Results of Proficiency Test
Transformer Oil (used)
November 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester
Corrector: dr. R.G. Visser & ing. R.J. Starink
Report: iis15L09 + iis15L09F

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1 INTRODUCTION

Since 2014, the Institute for Interlaboratory Studies organized a proficiency test for used Transformer Oil in combination with the PT on the analysis of Furanics in Transformer Oil. The PT on Furanics has been organized by the Institute since 2001 as a part of the PT on fresh Transformer Oil. During the annual proficiency testing program 2015/2016, it was decided to continue the round robin for used Transformer Oil and Furanics.
In this interlaboratory study 66 laboratories from 31 different countries for the PT on used Transformer Oil have participated and 43 laboratories from 21 different countries for the PT on Furanics. See appendix 2 for the number of participants per country. In this report, the results of the 2015 interlaboratory study on used Transformer Oil and Furanics are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. In this proficiency test the participants received, depending on the registration, 1*1 litre bottle of used Transformer Oil (labelled #15223) and/or 1*100 ml bottle (labelled #15224) for Furanics in Transformer Oil. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for the statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (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 organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis internet site 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

The necessary bulk material for the used oil sample #15223 was obtained from an European supplier. The approximately 90 litres bulk material was homogenised in a pre-cleaned drum. After homogenisation, 82 subsamples were transferred to 1 litre amber glass bottles and labelled #15223. The homogeneity of the subsamples #15223 was checked by determination Density in accordance with ASTM D4052 and Water in accordance with ASTM D6304 on 8 stratified randomly selected samples.

	Density at 20°C in kg/m^3	Water in mg/kg
Sample #15223-1	843.89	23
Sample #15223-2	843.89	27
Sample #15223-3	843.88	19
Sample #15223-4	843.87	21
Sample #15223-5	843.88	25
Sample #15223-6	843.88	25
Sample #15223-7	843.88	23
Sample #15223-8	843.87	23

Table 1: homogeneity test results of subsamples #15223

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference methods in agreement with the procedure of ISO 13528, Annex B2 in the next table.

	Density at 20°C in kg/m^3	Water in mg/kg
r (sample #15223)	0.02	7.0
reference method	ISO3675:98	D6304:07
$0.3xR_{(reference)}$	0.36	33.5

Table 2: repeatabilities of subsamples #15223

The necessary bulk material positive on Furanics was obtained from the same European supplier and mixed with a small amount of Transformer Oil, highly positive on Furanics, from a third party. After homogenisation, the bulk material of approx. 6 L was transferred to 50 amber glass bottles of 100 mL and labelled #15224. The homogeneity of the subsamples #15224 was checked by determination Density in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density at 20°C in kg/m^3
Sample #15224-1	843.90
Sample #15224-2	843.90
Sample #15224-3	843.90
Sample #15224-4	843.90
Sample #15224-5	843.90
Sample #15224-6	843.90
Sample #15224-7	843.90
Sample #15224-8	843.90

Table 3: homogeneity test results of subsamples #15224

From the above test results the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table.

	Density at 20°C in kg/m^3
r (sample #15224)	0.00
reference method	ISO3675:98
0.3x$R_{(reference)}$	0.36

Table 4: repeatability of subsamples #15224

Each of the calculated repeatabilities was equal or less than 0.3 times the corresponding reproducibility of the reference methods. Therefore, homogeneity of the subsamples of #15223 and #15224 was assumed.

To each of the participating laboratories, depending on the registration, 1*1 litre bottle (labelled #15223) and/or 1*100mL bottle (labelled #15224) was sent on November 4, 2015.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were asked to determine on sample #15223: Acid Number, Breakdown Voltage, Density at 20^{o}C, Di-electric loss at 90^{o}C (Di-electric Dissipation Factor and Specific Resistance), Flash Point Pensky-Martens Closed Cup, Interfacial Surface Tension, Kinematic Viscosity at 40^{o}C and Water.
On sample #15224, the participants were requested to determine the following Furanic Compounds: 2-acetylfuran, 2-furfural, 2-furfurylalcohol, 5-hydroxymethyl-2-furfural and 5-methyl-2-furfural.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/.
A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.
Directly after deadline, a reminder was sent to those laboratories that had not yet reported.
Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the (raw data of the) reported results.
Additional or corrected results have been used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

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

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

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and Rosner outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 3, no.16). Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for each determination (see appendix 1). On the Y-axis the reported analysis 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 standard. 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 (see appendix 3; nos.14 and 15). Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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

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

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

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

$\lvert z\rvert < 1$	good
$1 < \lvert z\rvert < 2$	satisfactory
$2 < \lvert z\rvert < 3$	questionable
$3 < \lvert z\rvert$	unsatisfactory

4 EVALUATION

In this proficiency test some problems were encountered during the execution.
For the used Transformer Oil iis15L09, seven participants reported test results after the final reporting date and two participants did not report any test results at all.
For the Furanics in Transformer Oil: four participants reported the test results after the final reporting date and two participants did not report any test results at all.

Sixty-four participants reported a numerical test result in the PT on used Transformer Oil and thirty-nine participants in the PT on Furanics. Some participants reported results for both PTs, while others reported results for only one PT. In both PTs sixty-eight different laboratories participated.
The total of 561 numerical results were reported by 68 participants for both the PT on used Transformer Oil and the PT on Furanics. Observed were 25 outlying results, which is 4.5% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per sample and per test. The specified test methods and requirements were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the reported data. The abbreviations, used in these tables, are listed in appendix 3.

For the Furanics the observed spreads were compared against the (strict) reproducibility estimated from the Horwitz equation. It is remarkable that the precision requirements of IEC 61198:94 are more strict than the requirements estimated from the Horwitz equation.

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

Acid Number: Thirty-four participants performed a potentiometric titration and eighteen participants a colorimetric titration. The group was divided in two groups: one performing a potentiometric titration and one performing a colorimetric titration. These two groups were evaluated separately. Four participants reported to have used an “in-house” or “EN62021” as test method without specifying the type of titration that was performed. Therefore the test results of these 4 participants were excluded in both evaluations.

Potentiometric titration: this determination was problematic. Two statistical outliers were observed and four other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN62021-1:03 or D664-A:11a.

Colorimetric titration: this determination may be problematic, depending on the method used. No statistical outliers were observed, but four test results were excluded. The calculated reproducibility after rejection of the suspect data is in good agreement with the requirements of D974:14e1, but not at all in agreement with the very strict requirements of EN62021-2:07.

Breakdown Voltage: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN60156:95.
The reproducibility of EN60156:95 was determined from Figure 3. The black line in Figure 3 shows the relative standard deviation as a function of the value of the mean based on six breakdown measurements. To calculate the repeatability, RSDr has to be multiplied with 2.8. The reproducibility can be estimated from the repeatability by multiplication with the empirical factor 3.

Density at 20°C: This determination was problematic for a number of laboratories. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO3675:98.

DD-Factor: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of EN60247:04.

Spec. Resistance: This determination was not problematic. No statistical outliers were observed. However, one test result was excluded as it was determined at a test temperature of 20°C instead of 90°C. The calculated reproducibility after rejection of the suspect data is in good agreement with the requirements of EN60247:04.

Flash Point PMcc: This determination was not problematic. Two test results were excluded as the test results were reported according to ASTM D92 which is not equivalent to ISO2719/ASTM D93/IP34 method B. One statistical outlier was observed. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ISO2719:02 method B.

Interf. Surf. Tension: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D971:12. One should be aware that ISO6295 is obsolete since February 2005.

Kinematic Viscosity: This determination was problematic. One statistical outlier was observed. The reproducibility for used oils is not present in ASTM D445:15a (see §17.3). Therefore the target reproducibility is calculated from the reproducibilities found in iis PTs on used oils (see appendix 3, ref. 17). The calculated reproducibility after rejection of the statistical outlier is not in agreement with the average reproducibility found for used oils in previous iis PTs.

Water: This determination may be problematic for a number of laboratories. Six statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN60814:98.

2-Furfural: This determination may be problematic. Three statistical outliers were observed. One test result (laboratory 1505) was excluded, because the test results for 2-furfurylalcohol and 5-methyl-2-furfural were outliers and the test result for 2-acetylfuran was a false positive result. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated requirements from the Horwitz equation.

2-Furfurylalcohol: This determination may be problematic. One statistical outlier was observed and two possible false negative test results were reported. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated requirements from the Horwitz equation.

5-Methyl-2-furfural: This determination may problematic for a number of laboratories. Four statistical outliers and two possible false negative test results were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated requirements calculated using the Horwitz equation.

Other Furanics : The concentrations of 2-Acetylfuran and 5-Hydroxymethyl-2-furfural may be near or below the detection limit. Therefore no significant conclusions were drawn. One laboratory (labcode 1264) may have identified the peak for 5-methyl-2-furfural incorrectly as 2-Acetylfuran.

Total Furanics: Using the web based result entry portal, it was possible this year to report the Total Furanics. Ten laboratories did so. Therefore Total Furanics was also statistically evaluated.
This determination may not be problematic. No statistical outliers were observed and one test result was excluded, because of a calculation error. However, the calculated reproducibility after rejection of the suspect data is in agreement with the estimated requirements calculated using the Horwitz equation.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM, ISO, EN and IEC standards) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acid Number (Potentiometric Titration)	g KOH/kg	32	0.030	0.015	0.011
Acid Number (Colorimetric Titration)	g KOH/kg	18	0.027	0.023	0.080
Breakdown Voltage	kV/2.5 mm	58	31.0	18.1	46.9
Density at 20°C	kg/m^3	41	843.9	1.0	1.2
Di-electric Dissipation Factor at 90°C		46	0.0327	0.0135	0.0339
Specific Resistance at 90°C	GΩm	36	13.66	4.53	14.34
Flash Point PMcc	°C	33	153	13	16
Interfacial Surface Tension	mN/m	40	24.6	6.6	2.5
Kinematic Viscosity at 40°C	mm^2/s	40	8.275	0.184	0.149
Water	mg/kg	56	28	8	8

table 5: Performance of the group on sample #15223

Parameter	unit	n	average	2.8 * sd	R(lit)
* 2-furfural	mg/kg	34	0.12	0.09	0.08
* 2-furfurylalcohol	mg/kg	29	0.10	0.09	0.06
* 5-methyl-2-furfural	mg/kg	31	0.18	0.08	0.11
* 2-acetylfuran	mg/kg	35	<0.05	n.a.	n.a.
* 5-hydroxymethyl-2-furfural	mg/kg	35	<0.05	n.a.	n.a.
Total Furanics	mg/kg	9	0.38	0.20	0.34

table 6: Performance of the group on sample #15224

Without further statistical calculations, it can be concluded that for several tests there is a good compliance of the group of participating laboratories with the relevant standards or the rather strict calculated estimates using the Horwitz equation. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE NOVEMBER 2015 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2015	November 2014
Number of reporting labs	68	63
Number of results reported	561	508
Statistical outliers	25	23
Percentage outliers	4.5%	4.5%

Table 7: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the target requirements. The conclusions are given the following table:

Parameter	November 2015	November 2014	November 2013	October 2012	November 2011
Acid number Potentiometric	-	--			
Acid number Colorimetric	++	n.e.			
Breakdown Voltage	++	+			
Density at 20°C	+	-			
Di-electric Dissipation Factor	++	++			
Specific Resistance	++	++			
Flash Point	+	+/-			
Interfacial Surface Tension	--	--			
Kinematic Viscosity at 40°C	-	--			
Water	+/-	-	-	-	+
2-furfural	-	+	+/-	--	+/-
2-furfurylalcohol	-	+	+	--	n.e.
5-methyl-2-furfural	+	++	+/-	++	n.e.
2-acetylfuran	n.e.	n.e.	n.e.	n.e.	n.e.
5-hydroxymethyl-2-furfural	n.e.	n.e.	n.e.	n.e.	n.e.
Total Furanics	+	n.e.	n.e.	n.e.	n.e.

table 8: comparison determinations against the standard requirements

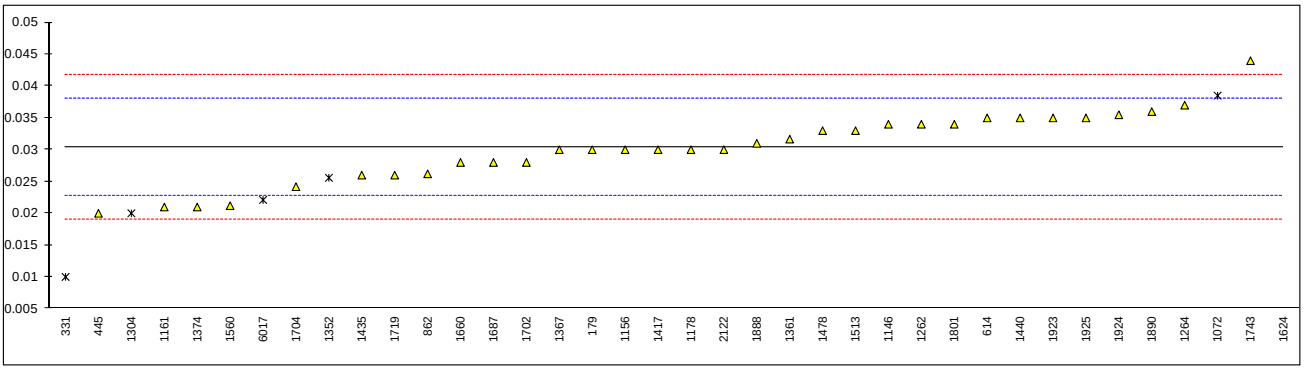
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

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

APPENDIX 1

Determination of Acid Number (Potentiometric Titration) on sample #15223; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks
179	D664	0.03		-0.10	
225		-----		-----	
237		-----		-----	
331	D664	0.01	R(0.05)	-5.37	
398		-----		-----	
445	IEC62021-1	0.02		-2.74	
511		-----		-----	
541	D664	<0.1		-----	
614	EN62021-1	0.035		1.21	
862	D664	0.0262		-1.10	
912		-----		-----	
963		-----		-----	
974		-----		-----	
1056		-----		-----	
1072	in house	0.0385	ex	2.13	excluded for it is uncertain if reported method is a potentiometric titration
1135		-----		-----	
1146	D664	0.034		0.95	
1156	IEC62021-1	0.030		-0.10	
1161	D664	0.021		-2.47	
1178	IEC62021-1	0.030		-0.10	
1262	EN62021-1	0.034		0.95	
1264	D664	0.037		1.74	
1303		-----		-----	
1304	INH-122	0.02	ex	-2.74	excluded for it is uncertain if reported method is a potentiometric titration
1306		-----		-----	
1352	IEC62021	0.02558	ex	-1.27	excluded for it is uncertain if reported method is a potentiometric titration
1361	EN62021-1	0.03167		0.33	
1367	EN62021-1	0.03		-0.10	
1374	IEC62021-1	0.021		-2.47	
1417	D664	0.03		-0.10	
1430		-----		-----	
1435	IEC62021-1	0.026		-1.16	
1440	EN62021-1	0.035		1.21	
1442		-----		-----	
1444		-----		-----	
1458		-----		-----	
1461		-----		-----	
1478	IEC62021-1	0.033		0.68	
1505		-----		-----	
1513	IEC62021-1	0.033		0.68	
1516		-----		-----	
1545		-----		-----	
1560	IEC62021-1	0.0212		-2.42	
1624	EN62021-1	0.088	R(0.01)	15.16	
1660	EN62021-1	0.028		-0.63	
1687	D664	0.028		-0.63	
1702	IEC62021-1	0.028		-0.63	
1704	IEC62021-1	0.02418		-1.64	
1719	D664	0.026		-1.16	
1720		-----		-----	
1743	EN62021-1	0.044	C	3.58	first reported: 0.069
1801	EN62021-1	0.034		0.95	
1888	EN62021-1	0.031		0.16	
1890	ISO6619	0.036		1.47	
1891		-----		-----	
1897		-----		-----	
1923	EN62021-1	0.035		1.21	
1924	EN62021-1	0.0355		1.34	
1925	EN62021-1	0.035		1.21	
1947		-----		-----	
1958		-----		-----	
1966		-----		-----	
2122	EN62021-1	0.03		-0.10	
3179		-----		-----	
6015		-----		-----	
6017	EN62021	0.0221	ex	-2.18	excluded for it is uncertain if reported method is a potentiometric titration
	normality	OK			
	n	32			
	outliers	2 (+4ex)			
	mean (n)	0.0304			
	st.dev. (n)	0.00541			
	R(calc.)	0.0152			
	R(EN62021-1:03)	0.0106			Compare R(D664-A:11a) = 0.0134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0.04
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445
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1687
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179
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1417
1178
2122
1888
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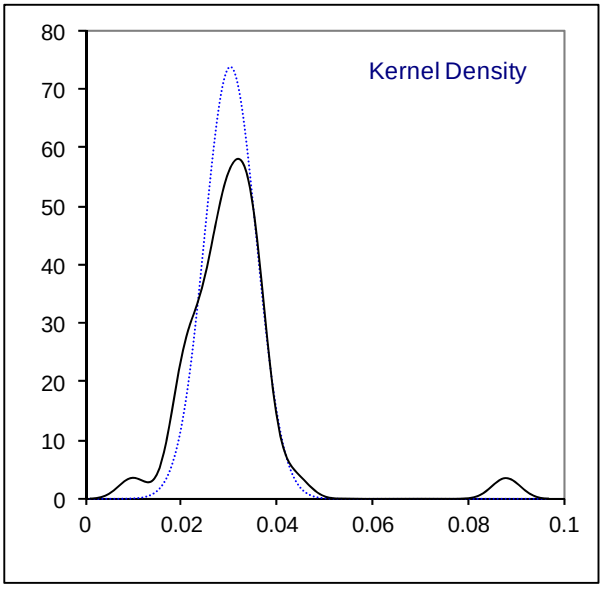
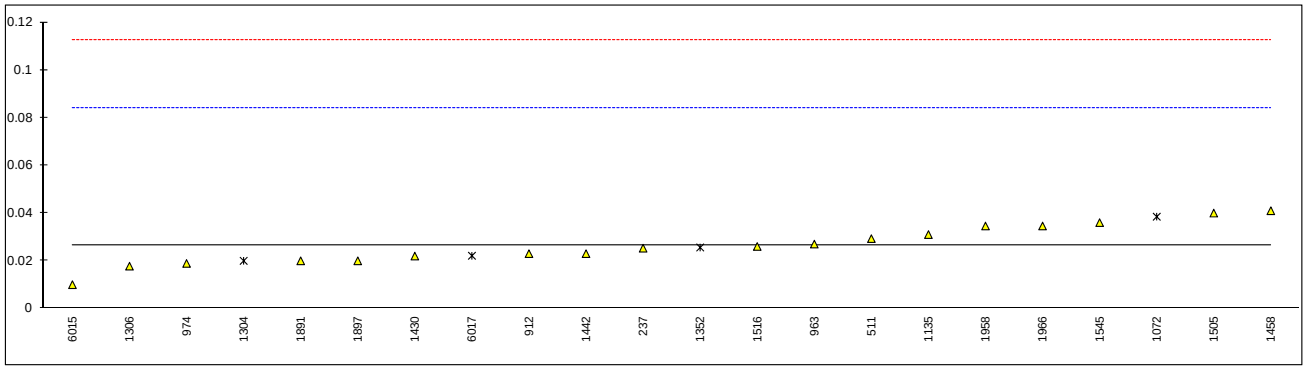
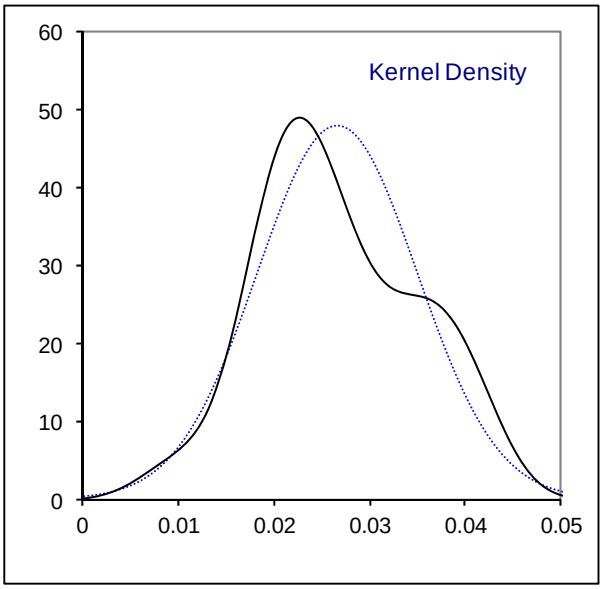

Kernel Density
80
70
60
50
40
30
20
10
0
0
0.02
0.04
0.06
0.08
0.1

Determination of Acid Number (Colorimetric Titration) on sample #15223; results in g KOH/kg

