Institute for
Interlaboratory Studies

Results of Proficiency Test
Hydraulic Oil (fresh)
November 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, The Netherlands

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1 INTRODUCTION

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Hydraulic Oil (fresh) every year. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of Hydraulic Oil (fresh).

In this interlaboratory study 47 laboratories in 33 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Hydraulic 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 Hydraulic Oil (fresh) in a 1-liter amber glass bottle labelled #22210.
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 100 liters of fresh Hydraulic Oil was obtained from a local supplier. After homogenization 60 amber glass bottles of 1 L were filled and labelled #22210.
The homogeneity of the subsamples was checked by determination of Density at 15 °C in accordance with ASTM ISO12185 and Kinematic Viscosity at 40 °C in accordance with ASTM D445 on 8 stratified randomly selected subsamples.

	Density at 15 °C in kg/L	Kinematic Viscosity at 40 °C in mm^2/s
sample #22210-1	0.86971	44.46
sample #22210-2	0.86971	44.46
sample #22210-3	0.86973	44.43
sample #22210-4	0.86971	44.39
sample #22210-5	0.86971	44.41
sample #22210-6	0.86971	44.46
sample #22210-7	0.86971	44.46
sample #22210-8	0.86971	44.43

Table 1: homogeneity test results of subsamples #22210

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15 °C in kg/L	Kinematic Viscosity at 40 °C in mm^2/s
r (observed)	0.00002	0.08
reference test method	ISO12185:96	D445:21e1
0.3 x R (reference test method)	0.00015	0.16

Table 2: evaluation of the repeatabilities of subsamples #22210

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one 1 L bottle of Hydraulic Oil (fresh) labelled #22210 was sent on October 12, 2022. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of fresh Hydraulic 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, Copper Corrosion 3 hrs at 50 °C, Density at 15 °C, Flash Point PMcc, Foaming Characteristics (Foaming Tendency, Foam Stability), Kinematic Viscosity at 40 °C and at 100 °C, Viscosity Index, Kinematic Viscosity Stabinger at 40 °C and at 100 °C, 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 determination of Total Acid Number (ASTM D664).

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. Four participants reported test results after the final reporting date and four other participants did not report any test results. Not all laboratories were able to report all tests requested. In total 43 participants reported 576 numerical test results. Observed were 25 outlying test results, which is 4.3%. In proficiency studies 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. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of Inflection Point at titration volume 60 mL and 125 mL and Buffer End Point at titration volume 60 mL and 125 mL of ASTM D664-A:18e2.

Copper Corrosion: This determination was not problematic. All reporting participants agreed on a test result of 1 (1A/1B).

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

Flash Point PMcc: This determination was not problematic. Three statistical outliers were observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D93-A:20.

Foaming Characteristics (Tendency and Stability): This determination was problematic. No statistical outliers were observed over Foaming Tendency parameters. Only the calculated reproducibility of Sequence I is in agreement with the requirements of ASTM D892:18. It was decided not to calculate z-scores for Foaming Tendency sequence III due to the large difference between the calculated and reference reproducibility.
All reporting participants reported 0 mL for Foam Stability.

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 full agreement with the requirements of ASTM D445:21e2.

Kinematic Viscosity at 100 °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 D445:21e2.

Viscosity Index: This determination was problematic for a number of laboratories. Three statistical outliers were observed and four other test results were excluded. The calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D2270:10R16.
One calculation difference was found between the reported test result of the participant and the value calculated by iis.

Kinematic Viscosity Stabinger at 40 °C: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D7042:21a.

Kinematic Viscosity Stabinger at 100 °C: 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 D7042:21a.

Pour Point Manual: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D97:17bR22.

Pour Point Automated 1 °C interval: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D5950:14R20.

Sulfur: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D4294:21.

Water: This determination may be problematic depending on the test method used. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6304-B:20 but not in agreement with ASTM D6304-A:20 and ASTM D6304-C:20.
A new version of ASTM D6304 was published in 2020 with major changes. In the 2016 version one precision statement was mentioned for test results based on mass with a broad application range and one based on volume. In the 2020 version all precision statements are based on mass with three different procedures (A - direct injection, B - oven accessory and C - evaporation accessory) each with a different application range. Although there is a new version of ASTM D6304 published in 2020 six participants mentioned to have used the A or C of the 2016 version.

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

Calcium as Ca: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation, but not at all with the strict requirements of ASTM D5185:18.

Phosphorus as P: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:18.

Zinc as Zn: 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 D5185:18.

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	29	0.50	0.12	0.23
Copper Corrosion 3 hrs at 50 °C		24	1 (1A/1B)	n.a.	n.a.
Density at 15 °C	kg/L	34	0.8697	0.0006	0.0005
Flash Point PMcc	°C	30	162.7	6.8	11.6
Foaming Tendency Seq. I	mL	21	7.5	15.0	18.1
Foaming Tendency Seq. II	mL	21	18.0	26.9	16.1
Foaming Tendency Seq. III	mL	21	8.9	22.9	(3.9)
Foam Stability Seq. I	mL	21	0	n.e.	n.e.
Foam Stability Seq. II	mL	21	0	n.e.	n.e.
Foam Stability Seq. III	mL	21	0	n.e.	n.e.
Kinematic Viscosity at 40 °C	mm^2/s	30	44.519	0.572	0.543
Kinematic Viscosity at 100 °C	mm^2/s	29	8.372	0.099	0.116
Viscosity Index		27	166.89	2.09	2
Kin.Viscosity Stabinger at 40 °C	mm^2/s	17	44.592	0.666	0.600
Kin.Viscosity Stabinger at 100 °C	mm^2/s	17	8.390	0.091	0.103
Pour Point Manual	°C	14	-45.4	12.7	9
Pour Point Automated 1 °C int.	°C	13	-49.4	8.1	4.5
Sulfur	mg/kg	20	5227	570	478
Water	mg/kg	29	53.2	71.4	119.0
Water Separability at 54 °C, distilled water					
Time ≤ 3 mL emulsion	minutes	17	11.4	5.8	20

Parameter	unit	n	average	2.8 * sd	R(lit)
Time 37 mL water	minutes	20	11.9	8.4	20
Complete Break (40-40-0)	minutes	10	12.9	7.0	20
Calcium as Ca	mg/kg	26	47.7	10.4	12.0
Phosphorus as P	mg/kg	30	313	61	76
Zinc as Zn	mg/kg	28	385	66	58

Table 3: reproducibilities of tests on sample #22210

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 NOVEMBER 2022 WITH PREVIOUS PTS

	November 2022	November 2021	November 2020	November 2019	November 2018
Number of reporting laboratories	43	36	41	35	35
Number of test results	576	462	533	504	465
Number of statistical outliers	25	21	23	23	18
Percentage of statistical outliers	4.3%	4.5%	4.3%	4.6%	3.9%

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	November 2022	November 2021	November 2020	November 2019	November 2018
Total Acid Number	+	+	+	+/-	-
Density at 15 °C	-	+	-	+	+
Flash Point PMcc	+	++	+/-	++	-
Foaming Tendency Seq. I	+	(--)	--	+	(--)
Foaming Tendency Seq. II	-	-	+/-	-	-
Foaming Tendency Seq. III	(--)	(--)	(--)	(--)	(--)
Kinematic Viscosity at 40 °C	+/-	+/-	++	+/-	++
Kinematic Viscosity at 100 °C	+	+	+/-	+	+/-
Viscosity Index	+/-	-	+/-	-	+/-
Kin.Viscosity Stabinger at 40 °C	-	++	++	+	++
Kin.Viscosity Stabinger at 100 °C	+	+/-	+	-	-
Pour Point Manual	-	+/-	+/-	+	-
Pour Point Automated 1 °C int.	-	-	-	-	+
Sulfur	-	-	+/-	-	+

Parameter	November 2022	November 2021	November 2020	November 2019	November 2018
Water	+	+/-	++	++	++
Water Separability	++	++	--	++	+
Calcium as Ca	+	+/-	+	+/-	-
Phosphorus as P	+	+	++	+/-	+
Zinc as Zn	-	-	-	-	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:

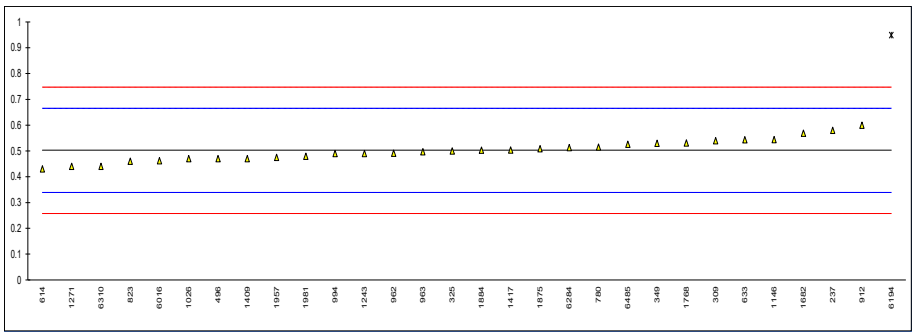
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

