Results of Proficiency Test
Grease
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CONTENTS

Appendices:

1 INTRODUCTION

Grease is a solid to semifluid product. It is a mixture of an oil (often mineral), a thickener (usually a metal soap) and an additive package. This formulation provides a low viscosity at application, will thin when shear is applied and will become semisolid again when the machine stops. Grease is used in machinery that cannot be lubricated by oil, because oil would drip out, water resistance while lubricating is required or when conditions are extreme in high temperature, pressure or variation of loads. Greases can also provide water resistance, for this the formation of an emulsion by the combination of oil and soap is important.

At the request of several participants, the Institute of Interlaboratory Studies decided to organise an interlaboratory study for Grease in the 2017-2018 PT program.
In this interlaboratory study 15 laboratories in 13 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2017 interlaboratory study on Grease 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 organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample of five litre of Grease in a metal can, labelled #17099. Participants were requested to report rounded and unrounded test results. The unrounded test 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 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 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 was obtained from a lubricant supplier. Two drums from one batch of multipurpose lithium grease were manually transferred to forty 5 L cans and labelled #17099. The homogeneity of the subsamples #17099 was checked by determination of Cone Penetration (worked) in accordance with ASTM D217 and Dropping Point according to ASTM D2665 on 3 stratified randomly selected samples.

	Penetration (worked) in 0.1mm	*Dropping Point in °C*
Sample #17099-1	274	200
Sample #17099-2	278	203
Sample #17099-3	274	201

Table 1: homogeneity test results of subsamples #17099

From the test results of table 1, the repeatabilities (r) 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:

	Penetration (worked) in 0.1mm	*Dropping Point in °C*
r (observed)	6	4
reference test method	D217:17	D2265:15
0.3 * R (ref. test method)	7	4

Table 2: evaluation of the repeatabilities of subsamples #17099

Each calculated repeatability was less than the corresponding repeatability of the reference test methods. Therefore, homogeneity of the subsamples #17099 was assumed.

To each of the participating laboratories one sample in a 5 litre metal can, labelled #17099, was sent on September 6, 2017. An SDS was added to the sample.

2.5 ANALYSES

The participants were asked to determine on sample #17099; Cone Penetration (unworked, worked and prolonged), Copper Corrosion 24 hrs at 100°C, Dropping Point, Extreme Pressure Properties (four-ball method), Leakage amount, Oil Separation (conical sieve), Oxidation Stability (100 hr) Pressure drop, PQ Index, Roll Stability Penetration Change (¼ and ½ scale penetrometer), Water by KF, Water Spray-Off, Water Washout at 79°C, Wear Preventative Characteristics, Metals as Lithium, Calcium, Phosphorus and Zinc.

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

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 reanalysis). 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.

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 no problems were encountered with the dispatch of the samples. Four participants reported the test results after the final reporting date and one participants did not report any test results at all. Not all laboratories were able to report all analyses requested. The 14 reporting participants sent in 92 numerical test results. Observed were 4 outlying test results, which is 4.3% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The different test methods that are reported by the laboratories are taken into account for explaining the observed differences when possible and applicable. These test methods are also mentioned in the tables in appendix 1 together with the original data. The abbreviations used in these tables are listed in appendix 3.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D2266) and an added designation for the year that the method was adopted or revised (e.g. D2266:01). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2266:01(2015)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D2266:01 will be used.

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.

Only one or two results for reported for the determination of Leakage amount, Oxidation stability, Roll Stability – Penetration change ¼ scale penetrometer and ½ scale penetrometer and Water Spray-off. These tests were not evaluated. The reported TEST results are summarized in Appendix 2.

Cone Penetration unworked: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier was in agreement with the requirements of ASTM D217:17.

Cone Penetration worked: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier was in full agreement with the requirements of ASTM D217:17.

Cone Penetration prolonged: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility was in good agreement with the requirements of ASTM D217:17.

Copper Corrosion: This determination was not problematic. All reporting participants agreed on a test result of 1 (1a/1b).

Dropping Point: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D2265:15 or with the less strict requirements of ASTM D566:17 or IP396:14.

Extreme Pressure Properties – Weld Point: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D2596:15.

Extreme Pressure Properties – Load Wear Index: Only one participant reported a test result. No evaluation was done.

Extreme Pressure Properties – Last Non-Seizure Load: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility after statistical outliers is in good agreement with the requirements of ASTM D2596:15.

Oil Separation: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6184:17.

PQ Index: Four laboratories reported a test result, one was excluded for reporting a zero (zero is not a real value). Since there is no known method for PQ Index, no target reproducibility is known. All test results showed that the PQ index is low (which is to be expected on a fresh grease).

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

Water washout: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1264:16.

Wear Preventative Characteristics: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D2266:01(2015).

Lithium: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D7303:17.

Calcium: 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 D7303:17.

Phosphorus: All reported test results are below the application range (50 – 2000 mg/kg) of ASTM D7303:17. Therefore no significant conclusions were drawn.

Zinc: All reported test results are below the application range (300– 2200 mg/kg) of ASTM D7303:17. Therefore no significant 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 reference test method and the reproducibility as found for the group of participating laboratories. The target reproducibilities derived from literature reference test methods (R (lit)) and the calculated reproducibilities (2.8 * sd) are compared in the next table;

