Results of Proficiency Test
Grease
September 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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.

Since 2017, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Grease every year. During the annual proficiency testing program 2019/2020, it was decided to continue the round robin for the analyzis of Grease according to the scope of the latest specification of ASTM D4950. In this interlaboratory study 15 laboratories in 12 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2019 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 organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one Grease sample of five kilograms labelled #19170. 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/IEC17043:2010. 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 organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). 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

A batch of grease samples of 5 kg was obtained from a third-party laboratory and labelled #19170. The homogeneity of the subsamples #19170 was checked by determination of Cone Penetration-worked in accordance with ASTMD217 on 3 stratified randomly selected samples.

	Cone Penetration-worked in 0.1mm
Sample #19170-1	274
Sample #19170-2	278
Sample #19170-3	274

Table 1: homogeneity test results of subsamples #19170

From the test results of table 1, the repeatability (r) was calculated and compared with 0.3 times the corresponding reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Cone Penetration-worked in 0.1mm
r (observed)	6.5
reference test method	ASTM D217:19a
0.3 * R (ref. test method)	6.9

Table 2: evaluation of the repeatability of subsamples #19170

The calculated repeatability was in agreement with the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #19170 was assumed.

To each of the participating laboratories one container of 5 kg, labelled #19170, was sent on Augustus 28, 2019. An SDS was added to the sample package.

2.5 ANALYZES

The participants were asked to determine on sample #19170: Cone Penetration (unworked, worked and prolonged), Copper Corrosion 24 hrs at 100°C, Dropping Point, Extreme Pressure Properties (Four-ball method), Oil Separation-Conical Sieve, Roll Stability-Penetration Change (¼ and ½ Scale Penetrometer), Water, Wear Preventive Characteristics, Elements Aluminum, Antimony, Barium, Calcium, Iron, Lithium, Magnesium, Molybdenum, Phosphorus, Silicon, Sodium, Sulfur and Zinc.

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

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 or 2 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 reanalyzes). Additional or corrected test results are used for data analyzis and original test results are placed under 'Remarks' in the test result tables in appendix 1 or 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

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

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation 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 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. One participant reported the test results after the final reporting date and two participants did not report any test results at all. Not all laboratories were able to report all analyzes requested. In total 13 participants reported 113 numerical test results. Observed were 4 outlying test results, which is 3.5% 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 “unknown”. 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 test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the reported test results. The abbreviations, used in these tables, are listed in appendix 4.

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. 2266:01(2015). In the results tables of appendix 1 only the method number and year of adoption or revision e.g. D2266:01 is used.

Cone Penetration-unworked: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D217:19a.

Cone Penetration-worked: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D217:19a.

Cone Penetration-prolonged: 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 D217:19a.

Copper Corrosion: This determination was not problematic. Nine participants agreed on a test result of 1a or 1b.

Dropping Point: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2265:19.

Weld Point: This determination may be very problematic. One statistical outlier was observed. The reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D2596:15.

Load Wear Index: No z-scores were calculated due to the low number of test results.

Last Non-Seizure Load: This determination may be very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D2596:15.

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

¼ and ½ Scale Penetrometer: These determinations were not problematic. No statistical outliers were observed. The calculated reproducibilities are in agreement with the requirements of ASTM D1813:19.

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

Wear Preventive Characteristics: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D2266:01(2015).

With respect to the elemental analyzes; test method ASTM D7303:17 states in section 5.2: “Although widely used in other sectors of the oil industry for metal analysis, ICP-AES based Test Methods D4951 or D5185 cannot be used for analyzing greases because of their insolubility in organic solvents used in these test methods. Hence, grease samples need to be brought into aqueous solution by acid decomposition before ICP-AES measurements.” Therefore, it was decided to exclude test results from test method ASTM D5185.

Calcium: This determination was problematic. No statistical outliers were observed. Two test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D7303:17.

Lithium: No z-scores were calculated due to the low number of test results.

Sodium: This determination was not problematic. One statistical outlier was observed. Two other test results were excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D7303:17.

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

For Aluminum, Antimony, Barium, Iron, Magnesium, Molybdenum, Phosphorus, Silicon and Zinc the majority of the participants agreed on concentrations near or below the limit of detections, see appendix 2. Therefore, no z-scores were calculated for these determinations.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the reproducibility derived from literature reference test methods (in casu ASTM reference test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Cone Penetration-unworked	0.1mm	12	271	23	22
Cone Penetration-worked	0.1mm	11	277	13	23
Cone Penetration-prolonged work	0.1mm	7	315	58	29
Copper Corrosion 24 hrs at 100°C		9	1a/1b	n.a.	n.a.
Dropping Point	°C	11	193	9	12
Weld Point	kgf	4	151	53	20
Load Wear Index	kgf	2	(18.3)	(13.9)	(8.0)
Last Non-Seizure Load	kgf	4	73	138	57
Oil Separation-Conical Sieve	%M/M	6	2.5	3.5	2.4
¼ Scale Penetrometer	0.1mm	3	33	20	45
½ Scale Penetrometer	0.1mm	3	25	27	45
Water	mg/kg	6	1973	2546	1602
Wear Preventive Characteristics	mm	5	0.56	0.39	0.37
Calcium as Ca	mg/kg	5	44	71	44
Lithium as Li	mg/kg	2	(2896)	(944)	(566)
Sodium as Na	mg/kg	3	586	97	280
Sulfur as S	mg/kg	4	4590	3593	2197

Table 3: reproducibilities of tests on sample #19170
Results between brackets is based on a low number of data.