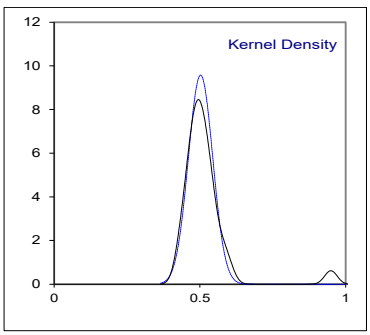
APPENDIX 1

Determination of Total Acid Number on sample #22210; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks	End point	Volume
178		-----		-----		---	---
179		-----		-----		---	---
237	D664-B	0.58		0.95		Inflection Point	125 mL
256		-----		-----		---	---
257		-----		-----		---	---
309	D664-A	0.54		0.46		Buffer End Point pH 10	125 mL
325	D664-A	0.50		-0.03		---	---
349	D664-A	0.53		0.34		Buffer End Point pH 10	125 mL
432		-----		-----		---	---
496	D664-A	0.47		-0.40		Buffer End Point pH 10	60 mL
614	D664-A	0.43		-0.89		---	60 mL
633	D664-A	0.54363		0.50		Inflection Point	125 mL
780	D664-A	0.515		0.15		Buffer End Point pH 10	60 mL
823	D664-A	0.46		-0.52		Inflection Point	125 mL
862		-----		-----		---	---
901		-----		-----		---	---
912	D664-A	0.6		1.19		---	---
962	D664-A	0.4914		-0.14		---	---
963	D664-B	0.497		-0.07		Inflection Point	60 mL
994	D664-A	0.49		-0.15		Buffer End Point pH 10	125 mL
1026	D664-B	0.47		-0.40		Buffer End Point pH 10	125 mL
1146	D664-A	0.544		0.51		Buffer End Point pH 10	125 mL
1174		-----		-----		---	---
1205		-----		-----		---	---
1243	ISO6618	0.49		-0.15		---	---
1271	ISO6618	0.44		-0.76		---	---
1381		-----		-----		---	---
1409	D664-A	0.47		-0.40		Buffer End Point pH 11	125 mL
1414		-----		-----		---	---
1417	D664-A	0.504		0.02		Inflection Point	60 mL
1444		-----		-----		---	---
1682	D664-A	0.569		0.81		Inflection Point	125 mL
1748		-----		-----		---	---
1768	ISO6618	0.53115		0.35		---	---
1799		-----		-----		---	---
1875	ISO6618	0.5086		0.07		---	---
1884	D664-A	0.5025		0.00		Buffer End Point pH 11	60 mL
1957	D664-A	0.475		-0.34		Buffer End Point pH 10	125 mL
1981	D664-A	0.48		-0.28		Inflection Point	60 mL
6016	D664-A	0.462		-0.50		Inflection Point	60 mL
6194	D664-A	0.9489	C,R(0.01)	5.46	fr. 2.9449	Inflection Point	125 mL
6257		-----		-----		---	---
6284	D664-A	0.513		0.13		Buffer End Point pH 11	60 mL
6310	D664-A	0.44		-0.76		---	---
6425		-----		-----		---	---
6442		-----		-----		---	---
6485	D974	0.526		0.29		---	---

	normality	OK
	n	29
	outliers	1
	mean (n)	0.5025
	st.dev. (n)	0.04167
	R(calc.)	0.1167
	st.dev.(D664-A:18e2, IP 60 mL)	0.08170
	R(D664-A:18e2, IP 60 mL)	0.2288
Compare		
	R(D664-A:18e2, IP 125 mL)	0.1067
	R(D664-A:18e2, BEP 60 mL)	0.2832
	R(D664-A:18e2, BEP 125 mL)	0.1533





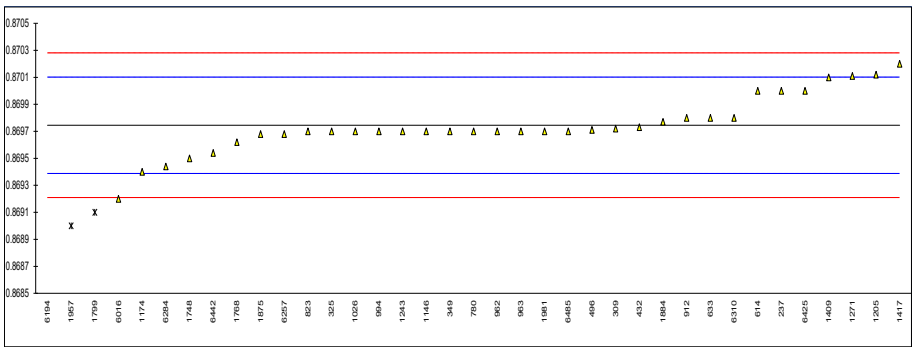
Determination of Copper Corrosion 3 hrs at 50 °C on sample #22210;

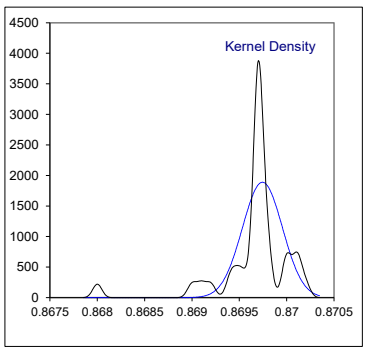
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D130	1A		-----	
256		-----		-----	
257		-----		-----	
309	D130	1A		-----	
325	D130	1A		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614	D130	1a		-----	
633	D130	1a		-----	
780	D130	1a		-----	
823	D130	1a		-----	
862		-----		-----	
901		-----		-----	
912	D130	1a		-----	
962	D130	1a		-----	
963		-----		-----	
994	D130	1a		-----	
1026		1A		-----	
1146		-----		-----	
1174		-----		-----	
1205		-----		-----	
1243	ISO2160	1a		-----	
1271	D130	1a		-----	
1381		-----		-----	
1409		-----		-----	
1414		-----		-----	
1417	IP154	1B		-----	
1444		-----		-----	
1682	D130	1a		-----	
1748	D130	1a		-----	
1768		-----		-----	
1799	D130	1A		-----	
1875		-----		-----	
1884	D130	1a		-----	
1957		-----		-----	
1981	D130	1A		-----	
6016		-----		-----	
6194	D130	1b		-----	
6257	ISO2160	1a		-----	
6284	D130	1a		-----	
6310		-----		-----	
6425		-----		-----	
6442	D130	1a		-----	
6485	D130	1a		-----	
	n	24			
	mean (n)	1 (1A/1B)			

Determination of Density at 15 °C on sample #22210; results in kg/L

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4052	0.8700		1.43	
256		-----		-----	
257		-----		-----	
309	D4052	0.86972		-0.14	
325	D4052	0.8697		-0.25	
349	D4052	0.8697		-0.25	
432	D4052	0.86973		-0.08	
496	ISO12185	0.86971		-0.20	
614	D4052	0.8700		1.43	
633	D4052	0.8698		0.31	
780	ISO12185	0.8697		-0.25	
823	ISO12185	0.8697		-0.25	
862		-----		-----	
901		-----		-----	
912	D4052	0.8698		0.31	
962	D4052	0.8697		-0.25	
963	D4052	0.8697		-0.25	
994	ISO12185	0.8697		-0.25	
1026		0.8697		-0.25	
1146	D4052	0.8697		-0.25	
1174	ISO3675	0.8694		-1.93	
1205	ISO12185	0.87012		2.10	
1243	ISO12185	0.8697		-0.25	
1271	D4052	0.87011		2.04	
1381		-----		-----	
1409	ISO12185	0.8701		1.99	
1414		-----		-----	
1417	IP365	0.8702		2.55	
1444		-----		-----	
1682		-----		-----	
1748	D4052	0.8695		-1.37	
1768	D4052	0.86962		-0.70	
1799	D7042	0.8691	DG(0.05)	-3.61	
1875	DIN51757	0.86968		-0.36	
1884	D4052	0.86977		0.14	
1957	D4052	0.869	DG(0.05)	-4.17	
1981	D4052	0.8697		-0.25	
6016	D7042	0.8692	C	-3.05	reported 869.2 kg/L
6194	D1298	0.868	C,G(0.01)	-9.77	first reported 867.2460 kg/m^3
6257	ISO12185	0.86968		-0.36	
6284	D4052	0.86944		-1.71	
6310	D4052	0.8698		0.31	
6425	D7042	0.870	C	1.43	first reported 869 kg/m^3
6442	D4052	0.86954		-1.15	
6485	D4052	0.8697		-0.25	

normality	OK
n	34
outliers	3
mean (n)	0.86974
st.dev. (n)	0.000211
R(calc.)	0.00059
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

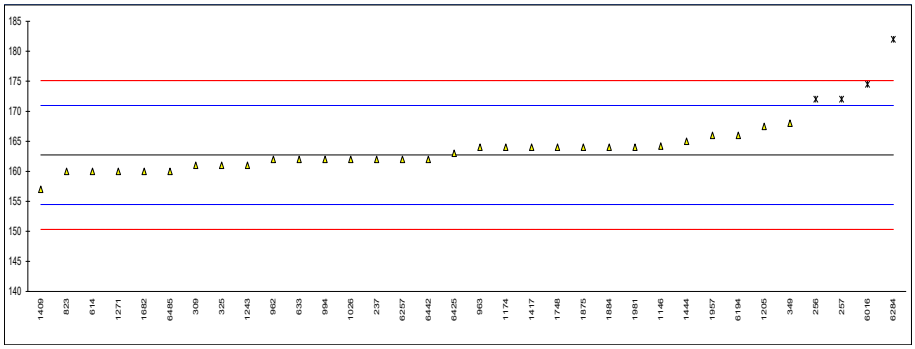


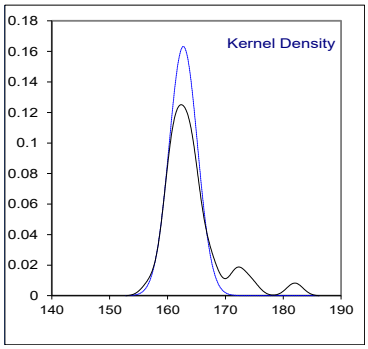


Determination of Flash Point PMcc on sample #22210; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D93-A	162.0		-0.18	
256	D3828	172	R(0.05)	2.25	
257	D3828	172.0	R(0.05)	2.25	
309	D93-A	161.0		-0.42	
325	D93-A	161.0		-0.42	
349	D93-A	168		1.28	
432		-----		-----	
496		-----		-----	
614	D93-A	160		-0.66	
633	D93-A	162.0		-0.18	
780		-----		-----	
823	ISO2719-A	160.0		-0.66	
862		-----		-----	
901		-----		-----	
912		-----		-----	
962	D93-A	162		-0.18	
963	D93-A	164.0		0.31	
994	D93-A	162.0		-0.18	
1026	D93-A	162.0	C	-0.18	first reported 198.0
1146	D93-B	164.2		0.36	
1174	ISO2719-A	164		0.31	
1205	D93-A	167.5		1.16	
1243	ISO2719-A	161		-0.42	
1271	ISO2719-A	160	C	-0.66	first reported 218
1381		-----		-----	
1409	D93-A	157.0		-1.39	
1414		-----		-----	
1417	D93-A	164		0.31	
1444	D93-A	165.0		0.55	
1682		160	C	-0.66	first reported 194
1748	D93-A	164		0.31	
1768		-----		-----	
1799		-----		-----	
1875	ISO2719-A	164		0.31	
1884	D93-A	164.0		0.31	
1957	D93-A	166		0.79	
1981	D93-A	164		0.31	
6016	D93-A	174.5	R(0.01)	2.85	
6194	D93-A	166	C	0.79	first reported 178.175
6257	ISO2719-A	162.0		-0.18	
6284	D93-A	182	ex	4.67	test result excluded, Flashpoint performed with COC
6310		-----		-----	
6425	ISO2719-A	163		0.07	
6442	D93-A	162.0		-0.18	
6485	D93-A	160		-0.66	

normality	OK
n	30
outliers	3+1ex
mean (n)	162.72
st.dev. (n)	2.445
R(calc.)	6.85
st.dev.(D93-A:20)	4.126
R(D93-A:20)	11.55

