

Results of Proficiency Test
Hydraulic Fluid (used)
November 2014

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Corrector: dr. R.G. Visser & ing. L. Sweere
Report no. iis14L05

February 2015

--Empty page --

CONTENTS

1	INTRODUCTION	4
2	SET UP	4
2.1	QUALITY SYSTEM	4
2.2	PROTOCOL	4
2.3	CONFIDENTIALITY STATEMENT	4
2.4	SAMPLES.....	5
2.5	STABILITY OF THE SAMPLES	6
2.6	ANALYSES	6
3	RESULTS.....	6
3.1	STATISTICS.....	7
3.2	GRAPHICS.....	8
3.3	Z-SCORES.....	8
4	EVALUATION.....	9
4.1	EVALUATION PER TEST	9
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	12
4.3	COMPARISON OF THE PT OF NOVEMBER 2014 WITH THE PREVIOUS PTS	14

Appendices:

1.	Data and statistical results	16
2.	Number of participants per country	49
3.	Abbreviations and literature	50

1 INTRODUCTION

Since 2003, the Institute for Interlaboratory Studies organized a proficiency test for the analysis of used Hydraulic Fluid every year. It was decided to continue this interlaboratory study during the annual program 2014/2015. In this interlaboratory study, 43 laboratories from 30 different countries have participated. See appendix 2 for the number of participants per country. In this report, the test results of the 2014 interlaboratory study on used Hydraulic Fluid are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. Analysis for fit-for-use and homogeneity testing were subcontracted. It was decided to send two different samples of used fluids: one sample of 1 litre used Tellus S46 Hydraulic Fluid and one sample of 0.1 litre used Tellus S46 Hydraulic Fluid especially for wear metals. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system on IEC/ISO17043:2010. 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 was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website www.iisnl.com.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary bulk materials for the two samples of used Hydraulic Fluid were obtained from a participating laboratory. The approximately 80 litre of the first bulk material was homogenised in a precleaned drum. After homogenisation, 80 subsamples of 1 liter amber glass bottles were filled and labelled #14220. The homogeneity of the subsamples #14220 was checked by determination of Density in accordance with ASTM D4052 and Viscosity at 40 °C according to ASTM D445 on 8 stratified randomly selected samples.

	<i>Density @15 °C in kg/L</i>	<i>Viscosity @40 °C in cSt</i>
Sample #14220-1	0.87438	41.14
Sample #14220-2	0.87437	41.13
Sample #14220-3	0.87438	41.14
Sample #14220-4	0.87438	41.14
Sample #14220-5	0.87438	41.14
Sample #14220-6	0.87438	41.14
Sample #14220-7	0.87437	41.13
Sample #14220-8	0.87438	41.13

Table 1: homogeneity test results of subsamples #14220

From the test results of table 1, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	<i>Density @15 °C in kg/L</i>	<i>Viscosity @40 °C in cSt</i>
r (Observed)	0.00001	0.015
reference method	ISO12185:96	D445:12
0.3 * R (ref. method)	0.00015	0.094

Table 2: repeatabilities of subsamples #14220

The approximately 5 litre of the second bulk material, positive on a number of metals, was homogenised. After homogenisation 78 subsamples of 100 mL HDPE containers were filled with approximately 70 mL material and labelled #14221. The homogeneity of the subsamples #14221 was checked by determination of Density in accordance with ASTM D4052 and Nickel and Phosphorus in accordance with ASTM D5185 on 8 stratified randomly selected samples.

	<i>Density @ 15 °C in kg/L</i>	<i>Phosphorus in mg/kg</i>	<i>Nickel in mg/kg</i>
Sample #14221-1	0.87467	342	8.4
Sample #14221-2	0.87463	343	8.8
Sample #14221-3	0.87466	348	8.7
Sample #14221-4	0.87465	334	8.8
Sample #14221-5	0.87466	341	8.6
Sample #14221-6	0.87464	335	8.3
Sample #14221-7	0.87465	347	8.4
Sample #14221-8	0.87465	333	8.0

Table 3: homogeneity test results of subsamples #14221

From the test results of table 3, the repeatability was calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	<i>Density @ 15 °C in kg/L</i>	<i>Phosphorus in mg/kg</i>	<i>Nickel in mg/kg</i>
r (Observed)	0.00003	16	0.8
reference method	ISO12185:96	D5185:13	D5185:13
0.3* R (ref. method)	0.00015	24	1.3

Table 4: repeatability of subsamples #14221

Each calculated repeatability was equal or less than 0.3 times the corresponding reproducibility of the respective test method. Therefore, homogeneity of the subsamples #14220 and #14221 was assumed.

To each of the participating laboratories was dispatched: One 1 litre amber glass bottle, labelled #14220 and one 100 mL HDPE container, labelled #14221 on October 29, 2014.

2.5 STABILITY OF THE SAMPLES

The stability of Hydraulic Fluid, packed in the brown glass bottles or in HDPE containers, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine Total Acid Number, Density @ 15 °C, Flash Point PMcc, Kinematic Viscosity @ 40 °C and @ 100 °C, Viscosity Stabinger @ 40 °C and @ 100°C, Pour Point (manual and automated), Sulphur, Water and Water Separability @ 54 °C on sample #14220 and 20 elements (17 wear metals and 3 additives) on sample #14221.

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

A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original results are tabulated per determination in the appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported results at that moment. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

Results that came in after deadline were not taken into account in the screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, April 2014 version 3.3). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<...>' or '>...>' were not used in the statistical evaluation.

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

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and Rosner outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 3, no.15). Stragglers are marked by D(0.05) for the Dixon 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 the averages and the standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.13 and 14). Also a normal Gauss curve was projected over the Kernel Density Graph.

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 spread 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 in accordance with:

$$Z_{(\text{target})} = (\text{result} - \text{average of PT}) / \text{target standard deviation}$$

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

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

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

4 EVALUATION

In this proficiency test, no serious problems were encountered during dispatch and execution. Only one laboratory (Brazil) received the samples late. Five laboratories reported the results after the final reporting date and one laboratory did not report any results at all.

The 42 reporting participants sent in 922 numerical results. Observed were 55 outlying results, which is 6.0% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D2086) and an added designation for the year that the method was adopted or revised (e.g. D2086-08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086-08 (2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

4.1 EVALUATION PER TEST

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

One should be aware that the sample “metals only” (#14221) contained a large number of elements, spectral interferences might explain part of the spread found for some elements.

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

Density @ 15°C: This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.

Flash Point PMcc: This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D93:13e1 method B.

Kin.Visco.@ 40°C: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D445:14e2.

Kin.Visco. @ 100°C: This determination was problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D445:14e2.

Visco. Stabinger @ 40°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D7042:14.

Visco. Stabinger @ 100°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D7042:14.

Pour Point (manual): This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D97:12. The rounding to 3 °C and the low number of test results may explain (part of) the large spread.

Pour Point (automated): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D5950:14.

Sulphur: 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 D4294:10.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D6304:07.

Water Separability: No significant conclusions were drawn as no results were reported. Twelve laboratories reported to have aborted the test.

Aluminium: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:13e1.

Barium: As the average concentration found by the group (8.34 mg/kg) is just above the application range given in ASTM D5185:13e1 table 3 (0.5 – 4 mg/kg), it was decided to use the estimated reproducibility calculated using the Horwitz equation instead of the reproducibility of ASTM D5185:13e1. This determination may be problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated requirements calculated using the Horwitz equation and the extrapolated reproducibility of ASTM D5185:13e1.

- Chromium: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:13e1.
- Copper: This determination was not problematic. Only one statistical outlier was observed and one result was excluded. However, the calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D5185:13e1.
- Iron: This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:13e1.
- Lead: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D5185:13e1.
- Lithium: This determination may be problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the estimated requirements calculated using the Horwitz equation.
- Magnesium: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:13e1.
- Manganese: This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:13e1.
- Molybdenum This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:13e1.
- Nickel: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:13e1.
- Sodium: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5185:13e1.

- Silicon: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5185:13e1.
- Silver: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5185:13e1.
- Tin: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5185:13e1.
- Titanium: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:13e1.
- Vanadium: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:13e1.
- Calcium: As the average concentration found by the group (13.07 mg/kg) is below the application range given in ASTM D5185:13e1 table 3 (40 – 9000 mg/kg), it was decided to use the estimated reproducibility calculated using the Horwitz equation instead of the reproducibility of ASTM D5185:13e1. This determination may be problematic for a number of laboratories. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is almost in agreement with the estimated requirements calculated using the Horwitz equation.
- Phosphorus: This determination was not problematic. One statistical outlier was observed and one result was excluded. The calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D5185:13e1.
- Zinc: This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:13e1.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and these parameters as found for the group of participating laboratories. The average results and the calculated reproducibilities are compared in the next tables with the reproducibilities, derived from literature standards (in casu the ASTM, IP, ISO and EN standards, see tables in appendix 1).

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R(lit)</i>
Total Acid Number	mg KOH/g	35	0.64	0.21	0.28
Density @ 15°C	kg/L	33	0.8744	0.0005	0.0005
Flash Point PMcc	°C	30	166.1	10.3	10.0
Kinematic viscosity @ 40°C	mm/s ²	33	41.350	0.358	0.314
Kinematic viscosity @ 100°C	mm/s ²	26	8.126	0.077	0.062
Viscosity Stabinger @ 40°C	mm/s ²	17	41.491	0.357	0.566
Viscosity Stabinger @ 100°C	mm/s ²	17	8.136	0.078	0.101
Pour Point (manual)	°C	13	-45.2	12.7	9.0
Pour Point (automated)	°C	8	-51.5	4.2	4.5
Sulphur	mg/kg	11	5904	873	517
Water	mg/kg	36	80	77	235

Table 5: reproducibilities of results of sample #14220.

<i>Parameter</i>	<i>Unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R (lit)</i>
Aluminium as Al	mg/kg	31	8.1	1.9	6.5
Barium as Ba	mg/kg	28	8.3	1.7	2.7
Chromium as Cr	mg/kg	32	7.7	1.6	2.8
Copper as Cu	mg/kg	33	31.8	7.4	7.6
Iron as Fe	mg/kg	32	9.2	1.7	3.1
Lead as Pb	mg/kg	33	12.3	3.3	7.4
Lithium as Li	mg/kg	10	8.9	3.6	2.9
Magnesium as Mg	mg/kg	30	8.6	2.3	3.8
Manganese as Mn	mg/kg	27	7.8	0.9	1.5
Molybdenum as Mo	mg/kg	29	7.8	1.8	2.7
Nickel as Ni	mg/kg	33	8.2	1.5	4.3
Sodium as Na	mg/kg	31	9.2	4.6	5.3
Silicon as Si	mg/kg	32	7.9	3.2	6.5
Silver as Ag	mg/kg	29	8.2	2.2	2.9
Tin as Sn	mg/kg	32	7.7	2.9	7.4
Titanium as Ti	mg/kg	26	7.5	1.1	6.5
Vanadium as V	mg/kg	32	7.9	1.8	2.7
Calcium as Ca	mg/kg	28	13.1	4.4	4.0
Phosphorus as P	mg/kg	32	341	72	79
Zinc as Zn	mg/kg	32	415	50	63

Table 6: reproducibilities of results of sample #14221

results between brackets to be used with care, result was lower (or above) than application range of reference method

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 standards. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2014 WITH THE PREVIOUS PTs.

	<i>November 2014</i>	<i>November 2013</i>	<i>November 2012</i>	<i>November 2011</i>
Number of reporting labs	42	42	40	36
Number of results reported	922	776	754	667
Statistical outliers	55	41	46	65
Percentage outliers	6.0%	5.3%	6.1%	9.7%

Table 7: 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 standards. The conclusions are given the following table:

Determination	November 2014	November 2013	November 2012	November 2011
Total Acid Number	++	(--)	+/-	--
Density @ 15°C	+/-	+/-	--	+/-
Flash Point PMcc	+/-	++	-	+/-
Kinematic viscosity @ 40°C	-	--	--	--
Kinematic viscosity @ 100°C	-	--	--	--
Viscosity Stabinger @ 40°C	++	n.e.	n.e.	n.e.
Viscosity Stabinger @ 100°C	++	n.e.	n.e.	n.e.
Pour Point (manual)	-	n.e.	n.e.	n.e.
Pour Point (automated)	+	n.e.	n.e.	n.e.
Sulphur	--	n.e.	n.e.	n.e.
Water	++	++	++	++
Aluminium as Al	++	(++)	++	+
Barium as Ba	++	-	(-)	(+)
Chromium as Cr	++	--	++	++
Copper as Cu	+/-	+	+/-	+
Iron as Fe	++	-	+/-	+/-
Lead as Pb	++	(++)	++	(+/-)
Lithium as Li	-	++	++	+
Magnesium as Mg	++	+	++	+/-
Manganese as Mn	++	(--)	--	--
Molybdenum as Mo	+	(--)	++	+
Nickel as Ni	++	(++)	++	++
Sodium as Na	+	-	--	(--)
Silicon as Si	++	+	++	(++)
Silver as Ag	+	--	--	+/-
Tin as Sn	++	(+)	++	(++)
Titanium as Ti	++	(++)	++	++
Vanadium as V	++	--	++	(-)
Calcium as Ca	-	--	--	(+/-)
Phosphorus as P	+	--	--	(-)
Zinc as Zn	++	-	--	(+/-)

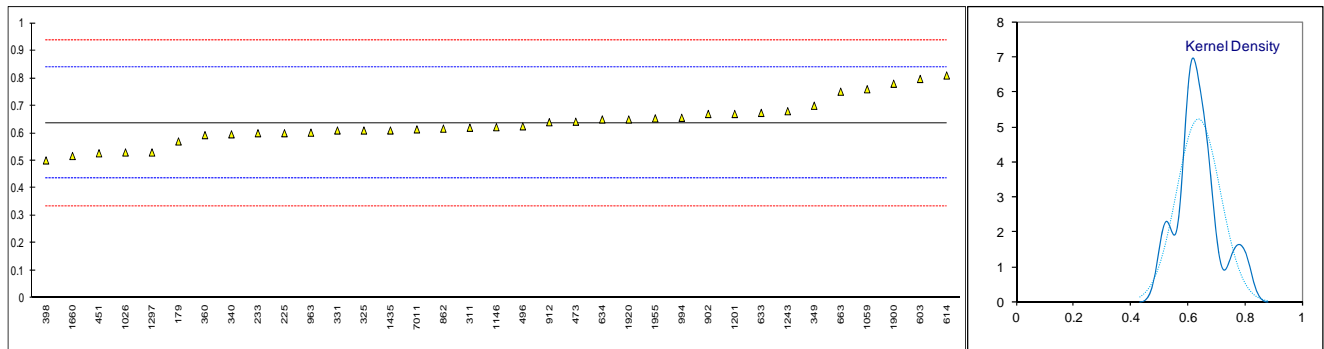
Table 8: comparison determinations against the standard

- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard

APPENDIX 1

Determination of Acid Number (Total) on sample #14220; results in mg KOH/g.

