.0 mg/L for sample PTA 1 and 38.0 – 60.8 mg/L for sample PTA 2.

Out of the 27 results submitted for sample PTA 1, five questionable results (2.0 < |z-score| < 3.0) were reported (laboratories 137, 222, 291, 656b and 683). Out of the 25 results submitted for sample PTA 2, two questionable results were reported (laboratories 256 and 656b).

Six outlier results (|z-score| ≥ 3.0) were obtained for sample PTA 1, requiring follow-up action by laboratories 101, 260, 435, 555a, 555b and 717. However, due to the proximity of high biasing labs 101, 260 and 435 to the doping concentration of PTA 1 at 87.91 mg/L ± 1.25 mg/L (see Figures 1 & 3), these three labs may choose not to investigate their performance in this case. Six outlier results were obtained for sample PTA 2, requiring follow-up action by laboratories 291, 319, 509, 555b, 683 and 717.

Of more concern are the low biasing results, where laboratories recovered less than 50% of the oil and grease in the samples; particularly in the context of wastewater discharge limits, and fitness for purpose of test methods. Laboratories receiving questionable and outlier ratings on low biasing results are strongly recommended to investigate their ability to adequately recover all the oil and grease adhered to the surfaces of the glass bottle and Teflon lid liner (Laboratories 291, 319, 509, 555a, 555b, 656b, 683, 717). Laboratories using the Soxhlet Extraction Method (APHA

5520 D) most likely had difficulty with the instruction to “wipe the sides and bottom of collecting vessel… with pieces of filter paper soaked in extraction solvent, taking care to remove all films caused by grease and to collect all solid material”. Closer inspection of the original bottles will likely show a smear of fatty material still adhering to the side of the glass bottles. Laboratories using solvent partition gravimetric methods APHA 5520 B (LLE) and APHA 5520 G (SPE) may have had a similar problem with insufficient rinsing of the sample container with extracting solvent.

With respect to sample PTA 2 which contained a mixture of vegetable oil (61%), anhydrous milk fat (30%) and stearic acid (9%); the Soxhlet extraction method (APHA 5520 D) is known in the industry for under-recovering vegetable oil from wastewater samples. It has been noted that vegetable oil can pass through the filter paper when the wastewater sample is filtered prior to analysis.

On the other end of the scale, incomplete evaporation of the solvent can cause erroneously high results for labs using the Liquid-Liquid Partition Gravimetric method (APHA 5520 B). Cooling in a desiccator and weighing to a constant weight is critical for obtaining acceptable results with this method. Also, if the sample is dried with sodium sulphate, the salt that passes into the flask becomes a positive interference in gravimetric methods. See the interferences section (d) in APHA 5520 A for additional instructions if you observe crystals in the flask after drying.

Laboratories using NIR methods have the additional difficulty of not knowing what kind of wastewater they are analysing, and the inherent assumptions they must make when calibrating their equipment can cause significant error. As mentioned previously, sample PTA 1 contained high purity mineral oil and stearic acid, and sample PTA 2 contained a mixture of vegetable oil, anhydrous milk fat (AMF) and stearic acid.

In general, the Quality Control practices identified in Table 5020:I of APHA section 5000 should be considered an integral part of each method. The use of Method Blanks (MB), Laboratory Fortified Blanks (LFB), and Laboratory fortified matrix (LFM) with duplicates (LFMD), are mandatory for all Oil and Grease methods. APHA recommends that all of these QC practices (MB, LFB, LFM & LFMD) be carried out daily (as a minimum) or with each batch of 20 samples.

The datasets for samples PTA 1 and PTA 2 formed an approximately normal distribution (Figures 1 and 2) around the median. The method most frequently used for Total Recoverable Oil and Grease testing in this round was APHA 5520 B (Liquid-Liquid, Partition-Gravimetric Method), which was used by approximately 50% of participants for both samples PTA 1 and PTA 2. Six laboratories used NIR methods APHA 5520 C, ASTM D 7066-04 and HORIBA; four laboratories used the APHA 5520 G Solid Phase Partition Gravimetric method; and three laboratories used Soxhlet extraction method APHA 5520 D.