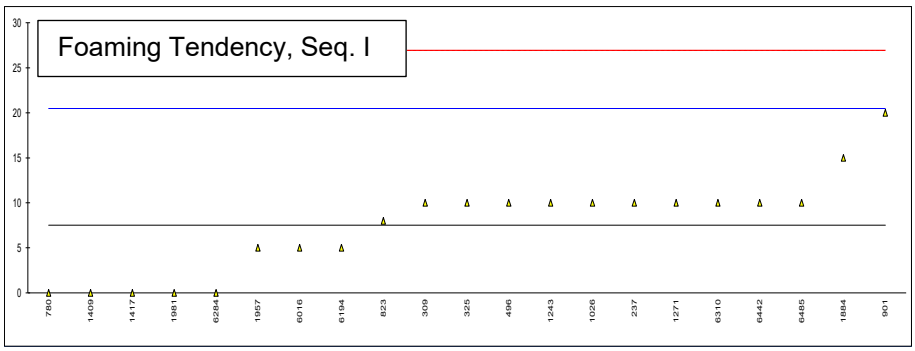


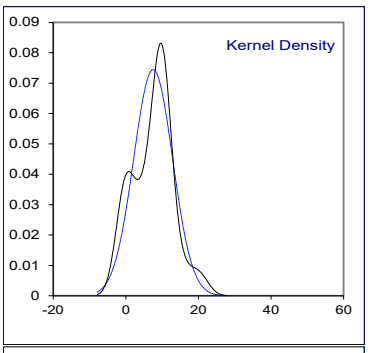


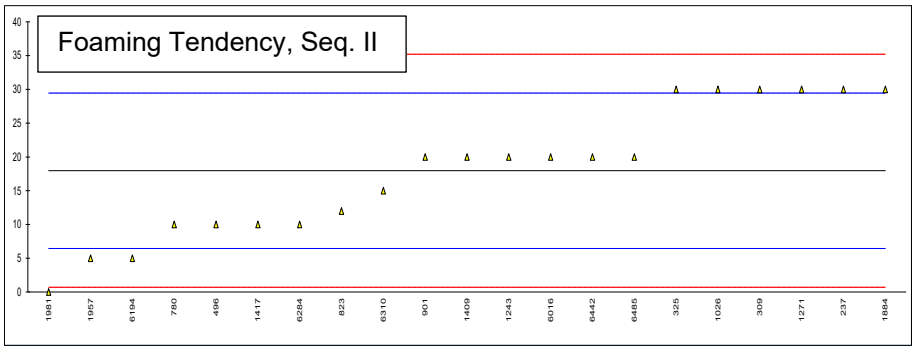
Determination of Foaming Characteristics, Foaming Tendency (5 minutes blowing period) on sample #22210; results in mL

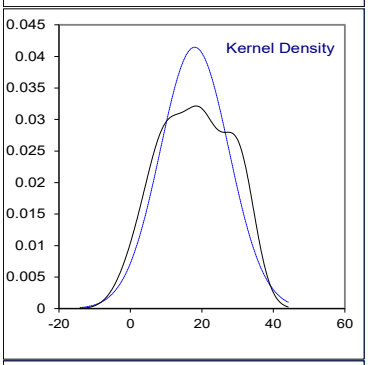
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237	D892	10		0.38	30		2.09	20		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	10		0.38	30		2.09	30		-----
325	D892	10		0.38	30		2.09	10		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	10		0.38	10		-1.38	0		-----
614		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
780	D892	0		-1.16	10		-1.38	0		-----
823	D892	8		0.07	12		-1.03	6		-----
862		-----		-----	-----		-----	-----		-----
901	D892	20		1.93	20		0.36	20		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1026	D892	10		0.38	30		2.09	10		-----
1146		-----		-----	-----		-----	-----		-----
1174		-----		-----	-----		-----	-----		-----
1205		-----		-----	-----		-----	-----		-----
1243	D892	10		0.38	20		0.36	10		-----
1271	ISO6247	10		0.38	30		2.09	10		-----
1381		-----		-----	-----		-----	-----		-----
1409	ISO6247	0		-1.16	20		0.36	0		-----
1414		-----		-----	-----		-----	-----		-----
1417	D892	0		-1.16	10		-1.38	0		-----
1444		-----		-----	-----		-----	-----		-----
1682		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1768		-----		-----	-----		-----	-----		-----
1799		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
1884	D892	15		1.15	30		2.09	20		-----
1957	D892	5		-0.39	5		-2.25	5		-----
1981	D892	0		-1.16	0		-3.12	0		-----
6016	D892	5		-0.39	20		0.36	10		-----
6194	D892	5		-0.39	5		-2.25	5		-----
6257		-----		-----	-----		-----	-----		-----
6284	D892	0		-1.16	10		-1.38	0		-----
6310	D892	10		0.38	15		-0.51	10		-----
6425		-----		-----	-----		-----	-----		-----
6442		10		0.38	20		0.36	10		-----
6485	D892	10		0.38	20		0.36	10		-----

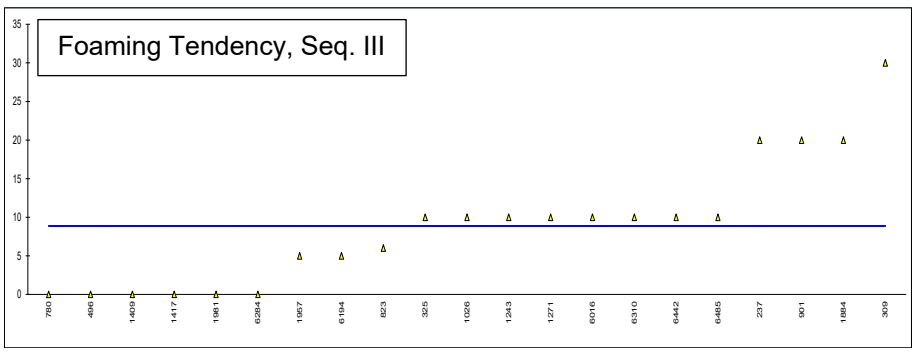
normality	OK	OK	OK
n	21	21	21
outliers	0	0	0
mean (n)	7.52	17.95	8.86
st.dev. (n)	5.363	9.620	8.181
R(calc.)	15.02	26.94	22.91
st.dev.(D892:18)	6.476	5.753	(1.392)
R(D892:18)	18.13	16.11	(3.90)

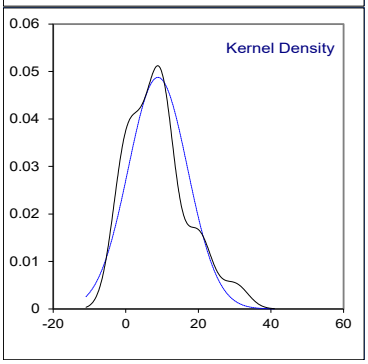

Foaming Tendency, Seq. I
780 1409 1417 1981 6284 1957 6016 6194 823 309 325 496 1243 1026 237 1271 6310 6442 6485 1884 901


Kernel Density


Foaming Tendency, Seq. II
1981 1957 6194 780 496 1417 6284 823 6310 901 1409 1243 6016 6442 6485 325 1026 309 1271 237 1884


Kernel Density


Foaming Tendency, Seq. III
780 496 1409 1417 1981 6284 1957 6194 823 325 1026 1243 1271 6016 6310 6442 6485 237 901 1884 309


Kernel Density

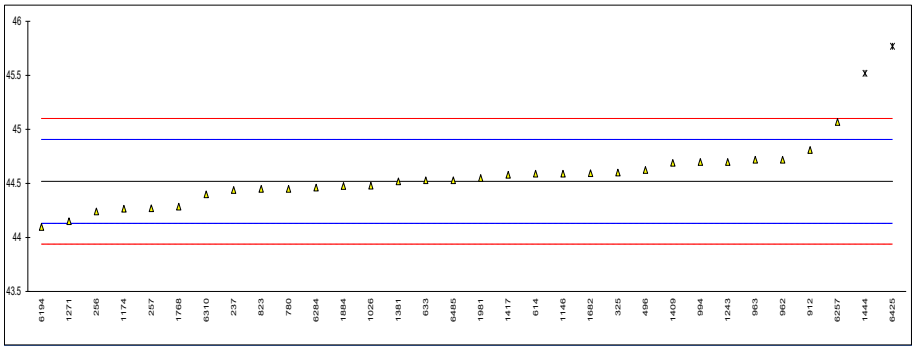
Determination of Foaming Characteristics, Foam Stability (10 minutes settling period) on sample #22210; results in mL

