Results of Proficiency Test
Transformer Oil
November 2011

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis11L04

January 2012

CONTENTS

1 INTRODUCTION

Since 2001, the Institute for Interlaboratory Studies organized a proficiency test for the analysis of Transformer Oil every year. It was decided to continue this interlaboratory study during the annual program 2011/2012. In this interlaboratory study, 61 laboratories from 30 different countries have participated. See appendix 2 for a list of number of participants per country order. In this report, the results of the interlaboratory study on unused transformer oil are presented and discussed.

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. In this proficiency test, two different samples were used. The participants received a bottle of 1 litre of an unused Transformer Oil (labelled #11104) and a bottle of 100 mL of used oil (labelled #11105) especially for the analysis of Furanic compounds.
Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO guide 43 and ILAC-G13:2007, (R007), since January 2000, by the Dutch Accreditation Council: RvA (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 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 January 2010 (iis-protocol, version 3.2).

2.3 CONFIDENTIALITY STATEMENT

All data present 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 for 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 (DIALA B dried) for the unused oil sample #11104 was obtained from a local supplier. The approximately 60 litre bulk material was homogenised. After homogenisation, 60 subsamples were transferred to 1 litre amber glass bottles and labelled #11104. The homogeneity of the subsamples #11104 was checked by determination Density and Water on 7 stratified randomly selected samples.

	Water in mg/kg	Density @ 15°C in kg/m^3
Sample #11104-1	17	883.42
Sample #11104-2	17	883.43
Sample #11104-3	18	883.43
Sample #11104-4	17	883.43
Sample #11104-5	17	883.42
Sample #11104-6	19	883.43
Sample #11104-7	20	883.42

Table 1: homogeneity test results of subsamples #11104

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

	Water in mg/kg	Density @ 15°C in kg/m^3
r (Observed)	3	0.01
reference method	EN60814:98	ISO3675:98
0.3 * R (ref. method)	3	0.36

Table 2: repeatabilities of subsamples #11104

The necessary bulk material for additional sample #11105, specifically for Furanic compounds was obtained from a participating laboratory. This material was also used in a previous proficiency test iis10L03. The spread found on the samples in 2010 was good and therefore it was decided to use this material again.
After homogenisation, the bulk material was transferred to 43 subsamples, 100 mL amber glass bottles and labelled #11105.

Each calculated repeatability was equal or less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories depending on the registration, 1*1 litre bottle (labelled #11104) and/or 1*100mL bottle (labelled #11105) was sent on October 26, 2011.

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 #11104: Acid Number (Neutralization Number), Breakdown Voltage, Colour, Density @ 20 ^{o}C, Di-electric loss 90 ^{o}C (Di-electric Dissipation Factor and Specific Resistance), Interfacial Surface Tension and Water.
On sample #11105 was asked to determine: Furanic Compounds (2-acetylfuran, 2-furfural, 2-furfurylalcohol, 5-hydroxymethyl-2-furfural and 5-methyl-2-furfural). To get comparable results, detailed report forms on which the units and the preferred test methods were printed, were sent together with each set of samples. Also, a letter of instructions and a SDS were added to the package.

3 RESULTS

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

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

3.1 STATISTICS

Statistical calculations were performed as described in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of January 2010 (iis-protocol, version 3.2).

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. 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 and Grubbs 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. Stragglers are marked by D(0.05)

for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs 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. 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 under the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a 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; nr.14 and 15).

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. The target standard deviation was calculated from the literature reproducibility by division with 2.8. 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.

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 the fit-for-useness of the reported test result.

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

4 EVALUATION

Some problems were encountered during the dispatch of the unused transformer oil sample #11104 and the sample #11105 “for furanics only” to India, Mexico and Sweden due to unknown reasons.
In total eight participants, reported results after the final reporting date and five participants did not report any results at all. Not all participants were able to report results for all tests.
In total 56 participants reported 378 numerical results. Observed were 27 outlying results, which is 7.1% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

Not all original data sets proved to have a normal distribution. Not normal distribution were found for the following determinations on sample #11104: Colour, Density and Di-electric Dissipation Factor. In these cases the statistical evaluations should be used with due care. In this section, the results are discussed per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

Acid Number: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D711:10 (see note 18, ASTM D974:11).

Breakdown Voltage: This determination was very problematic, as the results seem to be divided bimodally. No statistical outliers were observed. When the two groups are evaluated separately, both calculated reproducibilities are almost in agreement with the requirements of EN60156:98.

Colour: No analytical problems were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1500:08.

Density @ 20°C: This determination was problematic. Seven statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ISO3675:98.

DD-Factor: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of IEC60247:04.

Interf. Surf. Tension: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D971:04 and/or ISO6295:83. One should be aware that ISO6295 is obsolete since February 2005.

Spec. Resistance: This determination was problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the estimated requirements of IEC60247:04.

Water: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of EN60814:98.

Furanic Compounds: Sample #11105 was found to be positive only for 2-Furfural. This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated requirements calculated using the Horwitz equation.
It is remarkable that the precision requirements of IEC1198:93 are much smaller than precision data calculated using the Horwitz equation. This very strict precision seems to be impossible to meet as only few test results would be acceptable against IEC1198:98.
This material was also used in two previous proficiency tests iis07L03 and iis10L03. When the data are compared is it remarkable to notice that the consensus values for both rounds differ only very little, while the spread in the current PT iis11L04 is smaller then in the previous PT's.

	iis07L03 (#0784)	iis10L03 (#1086FC)	iis11L04 (#11105)
number of results	13	16	27
mean	0.463	0.474	0.450
st.dev	0.1133	0.0834	0.072
reproducibility	0.317	0.234	0.202

table 3: Comparison of Performance for 2-Furfural

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	g KOH/kg	39	0.0081	0.0125	0.0150
Breakdown Voltage *)	kV/2.5 mm	30/24	38 / 57	18 / 16	13 / 19
Colour		17	0.4	0.6	1.0
Density @ 20 °C	kg/m^3	38	880.0	1.4	1.2
Di-electric Dissipation Factor		36	0.0014	0.0019	0.0078
Interfacial Surface Tension	mN/m	35	43.2	7.0	4.3
Specific Resistance	GΩm	27	445.5	752.8	467.7
Water	mg/kg	50	21.0	6.5	6.9

table 4: Performance of the group on sample #11104

*) Data appears to be bimodally distributed

Parameter	unit	n	average	2.8 * sd	R(lit)
* 2-acetylfuran	mg/kg	5	0.005	n.a.	n.a.
* 2-furfural	mg/kg	27	0.45	0.20	0.23
* 2-furfurylalcohol	mg/kg	5	0.013	n.a.	n.a.
* 5-hydroxy-2-furfural	mg/kg	7	0.009	n.a.	n.a.
* 5-methyl-2-furfural	mg/kg	9	0.017	n.a.	n.a.

table 5: Performance of the group on sample #11105

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

4.3 COMPARISON OF THE NOVEMBER 2011 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2011	November 2010	November 2009	November 2008
Number of reporting labs	56	46	36	41
Number of results reported	378	289	348	410
Statistical outliers	27	18	21	16
Percentage outliers	7.1%	6.2%	6.0%	3.9%

table 6: 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 2011	November 2010	November 2009	November 2008
Acid number	+ *)	++	+	++
Breakdown Voltage	--	--	--	--
Colour	++	++	++	++
Density @ 20°C	-	+/-	++	--
Di-electric Dissipation Factor	++	++	++	++
Interfacial Surface Tension	--	--	--	--
Specific Resistance	--	--	-- / ++	+/-
Water	+	--	++	--
Furanic Compounds	+	+/-	--	--

table 7: comparison determinations against the standards

*) change of target reproducibility in comparison with previous evaluations

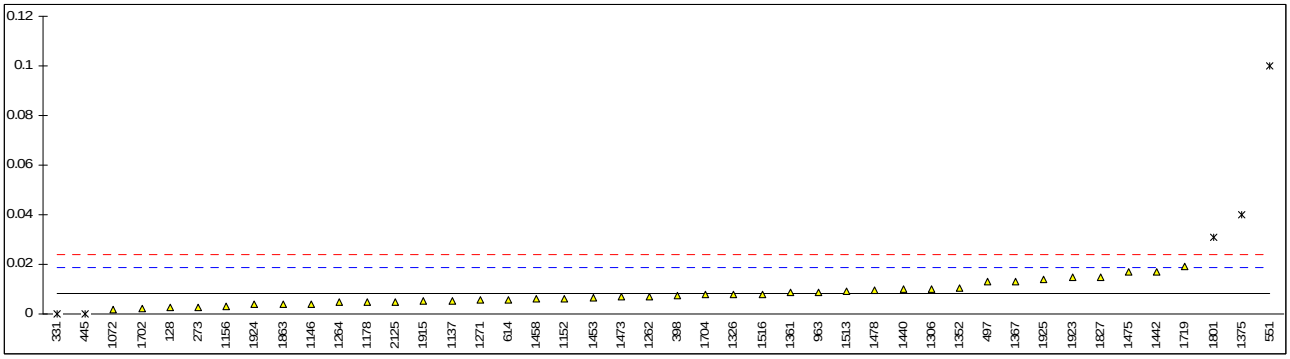
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