Without further statistical calculations it can be concluded that for most tests there is not a good compliance of the group of participating laboratories with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2019 WITH PREVIOUS PTS

	September 2019	September 2018	September 2017
Number of reporting laboratories	13	17	14
Number of test results	113	124	92
Number of statistical outliers	4	1	4
Percentage outliers	3.5%	0.8%	4.3%

Table 4: comparison with previous proficiency tests

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

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

Parameter	September 2019	September 2018	September 2017
Cone Penetration-unworked	+/-	+	+/-
Cone Penetration-worked	+	+	+/-
Cone Penetration-prolonged work	--	+	+
Dropping Point	+	--	-
Weld Point	--	+/-	+
Load Wear Index	(-)	+	n.e.
Last Non-Seizure Load	--	++	+
Oil Separation-Conical Sieve	-	+/-	+
¼ Scale Penetrometer	++	n.e.	n.e.
½ Scale Penetrometer	+	n.e.	n.e.
Water	-	+/-	+/-
Wear Preventive Characteristics	+/-	++	++
Calcium as Ca	-	+	-
Lithium as Li	(-)	-	-
Sodium as Na	++	n.e.	n.a.
Phosphorus as P	n.e.	+	n.e.
Sulfur as S	-	-	n.e.
Zinc as Zn	n.e.	--	n.e.

Table 5: comparison determinations against the reference test method

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

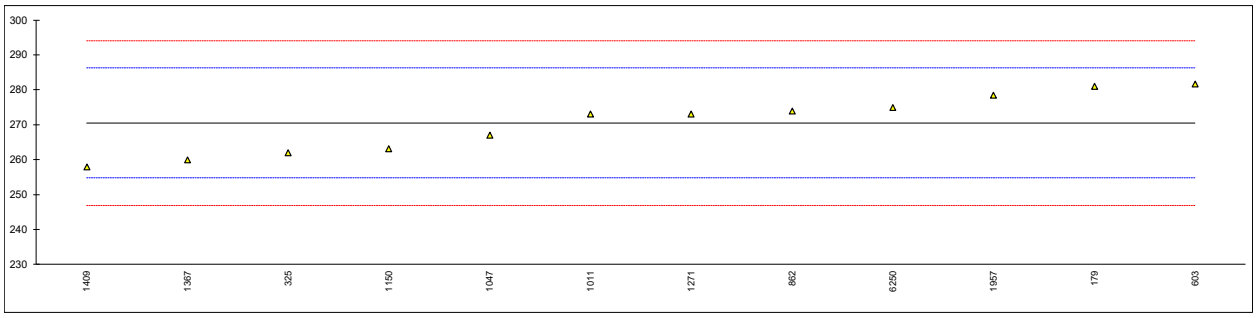
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test method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

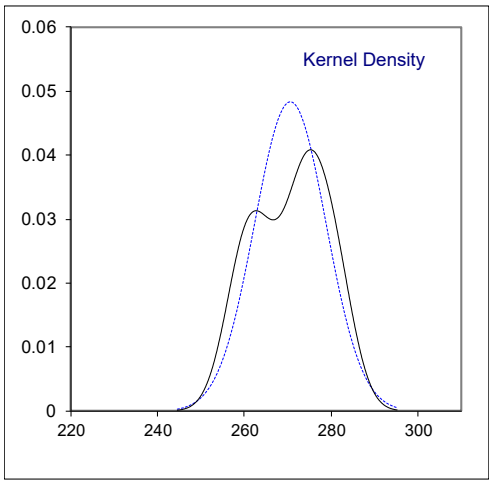
APPENDIX 1

Determination of Cone Penetration - unworked on sample #19170; results in 0.1mm

lab	method	value	mark	z(targ)	remarks
179	D217	281		1.33	
325	D217	262		-1.08	
349		-----		-----	
398		-----		-----	
603	D217	281.6		1.41	
862	D217	274		0.44	
1011	D217	273		0.32	
1047	ISO2137	267		-0.45	
1150	ISO2137	263.2		-0.93	
1271	D217	273		0.32	
1367	D217	260		-1.34	
1409	D217	258		-1.59	
1957	D217	278.5	C	1.02	first reported 108.5
6250	ISO2137	275		0.57	
6253		-----		-----	

normality	OK
n	12
outliers	0
mean (n)	270.52
st.dev. (n)	8.239
R(calc.)	23.07
st.dev.(D217:19a)	7.857
R(D217:19a)	22

