Results of Proficiency Test Transformer Oil (used) November 2017

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

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1 Introduction

Since 2014, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for <u>used</u> Transformer Oil in combination with the proficiency testing scheme on the analysis of Furanics in used Transformer Oil, in accordance with the latest applicable version of the specification IEC60296 and/or ASTM D3487. During the annual proficiency testing (PT) program 2017/2018, it was decided to continue the round robin for used Transformer Oil in combination with the PT on Furanics in Transformer Oil.

In this interlaboratory study 75 laboratories from 35 different countries for the PT on used Transformer Oil registered for participation and 46 laboratories from 25 different countries for the PT on Furanics in Transformer Oil. See appendix 3 for the number of participants per PT and per country. In this report, the results of the 2017 proficiency tests on used Transformer Oil in combination with the proficiency testing scheme on the analysis of Furanics in used Transformer Oil are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. 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 #17231) and/or 1x100 ml bottle (labelled #17232) for Furanics in Transformer Oil. The 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 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 March 2017 (iis-protocol, version 3.4). 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

Approximately 120 litre of used Transformer Oil was obtained from an European supplier. After homogenisation in a pre-cleaned drum, 120 amber glass bottles of 1 litre with inner and outer caps were filled and labelled #17231. The homogeneity of the sub samples #17231 was checked by determination Density in accordance with ASTM D4052 and Water in accordance with ASTM D6304 on resp. 8 and 7 stratified randomly selected samples.

	Density at 20°C in kg/m ³	Water in mg/kg
Sample #17231-1	871.12	33.53
Sample #17231-2	871.12	31.13
Sample #17231-3	871.09	32.08
Sample #17231-4	871.12	30.97
Sample #17231-5	871.08	31.36
Sample #17231-6	871.12	32.97
Sample #17231-7	871.12	32.02
Sample #17231-8	871.09	

Table 1: homogeneity test results of subsamples #17231

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

	Density at 20°C in kg/m ³	Water in mg/kg
r (observed)	0.05	2.68
reference test method	ISO3675:98	EN60814:98
0.3 x R (ref. test method)	0.36	2.55

Table 2: evaluation of the repeatabilities of subsamples #17231

Each of the calculated repeatabilities was equal to or less than 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples #172312 was assumed.

The necessary bulk material for the PT on Furanics was prepared by mixing 700ml of used Transformer Oil, highly positive on Furanics obtained from a third party, with approx. 6.2 kg of another batch fresh Transformer Oil obtained from a local supplier. After homogenisation in a pre-cleaned can 68 amber glass bottles of 100 mL were filled and labelled #17232. The homogeneity of the subsamples #17232 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 #17232-1	875.24
Sample #17232-2	875.24
Sample #17232-3	875.25
Sample #17232-4	875.23
Sample #17232-5	875.24
Sample #17232-6	875.23
Sample #17232-7	875.24
Sample #17232-8	875.25

Table 3: homogeneity test results of subsamples #17232

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 4: evaluation of the repeatability of subsamples #17232

The calculated repeatability was less than 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #17232 was assumed.

To each of the participating laboratories, depending on the registration, 1 * 1 litre bottle, labelled #17231 and/or 1 * 100mL bottle, labelled #17232 was sent on November 1, 2017. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of used Transformer Oil, packed in 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 #17231; 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.-Also, some analytical details were requested to be reported for the Breakdown Voltage determination.

The participants were requested to determine on sample #17232; Total Furanic Compounds, 2-Acetyl Furan, 2-Furfural, 2-Furfurylalcohol, 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, 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 and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no 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 March 2017 (iis-protocol, version 3.4).