++: 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

APPENDIX 1

Determination of Acid Number on sample #11104; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks
128	D974	0.0025		-1.04	
176		-----		-----	
273	D974	0.0026		-1.02	
331	D664	0.00	ex	-1.50	Result excluded, zero is not a real result
398	D974	0.0072		-0.16	
445	IEC62021	0.00	ex	-1.50	Result excluded, zero is not a real result
446	D974	<0.01		<0.36	
497	D974	0.013		0.92	
551	D974	0.10	G(0.01)	17.16	
614	IEC60422	0.0056		-0.46	
963	D974	0.0086		0.10	
1056		-----		-----	
1072	INH-04	0.0019		-1.15	
1137	D974	0.0054		-0.49	
1146	D664	0.0041		-0.74	
1152	D974	0.006		-0.38	
1156	D974	0.003		-0.94	
1178	IEC62021	0.005		-0.57	
1262	EN62021	0.007		-0.20	
1264	D974	0.005		-0.57	
1271	ISO6618	0.0055		-0.48	
1303	D974	<0.01		<0.36	
1304	INH-122	<0.01		<0.36	
1306	in house	0.01		0.36	
1326	D974	0.008		-0.01	
1352	INH-1767	0.0106		0.48	
1361	EN62021	0.0085		0.08	
1367	D974	0.013		0.92	
1375	IEC62021	0.040	G(0.01)	5.96	
1430		-----		-----	
1435	IEC62021	<0.01		<0.36	
1440	IEC62021	0.01		0.36	
1442	IEC62021	0.017		1.67	
1445		-----		-----	
1452	EN62021	<0.03		<4.10	
1453	D664	0.0066		-0.27	
1458	D974	0.006		-0.38	
1461		-----		-----	
1463	D974	<0.01		<0.36	
1471		-----		-----	
1473	D974	0.0070		-0.20	
1475	D664	0.017		1.67	
1478	IEC62021	0.0097		0.31	
1513	IEC62021	0.009		0.18	
1516	D974	0.008		-0.01	
1529	IEC62021	<0.01		<0.36	
1626		-----		-----	
1630		-----		-----	
1660	IEC62021	<0.01		<0.36	
1702	IEC62021	0.002		-1.13	
1704	IEC60296	0.00777		-0.05	
1719	D664Mod.	0.019		2.04	
1801	D974	0.031	G(0.01)	4.28	
1827	D664	0.015		1.30	
1863	D974	0.004		-0.76	
1915	D974	0.0054		-0.49	
1923	IEC62021	0.015		1.30	
1924	IEC62021	0.003965		-0.76	
1925	IEC62021	0.014		1.11	
2122	IEC62021	<0.01		<0.36	
2125	ISO6619	0.005		-0.57	
	normality	OK			
	n	39			
	outliers	3			
	mean (n)	0.0080			
	st.dev. (n)	0.00446			
	R(calc.)	0.0125			
	R(D711:10)	0.0150	(See note 18 in ASTM D974:2011), Compare R(IEC62021:07) = 0.0028		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0.1
0.08
0.06
0.04
0.02
0
331
445
1072
1702
128
273
1156
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1146
1264
1178
2125
1915
1137
1271
614
1458
1152
1453
1473
1262
398
1704
1326
1516
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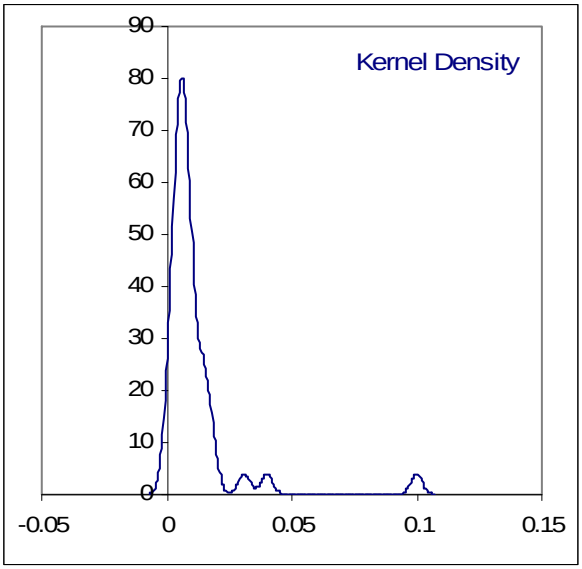
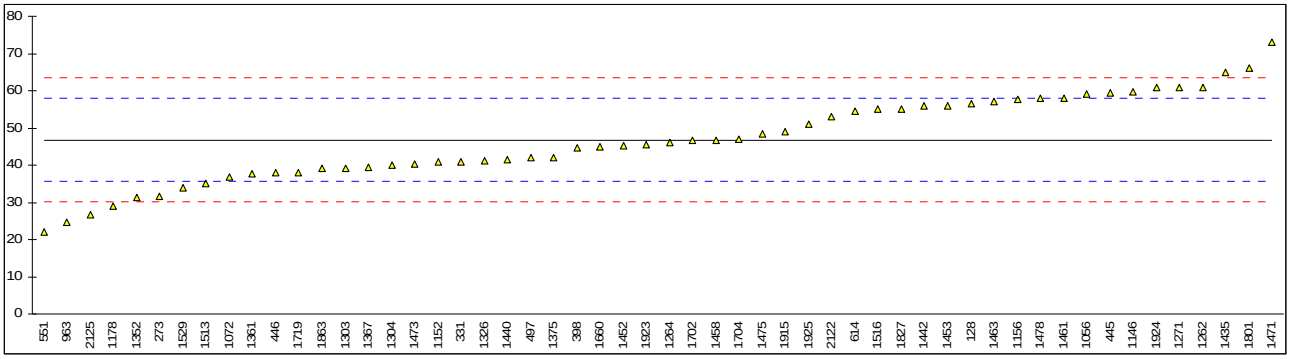
1923
1827
1475
1442
1719
1801
1375
551