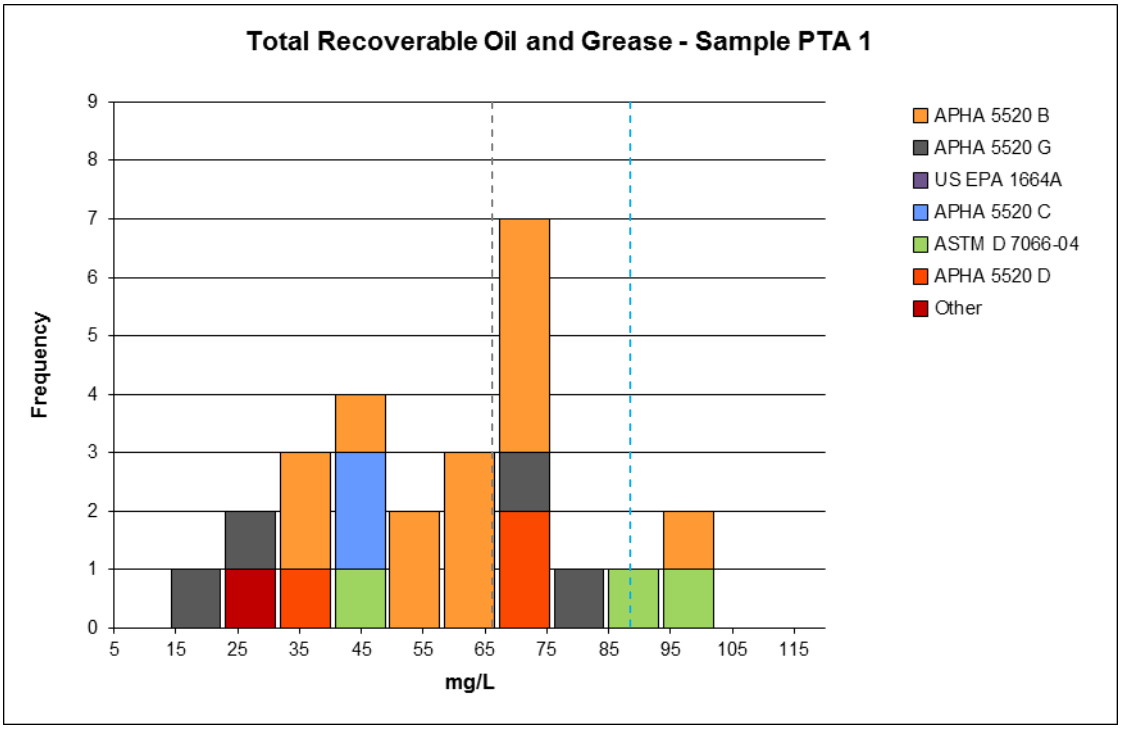


Figure 1. Spread of results for Total Recoverable Oil and Grease testing of sample PTA 1 with a median (- - -) of 66.10 mg/L and doping concentration (- - -) of 87.9 mg/L.

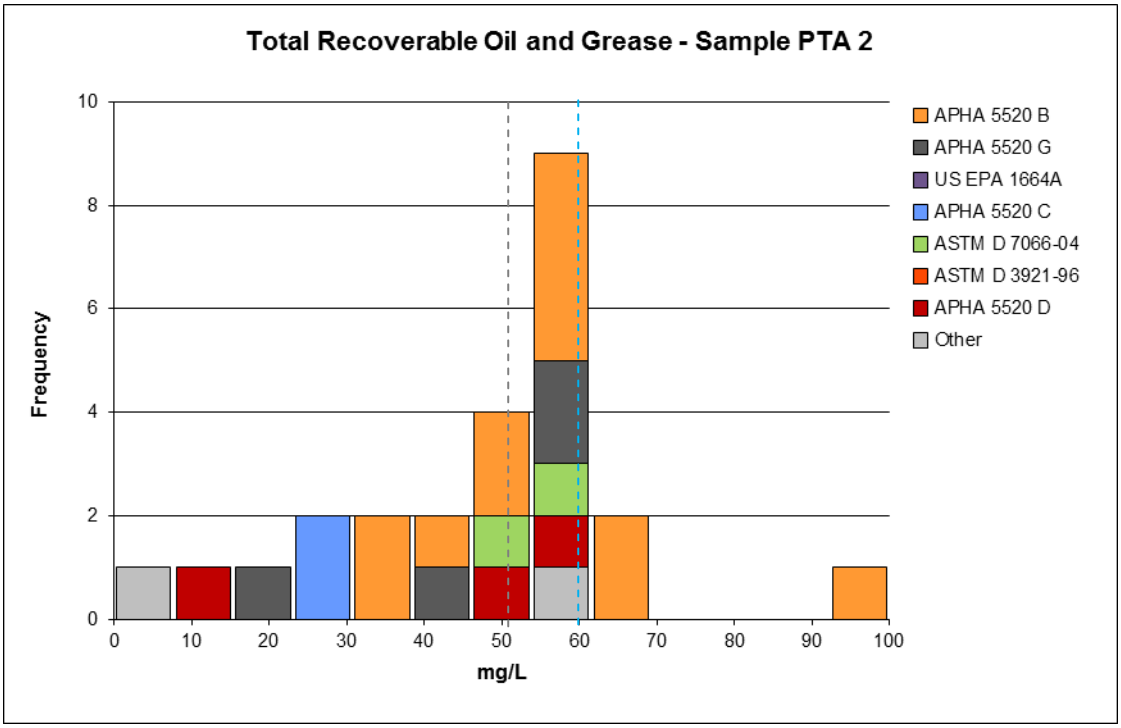


Figure 2. Spread of results for Total Recoverable Oil and Grease testing of sample PTA 2 with a median (- - -) of 51.40 mg/L and doping concentration (- - -) of 60.6 mg/L.