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

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve 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. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 - average of PT)} / \text{target standard deviation}
```

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. 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. For the main sample, five laboratories reported the test results after the final reporting date and one laboratory did not report any test results at all.

For the Furanic in Transformer Oil sample, seven laboratories reported the test results after the final reporting date. All participating laboratories reported test result.

Not all laboratories were able to report all analyses requested. The 74 reporting participants sent in 667 numerical test results. Observed were 36 outlying test results, which is 5.4% 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 reported test results are discussed per sample and per test. The test methods that are reported by the laboratories are taken into account for explaining the observed differences when possible and applicable. These test methods are also mentioned in the tables in appendix 1 together with the original 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.

Sample #17231

- Acidity, total (Potentiometric titration): This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of D664-A:17 nor with the requirements of EN62021-1:03.
- Acidity, total (Colorimetric titration): This determination was problematic depending on the test method used. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of D974:14e2, but not at all in agreement with the requirements of EN162021-2:07. It appears that the requirements of test method EN62021-2:07 are very strict.
- Breakdown Voltage: This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of EN60156:95. A number of analytical details were requested to be reported by the participants to try to clarify the large variation. For this sample, no conclusions could be drawn from the reported analytical details.
- <u>Density at 20°C</u>: 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 ISO3675:98.
- <u>DD-Factor:</u> This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN60247:04.

- <u>Spec. Resistance</u>: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN60247:04.
- <u>Flash Point COC</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D92:16b.
- Flash Point PMcc: 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 agreement with the requirements of ISO2719-B:16, and with the requirements of ISO2719-A:16, ASTM D93-A:16a and ASTM D93-B:16a.
- Interf. Surf. Tension: This determination was very problematic. Three 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: Till 2017 a precision statement for used oils was not present in ASTM D445. In the version of 2017 of ASTM D445 a precision statement is given for used (in-service) formulated oils. However, it appears that this reproducibility is very strict. It is decided to continue with the target reproducibility as calculated from the reproducibilities found in iis PT's on used oils (see appendix 4, ref. 17). This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the average reproducibility found for used oils in previous iis PTs (to 2015).

Water:

This determination was problematic depending on the test method used. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of EN60814:98. However, the calculated reproducibility is in good agreement with the requirements of ASTM D1533:12.

Sample #17232

Total Furanics:

This determination may not be problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the estimated requirements calculated using the Horwitz equation based on 1 component.

2-Furfural:

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 estimated requirements from the Horwitz equation.

Other Furanics: The concentrations of 2-Acetyl Furan, 2-Furfurylalcohol, 5-Hydroxymethyl-2-

Furfural and 5-Methyl-2-furfural may be near or below the detection limit.

Therefore, no significant conclusions were drawn for these 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 target reproducibilities derived from literature reference test methods (R (lit)) or based on previous iis PTs and the calculated reproducibilities (2.8 * sd) are compared in the next table:

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity, total (Potentiometric)	g KOH/kg	37	0.02	0.02	0.01
Acidity, total (Colorimetric)	g KOH/kg	32	0.02	0.02	0.04
Breakdown Voltage	kV/2.5 mm	61	29.9	19.2	19.5
Density at 20°C	kg/m³	51	871.1	1.0	1.2
Di-electric Dissipation Factor at 90°C		55	0.011	0.008	0.018
Specific Resistance at 90°C	GΩm	36	25.9	11.3	27.2
Flash Point COC	°C	21	160	12	18
Flash Point PMcc	°C	34	150	8	16
Interfacial Surface Tension	mN/m	48	26.7	4.6	2.7
Kinematic Viscosity at 40°C	mm²/s	40	10.56	0.32	0.19
Water	mg/kg	63	32.2	10.9	8.5

Table 5: reproducibilities of tests on sample #17231

Parameter	unit	n	average	2.8 * sd	R(lit) *)
Total Furanics	mg/kg	28	0.59	0.18	0.29
2-Furfural	mg/kg	44	0.57	0.21	0.28

Table 6: reproducibilities of tests on sample #17232

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

^{*)} target reproducibilities estimated from the Horwitz equation

4.3 COMPARISON OF THE NOVEMBER 2017 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2017	November 2016	November 2015	November 2014
