Institute for Interlaboratory Studies

Results of Proficiency Test
Turbine Oil (fresh)
May 2022

CONTENTS

Appendices:

1   INTRODUCTION

Since 2018 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of fresh Turbine Oil every year. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of fresh Turbine Oil.

In this interlaboratory study 29 laboratories in 20 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the fresh Turbine Oil 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 fresh Turbine Oil in a 1-liter bottle labelled #22076. 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 110 liters of fresh Turbine Oil was obtained from a third party. After homogenization 40 amber glass bottles of 1 L were filled and labelled #22076.
The homogeneity of the subsamples was checked by determination of Density at 15 °C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15 °C in kg/L
sample #22076-1	0.86282
sample #22076-2	0.86282
sample #22076-3	0.86282
sample #22076-4	0.86282
sample #22076-5	0.86282
sample #22076-6	0.86281
sample #22076-7	0.86282
sample #22076-8	0.86282

Table 1: homogeneity test results of subsamples #22076

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)	0.00001
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #22076

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

To each of the participating laboratories one 1 L bottle of fresh Turbine Oil labelled #22076 was sent on April 13, 2022. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of 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: Total Acid Number, Air Release time at 50 °C, Density at 15 °C, Flash Point C.O.C., Foam Characteristics (Foaming Tendency, Foam 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 Foam 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 reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

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

3.1 STATISTICS

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

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

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

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

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

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

Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3   Z-SCORES

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

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

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

The z-scores were calculated according to:

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

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

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

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

4   EVALUATION

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. Three participants reported test results after the extended reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 26 participants reported 377 numerical test results. Observed were 16 outlying test results, which is 4.2%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all 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 in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D2270) and an added designation for the year that the test method was adopted or revised (e.g. D2270:10). When a method has been reapproved an "R" will be added and the year of approval (e.g. D2270:10R16).

Total Acid Number: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D664-A:18e2 IP and BEP at 60 mL, but not with IP and BEP at 125 mL.
When the test results for IP and BEP were evaluated separately, the calculated reproducibilities of the test results are also not in agreement with the end points at 125 mL titration volume.
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 problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D3427:19.

Density at 15 °C: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers 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 but one test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D92:18.

Foam Characteristics (Tendency and Stability): This determination was problematic. In total one statistical outlier was observed. It was decided not to calculate z-scores for Foaming Tendency at sequence I and III due to the large difference between Calculated and Reference reproducibility. The Foaming Tendency determination for sequence II after rejection of the statistical outlier is in agreement with the requirements of ASTM D892:18.
All reported test results for Foam Stability were zero. Therefore, it was decided not to calculate z-scores.
The determination of the Foam 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. About 40% of the reporting participants used the sample as received and 54% after agitation. About 80% of the reporting participants used a metal diffuser.

Kinematic Viscosity at 40 °C: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:21e1.

Kinematic Viscosity at 100 °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 D445:21e1.

Viscosity Index: This determination was very problematic. No statistical outliers were observed but two test results were excluded. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ASTM D2270:10R16.

Pour Point Manual: 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 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 D5950:14R20.

Sulfur: 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 D4294:21.

Water: 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 D6304:20 procedure B and A, but not with procedure C.

Water Separability at 54 °C, distilled water: This determination was not problematic. Three statistical outliers were observed over 3 parameters. The calculated reproducibilities after rejection of the statistical outliers are in agreement with the requirements of ASTM D1401:21.

Calcium: This determination was not problematic. All reporting participants agreed on a consensus value below the application range of ASTM D5185:18. Therefore, no z-scores are 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 was not problematic. All reporting participants agreed on
                         a consensus value below the application range of ASTM D5185:18.
                         Therefore, no z-scores are calculated.

4.2   PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	18	0.159	0.063	0.089
Air Release time at 50 °C	minutes	13	3.2	2.6	2.2
Density at 15 °C	kg/L	22	0.8629	0.0005	0.0005
Flash Point C.O.C.	°C	18	219.7	15.4	18
Foaming Tendency Seq. I	mL	15	142.0	334.0	(56.4)
Foaming Tendency Seq. II	mL	12	15.4	13.9	18.8
Foaming Tendency Seq. III	mL	13	123.1	276.3	(73.7)
Foam Stability Seq. I	mL	15	0	n.e.	n.e.
Foam Stability Seq. II	mL	13	0	n.e.	n.e.
Foam Stability Seq. III	mL	13	0	n.e.	n.e.
Kinematic Viscosity at 40 °C	mm^2/s	21	31.342	0.269	0.382
Kinematic Viscosity at 100 °C	mm^2/s	21	5.401	0.099	0.075
Viscosity Index		20	106.09	5.05	2
Pour Point Manual	°C	12	-13.6	5.5	9
Pour Point Automated 1 °C interval	°C	6	-13.5	3.9	4.5
Sulfur	mg/kg	6	282	51	73
Water	mg/kg	20	34.0	23.5	99.5
Water Separability at 54 °C, distilled water					
Time to reach ≤ 3 mL emulsion	minutes	14	8.3	3.9	20
Time to reach 37 mL water	minutes	15	8.4	3.9	20
Time to reach complete break	minutes	11	9.2	3.7	20
Calcium as Ca	mg/kg	19	<10	n.e	n.e.
Phosphorus as P	mg/kg	18	49.5	7.1	30.3
Zinc as Zn	mg/kg	19	<10	n.e	n.e.

Table 3: reproducibilities of tests on sample #22076

For results between brackets no z-scores are calculated.

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2022 WITH PREVIOUS PTS

	May 2022	May 2021	May 2020	May 2019	May 2018
Number of reporting laboratories	26	27	17	20	19
Number of test results	377	368	254	271	281
Number of statistical outliers	16	8	8	4	12
Percentage of statistical outliers	4.2%	2.2%	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 to the requirements of the reference test methods. The conclusions are given in the following table.

Parameter	May 2022	May 2021	May 2020	May 2019	May 2018
Total Acid Number	+	--	+/-	-	++
Air Release time at 50 °C	-	+	+	-	++
Density at 15 °C	+/-	+	+/-	+	-
Flash Point C.O.C.	+	-	+	+	++
Foaming Tendency Seq. I	(--)	(--)	(--)	(--)	(--)
Foaming Tendency Seq. II	+	+/-	-	+/-	+
Foaming 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	++	+	++	+	++
Calcium as Ca	n.e.	n.e.	n.e.	n.e.	n.e.
Phosphorus as P	++	n.e.	+	n.e.	++
Zinc as Zn	n.e.	n.e.	n.e.	n.e.	n.e.

Table 5: comparison of determinations to the reference test methods

For results between brackets no z-scores are calculated.

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 #22076; results in mg KOH/g

lab	method	value	mark	z(targ)	End point determination	Volume titration solvent
178		-----		-----	---	---
179	D664-A	0.14		-0.60	---	---
237		-----		-----	---	---
325	D664-A	0.16		0.03	---	---
349	D664-A	0.16		0.03	Inflection Point	125 mL
432		-----		-----	---	---
496	D664-B	0.15		-0.28	Buffer End Point pH 10	60 mL
614	D664-A	0.15		-0.28	---	60 mL
862		-----		-----	---	---
912	D974	0.19		0.98	---	---
962		-----		-----	---	---
963	D664-B	0.166		0.22	Inflection Point	60 mL
1017		-----		-----	---	---
1146	D664-A	0.183		0.76	Buffer End Point pH 10	125 mL
1150	BDS9776	0.1766		0.56	---	---
1327		-----		-----	---	---
1384	ISO6618	0.156		-0.09	---	---
1409		-----		-----	---	---
1435	D664-A	0.124		-1.10	Buffer End Point pH 10	---
1743	D664-A	0.12		-1.22	Buffer End Point pH 11	60 mL
1875	ISO6618	0.1773		0.58	---	---
1943	ISO6618	0.179		0.63	---	---
1957	D664-A	0.139		-0.63	Buffer End Point pH 11	125 mL
6002	D664-A	0.163		0.13	Inflection Point	60 mL
6016	D664-A	0.197		1.20	Inflection Point	60 mL
6197		-----		-----	---	---
6253		-----		-----	---	---
6310	D664-A	0.13		-0.91	Buffer End Point pH 10	60 mL
6442		-----		-----	---	---