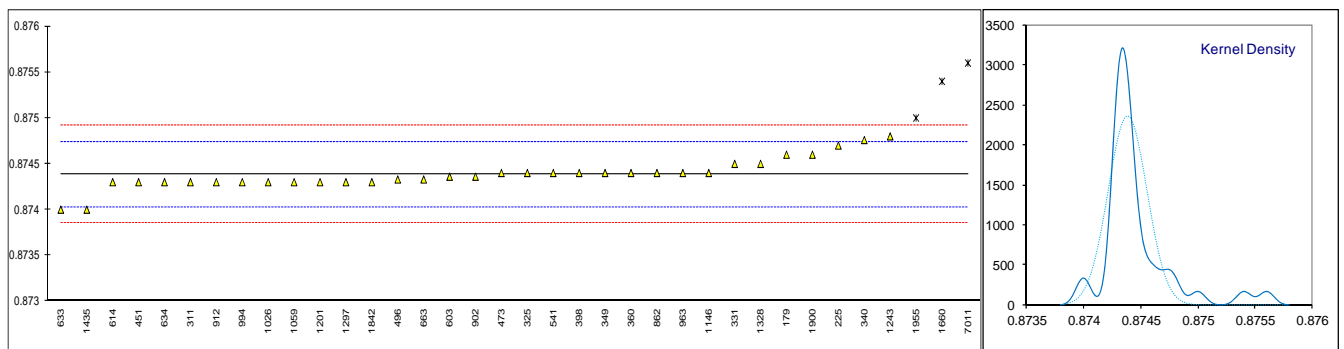
lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D664	0.57		-0.66	
225	D664	0.60		-0.37	
233	D664	0.60	C	-0.37	First reported 1.049
255		----		----	
271		----		----	
311	D664	0.62		-0.17	
325	D664	0.61		-0.27	
331	D664	0.61		-0.27	
340	D664	0.596		-0.41	
349	D664	0.70		0.63	
360	D974	0.593		-0.44	
398	D664	0.501		-1.35	
442		----		----	
451	D664	0.527		-1.09	
473	D664	0.6420		0.05	
496	D664	0.625		-0.12	
541		----		----	
551		----		----	
603	D664	0.7973		1.61	
614	D664	0.81		1.73	
633	D664	0.674		0.37	
634	D664	0.65	C	0.13	First reported 0.07
663	D664	0.751		1.14	
862	D664	0.6166		-0.20	
902	D664	0.670		0.33	
912	D664	0.64		0.03	
963	D974	0.602		-0.35	
994	D664	0.656		0.19	
1026	D664	0.53		-1.06	
1059	ISO6619	0.76		1.23	
1146	D664	0.622		-0.15	
1201	D664	0.67		0.33	
1243	D664	0.68		0.43	
1297	D664	0.53		-1.06	
1328		----		----	
1435	D664	0.61		-0.27	
1660	D664	0.517		-1.19	
1842		----		----	
1900	D664	0.78		1.43	
1920	D664	0.650		0.13	
1955	D664	0.654		0.17	
7011	D974	0.614		-0.23	
normality		OK			
n		35			
outliers		0			
mean (n)		0.6365			
st.dev. (n)		0.07638			
R(calc.)		0.2139			
R(D664:11a)		0.2801			



Determination of Density @ 15°C on sample #14220; results in kg/L.

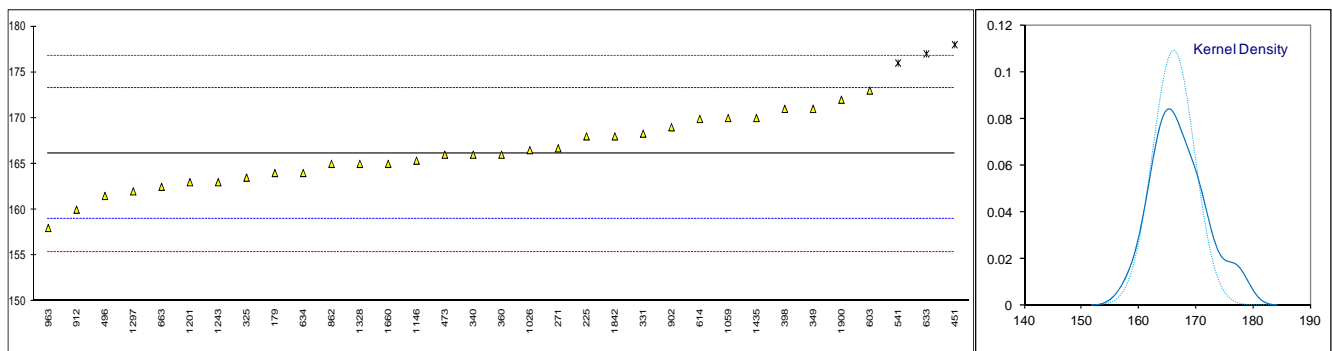
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4052	0.8746		1.20	
225	D4052	0.8747		1.76	
233		-----		-----	
255		-----		-----	
271		-----		-----	
311	D4052	0.8743		-0.48	
325	D4052	0.8744		0.08	
331	ISO12185	0.8745		0.64	
340	D4052	0.87476		2.10	
349	D4052	0.8744	C	0.08	First reported 0.8734
360	D4052	0.8744		0.08	
398	D4052	0.8744		0.08	
442		-----		-----	
451	D4052	0.8743	C	-0.48	First reported 874.3 (unit error)
473	D4052	0.8744		0.08	
496	D4052	0.87433		-0.31	
541	D4052	0.8744		0.08	
551		-----		-----	
603	D4052	0.87436		-0.14	
614	D4052	0.8743		-0.48	
633	D4052	0.8740	C	-2.16	First reported 874.0 (unit error)
634	D4052	0.8743		-0.48	
663	D4052	0.87433		-0.31	
862	D4052	0.8744		0.08	
902	D4052	0.87436		-0.14	
912	D4052	0.8743		-0.48	
963	D4052	0.8744		0.08	
994	D4052	0.8743		-0.48	
1026	D4052	0.8743		-0.48	
1059	ISO12185	0.8743		-0.48	
1146	D4052	0.8744		0.08	
1201	D4052	0.8743		-0.48	
1243	ISO12185	0.8748		2.32	
1297	D4052	0.8743		-0.48	
1328	GB/T1884	0.8745		0.64	
1435	D4052	0.874		-2.16	
1660	D7042	0.8754	R(0.01)	5.68	
1842	D4052	0.8743		-0.48	
1900	D4052	0.8746		1.20	
1920		-----		-----	
1955	D7042	0.8750	R(0.05)	3.44	
7011	D7042	0.8756	R(0.01)	6.80	

normality suspect
n 33
outliers 3
mean (n) 0.87439
st.dev. (n) 0.000169
R(calc.) 0.00047
R(ISO12185:96) 0.00050



Determination of Flash Point PMcc on sample #14220; results in °C.

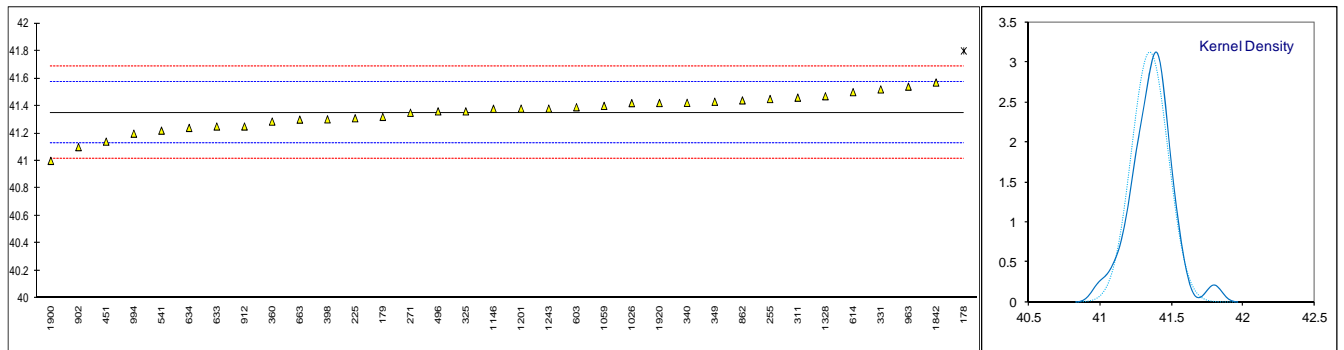
lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D93	164.0	C	-0.59	First reported 142
225	D93-A	168.0		0.53	
233		----		----	
255		----		----	
271	D93-A	166.7		0.17	
311		----		----	
325	D93-A	163.5		-0.73	
331	D93-A	168.3		0.61	
340	D93	166.0		-0.03	
349	D93-A	171		1.37	
360	D93-A	166.0		-0.03	
398	D93-A	171.0		1.37	
442		----		----	
451	D93-A	178	R(0.05)	3.33	
473	D93-A	166.0		-0.03	
496	D93-B	161.5		-1.29	
541	D93	176.0	R(0.05)	2.77	
551		----		----	
603	D3828	173.0		1.93	
614	D93-A	169.9		1.06	
633	D93-A	177.0	R(0.05)	3.05	
634	D93-A	164.0		-0.59	
663	D93-A	162.5		-1.01	
862	D93-A	165.0		-0.31	
902	D93-A	169.0		0.81	
912	D93-A	160.0		-1.71	
963	D93-B	158		-2.27	
994		----		----	
1026	D93-A	166.5		0.11	
1059	ISO2719	170.0		1.09	
1146	INH-93A	165.35		-0.21	
1201	D93-A	163.0		-0.87	
1243	D93-A	163		-0.87	
1297	D93-B	162		-1.15	
1328	GB/T261	165.0		-0.31	
1435	D93-A	170		1.09	
1660	D93-A	165.0		-0.31	
1842	D93-A	168		0.53	
1900	in house	172		1.65	
1920		----		----	
1955		----		----	
7011		----		----	
normality		OK			
n		30			
outliers		3			
mean (n)		166.11			
st.dev. (n)		3.663			
R(calc.)		10.26			
R(D93-B:13e1)		10.00			



Determination of Kinematic Viscosity @ 40°C on sample #14220; results in mm/s².

lab	method	value	mark	z(targ)	remarks
178	D445	41.8	R(0.05)	4.01	
179	D445	41.32		-0.27	
225	D445	41.31		-0.36	
233		----		----	
255	D7279	41.45	C	0.89	First reported 40.44
271	D445	41.35		0.00	
311	D445	41.46		0.98	
325	D445	41.36		0.09	
331	D7279	41.52		1.51	
340	D445	41.422		0.64	
349	D445	41.43		0.71	
360	D445	41.286		-0.57	
398	D445	41.302		-0.43	
442		----		----	
451	D7279	41.14		-1.87	
473		----		----	
496	D445	41.360		0.09	
541	D445	41.22		-1.16	
551		----		----	
603	D445	41.39		0.35	
614	D445	41.5		1.33	
633	D7279	41.25		-0.89	
634	D445	41.239850		-0.98	
663	D445	41.300		-0.45	
862	D445	41.44		0.80	
902	D445	41.10		-2.23	
912	D445	41.25		-0.89	
963	D445	41.54		1.69	
994	D445	41.198		-1.36	
1026	D445	41.42		0.62	
1059	ISO3104	41.40		0.44	
1146	D445	41.379		0.26	
1201	D445	41.38		0.27	
1243	D7279	41.38		0.27	
1297		----		----	
1328	GB/T265	41.47		1.07	
1435		----		----	
1660		----		----	
1842	IP71	41.57		1.96	
1900	D445	41.0		-3.12	
1920	D445	41.421		0.63	
1955		----		----	
7011		----		----	

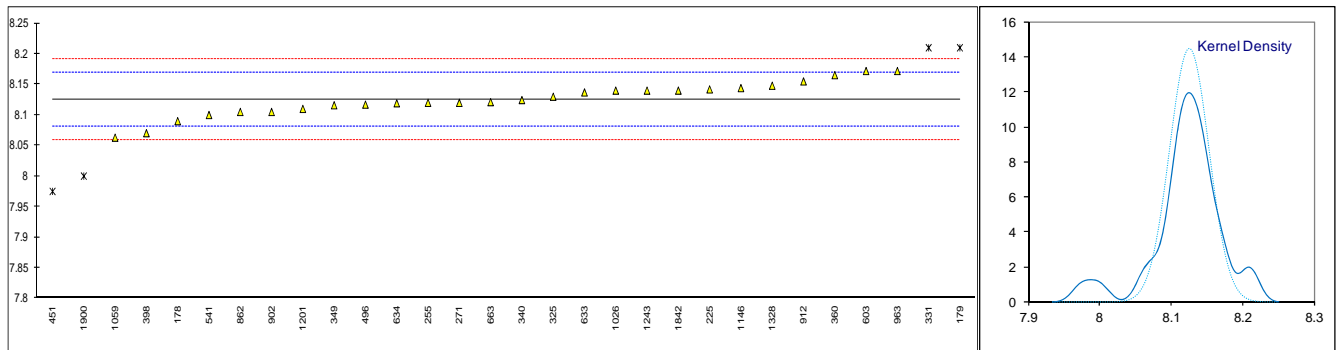
normality OK
n 33
outliers 1
mean (n) 41.3502
st.dev. (n) 0.12767
R(calc.) 0.3575
R(D445:14e2) 0.3143



Determination of Kinematic Viscosity @ 100°C on sample #14220; results in mm/s².

