

**Results of Proficiency Test
Transformer Oil (used)
November 2016**

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2014, the Institute for Interlaboratory Studies organizes (iis) a proficiency test (PT) for Used Transformer Oil in combination with the PT on the analysis of Furanics in used Transformer Oil in accordance with the latest applicable version of the specification IEC60296 and of ASTM D3487. The PT on Furanics has been organized by iis since 2001 as a part of the PT on Fresh Transformer Oil. During the annual proficiency testing program 2016/2017, it was decided to continue the round robin for used Transformer Oil in combination with the PT on Furanics.

In this interlaboratory study 76 laboratories from 35 different countries for the PT on used Transformer Oil did register for participation and 48 laboratories from 29 different countries for the PT on Furanics. See appendix 2 for the number of participants per PT and per country. In this report, the results of the 2016 interlaboratory study on used Transformer Oil and Furanics are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. In this proficiency test the participants received, depending on the registration, 1x1 litre bottle of used Transformer Oil (labelled #16251) and/or 1x100 ml bottle (labelled #16252) for Furanics in Transformer Oil. Participants were requested to report rounded and unrounded test results. The unrounded test 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 website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary bulk material for the used Transformer Oil sample #16251 was obtained from an European supplier. The approximately 82 litres bulk material was homogenised in a pre-cleaned drum. After homogenisation 90 amber glass bottles of 1 litre were filled with 900 ml used Transformer Oil and labelled #16251. The homogeneity of the sub samples #16251 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 #16251-1	869.67
Sample #16251-2	869.68
Sample #16251-3	869.68
Sample #16251-4	869.68
Sample #16251-5	869.69
Sample #16251-6	869.67
Sample #16251-7	869.67
Sample #16251-8	869.68

Table 1: homogeneity test results of sub samples #16251

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

	Density at 20°C in kg/m ³
r (observed)	0.02
reference test method	ISO3675:98
0.3 x R (ref. test method)	0.36

Table 2: evaluation of the repeatability of sub samples #16251

The necessary bulk material for the PT on Furanics was prepared by mixing 5 grams of used Transformer Oil, highly positive on Furanics obtained from a third party, with approx. 6 kg of another batch fresh Transformer Oil obtained from a local supplier. After homogenisation in a pre-cleaned vessel 68 amber glass bottles of 100 mL were filled and labelled #16252. The homogeneity of the sub samples #16252 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 #16252-1	876.36
Sample #16252-2	876.36
Sample #16252-3	876.36
Sample #16252-4	876.36
Sample #16252-5	876.35
Sample #16252-6	876.35
Sample #16252-7	876.36
Sample #16252-8	876.36

Table 3: homogeneity test results of sub samples #16252

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

	Density at 20°C in kg/m ³
r (observed)	0.01
reference test method	ISO3675:98
0.3 x R (ref. test method)	0.36

Table 4: evaluation of the repeatability of sub samples #16252

Each of the calculated repeatabilities was less than 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the sub samples of #16251 and #16252 was assumed.

To each of the participating laboratories, depending on the registration, 1 x 1 litre bottle (labelled #16251) and/or 1 x 100mL bottle (labelled #16252) was sent on November 2, 2016.

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 #16251; Total Acidity (Potentiometric and Colorimetric), Breakdown Voltage, Density at 20°C, Di-electric Dissipation Factor (DDF) at 90°C and Specific Resistance at 90°C, Flash Point (Cleveland Open Cup and Pensky-Martens Closed Cup), Interfacial Surface Tension, Kinematic Viscosity at 40°C and Water.

The participants were requested to determine on sample #16252; Total Furanic Compounds, 2-Acetyl Furan, 2-Furfural, 2-Furfuryl alcohol, 5-Hydroxy Methyl-2-Furfural and 5-Methyl-2-Furfural.

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

To get comparable test results a detailed report form, on which the units were prescribed as well as the required reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The laboratories were also requested to confirm the sample receipt on the same data entry portal. A SDS was added to the sample.

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

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

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

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. 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 one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.

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

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

3.3 Z-SCORES

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

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

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

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare.

The usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test some problems were encountered during the dispatch of the samples. Participants in Saudi Arabia and South of Africa received the samples late or not at all due to problems with clearance of the samples at customs. For the used Transformer Oil PT three participants reported test results after the final reporting date and four participants did not report any test results at all. For the Furanics in Transformer Oil PT one participant reported the test results after the final reporting date and four participants did not report any test results at all. Not all laboratories were able to report all analyses requested.

In total eighty different laboratories did register for participation for one or two PTs. Seventy-two participants reported numerical test results in the PT on used Transformer Oil and forty-four participants in the PT on Furanics. In total seventy-six participants reported 660 numerical test results on used Transformer Oil and Furanics PTs combined. Observed were 31 outlying test results, which is 4.7% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER SAMPLE AND 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 test methods are also in the tables together with the reported data. The abbreviations, used in these tables, are listed in appendix 3.

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.

For the Furanics the observed reproducibilities were compared against the (strict) reproducibilities estimated from the Horwitz equation. It is remarkable that the precision requirements of IEC 61198:93 are more strict than the requirements estimated from the Horwitz equation and the ASTM test methods.

Sample #16251

Acidity, total (Potentiometric titration): This determination was problematic. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of D664-A:11ae1 or EN62021-1:03. Initially test method EN62021-1 was selected as reference test method but it appeared that the reproducibility calculated over only ASTM D664 test results is much larger than calculated over only EN/IEC62021-1 test results. Therefore the precision of ASTM D664 is used to calculate the z-scores.

Acidity, total (Colorimetric titration): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of D974:14e2. It appears that the requirements of test method EN62021-2:07 are very strict.

Breakdown Voltage: This determination may not be problematic. No statistical outliers were observed. The calculated reproducibility is almost in agreement with the requirements of EN60156:95. The results may be divided in two groups (see the Kernel Density plot). Possible causes for this bimodal distribution may be one or more of the following: not all laboratories may have performed the test on the sample as received (drying or degassing is not allowed) and/or some of the electrodes were not properly maintained. The reproducibility of EN60156:95 is determined from Figure 3 in EN60156:95. The black line in this figure 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. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ISO3675:98.

DD-Factor: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of EN60247:04.

Spec. Resistance: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the outlier is in good agreement with the requirements of EN60247:04.

Flash Point COC: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D92:16a.

Flash Point PMcc: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO2719-B:16, but not with the requirements of ISO2719-A:16 or ASTM D93-A:16a or ASTM D93-B:16a.

Interf. Surf. Tension: This determination was very 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 D971:12. One should be aware that ISO6295 is obsolete since February 2005.

Kinematic Viscosity: Regretfully a reproducibility for used oils is not present in ASTM D445:15a (see §17 of ASTM D445). Therefore, the target reproducibility is calculated from the reproducibilities found in iis PT's on used oils (see app. 3, ref. 17). 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 average reproducibility found for used oils in previous iis PTs (to 2015).

Water: This determination may be problematic for a number of laboratories. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is almost in agreement with the requirements of EN60814:98, but in agreement with the requirements of ASTM D1533:12.

Sample #16252

Total Furanics: This determination may not be problematic. No statistical outliers were observed but five test results were excluded because of an outlier in one of the Furanic components. However, the calculated reproducibility after rejection of the suspect data is in agreement with the estimated requirements calculated using the Horwitz equation based on 2 components and the estimated requirements of ASTM D5837:15, but not with the strict requirements of IEC61198:93.

2-Furfural: This determination may be not problematic. One statistical outlier was observed and another test result was excluded, because this test result was probably a false negative test result. The calculated reproducibility after rejection of the suspect data is almost in agreement with the estimated requirements from the Horwitz equation and the estimated requirements of ASTM D5837:15, but not at all with the strict requirements of IEC61198:93.

2-Furfuryl alcohol: This determination may be not problematic. Six statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated requirements from the Horwitz equation and the estimated requirements of ASTM D5837:15, but not at all with the strict requirements of IEC61198:93.

5-Methyl-2-Furfural: This determination may be problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated requirements calculated using the Horwitz equation and the estimated requirements of ASTM D5837:15, but not at all with the strict requirements of IEC61198:93.

Other Furanics : The concentrations of 2-Acetyl Furan and 5-Hydroxymethyl-2-Furfural may be near or below the detection limit. Therefore no significant conclusions were drawn for these two components.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity, total (Potentiometric)	g KOH/kg	39	0.023	0.013	0.010
Acidity, total (Colorimetric)	g KOH/kg	28	0.019	0.015	0.040
Breakdown Voltage	kV/2.5 mm	68	17.5	21.1	19.1
Density at 20°C	kg/m ³	44	869.8	1.2	1.2
Di-electric Dissipation Factor at 90°C		52	0.0101	0.0050	0.0114
Specific Resistance at 90°C	GΩm	40	29.3	17.9	30.8
Flash Point COC	°C	19	160.7	17.3	18.0
Flash Point PMcc	°C	36	149.5	14.7	16.0
Interfacial Surface Tension	mN/m	44	26.9	5.8	2.7
Kinematic Viscosity at 40°C	mm ² /s	36	11.56	0.18	0.21
Water	mg/kg	61	36.7	10.1	9.1

Table 5: reproducibilities of tests on sample #16251

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Furanics	mg/kg	17	0.37	0.11	0.27
2-Furfural	mg/kg	35	0.02	0.02	0.02
2-Furfuryl alcohol	mg/kg	34	0.15	0.09	0.09
5-Methyl-2-Furfural	mg/kg	37	0.20	0.07	0.12
2-Acetyl Furan	mg/kg	37	<0.05	n.a.	n.a.
5-Hydroxy Methyl-2-Furfural	mg/kg	37	<0.05	n.a.	n.a.

Table 6: reproducibilities of tests on sample #16252

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 test methods 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 2016 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2016	November 2015	November 2014
Number of reporting labs	76	68	63
Number of test results reported	660	561	508
Statistical outliers	31	25	23
Percentage outliers	4.7%	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 requirements of the respective reference test methods. The conclusions are given the following table:

Parameter	November 2016	November 2015	November 2014	November 2013	October 2012
Acidity, total (Potentiometric)	-	-	--	n.e.	n.e.
Acidity, total (Colorimetric)	++	++	n.e.	n.e.	n.e.
Breakdown Voltage	+/-	++	+	n.e.	n.e.
Density at 20°C	+/-	+	-	n.e.	n.e.
Di-electric Dissipation Factor	++	++	++	n.e.	n.e.
Specific Resistance	++	++	++	n.e.	n.e.
Flash Point COC	+/-	n.e.	n.e.	n.e.	n.e.
Flash Point PMcc	+	+	+/-	n.e.	n.e.
Interfacial Surface Tension	--	--	--	n.e.	n.e.
Kinematic Viscosity at 40°C	+	-	--	n.e.	n.e.
Water	+/-	+/-	-	-	-
Total Furanics	++	+	n.e.	n.e.	n.e.
2-Furfural	+/-	-	+	+/-	--
2-Furfurylalcohol	+/-	-	+	+	--
5-Methyl-2-Furfural	+	+	++	+/-	++
2-Acetyl Furan	n.e.	n.e.	n.e.	n.e.	n.e.
5-Hydroxy Methyl-2-Furfural	n.e.	n.e.	n.e.	n.e.	n.e.

