Results of Proficiency Test
Turbine Oil (fresh)
May 2020

Organized by: **Institute for Interlaboratory Studies**
            Spijkenisse, the Netherlands

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Report: **iis20L04 == Revised ==**

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SUMMARY OF CHANGES

This revised report replaces the original report iis20L04 of August 2020.

It was discovered that z-scores were calculated in the results table of Zinc in appendix 1. Regretfully, this was done by mistake. In paragraph 4.1 can be read that for Zinc the consensus value was below the application range of ASTM D5185:18 and therefore no z-scores were calculated.

The following page in this report has been revised:

- Appendix 1: page 30 (page 29 in the original report)

CONTENTS

Appendices:

1   INTRODUCTION

In 2018 the Institute for Interlaboratory Studies (iis) has started a proficiency test (PT) for fresh Turbine Oil. During the annual proficiency testing program 2019/2020 it was decided to continue the proficiency test (PT) for the analysis of fresh Turbine Oil.

In this interlaboratory study 23 laboratories in 17 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the Turbine Oil (fresh) 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 sample of one liter of fresh Turbine Oil labelled #20066.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1   ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. 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 is electronically available through 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 approximately 120 liters of fresh Turbine Oil was obtained from a third party. After homogenization 45 amber glass bottles of 1L were filled and labelled #20066. The homogeneity of subsamples was checked by the determination of Density in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/L
sample #20066-1	0.86284
sample #20066-2	0.86284
sample #20066-3	0.86284
sample #20066-4	0.86284
sample #20066-5	0.86284
sample #20066-6	0.86284
sample #20066-7	0.86284
sample #20066-8	0.86284

Table 1: homogeneity test results of subsamples #20066

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.

	Density at 15°C in kg/L
r (observed)	0.00000
reference test method	ISO12185:96
0.3 * R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #20066

The calculated repeatability was 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 1L bottle labelled #20066 was sent on April 22, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of the fresh Turbine Oil packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine on sample #20066: Total Acid Number, Air Release Time at 50°C, Density at 15°C, Flash Point C.O.C., Foam Characteristics (Foaming Tendency, Foaming Stability), Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Pour Point (Manual and Automated, 1°C interval), Sulfur, Water, Water Separability at 54°C (distilled water), Calcium as Ca, Phosphorus as P and Zinc as Zn.
Some extra information was asked about the determinations of Total Acid Number and Foaming Characteristics.

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

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 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 the 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.

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

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

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

3.3 Z-SCORES

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

This target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

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

$$
\begin{array}{rll}
 & |z| < 1 & \text{good} \\
1 < & |z| < 2 & \text{satisfactory} \\
2 < & |z| < 3 & \text{questionable} \\
3 < & |z| & \text{unsatisfactory}
\end{array}
$$

4 EVALUATION

In this interlaboratory study some problems were encountered with the dispatch of the samples due to several reasons with transportation (e.g. closed borders due to COVID-19 pandemic). Therefore, the reporting time on the data entry portal was extended with another three weeks. Six participants did not report any test results. Not all participants were able to report test results for all requested tests.
In total 17 participants reported 254 numerical test results. Observed were 8 outlying test results, which is 3.1% 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 "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1   EVALUATION PER TEST

In this section the 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 original data. The abbreviations used in these tables are explained in appendix 3.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D2270) and an added designation for the year that the method was adopted or revised (e.g. D2270:10). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2270:10(2016)). In the results tables of appendix 1 only the method number and year of adoption or revision e.g. D2270:10 will be used.

Total Acid Number: This determination was problematic dependent on mode used of ASTM D664-A:18e2. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D664-A:18e2 IP 60mL and BEP 60mL but is not in agreement with IP 125mL and BEP 125mL.
When the test results for IP and BEP were evaluated separately, the calculated reproducibility of the test results for BEP 60mL is in agreement with the precision data of ASTM D664-A:18e2. The calculated reproducibility of the test results for BEP 125mL and IP 60 and 125mL are not in agreement.
Remarkably, two participants still used pH 11 for BEP instead of pH 10. In test method ASTM D664-A:18e2 pH 10 is mentioned.

Air Release time at 50°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D3427:19.

Density at 15°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ISO12185:96.

Flash Point C.O.C.: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D92:18.

Foaming Characteristics (Tendency and Stability): This determination was very problematic.
In total one statistical outlier was observed. It was decided not to calculate z-scores for Foam Tendency at sequence I and III due to the large reproducibility between the reported test results. The Foam Tendency determination for sequence II is after rejection of the statistical outlier not in agreement with the requirements of ASTM D892:18.
Almost all reported test results for Foam Stability were zero. Therefore, it was decided not to calculate z-scores.
The determination of the Foaming Characteristics is very sensitive in maintenance and execution. In ASTM D892:18 many tips and tricks are

given in the test method part X1. Possible sources for the large variation are the cleaning and checking of the air diffuser, air tubes and test cylinders, the air flow rate used during the blowing period. Almost all reporting participants mentioned to use the sample as received and with a metal diffuser.

Kinematic Viscosity at 40°C: This determination was problematic. One statistical outlier was observed. The calculated reproducibility is not in agreement with the requirements of ASTM D445:19a.

Kinematic Viscosity at 100°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:19a.

Viscosity Index: This determination was problematic. One statistical outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2270:10(2016).

Pour Point, Manual: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D97:17b.

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

Sulfur: 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 D4294:16e1.

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

Water Separability at 54°C, distilled water: This determination was not problematic. No statistical outliers were observed over 3 parameters. The calculated reproducibilities are in good agreement with the requirements of ASTM D1401:19.

Calcium: This determination may not be problematic. The consensus value was below the application range of ASTM D5185:18. Therefore, no z-scores were calculated.

Phosphorus: 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 D5185:18.

Zinc: This determination may not be problematic. The consensus value was below the application range of ASTM D5185:18. Therefore, no z-scores were calculated.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The 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, EN test methods) or previous proficiency tests are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	15	0.16	0.09	0.09
Air Release time at 50°C	minutes	9	3.2	1.9	2.2
Density at 15°C	kg/L	16	0.8628	0.0005	0.0005
Flash Point C.O.C.	°C	9	225.3	15.1	18
Foam Tendency Seq. I	mL	12	103.3	266.8	(43.9)
Foam Tendency Seq. II	mL	10	21.0	33.5	21.2
Foam Tendency Seq. III	mL	11	108.2	263.5	(67.5)
Foam Stability Seq. I	mL	11	0	n.e.	n.e.
Foam Stability Seq. II	mL	11	0	n.e.	n.e.
Foam Stability Seq. III	mL	11	0	n.e.	n.e.
Kinematic Viscosity at 40°C	mm^2/s	15	31.380	0.426	0.383
Kinematic Viscosity at 100°C	mm^2/s	14	5.404	0.077	0.075
Viscosity Index		13	106.4	3.6	2
Pour Point, Manual	°C	4	-15.8	8.0	9
Pour Point, Automated, 1°C interval	°C	5	-13.3	4.0	4.5
Sulfur	mg/kg	7	288	86	74
Water	mg/kg	15	38.0	40.1	149.8
Water Separability at 54°C, distilled water					
- Time to reach ≤ 3 ml emulsion	minutes	11	7.9	5.7	20
- Time to reach 37 ml water	minutes	10	7.9	5.9	20
- Time to reach complete break	minutes	9	9.4	4.4	20
Calcium as Ca	mg/kg	17	<40	n.e	n.e.
Phosphorus as P	mg/kg	16	51.4	16.8	30.8
Zinc as Zn	mg/kg	17	<60	n.e	n.e.

Table 3: reproducibilities of tests on sample #20066

Without further statistical calculations it can be concluded that for a number of tests there is a good compliance of the group of participants with the reference test methods. The tests that are problematic have been discussed in paragraph 4.1.

