Results of Proficiency Test Transformer Oil (fresh) November 2018

Organised by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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1 Introduction

Since 2001, the Institute for Interlaboratory Studies organizes a proficiency test for the analysis of Transformer Oil (fresh) every year. During the annual proficiency testing program 2018/2019, it was decided to continue the round robin for Transformer Oil (fresh) in accordance with the latest applicable version of the specification IEC60296 and ASTM D3487. In this interlaboratory study, 52 laboratories from 27 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2018 Transformer Oil (fresh) proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send a bottle of 1 litre of Transformer Oil (fresh), labelled #18230. Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/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 June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from 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 was obtained from a local supplier. The 70 litre bulk material was spiked with approximately 100 grams of DBPC and homogenised in a pre-cleaned drum. After homogenisation, 69 subsamples were transferred to 1 litre amber glass bottles and labelled #18230. The homogeneity of the subsamples #18230 was checked by determination of Density in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density at 20°C in kg/m ³
Sample #18230-1	876.47
Sample #18230-2	876.47
Sample #18230-3	876.47
Sample #18230-4	876.47
Sample #18230-5	876.47
Sample #18230-6	876.47
Sample #18230-7	876.47
Sample #18230-8	876.47

Table 1: homogeneity test results of subsamples #18230

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 (sample #18230)	0.00
reference test method	ISO3675:98
0.3 x R (ref. test method)	0.36

Table 2: evaluation of the repeatability of subsamples #18230

The calculated repeatability on sample #18230 was less than 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #18230 was assumed.

To each of the participating laboratories, 1*1 litre bottle (labelled #18230) was sent on October 31, 2018. An SDS was added to the sample package.

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 requested to determine tests mentioned in either ASTM D3487 or IEC 60296 on sample #18230: Acidity (Total, both potentiometric and colorimetric), Appearance, Breakdown Voltage, Color ASTM, Density at 20°C, Di-electric loss at 90°C (Di-electric Dissipation Factor and Specific Resistance), Flash Point (both COC and PMcc), Interfacial Surface Tension, Kinematic Viscosity at 40°C, Water and additives (DBP and DBPC, DBDS, BTA and Irgamet 39). Also some extra questions regarding the Breakdown Voltage determination were requested.

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

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate 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 reanalyses). Additional or corrected test results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, June 2018 version 3.5).

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgment 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.

In accordance to ISO 5725 the original test results per determination were submitted subsequently to Dixon's, Grubbs' and 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 the averages and the standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1, was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report. Finally, the reproducibilities were calculated from the standard deviations by multiplying 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 each determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected 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 as 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 in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used.

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

The z-scores were calculated in accordance with:

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. 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, no major problems were encountered with the dispatch of the samples. Two participants reported the results after the final reporting date and two participants did not report any test results at all. Not all participants were able to report results for all analyses requested.

In total 50 participants reported 371 numerical results. Observed were 24 outlying results, which is 6.5% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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, see also paragraph 3.1.

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The specified test methods and requirements were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 4.

Acidity, Potentiometric: No significant conclusions were drawn as the Acidity was below the quantification limit (0.014 g KOH/kg) of the test method EN62021-1:03.

Acidity, Colorimetric: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after the rejection of the statistical outliers is in agreement with the requirements of ASTM D974:14e2.

Appearance: All reporting laboratories agreed on the appearance of the oil being 'Bright and Clear' or remarked in similar words to this.

<u>Color ASTM</u>: The majority of the reporting laboratories agreed that the color was lower than 0.5.

<u>Breakdown Voltage</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN60156:95.

Some analytical details were requested when reporting the test result (see appendix 2). In the previous PT, a significant difference in consensus value was observed between stirring and not stirring during the test. In this PT, the difference was smaller and not significant.

The reproducibility of EN60156:95 was determined from Figure 3 of method EN60156:95, according to the iis memo of December 2017.

<u>Density at 20°C</u>: This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO3675:98.

<u>DD-Factor:</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>Spec. Resistance</u>: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility was not at all in agreement with the requirements of EN60247:04.

Please note that it is well known that specific resistance of new oils can vary over a wide range. This is due to randomly tiny amounts of impurities (maybe present in the air or in the test cell) which can dramatically change the value. In used oils, however, due to already present ion flow of the polar compounds, these problems are not observed.

- <u>Flash Point (COC)</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 ASTM D92:18.
- <u>Flash Point (PMcc)</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO2719-A:16.
- Interf. Surf. Tension: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D971:12.
- <u>Kinematic Viscosity:</u> This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is full in agreement with the requirements of ASTM D445:18, but not in agreement with the strict requirements of ISO3104:94.

<u>Water</u>: This determination was problematic for a number of laboratories. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of EN60814:98.

DBPC: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of IEC60666:10.

The average recovery of DBPC (theoretical increment of 0.177 mg DBPC/kg)

may be good: "less than 100%" (the actual blank DBPC content is not known).

Other anti-oxidant additives: The majority of the participants agreed that DBP (2.6-Di-tertiary-butyl phenol), DBDS (Dibenzyldisulphide), BTA (Benzotriazole) and Irgamet 39 were not present in the sample.

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 number of significant test results, the average result, calculated reproducibilities (2.8 * standard deviation) and reproducibilities, derived from test methods (in casu ASTM, ISO, EN and IEC test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity, Total (Potentiometric Titration)	g KOH/kg	22	0.007	0.015	(0.002)*
Acidity, Total (Colorimetric Titration)	g KOH/kg	15	0.009	0.020	0.04
Appearance		30	B&C	n.a.	n.a.
Color ASTM		38	<0.5	n.a.	n.a.
Breakdown Voltage	kV/2.5 mm	45	46.7	36.5	33.6
Density at 20°C	kg/m³	33	876.51	0.50	1.2
Di-electric Dissipation Factor at 90°C		33	0.0007	0.0010	0.0015
Specific Resistance at 90°C	$G\Omegam$	25	939.3	1812.9	986.3
Flash Point (COC)	°C	18	159.1	12.4	18
Flash Point (PMcc)	°C	31	151.8	7.6	10.8
Interfacial Surface Tension	mN/m	35	47.8	4.8	4.8
Kinematic Viscosity at 40°C	mm²/s	31	9.85	0.12	0.12
Water	mg/kg	41	22.7	7.2	7.2
DBPC Antioxidant Additive	%M/M	18	0.18	0.05	0.08

Table 3: reproducibilities of tests on sample #18230

Without further statistical calculations, it can be concluded that for most 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

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2018 WITH PREVIOUS PTs.