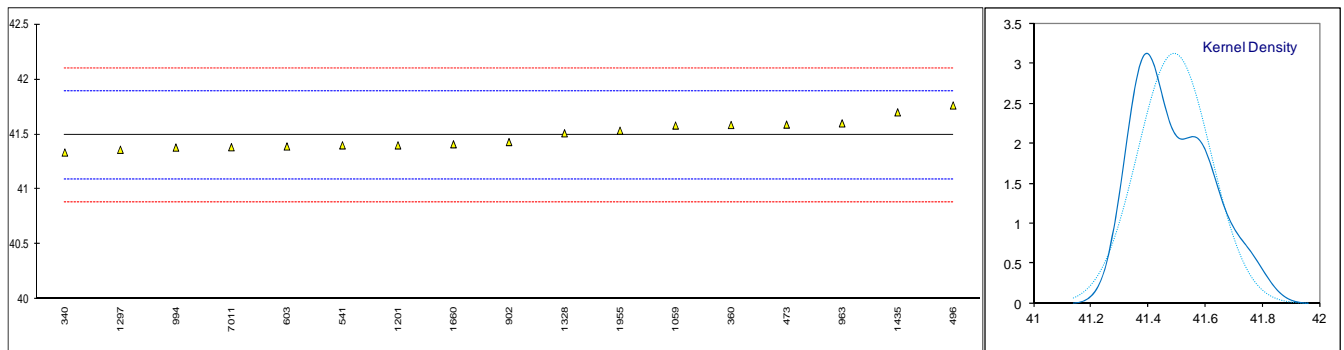
lab	method	value	mark	z(targ)	remarks
178	D445	8.09		-1.62	
179	D445	8.21	R(0.05)	3.83	
225	D445	8.142		0.74	
233		----		----	
255	D7279	8.12	C	-0.26	First reported 7.105
271	D445	8.12		-0.26	
311		----		----	
325	D445	8.130		0.20	
331	D7279	8.21	R(0.05)	3.83	
340	D445	8.1245		-0.05	
349	D445	8.116		-0.44	
360	D445	8.1653		1.80	
398	D445	8.0703		-2.51	
442		----		----	
451	D7279	7.975	R(0.05)	-6.83	
473		----		----	
496	D445	8.1170		-0.39	
541	D445	8.100		-1.16	
551		----		----	
603	D445	8.172		2.10	
614		----		----	
633	D7279	8.137		0.52	
634	D445	8.119239		-0.29	
663	D445	8.1211		-0.21	
862	D445	8.105		-0.94	
902	D445	8.105		-0.94	
912	D445	8.155		1.33	
963	D445	8.172		2.10	
994		----		----	
1026	D445	8.14		0.65	
1059	ISO3104	8.063		-2.84	
1146	D445	8.1441		0.84	
1201	D445	8.110		-0.71	
1243	D7279	8.14		0.65	
1297		----		----	
1328	GB/T265	8.148		1.01	
1435		----		----	
1660		----		----	
1842	IP71	8.140		0.65	
1900	D445	8.00	R(0.05)	-5.70	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 26
outliers 4
mean (n) 8.1256
st.dev. (n) 0.02751
R(calc.) 0.0770
R(D445:14e2) 0.0618



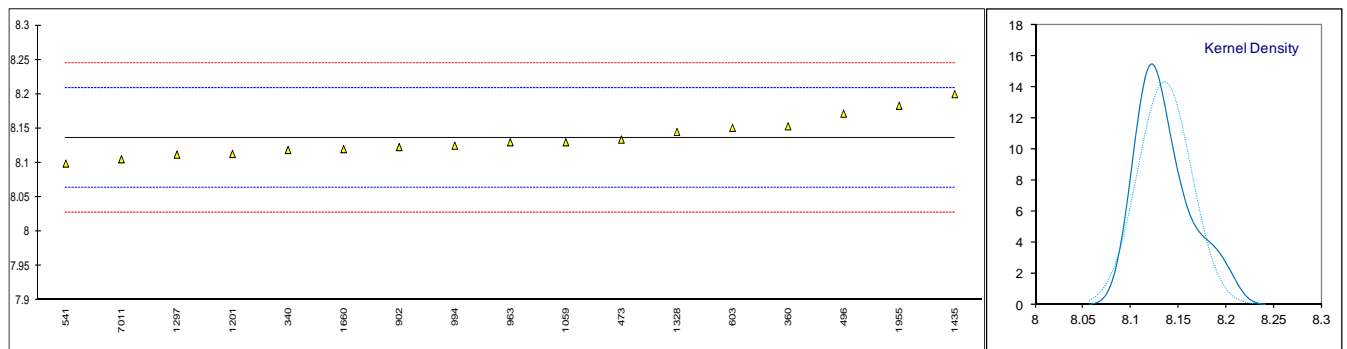
Determination of Viscosity Stabinger @ 40°C on sample #14220; results in mm/s².

lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
225		----		----	
233		----		----	
255		----		----	
271		----		----	
311		----		----	
325		----		----	
331		----		----	
340	D7042	41.335		-0.77	
349		----		----	
360	D7042	41.585		0.46	
398		----		----	
442		----		----	
451		----		----	
473	D7042	41.589		0.48	
496	D7042	41.763		1.34	
541	D7042	41.40		-0.45	
551		----		----	
603	D7042	41.39		-0.50	
614		----		----	
633		----		----	
634		----		----	
663		----		----	
862		----		----	
902	D7042	41.43		-0.30	
912		----		----	
963	D7042	41.60		0.54	
994	D7042	41.38		-0.55	
1026		----		----	
1059	D7042	41.58		0.44	
1146		----		----	
1201	D7042	41.40		-0.45	
1243		----		----	
1297	D7042	41.36		-0.65	
1328	SH/T0870	41.51		0.09	
1435	D7042	41.7		1.03	
1660	D7042	41.41		-0.40	
1842		----		----	
1900		----		----	
1920		----		----	
1955	D7042	41.5335		0.21	
7011	D7042	41.383		-0.53	
normality		OK			
n		17			
outliers		0			
mean (n)		41.4911			
st.dev. (n)		0.12762			
R(calc.)		0.3573			
R(D7042:14)		0.5662			



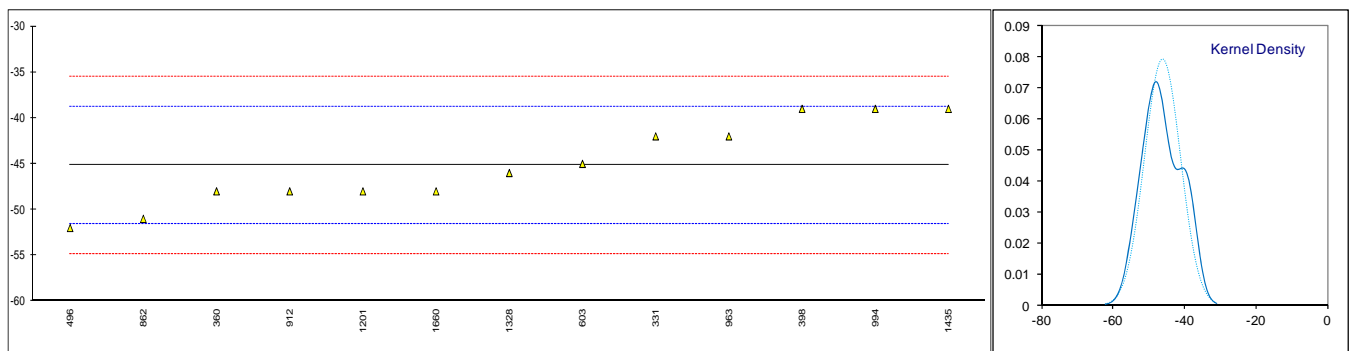
Determination of Viscosity Stabinger @ 100°C on sample #14220; results in mm/s².

lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
225		----		----	
233		----		----	
255		----		----	
271		----		----	
311		----		----	
325		----		----	
331		----		----	
340	D7042	8.1188		-0.48	
349		----		----	
360	D7042	8.1532		0.47	
398		----		----	
442		----		----	
451		----		----	
473	D7042	8.1339		-0.06	
496	D7042	8.1716		0.98	
541	D7042	8.099		-1.03	
551		----		----	
603	D7042	8.151		0.41	
614		----		----	
633		----		----	
634		----		----	
663		----		----	
862		----		----	
902	D7042	8.123		-0.36	
912		----		----	
963	D7042	8.130		-0.17	
994	D7042	8.125		-0.31	
1026		----		----	
1059	D7042	8.130		-0.17	
1146		----		----	
1201	D7042	8.113		-0.64	
1243		----		----	
1297	D7042	8.112		-0.67	
1328	SH/T0870	8.145		0.25	
1435	D7042	8.2		1.77	
1660	D7042	8.12		-0.45	
1842		----		----	
1900		----		----	
1920		----		----	
1955	D7042	8.1831		1.30	
7011	D7042	8.1053		-0.85	
normality		OK			
n		17			
outliers		0			
mean (n)		8.1361			
st.dev. (n)		0.02797			
R(calc.)		0.0783			
R(D7042:14)		0.1009			



Determination of Pour Point, manual on sample #14220; results in °C.

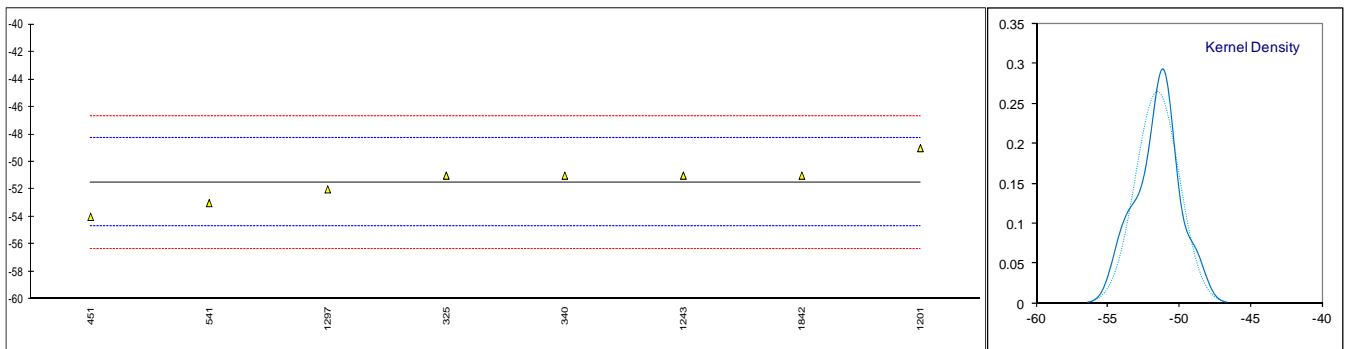
lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
225	D97	<-30		----	
233		----		----	
255		----		----	
271		----		----	
311		----		----	
325		----		----	
331	ISO3016	-42	C	0.98	Reported result as automated, test method is manual
340		----		----	
349		----		----	
360	D97	-48		-0.89	
398	D97	-39		1.91	
442		----		----	
451		----		----	
473		----		----	
496	D97	-52		-2.13	
541		----		----	
551		----		----	
603	D97	-45		0.05	
614		----		----	
633		----		----	
634		----		----	
663	D97	<-39		----	
862	D97	-51		-1.82	
902		----		----	
912	D97	-48		-0.89	
963	D97	-42		0.98	
994	D97	-39		1.91	
1026	D97	<-42		----	
1059	ISO3016	<-42		----	
1146		----		----	
1201	D97	-48		-0.89	
1243		----		----	
1297		----		----	
1328	GB/T3535	-46		-0.26	
1435	D97	-39		1.91	
1660	D97	-48		-0.89	
1842		----		----	
1900		----		----	
1920		----		----	
1955		----		----	
7011		----		----	
normality		OK			
n		13			
outliers		0			
mean (n)		-45.15			
st.dev. (n)		4.543			
R(calc.)		12.72			
R(D97:12)		9.00			



Determination of Pour Point, automated, 1°C interval on sample #14220; results in °C.

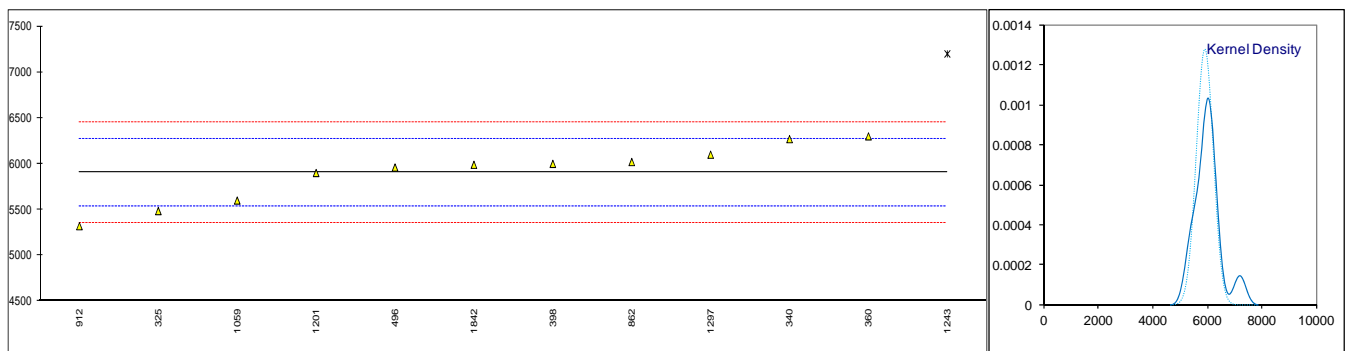
lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
225		----		----	
233		----		----	
255		----		----	
271		----		----	
311		----		----	
325	D5950	-51		0.31	
331		----		----	
340	D5950	-51.0		0.31	
349		----		----	
360		----		----	
398		----		----	
442		----		----	
451	D5949	-54	C	-1.56	Reported result as manual, test method is automated
473		----		----	
496		----		----	
541	D5950	-53		-0.93	
551		----		----	
603		----		----	
614		----		----	
633		----		----	
634		----		----	
663		----		----	
862		----		----	
902		----		----	
912		----		----	
963		----		----	
994		----		----	
1026	D5950	<-45		----	
1059		----		----	
1146		----		----	
1201	D5950	-49		1.56	
1243	D5950	-51		0.31	
1297	D5950	-52		-0.31	
1328		----		----	
1435		----		----	
1660		----		----	
1842	D5950	-51		0.31	
1900		----		----	
1920		----		----	
1955		----		----	
7011		----		----	

normality unknown
n 8
outliers 0
mean (n) -51.50
st.dev. (n) 1.512
R(calc.) 4.23
R(D5950:14) 4.50



