Results of Proficiency Test
Gear Oil (used)
April 2018

CONTENTS

Appendices:

1   INTRODUCTION

Since 2017, the Institute for Laboratory Studies (iis) organizes a proficiency test for used Gear Oil. During the annual proficiency testing program 2017/2018, it was decided to continue the proficiency test for the analysis of used Gear Oil.
In this interlaboratory study, 24 laboratories in 18 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2018 used Gear Oil proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send 0.5L for the main round (labelled #18036) and 50mL for metals determination only (labelled #18037).
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1   ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2   PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol can be downloaded from the iis website www.iisnl.com, from the FAQ page.

2.3   CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary bulk material for the main round, approximately 30 litre, of used Gear Oil was obtained from a local supplier. After homogenisation, 49 amber glass bottles of 0.5L were filled and labelled #18036. The homogeneity of the subsamples #18036 was checked by determination of Density at 15°C in accordance with ASTM D4052 and Water according to ASTM D6304-C on 8 stratified randomly selected samples.

	Density at 15 °C in kg/m³	Water in mg/kg
Sample #18036-1	891.49	162
Sample #18036-2	891.49	156
Sample #18036-3	891.48	168
Sample #18036-4	891.47	162
Sample #18036-5	891.47	166
Sample #18036-6	891.48	159
Sample #18036-7	891.47	162
Sample #18036-8	891.47	152

Table 1: homogeneity test results of subsamples #18036

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the reference test methods in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 15 °C in kg/m³	Water in mg/kg
r (observed)	0.02	15
reference test method	ASTM D4052:18	ASTM D6304:16e1
0.3 * R (ref. test method)	0.15	107

Table 2: repeatabilities of subsamples #18036

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

For the preparation of the metals sample #18037 approximately 2 litre was taken from the batch of used Gear oil used for the main sample and spiked with various Conostan standards with the metals listed in table 3.

Component	amount in mg/kg
Aluminium	16.19
Copper	12.80
Silicon	12.14
Tin	14.61

Table 3: components that were added to bulk material for sample #18037

After homogenisation, 38 amber glass bottles of 50mL were filled and labelled #18037. The homogeneity of the subsamples #18037 was checked by determination of Copper as Cu and Iron as Fe in accordance with ASTM D5185 on 8 stratified randomly selected samples.

	Copper as Cu in mg/kg	Iron as Fe in mg/kg
Sample #18037-1	16	189
Sample #18037-2	16	190
Sample #18037-3	16	189
Sample #18037-4	16	190
Sample #18037-5	16	186
Sample #18037-6	16	189
Sample #18037-7	16	188
Sample #18037-8	16	188

Table 4: homogeneity test results of subsamples #18037

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the reference test methods in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Copper as Cu in mg/kg	Iron as Fe in mg/kg
r (observed)	0.00	3.6
reference test method	ASTM D5185:18	ASTM D5185:18
0.3 * R (ref. test method)	1.15	10

Table 5: repeatabilities of subsamples #18037

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories, 1* 0.5L bottle, labelled #18036 and 1* 50mL bottle, labelled #18037 was sent on March 14, 2018. An SDS as added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of used Gear Oil packed in amber glass bottle was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine on sample #18036: Total Acid Number, Density at 15°C, Flash Point PMcc, Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Membrane Filtration 5µm, Water content by KF and Level of Contamination (counts/ml and scale number) and to determine on sample #18037: 24 elements (wear metals and additives).

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the results, but report as much significant figures as possible. It was also requested not to report 'less than' test results for test results which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared.

On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3   RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyses). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1   STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.
First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2   GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3   Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

This target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated in accordance with:

$z_{(target)}$ = (test result - average of PT) / target standard deviation

The $z_{(target)}$ scores are listed in the result tables of appendix 1.
Absolute values for z<2 are very common and absolute values for z>3 are very rare.

Therefore, the usual interpretation of z-scores is as follows:

$$|z| < 1 \quad \text{good}$$
$$1 < |z| < 2 \quad \text{satisfactory}$$
$$2 < |z| < 3 \quad \text{questionable}$$
$$3 < |z| \quad \text{unsatisfactory}$$

4   EVALUATION

In this proficiency test, some problems were encountered with the dispatch of the samples.
One participant reported after the final reporting date and two participants did not report any test results at all. Not all laboratories were able to report all analyses requested.
In total 22 participants reported 518 numerical test results. Of all reported numerical test results 391 numerical test results were statistically evaluated. Observed were 18 outlying test results, which is 4.6% of the statistically evaluated numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1   EVALUATION PER SAMPLE AND PER TEST

In this section, the reported test results are discussed per sample and per test.
The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported test results. The abbreviations, used in these tables, are listed in appendix 5.

Unfortunately, a suitable standard test method providing the precision data, is not available for all determinations. For the tests, that have no available precision data, the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

In the iis PT reports, ASTM test methods are referred to with a number (e.g. D7647) and an added designation for the year that the test method was adopted or revised (e.g. D7647:10). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D7647:10(2018)). In the test results tables of appendix 1 only the test method number and year of adoption or revision e.g. D7647:10 will be used.

Sample #18036

Acid Number (Total): This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D664-A:17a. No clear effect was observed between the different modes (determination of end point and volume of used in titration).

Density at 15°C: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D4052:18.

Flash Point PMcc: Both methods (A and B) of ASTM D93 may be applicable for this determination (in-use vs used lubricating oil). The majority of the participants used method A, only a few participants used method B (16 vs 3 participants respectively).
This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D93-A:16a.

Kin.Visco.at 40°C: This determination was not problematic. No statistical outliers were observed, but one test result was excluded from the statistical evaluation. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D445:17a.

Kin.Visco.at 100°C: This determination was very problematic. No statistical outliers were observed, but one test result was excluded from the statistical evaluation. The results vary over a broad range (15.17 – 19.51 mm^2/s). Therefore, it was decided not to calculate z-scores. No clear reason could be found for the variation in the test results. A possibility could be that the used Gear oil was heavily oxidized.

Viscosity Index: This determination was very problematic. No statistical outliers were observed. The Viscosity Index is a calculation in which the Kinematic Viscosity at 100°C is included. Therefore, also the results of Viscosity Index vary over a broad range (105 – 151) and it was decided not to calculate z-scores for this parameter as well.
Also, iis calculated the Viscosity Index from the test results reported for the Kinematic Viscosity as 40°C and 100°C. No calculation errors were observed.

Membrane Filtration 5µm: Only four participants reported a test result. Therefore no significant conclusions were drawn.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6304:16e1.

Level of Contamination: This determination was problematic. In total nine statistical outliers were observed over six parameters (7 in counts per ml and 2 in scale number) and twelve test results were excluded. The calculated reproducibilities after rejection of the suspect data is not in agreement with the requirements of ASTM D7647:10(2018). No clear conclusion could be drawn from the reported analytical details (see appendix 3).

Sample #18037

Aluminium: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:18.
The average recovery of Aluminium is good (<94%), the actual blank Aluminium content is unknown.

Boron: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D5185:18.

Chromium: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:18.

Copper: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:18.
The average recovery of Copper is good (<124%), the actual blank Copper content is unknown.

Iron: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5185:18.

Silicon: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:18.
The average recovery of Silicon is good (<104%), the actual blank Silicon content is unknown.

Tin: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5185:18.
The average recovery of Tin is good (<95%), the actual blank Tin content is unknown.

Calcium: This determination may be problematic at a level of 2.40 mg/kg. One statistical outlier was observed. The calculated reproducibility is not in agreement with the estimated reproducibility using the Horwitz equation. And is also not in agreement with the requirements of ASTM D5185:18 (application range 40 - 9000 mg/kg) and not in agreement with the requirements of ASTM D6595:17 (application range 3.7 - 11460 mg/kg).

Phosphorus: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:18.

Zinc: This determination is not problematic at a level of 2.69 mg/kg depending on the target method used. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility using the Horwitz equation. And is also in agreement with the requirements of ASTM D6595:17 (application range 5.3 - 1345 mg/kg), but is not at all in agreement with the requirements of ASTM D5185:18 (application range 60 - 1600 mg/kg).

4.2   PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities derived from literature reference test methods (in casu ASTM standards) are compared in the next tables.

Parameter	unit	n	average	2.8 * sd	R(target)
Acid Number (Total)	mg KOH/g	21	0.55	0.33	0.25
Density at 15°C	kg/m^3	18	891.5	0.7	0.5
Flash Point PMcc	°C	18	192.0	18.3	13.6
Kinematic Viscosity at 40°C	mm^2/s	19	147.01	1.77	3.21
Kinematic Viscosity at 100°C	mm^2/s	15	17.075	3.849	(0.214)
Viscosity Index		14	124.72	43.56	(2)
Membrane Filtration 5µm	%M/M	4	n.a.	n.a.	n.a.
Water	mg/kg	19	190	150	393
Level of contamination					
- ≥ 4µm (c)	counts/ml	6	266	471	301
- ≥ 6µm (c)	counts/ml	6	78	89	59
- ≥14µm (c)	counts/ml	6	12	28	16
- ≥ 4µm (c)	scale number	7	15.1	2.5	1.7
- ≥ 6µm (c)	scale number	7	13.4	2.2	1.2
- ≥14µm (c)	scale number	7	10.3	2.7	2

Table 6: reproducibilities of test results of sample #18036.

Reproducibility between brackets should be used with care (see paragraph 4.1).

Parameter	unit	n	average	2.8 * sd	R(target)
Aluminium as Al	mg/kg	21	15.3	4.0	7.7
Boron as B	mg/kg	18	17.2	7.5	13.4
Chromium as Cr	mg/kg	19	2.0	0.6	1.2
Copper as Cu	mg/kg	18	15.9	2.4	3.8
Iron as Fe	mg/kg	20	189	30	34
Silicon as Si	mg/kg	21	12.6	3.2	7.8
Tin as Sn	mg/kg	21	13.9	7.3	10.8
Calcium as Ca	mg/kg	16	2.4	1.3	0.9
Phosphorus as P	mg/kg	19	306	38	75
Zinc as Zn	mg/kg	19	2.7	1.1	1.0

Table 7: reproducibilities of test results of sample #18037.

