Results of Proficiency Test
Gear Oil (used)
April 2017

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

In 2015 Institute for Interlaboratory Studies organised a new proficiency test for the analyses of fresh Gear Oil. During the annual proficiency testing program 2016/2017 on request of several participants it was decided to organize also a proficiency test for the analyses of used Gear Oil. In this first interlaboratory study, 18 laboratories in 14 different countries registered for participation. See appendix 2 for the number of participants per country.
In this report, the results of the 2017 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 one bottle of 1L (labelled #17036) with used Gear Oil.
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 of used Gear Oil was obtained from a local supplier. The approximately 75 litre of the bulk material was homogenised in a precleaned drum. After homogenisation, 40 amber glass one litre bottles were filled and labelled #17036. The homogeneity of the subsamples #17036 was checked by determination of Density at 15°C in accordance with ASTM D4052 and Water according to ASTM D1364 on 8 stratified randomly selected samples.

	Density at 15 °C in kg/m^3	*Water in mg/kg*
Sample #17036-1	891.97	280
Sample #17036-2	891.97	270
Sample #17036-3	891.95	250
Sample #17036-4	891.94	250
Sample #17036-5	891.96	280
Sample #17036-6	891.96	280
Sample #17036-7	891.96	280
Sample #17036-8	891.97	270

Table 1: homogeneity test results of subsamples #17036

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/m^3	*Water in mg/kg*
r (observed)	0.03	37
reference test method	ASTM D4052:16	ASTM D6304:16e1
0.3 * R (ref. test method)	0.15	146

Table 2: repeatabilities of subsamples #17036

The calculated repeatabilities were each less than 0.3 times the reproducibility of the corresponding reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories, one sample of 1 L in a brown glass bottle (labelled #17036) was sent on March 15, 2017.

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 #17036: 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, Level of Contamination and 23 elements (20 wear metals and 3 additives).

It was explicitly requested to treat the samples as if they were routine samples. Therefore, each laboratory is advised to perform only those analyses that normally are done in daily routine (but the laboratories are allowed to do all analyses). Furthermore, it was requested to report the test results using the indicated units on the report form and not to round the test results more, 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 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 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. 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 of 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 of 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:

	$\lvert z\rvert < 1$	good
$1 <$	$\lvert z\rvert < 2$	satisfactory
$2 <$	$\lvert z\rvert < 3$	questionable
$3 <$	$\lvert z\rvert$	unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered during dispatch and reporting. All laboratories reported on time. Not all laboratories were able to report all analyses requested. The 17 reporting participants sent in 362 numerical test results. Observed were 23 outlying test results, which is 6.4% 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 TEST

In this section, the test results are discussed 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 3.

Acid Number (Total): 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 D664:11ae1.

Density at 15°C: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D4052:16.

Flash Point PMcc: Although both methods (A and B) of ASTM D93 may be applicable for this determination (in-use *vs* used lubricating oil), the consensus values of method A and method B differ significantly (191.5 vs 182.3°C) in this PT for sample #17036. Therefore, both data sets were evaluated separately.
For the group which followed method A, the determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D93:16a method A.
Also for the group of laboratories that followed method B, the 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 D93:16a method B.

Kin.Visco.at 40°C: This determination was not problematic. One statistical outlier was observed and one test result was excluded from the statistical evaluation, as the test result was not corrected to ASTM D445. However, the calculated reproducibility after rejection of the suspect data is in good agreement with the requirements of ASTM D445:17.

Kin.Visco.at 100°C: This determination was not problematic. Two statistical outliers were observed and one test result was excluded from the statistical evaluation, as the test result was not corrected to ASTM D445. However, the calculated reproducibility after rejection of the suspect data is in good agreement with the requirements of ASTM D445:17.

Viscosity Index: This determination was not problematic. One statistical outlier was observed and three test results were excluded from the statistical evaluation due to outliers in Kinematic Viscosity. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D2270:10.

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

Water: 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 D6304:16e1.

Level of Contamination: In total six statistical outliers were observed over six parameters (4 at counts per ml and 2 at scale number) and one test result was excluded. However, due to the low number of reported test results, it was difficult to draw a significant conclusion. Therefore, it was decided not to calculate z-scores for counts per ml and ISO4406-scale.

Boron: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5185:13e1.

Copper: Regretfully, the consensus value was below the application range of the test method ASTM D5185, therefore no significant conclusions were drawn.

Iron: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5185:13e1.

Calcium: Regretfully, the consensus value was below the application range of the test method ASTM D5185, therefore no significant conclusions were drawn.

Phosphorus: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:13e1.

Zinc: Regretfully, the consensus value was below the application range of the test method ASTM D5185, therefore no significant conclusions were drawn.

Other Wear Metals: No significant conclusions were drawn as the average concentration of each element was below the application range of the test method.

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 standards (in casu ASTM standards) are compared in the next tables.

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R(lit)*
Total Acid Number	mg KOH/g	15	0.62	0.31	0.27
Density at 15°C	kg/m^3	13	892.1	0.8	0.5
Flash Point PMcc – method A	°C	9	192.9	12.9	13.7
Flash Point PMcc – method B	°C	5	181.6	5.8	10
Kinematic Viscosity at 40°C	mm^2/s	14	145.53	0.91	1.78
Kinematic Viscosity at 100°C	mm^2/s	14	14.362	0.178	0.198
Viscosity Index		10	96.44	1.37	2
Membrane Filtration 5µm	%M/M	2	n.a.	n.a.	n.a.
Water	mg/kg	14	281	62	498
Level of contamination ≥ 4µm (c)	counts/ml	7	7779	15717	(8790)
Level of contamination ≥ 6µm (c)	counts/ml	9	1932	4815	(1468)
Level of contamination ≥14µm (c)	counts/ml	7	53	155	(72)
Level of contamination ≥ 4µm (c)	scale number	7	20.7	3.1	(1.7)
Level of contamination ≥ 6µm (c)	scale number	7	17.4	4.2	(1.2)
Level of contamination ≥14µm (c)	scale number	7	13.1	6.7	(2)
Boron as B	mg/kg	13	11.5	7.5	13.3
Copper as Cu	mg/kg	13	1.8	0.5	(0.4)
Iron as Fe	mg/kg	16	25.9	6.3	7.0
Calcium as Ca	mg/kg	15	5.5	2.9	(1.9)
Phosphorus as P	mg/kg	16	332	57	78
Zinc as Zn	mg/kg	13	3.1	1.5	(1.2)

Table 3: reproducibilities of test results of sample #17036.

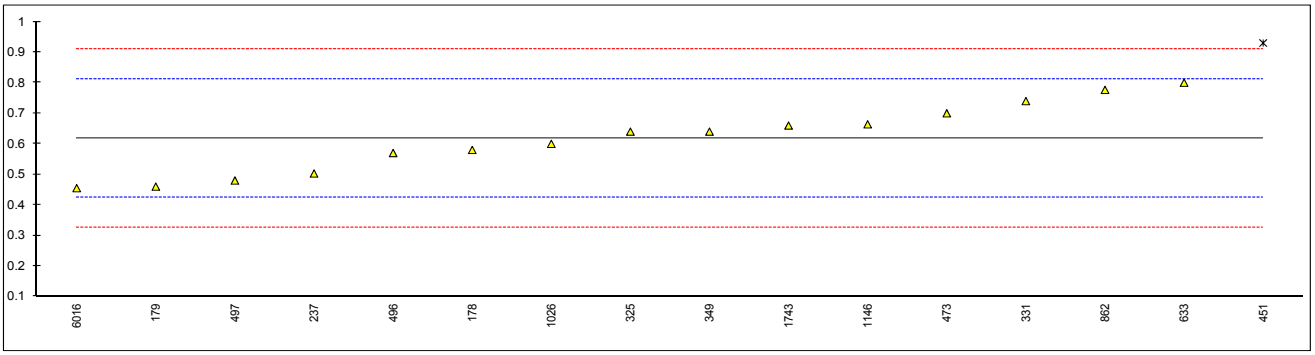
Evaluation between brackets is for concentrations near or below the detection limits

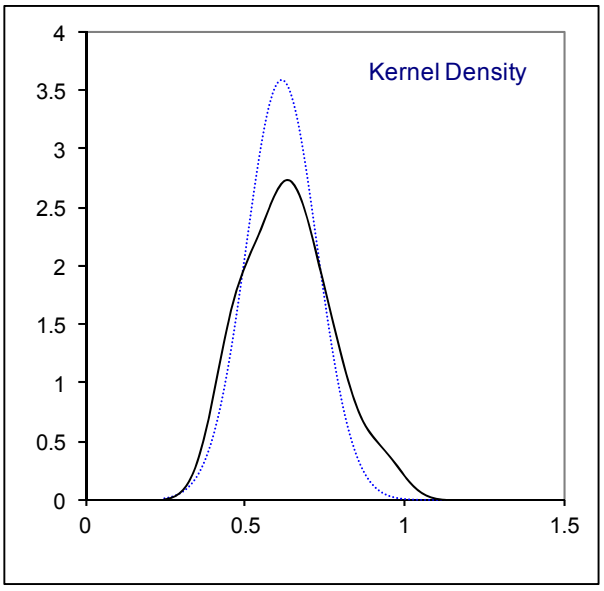
Without further statistical calculations, it could be concluded that for many tests there is a good compliance of the group of participating laboratories with the relevant reference test methods. The problematic tests have been discussed in paragraph 4.1.