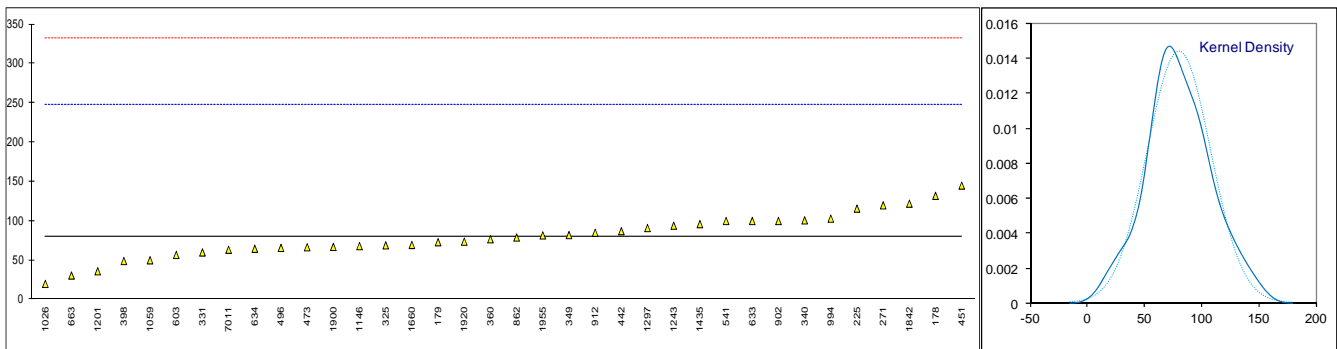
Determination of Sulphur on sample #14220; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
225		----		----	
233		----		----	
255		----		----	
271		----		----	
311		----		----	
325	INH-6443	5485		-2.27	
331		----		----	
340	ISO8754	6270		1.98	
349		----		----	
360	D4294	6300		2.14	
398	D4294	6000		0.52	
442		----		----	
451		----		----	
473		----		----	
496	D2622	5960		0.30	
541		----		----	
551		----		----	
603		----		----	
614		----		----	
633		----		----	
634		----		----	
663		----		----	
862	D2622	6020		0.63	
902		----		----	
912	D5185	5320		-3.16	
963		----		----	
994		----		----	
1026		----		----	
1059	ISO8754	5600		-1.65	
1146		----		----	
1201	ISO8754	5900	C	-0.02	First reported 0.5900 (unit error)
1243	ISO8754	7200	G(0.05)	7.01	
1297	D4294	6100		1.06	
1328		----		----	
1435		----		----	
1660		----		----	
1842	D1266	5990		0.46	
1900		----		----	
1920		----		----	
1955		----		----	
7011		----		----	
normality	OK				
n	11				
outliers	1				
mean (n)	5904.09				
st.dev. (n)	311.679				
R(calc.)	872.70				
R(D4294:10)	517.34				



Determination of Water on sample #14220; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D6304	132		0.62	
179	D6304-C	73		-0.09	
225	D6304-A	115.72		0.42	
233		----		----	
255		----		----	
271	D6304	119.9		0.47	
311		----		----	
325	D6304-C	69		-0.14	
331	D6304-C	60.0		-0.24	
340	D6304-A	100.8		0.24	
349	D6304-A	82.05		0.02	
360	D6304-A	76.7		-0.04	
398	D6304-C	49.05		-0.37	
442	IP438	87		0.08	
451	D6304-C	145		0.77	
473	D6304-C	66.6		-0.16	
496	D6304-C	65.8		-0.17	
541	D6304-A	100		0.23	
551		----		----	
603	D6304-C	56.8		-0.28	
614		----		----	
633	D6304-A	100		0.23	
634	D6304-A	64.6467		-0.19	
663	D6304-C	30.6		-0.59	
862	D6304-C	79		-0.02	
902	D6304-A	100		0.23	
912	D6304-C	85		0.06	
963		----		----	
994	D6304-C	103		0.27	
1026	D6304-C	20		-0.72	
1059	ISO12937	50		-0.36	
1146	D6304-C	68		-0.15	
1201	D6304-C	36		-0.53	
1243	ISO12937	94		0.16	
1297	D6304-A	91.0		0.13	
1328		----		----	
1435	D1744	96		0.19	
1660	IEC60814	69.5		-0.13	
1842	D6304-A	122		0.50	
1900	D6304-C	67		-0.16	
1920	D6304-C	73.7		-0.08	
1955	D6304-A	81.66		0.02	
7011	D6304	63.4		-0.20	
normality		OK			
n		36			
outliers		0			
mean (n)		80.39			
st.dev. (n)		27.610			
R(calc.)		77.31			
R(D6304:07)		234.85			



Determination of Water Separability at 54 °C on sample #14220; results in min.

lab	method	time to reach ≤3 ml emul.	mark	time to reach 37 ml water	mark	time to reach complete break	mark	test aborted	mark
178		----		----		----		----	
179	D1401	----		----		30		----	
225		----		----		----		----	
233		----		----		----		----	
255		----		----		----		----	
271		----		----		----		----	
311		----		----		----		----	
325	D1401	>60		>60		>60		Yes, at 60 min	
331		----		----		----		Yes	
340		----		----		----		----	
349		----		----		----		----	
360		----		----		----		Yes	
398	D1401	Not Reached		Not Reached		Not Reached		Yes	
442		----		----		----		----	
451		----		----		----		Yes, test limit exc.	
473		----		----		----		----	
496		----		----		----		----	
541		----		----		----		----	
551		----		----		----		----	
603		----		----		----		----	
614		----		----		----		Yes	
633		----		----		----		----	
634		----		----		----		----	
663		----		----		----		----	
862		----		----		----		Yes	
902		----		----		----		----	
912		----		----		----		Yes	
963		----		----		----		----	
994		----		----		----		----	
1026		----		----		----		----	
1059		----		----		----		----	
1146		----		----		----		Yes, 30	
1201		----		----		----		Yes	
1243		----		----		----		Yes	
1297		----		----		----		----	
1328		----		----		----		----	
1435		----		----		----		Yes, at 60 min	
1660		----		----		----		----	
1842		----		----		----		----	
1900		----		----		----		----	
1920		----		----		----		----	
1955		----		----		----		----	
7011		----		----		----		----	
	normality	n.a.		n.a.		n.a.			
	n	n.a.		n.a.		n.a.			
	outliers	n.a.		n.a.		n.a.			
	mean (n)	n.a.		n.a.		n.a.			
	st.dev. (n)	n.a.		n.a.		n.a.			
	R(calc.)	n.a.		n.a.		n.a.			
	R(D1401:12)	n.a.		n.a.		n.a.			

Determination of Water Separability at 54 °C on sample #14220; results in ml.

--- Continued ---

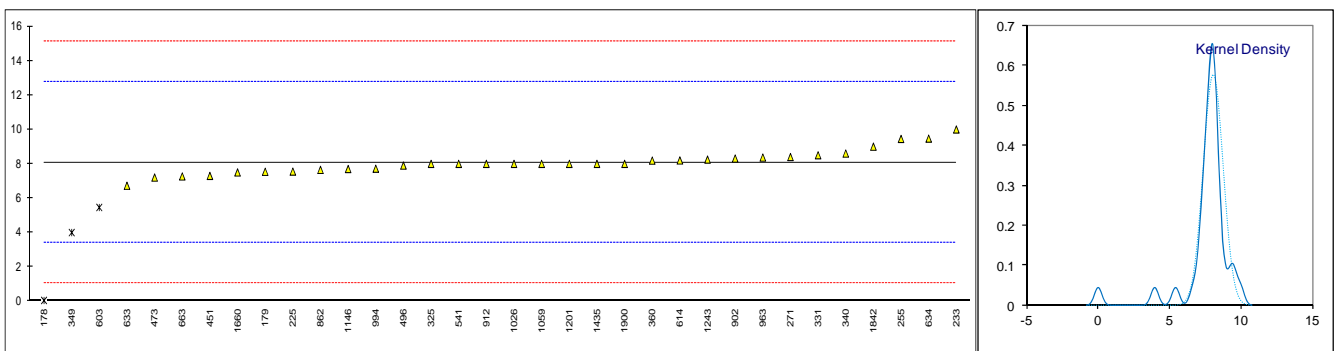
lab	method	volume oil phase	mark	volume water phase	mark	volume emul. phase	mark
178		----		----		----	
179	D1401	2		1		77	
225		----		----		----	
233		----		----		----	
255		----		----		----	
271		----		----		----	
311		----		----		----	
325	D1401	0		0		80	
331	D1401	1		1		78	
340	D1401	1.0		5.0		74.0	
349		----		----		----	
360	D1401	1		29		50	
398	D1401	1		0		79	
442		----		----		----	
451		----		----		----	
473		----		----		----	
496		----		----		----	
541		----		----		----	
551		----		----		----	
603		----		----		----	
614	D1401	2		0		78	
633		----		----		----	
634		----		----		----	
663		----		----		----	
862	D1401	1		0		79	
902		----		----		----	
912	D1401	0		0		80	
963		----		----		----	
994		----		----		----	
1026	D1401	0		0		80	
1059		----		----		----	
1146	D1401	2		0		78	
1201	D1401	1		5		74	
1243	D1401	1		0		79	
1297		----		----		----	
1328		----		----		----	
1435		1		3		76	
1660		----		----		----	
1842		----		----		----	
1900		----		----		----	
1920		----		----		----	
1955		----		----		----	
7011		----		----		----	

Determination of Aluminum (Al) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.05	R(0.01)	-3.44	
179	D5185	7.54		-0.24	
225	D6595	7.55		-0.23	
233	D6595	10		0.82	
255	INH-021	9.45		0.58	
271	D5185	8.40		0.13	
311		----		----	
325	D5185	8		-0.04	
331	D5185	8.5		0.18	
340	D5185	8.6		0.22	
349	D5185	4	R(0.01)	-1.75	
360	D5185	8.19		0.04	
398		----		----	
442		----		----	
451	D5185	7.3		-0.34	
473	D5185	7.203		-0.38	
496	DIN51399	7.9		-0.08	
541	D5185	8		-0.04	
551		----		----	
603	D5185	5.461	R(0.05)	-1.12	
614	D5185	8.2		0.05	
633	D6595	6.726		-0.58	
634	D6595	9.469		0.59	
663	D5185	7.27		-0.35	
862	D5185	7.650		-0.19	
902	D5185	8.313		0.10	
912	D5185	8.0		-0.04	
963	D5185	8.369		0.12	
994	D5185	7.72		-0.16	
1026	D5185	8		-0.04	
1059	in house	8		-0.04	
1146	in house	7.7		-0.17	
1201	D5185	8		-0.04	
1243	D5185	8.25		0.07	
1297		----		----	
1328		----		----	
1435	D5185	8		-0.04	
1660	D5185	7.5		-0.25	
1842	INH-01	9		0.39	
1900	D6596	8		-0.04	
1920		----		----	
1955		----		----	
7011		----		----	

normality suspect
n 31
outliers 3
mean (n) 8.090
st.dev. (n) 0.6927
R(calc.) 1.940
R(D5185:13e1) 6.544

Application range:6 – 40 mg/kg

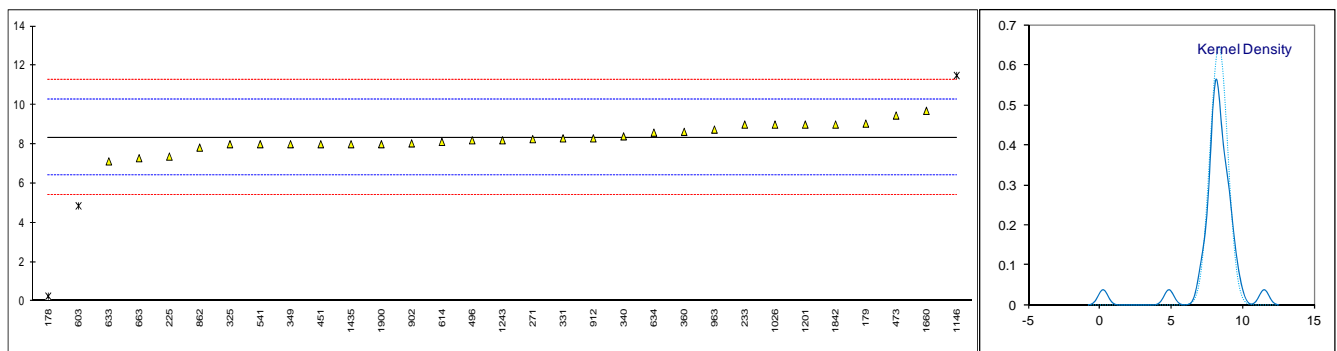


Determination of Barium (Ba) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.26	R(0.01)	-8.33	
179	D5185	9.05		0.73	
225	D6595	7.37		-1.00	
233	D6595	9		0.68	
255		----		----	
271	D5185	8.26		-0.09	
311		----		----	
325	D5185	8		-0.35	
331	D5185	8.3		-0.04	
340	D5185	8.4		0.06	
349	D5185	8		-0.35	
360	D5185	8.63		0.30	
398		----		----	
442		----		----	
451	D5185	8.0		-0.35	
473	D5185	9.462		1.15	
496	DIN51399	8.2		-0.15	
541	D5185	8		-0.35	
551		----		----	
603	D5185	4.860	R(0.01)	-3.59	
614	D5185	8.12		-0.23	
633	D6595	7.127		-1.25	
634	D6595	8.590		0.25	
663	D5185	7.29		-1.09	
862	D5185	7.828		-0.53	
902	D5185	8.043		-0.31	
912	D5185	8.30		-0.04	
963	D5185	8.745		0.41	
994		----		----	
1026	D5185	9		0.68	
1059		----		----	
1146	in house	11.5	R(0.01)	3.25	
1201	D5185	9		0.68	
1243	D5185	8.2		-0.15	
1297		----		----	
1328		----		----	
1435	D5185	8		-0.35	
1660	D5185	9.7		1.40	
1842	INH-01	9		0.68	
1900	D6595	8		-0.35	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 28
outliers 3
mean (n) 8.343
st.dev. (n) 0.6184
R(calc.) 1.731
R(Horwitz) 2.716