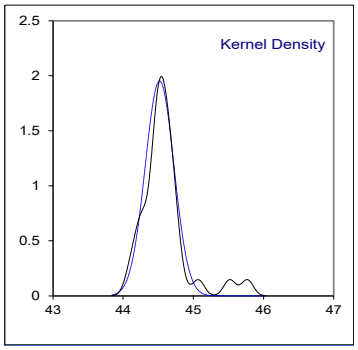
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237	D892	0		-----	0		-----	0		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	0		-----	0		-----	0		-----
325	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
614		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
780	D892	0		-----	0		-----	0		-----
823	D892	0		-----	0		-----	0		-----
862		-----		-----	-----		-----	-----		-----
901	D892	0		-----	0		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1026	D892	0		-----	0		-----	0		-----
1146		-----		-----	-----		-----	-----		-----
1174		-----		-----	-----		-----	-----		-----
1205		-----		-----	-----		-----	-----		-----
1243	D892	0		-----	0		-----	0		-----
1271	ISO6247	0		-----	0		-----	0		-----
1381		-----		-----	-----		-----	-----		-----
1409	ISO6247	0		-----	0		-----	0		-----
1414		-----		-----	-----		-----	-----		-----
1417	D892	0		-----	0		-----	0		-----
1444		-----		-----	-----		-----	-----		-----
1682		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1768		-----		-----	-----		-----	-----		-----
1799		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
1884		0		-----	0		-----	0		-----
1957	D892	0		-----	0		-----	0		-----
1981	D892	0		-----	0		-----	0		-----
6016	D892	0		-----	0		-----	0		-----
6194	D892	0		-----	0		-----	0		-----
6257		-----		-----	-----		-----	-----		-----
6284	D892	0		-----	0		-----	0		-----
6310	D892	0		-----	0		-----	0		-----
6425		-----		-----	-----		-----	-----		-----
6442	D892 (Alternative)	0		-----	0		-----	0		-----
6485	D892	0		-----	0		-----	0		-----
	n	21			21			21		
	mean (n)	0			0			0		

Determination of Kinematic Viscosity at 40 °C on sample #22210; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D445	44.44		-0.41	
256	D7279 corr to D445	44.24	C	-1.44	first reported 45.37
257	D7279 corr to D445	44.27		-1.29	
309		-----		-----	
325	D445	44.60		0.42	
349		-----		-----	
432		-----		-----	
496	D445	44.624		0.54	
614	D445	44.59		0.36	
633	D445	44.53		0.05	
780	D445	44.45		-0.36	
823	D445	44.45		-0.36	
862		-----		-----	
901		-----		-----	
912	D445	44.81		1.50	
962	D445	44.72		1.03	
963	D445	44.72		1.03	
994	D445	44.70		0.93	
1026	D445	44.48		-0.20	
1146	D445	44.59		0.36	
1174	ISO3104	44.2651		-1.31	
1205		-----		-----	
1243	D7279 corr to D445	44.70		0.93	
1271	ISO3104	44.15		-1.90	
1381	ISO3104	44.520		0.00	
1409	D445	44.69		0.88	
1414		-----		-----	
1417	D445	44.58		0.31	
1444	D445	45.520	R(0.01)	5.16	
1682	D445	44.595		0.39	
1748		-----		-----	
1768	ISO3104	44.285		-1.21	
1799		-----		-----	
1875		-----		-----	
1884	D445	44.476		-0.22	
1957		-----		-----	
1981	D445	44.55		0.16	
6016		-----		-----	
6194	D445	44.0969		-2.18	
6257	ISO3104	45.07		2.84	
6284	D445	44.4613		-0.30	
6310	D7279 corr to D445	44.4		-0.62	
6425	ISO3104	45.77	C,R(0.01)	6.45	first reported 45.60
6442		-----		-----	
6485	D445	44.53		0.05	

normality	OK
n	30
outliers	2
mean (n)	44.5194
st.dev. (n)	0.20445
R(calc.)	0.5725
st.dev.(D445:21e2)	0.19398
R(D445:21e2)	0.5431

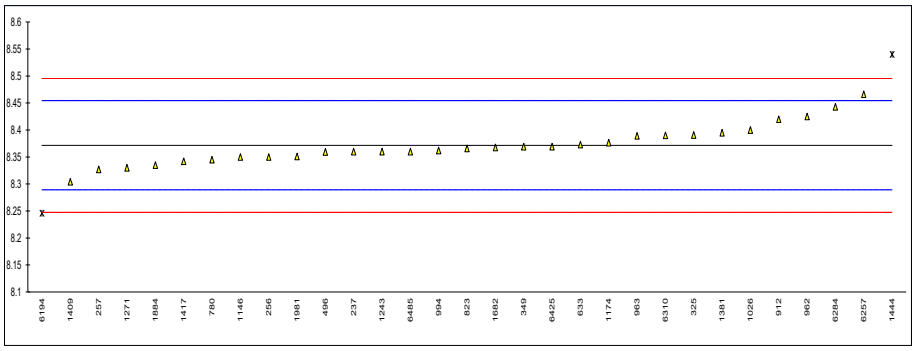


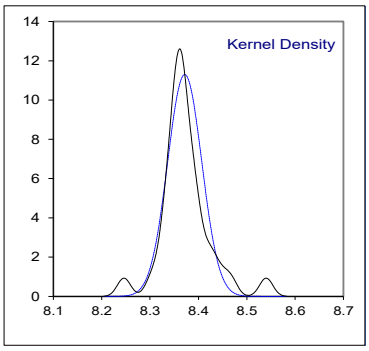


Determination of Kinematic Viscosity at 100 °C on sample #22210; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D445	8.360		-0.28	
256	D7279 corr to D445	8.35		-0.52	
257	D7279 corr to D445	8.327	C	-1.08	first reported 8.20
309		-----		-----	
325	D445	8.391		0.47	
349	D445	8.369		-0.06	
432		-----		-----	
496	D445	8.3593		-0.30	
614		-----		-----	
633	D445	8.373		0.03	
780	D445	8.345		-0.64	
823	ISO3104	8.366		-0.13	
862		-----		-----	
901		-----		-----	
912	D445	8.420		1.17	
962	D445	8.425		1.30	
963	D445	8.389		0.42	
994	D445	8.362		-0.23	
1026	D445	8.40		0.69	
1146	D445	8.350		-0.52	
1174	ISO3104	8.3766		0.12	
1205		-----		-----	
1243	D7279 corr to D445	8.36		-0.28	
1271	ISO3104	8.33	C	-1.01	first reported 8.23
1381	ISO3104	8.395		0.57	
1409	D445	8.304		-1.64	
1414		-----		-----	
1417	D445	8.342		-0.72	
1444	D445	8.540	R(0.01)	4.08	
1682	D445	8.3675		-0.10	
1748		-----		-----	
1768		-----		-----	
1799		-----		-----	
1875		-----		-----	
1884	D445	8.335		-0.89	
1957		-----		-----	
1981	D445	8.351		-0.50	
6016		-----		-----	
6194	D445	8.2459	R(0.05)	-3.05	
6257	ISO3104	8.466		2.29	
6284	D445	8.443	C	1.73	first reported 8.637
6310	D7279 corr to D445	8.39		0.45	
6425	ISO3104	8.369		-0.06	
6442		-----		-----	
6485	D445	8.36		-0.28	

normality	OK
n	29
outliers	2
mean (n)	8.3716
st.dev. (n)	0.03530
R(calc.)	0.0988
st.dev.(D445:21e2)	0.04126
R(D445:21e2)	0.1155

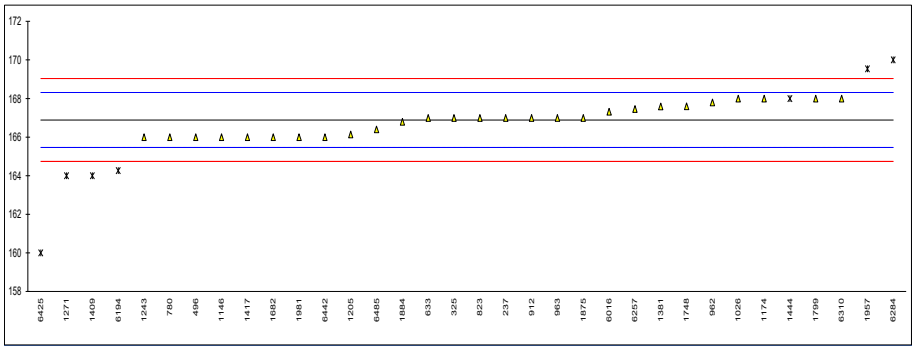


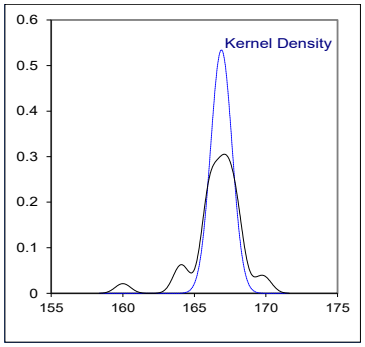


Determination of Viscosity Index on sample #22210;

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D2270	167		0.15	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D2270	167		0.15	
349		-----		-----	
432		-----		-----	
496	D2270	166.0		-1.25	
614		-----		-----	
633	D2270	167		0.15	
780	D2270	166		-1.25	
823	D2270	167		0.15	
862		-----		-----	
901		-----		-----	
912	D2270	167		0.15	
962	D2270	167.8		1.27	
963	D2270	167		0.15	
994		-----		-----	
1026	D2270	168		1.55	
1146	D2270	166		-1.25	
1174	ISO2909	168		1.55	
1205	ISO2909	166.138		-1.06	
1243	ISO2909	166		-1.25	
1271	ISO2909	164	ex	-4.05	test result excluded, outlier in KV Stabinger 100 °C
1381	ISO2909	167.595		0.98	
1409	D2270	164	R(0.05)	-4.05	
1414		-----		-----	
1417	D2270	166		-1.25	
1444	D2270	168	ex	1.55	test result excluded, outlier in KV 40 °C and KV 100 °C
1682	D2270	166		-1.25	
1748	D2270	167.6		0.99	
1768		-----		-----	
1799	D2270	168		1.55	
1875	ISO2909	167.0		0.15	
1884	D2270	166.8	E	-0.13	calculation difference, iis calculated 163.2
1957	D2270	169.54	R(0.05)	3.71	
1981	D2270	166		-1.25	
6016	D2270	167.32		0.60	
6194	D2270	164.2638	ex	-3.68	test result excluded, outlier in KV 100 °C
6257	D2270	167.46		0.79	
6284	D2270	170	C,R(0.05)	4.35	first reported 177
6310	D2270	168		1.55	
6425	D2270	160	C,ex	-9.65	first reported 162, test result excluded, outlier in KV 40 °C
6442	D2270	166		-1.25	
6485	D2270	166.4		-0.69	

normality	OK
n	27
outliers	3+4ex
mean (n)	166.89
st.dev. (n)	0.747
R(calc.)	2.09
st.dev.(D2270:10R16)	0.714
R(D2270:10R16)	2