Kernel Density
90
80
70
60
50
40
30
20
10
0
-0.05
0
0.05
0.1
0.15

Determination of Breakdown Voltage on sample #11104, results in kV/2.5 mm

lab	method	value	mark	z(targ)	remarks
128	D1816	56.4		-----	
176		-----		-----	
273	IEC60156	31.5		-----	
331	EN60156	41		-----	
398	IEC60156	44.5		-----	
445	IEC156	59.28		-----	
446	IEC60156	37.9		-----	
497	IEC60156	41.9		-----	
551	D877	22		-----	
614	IEC60156	54.5		-----	
963	D877	24.5		-----	
1056	IP265	59		-----	
1072	EN60156	36.7		-----	
1137		-----		-----	
1146	IEC60156	59.8		-----	
1152	IEC156	40.80		-----	
1156	IEC60156	57.6		-----	
1178	IEC60156	28.9		-----	
1262	EN60156	61.0		-----	
1264	IEC156	46.0		-----	
1271	IEC60156	61.0		-----	
1303	IEC60156	39.1		-----	
1304	INH-124	40		-----	
1306		-----		-----	
1326	IEC60156	41.2		-----	
1352	IEC60156	31.3		-----	
1361	EN60156	37.8		-----	
1367	IEC60156	39.55		-----	
1375	IEC60156	42		-----	
1430		-----		-----	
1435	IEC60156	64.9		-----	
1440	IEC60156	41.55		-----	
1442	IEC60156	55.8		-----	
1445		-----		-----	
1452	EN60156	45.1		-----	
1453	IEC60156	56.00		-----	
1458	IEC60156	46.8		-----	
1461	IEC60156	58		-----	
1463	D1816	57		-----	
1471	IEC60156	72.9		-----	
1473	IEC60156	40.4		-----	
1475	IP295	48.5		-----	
1478	IEC60156	58.0		-----	
1513	IEC60156	35.1		-----	
1516	IEC60156	55.0		-----	
1529	IEC60156	34		-----	
1626		-----		-----	
1630		-----		-----	
1660	IEC60156	44.9		-----	
1702	IEC60156	46.7		-----	
1704	IEC60156	46.9		-----	
1719	IEC60156	38.0		-----	
1801	IEC60156	66.2		-----	
1827	IEC60156	55		-----	
1863	IEC60156	39.0		-----	
1915	D877	49.00		-----	
1923	IEC60156	45.6		-----	
1924	IEC60156	60.78		-----	
1925	IEC60156	50.9		-----	
2122	IEC60156	53		-----	
2125	IEC60156	26.8		-----	

	Group 1	Group 2	Only EN60156 data:
normality	not OK	OK	OK
n	30	24	46
outliers	0	0	0
mean (n)	38.35	57.35	47.19
st.dev. (n)	6.582	5.798	10.875
R(calc.)	18.43	16.23	30.45
R(EN60156:95)	12.66	18.93	15.44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60
50
40
30
20
10
0
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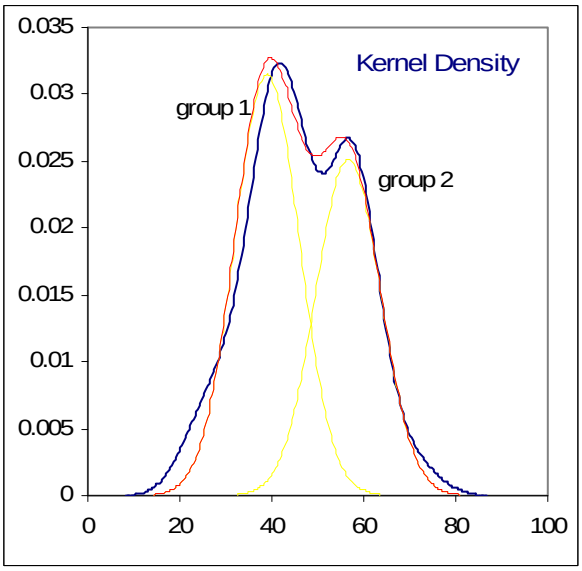
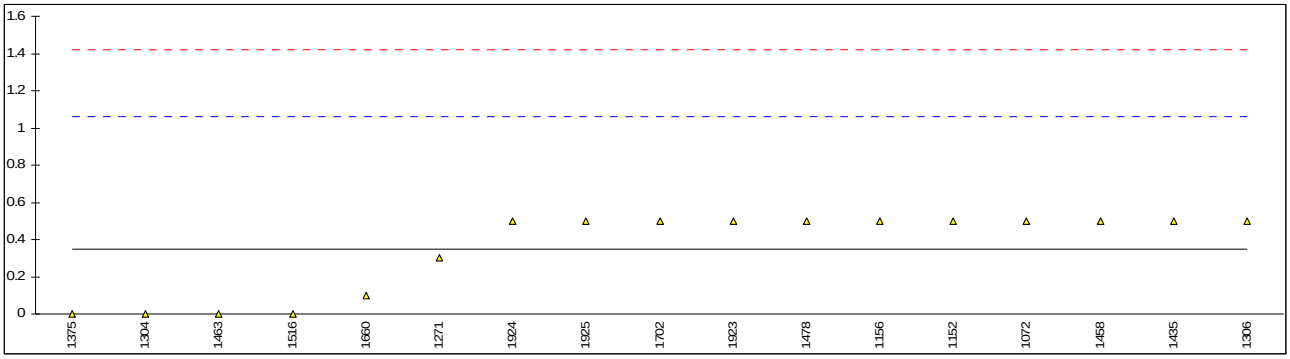
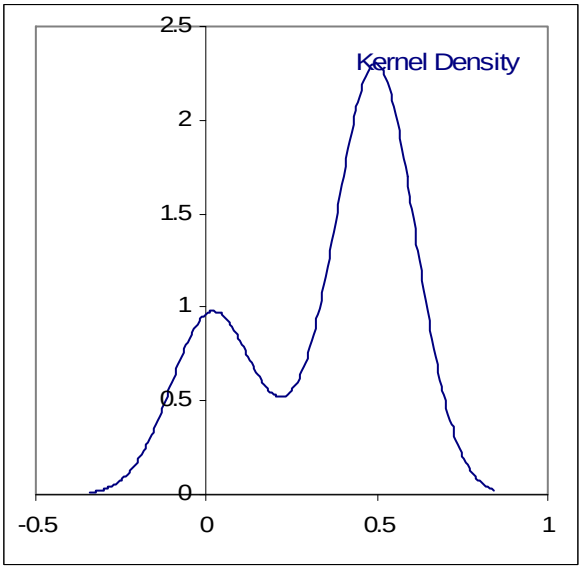

Kernel Density
group 1
group 2
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
0 20 40 60 80 100

Determination of Colour on samples #11104;

lab	method	value	mark	z(targ)	remarks
128	D1500	L0.5		-----	
176		-----		-----	
273	D1500	L0.5		-----	
331	D1500	<0.5		-----	
398	D1500	L0.5		-----	
445	D1500	<0.5		-----	
446	D1500	<0.5		-----	
497	D1500	<0.5		-----	
551	D1500	L0.5		-----	
614	INH-830	Straw		-----	
963	D1500	L0.5		-----	
1056		-----		-----	
1072	INH-21320	0.5		0.43	
1137		-----		-----	
1146		-----		-----	
1152	D1500	0.5		0.43	
1156	D1500	0.5		0.43	
1178	ISO2049	<0.5		-----	
1262	D1500	L0.5		-----	
1264	D1500	L0.5		-----	
1271	D6045	0.3		-0.13	
1303	D1500	L0.5		-----	
1304	INH-132	0.0		-0.97	
1306	D1500	0.5		0.43	
1326	D1500	<0.5		-----	
1352		-----		-----	
1361		-----		-----	
1367		-----		-----	
1375	ISO2049	0.0		-0.97	
1430		-----		-----	
1435	ISO2049	0.5		0.43	
1440	ISO2049	L0.5		-----	
1442	D1500	L0.5		-----	
1445		-----		-----	
1452	in house	<1		-----	
1453		-----		-----	
1458	D1500	0.5		0.43	
1461		-----		-----	
1463	D1500	0		-0.97	
1471		-----		-----	
1473	D1500	<0.5		-----	
1475		-----		-----	
1478	ISO2049	0.5		0.43	
1513	ISO2049	<0.5		-----	
1516	D1500	0		-0.97	
1529	ISO2049	<0.5		-----	
1626		-----		-----	
1630		-----		-----	
1660	D1500	0.1		-0.69	
1702	D1500	0.5		0.43	
1704	D1500	L0.5		-----	
1719	D1524	<0.5		-----	
1801	D1500	<0.5		-----	
1827		-----		-----	
1863	D1500	L0.5		-----	
1915	D1500	L0.5		-----	
1923	ISO2049	0.5		0.43	
1924	D6045	0.5		0.43	
1925	ISO2049	0.5		0.43	
2122	in house	pale straw		-----	
2125		-----		-----	

normality	not OK
n	17
outliers	0
mean (n)	0.35
st.dev. (n)	0.224
R(calc.)	0.63
R(D1500:08)	1.00