Compare R(D5185:13e1) = 4.154; range 0.5 – 4 ppm

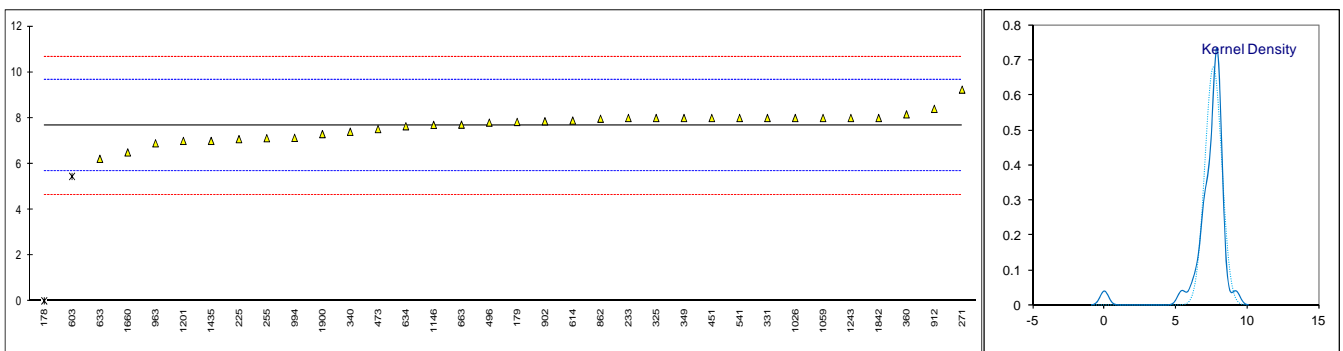


Determination of Chromium (Cr) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.02	R(0.01)	-7.63	
179	D5185	7.83		0.16	
225	D6595	7.08		-0.59	
233	D6595	8		0.33	
255	INH-021	7.12		-0.55	
271	D5185	9.24		1.57	
311		----		----	
325	D5185	8		0.33	
331	D5185	8.0		0.33	
340	D5185	7.4		-0.27	
349	D5185	8		0.33	
360	D5185	8.16		0.49	
398		----		----	
442		----		----	
451	D5185	8.0		0.33	
473	D5185	7.519		-0.15	
496	DIN51399	7.8		0.13	
541	D5185	8		0.33	
551		----		----	
603	D5185	5.450	R(0.05)	-2.21	
614	D5185	7.89		0.22	
633	D6595	6.219		-1.45	
634	D6595	7.642		-0.03	
663	D5185	7.71		0.04	
862	D5185	7.968		0.30	
902	D5185	7.859		0.19	
912	D5185	8.4		0.73	
963	D5185	6.897		-0.77	
994	D5185	7.134		-0.53	
1026	D5185	8		0.33	
1059	in house	8		0.33	
1146	in house	7.7		0.03	
1201	D5185	7		-0.67	
1243	D5185	8.0		0.33	
1297		----		----	
1328		----		----	
1435	D5185	7		-0.67	
1660	D5185	6.5		-1.17	
1842	INH-01	8		0.33	
1900	D6595	7.3		-0.37	
1920		----		----	
1955		----		----	
7011		----		----	

normality suspect
n 32
outliers 2
mean (n) 7.668
st.dev. (n) 0.5871
R(calc.) 1.644
R(D5185:13e1) 2.806

Application range:1 – 40 mg/kg

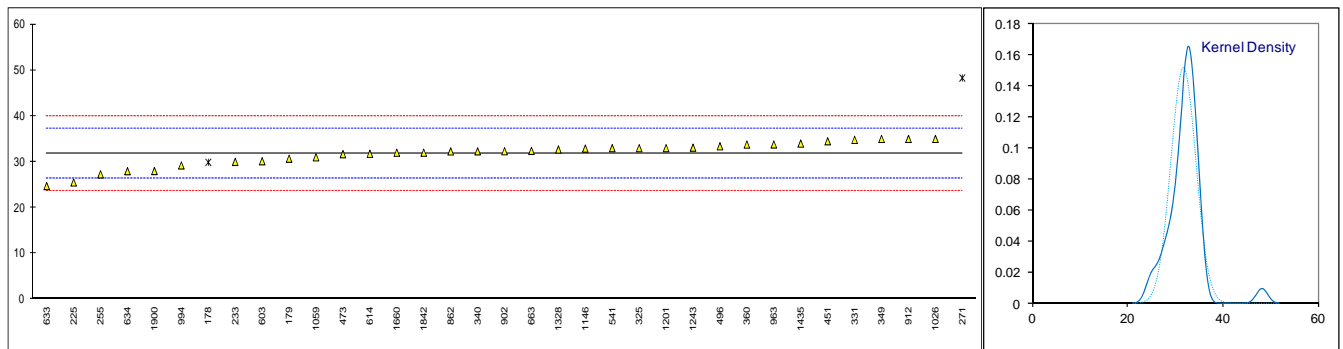


Determination of Copper (Cu) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	29.90	ex	-0.69	Result excluded, see §4.1
179	D5185	30.69		-0.39	
225	D6595	25.50		-2.30	
233	D6595	30		-0.65	
255	INH-021	27.28		-1.65	
271	D5185	48.3	C,R(0.01)	6.07	First reported 42.96
311		----		----	
325	D5185	33		0.45	
331	D5185	34.8		1.11	
340	D5185	32.3		0.20	
349	D5185	35		1.19	
360	D5185	33.78		0.74	
398		----		----	
442		----		----	
451	D5185	34.5		1.00	
473	D5185	31.68		-0.03	
496	DIN51399	33.4		0.60	
541	D5185	33		0.45	
551		----		----	
603	D5185	30.14		-0.60	
614	D5185	31.76		0.00	
633	D6595	24.721		-2.59	
634	D6595	27.965		-1.40	
663	D5185	32.4		0.23	
862	D5185	32.29		0.19	
902	D5185	32.32		0.20	
912	D5185	35.0		1.19	
963	D5185	33.800		0.75	
994	D5185	29.23		-0.93	
1026	D5185	35		1.19	
1059	in house	31		-0.28	
1146	in house	32.9		0.42	
1201	D5185	33		0.45	
1243	D5185	33.1		0.49	
1297		----		----	
1328	GB/T17476	32.7		0.34	
1435	D5185	34		0.82	
1660	D5185	32.0		0.09	
1842	INH-01	32		0.09	
1900	D6595	28		-1.38	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 33
outliers 1 (+1 excl)
mean (n) 31.765
st.dev. (n) 2.6539
R(calc.) 7.431
R(D5185:13e1) 7.624

Application range:2 –160 mg/kg

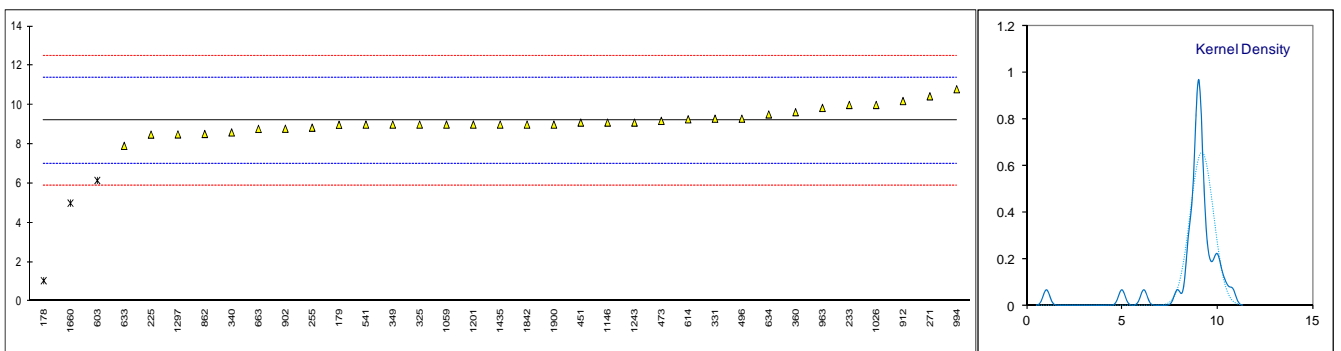


Determination of Iron (Fe) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	1.05	R(0.01)	-7.43	
179	D5185	8.99		-0.19	
225	D6595	8.49		-0.64	
233	D6595	10		0.74	
255	INH-021	8.843		-0.32	
271	D5185	10.44		1.14	
311		----		----	
325	D5185	9		-0.18	
331	D5185	9.3		0.10	
340	D5185	8.6		-0.54	
349	D5185	9		-0.18	
360	D5185	9.63		0.40	
398		----		----	
442		----		----	
451	D5185	9.1		-0.09	
473	D5185	9.189		-0.01	
496	DIN51399	9.3		0.10	
541	D5185	9		-0.18	
551		----		----	
603	D5185	6.153	R(0.01)	-2.78	
614	D5185	9.27		0.07	
633	D6595	7.916		-1.17	
634	D6595	9.517		0.29	
663	D5185	8.78		-0.38	
862	D5185	8.523		-0.61	
902	D5185	8.789		-0.37	
912	D5185	10.2		0.92	
963	D5185	9.848		0.60	
994	D5185	10.8		1.47	
1026	D5185	10		0.74	
1059	ISO14597Mod.	9		-0.18	
1146	in house	9.1		-0.09	
1201	D5185	9		-0.18	
1243	D5185	9.1		-0.09	
1297	D5708	8.5		-0.63	
1328		----		----	
1435	D5185	9		-0.18	
1660	D5185	5.0	R(0.01)	-3.83	
1842	INH-01	9		-0.18	
1900	D6595	9		-0.18	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 32
outliers 3
mean (n) 9.195
st.dev. (n) 0.6073
R(calc.) 1.701
R(D5185:13e1) 3.068

Application range:2 – 140 mg/kg

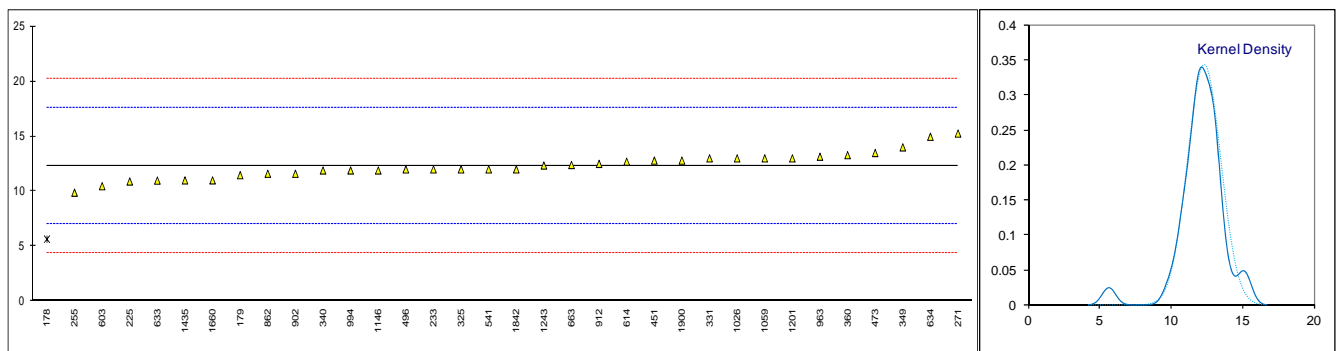


Determination of Lead (Pb) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	5.65	R(0.01)	-2.52	
179	D5185	11.47		-0.32	
225	D6595	10.90		-0.53	
233	D6595	12		-0.12	
255	INH-021	9.88		-0.92	
271	D5185	15.26		1.11	
311		----		----	
325	D5185	12		-0.12	
331	D5185	13.0		0.26	
340	D5185	11.9		-0.16	
349	D5185	14		0.64	
360	D5185	13.29		0.37	
398		----		----	
442		----		----	
451	D5185	12.8		0.18	
473	D5185	13.49		0.45	
496	DIN51399	12.0		-0.12	
541	D5185	12		-0.12	
551		----		----	
603	D5185	10.47		-0.70	
614	D5185	12.7		0.15	
633	D6595	10.979		-0.50	
634	D6595	14.962		1.00	
663	D5185	12.4		0.03	
862	D5185	11.60		-0.27	
902	D5185	11.60		-0.27	
912	D5185	12.5		0.07	
963	D5185	13.150		0.32	
994	D5185	11.9		-0.16	
1026	D5185	13		0.26	
1059	in house	13		0.26	
1146	in house	11.9		-0.16	
1201	D5185	13		0.26	
1243	D5185	12.35		0.01	
1297		----		----	
1328		----		----	
1435	D5185	11		-0.50	
1660	D5185	11.0		-0.50	
1842	INH-01	12		-0.12	
1900	D6595	12.8		0.18	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 33
outliers 1
mean (n) 12.312
st.dev. (n) 1.1648
R(calc.) 3.261
R(D5185:13e1) 7.410

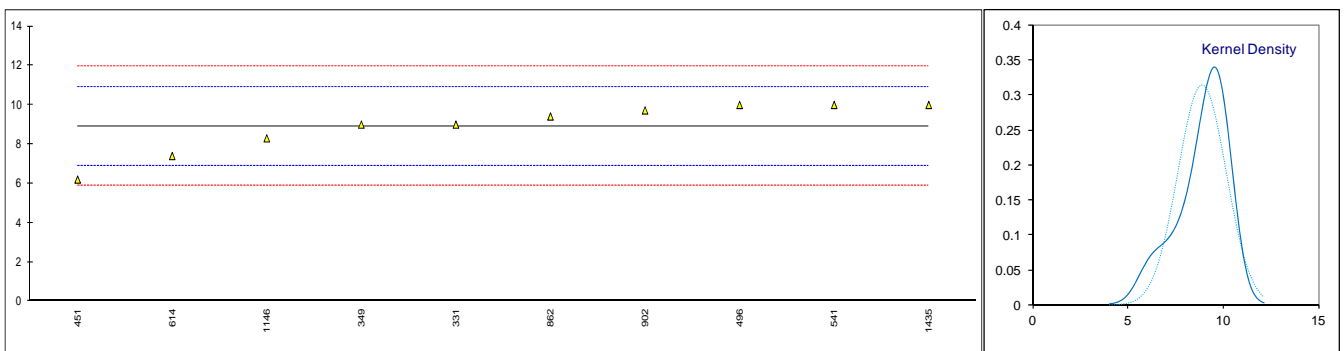
Application range:10 – 160 mg/kg



Determination of Lithium (Li) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
225		----		----	
233		----		----	
255		----		----	
271		----		----	
311		----		----	
325		----		----	
331	D5185	9.0		0.09	
340		----		----	
349	D5185	9		0.09	
360		----		----	
398		----		----	
442		----		----	
451	in house	6.2		-2.64	
473		----		----	
496	DIN51399	10.0		1.07	
541	D5185	10		1.07	
551		----		----	
603		----		----	
614	D5185	7.4		-1.47	
633		----		----	
634		----		----	
663		----		----	
862	D5185	9.413		0.50	
902	D5185	9.722		0.80	
912		----		----	
963		----		----	
994		----		----	
1026		----		----	
1059		----		----	
1146	in house	8.3		-0.59	
1201		----		----	
1243		----		----	
1297		----		----	
1328		----		----	
1435		10		1.07	
1660		----		----	
1842		----		----	
1900		----		----	
1920		----		----	
1955		----		----	
7011		----		----	

normality suspect
n 10
outliers 0
mean (n) 8.904
st.dev. (n) 1.2688
R(calc.) 3.553
R(Horwitz) 2.870