4.3   COMPARISON OF THE PROFICIENCY TEST OF MAY 2020 WITH PREVIOUS PTS

	May 2020	May 2019	May 2018
Number of reporting laboratories	17	20	19
Number of test results	254	271	281
Number of statistical outliers	8	4	12
Percentage of statistical outliers	3.1%	1.5%	4.3%

Table 4: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.
The performance of the determinations of the proficiency tests was compared against the requirements of the respective reference test methods. The conclusions are given in the following table.

Parameter	May 2020	May 2019	May 2018
Total Acid Number	+/-	-	++
Air Release time at 50°C	+	-	++
Density at 15°C	+/-	+	-
Flash Point C.O.C.	+	+	++
Foam Tendency Seq. I	(--)	(--)	(--)
Foam Tendency Seq. II	-	+/-	+
Foam Tendency Seq. III	(--)	(--)	(--)
Kinematic Viscosity at 40°C	-	+	+
Kinematic Viscosity at 100°C	+/-	+	+
Viscosity Index	-	-	-
Pour Point, Manual	+	+	++
Pour Point, Automated, 1°C interval	+	+	+
Sulfur	-	+/-	+
Water	++	++	++
Water Separability 54°C, dist. water	++	+	++
Calcium as Ca	n.e.	n.e.	n.e.
Phosphorus as P	+	n.e.	++
Zinc as Zn	n.e.	n.e.	n.e.

Table 5: comparison determinations against the reference test methods

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 Total Acid Number on sample #20066; results in mg KOH/g

lab	method	value	mark	z(targ)	Determination of end point	Volume of titration solvent
178	D664-A	0.13		-0.96	Inflection Point	60 mL
179	D664-A	0.09		-2.20	Inflection Point	60 mL
237	D664-A	<0.1		-----	Inflection Point	125 mL
325	D664-A	0.15		-0.33	Buffer End Point pH 10	125 mL
349	D664-A	0.16		-0.02	Inflection Point	125 mL
432		-----		-----	---	---
496	D664-A	0.16		-0.02	Buffer End Point pH 10	60 mL
614	D664-A	0.16		-0.02	---	60 mL
862		-----		-----	---	---
912		-----		-----	---	---
962		-----		-----	---	---
963		-----		-----	---	---
1011		-----		-----	---	---
1047	ISO6618	0.17		0.29	Inflection Point	125 mL
1146	D664-A	0.189		0.88	Buffer End Point pH 11	125 mL
1243	ISO6618	0.22		1.85	Inflection Point	60 mL
1435	D664-A	0.134		-0.83	---	---
1743	D664-A	0.15		-0.33	Buffer End Point pH 11	60 mL
1875	ISO6618	0.1725		0.37	Inflection Point	60 mL
1957	D664-A	0.20		1.23	Buffer End Point pH 10	125 mL
6016	D664-A	0.184		0.73	---	---
6253		-----		-----	---	---
6310	D664-A	0.14		-0.64	Buffer End Point pH 10	60 mL

					BEP (pH 10 and 11) only	Inflection point only
normality		OK			OK	OK
n		15			6	6
outliers		0			0	0
mean (n)		0.1606			0.1648	0.1571
st.dev. (n)		0.03156			0.02409	0.04383
R(calc.)		0.0884			0.0917	0.1227
st.dev.(D664-A:18e2 IP 60ml)		0.03207			---	---
R(D664-A:18e2 IP 60ml)		0.0898			---	0.0882
Compare						
D664-A:18e2 BEP 60ml		0.0931			0.0954	---
D664-A:18e2 IP 125ml		0.0325			---	0.0317
D664-A:18e2 BEP 125ml		0.0462			0.0475	---

Kernel Density
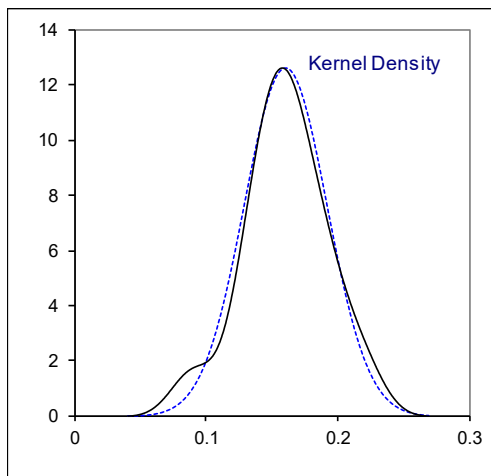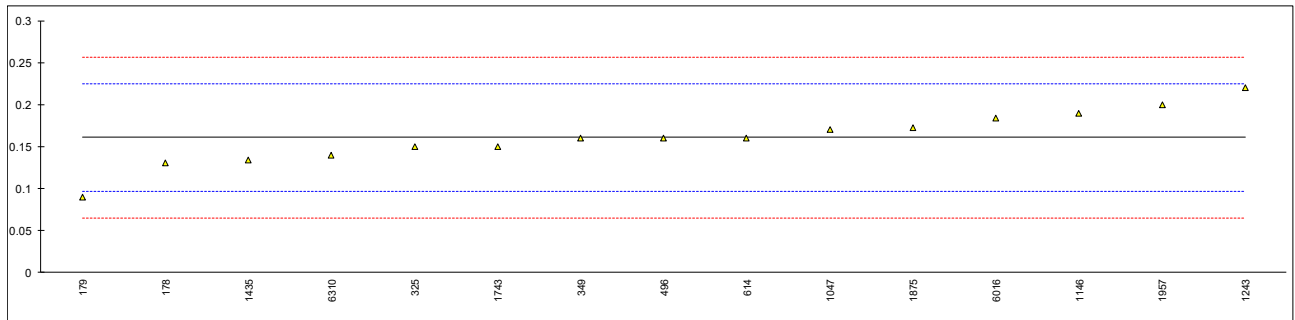
Determination of Air Release time at 50°C on sample #20066; results in minutes

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D3427	2.9		-0.40	
237		-----		-----	
325	D3427	3.366666666		0.19	
349		-----		-----	
432	ISO9120	4.5		1.62	
496	D3427	3.3		0.10	
614		-----		-----	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047		-----		-----	
1146		-----		-----	
1243	ISO9120	3.4		0.23	
1435	D3427	3.5		0.36	
1743	ISO9120	2.4		-1.03	
1875		-----		-----	
1957		-----		-----	
6016	D3427	3.4		0.23	
6253		-----		-----	
6310	D3427	2.2		-1.29	

normality	OK
n	9
outliers	0
mean (n)	3.2185
st.dev. (n)	0.67312
R(calc.)	1.8847
st.dev.(D3427:19)	0.79155
R(D3427:19)	2.2164