APPENDIX 1

Determination of Acid Number (Total) on sample #17036; results in mg KOH/g.

lab	method	value	mark	z(targ)	remarks
178	INH-1118	0.58		-0.39	
179	D664-A	0.46	C	-1.63	First reported 1.08
214		-----		-----	
237	D664-A	0.503		-1.18	
325	D664-A	0.64		0.23	
331	D664Mod.	0.74		1.26	
349	D664-A	0.64		0.23	
451	Inh-327	0.9	G(0.05)	3.21	
473	D664-A	0.7		0.85	
496	D664-A	0.57		-0.49	
497	D664-A	0.48		-1.42	
633	D664-A	0.80		1.88	
862	D664-A	0.7766		1.63	
902		-----		-----	
1026	D664-A	0.60		-0.18	
1146	D664-A	0.664		0.47	
1743	D664-A	0.66		0.43	
6016	D664-A	0.455		-1.68	
	normality	OK			
	n	15			
	outliers	1			
	mean (n)	0.6179			
	st.dev. (n)	0.11104			
	R(calc.)	0.3109			
	R(D664:11ae1)	0.2719			

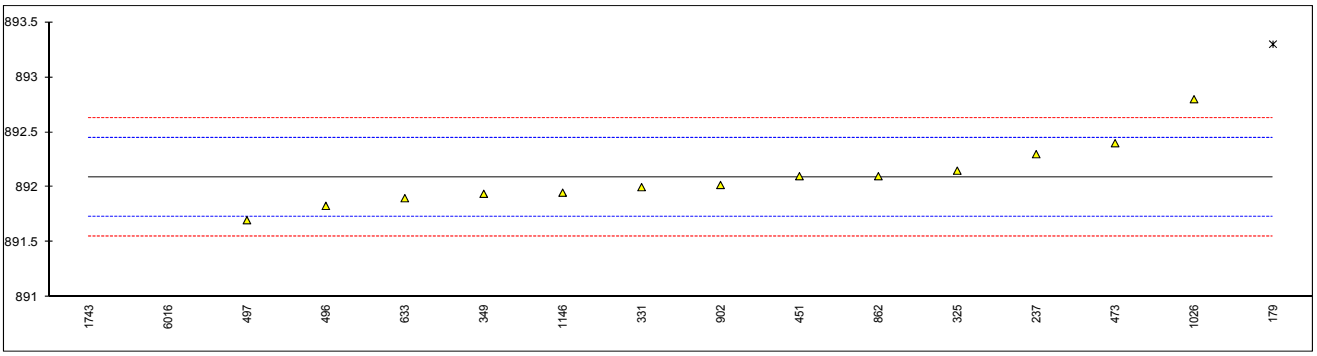


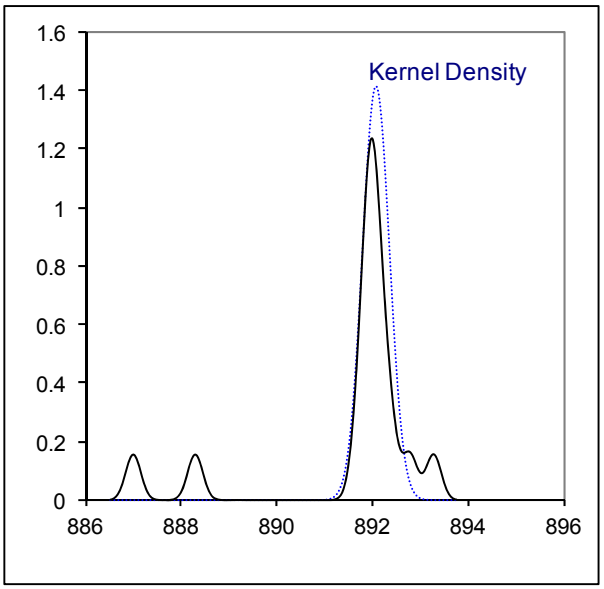


Determination of Density at 15°C on sample #17036; results in kg/m^3.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D1298	893.3	G(0.05)	6.77	
214		-----		-----	
237	D4052	892.3		1.17	
325	D4052	892.15		0.33	
331	ISO12185	892.0		-0.51	
349	D4052	891.94		-0.85	
451	D4052	892.1		0.05	
473	D4052	892.4		1.73	
496	D4052	891.83		-1.46	
497	D7042	891.7		-2.19	
633	D4052	891.9		-1.07	
862	D4052	892.1		0.05	
902	D4052	892.02		-0.40	
1026	D4052	892.8		3.97	
1146	D4052	891.95		-0.79	
1743	ISO12185	887	C,G(0.01)	-28.51	First reported 888
6016	D4052	888.3	C,G(0.01)	-21.23	Reported 0.8883 kg/m3

normality	not OK
n	13
outliers	3
mean (n)	892.092
st.dev. (n)	0.2827
R(calc.)	0.792
R(D4052:16)	0.500

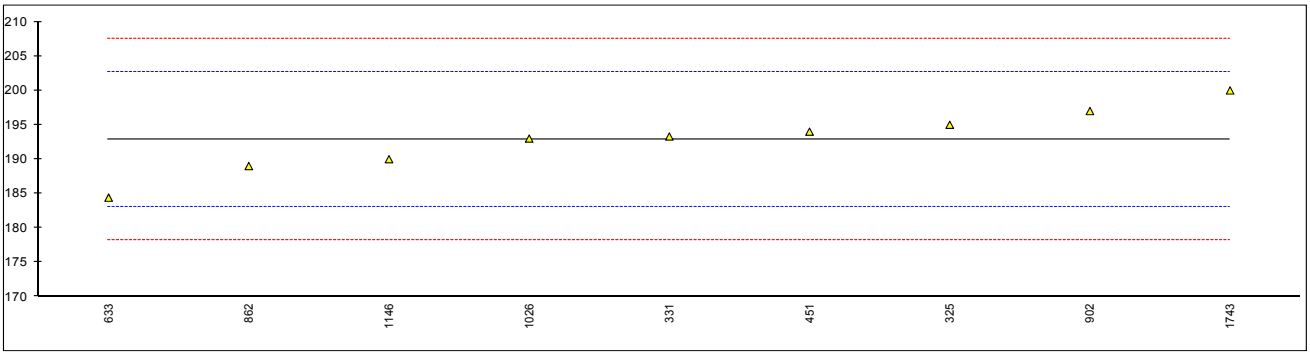


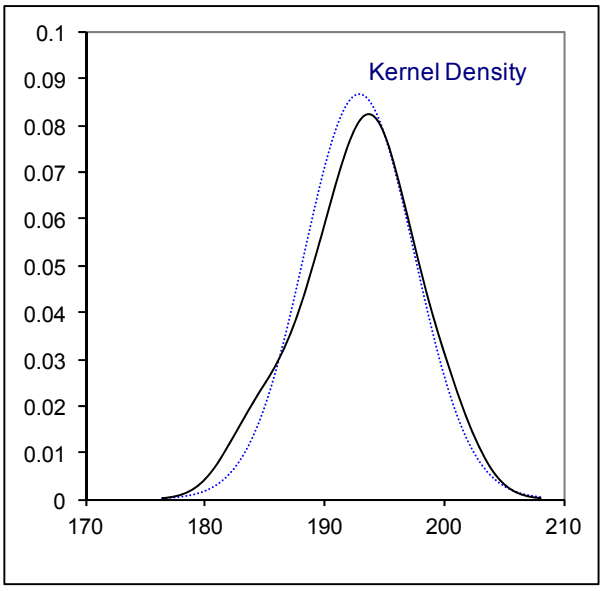


Determination of Flash Point PMcc (method A) on sample #17036; results in °C.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237		-----		-----	
325	D93-A	195.0		0.44	
331	D93-A	193.3		0.09	
349		-----		-----	
451	D93-A	194.0		0.23	
473		-----		-----	
496		-----		-----	
497		-----		-----	
633	D93-A	184.4		-1.73	
862	D93-A	189.0		-0.79	
902	D93-A	197.0		0.85	
1026	D93-A	193.0		0.03	
1146	D93-A	190.0		-0.58	
1743	ISO2719-A	200.0		1.46	
6016		-----		-----	

normality	OK
n	9
outliers	0
mean (n)	192.86
st.dev. (n)	4.594
R(calc.)	12.86
R(D93-A:16a)	13.69