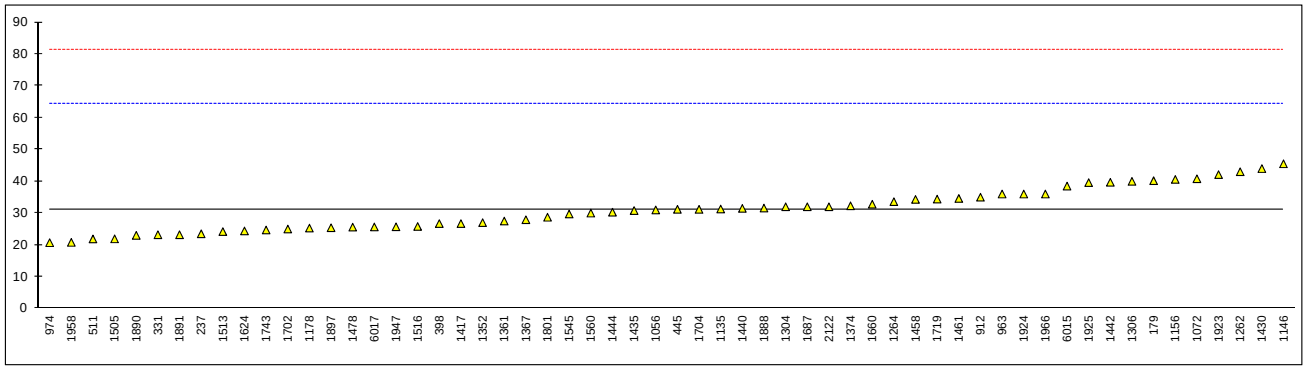
lab	method	value	mark	z(targ)	remarks
179		-----		-----	
225		-----		-----	
237	D974	0.025296		-0.05	
331		-----		-----	
398		-----		-----	
445		-----		-----	
511	D974	0.0293		0.09	
541		-----		-----	
614		-----		-----	
862		-----		-----	
912	D974	0.023		-0.13	
963	D974	0.027		0.01	
974	D974	0.0189		-0.27	
1056		-----		-----	
1072	in house	0.0385	ex	0.42	excluded for it is uncertain if reported method is a colorimetric titration
1135	D974	0.031		0.15	
1146		-----		-----	
1156		-----		-----	
1161		-----		-----	
1178		-----		-----	
1262		-----		-----	
1264		-----		-----	
1303		-----		-----	
1304	INH-122	0.02	ex	-0.23	excluded for it is uncertain if reported method is a colorimetric titration
1306	D974	0.01774		-0.31	
1352	IEC62021	0.02558	ex	-0.04	excluded for it is uncertain if reported method is a colorimetric titration
1361		-----		-----	
1367		-----		-----	
1374		-----		-----	
1417		-----		-----	
1430	EN62021-2	0.022		-0.16	
1435		-----		-----	
1440		-----		-----	
1442	IEC62021-2	0.023		-0.13	
1444		-----		-----	
1458	D974	0.041		0.50	
1461		-----		-----	
1478		-----		-----	
1505	D974	0.040		0.47	
1513		-----		-----	
1516	D974	0.026		-0.02	
1545	D974	0.036		0.33	
1560		-----		-----	
1624		-----		-----	
1660		-----		-----	
1687		-----		-----	
1702		-----		-----	
1704		-----		-----	
1719		-----		-----	
1720		-----		-----	
1743		-----		-----	
1801		-----		-----	
1888		-----		-----	
1890		-----		-----	
1891	IEC62021-2	0.02		-0.23	
1897	EN62021-2	0.02		-0.23	
1923		-----		-----	
1924		-----		-----	
1925		-----		-----	
1947		-----		-----	
1958	D974	0.0346		0.28	
1966	ISO6618	0.0346		0.28	
2122		-----		-----	
3179		-----		-----	
6015	ISO6618	0.01		-0.58	
6017	EN62021	0.0221	ex	-0.16	excluded for it is uncertain if reported method is a colorimetric titration
	normality	OK			
	n	18			
	outliers	0 (+4ex)			
	mean (n)	0.0266			
	st.dev. (n)	0.00833			
	R(calc.)	0.0233			
	R(D974:14e1)	0.0800			Compare R(EN62021-2:07) = 0.0053

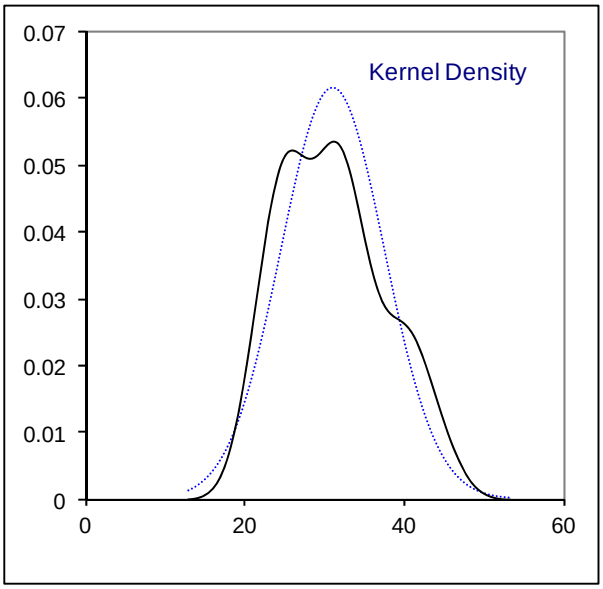

0.12
0.1
0.08
0.06
0.04
0.02
0
6015
1306
974
1304
1891
1897
1430
6017
912
1442
237
1352
1516
963
511
1135
1958
1966
1545
1072
1505
1458


Kernel Density
60
50
40
30
20
10
0
0
0.01
0.02
0.03
0.04
0.05

Determination of Breakdown Voltage on sample #15223, results in kV/2.5 mm

lab	method	value	mark	z(targ)	remarks
179	D877	40.2		0.55	
225		-----		-----	
237	D877	23.5		-0.45	
331	EN60156	23.2		-0.47	
398	EN60156	26.7		-0.26	
445	IEC60156	31.2		0.01	
511	D1816	21.87		-0.55	
541		-----		-----	
614		-----		-----	
862		-----		-----	
912	D877	35		0.24	
963	D877	36.0		0.30	
974	EN60156	20.7		-0.62	
1056	IP295	31		0.00	
1072	EN60156	40.8		0.59	
1135	IEC60156	31.3		0.02	
1146	IEC156	45.5		0.87	
1156	IEC60156	40.6		0.57	
1161		-----		-----	
1178	IEC60156	25.3		-0.34	
1262	EN60156	43.0		0.72	
1264	IEC60156	33.60		0.16	
1303		-----		-----	
1304	INH-124	32		0.06	
1306		40.0		0.54	
1352	IEC60156	27.0		-0.24	
1361	EN60156	27.5		-0.21	
1367	EN60156	27.9		-0.19	
1374	IEC60156	32.3		0.08	
1417	EN60156	26.7		-0.26	
1430	EN60156	44		0.78	
1435	IEC60156	30.8		-0.01	
1440	EN60156	31.5		0.03	
1442	IEC60156	39.7		0.52	
1444	IEC60156	30.3		-0.04	
1458	IEC60156	34.3		0.20	
1461	EN60156	34.6		0.22	
1478	IEC60156	25.6		-0.32	
1505	IEC60156	21.9		-0.54	
1513	IEC60156	24.2		-0.41	
1516	IEC60156	25.8		-0.31	
1545	IEC60156	29.71		-0.08	
1560	IEC60156	30		-0.06	
1624	IEC60156	24.4		-0.39	
1660	EN60156	32.8		0.11	
1687	EN60156	32		0.06	
1702	IEC60156	25.0		-0.36	
1704	IEC60156	31.2		0.01	
1719	IEC60156	34.4		0.20	
1720		-----		-----	
1743	IEC60156	24.7		-0.38	
1801	EN60156	28.7		-0.14	
1888	IEC60156	31.6		0.04	
1890	IEC60156	23		-0.48	
1891	IEC60156	23.2		-0.47	
1897	IEC60156	25.4		-0.33	
1923	EN60156	42.1		0.66	
1924	EN60156	36.0		0.30	
1925	EN60156	39.6		0.51	
1947	EN60156	25.7		-0.32	
1958	IEC60156	20.8		-0.61	
1966	IEC60156	36		0.30	
2122	EN60156	32		0.06	
3179		-----		-----	
6015	EN60156	38.5		0.45	
6017	EN60156	25.66		-0.32	
	normality	OK			
	n	58			
	outliers	0			
	mean (n)	31.00			
	st.dev. (n)	6.478			
	R(calc.)	18.14			
	R(EN60156:95)	46.87			

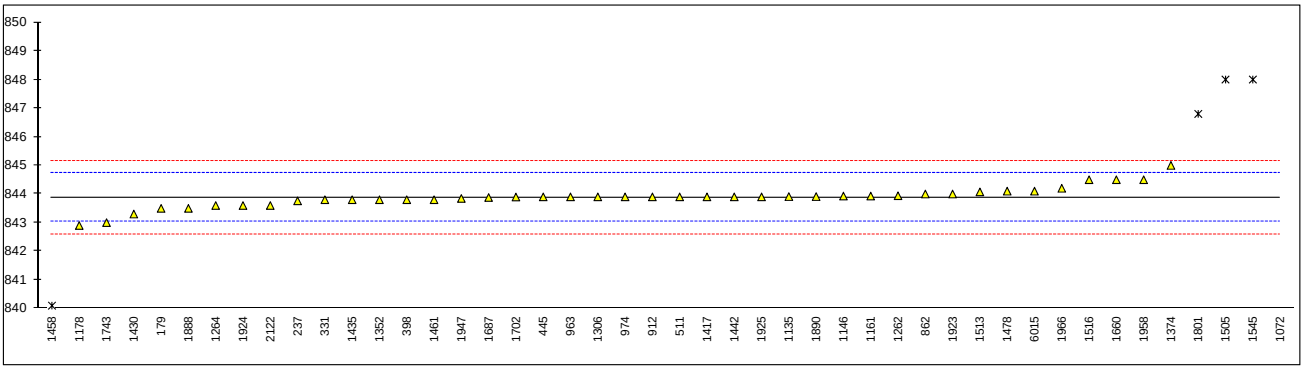

90
80
70
60
50
40
30
20
10
0
974 1958 511 1505 1890 331 1891 237 1513 1624 1743 1702 1178 1897 1478 6017 1947 1516 398 1417 1352 1361 1367 1801 1545 1560 1444 1435 1056 445 1704 1135 1440 1888 1304 1687 2122 1374 1660 1264 1458 1719 1461 912 963 1924 1966 6015 1925 1442 1306 179 1156 1072 1923 1262 1430 1146

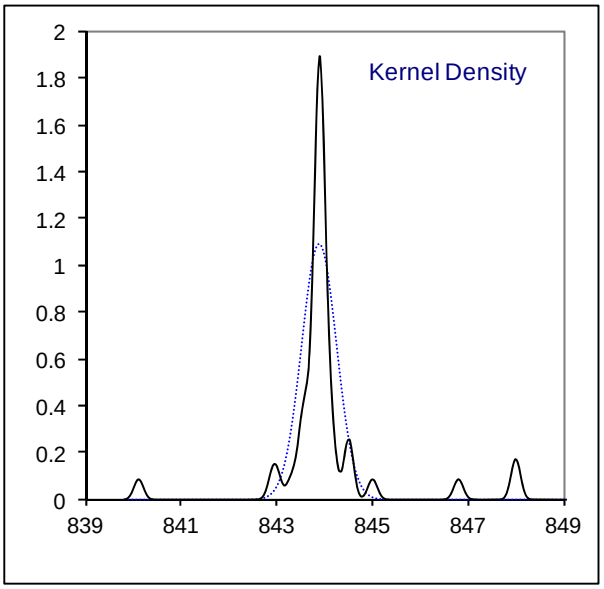

Kernel Density
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
0
20
40
60

Determination of Density at 20°C on sample #15223; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
179	D4052	843.5		-0.89	
225		-----		-----	
237	D4052	843.76		-0.28	
331	ISO12185	843.8		-0.19	
398	ISO12185	843.8		-0.19	
445	D4052	843.9		0.05	
511	D4052	843.9		0.05	
541		-----		-----	
614		-----		-----	
862	D4052	844.0		0.28	
912	D4052	843.9	C	0.05	reported: 0.8439 kg/m^3
963	D4052	843.9		0.05	
974	D4052	843.9		0.05	
1056		-----		-----	
1072	ISO3675	854.0	R(0.01)	23.61	
1135	ISO12185	843.91	C	0.07	first reported: 0.84391 kg/m^3
1146	ISO12185	843.93		0.12	
1156		-----		-----	
1161	ISO12185	843.93	C	0.12	first reported: 847.05
1178	ISO12185	842.90		-2.29	
1262	ISO3675	843.94		0.14	
1264	D4052	843.6		-0.65	
1303		-----		-----	
1304		-----		-----	
1306	D4052	843.9		0.05	
1352	D7042	843.8		-0.19	
1361		-----		-----	
1367		-----		-----	
1374	D7777	845		2.61	
1417	D4052	843.9		0.05	
1430	D4052	843.3		-1.35	
1435	D4052	843.8	C	-0.19	reported 0.8436 kg/m^3
1440		-----		-----	
1442	D7042	843.9		0.05	
1444		-----		-----	
1458	D4052	840.1	R(0.01)	-8.82	
1461	ISO3675	843.8		-0.19	
1478	ISO12185	844.1		0.51	
1505	D7042	848.0	R(0.01)	9.61	
1513	ISO12185	844.075		0.46	
1516	ISO3675	844.5		1.45	
1545	ISO3675	848.0	C,R(0.01)	9.61	first reported: 846.0
1560		-----		-----	
1624		-----		-----	
1660	D7042	844.5		1.45	
1687	ISO12185	843.88		0.00	
1702	ISO12185	843.897		0.04	
1704		-----		-----	
1719		-----		-----	
1720		-----		-----	
1743		843		-2.05	
1801	ISO3675	846.8	R(0.01)	6.81	
1888	ISO3675	843.5		-0.89	
1890	ISO12185	843.91		0.07	
1891		-----		-----	
1897		-----		-----	
1923	ISO3675	844.0		0.28	
1924	ISO3675	843.6		-0.65	
1925	ISO3675	843.9		0.05	
1947	ISO12185	843.845		-0.08	
1958	D4052	844.5	C	1.45	first reported: 0.8445 kg/m^3
1966	ISO3675	844.2	C	0.75	first reported: 842.2
2122	INH-12185	843.6		-0.65	
3179		-----		-----	
6015	ISO12185	844.1		0.51	
6017		-----		-----	