Without further statistical calculations, it could be concluded that for a number of tests there is a good compliance of the group of participating laboratories with the relevant reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3   COMPARISON OF THE PROFICIENCY TEST OF APRIL 2018 WITH PREVIOUS PT

	April 2018	April 2017
Number of reporting labs	22	17
Number of results reported	391	362
Statistical outliers	18	23
Percentage outliers	4.6%	6.4%

Table 8: comparison with previous proficiency test

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the respective reference test methods. The conclusions are given in the following table:

	April 2018	April 2017
Acid Number (Total)	-	-
Density at 15°C	-	-
Flash Point PMcc	-	+
Kinematic Viscosity at 40°C	+	++
Kinematic Viscosity at 100°C	--	+
Viscosity Index	--	+
Membrane Filtration 5µm	n.e.	n.e.
Water	++	++
Level of contamination		
counts/ml (≥4 / ≥6 / ≥14µm)	-	--
scale number (≥4 / ≥6 / ≥14µm)	-	--

	April 2018	April 2017
Aluminium as Al	+	n.e.
Antimony as Sb	n.e.	n.e.
Barium as Ba	n.e.	n.e.
Boron as B	+	+
Cadmium as Cd	n.e.	n.e.
Chromium as Cr	++	n.e.
Copper as Cu	+	(-)
Iron as Fe	+	+
Lead as Pb	n.e.	n.e.
Lithium as Li	n.e.	n.e.
Magnesium as Mg	n.e.	n.e.
Manganese as Mn	n.e.	n.e.
Molybdenum as Mo	n.e.	n.e.
Nickel as Ni	n.e.	n.e.
Potassium as K	n.e.	n.e.
Silicon as Si	++	n.e.
Silver as Ag	n.e.	n.e.
Sodium as Na	n.e.	n.e.
Tin as Sn	+	n.e.
Titanium as Ti	n.e.	n.e.
Vanadium as V	n.e.	n.e.
Calcium as Ca	-	(-)
Phosphorus as P	++	+
Zinc as Zn	+/-	(-)

Table 9: comparison against the requirements of the reference test methods

*) Consensus values between brackets were close or below the limit of detection

The performance of the determinations against the requirements of the respective reference test methods is listed in the above table. The following performance categories were used:

- ++:   group performed much better than the reference test method
- + :   group performed better than the reference test method
- +/-:   group performance equals the reference test method
- - :   group performed worse than the reference test method
- -- :   group performed much worse than the reference test method
- n.e.:   not evaluated

APPENDIX 1

Determination of Acid Number (Total) on sample #18036; results in mg KOH/g.

lab	method	value	mark	z(targ)	remarks	Determination of end point	Volume of titration solvent
178	INH-1118	0.41		-1.58		Inflection Point	---
179	D664-B	0.44		-1.25		Inflection Point	60 mL
237	D664-A	0.263		-3.21		Inflection Point	125 mL
325	D664-A	0.58		0.30		Buffer End Point (pH 11)	125 mL
331	D7279Mod.	0.67		1.29		Inflection Point	---
349	D664-A	0.67	C	1.29	first reported 0.87	Inflection Point	125 mL
451	In house	0.72		1.85		Buffer End Point (pH 11)	60 mL
496	D664-A	0.51		-0.48		Buffer End Point (pH 11)	60 mL
575	D664-A	0.53		-0.26		Inflection Point	60 mL
633	D664-A	0.80		2.73		Inflection Point	125 mL
663	D664-A	0.516		-0.41		Buffer End Point (pH 11)	125 mL
862	D664-A	0.6431		1.00		---	---
902	D664-A	0.52		-0.37		Inflection Point	60 mL
962	D974	0.58		0.30		---	---
963	D664-A	0.52		-0.37		Inflection Point	60 mL
974	D664-A	0.45		-1.14		Inflection Point	125 mL
1026	D664-A	0.63		0.85		Buffer End Point (pH 11)	60 mL
1146	D664-A	0.556		0.03		Buffer End Point (pH 11)	125 mL
1201	D664-A	0.60		0.52		Buffer End Point (pH 11)	125 mL
1435	D664-A	0.488		-0.72		Buffer End Point (pH 11)	---
1743	D664-A	0.52		-0.37		Buffer End Point (pH 11)	60 mL
1748		-----		-----		---	---
6016		-----		-----		---	---
6054		-----		-----		---	---

						BEP (pH 11) only	Inflection point only
normality	suspect					not OK	OK
n	21					9	10
outliers	0					0	0
mean (n)	0.5531					0.5689	0.5273
st.dev. (n)	0.11687					0.07334	0.15363
R(calc.)	0.3272					0.2053	0.4302
st.dev.(D664-A:17a)	0.09036					0.09286	0.08624
R(D664-A:17a)	0.2530	BEP (pH 11) – 60 mL				0.2600	
compare							
R(D664-A:17a)	0.2475	Inflection – 60 mL					0.2415

Kernel Density
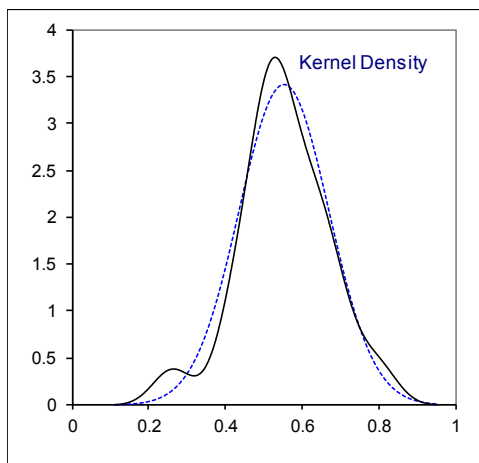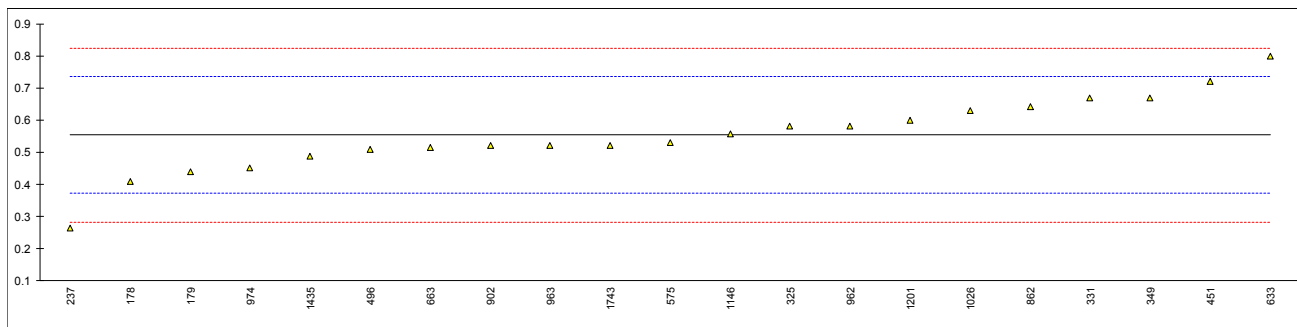
Determination of Density at 15°C on sample #18036; results in kg/m³.

lab	method	value	mark	z(targ)	remarks
178	D4052	892.5	R(0.05)	5.36	
179	D4052	891.6		0.32	
237	D4052	891.9		2.00	
325	D4052	891.54		-0.02	
331	ISO12185	891.75		1.16	
349	D4052	891.5		-0.24	
451	In house	892		2.56	
496	D4052	891.01	C	-2.99	first reported 887.55
575		-----		-----	
633		-----		-----	
663	D4052	891.48	C	-0.35	first reported 887.94
862	D4052	891.6		0.32	
902	D4052	891.50		-0.24	
962	D4052	891.9		2.00	
963	D4052	891.5		-0.24	
974	D4052	891.5		-0.24	
1026	D4052	891.5		-0.24	
1146	D4052	891.50	C	-0.24	first reported 861.5
1201	D4052	891.4		-0.80	
1435	D4052	891.1	C	-2.48	first reported 0.8911 kg/m³
1743	ISO12185	893.5	R(0.01)	10.96	
1748	D4052	891.5		-0.24	
6016		-----		-----	
6054		-----		-----	

normality	OK
n	18
outliers	2
mean (n)	891.543
st.dev. (n)	0.2462
R(calc.)	0.689
st.dev.(D4052:18)	0.1786
R(D4052:18)	0.50
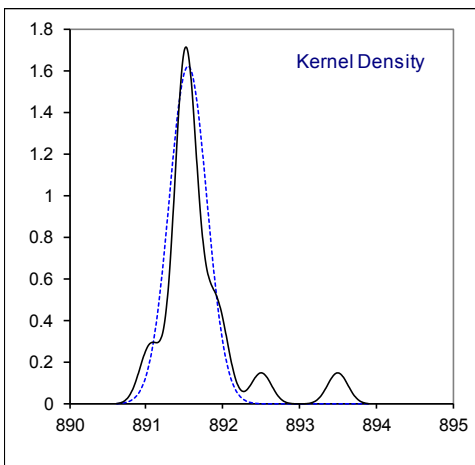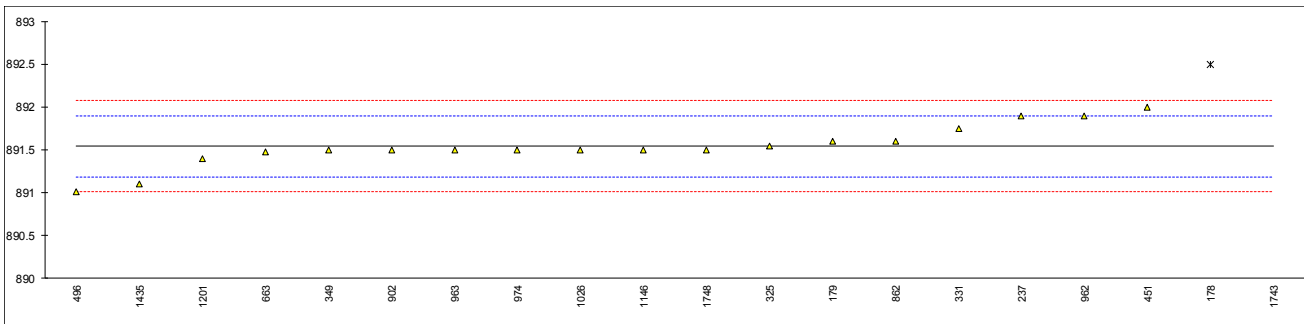
Determination of Flash Point PMcc on sample #18036; results in °C.

lab	method	value	mark	z(targ)	remarks
178	D93-A	193.5		0.31	
179	D93-A	182.0	C	-2.05	first reported 174.0
237	D93-B	190.0		-0.41	
325	D93-A	155.0	G(0.01)	-7.60	
331	D93-A	194.5		0.52	
349	D93-B	181		-2.26	
451	D93-A	206		2.88	
496	D93-A	194.0		0.41	
575		-----		-----	
633		-----		-----	
663	D93-A	195.3		0.68	
862	D93-A	181		-2.26	
902	D93-A	199.0		1.44	
962	D93-A	190		-0.41	
963	D93-A	190.0		-0.41	
974	D93-B	194		0.41	
1026	D93-A	194.0		0.41	
1146		-----		-----	
1201	D93-A	191.0		-0.20	
1435	D93-A	201.0		1.85	
1743	D93-A	188		-0.82	
1748	D93-A	191.5		-0.10	
6016		-----		-----	
6054		-----		-----	

normality	OK
n	18
outliers	1
mean (n)	191.99
st.dev. (n)	6.547
R(calc.)	18.33
st.dev.(D93-A:16a)	4.868
R(D93-A:16a)	13.63
compare	
R(D93-B:16a)	10
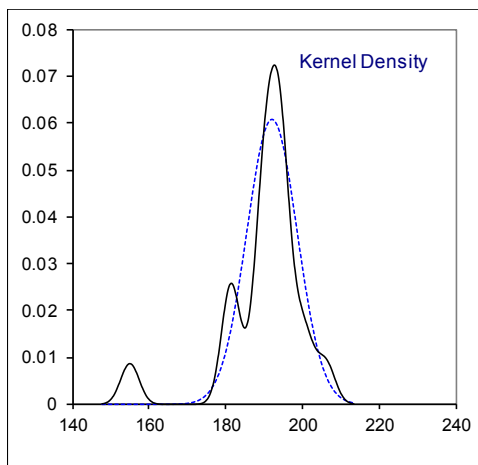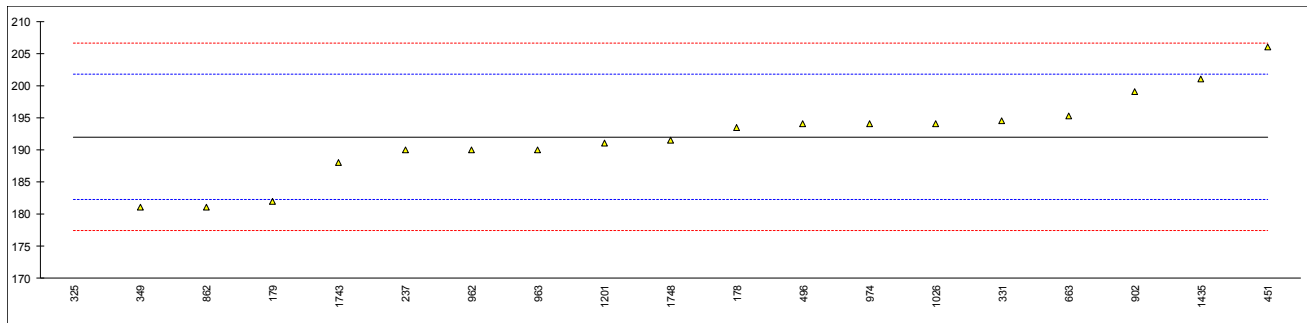
Determination of Kinematic Viscosity at 40°C on sample #18036; results in mm^2/s.

