Results of Proficiency Test
Grease
May 2021

Organized 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 the analysis of Grease in accordance with the latest specification of ASTM D4950 every year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of Grease.

In this interlaboratory study 16 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 Grease proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample 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 #21068.
The 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 24 Grease containers was obtained from a local supplier and labelled #21068. The homogeneity of the subsamples was checked by determination of Cone Penetration-worked in accordance with ASTM D217 on 4 stratified randomly selected subsamples.

	Cone Penetration-worked in 0.1mm
Sample #21068-1	266
Sample #21068-2	264
Sample #21068-3	266
Sample #21068-4	269

Table 1: homogeneity test results of subsamples #21068

From the above test results the repeatability was calculated and compared with 0.3 times the 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)	5.8
reference test method	ASTM D217:21
0.3 x R (reference test method)	6.9

Table 2: evaluation of the repeatability of subsamples #21068

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

To each of the participating laboratories one container of 5 kg Grease labelled #21068 was sent on April 21, 2021. An SDS was added to the sample package.

2.5 ANALYZES

The participants were requested to determine: Cone Penetration (unworked, worked and prolonged work), 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, Elemental Analysis: 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 reference test methods (when applicable) 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 appendices 1 and 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 analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

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

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

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

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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. Therefore, the usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. All participants reported test results, two participants reported the test results after the final reporting date. Not all laboratories were able to report all analyzes requested.
In total 16 participants reported 105 numerical test results. Observed was 1 outlying test result, which is 1.0% 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 reported test results are discussed per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported test results in appendix 1. The abbreviations, used in these tables, are explained 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.

A new version of ASTM D217 was published on July 1st, 2021. In this new version the precision statement was not changed and is the same as in the version from 2019. It was decided to refer to the 2021 version in the report.

Cone Penetration - unworked: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D217:21.

Cone Penetration - worked: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D217:21.

Cone Penetration - prolonged: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D217:21.

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

Dropping Point: This determination was very problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements of ASTM D2265:20.

Extreme-Pressure Properties (Four Ball method): Only four participants reported test results for this determination. No z-scores were calculated due to the low number of test results.

Oil Separation-Conical Sieve: Only three participants reported test results for this determination. No z-scores were calculated due to the low number of test results.

Roll Stability – Penetration change ¼ and ½ Scale Penetrometer: Only two participants reported test results for this determination. No z-scores were calculated due to the low number of test results.

Water: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D6304:16e1 and not at all in agreement with the requirements of ASTM D6304-B:20 or D6304-C:20.
A new version of ASTM D6304 was published in 2020 with major changes. In the 2016 version there was one precision statement for test results based on mass with a broad application range and one based on volume. In the 2021 version all precision statements are based on mass with three different procedures (A – direct injection, B – oven accessory and C – evaporation accessory) and with a different application range. It was decided to use for this PT the precision of D6304:16e1 for calculation of the z-scores and to add the precision of procedure B and C of D6304:20 for comparison.

Wear Preventive Characteristics: Only three participants reported test results for this determination. No z-scores were calculated due to the low number of test results.

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.” Two participants reported to use ASTM D5185.

Calcium: 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 D7303:17.

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

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

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

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

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

For all other elements 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. The reported test results are given in appendix 2.

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, the calculated reproducibility (2.8 * standard deviation) and the target 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	13	269	20	22
Cone Penetration-worked	0.1mm	14	274	24	23
Cone Penetration-prolonged work	0.1mm	6	282	36	29
Copper Corr. 24 hrs at 100°C		10	1 (1a/1b)	n.a.	n.a.
Dropping Point	°C	11	206	26	12
Weld Point	kgf	4	215	67	(20)
Load Wear Index	kgf	3	39	10	(17)
Last Non-Seizure Load	kgf	3	92	29	(72)
Oil Separation-Conical Sieve	%M/M	3	2.5	3.9	(2.4)
¼ Scale Penetrometer	0.1mm	2	n.e.	n.e.	n.e.
½ Scale Penetrometer	0.1mm	1	n.e.	n.e.	n.e.
Water	mg/kg	6	805	1402	936
Wear Preventive Characteristics	mm	3	n.e.	n.e.	n.e.
Calcium as Ca	mg/kg	7	1248	360	471
Lithium as Li	mg/kg	4	2685	1074	523
Molybdenum as Mo	mg/kg	7	191	104	68
Phosphorus as P	mg/kg	7	1028	486	437
Sulfur as S	mg/kg	4	11358	4100	4784
Zinc as Zn	mg/kg	7	1658	415	349

Table 3: reproducibilities of tests on sample #21068

Results between brackets in the last column is based on a low number of data.

Without further statistical calculations it can be concluded that for many 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 MAY 2021 WITH PREVIOUS PTS

	May 2021	September 2019	September 2018	September 2017
Number of reporting laboratories	16	13	17	14
Number of test results	105	113	124	92
Number of statistical outliers	1	4	1	4
Percentage of statistical outliers	1.0%	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 test was compared to the requirements of the reference test methods. The conclusions are given the following table.

Parameter	May 2021	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.	n.e.
½ Scale Penetrometer	n.e.	+	n.e.	n.e.
Water	-	-	+/-	+/-
Wear Preventive Characteristics	n.e.	+/-	++	++
Calcium as Ca	+	-	+	-
Lithium as Li	--	(-)	-	-
Molybdenum as Mo	-	n.a.	n.a.	n.a.
Phosphorus as P	-	n.e.	+	n.e.
Sodium as Na	n.e.	++	n.e.	n.a.
Sulfur as S	+	-	-	n.e.
Zinc as Zn	-	n.e.	--	n.e.