The majority of laboratories significantly underestimated their measurement uncertainty (MU), as can be seen in figures 3 & 4 where many laboratory results and their MU error bars do not encompass the median (- - -) or doping concentrations (——). These laboratories may have relied too heavily on their in-house repeatability

to estimate their measurement uncertainty, and may need to reassess their measurement uncertainty calculations; if they find over successive proficiency rounds, that their MU does not encompass the median or doping concentration.

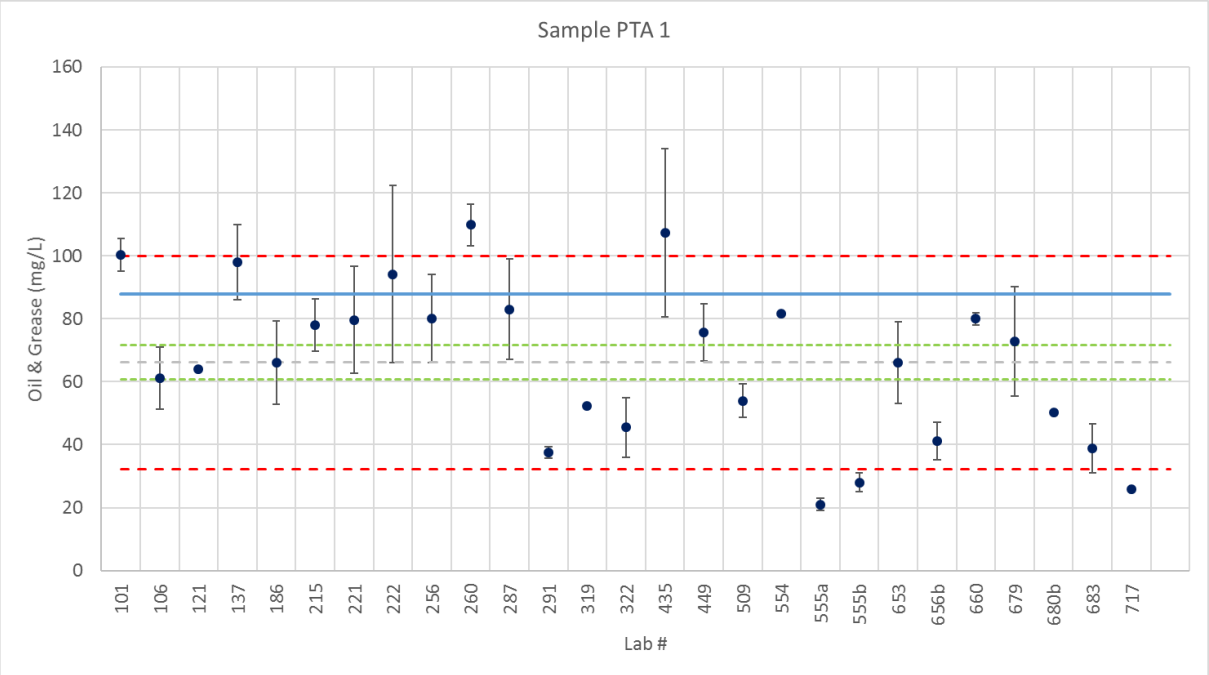


Figure 3. Spread of laboratory results (●) for Total Recoverable Oil and Grease testing of sample PTA 1, where laboratory MU show as error bars, the median is (- - -), the std error of median is (- - -), dope concentration is (——), and 3x NIQR is (- - -).