Number of reporting labs	74	76	68	63
Number of test results reported	667	660	561	508
Statistical outliers	35	31	25	23
Percentage outliers	5.2%	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 2017	November 2016	November 2015	November 2014
Acidity, total (Potentiometric)		-	-	
Acidity, total (Colorimetric)	++	++	++	n.e.
Breakdown Voltage	+/-	+/-	++	+
Density at 20°C	+	+/-	+	-
Di-electric Dissipation Factor	++	++	++	++
Specific Resistance	++	++	++	++
Flash Point COC	++	+/-	n.e.	n.e.
Flash Point PMcc	++	+	+	+/-
Interfacial Surface Tension				
Kinematic Viscosity at 40°C		+	-	
Water	-	+/-	+/-	-
Total Furanics	++	++	+	n.e.
2-Furfural	++	+/-	-	+
2-Furfurylalcohol	n.e.	+/-	-	+
5-Methyl-2-Furfural	n.e.	+	+	++
2-Acetyl Furan	n.e.	n.e.	n.e.	n.e.
5-Hydroxy Methyl-2-Furfural	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

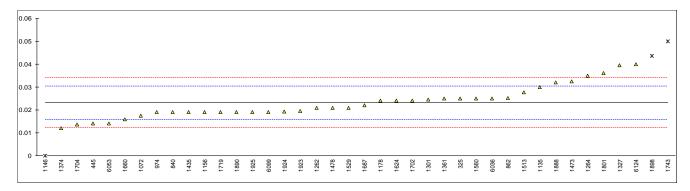
-- : group performed much worse than the reference test method

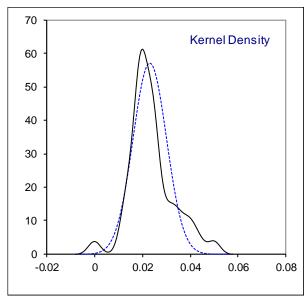
n.e: not evaluated

APPENDIX 1: Determination of Acidity, Total (Potentiometric Titration) on sample #17231; results in g KOH/kg

	•	•	•	•	Itration) on sample #17231; results in g KOH/kg
179	method D664-A	value <0.01	mark	z(targ)	remarks
225	D00+1A				
237	D664-A	<0.1			
325	D664-A	0.025		0.50	
331	D664Mod.	<0.05			
398 445	D664-A	0.014		-2.52	
511	D004-A	0.014		-2.52	
614					
840	D664-A	0.019		-1.15	
862	D664-A	0.0251		0.53	
912 913					
962					
963					
974	D664-A	0.019		-1.15	
1072	In house	0.0176		-1.53	
1135 1146	D664-A D664-A	0.03 0.000022	R(0.05)	1.88 -6.36	Reported in a different unit?
1156	EN62021-1	0.000022	K(0.03)	-1.15	Reported in a different drift!
1178	EN62021-1	0.024		0.23	
1262	D664-A	0.021		-0.60	
1264	D664-A	0.035		3.25	
1301	EN62021-1	0.0245		0.37	
1304 1306					
1327	D664-A	0.0395		4.49	
1352					
1361	EN62021-1	0.025		0.50	
1367 1374	D664-A	0.012		-3.07	
1430	D004-A	0.012		-3.07	
1435	IEC62021-1	0.019		-1.15	
1442					
1444					
1461	IEC62021 1	0.0334		2.54	
1473 1478	IEC62021-1 IEC62021-1	0.0324 0.0210		-0.60	
1513	IEC62021-1	0.0278		1.27	
1516					
1529	IEC62021-1	0.021		-0.60	
1545					
1551 1560	IEC62021-1	0.025		0.50	
1624	IEC62021-1	0.024		0.23	
1660	IEC62021-1	0.016		-1.97	
1687	D664-A	0.022		-0.32	
1702	IEC62021-1	0.024		0.23	
1704 1719	D664-A D664-A	0.01358 0.019		-2.63 -1.15	
1719	200170			-1.15	
1743	IEC62021-1	0.05	R(0.05)	7.37	
1801	EN62021-1	0.036		3.52	
1885 1888	EN62021-1	0.032		2.43	
1890	ISO6619	0.032		-1.15	
1891	-				
1897					
1898	EN62021-1	0.04361	R(0.05)	5.61	
1923 1924	EN62021-1 EN62021-1	0.0196 0.0192		-0.98 -1.09	
1924	EN62021-1 EN62021-1	0.0192		-1.09	
1943	- y - · ·				
2237					
6015	EN62024 4	0.005		0.50	
6036 6053	EN62021-1 IEC62021-1	0.025 0.014		0.50 -2.52	
6085	1L002021-1	0.014		-2.52	
6088					
6099	IEC62021-1	0.019		-1.15	
6124	IEC62021-1	0.04		4.62	
6137 6155					
6157					
6167					
					<u>Only EN62021-1</u> <u>Only ASTM D664</u>

normality	OK	OK	OK
n	37	22	13
outliers	3	2	1
mean (n)	0.0232	0.0239	0.0226
st.dev. (n)	0.00699	0.00646	0.00824
R(calc.)	0.0196	0.0181	0.0231
st.dev.(D664-A:17)	0.00364		0.00355
R(D664-A:17)	0.0102		0.0099
Compare			
R(EN62021-1:03)	0.0081	0.0084	

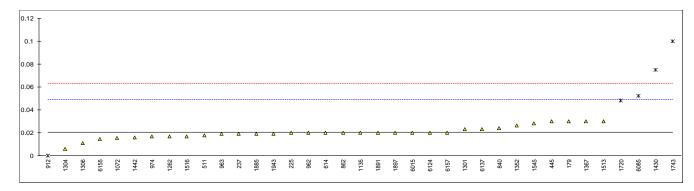


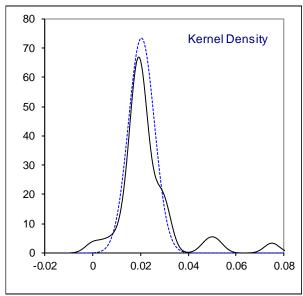


Determination of Acidity, Total (Colorimetric Titration) on sample #17231; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks		
179	D974	0.03		0.67			
225	D974	0.02		-0.03			
237	D974	0.019		-0.10			
325							
331							
398							
445	D974	0.03		0.67			
511	D974	0.0179		-0.17			
614	D974	0.02		-0.03			
840	D974	0.024		0.25			
862	D974	0.02		-0.03			
912	D974	0.000019	R(0.05)	-1.43			
913							
962	D974	0.02		-0.03			
963	D974	0.019		-0.10			
974	D974	0.017		-0.24			
1072	IEC62021-2	0.0156		-0.34			
1135	D974	0.02		-0.03			
1146							
1156							
1178	D974	0.017		-0.24			
1262 1264	דונט	0.017		-0.24			
1301	D974	0.023		0.18			
1301	INH-122	0.006		-1.01			
1306	D974	0.010772		-0.67			
1327	D014						
1352	INH-1767	0.0266		0.43			
1361							
1367	EN62021-2	0.03		0.67			
1374							
1430	EN62021-2	0.075	R(0.01)	3.82			
1435			, ,				
1442	IEC62021-2	0.016		-0.31			
1444							
1461							
1473							
1478							
1513	IEC62021-2	0.03		0.67			
1516	D974	0.017		-0.24			
1529							
1545	D974	0.028		0.53			
1551							
1560							
1624							
1660							
1687 1702							
1702							
1719							
1719	D974	0.048	R(0.01)	1.93			
1743	ISO6618	0.048	R(0.01)	5.57			
1801			(0.01)				
1885	D974	0.019		-0.10			
1888							
1890							
1891	IEC62021-2	0.02		-0.03			
1897	IEC62021-2	0.02		-0.03			
1898							
1923							
1924							
1925							
1943	ISO6618	0.019		-0.10			
2237	D074						
6015	D974	0.02		-0.03			
6036							
6053	D074	0.052	D(0.05)	2.24			
6085	D974	0.052	R(0.05)	2.21			
6088							
6099 6124	D974	0.02		-0.03			
6124 6137	EN62021-2	0.02		0.03			
6155	IEC62021-2	0.023		-0.41			
6157	IEC62021-2	0.02		-0.41			
6167	_						
-					Only D974 / ISO6618	Only EN62021-2	

normality	OK	suspect	OK
n	32	21	9
outliers	5	4	1
mean (n)	0.0204	0.0205	0.0210
st.dev. (n)	0.00544	0.00450	0.00575
R(calc.)	0.0152	0.0126	0.0161
st.dev.(D974:14e2)	0.01429	0.01429	
R(D974:14e2)	0.04	0.04	
Compare			
· R(EN62021-2:07)	0.0041		0.0041

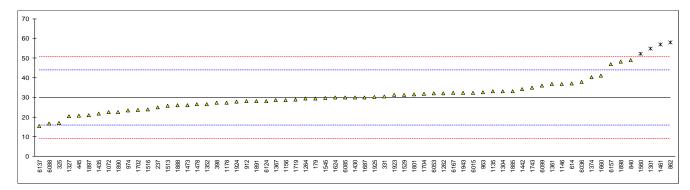


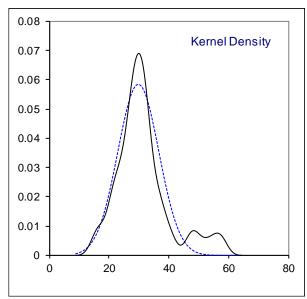


Determination of Breakdown Voltage on sample #17231, results in kV/2.5 mm