Table 8: comparison determinations against the reference test method

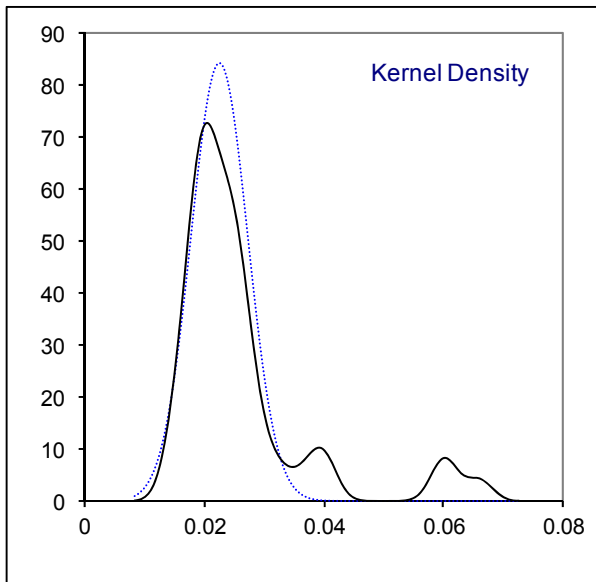
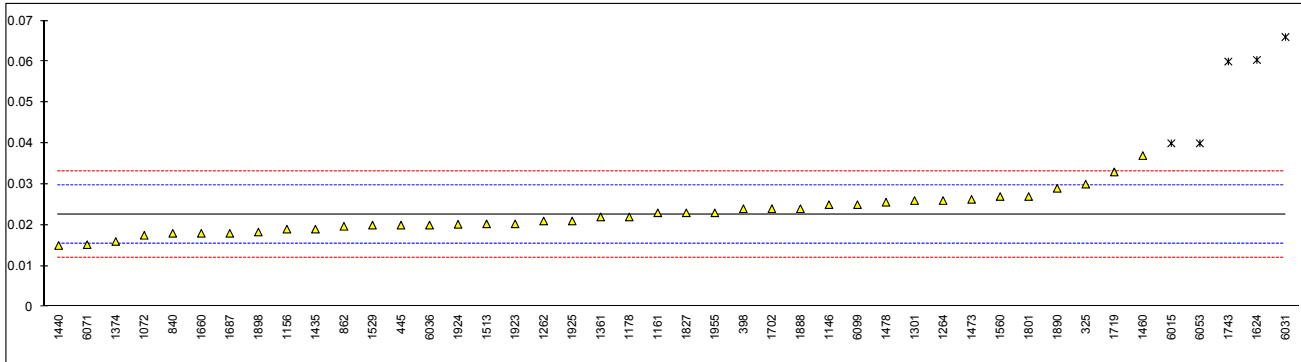
The performance of the determinations against the requirements of the respective reference test methods is listed in the above table. The following performance categories were used:

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

APPENDIX 1: Determination of Acidity, Total (Pot. Titration) on sample #16251; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks
179	D664-A	<0.01		<-3.54	Possibly a false negative test result
237	D664-A	<0.1		----	
325	D664-A	0.03		2.11	
331	D664-Amod.	<0.05		----	
398	EN62021-1	0.024		0.42	
445	D664-A	0.02		-0.71	
511		----		----	
593		----		----	
614		----		----	
840	IEC62021-1	0.018		-1.28	
862	IEC62021-1	0.0197		-0.80	
912		----		----	
962		----		----	
963		----		----	
974		----		----	
1072	EN62021-1	0.0175		-1.42	
1135		----		----	
1146	D664-A	0.025		0.70	
1156	IEC62021-1	0.019		-1.00	
1161	D664-A	0.023		0.13	
1178	IEC62021-1	0.022		-0.15	
1262	EN62021-1	0.021		-0.43	
1264	D664-A	0.026		0.98	
1301	EN62021-1	0.026		0.98	
1304		----		----	
1306		----		----	
1352		----		----	
1361	EN62021-1	0.022		-0.15	
1367		----		----	
1374	IEC62021-1	0.016		-1.84	
1430		----		----	
1435	EN62021-1	0.019		-1.00	
1440	EN62021-1	0.015		-2.13	
1442		----		----	
1444		----		----	
1458		----		----	
1460	D664-A	0.037		4.09	
1461		----		----	
1473	IEC62021-1	0.0263		1.07	
1478	IEC62021-1	0.0256		0.87	
1505		----		----	
1513	IEC62021-1	0.0203		-0.63	
1516		----		----	
1529	IEC62021-1	0.02		-0.71	
1545		----		----	
1560	IEC62021-1	0.027		1.26	
1595		----		----	
1624	IEC62021-1	0.0604	R(0.01)	10.70	
1660	IEC62021-1	0.018		-1.28	
1687	D664-A	0.018		-1.28	
1702	EN62021-1	0.024		0.42	
1719	D664-A	0.033		2.96	
1743	IEC62021-1	0.06	R(0.01)	10.59	
1801	EN62021-1	0.027		1.26	
1827	D664-A	0.023		0.13	
1888	IEC62021-1	0.024		0.42	
1890	ISO6619	0.029		1.83	
1891		----		----	
1897		----		----	
1898	EN62021-1	0.0183		-1.19	
1923	EN62021-1	0.0203		-0.63	
1924	EN62021-1	0.0202		-0.66	
1925	EN62021-1	0.021		-0.43	
1947		----		----	
1955	D664-A	0.023		0.13	
2122		----		----	
2237		----		----	
6015	D664-A	0.04	R(0.05)	4.94	
6029		----		----	
6030		----		----	
6031	IEC62021-1	0.066	C,R(0.01)	12.28	first reported: 0.000066 g KOH/g
6036	EN62021-1	0.02		-0.71	
6053	IEC62021-1	0.04	C,R(0.05)	4.94	first reported: 0.05
6071	IEC62021-1	0.0152	C	-2.07	first reported: 0.0092
6077		----		----	
6099	IEC62021-1	0.025		0.70	

		<u>Only EN/IEC62021-1</u>	<u>Only D664-A</u>
normality	suspect	OK	OK
n	39	28	10
outliers	5	4	1
mean (n)	0.0225	0.0211	0.0258
st.dev. (n)	0.00474	0.00353	0.00590
R(calc.)	0.0133	0.0099	0.0165
R(D664-A:11ae1)	0.0099	---	0.0114
Compare R(EN62021-1:03)	0.0079	0.0074	---

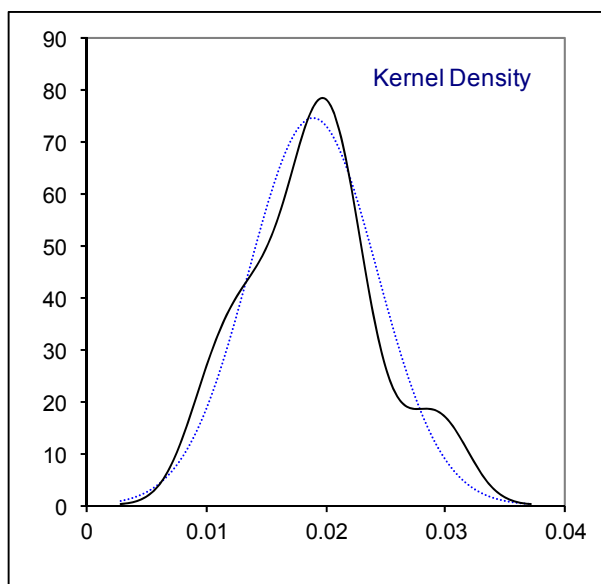
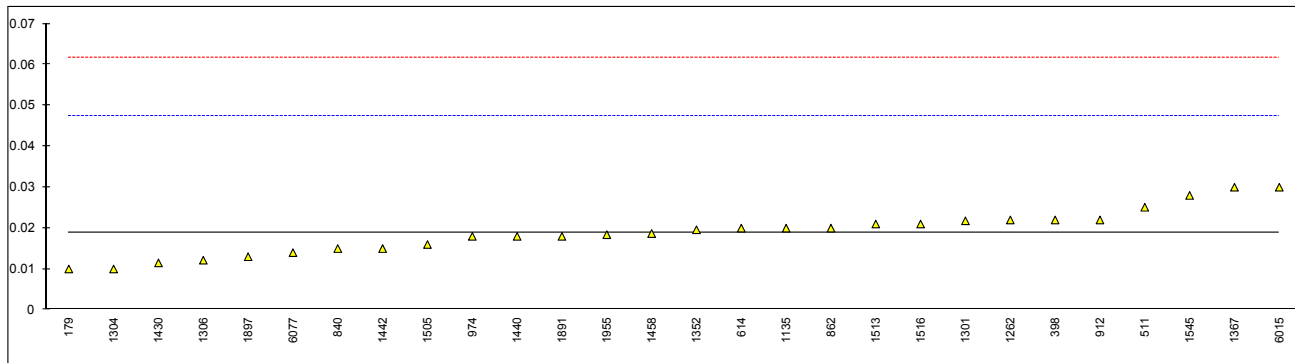


Determination of Acidity, Total(Colorimetric Titration) on sample #16251; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks
179	D974	0.01		-0.63	
237		----		----	
325		----		----	
331		----		----	
398	D974	0.022		0.21	
445		----		----	
511	D974	0.0251		0.43	
593		----		----	
614	D974	0.02		0.07	
840	D974	0.015		-0.28	
862	D974	0.020		0.07	
912	D974	0.022		0.21	
962		----		----	
963		----		----	
974	D974	0.018		-0.07	
1072		----		----	
1135	D974	0.02		0.07	
1146		----		----	
1156		----		----	
1161		----		----	
1178		----		----	
1262	ISO6618	0.022		0.21	
1264		----		----	
1301	D974	0.02178		0.20	
1304	INH-122	0.01		-0.63	
1306	D974	0.012156		-0.47	
1352	IEC62021-2	0.0196		0.05	
1361		----		----	
1367	D974	0.03		0.77	
1374		----		----	
1430	EN62021-2	0.0115		-0.52	
1435		----		----	
1440	ISO6618	0.018		-0.07	
1442	IEC62021-2	0.015		-0.28	
1444		----		----	
1458	D974	0.01867		-0.02	
1460		----		----	
1461		----		----	
1473		----		----	
1478		----		----	
1505	D974	0.016		-0.21	
1513	IEC62021-2	0.021		0.14	
1516	D974	0.021		0.14	
1529		----		----	
1545	D974	0.028		0.63	
1560		----		----	
1595		----		----	
1624		----		----	
1660		----		----	
1687		----		----	
1702		----		----	
1719		----		----	
1743		----		----	
1801		----		----	
1827		----		----	
1888		----		----	
1890		----		----	
1891	IEC62021-2	0.018		-0.07	
1897	IEC62021-2	0.013		-0.42	
1898		----		----	
1923		----		----	
1924		----		----	
1925		----		----	
1947		----		----	
1955	IEC62021-2	0.01840		-0.04	
2122	EN62021-2	<0.01		----	
2237		----		----	
6015	D974	0.03		0.77	
6029		----		----	
6030		----		----	
6031		----		----	
6036		----		----	
6053		----		----	
6071		----		----	
6077	IEC62021-2	0.01401		-0.34	
6099		----		----	

normality OK
 n 28
 outliers 0
 mean (n) 0.0189
 st.dev. (n) 0.00536
 R(calc.) 0.0150
 R(D974:14e2) 0.0400