	November 2018	November 2017	November 2016	November 2015	November 2014
Number of reporting labs	50	55	51	49	52
Number of results reported	371	405	383	330	340
Statistical outliers	24	18	29	26	13
Percentage outliers	6.5%	4.4%	7.6%	7.9%	3.8%

Table 4: comparison with previous proficiency tests

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

 $^{()^*}$ = Results were near or below detection limit, these results should be used with care

B&C = Bright and Clear

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

Parameter	November 2018	November 2017	November 2016	November 2015	November 2014
Acidity, Total (Potentiometric)	()	()	()	()	()
Acidity, Total (Colorimetric)	++	++	++	n.e.	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	+/-	-	+/-	-	-
DBPC Antioxidant Additive	+	+/-	n.e.	n.e.	n.e.

Table 5: 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:

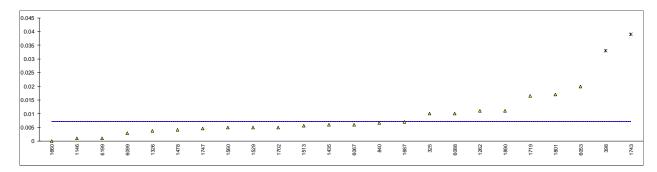
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- : group performed worse than the reference test method
- --: group performed much worse than the reference test method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

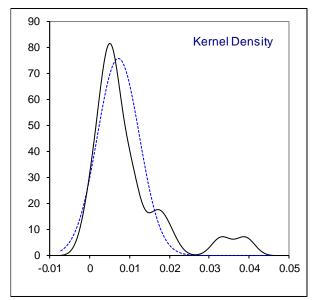
^{() =} Results between brackets were near or below detection limit, these results should be used with care

APPENDIX 1

Determination of Acidity, Total (Potentiometric Titration) on sample #18230; results in g KOH/kg

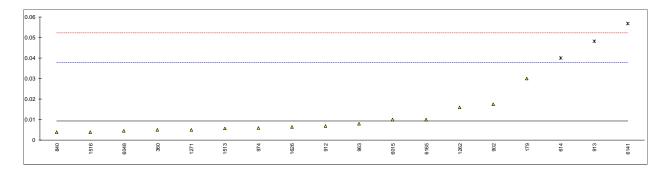
					ition) on sample #18230; results in g KOH/kg
173	method	value	mark	z(targ)	remarks
173	D664-A	<0.01			
325	D664-A	0.01			
360					
398	EN62021-1	0.033	R(0.01)		possibly a false positive test result?
446			, ,		
614					
840	IEC62021-1	0.0066			
862	D664-A	<0.1			
902					
912 913					
962					
963					
974					
1146	D664-A	0.001			
1262	EN62021-1	0.011			
1271					
1304					
1326	IEC62021-1	0.0038			
1435	IEC62021-1	0.006			
1442					
1444 1460					
1460					
1478	IEC62021-1	0.0041			
1513	IEC62021-1	0.0057			
1516					
1529	IEC62021-1	0.005			
1560	IEC62021-1	0.005			
1626					
1660	IEC62021-1	0.00			
1687	D664-A	0.007			
1702 1719	IEC62021-1 D664-A	0.005 0.0165			
1713	IEC62021-1	0.0103	R(0.01)		possibly a false positive test result?
1747	IEC62021-1	0.0047	11(0.01)		possibly a raise positive test result.
1801	EN62021-1	0.017			
1885					
1890	ISO6619	0.011			
6007					
6015					
6048	EN00004 4	0.00			
6053	EN62021-1	0.02			
6067 6085	EN62021-1	0.006			
6088	IEC62021-1	0.01			
6099	IEC62021-1	0.003			
6141					
6165					
6167					
6199	IEC62021-1	0.0011			
	na nana lite				
	normality	suspect			
	n outliers	22 2			
	mean (n)	0.0073			
	st.dev. (n)	0.0073			
	R(calc.)	0.0148			
	st.dev.(EN62021-1:03)	(0.00073)			
	R(EN62021-1:03)	(0.0020)			Quantification Limit EN62021-1:03: >0.014

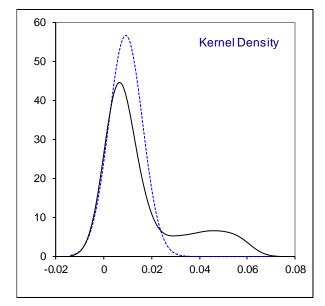




Determination of Acidity, Total (Colorimetric Titration) on sample #18230; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks
173					
179	D974	0.03		1.45	
325					
360	D974	0.005		-0.30	
398	20.1				
446	D974	< 0.02			
614	D974	0.04	G(0.05)	2.15	
840	D974	0.0038	G(0.00)	-0.38	
862	D974	<0.02		-0.50	
902	D974	0.02		0.57	
912	D974	0.0069		-0.17	
913	D974	0.0089	DG(0.05)	2.71	
962	D974	0.040	DG(0.03)	2.71	
963	D974	0.008		-0.09	
974	D974	0.006		-0.09	
	D974	0.006		-0.23	
1146	ISO6618	0.016		0.47	
1262					
1271	ISO6618	0.005		-0.30	
1304	INH-122	<0.01			
1326					
1435	IEC62024 2				
1442 1444	IEC62021-2	<0,01 			
1444					
1460					
1478					
	IEC62021 2	0.0058		-0.24	
1513	IEC62021-2			-0.24 -0.37	
1516	D974	0.004		-0.37	
1529 1560					
1626	D974	0.0064		-0.20	
1660	D974			-0.20	
1687					
1702					
1719					
1743	ISO6618	<0.1			
1747	1000010				
1801					
1885	D974	< 0.01			
1890	20				
6007					
6015	D974	0.010		0.05	
6048	D974	0.0046		-0.33	
6053					
6067					
6085					
6088					
6099					
6141	D974	0.0566	DG(0.05)	3.31	
6165	IEC62021-2	0.01	_ (() ()	0.05	
6167					
6199					
	normality	not OK			
	n	15			
	outliers	3			
	mean (n)	0.0093			
	st.dev. (n)	0.00705			
	R(calc.)	0.0197			
	st.dev.(D974:14e2)	0.01429			Compare R(IEC62021-2:07) = 0.032
	R(D974:14e2)	0.04			Quantification Limit IEC62021-2:07: >0.01