Parameter	unit	n	mean	2.8 * sd	R (lit)
Cone Penetration - unworked	0.1 mm	10	270.8	23.1	22
Cone Penetration - worked	0.1 mm	10	274.7	21.9	23
Cone Penetration – prolonged	0.1 mm	5	320.8	25.4	29
Copper Corrosion 24 hrs at 100°C		6	1	n.a.	n.a.
Dropping Point	°C	9	194.3	17.5	12
Extreme-Pressure Properties (four-ball method)					
- Weld Point	kgf	3	160	0	1
- Load Wear Index	kgf	1	n.a.	n.a.	n.a.
- Last Non-Seizure Load	kgf	3	52	27	41
Leakage amount	g	1	n.a.	n.a.	n.a.
Oil separation – Conical Sieve	%M/M	3	2.2	1.6	2.2
Oxidation stability - pressure drop	kPa	1	n.a.	n.a.	n.a.
PQ index	rating	3	5	3	n.a.
Roll Stability – Penetration change					
- ¼ scale penetrometer	0.1 mm	2	n.a.	n.a.	n.a.
- ½ scale penetrometer	0.1 mm	1	n.a.	n.a.	n.a.
Water by KF	mg/kg	6	2390	1830	1798
Water Spray-off	%M/M	0	n.a.	n.a.	n.a.
Water Washout at 79°C	%M/M	3	11.60	5.17	17.82
Wear Preventative Characteristics	mm	3	0.55	0.06	0.37
Lithium as Li	mg/kg	4	2397	565	465
Calcium as Ca	mg/kg	4	38	56	40
Phosphorus as P	mg/kg	6	<10	n.a.	n.a.
Zinc as Zn	mg/kg	5	<10	n.a.	n.a.

Table 3: reproducibilities of tests on sample #17099

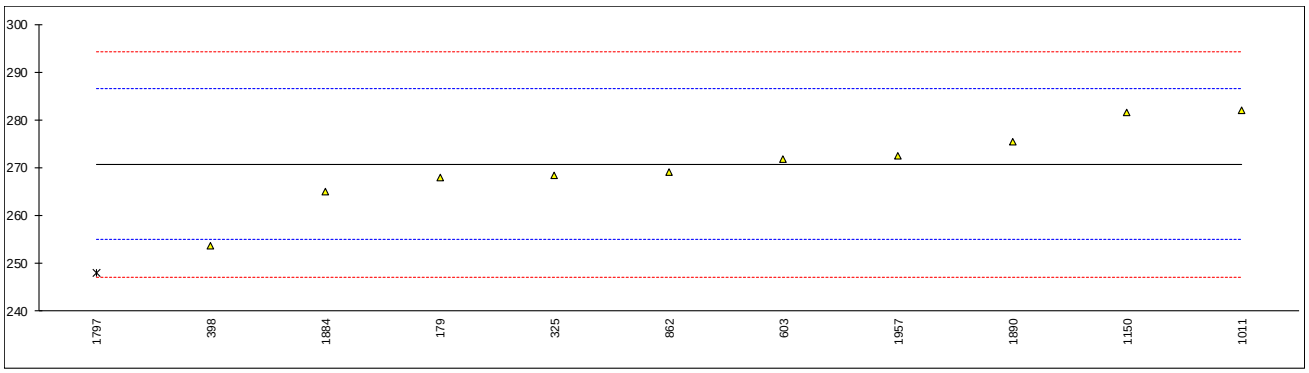
Without further statistical calculations, it could be concluded that for many tests there is a good compliance of the group of participating laboratories with the relevant reference test methods. Unfortunately, not all laboratories performed all tests, resulting in a low number of results for several tests. Hopefully in the next PT more test results will be reported.

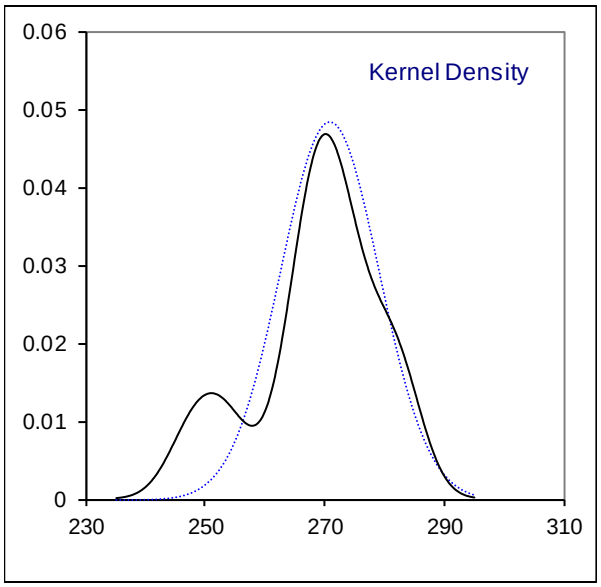
APPENDIX 1

Determination of Cone Penetration - unworked on sample #17099; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
179	D217	268		-0.35	
237		-----		-----	
325	D217	268.5		-0.29	
349		-----		-----	
398	D217	253.7		-2.17	
603	D217	271.9		0.14	
862	D217	269		-0.23	
962		-----		-----	
1011	D217	282		1.43	
1150	ISO2137	281.6		1.38	
1797	D217	248	C,G(0.05)	-2.90	first reported: 250
1884	D217	265.0		-0.73	
1890	ISO2137	275.4		0.59	
1957	D217	272.6		0.23	
6125		-----		-----	

normality	suspect
n	10
outliers	1
mean (n)	270.77
st.dev. (n)	8.233
R(calc.)	23.05
st.dev.(D217:17)	7.857
R(D217:17)	22