Compare R(EN62021-2:07)= 0.0038

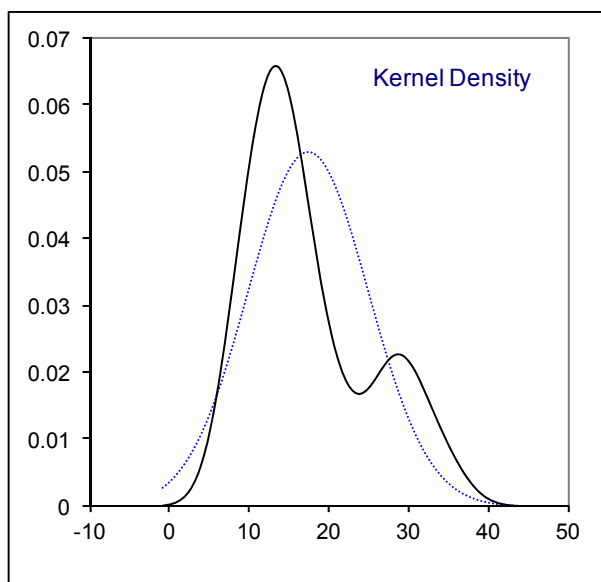
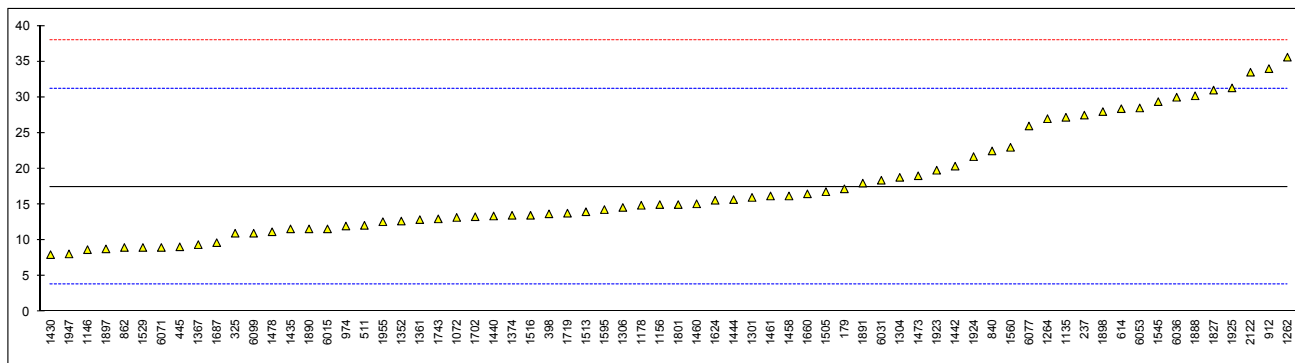


Determination of Breakdown Voltage on sample #16251, results in kV/2.5 mm

lab	method	value	mark	z(targ)	remarks
179	D877	17.2		-0.04	
237	IEC60156	27.5		1.47	
325	D1816	11		-0.95	
331		----		----	
398	EN60156	13.7		-0.55	
445	IEC60156	9.1		-1.23	
511	D1816	12.1		-0.79	
593		----		----	
614	IEC60156	28.4		1.60	
840	IEC60156	22.5		0.74	
862	IEC60156	9		-1.24	
912	IEC60156	34		2.42	
962		----		----	
963		----		----	
974	EN60156	12		-0.80	
1072	EN60156	13.2		-0.63	
1135	IEC60156	27.2		1.43	
1146	IEC60156	8.7		-1.29	
1156	IEC60156	15.0		-0.36	
1161		----		----	
1178	IEC60156	14.9		-0.38	
1262	EN60156	35.6		2.66	
1264	IEC60156	27		1.40	
1301	EN60156	16.0		-0.22	
1304	INH-124	18.8		0.19	
1306	EN60156	14.6		-0.42	
1352	IEC60156	12.7		-0.70	
1361	EN60156	12.9		-0.67	
1367	EN60156	9.4		-1.19	
1374	IEC60156	13.5		-0.58	
1430	EN60156	8		-1.39	
1435	IEC60156	11.6		-0.86	
1440	EN60156	13.4		-0.60	
1442	IEC60156	20.37 ¹		0.42	
1444	IEC60156	15.7		-0.26	
1458	IEC60156	16.2		-0.19	
1460	IEC60156	15.1		-0.35	
1461	EN60156	16.2		-0.19	
1473	IEC60156	19.03		0.23	
1478	IEC60156	11.2		-0.92	
1505	IEC60156	16.8		-0.10	
1513	IEC60156	14.0		-0.51	
1516	IEC60156	13.5		-0.58	
1529	IEC60156	9		-1.24	
1545	IEC60156	29.39		1.75	
1560	IEC60156	23		0.81	
1595	IEC60156	14.3		-0.47	
1624	EN60156	15.6		-0.28	
1660	IEC60156	16.5		-0.14	
1687	IEC60156	9.67		-1.15	
1702	IEC60156	13.3		-0.61	
1719	IEC60156	13.8		-0.54	
1743	IEC60156	13		-0.66	
1801	EN60156	15.0		-0.36	
1827	EN60156	31		1.98	
1888	IEC60156	30.2		1.87	
1890	IEC60156	11.6		-0.86	
1891	IEC60156	18.0		0.08	
1897	IEC60156	8.8		-1.27	
1898	EN60156	28		1.54	
1923	EN60156	19.8		0.34	
1924	EN60156	21.7		0.62	
1925	EN60156	31.3		2.03	
1947	IEC60156	8.1		-1.38	
1955	IEC60156	12.6		-0.72	
2122	EN60156	33.5		2.35	
2237		----		----	
6015		11.6		-0.86	
6029		----		----	
6030		----		----	
6031	IEC60156	18.4000		0.13	
6036	EN60156	30		1.84	
6053	IEC60156	28.5		1.62	
6071	IEC60156	9		-1.24	
6077	IEC60156	25.98		1.25	
6099	IEC60156	11.0		-0.95	

normality OK
 n 68
 outliers 0
 mean (n) 17.48
 st.dev. (n) 7.522
 R(calc.) 21.06
 R(EN60156:95) 19.09

Compare R(D1816:12)=8.22

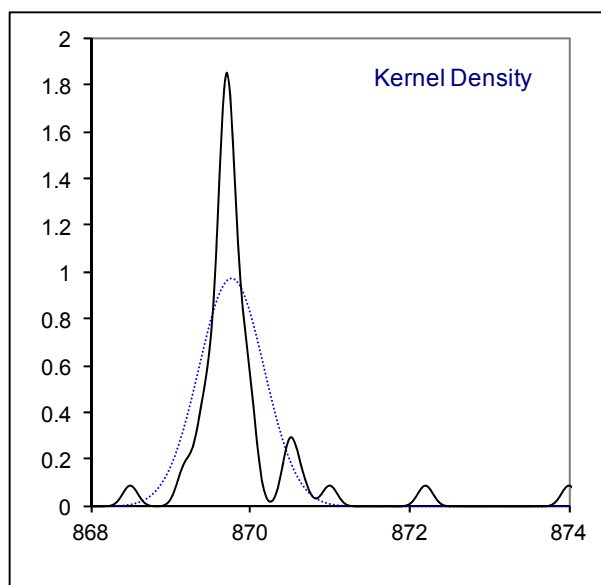
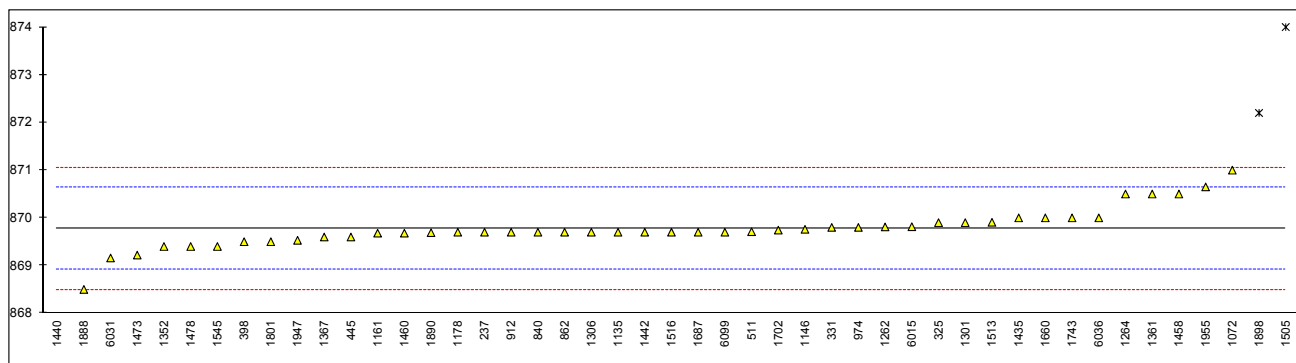


Determination of Density at 20°C on sample #16251; results in kg/m³

lab	method	value	mark	z(targ)	remarks
179		-----		-----	
237	D4052	869.7		-0.16	
325	D4052	869.9	C	0.30	reported: 0.8699 kg/m ³
331	ISO12185	869.8		0.07	
398	ISO12185	869.5		-0.63	
445	D4052	869.6		-0.40	
511	D4052	869.71		-0.14	
593		-----		-----	
614		-----		-----	
840	ISO12185	869.70		-0.16	
862	ISO12185	869.7		-0.16	
912	D4052	869.7		-0.16	
962		-----		-----	
963		-----		-----	
974	D4052	869.8		0.07	
1072	ISO3675	871.0		2.87	
1135	ISO12185	869.7	C	-0.16	first reported: 0.8697 kg/m ³
1146	D4052mod.	869.76		-0.02	
1156		-----		-----	
1161	ISO12185	869.68		-0.21	
1178	ISO12185	869.7	C	-0.16	first reported: 0.8697 kg/m ³
1262	ISO3675	869.81		0.09	
1264	D4052	870.5		1.70	
1301	D4052	869.9	C	0.30	first reported: 0.8699 kg/m ³
1304		-----		-----	
1306	D1298	869.7		-0.16	
1352	D4052	869.4		-0.86	
1361	ISO3675	870.5		1.70	
1367	ISO12185	869.6	C	-0.40	first reported: 0.8696 kg/m ³
1374		-----		-----	
1430		-----		-----	
1435	D7042	870.0		0.54	
1440		867.5	C,R(0.01)	-5.30	reported: 0.8675 kg/m ³
1442	D4052	869.7		-0.16	
1444		-----		-----	
1458	D4052	870.5		1.70	
1460	D4052	869.68	C	-0.21	first reported: 0.86968 kg/m ³
1461		-----		-----	
1473	D1217	869.22	C	-1.28	first reported: 0.86928 kg/m ³
1478	ISO12185	869.4		-0.86	
1505	D7042	874.0	R(0.01)	9.87	
1513	ISO12185	869.907	C	0.32	first reported: 0.869907 kg/m ³
1516	ISO3675	869.7		-0.16	
1529		-----		-----	
1545	ISO3675	869.4		-0.86	
1560		-----		-----	
1595		-----		-----	
1624		-----		-----	
1660	D7042	870.0	C	0.54	first reported: 0.8700 kg/m ³
1687	ISO12185	869.70		-0.16	
1702	ISO12185	869.743		-0.06	
1719		-----		-----	
1743		870		0.54	
1801	ISO3675	869.5		-0.63	
1827		-----		-----	
1888	ISO3675	868.5		-2.96	
1890	ISO12185	869.69		-0.19	
1891		-----		-----	
1897		-----		-----	
1898	ISO12185	872.2	R(0.01)	5.67	
1923		-----		-----	
1924		-----		-----	
1925		-----		-----	
1947	ISO12185	869.53		-0.56	
1955	D7042	870.65		2.05	
2122		-----		-----	
2237		-----		-----	
6015	ISO12185	869.815		0.11	
6029		-----		-----	
6030		-----		-----	
6031	D1298	869.16		-1.42	
6036	ISO3675	870	C	0.54	first reported: 0.870 kg/m ³
6053		-----		-----	
6071		-----		-----	
6077		-----		-----	
6099	ISO12185	869.7	C	-0.16	first reported: 872.4

normality not OK
 n 44
 outliers 3
 mean (n) 869.77
 st.dev. (n) 0.409
 R(calc.) 1.15
 R(ISO3675:98) 1.20