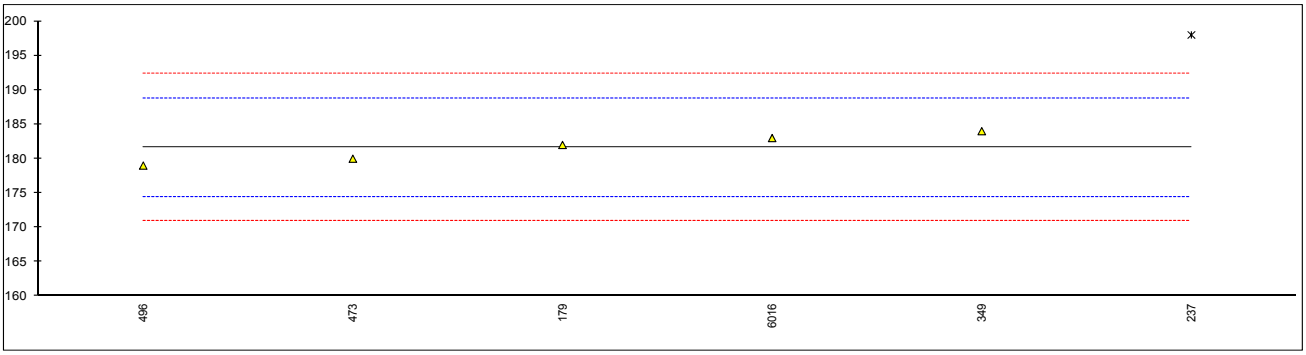




Determination of Flash Point PMcc (method B) on sample #17036; results in °C.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D93-B	182.0		0.11	
214		-----		-----	
237	D93-B	198	G(0.05)	4.59	
325		-----		-----	
331		-----		-----	
349	D93-B	184		0.67	
451		-----		-----	
473	D93-B	180.0		-0.45	
496	ISO2719-B	179.0		-0.73	
497		-----		-----	
633		-----		-----	
862		-----		-----	
902		-----		-----	
1026		-----		-----	
1146		-----		-----	
1743		-----		-----	
6016	D93-B	183		0.39	

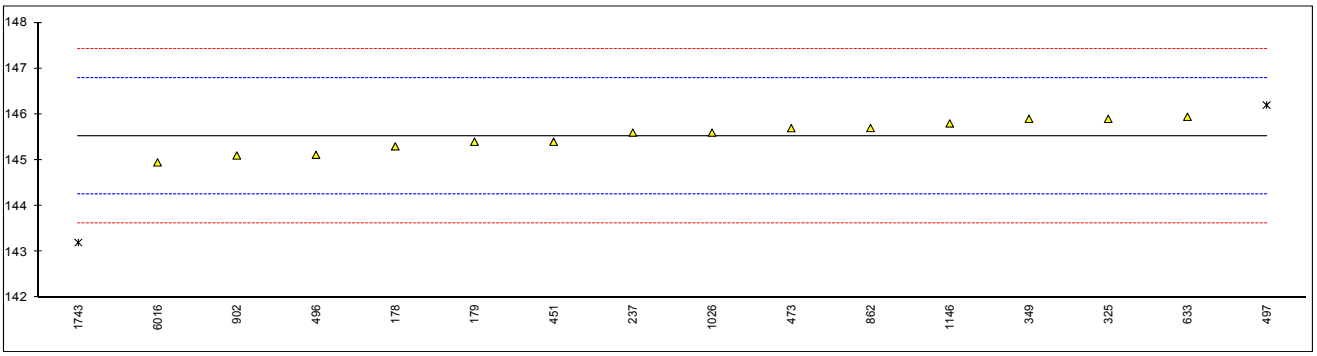
normality	unknown
n	5
outliers	1
mean (n)	181.60
st.dev. (n)	2.074
R(calc.)	5.81
R(D93-B:16a)	10

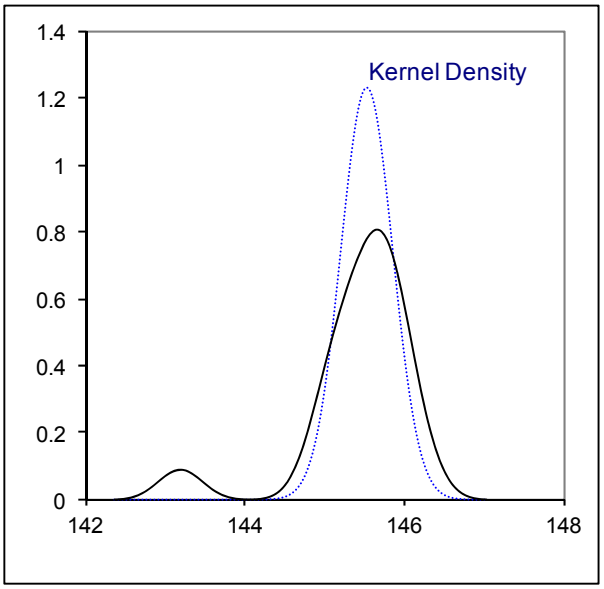


Determination of Kinematic Viscosity at 40°C on sample #17036; results in mm^2/s.

lab	method	value	mark	z(targ)	remarks
178	D445	145.3		-0.36	
179	D445	145.4		-0.20	
214		-----		-----	
237	D445	145.6		0.11	
325	D445	145.9		0.59	
331		-----		-----	
349	D445	145.9		0.59	
451	D7279	145.4		-0.20	
473	D7042	145.696		0.26	
496	D445	145.115		-0.65	
497	D7279	146.2	ex	1.06	Not corrected to D445, test result excluded
633	D7279	145.94532		0.66	
862	D445	145.7		0.27	
902	D445	145.1		-0.68	
1026	D445	145.6		0.11	
1146	D445	145.80		0.43	
1743	D7279	143.2	C,G(0.01)	-3.67	Corrected to D445, first reported 143.7
6016	D7042	144.95		-0.91	

normality	OK
n	14
outliers	1 (+1excl)
mean (n)	145.529
st.dev. (n)	0.3241
R(calc.)	0.908
R(D445:17)	1.775

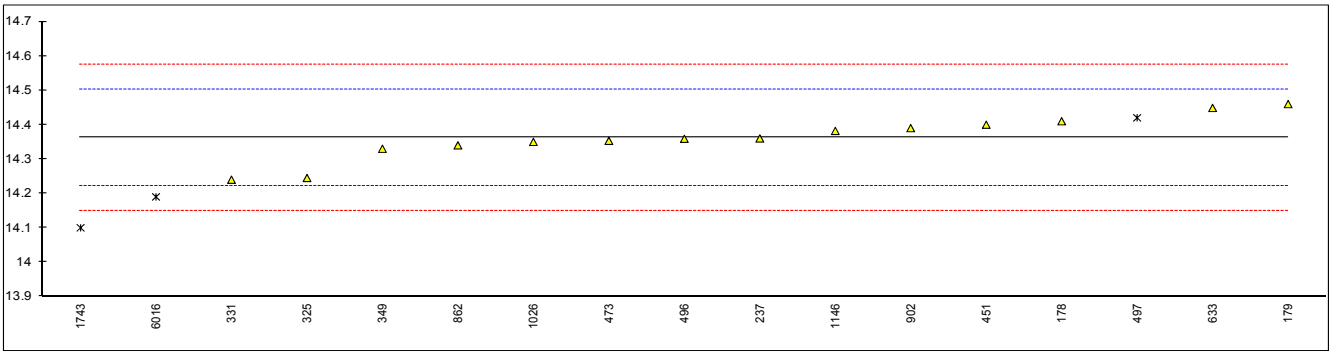


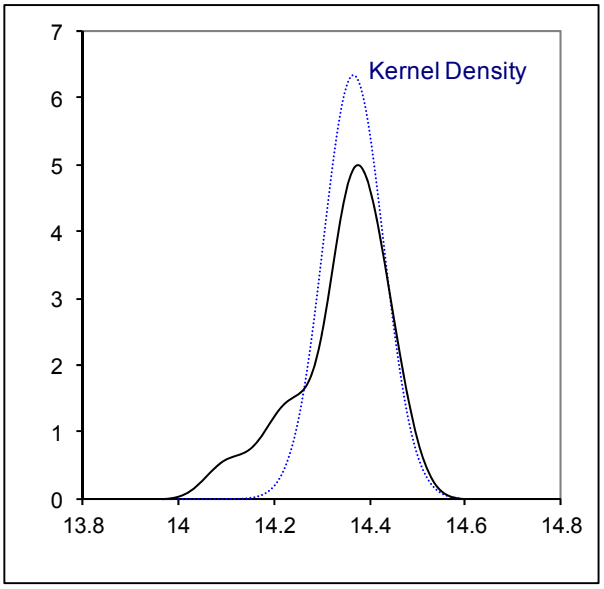


Determination of Kinematic Viscosity at 100°C on sample #17036; results in mm^2/s.

lab	method	value	mark	z(targ)	remarks
178	D445	14.41		0.68	
179	D445	14.46		1.38	
214		-----		-----	
237	D445	14.36		-0.03	
325	D445	14.245		-1.65	
331	D7279Mod.	14.24		-1.72	
349	D445	14.33		-0.45	
451	D7279	14.4		0.54	
473	D7042	14.353		-0.13	
496	D445	14.359		-0.04	
497	D7279	14.42	ex	0.82	Not corrected to D445, test result excluded
633	D7279	14.448852		1.23	Corrected to D445
862	D445	14.34		-0.31	
902	D445	14.39		0.40	
1026	D445	14.35		-0.17	
1146	D445	14.382		0.28	
1743	D7279	14.10	C,DG(0.05)	-3.70	Corrected to D445, first reported 14.57
6016	D7042	14.190	DG(0.05)	-2.43	

normality	OK
n	14
outliers	2 (+1excl)
mean (n)	14.3620
st.dev. (n)	0.06349
R(calc.)	0.1778
R(D445:17)	0.1982