lab	method	value	mark	z(targ)	remarks	
179	D877	29.4	IIIai N	-0.07	TOTII AT TOTI	
225	5011	23.4		-0.07		
237	IEC60156	25.0		-0.71		
325	D1816	17		-1.85		
331	EN60156	30.5		0.08		
398	EN60156	27.3		-0.38		
445	IEC60156	20.7		-1.32		
511 614	IEC60156	 37.1		1.03		
840	IEC60156 EN60156	49.0		2.74		
862	IEC60156	58	R(0.05)	4.04		
912		28	(0.00)	-0.27		
913						
962						
963	IEC60156	32.7		0.40		
974 1072	EN60156	23.2 22.6		-0.96 -1.05		
1135	EN60156 EN60156	33		0.44		
1146	IEC60156	36.9		1.00		
1156	EN60156	28.7		-0.17		
1178	EN60156	27.4		-0.36		
1262	EN60156	32.1		0.31		
1264	IEC60156	29.3	D(0.05)	-0.09		
1301 1304	IEC60156	54.8	R(0.05)	3.58 0.44		
1304	INH-124	33		0.44		
1327	IEC60156	20.3		-1.38		
1352	IEC60156	26.6		-0.48		
1361	EN60156	36.9		1.00		
1367	EN60156	28.6		-0.19		
1374	IEC60156	40.2		1.48		
1430	EN60156	30 21.7		0.01		
1435 1442	IEC60156 IEC60156	34.2		-1.18 0.62		
1444	12000100					
1461	EN60156	56.8	R(0.05)	3.86		
1473	IEC60156	26.1	, ,	-0.55		
1478	IEC60156	26.5		-0.49		
1513	IEC60156	25.6		-0.62		
1516 1529	IEC60156 IEC60156	23.9 31.3		-0.86 0.20		
1545	IEC60156	29.6		-0.04		
1551	12000100					
1560	IEC60156	52.2	R(0.05)	3.20		
1624	IEC60156	29.9		0.00		
1660	IEC60156	41.0		1.59		
1687	IEC60156 IEC60156	30 23.7		0.01		
1702 1704	EN60156	31.8		-0.89 0.27		
1719	IEC60156	29		-0.13		
1720						
1743	IEC60156	35		0.73		
1801	EN60156	31.5		0.23		
1885	IEC60156	33		0.44		
1888 1890	EN60156 IEC60156	26.0 22.6		-0.56 -1.05		
1891	IEC60156	28		-0.27		
1897	IEC60156	21		-1.28		
1898	EN60156	48.2		2.63		
1923	EN60156	31.2		0.19		
1924	EN60156	27.9		-0.29		
1925	EN60156	30.1 32.4		0.03		
1943 2237	EN60156	32.4 		0.36		
6015	EN60156	32.40		0.36		
6036	EN60156	38		1.16		
6053	IEC60156	32		0.30		
6085	IEC60156	29.9		0.00		
6088	IEC60156	16.8		-1.88		
6099 6124	IEC60156 IEC60156	36.0 28.2		0.87 -0.25		
6137	EN60156	26.2 15.4		-0.25 -2.08		
6155						
6157	IEC60156	46.83333		2.43		
6167	IEC60156	32.3		0.34	Did offer during a tractic	Did not otis dusting to the c
					Did stir during testing	Did not stir during testing

normality	suspect	OK	OK
n	61	34	11
outliers	4	3	1
mean (n)	29.910	29.721	31.236
st.dev. (n)	6.8448	5.0818	5.4637
R(calc.)	19.166	14.229	15.299
st.dev.(EN60156:95)	6.9602	6.9161	7.2687
R(EN60156:95)	19.489	19.365	20.352





Determination of Density at 20°C on sample #17231; results in kg/m³

1-1	d d			-(1)	
lab	method	value	mark	z(targ)	remarks
179	D4052	871.5	С	0.90	Reported 0.8715 kg/m3
225	D4052	871.2		0.20	
237	D4052	871.1		-0.04	
325	D4052	871.245		0.30	
331	ISO12185	871.2		0.20	
398	ISO12185	870.8		-0.74	
445	D4052	871.1		-0.04	
511	D4052	871.1		-0.04	
614	ISO3675	871.2		0.20	
840	D4052	871.18		0.15	
862	D4052	871.3		0.43	
912	ISO3675	871.1		-0.04	
913	D.4050	074.0			
962	D4052	871.2		0.20	
963	D4052	871.2		0.20	
974	D4052	871.2		0.20	
1072	ISO3675	871.9	_	1.83	Departed 0.0744 kg/m2
1135	ISO3675	871.1	С	-0.04	Reported 0.8711 kg/m3
1146	D4052	871.19		0.17	
1156	10040405	070.4		2.27	
1178	ISO12185	870.1		-2.37	
1262	D4052	871.11		-0.01	
1264	D4052	871.04	_	-0.18	Denovted 0.0742 kg/m2
1301	D4052	871.2	С	0.20	Reported 0.8712 kg/m3
1304	D4052	071 1		0.04	
1306	D4052	871.1		-0.04	
1327	D4052	871.2 971.0		0.20	
1352	D7042	871.0	0	-0.27	First remarked 000 0
1361	ISO3675	870.8	С	-0.74	First reported 869.0
1367	ISO3675	871.1		-0.04	
1374	D.4050	074.0			
1430	D4052	871.0	0	-0.27	Departure 0.0744 lan/m2
1435	D7042	871.1	С	-0.04	Reported 0.8711 kg/m3
1442	D7042	871.1		-0.04	
1444	1000075	070.7			
1461	ISO3675	870.7	0	-0.97	Departured 0.07070 log/cc 2
1473	D1217	870.70	С	-0.97	Reported 0.87070 kg/m3
1478	ISO12185	870.8		-0.74	
1513	ISO12185	871.346		0.54	
1516	ISO3675	871.0		-0.27	
1529	1000075	070.4		4.07	
1545	ISO3675	870.4	C D(0.04)	-1.67	Departured 0.07FF limites
1551	ISO3675	875.5	C,R(0.01)	10.23	Reported 0.8755 kg/m3
1560					
1624	D70.40		D(0.04)	4.40	
1660	D7042	873.0	R(0.01)	4.40	
1687	ISO12185	871.13		0.03	
1702	ISO12185	871.207		0.21	
1704	ISO3675	870.5		-1.44	
1719	D40E0	074.0		0.20	
1720	D4052	871.2		0.20	
1743	1000675	871		-0.27	
1801	ISO3675	872.4		3.00	
1885	1000675	 070 F0		4.44	
1888	ISO3675	870.50		-1.44	
1890	ISO12185	871.23		0.27	
1891					
1897	10012105	971 40		0.66	
1898	ISO12185	871.40		0.66	
1923					
1924					
1925	1902675	971 2		0.43	
1943	ISO3675	871.3 		0.43	
2237	ISO12105				
6015	ISO12185	871.40		0.66	
6036	ISO3675	872		2.06	
6053	D7042	971 05			
6085	D7042	871.05		-0.15 1.16	
6088	ISO3675	870.62		-1.16	
6099	ISO12185	871.4	D(0.04)	0.66	
6124	D4052	885	R(0.01)	32.40	
6137 6155					
6155 6157					
6167					
0107					

 normality
 not OK

 n
 51

 outliers
 3

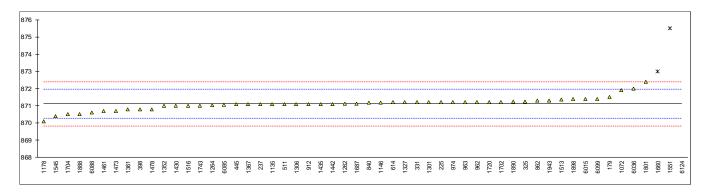
 mean (n)
 871.116

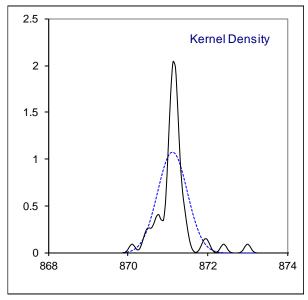
 st.dev. (n)
 0.3722

 R(calc.)
 1.042

 st.dev.(ISO3675:98)
 0.4286

 R(ISO3675:98)
 1.2

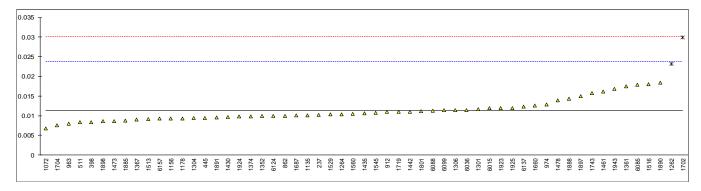


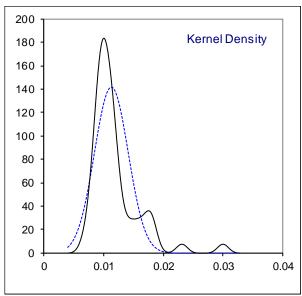


Determination of Di-electric Dissipation Factor (DDF) at 90°C on sample #17231

lah	method	value	mark	7(tara)	romarks
lab 179	method	value	mark	z(targ)	remarks
225					
237	IEC60247	0.0102		-0.17	
325					
331					
398	EN60247	0.008370		-0.46	
445 511	IEC60247	0.00947 0.0082968		-0.29 -0.47	
614		0.0062966		-0.47	
840					
862	IEC60247	0.0100		-0.20	
912	IEC60247	0.011		-0.04	
913					
962	15000015				
963	IEC60247 EN60247	0.0080 0.012849		-0.52 0.25	