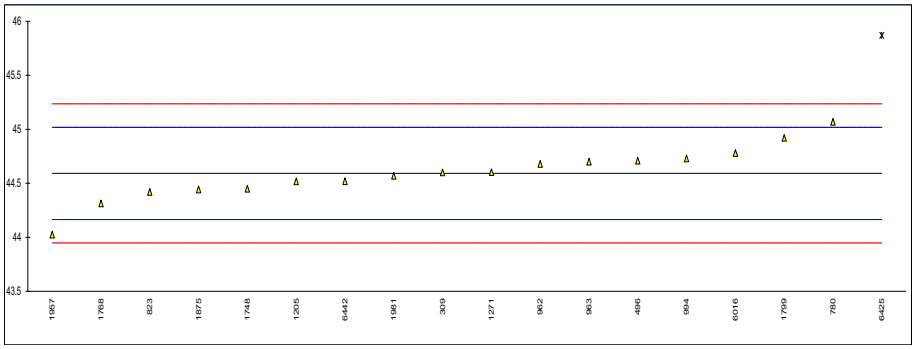


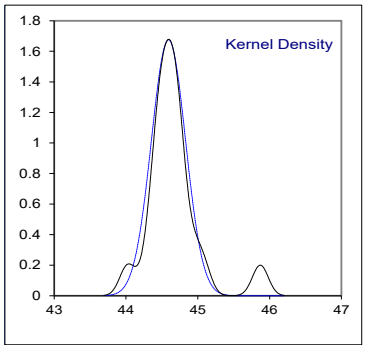


Determination of Kinematic Viscosity Stabinger at 40 °C on sample #22210; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309	D7042	44.601		0.04	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	D7042	44.710		0.55	
614		-----		-----	
633		-----		-----	
780	D7042	45.07		2.23	
823	D7042	44.42		-0.80	
862		-----		-----	
901		-----		-----	
912		-----		-----	
962	D7042	44.68		0.41	
963	D7042	44.701		0.51	
994	D7042	44.73		0.65	
1026		-----		-----	
1146		-----		-----	
1174		-----		-----	
1205	D7042	44.518		-0.34	
1243		-----		-----	
1271	D7042	44.604	C	0.06	first reported 43.876
1381		-----		-----	
1409		-----		-----	
1414		-----		-----	
1417		-----		-----	
1444		-----		-----	
1682		-----		-----	
1748	D7042	44.45		-0.66	
1768	D7042	44.314		-1.30	
1799	D7042	44.92		1.53	
1875	D7042	44.4428		-0.69	
1884		-----		-----	
1957	D7042	44.025		-2.64	
1981	D7042	44.57		-0.10	
6016	D7042	44.780		0.88	
6194		-----		-----	
6257		-----		-----	
6284		-----		-----	
6310		-----		-----	
6425	D7042	45.87	C,G(0.01)	5.97	first reported 45.60
6442	D7042	44.52		-0.33	
6485		-----		-----	

normality	suspect
n	17
outliers	1
mean (n)	44.5915
st.dev. (n)	0.23798
R(calc.)	0.6663
st.dev.(D7042:21a)	0.21426
R(D7042:21a)	0.5999

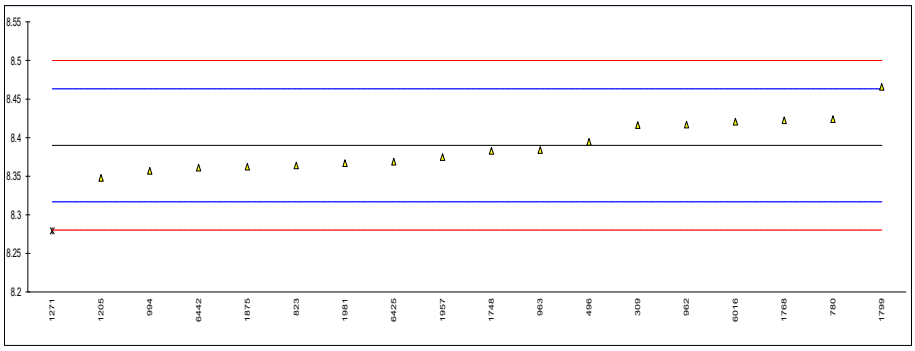


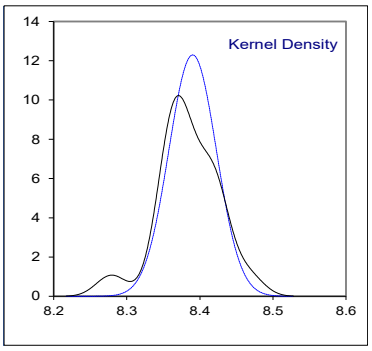


Determination of Kinematic Viscosity Stabinger at 100 °C on sample #22210; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309	D7042	8.4164		0.72	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	D7042	8.3946		0.12	
614		-----		-----	
633		-----		-----	
780	D7042	8.424		0.93	
823	D7042	8.364		-0.71	
862		-----		-----	
901		-----		-----	
912		-----		-----	
962	D7042	8.417		0.73	
963	D7042	8.384		-0.17	
994	D7042	8.357		-0.90	
1026		-----		-----	
1146		-----		-----	
1174		-----		-----	
1205	D7042	8.3478		-1.15	
1243		-----		-----	
1271	D7042	8.2792	C, D(0.05)	-3.03	first reported 8.1278
1381		-----		-----	
1409		-----		-----	
1414		-----		-----	
1417		-----		-----	
1444		-----		-----	
1682		-----		-----	
1748	D7042	8.383		-0.19	
1768	D7042	8.4227		0.89	
1799	D7042	8.466		2.07	
1875	D7042	8.36233		-0.76	
1884		-----		-----	
1957	D7042	8.375		-0.41	
1981	D7042	8.367		-0.63	
6016	D7042	8.4208		0.84	
6194		-----		-----	
6257		-----		-----	
6284		-----		-----	
6310		-----		-----	
6425	D7042	8.369		-0.58	
6442	D7042	8.361		-0.79	
6485		-----		-----	

normality	OK
n	17
outliers	1
mean (n)	8.3901
st.dev. (n)	0.032432
R(calc.)	0.0908
st.dev.(D7042:21a)	0.03663
R(D7042:21a)	0.1026

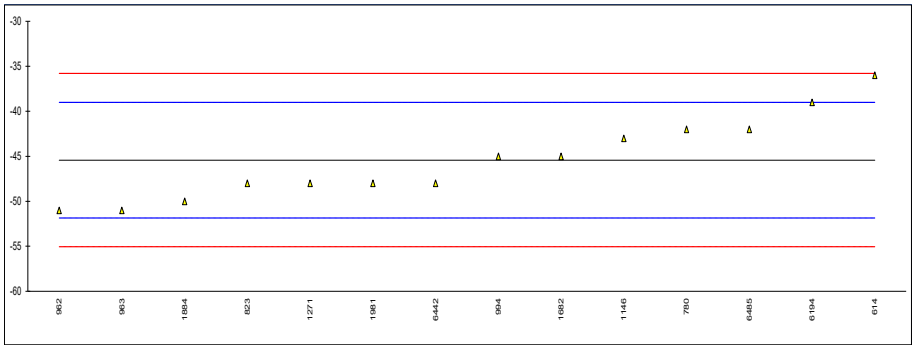


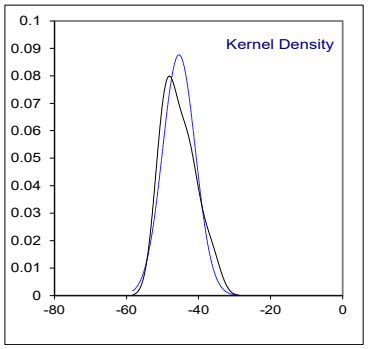


Determination of Pour Point Manual on sample #22210; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D97	<-21		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614	D97	-36		2.93	
633		-----		-----	
780	D97	-42		1.07	
823	ISO3016	-48		-0.80	
862		-----		-----	
901		-----		-----	
912		-----		-----	
962	D97	-51		-1.73	
963	D97	-51		-1.73	
994	D97	-45		0.13	
1026		-----		-----	
1146		-43		0.76	
1174		-----		-----	
1205		-----		-----	
1243		-----		-----	
1271	ISO3016	-48		-0.80	
1381		-----		-----	
1409		-----		-----	
1414		-----		-----	
1417		-----		-----	
1444		-----		-----	
1682	D97	-45		0.13	
1748		-----		-----	
1768		-----		-----	
1799		-----		-----	
1875		-----		-----	
1884	D97	-50		-1.42	
1957		-----		-----	
1981	D97	-48		-0.80	
6016		-----		-----	
6194	D97	-39		2.00	
6257		-----		-----	
6284		-----		-----	
6310		-----		-----	
6425		-----		-----	
6442	D97	-48		-0.80	
6485	D97	-42		1.07	

normality	OK
n	14
outliers	0
mean (n)	-45.43
st.dev. (n)	4.553
R(calc.)	12.75
st.dev.(D97:17bR22)	3.214
R(D97:17bR22)	9

