Results of Proficiency Test
Gear Oil (fresh)
April 2015

Organised by: Institute for Interlaboratory Studies
                Spijkenisse, the Netherlands

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Appendices:

1   INTRODUCTION

On request of several participants, the Institute for Interlaboratory Studies decided to organise a new proficiency test for the analysis of Gear Oil (fresh) during the annual proficiency testing program 2014/2015. In this interlaboratory study 13 laboratories in 12 different countries have participated. See appendix 2 for the number of participants per country.
In this report, the results of the 2015 Gear Oil (fresh) proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organizer of this proficiency test. It was decided to send one bottle of 1L (labelled #15057) of Gear Oil (fresh) that was purchased from a supplier. The analyses for fit-for-use and homogeneity were subcontracted. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1   QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. 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 a regular basis by sending out questionnaires.

2.2   PROTOCOL

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 Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3), which can be downloaded from www.iisnl.com.

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 was purchased from a supplier. The 200 litre bulk material was homogenized and part of this bulk was transferred into 30 brown glass bottles of 1 litre (labelled #15057). The homogeneity of the subsamples #15057 was checked by determination of Density at 15°C in accordance with ASTM D4052 on 4 stratified randomly selected samples.

	Density at 15 ^{o}C in kg/L
Sample #15057-1	886.99
Sample #15057-2	886.99
Sample #15057-3	887.00
Sample #15057-4	887.00

Table 1: homogeneity test results of subsamples #15057

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 15 ^{o}C in kg/L
r (sample #15057)	0.02
reference test	ASTM D4052:11
0.3 x R(reference test)	0.15

Table 2: evaluation of the repeatability of the subsamples #15057

The calculated repeatability was less than 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsample #15057 was assumed.

To each of the participating laboratories, one sample of 1 L in a brown glass bottle (labelled #15057) was sent on March 11, 2015.

2.5   ANALYSES

The participants were requested to determine on sample #15057: Acid Number (Total), Copper Corrosion, Density at 15°C, Flash Point PMcc, Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Pour Point Manual and Automated, Rust prevention (proc. A), Sulphur, Calcium, Phosphorus and Zinc.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The detailed report form was also made available for download on the iis website www.iisnl.com.

3   RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated per determination in the appendix of this report. The laboratories are presented by their code numbers.

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

Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1   STATISTICS

Statistical calculations were performed as described in the report 'iis. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 (1986 and 1994, lit. 3 and 4) the original results per determination were submitted to Dixon's and/or 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 (ref. 15). 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 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 analysis 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. The Kernel Density is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.13 and 14). 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 target standard deviation was calculated from the literature reproducibility by division with 2.8.
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:

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

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

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

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

4   EVALUATION

In this interlaboratory study no problems were encountered with the dispatch of the samples. All participants reported test results, although not all laboratories were able to report all analyses requested. Two participants reported test results after the final reporting date. In total 13 participants reported 125 test results. Observed were 6 outlying results, which is 4.8% of the numerical 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 results are discussed per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

Acid Number (total): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D664:11a.

Copper Corrosion: Six laboratories reported a test result. No problems have been observed. The participants agreed on classification 1A.

Density at 15°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 D4052:11. The low number of test results may explain part of this larger spread.

Flash Point PMcc: This determination was problematic. One statistical outlier was observed, this laboratory used the Cleveland Open Cup test (ISO2592). The calculated reproducibility after rejection of statistical outlier is not in agreement with ASTM D93A:15. The low number of test results may explain part of this larger spread.

Kin.Visco.at 40°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement full with the requirements of ASTM D445:15.

Kin.Visco.at 100°C: 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 D445:15.

Viscosity Index: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with ASTM D2270:10e1.
Also iis calculated the Viscosity Index from the test results reported for the kinematic viscosities at 40°C and 100°C. These calculated test results were compared to the reported test results. The results of two participants were marked as having a calculation error.

Pour Point manual: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with requirements of ASTM D97:12. The low number of test results and the rounding to 3°C may (partly) explain for the low precision.

Pour Point autom.:   This determination may not be problematic. Four participants reported a test result. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with requirements of ASTM D5950:14.
Three laboratories reported a test result for both D97 and D5950. Since both methods are different, it is possible that the test results from both tests are different. One laboratory did find very different results (a difference of 8°C), but the other two laboratories reported the same test result for both the manual and the automated test.

Rust prevention: Regretfully, only three participants reported a test result. One reported the absence of rust (No Rusting), while two participants reported the test as "Pass".

Sulphur: This determination may be problematic. Only three participants reported a test result. The calculated reproducibility is not in agreement with the requirements of ASTM D4951:14 (or ASTM D2622:10 and ASTM D4294:10). The low number of test results may explain part of this larger spread.

Water separability   The determination may not be problematic. Only five participants reported test results. No statistical outliers were observed. The calculated reproducibilities are in full agreement with the requirements of ASTM D1401:12.
For 'time aborted' different test results were reported. D1401 describes complete break only as '40-40-0', whereas a complete break also was interpreted as 'no emulsion layer present', for example '41-39-0'.

Calcium as Ca: Only five participants reported a numerical test result (with a mean of 1.87 mg/kg), one reported <0.1 mg/kg and another <0.01 mg/kg. The reproducibility described in ASTM D5185:13e1 has an application range

of 40-9000 mg/kg and cannot be used. Therefore no conclusions were drawn.

Phosphorus as P: This determination was very problematic. Six participants reported a test result. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D5185:13e1.

Zinc as Zn: Only five participants reported a numerical test result (with a mean of 2.43 mg/kg), one reported <1 mg/kg and another <0.01 mg/kg. The reproducibility described in ASTM D5185:13e1 has an application range of 60-1600 mg/kg and cannot be used. Therefore no conclusions were drawn.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories that participated. The average results, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM standards), are compared in the next table.