			BEP only	Inflection point only
normality	OK		unknown	unknown
n	18		6	4
outliers	0		0	0
mean (n)	0.1589		0.1410	0.1715
st.dev. (n)	0.02251		0.02324	0.01718
R(calc.)	0.0630		0.0651	0.0481
st.dev.(D664-A:18e2 IP 60 mL)	0.03180		---	0.03384
R(D664-A:18e2 IP 60 mL)	0.0890		---	0.0948
Compare:				
R(D664-A:18e2 BEP 60 mL)	0.0921		0.0819	---
R(D664-A:18e2 IP 125 mL)	0.0321		---	0.0348
R(D664-A:18e2 BEP 125 mL)	0.0457		0.0403	---
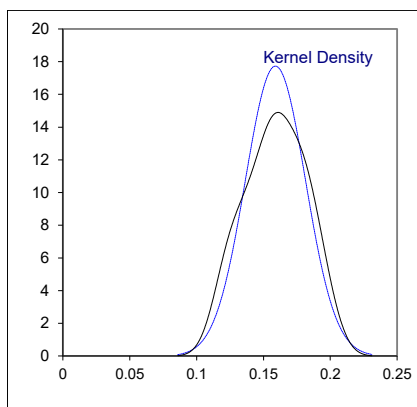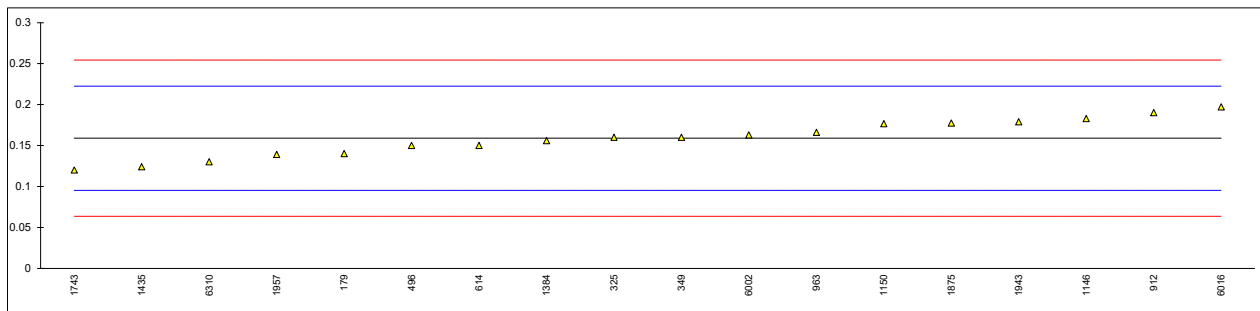
Determination of Air Release time at 50 °C on sample #22076; results in minutes

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D3427	2.2		-1.25	
237		-----		-----	
325	D3427	3.3166666		0.18	
349		-----		-----	
432		-----		-----	
496	D3427	3.4		0.29	
614	D3427	2.1		-1.38	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1017		-----		-----	
1146		-----		-----	
1150		-----		-----	
1327		-----		-----	
1384	ISO9120	2.9		-0.35	
1409	D3427	4.5		1.69	
1435	D3427	4.1		1.18	
1743	ISO9120	2.4		-0.99	
1875		-----		-----	
1943		-----		-----	
1957		-----		-----	
6002	ISO9120	2.98		-0.25	
6016		-----		-----	
6197	D3427	2.7		-0.61	
6253	ISO9120	5.0		2.33	
6310	D3427	2.0		-1.50	
6442	IP313	3.7		0.67	

normality	OK	
n	13	
outliers	0	
mean (n)	3.177	
st.dev. (n)	0.9449	
R(calc.)	2.646	
st.dev.(D3427:19)	0.7828	
R(D3427:19)	2.192	

Kernel Density
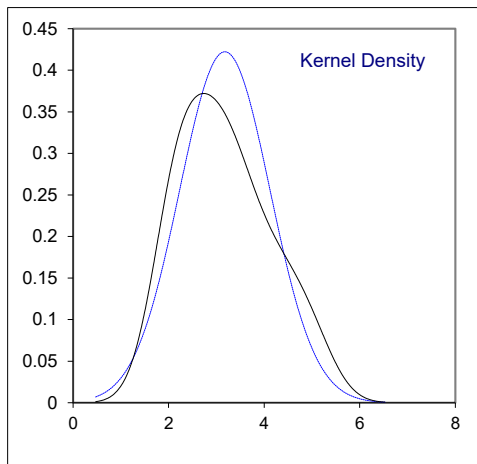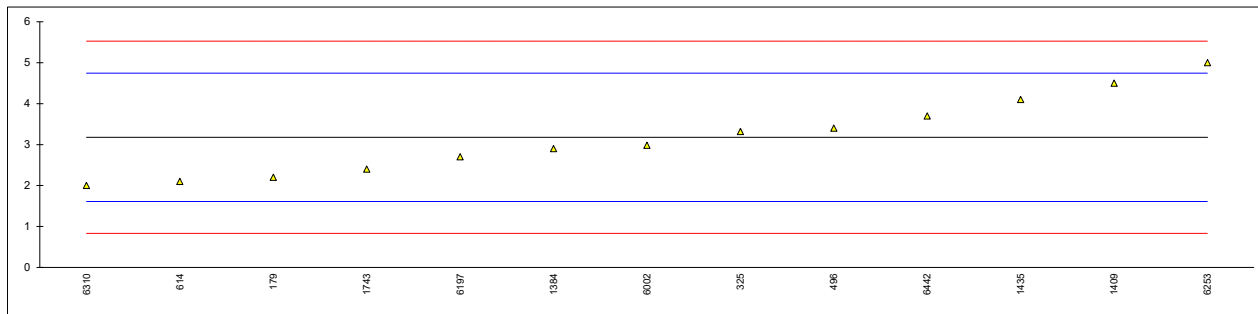
Determination of Density at 15 °C on sample #22076; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D4052	0.8628		-0.44	
179	D4052	0.8629		0.12	
237	D4052	0.8648	R(0.01)	10.76	
325	D4052	0.8629		0.12	
349	D4052	0.86278		-0.55	
432	ISO12185	0.86284		-0.22	
496	ISO12185	0.86291		0.18	
614	D4052	0.8632		1.80	
862		-----		-----	
912	D1298	0.8628		-0.44	
962		-----		-----	
963	D4052	0.8628		-0.44	
1017	ISO12185	0.8632		1.80	
1146	D4052	0.8628		-0.44	
1150	ISO12185	0.8628		-0.44	
1327		-----		-----	
1384	ISO3675	0.863		0.68	
1409		-----		-----	
1435	D4052	0.8630		0.68	
1743	ISO12185	0.8631		1.24	
1875	DIN51757	0.8627		-1.00	
1943	ISO3675	0.8617	C,R(0.01)	-6.60	first reported 0.8622
1957	D4052	0.8620	R(0.01)	-4.92	
6002	ISO12185	0.8628		-0.44	
6016	D4052	0.8631		1.24	
6197	D7042	0.86280		-0.44	
6253	ISO3675	0.8624	C	-2.68	first reported 862.4 kg/L
6310	D4052	0.8629	C	0.12	first reported 862.9 kg/L
6442	D4052	0.8628		-0.44	