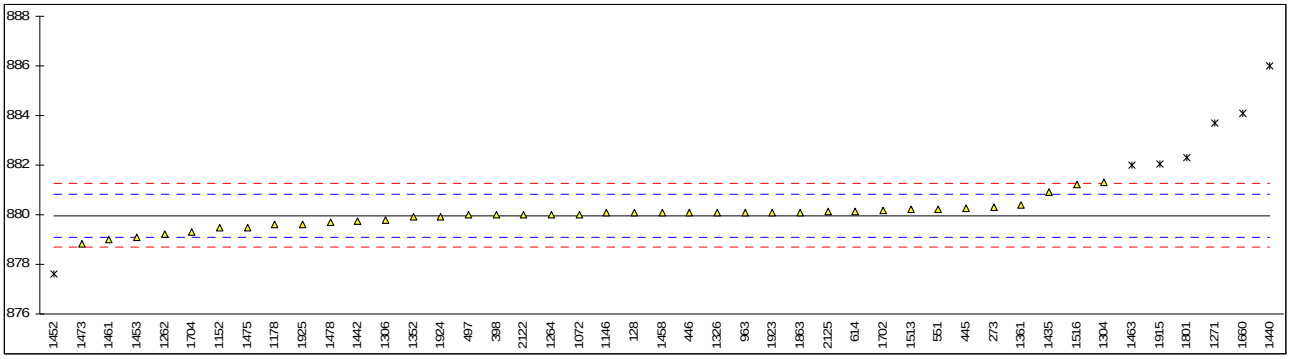

1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
1375
1304
1463
1516
1660
1271
1924
1925
1702
1923
1478
1156
1152
1072
1458
1435
1306

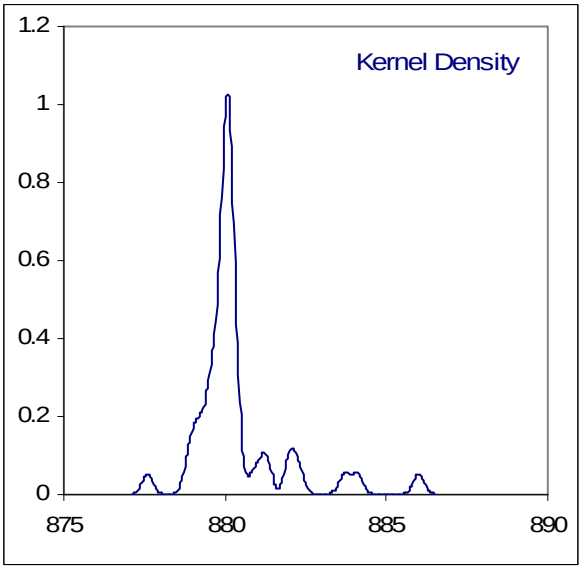

Kernel Density
2.5
2
1.5
1
0.5
0
-0.5
0
0.5
1

Determination of Density @20°C on sample #11104; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
128	ASTM D4052	880.1		0.32	
176		-----		-----	
273	ASTM D4052	880.3		0.78	
331		-----		-----	
398	ISO3675	880.0		0.08	
445	ASTM D4052	880.25		0.67	
446	ASTM D4052	880.1	C	0.32	First reported 0.8801
497	ASTM D4052	879.98		0.04	
551	ASTM D4052	880.2		0.55	
614	ISO3675	880.15		0.43	
963	ASTM D4052	880.1		0.32	
1056		-----		-----	
1072	ISO3675	880		0.08	
1137		-----		-----	
1146	ISO12185	880.09		0.29	
1152	ISO3675	879.5		-1.08	
1156		-----		-----	
1178	ISO3675	879.60	C	-0.85	First reported 0.87960
1262	ISO3675	879.2		-1.78	
1264	ASTM D4052	880.0		0.08	
1271	ASTM D4052	883.7	C,G(0.01)	8.72	First reported 878.5
1303		-----		-----	
1304	INH-102	881.3		3.12	
1306	ASTM D1298	879.8	C	-0.38	First reported 0.8739
1326	ASTM D4052	880.10		0.32	
1352	ASTM D7042	879.9		-0.15	
1361	ISO3675	880.4004		1.02	
1367		-----		-----	
1375		-----		-----	
1430		-----		-----	
1435	ASTM D4052	880.9	C	2.18	First reported 0.8809
1440	in house	886.0	C,G(0.01)	14.08	First reported 0.8860
1442	ASTM D7042	879.76		-0.48	
1445		-----		-----	
1452	DIN51757	877.6	G(0.05)	-5.52	
1453	ISO3675	879.1		-2.02	
1458	ASTM D4052	880.1		0.32	
1461	ISO3675	879.0	C	-2.25	First reported 8790,1
1463	ASTM D1298	882	DG(0.01)	4.75	
1471		-----		-----	
1473	ASTM D1298	878.84		-2.62	
1475	ASTM D1298	879.5		-1.08	
1478	ISO12185	879.7	C	-0.62	First reported 0.8797
1513	ISO12185	880.196		0.54	
1516	ISO3675	881.2		2.88	
1529		-----		-----	
1626		-----		-----	
1630		-----		-----	
1660	ISO3675	884.1	C,G(0.05)	9.65	First reported 0.8841
1702	ISO12185	880.155		0.45	
1704	ISO3675	879.303		-1.54	
1719		-----		-----	
1801	ISO3675	882.3	C,G(0.05)	5.45	First reported 0.8823
1827		-----		-----	
1863	ASTM D4052	880.1		0.32	
1915	ASTM D4052	882.05	DG(0.01)	4.87	
1923	ISO3675	880.1		0.32	
1924	ISO3675	879.91		-0.13	
1925	ISO3675	879.6		-0.85	
2122	ISO12185	880	C	0.08	First reported 0.880
2125	ISO12185	880.11		0.34	

normality	not OK
n	38
outliers	7
mean (n)	879.96
st.dev. (n)	0.513
R(calc.)	1.44
R(ISO3675:98)	1.20

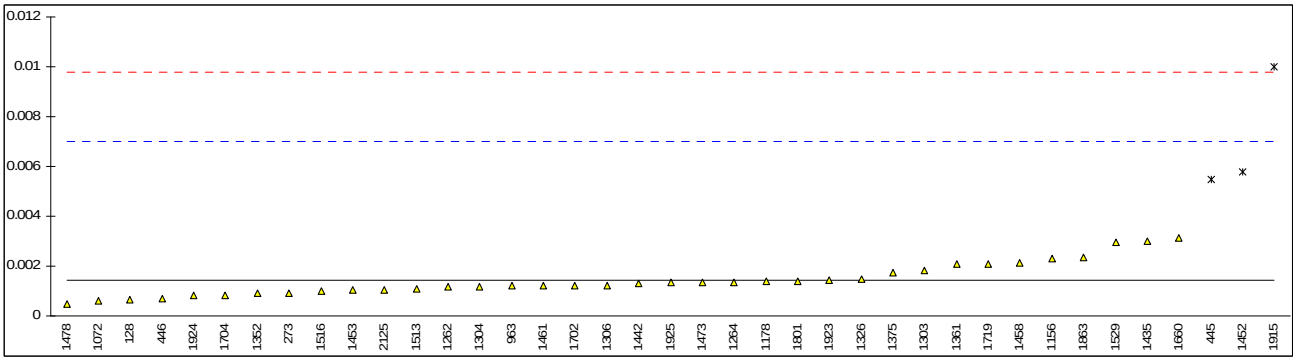

888
886
884
882
880
878
876
1452 1473 1461 1453 1262 1704 1152 1475 1178 1925 1478 1442 1306 1352 1924 497 398 2122 1264 1072 1146 128 1458 446 1326 963 1923 1863 2125 614 1702 1513 551 445 273 1361 1435 1516 1304 1463 1915 1801 1271 1660 1440