Determination of Appearance on sample #18230;

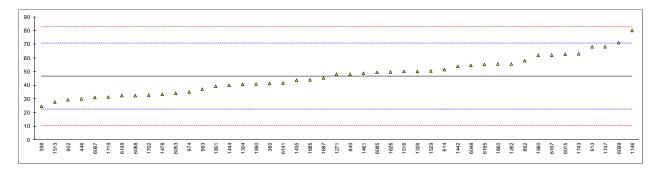
lab	method	value	mark	z(targ)	remarks
173	Visual	Clear & bright			
179					
325	Visual	water white			
360	Visual	Clear and Bright			
398	Visual	Clear			
446	Visual	Clear & Bright			
614					
840	Visual	Pass			
862	Visual	Colorless transparent liquid			
902	Visual	Pass			
912					
913	Visual	Clear and Bright			
962					
963	Visual	Bright & Clear			
974	Visual	Clear & Bright			
1146					
1262		bright and clear			
1271	Visual	clear			
1304					
1326					
1435	Visual	clear			
1442	Visual	clear			
1444	Visual	clear			
1460					
1461					
1478	IEC60296	clear			
1513	Visual	Limpid			
1516	Visual	Clear			
1529	Visual	clear			
1560	Visual	Clear & Bright			
1626	Visual	clear&bright			
1660	Visual	Clear			
1687	\ P = = 1	 Ola ::			
1702	Visual	Clear			
1719	\/:I	Linearial			
1743	Visual	Limpid			
1747					
1801					
1885 1890					
6007					
6015					
6048					
6053					
6067	Visual	clear			and free from sediments and suspended matter
6085	v iSuai				and nee nom sediments and suspended matter
6088	Visual	bright and clear			
6099	Visual	Claire			
6141	Visual	Clear & Bright			
6165					
6167					
6199	Visual	Clear			and free from sediments and suspended matter
		00			
	n	30			
	mean (n)	Bright and Clear			

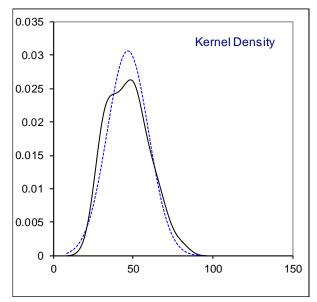
Determination of Color ASTM on sample #18230;

lab	method	value	mark	z(targ)	remarks
173	D1500	L0.5			
179	D1500	L 0.5			
325	D6045	L0.5			
360	ISO2049	< 0.5			
398					
446	D1500	L0.5			
614	D1500	<0.5			
840	D1500	L0.5			
862	D1500	L0.5			
902	D1500	L0.5			
912					
913	D1500	L0.5			
962					
963	D1500	<0.5			
974	D1500	L0.5			
1146					
1262	ISO2049	L 0.5			
1271	D6045	0.1			
1304	INH-132	0.0			
1326	_				
1435	D1500	0.5			
1442	ISO2049	0			
1444	ISO2049	0.3			
1460					
1461	1000010				
1478	ISO2049	0.1			
1513	ISO2049	L0,5			
1516	ISO2049	0			
1529	ISO2049	0-0.5			
1560	ISO2049	L0.5			
1626	D1500	<0,5			
1660	D1500	0.0			
1687 1702	D1500	L0.5			
1702	D1300	<0.5			
1713	ISO2049	L0.5			
1743	ISO2049	L0.5			
1801	ISO2049	<0.5			
1885	1002040				
1890	D1500	<0.5			
6007	2.000				
6015					
6048	D1500	0.0			
6053	2.000				
6067	ISO2049	<0.5			
6085	D1500	<0.5			
6088	D1500	L0.5			
6099	D1500	0.2			
6141	D1500	L0.5			
6165					
6167					
6199	D1500	L0.5			
	n	38			
	mean (n)	<0.5			

Determination of Breakdown Voltage on sample #18230, results in kV/2.5 mm

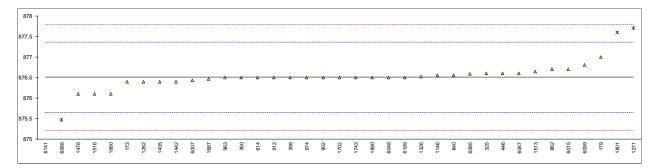
lab	method	value	mark z(targ)	remarks	
173					
179					
325					
360	EN60156	41.1	-0.47		
398	EN60156	24.6	-1.84		
446	EN60156	30	-1.39		
614	IEC60156	51.3	0.38		
840	IEC60156	48.0	0.11		
862 902	IEC60156 EN60156	58 29.4	0.94 -1.44		
912	LINOUISO	25.4	-1.44		
913	IEC60156	68	1.77		
962	12000100				
963	IEC60156	37.2	-0.79		
974	IEC60156	35	-0.97		
1146	IEC60156	80	2.77		
1262	EN60156	55.6	0.74		
1271	EN60156	48	0.11		
1304	INH-124	40.5	-0.52		
1326	IEC60156	50.0	0.28		
1435	IEC60156	43.7	-0.25		
1442	IEC60156	53.70	0.58		
1444	IEC60156	39.9	-0.57		
1460	IECC04EC	40.7	0.47		
1461	IEC60156	48.7	0.17 -1.12		
1478 1513	IEC60156 IEC60156	33.3 27.7	-1.58		
1516	IEC60156	50.0	0.28		
1529	IEC60156	50.5	0.32		
1560	IEC60156	62	1.27		
1626	IEC60156	49.7	0.25		
1660	IEC60156	55.5	0.73		
1687	EN60156	45.3	-0.12		
1702	IEC60156	32.8	-1.16		
1719	IEC60156	31.43	-1.27		
1743	IEC60156	63	1.36		
1747	IEC60156	68	1.77		
1801	EN60156	39.1	-0.63		
1885	IEC60156	44	-0.22		
1890	IEC60156	40.7	-0.50		
6007	ENICO4EC		4.00		
6015	EN60156	62.50	1.32 0.66		
6048 6053	IEC60156	54.6 34			
6067	EN60156 EN60156	30.9	-1.06 -1.31		
6085	IEC60156	49.2	0.21		
6088	IEC60156	32.4	-1.19		
6099	IEC60156	71.0	2.02		
6141	IEC60156	41.45	-0.44		
6165	IEC60156	55.0	0.69		
6167	IEC60156	62.0	1.27		
6199	IEC60156	32.3	-1.20		
				Results 'stirred'	Results 'not stirred'
	normality	OK		OK	OK
	n	45		33	9
	outliers	0		0	0
	mean (n)	46.69		46.01	50.85
	st.dev. (n)	13.033		13.841	10.992
	R(calc.)	36.49		38.75	30.78
	st.dev.(EN60156:95)	12.009		11.833	10.587
	R(EN60156:95)	33.62		33.13	29.64