lab	method	value	mark	z(targ)	remarks
178	D445	146.6		-0.36	
179	D445	146.9	C	-0.09	first reported 77.81
237		-----		-----	
325	D445	146.9		-0.09	
331	D7279Mod.	146.3	ex	-0.62	test result excluded: not corrected to D445
349	D445	146.8		-0.18	
451		-----		-----	
496	D445	146.32		-0.60	
575	D7279 corrected to D445	146.9		-0.09	
633	D7279 corrected to D445	145.66		-1.18	
663	D445	147.16		0.13	
862	D445	147.9		0.78	
902	D445	147.7		0.60	
962	D445	147.5		0.43	
963	D445	147.3		0.25	
974	D445	147.8		0.69	
1026	D445	147.1		0.08	
1146	D445	147.36		0.31	
1201	D445	146.3		-0.62	
1435	D7042	147.65		0.56	
1743	D7279 corrected to D445	147.4	C	0.34	first reported 154.7
1748	D7042	145.91		-0.96	
6016		-----		-----	
6054		-----		-----	

normality	OK
n	19
outliers	0 (+1 ex)
mean (n)	147.0084
st.dev. (n)	0.63212
R(calc.)	1.7699
st.dev.(D445:17a)	1.14496
R(D445:17a)	3.2059
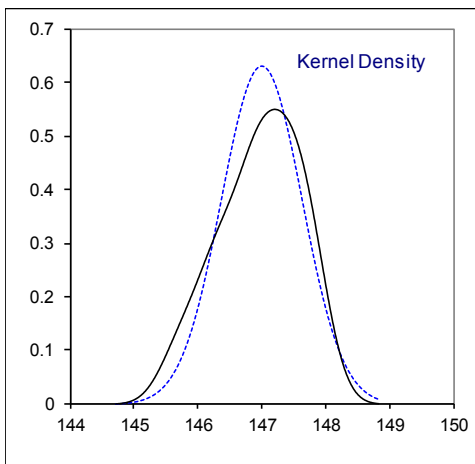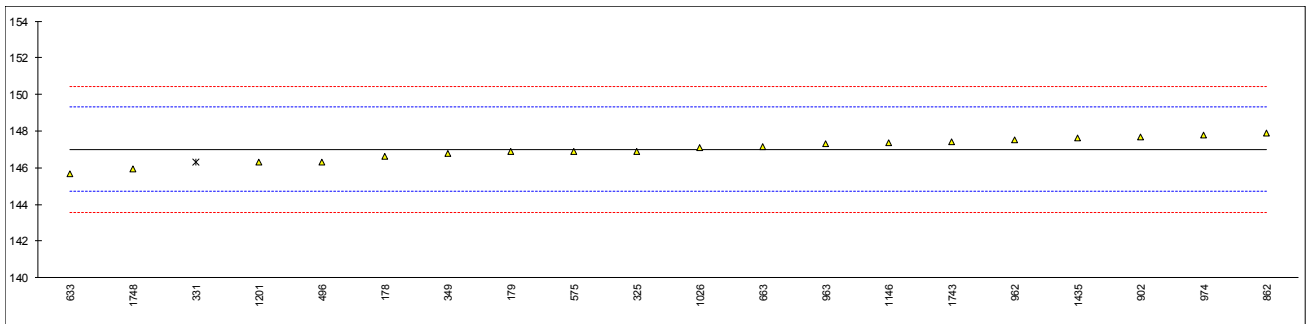
Determination of Kinematic Viscosity at 100°C on sample #18036; results in mm^2/s.

lab	method	value	mark	z(targ)	remarks
178	D445	15.57		-----	
179	D445	16.07		-----	
237		-----		-----	
325	D445	17.66		-----	
331	D7279Mod.	15.19	ex	-----	test result excluded: not corrected to D445
349		-----		-----	
451		-----		-----	
496		-----		-----	
575	D7279 corrected to D445	15.40		-----	
633	D7279 corrected to D445	15.892		-----	
663	D445	18.900		-----	
862	D445	19.05		-----	
902		-----		-----	
962		-----		-----	
963	D445	16.79		-----	
974	D445	19.51		-----	
1026	D445	17.75		-----	
1146	D445	16.443		-----	
1201	D445	15.17		-----	
1435	D7042	17.494		-----	
1743	D7279 corrected to D445	16.62		-----	
1748	D7042	17.80		-----	
6016		-----		-----	
6054		-----		-----	

normality	OK
n	15
outliers	0 (+1 ex)
mean (n)	17.0746
st.dev. (n)	1.37468
R(calc.)	3.8491
st.dev.(D445:17a)	(0.07631)
R(D445:17a)	(0.2137)
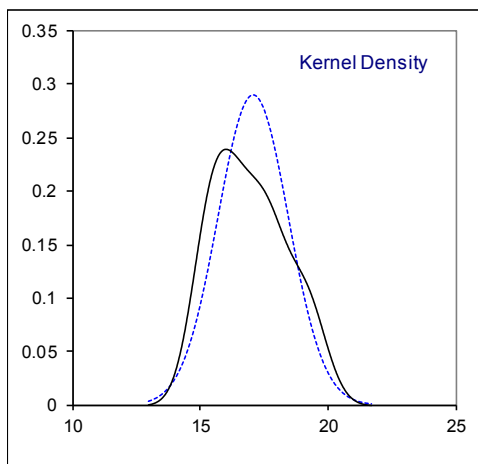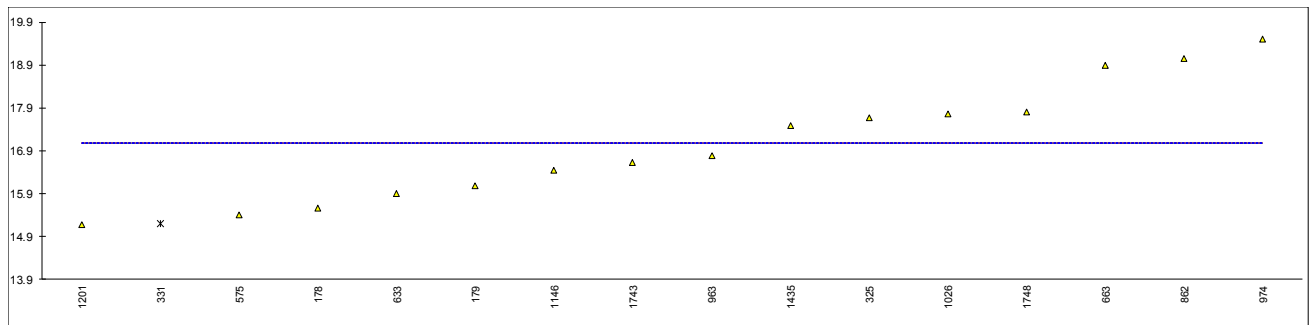
Determination of Viscosity Index (V.I.) on sample #18036

lab	method	value	mark	z(targ)	iis calc.	remarks
178	D2270	109		-----	109.16	
179	D2270	115	C	-----	114.65	first reported 222
237		-----		-----	-----	
325	D2270	132.5		-----	132.53	
331		-----		-----	104.97	
349		-----		-----	-----	
451		-----		-----	-----	
496		-----		-----	-----	
575	D2270	107		-----	106.94	
633	D2270	113.7		-----	113.71	
663	D2270	146		-----	145.62	
862	D2270	147		-----	146.49	
902		-----		-----	-----	
962		-----		-----	-----	
963	D2270	122		-----	122.50	
974	D2270	151		-----	151.35	
1026		-----		-----	133.31	
1146	D2270	118		-----	118.50	
1201	D2270	105		-----	104.75	
1435	D2270	129.92		-----	130.01	
1743	ISO2909	115		-----	120.45	KV 40°C was corrected without correcting VI
1748	D2270	134.987		-----	134.99	
6016		-----		-----	-----	
6054		-----		-----	-----	

normality	OK
n	14
outliers	0
mean (n)	124.722
st.dev. (n)	15.5559
R(calc.)	43.556
st.dev.(D2270:10)	(0.7143)
R(D2270:10)	(2)
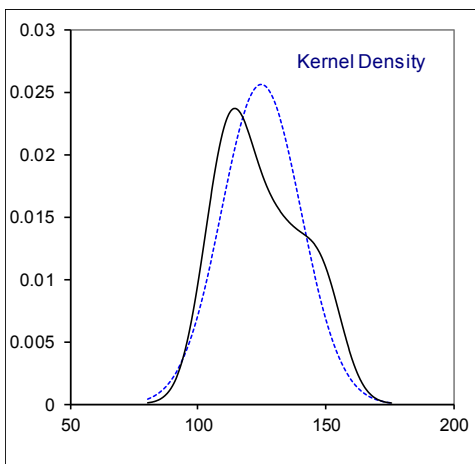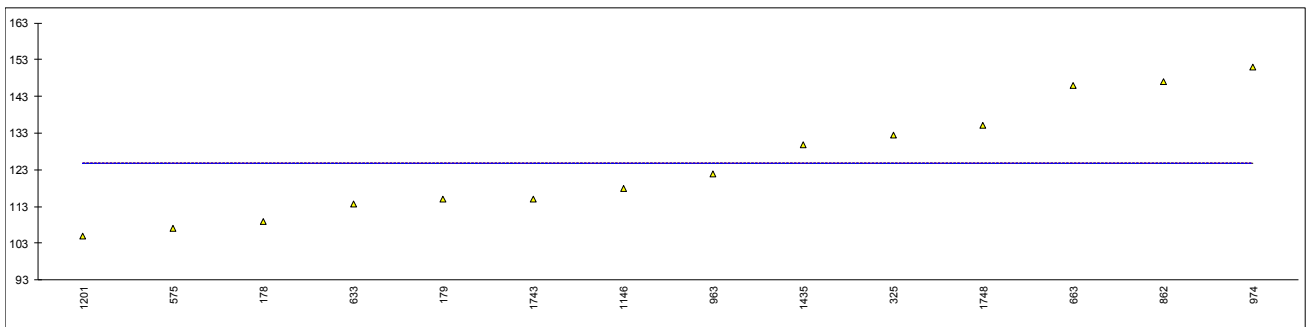
Determination of Membrane Filtration 5µm on sample #18036; results in %M/M.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
325	D4055	0.1010		-----	
331		-----		-----	
349		-----		-----	
451		-----		-----	
496		-----		-----	
575		-----		-----	
633		-----		-----	
663		-----		-----	
862	D4055	0.01		-----	
902		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D4898	0.025		-----	
1146		-----		-----	
1201		-----		-----	
1435	D4055	10		-----	
1743		-----		-----	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	unknown
n	4
outliers	n.a.
mean (n)	n.a.
st.dev. (n)	n.a.
R(calc.)	n.a.
st.dev.(D4055:04)	n.a.
R(D4055:04)	n.a.