974 1072	EN60247 EN60247	0.012849		-0.25	
1135	EN60247	0.0101		-0.19	
1146					
1156	EN60247	0.00925		-0.32	
1178	EN60247	0.00933		-0.31	
1262	EN60247	0.0231	R(0.01)	1.88	
1264	IEC60247	0.010394		-0.14	
1301	IEC60247	0.01171		0.07	
1304 1306	INH-125 IEC60247	0.009445 0.011395		-0.29 0.02	
1306	12000241	0.011393		0.02	
1352	IEC60247	0.00988	С	-0.22	First reported 9.88
1361	EN60247	0.017476		0.98	•
1367	EN60247	0.009057		-0.35	
1374	IEC60247	0.009868		-0.22	
1430	EN60247	0.009629		-0.26	
1435	EN60247	0.010632		-0.10	
1442 1444	IEC60247	0.01102		-0.04 	
1461	EN60247	0.01615	С	0.77	First reported 0.1615
1473	IEC60247	0.008600	O	-0.43	That reported out of the
1478	IEC60247	0.013929		0.42	
1513	IEC60247	0.00918		-0.33	
1516	IEC60247	0.01800		1.07	
1529	IEC60247	0.0103		-0.16	
1545	IEC60247	0.01070		-0.09	
1551 1560	IEC60247	0.0105		-0.12	
1624	12000247			-0.12	
1660	IEC60247	0.01264		0.22	
1687	IEC60247	0.010062		-0.19	
1702	IEC60247	0.02996	R(0.01)	2.97	
1704	EN60247	0.00757		-0.59	
1719	IEC60247	0.011		-0.04	
1720 1743	IEC60247	0.0157		0.70	
1801	EN60247	0.0157		-0.03	
1885	IEC60247	0.00880		-0.39	
1888	EN60247	0.0143		0.48	
1890	IEC60247	0.018391		1.13	
1891	IEC60247	0.0096		-0.27	
1897	IEC60247	0.015		0.59	
1898	IEC60247	0.008575		-0.43	
1923 1924	EN60247 EN60247	0.01199 0.00977		0.11 -0.24	
1924	EN60247	0.00977		0.24	
1943	EN60247	0.016878		0.89	
2237					
6015	EN60247	0.011966		0.11	
6036	EN60247	0.011422		0.02	
6053	IEC60047	0.047000		1.05	
6085 6088	IEC60247 IEC60247	0.017908 0.0112		1.05 -0.01	
6099	IEC60247	0.0112		0.01	
6124	IEC60247	0.009995		-0.20	
6137	EN60247	0.01234		0.17	
6155					
6157	IEC60247	0.009237		-0.32	
6167					

normality	suspect
n	55
outliers	2
mean (n)	0.01128
st.dev. (n)	0.002799
R(calc.)	0.00784
st.dev.(EN60247:04)	0.006295
R(EN60247:04)	0.01763
Compare	
R(D924:15)	0.02850

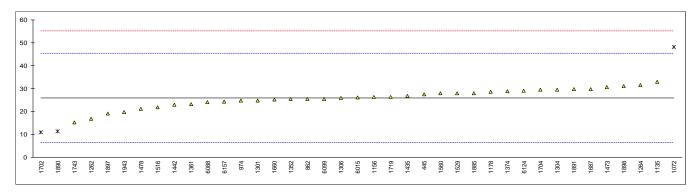


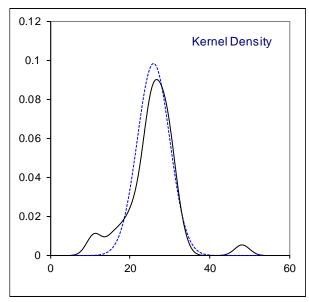


Determination of Specific Resistance at 90°C on sample #17231; results in $G\Omega m$

				4	
lab	method	value	mark	z(targ)	Remarks
179					
225 237					
325					
331					
398					
445	IEC60247	27.46		0.16	
511					
614					
840					
862	IEC60247	25.43		-0.05	
912					
913					
962					
963	EN60247	24.70		0.12	
974 1072	EN60247	24.70 48.09	R(0.01)	-0.13 2.28	
1135	EN60247	32.9	K(0.01)	0.72	
1146	LINOUZHI				
1156	EN60247	26.3		0.04	
1178	EN60247	28.7		0.29	
1262	EN60247	16.8		-0.94	
1264	IEC60247	31.50		0.57	
1301	IEC60247	24.7		-0.13	
1304	INH-125	29.565		0.38	
1306	IEC60247	25.88		0.00	
1327	IE0000 17				
1352	IEC60247	25.32		-0.06	
1361	EN60247	23.13		-0.29	
1367	IEC60247	20.72		0.20	
1374 1430	IEC60247	28.72		0.29	
1435	IEC60247	26.9		0.10	
1442	IEC60247	22.90		-0.31	
1444	12000247				
1461					
1473	IEC60247	30.70		0.49	
1478	IEC60247	21.10		-0.50	
1513					
1516	IEC60247	21.9		-0.41	
1529	IEC60247	28		0.21	
1545					
1551	IEC00047	07.0		0.00	
1560	IEC60247	27.9		0.20	
1624	IEC60247	25.3		0.06	
1660 1687	IEC60247	29.83		-0.06 0.40	
1702	IEC60247	10.83	R(0.05)	-1.55	
1704	EN60247	29.49	11(0.00)	0.37	
1719	IEC60247	26.3		0.04	
1720					
1743	IEC60247	15.3		-1.09	
1801					
1885	IEC60247	28		0.21	
1888			5 / :		
1890	IEC60247	11.37	R(0.05)	-1.50	
1891	IEC60247	29.7		0.39	
1897	IEC60247	19		-0.71	
1898 1923	IEC60247	31 		0.52	
1923					
1924					
1943	EN60247	19.68		-0.64	
2237					
6015	EN60247	26.20		0.03	
6036					
6053					
6085					
6088	IEC60247	24		-0.20	
6099	IEC60247	25.5		-0.04	
6124	IEC60247	28.98		0.31	
6137					
6155	IFC60047	24.20		0.47	
6157 6167	IEC60247	24.29		-0.17	
6167					

OK
36
3
25.9188
4.05282
11.3479
9.71953
27.2147

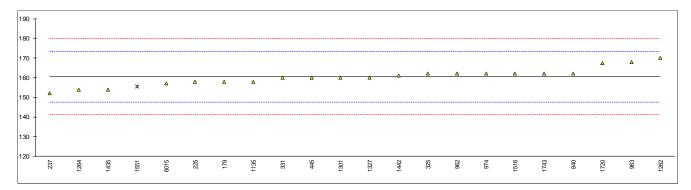


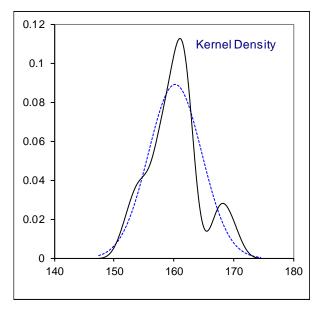


Determination of Flash Point COC on sample #17231; results in °C

lab	method	value	mark	z(targ)	remarks
			mark	-0.38	I OHIGH NO
179	D92	158			
225	D92	158		-0.38	
237	D92	152		-1.32	
325	D92	162		0.24	
331	D92	160.0		-0.07	
398					
445	D92	160		-0.07	
511					
614					
840	D92	162.1		0.26	
862					
912					
913					
962	D92	162		0.24	
963	D92	168		1.17	
974	D92	162		0.24	
1072					
1135	D92	158		-0.38	
1146					
1156					
1178					
1262	D92	170		1.48	
1264	D92	154		-1.00	
1301	D92	160.0		-0.07	
1304					
1306					
1327	D92	160		-0.07	
1352					
1361					
1367					
1374					
1430					
1435	D92	154.0		-1.00	
1442	ISO2592	161		0.08	
1444					
1461					
1473					
1478			С		First raparted 152.0
1513	D92	162	C	0.24	First reported 152.0
1516 1529	D92	102			
1545					
1551	IP523	155.5	ex	-0.77	Result excluded as test method reported is closed cup
1560	11 020		OX.		result excluded as test metrica reported is closed cap
1624					
1660					
1687					
1702					
1704					
1719					
1720	D92	167.5		1.10	
1743	ISO2592	162		0.24	
1801					
1885					
1888					
1890					
1891					
1897					
1898					
1923					
1924					
1925					
1943					
2237	ISO2592	157.0		-0.54	
6015 6036	1302392	157.0		-0.54	
6053 6085					
6088					
6099					
6124					
6137					
6155					
6157					
6167					

normality OK
n 21
outliers 0 (+1 excl)
mean (n) 160.46
st.dev. (n) 4.463
R(calc.) 12.49
st.dev.(D92:16b) 6.429
R(D92:16b) 18

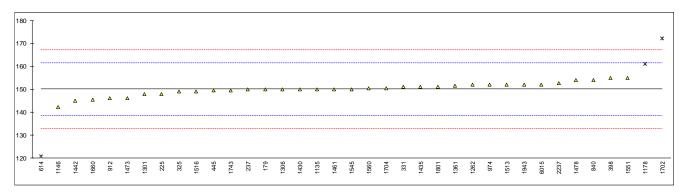


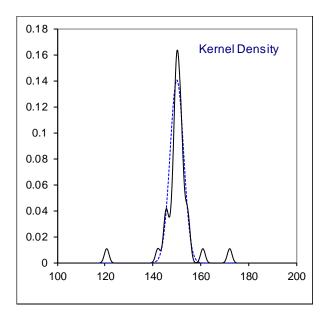


Determination of Flash Point PMcc on sample #17231; results in °C

lab	method	value	mark	z(tara)	remarks
179	D93-B	150.0	mark	z(targ) -0.02	IGHIGINS
225	D93-B D93-A	148		-0.02	
237	D93-B	150.0		-0.02	
325	D93-A	149		-0.20	