Compare R(D1298:12b)=1.20

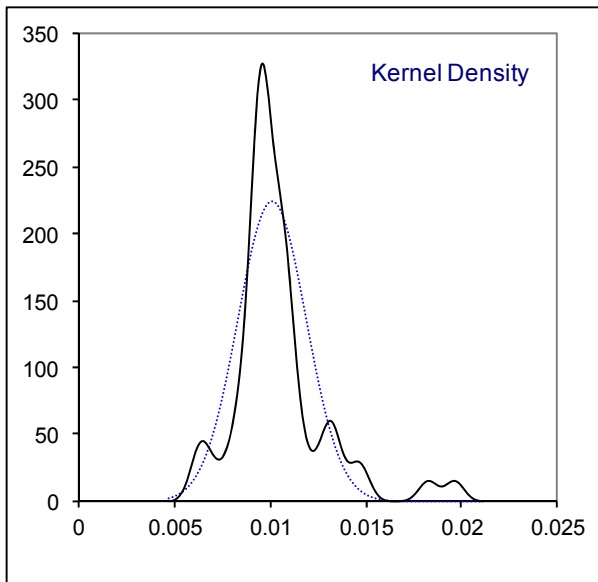
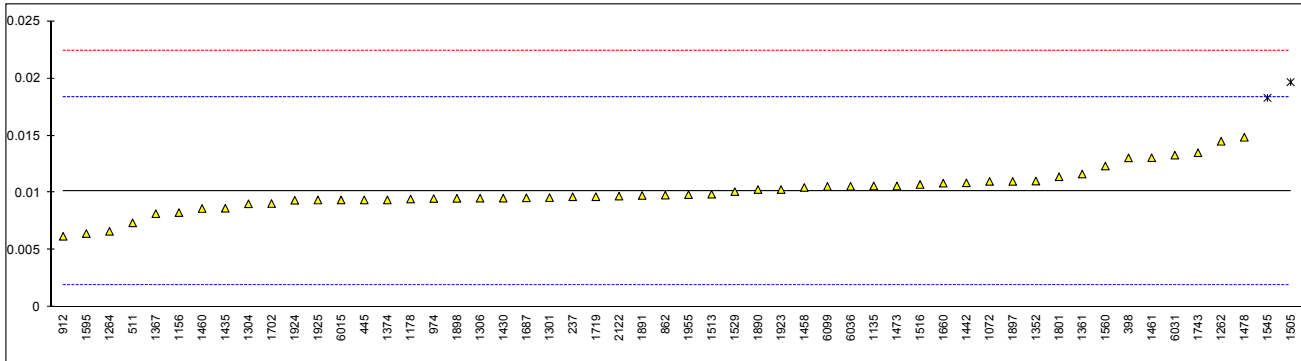


Determination of Di-electric Dissipation Factor at 90°C on sample #16251

lab	method	value	mark	z(targ)	remarks
179		----		----	
237	IEC60247	0.009653		-0.12	
325		----		----	
331		----		----	
398	EN60247	0.013053		0.71	reported: measured at 50Hz
445	IEC60247	0.009380		-0.18	
511	D924	0.007377	C	-0.67	first reported: 0.7377
593		----		----	
614		----		----	
840		----		----	
862	IEC60247	0.0098		-0.08	
912	IEC60247	0.0062		-0.96	
962		----		----	
963		----		----	
974	EN60247	0.0095		-0.15	
1072	EN60247	0.0110		0.21	
1135	IEC60247	0.0106		0.11	
1146		----		----	
1156	IEC60247	0.00827		-0.46	
1161		----		----	
1178	IEC60247	0.00946		-0.16	
1262	EN60247	0.01450		1.07	
1264	IEC60247	0.006639		-0.86	
1301	EN60247	0.00958		-0.14	
1304	INH-125	0.009041		-0.27	
1306	EN60247	0.009527		-0.15	
1352	IEC60247	0.01103		0.22	
1361	EN60247	0.011636		0.37	
1367	EN60247	0.008176		-0.48	
1374	IEC60247	0.00938		-0.18	
1430	EN60247	0.009534		-0.15	
1435	IEC60247	0.00865		-0.36	
1440		----		----	
1442	IEC60247	0.0108715		0.18	
1444		----		----	
1458	IEC60247	0.010468		0.08	
1460	IEC60247	0.008627		-0.37	
1461	EN60247	0.013064		0.72	
1473	IEC60247	0.010601		0.11	
1478	IEC60247	0.014851		1.16	
1505	IEC60247	0.01967	R(0.01)	2.33	
1513	IEC60247	0.009877		-0.06	
1516	IEC60247	0.010726		0.15	
1529	EN60247	0.0101		-0.01	
1545	IEC60247	0.01829	R(0.01)	2.00	
1560	IEC60247	0.01234		0.54	
1595	IEC60247	0.00644		-0.90	
1624		----		----	
1660	IEC60247	0.01084		0.17	
1687	IEC60247	0.009562		-0.14	
1702	IEC60247	0.00906		-0.26	
1719	IEC60247	0.00966		-0.12	
1743	IEC60247	0.0135		0.82	
1801	EN60247	0.0114		0.31	
1827		----		----	
1888		----		----	
1890	IEC60247	0.01028		0.04	
1891	IEC60247	0.00977		-0.09	
1897	IEC60247	0.0110		0.21	
1898	IEC60247	0.00952		-0.15	
1923	EN60247	0.010285		0.04	
1924	EN60247	0.009347		-0.19	
1925	EN60247	0.00937		-0.19	
1947		----		----	
1955	IEC60247	0.00984		-0.07	
2122	EN60247	0.009712		-0.10	
2237		----		----	
6015	EN60247	0.009377		-0.18	
6029		----		----	
6030		----		----	
6031	IEC60247	0.01329		0.77	
6036	EN60247	0.010569		0.11	
6053		----		----	
6071		----		----	
6077		----		----	
6099	IEC60247	0.010555		0.10	

normality suspect
 n 52
 outliers 2
 mean (n) 0.01013
 st.dev. (n) 0.001786
 R(calc.) 0.00500
 R(EN60247:04) 0.01144

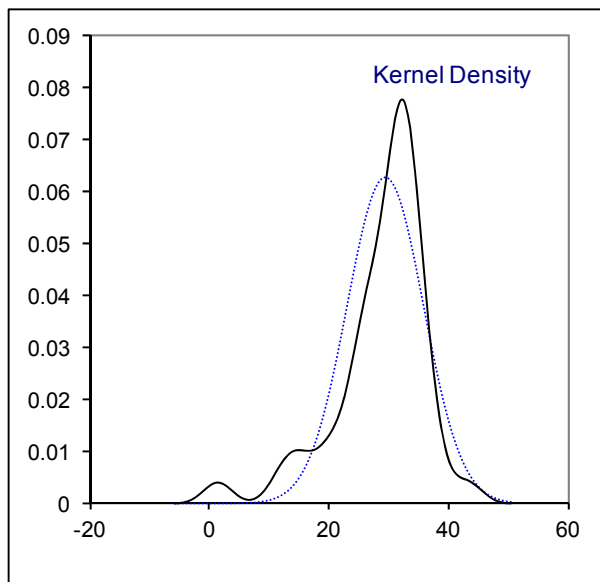
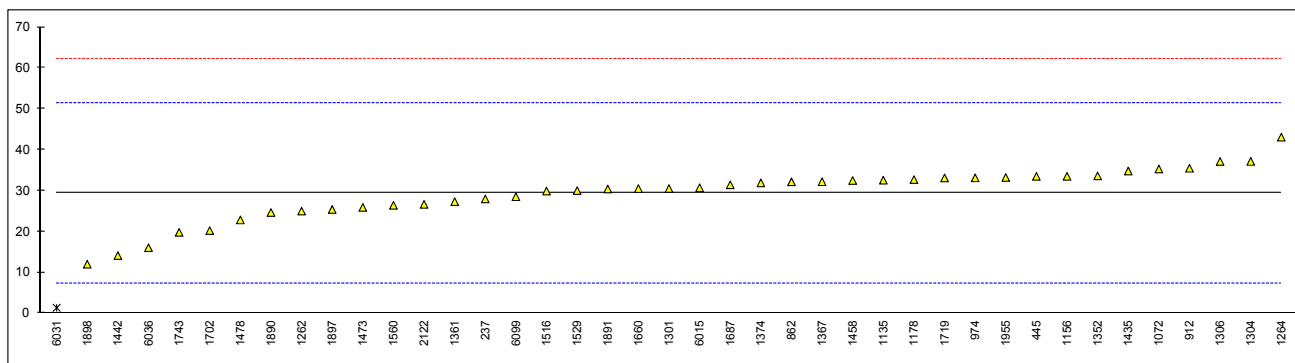
Compare R(D924:15)=0.02850



Determination of Specific Resistance at 90°C on sample #16251; results in GΩm

lab	method	value	mark	z(targ)	remarks
179		----		----	
237	IEC60247	27.98		-0.12	
325		----		----	
331		----		----	
398		----		----	
445	IEC60247	33.49		0.38	
511		----		----	
593		----		----	
614		----		----	
840		----		----	
862	IEC60247	32.15		0.26	
912	IEC60247	35.47		0.56	
962		----		----	
963		----		----	
974	EN60247	33.12		0.34	
1072	EN60247	35.3		0.54	
1135	IEC60247	32.57		0.29	
1146		----		----	
1156	IEC60247	33.5		0.38	
1161		----		----	
1178	IEC60247	32.7	C	0.31	first reported: 327
1262	EN60247	25		-0.39	
1264	IEC60247	43.09		1.25	
1301	EN60247	30.52		0.11	
1304	INH-125	37.155		0.71	
1306	EN60247	37.14		0.71	
1352	IEC60247	33.6		0.39	
1361	EN60247	27.31		-0.18	
1367	EN60247	32.18		0.26	
1374	IEC60247	31.9		0.23	
1430		----		----	
1435	IEC60247	34.8		0.50	
1440		----		----	
1442	IEC60247	14.12		-1.38	
1444		----		----	
1458	IEC60247	32.48		0.29	
1460		----		----	
1461		----		----	
1473	IEC60247	25.9		-0.31	
1478	IEC60247	22.83		-0.59	
1505		----		----	
1513		----		----	
1516	IEC60247	29.9		0.05	
1529	IEC60247	30		0.06	
1545		----		----	
1560	IEC60247	26.4		-0.27	
1595		----		----	
1624		----		----	
1660	IEC60247	30.5		0.11	
1687	IEC60247	31.42		0.19	
1702	IEC60247	20.215		-0.83	
1719	IEC60247	33.100	C	0.34	reported: 3310000000 GΩm, Probably reported in a different unit
1743	IEC60247	19.8		-0.87	
1801		----		----	
1827		----		----	
1888		----		----	
1890	IEC60247	24.65		-0.43	
1891	IEC60247	30.4		0.10	
1897	IEC60247	25.4		-0.36	
1898	IEC60247	11.99		-1.58	
1923		----		----	
1924		----		----	
1925		----		----	
1947		----		----	
1955	IEC60247	33.225		0.35	
2122	EN60247	26.63		-0.25	
2237		----		----	
6015	EN60247	30.70		0.12	
6029		----		----	
6030		----		----	
6031	IEC60247	1.337	C,R(0.01)	-2.55	first reported: 1337.3
6036	EN60247	16.046		-1.21	
6053		----		----	
6071		----		----	
6077		----		----	
6099	IEC60247	28.5		-0.08	

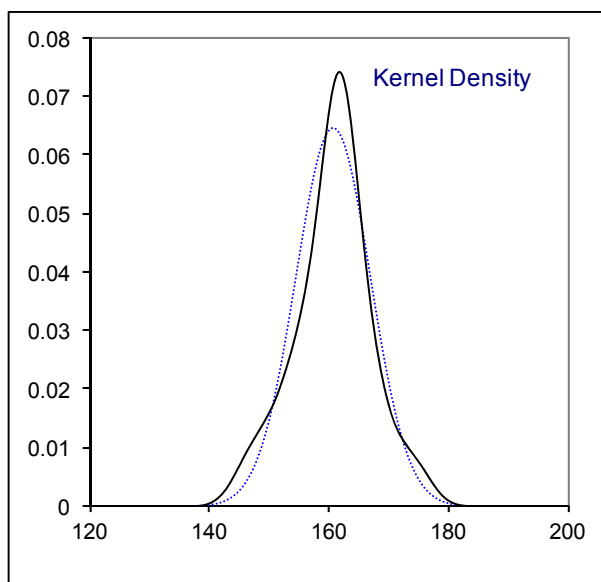
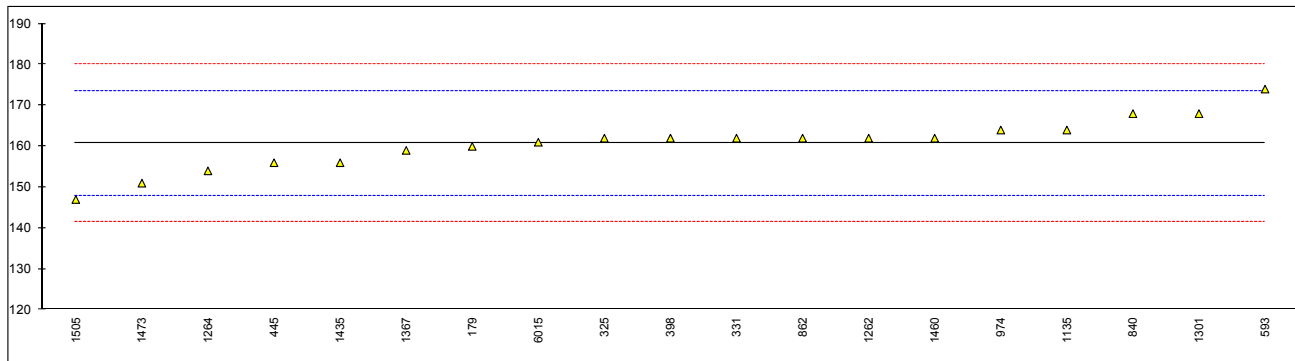
normality	suspect
n	40
outliers	1
mean (n)	29.3295
st.dev. (n)	6.38884
R(calc.)	17.8887
R(EN60247:04)	30.7960