Table 5: comparison determinations against the reference test method

The following performance categories were used:

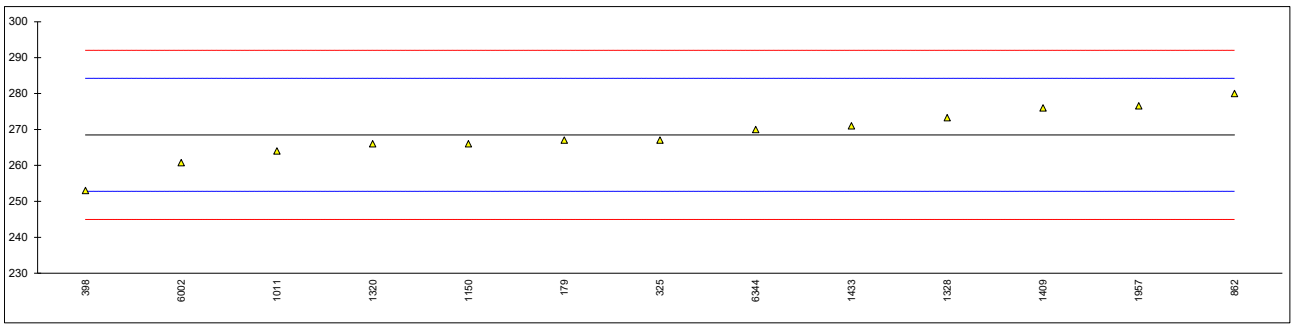
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

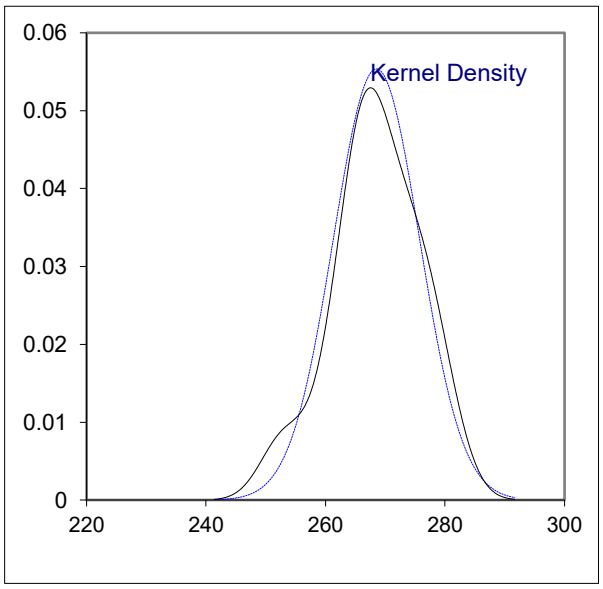
APPENDIX 1

Determination of Cone Penetration - unworked on sample #21068; results in 0.1mm

lab	method	value	mark	z(targ)	remarks
179	D217	267		-0.19	
325	D217	267		-0.19	
349		-----		-----	
398	D217	253		-1.97	
455		-----		-----	
862	D217	280		1.46	
1011	D217	264		-0.57	
1150	ISO2137	266		-0.32	
1213		-----		-----	
1320	D217	266		-0.32	
1328	D217	273.3		0.61	
1409	D217	276		0.95	
1433	D217	271		0.32	
1957	D217	276.6		1.03	
6002	D217	260.75		-0.99	
6344	ISO2137	270		0.19	

normality	OK
n	13
outliers	0
mean (n)	268.51
st.dev. (n)	7.207
R(calc.)	20.18
st.dev.(D217:21)	7.857
R(D217:21)	22

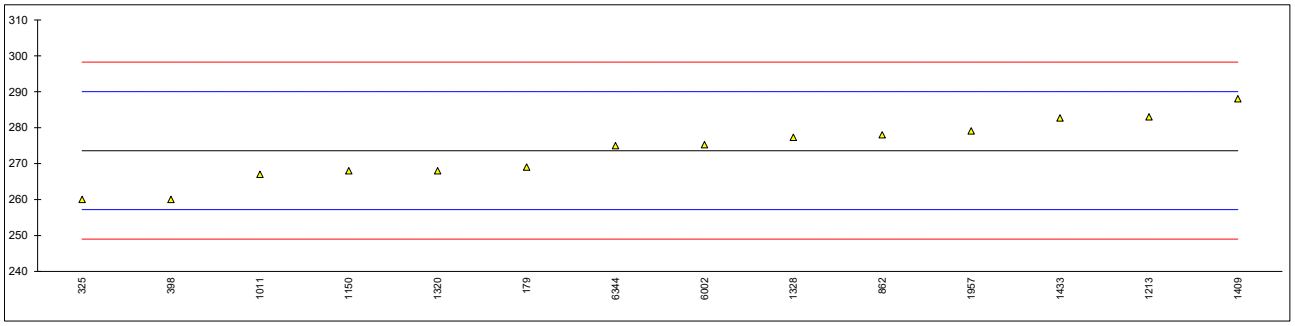


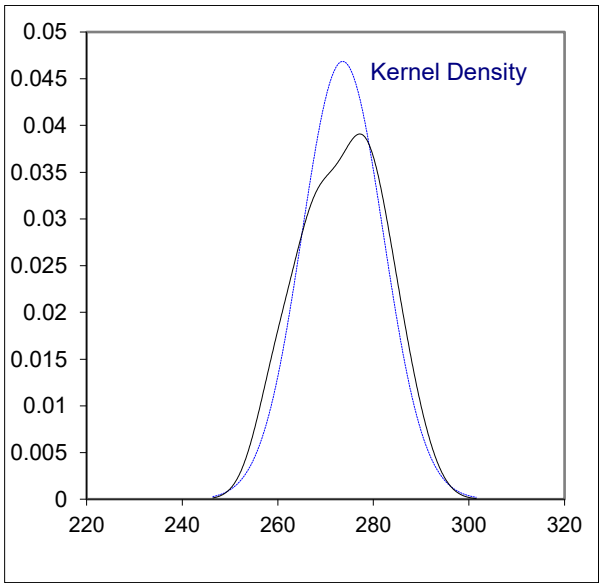


Determination of Cone Penetration - worked on sample #21068; results in 0.1mm

lab	method	value	mark	z(targ)	remarks
179	D217	269		-0.56	
325	D217	260		-1.65	
349		-----		-----	
398	D217	260		-1.65	
455		-----		-----	
862	D217	278		0.54	
1011	D217	267		-0.80	
1150	ISO2137	268		-0.68	
1213	D217	283		1.15	
1320	D217	268		-0.68	
1328	D217	277.3		0.45	
1409	D217	288		1.75	
1433	D217	282.67		1.10	
1957	D217	279.1		0.67	
6002	D217	275.25		0.20	
6344	ISO2137	275		0.17	

normality	OK
n	14
outliers	0
mean (n)	273.59
st.dev. (n)	8.518
R(calc.)	23.85
st.dev.(D217:21)	8.214
R(D217:21)	23

