Results of Proficiency Test
Hydraulic Fluid (used)
November 2018

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CONTENTS

Appendices:

1   INTRODUCTION

Since 2003, the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the analysis of used Hydraulic Fluid every year. It was decided to continue this interlaboratory study during the annual program 2018/2019.

In this interlaboratory study, 53 laboratories from 38 different countries did register for used Hydraulic Fluid main round (iis18L10) and 44 laboratories from 34 different countries did register for the determination of Metals in used Hydraulic Fluid (iis18L10M).

In this report the test results of the 2018 proficiency tests 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 in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analysis for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send two different samples of used Hydraulic fluids: one sample of 1 litre used Hydraulic Fluid, labelled #18226, and one sample of 50 mL used Hydraulic Fluid, labelled #18227, especially for wear metals. 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% confidentially 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 Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through 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

Preparation of samples for PT on used Hydraulic Fluid (main round), iis18L10

Approximately 90 litre of used Hydraulic Fluid was obtained from a local supplier. After homogenisation, 83 amber glass one litre bottles were filled and labelled #18226. The homogeneity of the subsamples #18226 was checked by determination of Density at 15°C in accordance with ASTM D4052 and Kinematic Viscosity at 40°C according to ASTM D445 on 8 stratified randomly selected samples.

	Density at 15°C in kg/L	Kinematic Viscosity at 40°C in mm^2/s
Sample #18226-1	0.89027	38.44
Sample #18226-2	0.89025	38.44
Sample #18226-3	0.89029	38.44
Sample #18226-4	0.89030	38.45
Sample #18226-5	0.89028	38.45
Sample #18226-6	0.89027	38.45
Sample #18226-7	0.89027	38.45
Sample #18226-8	0.89026	38.47

Table 1: homogeneity test results of subsamples #18226

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility 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/L	Kinematic Viscosity at 40°C in mm^2/s
r (observed)	0.00004	0.028
reference test method	ISO12185:96	iis, see ref. 17
0.3 * R (ref. test method)	0.00015	0.208

Table 2: evaluation of the repeatabilities of subsamples #18226

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

Preparation of samples for PT on Metals in used Hydraulic Fluid, iis18L10M

A batch of approximately 3.4 kg used Hydraulic Fluid was separated from the batch for the main round and spiked with Aluminum (approx. 12 mg/kg), Boron (approx. 10 mg/kg), Manganese (approx. 10 mg/kg), Molybdenum (approx. 10 mg/kg), Silicon (approx. 11 mg/kg) and Tin (approx. 10 mg/kg). After homogenisation, out of the batch 75 PE bottles of 50 mL were filled and labelled #18227. The homogeneity of the subsamples #18227 was checked by determination of Manganese, Molybdenum and Phosporus in accordance with ASTM D5185 on 8 stratified randomly selected samples.

	Manganese in mg/kg	Molybdenum in mg/kg	Phosphorus in mg/kg
Sample #18227-1	10.31	9.56	1130
Sample #18227-2	10.29	9.63	1145
Sample #18227-3	10.22	9.48	1135
Sample #18227-4	10.25	9.54	1160
Sample #18227-5	10.40	9.76	1160
Sample #18227-6	10.22	9.46	1160
Sample #18227-7	10.12	9.42	1155
Sample #18227-8	9.98	9.51	1175

Table 3: homogeneity test results of subsamples #18227

From the test results of table 3, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Manganese in mg/kg	Molybdenum in mg/kg	Phosphorus in mg/kg
r (observed)	0.35	0.30	42
reference test method	D5185:18	D5185:18	D5185:18
0.3* R (ref. test method)	0.63	0.95	44

Table 4: evaluation of the repeatabilities of subsamples #18227

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

Depending on the registration to each of the participating laboratories one 1 litre bottle, labelled #18226 and/or one 50 mL PE bottle, labelled #18227 was dispatched on October 24, 2018. A SDS was added to the sample package.

2.5   STABILITY OF THE SAMPLES

The stability of Hydraulic Fluid packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6   ANALYSES

The participants were requested to determine on sample #18226: Acid Number (Total), Density at 15°C, Flash Point PMcc, Kinematic Viscosity at 40°C and at 100°C, Viscosity Stabinger at 40°C and at 100°C, Sulfur, Water content by KF and Level of Contamination (counts/mL and scale number). The participants were requested to determine on sample #18227: 23 elements (wear metals and additives: Al, Ba, B, Cd, Cr, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, K, Si, Ag, Na, Sn, Ti, V, Ca, P and Zn).

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 test results, but report as much significant figures as possible. It was also requested not to report 'less than' 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 appropriate 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 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 June 2018 (iis-protocol, version 3.5).

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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualize 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. This 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. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The 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 according to:

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. The usual interpretation of z-scores is as follows:

$| z | < 1$ good
$1 < | z | < 2$ satisfactory
$2 < | z | < 3$ questionable
$3 < | z |$ unsatisfactory

4   EVALUATION

In this proficiency test, some problems were encountered with the dispatch of the samples. Three participants reported test results after the final reporting date and three laboratories did not report any test results at all. Not all participants were able to report all analyses requested. In total 52 participants reported test results. Of all reported numerical test results 1053 numerical test results were statistically evaluated. Observed were 49 outlying test results, which is 4.7% of the 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 original data. The abbreviations, used in these tables, are listed in appendix 4.

In the iis PT reports, 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. 7647:10). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D7647:10(2018)). In the tables of appendix 1 only the test method number and year of adoption or revision will be used.

The reference method for the analyses of wear metals is test method ASTM D5185:18. For some metals (Ba, Pb, Mg, Ni, K and P) the consensus values of the group are either above or below the respective application ranges on which the requirements of ASTM D5185:18 are based. However, it was decided to use the reproducibility from ASTM D5185:18 as the calculated reproducibility (after the rejection of the statistical outliers) is in general in good agreement with the requirements of ASTM D5185:18. More discussion per metal is given below.

Sample #18226

Acid Number (Total): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D664-A:17a (Buffer End Point, 60 and 125 mL). However, the calculated reproducibility is not in agreement with the precision data of the procedures Inflection Point (60 and 125 mL).

Density at 15°C: This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ISO12185:96.

Flash Point PMcc: This determination may be problematic depending on the test method used. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D93:18 method B but not with method A. When the test results of method A or B are evaluated separately, both calculated reproducibilities are in agreement with the requirements of ASTM D93:18 method A or B respectively. It is observed that for this type of oil sample with a flash point of 119°C the speed of heating (difference between method A or B) has a significant effect on the Flash Point determination.

Kin.Visco.at 40°C: Till 2017 a precision statement for used oils was not present in ASTM D445:15a. In the version of 2017 of ASTM D445 a precision statement is given for used (in-service) formulated oils. However, it appears that the reproducibility is very strict. It is decided to continue with the target reproducibility as calculated from the reproducibilities found in iis PT's on used oils (see appendix 4, ref. 17).
This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the average reproducibility found for used oils in previous iis PTs.

Kin.Visco.at 100°C: See explanation about selection of the target reproducibility at Kin.Visco.at 40°C. This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the average reproducibility found for used oils in previous iis PTs.

Visco. Stabinger at 40°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D7042:16e3.

Visco. Stabinger at 100°C: 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 D7042:16e3.

Sulfur: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D4294:16e1.

Water by KF: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6304:18a.

Level of Contamination: This determination was problematic. In total eighteen statistical outliers were observed over six parameters (14 for counts/mL and 4 for scale number) and another sixteen test results were excluded. However, the calculated reproducibilities of particle size ≥14 µm (counts/mL and scale number) after rejection of the suspect data are in agreement with the requirements of ASTM D7647:10(2018).

Sample #18227

Aluminum: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5185:18.
The average recovery of Aluminum (theoretical increment of 12.1 mg Al/kg) is good: "<less than 105%" (the actual blank Al content is unknown).

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

Boron: 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 Boron (theoretical increment of 10.4 mg B/kg) is good: "less than 111%" (the actual blank B content is unknown).

Copper: 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.

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

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

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

Manganese:

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

The average recovery of Manganese (theoretical increment of 10.0 mg Mn/kg) is good: "<less than 101%" (the actual blank Mn content is unknown).

Molybdenum:

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

The average recovery of Molybdenum (theoretical increment of 9.7 mg Mo/kg) is good: "<less than 101%" (the actual blank Mo content is unknown).

Nickel:

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

Potassium:

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.

Silicon:

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

The average recovery of Silicon (theoretical increment of 11.3 mg Si/kg) is good: "<less than 101%" (the actual blank Si content is unknown).

Sodium:

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

Tin:

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 Tin (theoretical increment of 9.8 mg Sn/kg) is good: "<less than 98%" (the actual blank Sn content is unknown).

Calcium:

This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D5185:18.

Phosphorus:

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

Zinc:

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

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 number of significant test results, the average results, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, EN standards) are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acid Number (Total)	mg KOH/g	40	2.49	1.17	1.09
Density at 15°C	kg/L	40	0.8903	0.0005	0.0005
Flash Point PMcc	°C	34	119	9.3	10
Kinematic Viscosity at 40°C	mm^2/s	36	38.526	0.529	0.694
Kinematic Viscosity at 100°C	mm^2/s	32	10.449	0.172	0.230
Viscosity Stabinger at 40°C	mm^2/s	18	38.517	0.552	0.534
Viscosity Stabinger at 100°C	mm^2/s	16	10.418	0.165	0.115
Sulfur	mg/kg	22	3059	671	339
Water by KF	mg/kg	37	758	471	902
Level of Contamination ≥ 4µm (c)	counts/mL	24	1154	1562	1319
Level of Contamination ≥ 6µm (c)	counts/mL	25	158	208	121
Level of Contamination ≥14µm (c)	counts/mL	21	9.9	11.0	13.5
Level of Contamination ≥ 4µm (c)	scale number	25	17.1	2.7	1.7
Level of Contamination ≥ 6µm (c)	scale number	26	14.1	2.3	1.2
Level of Contamination ≥14µm (c)	scale number	22	10.3	2.0	2.0

Table 5: reproducibilities of tests on sample #18226

Component	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	37	12.7	4.6	7.4
Barium as Ba	mg/kg	35	13.5	2.4	6.5
Boron as B	mg/kg	28	11.6	6.6	13.3
Copper as Cu	mg/kg	38	51.5	8.3	12.4
Iron as Fe	mg/kg	35	2.3	1.0	1.0
Lead as Pb	mg/kg	36	7.8	2.0	6.3
Magnesium as Mg	mg/kg	35	2.7	1.1	1.6
Manganese as Mn	mg/kg	34	10.1	2.0	2.1
Molybdenum as Mo	mg/kg	36	10.0	1.7	3.3
Nickel as Ni	mg/kg	17	0.4	0.9	1.0
Potassium as K	mg/kg	19	2.6	4.0	8.7
Silicon as Si	mg/kg	35	12.1	4.1	7.7
Sodium as Na	mg/kg	31	9.2	5.5	5.3
Tin as Sn	mg/kg	36	9.6	6.4	8.5
Calcium as Ca	mg/kg	39	479	115	46
Phosphorus as P	mg/kg	39	1230	237	151
Zinc as Zn	mg/kg	40	1400	280	240

Table 6: reproducibilities of tests on sample #18227

Without further statistical calculations, it could be concluded that for many 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 NOVEMBER 2018 WITH PREVIOUS PTS

	November 2018	November 2017	November 2016	November 2015	November 2014
Number of reporting labs	52	57	51	55	42
Number of test results reported	1053	1202	1168	1126	922
Statistical outliers	49	89	29	43	55
Percentage outliers	4.7%	7.4%	2.5%	3.8%	6.0%

Table 7: comparison with previous proficiency tests.

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 the following table.

Determination	November 2018	November 2017	November 2016	November 2015	November 2014
Acid Number (Total)	+/-	+	+	+	++
Density at 15°C	+/-	+/-	+/-	+/-	+/-
Flash Point PMcc	+/-	+/-	-	+	+/-
Kinematic Viscosity at 40°C	+	+	+	++	-
Kinematic Viscosity at 100°C	+	+	+/-	+/-	-
Viscosity Stabinger at 40°C	+/-	+	+/-	++	++
Viscosity Stabinger at 100°C	-	-	+/-	+	++
Sulfur	--	-	-	--	--
Water by KF	++	++	++	++	++
Level of Contamination – counts/mL	-	--	-	--	n.e.
Level of Contamination – scale no.	-	-	-	n.e.	n.e.
Aluminum as Al	+	(++)	++	++	++
Barium as Ba	(++)	(+)	++	+	++
Boron as B	++	++	n.e.	n.e.	n.e.
Cadmium as Cd	n.e.	+	n.e.	n.e.	n.e.
Chromium as Cr	n.e.	+/-	+	+	++
Copper as Cu	+	+	+/-	+	+/-
Iron as Fe	+/-	+	+	++	++
Lead as Pb	(++)	(++)	++	++	++
Lithium as Li	n.e.	+	(--)	-	-
Magnesium as Mg	(+)	+	+	+/-	++
Manganese as Mn	+/-	--	+/-	+/-	++
Molybdenum as Mo	++	+/-	+	+/-	+
Nickel as Ni	(+/-)	++	++	++	++
Potassium as K	(++)	(+)	n.e.	n.e.	n.e.

Determination	November 2018	November 2017	November 2016	November 2015	November 2014
Silicon as Si	+	+	++	++	++
Silver as Ag	n.e.	+	+/-	+	+
Sodium as Na	+/-	-	+/-	+/-	+
Tin as Sn	+	(++)	++	++	++
Titanium as Ti	n.e.	(++)	++	++	++
Vanadium as V	n.e.	+	+	++	++
Calcium as Ca	--	--	-	-	-
Phosphorus as P	(-)	-	+	+/-	+
Zinc as Zn	-	+/-	-	--	++

Table 8: comparison determinations against the reference test methods

The figures between brackets should be used with care as these were lower or above the application range of reference test method

++:   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

APPENDIX 1

Determination of Acid Number (Total) on sample #18226; results in mg KOH/g.

lab	method	value	mark	z(targ)	end point	volume	remarks
178	INH-1118	2.17		-0.81	---	60 mL	
179	D664-B	2.68		0.50	Inflection Point	60 mL	
225	D974	2.18		-0.78	---	---	
237	D664-A	2.51		0.06	Inflection Point	125 mL	
255		-----		-----	---	---	
311		-----		-----	---	---	
317	D974	1.69		-2.03	---	---	
325	D664-A	2.67		0.47	Buffer End Point (pH 11)	125 mL	
331	D664Mod.	2.76		0.70	Inflection Point	---	
335		-----		-----	---	---	
339		-----		-----	---	---	
349	D664-A	2.84		0.91	Inflection Point	125 mL	
360	D664-A	2.400		-0.22	Inflection Point	60 mL	
398	D664-A	1.73		-1.93	---	---	
432		-----		-----	---	---	
442		-----		-----	---	---	
451		1.88		-1.55	Buffer End Point (pH 11)	60 mL	
496	D664-A	2.46		-0.06	Buffer End Point (pH 11)	60 mL	
529	D664-A	2.64		0.40	Buffer End Point (pH 11)	125 mL	
541		-----		-----	---	---	
562	D664	2.695		0.54	---	60 mL	
575	D664-A	1.69		-2.03	Buffer End Point (pH 11)	60 mL	
603	D664-A	2.7016		0.55	Inflection Point	125 mL	
614	D664-A	2.85		0.93	---	60 mL	
634		-----		-----	---	---	
663	D664-A	2.390		-0.24	Buffer End Point (pH 11)	125 mL	
780	D664-A	2.94		1.16	Inflection Point	60 mL	
862	D664-A	2.89		1.04	Inflection Point	60 mL	
902	D664-A	1.90		-1.50	Inflection Point	60 mL	
912	D664-A	2.4		-0.22	---	---	
962	D974	2.39		-0.24	---	---	
963	D664-A	2.41		-0.19	Inflection Point	60 mL	
994	D664-A	2.99		1.29	Inflection Point	125 mL	
1059	ISO6619	3.44		2.44	Buffer End Point (pH 11)	60 mL	
1094	D664-A	2.3414		-0.37	Buffer End Point (pH 11)	60 mL	
1146	D664-A	2.758		0.70	Buffer End Point (pH 11)	125 mL	
1166	D664-A	3.0518		1.45	Inflection Point	60 mL	
1397	D664-A	2.59		0.27	Inflection Point	125 mL	
1435	D664-A	2.609		0.32	Buffer End Point (pH 11)	---	
1448		-----		-----	---	---	
1505	D664-A	2.910		1.09	Inflection Point	---	
1660		-----		-----	---	---	
1720	D974	1.8		-1.75	---	---	
1740	D664-A	2.34		-0.37	Inflection Point	60 mL	
1752		-----		-----	---	---	
1788		-----		-----	---	---	
1888	D664-A	2.312		-0.44	Buffer End Point (pH 11)	60 mL	
1890		-----		-----	---	---	
1900	D664-A	1.86		-1.60	---	60 mL	
1920	D664-A	2.330		-0.40	---	---	
1941	ISO6619	2.84		0.91	Inflection Point	60 mL	
6016	D664-A	2.332		-0.39	---	---	
6140	D664-A	3.041		1.42	Inflection Point	---	