Determination of Flash Point COC on sample #16251; results in °C

lab	method	value	mark	z(targ)	remarks
179	D92	160		-0.11	
237		----		----	
325	D92	162		0.20	
331	D92	162.0		0.20	
398	D92	162.0		0.20	
445	D92	156.0		-0.74	
511		----		----	
593	D92	174		2.06	
614		----		----	
840	ISO2592	168		1.13	
862	D92	162		0.20	
912		----		----	
962		----		----	
963		----		----	
974	D92	164.0		0.51	
1072		----		----	
1135	D92	164		0.51	
1146		----		----	
1156		----		----	
1161		----		----	
1178		----		----	
1262	ISO2592	162		0.20	
1264	D92	154	C	-1.05	first reported COC test result as PMcc result
1301	D92	168.0		1.13	
1304		----		----	
1306		----		----	
1352		----		----	
1361		----		----	
1367	D92	159		-0.27	
1374		----		----	
1430		----		----	
1435	D92	156		-0.74	
1440		----		----	
1442		----		----	
1444		----		----	
1458		----		----	
1460	D92	162		0.20	
1461		----		----	
1473	D92	151		-1.51	
1478		----		----	
1505	D92	147		-2.14	
1513		----		----	
1516		----		----	
1529		----		----	
1545		----		----	
1560		----		----	
1595		----		----	
1624		----		----	
1660		----		----	
1687		----		----	
1702		----		----	
1719		----		----	
1743		----		----	
1801		----		----	
1827		----		----	
1888		----		----	
1890		----		----	
1891		----		----	
1897		----		----	
1898		----		----	
1923		----		----	
1924		----		----	
1925		----		----	
1947		----		----	
1955		----		----	
2122		----		----	
2237		----		----	
6015	ISO2592	161		0.04	
6029		----		----	
6030		----		----	
6031		----		----	
6036		----		----	
6053		----		----	
6071		----		----	
6077		----		----	
6099		----		----	

normality	OK
n	19
outliers	0
mean (n)	160.74
st.dev. (n)	6.190
R(calc.)	17.33
R(D92:16a)	18.00

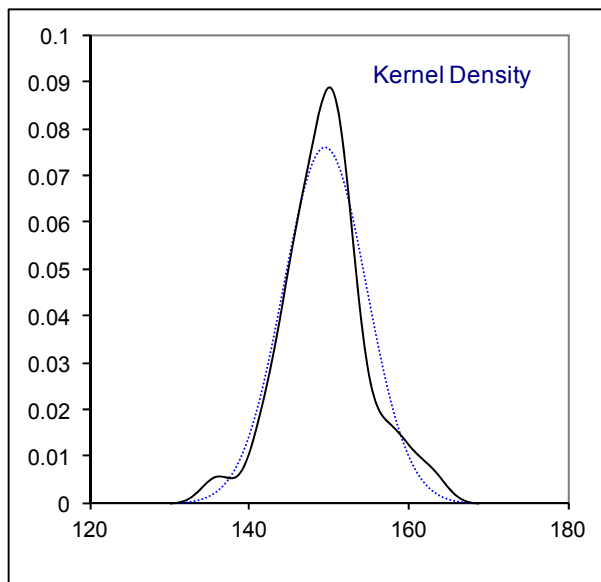
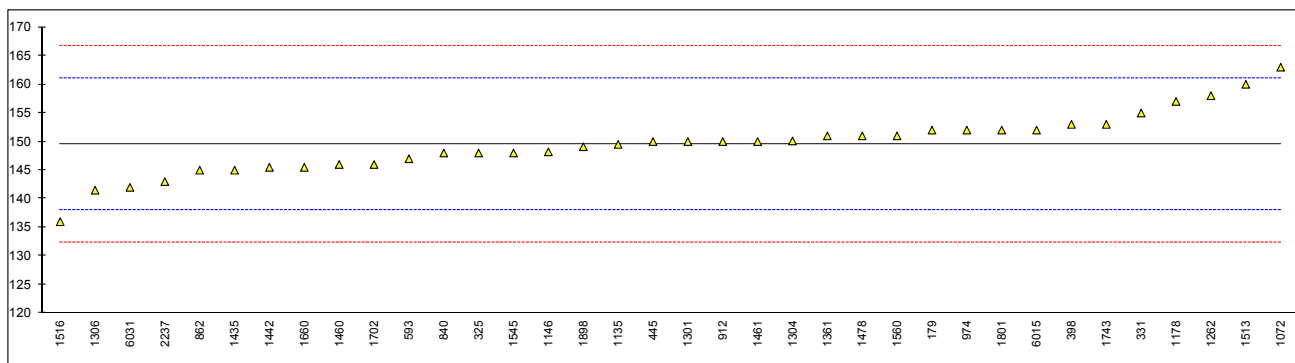


Determination of Flash Point PMcc on sample #16251; results in °C

lab	method	value	mark	z(targ)	remarks
179	D93-A	152.0		0.43	
237		----		----	
325	D93-A	148		-0.27	
331	D93	155.0		0.96	
398	ISO2719-A	153.0		0.61	
445	D93-A	150.0		0.08	
511		----		----	
593	D93	147.0		-0.44	
614		----		----	
840	ISO2719-B	148.0		-0.27	
862	D93-A	145.0		-0.79	
912	D93-A	150		0.08	
962		----		----	
963		----		----	
974	D93-A	152.0		0.43	
1072	ISO2719-A	163		2.36	
1135	D93-A	149.5		-0.01	
1146	D93-Amod.	148.2		-0.23	
1156		----		----	
1161		----		----	
1178	ISO2719-B	157.0		1.31	
1262	ISO2719-A	158.0		1.48	
1264		----		----	
1301	D93-A	150		0.08	
1304	INH-115	150.1		0.10	
1306	D7094	141.5	C	-1.41	first reported PMcc test result as COC result
1352		----		----	
1361	ISO2719-A	151.0		0.26	
1367		----		----	
1374		----		----	
1430		----		----	
1435	D93-A	145		-0.79	
1440		----		----	
1442	ISO2719-A	145.5		-0.71	
1444		----		----	
1458		----		----	
1460	D93-A	146		-0.62	
1461	ISO2719-A	150		0.08	
1473		----		----	
1478	ISO2719-A	151.0		0.26	
1505		----		----	
1513	ISO2719-A	160.0		1.83	
1516	D93-A	136		-2.37	
1529		----		----	
1545	ISO2719-A	148.0		-0.27	
1560	ISO2719	151		0.26	
1595		----		----	
1624		----		----	
1660	D93-A	145.5		-0.71	
1687		----		----	
1702	ISO2719-B	146.0		-0.62	
1719		----		----	
1743	ISO2719-A	153.0		0.61	
1801	ISO2719-A	152.0		0.43	
1827		----		----	
1888		----		----	
1890		----		----	
1891		----		----	
1897		----		----	
1898	ISO2719-C	149.1		-0.08	
1923		----		----	
1924		----		----	
1925		----		----	
1947		----		----	
1955		----		----	
2122		----		----	
2237	ISO2719-B	143.0		-1.14	
6015	D7236	152.0		0.43	
6029		----		----	
6030		----		----	
6031	D93-A	142		-1.32	
6036		----		----	
6053		----		----	
6071		----		----	
6077		----		----	
6099		----		----	

normality	suspect
n	36
outliers	0
mean (n)	149.54
st.dev. (n)	5.252
R(calc.)	14.71
R(ISO2719-B:16)	16.00

Compare R(ISO2719-A:16)=10.62
 Compare R(D93-A:16a)=10.62
 Compare R(D93-B:16a)=10.00

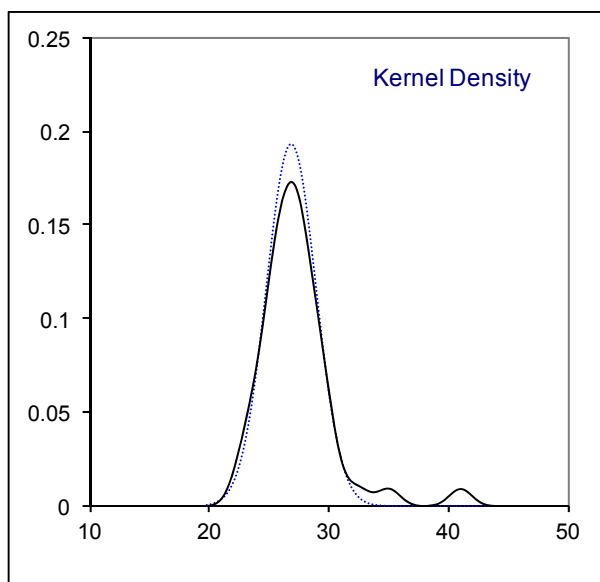
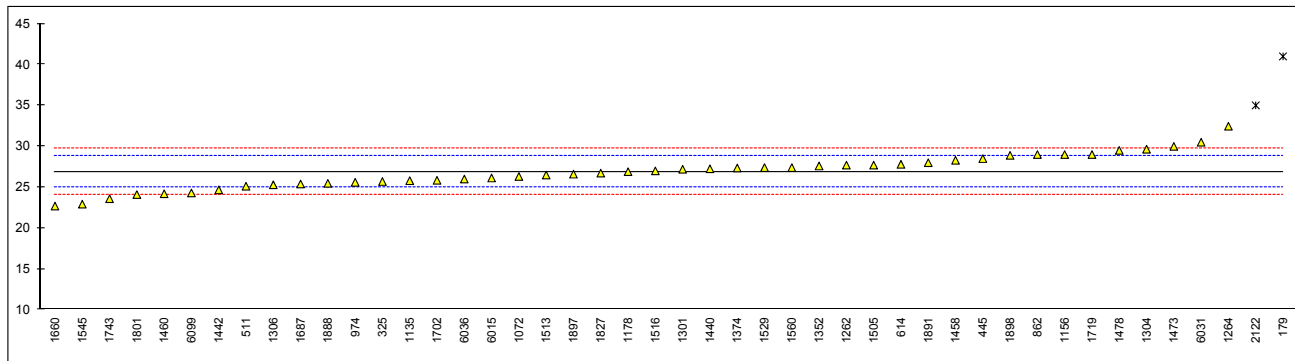