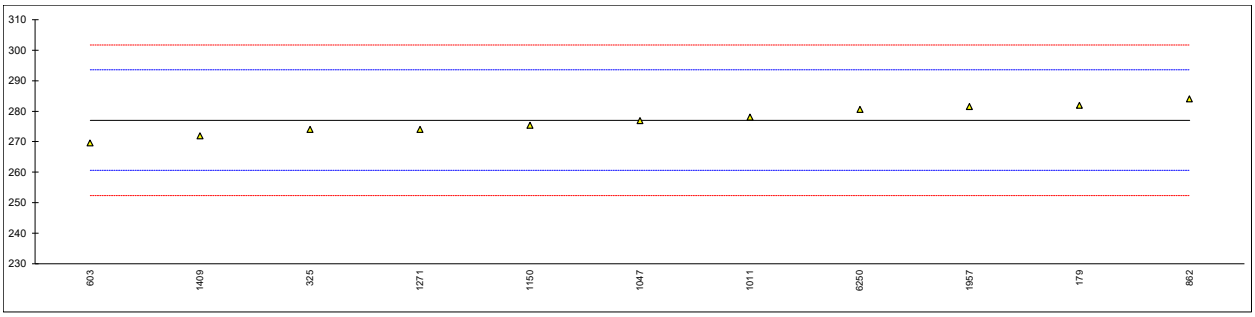


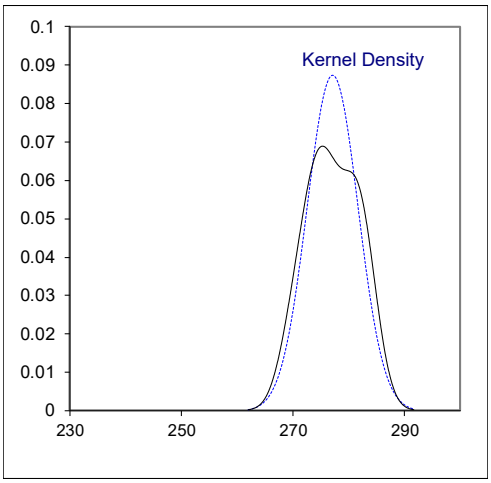


Determination of Cone Penetration - worked on sample #19170; results in 0.1mm

lab	method	value	mark	z(targ)	remarks
179	D217	282		0.60	
325	D217	274		-0.38	
349		-----		-----	
398		-----		-----	
603	D217	269.6		-0.91	
862	D217	284		0.84	
1011	D217	278		0.11	
1047	ISO2137	277		-0.01	
1150	ISO2137	275.4		-0.21	
1271	D217	274		-0.38	
1367		-----		-----	
1409	D217	272		-0.62	
1957	D217	281.6	C	0.55	first reported 111.1
6250	ISO2137	280.5		0.41	
6253		-----		-----	

normality	OK
n	11
outliers	0
mean (n)	277.10
st.dev. (n)	4.567
R(calc.)	12.79
st.dev.(D217:19a)	8.214
R(D217:19a)	23

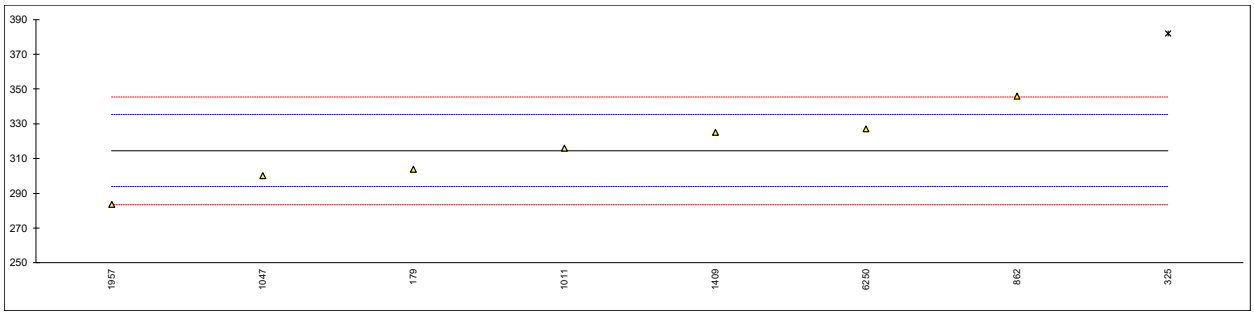


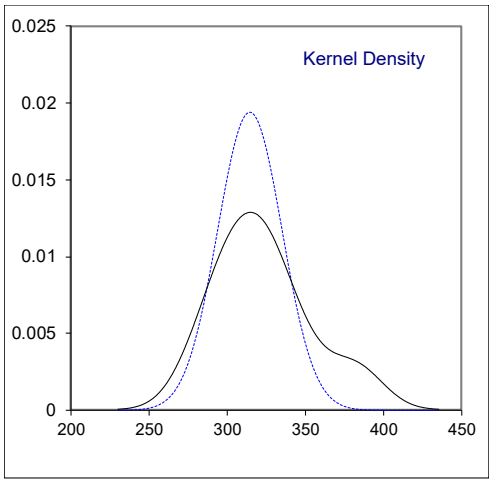


Determination of Cone Penetration – prolonged work on sample #19170; results in 0.1mm

lab	method	value	mark	z(targ)	remarks
179	D217	304		-1.01	
325	D217	382	D(0.05)	6.52	
349		-----		-----	
398		-----		-----	
603		-----		-----	
862	D217	346		3.04	
1011	D217	316		0.14	
1047	ISO2137	300		-1.40	
1150		-----		-----	
1271		-----		-----	
1367		-----		-----	
1409	D217	325		1.01	
1957	D217	283.5	C	-2.99	first reported 111.5
6250	ISO2137	327		1.21	
6253		-----		-----	

normality	unknown
n	7
outliers	1
mean (n)	314.50
st.dev. (n)	20.585
R(calc.)	57.64
st.dev.(D217:19a)	10.357
R(D217:19a)	29