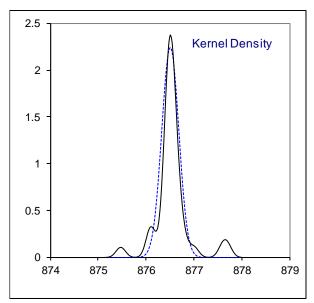




Determination of Density at 20°C on sample #18230; results in kg/m 3

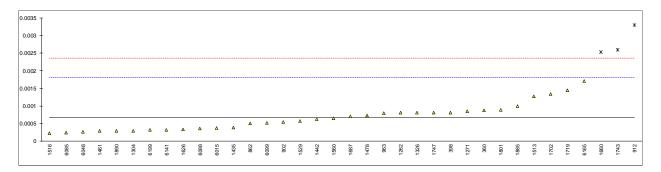
lab	method	value	mark	z(targ)	remarks
173	D4052	876.4		-0.24	
179	D4052	877.0		1.16	
325	D4052	876.6		0.22	
360	ISO12185	876.5		-0.01	
398	ISO12185	876.5	С	-0.01	first reported: 870.3
446	D4052	876.6		0.22	
614	D4052	876.5		-0.01	
840	D4052	876.55		0.11	
862	D4052	876.7		0.46	
902	D4052	876.5		-0.01	
912	D4052	876.5		-0.01	
913					
962	D 1050	070.5		0.04	
963	D4052	876.5		-0.01	
974	D4052	876.5		-0.01	
1146 1262	D4052 ISO3675	876.55 876.4		0.11 -0.24	
1202	D5002	877.7	C,R(0.01)	2.79	first reported: 878.9
1304	D3002		C,1X(0.01)	2.13	ilist reported. 070.9
1326	D4052	876.52		0.04	
1435	D7042	876.4	С	-0.24	first reported: 0.8764 kg/m ³
1442	ISO12185	876.4	Ü	-0.24	mot roported. o.or or ng/m
1444	10012100				
1460					
1461					
1478	ISO12185	876.1		-0.94	
1513	ISO12185	876.645		0.33	
1516	ISO3675	876.1		-0.94	
1529					
1560					
1626	_				
1660	D7042	876.1	_	-0.94	
1687	ISO12185	876.46	С	-0.10	first reported: 0.87646 kg/m ³
1702	ISO12185	876.5		-0.01	
1719		070 5			
1743		876.5		-0.01	
1747	1002675	877.6	D(0.01)	2.56	
1801 1885	ISO3675		R(0.01)	2.50	
1890	ISO12185	876.5		-0.01	
6007	DIN51757	876.435	С	-0.16	first reported: 0.8764 kg/m ³
6015	ISO12185	876.70	Ü	0.46	mot reported. 0.07 04 kg/m
6048	ISO12185	876.5		-0.01	
6053					
6067	D4052	876.6		0.22	
6085	D7042	876.59		0.20	
6088	ISO3675	875.47	C,R(0.01)	-2.41	first reported: 874.39
6099	ISO12185	876.8		0.69	
6141	D1298	870	R(0.01)	-15.18	
6165					
6167	_		_		
6199	D4052	876.5	С	-0.01	first reported: 0.8765 kg/m ³
	Pt.				
	normality	not OK			
	n outliers	33 4			
	mean (n)	4 876.505			
	st.dev. (n)	0.1776			
	R(calc.)	0.1776			
	st.dev.(ISO3675:98)	0.4286			Compare $R(D4052:18) = R(ISO12185:96) = 0.5$
	R(ISO3675:98)	1.2			Compare $R(D7042:16e3) = 1.3$
	()	-			1