Parameter	unit	n	Average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	8	0.35	0.17	0.19
Copper Corrosion		6	1A	n.a.	n.a.
Density at 15 °C	kg/m^3	9	887.2	0.9	0.5
Flash Point PMcc	^{o}C	10	199	18	14
Kinematic Viscosity at 40 °C	mm^2/s	13	218.7	2.3	2.7
Kinematic Viscosity at 100 °C	mm^2/s	11	19.30	0.18	0.37
Viscosity Index		10	99.8	1.7	2.0
Pour Point, Manual	^{o}C	7	-18	18	9
Pour Point, Autom.	^{o}C	3	-13	5	5
Rust Prevention (proc. A)		3	Pass	n.a.	n.a.
Sulphur	mg/kg	3	6580	1087	610
Water Separability at 82°C, time					
- to reach 3 ml or less emulsion	min.	4	13	5	20
- to reach 37 ml of water	min.	3	13	6	20
Calcium	mg/kg	5	1.9	5.1	n.a.
Phosphorus	mg/kg	6	294	501	74
Zinc	mg/kg	5	2.4	7.6	n.a.

Table 3: reproducibilities of results of sample #15057

Without further statistical calculations it can be concluded that for a number of tests there is a not a good compliance of the group of participants with the relevant standards. The tests that are problematic have been discussed in paragraph 4.1.

4.3   COMPARISON OF PT UNCERTAINTIES AGAINST THE STANDARDS.

The performance of the determinations of the proficiency tests was compared to the requirements of the respective standards. The conclusions are given in the following table:

Determination	March 2015
Total Acid Number	+
Copper Corrosion	n.e.
Density at 15 °C	-
Flash Point PMcc	-
Kinematic Viscosity at 40 °C	+
Kinematic Viscosity at 100 °C	+
Viscosity Index	+
Pour Point, Manual	--
Pour Point, Autom.	+/-
Rust Prevention (proc. A)	n.e.
Sulphur	-
Water Separability at 82°C	++
Calcium	n.e.
Phosphorus	--
Zinc	n.e.

Table 5: comparison PT uncertainties against the standards

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:
- ++:   group performed much better than the standard
- + :   group performed better than the standard
- +/-:   group performance equals the standard
- - :   group performed worse than the standard
- -- :   group performed much worse than the standard
- n.e.:   not evaluated

APPENDIX 1

Determination of Acid Number (Total) on sample #15057; results in mg KOH/g

—lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D664	0.24		-1.67	
237	D664	0.33		-0.35	
255		-----		-----	
432		-----		-----	
473	D664	0.4		0.68	
621	D664	0.31		-0.64	
862	D664	0.39		0.53	
1201	D664	0.40		0.68	
1461		-----		-----	
1748	D664	0.411		0.84	
1807	D664	0.35		-0.06	
1877		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	0.354
st.dev. (n)	0.0589
R(calc.)	0.165
R(D664:11a)	0.191
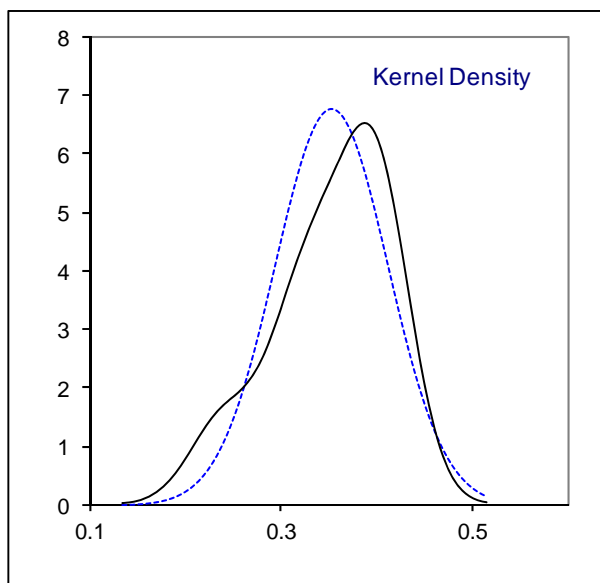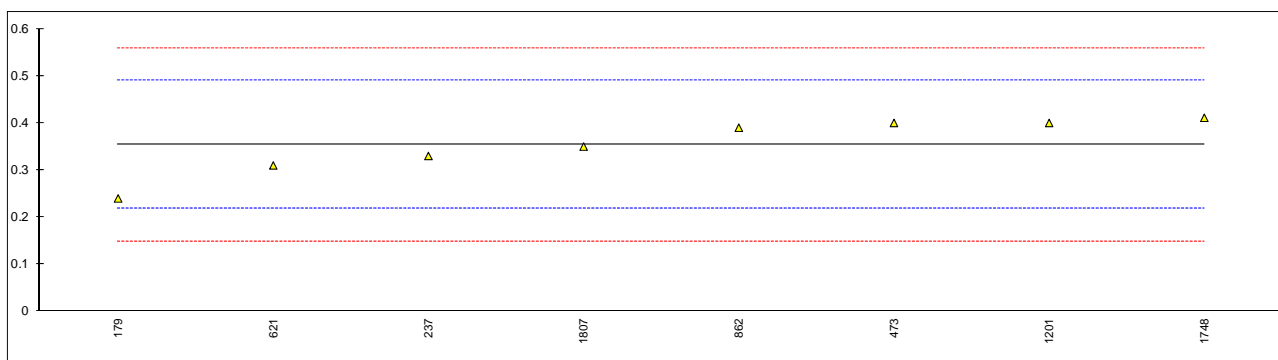
Determination of Copper Corrosion 3 hours at 100°C on sample #15057;

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D130	1A		-----	
237		-----		-----	
255		-----		-----	
432		-----		-----	
473		-----		-----	
621	D130	1A		-----	
862	D130	1A		-----	
1201	D130	1A		-----	
1461	ISO2160	1A		-----	
1748	D130	1A		-----	
1807		-----		-----	
1877		-----		-----	

normality	n.a.	
n	6	
outliers	n.a.	
mean (n)	1A	
st.dev. (n)	n.a.	
R(calc.)	n.a.	
R(lit)	n.a.	