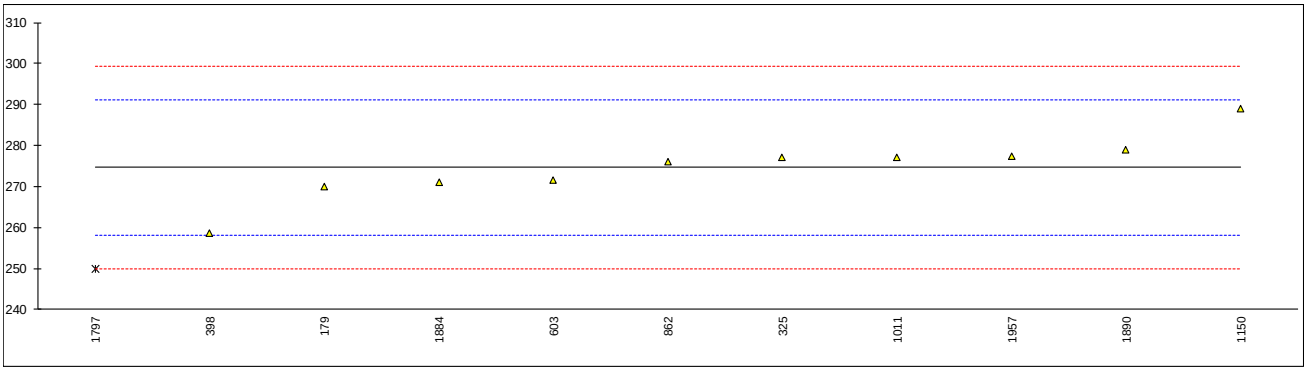


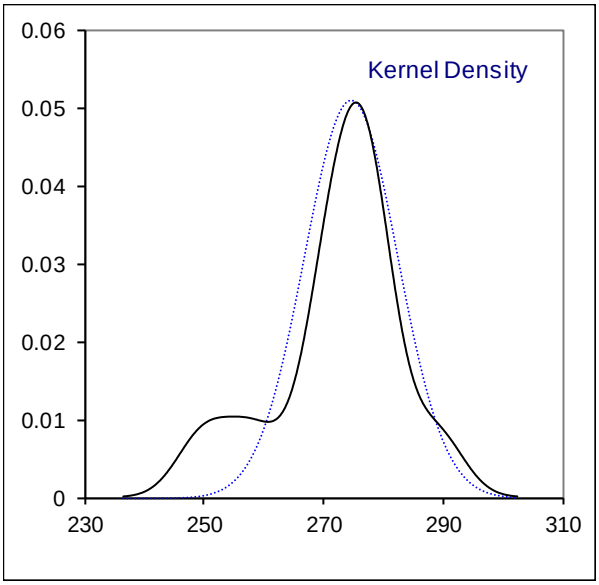


Determination of Cone Penetration - worked on sample #17099; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
179	D217	270		-0.57	
237		-----		-----	
325	D217	277		0.29	
349		-----		-----	
398	D217	258.5		-1.97	
603	D217	271.5		-0.38	
862	D217	276		0.16	
962		-----		-----	
1011	D217	277		0.29	
1150	ISO2137	289		1.75	
1797	D217	250	C,G(0.05)	-3.00	
1884	D217	271.0		-0.44	
1890	ISO2137	279.0		0.53	
1957	D217	277.5		0.35	
6125		-----		-----	

normality	not OK
n	10
outliers	1
mean (n)	274.65
st.dev. (n)	7.828
R(calc.)	21.92
st.dev.(D217:17)	8.214
R(D217:17)	23

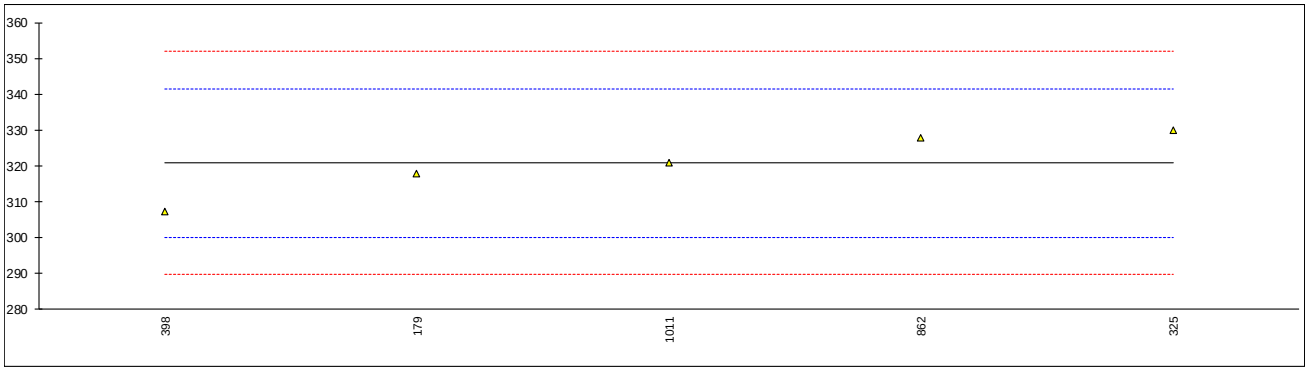




Determination of Cone Penetration – prolonged work on sample #17099; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
179	D217	318		-0.27	
237		-----		-----	
325	D217	330		0.88	
349		-----		-----	
398	D217	307.2		-1.32	
603		-----		-----	
862	D217	328		0.69	
962		-----		-----	
1011	D217	321		0.02	
1150		-----		-----	
1797		-----		-----	
1884		-----		-----	
1890		-----		-----	
1957		-----		-----	
6125		-----		-----	

normality	unknown
n	5
outliers	0
mean (n)	320.84
st.dev. (n)	9.073
R(calc.)	25.41
st.dev.(D217:17)	10.357
R(D217:17)	29