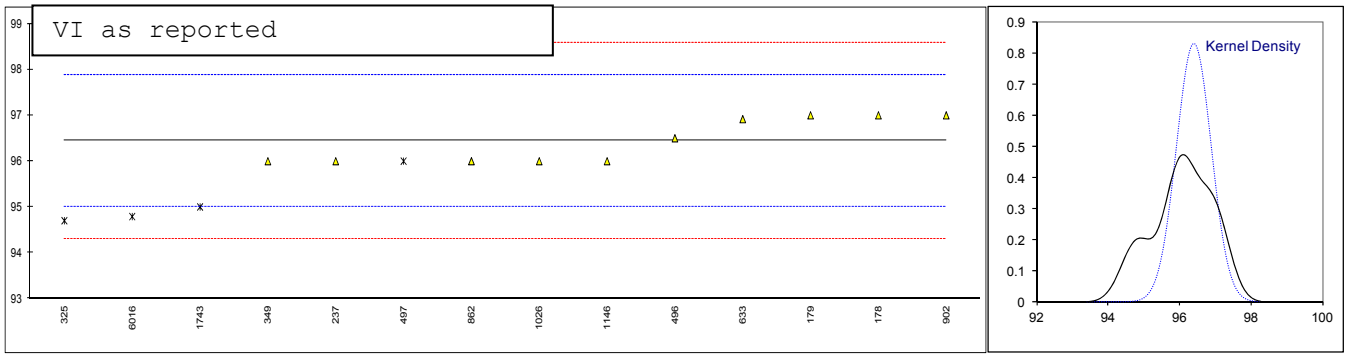


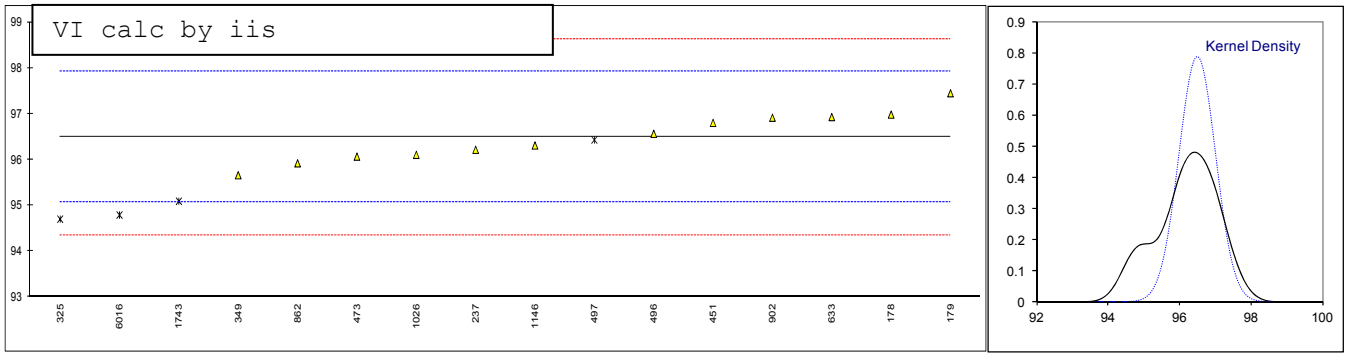


Determination of Viscosity Index (V.I.) on sample #17036

lab	method	value	mark	z(targ)	iis calc	remarks
178	D2270	97		0.78	96.984	
179	D2270	97		0.78	97.449	
214		-----		-----	-----	
237	D2270	96		-0.62	96.215	
325	D2270	94.7	D(0.05)	-2.44	94.701	
331		-----		-----	-----	
349	D2270	96		-0.62	95.660	
451		-----		-----	96.801	
473		-----		-----	96.067	
496	D2270	96.5		0.08	96.564	
497	D2270	96	ex	-0.62	96.429	Result excluded, see Visco at 40 and 100°C
633	D2270	96.917		0.67	96.929	
862	D2270	96		-0.62	95.920	
902	D2270	97		0.78	96.914	
1026	D2270	96		-0.62	96.105	
1146	D2270	96		-0.62	96.309	
1743	ISO2909	95	C,ex	-2.02	95.092	Result excluded, outlier in Visco at 40 and 100°C, f.r.101
6016	D2270	94.79	ex	-2.31	94.791	Result excluded, outlier in Visco at 100°C

normality	OK	OK
n	10	12
outliers	1 (+3excl)	1 (+3excl)
mean (n)	96.44	96.49
st.dev. (n)	0.488	0.530
R(calc.)	1.37	1.48
R(D2270:10)	2	2





Determination of Membrane Filtration 5µm on sample #17036; results in %M/M.

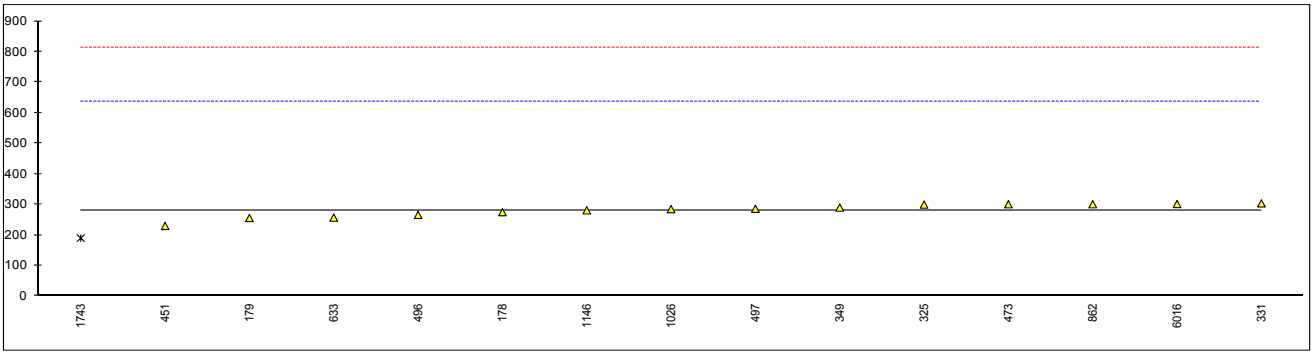
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237		-----		-----	
325		-----		-----	
331		-----		-----	
349		-----		-----	
451		-----		-----	
473		-----		-----	
496		-----		-----	
497		-----		-----	
633		-----		-----	
862	D4055	<0.01		-----	
902		-----		-----	
1026	D4898	33.6		-----	Used Heptane as solvent, unit error?
1146		-----		-----	
1743		-----		-----	
6016		-----		-----	

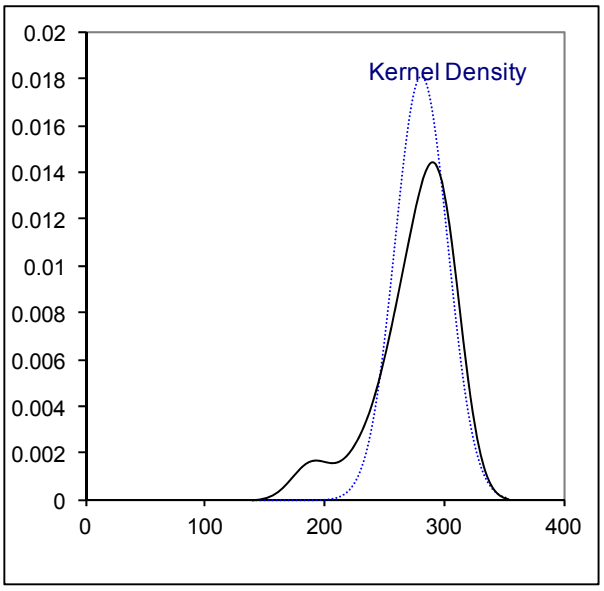
normality	unknown
n	2
outliers	n.a.
mean (n)	n.a.
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D4055:04)	n.a.

Determination of Water by KF on sample #17036; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D6304-C	275		-0.03	
179	D6304-C	256		-0.14	
214		-----		-----	
237		-----		-----	
325	D6304-C	300		0.11	
331	In house	303.8		0.13	
349	D6304-A	290		0.05	
451	D6304-C	230		-0.29	
473	D6304-C	301		0.11	
496	D6304-C	266		-0.08	
497	D6304-C	286		0.03	
633	D6304-C	257		-0.13	
862	D6304-C	301		0.11	
902		-----		-----	
1026	D6304-A	285		0.02	
1146	D6304-C	281		0.00	
1743	ISO12937	190	C,G(0.05)	-0.51	First reported 230
6016	D6304-A	301.6		0.12	

normality	OK
n	14
outliers	1
mean (n)	280.96
st.dev. (n)	21.997
R(calc.)	61.59
R(D6304:16e1)	497.57