Determination of Density at 15°C on sample #15057; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4052	887.5		1.84	
237	D4052	887.4		1.30	
255		-----		-----	
432	D4052	887.16		0.01	
473	D4052	887.5	C	1.84	first reported: 0.8875 kg/m^3
621	D4052	887.3		0.76	
862	D4052	886.9		-1.39	
1201	D4052	887.0		-0.86	
1461	ISO3675	889.0	C,G(0.01)	9.91	first reported: 888.7, method not equivalent
1748	D4052	887.1	C	-0.32	first reported: 0.8871 kg/m^3
1807		-----		-----	
1877	D4052	886.57		-3.17	

normality	OK
n	9
outliers	1
mean (n)	887.16
st.dev. (n)	0.307
R(calc.)	0.86
R(D4052:11)	0.52
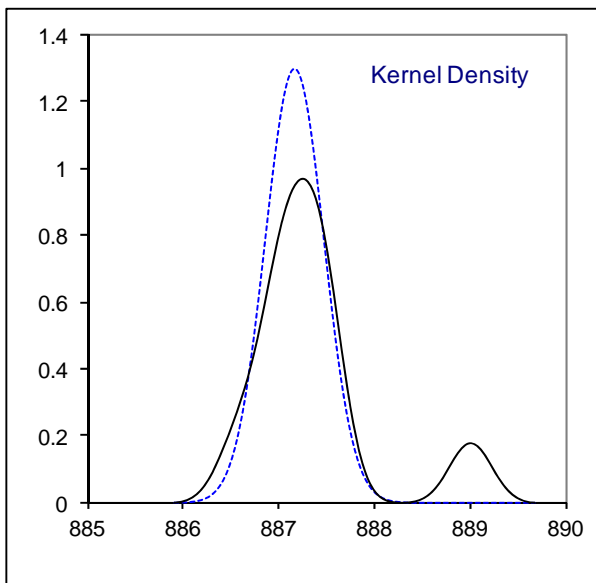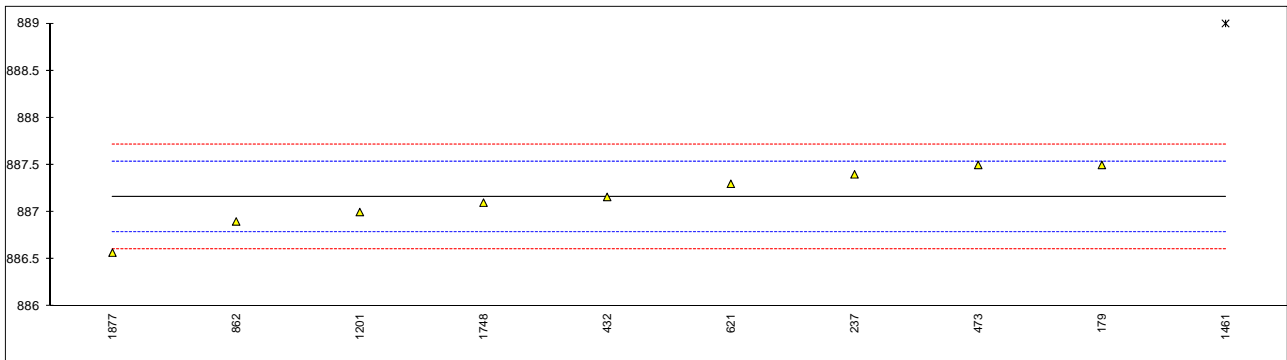
Determination of Flash Point PMcc on sample #15057; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D93	185.0		-2.77	
237	D93-A	198		-0.20	
255		-----		-----	
432	D93-A	207.0		1.59	
473	D93-A	205.5		1.29	
621	D93-B	194.0		-0.99	
862	D93-A	202.0		0.59	
1201	D93-A	200.5		0.30	
1461	ISO2592	256	G(0.01)	11.30	method not equivalent
1748	D93-A	195		-0.79	
1807	D93	203		0.79	
1877	D93-A	200		0.20	

normality	suspect
n	10
outliers	1
mean (n)	199.0
st.dev. (n)	6.43
R(calc.)	18.0
R(D93-A:15)	14.1