Determination of Interfacial Surface Tension on sample #16251; results in mN/m

lab	method	value	mark	z(targ)	remarks
179	D971	41	C,R(0.01)	14.69	first reported: 43
237		----		----	
325	In house	25.7		-1.24	
331		----		----	
398		----		----	
445	D971	28.5		1.68	
511	D971	25.12		-1.84	
593		----		----	
614	D971	27.8		0.95	
840		----		----	
862	ISO6295	29		2.20	
912		----		----	
962		----		----	
963		----		----	
974	D971	25.6		-1.34	
1072	ISO6295	26.32		-0.59	
1135	ISO6295	25.8		-1.13	
1146		----		----	
1156	D971	29.0		2.20	
1161		----		----	
1178	D971	26.9		0.01	
1262	D971	27.7		0.84	
1264	D971	32.46		5.80	
1301	ISO6295	27.2		0.32	
1304	INH-123	29.65		2.87	
1306	D971	25.30		-1.66	
1352	D971	27.6		0.74	
1361		----		----	
1367		----		----	
1374	D971	27.35		0.48	
1430		----		----	
1435		----		----	
1440	D971	27.26		0.39	
1442	EN14210	24.68		-2.30	
1444		----		----	
1458	D971	28.3		1.47	
1460	D971	24.2		-2.80	
1461		----		----	
1473	D971	30	C	3.24	first reported: 33.5467
1478	D971	29.5	C	2.72	first reported: 34.5
1505	D971	27.7		0.84	
1513	D971	26.49		-0.42	
1516	D971	27.0		0.12	
1529	D971	27.4		0.53	
1545	D971	22.93		-4.12	
1560	D971	27.4		0.53	
1595		----		----	
1624		----		----	
1660	D971	22.7		-4.36	
1687	D971	25.4		-1.55	
1702	D971	25.848		-1.08	
1719	D2285	29		2.20	
1743	D971	23.6		-3.43	
1801	ISO6295	24.1		-2.90	
1827	D971	26.73		-0.17	
1888	ISO6295	25.47		-1.48	
1890		----		----	
1891	D971	28.0		1.16	
1897	D971	26.6		-0.30	
1898	D971	28.9		2.09	
1923		----		----	
1924		----		----	
1925		----		----	
1947		----		----	
1955		----		----	
2122	EN14210	35.01	R(0.05)	8.46	
2237		----		----	
6015	D971	26.13		-0.79	
6029		----		----	
6030		----		----	
6031	D971	30.5		3.76	
6036	D971	26		-0.93	
6053		----		----	
6071		----		----	
6077		----		----	
6099	EN14210	24.3		-2.70	

normality OK
 n 44
 outliers 2
 mean (n) 26.890
 st.dev. (n) 2.0623
 R(calc.) 5.774
 R(D971:12) 2.689

Compare R(ISO6295:83)=2.689

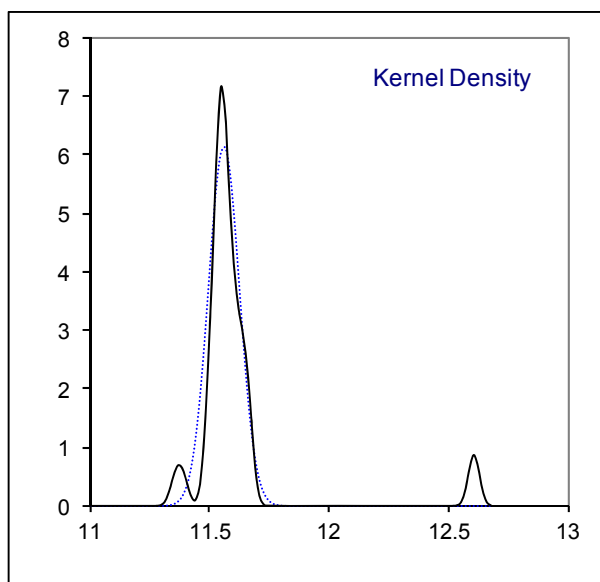
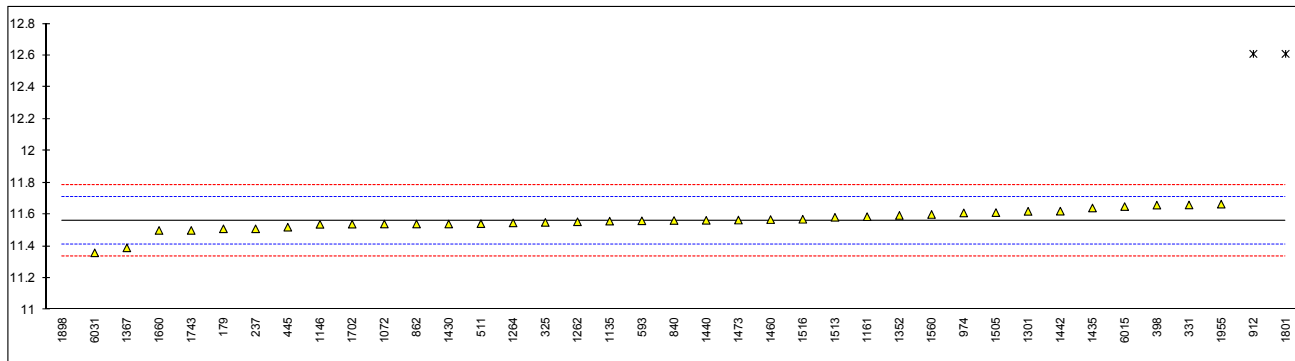


Determination of Kinematic Viscosity at 40°C on sample #16251; results in mm²/s

lab	method	value	mark	z(targ)	remarks
179	D445	11.51		-0.70	
237	D445	11.51		-0.70	
325	D445	11.55		-0.16	
331	D7279Mod.	11.66		1.32	
398	ISO3104	11.6589		1.31	
445	ISO3104	11.52		-0.56	
511	D445	11.542		-0.27	
593	D445	11.56		-0.02	
614		----		----	
840	ISO3104	11.562		0.00	
862	ISO3104	11.54		-0.29	
912	D445	12.61	R(0.01)	14.10	
962		----		----	
963		----		----	
974	D445	11.61		0.65	
1072	ISO3104	11.54		-0.29	
1135	ISO3104	11.558		-0.05	
1146	ISO3104	11.538		-0.32	
1156		----		----	
1161	ISO3104	11.586		0.33	
1178		----		----	
1262	ISO3104	11.554		-0.11	
1264	D7042	11.5470		-0.20	
1301	ISO3104	11.62		0.78	
1304		----		----	
1306		----		----	
1352	D7042	11.594		0.43	
1361		----		----	
1367	D445	11.39	C	-2.31	first reported: 12.7
1374		----		----	
1430	D445	11.54		-0.29	
1435	D7042	11.641		1.07	
1440	ISO3104	11.5634		0.02	
1442	D7042	11.621		0.80	
1444		----		----	
1458		----		----	
1460	D7042	11.568		0.08	
1461		----		----	
1473	D445	11.5648		0.04	
1478		----		----	
1505	D7042	11.612		0.68	
1513	ISO3104	11.5822		0.27	
1516	ISO3104	11.57	C	0.11	first reported: 11.27
1529		----		----	
1545		----		----	
1560	ISO3104	11.6		0.51	
1595		----		----	
1624		----		----	
1660	D7042	11.5		-0.83	
1687		----		----	
1702	D7042	11.539		-0.31	
1719		----		----	
1743	D7279	11.5		-0.83	
1801	ISO3104	12.61	R(0.01)	14.10	
1827		----		----	
1888		----		----	
1890		----		----	
1891		----		----	
1897		----		----	
1898	ISO3104	8.1715	R(0.01)	-45.61	
1923		----		----	
1924		----		----	
1925		----		----	
1947		----		----	
1955	D7042	11.665		1.39	
2122		----		----	
2237		----		----	
6015	D7279	11.65		1.19	
6029		----		----	
6030		----		----	
6031	D445	11.359		-2.73	
6036		----		----	
6053		----		----	
6071		----		----	
6077		----		----	
6099		----		----	

normality	not OK
n	36
outliers	3
mean (n)	11.5618
st.dev. (n)	0.06500
R(calc.)	0.1820
R(iis)	0.2081

R(iis) = 1.8% of the mean for used oils at 40°C (see lit. 17)

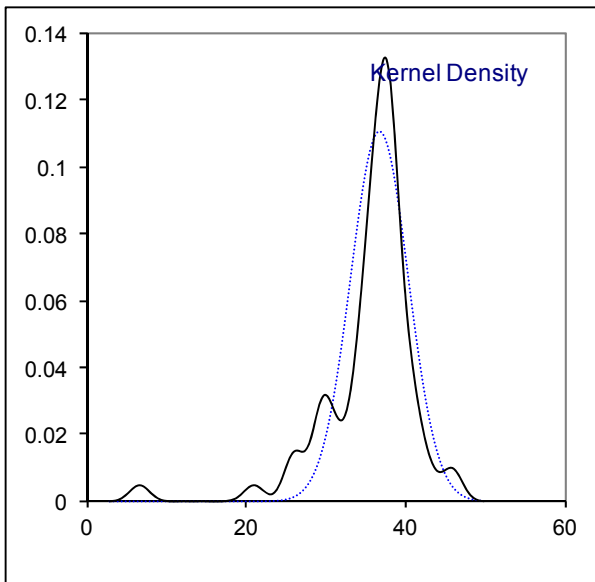
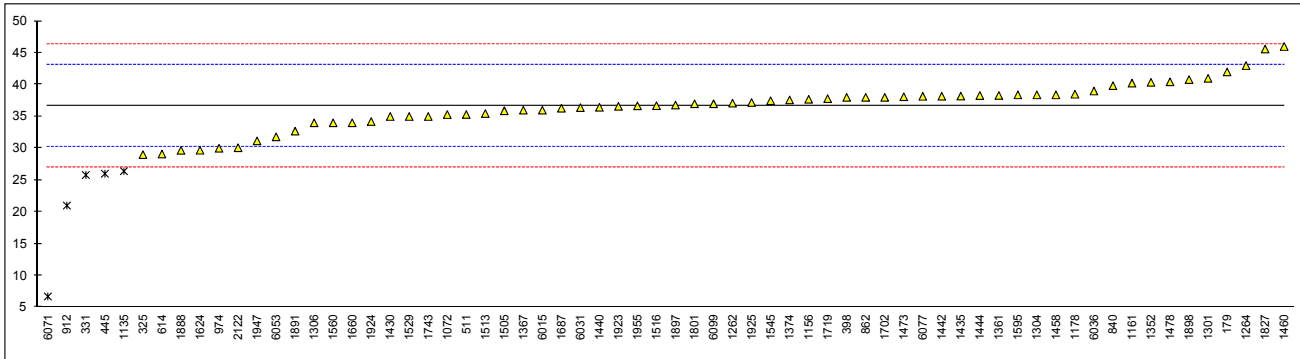