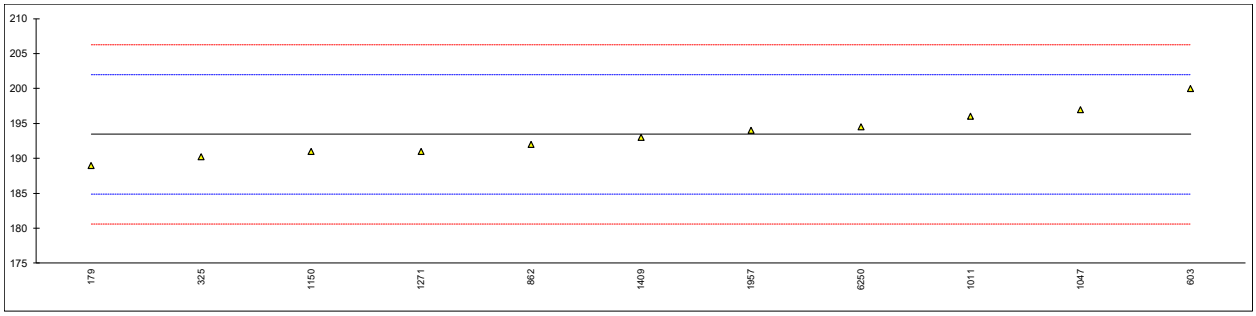
Determination of Copper Corrosion 24 hrs at 100°C on sample #19170

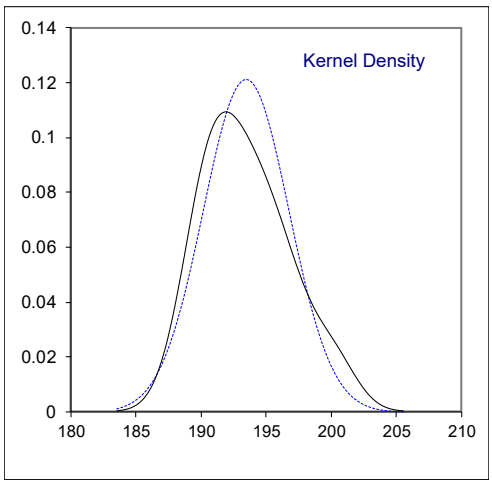
lab	method	value	mark	z(targ)	remarks
179	D4048	1A		-----	
325	DIN51811	1B-slight		-----	
349		-----		-----	
398		-----		-----	
603		-----		-----	
862	D4048	1a		-----	
1011	D4048	1a		-----	
1047	D4048	1A		-----	
1150		-----		-----	
1271	D130	1a		-----	
1367	D130	1A		-----	
1409		-----		-----	
1957	D130	1a		-----	
6250	D4048	1a		-----	
6253		-----		-----	
	n	9			
	mean (n)	1 (1a/1b)			

Determination of Dropping Point on sample #19170; results in °C

lab	method	value	mark	z(targ)	remarks
179	D2265	189.0		-1.03	
325	INH-566	190.2		-0.75	
349		-----		-----	
398		-----		-----	
603	D2265	200.0		1.53	
862	D2265	192		-0.33	
1011	D2265	196		0.60	
1047	ISO2176	197		0.83	
1150	ISO2176	191		-0.57	
1271		191		-0.57	
1367		-----		-----	
1409	D2265	193		-0.10	
1957	D2265	194		0.13	
6250	ISO6299	194.5		0.25	
6253		-----		-----	

normality	OK
n	11
outliers	0
mean (n)	193.43
st.dev. (n)	3.288
R(calc.)	9.21
st.dev.(D2265:19)	4.286
R(D2265:19)	12

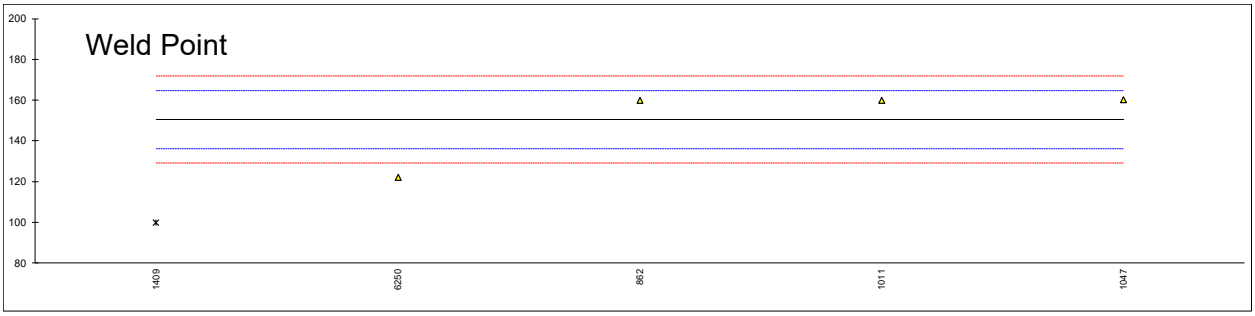


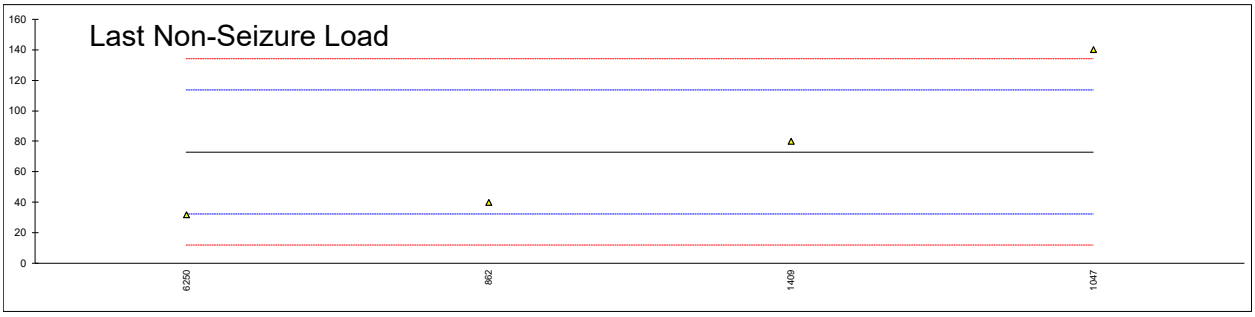


Determination of Extreme-Pressure Properties (Four-ball method) on sample #19170; Weld Point, Load Wear Index (LWI) and Last Non-Seizure Load; results in kgf