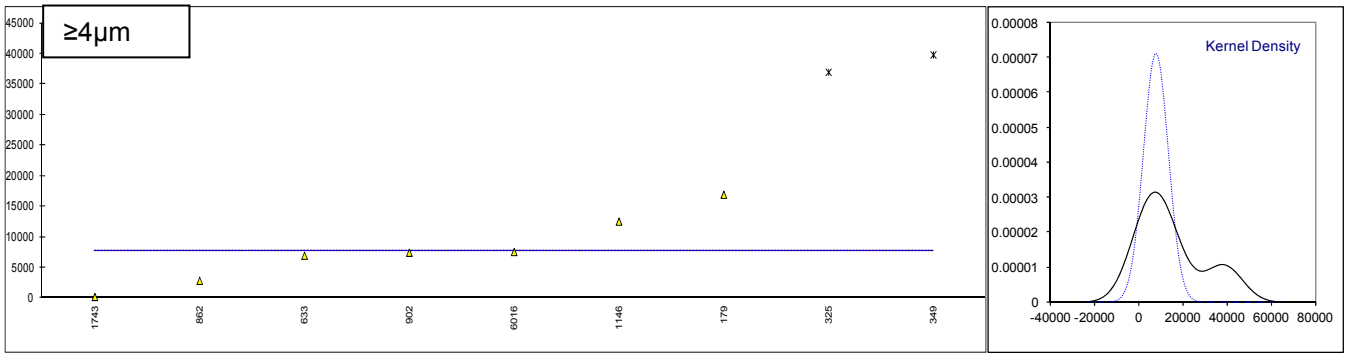


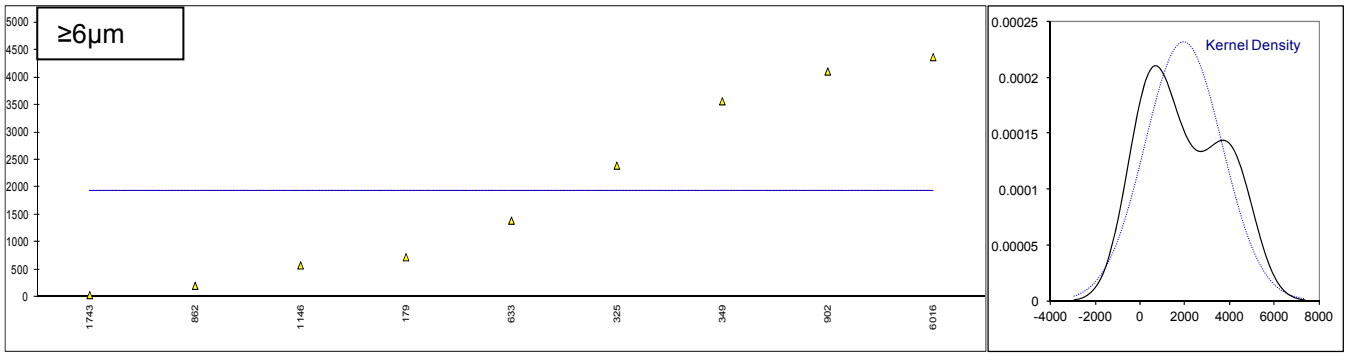


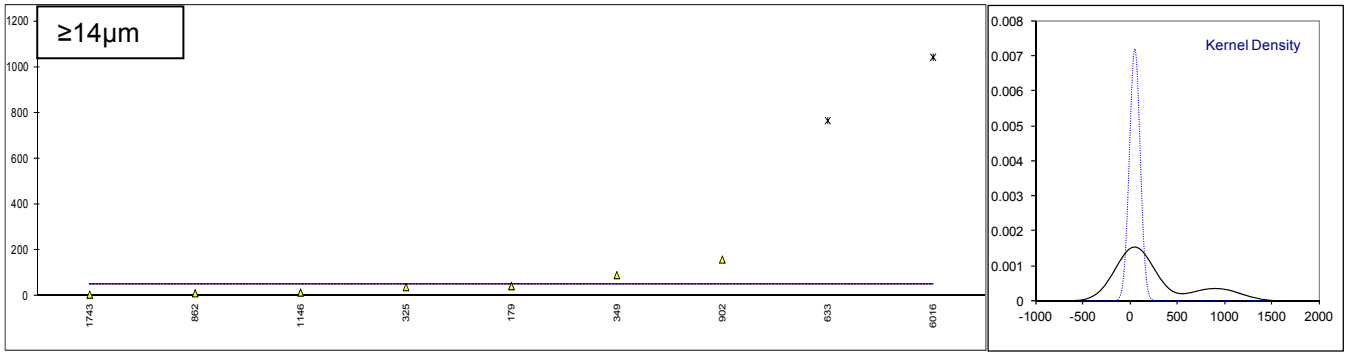
Determination of Level of Contamination on sample #17036; results in counts per ml.

lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D7647	16935		-----	729		-----	44		-----
214		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
325	ISO11500/ISO4406	36966.07	DG(0.05)	-----	2394.47		-----	39.73		-----
331		-----		-----	-----		-----	-----		-----
349	ISO4407	39819	DG(0.05)	-----	3565		-----	92		-----
451		-----		-----	-----		-----	-----		-----
473		-----		-----	-----		-----	-----		-----
496		-----		-----	-----		-----	-----		-----
497		-----		-----	-----		-----	-----		-----
633	D7647	6947.49		-----	1393.48		-----	766.30	DG(0.01)	-----
862	ISO11500	2837		-----	209		-----	13		-----
902	D7647	7439		-----	4106		-----	160.1		-----
1026		-----		-----	-----		-----	-----		-----
1146	ISO11500	12533		-----	580		-----	16		-----
1743	ISO4407	207		-----	40		-----	7		-----
6016	ISO4406	7555	C	-----	4368	C	-----	1043	C,DG(0.01)	-----
	normality	OK			OK			not OK		
	n	7			9			7		
	outliers	2			0			2		
	mean (n)	7779.07			1931.66			53.12		
	st.dev. (n)	5613.208			1719.537			55.277		
	R(calc.)	15716.98			4814.70			154.78		
	R(D7647:10)	8790.35			1468.06			71.71		

Lab 6016: test results were first reported in table of ISO4406 scale



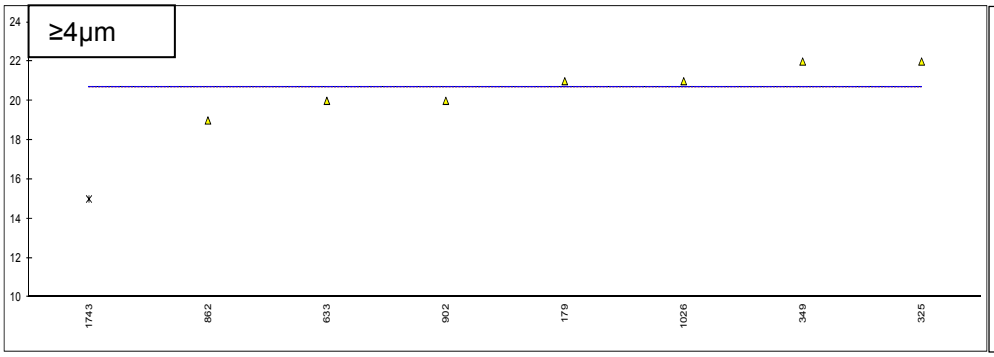


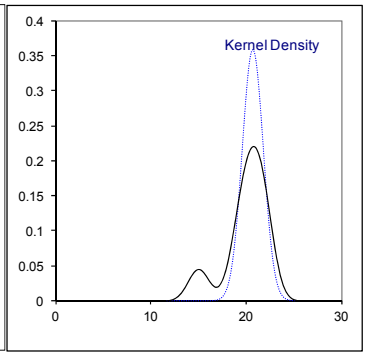


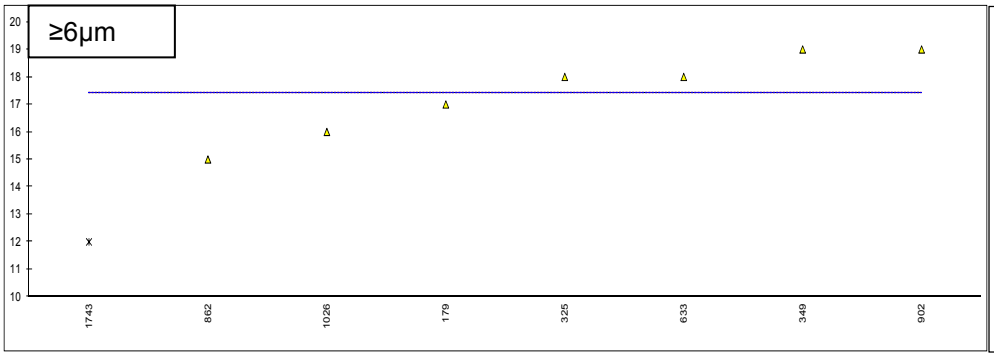
Determination of Level of Contamination acc. to ISO4406 scale on sample #17036; results in scale number

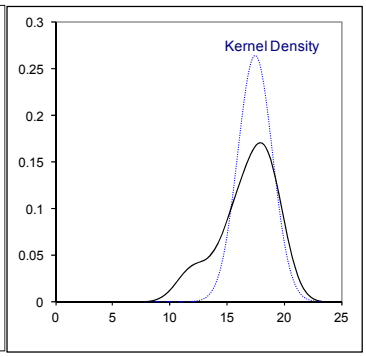
lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	ISO4406	21		-----	17		-----	13		-----
214		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
325	ISO4406	22		-----	18		-----	12		-----
331		-----		-----	-----		-----	-----		-----
349	ISO4406	22		-----	19		-----	14		-----
451		-----		-----	-----		-----	-----		-----
473		-----		-----	-----		-----	-----		-----
496		-----		-----	-----		-----	-----		-----
497		-----		-----	-----		-----	-----		-----
633	ISO4406	20		-----	18		-----	17		-----
862	ISO4406	19		-----	15		-----	11		-----
902	ISO4406	20		-----	19		-----	15		-----
1026	ISO4406	21		-----	16		-----	10		-----
1146		-----		-----	-----		-----	-----		-----
1743	ISO4406	15	G(0.05)	-----	12	G(0.05)	-----	10	ex	-----
6016		-----		-----	-----		-----	-----		-----
	normality	unknown			unknown			unknown		
	n	7			7			7		
	outliers	1			1			0 (+1excl)		
	mean (n)	20.7			17.4			13.1		
	st.dev. (n)	1.11			1.51			2.41		
	R(calc.)	3.1			4.2			6.7		
	R(D7647:10)	1.7			1.2			2		