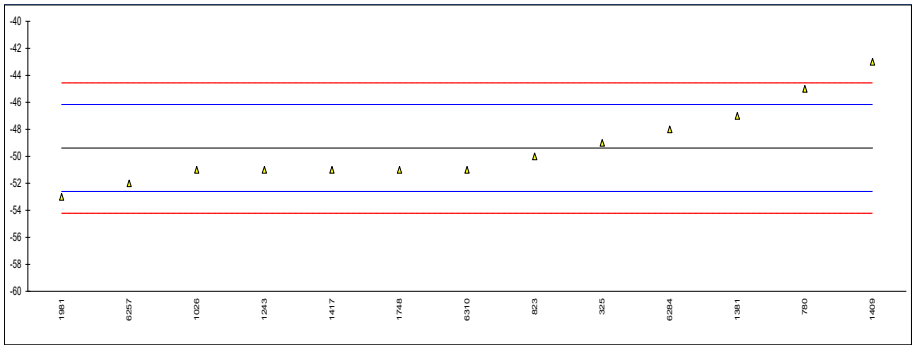


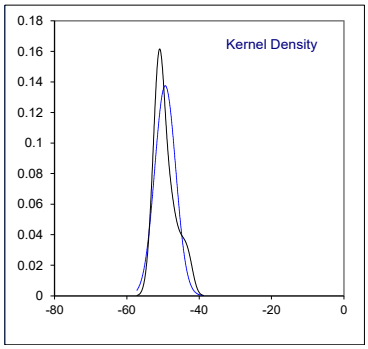


Determination of Pour Point Automated 1 °C interval on sample #22210; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D5950	-49		0.24	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614		-----		-----	
633		-----		-----	
780	D5950	-45	C	2.73	first reported -42
823	D5950	-50		-0.38	
862		-----		-----	
901		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
994		-----		-----	
1026	D5950	-51		-1.01	
1146		-----		-----	
1174		-----		-----	
1205		-----		-----	
1243	D7346	-51		-1.01	
1271		-----		-----	
1381	D6749	-47.0		1.48	
1409	D5950	-43		3.97	
1414		-----		-----	
1417	D5950	-51		-1.01	
1444		-----		-----	
1682		-----		-----	
1748	D7346	-51		-1.01	
1768		-----		-----	
1799		-----		-----	
1875		-----		-----	
1884		-----		-----	
1957		-----		-----	
1981	D5950	-53		-2.25	
6016		-----		-----	
6194		-----		-----	
6257		-52.0		-1.63	
6284	D5950	-48		0.86	
6310	D5950	-51		-1.01	
6425		-----		-----	
6442		-----		-----	
6485		-----		-----	

normality	suspect
n	13
outliers	0
mean (n)	-49.38
st.dev. (n)	2.902
R(calc.)	8.13
st.dev.(D5950:14R20)	1.607
R(D5950:14R20)	4.5

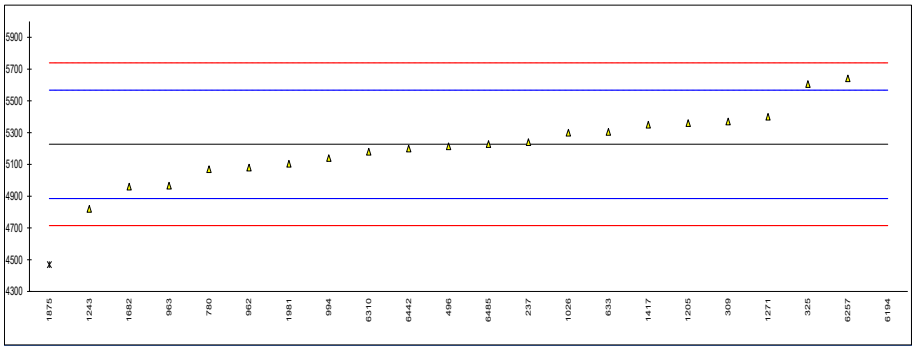


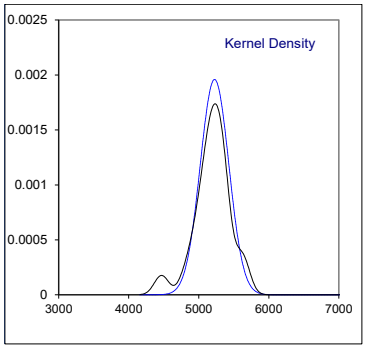


Determination of Sulfur on sample #22210; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4294	5240		0.08	
256		-----		-----	
257		-----		-----	
309	D4294	5370		0.84	
325	D5185	5606		2.22	
349		-----		-----	
432		-----		-----	
496	D2622	5213.6		-0.08	
614		-----		-----	
633	D4294	5305		0.46	
780	D4294	5070		-0.92	
823		-----		-----	
862		-----		-----	
901		-----		-----	
912		-----		-----	
962	D4294	5080	C	-0.86	first reported 508
963	D4294	4966		-1.53	
994	D4294	5140		-0.51	
1026	D2622	5300		0.43	
1146		-----		-----	
1174		-----		-----	
1205	ISO14596	5360.0	C	0.78	first reported 0.536 mg/kg
1243	ISO8754	4820		-2.38	
1271	D4294	5400		1.01	
1381		-----		-----	
1409		-----		-----	
1414		-----		-----	
1417	In house	5350		0.72	
1444		-----		-----	
1682	D4294	4960	C	-1.56	first reported 496
1748		-----		-----	
1768		-----		-----	
1799		-----		-----	
1875	DIN51724	4469	R(0.05)	-4.44	
1884		-----		-----	
1957		-----		-----	
1981	D4294	5104	C	-0.72	first reported 0.5104 mg/kg
6016		-----		-----	
6194	D5185	9430.556	C,R(0.01)	24.61	first reported 7238.3229
6257	ISO8754	5641.8		2.43	
6284		-----		-----	
6310	D7751	5180		-0.27	
6425		-----	W	-----	test result withdrawn, reported 8010
6442	D6481	5200		-0.16	
6485	D7751	5228		0.01	

normality	OK
n	20
outliers	2
mean (n)	5226.72
st.dev. (n)	203.600
R(calc.)	570.08
st.dev.(D4294:21)	170.807
R(D4294:21)	478.26

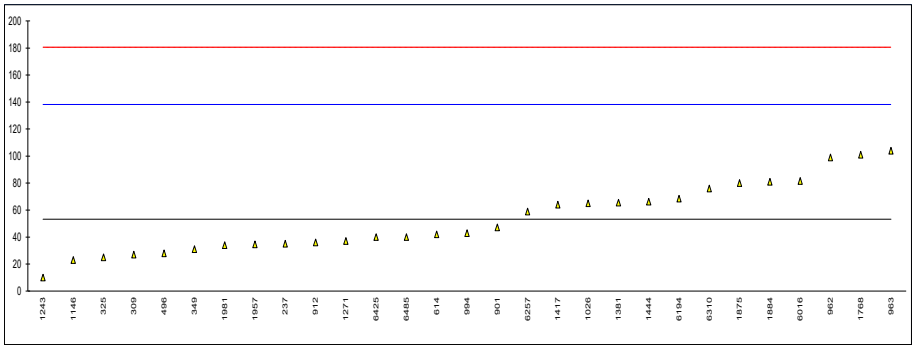


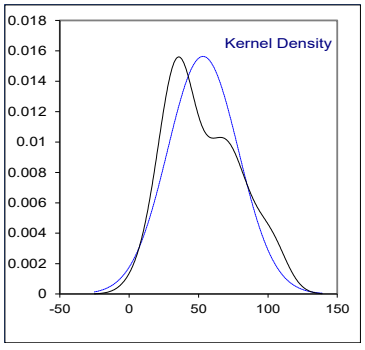


Determination of Water on sample #22210; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D6304-C:16e1	35		-0.43	
256		-----		-----	
257		-----		-----	
309	D6304-C:20	27		-0.62	
325	D6304-C:20	25		-0.66	
349	D6304-C:20	31		-0.52	
432		-----		-----	
496	D6304-B:20	28		-0.59	
614	D6304-B:20	42		-0.26	
633		-----		-----	
780	D6304-B:20	< 30		-----	
823		-----		-----	
862		-----		-----	
901	D6304-A:20	47.2		-0.14	
912	D6304-C:20	36		-0.41	
962	D6304-A:20	99		1.08	
963	D6304-A:20	104		1.20	
994	D6304-C:20	43		-0.24	
1026	D6304-C:20	65		0.28	
1146	D6304-B:20	23		-0.71	
1174		-----		-----	
1205		-----		-----	
1243	ISO12937	10		-1.02	
1271	E203	37		-0.38	
1381	EN60814	65.5		0.29	
1409		-----		-----	
1414		-----		-----	
1417	D6304-A:20	64		0.25	
1444	D6304-A:16e1	66.2		0.31	
1682		-----		-----	
1748		-----		-----	
1768	ISO3733	101		1.12	
1799		-----		-----	
1875	ISO12937	80		0.63	
1884	D6304-A:20	81		0.65	
1957	D6304-A:16e1	34.6		-0.44	
1981	D6304-C:20	34		-0.45	
6016	D6304-A:16e1	81.5		0.67	
6194	D6304-A:20	68.4886		0.36	
6257	ISO12937	58.93		0.13	
6284		-----		-----	
6310	D6304-C:16e1	76		0.54	
6425	D6304-A:20	40		-0.31	
6442		-----		-----	
6485	D6304-A:16e1	40	C	-0.31	first reported 170

	normality	OK
	n	29
	outliers	0
	mean (n)	53.221
	st.dev. (n)	25.5116
	R(calc.)	71.433
	st.dev.(D6304-B:20)	42.4849
	R(D6304-B:20)	118.958
Compare		
	R(D6304-A:20)	38.571
	R(D6304-C:20)	26.872