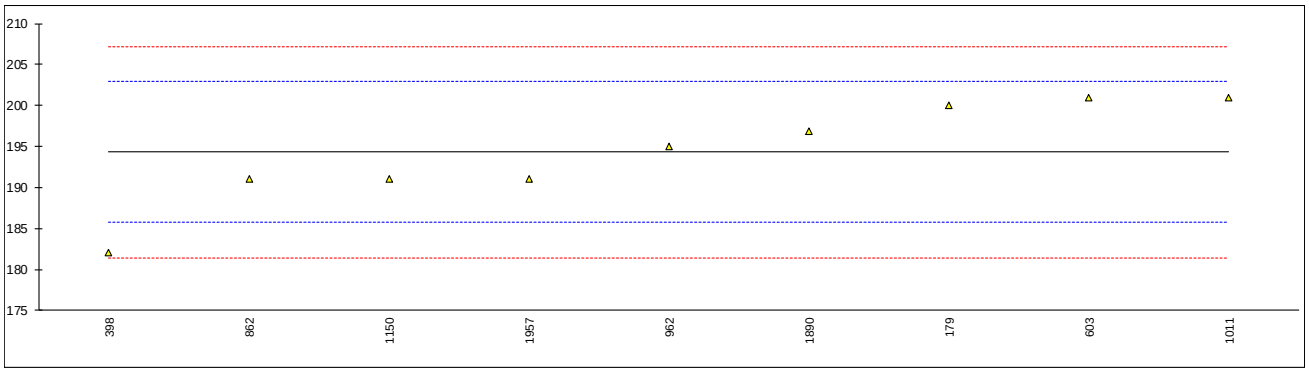


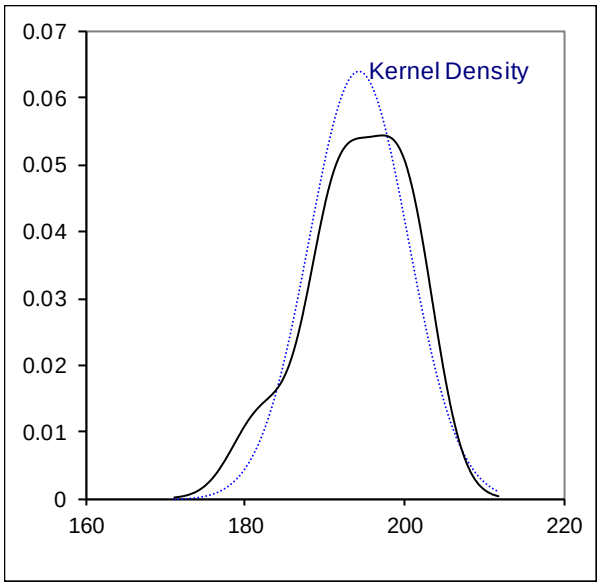
Determination of Copper Corrosion 24 hrs at 100°C on sample #17099;

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
237		-----		-----	
325	DIN51811	1A_slight		-----	
349		-----		-----	
398		-----		-----	
603		-----		-----	
862	D4048	1a		-----	
962		-----		-----	
1011	D4048	1a		-----	
1150	D4048	1b		-----	
1797	ISO2160	1b		-----	
1884		-----		-----	
1890		-----		-----	
1957	D4048	1a		-----	
6125		-----		-----	
	n	6			
	mean (n)	1			

Determination of Dropping Point on sample #17099; results in °C

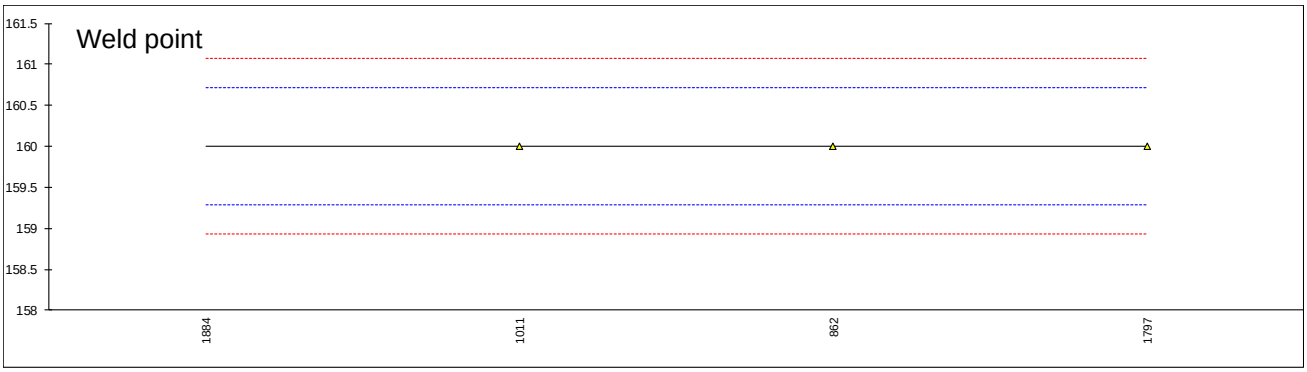
lab	method	value	mark	z(targ)	remarks
179	D2265	200.0		1.33	
237		-----		-----	
325	INH-396/566	>300		>24.66	possible false positive?
349		-----		-----	
398	D2265	182		-2.87	
603	D2265	200.9		1.54	
862	D2265	191		-0.77	
962	D566	195		0.16	
1011	D2265	201		1.56	
1150	ISO2176	191		-0.77	
1797		-----		-----	
1884		-----		-----	
1890	IP396	196.9		0.60	
1957	D566	191		-0.77	
6125		-----		-----	
	normality	OK			
	n	9			
	outliers	0			
	mean (n)	194.31			
	st.dev. (n)	6.246			
	R(calc.)	17.49			
	st.dev.(D2265:15)	4.286			
	R(D2265:15)	12			Compare R(D566:17) = 13°C and R(IP396:14) = 14.48°C