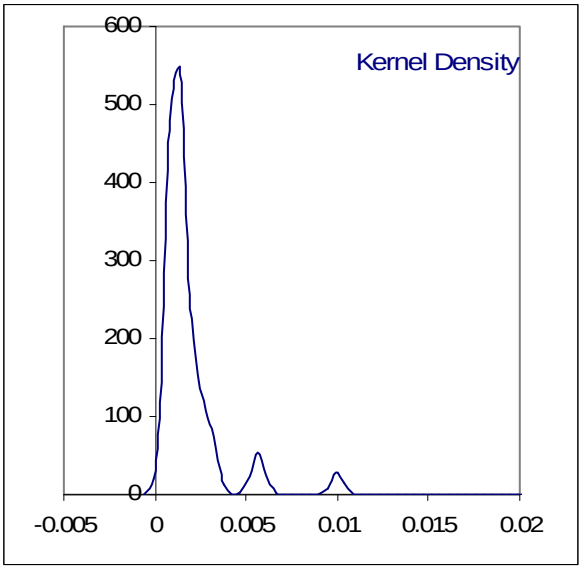

Kernel Density
1.2
1
0.8
0.6
0.4
0.2
0
875
880
885
890

Determination of Di-electric Dissipation Factor on sample #11104;

lab	method	value	mark	z(targ)	remarks
128	D924	0.00067		-0.28	
176		-----		-----	
273	IEC60247	0.00093		-0.18	
331		-----		-----	
398		-----		-----	
445	IEC60247	0.0055	G(0.01)	1.46	
446	IEC60247	0.0007		-0.27	
497		-----		-----	
551		-----		-----	
614		-----		-----	
963	ASTM D924	0.0012	U	-0.09	Reported 0.12, probably an unit error?
1056		-----		-----	
1072	EN60247	0.00060		-0.30	
1137		-----		-----	
1146		-----		-----	
1152		-----		-----	
1156	IEC60247	0.00231		0.31	
1178	IEC60247	0.0014		-0.01	
1262	IEC60247	0.00116		-0.10	
1264	IEC60247	0.00136		-0.03	
1271		-----		-----	
1303	IEC60247	0.00182		0.14	
1304	INH-125	0.00117		-0.10	
1306	IEC60247	0.00123		-0.08	
1326	IEC60247	0.00146		0.01	
1352	IEC60247	0.000893		-0.20	
1361	IEC60247	0.002082		0.23	
1367		-----		-----	
1375	IEC60247	0.00174		0.11	
1430		-----		-----	
1435	IEC60247	0.003		0.56	
1440		-----		-----	
1442	IEC60247	0.0013		-0.05	
1445		-----		-----	
1452	EN60247	0.0058	G(0.01)	1.56	
1453	IEC60247	0.00103		-0.15	
1458	IEC60247	0.00211		0.24	
1461	IEC60247	0.0012		-0.09	
1463	ASTM D924	<0.04		-----	
1471		-----		-----	
1473	IEC60247	0.001352		-0.03	
1475		-----		-----	
1478	IEC60247	0.000472		-0.35	
1513	IEC60247	0.00107		-0.13	
1516	IEC60247	0.001010		-0.15	
1529	IEC60247	0.00297		0.55	
1626		-----		-----	
1630		-----		-----	
1660	IEC60247	0.00312		0.60	
1702	IEC60247	0.001209		-0.08	
1704	IEC60247	0.000835		-0.22	
1719	IEC60247	0.00210		0.24	
1801	IEC60247	0.00140		-0.01	
1827		-----		-----	
1863	IEC60247	0.00233		0.32	
1915	ASTM D924	0.010	G(0.01)	3.07	
1923	IEC60247	0.00145		0.00	
1924	IEC60247	0.000817		-0.22	
1925	IEC60247	0.00134		-0.04	
2122		-----		-----	
2125	IEC60247	0.00104		-0.14	

normality	not OK
n	36
outliers	3
mean (n)	0.0014
st.dev. (n)	0.00067
R(calc.)	0.0019
R(IEC60247:04)	0.0078

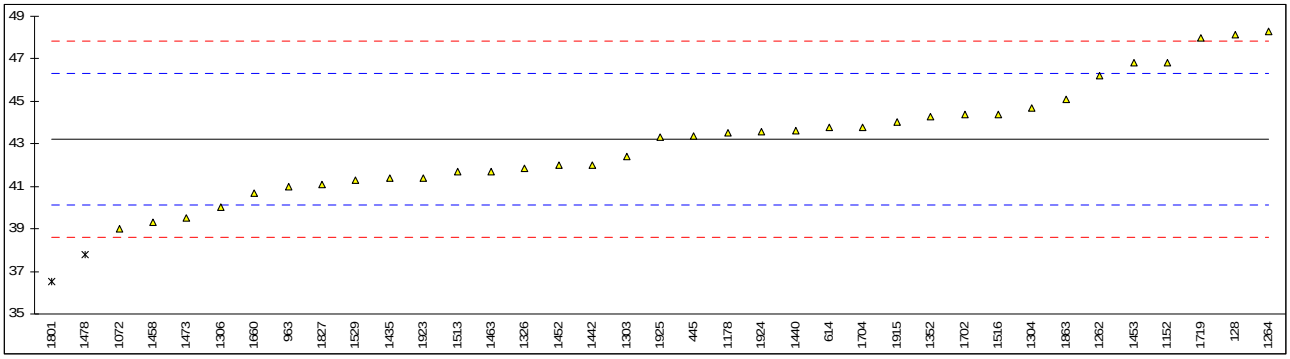

0.012
0.01
0.008
0.006
0.004
0.002
0
1478
1072
128
446
1924
1704
1352
273
1516
1453
2125
1513
1262
1304
963
1461
1702
1306
1442
1925
1473
1264
1178
1801
1923
1326
1375
1303
1361
1719
1458
1156
1863
1529
1435
1660
445
1452
1915