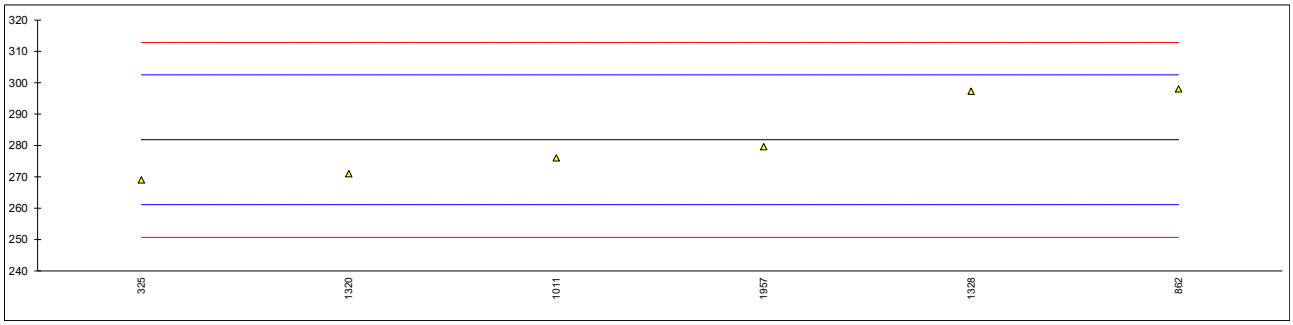




Determination of Cone Penetration – prolonged work on sample #21068; results in 0.1mm

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
325	D217	269		-1.24	
349		-----		-----	
398		-----		-----	
455		-----		-----	
862	D217	298		1.56	
1011	D217	276		-0.56	
1150		-----		-----	
1213		-----		-----	
1320	D217	271		-1.04	
1328	D217	297.3		1.49	
1409		-----		-----	
1433		-----	W	-----	first reported: 310.67
1957	D217	279.6		-0.21	
6002		-----		-----	
6344		-----		-----	

normality	unknown
n	6
outliers	0
mean (n)	281.82
st.dev. (n)	12.819
R(calc.)	35.89
st.dev.(D217:21)	10.357
R(D217:21)	29



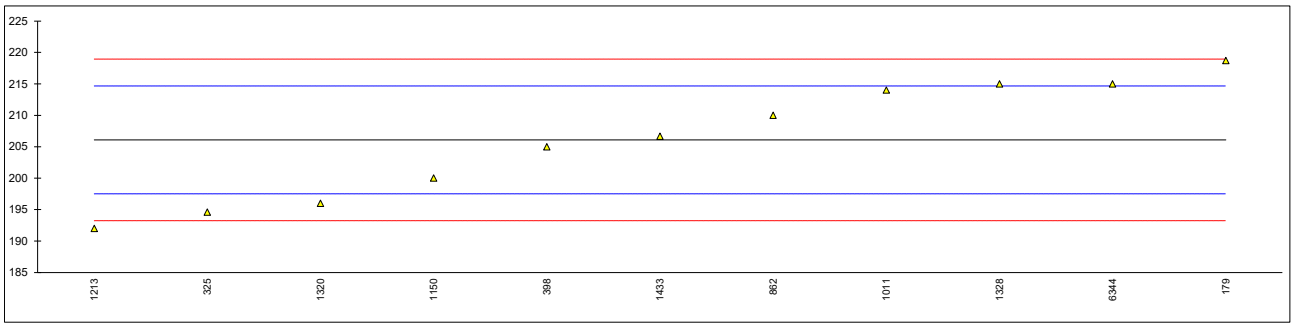
Determination of Copper Corrosion 24 hrs at 100°C on sample #21068

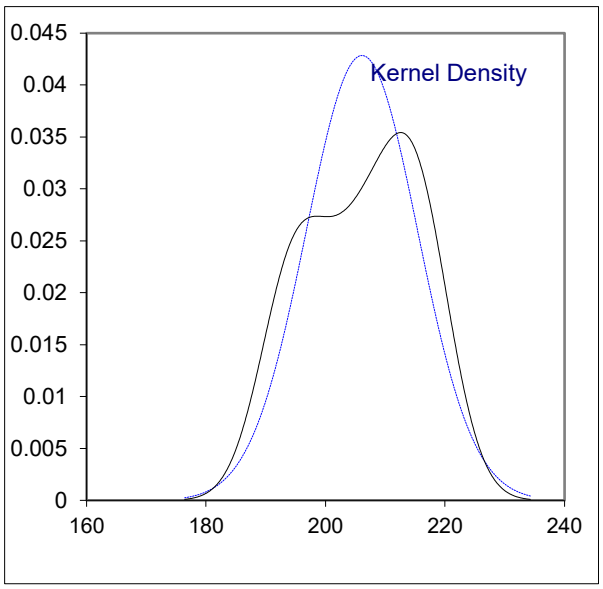
lab	method	value	mark	z(targ)	remarks
179		-----		-----	
325	D4048	1B - slight		-----	
349		-----		-----	
398	D4048	1a		-----	
455		-----		-----	
862	D4048	1a		-----	
1011	D4048	1b		-----	
1150		-----		-----	
1213	D130	1a		-----	
1320	D4048	1a		-----	
1328	D4048	1b		-----	
1409		-----		-----	
1433	D4048	1a		-----	
1957	D130	1a		-----	
6002	D130	1a		-----	
6344	D4048	2c	f+?	-----	possible false positive result?
	n	10			
	mean (n)	1 (1a/1b)			

Determination of Dropping Point on sample #21068; results in °C

lab	method	value	mark	z(targ)	remarks
179	D2265	218.7		2.94	
325	IP396/D566	194.6		-2.68	
349		-----		-----	
398	D2265	205		-0.25	
455		-----		-----	
862	D2265	210		0.91	
1011	D2265	214		1.85	
1150	D2265	200		-1.42	
1213	D566	192.0		-3.29	
1320	D2265	196		-2.35	
1328	D2265	215		2.08	
1409		-----		-----	
1433	D2265	206.67		0.14	
1957		-----		-----	
6002		-----		-----	
6344	D2265	215		2.08	