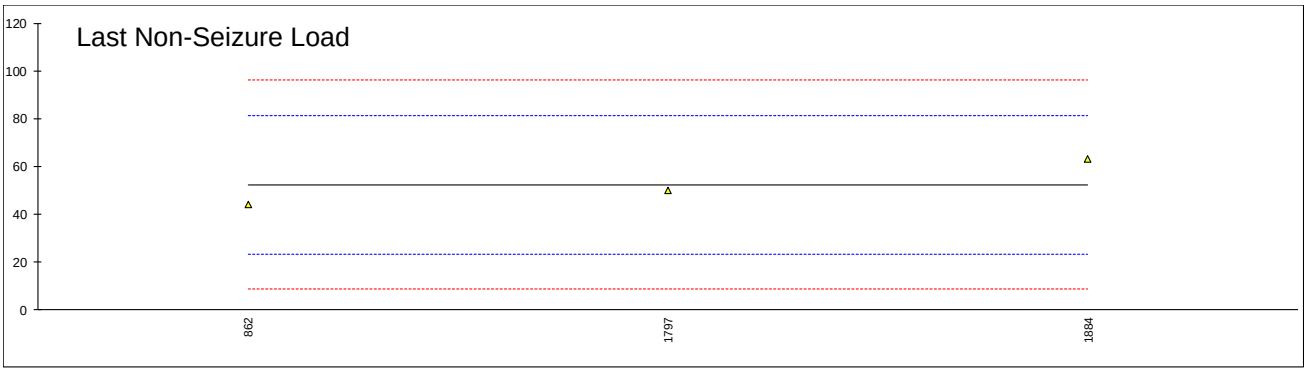




Determination of Extreme-Pressure Properties (four-ball method) on sample #17099; Weld Point, Load Wear Index and Last Non-Seizure Load results in kgf

lab	method	Weld Point	mark	z(targ)	LWI	mark	z(targ)	Last Non-Seizure Load	mark	z(targ)
179		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
398		-----		-----	-----		-----	-----		-----
603		-----		-----	-----		-----	-----		-----
862	D2596	160		0.00	21		-----	44		-0.57
962		-----		-----	-----		-----	-----		-----
1011	D2596	160		0.00	-----		-----	-----		-----
1150		-----		-----	-----		-----	-----		-----
1797	D2596	160		0.00	-----		-----	50		-0.16
1884	D2596	126	G(0.01)	-95.20	-----		-----	63		0.73
1890		-----		-----	-----		-----	-----		-----
1957		-----		-----	-----		-----	-----		-----
6125		-----		-----	-----		-----	-----		-----
	normality	unknown			n.a.			unknown		
	n	3			1			3		
	outliers	1			n.a.			0		
	mean (n)	160.0			n.a.			52.3		
	st.dev. (n)	0.00			n.a.			9.71		
	R(calc.)	0.0			n.a.			27.2		
	st.dev.(D2596:15)	0.36			n.a.			14.58		
	R(D2596:15)	1			n.a.			40.8		

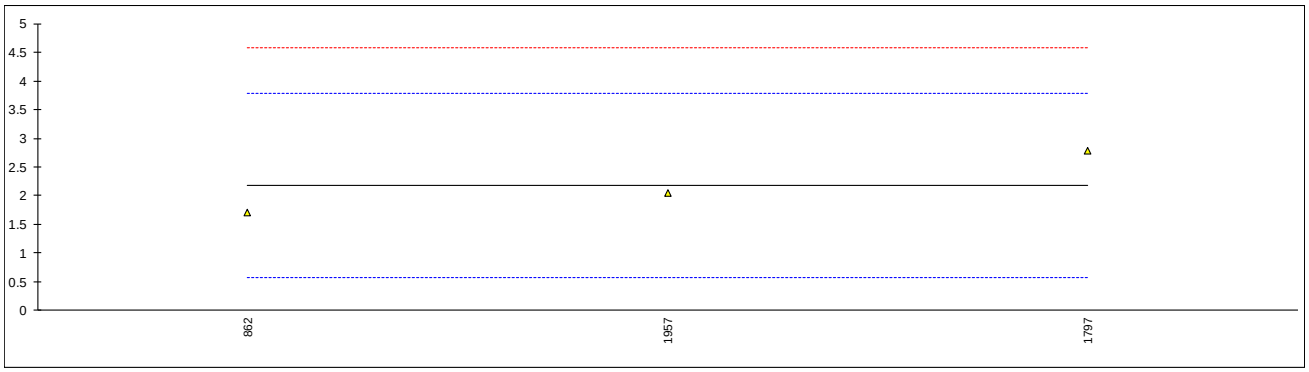




Determination of Oil separation – Conical Sieve on sample #17099; results in %M/M

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
237		-----		-----	
325		-----		-----	
349		-----		-----	
398		-----		-----	
603		-----		-----	
862	D6184	1.7		-0.60	
962		-----		-----	
1011		-----		-----	
1150		-----		-----	
1797	D6184	2.79		0.76	
1884		-----		-----	
1890		-----		-----	
1957	D6184	2.05		-0.16	
6125		-----		-----	

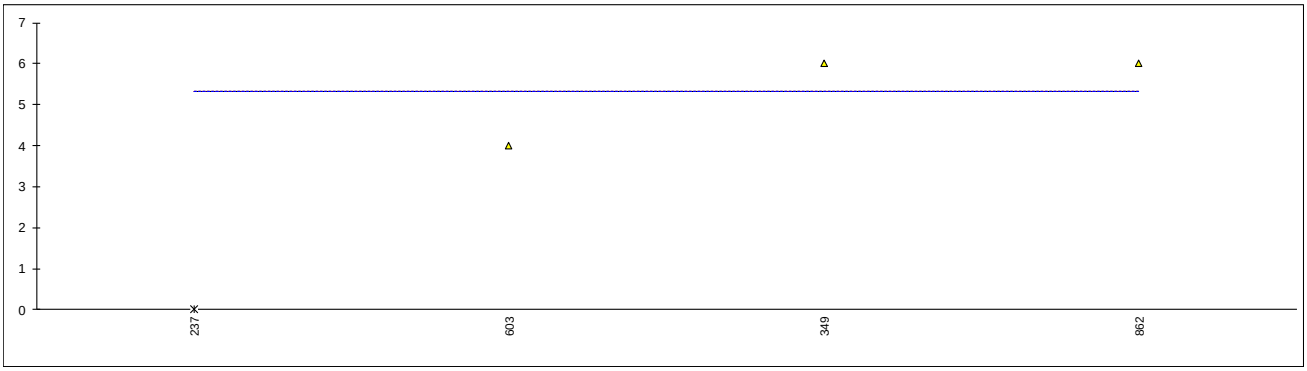
normality	unknown
n	3
outliers	0
mean (n)	2.18
st.dev. (n)	0.557
R(calc.)	1.56
st.dev.(D6184:17)	0.800
R(D6184:17)	2.24