Kernel Density
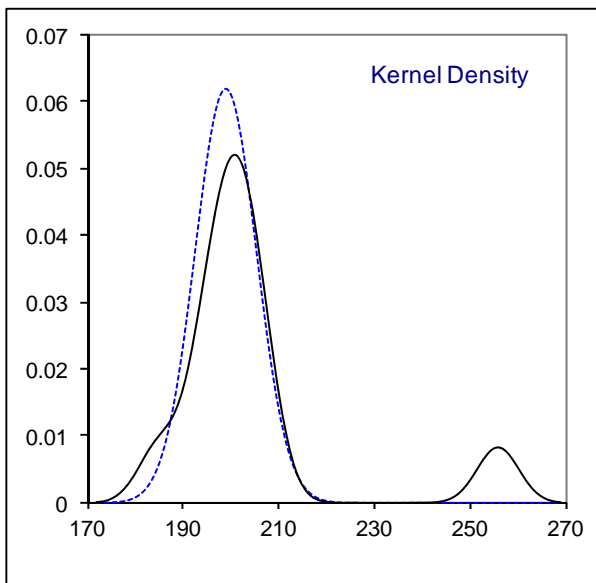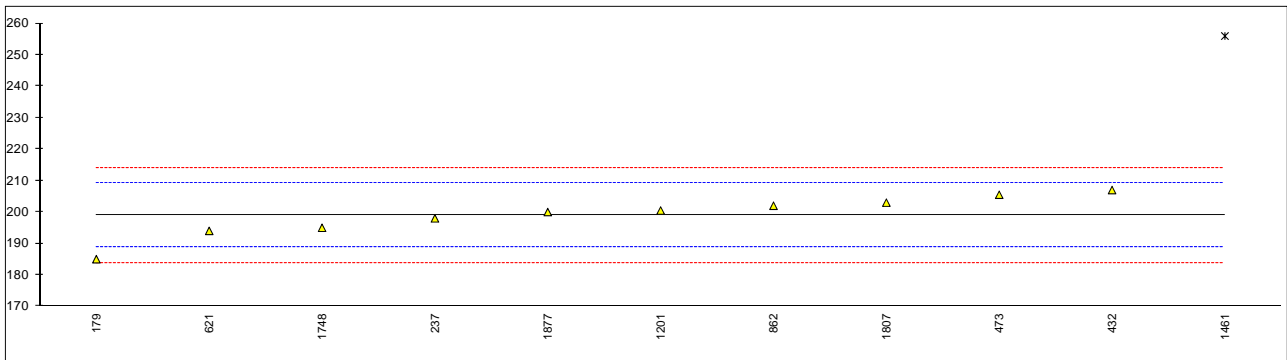
Determination of Kinematic Viscosity at 40°C on sample #15057; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	218.9		0.21	
179	D445	218.2		-0.52	
237	D445	217.7		-1.05	
255	D7279	218.6		-0.10	
432	D445	219.45		0.79	
473	D7042	220.4		1.79	
621	D445	219.3		0.63	
862	D445	217.7		-1.05	
1201	D445	219.2		0.53	reported also: Stabinger Viscosity D7042 219.9 mm^2/s
1461	ISO3140	219.2376		0.57	
1748	D445	218.3		-0.42	
1807	D445	217.7		-1.05	
1877	D445	218.4		-0.31	

normality	OK
n	13
outliers	0
mean (n)	218.70
st.dev. (n)	0.811
R(calc.)	2.27
R(D445:15)	2.67

Kernel Density
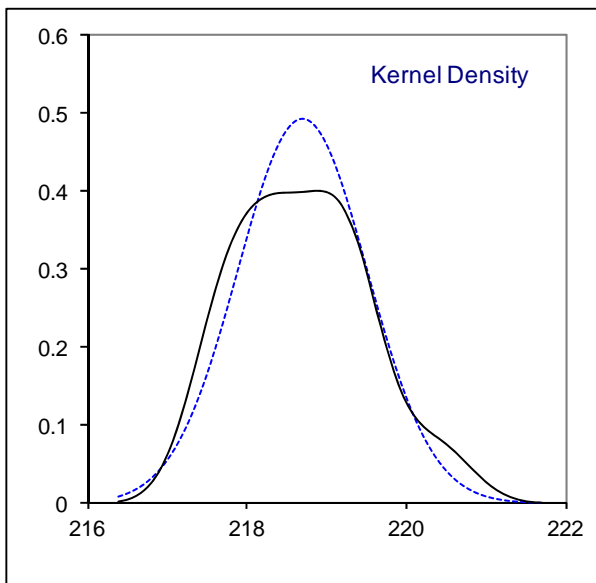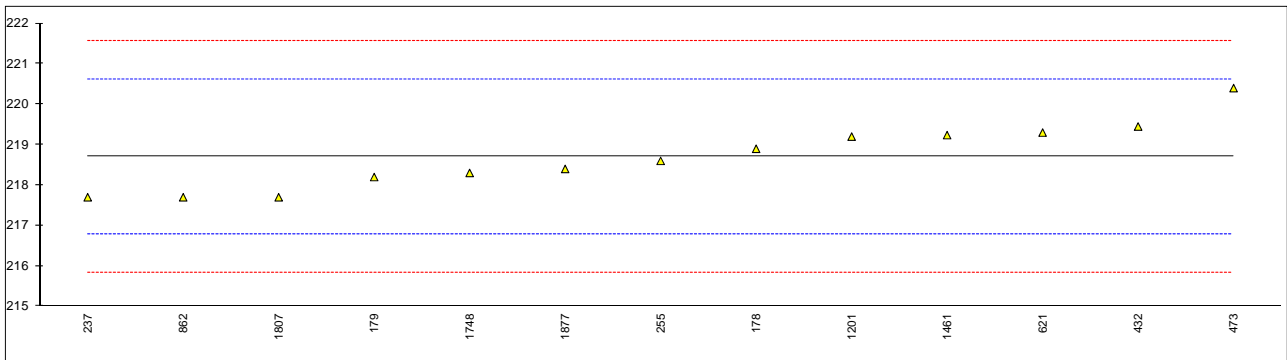
Determination of Kinematic Viscosity at 100°C on sample #15057; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	19.25		-0.53	
179	D445	19.32		0.21	
237	D445	19.26		-0.42	
255	D7279	18.96	DG(0.01)	-3.58	
432	D445	19.265		-0.37	
473	D7042	19.338		0.40	
621	D445	18.85	DG(0.01)	-4.73	
862	D445	19.24		-0.63	
1201	D445	19.22		-0.84	reported also: Stabinger Viscosity D7042 19.69 mm^2/s
1461	ISO3140	19.3901		0.94	
1748	D445	19.42		1.26	
1807	D445	19.33		0.31	
1877	D445	19.27		-0.32	

normality	OK
n	11
outliers	2
mean (n)	19.300
st.dev. (n)	0.0644
R(calc.)	0.180
R(D445:15)	0.266