normality	suspect
n	22
outliers	3
mean (n)	0.86288
st.dev. (n)	0.000177
R(calc.)	0.00050
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005
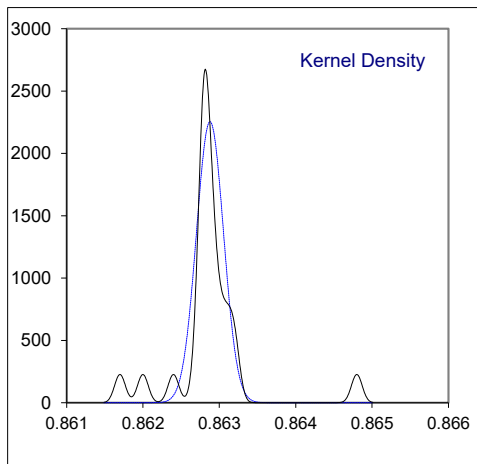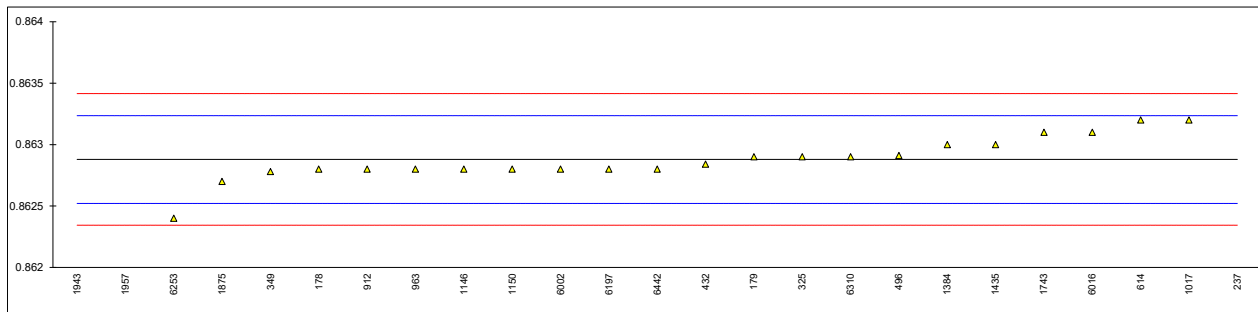
Determination of Flash Point C.O.C. on sample #22076; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D92	216		-0.58	
237	D92	218		-0.27	
325	D92	218		-0.27	
349	D92	217		-0.42	
432		-----		-----	
496	D92	222.0		0.36	
614		-----		-----	
862		-----		-----	
912	D92	212		-1.20	
962		-----		-----	
963	D92	226.0		0.98	
1017	D92	213		-1.04	
1146		-----		-----	
1150	ISO2592	217.3		-0.38	
1327		-----		-----	
1384	ISO2592	220		0.04	
1409		-----		-----	
1435	D92	212.0		-1.20	
1743	ISO2592	230		1.60	
1875	ISO2719-A	212	ex	-1.20	test result excluded as used test method is Flash Point PMcc
1943	ISO2592	218		-0.27	
1957	D92	224		0.67	
6002	ISO2592	215.6		-0.64	
6016		-----		-----	
6197	D92	230		1.60	
6253	ISO2592	224		0.67	
6310		-----		-----	
6442	D92	222		0.36	

normality	OK
n	18
outliers	0 +1ex
mean (n)	219.72
st.dev. (n)	5.505
R(calc.)	15.41
st.dev.(D92:18)	6.429
R(D92:18)	18
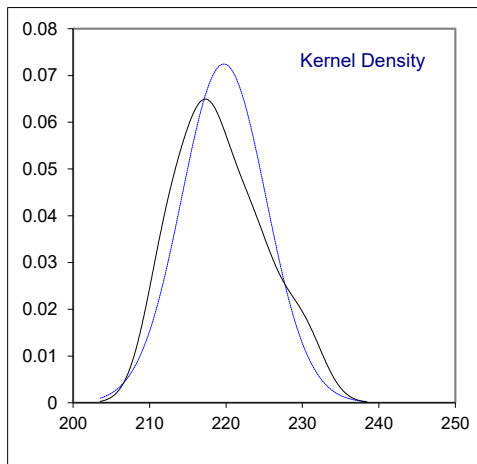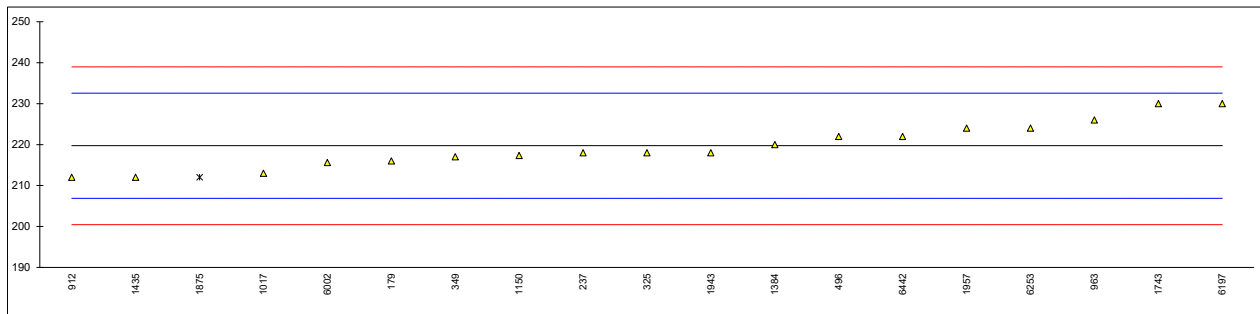
Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #22076; results in mL

lab	method	Sample used	Diffuser	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		---	---	-----		-----	-----		-----	-----		-----
179	D892	---	---	300		-----	40	G(0.01)	3.66	350		-----
237	D892	Heated at 49°C	Metal	170		-----	10		-0.81	110		-----
325	D892	---	---	290		-----	20		0.68	110		-----
349		---	---	-----		-----	-----		-----	-----		-----
432		---	---	-----		-----	-----		-----	-----		-----
496	D892	After agitation	Metal	40		-----	10		-0.81	30		-----
614		---	---	-----		-----	-----		-----	-----		-----
862		---	---	-----		-----	-----		-----	-----		-----
912		---	---	-----		-----	-----		-----	-----		-----
962		---	---	-----		-----	-----		-----	-----		-----
963		---	---	-----		-----	-----		-----	-----		-----
1017		---	---	-----		-----	-----		-----	-----		-----
1146		---	---	-----		-----	-----		-----	-----		-----
1150		---	---	-----		-----	-----		-----	-----		-----
1327		---	---	-----		-----	-----		-----	-----		-----
1384	ISO6247	As received	Metal	300		-----	20		0.68	250		-----
1409	ISO6247	As received	Metal	220		-----	10		-0.81	150		-----
1435	D892	As received	Stone	300		-----	20		0.68	170		-----
1743	ISO6247	As received	Metal	10		-----	-----		-----	-----		-----
1875		---	---	-----		-----	-----		-----	-----		-----
1943		---	---	-----		-----	-----		-----	-----		-----
1957	D892	After agitation	Metal	20		-----	10		-0.81	20		-----
6002	ISO6247	After agitation	Metal	20		-----	10		-0.81	20		-----
6016	D892	After agitation	Stone	120		-----	20		0.68	150		-----
6197	D892	After agitation	Metal	220		-----	20		0.68	160		-----
6253	ISO6247	As received	Stone	30		-----	-----		-----	-----		-----
6310	D892	After agitation	Metal	40		-----	15		-0.06	30		-----
6442	D892	After agitation	Metal	50		-----	20		0.68	50		-----
	normality			OK			OK			OK		
	n			15			12			13		
	outliers			0			1			0		
	mean (n)			142.00			15.42			123.08		
	st.dev. (n)			119.29			4.981			98.690		
	R(calc.)			334.03			13.95			276.33		
	st.dev.(D892:18)			(20.142)			6.708			(26.334)		
	R(D892:18)			(56.40)			18.78			(73.73)		

Foaming Tendency, Sequence I (5 min. blowing period)

Foaming Tendency, Sequence II (5 min. blowing period)