Determination of PQ index on sample #17099; rating

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
237	D7690	0	ex	-----	excluded for zero is not a real value
325		-----		-----	
349		6		-----	
398		-----		-----	
603		4		-----	
862	In house	6		-----	
962		-----		-----	
1011		-----		-----	
1150		-----		-----	
1797		-----		-----	
1884		-----		-----	
1890		-----		-----	
1957		-----		-----	
6125		-----		-----	

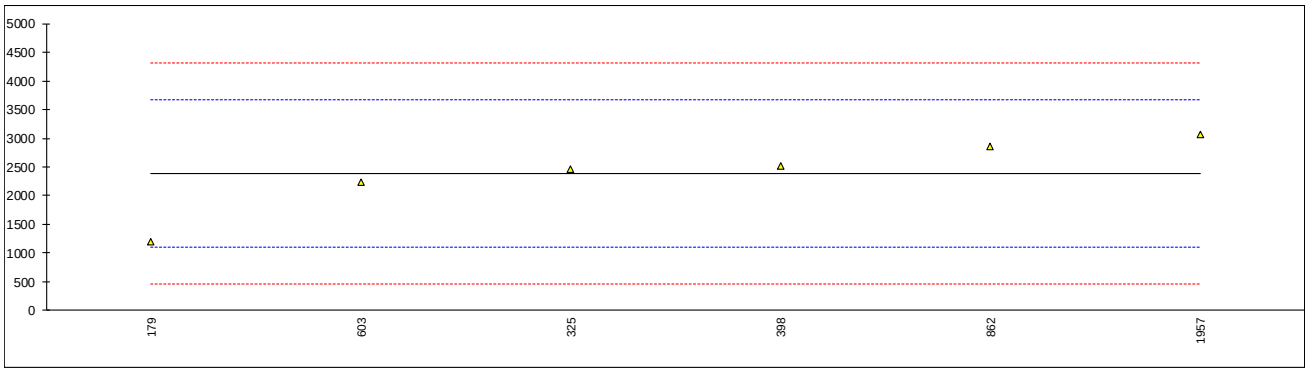
normality	unknown
n	3
outliers	0 (+1ex)
mean (n)	5.3
st.dev. (n)	1.15
R(calc.)	3.2
st.dev.(lit)	unknown
R(lit)	unknown

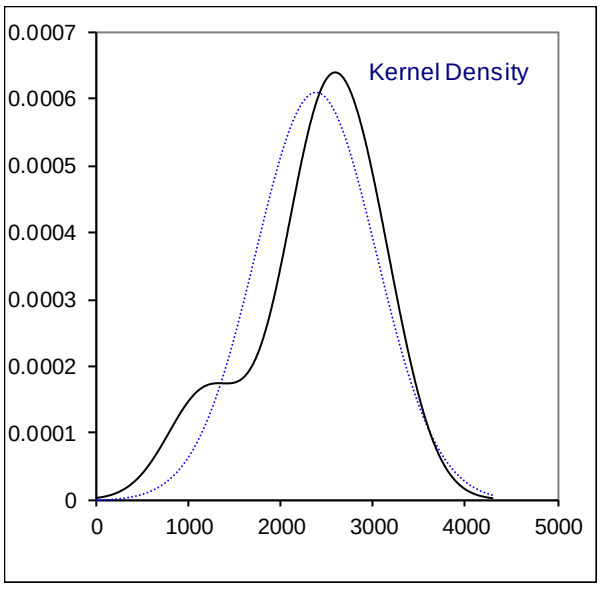


Determination of Water by KF on sample #17099; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D6304-C	1200		-1.85	
237		-----		-----	
325	D6304-C	2469		0.12	
349		-----		-----	
398	D6304-C	2513		0.19	
603	D6304-C	2236		-0.24	
862	D6304-C	2853		0.72	
962		-----		-----	
1011		-----		-----	
1150		-----		-----	
1797		-----		-----	
1884		-----		-----	
1890		-----		-----	
1957	D6304-C	3068		1.06	
6125		-----		-----	

normality	unknown
n	6
outliers	0
mean (n)	2389.8
st.dev. (n)	653.49
R(calc.)	1829.8
st.dev.(D6304:16e1)	642.00
R(D6304:16e1)	1797.6

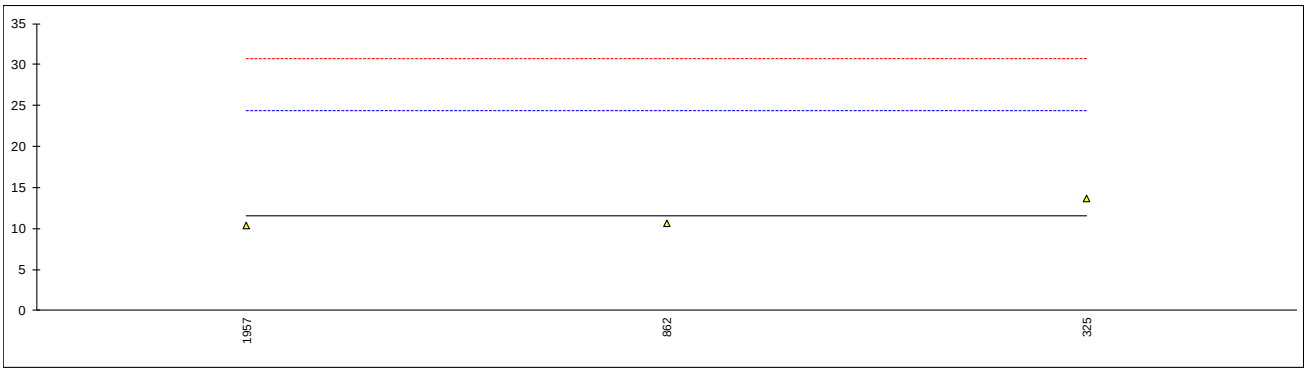




Determination of Water Washout at 79°C on sample #17099; results in %M/M

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
237		-----		-----	
325	D1264	13.72		0.33	
349		-----		-----	
398		-----		-----	
603		-----		-----	
862	D1264	10.68		-0.14	
962		-----		-----	
1011		-----		-----	
1150		-----		-----	
1797		-----		-----	
1884		-----		-----	
1890		-----		-----	
1957	D1264	10.39		-0.19	
6125		-----		-----	