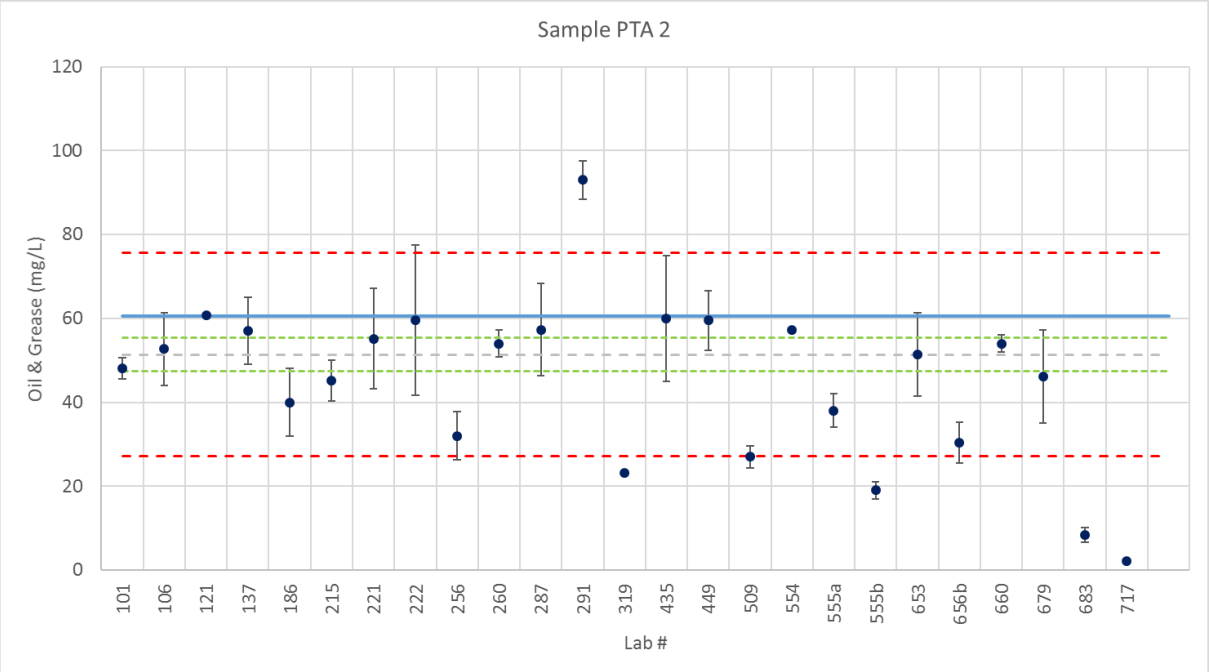


Figure 4. Spread of laboratory results (●) for Total Recoverable Oil and Grease testing of sample PTA 2, where laboratory MU show as error bars, the median is (- - -), the std error of median is (- - -), dope concentration is (——), and 3x NIQR is (- - -).

The importance of including data from QC samples comprising of spiked samples containing a variety of fats, oils and greases in a variety of wastewater matrices needs to be emphasised.

The majority of labs submitted MU values between 10% - 20%, however some laboratories gave MUs as small as 2.5% or as wide as 30%. An MU between 20%-30% may be a more realistic benchmark for laboratories to compare their in-house calculations with.

4.3 Measurement Uncertainty (MU)

The majority of participants in this round (88%) reported the measurement uncertainty (MU) associated with their results. Table 3 below presents the number and percentage of laboratories reporting the MU for each sample.

Table 3. The number and percentage of laboratories reporting a MU in round 220

Sample	Total participants	Participants reporting MU (percentage)
PTA 1	27	23 (85%)
PTA 2	25	22 (88%)

Many of the stated MUs did not accurately reflect the difference between the median and the participant's result for these proficiency samples.

Some laboratories may have under estimated their MU, as they indicated that their MU was less than two times the uncertainty of the median, however their results were further from the median than the MU value.[1]

4.4 Analysis of Results by Method Groups

Further analysis of results by method groups was undertaken to provide specific information on individual method performance.

In order for methods to be grouped for analysis, PTA requires at least 11 sets of results from the same method group. Please note that, for methods other than the one presented below, there were less than 11 results submitted for each method and reliable conclusions cannot be drawn from analysing them on this occasion.

The method APHA 5520 B (Liquid-Liquid, Partition-Gravimetric Method - method code 1), was most frequently employed for both samples.

Table 4 below presents the median and uncertainty of the median for results obtained by method 1. These results showed similarity to the overall data set.

Table 4. Variability and proficiency medians obtained by method 1.

Sample	Method code	Participants	Median ± Uncertainty of the Median (mg/L)	Robust CV (%)
PTA 1	1	13	66.10 ± 4.84	21.1
PTA 2	1	12	53.35 ± 3.57	18.5

5. Outlier Results

Laboratories reporting results that have been identified as outliers are listed in Table 3 below.

Table 4: Laboratory results identified as outliers for each sample tested.

Lab Code	Total Recoverable Oil and Grease	
	PTA 1	PTA 2
101	§	
260	§	
291		§
319		§
435	§	
509		§
555a	§	
555b	§	§
683		§
717	§	§

Note:
1. A "§" indicates the occurrence of a z-score outlier result (i.e. those results for which |z-score| ≥ 3.0).

6. References

[1] *Guide to Proficiency Testing Australia*, 2016 (This document can be found on the PTA website, www.pta.asn.au)

[2] Standard Methods for the Examination of Water and Wastewater, 2012. APHA, AWWA, WEF, 22nd Edition

APPENDIX A

Results and Data Analysis

Total Recoverable Oil and Grease Results

Samples PTA 1 and PTA 2

Total Recoverable Oil and Grease

Results by Laboratory Code

Lab Code	Sample PTA 1				
	Result ± MU[1] mg/L			Robust z-score[2]	Method Code [3]
101	100.4	±	5.2	3.04 §	5
106	61.2	±	9.92	-0.43	1
121	64		#	-0.19	1
137	98	±	12	2.82	#
186	66.0	±	13.2	-0.01	1
215	78.0	±	8.3	1.05	9
221	79.6	±	17	1.19	9
222	94.2	±	28.2	2.49	2
256	80.0	±	14	1.23	1
260	109.9	±	6.6	3.88 §	5
287	83.0	±	16.0	1.50	1
291	37.5	±	1.9	-2.53	1
319	52.3		#	-1.22	4
322	45.5	±	9.5	-1.82	5
435	107.3	±	26.8	3.65 §	1
449	75.6	±	9.1	0.84	2
509	54.0	±	5.4	-1.07	4
554	81.6		#	1.37	1
555a	21.0	±	2.0	-3.99 §	2
555b	28	±	3.0	-3.37 §	2
653	66.1	±	13.0	0.00	1
656b	41.1	±	6.0	-2.21	1
660	80	±	2	1.23	1

[1] Where reported, results are shown with their corresponding measurement uncertainty (MU).