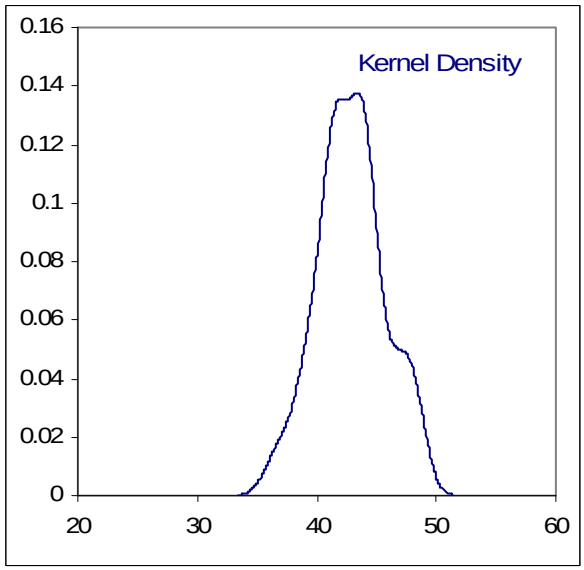

Kernel Density
600
500
400
300
200
100
0
-0.005
0
0.005
0.01
0.015
0.02

Determination of Interfacial Surface Tension on sample #11104; results in mN/m

lab	method	value	mark	z(targ)	remarks
128	ASTM D971	48.12		3.18	
176		-----		-----	
273		-----		-----	
331		-----		-----	
398		-----		-----	
445	ASTM D971	43.35		0.09	
446		-----		-----	
497		-----		-----	
551		-----		-----	
614	ISO6295	43.8		0.38	
963	ASTM D971	41		-1.43	
1056		-----		-----	
1072	INH-21320	39.0		-2.73	
1137		-----		-----	
1146		-----		-----	
1152	ASTM D971	46.84	C	2.35	First reported 51.33
1156		-----		-----	
1178	ASTM D971	43.5		0.19	
1262	ASTM D971	46.2		1.93	
1264	ASTM D971	48.3		3.30	
1271		-----		-----	
1303	ASTM D971	42.4		-0.53	
1304	INH-123	44.7		0.96	
1306	ISO6295	40		-2.08	
1326	ISO6295	41.85		-0.88	
1352	ASTM D971	44.3		0.70	
1361		-----		-----	
1367		-----		-----	
1375		-----		-----	
1430		-----		-----	
1435	ASTM D971	41.40		-1.18	
1440	ISO6295	43.6		0.25	
1442	EN14210	42.0		-0.79	
1445		-----		-----	
1452	ASTM D971	42		-0.79	
1453	ISO6295	46.8		2.32	
1458	ASTM D971	39.3		-2.54	
1461		-----		-----	
1463	ASTM D971	41.7		-0.98	
1471		-----		-----	
1473	ISO6295	39.5		-2.41	
1475		-----		-----	
1478	ASTM D971	37.8	G(0.05)	-3.51	
1513	ASTM D971	41.7		-0.98	
1516	ASTM D971	44.4		0.77	
1529	ASTM D971	41.3		-1.24	
1626		-----		-----	
1630		-----		-----	
1660	ISO6295	40.7		-1.63	
1702	ASTM D971	44.397		0.77	
1704	ISO6295	43.8		0.38	
1719	ASTM D2285	48		3.10	
1801	ISO6295	36.5	G(0.05)	-4.35	
1827	ASTM D971	41.09		-1.38	
1863	ASTM D971	45.1		1.22	
1915	ASTM D971	44.044		0.54	
1923	ASTM D971	41.4		-1.18	
1924	ASTM D971	43.589		0.24	
1925	ASTM D971	43.3		0.06	
2122		-----		-----	
2125		-----		-----	

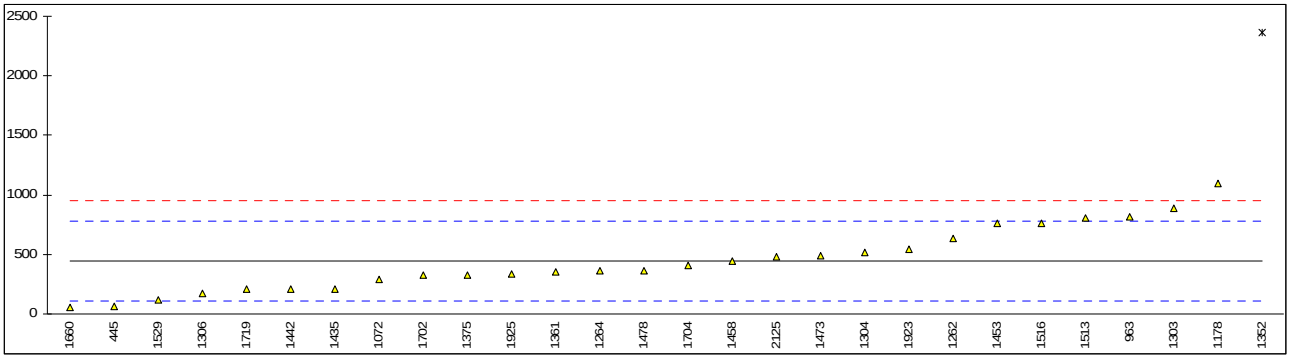
normality	OK
n	35
outliers	2
mean (n)	43.21
st.dev. (n)	2.500
R(calc.)	7.00
R(ISO6295:83)	4.32
R(ASTM D971)	4.32

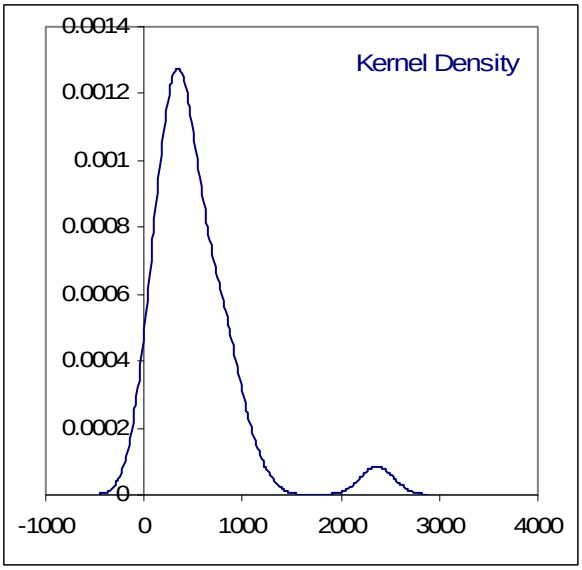

49
47
45
43
41
39
37
35
1801
1478
1072
1458
1473
1306
1660
963
1827
1529
1435
1923
1513
1463
1326
1452
1442
1303
1925
445
1178
1924
1440
614
1704
1915
1352
1702
1516
1304
1863
1262
1453
1152
1719
128
1264


Kernel Density
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
20
30
40
50
60

Determination of Specific Resistance on sample #11104; results in GΩm

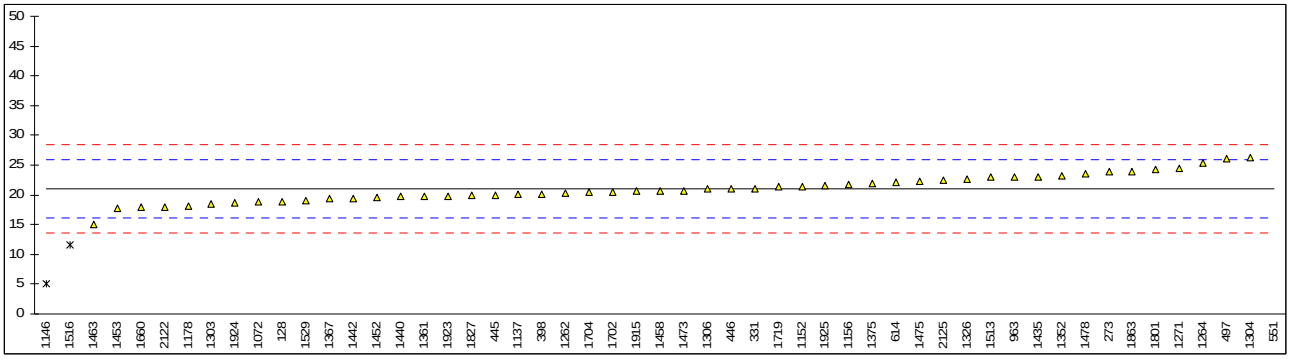
lab	method	value	mark	z(targ)	remarks
128		-----		-----	
176		-----		-----	
273		-----		-----	
331		-----		-----	
398		-----		-----	
445	IEC60247	63.55		-2.29	
446		-----		-----	
497		-----		-----	
551		-----		-----	
614		-----		-----	
963	IEC60247	813.06		2.20	
1056		-----		-----	
1072	EN60247	289		-0.94	
1137		-----		-----	
1146		-----		-----	
1152		-----		-----	
1156		-----		-----	
1178	IEC60247	1098.0		3.91	
1262	IEC60247	630.1		1.11	
1264	IEC60247	359.3		-0.52	
1271		-----		-----	
1303	IEC60247	889.1		2.66	
1304	INH-125	518.05		0.43	
1306	IEC60247	168.2		-1.66	
1326		-----		-----	
1352	IEC60247	2366.67	G(0.01)	11.50	
1361	IEC60247	353.73		-0.55	
1367		-----		-----	
1375	IEC60247	330.5		-0.69	
1430		-----		-----	
1435	IEC60247	208.3		-1.42	
1440		-----		-----	
1442	IEC60247	206.2		-1.43	
1445		-----		-----	
1452		-----		-----	
1453	IEC60247	756.4		1.86	
1458	IEC60247	446.0		0.00	
1461		-----		-----	
1463		-----		-----	
1471		-----		-----	
1473	IEC60247	493		0.28	
1475		-----		-----	
1478	IEC60247	362.23		-0.50	
1513	IEC60247	809.8	C	2.18	First reported 888.8
1516	IEC60247	761.4		1.89	
1529	IEC60247	121		-1.94	
1626		-----		-----	
1630		-----		-----	
1660	IEC60247	58.0		-2.32	
1702	IEC60247	322.0		-0.74	
1704	IEC60247	404		-0.25	
1719	IEC60247	205.7		-1.44	
1801		-----		-----	
1827		-----		-----	
1863		-----		-----	
1915		-----		-----	
1923	IEC60247	546.1		0.60	
1924		-----		-----	
1925	IEC60247	332.0		-0.68	
2122		-----		-----	
2125	IEC60247	482.8		0.22	
	normality	OK			
	n	27			
	outliers	1			
	mean (n)	445.46			
	st.dev. (n)	268.868			
	R(calc.)	752.83			
	R(IEC60247:04)	467.74			