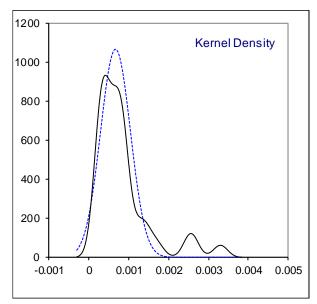




Determination of Di-electric Dissipation Factor (DDF) at 90°C on sample #18230

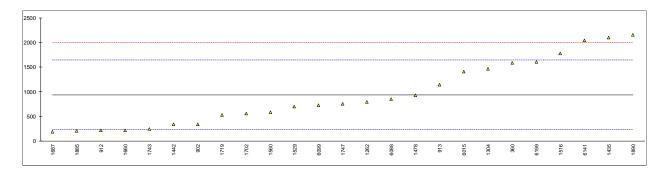
lab	method	value	mark	z(targ)	remarks
173					
179					
325					
360	EN60247	0.00087		0.36	
398	EN60247	0.000814		0.26	measured at 50 Hz
446	EN60247	< 0.0010			
614					
840					
862	IEC60247	0.0005		-0.29	
902	EN60247	0.00055		-0.20	
912	IEC60247	0.0033	R(0.01)	4.66	
913					
962	=11000.				
963	EN60247	0.0008		0.24	
974					
1146	EN00047	0.00004		0.00	
1262	EN60247	0.00081		0.26	
1271	EN60247	0.000847		0.32	
1304	INH-125	0.000298		-0.65	
1326 1435	IEC60247 IEC60247	0.0008105 0.00039		0.26 -0.49	
1435	IEC60247 IEC60247			-0.49 -0.07	
1442	IEC00247	0.000626		-0.07	
1444					
1461	IEC60247	0.000294		-0.66	
1478	IEC60247	0.000294		0.11	
1513	IEC60247	0.00127		1.07	
1516	IEC60247	0.000230		-0.77	
1529	IEC60247	0.00057	С	-0.17	first reported: 5.71
1560	IEC60247	0.000655	Ü	-0.02	mot ropolitod. G.1 1
1626	IEC60247	0.00033		-0.59	
1660	IEC60247	0.00253	R(0.01)	3.30	
1687	EN60247	0.00070	,	0.06	measured at 50 Hz
1702	IEC60247	0.00134		1.19	
1719	IEC60247	0.00144666		1.38	
1743	IEC60247	0.00259	R(0.01)	3.40	
1747	IEC60247	0.000813	C	0.26	first reported: 0.0813
1801	EN60247	0.00089		0.40	
1885	IEC60247	0.00099		0.57	
1890	IEC60247	0.000296		-0.65	
6007					
6015	EN60247	0.0003710		-0.52	
6048	IEC60247	0.000263		-0.71	
6053					
6067	IE000047	0.000040		0.75	
6085	IEC60247	0.000243		-0.75	
6088	IEC60247	0.00036		-0.54	
6099 6141	IEC60247 IEC60247	0.000514		-0.27	
6141 6165	IEC60247 IEC60247	0.000324 0.00171	С	-0.60 1.85	first reported: 0.1711
6167	IEC00247	0.00171	U	1.85	ilist reported. U. 17 11
6199	IEC60247	0.000320		-0.61	
0199	ILOUUZ41	0.000320		-0.01	
	normality	suspect			
	n	33			
	outliers	3			
	mean (n)	0.000666			
	st.dev. (n)	0.0003741			
	R(calc.)	0.001048			
	st.dev.(EN60247:04)	0.0005652			
	R(EN60247:04)	0.001583			
	. ,				

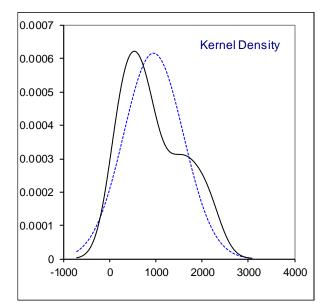




Determination of Specific Resistance at 90°C on sample #18230; results in $\mbox{G}\Omega\mbox{m}$

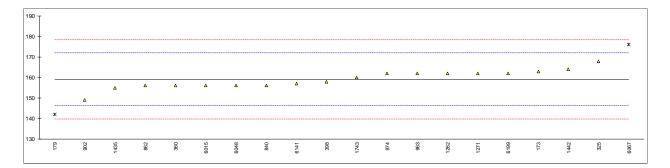
lab	method	value	mark	z(targ)	remarks
173					
179					
325	EN 1000 17	4500		4.05	
360	EN60247	1590		1.85	
398					
446					
614					
840					
862	.=				
902	IEC60247	340.4	_	-1.70	
912	IEC60247	217.5	С	-2.05	first reported: 0.02175 GΩm
913	IEC60247	1144		0.58	
962					
963					
974					
1146	EN1000.47	700.7		0.40	
1262	EN60247	796.7		-0.40	
1271	INUL 405	4.405		4.40	
1304	INH-125	1465		1.49	
1326	EN1000.47				
1435	EN60247	2100		3.30	
1442	IEC60247	339.040		-1.70	
1444					
1460					
1461	15000047			0.04	
1478	IEC60247	925.13		-0.04	
1513	IEC00047	4775		0.07	
1516	IEC60247	1775		2.37	
1529	IEC60247	703		-0.67	
1560 1626	IEC60247	584.8 		-1.01 	
	IEC60247				
1660	IEC60247	222.9		-2.03	
1687	EN60247	192.73		-2.12	
1702	IEC60247	557.40	0	-1.08	reported, E26 426666E0 COm (neceibly a unit array2)
1719 1743	EN60247 IEC60247	526.426667 249	С	-1.17 -1.96	reported: 526.426666E9 G Ω m (possibly a unit error?)
		754.36		-0.53	
1747 1801	IEC60247	754.56		-0.55	
1885	IEC60247	212		-2.06	
1890	IEC60247	2153.5		3.45	
6007	ILC00247	2100.0		3.43	
6015	EN60247	1405.0		1.32	
6048	LINOOZHI				
6053					
6067					
6085					
6088	IEC60247	850		-0.25	
6099	IEC60247	728.22		-0.60	
6141	IEC60247	2040		3.12	
6165					
6167					
6199	IEC60247	1610		1.90	
	normality	OK			
	n	25			
	outliers	0			
	mean (n)	939.28			
	st.dev. (n)	647.462			
	R(calc.)	1812.89			
	st.dev.(EN60247:04)	352.232			
	R(EN60247:04)	986.25			