Kernel Density
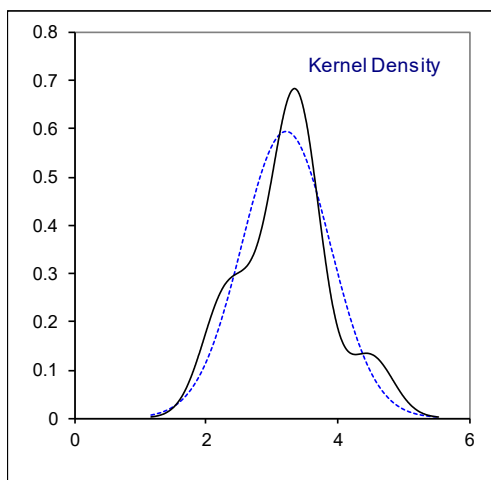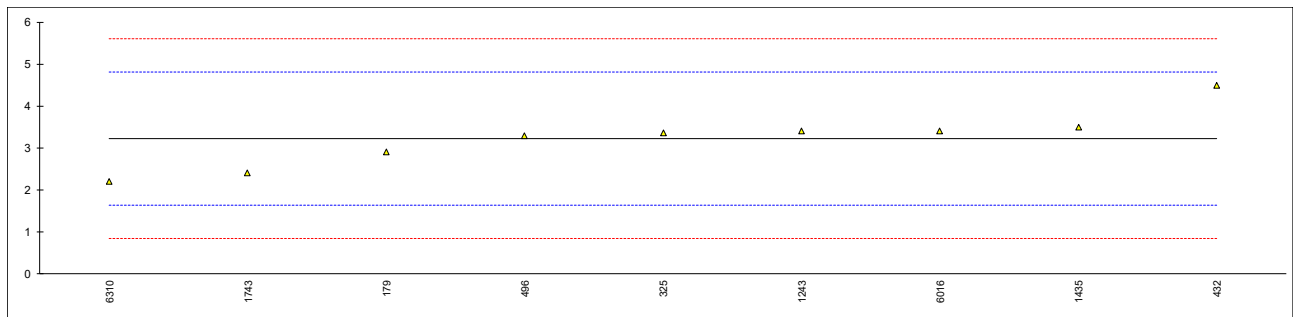
Determination of Density at 15°C on sample #20066; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D4052	0.8625	C	-1.81	first reported 862.5 kg/L
179	D4052	0.863		0.99	
237	D4052	0.8631		1.55	
325	D4052	0.8629		0.43	
349	D4052	0.8627		-0.69	
432	D4052	0.86283		0.04	
496	ISO12185	0.86284		0.09	
614	D4052	0.8626		-1.25	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047	ISO12185	0.8627	C	-0.69	first reported 0.8318
1146	D4052	0.8628		-0.13	
1243	ISO12185	0.8629	C	0.43	first reported without a unit
1435	D4052	0.8629		0.43	
1743	In house	0.867	C,G(0.01)	23.39	first reported 862.0 kg/m^3
1875	D7042	0.8631	C	1.55	first reported 0.8631 kg/m^3
1957	D4052	0.8627	C	-0.69	reported without a unit
6016	D4052	0.8628		-0.13	
6253		-----		-----	
6310	D4052	0.8628		-0.13	

normality	OK
n	16
outliers	1
mean (n)	0.86282
st.dev. (n)	0.000164
R(calc.)	0.00046
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005
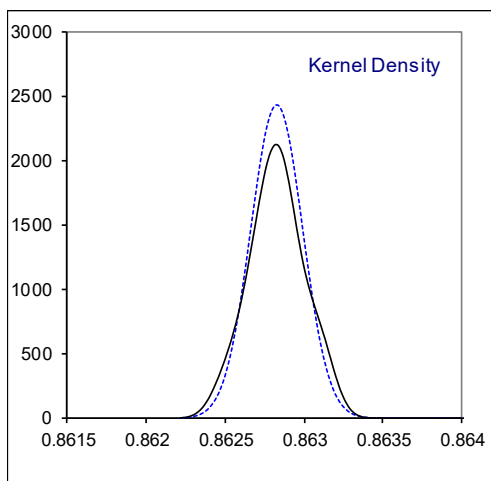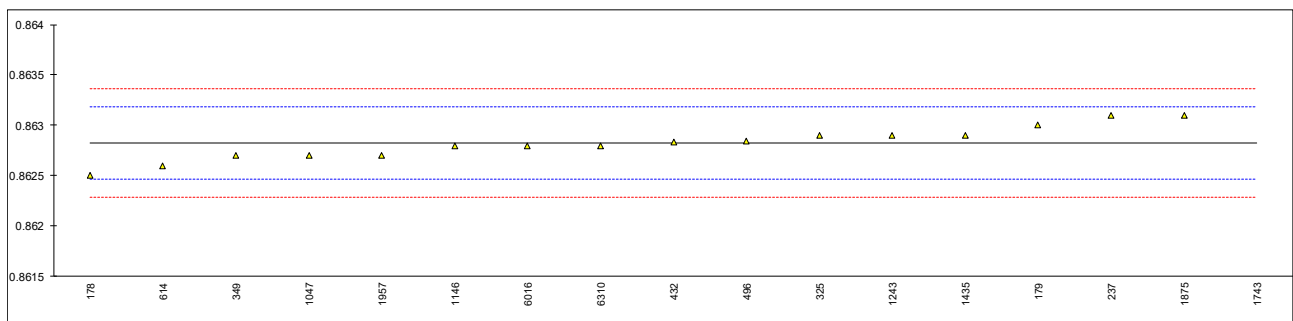
Determination of Flash Point C.O.C. on sample #20066; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D92	230		0.73	
237	D92	222		-0.52	
325	D92	224		-0.21	
349	D92	230		0.73	
432	D92	228.0		0.41	
496	D92	222		-0.52	
614		-----		-----	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047		-----		-----	
1146		-----		-----	
1243	ISO2592	230		0.73	
1435	D92	214.0		-1.76	
1743	ISO2592	228		0.41	
1875		-----		-----	
1957		-----		-----	
6016		-----		-----	
6253		-----		-----	
6310		-----		-----	

normality	suspect
n	9
outliers	0
mean (n)	225.33
st.dev. (n)	5.385
R(calc.)	15.08
st.dev.(D92:18)	6.429
R(D92:18)	18
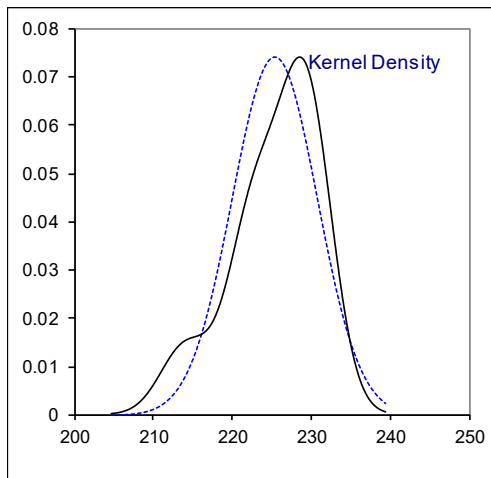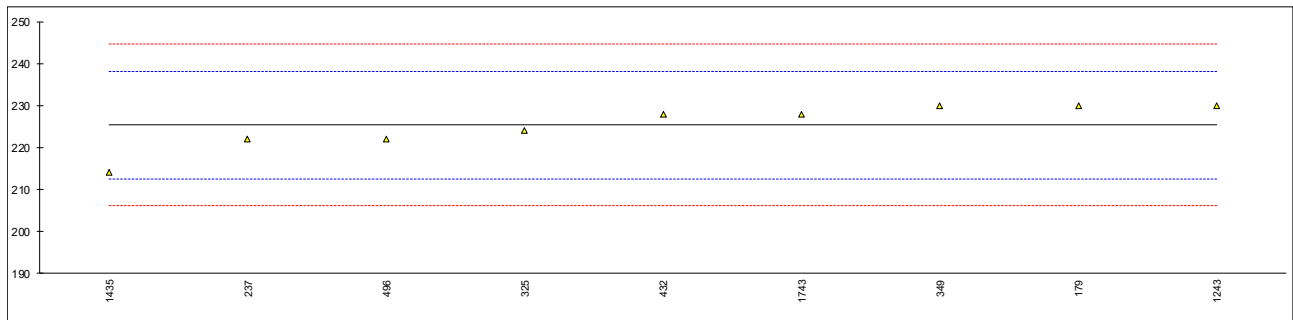
Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #20066; results in mL