lab	method	Weld Point	mark	z(targ)	LWI	mark	z(targ)	Last Non-Seizure Load	mark	z(targ)
179		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
398		-----		-----	-----		-----	-----		-----
603		-----		-----	-----		-----	-----		-----
862	D2596	160		1.33	21.8		-----	40		-1.62
1011	D2596	160		1.33	-----		-----	-----		-----
1047	ISO20623	160.06		1.34	14.76		-----	139.99		3.29
1150		-----		-----	-----		-----	-----		-----
1271		-----		-----	-----		-----	-----		-----
1367		-----		-----	-----		-----	-----		-----
1409	D2596	100	DG(0.01)	-7.07	-----		-----	80		0.34
1957		-----		-----	-----		-----	-----		-----
6250	DIN51350-4	122		-3.99	-----		-----	32		-2.02
6253		-----		-----	-----		-----	-----		-----
	normality	unknown			unknown			unknown		
	n	4			2			4		
	outliers	1			0			0		
	mean (n)	150.5			18.3			73.0		
	st.dev. (n)	19.01			4.98			49.35		
	R(calc.)	53.2			13.9			138.2		
	st.dev.(D2596:15)	7.14			(2.87)			20.34		
	R(D2596:15)	20			(8.0)			56.9		

Lab 6250 reported 1200N for Weld Point

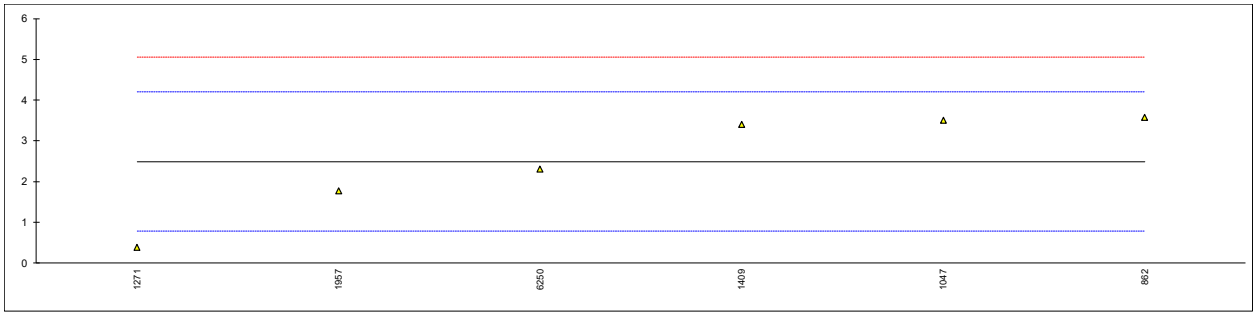




Determination of Oil Separation – Conical Sieve on sample #19170; results in %M/M

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
325		-----		-----	
349		-----		-----	
398		-----		-----	
603		-----		-----	
862	D6184	3.58		1.27	
1011		-----		-----	
1047	PN-V-04047	3.5		1.18	
1150		-----		-----	
1271		0.39		-2.46	
1367		-----		-----	
1409	D6184	3.4		1.06	
1957	D6184	1.7758		-0.84	
6250	DIN51817	2.30		-0.22	
6253		-----		-----	

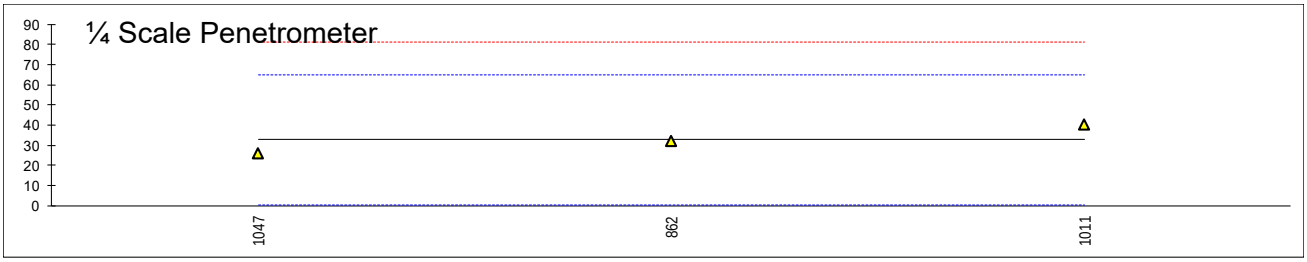
normality	unknown
n	6
outliers	0
mean (n)	2.49
st.dev. (n)	1.264
R(calc.)	3.54
st.dev.(D6184:17)	0.855
R(D6184:17)	2.39