Determination of Water by KF on sample #18036; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D6304-C	193		0.02	
179	D6304-C	214		0.17	
237		-----		-----	
325	D6304-C	201		0.08	
331	In house	139.3		-0.36	
349	D6304-A	198		0.06	
451	D6304-C	281		0.65	
496	D6304-C	185		-0.03	
575		-----		-----	
633	D6304-C	157.7		-0.23	
663	D6304-C	300		0.79	
862	D6304-C	156		-0.24	
902	D6304-A	203		0.10	
962	D6304-A	189		0.00	
963	D6304-A	179		-0.08	
974	D6304-A	239		0.35	
1026	D6304-C	52		-0.98	
1146	D6304-C	195		0.04	
1201	D6304-A	211		0.15	
1435	D6304-A	178.56		-0.08	
1743	ISO12937	130		-0.42	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	not OK
n	19
outliers	0
mean (n)	189.556
st.dev. (n)	53.6837
R(calc.)	150.314
st.dev.(D6304:16e1)	140.3322
R(D6304:16e1)	392.930
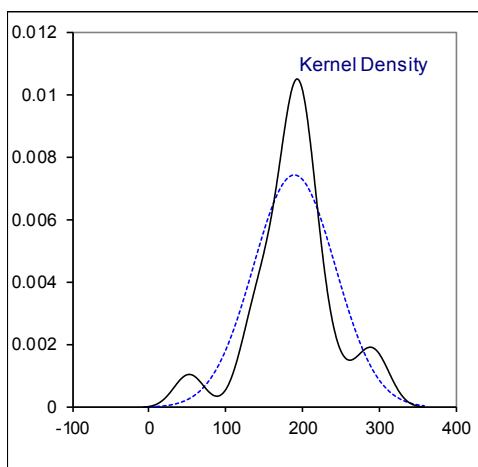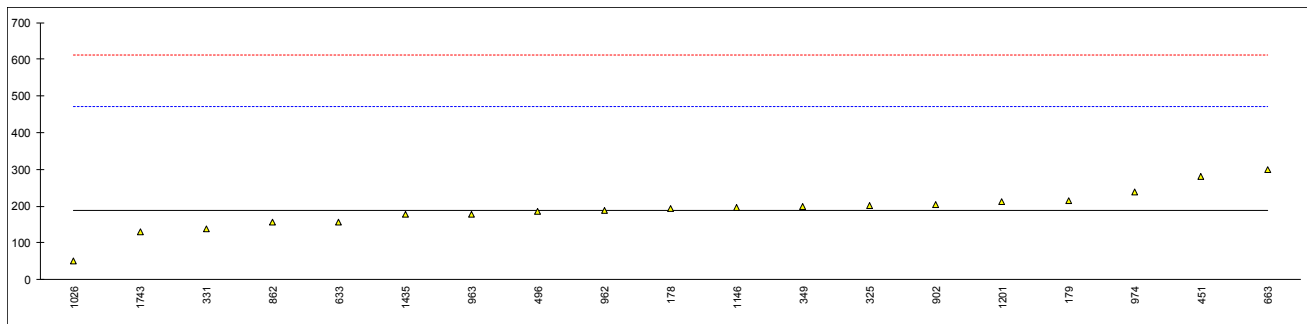
Determination of Level of Contamination on sample #18036; results in counts per ml.

lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)
178	INH-1185	23679	G(0.01)	217.77	4309	G(0.01)	200.50	916	DG(0.01)	158.25
179	D7647	1478	C, DG(0.01)	11.27	171	C, ex	4.42	59	C, ex	8.25
237		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
451		180		-0.80	39		-1.84	7		-0.85
496	D7647	110		-1.45	43		-1.65	3		-1.55
575		-----		-----	-----		-----	-----		-----
633	D7647	1703.5	DG(0.01)	13.37	1000.4	G(0.01)	43.72	782.7	DG(0.01)	134.92
663		-----		-----	-----		-----	-----		-----
862	ISO11500	504		2.21	79		0.06	9		-0.50
902	D7596	430.4		1.53	120.5		2.03	5.1		-1.18
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1026		-----		-----	-----		-----	-----		-----
1146		270		0.03	100		1.05	18		1.08
1201		-----		-----	-----		-----	-----		-----
1435		-----		-----	-----		-----	-----		-----
1743	ISO4407	104		-1.51	85		0.34	29		3.00
1748		-----		-----	-----		-----	-----		-----
6016		-----		-----	-----		-----	-----		-----
6054		-----		-----	-----		-----	-----		-----
	normality	OK			OK			suspect		
	n	6			6			6		
	outliers	3			2 (+1 ex)			2 (+1 ex)		
	mean (n)	266.40			77.75			11.85		
	st.dev. (n)	168.323			31.893			9.874		
	R(calc.)	471.30			89.30			27.65		
	st.dev.(D7647:10)	107.511			21.104			5.713		
	R(D7647:10)	301.03			59.09			16.00		

Lab 179: first reported 147778, 17147 and 5886 respectively. Excluded as one test result is a statistical outlier.
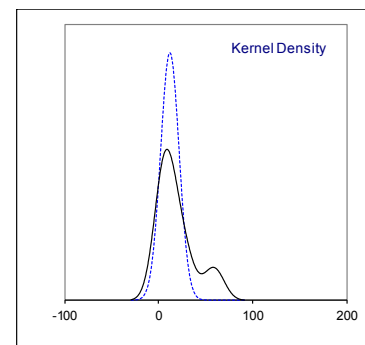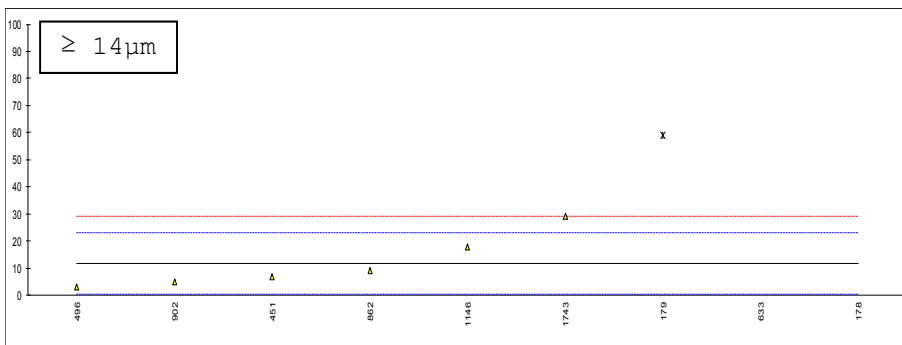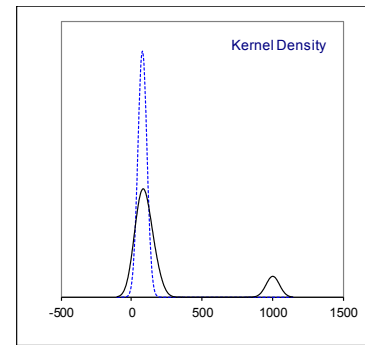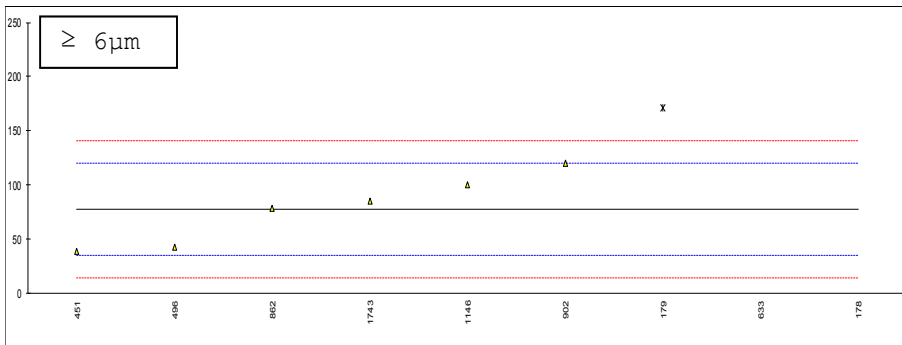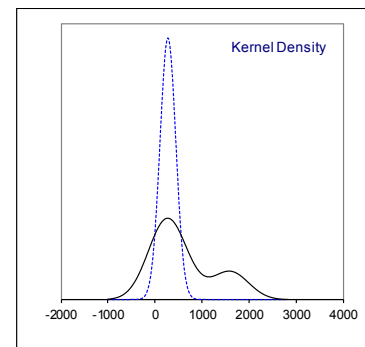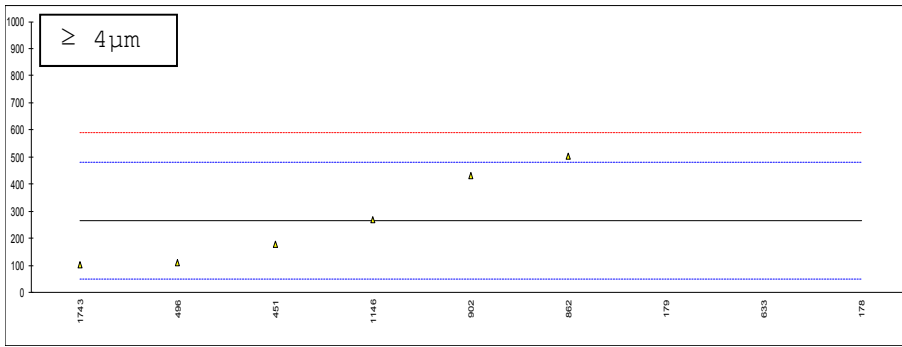
Determination of Level of Contamination acc. to ISO4406 scale on sample #18036; results in scale number

lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)
178	ISO4406	15	ex	-0.24	13	ex	-1.00	11	ex	1.00
179	ISO4406	18	ex	4.71	15	ex	3.67	13	ex	3.80
237		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
451	ISO4406	15		-0.24	12		-3.33	10		-0.40
496	ISO4406	14		-1.88	13		-1.00	9		-1.80
575		-----		-----	-----		-----	-----		-----
633	ISO4406	18	ex	4.71	17	ex	8.33	17	ex	9.40
663		-----		-----	-----		-----	-----		-----
862	ISO11500	16		1.41	13		-1.00	10		-0.40
902	ISO4406	16		1.41	14		1.33	10		-0.40
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1026	ISO4406	17	ex	3.06	16	D(0.05)	6.00	14	D(0.05)	5.20
1146	ISO4406	15		-0.24	14		1.33	11		1.00
1201		-----		-----	-----		-----	-----		-----
1435	ISO4406	16		1.41	14		1.33	10		-0.40
1743	ISO4406	14		-1.88	14		1.33	12		2.40
1748		-----		-----	-----		-----	-----		-----
6016		-----		-----	-----		-----	-----		-----
6054		-----		-----	-----		-----	-----		-----
	normality	OK			OK			suspect		
	n	7			7			7		
	outliers	0 (+4 ex)			1 (+3 ex)			1 (+3 ex)		
	mean (n)	15.1			13.4			10.3		
	st.dev. (n)	0.90			0.79			0.95		
	R(calc.)	2.5			2.2			2.7		
	st.dev.(D7647:10)	0.6			0.4			0.7		
	R(D7647:10)	1.7			1.2			2		