| lab	method	Sample used	Diffuser	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		As received	Metal	-----		-----	-----		-----	-----		-----
179	D892	As received	Metal	150		-----	10		-1.45	220		-----
237	D892	As received	Metal	20		-----	30		1.19	20		-----
325	D892	As received	Metal	80		-----	20		-0.13	80		-----
349		---	---	-----		-----	-----		-----	-----		-----
432	D892	As received	Metal	20		-----	20		-0.13	30		-----
496		---	---	-----		-----	-----		-----	-----		-----
614		---	Metal	-----		-----	-----		-----	-----		-----
862		---	---	-----		-----	-----		-----	-----		-----
912		---	---	-----		-----	-----		-----	-----		-----
962		---	---	-----		-----	-----		-----	-----		-----
963		---	---	-----		-----	-----		-----	-----		-----
1011		---	---	-----		-----	-----		-----	-----		-----
1047	D892	---	Metal	230		-----	10		-1.45	130		-----
1146	ISO6247	As received	Metal	220		-----	90	G(0.01)	9.09	200		-----
1243	D892	As received	Stone	30		-----	20		-0.13	20		-----
1435		---	---	290		-----	20		-0.13	290		-----
1743	ISO6247	As received	Stone	20		-----	-----		-----	-----		-----
1875		---	---	-----		-----	-----		-----	-----		-----
1957	D892	As received	Metal	40		-----	20		-0.13	50		-----
6016	D892	---	---	80		-----	50		3.82	20		-----
6253		---	---	-----		-----	-----		-----	-----		-----
6310	D892	After agitation	Metal	60		-----	10		-1.45	130		-----
	normality			OK			not OK			OK		
	n			12			10			11		
	outliers			0			1			0		
	mean (n)			103.33			21.00			108.18		
	st.dev. (n)			95.283			11.972			94.108		
	R(calc.)			266.79			33.52			263.50		
	st.dev.(D892:18)			(15.66)			7.59			(24.10)		
	R(D892:18)			(43.862)			21.244			(67.479)		
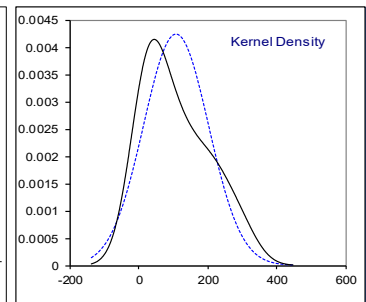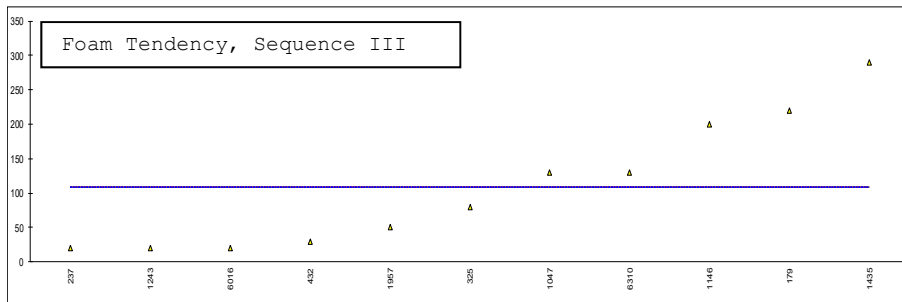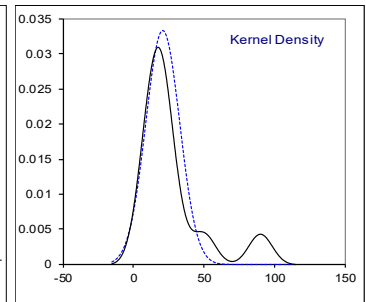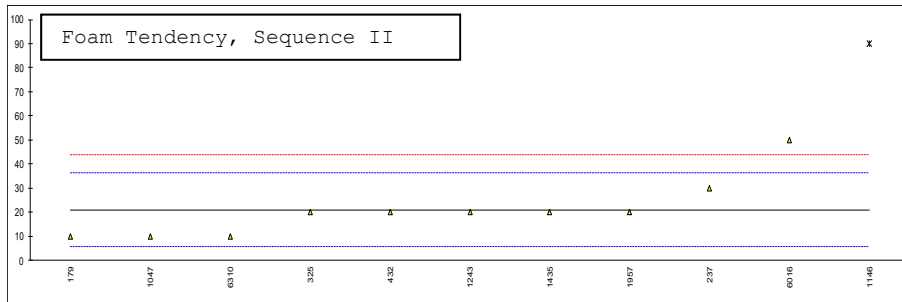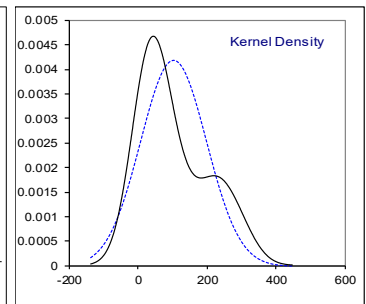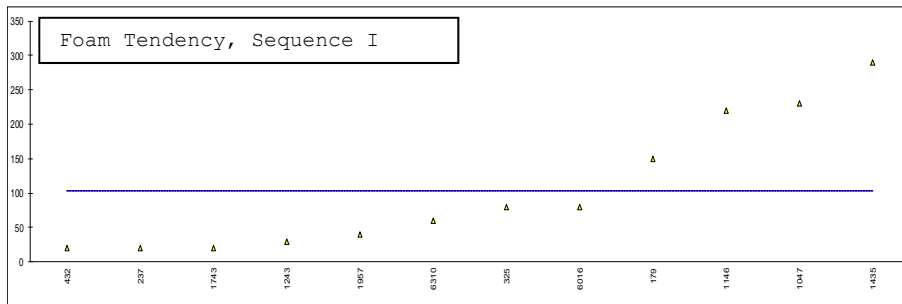
Determination of Foaming Stability, Sequence I, II and III (10 min. settling period) on sample #20066; results in mL

lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	20	f+?	-----	0		-----	0		-----
237	D892	0		-----	0		-----	0		-----
325	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432	D892	0		-----	0		-----	0		-----
496		-----		-----	-----		-----	-----		-----
614		-----		-----	-----		-----	-----		-----
862		-----		-----	-----		-----	-----		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1047	D892	0		-----	0		-----	0		-----
1146	ISO6247	0		-----	0		-----	0		-----
1243	D892	0		-----	0		-----	0		-----
1435		0		-----	0		-----	0		-----
1743	ISO6247	0		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
1957	D892	0		-----	0		-----	0		-----
6016	D892	0		-----	0		-----	0		-----
6253		-----		-----	-----		-----	-----		-----
6310	D892	0		-----	0		-----	0		-----
	n	11			11			11		
	mean (n)	0			0			0		

Determination of Kinematic Viscosity at 40°C on sample #20066; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	31.3		-0.58	
179	D445	31.30		-0.58	
237	D445	31.59		1.54	
325	D445	31.25		-0.95	
349	D445	31.29		-0.66	
432		-----		-----	
496	D445	31.499		0.87	
614	D7042	31.50		0.88	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047	ISO3104	31.38		0.00	
1146	D445	31.310		-0.51	
1243	D7279 corr. to D445	31.33		-0.36	
1435	D7042	31.553		1.27	
1743	D7279 corr. to D445	31.11		-1.97	
1875	D7042	31.3035		-0.56	
1957	D7042	31.93	C,G(0.05)	4.02	first reported 32.04
6016	D7042	31.681		2.20	
6253		-----		-----	
6310	D7279 corr. to D445	31.3		-0.58	

normality	OK
n	15
outliers	1
mean (n)	31.3798
st.dev. (n)	0.15202
R(calc.)	0.4257
st.dev.(D445:19a)	0.13688
R(D445:19a)	0.3833