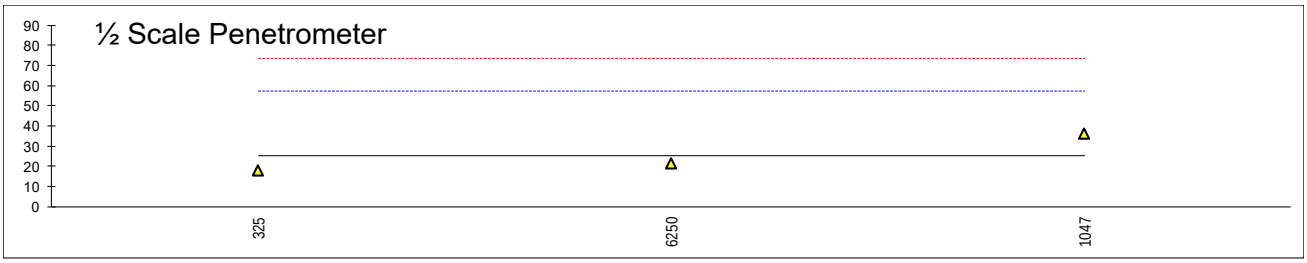


Determination of Roll Stability - Penetration change on sample #19170; results in 0.1mm

lab	method	¼ SP	mark	z(targ)	½ SP	mark	z(targ)	remarks
179		-----		-----	-----		-----	
325	D1831	-----		-----	18		-0.45	
349		-----		-----	-----		-----	
398		-----		-----	-----		-----	
603		-----		-----	-----		-----	
862	D1831	32		-0.04	-----		-----	
1011	D1831	40		0.46	-----		-----	
1047	D1831	26		-0.41	36		0.67	
1150		-----		-----	-----		-----	
1271		-----		-----	-----		-----	
1367		-----		-----	-----		-----	
1409		-----		-----	-----		-----	
1957		-----		-----	-----		-----	
6250	D1831	-----		-----	21.5		-0.23	
6253		-----		-----	-----		-----	

normality	unknown	unknown
n	3	3
outliers	0	0
mean (n)	32.7	25.2
st.dev. (n)	7.02	9.54
R(calc.)	19.7	26.7
st.dev.(D1813:19)	16.07	16.07
R(D1813:19)	45.0	45

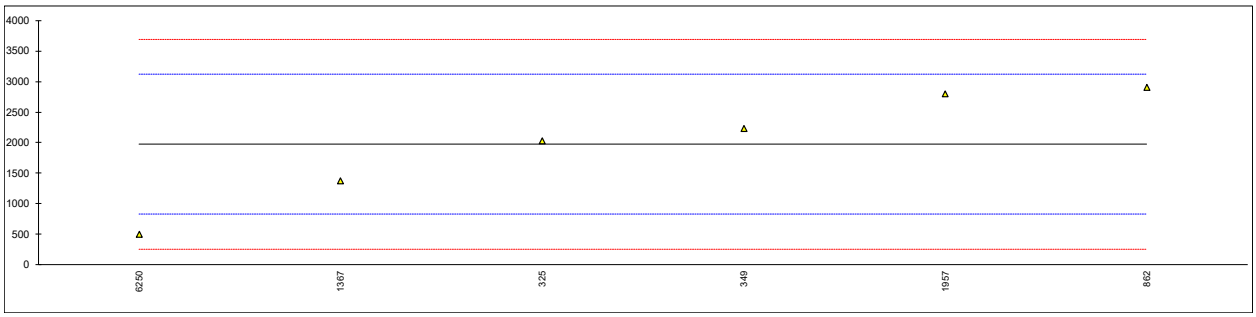




Determination of Water on sample #19170; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
325	D6304-C	2030		0.10	
349	D6304-C	2233		0.45	
398		-----		-----	
603		-----		-----	
862	D6304	2900.9		1.62	
1011		-----		-----	
1047		-----		-----	
1150		-----		-----	
1271		-----		-----	
1367	D6304-C	1378		-1.04	
1409		-----		-----	
1957	D6304	2797		1.44	
6250	ISO3733	500		-2.57	
6253		-----		-----	

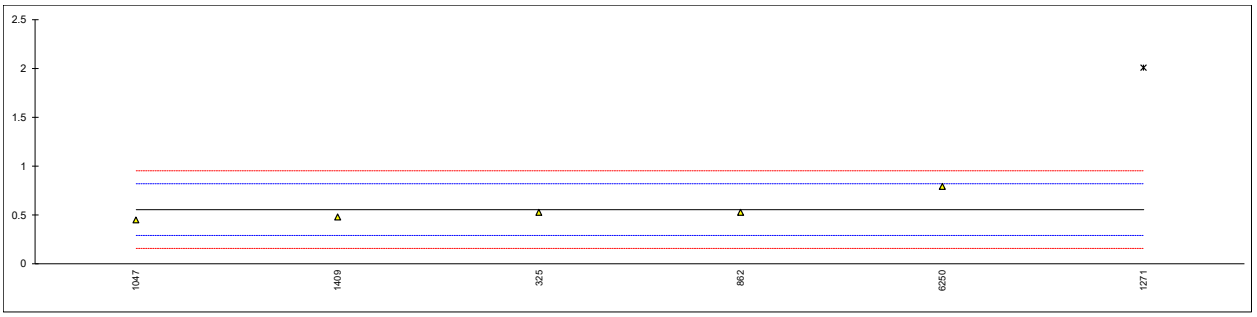
normality	unknown
n	6
outliers	0
mean (n)	1973.2
st.dev. (n)	909.35
R(calc.)	2546.2
st.dev.(D6304:16e1)	572.28
R(D6304:16e1)	1602.4