[2] "**§**" denotes an outlier (i.e. those results for which |*z-score*| ≥ 3.0). Robust *z-scores* are calculated *as:* $z = (A - median) \div normalised\ IQR$, where *A* is the participant laboratory's result.

[3] Please refer to Appendix C (page C3) for method code descriptions.

Total Recoverable Oil and Grease

Results by Laboratory Code

Lab Code	Sample PTA 1 Result ± MU[1] mg/L			Robust z-score[2]	Method Code [3]
679	72.8	±	17.47	0.59	1
680b	50.2		#	-1.41	1
683	38.8	±	7.8	-2.42	9
717	25.8	±	2.3	-3.57 **§**	10

No of Results:	27
Median:	66.10
Normalised IQR:	11.30
Uncertainty of the Median:	2.73
Robust CV:	17.1%
Minimum:	21.0
Maximum:	109.9
Range:	88.9

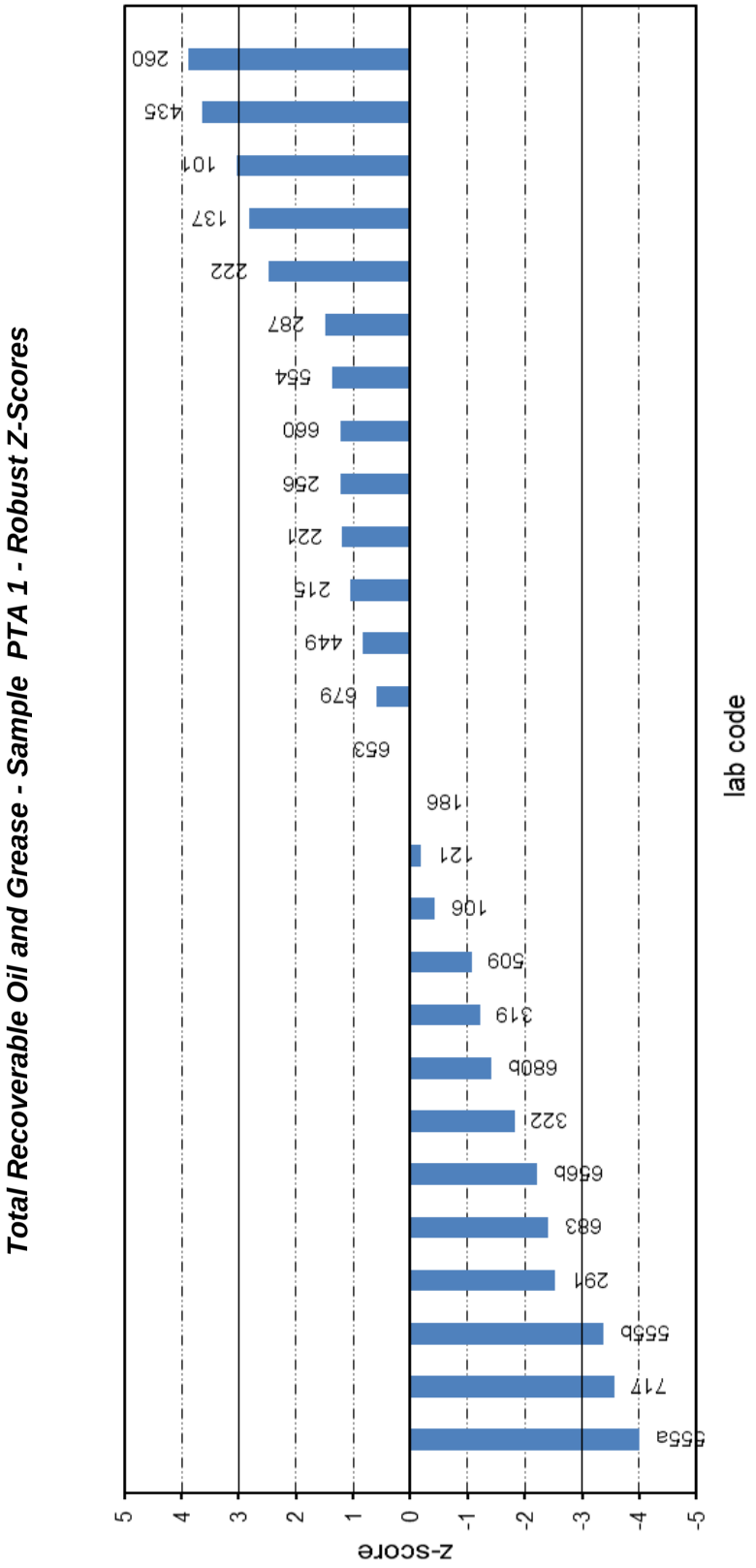
[1] Where reported, results are shown with their corresponding measurement uncertainty (MU).

[2] "**§**" denotes an outlier (i.e. those results for which |*z-score*| ≥ 3.0). Robust *z-scores* are calculated *as:* $z = (A - median) \div normalised\ IQR$, where *A* is the participant laboratory's result.