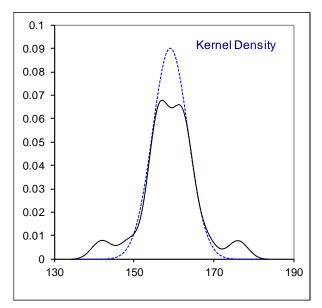




Determination of Flash Point (COC) on sample #18230; results in °C

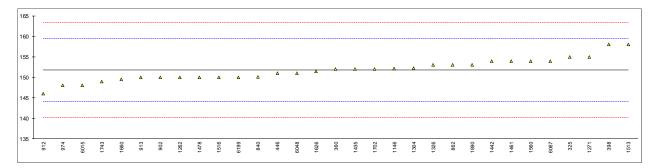
lab	method	value	mark	z(targ)	remarks
173	D92	163		0.60	
179	D92	142	R(0.05)	-2.66	
325	D92	168	11(0.00)	1.38	
360	D92	156		-0.48	
398	ISO2592	158		-0.17	
446	1002332				
614					
	Dos	156.1		-0.47	
840	D92				
862	D92	156		-0.48	
902	D92	149.0		-1.57	
912					
913					
962	D02	160		0.45	
963	D92	162		0.45	
974	D92	162		0.45	
1146	1000500	400			
1262	ISO2592	162		0.45	
1271	ISO2592	162		0.45	
1304					
1326					
1435	D92	155.0		-0.64	
1442	D92	164		0.76	
1444					
1460					
1461					
1478					
1513					
1516					
1529					
1560					
1626					
1660					
1687					
1702					
1719					
1743	ISO2592	160		0.14	
1747					
1801					
1885					
1890					
6007					
6015	D92	156.0		-0.48	
6048	ISO2592	156		-0.48	
6053					
6067	D92	176	R(0.05)	2.63	
6085					
6088					
6099					
6141	D92	157		-0.33	
6165					
6167					
6199	D92	162		0.45	
	normality	OK			
	n	18			
	outliers	2			
	mean (n)	159.12			
	st.dev. (n)	4.427			
	R(calc.)	12.40			
	st.dev.(D92:18)	6.429			
	R(D92:18)	18			R(D92:18) = R(ISO2592:17)
	•				•

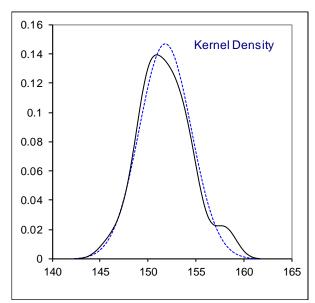




Determination of Flash Point (PMcc) on sample #18230; results in °C

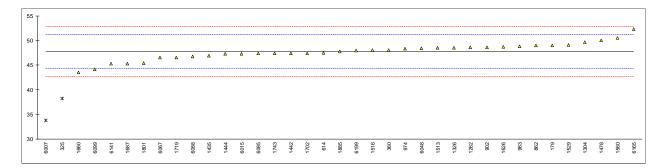
lab	method	value	mark	z(targ)	remarks
173					
179					
325	D93-A	155		0.83	
360	ISO2719-A	152.0		0.06	
398	ISO2719-A	158		1.61	
446	D93-A	151		-0.20	
614	D00.4	450.4			
840	D93-A	150.1	0	-0.44	Continuo anta di 400
862	D93-A	153	С	0.32	first reported: 139
902	D93-A	150.0		-0.46	
912 913	D93-A D93-A	146 150		-1.50 -0.46	
962	D93-A			-0.40	
963					
974	D93-A	148.0		-0.98	
1146	D93-A	152.1		0.08	
1262	ISO2719-A	150		-0.46	
1271	ISO2719-A	155		0.83	
1304	INH-115	152.2		0.11	
1326	D93-A	153.0		0.32	
1435	D93-A	152.0		0.06	
1442	ISO2719-A	154		0.57	
1444					
1460	1000740 4	454		0.57	
1461	ISO2719-A	154		0.57	
1478	ISO2719-A	150.0		-0.46	
1513	ISO2719-A	158.0 150		1.61 -0.46	
1516 1529	D93-A	130		-0.40	
1560	ISO2719-A	154		0.57	
1626	D93-A	151.5		-0.07	
1660	D93-A	149.5		-0.59	
1687					
1702	ISO2719-A	152.0		0.06	
1719					
1743	D93-A	149		-0.72	
1747					
1801					
1885	D00 A	450			
1890	D93-A	153		0.32	
6007	ISO2710 A	1400		0.00	
6015 6048	ISO2719-A D93-A	148.0 151.0		-0.98 -0.20	
6053	D90-W	151.0		-0.20	
6067	D93-C	154		0.57	
6085	_000				
6088					
6099					
6141					
6165					
6167					
6199	ISO2719-A	150		-0.46	
	P.	014			
	normality	OK			
	n outliere	31			
	outliers	0 151.79			
	mean (n) st.dev. (n)	2.713			
	R(calc.)	7.60			
	st.dev.(ISO2719-A:16)	3.849			
	R(ISO2719-A:16)	10.78			R(ISO2719-A:16) = R(D93-A:18) = R(IP34-A:16)
	-,	-			, , , , , , , , , , , , , , , , , , , ,

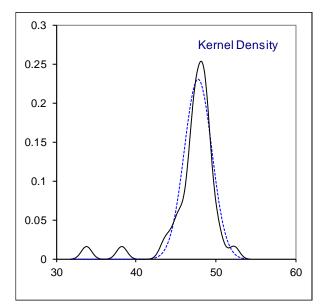




Determination of Interfacial Surface Tension on sample #18230; results in mN/m

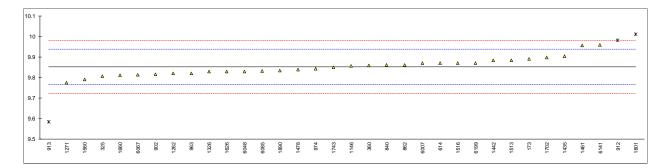
lab	method	value	mark	z(targ)	remarks
173					
179	D971	49	С	0.72	first reported: 42
325	In house	38.2	R(0.01)	-5.61	
360	D971	48.1		0.19	
398					
446					
614	D971	47.5		-0.16	
840					
862	D971	49		0.72	
902	D971	48.63		0.50	
912					
913					
962	_				
963	D971	48.8		0.60	
974	D971	48.3		0.31	
1146	_				
1262	D971	48.6		0.48	
1271					
1304	INH-123	49.7		1.13	
1326	D971	48.56		0.46	
1435	D971	46.9		-0.51	
1442	EN14210	47.420		-0.21	
1444	D971	47.27		-0.30	
1460					
1461	_				
1478	D971	50.0		1.30	
1513	D971	48.51		0.43	
1516	D971	48.1		0.19	
1529	D971	49.1		0.77	
1560	D971	50.5		1.60	
1626	D971	48.71		0.55	
1660	D971	43.5	0	-2.51	Continuo esta di OAO
1687	D971	45.3	С	-1.45	first reported: 34.8
1702	D971	47.430		-0.20	
1719	D2285	46.58		-0.70	
1743	D971	47.4		-0.22	
1747	D971	 45.42		-1.38	
1801 1885		45.42 47.8			
1890	D971	47.0		0.01	
6007	DIN14370	33.7665	C,R(0.01)	-8.21	first reported: 30.774
6015	D971	47.305	C,K(0.01)	-0.21	ilist reported. 30.774
6048	D971	48.43		0.38	
6053	ווטע	40.43		0.36	
6067	D971	46.5		-0.75	
6085	D971	47.371		-0.73	
6088	ISO6295	46.7		-0.63	
6099	EN14210	44.2		-2.10	
6141	D971	45.28		-1.46	
6165	D971	52.3		2.65	
6167	-311				
6199	D971	48		0.13	
	normality	suspect			
	n	35			
	outliers	2			
	mean (n)	47.778			
	st.dev. (n)	1.7304			
	R(calc.)	4.845			
	st.dev.(D971:12)	1.7063			
	R(D971:12)	4.778			