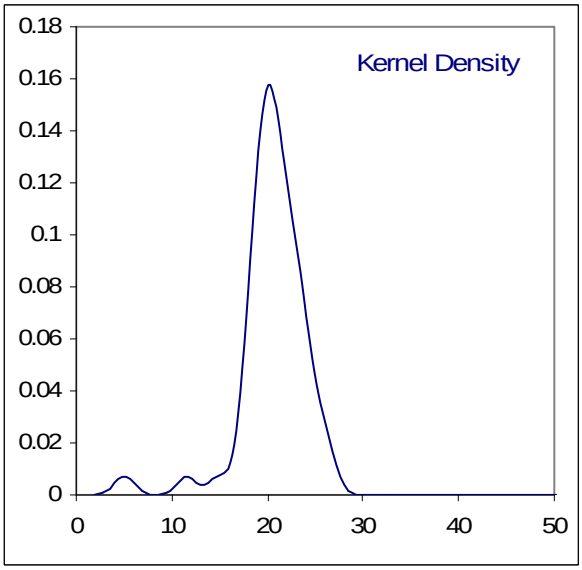

2500
2000
1500
1000
500
0
1660
445
1529
1306
1719
1442
1435
1072
1702
1375
1925
1361
1264
1478
1704
1458
2125
1473
1304
1923
1262
1453
1516
1513
963
1303
1178
1352


Kernel Density
0.0014
0.0012
0.001
0.0008
0.0006
0.0004
0.0002
0
-1000
0
1000
2000
3000
4000

Determination of Water on sample #11104; results in mg/kg

lab	method	value	mark	z(targ)	remarks
128	ASTM D1533	18.9		-0.87	
176		-----		-----	
273	IEC60814	23.9		1.17	
331	ASTM D6304	21		-0.01	
398	IEC60814	20.1		-0.38	
445	IEC60814	19.9		-0.46	
446	IEC60814	21.0		-0.01	
497	ASTM D6304	26		2.02	
551	ASTM D203	210	G(0.01)	76.91	
614	IEC60814	22.1		0.43	
963	ASTM D1533	23		0.80	
1056		-----		-----	
1072	EN60814	18.8		-0.91	
1137	ISO10337	20.043		-0.40	
1146	ASTM D6304	5	G(0.01)	-6.53	
1152	IEC60814	21.38		0.14	
1156	IEC60814	21.8		0.31	
1178	IEC60814	18.1		-1.19	
1262	EN60814	20.3		-0.30	
1264	ASTM D1533	25.4		1.78	
1271	ISO12937	24.4		1.37	
1303	IEC60814	18.5		-1.03	
1304	INH-121	26.35		2.16	
1306	IEC60814	21		-0.01	
1326	ASTM D1533	22.6		0.64	
1352	IEC60814	23.1		0.84	
1361	EN60814	19.7		-0.54	
1367	IEC60814	19.33		-0.69	
1375	IEC60814	22		0.39	
1430		-----		-----	
1435	IEC60814	23		0.80	
1440	IEC60814	19.7		-0.54	
1442	IEC60814	19.4		-0.66	
1445		-----		-----	
1452	EN60814	19.5		-0.62	
1453	IEC60814	17.8		-1.32	
1458	IEC60814	20.6		-0.18	
1461		-----		-----	
1463	ASTM D1533	15		-2.46	
1471		-----		-----	
1473	IEC60814	20.7		-0.14	
1475	ASTM D6304	22.2		0.47	
1478	IEC60814	23.6		1.04	
1513	IEC60814	23.0		0.80	
1516	IEC60814	11.6	G(0.01)	-3.84	
1529	IEC60814	19		-0.83	
1626		-----		-----	
1630		-----		-----	
1660	IEC60814	18		-1.23	
1702	IEC60814	20.51		-0.21	
1704	IEC60814	20.476		-0.23	
1719	IEC60814	21.3		0.11	
1801	IEC60814	24.3		1.33	
1827	ASTM D6304	19.85		-0.48	
1863	ASTM D6304	24		1.21	
1915	ASTM D1533	20.6		-0.18	
1923	IEC60814	19.75		-0.52	
1924	IEC60814	18.72		-0.94	
1925	IEC60814	21.5		0.19	
2122	IEC60814	18		-1.23	
2125	IEC60814	22.46		0.58	
	normality	OK			
	n	50			
	outliers	3			
	mean (n)	21.03			
	st.dev. (n)	2.308			
	R(calc.)	6.46			
	R(EN60814:98)	6.88			


50
45
40
35
30
25
20
15
10
5
0
1146
1516
1463
1453
1660
2122
1178
1303
1924
1072
128
1529
1367
1442
1452
1440
1361
1923
1827
445
1137
398
1282
1704
1702
1915
1458
1473
1306
446
331
1719
1152
1925
1156
1375
614
1475
2125
1326
1513
963
1435
1352
1478
273
1863
1801
1271
1264
497
1304
551

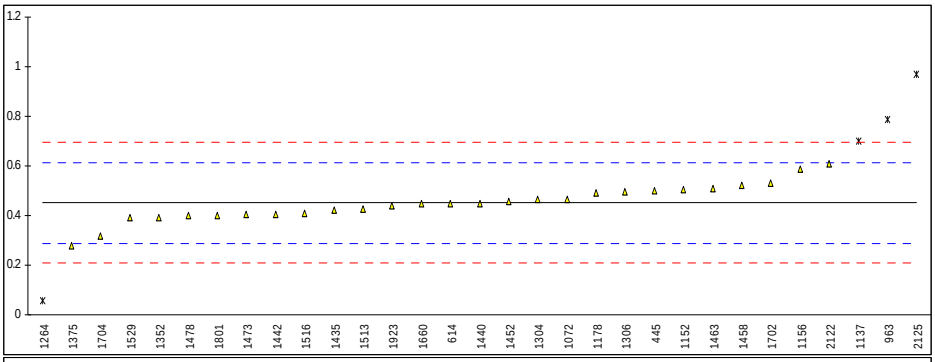

Kernel Density
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0
10
20
30
40
50

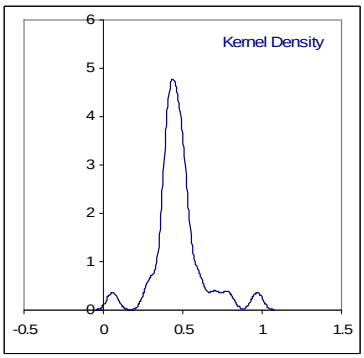
Determination of 2-Furfural on sample #11105; results in mg/kg