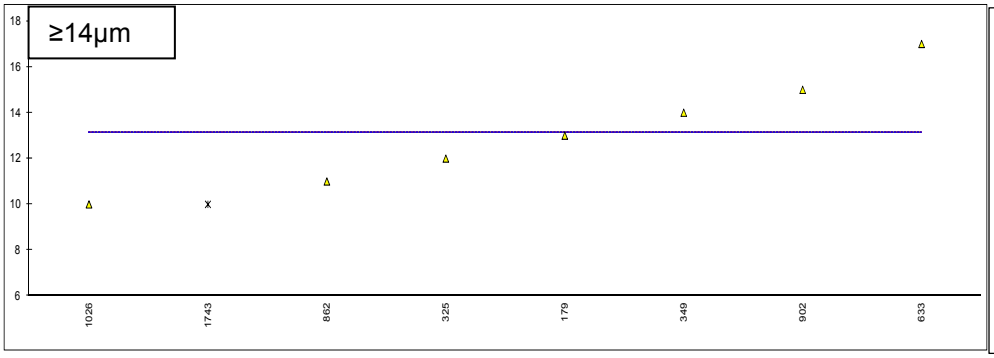
Lab 1743: test result was excluded as two other test results were statistical outliers.

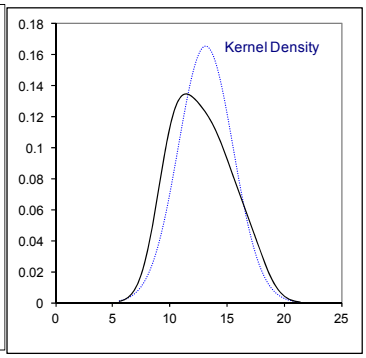








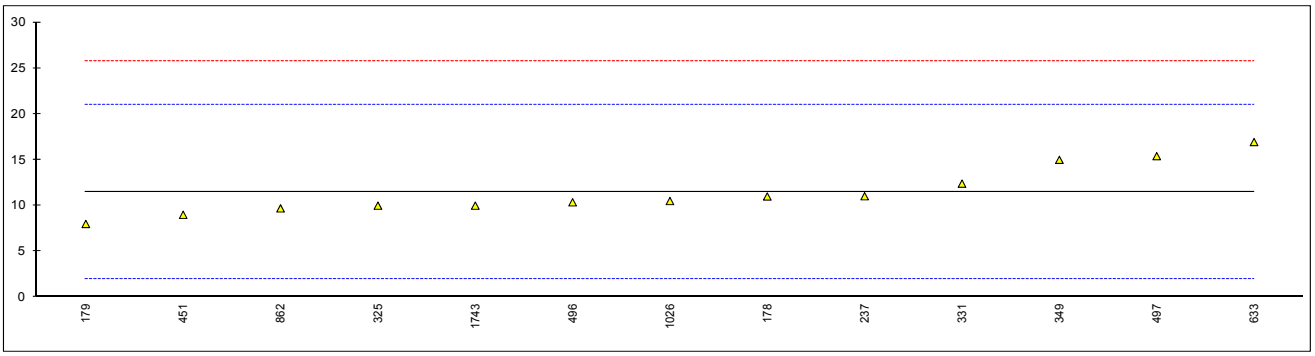


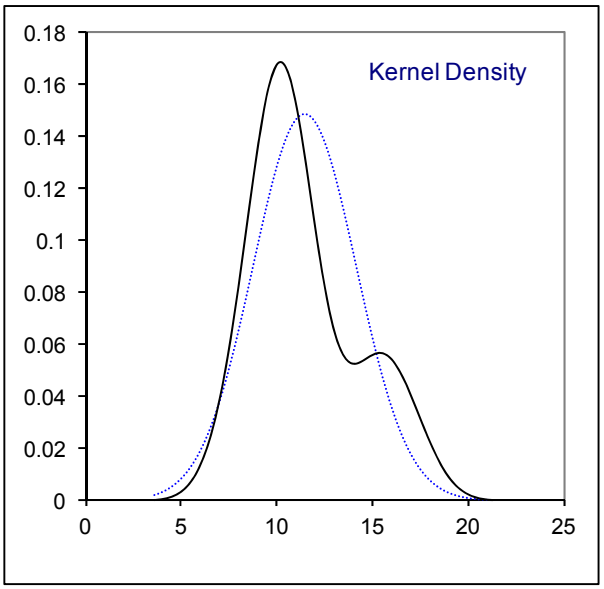


Determination of Boron as B on sample #17036; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	11		-0.10	
179	D5185	8		-0.73	
214		-----		-----	
237	D5185	11.03		-0.10	
325	D5185	10		-0.31	
331	D5185Mod.	12.4		0.19	
349	D5185	15		0.74	
451	D5185	9		-0.52	
473		-----		-----	
496	D5185	10.362		-0.24	
497	D5185	15.4		0.82	
633	D6595	16.935		1.15	
862	D5185	9.7		-0.38	
902		-----		-----	
1026	D5185	10.5		-0.21	
1146		-----		-----	
1743	D5185	10		-0.31	
6016		-----		-----	

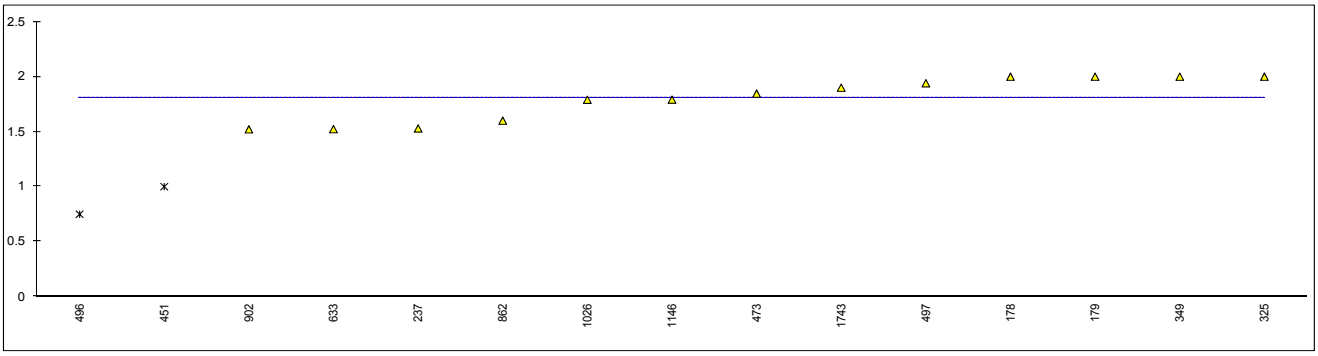
normality	OK
n	13
outliers	0
mean (n)	11.487
st.dev. (n)	2.6897
R(calc.)	7.531
R(D5185:13e1)	13.321

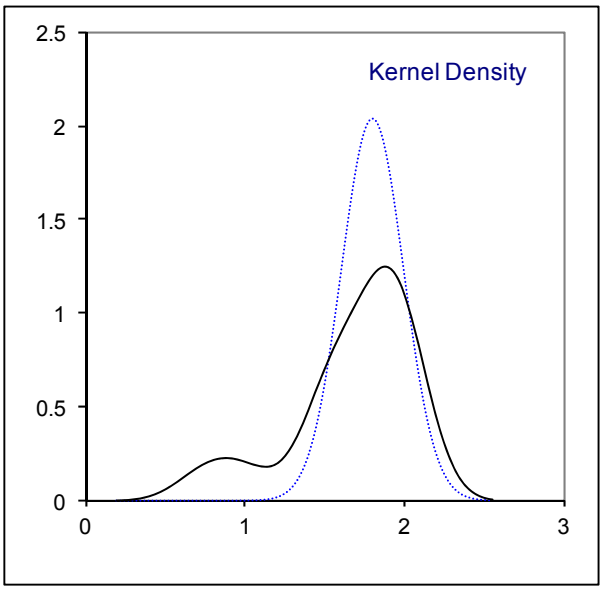




Determination of Copper as Cu on sample #17036; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	2		-----	
179	D5185	2		-----	
214		-----		-----	
237	D5185	1.529		-----	
325	D5185	2		-----	
331	D5185Mod.	<1		-----	
349	D5185	2		-----	
451	D5185	1	DG(0.01)	-----	
473	D5185	1.846		-----	
496	D5185	0.749	DG(0.01)	-----	
497	D5185	1.94		-----	
633	D6595	1.523		-----	
862	D5185	1.6		-----	
902	D5185	1.521		-----	
1026	D5185	1.79		-----	
1146	INH-5185	1.791		-----	
1743	D5185	1.9		-----	
6016		-----		-----	
	normality	OK			
	n	13			
	outliers	2			
	mean (n)	1.803			
	st.dev. (n)	0.1957			
	R(calc.)	0.548			
	R(D5185:13e1)	(0.433)			Application range: 2 – 160 mg/kg