				IP (60+125 mL) only	BEP pH 11 (60+125 mL) only
normality	OK		suspect	suspect
n	40		17	11
outliers	0		0	0
mean (n)	2.4853		2.6343	2.4719
st.dev. (n)	0.41913		0.39081	0.46006
R(calc.)	1.1736		1.0943	1.2882
st.dev.(D664-A:17a BEP 60 mL)	0.39088		--	0.38882
R(D664-A:17a BEP 60 mL)	1.0945		--	1.0887
compare				
R(D664-A:17a IP 60 mL)	0.8484		0.8899	--
R(D664-A:17a IP 125 mL)	0.4914		0.5124	--
R(D664-A:17a BEP 125 mL)	1.0935		--	1.0876

Kernel Density
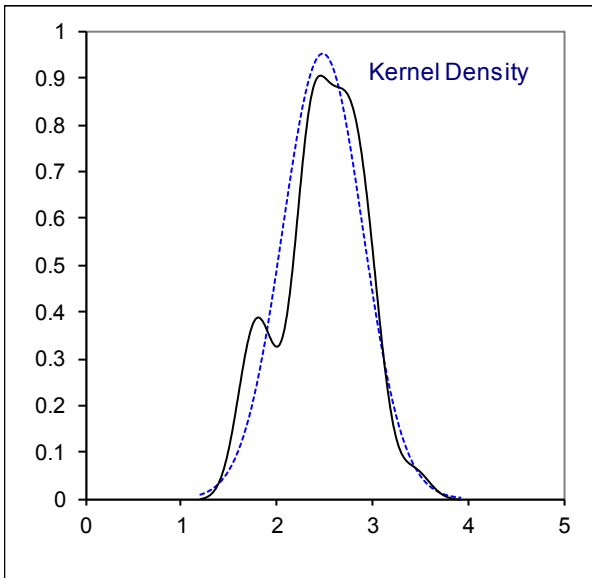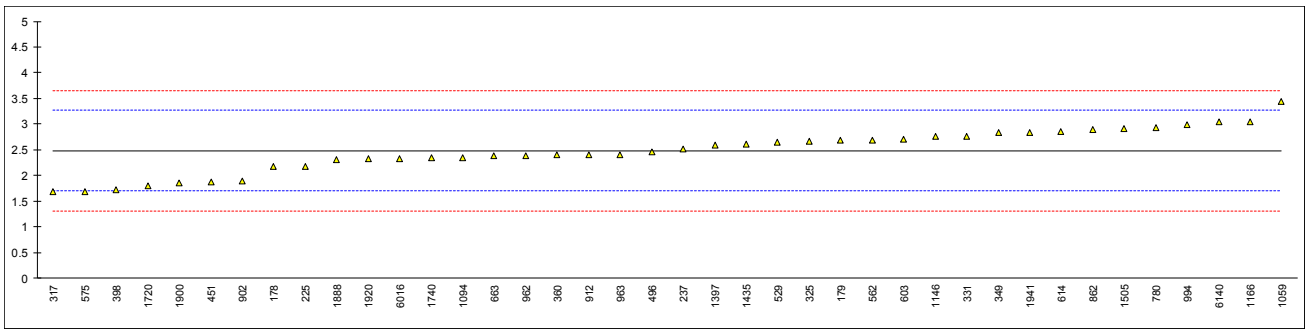
Determination of Density at 15°C on sample #18226; results in kg/L.

lab	method	value	mark	z(targ)	remarks
178	D4052	0.8899		-2.20	
179	D4052	0.8908		2.84	
225		-----		-----	
237	D4052	0.8903		0.04	
255		-----		-----	
311	D4052	0.8903		0.04	
317	D4052	0.8903		0.04	
325	D4052	0.8886	R(0.01)	-9.48	
331	ISO12185	0.88950	R(0.05)	-4.44	
335		-----		-----	
339		-----		-----	
349	D4052	0.8902		-0.52	
360	ISO12185	0.8903		0.04	
398	ISO12185	0.8903		0.04	
432	ISO12185	0.89051		1.21	
442		-----		-----	
451	D4052	0.8900		-1.64	
496	ISO12185	0.89027		-0.13	
529	D4052	0.8903		0.04	
541		-----		-----	
562	D4052	0.8909		3.40	
575		-----		-----	
603	D4052	0.8904		0.60	
614	D4052	0.8904		0.60	
634	D4052	0.89028		-0.07	
663	D4052	0.89024		-0.30	
780	ISO12185	0.8903		0.04	
862	D4052	0.8904		0.60	
902	D4052	0.8903		0.04	
912	D4052	0.8903		0.04	
962	D4052	0.8903		0.04	
963	D4052	0.8903		0.04	
994	ISO12185	0.8901		-1.08	
1059	ISO12185	0.8903		0.04	
1094	D4052	0.89027		-0.13	
1146	D4052	0.89027		-0.13	
1166	In house	0.891	C,R(0.05)	3.96	first reported 0.888 (at 21°C)
1397	D4052	0.8904		0.60	
1435	D4052	0.8900		-1.64	
1448	D4052	0.8903		0.04	
1505	D7042	0.8903	C	0.04	reported 0.8903 kg/m^3
1660		-----		-----	
1720	D4052	0.8901		-1.08	
1740	D7042	0.8903		0.04	
1752	D4052	0.89007		-1.25	
1788	D4052	0.89027		-0.13	
1888	ISO3675	0.893	R(0.01)	15.16	
1890	ISO12185	0.8904		0.60	
1900	D4052	0.8903		0.04	
1920		-----		-----	
1941	D4052	0.89012		-0.97	
6016	D4052	0.89032		0.15	
6140	D7042	0.8903	C	0.04	reported 0.8903 kg/m^3

normality	not OK
n	40
outliers	4
mean (n)	0.89029
st.dev. (n)	0.000177
R(calc.)	0.00050
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005
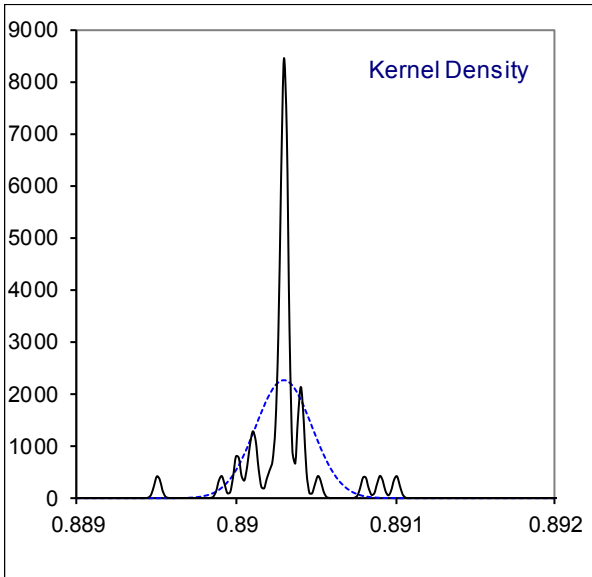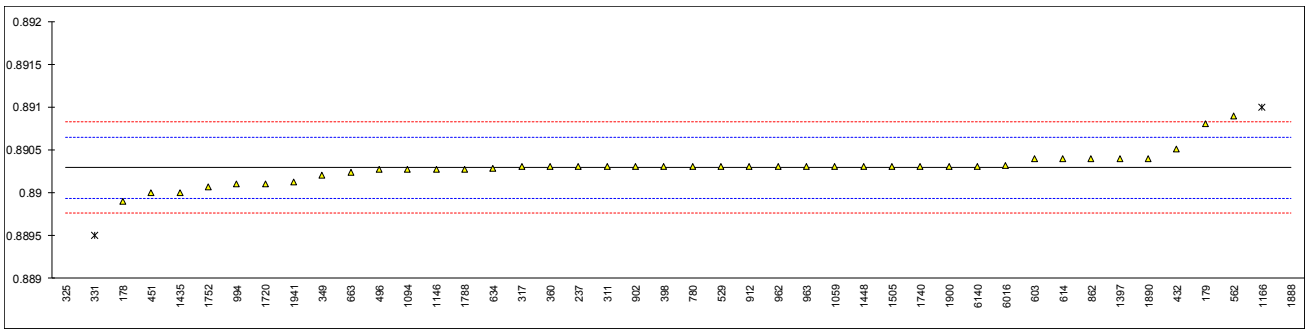
Determination of Flash Point PMcc on sample #18226; results in °C.

lab	method	value	mark	z(targ)	remarks
178	D93-A	119.5		0.07	
179	D93-A	120	C	0.21	first reported 138.0
225	D93-B	118.0		-0.35	
237	D93-B	122		0.77	
255		-----		-----	
311		-----		-----	
317		-----		-----	
325	D93-A	115		-1.19	
331	D93-A	122.5		0.91	
335		-----		-----	
339		-----		-----	
349		-----		-----	
360	D93-B	120.0		0.21	
398	D93-A	122		0.77	
432		-----		-----	
442		-----		-----	
451	D93-A	120		0.21	
496	D93-B	120.2		0.27	
529	D93-A	116		-0.91	
541		-----		-----	
562		-----		-----	
575		-----		-----	
603	D3828	118.0		-0.35	
614	D93-A	125		1.61	
634	D93-A	114.0		-1.47	
663	D93-A	119.38		0.04	
780	D93-B	127.5		2.31	
862	D93-A	113		-1.75	
902	D93-A	119.0		-0.07	
912		-----		-----	
962	D93-A	118		-0.35	
963	D93-A	120.0		0.21	
994	D93-B	125.0		1.61	
1059	ISO2719-A	115.5		-1.05	
1094	D93-A	115.0		-1.19	
1146	D93-A	116.2		-0.85	
1166		-----		-----	
1397	D93-A	115.0		-1.19	
1435	D93-A	117.0		-0.63	
1448		-----		-----	
1505	D93-A	122		0.77	
1660		-----		-----	
1720	D93-A	122.5		0.91	
1740	D93-B	119.5		0.07	
1752		-----		-----	
1788	D93-B	119.0		-0.07	
1888	ISO2719-A	121		0.49	
1890		-----		-----	
1900	D7094	117.6		-0.46	
1920		-----		-----	
1941		-----		-----	
6016	D93-B	121.9		0.74	
6140	D93-A	118		-0.35	

				D93-A / ISO2719-A only	D93-B only
normality	OK			OK	suspect
n	34			23	9
outliers	0			0	0
mean (n)	119.24			118.50	121.46
st.dev. (n)	3.304			3.193	3.064
R(calc.)	9.25			8.94	8.58
st.dev.(D93-B:18)	3.571			--	3.571
R(D93-B:18)	10			--	10
compare					
R(D93-A:18)	8.47			8.41	--

Kernel Density

Determination of Kinematic Viscosity at 40°C on sample #18226; results in mm^2/s.

lab	method	value	mark	z(targ)	remarks
178	D445	38.30		-0.91	
179	D445	38.52		-0.03	
225	D445	38.56		0.14	
237	D445	38.44		-0.35	
255	D7279	37.69	R(0.05)	-3.38	corrected to D445
311	D445	38.42		-0.43	
317	D445	38.47		-0.23	
325	D445	38.45		-0.31	
331	D7279Mod.	37.78	R(0.05)	-3.01	
335		-----		-----	
339		-----		-----	
349	D445	39.27	R(0.05)	3.00	
360	D445	38.496		-0.12	
398		-----		-----	
432	D445	38.48		-0.19	
442		-----		-----	
451		-----		-----	
496	D445	38.615		0.36	
529	D445	38.47		-0.23	
541		-----		-----	
562	D7279	38.72333		0.80	
575	D7279	38.51		-0.07	corrected to D445
603		-----		-----	
614	D445	38.59		0.26	
634	D7279	38.09		-1.76	corrected to D445
663	D445	38.419		-0.43	
780	D445	38.46		-0.27	
862	D445	38.50		-0.11	
902	D445	38.52		-0.03	
912	D445	38.64		0.46	
962	D445	38.81		1.15	
963	D445	38.51		-0.07	
994	D445	38.45		-0.31	
1059	ISO3104	38.37		-0.63	
1094	D445	38.455		-0.29	
1146	D445	38.48		-0.19	
1166	ISO3104	38.70702		0.73	
1397		-----		-----	
1435		-----		-----	
1448		-----		-----	
1505	D445	38.832		1.23	
1660		-----		-----	
1720		-----		-----	
1740	D445	38.50		-0.11	
1752	D7279	38.58		0.22	corrected to D445
1788	D445	38.71		0.74	
1888	D445	40.37	R(0.01)	7.44	
1890	ISO3104	38.414		-0.45	
1900	D445	37.99		-2.17	
1920	D445	38.968		1.78	
1941	ISO3104	38.663		0.55	
6016		-----		-----	
6140	D445	38.834		1.24	

normality	suspect	
n	36	
outliers	4	
mean (n)	38.5263	
st.dev. (n)	0.18880	
R(calc.)	0.5287	
st.dev.(iis:15)	0.24767	
R(iis:15)	0.6935	see appendix 4, ref 17
compare		
R(D445:17a)	0.3195	

Kernel Density
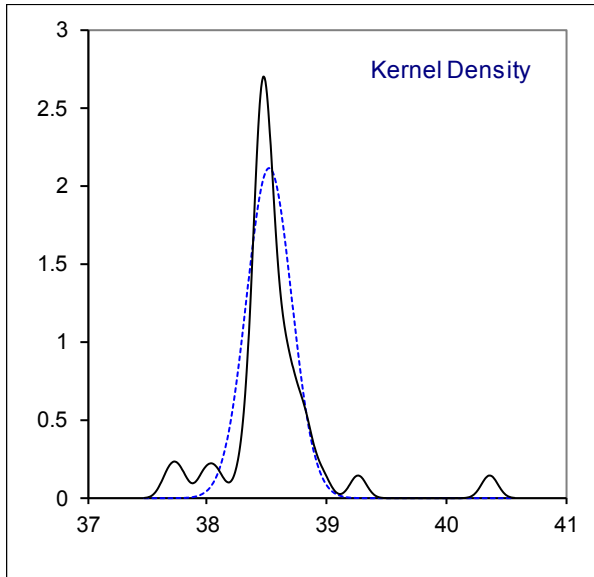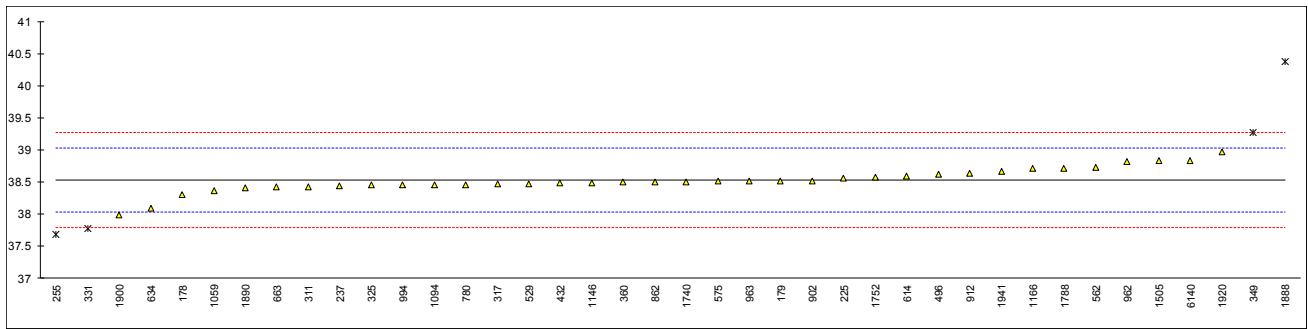
Determination of Kinematic Viscosity at 100°C on sample #18226; results in mm^2/s.