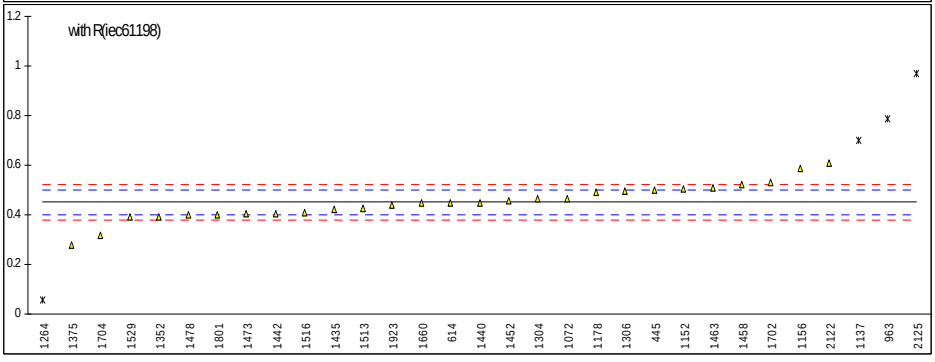
lab	method	value	mark	z(targ)	z(IEC61198)	remarks
445	IEC61198	0.50		0.61	2.06	
497		-----		-----	-----	
614	IEC61198	0.45		0.00	-0.01	
963	D5837	0.785	G(0.05)	4.12	13.88	
1072	EN61198	0.467		0.21	0.69	
1137	D5837	0.699526	G(0.05)	3.07	10.33	
1152	IEC61198	0.5052		0.68	2.28	
1156	IEC61198	0.586		1.67	5.63	
1178	IEC61198	0.490		0.49	1.65	
1264	D5837	0.0571	G(0.05)	-4.84	-16.30	
1304	INH-126	0.4631		0.16	0.53	
1306	IEC61198	0.4941		0.54	1.82	
1352	IEC61198	0.391		-0.73	-2.46	
1367		-----		-----	-----	
1375	IEC61198	0.28		-2.10	-7.06	
1430		-----		-----	-----	
1435	IEC61198	0.42		-0.37	-1.25	
1440	IEC61198	0.45		0.00	-0.01	
1442	IEC61198	0.403		-0.58	-1.96	
1445		-----		-----	-----	
1452	EN61198	0.457		0.08	0.28	
1458	IEC61198	0.520		0.86	2.89	
1463	ASTM583	0.508		0.71	2.39	
1473	IEC61198	0.4029		-0.58	-1.96	
1478	IEC61198	0.399		-0.63	-2.12	
1513	IEC61198	0.4268		-0.29	-0.97	
1516	IEC61198	0.410		-0.50	-1.67	
1529	IEC61198	0.39		-0.74	-2.50	
1660	IEC61198	0.449		-0.02	-0.05	
1702	IEC61198	0.528542		0.96	3.25	
1704	IEC61198	0.316		-1.65	-5.57	
1801	IEC61198	0.40		-0.62	-2.08	
1923	IEC61198	0.44	C	-0.13	-0.42	First reported 0.362
2122	IEC61198	0.61		1.97	6.62	
2125	IEC61198	0.97	G(0.05)	6.40	21.55	

normality	OK
n	27
outliers	4
mean (n)	0.450
st.dev. (n)	0.0720
R(calc.)	0.202
R(Horwitz)	0.228

Compare R(IEC1198) = 0.068







Determination of other Furanic compounds on sample #11105; results in mg/kg

lab	method	2-af	mark	z(targ)	2-fa	mark	z(targ)	5-hm-2-f	mark	z(targ)	5-m-2-f	mark	z(targ)
445	IEC61198	0.01		-----	0.01		-----	<0.05		-----	0.01		-----
497		-----		-----	-----		-----	-----		-----	-----		-----
614	IEC61198	<0.01		-----	<0.01		-----	0.02		-----	<0.01		-----
963	D5837	n.d.		-----	n.d.		-----	n.d.		-----	n.d.		-----
1072	EN61198	0.178		-----	<0.01		-----	<0.01		-----	0.015		-----
1137		-----		-----	-----		-----	-----		-----	-----		-----
1152	IEC61198	0.2435	G(0.01)	-----	0.0135		-----	0.0128		-----	0.0100		-----
1156	IEC61198	0.00		-----	0.00		-----	0.00		-----	0.00		-----
1178		-----		-----	-----		-----	-----		-----	-----		-----
1264	D5837	0.0063		-----	0.0243		-----	0.0292		-----	0.0052		-----
1304	INH-126	<0.01		-----	<0.01		-----	0.0132		-----	0.0401		-----
1306	IEC61198	<0.02		-----	<0.02		-----	<0.02		-----	<0.02		-----
1352	IEC61198	<0.01		-----	<0.01		-----	<0.01		-----	<0.01		-----
1367		-----		-----	-----		-----	-----		-----	-----		-----
1375	IEC61198	<0.03		-----	<0.03		-----	<0.03		-----	<0.03		-----
1430		-----		-----	-----		-----	-----		-----	-----		-----
1435	IEC61198	<0.03		-----	<0.03		-----	<0.03		-----	0.04		-----
1440	IEC61198	<0.01	C	-----	<0.01		-----	<0.01		-----	<0.01		-----
1442	IEC61198	<0.01		-----	<0.01		-----	<0.01		-----	<0.01		-----
1445		-----		-----	-----		-----	-----		-----	-----		-----
1452	EN61198	<0.05	C	-----	<0.05		-----	<0.05		-----	<0.05		-----
1458	IEC61198	<0.01		-----	<0.01		-----	<0.01		-----	<0.01		-----
1463		-----		-----	-----		-----	-----		-----	-----		-----
1473	IEC61198	0.0139	G(0.01)	-----	-----		-----	<0.03		-----	<0.03		-----
1478	IEC61198	<0.01		-----	<0.01		-----	<0.01		-----	<0.01		-----
1513	IEC61198	<0.05		-----	<0.05		-----	<0.05		-----	<0.05		-----
1516	IEC61198	<0.05		-----	<0.05		-----	<0.05		-----	<0.05		-----
1529	IEC61198	<0.1		-----	<0.1		-----	<0.1		-----	<0.1		-----
1660	IEC61198	0.007		-----	0.034		-----	0.018		-----	0.044		-----
1702	IEC61198	n.d.		-----	n.d.		-----	n.d.		-----	n.d.		-----
1704		-----		-----	-----		-----	-----		-----	-----		-----
1801	IEC61198	<0.01		-----	<0.1		-----	<0.01		-----	<0.01		-----
1923	IEC61198	0.09	G(0.01)	-----	0.67	G(0.01)	-----	0.02		-----	0.01		-----
2122		-----		-----	-----		-----	-----		-----	-----		-----
2125	IEC61198	<0.01		-----	<0.01		-----	<0.01		-----	<0.01		-----
	normality	n.a.			n.a.			n.a.			n.a.		
	n	5			5			7			9		
	nutliers	3			1			0			0		
	mean (n)	0.0051			0.0131			0.0090			0.0170		
	st.dev. (n)	n.a.			n.a.			n.a.			n.a.		
	R(calc.)	n.a.			n.a.			n.a.			n.a.		
	R(Horwitz)	n.a.			n.a.			n.a.			n.a.		

Abbreviations:

2-af	= 2-acetylfuran
2-fa	= 2-furfurylalcohol
5-hm-2-f	= 5-hydroxymethyl-2-furfural
5-m-2-f	= 5-methyl-2-furfural

APPENDIX 2

Number of participants per country

5 laboratories in	AUSTRALIA
2 laboratories in	BELGIUM
1 laboratory in	BOSNIA and HERZEGOVINA
1 laboratory in	BRAZIL
7 laboratories in	BULGARIA
1 laboratory in	CANADA
1 laboratory in	CROATIA
3 laboratories in	FRANCE
3 laboratories in	GERMANY
2 laboratories in	INDIA
2 laboratories in	ITALY
1 laboratory in	KINGDOM OF BAHRAIN
1 laboratory in	KOREA
3 laboratories in	MALAYSIA
1 laboratory in	MEXICO
1 laboratory in	NEW ZEALAND
1 laboratory in	NORWAY
1 laboratory in	P.R. of CHINA
2 laboratories in	PORTUGAL
2 laboratories in	SAUDI ARABIA
1 laboratory in	SLOVENIA
2 laboratories in	SOUTH AFRICA
5 laboratories in	SPAIN
1 laboratory in	SWEDEN
2 laboratories in	THE NETHERLANDS
1 laboratory in	TURKEY
1 laboratory in	U.A.E.
2 laboratories in	U.S.A.
4 laboratories in	UNITED KINGDOM
1 laboratory in	VIETNAM

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
ex	= excluded from calculations
n.a.	= not applicable
W	= withdrawn on request participant
U	= reported in a deviating unit
E	= error in calculations
SDS	= Safety Data Sheet
fr.	= first reported

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
2 prNEN 12766-2:2001
3 ASTM E178-02
4 ASTM E1301-03
5 ISO 5725-86
6 ISO 5725, parts 1-6, 1994
7 ISO13528-05
8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
10 IP 367/96
11 DIN 38402 T41/42
12 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
13 J.N. Miller, Analyst, 118, 455, (1993)
14 Analytical Methods Committee Technical Brief, No4 January 2001
15 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)