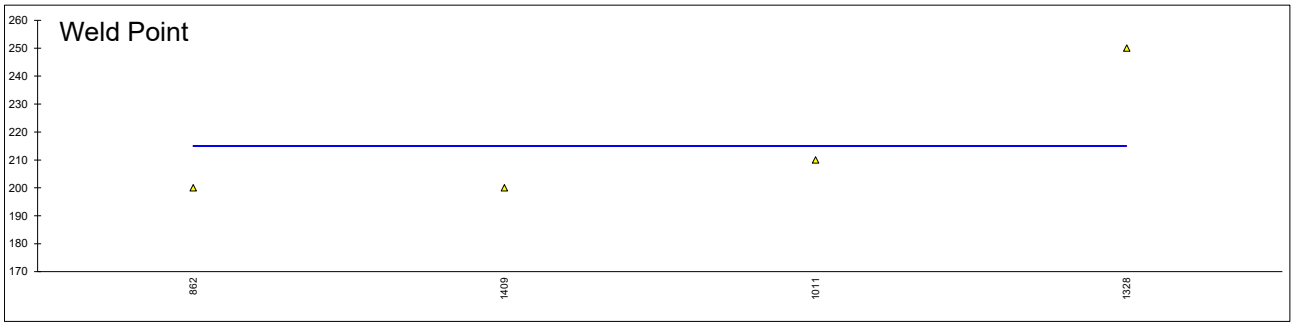
normality	OK
n	11
outliers	0
mean (n)	206.09
st.dev. (n)	9.309
R(calc.)	26.07
st.dev.(D2265:20)	4.286
R(D2265:20)	12

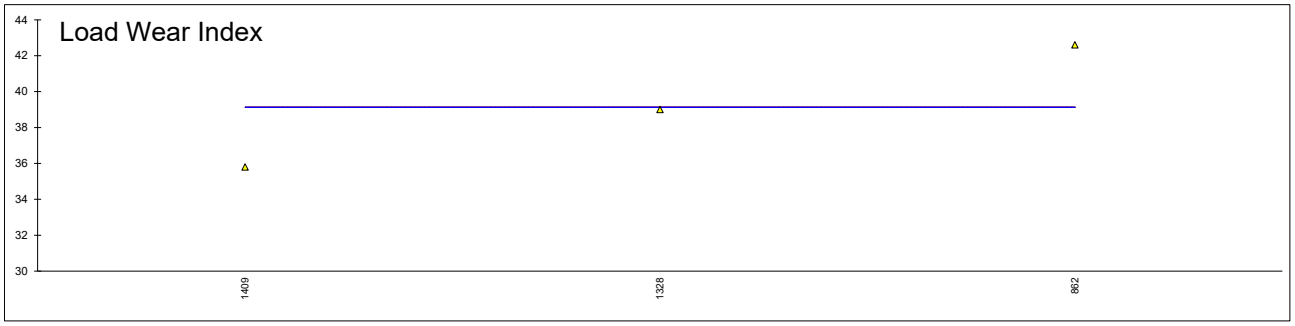


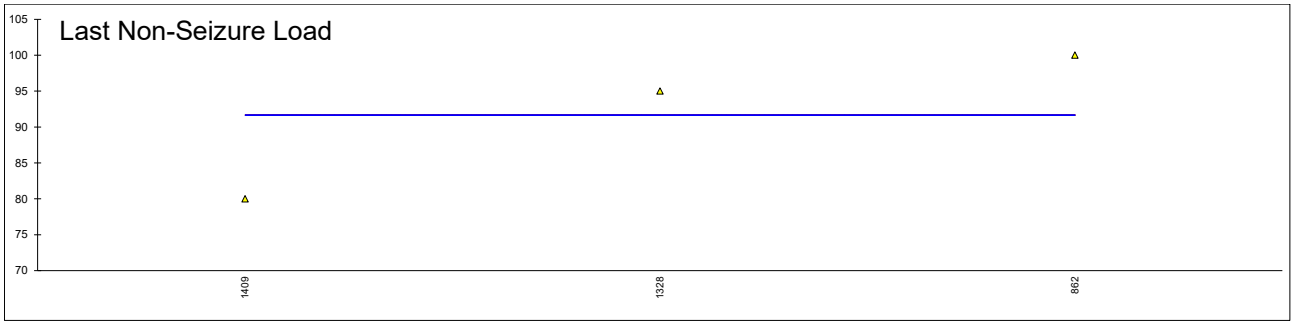


Determination of Extreme-Pressure Properties (Four-ball method) on sample #21068; Weld Point, Load Wear Index (LWI) and Last Non-Seizure Load; results in kgf

lab	method	Weld Point	mark	z(targ)	LWI	mark	z(targ)	Last N-S Load	mark	z(targ)
179		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
398		-----		-----	-----		-----	-----		-----
455		-----		-----	-----		-----	-----		-----
862	D2596	200		-----	42.6		-----	100		-----
1011	D2596	210		-----	-----		-----	-----		-----
1150		-----		-----	-----		-----	-----		-----
1213		-----		-----	-----		-----	-----		-----
1320		-----		-----	-----		-----	-----		-----
1328	D2596	250		-----	39		-----	95		-----
1409	D2596	200		-----	35.8		-----	80		-----
1433		-----		-----	-----		-----	-----		-----
1957		-----		-----	-----		-----	-----		-----
6002		-----		-----	-----		-----	-----		-----
6344		-----		-----	-----		-----	-----		-----
	normality	unknown			unknown			unknown		
	n	4			3			3		
	outliers	0			0			0		
	mean (n)	215.0			39.13			91.7		
	st.dev. (n)	23.80			3.402			10.41		
	R(calc.)	66.7			9.53			29.1		
	st.dev.(D2596:15)	(7.14)			(6.150)			(25.54)		
	R(D2596:15)	(20)			(17.22)			(71.5)		