331	D93-B	151.0		0.15	
398	ISO2719-A	155.0		0.85	
445	D93-B	149.5		-0.11	
511					
614	D93-A	120.8	R(0.01)	-5.13	
840	D93-B	154.1	, ,	0.70	
862					
912	D93-A	146		-0.72	
913					
962					
963					
974	D93-A	152.0		0.33	
1072	1000740 4	450.0			
1135	ISO2719-A	150.0		-0.02	
1146	D93-A	142.2 		-1.39	
1156 1178	ISO2719-B	161.0	R(0.05)	1.90	
1262	D93-A	152.0	K(0.03)	0.33	
1264	D33 A				
1301	D93-A	148.0		-0.37	
1304					
1306	D93-A	150		-0.02	
1327					
1352					
1361	ISO2719-A	151.5		0.24	
1367					
1374					
1430	ISO2719-A	150		-0.02	
1435	D93-A	151.0		0.15	
1442	ISO2719-B	145.00		-0.90	
1444	1000740 4	450			
1461 1473	ISO2719-A D93-A	150 146.0		-0.02 -0.72	
1478	ISO2719-A	154.0		0.68	
1513	ISO2719	152.0	С	0.33	Reported first 152.0 as Flash Point COC
1516	ISO2719	149	Ü	-0.20	Troportou mot 102.0 do Flacil Folia 000
1529	.0020				
1545	ISO2719-A	150.0		-0.02	
1551	IP523	155.0		0.85	
1560	ISO2719-A	150.5		0.07	
1624					
1660	D93-A	145.5		-0.81	
1687	1000740 B	470.0	D(0.04)	2.00	
1702 1704	ISO2719-B ISO2719	172.2 150.5	R(0.01)	3.86 0.07	
1704	1302719				
1719					
1743	ISO2719-A	149.5		-0.11	
1801	ISO2719-A	151.2		0.19	
1885					
1888					
1890					
1891					
1897					
1898					
1923					
1924					
1925	ICO0740 A	450		0.22	
1943 2237	ISO2719-A ISO2719-B	152 152.7		0.33 0.45	
6015	D7236	152.7		0.43	
6036	D7230				
6053					
6085					
6088					
6099					
6124					
6137					
6155 6157					
6167					
0101					

	normality	OK
	n	34
	outliers	3
	mean (n)	150.12
	st.dev. (n)	2.830
	R(calc.)	7.93
	st.dev.(ISO2719-B:16)	5.714
	R(ISO2719-B:16)	16
Compa	are	
	R(ISO2719-a:16)	10.65
	R(D93-A:16a)	10.65
	R(D93-B:16a)	10.00

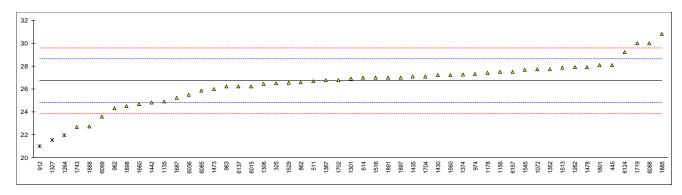


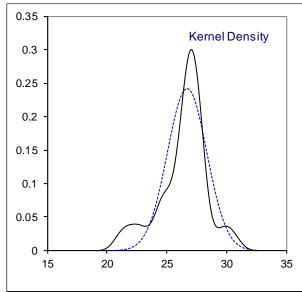


Determination of Interfacial Surface Tension on sample #17231; results in mN/m

lab	method	value	mark	z(targ)	remarks
179					
225					
237					
325	In house	26.5		-0.24	
331 398					
445	D971	28.1		1.43	
511	D971	26.69		-0.04	
614	D971	27		0.28	
840					
862	D971	26.6	C D(0.05)	-0.14	First reported 10.0
912 913	D971	21	C,R(0.05)	-6.00 	First reported 18.8
962	D971	24.3		-2.55	
963	D971	26.2		-0.56	
974	D971	27.3		0.60	
1072	D971	27.70		1.02	
1135	D971	24.9		-1.92	
1146 1156	D971	27.5		0.81	
1178	D971	27.4		0.70	
1262	D971	27.9		1.22	
1264	D971	21.942	C,R(0.05)	-5.02	First reported 21.647
1301	ISO6295	26.9	С	0.18	First reported 0.0279
1304 1306	D971	26.46		-0.28	
1327	D971	21.56	R(0.05)	-5.42	
1352	D971	27.7	(0.00)	1.02	
1361					
1367		26.75		0.02	
1374	D971	27.27		0.56	
1430 1435	D971 D971	27.2 27.1		0.49 0.39	
1442	EN14210	24.8		-2.02	
1444	-				
1461	_				
1473	D971	26.0		-0.77	
1478 1513	D971 D971	27.9 27.83		1.22 1.15	
1516	D971	27.0		0.28	
1529	D971	26.55		-0.19	
1545	D971	27.69		1.00	
1551					
1560	D971	27.2		0.49	
1624 1660	D971	24.7		-2.13	
1687	D971	25.2		-1.60	
1702	D971	26.754		0.02	
1704	D971	27.1		0.39	
1719	D2285	30		3.42	
1720 1743	D971	22.7	С	-4.22	First reported 21.8
1801	ISO6295	28.06	J	1.39	Sported 21.0
1885	D971	30.8		4.26	
1888	ISO6295	22.75		-4.17	
1890	D971	27		0.28	
1891 1897	D971	27 27		0.28	
1898	D971	24.5		-2.34	
1923					
1924					
1925					
1943 2237					
6015	D971	26.215		-0.54	
6036	D971	25.5		-1.29	
6053	D				
6085	D971	25.852	C	-0.92	First reported 24.2
6088 6099	ISO6295 EN14210	30 23.6	С	3.42 -3.28	First reported 31.3
6124	D971	29.2		2.59	
6137	D971	26.2		-0.56	
6155					
6157	EN14210	27.50		0.81	
6167					

normality	OK
n	48
outliers	3
mean (n)	26.731
st.dev. (n)	1.6493
R(calc.)	4.618
st.dev.(D971:12)	0.9547
R(D971:12)	2.673
Compare	
R(ISO6295:83)	2.673



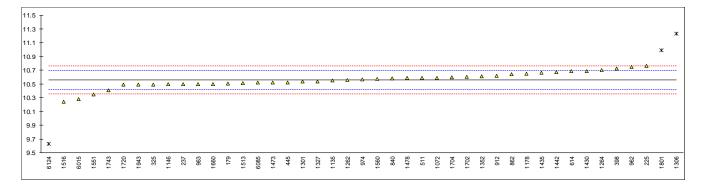


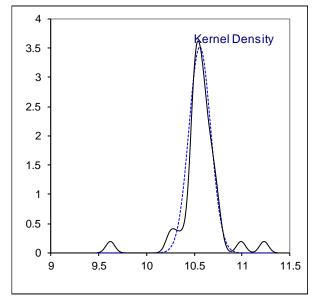
Determination of Kinematic Viscosity at 40°C on sample #17231; results in mm²/s

lab	wa a tha a d	volue	w o =1 -	=/46.00)	vamavira
lab	method D445	value	mark	z(targ)	remarks
179 225	D445 D445	10.51 10.76		-0.73 2.95	
237	D445	10.70		-0.88	
325	D445	10.4925		-0.99	
331	D-1-10				
398	D7042	10.726		2.45	
445	D445	10.52		-0.59	
511	D445	10.588		0.42	
614	D445	10.69		1.92	
840	D445	10.582		0.33	
862	D445	10.64		1.18	
912	D445	10.62	С	0.89	First reported 10.92
913	_				
962	D445	10.75		2.80	
963	D445	10.50		-0.88	
974	D445	10.57		0.15	
1072	ISO3104	10.59		0.45	
1135	ISO3104 D445	10.55 10.496		-0.14 -0.94	
1146 1156	D443	10.490		-0.94	
1178	ISO3104	10.65		1.33	
1262	D445	10.56		0.00	
1264	D7042	10.706		2.15	
1301	ISO3104	10.54		-0.29	
1304					
1306	D445	11.23	R(0.01)	9.87	
1327	D445	10.54		-0.29	
1352	D7042	10.614		0.80	
1361					
1367					
1374	1000404	40.00		4.00	
1430	ISO3104	10.69		1.92	
1435 1442	D7042 D7042	10.667 10.6730		1.58 1.67	
1444	D7042	10.0730		1.07	
1461					
1473	D445	10.5192	С	-0.60	First reported 10.1520
1478	D7042	10.587	_	0.40	
1513	ISO3104	10.514		-0.67	
1516	ISO3104	10.24		-4.71	
1529					
1545					
1551	IP71	10.3450		-3.16	
1560	ISO3104	10.5766		0.25	
1624	D7040	10 F		0.00	
1660 1687	D7042	10.5		-0.88	
1702	D7042	10.603		0.64	
1704	ISO3104	10.596		0.53	
1719					
1720	D7042	10.488		-1.06	
1743	D7279 cor D445	10.41		-2.21	
1801	ISO3104	10.99	R(0.05)	6.34	
1885					
1888					
1890					
1891					
1897					
1898 1923					
1923					
1925					
1943	ISO3104	10.49		-1.03	
2237					
6015	D7279 cor D445	10.280	С	-4.12	First reported 9.9900
6036					
6053					
6085	D7042	10.518		-0.62	
6088					
6099	D7042	0.627	D/0.04\	12 74	
6124 6137	D7042	9.627	R(0.01)	-13.74	
6155					
6157					
6167					