[3] Please refer to Appendix C (page C3) for method code descriptions.

Total Recoverable Oil and Grease - Sample PTA 1

Ordered Robust Z-Score Charts



Total Recoverable Oil and Grease

Results by Laboratory Code

Lab Code	Sample PTA 2			
	Result ± MU[1] mg/L		Robust z-score[2]	Method Code [3]
101	48.1	± 2.5	-0.41	5
106	52.7	± 8.6	0.16	1
121	60.8	#	1.17	1
137	57	± 8	0.70	#
186	40	± 8	-1.41	1
215	45.2	± 4.8	-0.77	9
221	55.2	± 12	0.47	9
222	59.6	± 17.9	1.02	2
256	32.0	± 5.7	-2.41	1
260	54.0	± 3.2	0.32	5
287	57.3	± 11.0	0.73	1
291	93.0	± 4.6	5.16 **§**	1
319	23.1	#	-3.51 **§**	4
435	60.0	15.0	1.07	1
449	59.5	± 7.1	1.01	2
509	27.0	± 2.7	-3.03 **§**	4
554	57.2	± #	0.72	1
555a	38.0	4.0	-1.66	2
555b	19	± 2.0	-4.02 **§**	2
653	51.4	± 10.0	0.00	1
656b	30.4	± 4.8	-2.61	1

[1] Where reported, results are shown with their corresponding measurement uncertainty (MU).

[2] "**§**" denotes an outlier (i.e. those results for which |*z-score*| ≥ 3.0). Robust *z-scores* are calculated *as:* $z = (A - median) \div normalised\ IQR$, where *A* is the participant laboratory's result.

[3] Please refer to Appendix C (page C3) for method code descriptions.

Total Recoverable Oil and Grease

Results by Laboratory Code

Lab Code	Sample PTA 2 Result ± MU[1] mg/L	Robust z-score[2]	Method Code [3]
660	54 ± 2	0.32	1
679	46.2 ± 11.09	-0.65	1
683	8.4 ± 1.7	-5.34 §	9
717	2.1 0.2	-6.12 §	10

No of Results:	25
Median:	51.40
Normalised IQR:	8.06
Uncertainty of the Median:	2.02
Robust CV:	15.7%
Minimum:	2.1
Maximum:	93.0
Range:	90.9

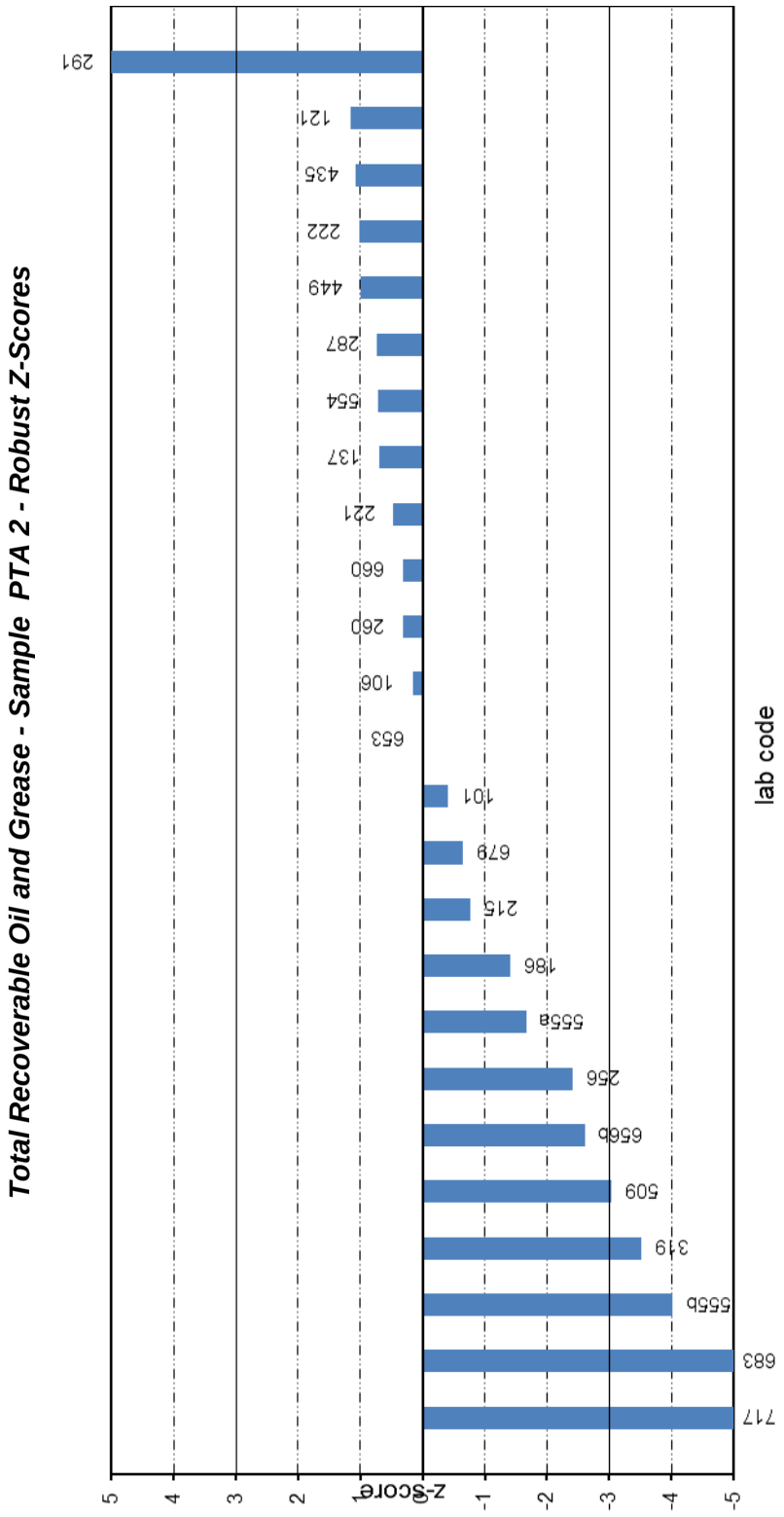
[1] Where reported, results are shown with their corresponding measurement uncertainty (MU).