Kernel Density
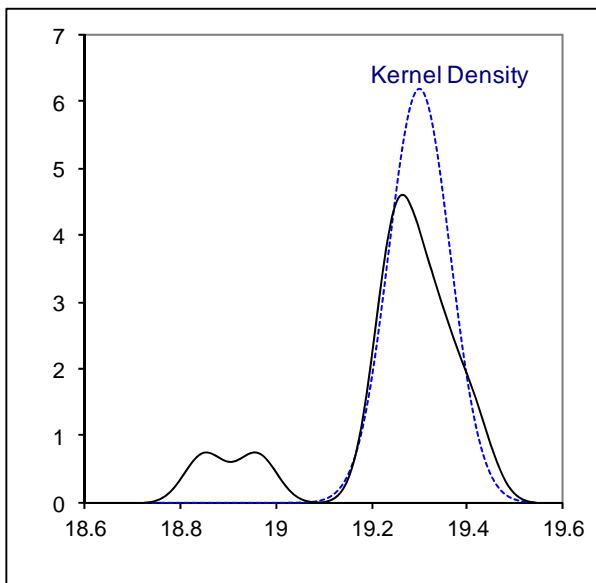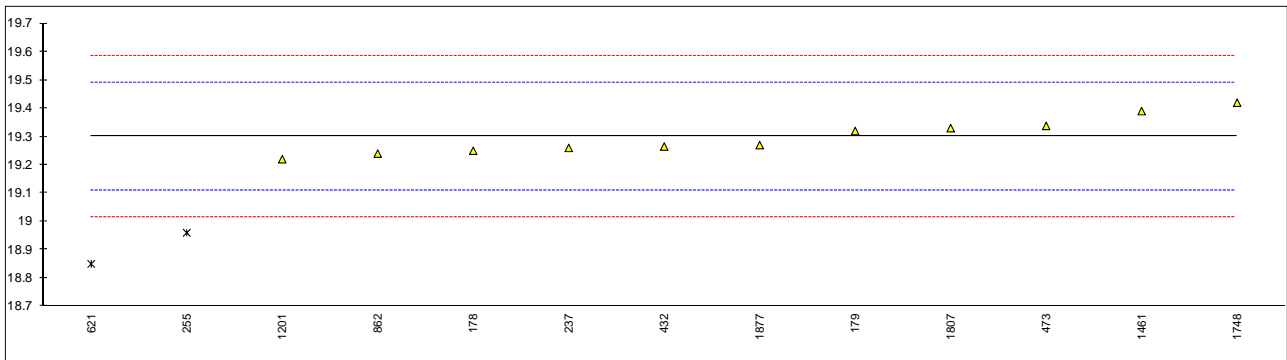
Determination of Viscosity Index on sample #15057

lab	method	value	mark	z(targ)	calc. iis	mark	remarks
178	D2270	99		-1.06	99.18		
179	D2270	100		0.34	99.93		
237	D2270	100		0.34	99.74		
255		-----		-----	-----		
432	D2270	99.1		-0.92	99.05		
473	D2270	99.5		-0.36	99.16	E	iis calculated different VI
621	D2270	95.7	G(0.01)	-5.68	96.08	G(0.01),E	outlier in D445 100°C, iis calculated different VI
862	D2270	100		0.34	99.61		
1201	D2270	99		-1.06	98.86		
1461	ISO2909	100		0.34	100.00		
1748	D2270	101		1.74	100.72		
1807		-----		-----	100.25		
1877	D2270	100		0.34	99.50		

normality	OK		OK	
n	10		11	
outliers	1		1	
mean (n)	99.76		99.64	
st.dev. (n)	0.622		0.564	
R(calc.)	1.74		1.58	
R(D2270:10e1)	2.00		2.00	

Kernel Density
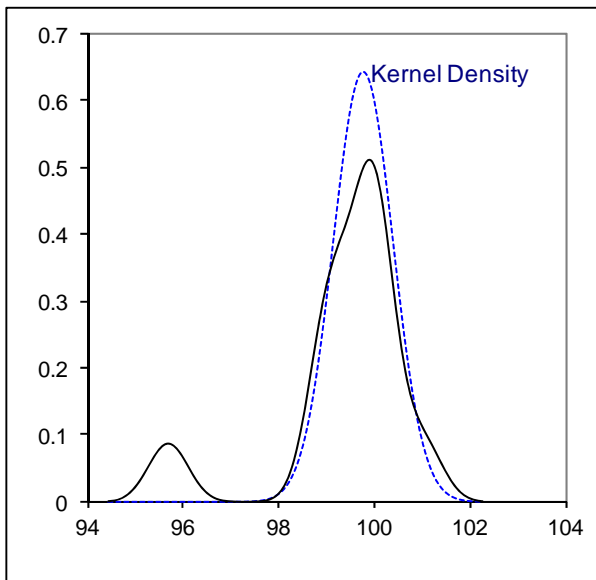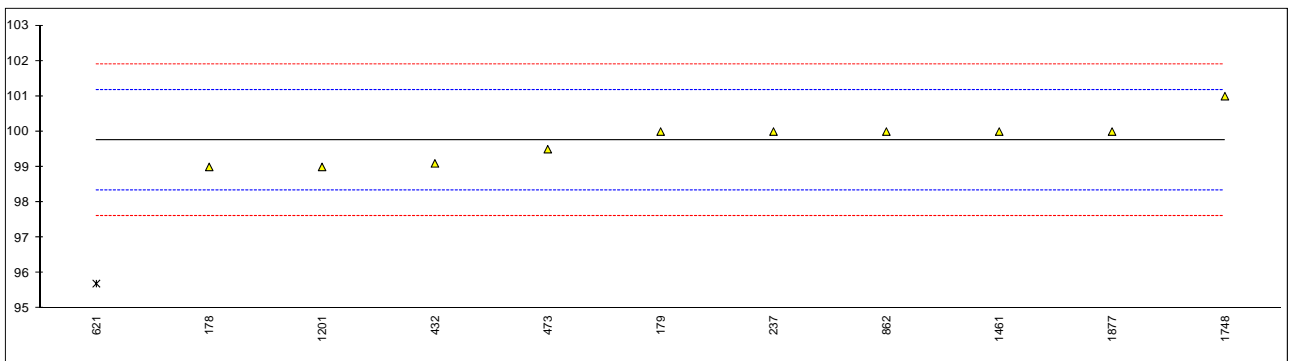
Determination of Pour Point, manual on sample #15057; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D97	<-15		-----	
255		-----		-----	
432	D97	-23		-1.51	
473		-----		-----	
621		-----		-----	
862	D97	-12		1.91	
1201	ISO3016	-12		1.91	
1461	ISO3016	-12		1.91	
1748	D97	-23		-1.51	
1807	D97	-18		0.04	
1877	D97	-27		-2.76	

normality	unknown
n	7
outliers	0
mean (n)	-18.1
st.dev. (n)	6.31
R(calc.)	17.7
R(D97:12)	9.0