```
normality
                          suspect
                          40
       n
       outliers
                         3
                          10.5598
       mean (n)
                          0.11323
       st.dev. (n)
       R(calc.)
                         0.3170
                         0.06788
       st.dev.(iis)
       R(iis)
                         0.1901
Compare
       R(D445:17a)
                         0.0344
```

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

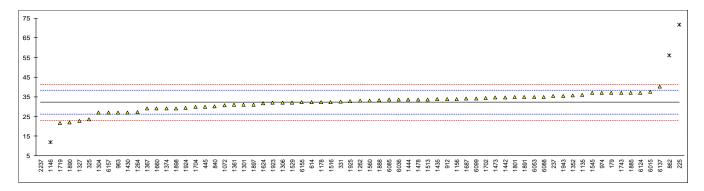


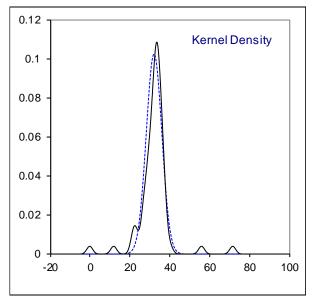


Determination of Water on sample #17231; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D6304-C	37	mun	1.57	Tomano
225	D6304-A	71.61	R(0.01)	12.95	
237	D6304-A	35.48	c` ´	1.07	First reported 83.85
325	D6304-C	23.5		-2.87	·
331	In house	32.5		0.09	
398					
445	IEC60814	30		-0.73	
511	FNOOGAA				
614	EN60814	32.4		0.05	
840 862	IEC60814 D6304-A	30.25 56	R(0.01)	-0.65 7.81	
912	D6304-A	34	K(0.01)	0.58	
913	D000+ 0				
962					
963	D1533	27		-1.72	
974	D1533	37		1.57	
1072	EN60814	30.6		-0.54	
1135	ISO12937	36	5(0.04)	1.24	
1146	D6304-C	12	R(0.01)	-6.65	
1156 1178	EN60814 EN60814	34.0 32.4		0.58 0.05	
1262	EN60814	33		0.05	
1264	D1533	27.2		-1.65	
1301	IEC60814	31		-0.41	
1304	INH-121	26.9		-1.75	
1306	D1533	32		-0.08	
1327	D6304-A	22.835		-3.09	
1352	IEC60814	35.68		1.13	
1361	EN60814	30.9		-0.44	
1367	IECC004.4	29		-1.06	
1374 1430	IEC60814 EN60814	29.2 27		-1.00 -1.72	
1435	IEC60814	34		0.58	
1442	IEC60814	34.6		0.78	
1444	IEC60814	33.6574		0.47	
1461					
1473	D1533	34.55		0.76	
1478	IEC60814	33.7		0.48	
1513	IEC60814	33.72		0.49	
1516	IEC60814	32.4		0.05	
1529 1545	IEC60814 IEC60814	32.1 36.97		-0.04 1.56	
1551	IP74	<1000			
1560	IEC60814	33		0.25	
1624	IEC60814	31.88		-0.12	
1660	IEC60814	29		-1.06	
1687	IEC60814	34.142		0.63	
1702	IEC60814	34.5		0.75	
1704	EN60814	29.8		-0.80	
1719 1720	IEC60814	21.8		-3.43	
1720	IEC60814	37		1.57	
1801	IEC60814	35		0.91	
1885	D1533	37		1.57	
1888	EN60814	33.25		0.33	
1890	IEC60814	21.95		-3.38	
1891	IEC60814	35		0.91	
1897	IEC60814	31		-0.41	
1898	EN60814	29.2		-1.00	
1923 1924	EN60814 EN60814	31.9 29.3		-0.11 -0.96	
1924	EN60814 EN60814	32.9		0.22	
1943	EN60814	35.57		1.10	
2237	DIN51777-1	0.01	C,R(0.01)	-10.59	First reported 0
6015	DIN51777	37.50	., (5.5.)	1.73	• • • • • • • • • • • • • • • • • • • •
6036	EN60814	33.5		0.42	
6053	IEC60814	35		0.91	
6085	IEC60814	33.4867		0.41	
6088	D1533	35		0.91	
6099	IEC60814	34.2		0.65	
6124 6137	IEC60814 EN60814	37 40.16		1.57 2.61	
6155	D1533	32.2		-0.01	
6157	EN60814	26.90		-1.75	
6167					

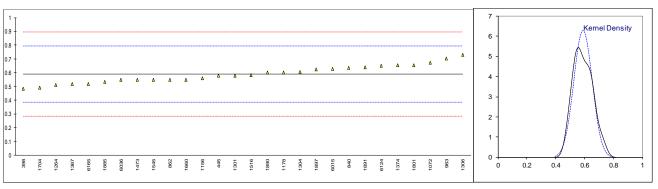
	normality	OK
	n	63
	outliers	4
	mean (n)	32.233
	st.dev. (n)	3.8877
	R(calc.)	10.886
	st.dev.(EN60814:98)	3.0415
	R(EN60814:98)	8.516
Compar	e .	
	R(D1533:12)	14





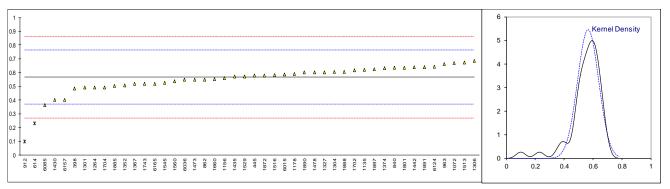
Determination of Total Furanics on sample #17232; results in mg/kg

lab	method	value	mark	z(targ)	remarks
398	EN61198	0.484		-1.03	
445	IEC61198	0.579		-0.10	
614	IEC61198	<0.01		<-5.67	False negative test result?
840	D5837	0.634		0.43	•
862	IEC61198	0.55		-0.39	
912					
963	D5837	0.705		1.13	
1072	EN61198	0.673		0.82	
1135	=110.440				
1156	EN61198	0.559		-0.30	
1178	EN61198	0.6017		0.12	
1264	D5837 IEC61198	0.511 0.58	С	-0.77	First reported 0.027
1301 1304	INH-126	0.6035	C	-0.09 0.14	First reported 0.037
1304	IEC61198	0.7297159		1.37	
1327	12001130				
1352					
1367	EN61198	0.52		-0.68	
1374	D5837	0.656		0.65	
1430					
1435					
1442					
1473	IEC61198	0.5492	С	-0.40	First reported 0.7078
1478					
1513					
1516	IEC61198	0.582		-0.07	
1529	IECC1400	0.5400		0.00	
1545	IEC61198	0.5498		-0.39	
1560 1660	IEC61198	0.55		-0.39	
1702	12001190	0.55		-0.59	
1704	IEC61198	0.49265		-0.95	
1743	12001100				
1801	EN61198	0.656	С	0.65	First reported 0.00
1872					'
1885	D5837	0.534		-0.54	
1888					
1890	IEC61198	0.60		0.10	
1891	IEC61198	0.64		0.49	
1897	IEC61198	0.625		0.35	
6015	EN61198	0.627		0.37	
6036	EN61198	0.549		-0.40	
6085 6124	IEC61108	0.6495		0.59	
6124 6157	IEC61198	0.6495		0.59	
6165	IEC61198	0.52		-0.68	
0100	12301100	0.02		0.00	
	normality	OK			
	n	28			
	outliers	0			
	mean (n)	0.5896			
	st.dev. (n)	0.06343			
	R(calc.)	0.1776			
	st.dev.(Horwitz 1 comp)	0.10215			
•	R(Horwitz 1 comp)	0.2860			
Compa	D(IEC64400:02)	0.0640			
	R(IEC61198:93)	0.0649			



Determination of 2-Furfural on sample #17232; results in mg/kg

lab	method	value	mark	z(targ)	remarks
398	EN61198	0.484	man	-0.84	Tomario
445	IEC61198	0.579		0.12	
614	IEC61198	0.23	R(0.01)	-3.41	
840	D5837	0.634	(5.5.)	0.67	
862	IEC61198	0.55		-0.17	
912	IEC61198	0.100	R(0.01)	-4.73	
963	D5837	0.663	, ,	0.97	
1072	EN61198	0.670		1.04	
1135	IEC61198	0.62	С	0.53	First reported 598
1156	EN61198	0.559		-0.08	
1178	EN61198	0.5914		0.24	
1264	D5837	0.491		-0.77	
1301	IEC61198	0.49	С	-0.78	First reported 0.037
1304	INH-126	0.6035		0.37	
1306	IEC61198	0.683689		1.18	
1327	IEC61198	0.6019		0.35	
1352	IEC61198	0.5064		-0.62	
1367	EN61198	0.52		-0.48	
1374	D5837	0.633		0.66	
1430	EN61198	0.4		-1.69	
1435	IEC61198	0.57		0.03	
1442	IEC61198	0.64		0.74	
1473	IEC61198	0.5492		-0.18	
1478 1513	IEC61198 IEC61198	0.601 0.6735		0.34 1.07	
1516	IEC61198	0.6733		0.15	
1529	IEC61198	0.562		0.13	
1545	IEC61198	0.5240		-0.44	
1560	IEC61198	0.537		-0.31	
1660	IEC61198	0.552		-0.15	
1702	IEC61198	0.615		0.48	
1704	IEC61198	0.49265		-0.76	
1743	IEC61198	0.52		-0.48	
1801	EN61198	0.635		0.68	
1872	IEC61198	0.58		0.13	
1885	D5837	0.504		-0.64	
1888	EN61198	0.606		0.39	
1890	IEC61198	0.60		0.33	
1891	IEC61198	0.64		0.74	
1897	IEC61198	0.625		0.58	
6015	EN61198	0.588		0.21	
6036	EN61198	0.549		-0.19	
6085	In house	0.364		-2.06	
6124	IEC61198	0.6435		0.77	
6157	IEC64400	0.40		-1.69	
6165	IEC61198	0.52		-0.48	
	normality	OK			
	n	44			
	outliers	2			
	mean (n)	0.5673			
	st.dev. (n)	0.07321			
	R(calc.)	0.2050			
	st.dev.(Horwitz)	0.09885			
	R(Horwitz)	0.2768			
Compa	are				
	R(IEC61198:93)	0.0851			



Determination of other Furanic compounds on sample #17232; results in mg/kg

leb	moth od	2 of	2.60	Ehm 2 f	E 2 f
lab	method	2-af	2-fa	5-hm-2-f	5-m-2-f
398	EN61198	<0,05	<0,05	<0,05	<0,05
445	IEC61198	<0.05	<0.05	<0.05	<0.05
614	IEC61198	1.40	<0.01	<0.01	<0.01
840	D5837	<0.01	<0.01	<0.01	<0.01
862	IEC61198	<0.05	<0.05	<0.05	< 0.05
912	IEC61198	0.022	0.052	0.032	0.084
963	D5837	<0.05	<0.05	0.036	0.006
1072	EN61198	<0,01	<0,01	<0,01	<0,01
1135	IEC61198	0.0008	0.0078	<1	0.0038
1156	EN61198	0	0	0	0
1178	EN61198	0.0004	0.0028	0.0047	0.0024
1264	D5837	Nil	Nil	0.02	Nil
1301	IEC61198	<0.01	<0.01	0.09	<0.01
1304	INH-126	< 0.01	<0.01	<0.01	<0.01
1306	IEC61198	0.0116989	0.0343280	<0.01	< 0.01
1327					
1352	IEC61198	< 0.0500	0.0707	< 0.0500	< 0.0500
1367	EN61198	0.00	0.00	0.00	0.00
1374	D5837	0	0	0.023	0
1430	EN61198	0.14	<0.04	< 0.02	< 0.03
1435	IEC61198	0.000	0.000	0.000	0.000
1442	IEC61198	<0,05	<0,05	<0,05	<0,05
1473	IEC61198	<0.1	<0.1	<0.1	<0.1
1478	IEC61198	0.00	0.011	0.032	0.00
1513	IEC61198	< 0.05	<0.05	<0.05	< 0.05
1516	IEC61198	<0.05	<0.05	<0.05	<0.05
1529	IEC61198	0.00	0.00	0.03	0.00
1545	IEC61198	0.0000	0.0055	0.0204	0.0000
1560	IEC61198	Not detected	Not detected	0.02	Not detected
1660	IEC61198	0.000	0.000	0.000	0.010
1702	IEC61198	Not detected	Not detected	not detected	not detected
1704	IEC61198	0	0	0	0
1743	IEC61198	< 0.05	<0.05	<0.05	< 0.05
1801	EN61198	0.00		N.D.	0.0246
1872	IEC61198	<0.05	<0.05	<0,05	<0.05
1885	D5837	<0.001	<0.001	0.034	<0.001
1888	EN61198	<0,05	<0,05	<0,05	<0,05
1890	IEC61198	0	0	0	0
1891	IEC61198	<0.01	<0.01	<0.01	<0.01
1897	IEC61198	<0.01	<0.01	<0.01	<0.01
6015	EN61198	< 0.05	< 0.05	0.040	< 0.05
6036	EN61198	0	0	0	0
6085	In house	0	ő	0	0
6124	IEC61198	0	0.0015	0.0005	0.004
6157	001100				
6165	IEC61198	0.00	0.00	0.00	0.00
5.00			1	1	1

Abbreviations:

= 2-Acetyl Furan 2-af 2-fa = z-1 a... 5-hm-2-f = 5-Hydroxymetnyı-z = 5-Methyl-2-Furfural

= 2-Furfurylalcohol = 5-Hydroxymethyl-2-Furfural

APPENDIX 2

Analytical details of the determination: Breakdown Voltage

			3. The determination				
lab	1. What is the brand of your test equipment?	2. What type of electrodes were used?	was started after	during the	5. temperature of sample during the determination?	6. How did you prepare the sample before filling the test cell?	Any remark
179							
225							
237							
325	Foster OTS 100 AF	VDE Electrodes	Automatically device according ASTM D1816	Yes	Room Temperature (between 20° to 30°C)	Cell cleaning - drye at 60°C - 5 min.	
331	MEGGER	SPHERICAL	3	Yes	23	SHAKING	
398	BAUR		Immediately	Yes	24.6	It shakes softly preventing formig of air's bubbles	
445	Megger	Spherical, Icm(?) diameter	5 minutes	Yes	20	Washed with heptane, dried. Conditioned with sample, emptied & refilled	No