Kernel Density
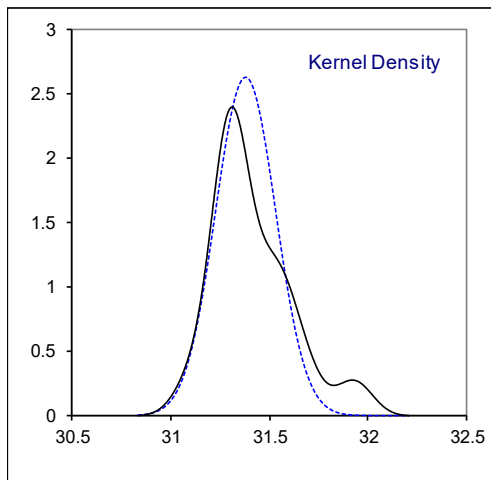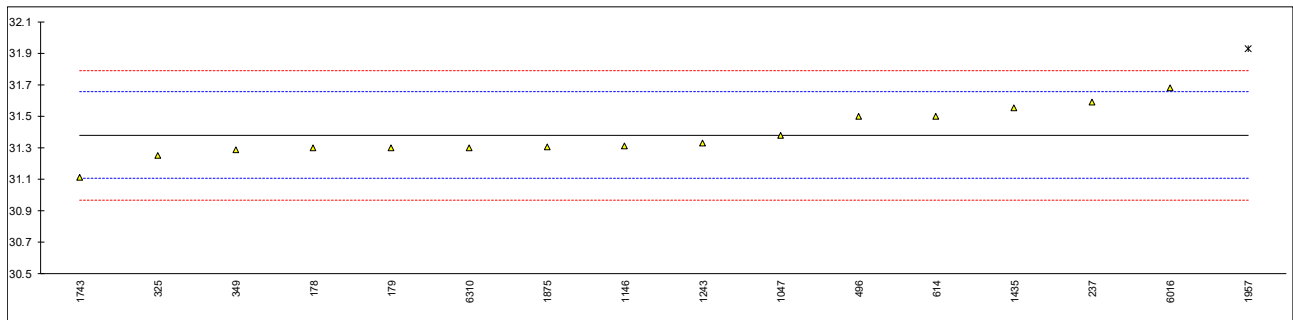
Determination of Kinematic Viscosity at 100°C on sample #20066; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	5.40		-0.15	
179	D445	5.39		-0.53	
237	D445	5.340		-2.40	
325	D445	5.425		0.79	
349	D445	5.397		-0.26	
432		-----		-----	
496	D445	5.3913		-0.48	
614	D7042	5.417		0.49	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047	ISO3104	5.428		0.90	
1146	D445	5.3878		-0.61	
1243	D7279 corr. to D445	5.39		-0.53	
1435	D7042	5.409		0.19	
1743	D7279 corr. to D445	5.261	C,G(0.05)	-5.37	first reported 5.247
1875	D7042	5.4182		0.53	
1957	D7042	5.512	C,G(0.05)	4.05	first reported 5.555
6016	D7042	5.463		2.21	
6253		-----		-----	
6310	D7279 corr. to D445	5.4		-0.15	

normality	not OK
n	14
outliers	2
mean (n)	5.4040
st.dev. (n)	0.02758
R(calc.)	0.0772
st.dev.(D445:19a)	0.02663
R(D445:19a)	0.0746

Kernel Density
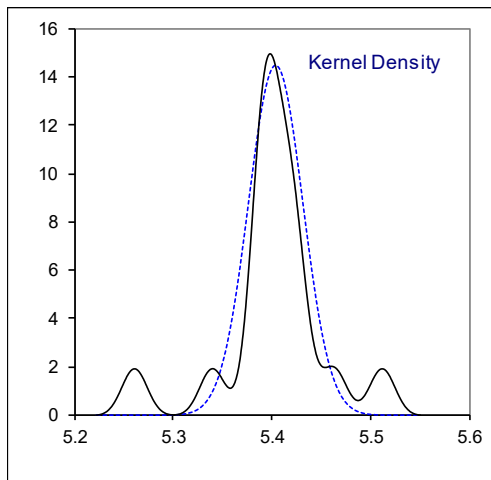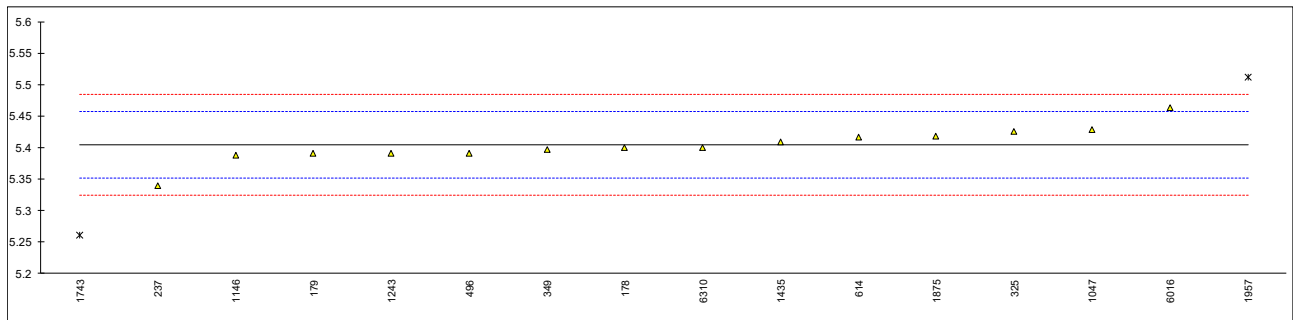
Determination of Viscosity Index on sample #20066

lab	method	value	mark	z(targ)	remarks
178	D2270	107		0.78	
179	D2270	106		-0.62	
237	D2270	104	E, C	-3.42	first reported 101.2, iis calculated 101
325	D2270	108		2.18	
349	D2270	106		-0.62	
432		-----		-----	
496	D2270	104.9		-2.16	
614		-----		-----	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047	ISO2909	108		2.18	
1146	D2270	106		-0.62	
1243	ISO2909	105.8		-0.90	
1435	D2270	105.5		-1.32	
1743	ISO2909	100	E,C,G(0.01)	-9.02	first reported 99, iis calculated 99
1875	ISO2909	107.75		1.83	
1957	D2270	109.1	ex,C	3.72	first reported 111.1, excluded as outlier in Viscosity at 100°C
6016	D2270	107.8		1.90	
6253		-----		-----	
6310	D2270	107		0.78	

normality	OK
n	13
outliers	1 (+1 ex)
mean (n)	106.44
st.dev. (n)	1.267
R(calc.)	3.55
st.dev.(D2270:10)	0.714
R(D2270:10)	2
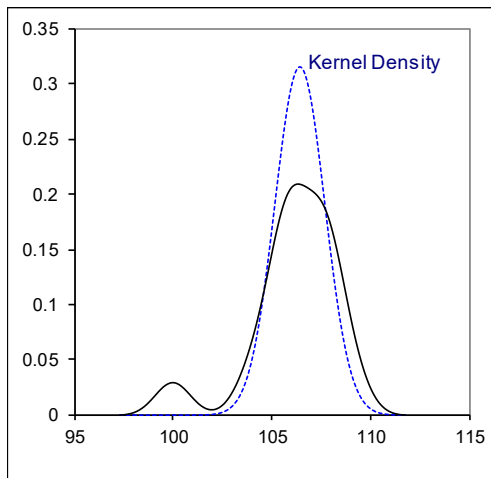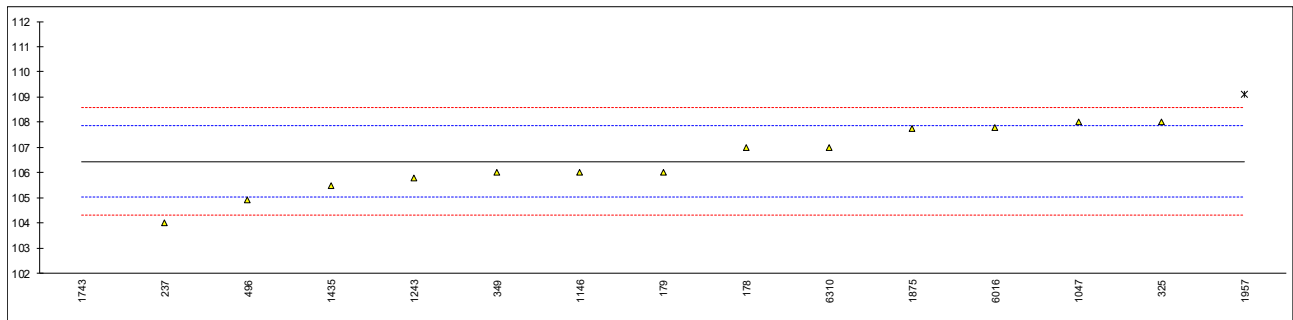
Determination of Pour Point, Manual on sample #20066; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-18		-0.70	
179	D97	-15		0.23	
237		-----		-----	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614	D97	-18		-0.70	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047		-----		-----	
1146		-----		-----	
1243		-----		-----	
1435	ISO3016	-12		1.17	
1743		-----		-----	
1875		-----		-----	
1957		-----		-----	
6016		-----		-----	
6253		-----		-----	
6310		-----		-----	