Determination of Wear Preventive Characteristics on sample #19170; results in mm

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
325	D2266	0.53		-0.20	
349		-----		-----	
398		-----		-----	
603		-----		-----	
862	D2266	0.53		-0.20	
1011		-----		-----	
1047	ISO20623	0.45		-0.81	
1150		-----		-----	
1271	D2266	2.01	D(0.01)	11.00	
1367		-----		-----	
1409	D2266	0.48		-0.58	
1957		-----		-----	
6250	D2266	0.795		1.80	
6253		-----		-----	

normality	unknown
n	5
outliers	1
mean (n)	0.557
st.dev. (n)	0.1374
R(calc.)	0.385
st.dev.(D2266:01)	0.1321
R(D2266:01)	0.37

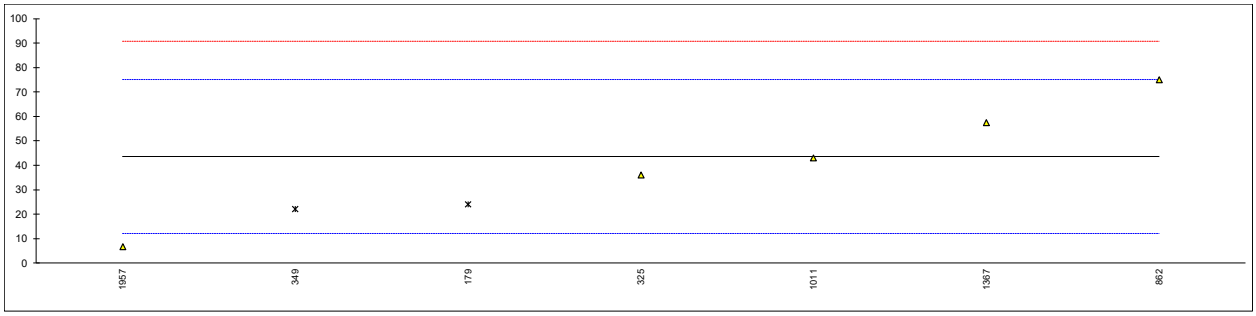


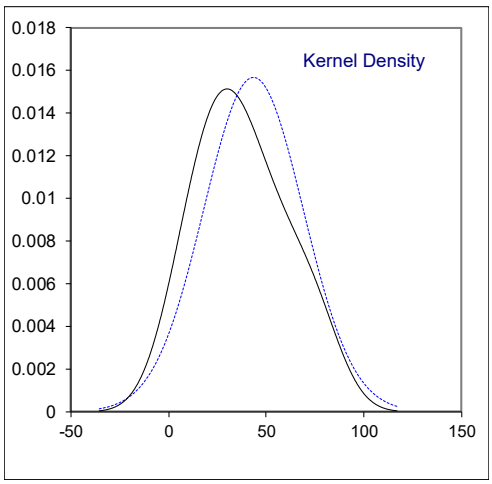
Determination of Calcium as Ca on sample #19170; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D5185	24	ex	-1.25	see § 4.1
325	In-house	36		-0.48	
349	D5185	22	ex	-1.37	see § 4.1
398		-----		-----	
603		-----		-----	
862	D7303	75		1.99	
1011		43		-0.04	
1047		-----		-----	
1150		-----		-----	
1271		-----		-----	
1367		57.35	C	0.87	first reported 81.5
1409		-----		-----	
1957		6.8		-2.34	
6250		-----		-----	
6253		-----		-----	

	normality	unknown
	n	5
	outliers	0 +2ex
	mean (n)	43.6
	st.dev. (n)	25.43
	R(calc.)	71.2
	st.dev.(D7303:17)	15.74
	R(D7303:17)	44.1

application range D7303: 20-50000 mg/kg





Determination of Lithium as Li on sample #19170; results in mg/kg

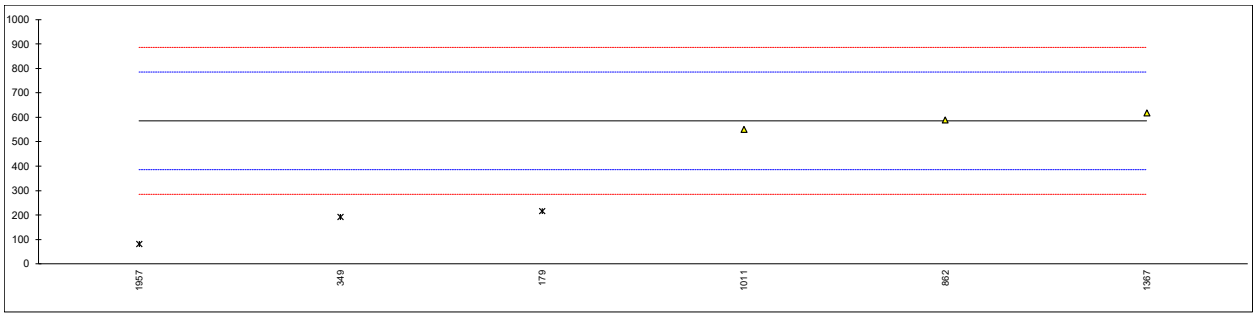
lab	method	value	mark	z(targ)	remarks
179	D5185	1798	ex	-----	see § 4.1
325		-----		-----	
349		-----		-----	
398		-----		-----	
603		-----		-----	
862	D7303	2657		-----	
1011		-----		-----	
1047		-----		-----	
1150		-----		-----	
1271		-----		-----	
1367		3134		-----	
1409		-----		-----	
1957		<1		-----	possibly a false negative test result?
6250		-----		-----	
6253		-----		-----	
	normality	unknown			
	n	2			
	outliers	0 +1ex			
	mean (n)	2895.5			
	st.dev. (n)	337.29			
	R(calc.)	944.4			
	st.dev.(D7303:17)	(202.1)			
	R(D7303:17)	(565.80)			application range D7303: 300-3200 mg/kg