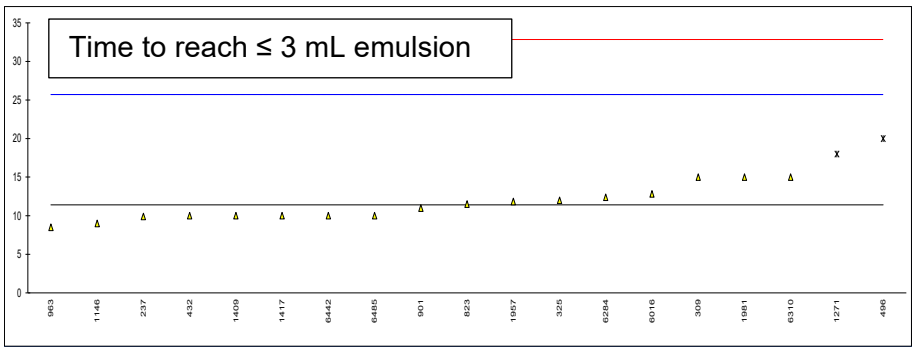


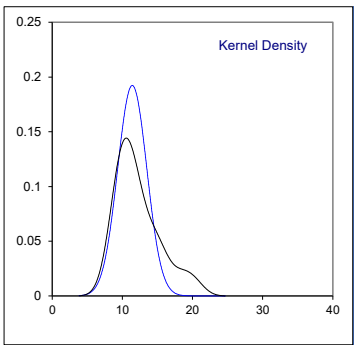


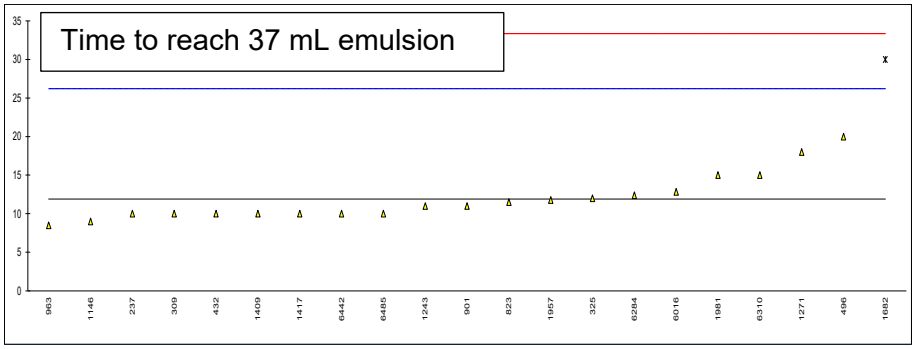
Determination of Water Separability at 54 °C, distilled water on sample #22210; results in minutes

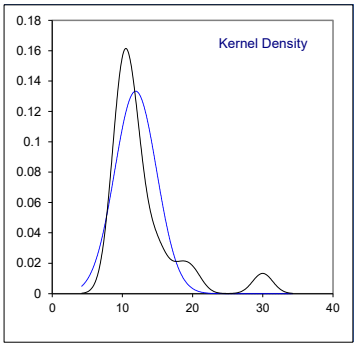
lab	method	≤3 mL emulsion	m.	z(targ)	37 mL water	m.	z(targ)	complete break (40-40-0)	m.	z(targ)	test aborted	time aborted
178		-----		-----	-----		-----	-----		-----		-----
179		-----		-----	-----		-----	-----		-----		-----
237	D1401	9.9		-0.21	10.0		-0.27	16.4		0.48	No	-----
256		-----		-----	-----		-----	-----		-----		-----
257		-----		-----	-----		-----	-----		-----		-----
309	D1401	15		0.50	10		-0.27	15		0.29	Yes	15
325	D1401	12		0.08	12		0.01	12		-0.13		-----
349		-----		-----	-----		-----	-----		-----		-----
432	D1401	10		-0.20	10		-0.27	>30		-----	Yes	30
496	D1401	20	DG5	1.20	20		1.13	-----		-----	Yes	20
614		-----		-----	-----		-----	-----		-----		-----
633		-----		-----	-----		-----	-----		-----		-----
780		-----		-----	-----		-----	-----		-----	Yes	30
823	D1401	11.5		0.01	11.5		-0.06	>30		-----	Yes	30
862		-----		-----	-----		-----	-----		-----		-----
901	D1401	11		-0.06	11		-0.13	-----		-----	Yes	30
912		-----		-----	-----		-----	-----		-----		-----
962		-----		-----	-----		-----	-----		-----		-----
963	D1401	8.5		-0.41	8.5		-0.48	9.3		-0.51	No	-----
994		-----		-----	-----		-----	-----		-----		-----
1026		-----		-----	-----		-----	-----		-----	Yes	15
1146	D1401	9		-0.34	9		-0.41	-----		-----	No	-----
1174		-----		-----	-----		-----	-----		-----		-----
1205		-----		-----	-----		-----	-----		-----		-----
1243		-----		-----	11		-0.13	-----		-----	Yes	30
1271	ISO6614	18	DG5	0.92	18		0.85	>30		-----		-----
1381		-----		-----	-----		-----	-----		-----		-----
1409	ISO6614	10		-0.20	10		-0.27	10		-0.41		-----
1414		-----		-----	-----		-----	-----		-----		-----
1417	D1401	10		-0.20	10		-0.27	10		-0.41	No	-----
1444		-----		-----	-----		-----	-----		-----		-----
1682		-----		-----	30	G1	2.53	-----		-----	No	-----
1748		-----		-----	-----		-----	-----		-----		-----
1768		-----		-----	-----		-----	-----		-----		-----
1799		-----		-----	-----		-----	-----		-----		-----
1875		-----		-----	-----		-----	-----		-----		-----
1884		-----		-----	-----		-----	-----		-----		-----
1957		11.84		0.06	11.77		-0.02	13.67		0.10	No	-----
1981	D1401	15		0.50	15		0.43	-----		-----	No	30
6016	D1401	12.82		0.20	12.82		0.13	-----		-----	Yes	30
6194		-----		-----	-----		-----	-----		-----		-----
6257		-----		-----	-----		-----	-----		-----		-----
6284	D1401	12.4		0.14	12.4		0.07	13.1		0.02	Yes	30
6310		15		0.50	15		0.43	15		0.29	No	-----
6425		-----		-----	-----		-----	-----		-----		-----
6442		10		-0.20	10		-0.27	15		0.29	No	-----
6485	D1401	10		-0.20	10		-0.27	-----		-----	Yes	15

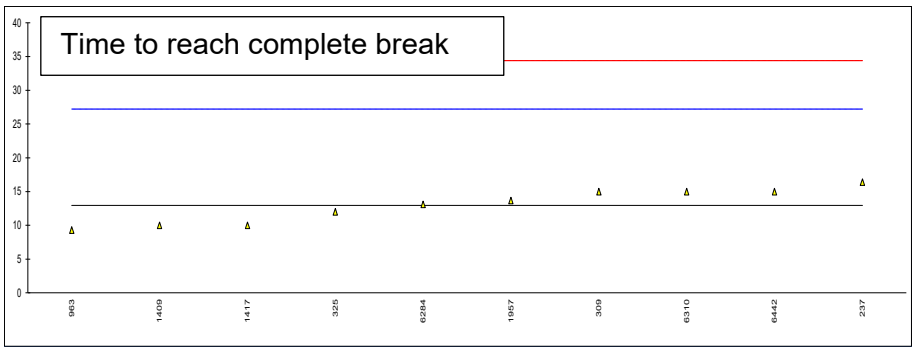
	≤3 mL emulsion	37 mL water	complete break (40-40-0)
normality	OK	not OK	OK
n	17	20	10
outliers	2	1	0
mean (n)	11.41	11.90	12.95
st.dev. (n)	2.074	2.993	2.510
R(calc.)	5.81	8.38	7.03
st.dev.(D1401:21)	7.143	7.143	7.143
R(D1401:21)	20	20	20

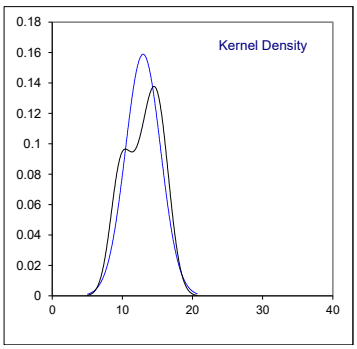

Time to reach ≤ 3 mL emulsion
963
1146
237
432
1409
1417
6442
6485
901
823
1957
325
6284
6016
309
1981
6310
1271
496


Kernel Density


Time to reach 37 mL emulsion
963
1146
237
309
432
1409
1417
6442
6485
1243
901
823
1957
325
6284
6016
1981
6310
1271
496
1682


Kernel Density


Time to reach complete break
963
1409
1417
325
6284
1957
309
6310
6442
237


Kernel Density

Determination of Water Separability at 54 °C, distilled water on sample #22210; results in mL
--- Continued ----

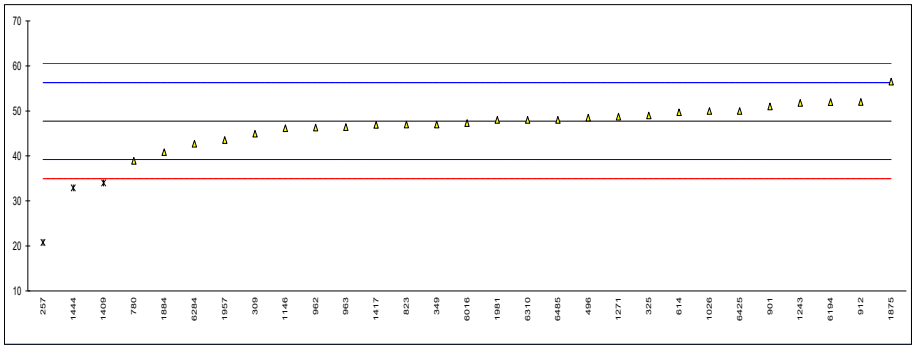
lab	method	oil	mark	z(targ)	water	mark	z(targ)	emulsion	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237	D1401	40.0		-----	40.0		-----	0		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D1401	40		-----	40		-----	0		-----
325		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432	D1401	41		-----	39		-----	0		-----
496	D1401	40		-----	37		-----	3		-----
614		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
780	D1401	41		-----	39		-----	0		-----
823	D1401	42		-----	38		-----	0		-----
862		-----		-----	-----		-----	-----		-----
901	D1401	41		-----	39		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1026	D1401	40		-----	40		-----	0		-----
1146	D1401	43		-----	37		-----	0		-----
1174		-----		-----	-----		-----	-----		-----
1205		-----		-----	-----		-----	-----		-----
1243	ISO6614	41		-----	39		-----	0		-----
1271		-----		-----	-----		-----	-----		-----
1381		-----		-----	-----		-----	-----		-----
1409		-----		-----	-----		-----	-----		-----
1414		-----		-----	-----		-----	-----		-----
1417	D1401	40		-----	40		-----	0		-----
1444		-----		-----	-----		-----	-----		-----
1682		43		-----	37		-----	0		-----
1748		-----		-----	-----		-----	-----		-----
1768		-----		-----	-----		-----	-----		-----
1799		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
1884		-----		-----	-----		-----	-----		-----
1957		40		-----	40		-----	0		-----
1981	D1401	42		-----	38		-----	0		-----
6016	D1401	41		-----	39		-----	0		-----
6194		-----		-----	-----		-----	-----		-----
6257		-----		-----	-----		-----	-----		-----
6284	D1401	40.25		-----	37.5		-----	2.2		-----
6310		40		-----	40	C	-----	0		-----
6425		-----		-----	-----		-----	-----		-----
6442		-----		-----	-----		-----	-----		-----
6485	D1401	41		-----	39		-----	1		-----