normality	unknown
n	4
outliers	0
mean (n)	-15.75
st.dev. (n)	2.872
R(calc.)	8.04
st.dev.(D97:17b)	3.214
R(D97:17b)	9
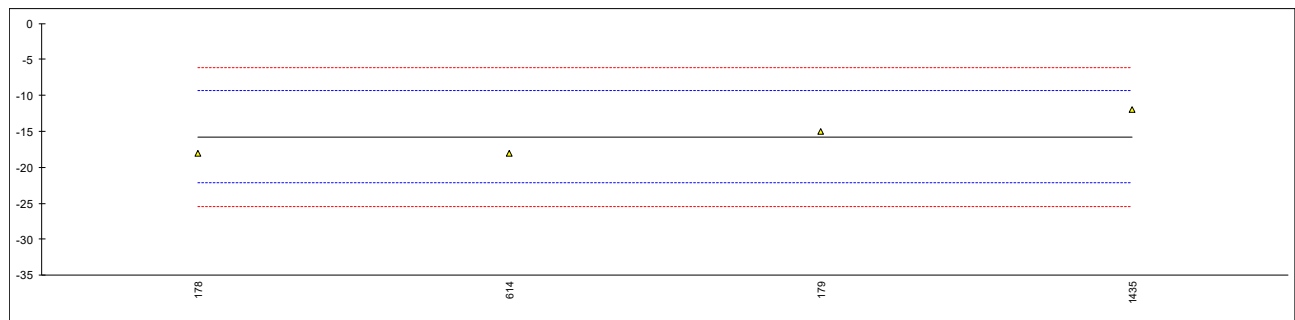
Determination of Pour Point, Automated, 1°C interval on sample #20066; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
325	D5950	-12		0.78	
349		-----		-----	
432		-----		-----	
496	D5950	-13		0.16	
614		-----		-----	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047		-----		-----	
1146		-----		-----	
1243	D7346	-15.3		-1.27	
1435		-----		-----	
1743	NF T60-105	-12		0.78	
1875		-----		-----	
1957		-----		-----	
6016		-----		-----	
6253		-----		-----	
6310	D5950	-14		-0.46	

normality	unknown
n	5
outliers	0
mean (n)	-13.26
st.dev. (n)	1.410
R(calc.)	3.95
st.dev.(D5950:14)	1.607
R(D5950:14)	4.5
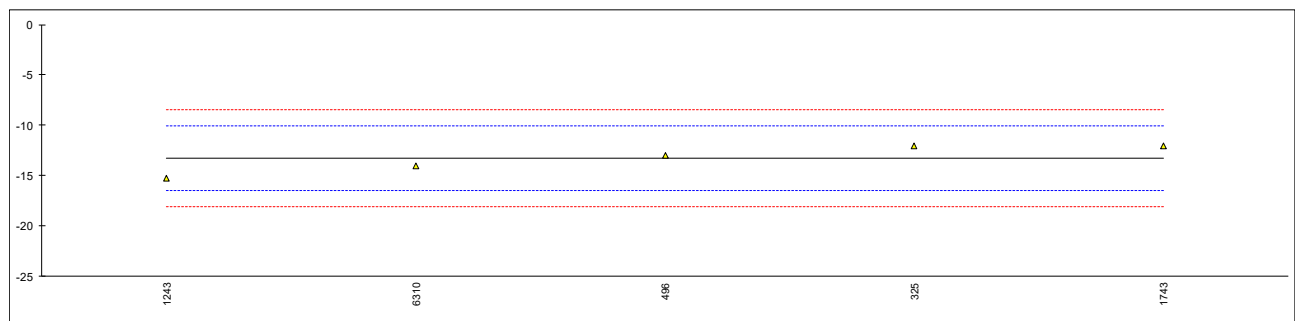
Determination of Sulfur on sample #20066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5453	285		-0.13	
325	D5185	245		-1.65	
349	D2622	294		0.21	
432		-----		-----	
496	D4294	2980	G(0.01)	101.98	
614		-----		-----	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047		-----		-----	
1146	D4294	270		-0.70	
1243	ISO8754	299		0.40	
1435		-----		-----	
1743		-----		-----	
1875	DIN51724	345		2.14	
1957		-----		-----	
6016		-----		-----	
6253		-----		-----	
6310	D7751	281	C	-0.28	first reported 28.1

normality	unknown
n	7
outliers	1
mean (n)	288.43
st.dev. (n)	30.648
R(calc.)	85.81
st.dev.(D4294:16e1)	26.392
R(D4294:16e1)	73.90
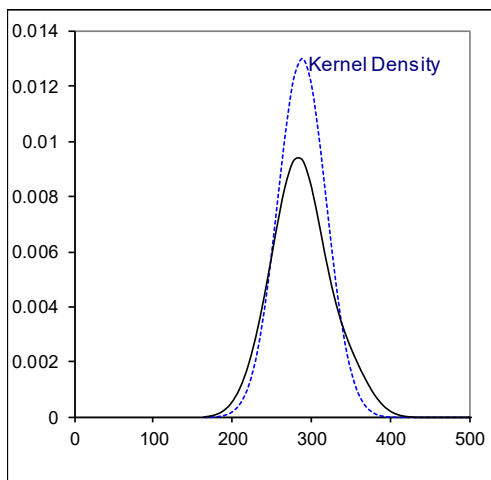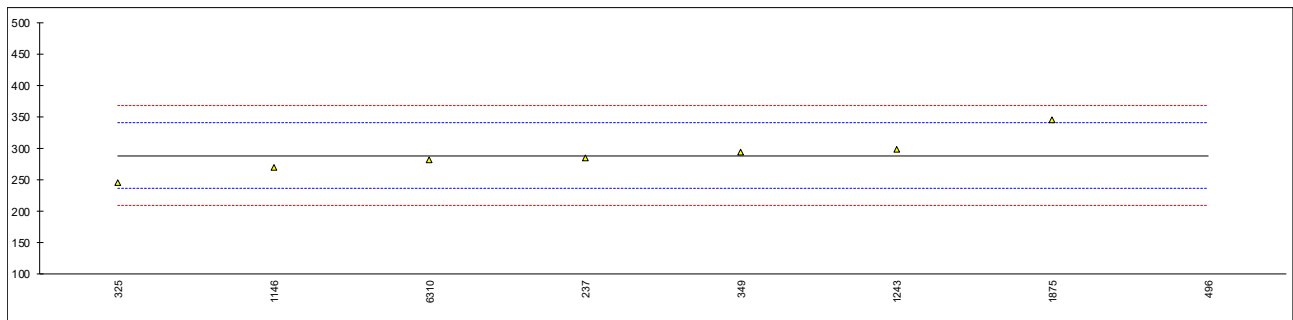
Determination of Water on sample #20066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-A	48		0.19	
179	D6304-C	48		0.19	
237	D6304-C	45		0.13	
325	D6304-C	24		-0.26	
349	D6304-A	37		-0.02	
432		-----		-----	
496	D6304-C	25		-0.24	
614	D6304-C	41		0.06	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047		-----		-----	
1146	D6304-C	20		-0.34	
1243	ISO12937	13		-0.47	
1435	D6304-A	39		0.02	
1743	ISO12937	50		0.22	
1875	ISO12937	62	C	0.45	first reported 183
1957	D6304-A	39.8		0.03	
6016	D6304-A	56		0.34	
6253		-----		-----	
6310	D6304-C	22		-0.30	