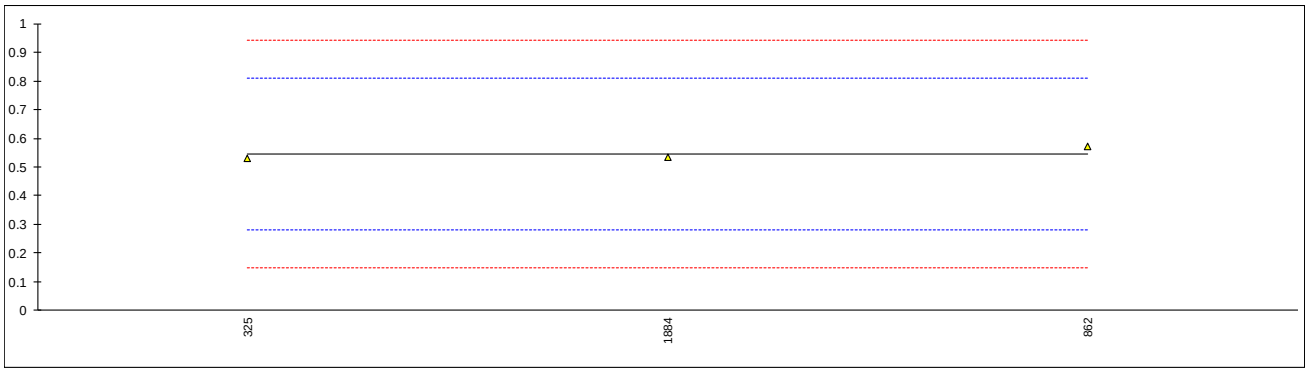
normality	unknown
n	3
outliers	0
mean (n)	11.597
st.dev. (n)	1.8446
R(calc.)	5.165
st.dev.(D1264:16)	6.3630
R(D1264:16)	17.816



Determination of Wear Preventative Characteristics on sample #17099; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
237		-----		-----	
325	D2266	0.53		-0.11	
349		-----		-----	
398		-----		-----	
603		-----		-----	
862	D2266	0.57		0.19	
962		-----		-----	
1011		-----		-----	
1150		-----		-----	
1797		-----		-----	
1884	D2266	0.534		-0.08	
1890		-----		-----	
1957		-----		-----	
6125		-----		-----	

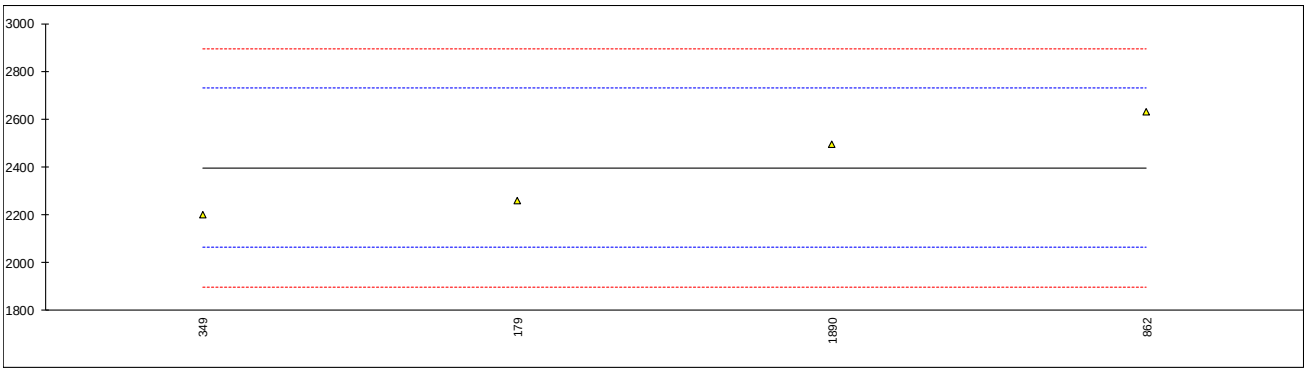
normality	unknown
n	3
outliers	0
mean (n)	0.545
st.dev. (n)	0.0220
R(calc.)	0.062
st.dev.(D2266:01)	0.1321
R(D2266:01)	0.37



Determination of Lithium as Li on sample #17099; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
179	D5185	2259		-0.83	
237		-----		-----	
325		-----		-----	
349		2200		-1.18	
398		-----		-----	
603		-----		-----	
862	D7303	2630		1.41	
962		-----		-----	
1011		-----		-----	
1150		-----		-----	
1797		-----		-----	
1884		-----		-----	
1890		2497		0.60	
1957		-----		-----	
6125		-----		-----	

normality	unknown
n	4
outliers	0
mean (n)	2396.5
st.dev. (n)	201.78
R(calc.)	565.0
st.dev.(D7303:17)	166.14
R(D7303:17)	465.2