lab	method	value	mark	z(targ)	remarks
178	D445	10.49		0.49	
179	D445	10.51		0.74	
225	D445	10.49		0.49	
237	D445	10.45		0.01	
255	D7279	10.68	R(0.05)	2.81	corrected to D445
311	D445	10.41		-0.48	
317		-----		-----	
325	D445	10.43		-0.24	
331	D7279Mod.	10.60		1.83	
335		-----		-----	
339		-----		-----	
349	D445	10.44	C	-0.11	first reported 8.703
360	D445	10.462		0.15	
398		-----		-----	
432	D445	10.45		0.01	
442		-----		-----	
451		-----		-----	
496	D445	10.435		-0.18	
529	D445	10.42		-0.36	
541		-----		-----	
562	D7279	10.43166		-0.22	
575	D7279	10.30		-1.82	corrected to D445
603		-----		-----	
614	D445	10.7	C,R(0.05)	3.05	first reported 10.90
634	D7279	10.477		0.34	corrected to D445
663	D445	10.413		-0.44	
780	D445	10.54		1.10	
862	D445	10.46		0.13	
902	D445	10.45		0.01	
912	D445	10.48		0.37	
962		-----		-----	
963	D445	10.51		0.74	
994		-----		-----	
1059	ISO3104	10.49		0.49	
1094	D445	10.492		0.52	
1146	D445	10.411		-0.47	
1166		-----		-----	
1397		-----		-----	
1435		-----		-----	
1448		-----		-----	
1505	D445	10.404		-0.55	
1660		-----		-----	
1720		-----		-----	
1740	D445	10.52		0.86	
1752	D7279	10.485		0.43	corrected to D445
1788	D445	10.38		-0.85	
1888	D445	10.9	R(0.01)	5.49	
1890	ISO3104	10.418		-0.38	
1900	D445	10.29		-1.94	
1920		-----		-----	
1941	ISO3104	10.425		-0.30	
6016		-----		-----	
6140	D445	10.418		-0.38	

	normality	suspect	
	n	32	
	outliers	3	
	mean (n)	10.4494	
	st.dev. (n)	0.06133	
	R(calc.)	0.1717	
	st.dev.(iis:15)	0.08210	
	R(iis:15)	0.2299	see appendix 4, ref 17
	compare		
	R(D445:17a)	0.1042	

Kernel Density
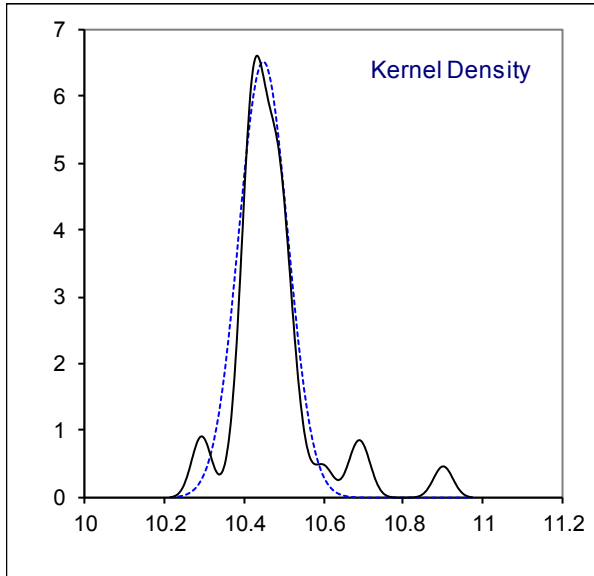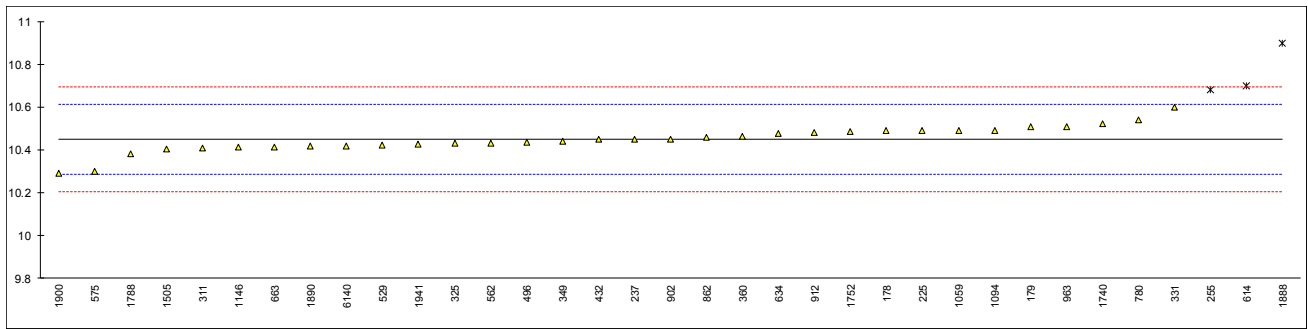
Determination of Viscosity Stabinger at 40°C on sample #18226; results in mm^2/s.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D7042	38.60		0.44	
225		-----		-----	
237		-----		-----	
255		-----		-----	
311		-----		-----	
317		-----		-----	
325		-----		-----	
331		-----		-----	
335		-----		-----	
339		-----		-----	
349		-----		-----	
360	D7042	38.517		0.00	
398	D7042	38.15		-1.92	
432		-----		-----	
442		-----		-----	
451		-----		-----	
496	D7042	38.488		-0.15	
529		-----		-----	
541		-----		-----	
562		-----		-----	
575		-----		-----	
603	D7042	38.637		0.63	
614		-----		-----	
634		-----		-----	
663		-----		-----	
780	D7042	38.27		-1.29	
862	D7042	38.63		0.59	
902		-----		-----	
912		-----		-----	
962		-----		-----	
963	D7042	38.06		-2.39	
994	D7042	38.48		-0.19	
1059	D7042	38.73		1.12	
1094		-----		-----	
1146		-----		-----	
1166		-----		-----	
1397	D7042	38.49		-0.14	
1435	D7042	38.674		0.83	
1448	D7042	38.57		0.28	
1505	D7042	38.616		0.52	
1660		-----		-----	
1720	D7042	38.88		1.91	
1740	D7042	38.475		-0.22	
1752		-----		-----	
1788		-----		-----	
1888		-----		-----	
1890		-----		-----	
1900		-----		-----	
1920		-----		-----	
1941		-----		-----	
6016	D7042	38.556		0.21	
6140	D7042	38.477		-0.21	

|		
normality	suspect	
n	18	
outliers	0	
mean (n)	38.5167	
st.dev. (n)	0.19716	
R(calc.)	0.5520	
st.dev.(D7042:16e3)	0.19068	
R(D7042:16e3)	0.5339	
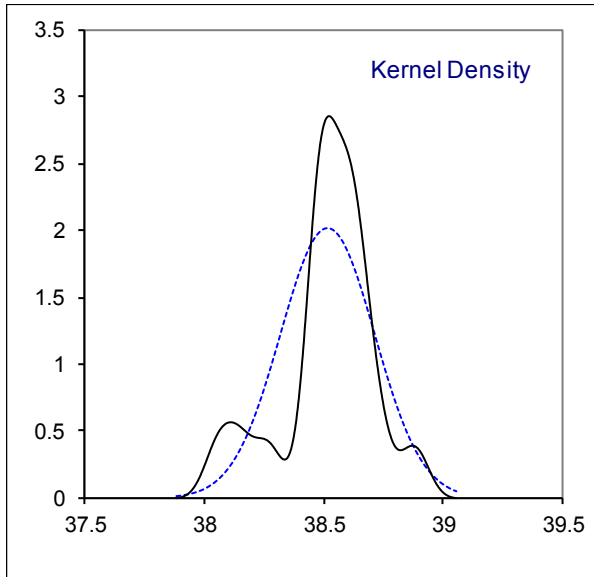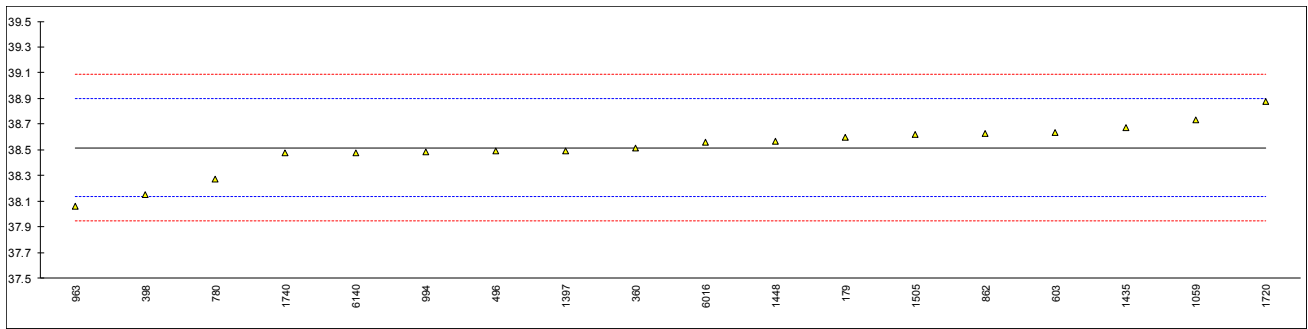
Determination of Viscosity Stabinger at 100°C on sample #18226; results in mm^2/s.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D7042	10.52		2.47	
225		-----		-----	
237		-----		-----	
255		-----		-----	
311		-----		-----	
317		-----		-----	
325		-----		-----	
331		-----		-----	
335		-----		-----	
339		-----		-----	
349		-----		-----	
360	D7042	10.474		1.35	
398	D7042	10.31		-2.62	
432		-----		-----	
442		-----		-----	
451		-----		-----	
496	D7042	10.391		-0.66	
529		-----		-----	
541		-----		-----	
562		-----		-----	
575		-----		-----	
603	D7042	10.496		1.89	
614		-----		-----	
634		-----		-----	
663		-----		-----	
780	D7042	9.949	D(0.01)	-11.38	
862	D7042	10.47		1.26	
902		-----		-----	
912		-----		-----	
962		-----		-----	
963	D7042	10.44		0.53	
994		-----		-----	
1059	D7042	10.41		-0.20	
1094		-----		-----	
1146		-----		-----	
1166		-----		-----	
1397	D7042	10.37		-1.17	
1435	D7042	10.433		0.36	
1448	D7042	10.45		0.77	
1505	D7042	10.325		-2.26	
1660		-----		-----	
1720	D7042	10.451		0.80	
1740	D7042	10.39		-0.68	
1752		-----		-----	
1788		-----		-----	
1888		-----		-----	
1890		-----		-----	
1900		-----		-----	
1920		-----		-----	
1941		-----		-----	
6016	D7042	10.390		-0.68	
6140	D7042	10.371		-1.14	

	normality	OK	
	n	16	
	outliers	1	
	mean (n)	10.4182	
	st.dev. (n)	0.05907	
	R(calc.)	0.1654	
	st.dev.(D7042:16e3)	0.04122	
	R(D7042:16e3)	0.1154	

Kernel Density
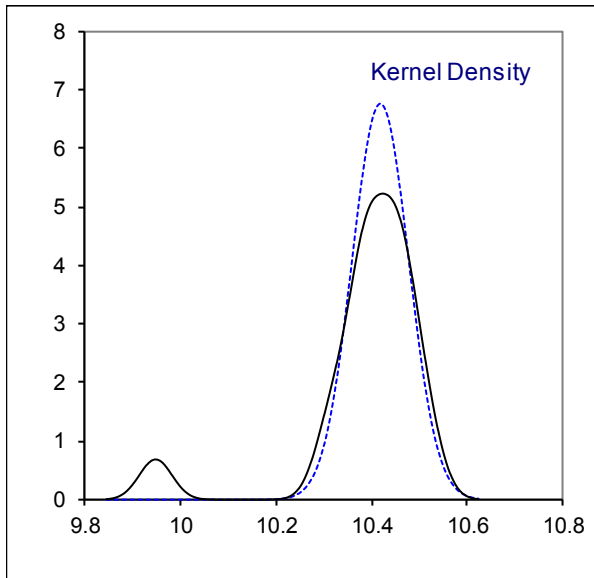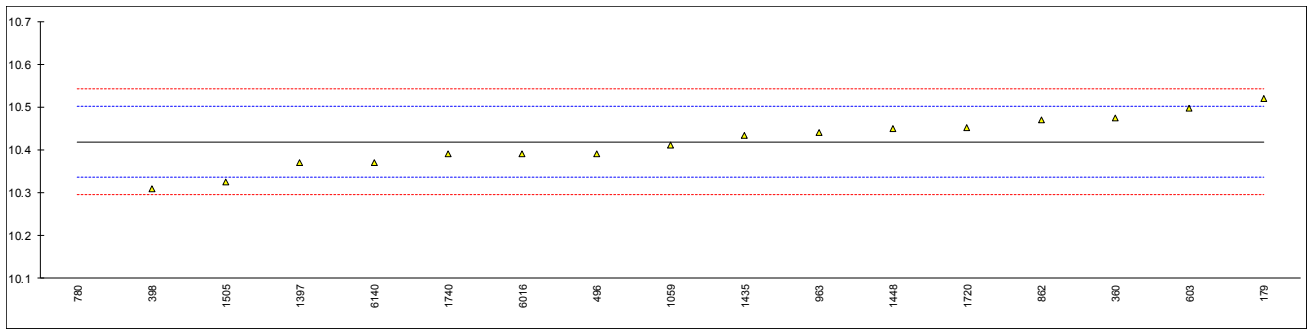
Determination of Sulfur on sample #18226; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4294	3167		0.89	
225		-----		-----	
237	D4294	3368		2.56	
255		-----		-----	
311		-----		-----	
317		-----		-----	
325		-----		-----	
331	ISO8754	3020		-0.32	
335		-----		-----	
339	In house	2750		-2.55	
349		-----		-----	
360	D4294	2950		-0.90	
398		-----		-----	
432	D5185	3106		0.39	
442		-----		-----	
451		-----		-----	
496	D2622	3027		-0.26	
529		-----		-----	
541		-----		-----	
562		-----		-----	
575		-----		-----	
603		-----		-----	
614		-----		-----	
634	D4294	3168		0.90	
663		-----		-----	
780	D4294	3240		1.50	
862	D4294	2930		-1.07	
902	D4294	3527		3.87	
912		-----		-----	
962		-----		-----	
963		-----		-----	
994	D4294	3329		2.23	
1059	ISO14596	3000		-0.49	
1094		-----		-----	
1146	D4294	2720	C	-2.80	first reported 0.272 mg/kg
1166	In house	2998.99		-0.50	
1397	D2622	3000	C	-0.49	first reported 0.30 mg/kg
1435	D5185	3875	D(0.05)	6.75	
1448		-----		-----	
1505	D5185	2670		-3.22	
1660		-----		-----	
1720	D4294	3170	C	0.92	reported 0.317 mg/kg
1740	D4294	3000		-0.49	
1752		-----		-----	
1788		-----		-----	
1888	D5185	3062.9		0.03	
1890		-----		-----	
1900		-----		-----	
1920		-----		-----	
1941	ISO8754	3460		3.32	
6016		-----	W	-----	first reported 3433.9
6140	D1266	2633		-3.52	

	normality	OK			
	n	22			
	outliers	1			
	mean (n)	3058.9			
	st.dev. (n)	239.78			
	R(calc.)	671.4			
	st.dev.(D4294:16e1)	120.93			
	R(D4294:16e1)	338.6			