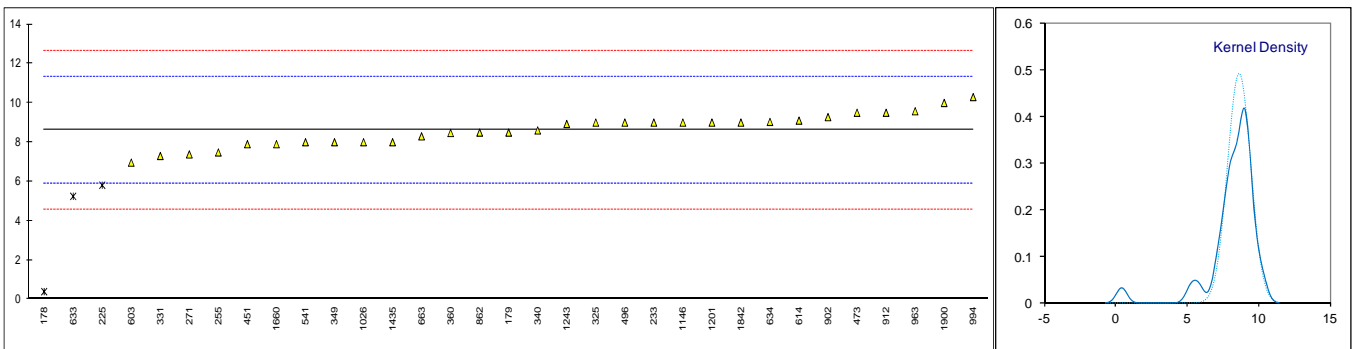


Determination of Magnesium (Mg) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.39	R(0.01)	-6.09	
179	D5185	8.49		-0.09	
225	D6595	5.81	DG(0.01)	-2.08	
233	D6595	9		0.28	
255	INH-021	7.48		-0.84	
271	D5185	7.38		-0.92	
311		----		----	
325	D5185	9		0.28	
331	D5185	7.3		-0.98	
340	D5185	8.6		-0.01	
349	D5185	8		-0.46	
360	D5185	8.47		-0.11	
398		----		----	
442		----		----	
451	D5185	7.9		-0.53	
473	D5185	9.500		0.65	
496	DIN51399	9.0		0.28	
541	D5185	8		-0.46	
551		----		----	
603	D5185	6.961		-1.23	
614	D5185	9.1		0.36	
633	D6595	5.244	DG(0.01)	-2.50	
634	D6595	9.036		0.31	
663	D5185	8.30		-0.23	
862	D5185	8.489		-0.09	
902	D5185	9.279		0.49	
912	D5185	9.50		0.65	
963	D5185	9.578		0.71	
994	D5185	10.3		1.25	
1026	D5185	8		-0.46	
1059	in house	<50		----	
1146	in house	9.0		0.28	
1201	D5185	9		0.28	
1243	D5185	8.93		0.23	
1297		----		----	
1328		----		----	
1435	D5185	8		-0.46	
1660	D5185	7.9		-0.53	
1842	INH-01	9		0.28	
1900	D6595	10		1.02	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 30
outliers 3
mean (n) 8.616
st.dev. (n) 0.8107
R(calc.) 2.270
R(D5185:13e1) 3.780

Application range:5 – 1700 mg/kg

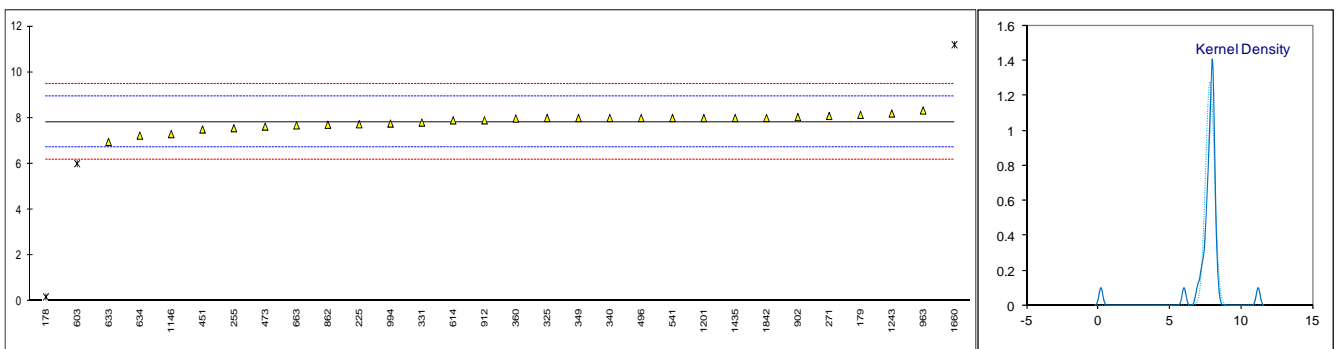


Determination of Manganese (Mn) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.19	R(0.01)	-13.92	
179	D5185	8.14		0.56	
225	D6595	7.73		-0.18	
233		----		----	
255	INH-021	7.556		-0.50	
271	D5185	8.09		0.47	
311		----		----	
325	D5185	8		0.31	
331	D5185	7.8		-0.06	
340	D5185	8.0		0.31	
349	D5185	8		0.31	
360	D5185	7.98		0.27	
398		----		----	
442		----		----	
451	D5185	7.5		-0.60	
473	D5185	7.627		-0.37	
496	DIN51399	8.0		0.31	
541	D5185	8		0.31	
551		----		----	
603	D5185	6.010	R(0.01)	-3.32	
614	D5185	7.9		0.13	
633	D6595	6.957		-1.59	
634	D6595	7.234		-1.09	
663	D5185	7.68		-0.27	
862	D5185	7.708		-0.22	
902	D5185	8.042		0.38	
912	D5185	7.9		0.13	
963	D5185	8.330		0.91	
994	D5185	7.76		-0.13	
1026		----		----	
1059		----		----	
1146	in house	7.3		-0.97	
1201	D5185	8		0.31	
1243	D5185	8.20		0.67	
1297		----		----	
1328		----		----	
1435	D5185	8		0.31	
1660	D5185	11.2	R(0.01)	6.14	
1842	INH-01	8		0.31	
1900		----		----	
1920		----		----	
1955		----		----	
7011		----		----	

normality suspect
n 27
outliers 3
mean (n) 7.831
st.dev. (n) 0.3123
R(calc.) 0.874
R(D5185:13e1) 1.536

Application range:5 – 700 mg/kg

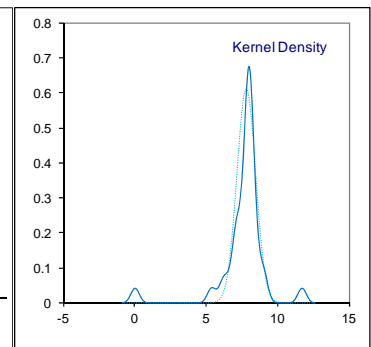
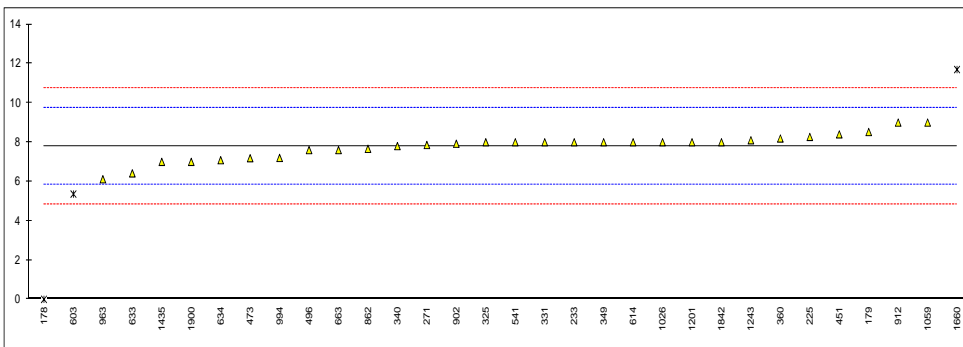


Determination of Molybdenum (Mo) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.00	R(0.01)	-7.93	
179	D5185	8.52		0.74	
225	D6595	8.27		0.49	
233	D6595	8		0.21	
255		----		----	
271	D5185	7.86		0.07	
311		----		----	
325	D5185	8		0.21	
331	D5185	8.0		0.21	
340	D5185	7.8		0.01	
349	D5185	8		0.21	
360	D5185	8.19		0.41	
398		----		----	
442		----		----	
451	D5185	8.4		0.62	
473	D5185	7.185		-0.62	
496	DIN51399	7.6		-0.19	
541	D5185	8		0.21	
551		----		----	
603	D5185	5.359	R(0.05)	-2.48	
614	D5185	8.0		0.21	
633	D6595	6.414		-1.40	
634	D6595	7.083	C	-0.72	First reported 11.783
663	D5185	7.60		-0.19	
862	D5185	7.665		-0.13	
902	D5185	7.923		0.13	
912	D5185	9.0		1.23	
963	D5185	6.114		-1.71	
994	D5185	7.20		-0.60	
1026	D5185	8		0.21	
1059	in house	9		1.23	
1146		----		----	
1201	D5185	8		0.21	
1243	D5185	8.10		0.32	
1297		----		----	
1328		----		----	
1435	D5185	7		-0.81	
1660	D5185	11.7	R(0.01)	3.98	
1842	INH-01	8		0.21	
1900	D6595	7		-0.81	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 29
outliers 3
mean (n) 7.790
st.dev. (n) 0.6544
R(calc.) 1.832
R(D5185:13e1) 2.749

Application range:5 – 200 mg/kg

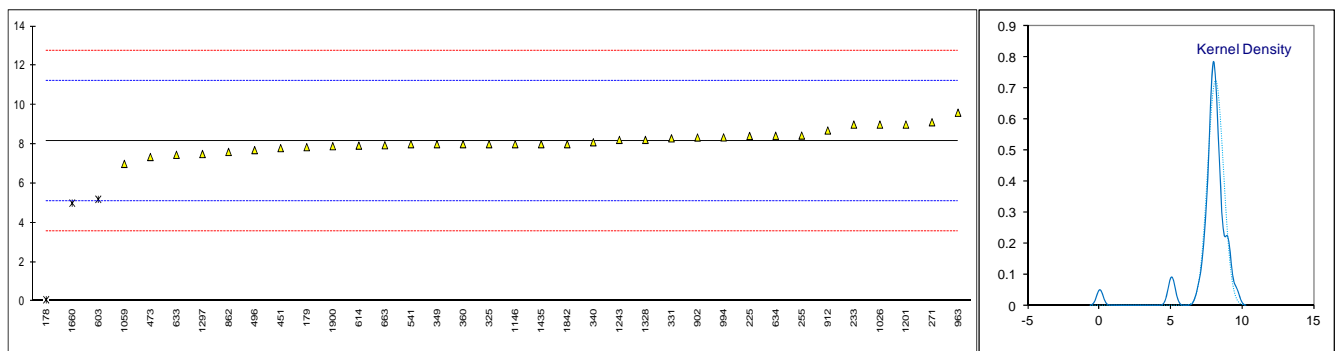


Determination of Nickel (Ni) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.08	R(0.01)	-5.28	
179	D5185	7.85		-0.20	
225	D6595	8.42		0.17	
233	D6595	9		0.55	
255	INH-021	8.44		0.18	
271	D5185	9.12		0.63	
311		----		----	
325	D5185	8		-0.10	
331	D5185	8.3		0.09	
340	D5185	8.1		-0.04	
349	D5185	8		-0.10	
360	D5185	8.00		-0.10	
398		----		----	
442		----		----	
451	D5185	7.8		-0.24	
473	D5185	7.348		-0.53	
496	DIN51399	7.7		-0.30	
541	D5185	8		-0.10	
551		----		----	
603	D5185	5.194	R(0.01)	-1.94	
614	D5185	7.93		-0.15	
633	D6595	7.461		-0.46	
634	D6595	8.430		0.18	
663	D5185	7.95		-0.14	
862	D5185	7.607		-0.36	
902	D5185	8.343		0.12	
912	D5185	8.7		0.35	
963	D5185	9.605		0.94	
994	D5185	8.35		0.12	
1026	D5185	9		0.55	
1059	ISO14597Mod.	7		-0.76	
1146	in house	8.0		-0.10	
1201	D5185	9		0.55	
1243	D5185	8.22		0.04	
1297	D5708	7.5		-0.43	
1328	GB/T/17476	8.22		0.04	
1435	D5185	8		-0.10	
1660	D5185	5.0	R(0.01)	-2.07	
1842	INH-01	8		-0.10	
1900	D6595	7.9		-0.17	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 33
outliers 3
mean (n) 8.160
st.dev. (n) 0.5521
R(calc.) 1.546
R(D5185:13e1) 4.285

Application range:5 – 40 mg/kg

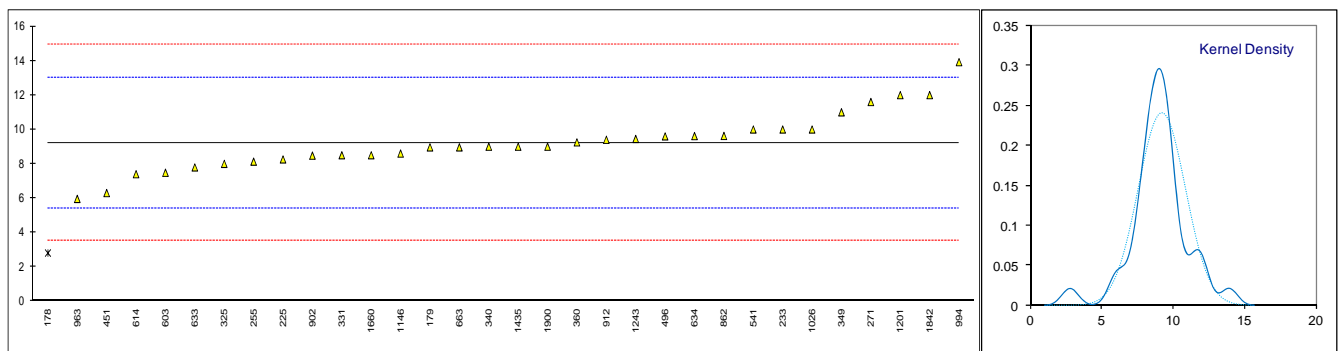


Determination of Sodium (Na) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	2.81	R(0.05)	-3.37	
179	D5185	8.96		-0.14	
225	D6595	8.26		-0.50	
233	D6595	10		0.41	
255	INH-021	8.13		-0.57	
271	D5185	11.60		1.25	
311		----		----	
325	D5185	8		-0.64	
331	D5185	8.5		-0.38	
340	D5185	9.0		-0.12	
349	D5185	11		0.94	
360	D5185	9.25		0.02	
398		----		----	
442		----		----	
451	D5185	6.3		-1.53	
473		----		----	
496	DIN51399	9.6		0.20	
541	D5185	10		0.41	
551		----		----	
603	D5185	7.482		-0.91	
614	D5185	7.4		-0.96	
633	D6595	7.794		-0.75	
634	D6595	9.617		0.21	
663	D5185	8.96		-0.14	
862	D5185	9.624		0.21	
902	D5185	8.478		-0.39	
912	D5185	9.4		0.10	
963	D5185	5.961		-1.71	
994	D5185	13.92		2.47	
1026	D5185	10		0.41	
1059		----		----	
1146	in house	8.6		-0.33	
1201	D5185	12		1.46	
1243	D5185	9.45		0.12	
1297		----		----	
1328		----		----	
1435	D5185	9		-0.12	
1660	D5185	8.5		-0.38	
1842	INH-01	12		1.46	
1900	D6595	9		-0.12	
1920		----		----	
1955		----		----	
7011		----		----	

normality suspect
n 31
outliers 1
mean (n) 9.219
st.dev. (n) 1.6545
R(calc.) 4.633
R(D5185:13e1) 5.325