normality	not OK
n	41
outliers	5
mean (n)	843.88
st.dev. (n)	0.365
R(calc.)	1.02
R(ISO3675:98)	1.20

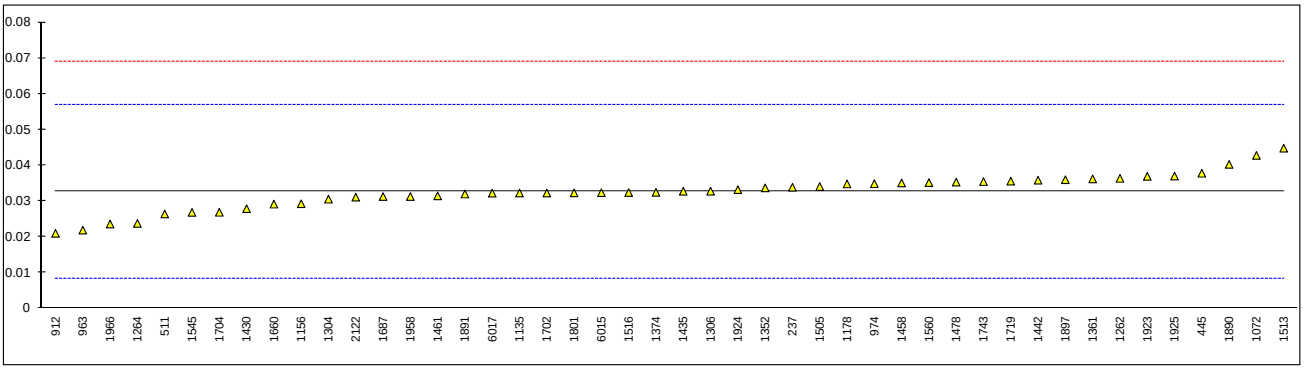

850
849
848
847
846
845
844
843
842
841
840
1458
1178
1743
1430
179
1888
1264
1924
2122
237
331
1435
1352
398
1461
1947
1687
1702
445
963
1306
974
912
511
1417
1442
1925
1135
1890
1146
1161
1262
862
1923
1513
1478
6015
1966
1516
1660
1958
1374
1801
1505
1545
1072

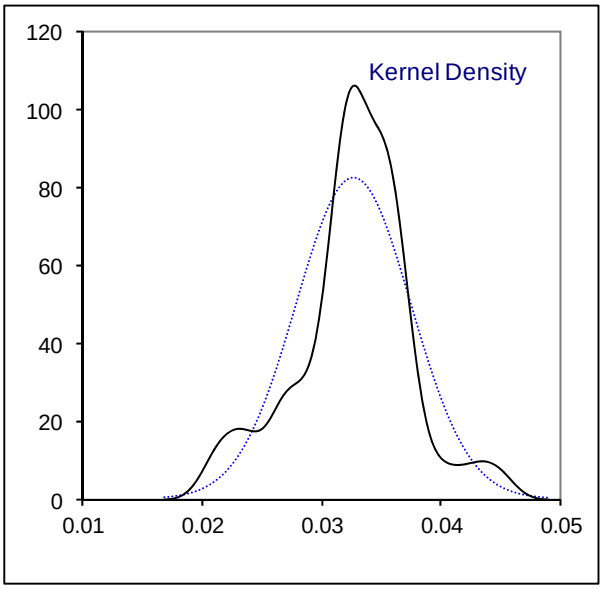

Kernel Density
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
839
841
843
845
847
849

Determination of Di-electric Dissipation Factor at 90°C on sample #15223

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
225		-----		-----	
237	IEC60247	0.033904		0.10	
331		-----		-----	
398		-----		-----	
445	IEC60247	0.03781		0.43	
511	D924	0.02643		-0.51	
541		-----		-----	
614		-----		-----	
862		-----		-----	
912	D924	0.021		-0.96	
963	IEC60247	0.0219		-0.89	
974	EN60247	0.0349		0.19	
1056		-----		-----	
1072	EN60247	0.04280		0.84	
1135	IEC60247	0.03227		-0.03	
1146		-----		-----	
1156	IEC60247	0.02929		-0.28	
1161		-----		-----	
1178	IEC60247	0.03481		0.18	
1262	EN60247	0.03638		0.31	
1264	IEC60247	0.02379		-0.73	
1303		-----		-----	
1304	INH-125	0.030567		-0.17	
1306	IEC60247	0.032792		0.01	
1352	IEC60247	0.0337		0.09	
1361	EN60247	0.036238		0.30	
1367		-----		-----	
1374	IEC60247	0.03247		-0.02	
1417		-----		-----	
1430	EN60247	0.02791		-0.39	
1435	EN60247	0.03277		0.01	
1440		-----		-----	
1442	IEC60247	0.0358684		0.27	
1444		-----		-----	
1458	IEC60247	0.0351		0.20	
1461	EN60247	0.031475		-0.10	
1478	IEC60247	0.035316		0.22	
1505	IEC60247	0.03409		0.12	
1513	IEC60247	0.0448		1.00	
1516	IEC60247	0.03243		-0.02	
1545	IEC60247	0.02687		-0.48	
1560	IEC60247	0.0352		0.21	
1624		-----		-----	
1660	EN60247	0.02918		-0.29	
1687	EN60247	0.031308		-0.11	
1702	IEC60247	0.0322835		-0.03	
1704	IEC60247	0.0269		-0.48	
1719	IEC60247	0.03559		0.24	
1720		-----		-----	
1743	IEC60247	0.035479		0.23	
1801	EN60247	0.032339		-0.03	
1888		-----		-----	
1890	IEC60247	0.040282		0.63	
1891	IEC60247	0.032		-0.05	
1897	IEC60247	0.036		0.28	
1923	EN60247	0.03694		0.35	
1924	EN60247	0.033215		0.05	
1925	EN60247	0.03702		0.36	
1947		-----		-----	
1958	IEC60247	0.03131		-0.11	
1966	IEC60247	0.02361		-0.75	
2122	EN60247	0.031120		-0.13	
3179		-----		-----	
6015	EN60247	0.032374		-0.02	
6017	EN60247	0.03224		-0.03	

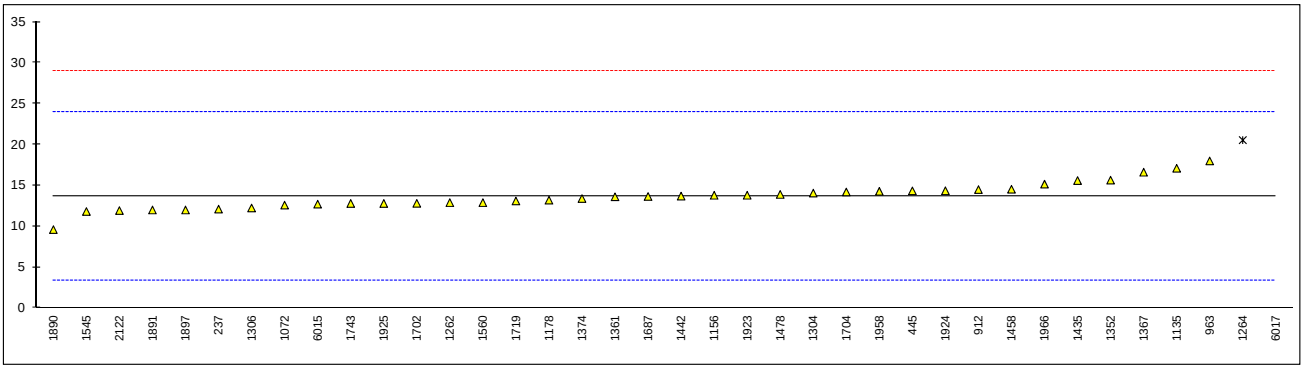
normality	OK
n	46
outliers	0
mean (n)	0.03265
st.dev. (n)	0.004825
R(calc.)	0.01351
R(EN60247:04)	0.03390


0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
912 963 1966 1264 511 1545 1704 1430 1660 1156 1304 2122 1687 1958 1461 1891 6017 1135 1702 1801 6015 1516 1374 1435 1306 1924 1352 237 1505 1178 974 1458 1560 1478 1743 1719 1442 1897 1361 1262 1923 1925 445 1890 1072 1513


Kernel Density
120
100
80
60
40
20
0
0.01
0.02
0.03
0.04
0.05

Determination of Specific Resistance at 90°C on sample #15223; results in GΩm

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
225		-----		-----	
237	IEC60247	12.11		-0.30	
331		-----		-----	
398		-----		-----	
445	IEC60247	14.335		0.13	
511		-----		-----	
541		-----		-----	
614		-----		-----	
862		-----		-----	
912	D1169	14.5	C	0.16	reported $1.45*10^{12}$ GΩm (possible unit error?)
963	D1169	18.0		0.85	
974		-----		-----	
1056		-----		-----	
1072	EN60247	12.6		-0.21	
1135	IEC60247	17.1		0.67	
1146		-----		-----	
1156	IEC60247	13.8		0.03	
1161		-----		-----	
1178	IEC60247	13.2		-0.09	
1262	EN60247	12.9		-0.15	
1264	IEC60247	20.54	R(0.01)	1.34	
1303		-----		-----	
1304	INH-125	14.07		0.08	
1306	IEC60247	12.24		-0.28	
1352	IEC60247	15.65		0.39	
1361	EN60247	13.61		-0.01	
1367	EN60247	16.62		0.58	
1374	IEC60247	13.4		-0.05	
1417		-----		-----	
1430		-----		-----	
1435	IEC60247	15.6		0.38	
1440		-----		-----	
1442	IEC60247	13.7		0.01	
1444		-----		-----	
1458	IEC60247	14.54		0.17	
1461		-----		-----	
1478	IEC60247	13.9		0.05	
1505		-----		-----	
1513		-----		-----	
1516		-----		-----	
1545	IEC60247	11.82		-0.36	
1560	IEC60247	12.9		-0.15	
1624		-----		-----	
1660		-----		-----	
1687	EN60247	13.65		0.00	
1702	IEC60247	12.81		-0.17	
1704	IEC60247	14.2		0.11	
1719	IEC60247	13.1	C	-0.11	reported: 13.1E9 GΩm (possible unit error?)
1720		-----		-----	
1743	IEC60247	12.80		-0.17	
1801		-----		-----	
1888		-----		-----	
1890	IEC60247	9.6		-0.79	
1891	IEC60247	12		-0.32	
1897	IEC60247	12		-0.32	
1923	EN60247	13.8		0.03	
1924	EN60247	14.35		0.13	
1925	EN60247	12.8		-0.17	
1947		-----		-----	
1958	IEC60247	14.28		0.12	
1966	IEC60247	15.16		0.29	
2122	EN60247	11.93		-0.34	
3179		-----		-----	
6015	EN60247	12.71		-0.19	
6017	EN60247	$4.336*10^{10}$	ex	-----	excluded, test temperature used was 20°C
	normality	suspect			
	n	36			
	outliers	1 (+1ex)			
	mean (n)	13.6607			
	st.dev. (n)	1.61713			
	R(calc.)	4.5280			
	R(EN60247:04)	14.3437			