Foaming Tendency, Sequence III (5 min. blowing period)
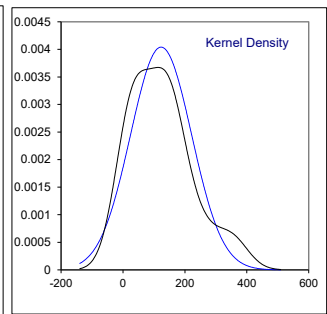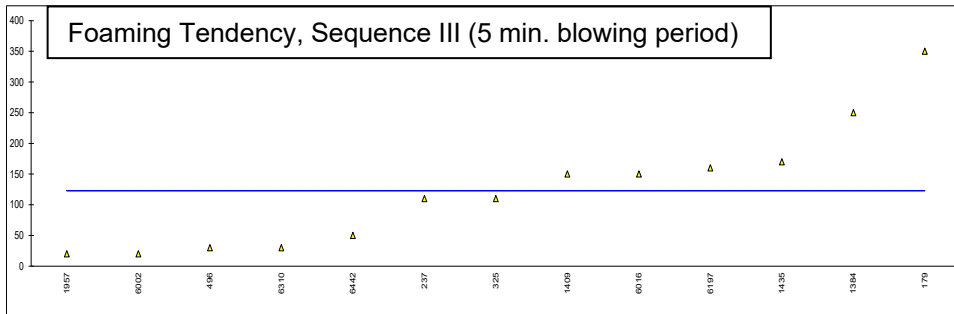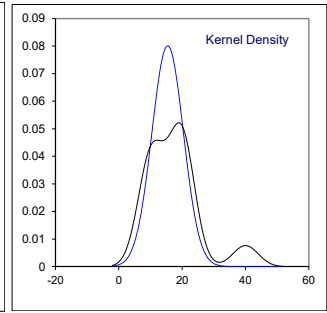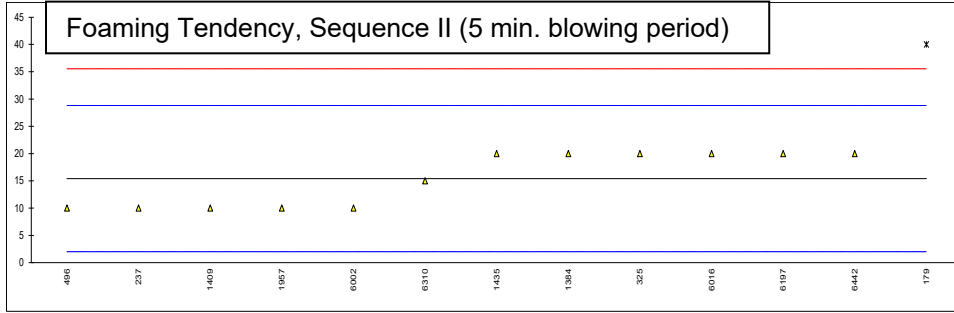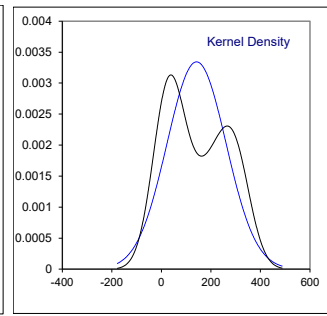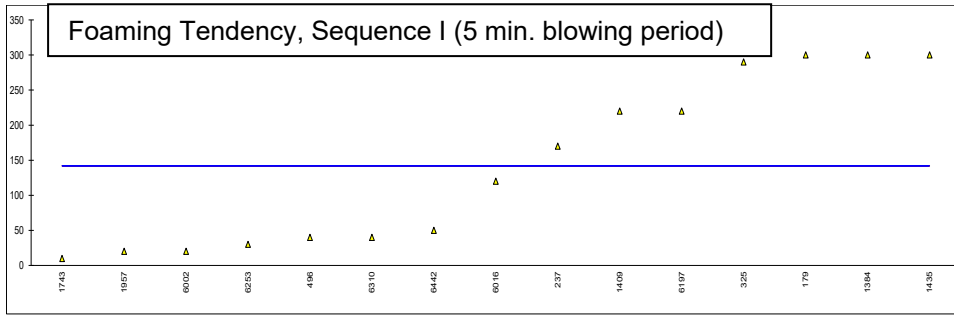
Determination of Foam Stability, Sequence I, II and III (10 min. settling period) on sample #22076; results in mL

lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----			-----			-----		
179	D892	0			0			0		
237	D892	0			0			0		
325	D892	0			0			0		
349		-----			-----			-----		
432		-----			-----			-----		
496	D892	0			0			0		
614		-----			-----			-----		
862		-----			-----			-----		
912		-----			-----			-----		
962		-----			-----			-----		
963		-----			-----			-----		
1017		-----			-----			-----		
1146		-----			-----			-----		
1150		-----			-----			-----		
1327		-----			-----			-----		
1384	ISO6247	0			0			0		
1409		0			0			0		
1435	D892	0			0			0		
1743	ISO6247	0			-----			-----		
1875		-----			-----			-----		
1943		-----			-----			-----		
1957	D892	0			0			0		
6002	ISO6247	0			0			0		
6016	D892	0			0			0		
6197	D892	0			0			0		
6253	ISO6247	0			-----			-----		
6310	D892	0			0			0		
6442	D892	0			0			0		
	n	15			13			13		
	mean (n)	0			0			0		

lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)

Determination of Kinematic Viscosity at 40 °C on sample #22076; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D7279 corrected to D445	30.84	R(0.01)	-3.68	
179	D445	31.37		0.21	
237	D445	31.13		-1.55	
325	D445	31.35		0.06	
349	D445	31.36		0.13	
432		-----		-----	
496	D445	31.424		0.60	
614		-----		-----	
862		-----		-----	
912	D445	31.28	C	-0.45	first reported 31.75
962		-----		-----	
963	D445	31.351		0.07	
1017	D445	31.31		-0.23	
1146	D445	31.387		0.33	
1150	ISO3104	31.43		0.64	
1327		-----		-----	
1384	ISO3104	31.41		0.50	
1409		-----		-----	
1435	D7042	31.542		1.47	
1743	D445	31.25		-0.67	
1875	D7042	31.29		-0.38	
1943	ISO3104	31.37		0.21	
1957	D7042	31.791	R(0.01)	3.29	
6002	ISO3104	31.3464		0.03	
6016	D7042	31.420		0.57	
6197	D445	31.34		-0.01	
6253	ISO3104	31.12		-1.63	
6310	D7279 corrected to D445	31.3		-0.31	
6442	D7042	31.40		0.43	

	normality	suspect
	n	21
	outliers	2
	mean (n)	31.3419
	st.dev. (n)	0.09609
	R(calc.)	0.2690
	st.dev.(D445:21e1)	0.13656
	R(D445:21e1)	0.3824
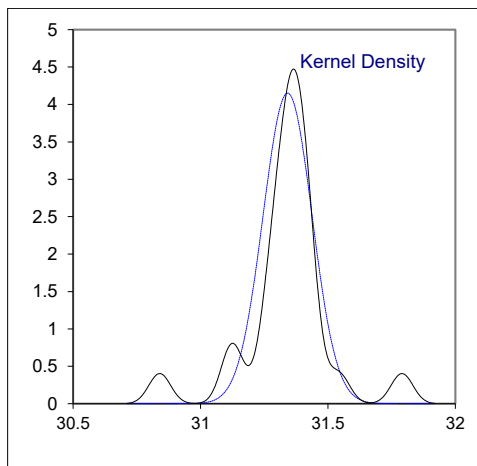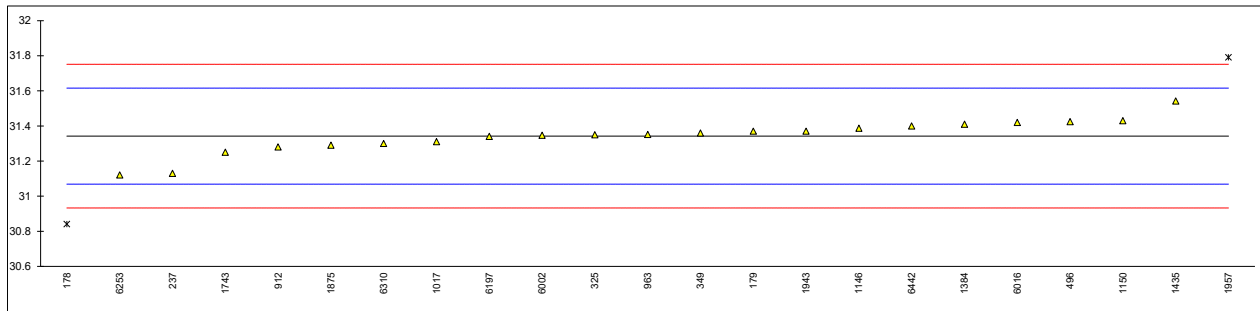
Determination of Kinematic Viscosity at 100 °C on sample #22076; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D7279 corrected to D445	5.46		2.23	
179	D445	5.44		1.48	
237	D445	5.380		-0.78	
325	D445	5.382		-0.70	
349	D445	5.396		-0.18	
432		-----		-----	
496		-----		-----	
614		-----		-----	
862		-----		-----	
912	D445	5.430	C	1.10	first reported 5.528
962		-----		-----	
963	D445	5.443		1.59	
1017	D445	5.388		-0.48	
1146	D445	5.3823		-0.69	
1150	ISO3104	5.421		0.76	
1327		-----		-----	
1384	ISO3104	5.389		-0.44	
1409		-----		-----	
1435	D7042	5.458		2.15	
1743	D445	5.331		-2.62	
1875	D7042	5.4218		0.79	
1943	ISO3104	5.36		-1.53	
1957	D7042	5.5673	R(0.01)	6.26	
6002	ISO3104	5.4044		0.14	
6016	D7042	5.404		0.12	
6197	D445	5.397	C	-0.14	first reported 5.937
6253	ISO3104	5.391		-0.36	
6310	D7279 corrected to D445	5.33		-2.66	
6442	D7042	5.406		0.20	

normality	OK
n	21
outliers	1
mean (n)	5.4007
st.dev. (n)	0.03533
R(calc.)	0.0989
st.dev.(D445:21e1)	0.02662
R(D445:21e1)	0.0745