Determination of Sodium as Na on sample #19170; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D5185	217	ex	-3.69	see § 4.1
325		-----		-----	
349	D5185	193	ex	-3.93	see § 4.1
398		-----		-----	
603		-----		-----	
862	D7303	588		0.02	
1011		550		-0.36	
1047		-----		-----	
1150		-----		-----	
1271		-----		-----	
1367		619		0.33	
1409		-----		-----	
1957		82	D(0.05)	-5.04	
6250		-----		-----	
6253		-----		-----	

	normality	unknown
	n	3
	outliers	1 +2ex
	mean (n)	585.7
	st.dev. (n)	34.56
	R(calc.)	96.8
	st.dev.(D7303:17)	100.01
	R(D7303:17)	280.0

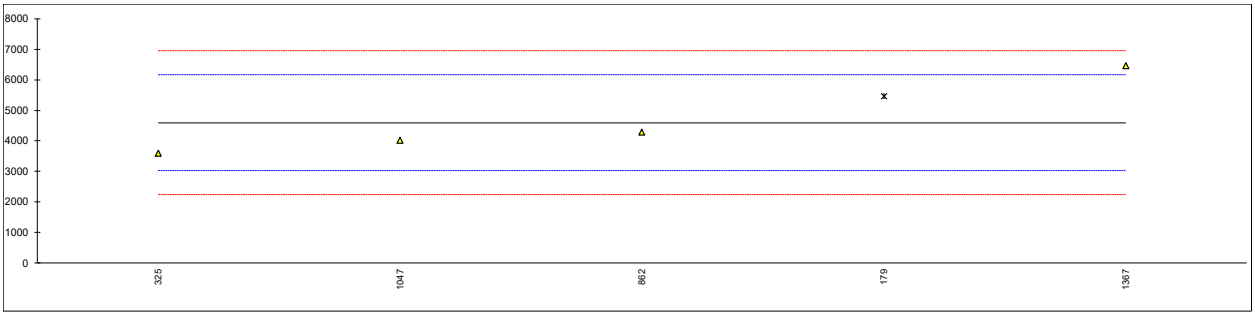
application range D7303: 30-1500 mg/kg



Determination of Sulfur as S on sample #19170; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D5185	5467	ex	1.12	see § 4.1
325	In-house	3590		-1.27	
349		-----		-----	
398		-----		-----	
603		-----		-----	
862	D7303	4280		-0.40	
1011		-----		-----	
1047	ISO8754	4024		-0.72	
1150		-----		-----	
1271		-----		-----	
1367		6467		2.39	
1409		-----		-----	
1957		-----		-----	
6250		-----		-----	
6253		-----		-----	

normality	unknown	
n	4	
outliers	0 +1ex	
mean (n)	4590.3	
st.dev. (n)	1283.17	
R(calc.)	3592.9	
st.dev.(D7303:17)	784.72	
R(D7303:17)	2197.2	application range D7303: 1600-28000 mg/kg



APPENDIX 2: Other reported test results

All reported test results of Aluminum, Antimony, Barium, Iron, Magnesium, Molybdenum, Phosphorus as P, Silicon and Zinc on sample #19170; results in mg/kg

Lab	Al	Sb	Ba	Fe	Mg	Mo	P	Si	Zn
179	1	1	<1	1	<1	<1	<1	1	<1
325	-----	-----	-----	-----	-----	-----	-----	-----	-----
349	<4	<1	<1	2	<4	<4	<4	<4	<4
398	-----	-----	-----	-----	-----	-----	-----	-----	-----
603	-----	-----	-----	-----	-----	-----	-----	-----	-----
862	12	<10	<50	<10	<30	<50	<50	64	<10
1011	<10	-----	<10	<10	<10	<10	11	<10	<10
1047	-----	-----	-----	-----	-----	-----	-----	-----	-----
1150	-----	-----	-----	-----	-----	-----	-----	-----	-----
1271	-----	-----	-----	-----	-----	-----	-----	-----	-----
1367	4.35	-----	0	2.7	0.65	0.1	5.0	16.5	0.7
1409	-----	-----	-----	-----	-----	-----	-----	-----	-----
1957	<1	-----	<1	<1	<1	<1	<1	<1	<1
6250	-----	-----	-----	-----	-----	-----	-----	-----	-----
6253	-----	-----	-----	-----	-----	-----	-----	-----	-----

APPENDIX 3

Number of participants per country

2 labs in BELGIUM
1 lab in BOSNIA and HERZEGOVINA
2 labs in BULGARIA
1 lab in CHINA, People's Republic
1 lab in ITALY
2 labs in MALAYSIA
1 lab in MOROCCO
1 lab in POLAND
1 lab in PORTUGAL
1 lab in SPAIN
1 lab in UNITED KINGDOM
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	= possibly an error in calculations
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, June 2018
2 ASTM E178:16
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, Analyst, 127, 1359-1364 (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)