Kernel Density
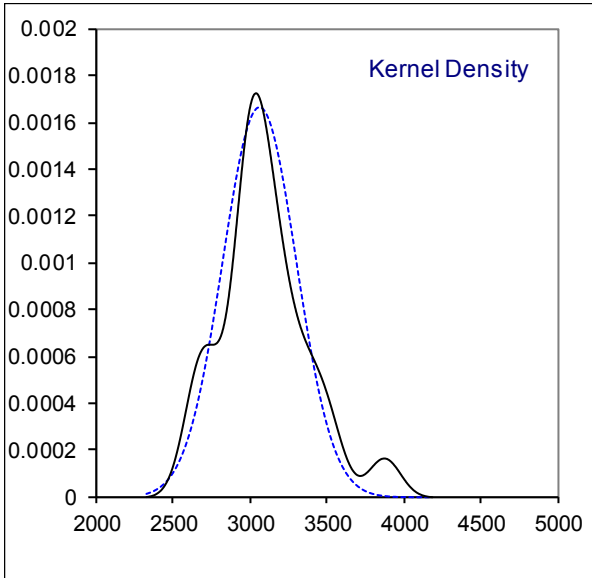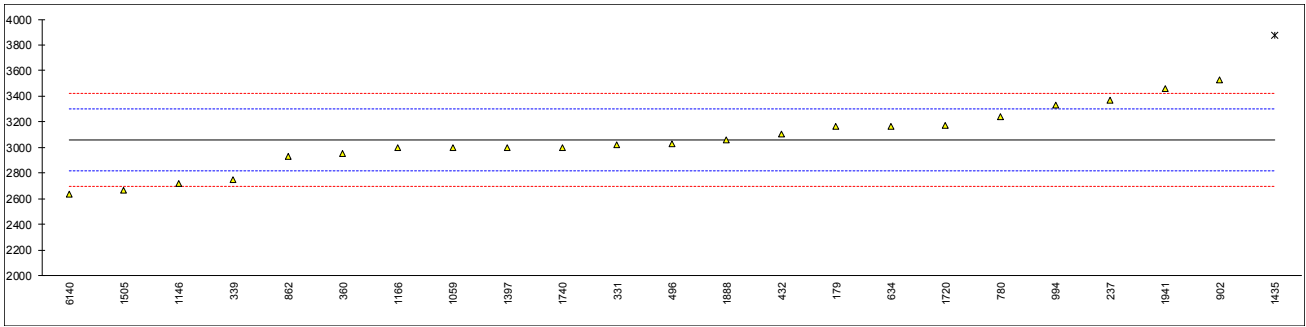
Determination of Water by KF on sample #18226; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D6304-C	659		-0.31	
179	D6304-C	721		-0.11	
225	D6304-A	887		0.40	
237		-----		-----	
255		-----		-----	
311	D6304-A	920		0.50	
317	D6304-A	840		0.26	
325	D6304-C	536.5		-0.69	
331	In house	629.3		-0.40	
335		-----		-----	
339		-----		-----	
349	D6304-A	928		0.53	
360	D6304-A	730.0		-0.09	
398	D6304-C	728		-0.09	
432	D6304-C	646.8		-0.34	
442	IP438	825		0.21	
451	D6304-C	570		-0.58	
496	D6304-C	589		-0.52	
529		-----		-----	
541		-----		-----	
562		-----		-----	
575		-----		-----	
603	D6304-C	607.2		-0.47	
614	D6304-C	567		-0.59	
634	D6304-A	800		0.13	
663	D6304-C	596.05		-0.50	
780	D6304-C	627		-0.41	
862	D6304-C	613.9		-0.45	
902		-----		-----	
912	D6304-C	630		-0.40	
962	D6304-A	941		0.57	
963	D6304-A	955		0.61	
994		-----		-----	
1059		-----		-----	
1094	D6304-A	1167		1.27	
1146	D6304-C	648		-0.34	
1166	In house	624.5	C	-0.41	first reported 0.06245 mg/kg
1397	ISO12937	1260		1.56	
1435	D6304-A	825.1		0.21	
1448		-----		-----	
1505	D6304-C	827.4		0.22	
1660		-----		-----	
1720		-----		-----	
1740	D6304-C	631		-0.39	
1752		-----		-----	
1788		-----		-----	
1888	EN60814	873.2		0.36	
1890	EN60814	945.74		0.58	
1900	D6304-C	759		0.00	
1920	D6304-C	691		-0.21	
1941	D6304-C	650		-0.33	
6016	D6304-A	679.2		-0.24	
6140	D6304-C	904.3		0.46	

normality	not OK	
n	37	
outliers	0	
mean (n)	757.627	
st.dev. (n)	168.2016	
R(calc.)	470.964	
st.dev.(D6304:18a)	322.2465	
R(D6304:18a)	902.290	

Kernel Density
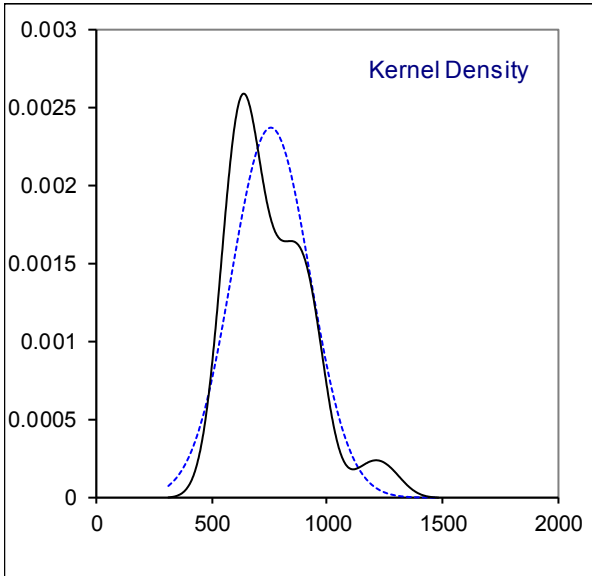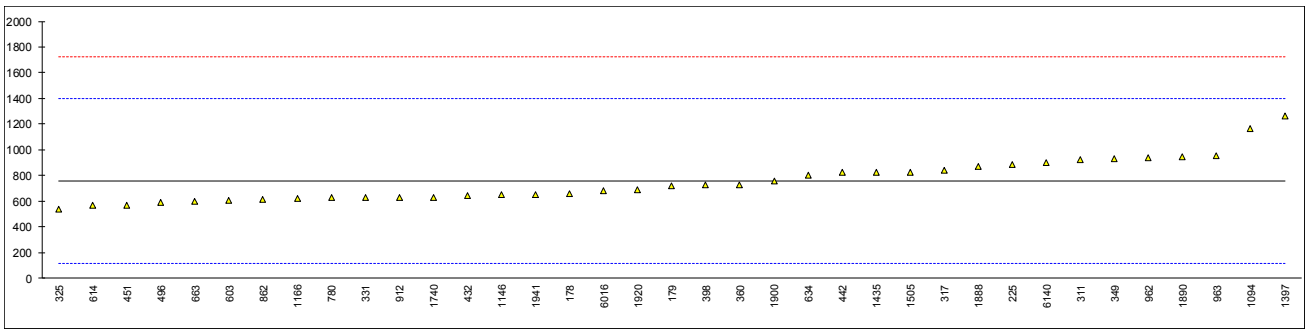
Determination of Level of Contamination on sample #18226; results in counts/mL.

lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178	INH-1185	1066		-0.19	76		-1.89	7		-0.60
179	D7647	1878		1.54	278		2.78	43	R(0.01)	6.89
225		-----		-----	-----		-----	-----		-----
237				-----	-----		-----	-----		-----
255		-----		-----	-----		-----	-----		-----
311		-----		-----	-----		-----	-----		-----
317		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
335		-----		-----	-----		-----	-----		-----
339		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
360	ISO4406	1880.7		1.54	140.6		-0.40	10.8		0.19
398		971	C	-0.39	119	C	-0.89	8	C	-0.39
432	ISO11500	1293		0.29	202		1.02	11		0.24
442		-----		-----	-----		-----	-----		-----
451		906		-0.53	62		-2.21	1	ex	-1.84
496	D7647	469		-1.46	42		-2.67	3		-1.43
529		-----		-----	-----		-----	-----		-----
541		-----		-----	-----		-----	-----		-----
562	ISO4406	686		-0.99	186		0.65	15		1.07
575	ISO11500	1442.2		0.61	295.0		3.17	50.2	R(0.01)	8.39
603		-----		-----	-----		-----	-----		-----
614		897	C	-0.55	136	C	-0.50	13	C	0.65
634	ISO11500	1802		1.38	232		1.72	99	R(0.01)	18.54
663	D7647	365.7		-1.67	123.1		-0.80	18.73		1.84
780	ISO11500	1803		1.38	173		0.35	10		0.03
862	ISO11500	1410		0.54	152		-0.13	12		0.44
902	D7647	419.9		-1.56	95.4		-1.44	9.2		-0.14
912	D7647	771		-0.81	72		-1.98	7		-0.60
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1059	D7647	1006		-0.32	229		1.65	15		1.07
1094	D7647	1208.65		0.12	137.30		-0.47	7.92		-0.40
1146	ISO11500	1600		0.95	210		1.21	14		0.86
1166	D7647	1134.47		-0.04	134.97		-0.53	7.13		-0.57
1397	D7647	3717.5	R(0.01)	5.44	157.6		0.00	4.8		-1.05
1435	D7647	2394.27		2.63	272.93		2.66	12.63		0.58
1448		-----		-----	-----		-----	-----		-----
1505	D7647	3900	ex	5.83	1525	R(0.01)	31.60	271	R(0.01)	54.32
1660		-----		-----	-----		-----	-----		-----
1720		-----		-----	-----		-----	-----		-----
1740		-----		-----	-----		-----	-----		-----
1752		1445.3		0.62	262.6		2.42	7.1		-0.57
1788		-----		-----	-----		-----	-----		-----
1888	ISO4407	38063.0	R(0.01)	78.38	5294.7	R(0.01)	118.73	1207.0	R(0.01)	249.02
1890		-----		-----	-----		-----	-----		-----
1900	D7596	300		-1.81	74		-1.93	9		-0.18
1920	D7596	555.9833		-1.27	79.8433		-1.80	4.8333		-1.05
1941		-----		-----	-----		-----	-----		-----
6016		3690	ex	5.38	1450	C,R(0.01)	29.87	232	C,R(0.01	46.21
6140	D7647	18382	R(0.01)	36.58	7147	R(0.01)	161.54	1270	R(0.01)	262.13
	normality	OK			OK			OK		
	n	24			25			21		
	outliers	3 (+2 ex)			4			7 (+1 ex)		
	mean (n)	1154.382			157.694			9.864		
	st.dev. (n)	557.9304			74.1279			3.9416		
	R(calc.)	1562.205			207.558			11.037		
	st.dev.(D7647:10)	470.9213			43.2661			4.8073		
	R(D7647:10)	1318.580			121.145			13.461		

Lab 398: first reported as scale number
Lab 451: test value excluded as statistical outlier in scale number
Lab 614: first reported as scale number: 769, 75 and 2 respectively
Lab 1505: test value excluded as two other test results are statistical outliers
Lab 6016: first reported 232,1450. Test value excluded as two other test results are statistical outliers
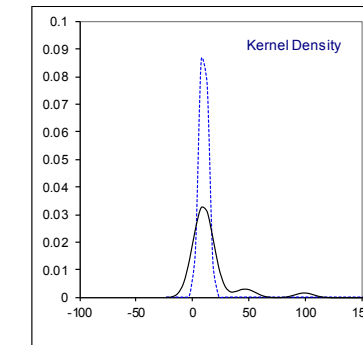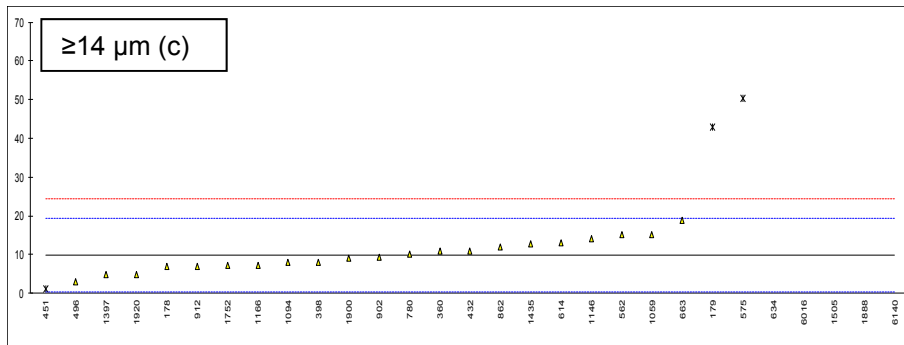
Determination of Level of Contamination acc. to ISO4406 scale on sample #18226;
results in scale number.

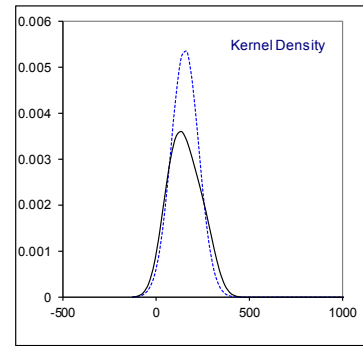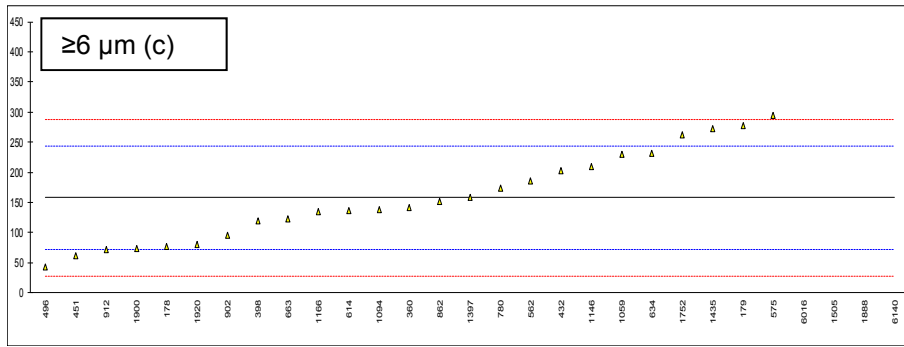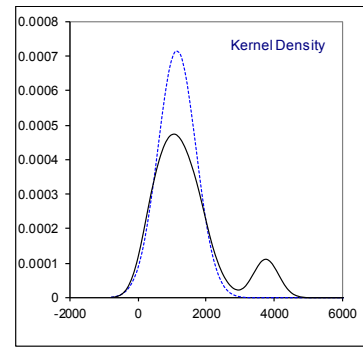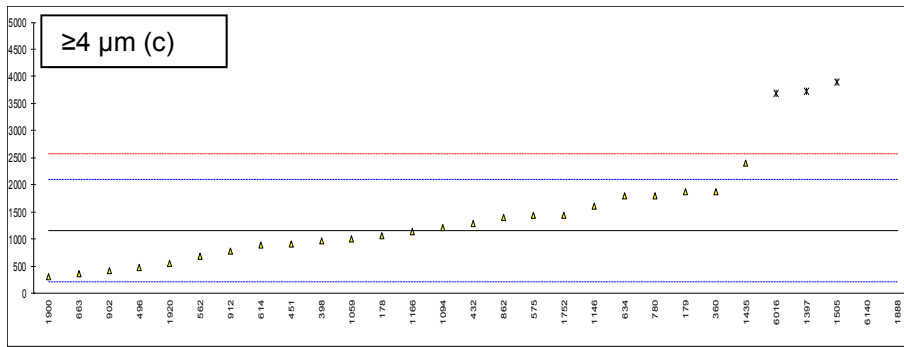| lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178	ISO4406	18		1.53	13		-2.64	10		-0.44
179	D7647	18		1.53	15		1.98	13	ex	3.71
225		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
255		-----		-----	-----		-----	-----		-----
311		-----		-----	-----		-----	-----		-----
317		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
335		-----		-----	-----		-----	-----		-----
339		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
360	ISO4406	18		1.53	14		-0.33	11		0.94
398	ISO4406	-----		-----	-----		-----	-----		-----
432	ISO4406	17		-0.10	15		1.98	11		0.94
442		-----		-----	-----		-----	-----		-----
451		17		-0.10	13		-2.64	7	R(0.01)	-4.60
496	ISO4406	16		-1.73	13		-2.64	9		-1.83
529	ISO4406	17		-0.10	14		-0.33	10		-0.44
541		-----		-----	-----		-----	-----		-----
562	ISO4406	16.5		-0.91	14.75		1.40	11		0.94
575	ISO4406	18		1.53	15		1.98	13	ex	3.71
603		-----		-----	-----		-----	-----		-----
614	ISO4406	-----		-----	-----		-----	-----		-----
634	ISO4406	18		1.53	15		1.98	14	ex	5.10
663	ISO4406	16		-1.73	14		-0.33	11		0.94
780	ISO11500	18		1.53	15		1.98	11		0.94
862	ISO4406	18		1.53	14		-0.33	11		0.94
902	ISO4406	16		-1.73	14		-0.33	10		-0.44
912	ISO4406	17		-0.10	13		-2.64	10		-0.44
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1059	D7647	17		-0.10	15		1.98	11		0.94
1094	ISO4406	17		-0.10	14		-0.33	10		-0.44
1146	ISO4406	18		1.53	15		1.98	11		0.94
1166	ISO4406	17		-0.10	14		-0.33	10		-0.44
1397	D7647	19	ex	3.16	14		-0.33	9		-1.83
1435	ISO4406	18		1.53	15		1.98	11		0.94
1448		-----		-----	-----		-----	-----		-----
1505	ISO4406	19	ex	3.16	18	R(0.01)	8.90	15	ex	6.48
1660		-----		-----	-----		-----	-----		-----
1720		-----		-----	-----		-----	-----		-----
1740	ISO4406	15		-3.36	13		-2.64	10		-0.44
1752		18		1.53	15		1.98	10		-0.44
1788		-----		-----	-----		-----	-----		-----
1888	ISO4406	19	ex	3.16	16	ex	4.28	14	ex	5.10
1890		-----		-----	-----		-----	-----		-----
1900		15		-3.36	13		-2.64	10		-0.44
1920	ISO4406	16		-1.73	13		-2.64	9		-1.83
1941	ISO4406	17		-0.10	15		1.98	11		0.94
6016	ISO4406	19	ex	3.16	18	R(0.01)	8.90	15	ex	6.48
6140	ISO4406	21	ex	6.42	20	R(0.01)	13.52	17	ex	9.25
	normality	OK			OK			OK		
	n	25			26			22		
	outliers	0 (+5 ex)			3 (+1 ex)			1 (+7 ex)		
	mean (n)	17.06			14.14			10.318		
	st.dev. (n)	0.961			0.825			0.7162		
	R(calc.)	2.69			2.31			2.005		
	st.dev.(D7647:10)	0.614			0.433			0.7220		
	R(D7647:10)	1.72			1.21			2.022		