Kernel Density
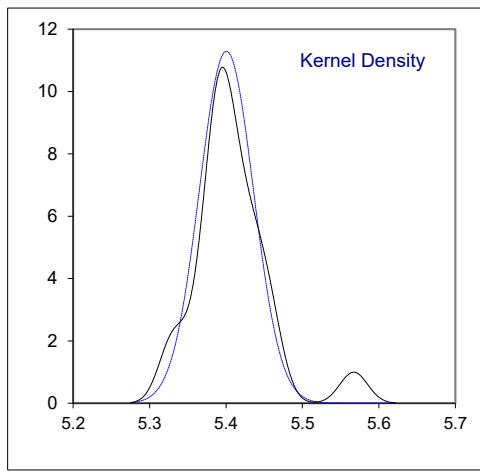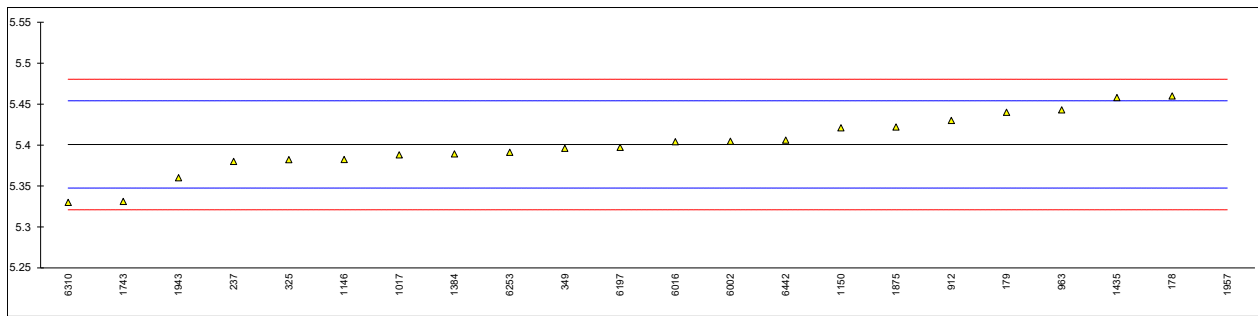
Determination of Viscosity Index on sample #22076

lab	method	value	mark	z(targ)	remarks
178	D2270	112	ex, E	8.28	test result excluded as statistical outlier in KV 40 °C, iis calc. 113
179	D2270	108		2.68	
237	D2270	106		-0.12	
325	D2270	105		-1.52	
349	D2270	106		-0.12	
432		-----		-----	
496		-----		-----	
614		-----		-----	
862		-----		-----	
912	D2270	108	C	2.68	first reported 111
962		-----		-----	
963	D2270	109		4.08	
1017	D2270	106		-0.12	
1146	D2270	105		-1.52	
1150	ISO2909	107		1.28	
1327		-----		-----	
1384	ISO2909	105		-1.52	
1409		-----		-----	
1435	D2270	108.431		3.28	
1743	D2270	103		-4.32	
1875	ISO2909	108		2.68	
1943	ISO2909	103.8		-3.20	
1957	D2270	108.3	ex, E	3.10	test result excl. as statistical outlier in KV 40 and 100 °C, iis calc. 113
6002	ISO2909	106.5		0.58	
6016	D2270	106.041		-0.07	
6197	D2270	106		-0.12	
6253	ISO2909	107		1.28	
6310	D2270	102		-5.72	
6442	D2270	106		-0.12	

normality	OK	
n	20	
outliers	0 +2ex	
mean (n)	106.09	
st.dev. (n)	1.804	
R(calc.)	5.05	
st.dev.(D2270:10R16)	0.714	
R(D2270:10R16)	2	
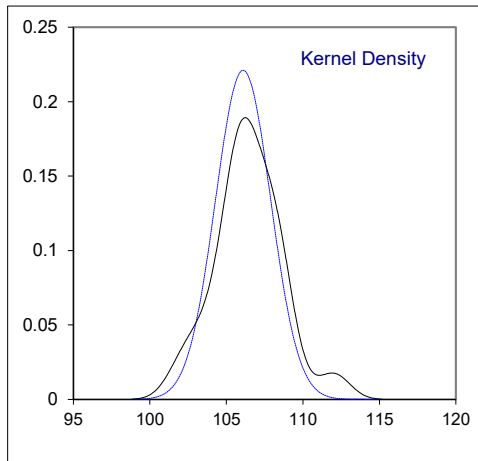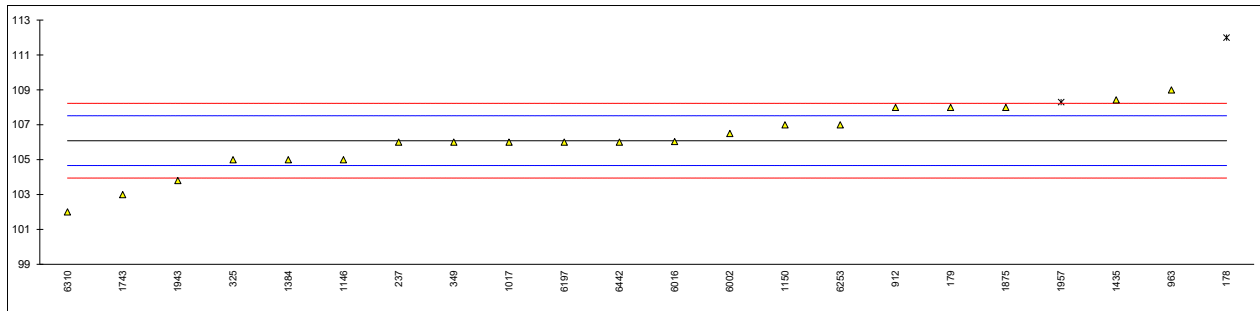
Determination of Pour Point Manual on sample #22076; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-15		-0.44	
179	D97	-30	G(0.01)	-5.11	
237	D97	-15		-0.44	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614	D97	-13		0.18	
862		-----		-----	
912	D97	-12		0.49	
962		-----		-----	
963	D97	-12		0.49	
1017		-----		-----	
1146	D6892	-12		0.49	
1150	ISO3016	-12		0.49	
1327		-----		-----	
1384		-----		-----	
1409		-----		-----	
1435	D97	-15		-0.44	
1743		-----		-----	
1875		-----		-----	
1943		-----		-----	
1957		-----		-----	
6002	ISO3016	-18		-1.37	
6016		-----		-----	
6197	D97	-12		0.49	
6253	NF T 60-105	-15		-0.44	
6310		-----		-----	
6442	D97	-12		0.49	

normality	OK
n	12
outliers	1
mean (n)	-13.58
st.dev. (n)	1.975
R(calc.)	5.53
st.dev.(D97:17b)	3.214
R(D97:17b)	9