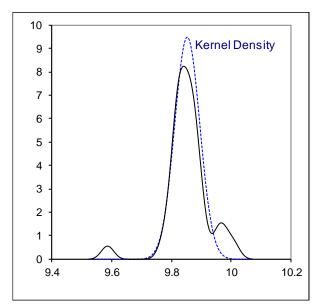




Determination of Kinematic Viscosity at 40°C on sample #18230; results in mm²/s

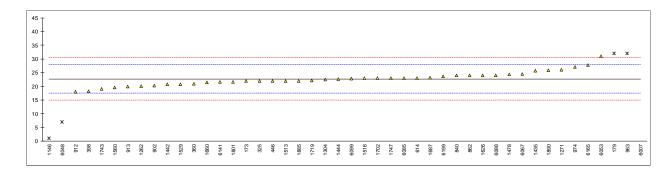
lab	method	value	mark	z(targ)	remarks
173	D445	9.890		0.88	
179					
325	D445	9.807		-1.06	
360	D445	9.8594		0.16	
398					
446	D.445		•		" · · · · · · · · · · · · · · · · · · ·
614	D445	9.87	С	0.41	first reported: 9.58
840	D445	9.8602		0.18	
862 902	D445	9.861		0.20	
902	D445 D445	9.815 9.981	DG(0.05)	-0.87 3.00	
912	D445 D445	9.585	C,G(0.03)	-6.23	first reported: 10.01
962	2710		5,5(0.01)		
963	D445	9.820		-0.75	
974	D445	9.843		-0.22	
1146	D445	9.8559		0.08	
1262	ISO3104	9.82		-0.75	
1271	D7042	9.774		-1.82	
1304	5				
1326	D445	9.828		-0.57	
1435	D7042	9.904		1.20	
1442 1444	D7042	9.883		0.71	
1444					
1460	ISO3104	9.9571		2.44	
1478	D7042	9.8380		-0.33	
1513	ISO3104	9.88442		0.75	
1516	ISO3104	9.87		0.41	
1529					
1560	ISO3104	9.791	С	-1.43	first reported: 9.701
1626	D445	9.829		-0.54	
1660	D7042	9.81		-0.99	
1687	1002104	0.9072		1.04	
1702 1719	ISO3104	9.8972 		1.04	
1719	D7279 corr. to D445	9.85	С	-0.05	first reported: 9.49
1743	5,213 con. to 5443	9.05	J	-0.05	mot reported. 0.70
1801	ISO3104	10.0094	DG(0.05)	3.66	
1885			- (3.22)		
1890	ISO3104	9.8338		-0.43	
6007	DIN51562	9.8690		0.39	
6015					
6048	D445	9.829		-0.54	
6053	D445	0.044		0.00	
6067	D445	9.814		-0.89	
6085 6088	D7042	9.8306		-0.51 	
6099					
6141	D445	9.959		2.48	
6165	•				
6167					
6199	D7042	9.87	С	0.41	first reported: 9.68
	normality	OK			
	n 	31			
	outliers	3			
	mean (n) st.dev. (n)	9.852			
	R(calc.)	0.0422 0.118			
	st.dev.(D445:18)	0.116			Compare R(ISO3104:94) = 0.075
	R(D445:18)	0.0429			Compare R(D7042:16e3) = 0.222
	(5 / 10.10)	0.120			33pa.3 (4)5/3/12.1000/ = 0.1112