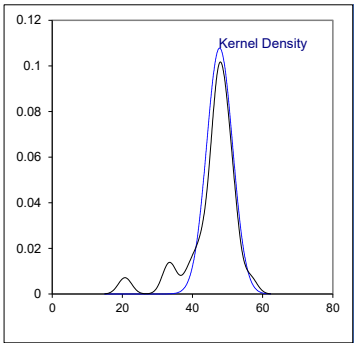
Lab 6310 first reported 0

Determination of Calcium as Ca on sample #22210; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257	D6595	20.76	C,R(0.01)	-6.32	first reported 29.26
309	D5185	44.945		-0.65	
325	D5185	49		0.30	
349	D5185	47		-0.17	
432		-----		-----	
496	D5185	48.49		0.18	
614	D5185	49.7		0.46	
633		-----		-----	
780	D5185	38.9		-2.07	
823	D5185	47		-0.17	
862		-----		-----	
901	D5185	51		0.76	
912		52		1.00	
962	D5185	46.3		-0.34	
963	D5185	46.41		-0.31	
994	D5185	<40		-----	
1026	D5185	50		0.53	
1146	D4951	46.173		-0.37	
1174		-----		-----	
1205		-----		-----	
1243	DIN51399-1	51.8		0.95	
1271	D5185	48.7		0.23	
1381		-----		-----	
1409	D5185	34	R(0.05)	-3.22	
1414		-----		-----	
1417	D5185	46.9		-0.20	
1444	D5185	32.9	R(0.05)	-3.48	
1682		-----		-----	
1748		-----		-----	
1768		-----		-----	
1799		-----		-----	
1875	DIN11885	56.5		2.05	
1884		40.86		-1.61	
1957	D5185	43.518		-0.99	
1981	D5185	48		0.06	
6016	D5185	47.31		-0.10	
6194	D5185	51.9582		0.99	
6257		-----		-----	
6284	D5185	42.7068		-1.18	
6310	D7751	48		0.06	
6425	D6481	50		0.53	
6442		-----		-----	
6485	D7751	48		0.06	

	normality	suspect
	n	26
	outliers	3
	mean (n)	47.737
	st.dev. (n)	3.6979
	R(calc.)	10.354
	st.dev.(Horwitz)	4.2686
	R(Horwitz)	11.952
Compare		
	R(D5158:18)	2.284

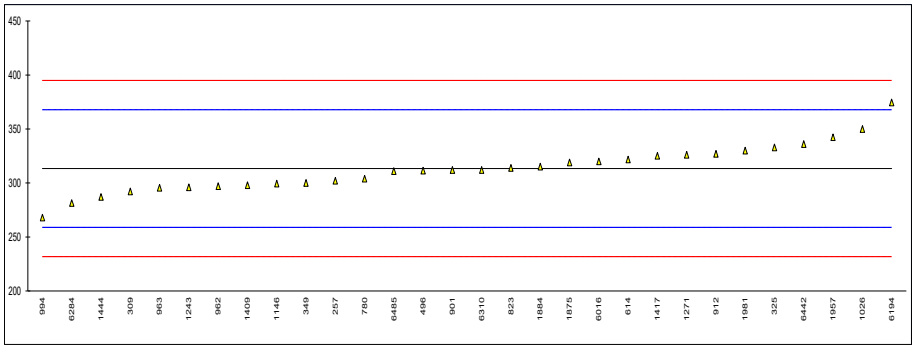


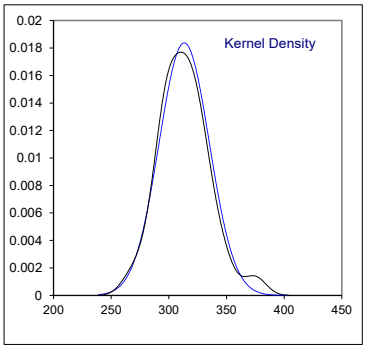


Determination of Phosphorus as P on sample #22210; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257	D6595	302		-0.42	
309	D5185	292.155		-0.78	
325	D5185	333		0.72	
349	D5185	300		-0.49	
432		-----		-----	
496	D5185	311.4		-0.07	
614	D5185	321.8		0.31	
633		-----		-----	
780	D5185	304		-0.34	
823	D5185	314		0.02	
862		-----		-----	
901	D5185	312		-0.05	
912		327		0.50	
962	D5185	297		-0.60	
963	D5185	295.6		-0.65	
994	D5185	268		-1.67	
1026	D5185	350		1.35	
1146	D4951	299.37		-0.51	
1174		-----		-----	
1205		-----		-----	
1243	DIN51399-1	296		-0.64	
1271	D5185	326.2		0.47	
1381		-----		-----	
1409	D5185	298		-0.57	
1414		-----		-----	
1417	D5185	325.1		0.43	
1444	D5185	287		-0.97	
1682		-----		-----	
1748		-----		-----	
1768		-----		-----	
1799		-----		-----	
1875	DIN11885	318.9		0.20	
1884		315.2		0.07	
1957	D5185	342.328		1.07	
1981	D5185	330		0.61	
6016	D5185	320.0		0.24	
6194	D5185	374.5010		2.25	
6257		-----		-----	
6284	D5185	281.49		-1.17	
6310	D7751	312		-0.05	
6425		-----	W	-----	test result withdrawn, reported 220
6442	D5185	336		0.83	
6485	D7751	311		-0.09	

normality	suspect
n	30
outliers	0
mean (n)	313.368
st.dev. (n)	21.7158
R(calc.)	60.804
st.dev.(D5185:18)	27.1855
R(D5185:18)	76.119

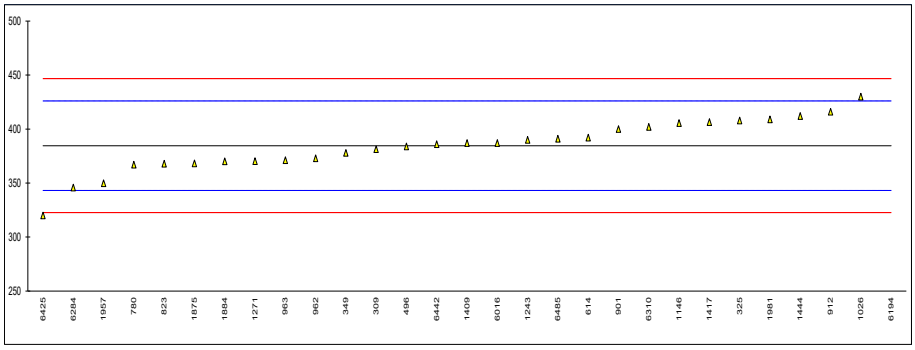


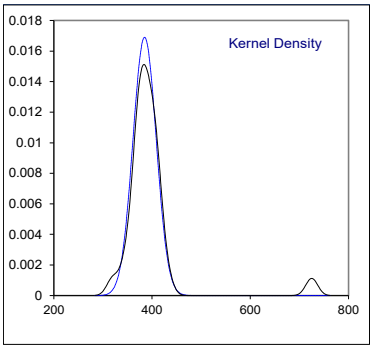


Determination of Zinc as Zn on sample #22210; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309	D5185	381.317		-0.16	
325	D5185	408		1.13	
349	D5185	378		-0.32	
432		-----		-----	
496	D5185	383.9		-0.03	
614	D5185	392.1		0.36	
633		-----		-----	
780	D5185	367		-0.85	
823	D5185	368		-0.80	
862		-----		-----	
901	D5185	400		0.74	
912		416		1.52	
962	D5185	373		-0.56	
963	D5185	371.2		-0.65	
994		-----		-----	
1026	D5185	430		2.20	
1146	D4951	405.63		1.02	
1174		-----		-----	
1205		-----		-----	
1243	DIN51399-1	390		0.26	
1271	D5185	370.3		-0.69	
1381		-----		-----	
1409	D5185	387		0.12	
1414		-----		-----	
1417	D5185	406.4		1.05	
1444	D5185	412		1.32	
1682		-----		-----	
1748		-----		-----	
1768		-----		-----	
1799		-----		-----	
1875	DIN11885	368.2		-0.79	
1884		370.2		-0.70	
1957	D5185	349.917		-1.68	
1981	D5185	409		1.18	
6016	D5185	387.0		0.12	
6194	D5185	724.882	C,R(0.01)	16.46	first reported 801.1405
6257		-----		-----	
6284	D5185	345.89		-1.87	
6310	D7751	402		0.84	
6425	D6481	320		-3.13	
6442	D5185	386		0.07	
6485	D7751	391		0.31	

normality	OK
n	28
outliers	1
mean (n)	384.609
st.dev. (n)	23.6077
R(calc.)	66.101
st.dev.(D5185:18)	20.6748
R(D5185:18)	57.889





APPENDIX 2

Number of participants per country

1 lab in ALGERIA
1 lab in AUSTRALIA
1 lab in AUSTRIA
1 lab in AZERBAIJAN
4 labs in BELGIUM
1 lab in BOSNIA and HERZEGOVINA
1 lab in BULGARIA
1 lab in CHINA, People's Republic
1 lab in ESTONIA
1 lab in GEORGIA
3 labs in GERMANY
1 lab in GREECE
1 lab in INDIA
1 lab in JORDAN
1 lab in KAZAKHSTAN
1 lab in KOREA, Republic of
2 labs in MALAYSIA
3 labs in NETHERLANDS
1 lab in NIGERIA
1 lab in OMAN
1 lab in PHILIPPINES
2 labs in POLAND
1 lab in PORTUGAL
1 lab in ROMANIA
1 lab in RUSSIAN FEDERATION
3 labs in SAUDI ARABIA
1 lab in SERBIA
1 lab in SPAIN
2 labs in TANZANIA
2 labs in TURKEY
1 lab in UNITED ARAB EMIRATES
1 lab in UNITED KINGDOM
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)/G1	= 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)/DG5	= 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
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)