35
30
25
20
15
10
5
0
1890
1545
2122
1891
1897
237
1306
1072
6015
1743
1925
1702
1262
1560
1719
1178
1374
1361
1687
1442
1156
1923
1478
1304
1704
1958
445
1924
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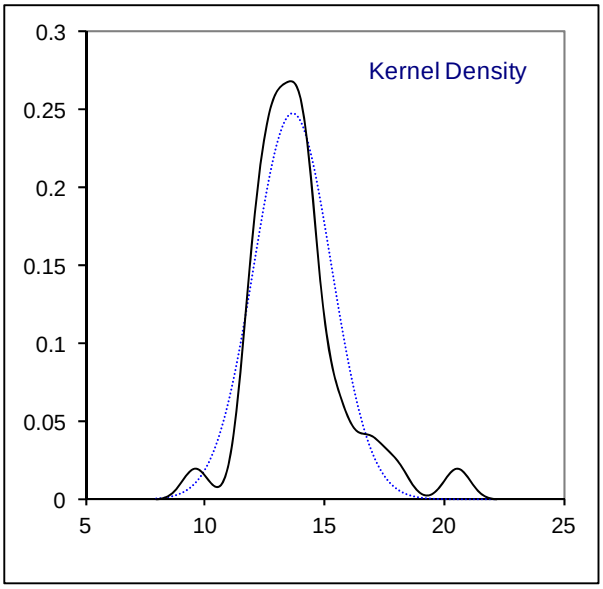
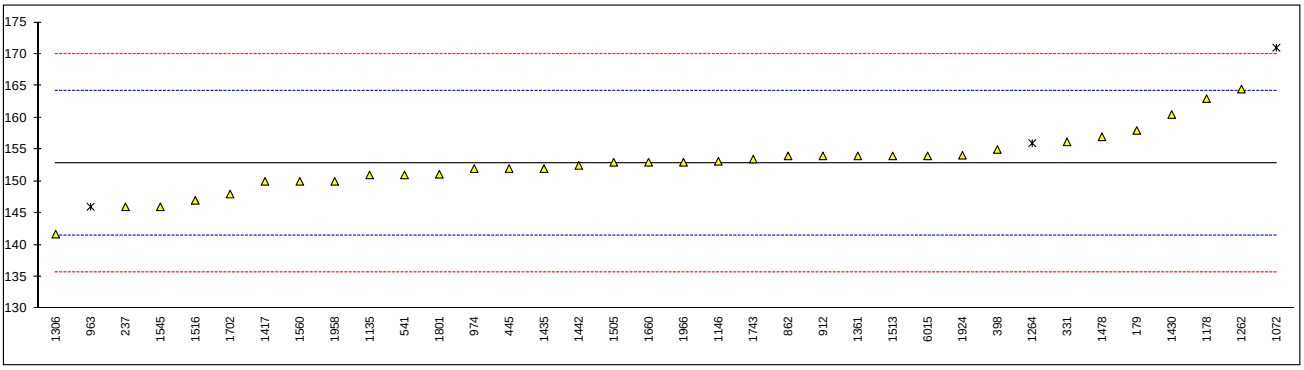
1966
1435
1352
1367
1135
963
1264
6017


Kernel Density
0.3
0.25
0.2
0.15
0.1
0.05
0
5
10
15
20
25

Determination of Flash Point PMcc on sample #15223; results in °C

lab	method	value	mark	z(targ)	remarks
179	D93	158.0		0.90	
225		-----		-----	
237	D93	146.0		-1.20	
331	D93	156.2		0.59	
398	ISO2719	155		0.38	
445	ISO2719	152.0		-0.15	
511		-----		-----	
541	ISO2719	151.0		-0.32	
614		-----		-----	
862	D93	154.0		0.20	
912	D93	154		0.20	
963	D92	146	ex	-1.20	excluded, method is not equivalent to Flash Point PMcc
974	D93	152.0		-0.15	
1056		-----		-----	
1072	ISO2719	171	R(0.05)	3.18	
1135	ISO2719	151.0		-0.32	
1146	in house	153.15		0.05	
1156		-----		-----	
1161		-----		-----	
1178	ISO2719	163.0		1.78	
1262	D93	164.5		2.04	
1264	D92	156	ex	0.55	excluded, method is not equivalent to Flash Point PMcc
1303		-----		-----	
1304		-----		-----	
1306	D93	141.7		-1.95	
1352		-----		-----	
1361	ISO2719	154.0		0.20	
1367		-----		-----	
1374		-----		-----	
1417	IP34	150		-0.50	
1430	ISO2719	160.5		1.34	
1435	D93	152		-0.15	
1440		-----		-----	
1442	ISO2719	152.5		-0.06	
1444		-----		-----	
1458		-----		-----	
1461		-----		-----	
1478	ISO2719	157.0		0.73	
1505	D93	153		0.03	
1513	ISO2719	154.0		0.20	
1516	ISO2719	147		-1.02	
1545	ISO2719	146.0		-1.20	
1560	ISO2719	150		-0.50	
1624		-----		-----	
1660	D93	153		0.03	
1687		-----		-----	
1702	ISO2719	148.0		-0.85	
1704		-----		-----	
1719		-----		-----	
1720		-----		-----	
1743	ISO2719	153.5		0.11	
1801	ISO2719	151.1		-0.31	
1888		-----		-----	
1890		-----		-----	
1891		-----		-----	
1897		-----		-----	
1923		-----		-----	
1924	ISO2719	154.1		0.22	
1925		-----		-----	
1947		-----		-----	
1958	D93	150		-0.50	
1966	ISO2719	153		0.03	
2122		-----		-----	
3179		-----		-----	
6015	D7236	154.0		0.20	
6017		-----		-----	
	normality	suspect			
	n	33			
	outliers	1 (+2ex)			
	mean (n)	152.86			
	st.dev. (n)	4.603			
	R(calc.)	12.89			
	R(ISO2719-B:02)	16.00			Compare R(D93-B:15) = 10.00


175
170
165
160
155
150
145
140
135
130
1306
963
237
1545
1516
1702
1417
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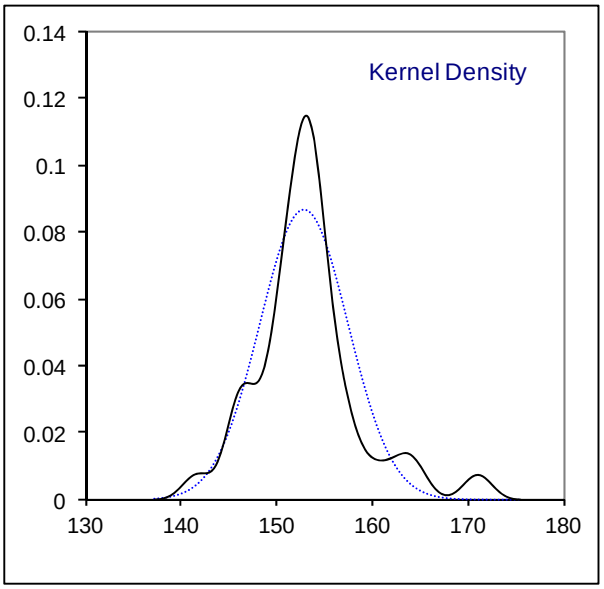
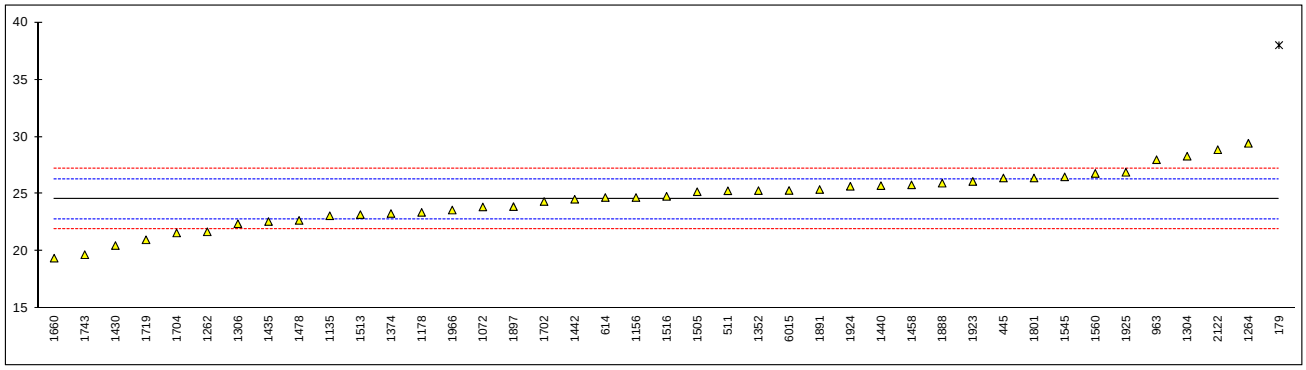
1958
1135
541
1801
974
445
1435
1442
1505
1660
1966
1146
1743
862
912
1361
1513
6015
1924
398
1264
331
1478
179
1430
1178
1262
1072


Kernel Density
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
130
140
150
160
170
180