Kernel Density
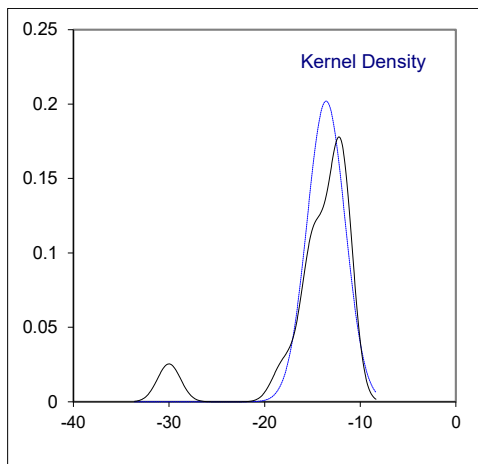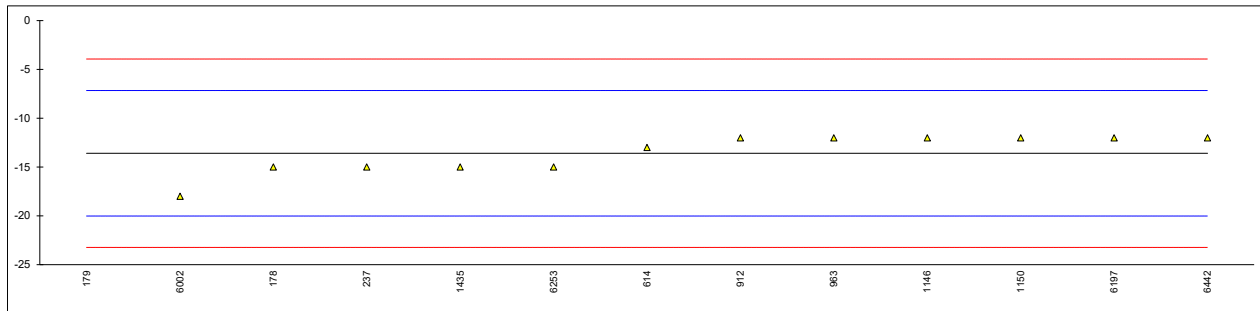
Determination of Pour Point Automated 1 °C interval on sample #22076; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
325	D5950	-12		0.93	
349	D5950	-12		0.93	
432		-----		-----	
496		-----		-----	
614		-----		-----	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1017	D5950	-14		-0.31	
1146		-----		-----	
1150		-----		-----	
1327		-----		-----	
1384		-----		-----	
1409		-----		-----	
1435		-----		-----	
1743	NF T60-105	-15		-0.93	
1875		-----		-----	
1943		-----		-----	
1957		-----		-----	
6002		-----		-----	
6016		-----		-----	
6197		-----		-----	
6253		-----		-----	
6310	D5950	-15		-0.93	
6442	D6892	-13		0.31	

normality	unknown
n	6
outliers	0
mean (n)	-13.50
st.dev. (n)	1.378
R(calc.)	3.86
st.dev.(D5950:14R20)	1.607
R(D5950:14R20)	4.5
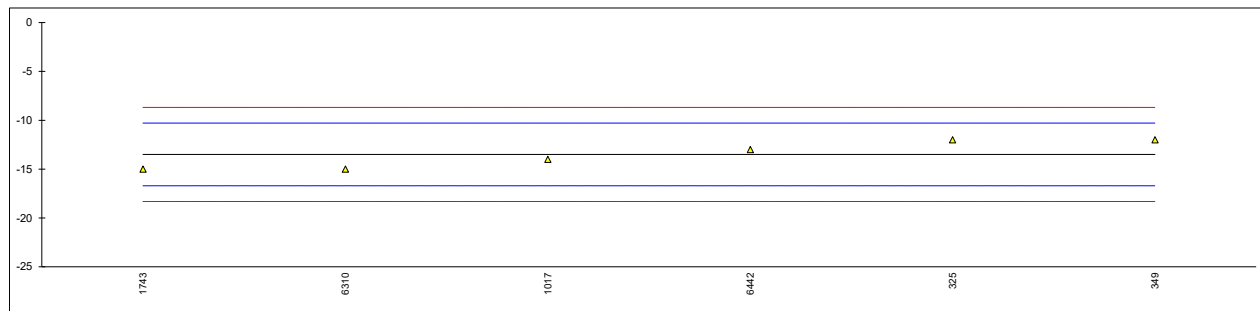
Determination of Sulfur on sample #22076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4294	316		1.30	
237	D4294	270	C	-0.47	first reported 0.0270 mg/kg
325	D5185	274		-0.32	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614		-----		-----	
862		-----		-----	
912	D4294	267.2		-0.58	
962		-----		-----	
963		-----		-----	
1017		-----		-----	
1146		-----		-----	
1150		-----		-----	
1327		-----		-----	
1384	In house	276		-0.24	
1409		-----		-----	
1435		-----		-----	
1743		-----		-----	
1875	DIN51724-1	429	G(0.01)	5.64	
1943		-----		-----	
1957		-----		-----	
6002	D5185	290		0.30	
6016		-----		-----	
6197		-----		-----	
6253		-----		-----	
6310		-----		-----	
6442		-----		-----	

normality	unknown
n	6
outliers	1
mean (n)	282.20
st.dev. (n)	18.346
R(calc.)	51.37
st.dev.(D4294:21)	26.023
R(D4294:21)	72.87
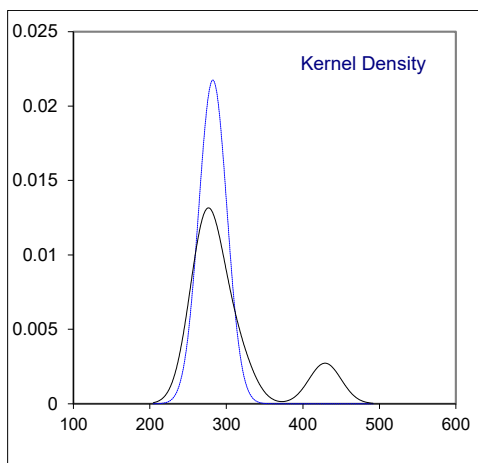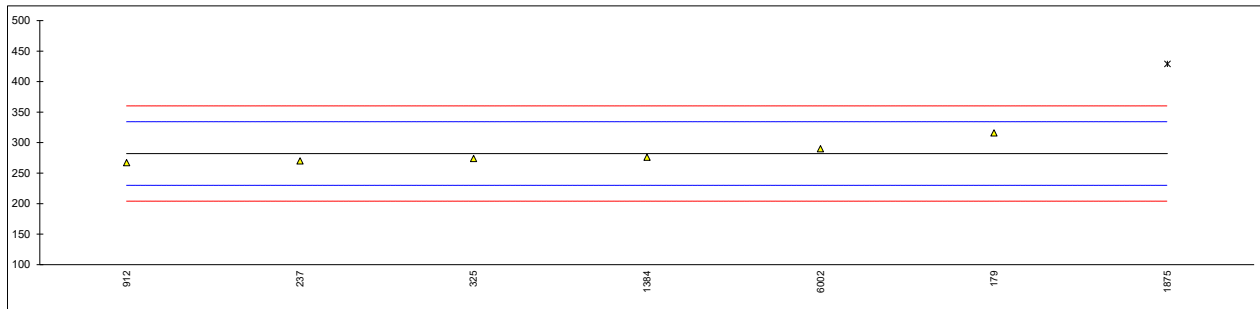
Determination of Water on sample #22076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C:20	20		-0.40	
179	D6304-C:20	27		-0.20	
237	D6304-C:16e1	34		0.00	
325	D6304-C:20	23		-0.31	
349	D6304-A:16e1	38		0.11	
432	D6304-B:20	39.84		0.16	
496	D6304-B:20	20		-0.40	
614	D6304-B:20	39		0.14	
862		-----		-----	
912	D6304-C:20	40		0.17	
962		-----		-----	
963	D6304-A	47.1		0.37	
1017		-----		-----	
1146	D6304-B:20	21		-0.37	
1150	ISO12937	37		0.08	
1327		-----		-----	
1384	In house	42	C	0.22	first reported 62
1409		-----		-----	
1435	D6304-A:20	38.0		0.11	
1743	ISO12937	40		0.17	
1875	ISO12937	36.7		0.07	
1943	EN60814	24.35		-0.27	
1957	D6304-A:16e1	43.6		0.27	
6002	ISO12937	33.02		-0.03	
6016	D6304-A:20	37.2		0.09	
6197		-----		-----	
6253		-----		-----	
6310	D6304-C:20	75	C,R(0.01)	1.15	first reported 275
6442		-----		-----	

normality	OK	
n	20	
outliers	1	
mean (n)	34.04	
st.dev. (n)	8.406	
R(calc.)	23.54	
st.dev.(D6304-B:20)	35.542	
R(D6304-B:20)	99.52	range 30-2100 mg/kg

Compare:

R(D6304-A:20)	28.14	range 20-25000 mg/kg
R(D6304-C:20)	18.75	range 20-360 mg/kg
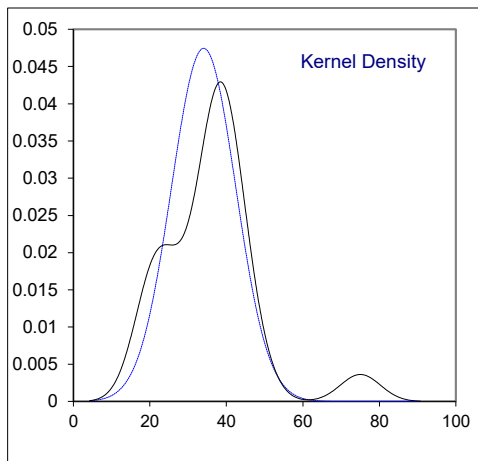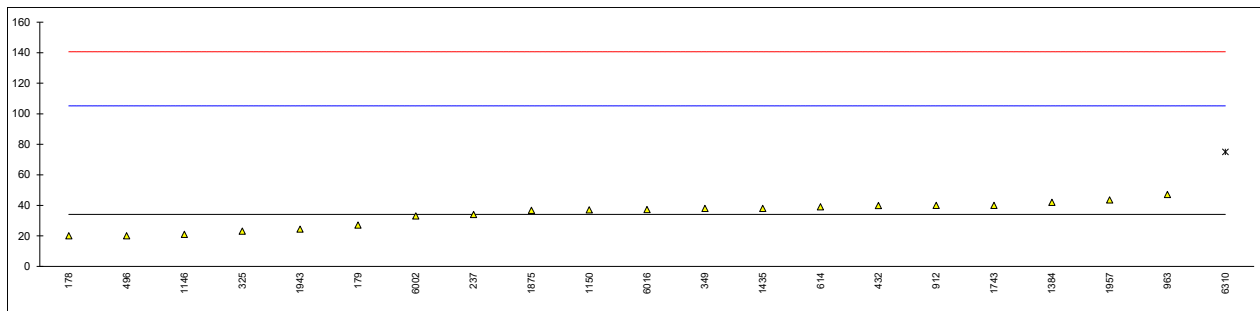
Kernel Density

Determination of Water Separability at 54 °C, distilled water on sample #22076; results in minutes

lab	method	3 mL or less 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	D1401	0	G(1)	-1.16	10		0.22	10		0.11	Yes	10
237	D1401	6.8		-0.20	6.9		-0.22	8.0		-0.17	No	-----
325	D1401	9		0.10	9		0.08	10		0.11	No	-----
349		-----		-----	-----		-----	-----		-----		-----
432		-----		-----	-----		-----	-----		-----		-----
496	D1401	10		0.24	10		0.22	0	G(5)	-1.29	No	-----
614	D1401	6.6	C	-0.23	6.6	C	-0.26	7.4	C	-0.25	No	-----
862		-----		-----	-----		-----	-----		-----		-----
912		-----		-----	-----		-----	-----		-----		-----
962		-----		-----	-----		-----	-----		-----		-----
963		-----		-----	-----		-----	-----		-----		-----
1017		7.10		-0.16	7.10		-0.19	-----		-----		-----
1146	D1401	8		-0.04	8		-0.06	-----		-----	Yes	8
1150		-----		-----	-----		-----	-----		-----		-----
1327		-----		-----	-----		-----	-----		-----		-----
1384		-----		-----	-----		-----	-----		-----		-----
1409	ISO6614	10		0.24	10		0.22	10		0.11	No	-----
1435		9		0.10	9		0.08	-----		-----		-----
1743	ISO6614	-----		-----	-----		-----	10		0.11	No	-----
1875		-----		-----	-----		-----	-----		-----		-----
1943		-----		-----	-----		-----	-----		-----		-----
1957		-----		-----	-----		-----	-----		-----		-----
6002	ISO6614	6.7		-0.22	6.5		-0.27	7.0		-0.31	No	-----
6016	D1401	8.5		0.03	8.5		0.01	10.9		0.24	No	-----
6197		7		-0.18	8		-0.06	8		-0.17	No	-----
6253	ISO6614	10		0.24	10		0.22	15	G(5)	0.81		-----
6310	D1401	10		0.24	10		0.22	10		0.11	No	-----
6442	D1401	7		-0.18	7		-0.20	10		0.11		-----
normality		OK			OK			OK				
n		14			15			11				
outliers		1			0			2				
mean (n)		8.26			8.44			9.21				
st.dev. (n)		1.392			1.378			1.330				
R(calc.)		3.90			3.86			3.72				
st.dev.(D1401:21)		7.143			7.143			7.143				
R(D1401:21)		20			20			20				

Lab 614 first reported 6 min 35 sec / 6 min 35 sec / 7 min 22 sec respectively
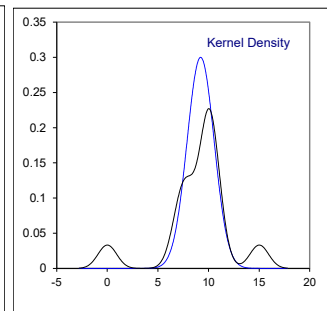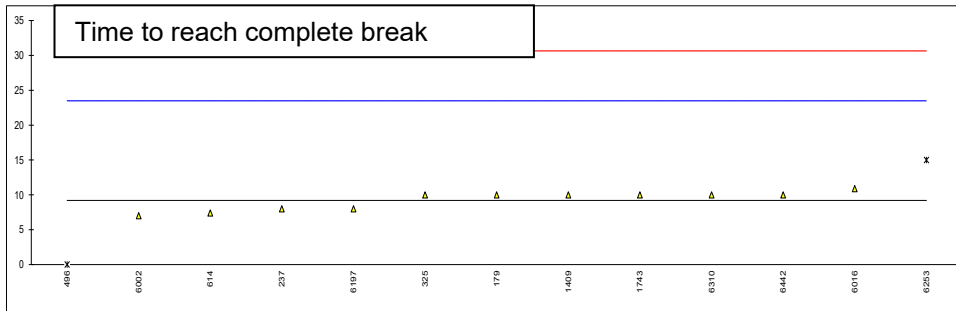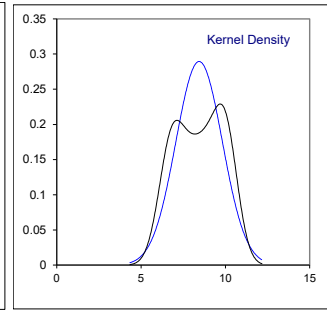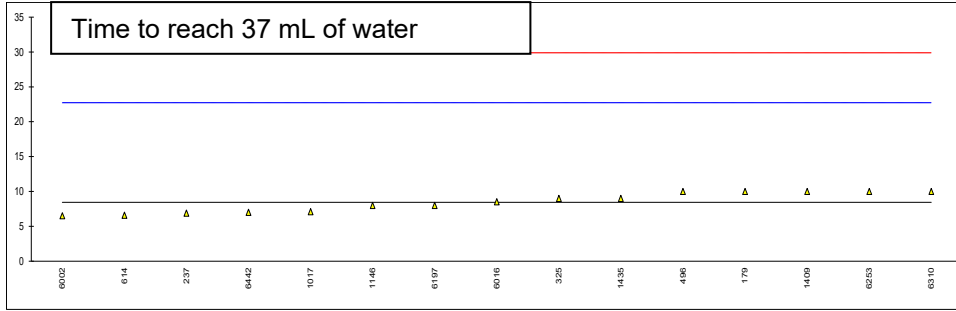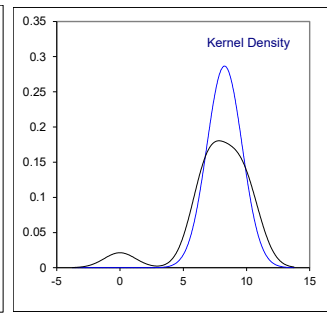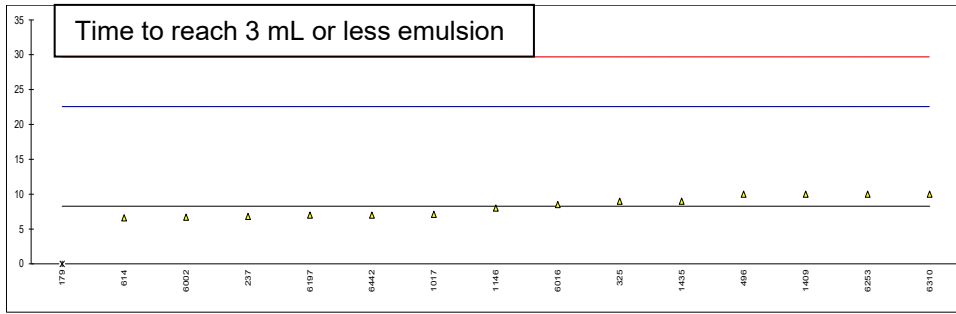
Time to reach 3 mL or less emulsion