Determination of Water on sample #16251; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D6304-C	42	C	1.63	first reported: 66
237		----		----	
325	D6304-C	29		-2.37	
331	D6304-A	25.8	R(0.05)	-3.36	
398	EN60814	38		0.40	
445	D6304-B	26	R(0.05)	-3.30	
511	D1533	35.317		-0.42	
593		----		----	
614	IEC60814	29.1		-2.34	
840	IEC60814	39.85		0.97	
862	D6304-A	38		0.40	
912	D6304-A	21	R(0.01)	-4.84	
962		----		----	
963		----		----	
974	D1533	30		-2.06	
1072	EN60814	35.3		-0.43	
1135	EN60814	26.4	R(0.05)	-3.17	
1146		----		----	
1156	IEC60814	37.7		0.31	
1161	ISO12937	40.238		1.09	
1178	IEC60814	38.5		0.56	
1262	D6304-A	37.1		0.12	
1264	D1533	43		1.94	
1301	IEC60814	41.0		1.33	
1304	INH-121	38.4		0.53	
1306	EN60814	34		-0.83	
1352	IEC60814	40.37		1.13	
1361	EN60814	38.3		0.49	
1367	D4928	36		-0.21	
1374	IEC60814	37.6		0.28	
1430	EN60814	35		-0.52	
1435	IEC60814	38.2		0.46	
1440	EN60814	36.45		-0.08	
1442	IEC60814	38.18		0.46	
1444	IEC60814	38.2930		0.49	
1458	IEC60814	38.4		0.53	
1460	D1533	46		2.87	
1461		----		----	
1473	D1533	38.10		0.43	
1478	IEC60814	40.45		1.16	
1505	D1533	35.9		-0.25	
1513	IEC60814	35.46		-0.38	
1516	IEC60814	36.7		0.00	
1529	IEC60814	35		-0.52	
1545	IEC60814	37.47		0.24	
1560	IEC60814	34		-0.83	
1595	IEC60814	38.395		0.52	
1624	IEC60814	29.69		-2.16	
1660	IEC60814	34		-0.83	
1687	IEC60814	36.291		-0.12	
1702	IEC60814	38		0.40	
1719	IEC60814	37.8		0.34	
1743	IEC60814	35		-0.52	
1801	IEC60814	37		0.09	
1827	D6304-A	45.6		2.74	
1888	IEC60814	29.67		-2.16	
1890		----		----	
1891	IEC60814	32.7		-1.23	
1897	IEC60814	36.8		0.03	
1898	EN60814	40.8		1.26	
1923	EN60814	36.6		-0.03	
1924	EN60814	34.2		-0.77	
1925	EN60814	37.2		0.16	
1947	IEC60814	31.154		-1.71	
1955	IEC60814	36.67		-0.01	
2122	IEC60814	30.09		-2.04	
2237		----		----	
6015	DIN51777	36.0		-0.21	
6029		----		----	
6030		----		----	
6031	IEC60814	36.395		-0.09	
6036	EN60814	39		0.71	
6053	IEC60814	31.8		-1.51	
6071	IEC60814	6.7	C,R(0.01)	-9.24	first reported: 3.7272
6077	D1533	38.17		0.45	
6099	IEC60814	37.0		0.09	

normality OK
 n 61
 outliers 5
 mean (n) 36.695
 st.dev. (n) 3.6009
 R(calc.) 10.083
 R(EN60814:98) 9.086

Compare R(D1533:12)=14

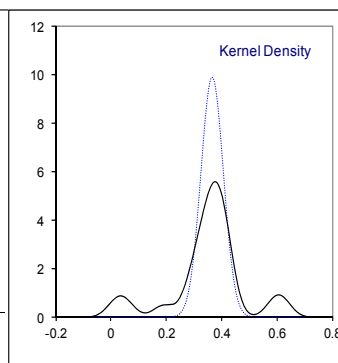
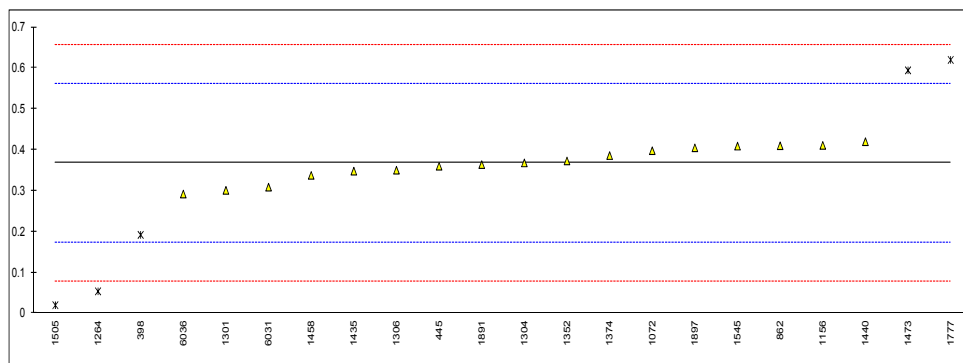


Determination of Total Furanics on sample #16252; results in mg/kg

lab	method	value	mark	z(targ)	remarks
237		----		----	
398	IEC61198	0.192	ex	-1.81	ex: an outlier in one of the Furanic components
445	EN61198	0.36		-0.07	
614		----		----	
840		----		----	
862	IEC61198	0.41		0.44	
912		----		----	
963		----		----	
1072	EN61198	0.398		0.32	
1135		----		----	
1137			W	----	first reported: 0.016197
1156	IEC61198	0.411		0.45	
1178		----		----	
1264	D5837	0.054	ex, E	-3.24	ex: an outlier in one of the Furanic components; E: iis calculated: 0.081
1301	D5837	0.301		-0.68	
1304	INH-126	0.368		0.01	
1306	IEC61198	0.3502706		-0.17	
1352	IEC61198	0.3729		0.06	
1367		----		----	
1374	D5837	0.386		0.19	
1430		----		----	
1435	IEC61198	0.348		-0.20	
1440	EN61198	0.42		0.55	
1442		----		----	
1458	IEC61198	0.3377		-0.31	
1473	IEC61198	0.5945	ex	2.35	ex: an outlier in one of the Furanic components
1478		----		----	
1505	D5837	0.02	ex	-3.59	ex: false negative test results in almost all Furanic components
1513		----		----	
1516		----		----	
1529		----		----	
1545	IEC61198	0.4091		0.43	
1560		----		----	
1660		----		----	
1702		----		----	
1743		----		----	
1777	IEC61198	0.62	ex	2.62	ex: an outlier in one of the Furanic components
1801		----		----	
1872		----		----	
1888		----		----	
1890		----		----	
1891	IEC61198	0.364		-0.03	
1897	IEC61198	0.405		0.39	
2122		----		----	
6015		----		----	
6031	IEC61198	0.30882		-0.60	
6036	EN61198	0.292		-0.78	
6073		----		----	

normality OK
n 17
outliers 0+5ex
mean (n) 0.3672
st.dev. (n) 0.04038
R(calc.) 0.1131
R(Horwitz 2 comp.) 0.2705

Compare R(IEC61198:93) = 0.0404
Compare R(D5837:15; liquid/liquid extr)= 0.3087 *)
Compare R(D5837:15; solid state extr)= 0.8176 *)
*) R(D5837:15) estimated on 3 times the repeatabilities of 2-Furfural, 2-Furfuryl alcohol and 5-Methyl-2-Furfural

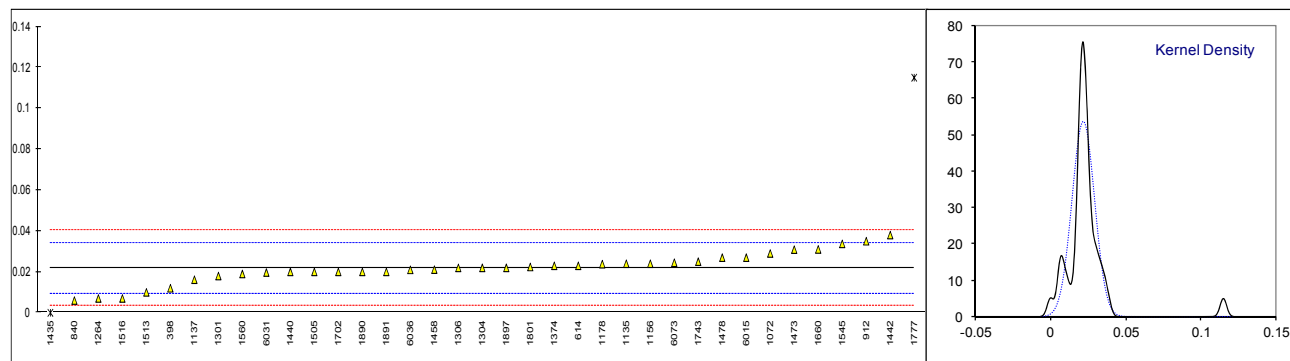


Determination of 2-Furfural on sample #16252; results in mg/kg

lab	method	value	mark	z(targ)	remarks
237		----		----	
398	IEC61198	0.012		-1.58	
445	EN61198	<0.05		----	
614	IEC61198	0.023		0.19	
840	IEC61198	0.006		-2.55	
862	IEC61198	<0.05		----	
912	D5837	0.035		2.12	
963		----		----	
1072	EN61198	0.029		1.15	
1135	IEC61198	0.024		0.35	
1137	D5837	0.016197		-0.91	
1156	IEC61198	0.024		0.35	
1178	IEC61198	0.0238		0.32	
1264	D5837	0.007		-2.39	
1301	D5837	0.018		-0.62	
1304	INH-126	0.022		0.03	
1306	IEC61198	0.0219606		0.02	
1352	IEC61198	nd	C	----	first reported: 0.0478
1367		----		----	
1374	D5837	0.023		0.19	
1430	EN61198	<0.03	C	----	first reported: 0
1435	IEC61198	0.000	ex	-3.51	ex: probably a false negative test result?
1440	EN61198	0.02		-0.30	
1442	IEC61198	0.038		2.60	
1458	IEC61198	0.0211		-0.12	
1473	IEC61198	0.0309		1.46	
1478	IEC61198	0.0270		0.83	
1505	D5837	0.02		-0.30	
1513	IEC61198	0.010		-1.91	
1516	IEC61198	0.007		-2.39	
1529	IEC61198	< 0.05		----	
1545	IEC61198	0.0337		1.91	
1560	IEC61198	0.019		-0.46	
1660	IEC61198	0.031		1.48	
1702	IEC61198	0.020		-0.30	
1743	IEC61198	0.025		0.51	
1777	IEC61198	0.115	R(0.01)	15.00	
1801	EN61198	0.0224		0.09	
1872	EN61198	<0,05		----	
1888	EN61198	< 0,05		----	
1890	IEC61198	0.02		-0.30	
1891	IEC61198	0.020		-0.30	
1897	IEC61198	0.022		0.03	
2122		----		----	
6015	EN61198	0.027		0.83	
6031	IEC61198	0.01969		-0.35	
6036	EN61198	0.021		-0.13	
6073	D5837	0.02450		0.43	

normality OK
n 35
outliers 1+1ex
mean (n) 0.0218
st.dev. (n) 0.00745
R(calc.) 0.0208
R(Horwitz) 0.0174

Compare R(IEC61198:93) = 0.0033
Compare R(D5837:15; liquid/liquid extr)= 0.0524 *)
Compare R(D5837:15; solid state extr)= 0.0393 *)
Compare R(D5837:15; Direct inj.)= 0.0694 *)
*) R(D5837:15) estimated on 3 times the repeatability