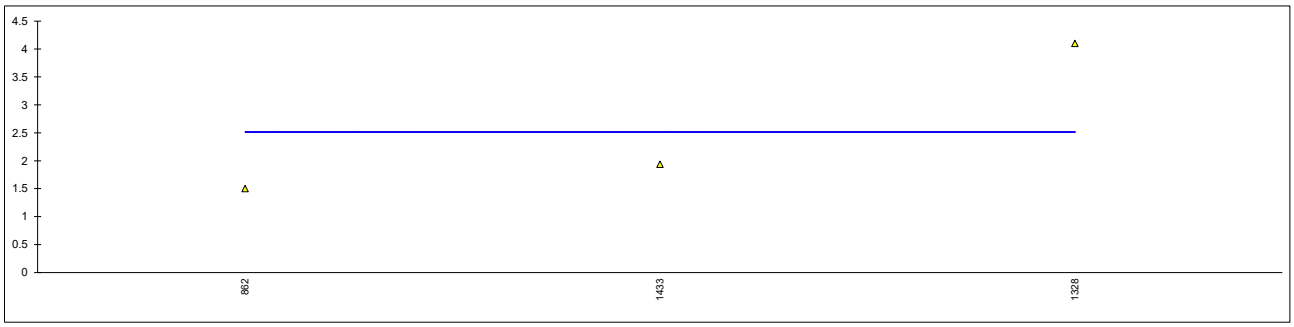




Determination of Oil Separation – Conical Sieve on sample #21068; results in %M/M

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
325		-----		-----	
349		-----		-----	
398		-----		-----	
455		-----		-----	
862	D6184	1.5		-----	
1011		-----		-----	
1150		-----		-----	
1213		-----		-----	
1320		-----		-----	
1328	D6184	4.1		-----	
1409		-----		-----	
1433	In house	1.9356		-----	
1957		-----		-----	
6002		-----		-----	
6344		-----		-----	

normality	unknown
n	3
outliers	0
mean (n)	2.51
st.dev. (n)	1.393
R(calc.)	3.90
st.dev.(D6184:17)	(0.859)
R(D6184:17)	(2.40)

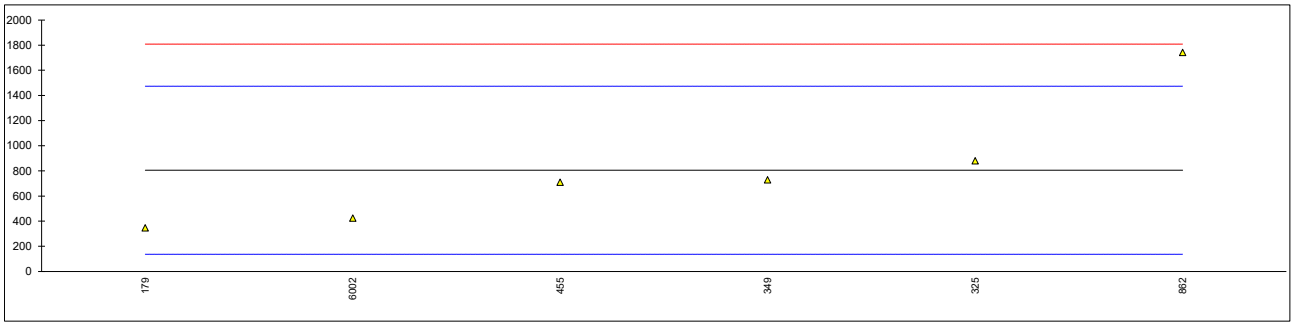


Determination of Roll Stability - Penetration change on sample #21068; results in 0.1mm

lab	method	¼ Scale Pen.	mark	z(targ)	½ Scale Pen.	mark	z(targ)	remarks
179		-----		-----	-----		-----	
325		-----		-----	-----		-----	
349		-----		-----	-----		-----	
398		-----		-----	-----		-----	
455		-----		-----	-----		-----	
862	D1831	10		-----	9		-----	
1011	D1831	4		-----	-----		-----	
1150		-----		-----	-----		-----	
1213		-----		-----	-----		-----	
1320		-----		-----	-----		-----	
1328		-----		-----	-----		-----	
1409		-----		-----	-----		-----	
1433		-----		-----	-----		-----	
1957		-----		-----	-----		-----	
6002		-----		-----	-----		-----	
6344		-----		-----	-----		-----	
	n	2			1			

Determination of Water on sample #21068; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D6304-C	347		-1.37	
325	D6304-C	880		0.22	
349	D6304-C	729		-0.23	
398		-----		-----	
455	D6304-B	709.6		-0.29	
862	D6304-B	1742		2.80	
1011		-----		-----	
1150		-----		-----	
1213		-----		-----	
1320		-----		-----	
1328		-----		-----	
1409		-----		-----	
1433		-----		-----	
1957		-----		-----	
6002	In house	424.35		-1.14	
6344		-----		-----	
	normality	unknown			
	n	6			
	outliers	0			
	mean (n)	805.33			
	st.dev. (n)	500.625			
	R(calc.)	1401.75			
	st.dev.(D6304:16e1)	334.270			
	R(D6304:16e1)	935.96			Range: 10-25000 mg/kg
compare					
	R(D6304-B:20)	368.56			Range: 33-2094 mg/kg
	R(D6304-C:20)	239.72			Range: 20-359 mg/kg



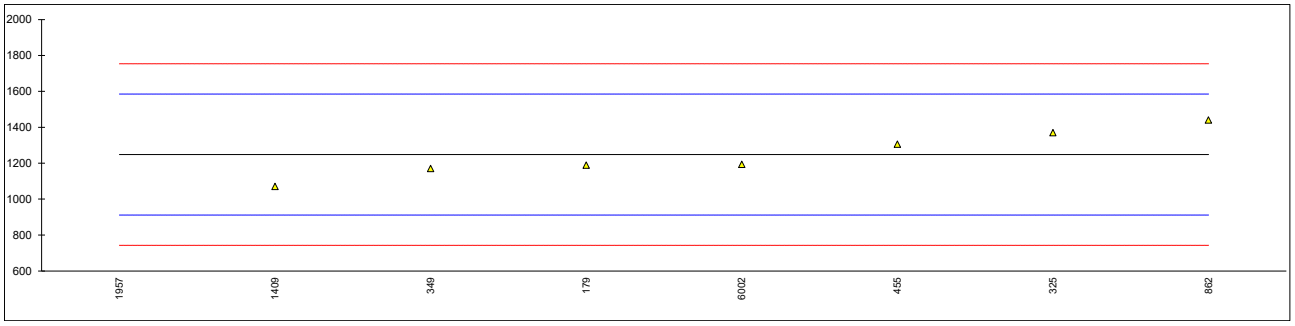
Determination of Wear Preventive Characteristics on sample #21068; results in mm