[2] "**§**" denotes an outlier (i.e. those results for which |*z-score*| ≥ 3.0). Robust *z-scores* are calculated *as:* $z = (A - median) \div normalised\ IQR$, where *A* is the participant laboratory's result.

[3] Please refer to Appendix C (page C3) for method code descriptions.

Total Recoverable Oil and Grease - Sample PTA 2

Ordered Robust Z-Score Charts



APPENDIX B

Sample Homogeneity and Stability

Homogeneity and Stability Testing

Samples for this program were obtained from Global Proficiency Ltd, New Zealand. As such, all samples are subjected to rigorous quality control and homogeneity / stability testing. All samples underwent quality control weight checks to ensure a consistent mass of oil and grease was delivered to each bottle. Sample PTA 1 was doped with 43.95 ± 0.63 mg (95% CI) of mineral oil and stearic acid per 500mL sample bottle, and sample PTA 2 was doped with 30.3 ± 0.91 mg (95% CI) of a mixture of vegetable oil, anhydrous milk fat and stearic acid per 500mL sample bottle. All samples were acidified to pH < 2 using H_2SO_4. Results are displayed in Table B1.

From statistical analysis based on the results of the quality control weight checks and reliable historical data, it was considered that all samples were sufficiently homogenous and stable, so that any results later identified as outliers should not be attributed to any notable sample variability.

Table B1. Homogeneity weight checks during sample manufacture.

PTA 1		**PTA 1**		**PTA 2**		**PTA 2**	
No.	Wgt (mg)	No.	Wgt (mg)	No.	Wgt (mg)	No.	Wgt (mg)
1	44.0	38	44.8	1	29.9	38	29.8
2	43.7	39	43.7	2	30.4	39	29.6
3	44.0	40	43.5	3	31.1	40	30.0
4	44.3	41	43.2	4	29.4	41	30.0
5	44.0	42	43.6	5	29.9	42	29.8
6	44.1	43	44.1	6	30.2	43	31.2
7	43.9	44	43.6	7	30.5	44	30.7
8	44.0	45	43.4	8	30.2	45	30.5
9	43.9	46	43.9	9	29.6	46	30.8
10	43.9	47	44.3	10	30.7	47	30.1
11	44.5	48	44.2	11	30.4	48	30.7
12	44.0	49	44.2	12	30.5	49	30.4
13	44.4	50	43.6	13	30.5	50	30.5
14	43.9	51	43.9	14	30.6	51	30.3
15	43.6	52	43.6	15	30.7	52	30.5
16	43.9	53	44.5	16	30.0	53	30.6
17	43.9			17	30.5		
18	44.1			18	30.7		
19	43.8			19	30.8		
20	44.1			20	31.0		
21	43.8			21	30.6		
22	44.0			22	30.0		
23	44.3			23	30.0		
24	43.9			24	29.8		
25	44.1			25	30.9		
26	43.7			26	30.2		
27	43.9			27	30.0		
28	44.0			28	29.5		
29	43.8		Homogeneity	29	29.9		Homogeneity
30	44.2		Stability	30	30.1		Stability
31	43.7	**Mean**	**43.95283**	31	29.1	**Mean**	**30.30000**
32	44.1	**Stdev**	**0.31293**	32	30.2	**Stdev**	**0.45362**
33	44.5	**CV**	**0.71%**	33	30.5	**CV**	**1.50%**
34	43.5			34	30.7		
35	44.2			35	30.9		
36	43.5			36	30.5		
37	44.2			37	29.9		

APPENDIX C

Documentation

PROFICIENCY TESTING AUSTRALIA

WATERS PROFICIENCY TESTING PROGRAM

CHEMICAL ANALYSIS ROUND 220 **OCTOBER, 2017**

Total Recoverable Oil and Grease

INSTRUCTIONS TO PARTICIPANTS

*****Please record (on the Results Sheet) the approximate temperature of the samples upon receipt*****

Please note the following before commencing the analysis of the samples.