Lab 179: excluded as statistical outlier in counts/mL
Lab 575: excluded as statistical outlier in counts/mL
Lab 634: excluded as statistical outlier in counts/mL
Lab 1397: excluded as statistical outlier in counts/mL
Lab 1505: excluded as statistical outliers in counts/mL
Lab 1888: excluded as statistical outliers in counts/mL
Lab 6016: excluded as statistical outliers in counts/mL
Lab 6140: excluded as statistical outliers in counts/mL
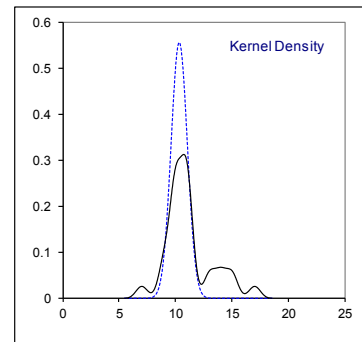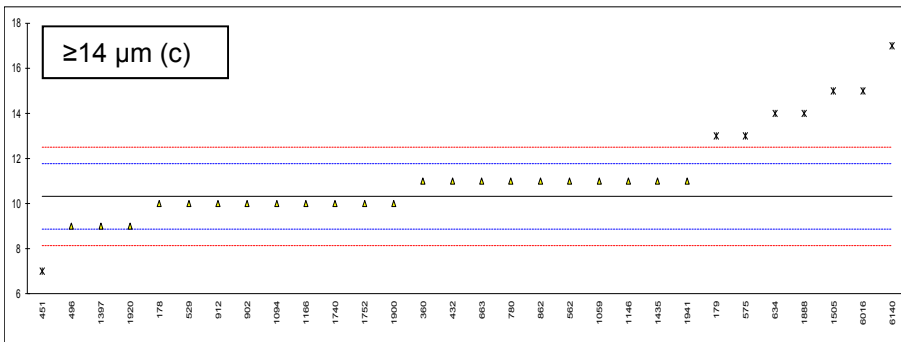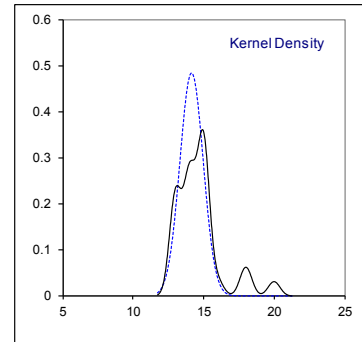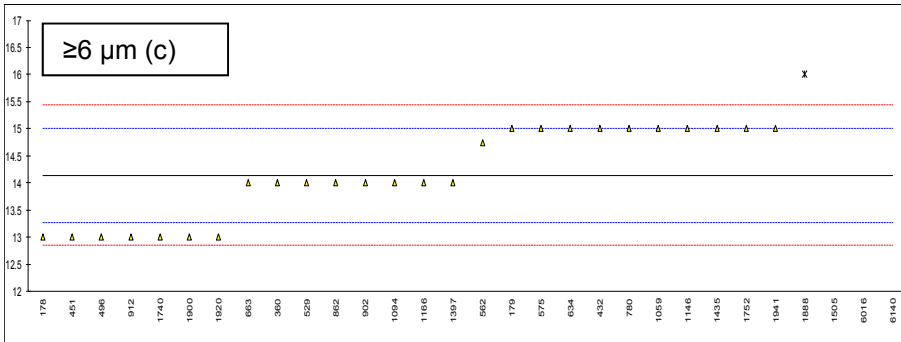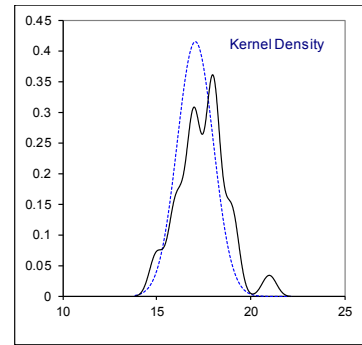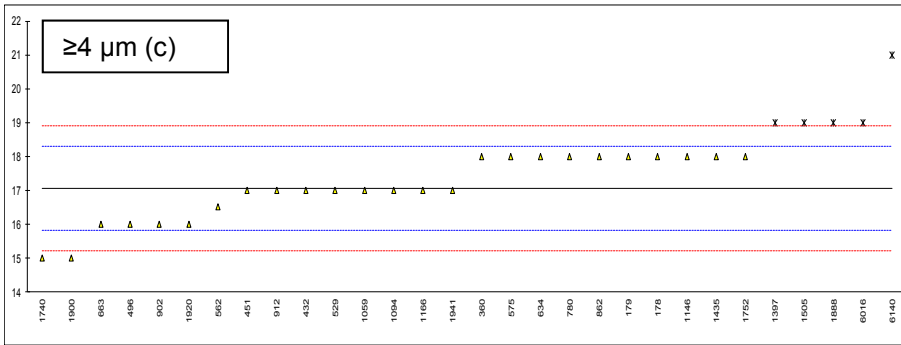
Determination of Aluminum (Al) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	14		0.50	
179	D5185	12		-0.26	
237	D5185	8.729		-1.50	
255	INH-OL1	11.80		-0.33	
311	D5185	10		-1.02	
325	D5185	12		-0.26	
331	D5185Mod.	12.0		-0.26	
339	In house	<5		<-2.92	possibly a false negative test result?
349	D5185	14		0.50	
360	D5185	11.8		-0.33	
398	D5185	14.0		0.50	
432	D5185	12.9		0.09	
451	D5185	11		-0.64	
496	D5185	12.970		0.11	
529	D5185	13.063		0.15	
541		-----		-----	
575	D6595	14.8		0.81	
603		-----		-----	
614	D5185	13.08		0.15	
634	D6595	14		0.50	
663	D5185	12.14		-0.20	
780	D5185	13		0.12	
862	D5185	11.8		-0.33	
902	D5185	13.24		0.22	
912	D5185	12		-0.26	
962	D5185	13		0.12	
963	D5185	12.04		-0.24	
994	D5185	13.17		0.19	
1059	In house	16		1.27	
1146	In house	11.92		-0.29	
1166	In house	15.78		1.18	
1370		<10		-----	
1435	D5185	8.398		-1.63	
1505	D5185	11.46		-0.46	
1660		-----		-----	
1740	D5185	13		0.12	
1743	D5185	14		0.50	
1752	D6595	15.0		0.89	
1888	D5185	18.7	R(0.05)	2.29	
1890		-----		-----	
1900	D5185	14.12		0.55	
1941	D5185	11.94		-0.28	
6016	D5185	13.50		0.31	
6140	D5185	11.32		-0.52	

	normality	OK		
	n	37		
	outliers	1		spike
	mean (n)	12.675		12.07 (recovery <105%)
	st.dev. (n)	1.6377		
	R(calc.)	4.586		
	st.dev.(D5185:18)	2.6266		
	R(D5185:18)	7.354		
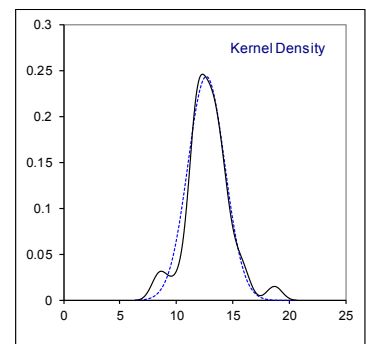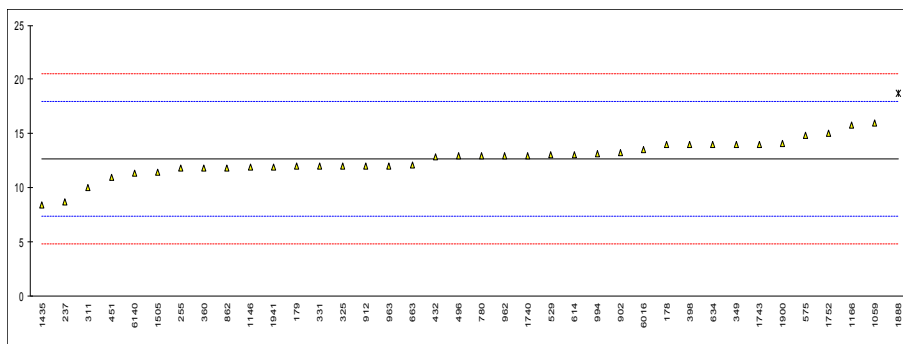
Determination of Barium (Ba) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	13		-0.22	
179	D5185	13		-0.22	
237	D5185	12.96		-0.24	
255		-----		-----	
311	D5185	15		0.64	
325	D5185	14		0.21	
331	D5185Mod.	14.0		0.21	
339	In house	13		-0.22	
349	D5185	14		0.21	
360	D5185	12.67		-0.37	
398	D5185	13.7		0.08	
432	D5185	12.6		-0.40	
451	D5185	12		-0.66	
496	D5185	14.637		0.48	
529	D5185	12.868		-0.28	
541		-----		-----	
575	D6595	15.9		1.03	
603		-----		-----	
614	D5185	14.31		0.34	
634	D6595	20	C,R(0.01)	2.80	first reported 27
663	D5185	13.81		0.13	
780	D5185	14		0.21	
862	D5185	12.0		-0.66	
902	D5185	13.68		0.07	
912	D5185	13		-0.22	
962	D5185	13.1		-0.18	
963	D5185	13.60		0.03	
994	D5185	14.00		0.21	
1059		-----		-----	
1146	In house	12.75		-0.33	
1166	In house	12.85		-0.29	
1370		<10		-----	
1435	D5185	14.10		0.25	
1505	D5185	13.48		-0.02	
1660		-----		-----	
1740	D5185	13		-0.22	
1743	D5185	14.1		0.25	
1752	D6595	<50		-----	
1888	D5185	12.7		-0.35	
1890		-----		-----	
1900	D5185	15.458		0.84	
1941	D5185	13.55		0.01	
6016	D5185	12.99		-0.23	
6140	D5185	13.38		-0.06	

normality	OK	
n	35	
outliers	1	
mean (n)	13.520	
st.dev. (n)	0.8733	
R(calc.)	2.445	
st.dev.(D5185:18)	2.3131	
R(D5185:18)	6.477	application range: 0.5 – 4 mg/kg
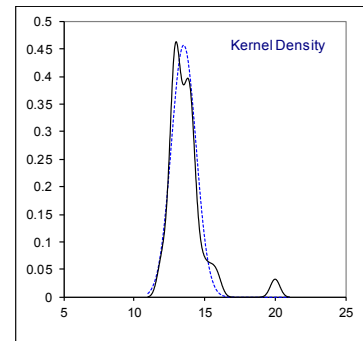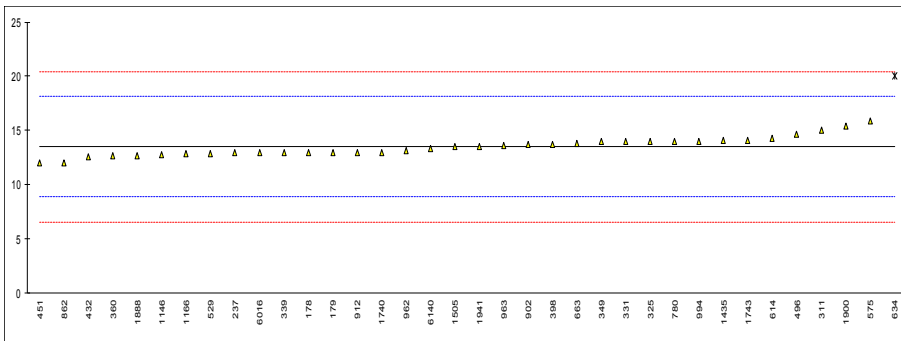
Determination of Boron (B) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	12		0.08	
179	D5185	11		-0.13	
237	D5185	9.7895		-0.38	
255		-----		-----	
311		-----		-----	
325	D5185	13		0.29	
331	D5185Mod.	11.5		-0.02	
339		-----		-----	
349	D5185	8		-0.76	
360	D5185	15.6	C	0.84	first reported 35.6
398	D5185	10.5		-0.23	
432	D5185	10.9		-0.15	
451	D5185	10		-0.34	
496	D5185	11.680		0.02	
529	D5185	12.981		0.29	
541		-----		-----	
575	D6595	11.5		-0.02	
603		-----		-----	
614	D5185	14.4		0.59	
634	D6595	8.72		-0.61	
663		-----		-----	
780	D5185	14		0.50	
862	D5185	10.0		-0.34	
902		-----		-----	
912		-----		-----	
962	D5185	13		0.29	
963	D5185	12.26		0.14	
994	D5185	14.0		0.50	
1059		-----		-----	
1146		-----		-----	
1166		-----		-----	
1370		<5		-----	
1435	D5185	12.40		0.17	
1505		-----		-----	
1660		-----		-----	
1740	D5185	7		-0.97	
1743	D5185	13		0.29	
1752	D6595	8.7		-0.61	
1888	D5185	17.8		1.30	
1890		-----		-----	
1900	D5185	9.239		-0.50	
1941	D5185	11.03		-0.12	
6016	D5185	10.89		-0.15	
6140		-----		-----	

normality	OK			
n	28			
outliers	0		spike	
mean (n)	11.603		10.42 (recovery <111%)	
st.dev. (n)	2.3612			
R(calc.)	6.611			
st.dev.(D5185:18)	4.7581			
R(D5185:18)	13.323			
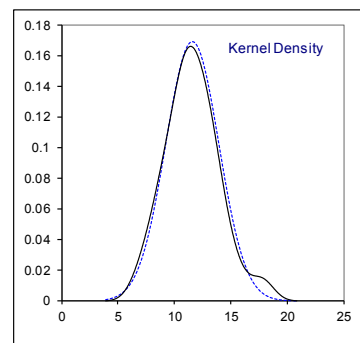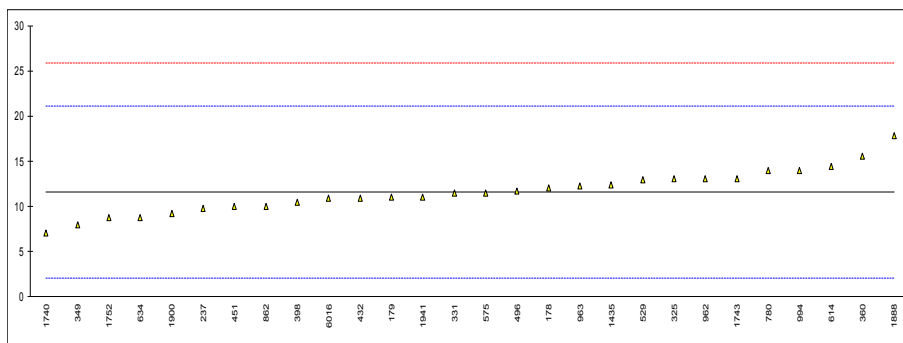
Determination of Copper (Cu) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	52		0.11	
179	D5185	48		-0.80	
237	D5185	52.045		0.12	
255	INH-OL1	47.29		-0.96	
311	D5185	50		-0.35	
325	D5185	53		0.33	
331	D5185Mod.	51.5		-0.01	
339	In house	51		-0.12	
349	D5185	51		-0.12	
360	D5185	47.3		-0.96	
398	D5185	49.0		-0.57	
432	D5185	54.6		0.70	
451	D5185	45		-1.48	
496	D5185	56.652		1.16	
529	D5185	52.703		0.27	
541		-----		-----	
575	D6595	72	C,R(0.01)	4.64	first reported 75.5
603		-----		-----	
614	D5185	54.16		0.60	
634	D6595	45	C	-1.48	first reported 35
663	D5185	50.15		-0.31	
780	D5185	53		0.33	
862	D5185	53.6		0.47	
902	D5185	56.3		1.08	
912	D5185	50		-0.35	
962	D5185	50		-0.35	
963	D5185	51.39		-0.03	
994	D5185	49.04		-0.56	
1059	In house	52		0.11	
1146	In house	49.61		-0.43	
1166	In house	49.02		-0.57	
1370		48.68		-0.64	
1435	D5185	52.22		0.16	
1505	D5185	53.76		0.51	
1660		-----		-----	
1740	D5185	55		0.79	
1743	D5185	57		1.24	
1752	D6595	51.9		0.08	
1888	D5185	55.7		0.95	
1890		-----		-----	
1900	D5185	64.501	R(0.01)	2.94	
1941	D5185	51.2		-0.07	
6016	D5185	54.23		0.61	
6140	D5185	53.92		0.54	