Determination of Interfacial Surface Tension on sample #15223; results in mN/m

lab	method	value	mark	z(targ)	remarks
179	D971	38	R(0.01)	15.32	
225		-----		-----	
237		-----		-----	
331		-----		-----	
398		-----		-----	
445	D971	26.4		2.10	
511	D971	25.28		0.82	
541		-----		-----	
614	ISO6295	24.7		0.16	
862		-----		-----	
912		-----		-----	
963	D971	28.0		3.92	
974		-----		-----	
1056		-----		-----	
1072	ISO6295	23.87		-0.79	
1135	ISO6295	23.1		-1.66	
1146		-----		-----	
1156	D971	24.7		0.16	
1161		-----		-----	
1178	D971	23.4		-1.32	
1262	D971	21.7		-3.26	
1264	D971	29.43		5.55	
1303		-----		-----	
1304	INH-123	28.3		4.26	
1306	D971	22.4		-2.46	
1352	D971	25.3		0.84	
1361		-----		-----	
1367		-----		-----	
1374	D971	23.3		-1.44	
1417		-----		-----	
1430	D971	20.5		-4.63	
1435	ISO6295	22.6		-2.23	
1440	D971	25.73		1.33	
1442	EN14210	24.54		-0.02	
1444		-----		-----	
1458	D971	25.8		1.41	
1461		-----		-----	
1478	D971	22.7		-2.12	
1505	D971	25.2		0.73	
1513	D971	23.2		-1.55	
1516	D971	24.8		0.27	
1545	D971	26.50		2.21	
1560	D971	26.8		2.55	
1624		-----		-----	
1660	D971	19.4		-5.88	
1687		-----		-----	
1702	D971	24.351		-0.24	
1704	ISO6295	21.6		-3.37	
1719	INH-2285	21		-4.06	
1720		-----		-----	
1743	D971	19.7		-5.54	
1801	ISO6295	26.4		2.10	
1888	ISO6295	25.95		1.58	
1890		-----		-----	
1891	D971	25.4		0.96	
1897	D971	23.9		-0.75	
1923	D971	26.1		1.76	
1924	D971	25.68		1.28	
1925	D971	26.9		2.67	
1947		-----		-----	
1958		-----		-----	
1966	D971	23.6		-1.09	
2122	ISO6295	28.87		4.91	
3179		-----		-----	
6015	D971	25.31		0.85	
6017		-----		-----	

normality	OK
n	40
outliers	1
mean (n)	24.560
st.dev. (n)	2.3618
R(calc.)	6.613
R(D971:12)	2.456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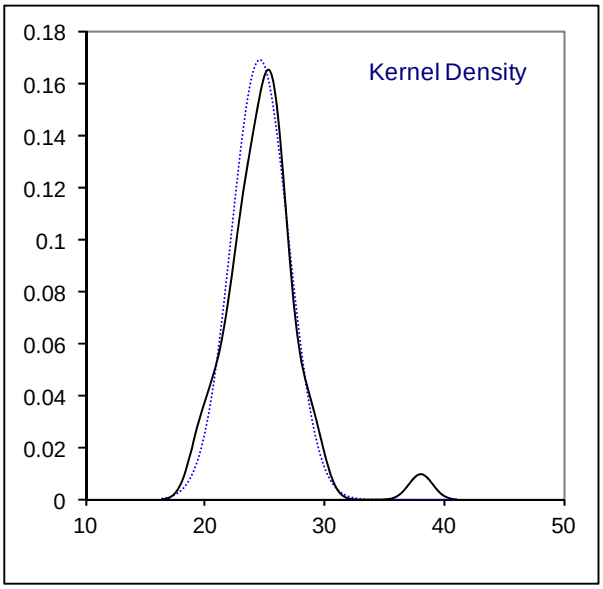
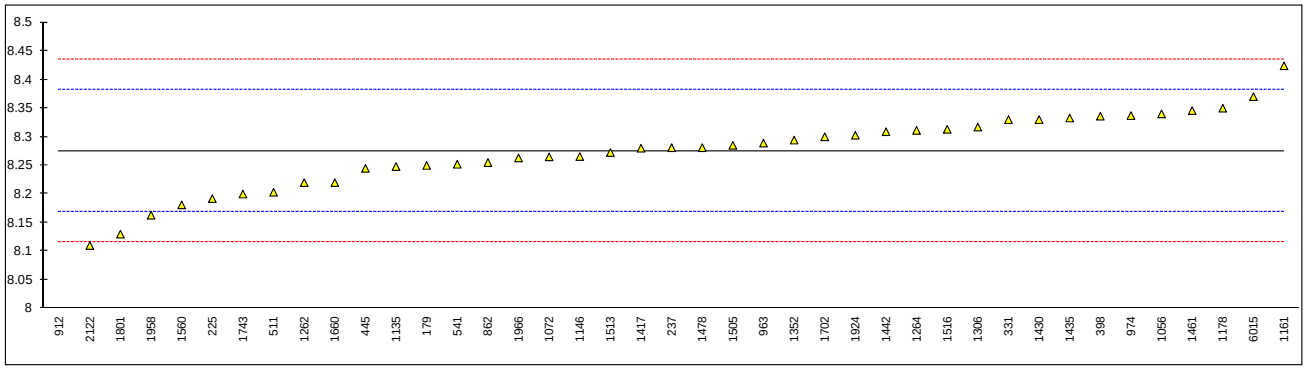
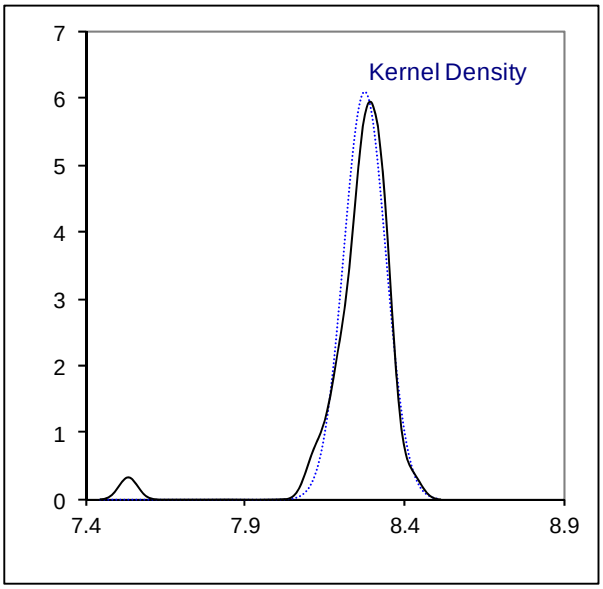
35
30
25
20
15
1660
1743
1430
1719
1704
1262
1306
1435
1478
1135
1513
1374
1178
1966
1072
1897
1702
1442
614
1156
1516
1505
511
1352
6015
1891
1924
1440
1458
1888
1923
445
1801
1545
1560
1925
963
1304
2122
1264
179


Kernel Density
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
10
20
30
40
50

Determination of Kinematic Viscosity at 40°C on sample #15223; results in mm²/s

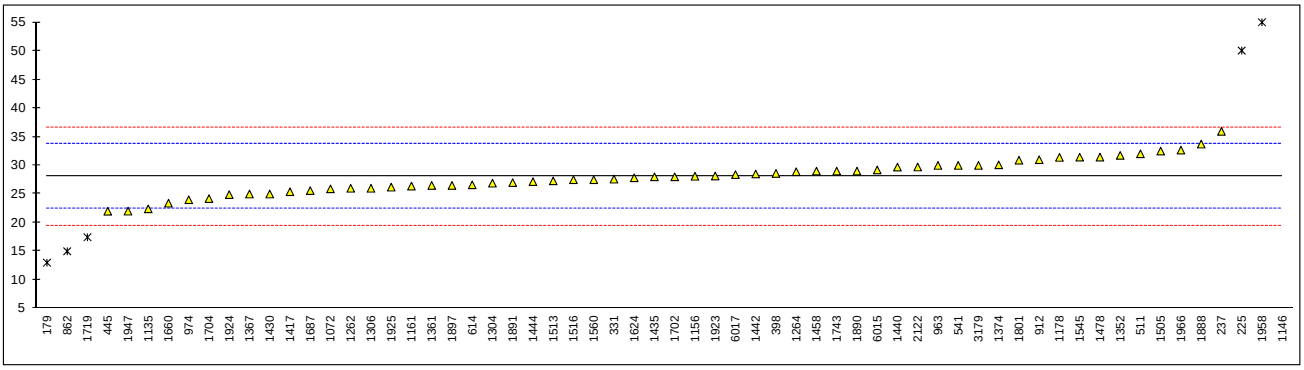
lab	method	value	mark	z(targ)	remarks
179	D445	8.25		-0.47	
225	D445	8.192		-1.56	
237	D445	8.281		0.12	
331	D7279	8.33		1.04	
398	ISO3104	8.336		1.15	
445	ISO3104	8.2448		-0.56	
511	D445	8.2032		-1.35	
541	ISO3104	8.252		-0.43	
614		-----		-----	
862	D445	8.255		-0.37	
912	D445	7.536	R(0.01)	-13.89	
963	D445	8.289		0.27	
974	D445	8.337		1.17	
1056	D7042	8.34		1.22	
1072	ISO3104	8.265		-0.18	
1135	ISO3104	8.2480		-0.50	
1146	D445	8.2655		-0.18	
1156		-----		-----	
1161	ISO3104	8.424	C	2.80	first reported: 8.097
1178	DIN53015	8.35		1.41	
1262	ISO3104	8.22		-1.03	
1264	D7042	8.3112		0.68	
1303		-----		-----	
1304		-----		-----	
1306	D445	8.317		0.79	
1352	D7042	8.2942		0.36	
1361		-----		-----	
1367		-----		-----	
1374		-----		-----	
1417	D7279	8.280		0.10	
1430	ISO3104	8.33		1.04	
1435	D7042	8.3328		1.09	
1440		-----		-----	
1442	D7042	8.3089		0.64	
1444		-----		-----	
1458		-----		-----	
1461	ISO3104	8.3457		1.33	
1478	ISO3104	8.281		0.12	
1505	D7042	8.285		0.19	
1513	ISO3104	8.27247		-0.04	
1516	ISO3104	8.313		0.72	
1545		-----		-----	
1560	ISO3104	8.181		-1.76	
1624		-----		-----	
1660	D7042	8.22		-1.03	
1687		-----		-----	
1702	ISO3104	8.3		0.47	
1704		-----		-----	
1719		-----		-----	
1720		-----		-----	
1743	ISO3104	8.2		-1.41	
1801	ISO3104	8.13		-2.72	
1888		-----		-----	
1890		-----		-----	
1891		-----		-----	
1897		-----		-----	
1923		-----		-----	
1924	ISO3104	8.30270		0.52	
1925		-----		-----	
1947		-----		-----	
1958	D445	8.163		-2.10	
1966	ISO3104	8.263		-0.22	
2122	INH-445	8.110		-3.10	
3179		-----		-----	
6015	D7279	8.37		1.79	
6017		-----		-----	
	normality	OK			
	n	40			
	outliers	1			
	mean (n)	8.2748			
	st.dev. (n)	0.06558			
	R(calc.)	0.1836			
	R(iis)	0.1489			R(iis) = 1.8% of mean for used oils at 40°C (see lit. 17)


8.5
8.45
8.4
8.35
8.3
8.25
8.2
8.15
8.1
8.05
8
912 2122 1801 1958 1560 225 1743 511 1262 1660 445 1135 179 541 862 1966 1072 1146 1513 1417 237 1478 1505 963 1352 1702 1924 1442 1264 1516 1306 331 1430 1435 398 974 1056 1461 1178 6015 1161