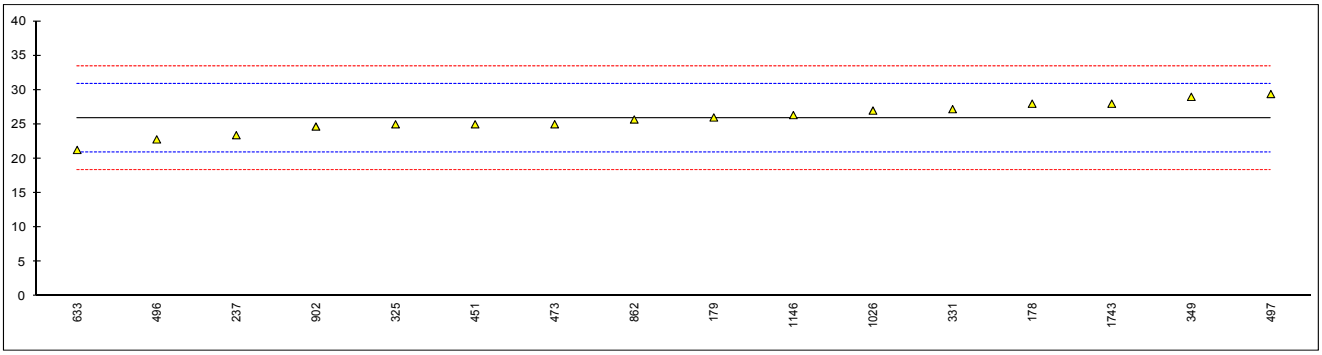


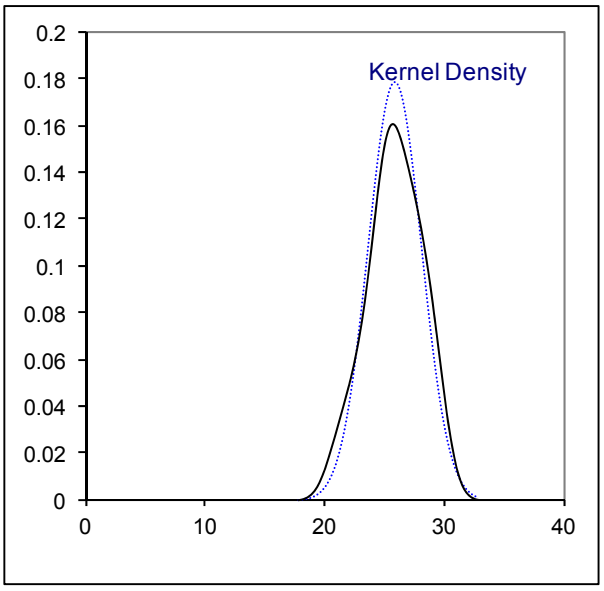
Determination of Iron as Fe on sample #17036; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	28		0.85	
179	D5185	26		0.05	
214		-----		-----	
237	D5185	23.42		-0.98	
325	D5185	25		-0.34	
331	D5185Mod.	27.2		0.53	
349	D5185	29		1.25	
451	D5185	25		-0.34	
473	D5185	25.01		-0.34	
496	D5185	22.808		-1.22	
497	D5185	29.4		1.41	
633	D6595	21.269		-1.83	
862	D5185	25.7		-0.07	
902	D5185	24.68		-0.47	
1026	D5185	27.0		0.45	
1146	INH-5185	26.34		0.19	
1743	D5185	28		0.85	
6016		-----		-----	

normality	OK
n	16
outliers	0
mean (n)	25.864
st.dev. (n)	2.2340
R(calc.)	6.255
R(D5185:13e1)	7.017

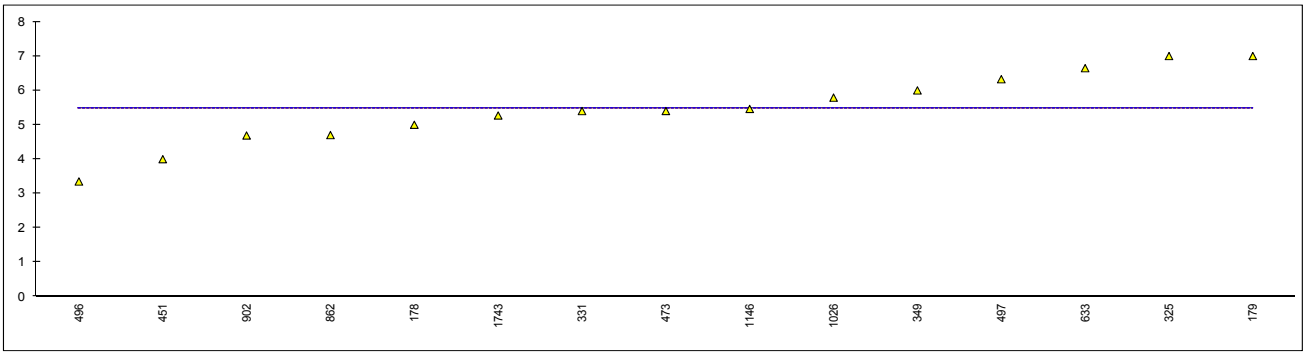
Application range : 2 – 140 mg/kg

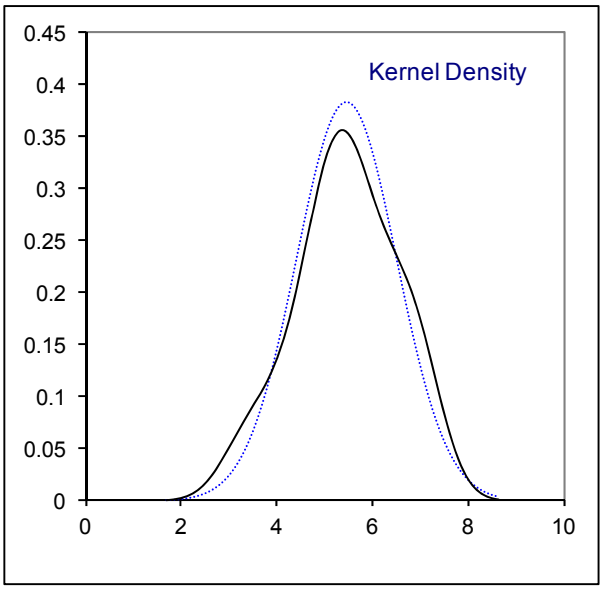




Determination of Calcium as Ca on sample #17036; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	5		-----	
179	D5185	7		-----	
214		-----		-----	
237	D5185	<10		-----	
325	D5185	7		-----	
331	D5185Mod.	5.4		-----	
349	D5185	6		-----	
451	D5185	4		-----	
473	D5185	5.401		-----	
496	D5185	3.351		-----	
497	D5185	6.326		-----	
633	D6595	6.648		-----	
862	D5185	4.7		-----	
902	D5185	4.689		-----	
1026	D5185	5.79		-----	
1146	INH-5185	5.461		-----	
1743	D5185	5.27		-----	
6016		-----		-----	
	normality	OK			
	n	15			
	outliers	0			
	mean (n)	5.469			
	st.dev. (n)	1.0457			
	R(calc.)	2.928			
	R(Horwitz)	(1.897)			Compare R(D5185:13e1) = 0.137 (appl. range 40 – 9000 mg/kg)



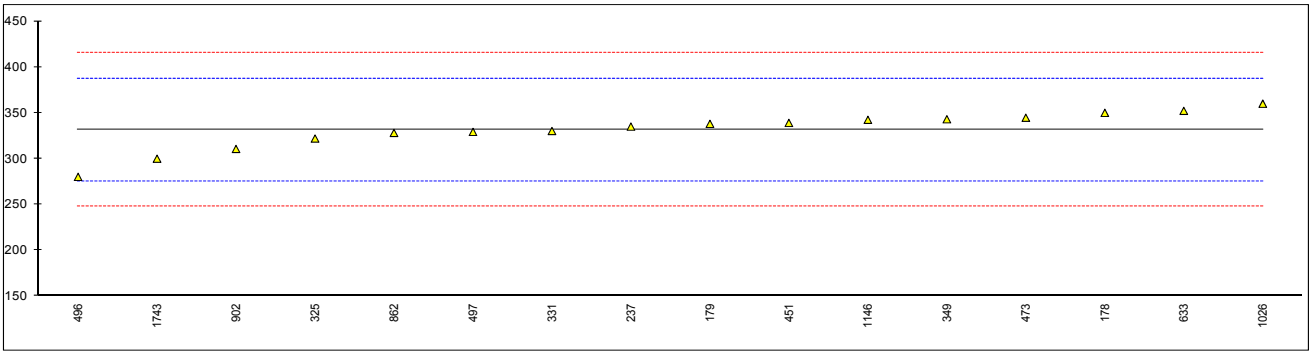


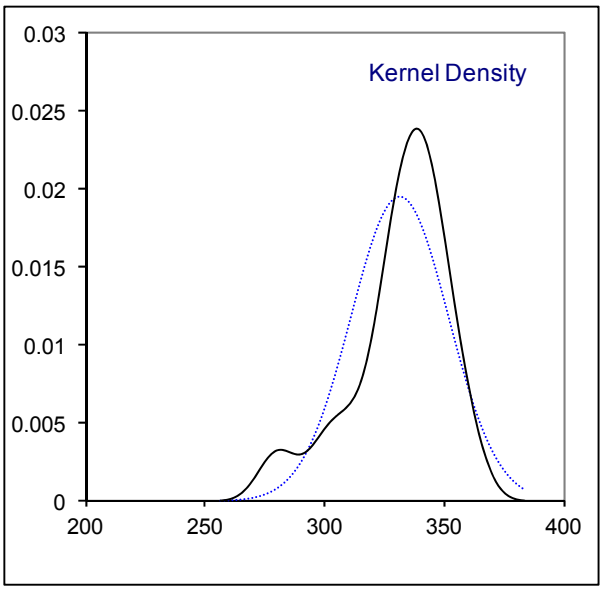
Determination of Phosphorus as P on sample #17036; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	350		0.66	
179	D5185	338		0.23	
214		-----		-----	
237	D5185	335		0.12	
325	D5185	322		-0.34	
331	D5185Mod.	330.2		-0.05	
349	D5185	343		0.41	
451	D5185	339		0.27	
473	D5185	344.7		0.47	
496	D5185	280.15		-1.84	
497	D5185	329.2		-0.08	
633	D6595	352.2		0.74	
862	D5185	328.1		-0.12	
902	D5185	310.6		-0.75	
1026	D5185	360		1.02	
1146	INH-5185	342.4		0.39	
1743	D5185	300		-1.13	
6016		-----		-----	

	normality	suspect
	n	16
	outliers	0
	mean (n)	331.534
	st.dev. (n)	20.4836
	R(calc.)	57.354
	R(D5185:13e1)	78.295