Kernel Density
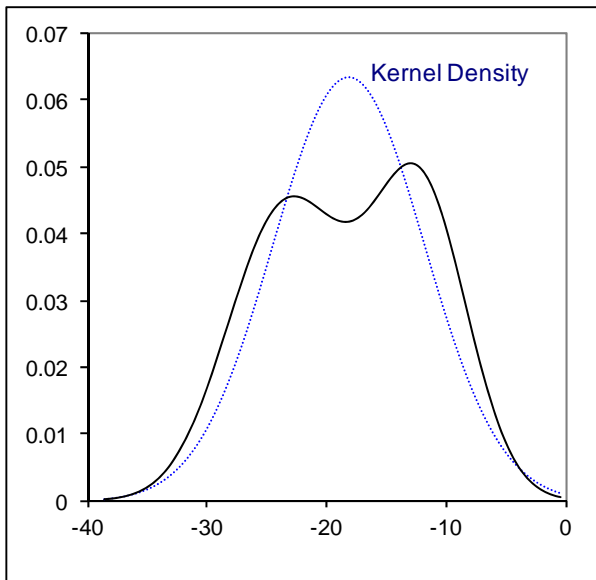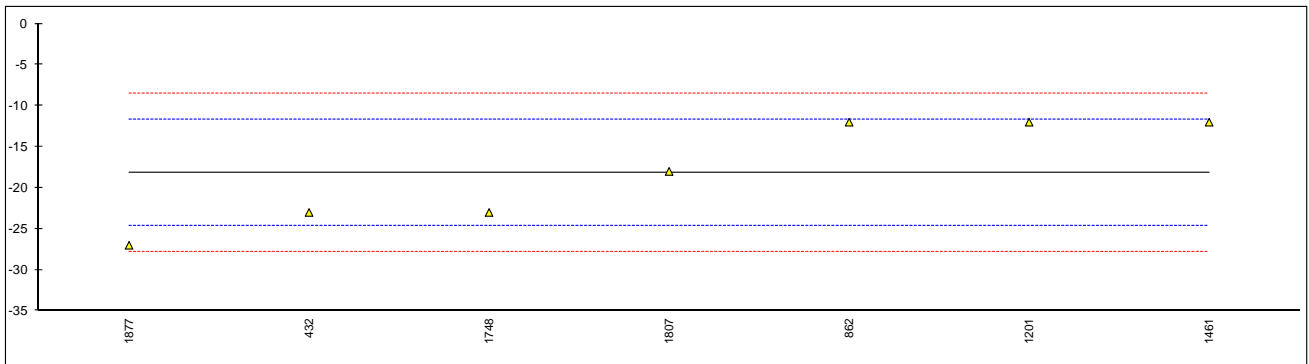
Determination of Pour Point, automated, 1°C interval on sample #15057; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	in house	-33	C,G(0.05)	-12.44	first reported as D97, manual
237		-----		-----	
255		-----		-----	
432	D5950	-15		-1.24	
473		-----		-----	
621		-----		-----	
862	D5950	-12		0.62	
1201	D5950	-12		0.62	
1461		-----		-----	
1748		-----		-----	
1807		-----		-----	
1877		-----		-----	

normality	unknown
n	3
outliers	1
mean (n)	-13.0
st.dev. (n)	1.73
R(calc.)	4.8
R(D5950:14)	4.5
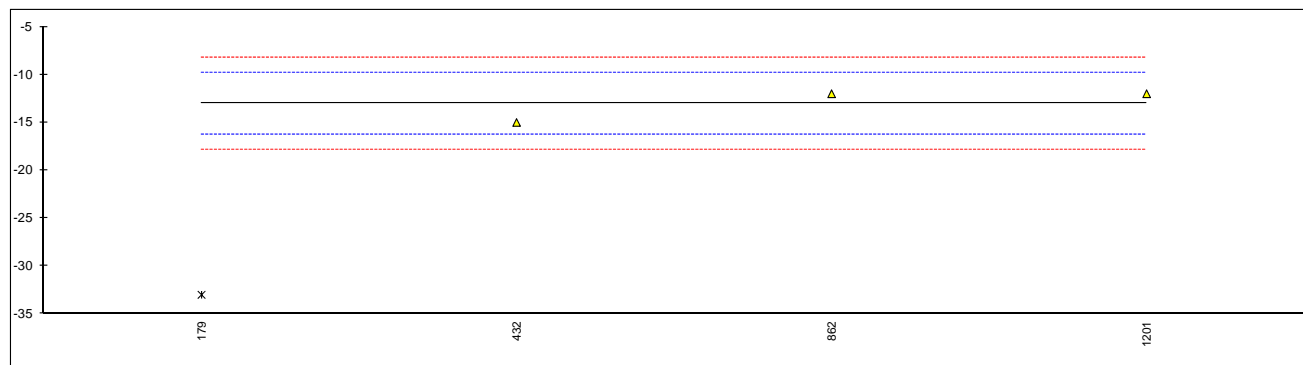
Determination of Rust prevention (procedure A) on sample #15057

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D665	Pass		-----	
237	D665	Pass		-----	
255		-----		-----	
432		-----		-----	
473		-----		-----	
621		-----		-----	
862	D665	No Rusting		-----	
1201		-----		-----	
1461		-----		-----	
1748		-----		-----	
1807		-----		-----	
1877		-----		-----	

normality	unknown
n	3
outliers	n.a.
mean (n)	Pass
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit)	unknown