normality	OK	
n	15	
outliers	0	
mean (n)	37.99	
st.dev. (n)	14.336	
R(calc.)	40.14	
st.dev.(D6304:16e1)	53.493	
R(D6304:16e1)	149.78	
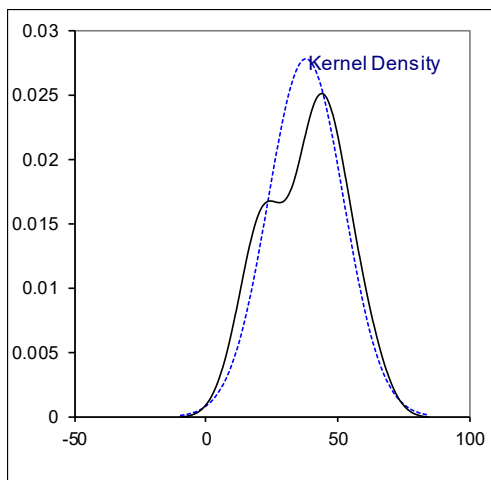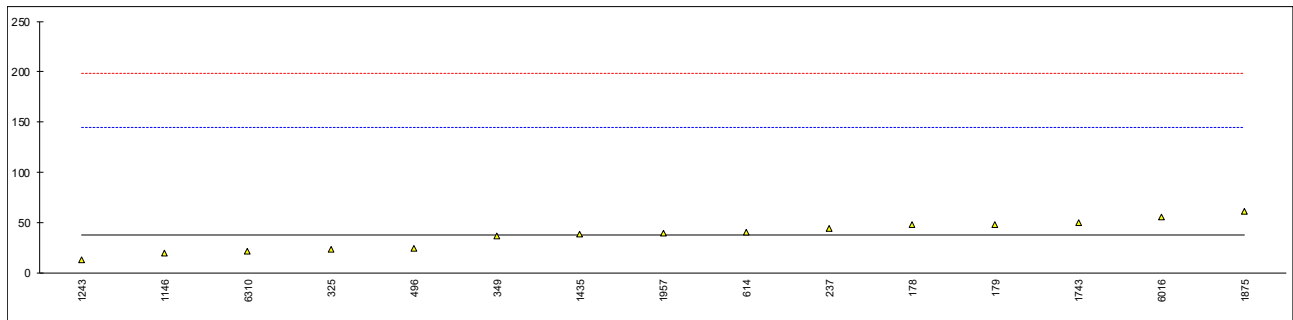
Determination of Water Separability at 54°C, distilled water on sample #20066; results in minutes

lab	method	≤ 3 mL emulsion mark	z(targ)	37 mL of water mark	z(targ)	complete break (40-40-0) mark	z(targ)	test aborted	time test aborted
178		-----	-----	-----	-----	-----	-----	-----	-----
179		-----	-----	-----	-----	7	-0.34	NO	-----
237	D1401	7.29	-0.08	7.47	-0.06	12.10	0.37	NO	-----
325	D1401	8	0.02	8	0.02	10	0.08		-----
349		-----	-----	-----	-----	-----	-----		-----
432	D1401	10	0.30	10	0.30	>30	-----	YES	>30
496	D1401	-----	-----	-----	-----	10	0.08	NO	10
614	D1401	8.75	0.13	8.75	0.12	9.50	0.01	NO	-----
862		-----	-----	-----	-----	-----	-----		-----
912		-----	-----	-----	-----	-----	-----		-----
962		-----	-----	-----	-----	-----	-----		-----
963		-----	-----	-----	-----	-----	-----		-----
1011		-----	-----	-----	-----	-----	-----		-----
1047		10	0.30	10	0.30	-----	-----		10
1146		8	0.02	-----	-----	-----	-----	YES	8
1243	ISO6614	7	-0.12	7	-0.12	-----	-----	YES	30
1435	D1401	3	-0.68	3	-0.68	-----	-----	NO	-----
1743	ISO6614	-----	-----	-----	-----	10	0.08	NO	-----
1875		-----	-----	-----	-----	-----	-----		-----
1957		7.62	-0.03	7.82	-0.01	9	-0.06	NO	-----
6016	D1401	6.7	-0.16	6.7	-0.16	7.2	-0.31	NO	-----
6253		-----	-----	-----	-----	-----	-----		-----
6310	D1401	10	0.30	10	0.30	10	0.08	NO	10
normality		not OK		not OK		OK			
n		11		10		9			
outliers		0		0		0			
mean (n)		7.85		7.87		9.42			
st.dev. (n)		2.016		2.119		1.561			
R(calc.)		5.65		5.93		4.37			
st.dev.(D1401:19)		7.143		7.143		7.143			
R(D1401:19)		20		20		20			
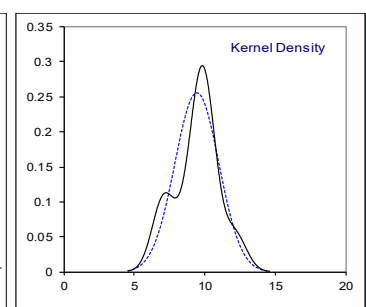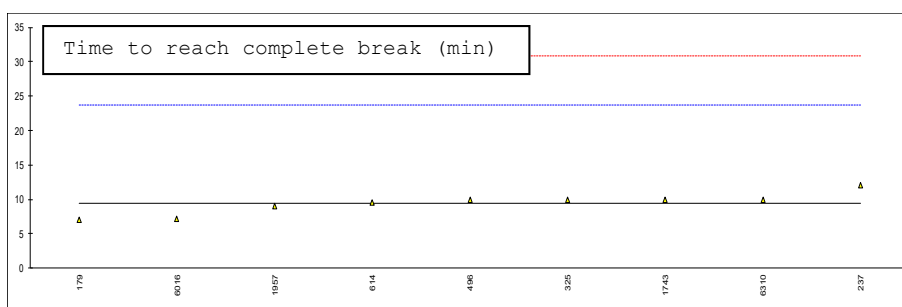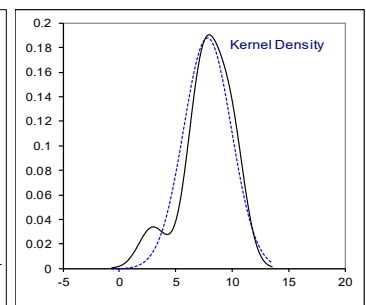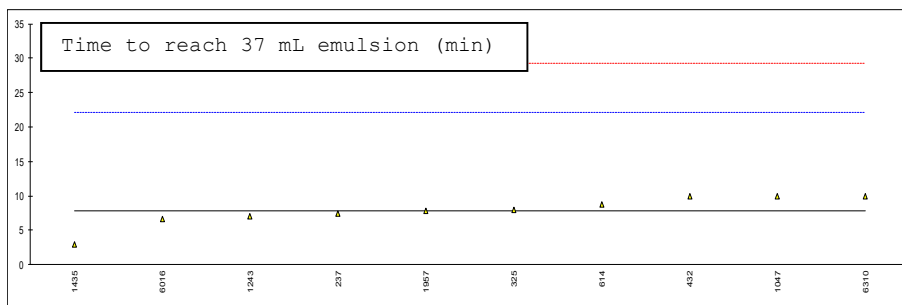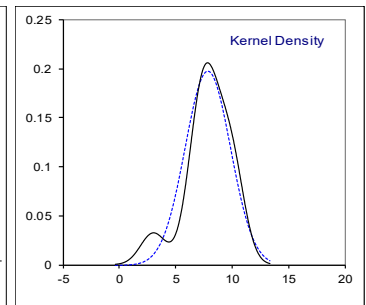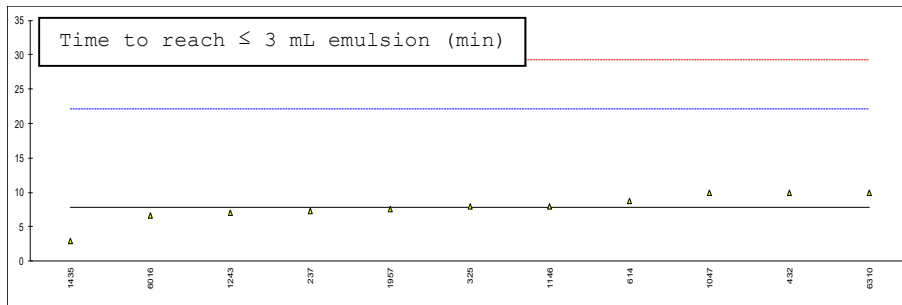
Time to reach ≤ 3 mL emulsion (min) — Kernel Density