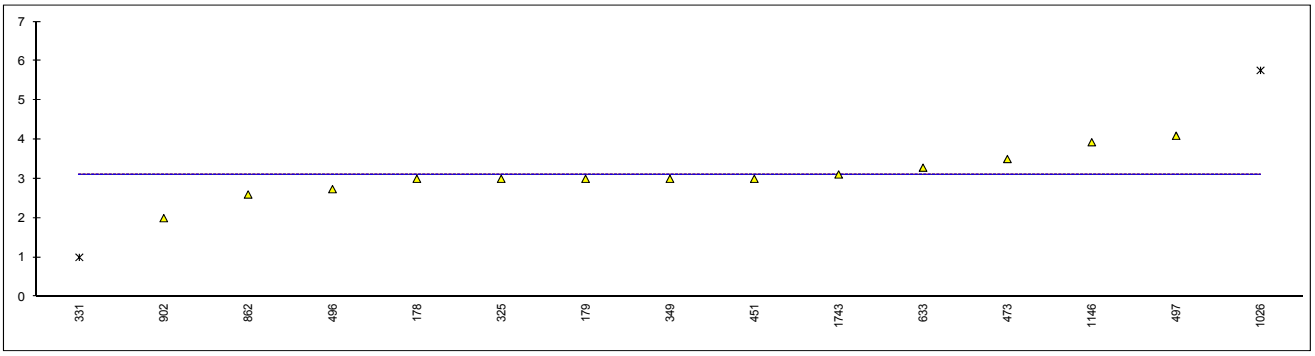
Application range: 10 – 1000 mg/kg

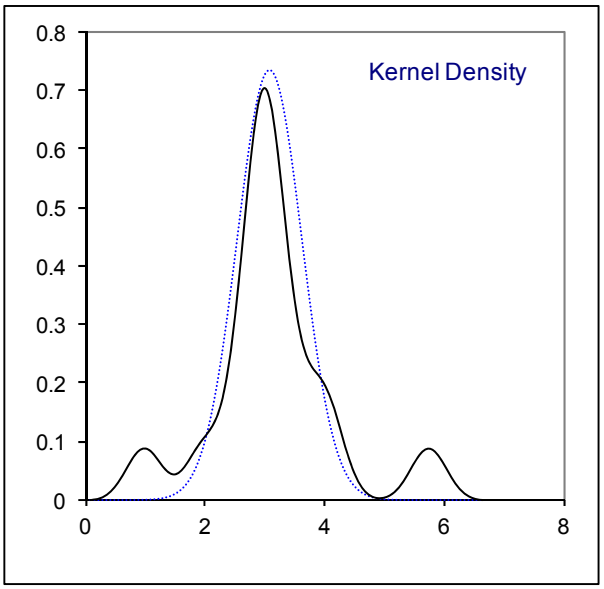




Determination of Zinc as Zn on sample #17036; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	3		-----	
179	D5185	3		-----	
214		-----		-----	
237	D5185	<5		-----	
325	D5185	3		-----	
331	D5185Mod.	1.0	D(0.05)	-----	
349	D5185	3		-----	
451	D5185	3		-----	
473	D5185	3.504		-----	
496	D5185	2.736		-----	
497	D5185	4.096		-----	
633	D6595	3.283		-----	
862	D5185	2.6		-----	
902	D5185	1.998		-----	
1026	D5185	5.76	D(0.05)	-----	
1146	INH-5185	3.931		-----	
1743	D5185	3.11		-----	
6016		-----		-----	
	normality	OK			
	n	13			
	outliers	2			
	mean (n)	3.097			
	st.dev. (n)	0.5421			
	R(calc.)	1.518			
	R(Horwitz)	(1.170)			Compare R(D5185:13e1) = 0.288 (appl. range 60 – 1600 mg/kg)





Determination of other elements on sample #17036; results in mg/kg.

Lab	method	Al	Sb	Ba	Cr	Pb	Li	Mg	Mn	Mo	Ni
178	D5185	<1	<1	<1	<1	<1	-----	<1	-----	<1	<1
179	D5185	<1	<1	<1	<1	<1	-----	<1	<1	<1	<1
214		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
237	D5185	1.27	<1	<1	<1	-----	-----	<1	<1	<1	<1
325	D5185	0	0	0	0	0	-----	0	0	0	0
331	D5185Mod.	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
349	D5185	0	0	0	0	0	0	0	0	0	0
451	D5185	1	0	0.000	0	0	0	0	0	0	0
473	D5185	<1	-----	***1.592***	<1	<1	-----	<1	<1	<1	<1
496	D5185	<6	<1	<0.5	<1	<10	0	0.713	<5	<5	<5
497	D5185	0.3	0.1	0.3	0.2	0.3	0.1	0.85	0.3	0.4	0.2
633	D6595	0.106	-----	0.0615	0.259	0.034	0.018	0.298	0.486	0.521	0.308
862	D5185	<0.1	-----	<0.1	0.3	<0.1	<0.1	0.1	0.2	<0.1	0.1
902	D5185	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1026	D5185	<1	-----	0.072	0.355	0.120	-----	0.223	-----	0.050	0.149
1146	INH-5185	0.3405	-----	0.0963	0.3115	-----	0.0528	0.3183	0.2550	-----	0.1709
1743	D5185	0	0.95	0.09	0.3	0	0	0	0.2	0	0.1
6016		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
	normality	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	n	14	10	10	15	12	9	15	12	13	14
	outliers	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean (n)	<2	<2	<0.5	<1	<2	<1	<1	<2	<2	<2
	st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	R(lit)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

Lab	method	K	Si	Ag	Na	Sn	Ti	V
178	D5185	<3	1	<0.10	3	<1	-----	<1
179	D5185	<1	1	<0.10	<3	<1	<1	<1
214		-----	-----	-----	-----	-----	-----	-----
237	D5185	<1	<1	<1	<1	<1	<1	<1
325	D5185	0	0	0	0	0	-----	0
331	D5185Mod.	<1	<1	<1	<1	<1	<1	<1
349	D5185	***6***	0 **f.r.3**	0	1 **f.r.6**	0	0	0
451	D5185	3	1	0	3	0.000	0	0
473	D5185	-----	<1	<1	***8.793***	2.519	<1	<1
496	D5185	<40	<8	<0.5	<7	<10	<5	<1
497	D5185	0.2	0.9	0.1	1.23	0.5	0.1	0.1
633	D6595	0.227	1.374	0.090	1.782	0.438	0.250	0.171
862	D5185	0.3	0.7	<0.1	0.6	<0.1	<0.1	<0.1
902	D5185	-----	-----	-----	-----	-----	-----	-----
1026	D5185	-----	<1	-----	<1	0.337	0.54	0.044
1146	INH-5185	-----	0.6508	0.1719	0.4749	0.2800	0.0664	0.1934
1743	D5185	1.2	0.5	0	1.4	0.8	0.1	0
6016		-----	-----	-----	-----	-----	-----	-----
	normality	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	n	11	14	11	13	15	15	15
	outliers	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	mean (n)	<5	<2	<0.5	<5	<5	<5	<1
	st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	R(lit)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

Test results in Bold, Italic and underlined are marked as “False positive test result”

APPENDIX 2

Number of participants per country

1 lab in ALGERIA
1 lab in BELGIUM
1 lab in CHINA, People's Republic
2 labs in FRANCE
2 labs in GERMANY
1 lab in KAZAKHSTAN
2 labs in NETHERLANDS
1 lab in NIGERIA
1 lab in NORWAY
1 lab in PHILIPPINES
1 lab in SPAIN
1 lab in TURKEY
1 lab in UNITED KINGDOM
2 labs in UNITED STATES OF AMERICA

APPENDIX 3

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	= 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 statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
f.r.	= first reported
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, March 2017
2 ASTM E178:02
3 ASTM E1301:95(2003)
4 ISO 5725:86
5 ISO 5725, parts 1-6, 1994
6 ISO 13528:05
7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9 IP 367:84
10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee Technical Brief, No 4 January 2001
14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analys, 127, 1359-1364 (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2),165-172, (1983)
16 Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)