Kernel Density

Time to reach 37 mL of water

Kernel Density

Time to reach complete break

Kernel Density

Determination of Water Separability at 54 °C, distilled water on sample #22076; results in mL

lab	method	oil phase	mark	z(targ)	water phase	mark	z(targ)	emulsion phase	mark	z(targ)
178		-----			-----			-----		
179	D1401	40			40			0		
237	D1401	40.0			40.0			0		
325	D1401	-----			-----			-----		
349		-----			-----			-----		
432		-----			-----			-----		
496	D1401	39			39			2		
614	D1401	40			40			0		
862		-----			-----			-----		
912		-----			-----			-----		
962		-----			-----			-----		
963		-----			-----			-----		
1017		-----			-----			-----		
1146	D1401	40			37			3		
1150		-----			-----			-----		
1327		-----			-----			-----		
1384		-----			-----			-----		
1409	ISO6614	-----			-----			-----		
1435		-----			-----			-----		
1743	ISO6614	40			40			0		
1875		-----			-----			-----		
1943		-----			-----			-----		
1957		-----			-----			-----		
6002	ISO6614	40			40			0		
6016	D1401	40			40			0		
6197		-----			-----			-----		
6253	ISO6614	-----			-----			-----		
6310	D1401	40			40			0		
6442	D1401	-----			-----			-----		

Determination of Calcium as Ca on sample #22076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	0		-----	
179		-----		-----	
237		-----		-----	
325	D5185	<1		-----	
349		1		-----	
432	D5185	<1		-----	
496	D5185	0.64		-----	
614	D5185	<1		-----	
862		-----		-----	
912	D5185	<1		-----	
962		-----		-----	
963	D5185	0.210		-----	
1017		-----		-----	
1146		<5		-----	
1150		-----		-----	
1327		-----		-----	
1384	D5185	<10		-----	
1409		-----		-----	
1435		<1		-----	
1743	NF T60-106	0.07		-----	
1875	EN11885	<1		-----	
1943		-----		-----	
1957	D5185	<1		-----	
6002	D5185	0.25		-----	
6016	D6595	0.1		-----	
6197	D4951	<1		-----	
6253		-----		-----	
6310	D7751	<1		-----	
6442	D5185	< 10		-----	
	n	19			
	mean (n)	<10			Application range D5185:18: 40-9000 mg/kg

Determination of Phosphorus as P on sample #22076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	47		-0.23	
179		-----		-----	
237		-----		-----	
325	D5185	52		0.23	
349		50		0.04	
432	D5185	51.42		0.17	
496	D5185	46.33		-0.30	
614	D5185	49.35	C	-0.02	first reported 59.6
862		-----		-----	
912	D5185	50	C	0.04	first reported 35
962		-----		-----	
963	D5185	51.31		0.16	
1017		-----		-----	
1146		49		-0.05	
1150		-----		-----	
1327		-----		-----	
1384	D5185	51.2		0.15	
1409		-----		-----	
1435		53		0.32	
1743	NF T60-106	50.6		0.10	
1875	EN11885	44		-0.51	
1943		-----		-----	
1957	D5185	30	G(0.01)	-1.81	
6002	D5185	46.5		-0.28	
6016	D6595	51.1		0.14	
6197	D4951	46		-0.33	
6253		-----		-----	
6310	D7751	51		0.14	
6442	D5185	51.8		0.21	

normality	OK	
n	18	
outliers	1	
mean (n)	49.53	
st.dev. (n)	2.523	
R(calc.)	7.06	
st.dev.(D5185:18)	10.808	
R(D5185:18)	30.26	Application range D5185:18: 10-1000 mg/kg
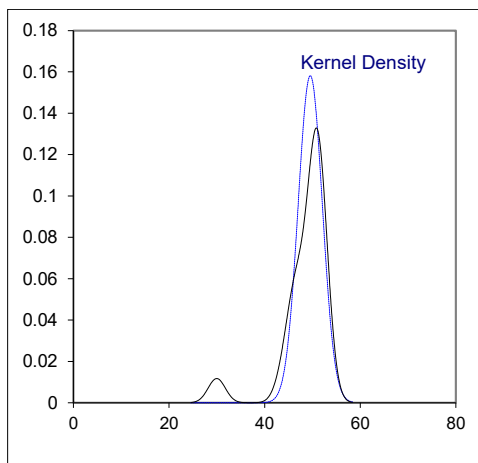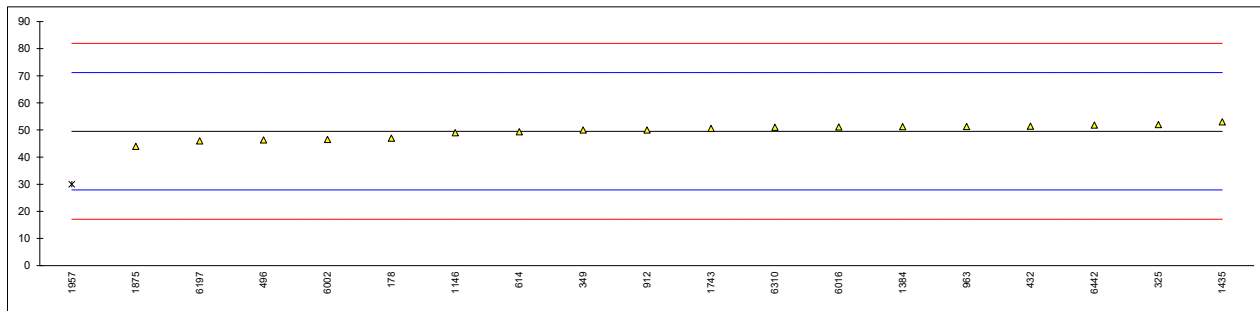
Determination of Zinc as Zn on sample #22076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	3		-----	
179		-----		-----	
237		-----		-----	
325	D5185	<1		-----	
349		1		-----	
432	D5185	<1		-----	
496	D5185	0.55		-----	
614	D5185	<1		-----	
862		-----		-----	
912	D5185	<1		-----	
962		-----		-----	
963	D5185	0.430		-----	
1017		-----		-----	
1146		<5		-----	
1150		-----		-----	
1327		-----		-----	
1384	D5185	<0,5		-----	
1409		-----		-----	
1435		<1		-----	
1743	NF T60-106	0.38		-----	
1875	EN11885	4		-----	
1943		-----		-----	
1957	D5185	<1		-----	
6002	D5185	0.01		-----	
6016	D6595	0.1		-----	
6197	D4951	<1		-----	
6253		-----		-----	
6310	D7751	<1		-----	
6442	D5185	< 10		-----	
	n	19			
	mean (n)	<10			Application range D5185:18: 60-1600 mg/kg

APPENDIX 2

Number of participants per country

1 lab in AUSTRALIA
1 lab in AUSTRIA
5 labs in BELGIUM
1 lab in BULGARIA
2 labs in CHINA, People's Republic
1 lab in FRANCE
2 labs in GERMANY
1 lab in INDIA
1 lab in KAZAKHSTAN
1 lab in LATVIA
1 lab in MALAYSIA
1 lab in MOROCCO
1 lab in NETHERLANDS
1 lab in NIGERIA
2 labs in POLAND
1 lab in PORTUGAL
2 labs in SAUDI ARABIA
1 lab in SINGAPORE
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) or G(1)	= outlier in Grubbs' outlier test
G(0.05) or G(5)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

1   iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2   ISO5725:86
3   ISO5725 parts 1-6:94
4   ISO13528:05
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7   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8   J.N. Miller, Analyst, 118, 455, (1993)
9   Analytical Methods Committee, Technical Brief, No 4, January 2001
10   P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11   W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)