normality	OK	
n	38	
outliers	2	
mean (n)	51.526	
st.dev. (n)	2.9769	
R(calc.)	8.335	
st.dev.(D5185:18)	4.4165	
R(D5185:18)	12.366	
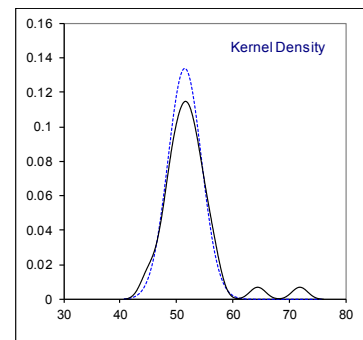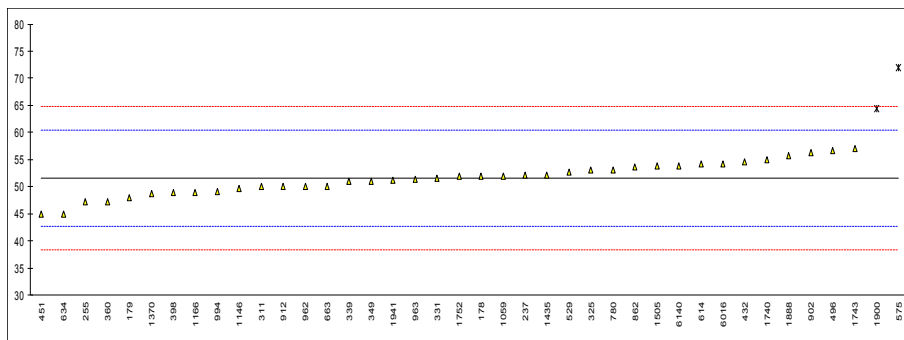
Determination of Iron (Fe) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	2		-0.95	
179	D5185	3		1.78	
237	D5185	2.3425		-0.01	
255	INH-OL1	2.65		0.82	
311	D5185	3		1.78	
325	D5185	2		-0.95	
331	D5185Mod.	2.5		0.41	
339	In house	<5		-----	
349	D5185	2		-0.95	
360	D5185	2.18		-0.46	
398	D5185	2.3		-0.13	
432	D5185	2.4		0.14	
451	D5185	2		-0.95	
496	D5185	2.421		0.20	
529	D5185	1.471		-2.38	
541		-----		-----	
575	D6595	2.90		1.50	
603		-----		-----	
614	D5185	2.67		0.88	
634	D6595	2.2		-0.40	
663	D5185	2.43		0.22	
780	D5185	2		-0.95	
862	D5185	2.3		-0.13	
902	D5185	2.18		-0.46	
912	D5185	2.5		0.41	
962	D5185	2.3		-0.13	
963	D5185	2.59		0.66	
994	D5185	2.42		0.20	
1059	In house	<3		-----	
1146	In house	2.366		0.05	
1166	In house	3.91	R(0.01)	4.25	
1370		<5		-----	
1435	D5185	2.264		-0.23	
1505	D5185	2.38		0.09	
1660		-----		-----	
1740	D5185	2		-0.95	
1743	D5185	2.9	C	1.50	first reported 3.7
1752	D6595	2.3		-0.13	
1888	D5185	4.1	R(0.01)	4.77	
1890		-----		-----	
1900	D5185	2.943		1.62	
1941	D5185	1.46		-2.41	
6016	D5185	2.467		0.32	
6140	D5185	2.33		-0.05	

normality	OK	
n	35	
outliers	2	
mean (n)	2.348	
st.dev. (n)	0.3649	
R(calc.)	1.022	
st.dev.(D5185:18)	0.3676	
R(D5185:18)	1.029	
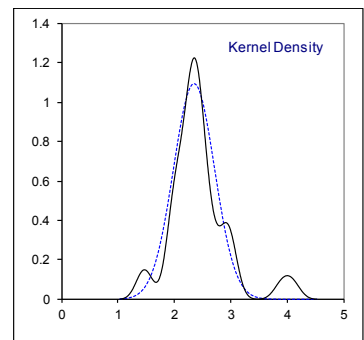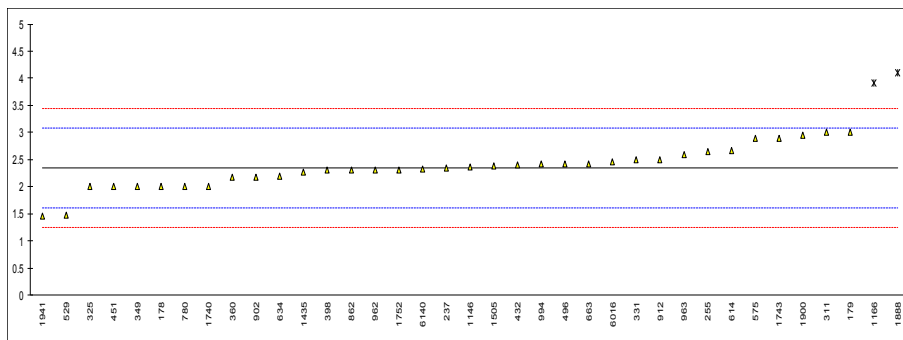
Determination of Lead (Pb) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	9		0.55	
179	D5185	9		0.55	
237	D5185	7.504		-0.12	
255	INH-OL1	9.08		0.58	
311	D5185	8		0.10	
325	D5185	7		-0.35	
331	D5185Mod.	7.8		0.01	
339	In house	8		0.10	
349	D5185	6		-0.79	
360	D5185	7.06		-0.32	
398	D5185	7.8		0.01	
432	D5185	7.9		0.06	
451	D5185	7		-0.35	
496	D5185	7.617		-0.07	
529		-----		-----	
541		-----		-----	
575		-----		-----	
603		-----		-----	
614	D5185	8.39		0.27	
634	D6595	7.3		-0.21	
663	D5185	7.56		-0.10	
780	D5185	7		-0.35	
862	D5185	7.4		-0.17	
902	D5185	8.01		0.10	
912	D5185	8.6		0.37	
962	D5185	7.9		0.06	
963	D5185	7.80		0.01	
994	D5185	6.82		-0.43	
1059	In house	<8		-----	
1146	In house	7.227		-0.24	
1166	In house	7.63		-0.06	
1370		8.468		0.31	
1435	D5185	6.808		-0.43	
1505	D5185	7.58		-0.09	
1660		-----		-----	
1740	D5185	8		0.10	
1743	D5185	8.7		0.41	
1752	D6595	10.8	R(0.01)	1.35	
1888	D5185	9.2		0.64	
1890		-----		-----	
1900	D5185	8.222		0.20	
1941	D5185	7.36		-0.19	
6016	D5185	7.7	C	-0.03	first reported 22.32
6140	D5185	7.49		-0.13	

|		
normality	OK	
n	36	
outliers	1	
mean (n)	7.776	
st.dev. (n)	0.7173	
R(calc.)	2.009	
st.dev.(D5185:18)	2.2420	
R(D5185:18)	6.278	application range: 10 – 160 mg/kg
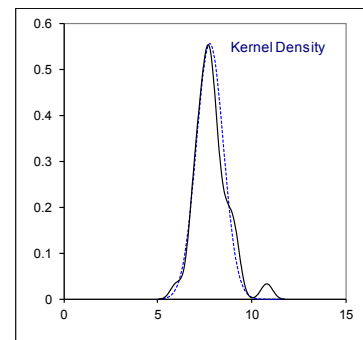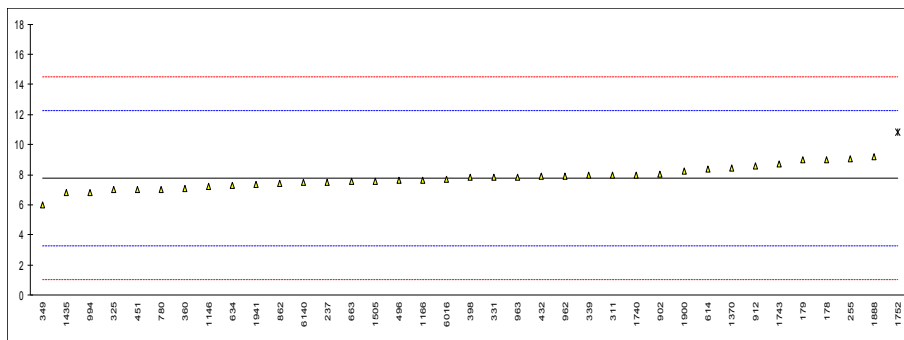
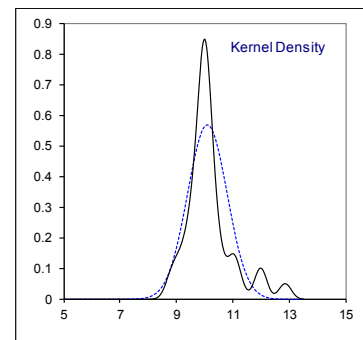Determination of Magnesium (Mg) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	2		-1.28	
179	D5185	3		0.52	
237	D5185	3.2255		0.93	
255	INH-OL1	2.93		0.39	
311	D5185	3		0.52	
325	D5185	3		0.52	
331	D5185Mod.	3.0		0.52	
339	In house	<50		-----	
349	D5185	3		0.52	
360	D5185	2.725		0.02	
398	D5185	3.0		0.52	
432	D5185	2.8		0.16	
451	D5185	2		-1.28	
496	D5185	2.794		0.15	
529	D5185	1.234	R(0.05)	-2.67	
541		-----		-----	
575		-----		-----	
603		-----		-----	
614	D5185	2.6		-0.20	
634	D6595	2.7		-0.02	
663	D5185	2.784		0.13	
780	D5185	2		-1.28	
862	D5185	2.6		-0.20	
902	D5185	2.56		-0.27	
912	D5185	2.6		-0.20	
962	D5185	2.49		-0.40	
963	D5185	2.04		-1.21	
994	D5185	2.90		0.34	
1059		-----		-----	
1146	In house	2.720		0.02	
1166	In house	1.92		-1.43	
1370		<5		-----	
1435	D5185	2.268		-0.80	
1505	D5185	2.97		0.47	
1660		-----		-----	
1740	D5185	3		0.52	
1743	D5185	2.95		0.43	
1752	D6595	2.9		0.34	
1888	D5185	3.6		1.60	
1890		-----		-----	
1900	D5185	2.801		0.16	
1941	D5185	2.39		-0.58	
6016	D5185	2.717		0.01	
6140	D5185	2.91		0.36	

	normality	OK	
	n	35	
	outliers	1	
	mean (n)	2.711	
	st.dev. (n)	0.3816	
	R(calc.)	1.069	
	st.dev.(D5185:18)	0.5543	
	R(D5185:18)	1.552	application range: 5 – 1700 mg/kg
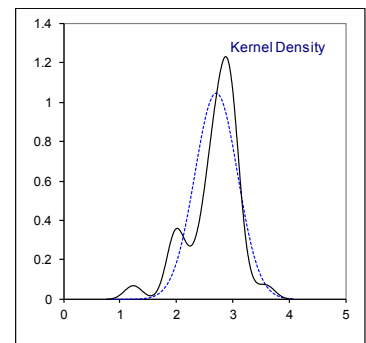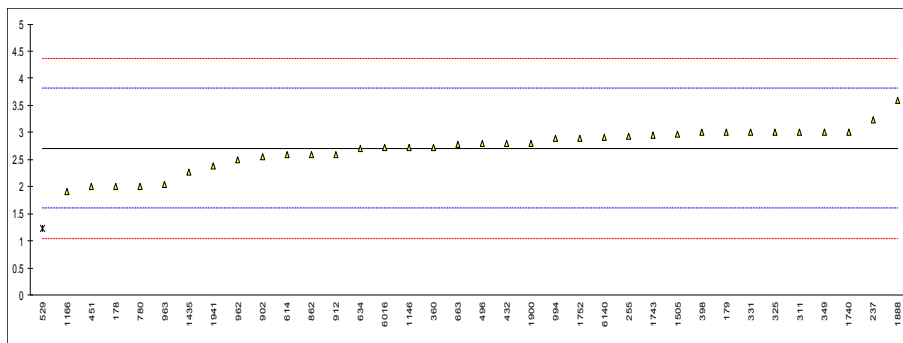
Determination of Manganese (Mn) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	10		-0.15	
237	D5185	10.15		0.05	
255	INH-OL1	9.77		-0.46	
311	D5185	10		-0.15	
325	D5185	10		-0.15	
331	D5185Mod.	10.0		-0.15	
339	In house	10		-0.15	
349	D5185	10		-0.15	
360	D5185	9.34		-1.03	
398	D5185	9.9		-0.28	
432	D5185	10.6		0.66	
451		-----		-----	
496	D5185	10.177		0.09	
529		-----		-----	
541		-----		-----	
575	D6595	11.2		1.46	
603		-----		-----	
614	D5185	10.4		0.39	
634	D6595	12		2.53	
663	D5185	10.13		0.03	
780	D5185	11		1.19	
862	D5185	10.0		-0.15	
902	D5185	10.19		0.11	
912	D5185	9		-1.49	
962	D5185	10		-0.15	
963	D5185	10.46		0.47	
994	D5185	10.26		0.20	
1059	In house	11		1.19	
1146	In house	9.678		-0.58	
1166	In house	12.87	R(0.05)	3.70	
1370		9.468		-0.86	
1435	D5185	9.630		-0.64	
1505	D5185	10.02		-0.12	
1660		-----		-----	
1740	D5185	10		-0.15	
1743	D5185	12		2.53	
1752		-----		-----	
1888	D5185	8.8		-1.76	
1890		-----		-----	
1900	D5185	1.205	R(0.01)	-11.94	
1941	D5185	9.2		-1.22	
6016	D5185	9.608		-0.67	
6140	D5185	9.76		-0.47	

normality	not OK			
n	34			
outliers	2		spike	
mean (n)	10.110		10.00 (recovery <101%)	
st.dev. (n)	0.7017			
R(calc.)	1.965			
st.dev.(D5185:18)	0.7456			
R(D5185:18)	2.088			
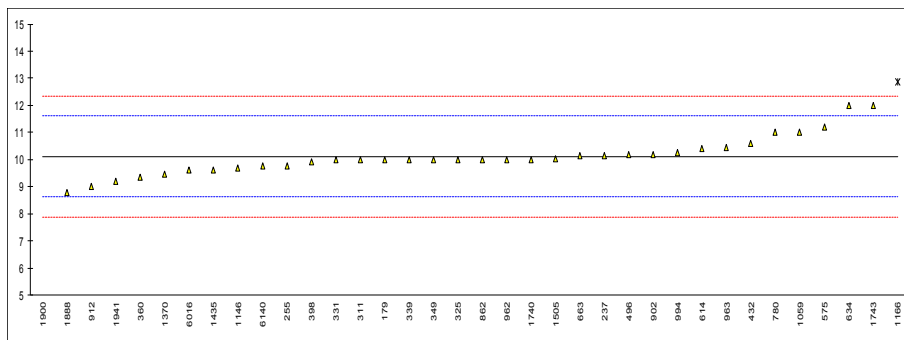
Determination of Molybdenum (Mo) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	10		0.01	
179	D5185	10		0.01	
237	D5185	9.960		-0.02	
255		-----		-----	
311	D5185	10		0.01	
325	D5185	10		0.01	
331	D5185Mod.	10.0		0.01	
339	In house	10		0.01	
349	D5185	10		0.01	
360	D5185	9.74		-0.21	
398	D5185	10.8		0.70	
432	D5185	10.4		0.36	
451	D5185	10		0.01	
496	D5185	10.361		0.32	
529	D5185	9.267		-0.61	
541		-----		-----	
575	D6595	11.0		0.87	
603		-----		-----	
614	D5185	9.86		-0.11	
634	D6595	8.9		-0.93	
663	D5185	9.82		-0.14	
780	D5185	10		0.01	
862	D5185	9.2		-0.67	
902	D5185	10.16		0.15	
912	D5185	7	R(0.01)	-2.55	
962	D5185	9.8		-0.16	
963	D5185	9.96		-0.02	
994	D5185	10.07		0.07	
1059	In house	<3		<-5.96	possibly a false negative test result?
1146		-----		-----	
1166	In house	8.72		-1.08	
1370		9.8675		-0.10	
1435	D5185	9.0		-0.84	
1505	D5185	9.58		-0.35	
1660		-----		-----	
1740	D5185	10		0.01	
1743	D5185	11		0.87	
1752	D6595	9.1		-0.76	
1888	D5185	11.1		0.95	
1890		-----		-----	
1900	D5185	11.392		1.20	
1941	D5185	10.2		0.18	
6016	D5185	10.64		0.56	
6140	D5185	9.53		-0.39	