Lab 178: excluded as statistical outliers in counts per ml.
Lab 179: excluded as statistical outliers in counts per ml.
Lab 633: excluded as statistical outliers in counts per ml.
Lab 1026: excluded as two test results are statistical outliers.
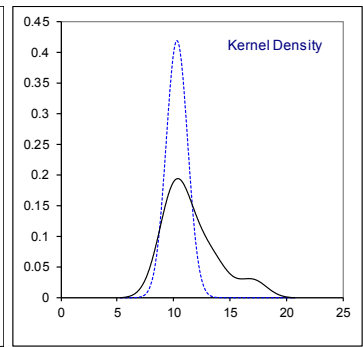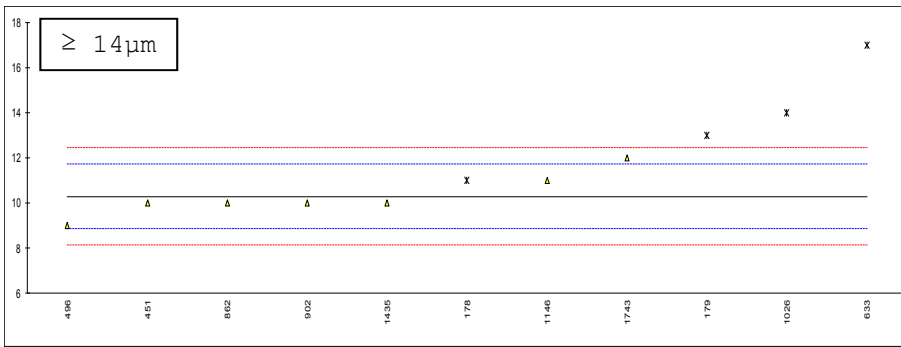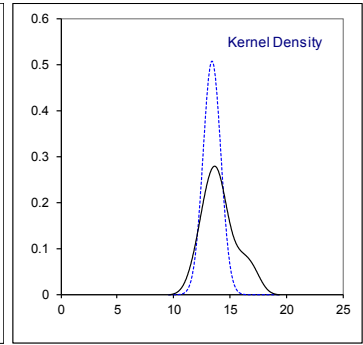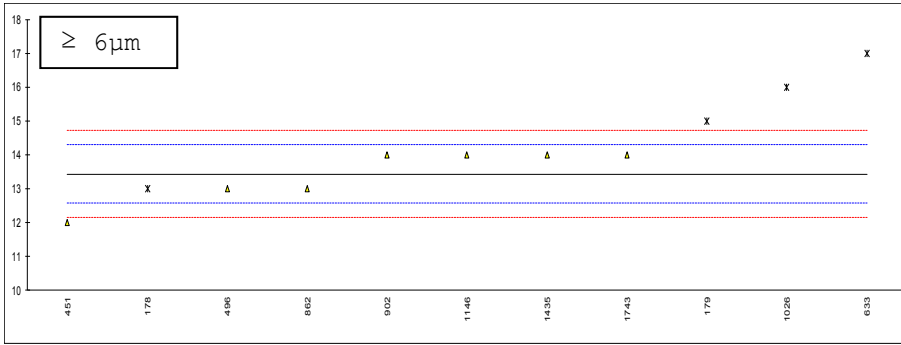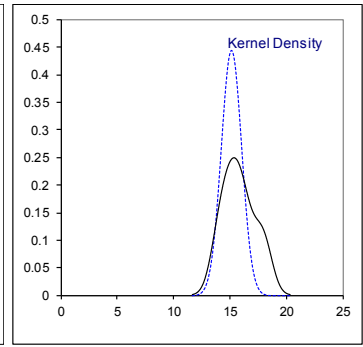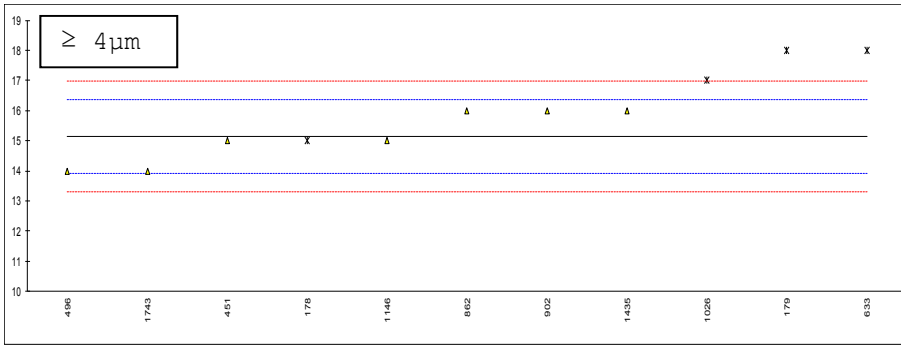
Determination of Aluminium as Al on sample #18037; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	17		0.62	
179	D5185	18		0.98	
237	D5185	14.46		-0.30	
325	D5185	16		0.26	
331	D5185Mod,	13.5		-0.65	
349	D5185	17		0.62	
451	D5185Mod.	15		-0.10	
496	D5185	15.81		0.19	
575	D6595	14.9		-0.14	
633	D6595	13.44		-0.67	
663	D5185	13.75		-0.56	
862	D5185	14.6		-0.25	
902	D5185	14.31		-0.35	
962	D5185	15		-0.10	
963	D5185	15.2		-0.03	
974		15		-0.10	
1026	D5185	16		0.26	
1146	In house	16.03		0.27	
1201	D5185	16.8	C	0.55	first reported 0.8
1435	D5185	12.24		-1.11	
1743	D5185	17		0.62	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	OK			
n	21			
outliers	0		spike:	
mean (n)	15.288		16.19	Recovery < 94%
st.dev. (n)	1.4329			
R(calc.)	4.012			
st.dev.(D5185:18)	2.7577			
R(D5185:18)	7.722			

Kernel Density
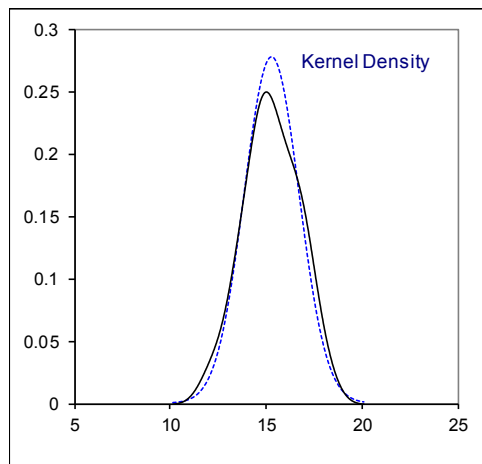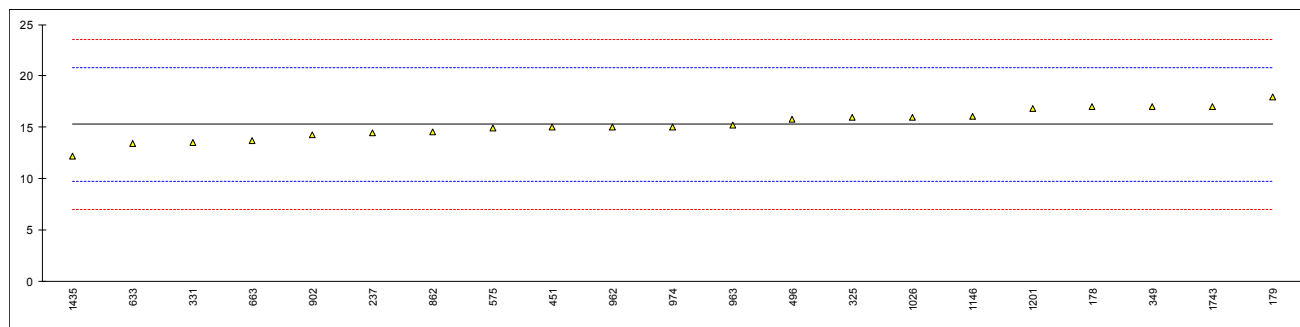
Determination of Boron as B on sample #18037; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	16		-0.25	
179	D5185	16		-0.25	
237	D5185	27.055	G(0.05)	2.06	
325	D5185	17		-0.04	
331	D5185Mod,	14.7		-0.52	
349	D5185	20		0.58	
451	D5185Mod.	15		-0.46	
496	D5185	15.8		-0.29	
575	D6595	14.9		-0.48	
633	D6595	20.95		0.78	
663	D5185	22.99	C	1.21	first reported 45.98
862	D5185	14.8		-0.50	
902	D5185	18.75		0.32	
962	D5185	15		-0.46	
963	D5185	14.8		-0.50	
974		-----		-----	
1026	D5185	20.9		0.77	
1146		-----		-----	
1201	D5185	17		-0.04	
1435	D5185	15.15		-0.43	
1743	D5185	20		0.58	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	OK
n	18
outliers	1
mean (n)	17.208
st.dev. (n)	2.6703
R(calc.)	7.477
st.dev.(D5185:18)	4.7769
R(D5185:18)	13.375
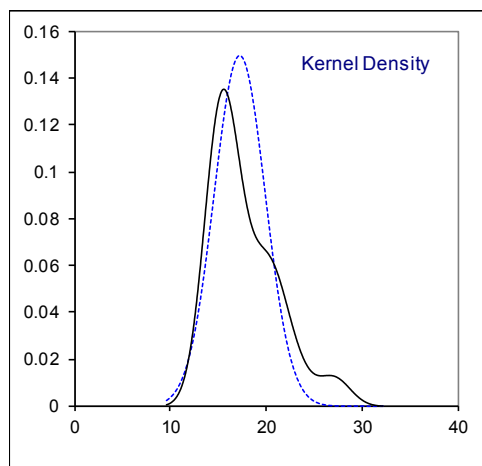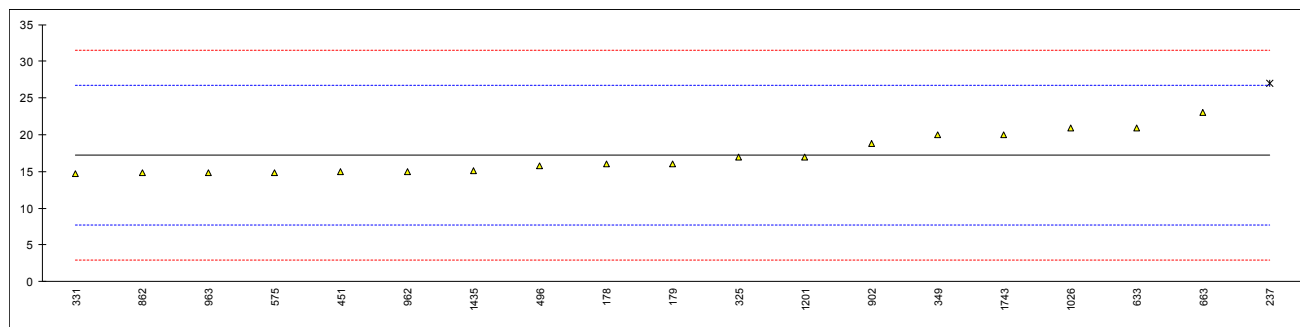
Determination of Chromium as Cr on sample #18037; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	2		0.00	
179	D5185	2		0.00	
237	D5185	2.09		0.20	
325	D5185	2		0.00	
331	D5185Mod,	2.0		0.00	
349	D5185	<4		-----	
451	D5185Mod.	2		0.00	
496	D5185	1.92		-0.18	
575	D6595	2.11		0.25	
633	D6595	1.47		-1.20	
663	D5185	1.95		-0.11	
862	D5185	1.8		-0.45	
902	D5185	1.64		-0.82	
962	D5185	2		0.00	
963	D5185	1.99		-0.02	
974		<1		<-2.27	possible false negative test result?
1026	D5185	2.20		0.45	
1146	In house	2.018		0.04	
1201	D5185	2.5		1.13	
1435	D5185	2.123		0.28	
1743	D5185	2.2		0.45	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	not OK
n	19
outliers	0
mean (n)	2.001
st.dev. (n)	0.2141
R(calc.)	0.599
st.dev.(D5185:18)	0.4416
R(D5185:18)	1.236