Kernel Density
7
6
5
4
3
2
1
0
7.4
7.9
8.4
8.9

Determination of Water on sample #15223; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D6304	13	R(0.05)	-5.30	
225	D6304	50.06	R(0.01)	7.76	
237	D6304	35.95	C	2.79	first reported: 55.95
331	D6304	27.6		-0.15	
398	EN60814	28.6		0.20	
445	IEC60814	22		-2.13	
511	D1533	32.02		1.40	
541	D6304	30		0.69	
614	EN60814	26.6		-0.51	
862	D6304	15	C,R(0.05)	-4.60	first reported: 9.95
912	D6304	31		1.05	
963	D1533	30		0.69	
974	D6304	24		-1.42	
1056		-----		-----	
1072	EN60814	25.9		-0.75	
1135	EN60814	22.4		-1.99	
1146	D6304	110	R(0.01)	28.90	reported: 0.011% M/M
1156	IEC60814	28.1		0.02	
1161	D6304	26.390		-0.58	
1178	IEC60814	31.4		1.19	
1262	EN60814	26.0		-0.72	
1264	D1533	28.9		0.31	
1303		-----		-----	
1304	INH-121	26.9		-0.40	
1306		26		-0.72	
1352	IEC90814	31.75		1.31	
1361	EN60814	26.5		-0.54	
1367	EN60814	25		-1.07	
1374	IEC60814	30.1		0.73	
1417	D6304	25.4		-0.93	
1430	EN60814	25		-1.07	
1435	IEC60814	28		-0.01	
1440	EN60814	29.7		0.59	
1442	IEC60814	28.5		0.16	
1444	IEC60567	27.1649		-0.31	
1458	IEC60814	29		0.34	
1461		-----		-----	
1478	IEC60814	31.45		1.20	
1505	D1533	32.5		1.57	
1513	IEC60814	27.3		-0.26	
1516	IEC60814	27.5		-0.19	
1545	IEC60814	31.44		1.20	
1560	IEC60814	27.5		-0.19	
1624	IEC60814	27.84		-0.07	
1660	EN60814	23.4		-1.63	
1687	IEC60814	25.589		-0.86	
1702	IEC60814	28		-0.01	
1704	IEC60814	24.2		-1.35	
1719	IEC60814	17.45	R(0.05)	-3.73	
1720		-----		-----	
1743	IEC60814	29		0.34	
1801	EN60814	30.9		1.01	
1888	IEC60814	33.72		2.00	
1890	IEC60814	29		0.34	
1891	IEC60814	27		-0.36	
1897	IEC60814	26.5		-0.54	
1923	EN60814	28.15		0.04	
1924	EN60814	24.89		-1.11	
1925	EN60814	26.2		-0.65	
1947	IEC60814	22.008		-2.12	
1958	IEC60814	55	C,R(0.01)	9.51	first reported: 49
1966	IEC60814	32.67		1.63	
2122	EN60814	29.72		0.59	
3179	EN60814	30		0.69	
6015	DIN51777	29.2		0.41	
6017	EN60814	28.397		0.13	
	normality	OK			
	n	56			
	outliers	6			
	mean (n)	28.035			
	st.dev. (n)	2.9327			
	R(calc.)	8.212			
	R(EN60814:98)	7.942			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35
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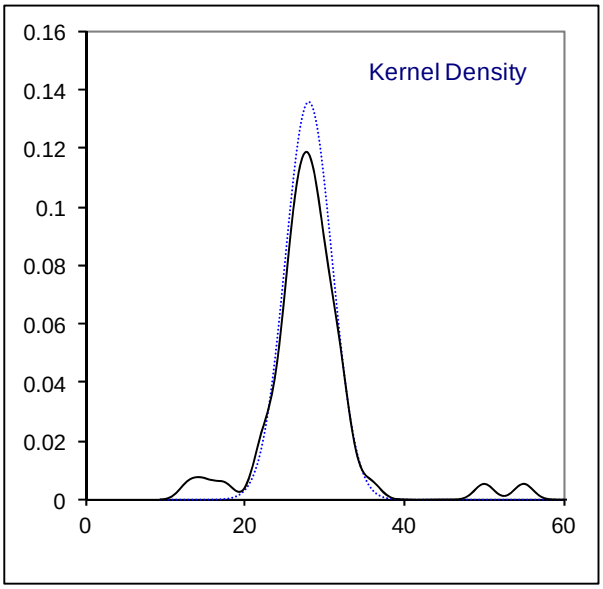
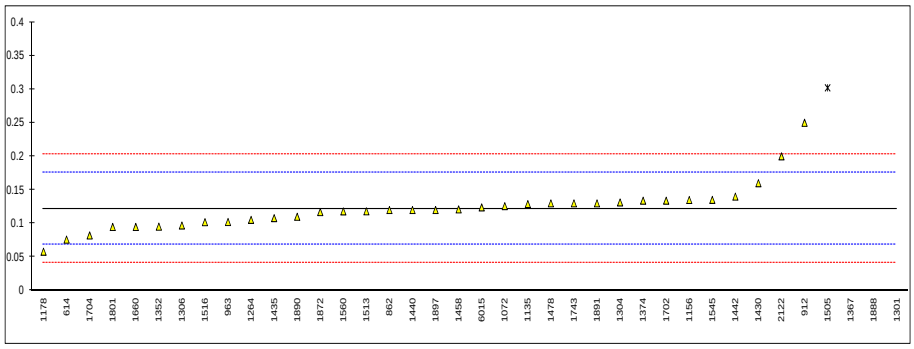
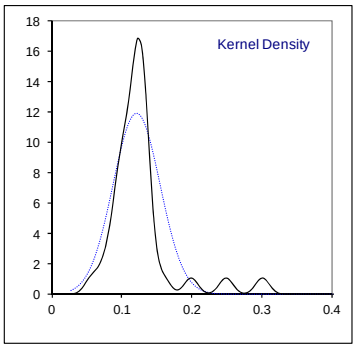
25
20
15
10
5
179 862 1719 445 1947 1135 1660 974 1704 1924 1367 1430 1417 1687 1072 1262 1306 1925 1161 1361 1897 614 1304 1891 1444 1513 1516 1560 331 1624 1435 1702 1156 1923 6017 1442 398 1264 1458 1743 1890 6015 1440 2122 963 541 3179 1374 1801 912 1178 1545 1478 1352 511 1505 1966 1888 237 225 1958 1146


Kernel Density
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0
20
40
60

Determination of 2-Furfural on sample #15224; results in mg/kg

lab	method	value	mark	z(targ)	remarks
237		-----		-----	
398		-----		-----	
445	EN61198	<0.05		<-2.69	possible false negative result?
614	D5837	0.076		-1.72	
862	IEC61198	0.12		-0.08	
912	D5837	0.25		4.78	
963	D5837	0.1022		-0.74	
1072	EN61198	0.1259		0.15	
1135	IEC61198	0.129		0.26	
1156	IEC61198	0.135		0.48	
1178	IEC61198	0.058		-2.39	
1264	D5837	0.1054		-0.62	
1301	D5837	1.426	C,R(0.01)	48.67	first reported: 0.7875
1304	INH-126	0.1314		0.35	
1306	INH-223	0.09705		-0.93	
1352	IEC61198	0.0953		-1.00	
1367	IEC61198	0.78	R(0.01)	24.56	
1374	D5837	0.134		0.45	
1417		-----		-----	
1430	EN61198	0.16		1.42	
1435	IEC61198	0.108		-0.52	
1440	EN61198	0.12		-0.08	
1442	IEC61198	0.140		0.67	
1458	IEC61198	0.121		-0.04	
1478	IEC61198	0.130		0.30	
1505	D5837	0.302	ex	6.72	excluded, see §4.1
1513	IEC61198	0.1181		-0.15	
1516	IEC61198	0.102		-0.75	
1545	IEC61198	0.1352		0.49	
1560	IEC61198	0.118		-0.15	
1660	IEC61198	0.09485		-1.01	
1702	IEC61198	0.134		0.45	
1704	IEC61198	0.0822	C	-1.49	first reported: 0.08305
1743	IEC61198	0.13		0.30	
1801	IEC61198	0.0948		-1.02	
1872	EN61198	0.117		-0.19	
1888	EN61198	1.3347	R(0.01)	45.26	
1890	IEC61198	0.11		-0.45	
1891	IEC61198	0.13		0.30	
1897	IEC61198	0.12		-0.08	
2122	INH-61198	0.2		2.91	
6015	EN61198	0.124		0.07	
6017		-----		-----	
	normality	not OK			
	n	34			
	outliers	3 (+1ex)			
	mean (n)	0.1220			
	st.dev. (n)	0.03362			
	R(calc.)	0.0941			
	R(Horwitz)	0.0750			Compare R(IEC61198:96) = 0.0183



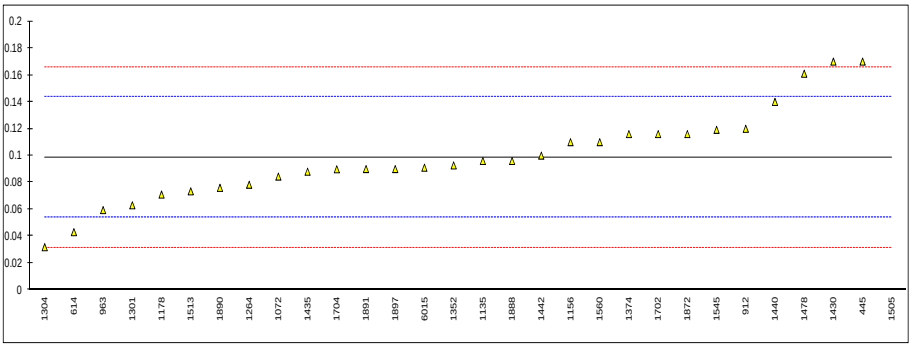


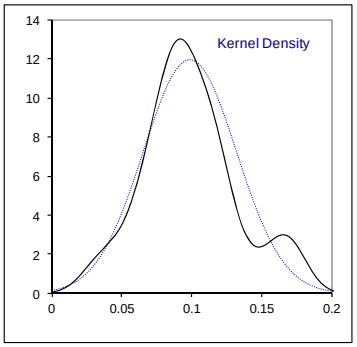
Determination of 2-Furfurylalcohol on sample #15224; results in mg/kg

lab	method	value	mark	z(targ)	remarks
237		-----		-----	
398		-----		-----	
445	IEC61198	0.17		3.19	
614	D5837	0.043		-2.49	
862	IEC61198	<0.05		-----	
912	D5837	0.12		0.95	
963	D5837	0.0594		-1.76	
1072	EN61198	0.0844		-0.64	
1135	IEC61198	0.096		-0.12	
1156	IEC61198	0.110		0.51	
1178	IEC61198	0.071		-1.24	
1264	D5837	0.0783		-0.91	
1301	D5837	0.063		-1.60	
1304	INH-126	0.0318		-2.99	
1306	INH-223	<0.03		<-3.07	possible false negative result?
1352	IEC61198	0.0927		-0.27	
1367		-----		-----	
1374	D5837	0.116		0.77	
1417		-----		-----	
1430	EN61198	0.17		3.19	
1435	IEC61198	0.088		-0.48	
1440	EN61198	0.14		1.85	
1442	IEC61198	0.100		0.06	
1458	IEC61198	<0.01		<-3.96	possible false negative result?
1478	IEC61198	0.161		2.78	
1505	D5837	0.753	R(0.01)	29.24	
1513	IEC61198	0.0734		-1.13	
1516	IEC61198	<0.05		-----	
1545	IEC61198	0.1192		0.92	
1560	IEC61198	0.11		0.51	
1660	IEC61198	<0.05		-----	
1702	IEC61198	0.116		0.77	
1704	IEC61198	0.0898	C	-0.40	first reported: 0.0886
1743	IEC61198	<0.05		-----	
1801		-----		-----	
1872	EN61198	0.116		0.77	
1888	EN61198	0.0960		-0.12	
1890	IEC61198	0.076		-1.01	
1891	IEC61198	0.09		-0.39	
1897	IEC61198	0.09		-0.39	
2122		-----		-----	
6015	EN61198	0.091		-0.34	
6017		-----		-----	

normality	OK
n	29
outliers	1
mean (n)	0.0987
st.dev. (n)	0.03337
R(calc.)	0.0934
R(Horwitz)	0.0627