Determination of Sulphur on sample #15057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4294	6953		1.71	
255		-----		-----	
432	D4951	6178		-1.85	
473		-----		-----	
621		-----		-----	
862	D2622	6610		0.14	
1201		-----		-----	
1461		-----		-----	
1748		-----		-----	
1807		-----		-----	
1877		-----		-----	

normality	unknown	
n	3	
outliers	0	
mean (n)	6580	
st.dev. (n)	388.4	
R(calc.)	1087	
R(D4951:14)	610	compare R(D4294:10) = 555 and R(D2622:10) = 491
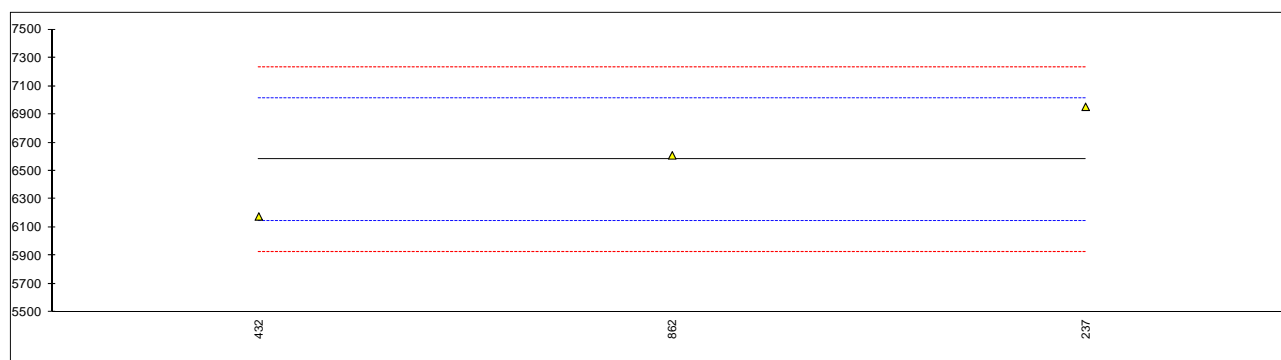
Determination of Water Separability at 82°C on sample #15057; results in minutes

lab	method	time to reach 3 ml or less emulsion	z(targ)	time to reach 37 ml of water	z(targ)	time to reach complete break (40-40-0)	time test aborted
178		-----	-----	-----	-----	-----	-----
179	D1401	-----	-----	-----	-----	15	-----
237	D1401	13.18	0.05	12.37	-0.06	16.50	-----
255		-----	-----	-----	-----	-----	-----
432	D1401	15	0.31	15	0.31	>60	60
473		-----	-----	-----	-----	-----	-----
621		-----	-----	-----	-----	-----	-----
862	D1401	11	-0.25	-----	-----	-----	15
1201		-----	-----	-----	-----	-----	-----
1461		-----	-----	-----	-----	-----	-----
1748	D1401	12	-0.11	11	-0.25	15	-----
1807		-----	-----	-----	-----	-----	-----
1877		-----	-----	-----	-----	-----	-----

normality	unknown	unknown
n	4	3
outliers	0	0
mean (n)	12.8	12.8
st.dev. (n)	1.72	2.03
R(calc.)	4.8	5.7
R(D1401:12)	20.0	20.0

Determination of Water Separability at 82°C after 15 minutes on sample #15057; results in ml

lab	method	volume oil phase	volume water phase	volume emulsion phase	reported:
178		-----	-----	-----	-----
179	D1401	40	40	0	40-40-0 (15)
237		-----	-----	-----	-----
255		-----	-----	-----	-----
432	D1401	42	38	0	42-38-0(15)
473		-----	-----	-----	-----
621		-----	-----	-----	-----
862	D1401	41	39	0	41-39-0(15min)
1201		-----	-----	-----	-----
1461		-----	-----	-----	-----
1748		-----	-----	-----	-----
1807		-----	-----	-----	-----
1877		-----	-----	-----	-----

Determination of Calcium (Ca) on sample #15057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5185	2.184		-----	
255	INH-1	<0.01		-----	Maybe unit error?
432	D5185	4.8		-----	
473	D5185	0.96		-----	
621		-----		-----	
862	D5185	<0.1		-----	
1201		-----		-----	
1461		-----		-----	
1748	D6481	0.0		-----	
1807	D5185	1.4		-----	
1877		-----		-----	

|		
normality	unknown	
n	5	
outliers	0	
mean (n)	1.87	
st.dev. (n)	1.819	
R(calc.)	5.09	
R(D5185:13e1)	n.a.	R(D5185:13e1) = 0.03 for application range 40-9000 mg/kg
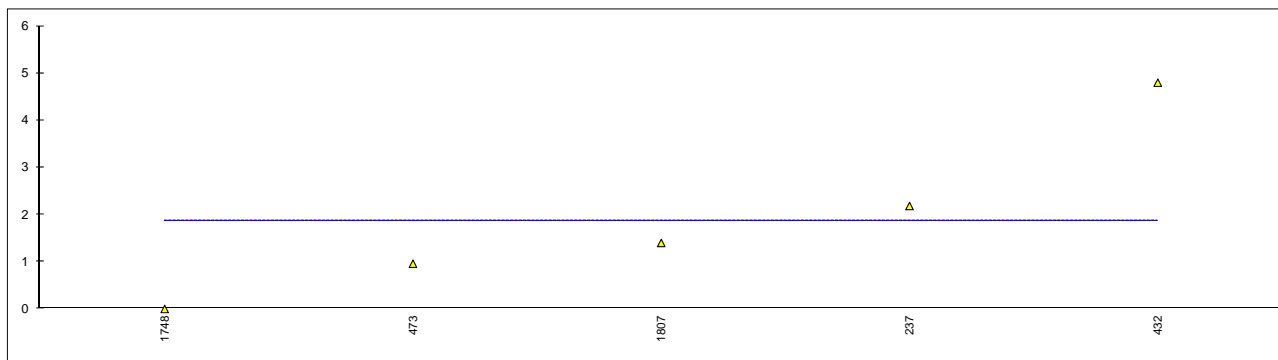
Determination of Phosphorus (P) on sample #15057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5185	51.71		-9.20	
255		-----		-----	
432	D5185	290.9		-0.12	
473	D5185	273.8	C	-0.77	first reported: 0.932
621		-----		-----	
862	D5185	269		-0.95	
1201		-----		-----	
1461		-----		-----	
1748	D6481	610		12.00	
1807	D5185	269		-0.95	
1877		-----		-----	