Application range:7 – 70 mg/kg

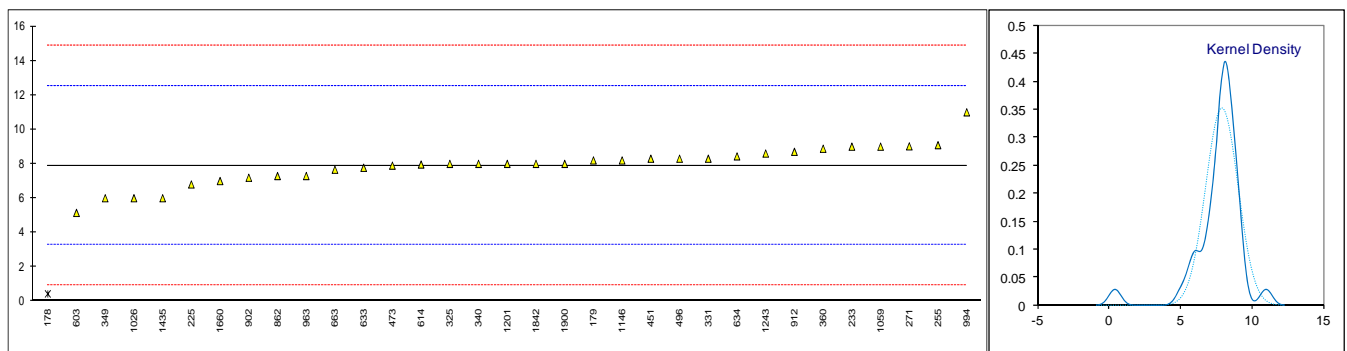


Determination of Silicon (Si) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.43	R(0.01)	-3.22	
179	D5185	8.20		0.13	
225	D6595	6.80		-0.48	
233	D6595	9		0.47	
255	INH-021	9.087		0.51	
271	D5185	9.02		0.48	
311		----		----	
325	D5185	8		0.04	
331	D5185	8.3		0.17	
340	D5185	8.0		0.04	
349	D5185	6		-0.82	
360	D5185	8.88		0.42	
398		----		----	
442		----		----	
451	D5185	8.3		0.17	
473	D5185	7.895		-0.01	
496	DIN51399	8.3		0.17	
541	D5185	<8		----	
551		----		----	
603	D5185	5.145		-1.19	
614	D5185	7.96		0.02	
633	D6595	7.770		-0.06	
634	D6595	8.437		0.23	
663	D5185	7.66		-0.11	
862	D5185	7.290		-0.27	
902	D5185	7.192		-0.31	
912	D5185	8.7		0.34	
963	D5185	7.293		-0.26	
994	D5185	11.0		1.33	
1026	D5185	6		-0.82	
1059	in house	9		0.47	
1146	in house	8.2		0.13	
1201	D5185	8		0.04	
1243	D5185	8.60		0.30	
1297		----		----	
1328		----		----	
1435	D5185	6		-0.82	
1660	D5185	7.0		-0.39	
1842	INH-01	8		0.04	
1900	D6595	8		0.04	
1920		----		----	
1955		----		----	
7011		----		----	

normality suspect
n 32
outliers 1
mean (n) 7.907
st.dev. (n) 1.1294
R(calc.) 3.162
R(D5185:13e1) 6.496

Application range:8 – 50 mg/kg

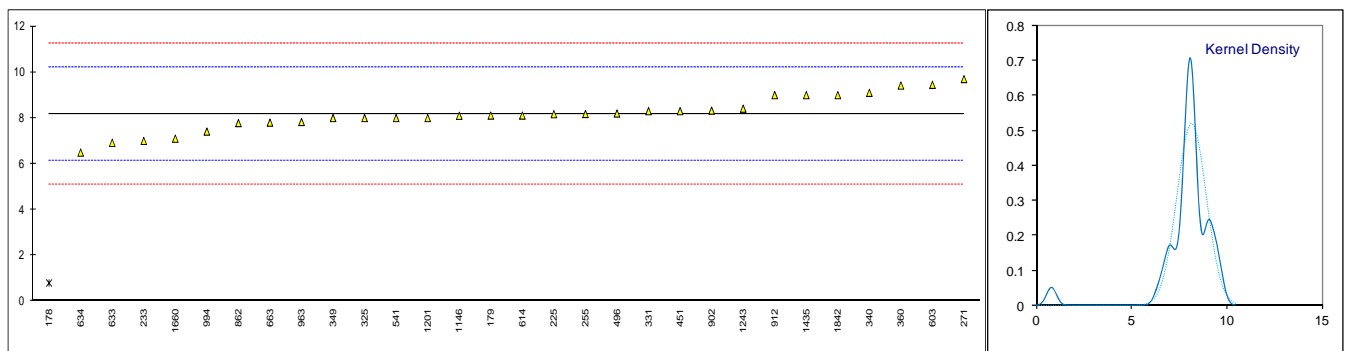


Determination of Silver (Ag) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.80	R(0.01)	-7.22	
179	D5185	8.11		-0.07	
225	D6595	8.17		-0.01	
233	D6595	7		-1.15	
255	INH-021	8.179		0.00	
271	D5185	9.70		1.49	
311		----		----	
325	D5185	8	C	-0.18	First reported 0
331	D5185	8.3		0.12	
340	D5185	9.1		0.90	
349	D5185	8		-0.18	
360	D5185	9.42		1.21	
398		----		----	
442		----		----	
451	D5185	8.3		0.12	
473		----		----	
496	DIN51399	8.2		0.02	
541	D5185	8		-0.18	
551		----		----	
603	D5185	9.45		1.24	
614	D5185	8.11		-0.07	
633	D6595	6.918		-1.23	
634	D6595	6.492		-1.65	
663	D5185	7.80		-0.37	
862	D5185	7.781		-0.39	
902	D5185	8.325		0.14	
912	D5185	9.0		0.80	
963	D5185	7.829		-0.34	
994	D5185	7.41		-0.75	
1026		----		----	
1059		----		----	
1146	in house	8.1		-0.08	
1201	D5185	8		-0.18	
1243	D5185	8.41		0.23	
1297		----		----	
1328		----		----	
1435	D5185	9		0.80	
1660	D5185	7.1		-1.06	
1842	INH-01	9		0.80	
1900		----		----	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 29
outliers 1
mean (n) 8.179
st.dev. (n) 0.7698
R(calc.) 2.156
R(D5185:13e1) 2.863

Application range:0.5 – 50 mg/kg

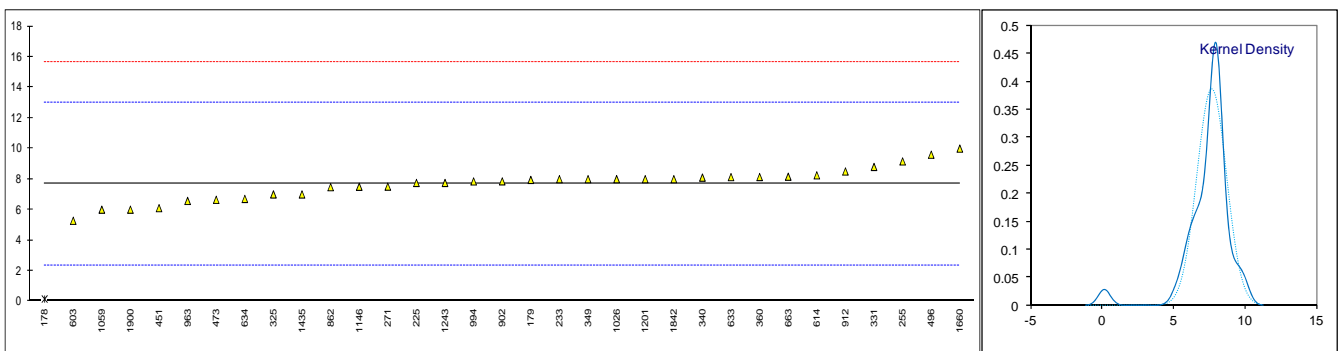


Determination of Tin (Sn) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.15	R(0.01)	-2.84	
179	D5185	7.95		0.10	
225	D6595	7.75		0.03	
233	D6595	8		0.12	
255	INH-021	9.160		0.56	
271	D5185	7.51		-0.06	
311		----		----	
325	D5185	7		-0.26	
331	D5185	8.8		0.42	
340	D5185	8.1		0.16	
349	D5185	8		0.12	
360	D5185	8.14		0.17	
398		----		----	
442		----		----	
451	D5185	6.1		-0.60	
473	D5185	6.646		-0.39	
496	DIN51399	9.6		0.72	
541	D5185	<10		----	
551		----		----	
603	D5185	5.273		-0.91	
614	D5185	8.25		0.21	
633	D6595	8.132		0.17	
634	D6595	6.705		-0.37	
663	D5185	8.16		0.18	
862	D5185	7.474		-0.08	
902	D5185	7.855		0.07	
912	D5185	8.5		0.31	
963	D5185	6.572		-0.42	
994	D5185	7.85		0.06	
1026	D5185	8		0.12	
1059	in house	6		-0.63	
1146	in house	7.5		-0.07	
1201	D5185	8		0.12	
1243	D5185	7.75		0.03	
1297		----		----	
1328		----		----	
1435	D5185	7		-0.26	
1660	D5185	10.0		0.87	
1842	INH-01	8		0.12	
1900	D6595	6		-0.63	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 32
outliers 1
mean (n) 7.681
st.dev. (n) 1.0349
R(calc.) 2.898
R(D5185:13e1) 7.433

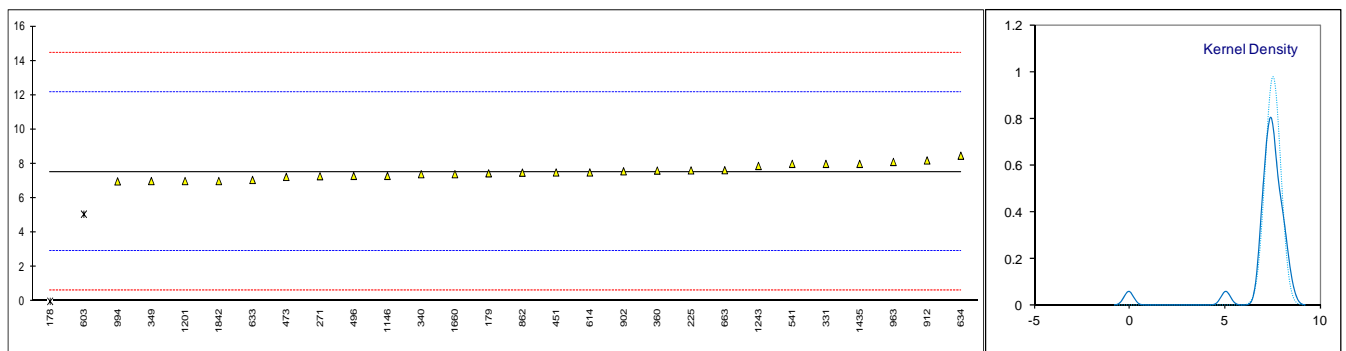
Application range:10 – 40 mg/kg



Determination of Titanium (Ti) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.00	R(0.01)	-3.27	
179	D5185	7.45		-0.04	
225	D6595	7.62		0.04	
233		----		----	
255		----		----	
271	D5185	7.28		-0.11	
311		----		----	
325		----		----	
331	D5185	8.0		0.20	
340	D5185	7.4		-0.06	
349	D5185	7		-0.23	
360	D5185	7.61		0.03	
398		----		----	
442		----		----	
451	D5185	7.5		-0.02	
473	D5185	7.241		-0.13	
496	DIN51399	7.3		-0.10	
541	D5185	8		0.20	
551		----		----	
603	D5185	5.069	R(0.01)	-1.07	
614	D5185	7.5		-0.02	
633	D6595	7.065		-0.21	
634	D6595	8.475		0.41	
663	D5185	7.63		0.04	
862	D5185	7.489		-0.02	
902	D5185	7.568		0.01	
912	D5185	8.2		0.29	
963	D5185	8.112		0.25	
994	D5185	6.98		-0.24	
1026		----		----	
1059		----		----	
1146	in house	7.3		-0.10	
1201	D5185	7		-0.23	
1243	D5185	7.88		0.15	
1297		----		----	
1328		----		----	
1435	D5185	8		0.20	
1660	D5185	7.4		-0.06	
1842	INH-01	7		-0.23	
1900		----		----	
1920		----		----	
1955		----		----	
7011		----		----	
normality		OK			
n		26			
outliers		2			
mean (n)		7.538			
st.dev. (n)		0.4073			
R(calc.)		1.140			
R(D5185:13e1)		6.460			