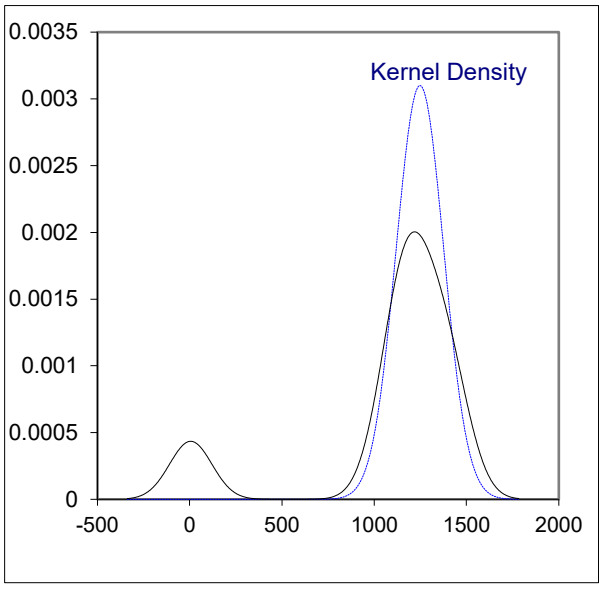
lab	method	value	mark	z(targ)	remarks
179		-----		-----	
325		-----		-----	
349		-----		-----	
398		-----		-----	
455		-----		-----	
862	D7303	<10		-----	
1011		-----		-----	
1150		-----		-----	
1213		-----		-----	
1320		-----		-----	
1328	D2266	0.39		-----	
1409	D2266	0.40		-----	
1433		-----		-----	
1957		-----		-----	
6002		-----		-----	
6344		-----		-----	
	n	3			

Determination of Calcium as Ca on sample #21068; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D5185	1189		-0.35	
325	D6443/D4927	1370		0.72	
349	D5185	1170		-0.46	
398		-----		-----	
455	D7303	1306		0.34	
862	D7303	1440		1.14	
1011		-----		-----	
1150		-----		-----	
1213		-----		-----	
1320		-----		-----	
1328		-----		-----	
1409		1070		-1.06	
1433		-----		-----	
1957	D7303	5.4	C,G(0.01)	-7.38	first reported: 214
6002	D6595	1193		-0.33	
6344		-----		-----	

normality	unknown	
n	7	
outliers	1	
mean (n)	1248	
st.dev. (n)	128.7	
R(calc.)	360	
st.dev.(D7303:17)	168.4	
R(D7303:17)	471	Range ASTM D7303: 20-50000 mg/kg

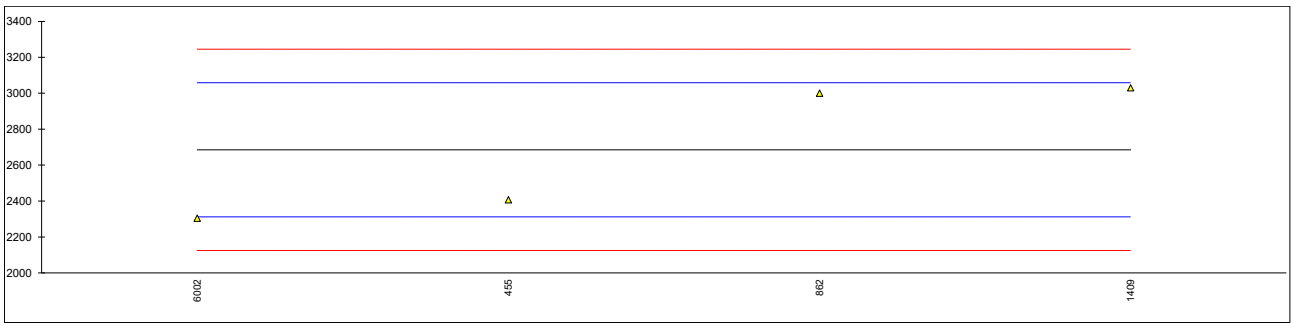




Determination of Lithium as Li on sample #21068; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
325		-----		-----	
349		-----		-----	
398		-----		-----	
455	D7303	2406		-1.49	
862	D7303	3000		1.69	
1011		-----		-----	
1150		-----		-----	
1213		-----		-----	
1320		-----		-----	
1328		-----		-----	
1409		3030		1.85	
1433		-----		-----	
1957	D7303	<1		<-14.37	possible false negative test result?
6002	D6595	2304		-2.04	
6344		-----		-----	

normality	unknown	
n	4	
outliers	0	
mean (n)	2685	
st.dev. (n)	383.5	
R(calc.)	1074	
st.dev.(D7303:17)	186.9	
R(D7303:17)	523	Range D7303: 300-3200 mg/kg