1. Samples

i) Two 500 mL glass bottles, labelled PTA 1 and PTA 2, supplied by Global Proficiency Ltd. The bottles contain oil and grease (in water) in the range of 10-50 milligrams (mg). These have been acid preserved and should be refrigerated until ready to test.

ii) The sample must be thoroughly mixed prior to analysis.

Please Note: Where possible, proficiency testing samples should be treated as a routine laboratory sample.

2. Sample Preparation

Caution: Analysis must begin immediately after bottle is opened.

i) Adjust bottle temperature to 20º C.

ii) Record bottle ID number.

iii) Shake the bottle prior to opening.

iv) Each bottle is ready to test according to your normal procedures.

Note: Please treat as “unknown oil”.

3. Tests Requested

i) Total Recoverable Oil and Grease.

If unable to perform the above please note this on your Results Sheet.

4. Safety

i) Samples are for laboratory use only.

ii) Participants should have sufficient experience and training to take the necessary precautions when handling the samples and reagent chemicals and during disposal.

iii) Use of safety glasses, gloves, and fume hoods, where appropriate during the determinations, is recommended.

5. Reporting

i) Report results using one decimal place.

ii) Report results in milligrams per litre (mg/L).

iii) Do not correct results for recovery.

iv) Select the appropriate method code from the Method Code Table and record it on the Results Sheet.

v) Calculate the measurement uncertainty (MU) for each reported result. All estimates of MU must be given as a 95% confidence interval (coverage factor k ≈ 2) and reported in mg/L. Report MU using the same number of decimal places as for the result.

6. Testing should commence as soon as possible after receiving the samples and results reported **NO LATER THAN 10 NOVEMBER 2017** to:

Karen Cividin
Proficiency Testing Australia
PO Box 7507
SILVERWATER NSW 2128
AUSTRALIA
Phone: +612 9736 8397
Fax: +612 9743 6664
Email: kcividin@pta.asn.au

7. 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.

8. As a guide, ranges for the samples can be expected to be (in mg/L):

Analyte	Range
Total Recoverable Oil and Grease	10 – 100 mg/L

Method Codes to be used for the Results Sheet

ANALYSIS	METHOD	METHOD DESCRIPTION	CODE
Total Recoverable Oil and Grease	Partition-Gravimetric	APHA 5520 B. Liquid-Liquid, Partition-Gravimetric Method	1
		APHA 5520 G. Solid-Phase, Partition-Gravimetric Method	2
	Hexane Extraction & Gravimetry	US EPA 1664A Oil & Grease (HEM/SGT-HEM) by extraction	3
	Infrared	APHA 5520 C. Partition-Infrared Method	4
		ASTM D 7066-04 Standard Test Method for dimer/trimer of chlorotrifluoroethylene (S-316) Recoverable Oil and Grease and Nonpolar Material by Infrared Determination	5
		ASTM D 3921-96 Standard Test Method for Oil and Grease and Petroleum Hydrocarbons in Water	6
		US EPA 413.2 Oil & Grease, Total Recoverable - Spectrophotometric, Infrared)	7
		US EPA 418.1 Petroleum Hydrocarbons, Total Recoverable	8
	Soxhlet Extraction	APHA 5520 D. Soxhlet Extraction Method	9
	Other	Please specify	10

Method Reference Key

i) APHA APHA “Standard Methods for the Examination of Water and Wastewater” (18, 19, 20, 21, 22 Edition) (http://www.standardmethods.org/).

ii) ASTM American Society for Testing and Materials, Annual Book of ASTM Standards, Vol. 11.01 and Vol. 11.02.

iii) US EPA U.S Environmental Protection Agency. (http://www.epa.gov/osa).

PROFICIENCY TESTING AUSTRALIA

WATERS PROFICIENCY TESTING PROGRAM

CHEMICAL ANALYSIS ROUND 220

Total Recoverable Oil and Grease

OCTOBER, 2017

RESULTS SHEET
(mg/L)

Laboratory Code	«Code»

*Approximate temperature of samples upon receipt:	

ANALYSIS	**SAMPLE PTA 1**		**SAMPLE PTA 2**		**METHOD CODE**
	Result (mg/L)	**±MU (mg/L)**	**Result (mg/L)**	**±MU (mg/L)**	
Total Recoverable Oil and Grease					

Please note: Where possible, proficiency testing samples should be treated as a routine laboratory sample.

i) For each sample only a single result is requested.

ii) Report results using one decimal place.

iii) Report results in milligrams per litre (mg/L) - i.e., the total mg extracted/sample treated as 1L for reporting.

iv) Do not correct results for recovery.

v) MU* Laboratories Measurement Uncertainty (MU) if known for the result.
Please report in mg/L and use a coverage factor of k=2.

DATE: _____ **SIGNATURE:** _____

Return results **NO LATER THAN 10 NOVEMBER 2017** to:

Karen Cividin
Proficiency Testing Australia
PO Box 7507
SILVERWATER NSW 2128
AUSTRALIA

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

- End of Report -