Compare R(IEC61198:96) = 0.0148



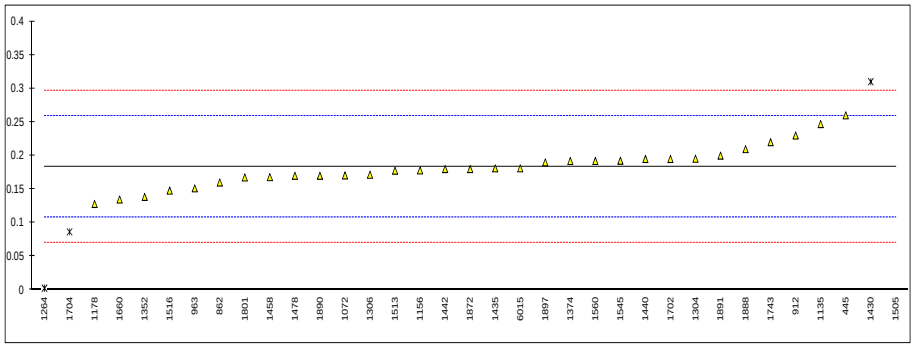


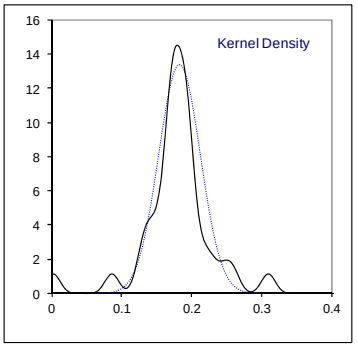
Determination of 5-Methyl-2-furfural on sample #15224; results in mg/kg

lab	method	value	mark	z(targ)	remarks
237		-----		-----	
398		-----		-----	
445	IEC61198	0.26		2.04	
614	D5837	<0.01		<-4.57	possible false negative result?
862	IEC61198	0.16		-0.61	
912	D5837	0.23		1.24	
963	D5837	0.1512		-0.84	
1072	EN61198	0.1705		-0.33	
1135	IEC61198	0.247		1.69	
1156	IEC61198	0.178		-0.13	
1178	IEC61198	0.128		-1.46	
1264	D5837	0.0026	R(0.01)	-4.77	
1301	D5837	<0.01		<-4.57	possible false negative result?
1304	INH-126	0.1954		0.33	
1306	INH-223	0.1716		-0.30	
1352	IEC61198	0.1385		-1.18	
1367		-----		-----	
1374	D5837	0.192		0.24	
1417		-----		-----	
1430	EN61198	0.31	R(0.01)	3.36	
1435	IEC61198	0.181		-0.05	
1440	EN61198	0.195		0.32	
1442	IEC61198	0.180		-0.08	
1458	IEC61198	0.168		-0.40	
1478	IEC61198	0.170		-0.34	
1505	D5837	1.035	R(0.01)	22.53	
1513	IEC61198	0.1776		-0.14	
1516	IEC61198	0.148		-0.93	
1545	IEC61198	0.1924		0.25	
1560	IEC61198	0.192		0.24	
1660	IEC61198	0.13464		-1.28	
1702	IEC61198	0.195		0.32	
1704	IEC61198	0.0864	C,R(0.01)	-2.56	first reported: 0
1743	IEC61198	0.22		0.98	
1801	IEC61198	0.1675		-0.41	
1872	EN61198	0.180		-0.08	
1888	EN61198	0.2096		0.70	
1890	IEC61198	0.17		-0.34	
1891	IEC61198	0.20		0.45	
1897	IEC61198	0.19		0.18	
2122		-----		-----	
6015	EN61198	0.181		-0.05	
6017		-----		-----	

	normality	OK
	n	31
	outliers	4
	mean (n)	0.1830
	st.dev. (n)	0.02979
	R(calc.)	0.0834
	R(Horwitz)	0.1059

Compare R(IEC61198:96) = 0.0275





Determination of other Furanic compounds on sample #15224; results in mg/kg

lab	method	2-af	mark	z(targ)	5-hm-2-f	mark	z(targ)	remarks
237		-----		-----	-----		-----	
398		-----		-----	-----		-----	
445	IEC61198	<0.05		-----	<0.05		-----	
614	D5837	<0.01		-----	0.105		-----	
862	IEC61198	<0.05		-----	<0.05		-----	
912	D5837	0.06		-----	0.11		-----	
963	D5837	<0.01		-----	<0.01		-----	
1072	EN61198	<0.01		-----	<0.01		-----	
1135	IEC61198	0.002		-----	<0.01		-----	
1156	IEC61198	0.00		-----	0.00		-----	
1178	IEC61198	0.0008		-----	0.0008		-----	
1264	D5837	0.1675	false +?	-----	0.0075		-----	
1301	D5837	<0.01		-----	<0.01		-----	
1304	INH-126	<0.01		-----	<0.01		-----	
1306	INH-223	<0.03		-----	<0.03		-----	
1352	IEC61198	n.d.		-----	n.d.		-----	
1367		-----		-----	-----		-----	
1374	D5837	<0.01		-----	<0.01		-----	
1417		-----		-----	-----		-----	
1430	EN61198	0		-----	0		-----	
1435	IEC61198	0.000		-----	0.000		-----	
1440	EN61198	0.005		-----	0.00		-----	
1442	IEC61198	<0.05		-----	<0.05		-----	
1458	IEC61198	<0.01		-----	<0.01		-----	
1478	IEC61198	< 0.01		-----	< 0.01		-----	
1505	D5837	0.709	false +?	-----	0.008		-----	
1513	IEC61198	< 0.05		-----	< 0.05		-----	
1516	IEC61198	<0.05		-----	<0.05		-----	
1545	IEC61198	<0.01		-----	0.0051	C	-----	first reported: 0.0232
1560	IEC61198	n.d		-----	n.d.		-----	
1660	IEC61198	<0.05		-----	<0.05		-----	
1702	IEC61198	n.d.		-----	n.d.		-----	
1704	IEC61198	0.00		-----	0.00		-----	
1743	IEC61198	<0.05		-----	<0.05		-----	
1801		-----		-----	-----		-----	
1872	EN61198	<0.05		-----	<0.05		-----	
1888	EN61198	<0.05		-----	<0.05		-----	
1890		-----		-----	-----		-----	
1891	IEC61198	0		-----	0		-----	
1897	IEC61198	0		-----	0		-----	
2122		-----		-----	-----		-----	
6015	EN61198	<0.05		-----	<0.05		-----	
6017		-----		-----	-----		-----	
	n	35			35			
	mean (n)	<0.05			<0.05			

Abbreviations:

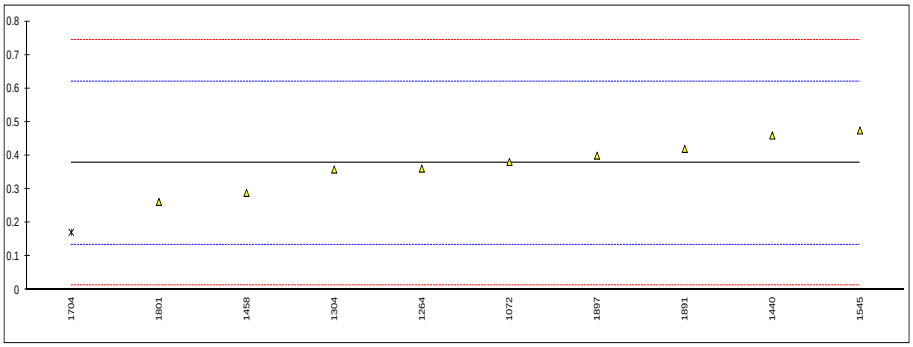
2-af	= 2-acetylfuran
5-hm-2-f	= 5-hydroxymethyl-2-furfural

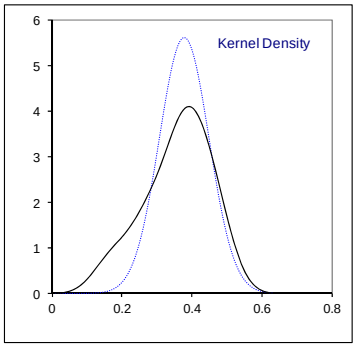
Determination of Total Furanics on sample #15224; results in mg/kg

lab	method	value	mark	z(targ)	remarks
237		-----		-----	
398		-----		-----	
445		-----		-----	
614		-----		-----	
862		-----		-----	
912		-----		-----	
963		-----		-----	
1072		0.3813		0.02	
1135		-----		-----	
1156		-----		-----	
1178		-----		-----	
1264		0.3614		-0.14	
1301		-----		-----	
1304		0.3586		-0.16	
1306		-----		-----	
1352		-----		-----	
1367		-----		-----	
1374		-----		-----	
1417		-----		-----	
1430		-----		-----	
1435		-----		-----	
1440		0.46		0.67	
1442		-----		-----	
1458		0.289		-0.74	
1478		-----		-----	
1505		-----		-----	
1513		-----		-----	
1516		-----		-----	
1545		0.4750		0.79	
1560		-----		-----	
1660		-----		-----	
1702		-----		-----	
1704		0.17165	ex	-1.70	excluded for calculation error, iis calculated: 0.2584
1743		-----		-----	
1801		0.2623		-0.96	
1872		-----		-----	
1888		-----		-----	
1890		-----		-----	
1891		0.42		0.34	
1897		0.40		0.18	
2122		-----		-----	
6015		-----		-----	
6017		-----		-----	

	normality	OK
	n	9
	outliers	0 (+1ex)
	mean (n)	0.3786
	st.dev. (n)	0.07102
	R(calc.)	0.1988
	R(Horwitz)	0.3400

Compare R(IEC61198:96) = 0.0568





APPENDIX 2

Number of participants per country

iis15L09	
1	ARGENTINA
7	AUSTRALIA
3	BELGIUM
6	BULGARIA
1	CHINA, People's Republic
1	COTE D'IVOIRE
1	CROATIA
4	FRANCE
1	GEORGIA
5	GERMANY
1	GREECE
2	INDIA
2	ITALY
2	MALAYSIA
1	MOROCCO
2	NETHERLANDS
1	NEW ZEALAND
1	NIGERIA
1	NORWAY
1	PERU
2	PORTUGAL
1	SAUDI ARABIA
1	SINGAPORE
1	SLOVENIA
1	SOUTH AFRICA
4	SPAIN
1	SUDAN
1	TURKEY
5	UNITED ARAB EMIRATES
4	UNITED KINGDOM
1	UNITED STATES OF AMERICA

iis15L09F	
6	AUSTRALIA
3	BELGIUM
1	CHINA, People's Republic
1	CROATIA
3	FRANCE
2	GERMANY
1	GREECE
2	INDIA
2	ITALY
2	MALAYSIA
1	MOROCCO
1	NIGERIA
1	POLAND
2	PORTUGAL
1	SAUDI ARABIA
1	SINGAPORE
1	SLOVENIA
1	SOUTH AFRICA
4	SPAIN
3	UNITED ARAB EMIRATES
4	UNITED KINGDOM

APPENDIX 3

Abbreviations:

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

Literature:

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