normality	unknown	
n	6	
outliers	0	
mean (n)	294.1	
st.dev. (n)	179.01	
R(calc.)	501.2	
R(D5185:13e1)	73.7	application range 10-1000 mg/kg
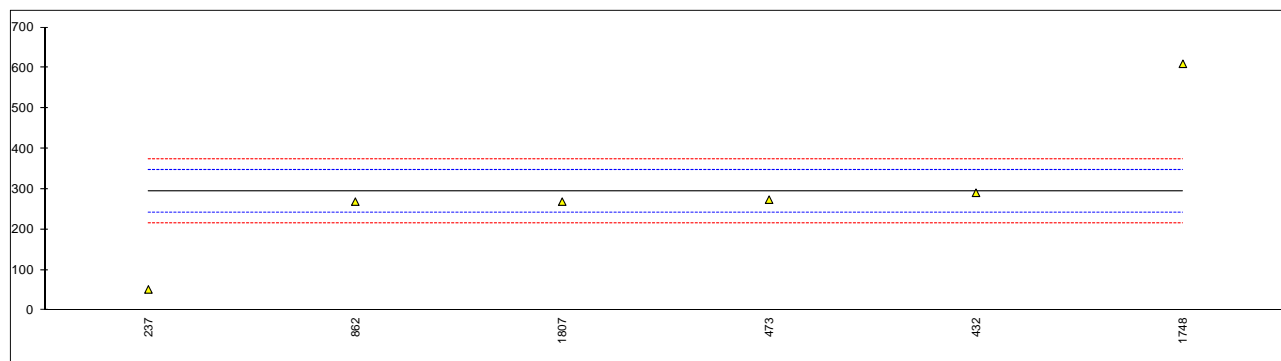
Determination of Zinc (Zn) on sample #15057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5185	2.791		-----	
255	INH-1	<0.01		-----	Maybe unit error?
432	D5185	<1		-----	
473	D5185	0.932	C	-----	first reported: 273.8
621		-----		-----	
862	D5185	0.9		-----	
1201		-----		-----	
1461		-----		-----	
1748	D6481	7		-----	
1807	D5185	0.51		-----	
1877		-----		-----	

normality	unknown
n	5
outliers	0
mean (n)	2.43
st.dev. (n)	2.706
R(calc.)	7.58
R(D5185:13e1)	n.a.

R(D5185:13e1) = 0.22 for application range 60-1600 mg/kg
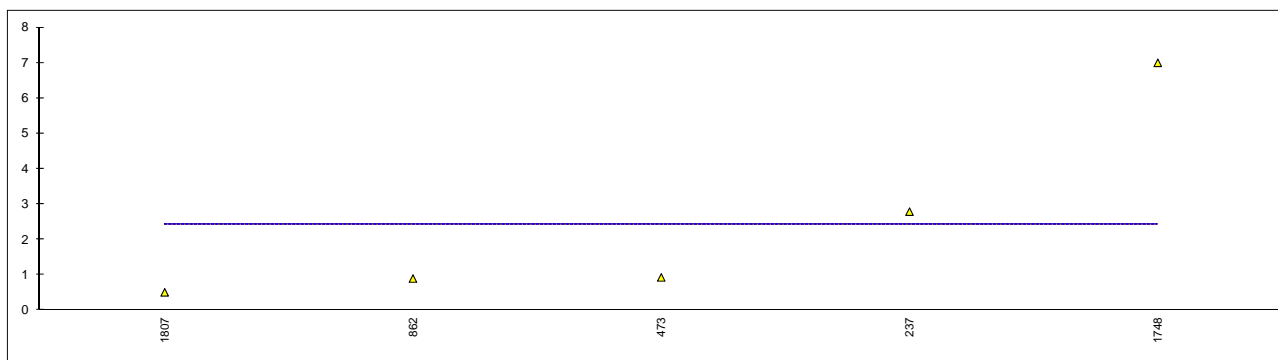
APPENDIX 2

Number of participants per country

    1 lab in AUSTRIA
    1 lab in BELGIUM
    1 lab in BULGARIA
    1 lab in CHINA, People's Republic
    1 lab in INDONESIA
    1 lab in JORDAN
    1 lab in NETHERLANDS
    1 lab in NIGERIA
    1 lab in NORWAY
    1 lab in SPAIN
    1 lab in TANZANIA
    2 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C   = final result after checking of first reported suspect 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 outlier test
R(0.05)   = straggler in Rosner outlier test
ex   = excluded from calculations
U   = reported in different unit
W   = result withdrawn on request of the participants
fr.   = first reported
S   = scope of the reported method is not applicable
n.a.   = not applicable
n.e.   = not evaluated
SDS   = Material Safety Data Sheet

Literature:

1   iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, April 2014
2   ASTM E178:08
3   ISO 5725-86
4   ISO 5725, parts 1-6, 1994
5   ISO13528:05
6   ISO17043:2010
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, First reported Z. Anal. Chem, 331, 513, (1988)
12   J.N. Miller, Analyst, 118, 455, (1993)
13   Analytical Methods Committee Technical brief, No4 January 2001.
14   The Royal Society of Chemistry 2002, Analyst 2002, 127 pages 1359-1364, P.J. Lowthian and M. Thompson (see http://www.rsc.org/suppdata/an/b2/b205600n/).
15   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)