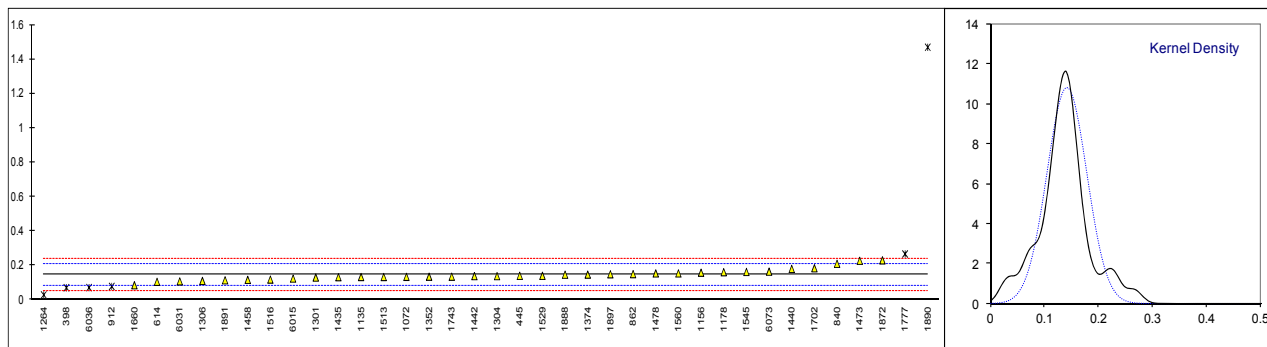


Determination of 2-Furfuryl alcohol on sample #16252; results in mg/kg

lab	method	value	mark	z(targ)	remarks
237		----		----	
398	IEC61198	0.07		-2.43	
445	EN61198	0.14		-0.18	
614	IEC61198	0.105		-1.30	
840	IEC61198	0.210	C	2.07	first reported: 0.01
862	IEC61198	0.15		0.15	
912	D5837	0.078		-2.17	
963		----		----	
1072	EN61198	0.134		-0.37	
1135	IEC61198	0.133		-0.40	
1137		----		----	
1156	IEC61198	0.158		0.40	
1178	IEC61198	0.1612		0.51	
1264	D5837	0.029		-3.74	
1301	D5837	0.130		-0.50	
1304	INH-126	0.138		-0.24	
1306	IEC61198	0.110112		-1.14	
1352	IEC61198	0.1349		-0.34	
1367		----		----	
1374	D5837	0.147		0.05	
1430	EN61198	<0.04	C	<-3.39	possibly a false negative test result? first reported: 0
1435	IEC61198	0.132		-0.43	
1440	EN61198	0.18		1.11	
1442	IEC61198	0.138		-0.24	
1458	IEC61198	0.1165		-0.93	
1473	IEC61198	0.2274		2.63	
1478	IEC61198	0.1539		0.27	
1505	D5837	<0.01		<-4.35	possibly a false negative test result?
1513	IEC61198	0.133		-0.40	
1516	IEC61198	0.118		-0.88	
1529	IEC61198	0.14		-0.18	
1545	IEC61198	0.1623		0.54	
1560	IEC61198	0.154		0.27	
1660	IEC61198	0.085		-1.94	
1702	IEC61198	0.184		1.24	
1743	IEC61198	0.135		-0.34	
1777	IEC61198	0.267		3.91	
1801		----	W	----	first reported: n.d.
1872	EN61198	0.23		2.72	
1888	EN61198	0.1466		0.04	
1890	IEC61198	1.47	R(0.01)	42.57	
1891	IEC61198	0.114		-1.01	
1897	IEC61198	0.149		0.11	
2122		----		----	
6015	EN61198	0.124		-0.69	
6031	IEC61198	0.10793		-1.21	
6036	EN61198	0.071		-2.39	
6073	D5837	0.1644		0.61	

normality not OK
n 34
outliers 6
mean (n) 0.1455
st.dev. (n) 0.03218
R(calc.) 0.0901
R(Horwitz) 0.0871

Compare R(IEC61198:93) = 0.0210
Compare R(D5837:15; liquid/liquid extr)= 0.2184 *)
Compare R(D5837:15; solid state extr)= 0.7016 *)
*) R(D5837:15) estimated on 3 times the repeatability

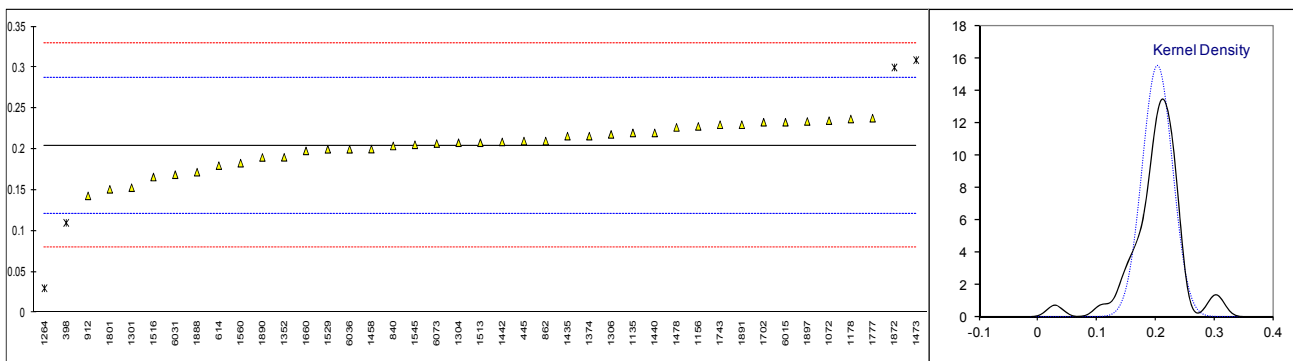


Determination of 5-Methyl-2-Furfural on sample #16252; results in mg/kg

lab	method	value	mark	z(targ)	remarks
237		----		----	
398	IEC61198	0.11	R(0.05)	-2.28	
445	EN61198	0.21		0.13	
614	IEC61198	0.180		-0.59	
840	IEC61198	0.204	C	-0.02	first reported: 0.011
862	IEC61198	0.21		0.13	
912	D5837	0.143		-1.48	
963		----		----	
1072	EN61198	0.235		0.73	
1135	IEC61198	0.220		0.37	
1137		----		----	
1156	IEC61198	0.228		0.56	
1178	IEC61198	0.2370		0.78	
1264	D5837	0.03	R(0.01)	-4.20	
1301	D5837	0.153		-1.24	
1304	INH-126	0.208		0.08	
1306	IEC61198	0.218198		0.33	
1352	IEC61198	0.1902		-0.35	
1367		----		----	
1374	D5837	0.216		0.27	
1430	EN61198	<0.03	C	<-4.20	possibly a false negative test result? first reported: 0
1435	IEC61198	0.216		0.27	
1440	EN61198	0.22		0.37	
1442	IEC61198	0.209		0.10	
1458	IEC61198	0.2001		-0.11	
1473	IEC61198	0.309	C,R(0.05)	2.51	first reported: 0.3362
1478	IEC61198	0.2267		0.53	
1505	D5837	<0.01		<-4.68	possibly a false negative test result?
1513	IEC61198	0.208		0.08	
1516	IEC61198	0.166		-0.93	
1529	IEC61198	0.20		-0.11	
1545	IEC61198	0.2055		0.02	
1560	IEC61198	0.183		-0.52	
1660	IEC61198	0.198		-0.16	
1702	IEC61198	0.233		0.68	
1743	IEC61198	0.23		0.61	
1777	IEC61198	0.238		0.80	
1801	EN61198	0.151		-1.29	
1872	EN61198	0.30	R(0.05)	2.29	
1888	EN61198	0.1720		-0.79	
1890	IEC61198	0.19		-0.35	
1891	IEC61198	0.230		0.61	
1897	IEC61198	0.234		0.71	
2122		----		----	
6015	EN61198	0.233		0.68	
6031	IEC61198	0.16890		-0.86	
6036	EN61198	0.200		-0.11	
6073	D5837	0.2070		0.06	

normality OK
n 37
outliers 4
mean (n) 0.2046
st.dev. (n) 0.02564
R(calc.) 0.0718
R(Horwitz) 0.1164

Compare R(IEC61198:93) = 0.0307
Compare R(D5837:15; liquid/liquid extr)= 0.2026 *)
Compare R(D5837:15; solid state extr)= 0.3683 *)
Compare R(D5837:15; Direct inj.)= 0.1289 *)
*) R(D5837:15) estimated on 3 times the repeatability



Determination of other Furanic compounds on sample #16252; results in mg/kg

lab	method	2-af	mark	z(targ)	5-hm-2-f	mark	z(targ)	remarks
237		----		----	----		----	
398	IEC61198	<0.05		----	<0.05		----	
445	EN61198	<0.05		----	<0.05		----	
614	IEC61198	<0.01		----	<0.01		----	
840	IEC61198	0.007		----	0.002		----	
862	IEC61198	<0.05		----	<0.05	C	----	first reported: 0.05 for 5-hm-2-f
912	D5837	0.010		----	0.030	C	----	first reported: 0.058 05 for 5-hm-2-f
963		----		----	----		----	
1072	EN61198	<0,01		----	<0,01		----	
1135	IEC61198	0.001		----	0.001		----	
1137		----		----	----		----	
1156	IEC61198	0.00		----	0.00		----	
1178	IEC61198	0.0023		----	0.0000		----	
1264	D5837	0.002		----	0.013		----	
1301	D5837	<0.01		----	<0.01		----	
1304	INH-126	<0.01		----	<0.01		----	
1306	IEC61198	<0.03		----	<0.03		----	
1352	IEC61198	nd		----	nd		----	
1367		----		----	----		----	
1374	D5837	<0.01		----	<0.01		----	
1430	EN61198	<0.01	C	----	<0.02	C	----	first reported: 0 for both comp.
1435	IEC61198	0.000		----	0.000		----	
1440	EN61198	0.00		----	0.00		----	
1442	IEC61198	0.000		----	0.000		----	
1458	IEC61198	<0.01		----	<0.01		----	
1473	IEC61198	<0.10		----	<0.1		----	
1478	IEC61198	0.00		----	0.00		----	
1505	D5837	<0.01		----	<0.01		----	
1513	IEC61198	< 0,05		----	< 0,05		----	
1516	IEC61198	<0,05		----	<0,05		----	
1529	IEC61198	< 0.05		----	< 0.05		----	
1545	IEC61198	0.0009		----	0.0076		----	
1560	IEC61198	n.d.		----	n.d.		----	
1660	IEC61198	0.00		----	0.006		----	
1702	IEC61198	not detected		----	not detected		----	
1743	IEC61198	<0.05		----	0		----	
1777	IEC61198	0		----	<0.05		----	
1801	EN61198	n.d.		----	n.d.		----	
1872	EN61198	<0,05		----	<0,05		----	
1888	EN61198	< 0,05		----	< 0,05		----	
1890	IEC61198	<0,01		----	<0,01		----	
1891	IEC61198	<0,01		----	<0,01		----	
1897	IEC61198	<0,01		----	<0,01		----	
2122		----		----	----		----	
6015	EN61198	0.003		----	0.021		----	
6031	IEC61198	0.00116		----	0.01114		----	
6036	EN61198	0		----	0		----	
6073		----		----	----		----	
n		37			37			
mean (n)		<0.05			<0.05			

Abbreviations:

2-af = 2-Acetyl Furan

5-hm-2-f = 5-Hydroxymethyl-2-Furfural

APPENDIX 2**Number of participants per country****iis16L09 (main round)**

6 labs in AUSTRALIA
 4 labs in BELGIUM
 6 labs in BULGARIA
 1 lab in CHINA, People's Republic
 1 lab in CROATIA
 1 lab in ECUADOR
 3 labs in FRANCE
 2 labs in GEORGIA
 5 labs in GERMANY
 1 lab in GREECE
 1 lab in HONG KONG
 1 lab in INDIA
 1 lab in ISRAEL
 2 labs in ITALY
 1 lab in KINGDOM OF BAHRAIN
 3 labs in MALAYSIA
 1 lab in MOROCCO
 3 labs in NETHERLANDS
 1 lab in NEW ZEALAND
 1 lab in NIGERIA
 1 lab in PAKISTAN
 1 lab in PERU
 2 labs in PORTUGAL
 3 labs in SAUDI ARABIA
 2 labs in SINGAPORE
 1 lab in SLOVENIA
 1 lab in SOUTH AFRICA
 1 lab in SOUTH KOREA
 4 labs in SPAIN
 2 labs in SWITZERLAND
 2 labs in TURKEY
 5 labs in UNITED ARAB EMIRATES
 3 labs in UNITED KINGDOM
 1 lab in UNITED STATES OF AMERICA
 2 labs in VIETNAM

iis16L09F (Furanics round)

6 labs in AUSTRALIA
 3 labs in BELGIUM
 1 lab in CANADA
 1 lab in CHINA, People's Republic
 1 lab in CROATIA
 2 labs in FRANCE
 2 labs in GERMANY
 1 lab in GREECE
 1 lab in INDIA
 2 labs in ITALY
 1 lab in KINGDOM OF BAHRAIN
 2 labs in MALAYSIA
 1 lab in MOROCCO
 1 lab in NETHERLANDS
 1 lab in NIGERIA
 1 lab in PAKISTAN
 1 lab in POLAND
 2 labs in PORTUGAL
 1 lab in SAUDI ARABIA
 1 lab in SINGAPORE
 1 lab in SLOVENIA
 1 lab in SOUTH AFRICA
 1 lab in SOUTH KOREA
 4 labs in SPAIN
 1 lab in SWITZERLAND
 1 lab in TURKEY
 3 labs in UNITED ARAB EMIRATES
 3 labs in UNITED KINGDOM
 1 lab in VIETNAM

APPENDIX 3

Abbreviations:

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

Literature:

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