Kernel Density
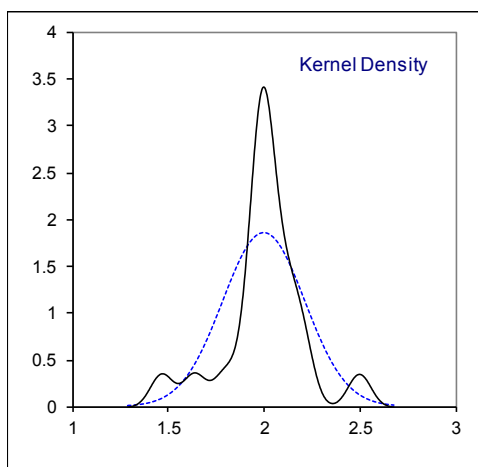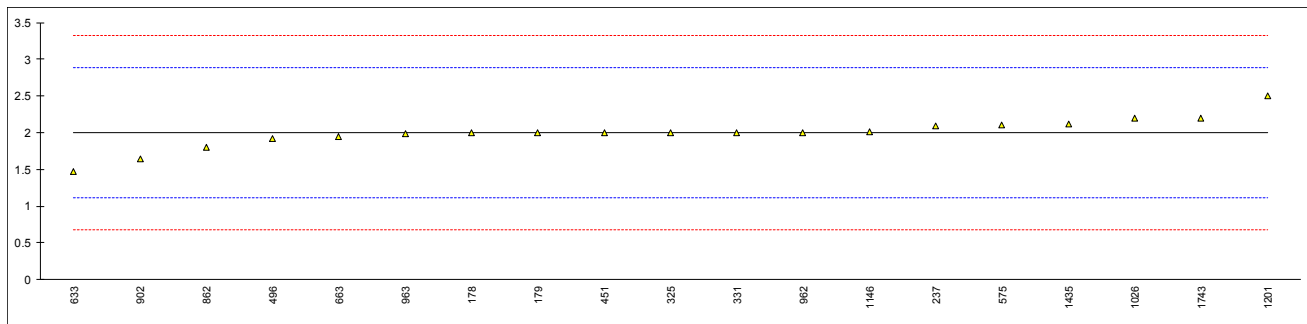
Determination of Copper as Cu on sample #18037; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	17		0.79	
179	D5185	17		0.79	
237	D5185	16.54		0.45	
325	D5185	16		0.05	
331	D5185Mod,	14.5		-1.04	
349	D5185	17		0.79	
451	D5185Mod.	16		0.05	
496	D5185	16.11		0.13	
575	D6595	14.8		-0.82	
633	D6595	12.64	R(0.05)	-2.41	
663	D5185	14.77		-0.85	
862	D5185	15.9		-0.02	
902	D5185	14.97		-0.70	
962	D5185	15		-0.68	
963	D5185	15.6		-0.24	
974		<1		<-10.93	possible false negative test result?
1026	D5185	16.4		0.35	
1146	In house	15.73		-0.14	
1201	D5185	19	R(0.05)	2.25	
1435	D5185	16.35		0.31	
1743	D5185	17		0.79	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	OK			
n	18			
outliers	2		spike:	
mean (n)	15.926		12.80	Recovery < 124%
st.dev. (n)	0.8400			
R(calc.)	2.352			
st.dev.(D5185:18)	1.3651			
R(D5185:18)	3.822			
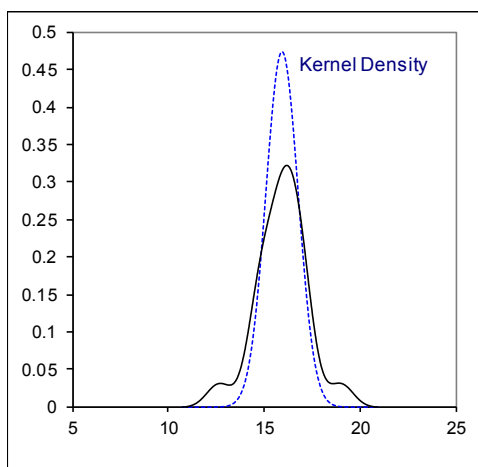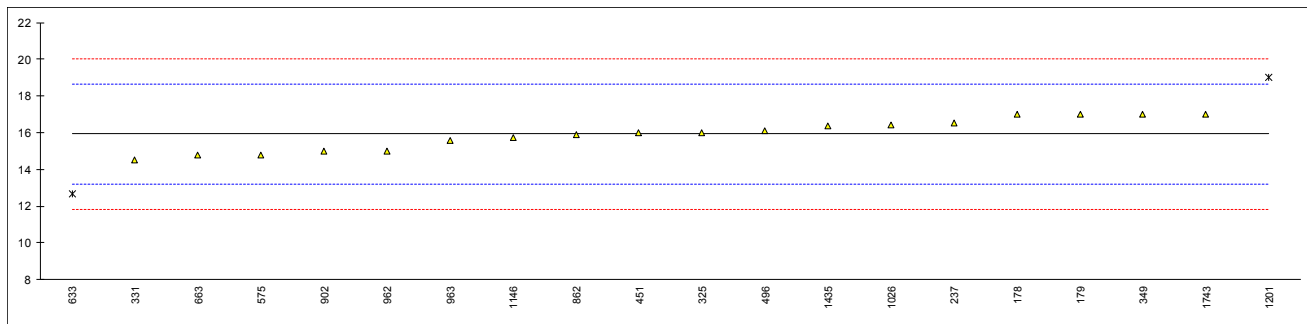
Determination of Iron as Fe on sample #18037; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	187		-0.14	
179	D5185	192		0.26	
237	D5185	>140		-----	
325	D5185	187		-0.14	
331	D5185Mod,	172.5		-1.32	
349	D5185	206		1.40	
451	D5185Mod.	178		-0.88	
496	D5185	184.2		-0.37	
575	D6595	210.8		1.79	
633	D6595	177.26		-0.94	
663	D5185	182.31		-0.52	
862	D5185	192.6		0.31	
902	D5185	176		-1.04	
962	D5185	182		-0.55	
963	D5185	189.8		0.08	
974		196		0.59	
1026	D5185	200		0.91	
1146	In house	182.7		-0.49	
1201	D5185	200		0.91	
1435	D5185	179.0		-0.79	
1743	D5185	200		0.91	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	OK
n	20
outliers	0
mean (n)	188.759
st.dev. (n)	10.6678
R(calc.)	29.870
st.dev.(D5185:18)	12.2905
R(D5185:18)	34.413

Kernel Density
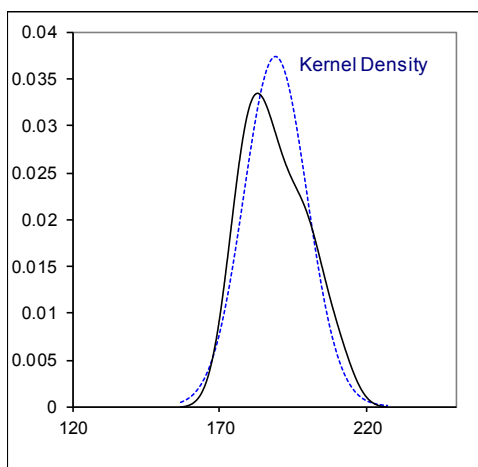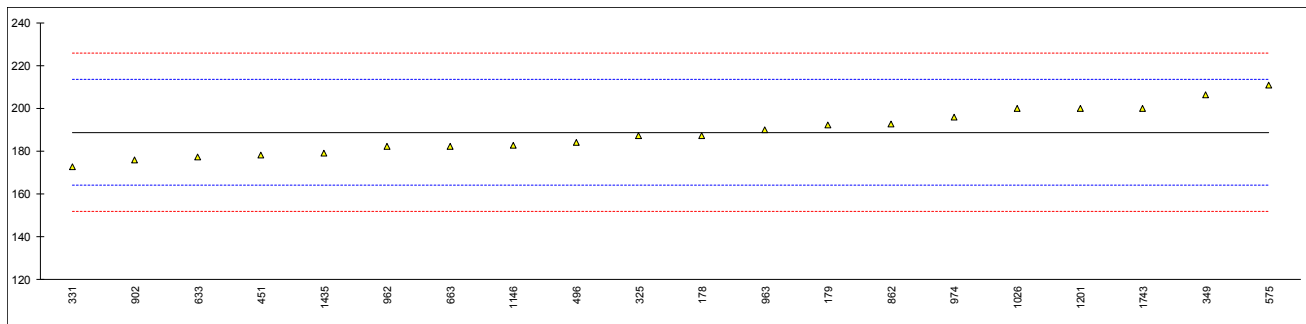
Determination of Silicon as Si on sample #18037; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	13		0.15	
179	D5185	13		0.15	
237	D5185	11.57		-0.36	
325	D5185	13		0.15	
331	D5185Mod,	11.2		-0.50	
349	D5185	13		0.15	
451	D5185Mod.	12		-0.21	
496	D5185	13.52		0.34	
575	D6595	11.8		-0.28	
633	D6595	15.13		0.92	
663	D5185	10.46		-0.76	
862	D5185	12.0		-0.21	
902	D5185	13.72		0.41	
962	D5185	12		-0.21	
963	D5185	12.2		-0.14	
974		14		0.51	
1026	D5185	11.0		-0.57	
1146	In house	12.26		-0.11	
1201	D5185	14		0.51	
1435	D5185	12.27		-0.11	
1743	D5185	13		0.15	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	OK				
n	21				
outliers	0		spike:		
mean (n)	12.578		12.14	Recovery < 104%	
st.dev. (n)	1.1286				
R(calc.)	3.160				
st.dev.(D5185:18)	2.7802				
R(D5185:18)	7.785				