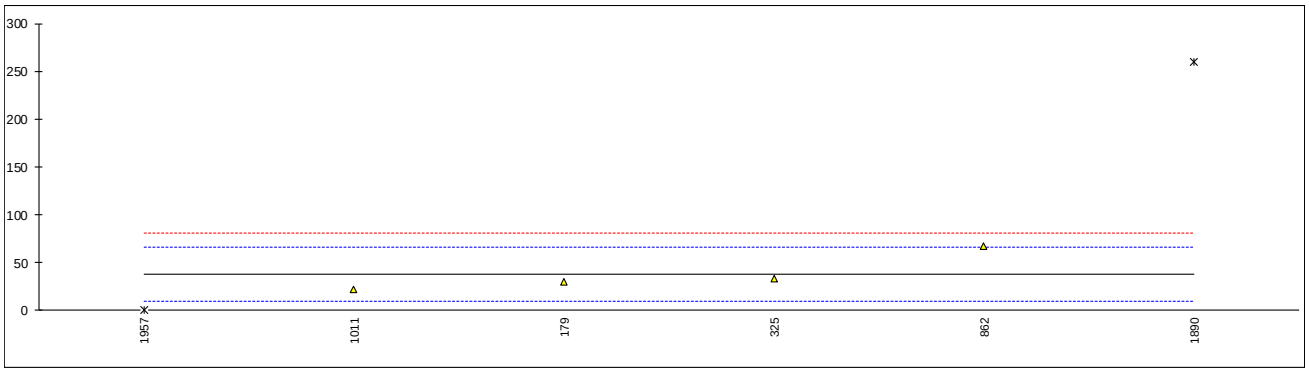


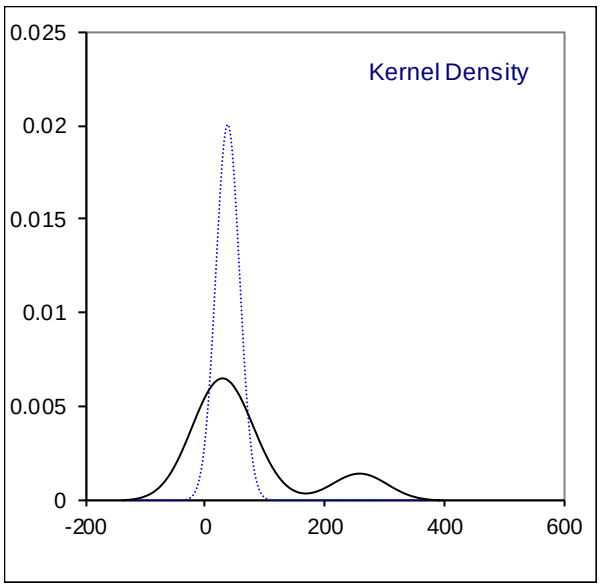
Determination of Calcium as Ca on sample #17099; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D5185	30		-0.56	
237		-----		-----	
325	In house	33		-0.35	
349		<4		-----	
398		-----		-----	
603		-----		-----	
862	D7303	67		2.03	
962		-----		-----	
1011	D5185	22		-1.12	
1150		-----		-----	
1797		-----		-----	
1884		-----		-----	
1890		260	C,G(0.05)	15.55	first reported: 116
1957		0	ex	-2.66	excluded for zero is not a real value
6125		-----		-----	

	normality	unknown
	n	4
	outliers	1 (+1ex)
	mean (n)	38.0
	st.dev. (n)	19.88
	R(calc.)	55.7
	st.dev.(D7303:17)	14.28
	R(D7303:17)	40.0

application range D7303: 20 – 50000 mg/kg





Determination of Phosphorus as P on sample #17099; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
179	D5185	3		-----	
237		-----		-----	
325	In house	<10		-----	
349		<4		-----	
398		-----		-----	
603		-----		-----	
862	D7303	<50		-----	
962		-----		-----	
1011	D5185	1		-----	
1150		-----		-----	
1797		-----		-----	
1884		-----		-----	
1890		1		-----	
1957		9		-----	
6125		-----		-----	
	n	6			
	mean (n)	<10			application range D7303: 50 – 2000 mg/kg

Determination of Zinc as Zn on sample #17099; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
179	D5185	3		-----	
237		-----		-----	
325	In house	<10		-----	
349		<4		-----	
398		-----		-----	
603		-----		-----	
862	D7303	<50		-----	
962		-----		-----	
1011	D5185	0		-----	
1150		-----		-----	
1797		-----		-----	
1884		-----		-----	
1890		32		-----	
1957		5		-----	
6125		-----		-----	
	n	5			
	mean (n)	<10			application range D7303: 300 – 2200 mg/kg

Appendix 2: Determinations with less than 3 reported results

Determination of Leakage amount (g), Oxidation stability (100 hr), Roll Stability – Penetration change ¼ scale penetrometer and ½ scale penetrometer (0.1 mm) and Water Spray-off (%M/M) on sample #17099

lab	Leakage amount	Oxidation Stab.	Roll stability ¼	Roll stability ½	Water Spray
179	-----	-----	-----	-----	-----
237	-----	-----	-----	-----	-----
325	-----	-----	-----	148.8	-----
349	-----	-----	-----	-----	-----
398	-----	-----	-----	-----	-----
603	-----	-----	-----	-----	-----
862	-----	26.0	35	-----	-----
962	-----	-----	-----	-----	-----
1011	4.4	-----	41	-----	-----
1150	-----	-----	-----	-----	-----
1797	-----	-----	-----	-----	-----
1884	-----	-----	-----	-----	-----
1890	-----	-----	-----	-----	-----
1957	-----	-----	-----	-----	-----
6125	-----	-----	-----	-----	-----

APPENDIX 3

Number of participants per country

2 labs in	BELGIUM
1 lab in	BULGARIA
1 lab in	CHINA, People's Republic
1 lab in	GEORGIA
1 lab in	ITALY
2 labs in	MALAYSIA
1 lab in	NIGERIA
1 lab in	PORTUGAL
1 lab in	ROMANIA
1 lab in	SAUDI ARABIA
1 lab in	SPAIN
1 lab in	U.S.A.
1 lab in	UNITED STATES OF AMERICA

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	= 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
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, March 2017
2 ASTM E178:16
3 ASTM E1301:95(2003)
4 ISO 5725:86
5 ISO 5725, parts 1-6, 1994
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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)
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13 Analytical Methods Committee Technical Brief, No 4 January 2001
14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)