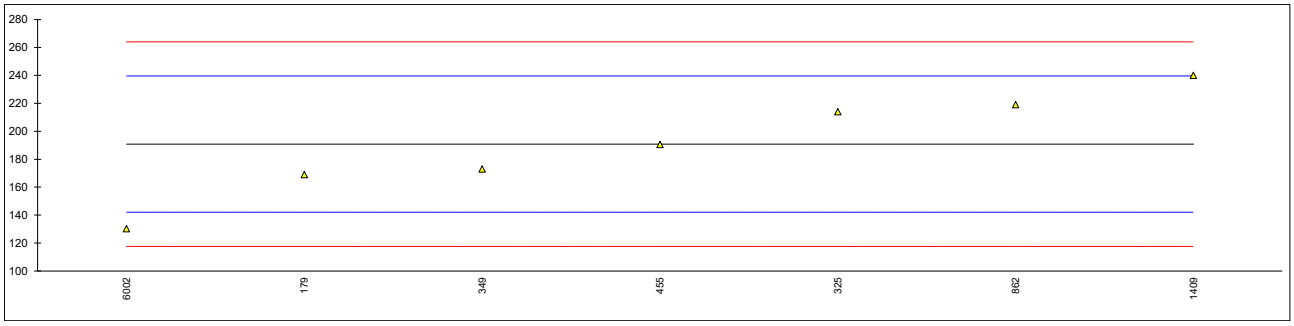


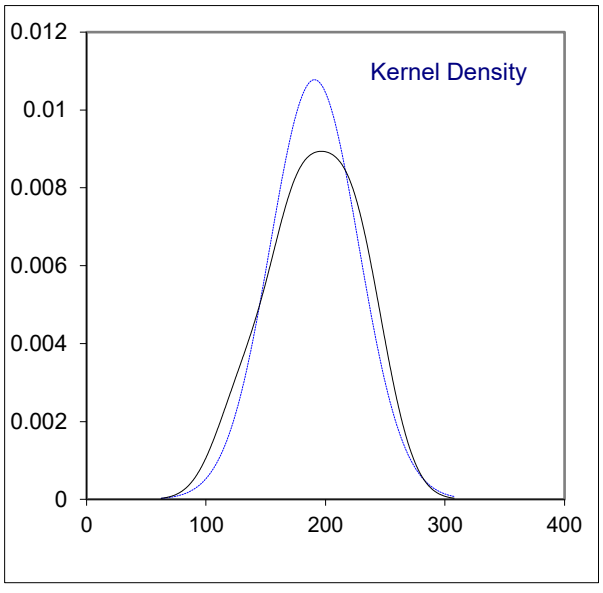
Determination of Molybdenum as Mo on sample #21068; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D5185	169		-0.89	
325	D6443/D4927	214		0.95	
349	D5185	173		-0.73	
398		-----		-----	
455	D7303	190.5		-0.01	
862	D7303	219		1.16	
1011		-----		-----	
1150		-----		-----	
1213		-----		-----	
1320		-----		-----	
1328		-----		-----	
1409		240		2.02	
1433		-----		-----	
1957	D7303	<1		<-7.78	possible false negative test result?
6002	D6595	130.19		-2.49	
6344		-----		-----	

	normality	unknown
	n	7
	outliers	0
	mean (n)	190.8
	st.dev. (n)	37.01
	R(calc.)	103.6
	st.dev.(D7303:17)	24.39
	R(D7303:17)	68.3

Range ASTM D7303: 50-22000 mg/kg



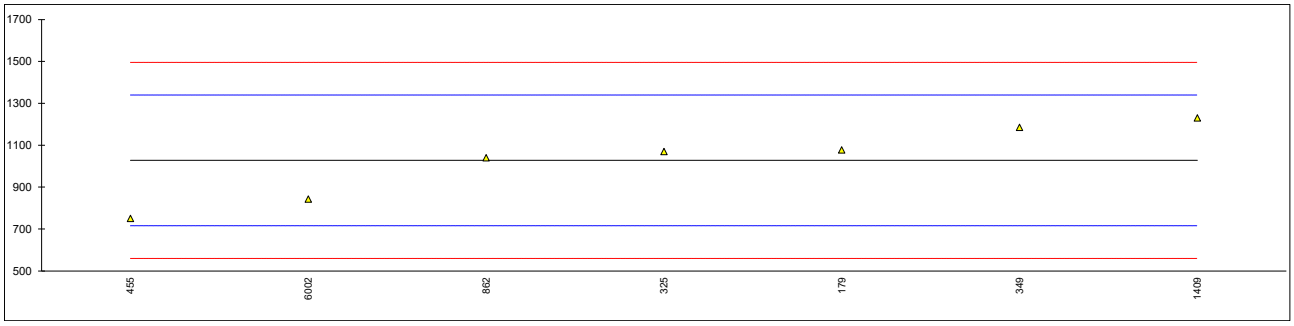


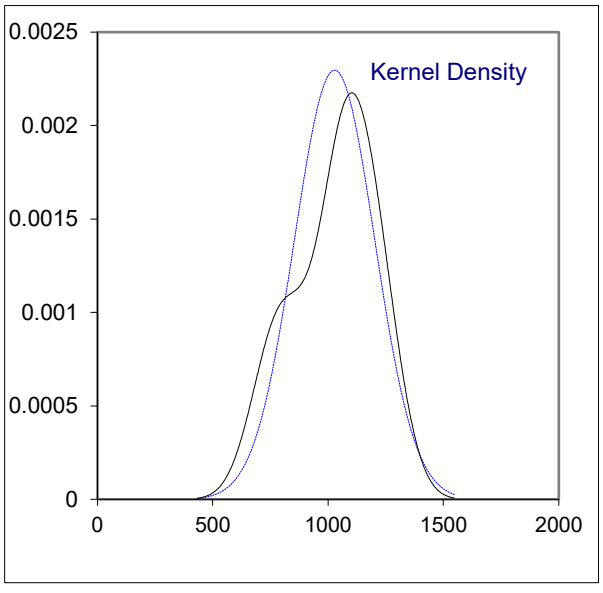
Determination of Phosphorus as P on sample #21068; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D5185	1077		0.31	
325	D6443/D4927	1070		0.27	
349	D5185	1185		1.01	
398		-----		-----	
455	D7303	750.4		-1.78	
862	D7303	1040		0.08	
1011		-----		-----	
1150		-----		-----	
1213		-----		-----	
1320		-----		-----	
1328		-----		-----	
1409		1230		1.30	
1433		-----		-----	
1957	D7303	<1	C	<-6.58	first reported: 307, possible false negative test result?
6002	D6595	842.91		-1.19	
6344		-----		-----	

	normality	unknown
	n	7
	outliers	0
	mean (n)	1027.9
	st.dev. (n)	173.72
	R(calc.)	486.4
	st.dev.(D7303:17)	156.00
	R(D7303:17)	436.8

Range ASTM D7303: 50-2000 mg/kg

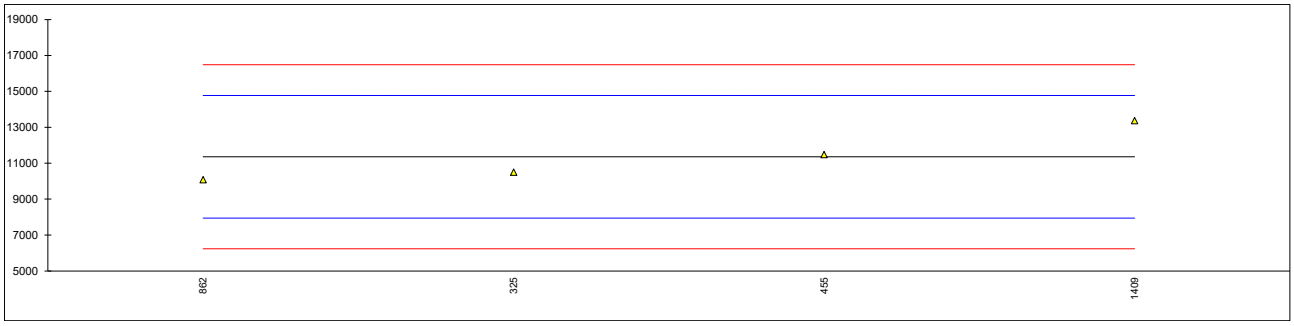




Determination of Sulfur as S on sample #21068; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
325	D6443/D4927	10500		-0.50	
349		-----		-----	
398		-----		-----	
455	D7303	11482		0.07	
862	D7303	10080		-0.75	
1011		-----		-----	
1150		-----		-----	
1213		-----		-----	
1320		-----		-----	
1328		-----		-----	
1409		13370		1.18	
1433		-----		-----	
1957		-----		-----	
6002		-----		-----	
6344		-----		-----	