Kernel Density
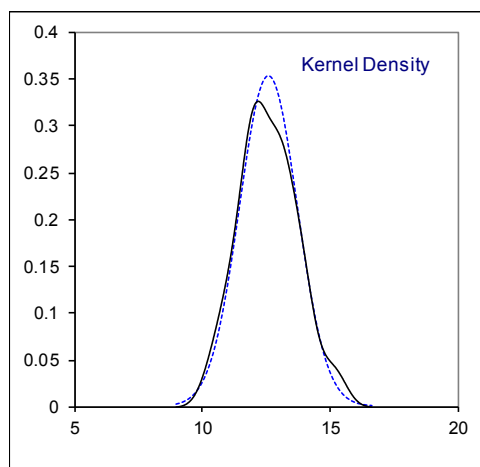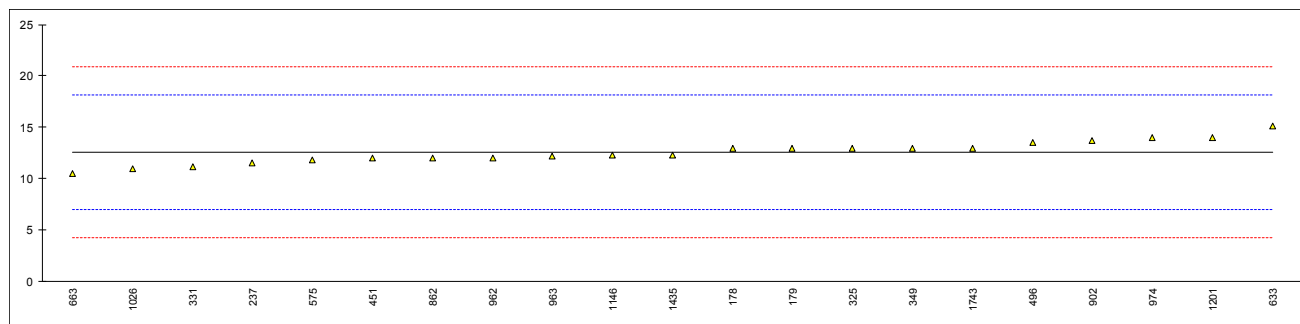
Determination of Tin as Sn on sample #18037; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	14		0.02	
179	D5185	15		0.28	
237	D5185	14.46		0.14	
325	D5185	16		0.54	
331	D5185Mod,	13.4		-0.14	
349	D5185	9		-1.28	
451	D5185Mod.	15		0.28	
496	D5185	9.0		-1.28	
575	D6595	12.1		-0.48	
633	D6595	11.67		-0.59	
663	D5185	14.13		0.05	
862	D5185	14.0		0.02	
902	D5185	14.67		0.19	
962	D5185	15		0.28	
963	D5185	16.4		0.64	
974		18		1.06	
1026	D5185	16.3		0.62	
1146	In house	8.181		-1.50	
1201	D5185	16		0.54	
1435	D5185	14.25		0.08	
1743	D5185	16		0.54	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	OK		
n	21		
outliers	0	spike:	
mean (n)	13.931	14.61	Recovery < 95%
st.dev. (n)	2.6072		
R(calc.)	7.300		
st.dev.(D5185:18)	3.8401		
R(D5185:18)	10.752		
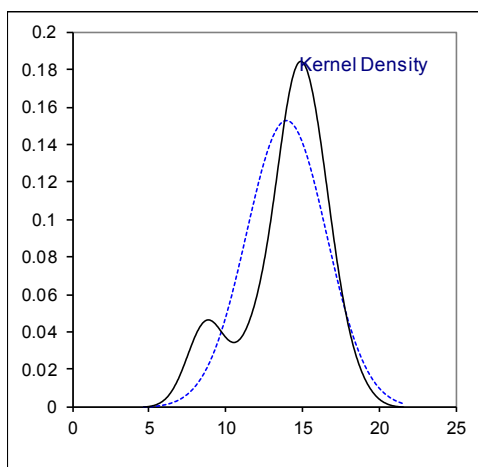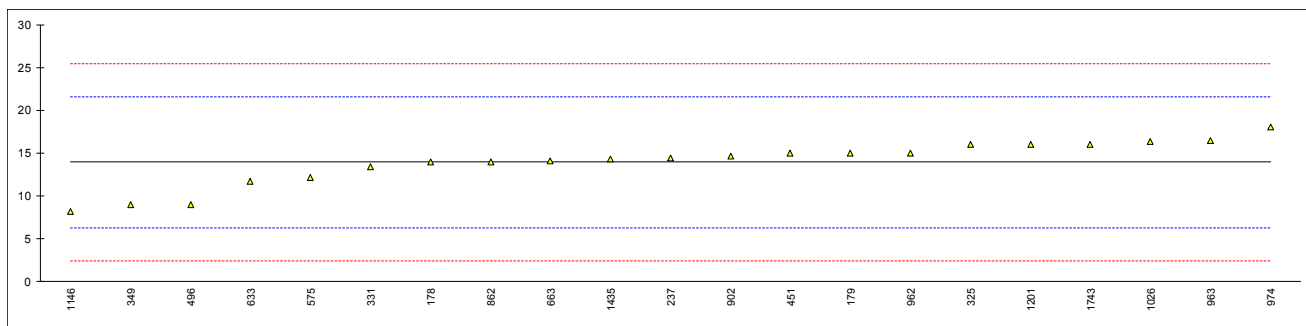
Determination of Calcium as Ca on sample #18037; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	2		-1.20	
179	D5185	<1		<-4.16	possible false negative test result?
237	D5185	<40		-----	
325	D5185	3		1.77	
331	D5185Mod,	2.2		-0.60	
349	D5185	<4		-----	
451	D5185Mod.	10	G(0.01)	22.54	
496	D5185	2.52		0.35	
575	D6595	< LOD		<-7.13	reported: 0.00, below application range ASTM D6595; **-f?**
633	D6595	3.2		2.36	
663	D5185	2.24		-0.48	
862	D5185	2.0		-1.20	
902	D5185	2.84	C	1.30	first reported 5.19
962	D5185	2		-1.20	
963	D5185	2.37		-0.10	
974		2		-1.20	
1026	D5185	2		-1.20	
1146	In house	2.410		0.02	
1201	D5185	3.3		2.66	
1435	D5185	1.952		-1.34	
1743	D5185	2.42		0.05	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	OK	
n	16	
outliers	1	
mean (n)	2.403	
st.dev. (n)	0.4535	
R(calc.)	1.270	
st.dev.(Horwitz)	0.3370	
R(Horwitz)	0.944	
compare		
R(D5185:18)	0.047	application range: 40 – 9000 mg/kg
R(D6595:17)	1.353	application range: 3.7 – 11460 mg/kg
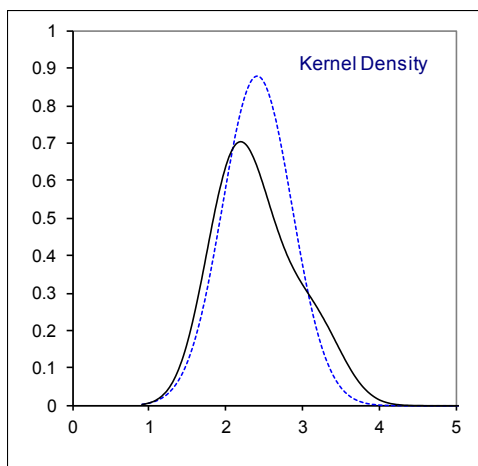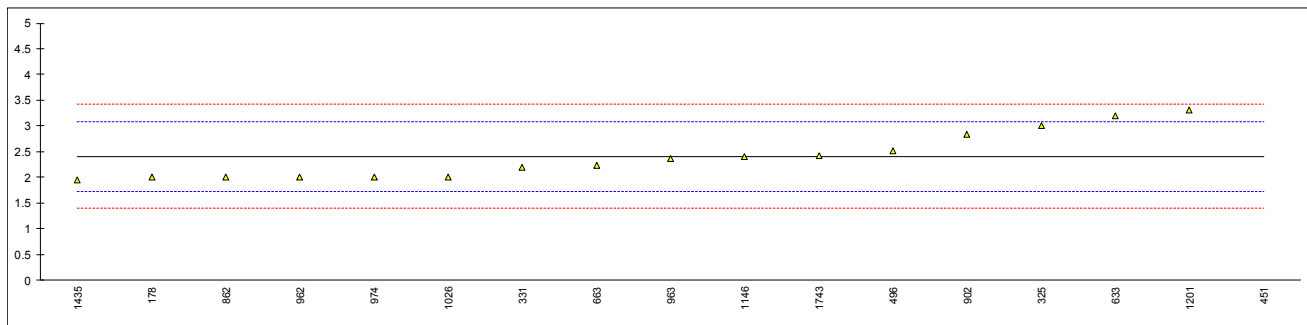
Determination of Phosphorus as P on sample #18037; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	302		-0.16	
179	D5185	312		0.21	
237	D5185	310		0.13	
325	D5185	312		0.21	
331	D5185Mod,	280.3		-0.97	
349	D5185	301		-0.20	
451	D5185Mod.	294		-0.46	
496	D5185	292.0		-0.54	
575	D6595	240.6	R(0.01)	-2.45	
633	D6595	368.9	R(0.01)	2.32	
663	D5185	291.13		-0.57	
862	D5185	295.5		-0.41	
902	D5185	308		0.06	
962	D5185	307		0.02	
963	D5185	307.7		0.05	
974		300		-0.24	
1026	D5185	338		1.18	
1146	In house	310.6		0.16	
1201	D5185	320		0.51	
1435	D5185	309.60		0.12	
1743	D5185	331		0.91	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	OK	
n	19	
outliers	2	
mean (n)	306.412	
st.dev. (n)	13.7045	
R(calc.)	38.373	
st.dev.(D5185:18)	26.8821	
R(D5185:18)	75.270	