511	BAUR / DPA 75C		Immediately after fill the test cell	Yes	20°C	We homogenize the sample before introducing it into the analysis cell.	The readings of breakdown voltage equipment are very variable
614	Megger	partially spherical electrodes	5 minutes	Yes	22.4 °C	no preparation	
840	BA100 - Portable Breakdown Analyzer for Oil Testing	GB0055 Mushroom	5 min	Yes	30 deg C	Clean outside the bottle of sample. Test Breakdown Voltage at the first.	
862	BAUR	brass polished, partially spherical of shape(13mm diameter)	5 min	No	20°C	The sample container is gently agitated and turn over without causing the formation of air bubbles	
912	DAOIX	diameter)	3 111111		20 0	Over without causing the formation of all bubbles	
913							
962							
		2.5 mm spherical	_		_	agitated & turned over several times to make	
963	Baur DTA 100 C	electrodes	2 min	No	21°C	homogeneous	
974	BAUR OIL TESTER DTA	Spherical Electrode	5 MINUTES	Yes	24.0	Prior starting the test sample was inspected for the presence of water, sludge & any foriegn matter	If the free water is observed the test for BDV shoul be waived
1072	BAUR	EN60156	5	Yes	20°C±5	SHAKE AND HOMOGENIZE THE SAMPLE	
1135							
1146	Megger Foster OTS80 AF/2	Bol electroden	circa 15 minuten	Yes	20	15minuten schudden hierna 2uur rusten	
1156	Baur	VDE	5	No	20,9	the bottle was turn over several times	
1178	Megger OTS100AF	36 mm height mushroom	5 min	Yes	22	Sample was conditioned to room temperature and homogenized by turning of bottle	
1262	BAUR DTA 100C	IEC 60156, fig. II	5 min	Yes	25°C	tempered at room temperature and fill the cell	no
1264	BAUR DPA 75C	IEC 60156 Figure-II	10 Minutes	Yes	22	NA	NA
1301							
1304	Foster	Small round balls	3	Yes	19-22	Gentle turning of the sample bottle to mix sample without adding air bubbles	

lab	1. What is the brand of your test equipment?	2. What type of electrodes were used?	3. The determination was started after test cell was filled in mintes?	during the	5. temperature of sample during the determination?	6. How did you prepare the sample before filling the test cell?	Any remark
1306							
1327				No			
	Megger Foster	Partially spheriical		- 119		Mix the sample well then pour the sample into the	
1352	OTS 100 AF/2	electrodes	4 min	No	22°C	test cell.	
						The bottle was brought to ambient temperature	
1361	Baur	Brass electrodes	5	Yes	25	and gently shaken before filling the cell.	
1367	MEGGER	SFERICAL	5 MINUTES	Yes	21	BOTTLE AGITATION	
1374	Baur	Mushroom shaped	automatically starts with a 5 minute wait before testing.	Yes	20	Drain previous oil sample, rinsed with new sample and drained then sample introduced into cell	20
1430	BAUR	sferical	5	Yes	22		
1435							6 measurments SD:3.7 kV
1442	Baur	spherical electrodes accoreding IEC 60156	5 Minutes according IEC 60156	Yes	21,1	gently agitated and turn the bottle, fill without bubbles	
1444		3			,		
1461							
1473	Magger	Mushroom Electrodes	5 minutes	Yes	20	shaking gently	RSD% was high (12 %)
	33	partially spherical				3 9 ,	J 12 202 J (22)
1478	BAUR DTA100	electrodes	10 minutes	Yes	25C	we turn sample bottle over 3 times	
1513 1516	Baur	Spherical electrodes	5	Yes	21	Gently agitated and turned over several times	The six individuals measures were: 33,9; 24,9;27,0;20,5;22,8 and 24,5
1529	Bauer	Spherical electrodes	10 min	Yes	20°C	homogenize sample without introducing air bubbles	
1545	Megger 80 kV	Mushroom Type	1 min	Yes	25	Shake	
1551		-					
1560	Megger	IEC60156	5 Minutes	Yes	24	As per IEC 60156	-
1624	Baur DTA 100C			Yes	22	gently turn around	
1660	B2 Electronics	VDE partially spherical electrodes acc. to IEC	5	No	25	Gentle shaking (rolling)	
1687	EA-Electronics	60156, Fig. 2	12	Yes	23,3°C	Homogenization by gentle rotation of the bottle.	
1702							
1704							
1719 1720	Megger	round	5 mins	Yes	21	invert sample once, rinse empty cell then fill to clean empty cell	n/a
1743	BAUR	spherical electrodes		No	21	The sample is mixt before filling the test cell	
1801						, , , , , , , , , , , , , , , , , , ,	
1885	Megger	Mushroom	1	No	22	nothing	no
1888	BAUR DPA75	SPHERE BRONZE	20 MIN	Yes	24.6	AS DESCRIBED IN THE STANDARD	NO REMARK
1890	baur DTA 100C	VDE	10min	Yes	19°C	homogonise on rolling equipment	

lab	1. What is the brand of your test equipment?	2. What type of electrodes were used?	3. The determination was started after test cell was filled in mintes?	during the	5. temperature of sample during the determination?	filling the test cell?	Any remark
		Brass mushroom shape				Gently invert bottle several times before rinsing	A new, better designed test is
1891	Baur DTA 100C	electrodes	5 min	Yes	22°C	electrodes and cell prior to filling	needed
		Brass mushroom shaped				Gently invert bottle several times before rinsing	A new, better designed test is
1897	Baur DTA 100C	electrodes	5 min	Yes	22°C	electrodes and cell prior to filling	needed
						After we turned the sample over, we filled the	
1898	Schleich	Mushro0m	15	Yes	20	sample bowl.	
	OTS 80AF/2					The sample equilibrate to ambient temperature.	•
1923	MEGGER	Mushroom electrodes	5 (five) minutes	Yes	22,5°C	Before filling we gently invert the sample container.	22,5°C
1924	MEGGER®, FOSTER, AVO INTERNATIONAL - OTS 80AF/2	mushroom-shape electrodes	1	No	24	The bottle with the sample was shacked softly and the oil was spilled slowly in the test cell	24
4005	BAUR OIL Tester	Marshara and also days days	F (Co.)	NI-	00.000	The sample equilibrate to ambient temperature.	23.2°C
1925	DTA 100	Mushroom electrodes Partially spheric electrodes	5 (five) minutes	No	23.2°C	Before filling we gently invert the sample container	23.2A°C
1943 2237	DPA 75 (BAUR) Metrohm	(r=25mm, diameter=36mm, gap between electrodes=2.50mm).	5 minutes.	Yes Yes	23.8-25.0 C.	Test is carried out without drying or degassing, at the room temperature. The sample was shaken	
2231	Metronin	multi electrod	1 min	res	7.0	The sample was shaken	A
6015	Baur	polished round electrodes, made of brass	5	Yes	25	The closed bottle was moved and turned softly several times without creating bubbles.	Automatic measuring program including stirring and waiting times.
6036	Baur DTA 100 C	spherical	30	Yes	20	Just pour in, no agitation or turning over of bottle	
6053	Megger		1	Yes	25		
6085	BAUR DTA E	PARTIALLY SPHERICAL	5	Yes	24	THE SAMPLE CONTAINER WAS GENTLY AGITATED & TURNED OVER SEVERAL TIMES	MEAN VALUE OUT OF 6 MEASUREMENTS
6088							
6099	Baur DTA 100 C	Spherical (figure 1 IEC60156)	5 minutes	No	21°C	shaked	
6124	BAUR	Partially Spherical of the shape and dinmentions descibed at IEC 60156/95 pag 20 fig 2.	5 min.	Yes	24°C	Tempering the sample to the conditions of the lab. and rotating slowly to homegeinize it.	If the S/M value doesn 't fit whit IEC 60156/95 fig 3, the test is repeated once more.
6137							
6155 6157	Foster OTS60	Spherical 2.5mm gap	at least 15	Yes	n/a	Sample was agitated to ensure a homogeneous representative sample	Some fine particles are visible in the sample
6167	FOSTER (AVO) MEGGER, UK	2.5 mm Gap Spheres	5	No	23	Clean dried test cell is rinced with a portion of the sample liquid before filling the sample	No

APPENDIX 3

Number of participants per country

iis17L10 (main round)

6 labs in AUSTRALIA 4 labs in BELGIUM 6 labs in BULGARIA

2 labs in CHINA, People's Republic

1 lab in COTE D'IVOIRE 1 lab in CROATIA 2 labs in FRANCE

5 labs in GERMANY 1 lab in GREECE

3 labs in INDIA 2 labs in ITALY

1 lab in KINGDOM OF BAHRAIN

1 lab in KUWAIT
1 lab in LATVIA
2 labs in MALAYSIA
1 lab in MOROCCO
3 labs in NETHERLANDS
1 lab in NEW ZEALAND

1 lab in NIGERIA
1 lab in PERU
2 labs in PORTUGAL
2 labs in SAUDI ARABIA

2 labs in SINGAPORE
1 lab in SLOVENIA
1 lab in SOUTH AFRICA

1 lab in SOUTH KOREA

5 labs in SPAIN
1 lab in SUDAN
2 labs in SWITZERLAND

1 lab in TURKEY 5 labs in UNITED ARAB EMIRATES

4 labs in UNITED KINGDOM

1 lab in UNITED STATES OF AMERICA

1 lab in URUGUAY 1 lab in VIETNAM

iis17L10F (Furanics round)

6 labs in AUSTRALIA 3 labs in BELGIUM

2 labs in CHINA, People's Republic

1 lab in CROATIA
1 lab in FRANCE
2 labs in GERMANY
1 lab in GREECE
1 lab in INDIA
2 labs in ITALY

2 labs in MALAYSIA

1 lab in KINGDOM OF BAHRAIN

1 lab in MOROCCO
1 lab in NETHERLANDS
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

4 labs in SPAIN
1 lab in SWITZERLAND

3 labs in UNITED ARAB EMIRATES

4 labs in UNITED KINGDOM

1 lab in URUGUAY

1 lab in VIETNAM

APPENDIX 4

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 D(0.01) = outlier in Grubbs' outlier test D(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 of 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

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