	normality	OK		
	n	36		
	outliers	1		spike
	mean (n)	9.984		9.73 (recovery <103%)
	st.dev. (n)	0.6086		
	R(calc.)	1.704		
	st.dev.(D5185:18)	1.1709		
	R(D5185:18)	3.279		
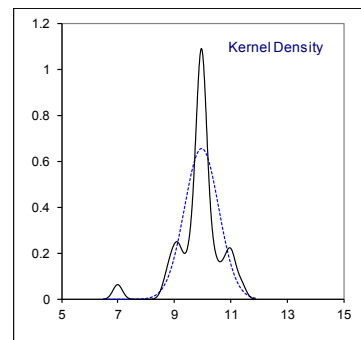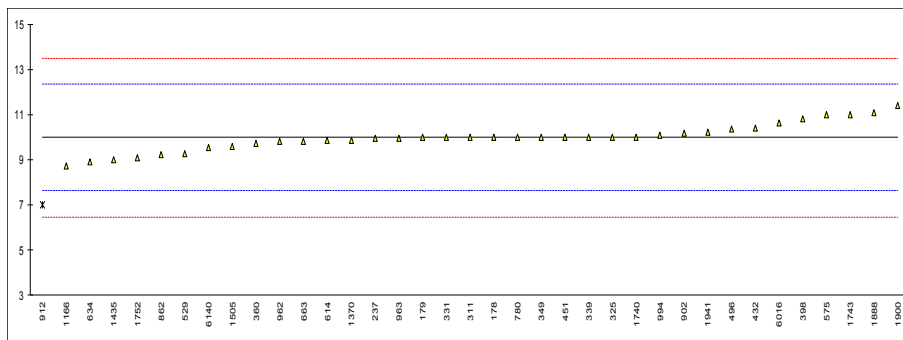
Determination of Nickel (Ni) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	<1		-----	
179	D5185	<1		-----	
237	D5185	0.59255		0.54	
255	INH-OL1	0.75		1.00	
311	D5185	<2		-----	
325	D5185	0		-1.19	
331	D5185Mod.	<1		-----	
339	In house	<5		-----	
349	D5185	<1		-----	
360	D5185	< 1.0		-----	
398	D5185	<1,0		-----	
432	D5185	<1		-----	
451	D5185	n/d		-----	
496	D5185	0.327		-0.24	
529		-----		-----	
541		-----		-----	
575		-----		-----	
603		-----		-----	
614	D5185	0.48		0.21	
634	D6595	0.83		1.23	
663	D5185	0.45		0.12	
780	D5185	<1		-----	
862	D5185	<1		-----	
902		-----		-----	
912	D5185	<1		-----	
962	D5185	<1		-----	
963	D5185	0.22		-0.55	
994	D5185	<5		-----	
1059	In house	<3		-----	
1146	In house	0.3207		-0.26	
1166	In house	0.28		-0.37	
1370		<5		-----	
1435	D5185	0.6865		0.81	
1505	D5185	0.0		-1.19	
1660		-----		-----	
1740	D5185	<1		-----	
1743	D5185	0.30		-0.32	
1752	D6595	<2		-----	
1888	D5185	1.1		2.02	
1890		-----		-----	
1900	D5185	0		-1.19	
1941	D5185	0.6		0.56	
6016	D5185	<0.01		-----	
6140	D5185	0.0		-1.19	

|	
normality	OK
n	17
outliers	0
mean (n)	0.408
st.dev. (n)	0.3233
R(calc.)	0.905
st.dev.(D5185:18)	0.3422
R(D5185:18)	0.958

application range: 5 – 40 mg/kg
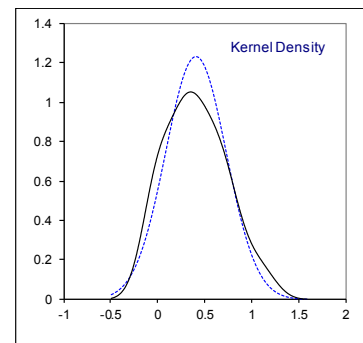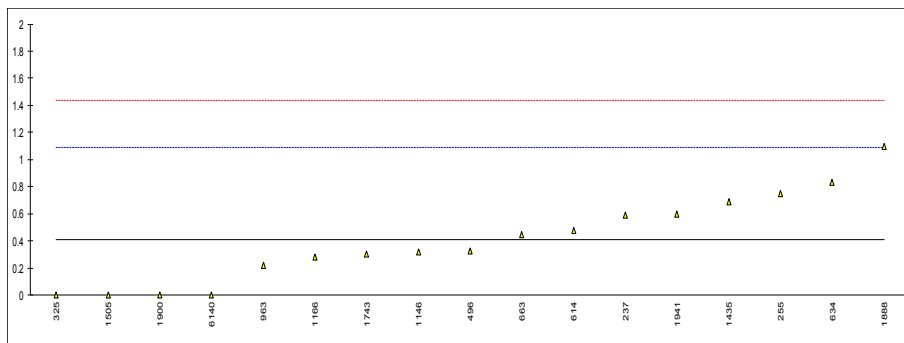
Determination of Potassium (K) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	1		-0.52	
237	D5185	3.565		0.30	
255		-----		-----	
311		-----		-----	
325	D5185	2		-0.20	
331	D5185Mod.	3.0		0.12	
339	In house	<50		-----	
349	D5185	12	G(0.05)	3.01	
360	D5185	2.70		0.02	
398		-----		-----	
432	D5185	3.7		0.34	
451	D5185	2		-0.20	
496	D5185	3.668		0.33	
529		-----		-----	
541		-----		-----	
575		-----		-----	
603		-----		-----	
614	D5185	4.9		0.73	
634	D6595	3.4		0.25	
663	D5185	5.58		0.95	
780	D5185	<1		-----	
862	D5185	2.1		-0.17	
902		-----		-----	
912		-----		-----	
962		-----		-----	
963	D5185	0.78		-0.59	
994		-----		-----	
1059		-----		-----	
1146		-----		-----	
1166	In house	2.27		-0.11	
1370		<5		-----	
1435	D5185	3.808		0.38	
1505		-----		-----	
1660		-----		-----	
1740	D5185	2		-0.20	
1743	D5185	0		-0.84	
1752		-----		-----	
1888		-----		-----	
1890		-----		-----	
1900	D5185	2.665		0.01	
1941	D5185	9.98	G(0.01)	2.36	
6016	D5185	0.774		-0.59	
6140		-----		-----	

|		
normality	OK	
n	19	
outliers	2	
mean (n)	2.627	
st.dev. (n)	1.4360	
R(calc.)	4.021	
st.dev.(D5185:18)	3.1190	
R(D5185:18)	8.733	application range: 40 – 1200 mg/kg
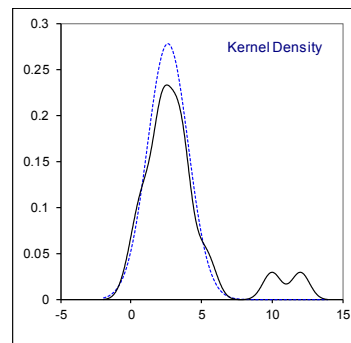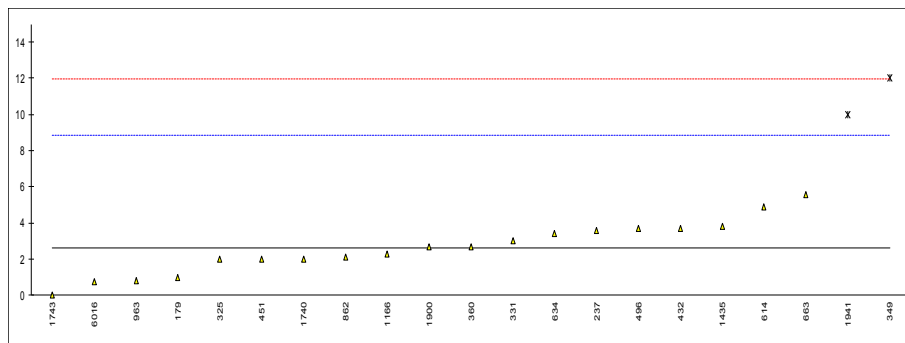
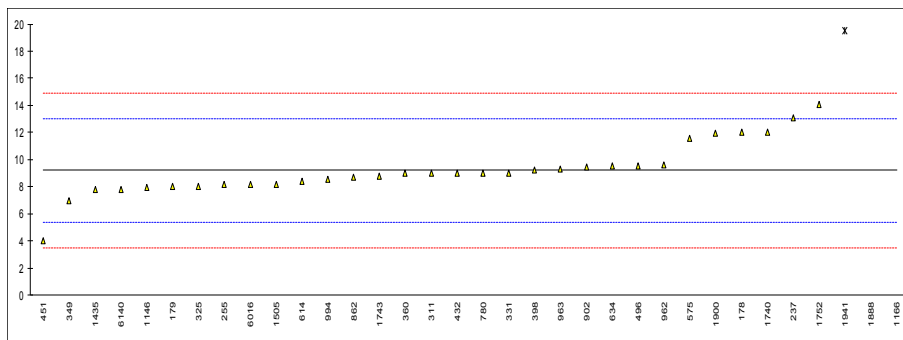## Determination of Silicon (Si) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	12		-0.03	
179	D5185	12		-0.03	
237	D5185	9.093	C	-1.09	first reported 26.085
255		-----		-----	
311	D5185	15		1.07	
325	D5185	13		0.34	
331	D5185Mod.	13.0		0.34	
339	In house	<50		-----	
349	D5185	13		0.34	
360	D5185	12.5		0.16	
398	D5185	11.2		-0.32	
432	D5185	13.1		0.38	
451	D5185	11		-0.39	
496	D5185	12.706		0.23	
529	D5185	11.899		-0.06	
541		-----		-----	
575	D6595	14.6		0.92	
603		-----		-----	
614	D5185	12.12		0.02	
634	D6595	18	R(0.05)	2.17	
663	D5185	12.45		0.14	
780	D5185	12		-0.03	
862	D5185	11.8		-0.10	
902	D5185	13.53		0.53	
912		-----		-----	
962	D5185	11		-0.39	
963	D5185	12.97		0.33	
994	D5185	12.34		0.10	
1059	In house	9		-1.12	
1146	In house	11.70		-0.14	
1166	In house	10.14		-0.71	
1370		<5		-----	
1435	D5185	11.39		-0.25	
1505	D5185	10.76		-0.48	
1660		-----		-----	
1740	D5185	12.5		0.16	
1743	D5185	13		0.34	
1752	D6595	14.5		0.89	
1888	D5185	9.1		-1.09	
1890		-----		-----	
1900	D5185	14.043		0.72	
1941	D5185	11.7		-0.14	
6016	D5185	11.55		-0.19	
6140	D5185	10.78		-0.47	

normality	OK		
n	35		
outliers	1		spike
mean (n)	12.071		11.26 (recovery <107%)
st.dev. (n)	1.4514		
R(calc.)	4.064		
st.dev.(D5185:18)	2.7360		
R(D5185:18)	7.661		
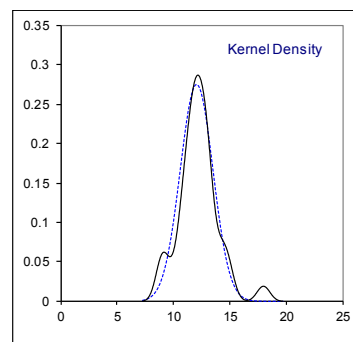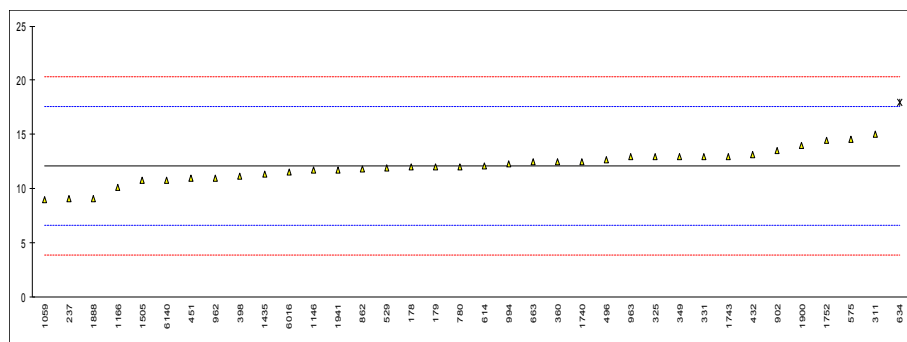
Determination of Sodium (Na) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	12		1.46	
179	D5185	8		-0.64	
237	D5185	13.12		2.05	
255	ONH-OL1	8.14		-0.57	
311	D5185	9		-0.11	
325	D5185	8		-0.64	
331	D5185Mod.	9.0		-0.11	
339	In house	<50		-----	
349	D5185	7		-1.17	
360	D5185	8.99		-0.12	
398	D5185	9.2		-0.01	
432	D5185	9.0		-0.11	
451	D5185	4		-2.74	
496	D5185	9.510		0.15	
529		-----		-----	
541		-----		-----	
575	D6595	11.6		1.25	
603		-----		-----	
614	D5185	8.4		-0.43	
634	D6595	9.5		0.15	
663		-----		-----	
780	D5185	9		-0.11	
862	D5185	8.7		-0.27	
902	D5185	9.46		0.13	
912		-----		-----	
962	D5185	9.6		0.20	
963	D5185	9.28		0.03	
994	D5185	8.525		-0.36	
1059		-----		-----	
1146	In house	7.954		-0.66	
1166	In house	303.90	R(0.01)	154.97	
1370		<10		-----	
1435	D5185	7.790		-0.75	
1505	D5185	8.17		-0.55	
1660		-----		-----	
1740	D5185	12		1.46	
1743	D5185	8.8		-0.22	
1752	D6595	14.1		2.57	
1888	D5185	100.8	R(0.01)	48.16	
1890		-----		-----	
1900	D5185	11.944		1.43	
1941	D5185	19.5	R(0.01)	5.41	
6016	D5185	8.154		-0.56	
6140	D5185	7.81		-0.74	

normality	suspect	
n	31	
outliers	3	
mean (n)	9.218	
st.dev. (n)	1.9492	
R(calc.)	5.458	
st.dev.(D5185:18)	1.9016	
R(D5185:18)	5.324	
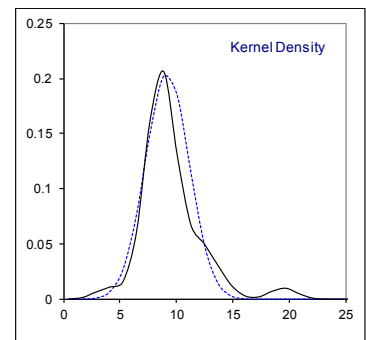
Determination of Tin (Sn) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	10		0.14	
179	D5185	12		0.80	
237	D5185	9.6585		0.03	
255		-----		-----	
311	D5185	10		0.14	
325	D5185	10		0.14	
331	D5185Mod.	10.3		0.24	
339	In house	9		-0.19	
349	D5185	6		-1.17	
360	D5185	10.2		0.21	
398	D5185	4.0		-1.83	
432	D5185	10.9		0.44	
451	D5185	9		-0.19	
496	D5185	5.151		-1.45	
529	D5185	6.755		-0.92	
541		-----		-----	
575		-----		-----	
603		-----		-----	
614	D5185	9.93		0.12	
634	D6595	5.4		-1.37	
663	D5185	10.19		0.21	
780	D5185	10		0.14	
862	D5185	8.5		-0.35	
902	D5185	11.25		0.55	
912		-----		-----	
962	D5185	9.9		0.11	
963	D5185	9.87		0.10	
994	D5185	9.8		0.08	
1059	In house	<6		-----	
1146	In house	4.830		-1.56	
1166	In house	9.97		0.13	
1370		12.354		0.92	
1435	D5185	9.427		-0.05	
1505	D5185	8.76		-0.26	
1660		-----		-----	
1740	D5185	10		0.14	
1743	D5185	11		0.47	
1752	D6595	10.2		0.21	
1888	D5185	13.2		1.20	
1890		-----		-----	
1900	D5185	11.199		0.54	
1941	D5185	11.8		0.73	
6016	D5185	14.57		1.65	
6140	D5185	9.22		-0.11	