Kernel Density
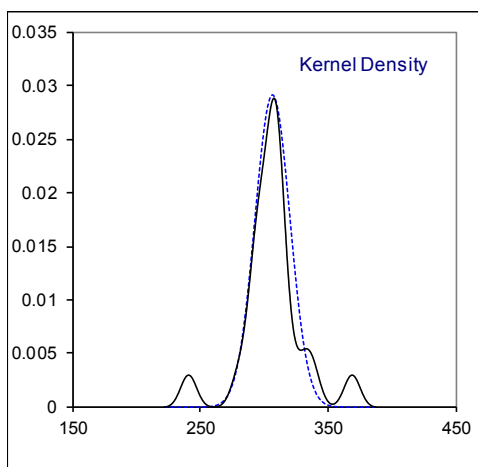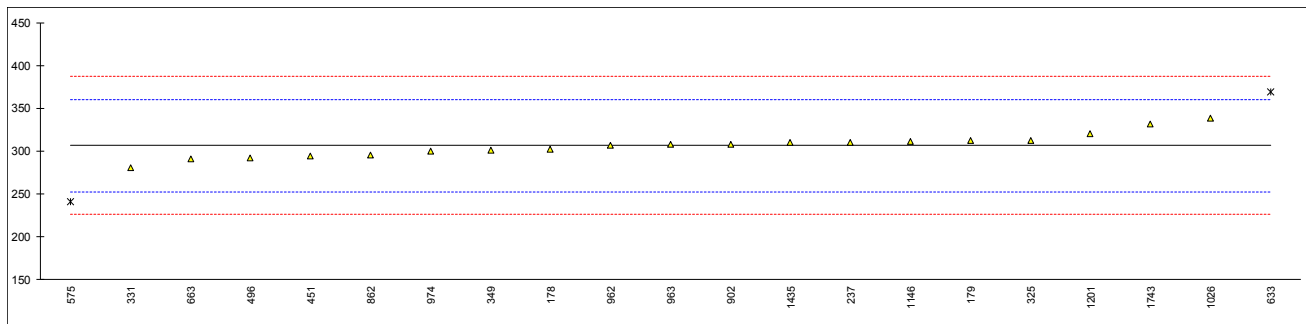
Determination of Zinc as Zn on sample #18037; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	2		-1.85	
179	D5185	2		-1.85	
237	D5185	<60.0		-----	
325	D5185	3		0.84	
331	D5185Mod,	2.2		-1.31	
349	D5185	<4		-----	
451	D5185Mod.	3		0.84	
496	D5185	2.76		0.20	
575	D6595	2.07		-1.67	
633	D6595	2.6		-0.24	
663	D5185	2.71		0.06	
862	D5185	2.4		-0.78	
902	D5185	2.93		0.66	
962	D5185	3		0.84	
963	D5185	2.61		-0.21	
974		3	C	0.84	first reported 5
1026	D5185	3		0.84	
1146	In house	2.743		0.15	
1201	D5185	3.4		1.92	
1435	D5185	2.904		0.59	
1743	D5185	2.73		0.12	
1748		-----		-----	
6016		-----		-----	
6054		-----		-----	

normality	OK		
n	19		
outliers	0		
mean (n)	2.687		
st.dev. (n)	0.3925		
R(calc.)	1.099		
st.dev.(Horwitz)	0.3705		
R(Horwitz)	1.037		
compare			
R(D5185:18)	0.246	application range: 60 – 1600 mg/kg	
R(D6595:17)	2.066	application range: 5.3 – 1345 mg/kg	
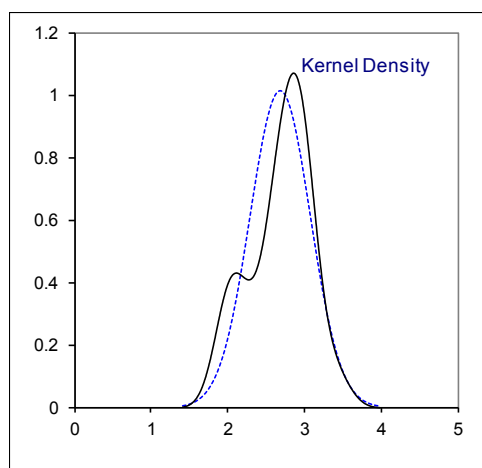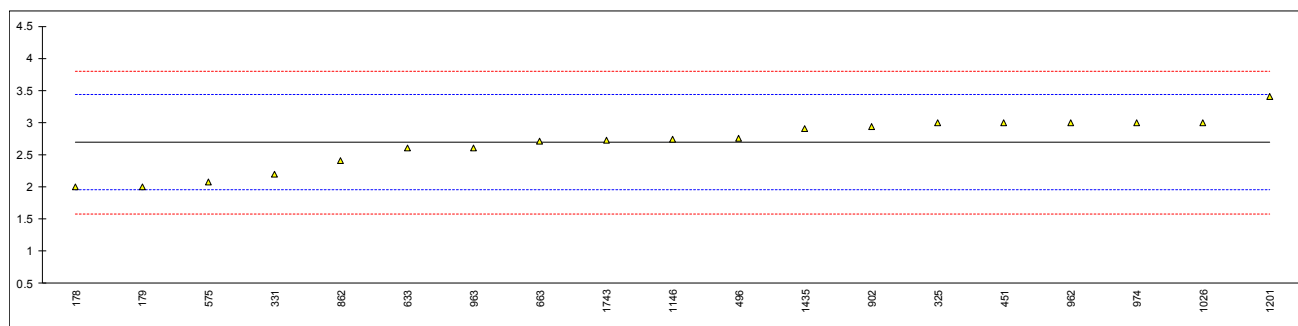
APPENDIX 2 Summary of other reported test results

Reported test results of other elements on sample #18037; results in mg/kg.

lab	method	Antimony as Sb	Barium as Ba	Cadmium as Cd	Lead as Pb	Lithium as Li	Magnesium as Mg	Manganese as Mn
178	D5185	<1	<1	-----	<1	-----	<1	-----
179	D5185	<1	<1	<1	<1	<1	<1	1
237	D5185	-----	<0.5	-----	<1	-----	<5.0	<5.0
325	D5185	0	0	-----	0	-----	0	1
331	D5185Mod.	<1	<1	-----	<1	<1	<1	<2
349	D5185	<4	<4	<4	<4	<4	<4	<4
451	D5185Mod.	0	0	0	0	0	0	0
496	D5185	-----	0	0.275	0	0.159	0	0.67
575	D6595	-----	0.00	-----	0.00	-----	0.00	0.00
633	D6595	-----	0.02	0.20	1.50	0.00	0.3	0.56
663	D5185	<1.0	0.030	-----	0.04	-----	0.19	0.74
862	D5185	<1	<1	<1	<1	<1	<1	<1
902	D5185	-----	0.17	-----	-----	-----	-----	1.18
962	D5185	-----	<1	-----	<1	-----	<1	<1
963	D5185	-----	<0.100	<0.10	<0.10	-----	0.280	0.78
974		-----	<1	<1	<1	-----	<1	<1
1026	D5185	-----	0	-----	0.204	-----	0	-----
1146	In house	-----	0.0750	-----	0.4416	0.0199	0.2037	0.7020
1201	D5185	-----	0.3	0.1	0.4	-----	0.6	0.8
1435	D5185	<1	<1	<1	<1	<1	<1	<1
1743	D5185	0.15	0.05	0.03	0.23	0.63	0.37	1.0
1748		-----	-----	-----	-----	-----	-----	-----
6016		-----	-----	-----	-----	-----	-----	-----
6054		-----	-----	-----	-----	-----	-----	-----

lab	method	Molybdenum as Mo	Nickel as Ni	Potassium as K	Silver as Ag	Sodium as Na	Titanium as Ti	Vanadium as V
178	D5185	<1	<1	<3	<0.10	<3	-----	<1
179	D5185	<1	1	<1	<0.10	<3	<1	<1
237	D5185	<5.0	<5.0	<40.0	<0.5	<7.0	<10.0	<1.0
325	D5185	0	0	1	0	0	-----	0
331	D5185Mod.	<1	<1	<1	<1	<1	<1	<1
349	D5185	<4	<4	<4	<4	<4	<4	<4
451	D5185Mod.	0	0	0	0	0	0	0
496	D5185	0	0.86	0	0	0.72	0	0
575	D6595	0.00	0.00	-----	0.00	0.00	0.00	0.00
633	D6595	0.3	0.27	0.13	0.00	1.00	0.00	0.39
663	D5185	0.08	0.41	0.29	<1.0	<1.0	0.03	0.04
862	D5185	<1	<1	<1	<1	<1	<1	<1
902	D5185	-----	0.17	-----	2.90	0.17	-----	-----
962	D5185	<1	<1	<1	<1	<1	<1	<1
963	D5185	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
974		1	<1 C	<1	<1 C	1 C	<1	<1
1026	D5185	0.059	0.382	-----	-----	0.394	0.031	0.021
1146	In house	-----	0.3686	-----	0.0494	0.2018	0.0334	0.1063
1201	D5185	0.7	0.2	1.5	0.8	2.4	0.3	0.3
1435	D5185	<1	2.641	1.006	<1	<1	<1	<1
1743	D5185	0.07	0.48	0.0	0.0	0.43	0.03	0.04
1748		-----	-----	-----	-----	-----	-----	-----
6016		-----	-----	-----	-----	-----	-----	-----
6054		-----	-----	-----	-----	-----	-----	-----

Lab 974: first reported 2, 24 and 3 respectively

APPENDIX 3 Summary of reported analytical details

Level of Contamination

lab	Sample rolled	Sample diluted	Dilution ratio	Manufacturer / model of equipment
178	Yes	No	---	---
179	Yes	No	---	---
237	---	---	---	---
325	Yes	No	---	Methrom
331	---	---	---	---
349	---	---	---	---
451	No	No	---	---
496	Yes	No	---	Pamas SBSS-C
575	---	---	---	---
633	Yes	Yes	20:80 Sample:Diluent	Pamas S-40
663	---	---	---	---
862	---	---	---	---
902	Yes	No	---	Lasernet Fines Spectro LNF Q200
962	---	---	---	---
963	---	---	---	---
974	---	---	---	---
1026	No	No	---	---
1146	No	Yes	18.9167g sample in 125ml	Metrohm 716 titrino and 730 sample changer
1201	---	---	---	---
1435	Yes	No	---	---
1743	Yes	No	---	Microscope Olympus BH-2
1748	---	---	---	---
6016	---	---	---	---
6054	---	---	---	---

APPENDIX 4

Number of participants per country

2 labs in BELGIUM

1 lab in CHINA, People's Republic

1 lab in COLOMBIA

1 lab in COTE D'IVOIRE

2 labs in FRANCE

1 lab in GERMANY

1 lab in JORDAN

1 lab in KAZAKHSTAN

3 labs in NETHERLANDS

1 lab in NIGERIA

1 lab in PHILIPPINES

2 labs in SAUDI ARABIA

1 lab in SPAIN

1 lab in THAILAND

1 lab in TURKEY

1 lab in UNITED ARAB EMIRATES

1 lab in UNITED KINGDOM

2 labs in UNITED STATES OF AMERICA

APPENDIX 5

Abbreviations:

C   = final result after checking of first reported suspect test result
D(0.01)   = outlier in Dixon's outlier test
D(0.05)   = straggler in Dixon's outlier test
G(0.01)   = outlier in Grubbs' outlier test
G(0.05)   = straggler in Grubbs' outlier test
DG(0.01)   = outlier in Double Grubbs' outlier test
DG(0.05)   = straggler in Double Grubbs' outlier test
R(0.01)   = outlier in Rosner's outlier test
R(0.05)   = straggler in Rosner's outlier test
E   = probably an error in calculations
U   = test result probably reported in a different unit
W   = test result withdrawn on request of participant
ex   = test result excluded from the statistical evaluation
n.a.   = not applicable
n.e.   = not evaluated
n.d.   = not detected
fr.   = first reported
SDS   = Safety Data Sheet

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