Time to reach 37 mL emulsion (min) — Kernel Density

Time to reach complete break (min) — Kernel Density

Determination of Water Separability at 54°C, distilled water on sample #20066; results in mL

lab	method	oil phase	mark	z(targ)	water phase	mark	z(targ)	emulsion phase	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		40		-----	40		-----	0		-----
237	D1401	40		-----	40		-----	0		-----
325	D1401	-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432	D1401	43		-----	37		-----	0		-----
496	D1401	41		-----	39		-----	-----		-----
614	D1401	40		-----	40		-----	0		-----
862		-----		-----	-----		-----	-----		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1047		42		-----	37		-----	1		-----
1146		41		-----	39		-----	0		-----
1243	ISO6614	41		-----	39		-----	0		-----
1435	D1401	-----		-----	-----		-----	-----		-----
1743	ISO6614	40		-----	40		-----	0		-----
1875		-----		-----	-----		-----	-----		-----
1957		40		-----	40		-----	0		-----
6016	D1401	40		-----	40		-----	0		-----
6253		-----		-----	-----		-----	-----		-----
6310	D1401	40		-----	40		-----	0		-----

Determination of Calcium as Ca on sample #20066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		1		-----	
179	D5185	<1		-----	
237	D5185	<40		-----	
325	D5185	<1		-----	
349	D5185	0		-----	
432	D5185	<1		-----	
496	D5185	<40		-----	
614	D5185	<1		-----	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047	D5185	<2,0		-----	
1146	In house	0.2		-----	
1243	DIN51399-1	<1		-----	
1435		<1		-----	
1743	D5185	0.51		-----	
1875		<2		-----	
1957	D5185	<1		-----	
6016	D5185	<1.0		-----	
6253		-----		-----	
6310	D7751	<1		-----	

n	17			
mean(n)	<40		Application range D5185:18: 40 – 9000 mg/kg	

Determination of Phosphorus as P on sample #20066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		50		-0.13	
179	D5185	51		-0.04	
237	D5185	48		-0.31	
325	D5185	42		-0.86	
349	D5185	42		-0.86	
432	D5185	49.67		-0.16	
496	D5185	48.78		-0.24	
614	D5185	65.6		1.29	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047	D5185	53.0		0.14	
1146	In house	49.9		-0.14	
1243	DIN51399-1	51.3		-0.01	
1435		49.97		-0.13	
1743	D5185	51.7		0.02	
1875		62		0.96	
1957	D5185	25	G(0.05)	-2.40	
6016	D5185	56		0.41	
6253		-----		-----	
6310	D7751	52	C	0.05	

normality	suspect
n	16
outliers	1
mean (n)	51.43
st.dev. (n)	6.015
R(calc.)	16.84
st.dev.(D5185:18)	11.014
R(D5185:18)	30.84

Kernel Density
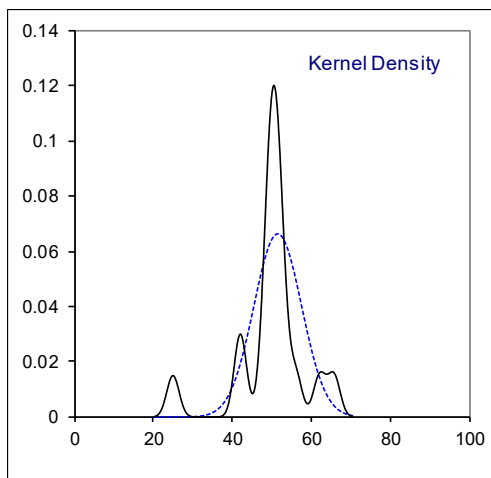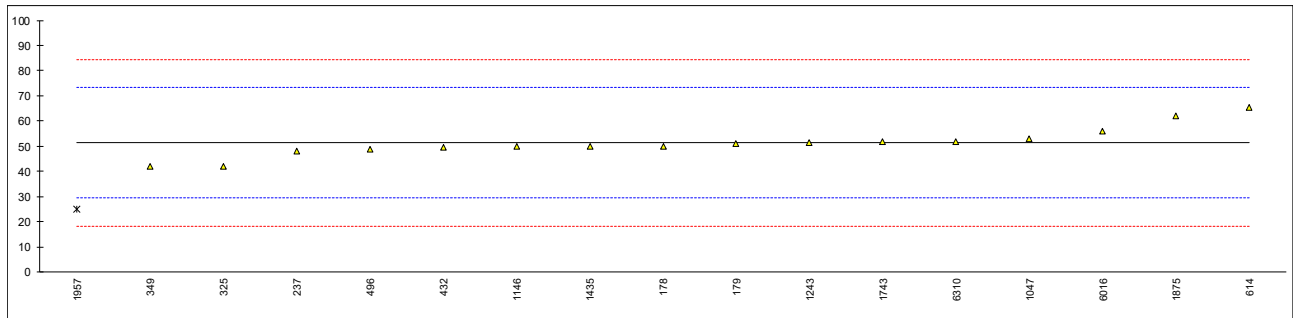
Determination of Zinc as Zn on sample #20066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		1		-----	
179	D5185	<1		-----	
237	D5185	<60		-----	
325	D5185	<1		-----	
349	D5185	0		-----	
432	D5185	<1		-----	
496	D5185	<60		-----	
614	D5185	<1		-----	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1047	D5185	<2,0		-----	
1146	In house	0.1		-----	
1243	DIN51399-1	<1		-----	
1435		<1		-----	
1743	D5185	0.51		-----	
1875		1		-----	
1957	D5185	<1		-----	
6016	D5185	<1.0		-----	
6253		-----		-----	
6310	D7751	<1		-----	
	n	17			
	mean (n)	<60			Application range D5185:18: 60 – 1600 mg/kg

APPENDIX 2

Number of participants per country

    1 lab in AUSTRALIA

    1 lab in AUSTRIA

    3 labs in BELGIUM

    1 lab in CHINA, People's Republic

    1 lab in FRANCE

    3 labs in GERMANY

    1 lab in INDIA

    1 lab in KAZAKHSTAN

    1 lab in MALAYSIA

    1 lab in MOROCCO

    1 lab in NETHERLANDS

    1 lab in NIGERIA

    1 lab in POLAND

    1 lab in PORTUGAL

    2 labs in SAUDI ARABIA

    1 lab in SPAIN

    2 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations

C   = final test result after checking of first reported suspect test result
D(0.01)   = outlier in Dixon's outlier test
D(0.05)   = straggler in Dixon's outlier test
G(0.01)   = outlier in Grubbs' outlier test
G(0.05)   = straggler in Grubbs' outlier test
DG(0.01)   = outlier in Double Grubbs' outlier test
DG(0.05)   = straggler in Double Grubbs' outlier test
R(0.01)   = outlier in Rosner's outlier test
R(0.05)   = straggler in Rosner's outlier test
E   = possibly an error in calculations
W   = test result withdrawn on request of participant
ex   = test result excluded from statistical evaluation
n.a.   = not applicable
n.e.   = not evaluated
n.d.   = not detected
fr.   = first reported
f+?   = possible false positive test results?
f-?   = possible false negative test results?
SDS   = Safety Data Sheet

Literature

1   iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2   ASTM E178:89
3   ASTM E1301:89
4   ISO5725:86
5   ISO5725, parts 1-6, 1994
6   ISO13528:05
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   IP367:84
10   DIN38402 T41/42
11   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12   J.N. Miller, Analyst, 118, 455, (1993)
13   Analytical Methods Committee Technical brief, No 4, January 2001
14   P.J. Lowthian and M.Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
15   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
16   Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)