normality	OK			
n	36			
outliers	0		spike	
mean (n)	9.565		9.81 (recovery <98%)	
st.dev. (n)	2.2863			
R(calc.)	6.402			
st.dev.(D5185:18)	3.0415			
R(D5185:18)	8.516			
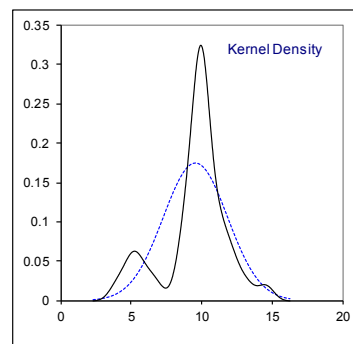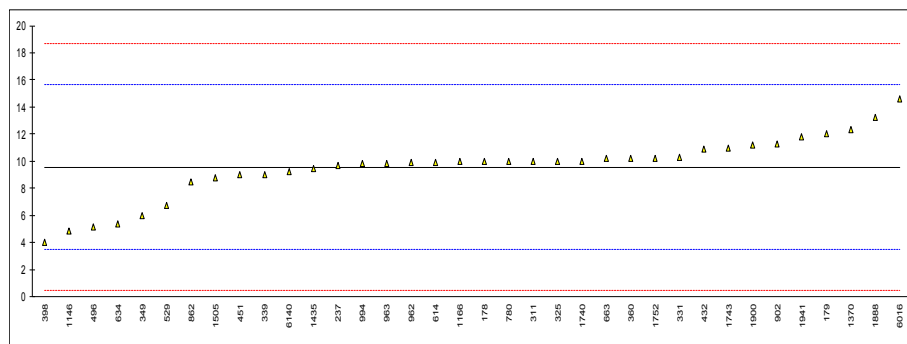
Determination of Calcium (Ca) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	472		-0.43	
179	D5185	446		-2.02	
237	D5185	537.3		3.57	
255	ONH-OL1	390		-5.45	
311	D5185	475		-0.25	
325	D5185	481		0.12	
331	D5185Mod.	504.8		1.58	
339	In house	508		1.77	
349	D5185	492		0.79	
360	D5185	458.6		-1.25	
398	D5185	515		2.20	
432	D5185	523		2.69	
451	D5185	450		-1.77	
496	D5185	511.9		2.01	
529	D5185	475.230		-0.23	
541		-----		-----	
575	D6595	515		2.20	
603		-----		-----	
614	D5185	496.1		1.05	
634	D6595	300	C,R(0.01)	-10.95	first reported 260
663	D5185	460.02		-1.16	
780	D5185	536		3.49	
862	D5185	466.4		-0.77	
902	D5185	551		4.40	
912	D5185	428		-3.12	
962	D5185	477		-0.12	
963	D5185	491.32		0.75	
994	D5185	522		2.63	
1059	In house	441		-2.33	
1146	In house	490.4		0.70	
1166	In house	411.43		-4.13	
1370		478.81		-0.01	
1435	D5185	485.3		0.39	
1505	D5185	437.7		-2.53	
1660		-----		-----	
1740	D5185	465		-0.86	
1743	D5185	531		3.18	
1752	D6595	433		-2.81	
1888	D5185	384.1		-5.81	
1890		-----		-----	
1900	D5185	548.05		4.22	
1941	D5185	476		-0.18	
6016	D5185	485.4		0.39	
6140	D5185	431.4		-2.91	

normality	OK
n	39
outliers	1
mean (n)	479.007
st.dev. (n)	40.9994
R(calc.)	114.798
st.dev.(D5185:18)	16.3447
R(D5185:18)	45.765
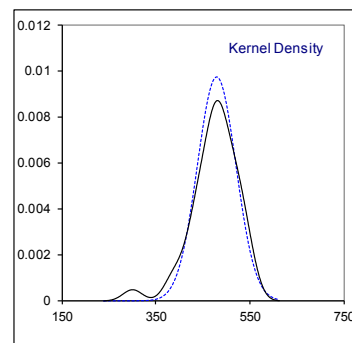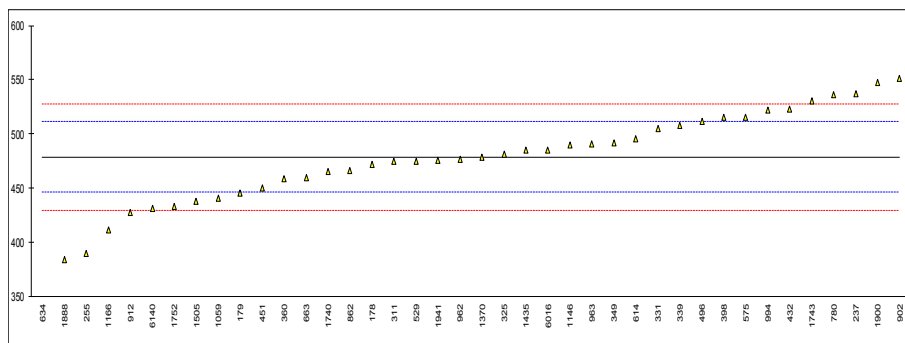
Determination of Phosphorus (P) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	1229		-0.02	
179	D5185	1313		1.54	
237	D5185	1126		-1.93	
255		-----		-----	
311	D5185	1255		0.46	
325	D5185	1229		-0.02	
331	D5185Mod.	1279.3		0.91	
339	In house	1200		-0.56	
349	D5185	1181		-0.91	
360	D5185	1151		-1.47	
398	D5185	1285		1.02	
432	D5185	1276		0.85	
451	D5185	1163		-1.25	
496	D5185	1251.8		0.40	
529	D5185	1364.270		2.49	
541		-----		-----	
575	D6595	1316	C	1.59	first reported 1516
603		-----		-----	
614	D5185	1270		0.74	
634	D6595	1300	C	1.30	first reported 2030
663	D5185	1229.7		-0.01	
780	D5185	1260		0.55	
862	D5185	1204		-0.49	
902	D5185	1227		-0.06	
912	D5185	1236		0.11	
962	D5185	1189		-0.76	
963	D5185	1245.49		0.28	
994	D5185	1245		0.27	
1059	In house	1119		-2.06	
1146	In house	1200		-0.56	
1166	In house	1250.51		0.38	
1370		1053.25		-3.28	
1435	D5185	1219		-0.21	
1505	D5185	1134		-1.79	
1660		-----		-----	
1740	D5185	1130		-1.86	
1743	D5185	1280		0.92	
1752	D6595	1055		-3.25	
1888	D5185	1319.5		1.66	
1890		-----		-----	
1900	D5185	1422.5		3.57	
1941	D5185	1201		-0.54	
6016	D5185	1436		3.82	
6140	D5185	1132		-1.82	

normality	OK	
n	39	
outliers	0	
mean (n)	1230.188	
st.dev. (n)	84.5097	
R(calc.)	236.627	
st.dev.(D5185:18)	53.8637	
R(D5185:18)	150.818	application range: 10 – 1000 mg/kg
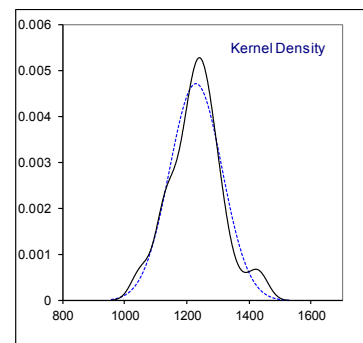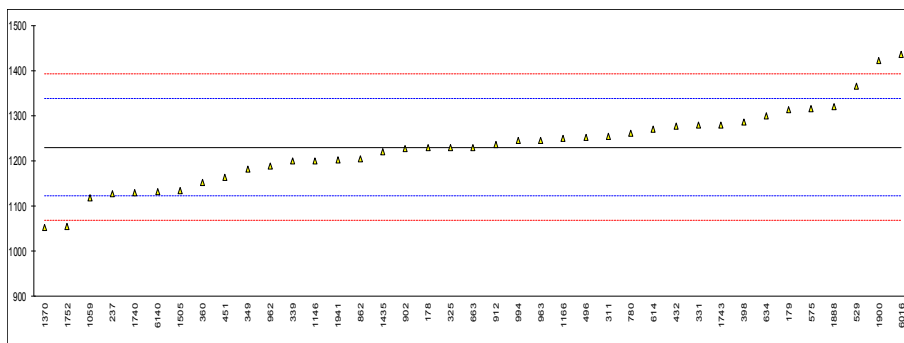
Determination of Zinc (Zn) on sample #18227; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	1360		-0.47	
179	D5185	1404		0.04	
237	D5185	1233.3		-1.95	
255	ONH-OL1	1398		-0.03	
311	D5185	1465		0.75	
325	D5185	1419		0.22	
331	D5185Mod.	1511.8		1.30	
339	In house	1316		-0.99	
349	D5185	1265		-1.58	
360	D5185	1261		-1.63	
398	D5185	1473		0.85	
432	D5185	1495		1.10	
451	D5185	1254		-1.71	
496	D5185	1461.6		0.71	
529	D5185	1368.710		-0.37	
541		-----		-----	
575	D6595	1526		1.47	
603		-----		-----	
614	D5185	1461		0.71	
634	D6595	1150	C	-2.92	first reported 1930
663	D5185	1488.7		1.03	
780	D5185	1495		1.10	
862	D5185	1376		-0.29	
902	D5185	1430		0.34	
912	D5185	1341		-0.69	
962	D5185	1333		-0.79	
963	D5185	1396.56		-0.05	
994	D5185	1467		0.78	
1059	In house	1330		-0.82	
1146	In house	1451		0.59	
1166	In house	1429.42		0.34	
1370		1271.54		-1.51	
1435	D5185	1305		-1.11	
1505	D5185	1402		0.02	
1660		-----		-----	
1740	D5185	1380		-0.24	
1743	D5185	1500		1.16	
1752	D6595	1500		1.16	
1888	D5185	1505.3		1.22	
1890		-----		-----	
1900	D5185	1656.0		2.98	
1941	D5185	1290		-1.29	
6016	D5185	1451		0.59	
6140	D5185	1398		-0.03	

normality	OK	
n	40	
outliers	0	
mean (n)	1400.473	
st.dev. (n)	100.0120	
R(calc.)	280.034	
st.dev.(D5185:18)	85.6687	
R(D5185:18)	239.872	
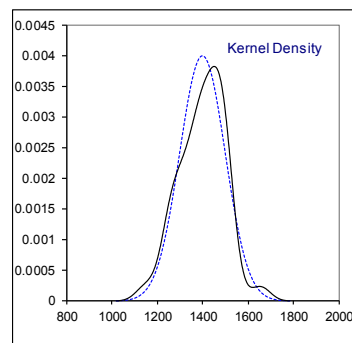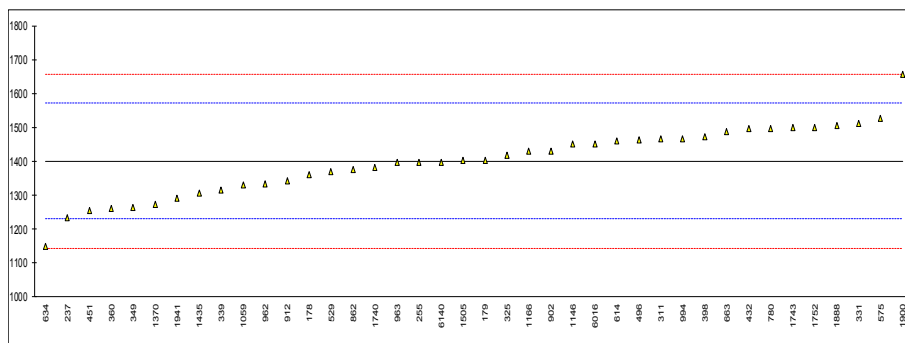
APPENDIX 2

Other reported Metals in sample #18227; results in mg/kg

lab	Cadmium (Cd)		Chromium (Cr)	Lithium (Li)	Silver (Ag)	Titanium (Ti)	Vanadium (V)
178	-----		<1	-----	0.11	-----	<1
179	-----		<1	-----	<0.10	<1	<1
237	-----		<1	-----	0.05875	<5	<1
255	-----		0.86	-----	0.79	-----	-----
311	-----		<2	-----	<2	<2	<2
325	-----		0	-----	0	0	0
331	<1		<1	<1	<1	<1	<1
339	<1		<5	<50	<5	<5	<5
349	1		<1	<1	3	<1	<1
360	< 1.0		< 1.0	-----	-----	< 1.0	< 1.0
398	<1,0		<1,0	-----	1.9	<1,0	<1,0
432	<1		<1	-----	<1	<1	<1
451	-----		n/d	-----	n/d	n/d	n/d
496	0.193		0.233	0.065	<0.5	0.019	<1
529	-----		-----	-----	-----	-----	-----
541	-----		-----	-----	-----	-----	-----
575	-----		0.54	-----	-----	-----	1.00
603	-----		-----	-----	-----	-----	-----
614	0.43		0.35	0.2	0.3	0	0
634	0.0		0.35	-----	0.13	0.0	1.6
663	-----		0.28	-----	0.05	0.00	0.00
780	<1		<1	-----	<1	<1	<1
862	<1		<1	<1	<1	<1	<1
902	-----		-----	-----	-----	-----	-----
912	1		<1	-----	1	<1	<1
962	-----		<1	-----	<0.5	<1	<1
963	0.25		0.21	-----	0	0.05	0.04
994	-----		<1	-----	<0.5	<5	<1
1059	-----		<4	-----	-----	-----	<4
1146	-----		0.2723	0.1533	0.0666	0.000	0.0460
1166	1.52		0.00	-----	3.08	0.00	0.00
1370	<5		<5	<5	<5	<5	<5
1435	0.5485		0.3049	0.3954	0.3108	0.0239	0.0
1505	0.19		0.07	0.39	0.98	0.0	0.0
1660	-----		-----	-----	-----	-----	-----
1740	-----		<1	-----	<1	<1	<1
1743	0.21		0.30	1.22	0.03	0.01	0.01
1752	-----		<1	-----	<1	<1	<3
1888	0.8		11.8	-----	-----	0.5	11.7
1890	-----		-----	-----	-----	-----	-----
1900	-----		0.090	-----	0	0	0
1941	-----	W	0.8	-----	0.0	0.77	0.09
6016	-----		0.438	-----	-----	0.04	<0.01
6140	0.32		0.29	0.38	0.0	0.0	0.0

Lab 1941: W = test result withdrawn on request of participant, first reported 5.99

APPENDIX 3

Number of participants per country of main round

```
1 lab in  ARGENTINA
1 lab in  AUSTRALIA
1 lab in  AUSTRIA
1 lab in  AZERBAIJAN
3 labs in  BELGIUM
2 labs in  BULGARIA
1 lab in  CHILE
1 lab in  CHINA, People's Republic
1 lab in  COLOMBIA
1 lab in  COTE D'IVOIRE
1 lab in  CROATIA
3 labs in  FRANCE
1 lab in  GERMANY
2 labs in  GREECE
1 lab in  INDIA
2 labs in  ITALY
1 lab in  KAZAKHSTAN
2 labs in  MALAYSIA
1 lab in  MEXICO
1 lab in  MOROCCO
3 labs in  NETHERLANDS
1 lab in  NIGERIA
1 lab in  NORWAY
1 lab in  PHILIPPINES
2 labs in  POLAND
1 lab in  PORTUGAL
1 lab in  RUSSIAN FEDERATION
2 labs in  SAUDI ARABIA
1 lab in  SERBIA
1 lab in  SLOVENIA
1 lab in  SPAIN
1 lab in  SUDAN
2 labs in  SWEDEN
1 lab in  TANZANIA
1 lab in  THAILAND
1 lab in  TURKEY
2 labs in  UNITED KINGDOM
2 labs in  UNITED STATES OF AMERICA
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APPENDIX 4

Abbreviations

C   = final test 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   = possibly an error in calculations
U   = test result possibly reported in a different unit
W   = test result withdrawn on request of participant
ex   = test result excluded from 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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