Application range:5 – 40 mg/kg

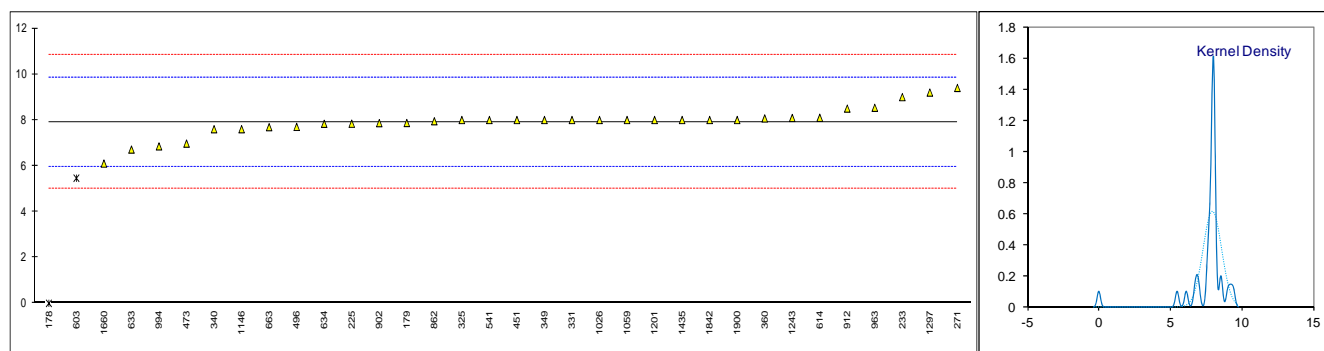


Determination of Vanadium (V) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	0.00	R(0.01)	-8.13	
179	D5185	7.87		-0.05	
225	D6595	7.84		-0.08	
233	D6595	9		1.11	
255		----		----	
271	D5185	9.40		1.52	
311		----		----	
325	D5185	8		0.08	
331	D5185	8.0		0.08	
340	D5185	7.6		-0.33	
349	D5185	8		0.08	
360	D5185	8.07		0.15	
398		----		----	
442		----		----	
451	D5185	8.0		0.08	
473	D5185	6.972		-0.97	
496	DIN51399	7.7		-0.23	
541	D5185	8		0.08	
551		----		----	
603	D5185	5.461	R(0.05)	-2.53	
614	D5185	8.1		0.18	
633	D6595	6.711		-1.24	
634	D6595	7.835		-0.09	
663	D5185	7.69		-0.24	
862	D5185	7.948		0.03	
902	D5185	7.865		-0.06	
912	D5185	8.5		0.59	
963	D5185	8.535		0.63	
994	D5185	6.85		-1.10	
1026	D5185	8		0.08	
1059	ISO145987Mod.	8		0.08	
1146	in house	7.6		-0.33	
1201	D5185	8		0.08	
1243	D5185	8.09		0.17	
1297	D5708	9.2		1.31	
1328		----		----	
1435	D5185	8		0.08	
1660	D5185	6.1		-1.87	
1842	INH-01	8		0.08	
1900	D6595	8		0.08	
1920		----		----	
1955		----		----	
7011		----		----	

normality not OK
n 32
outliers 2
mean (n) 7.921
st.dev. (n) 0.6478
R(calc.) 1.814
R(D5185:13e1) 2.728

Application range:1 – 50 mg/kg

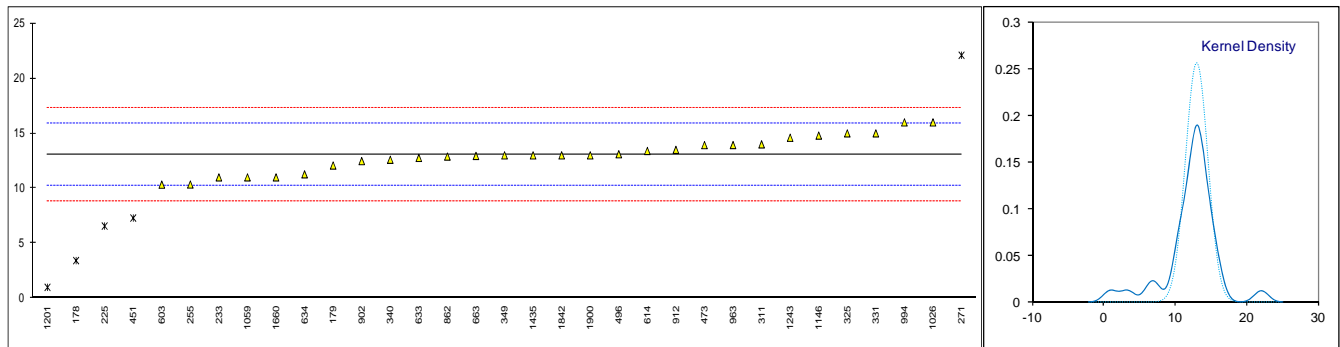


Determination of Calcium (Ca) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	3.43	R(0.01)	-6.79	
179	D5185	12.08		-0.70	
225	D6595	6.57	R(0.05)	-4.58	
233	D6595	11		-1.46	
255	INH-021	10.35		-1.92	
271	D5185	22.10	R(0.01)	6.36	
311	D5185	14		0.65	
325	D5185	15		1.36	
331	D5185	15.0		1.36	
340	D5185	12.6		-0.33	
349	D5185	13		-0.05	
360		----		----	
398		----		----	
442		----		----	
451	D5185	7.3	R(0.05)	-4.06	
473	D5185	13.93		0.60	
496	DIN51399	13.1		0.02	
541	D5185	<40		----	
551		----		----	
603	D5185	10.34		-1.92	
614	D5185	13.4		0.23	
633	D6595	12.765		-0.22	
634	D6595	11.274		-1.27	
663	D5185	12.95		-0.09	
862	D5185	12.89		-0.13	
902	D5185	12.48		-0.42	
912	D5185	13.5		0.30	
963	D5185	13.940		0.61	
994	D5185	15.998		2.06	
1026	D5185	16		2.06	
1059	in house	11		-1.46	
1146	in house	14.8		1.22	
1201	D5185	1	C,R(0.01)	-8.50	First reported 7
1243	D5185	14.6		1.08	
1297		----		----	
1328		----		----	
1435	D5185	13		-0.05	
1660	D5185	11.0		-1.46	
1842	INH-01	13		-0.05	
1900	D6595	13		-0.05	
1920		----		----	
1955		----		----	
7011		----		----	

normality OK
n 28
outliers 5
mean (n) 13.071
st.dev. (n) 1.5544
R(calc.) 4.352
R(Horwitz) 3.977

Compare R(D5185:13e1) = 0.424, range 40 – 9000 mg/kg

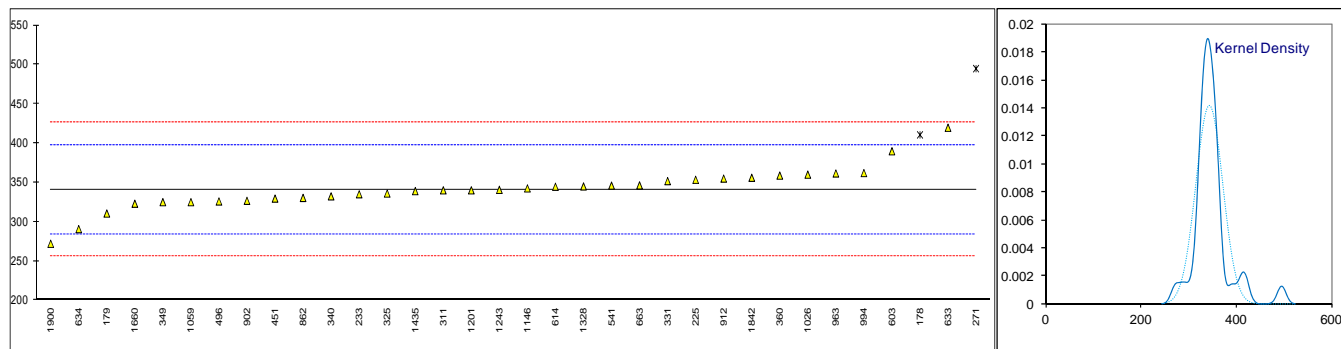


Determination of Phosphorus (P) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	410.61	ex	2.45	Result excluded, see §4.1
179	D5185	310.51		-1.08	
225	D6595	353.44		0.44	
233	D6595	335		-0.21	
255		----		----	
271	D5185	494.9	C,R(0.01)	5.42	First reported 449.69
311	D5185	340		-0.04	
325	D5185	336		-0.18	
331	D5185	351.8		0.38	
340	D5185	332.4		-0.31	
349	D5185	325		-0.57	
360	D5185	358.8		0.63	
398		----		----	
442		----		----	
451	D5185	329.5		-0.41	
473		----		----	
496	DIN51399	325.9		-0.53	
541	D5185	346		0.17	
551		----		----	
603	D5185	389.8		1.72	
614	D5185	344.6		0.12	
633	D6595	419.71		2.77	
634	D6595	290.67		-1.78	
663	D5185	346.3		0.18	
862	D5185	330.4		-0.38	
902	D5185	326.7		-0.51	
912	D5185	355		0.49	
963	D5185	361.5		0.72	
994	D5185	362	C	0.74	First reported 518.3
1026	D5185	360		0.67	
1059	in house	325		-0.57	
1146	in house	342.5		0.05	
1201	D5185	340		-0.04	
1243	D5185	340.7		-0.01	
1297		----		----	
1328	GB/T/17476	345		0.14	
1435	D5185	339		-0.07	
1660	D5185	323		-0.64	
1842	INH-01	356		0.53	
1900	D6595	272		-2.44	
1920		----		----	
1955		----		----	
7011		----		----	

normality not OK
n 32
outliers 1 (+1 excl)
mean (n) 341.070
st.dev. (n) 25.8845
R(calc.) 72.477
R(D5185:13e1) 79.413

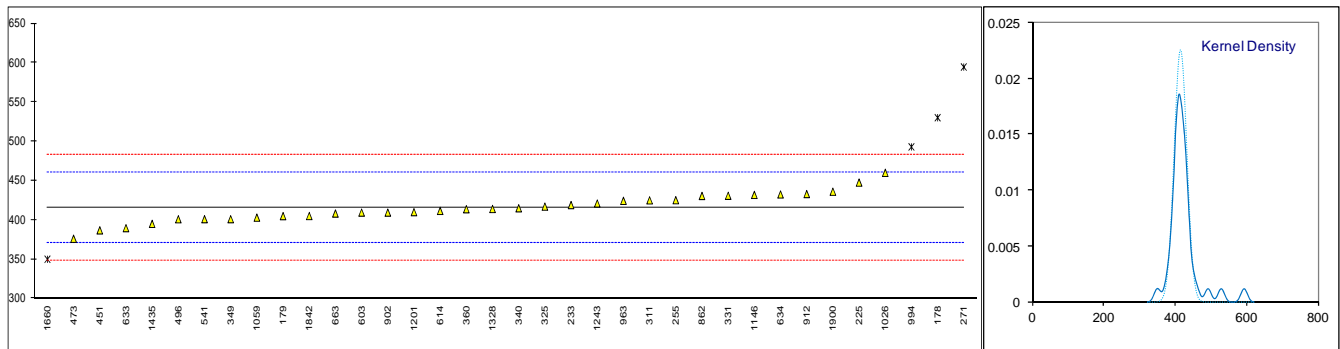
Application range:10 – 1000 mg/kg



Determination of Zinc (Zn) on sample #14221; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	INH-1158	530.14	R(0.01)	5.11	
179	D5185	404.91		-0.46	
225	D6595	447.61		1.44	
233	D6595	419		0.17	
255	INH-021	425.18		0.44	
271	D5185	594.7	C,R(0.01)	7.98	First reported 555.42
311	D5185	425		0.43	
325	D5185	417		0.08	
331	D5185	430.8		0.69	
340	D5185	414.9		-0.02	
349	D5185	401		-0.63	
360	D5185	413.7		-0.07	
398		----		----	
442		----		----	
451	D5185	386.8		-1.27	
473	D5185	376.1		-1.74	
496	DIN51399	400.9		-0.64	
541	D5185	401		-0.63	
551		----		----	
603	D5185	409.4		-0.26	
614	D5185	411.5		-0.17	
633	D6595	389.61		-1.14	
634	D6595	432.46		0.76	
663	D5185	408.2		-0.31	
862	D5185	430.6		0.68	
902	D5185	409.4		-0.26	
912	D5185	433		0.79	
963	D5185	424.4		0.41	
994	D5185	493	C,R(0.05)	3.46	First reported 535.2
1026	D5185	460		1.99	
1059	in house	403		-0.55	
1146	in house	431.9		0.74	
1201	D5185	410		-0.23	
1243	D5185	421.0		0.26	
1297		----		----	
1328	GB/T/17476	414		-0.06	
1435	D5185	395		-0.90	
1660	D5185	350	R(0.05)	-2.90	
1842	INH-01	405		-0.46	
1900	D6595	436		0.92	
1920		----		----	
1955		----		----	
7011		----		----	
normality		OK			
n		32			
outliers		4			
mean (n)		415.262			
st.dev. (n)		17.7085			
R(calc.)		49.584			
R(D5185:13e1)		62.984			

Application range:60 – 1600 mg/kg



APPENDIX 2**Number of participants per country**

1 laboratory in	ARGENTINA
1 laboratory in	AUSTRALIA
1 laboratory in	AZERBAIJAN
2 laboratories in	BELGIUM
1 laboratory in	BRAZIL
1 laboratory in	BULGARIA
2 laboratories in	P.R. of CHINA
1 laboratory in	COTE D'IVOIRE
2 laboratory in	FRANCE
2 laboratory in	GERMANY
1 laboratory in	GHANA
1 laboratory in	GREECE
1 laboratory in	INDIA
1 laboratory in	IRAN
1 laboratory in	ISRAEL
2 laboratories in	ITALY
1 laboratory in	MALAYSIA
4 laboratories in	NETHERLANDS
2 laboratories in	NORWAY
2 laboratories in	PHILIPPINES
1 laboratory in	SAUDI ARABIA
1 laboratory in	SLOVENIA
1 laboratory in	SOUTH AFRICA
1 laboratory in	SPAIN
1 laboratory in	SWEDEN
1 laboratory in	TANZANIA
1 laboratory in	TURKEY
3 laboratories in	UNITED KINGDOM
2 laboratories in	UNITED STATES OF AMERICA

APPENDIX 3**Abbreviations:**

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from calculations
n.a.	= not applicable
E	= error in calculations
U	= reported wrong unit
W	= withdrawn result on request of participant
SDS	= Safety Data Sheet

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, April 2014
- 2 ASTM E178-89
- 3 ASTM E1301-89
- 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, First reported Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical Brief, No4 January 2001
- 14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>).
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)