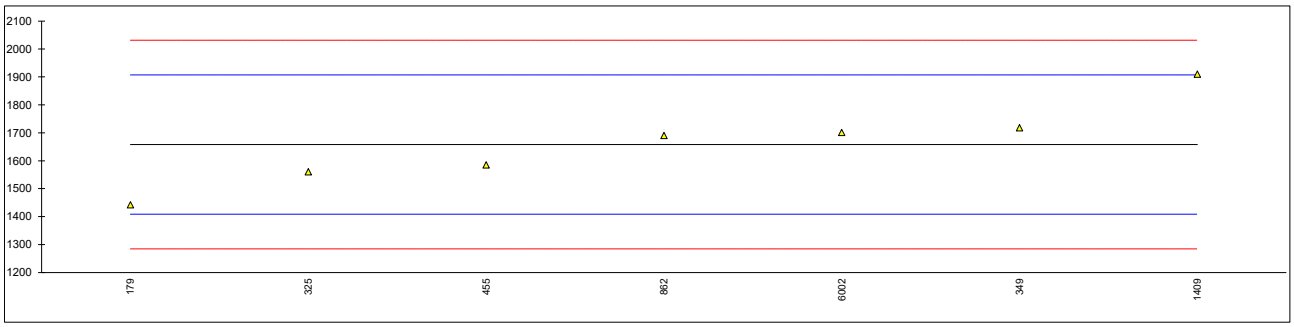
normality	unknown	
n	4	
outliers	0	
mean (n)	11358	
st.dev. (n)	1464.4	
R(calc.)	4100	
st.dev.(D7303:17)	1708.5	
R(D7303:17)	4784	Range ASTM D7303: 1600-28000 mg/kg

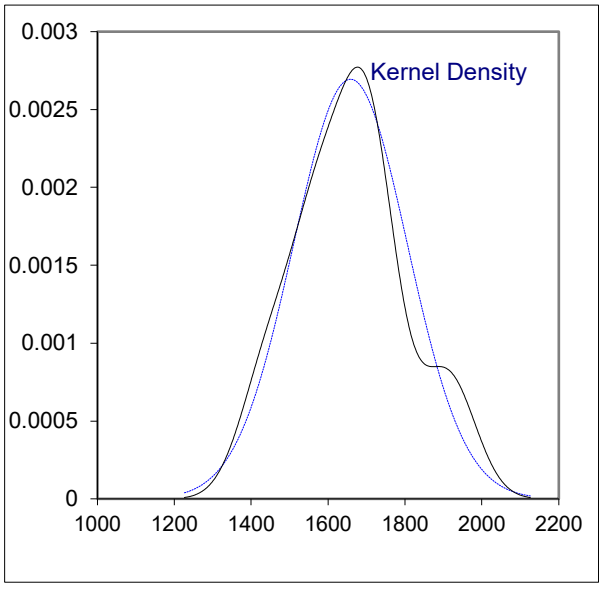


Determination of Zinc as Zn on sample #21068; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D5185	1442		-1.73	
325	D6443/D4927	1560		-0.79	
349	D5185	1718		0.48	
398		-----		-----	
455	D7303	1585		-0.59	
862	D7303	1690		0.26	
1011		-----		-----	
1150		-----		-----	
1213		-----		-----	
1320		-----		-----	
1328		-----		-----	
1409		1910		2.02	
1433		-----		-----	
1957	D7303	<1	C	<-13.29	first reported: 398, possible false negative test result?
6002	D6595	1701		0.34	
6344		-----		-----	

normality	unknown	
n	7	
outliers	0	
mean (n)	1658	
st.dev. (n)	148.1	
R(calc.)	415	
st.dev.(D7303:17)	124.6	
R(D7303:17)	349	Range ASTM D7303: 300-2200 mg/kg





APPENDIX 2

Other reported test results on sample #21068; results in mg/kg

Lab	Al	Sb	Ba	Fe	Mg	Si	Na
179	<1	-----	<1	2	7	8	4
325	-----	-----	-----	-----	-----	-----	-----
349	<4	<1	<1	<2 C	6	<4	2
398	-----	-----	-----	-----	-----	-----	-----
455	4.33	-----	2.529	2.755	8.153	21.17	12.18
862	<10	<10	<50	<10	<30	10	<30
1011	-----	-----	-----	-----	-----	-----	-----
1150	-----	-----	-----	-----	-----	-----	-----
1213	-----	-----	-----	-----	-----	-----	-----
1320	-----	-----	-----	-----	-----	-----	-----
1328	-----	-----	-----	-----	-----	-----	-----
1409	0	0	0	0	10	0	0
1433	-----	-----	-----	-----	-----	-----	-----
1957	<1	-----	<1	<1	<1	<1	24
6002	1.23	0	0	0	6.77	4.31	20.85
6344	-----	-----	-----	-----	-----	-----	-----
Range *	10-600	10-2300	50-800	10-360	30-10000	10-15000	30-1500

*) of D7303 for the different elements in mg/kg

Lab 349 first reported for Fe: 8

APPENDIX 3

Number of participants per country

2 labs in	BELGIUM
1 lab in	BULGARIA
2 labs in	CHINA, People's Republic
1 lab in	ITALY
1 lab in	MALAYSIA
2 labs in	POLAND
1 lab in	PORTUGAL
2 labs in	SLOVAKIA
1 lab in	SPAIN
1 lab in	UNITED KINGDOM
1 lab in	UNITED STATES OF AMERICA
1 lab in	VIETNAM

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	= calculation difference between reported test result and result calculated by iis
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 Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)