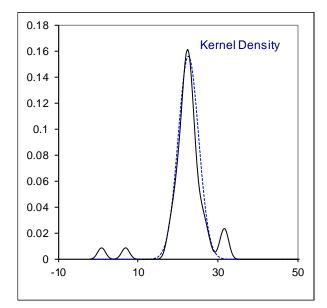




Determination of Water on sample #18230; results in mg/kg

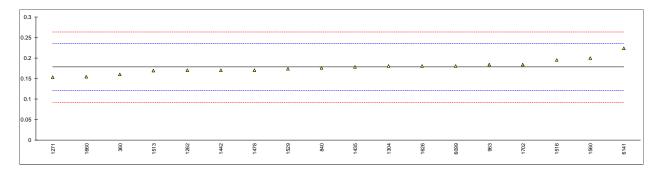
lab	method	value	mark	z(targ)	remarks
173	D6304-C	22		-0.29	
179	D6304-C	32	R(0.05)	3.63	
325	D6304-C	22		-0.29	
360	EN60814	21.0		-0.68	
398	D6304-C	18.2		-1.77	
446	EN60814	22		-0.29	
614	IEC60814	23.05		0.13	
840	D1533	24.0		0.50	
862	D6304-A D1533	24		0.50	
902 912	D6304-C	20.2 18		-0.99 -1.85	
912	D1533	20	С	-1.03 -1.07	first reported: 31
962	D1000		C	-1.07	ilist reported. 31
963	D1533	32	R(0.05)	3.63	
974	D1533	27	C	1.67	first reported: 37
1146	D6304-C	1	R(0.01)	-8.51	
1262	EN60814	20.1	(,	-1.03	
1271	ISO12937	26		1.28	
1304	INH-121	22.45		-0.11	
1326					
1435	IEC60814	25.65		1.14	
1442	IEC60814	20.70		-0.79	
1444	IEC60567	22.5995		-0.05	
1460					
1461	IE000044	04.4		0.05	
1478	IEC60814	24.4		0.65	
1513	IEC60814 IEC60814	22.0		-0.29 0.07	
1516 1529	EN60814	22.9 20.7		-0.79	
1560	IEC60814	19.5		-1.26	
1626	IEC60814	24		0.50	
1660	IEC60814	21.4		-0.52	
1687	EN60814	23.12		0.15	
1702	IEC60814	22.9		0.07	
1719	IEC60814	22.1		-0.25	
1743	IEC60814	19		-1.46	
1747	IEC60814	23		0.11	
1801	EN60814	21.7		-0.40	
1885	D1533	22		-0.29	
1890	IEC60814	25.92	0.5(0.04)	1.25	
6007 6015	ISO760	87.0 	C,R(0.01)	25.16 	first reported: 0
6048	ISO12937	7	C,R(0.01)	-6.16	first reported: <10
6053	EN60814	31	-, (-,-,	3.24	
6067	EN60814	24.5		0.69	
6085	IEC60814	23.002		0.11	
6088	D1533	24		0.50	
6099	IEC60814	22.8		0.03	
6141	D1533	21.6		-0.44	
6165	IEC60814	27.7		1.95	
6167 6199	IEC60814	23.7		0.38	
0199	IEC00014	23.1		0.38	
	normality	suspect			
	n	41			
	outliers	5			
	mean (n)	22.729			
	st.dev. (n)	2.5545			0
	R(calc.) st.dev.(EN60814:98)	7.152 2.5540			Compare R(D1533:12) = 14, range 0-50
	R(EN60814:98)	2.5540 7.151			Compare R(D6304-C:16e1) = 110.058, range 10-25000 Compare R(ISO12937:00) = 32.786, range 30-10000
	11(1100014.30)	7.101			Joinpard Miloo 12001.00) - 02.100, larige 00-10000

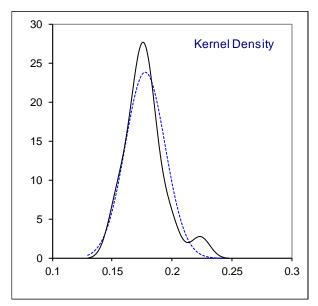




Determination of 2,6-Ditertiary-butyl paracresol (DBPC) on sample #18230; results in %M/M

lab	method	value	mark	z(targ)	remarks
173			mark	<u> </u>	
179					
325					
360	IEC60666	0.16		-0.62	
398					
446					
614					
840	IEC60666	0.176		-0.07	
862	IEC60666	<0.01	•	<-5.88	" · · · · · · · · · · · · · · · · · · ·
902	D2668	<0.05	С	<-4.48	first reported 0.06
912					
913 962					
963	IEC60666	0.184		0.21	
974	12000000				
1146					
1262	IEC60666	0.17		-0.28	
1271	IEC60666	0.1537		-0.85	
1304	INH-129	0.18		0.07	
1326					
1435	IEC60666	0.178		0.00	
1442	IEC60666	0.17		-0.28	
1444					
1460					
1461	IECeoeee	0.17		0.29	
1478 1513	IEC60666 IEC60666	0.17		-0.28 -0.32	
1516	IEC60666	0.1000		0.60	
1529	IEC60666	0.174		-0.14	
1560	IEC60666	0.2		0.77	
1626	IEC60666	0.18		0.07	
1660	IEC60666	0.154		-0.83	
1687					
1702	IEC60666	0.184		0.21	
1719					
1743					
1747 1801					
1885					
1890					
6007					
6015					
6048					
6053					
6067					
6085					
6088	IE000000	0.40		0.07	
6099	IEC60666	0.18		0.07	
6141 6165	D2668	0.224		1.61 	
6167					
6199					
0100					
	normality	not OK			
	n	18			
	outliers	0	Spike:		
	mean (n)	0.178	0.177	recovery	<100%
	st.dev. (n)	0.0167			
	R(calc.)	0.047			
	st.dev.(IEC60666:10) R(IEC60666:10)	0.0286 0.080			
	11(11000000.10)	0.000			





Determination of 2,6-Ditertiary-butyl phenol (DBP); results in %M/M and determination of Dibenzyl disulphide (DBDS), Benzotriazole (BTA) and Irgamet 39; results in mg/kg, all on sample #18230

lab	DBP	DBDS	ВТА	Irgamet 39	remarks
173					
179					
325					
360					
398					
446					
614					
840					
862	<0.01	<5	<5		
902					
912					
913					
962					
963	<0.02	<5		<5	
974					
1146					
1262					
1271					
1304					
1326			4.40		
1435		0	1.16	0	
1442	<0,0001	<5	<0,03	<5	
1444					
1460					
1461					
1478		 <5		 -5	
1513				<5 0	
1516 1529	0	0	0	0 0.4	
1529	·	·		0.4	
1626					
1660	0.00	0	0.00	0.00	
1687					
1702		Not Detected		Not Detected	
1719					
1743		<3			
1747					
1801					
1885	0.172				
1890					
6007					
6015					
6048					
6053					
6067	0.1168	<5	<5		
6085					
6088					
6099					
6141		0	<1	<1	
6165					
6167					
6199					

APPENDIX 2 Analytical details for the test method on Breakdown Voltage

				1	1	
			Test	0	_	
	Drand of toot		started	Stirring	Temp.	
lab	Brand of test equipment	Type of electrode	after min	during test	sample in °C	Preparation of the sample
	equipinient	Type of electrode			C	Freparation of the sample
173						
179						
325						
		Pairs of electrodes in				
000	DALLE DTA 4000	accordance with EN	_	\ ,		As received - it is mixed carefully without air
360	BAUR DTA 100C	60156 Fig.2	5	Yes	29	bubbles
		brass made and hemi- spherical shape. 3cm				gently agitated by turning-over of the bottle (approximatively 10 times). No bubble air
398	BAUR DPA	diameter	<1	Yes	23,8	formed.
	BAUR	ulailloto!	5	Yes	19.8	Torriod.
		Spherical electrodes	5	Yes	21.2	Rinsed cell out with new oil and wiped it clean
	00					Clean outside the bottle of sample. Test
840	BA100	GB0055 Mushroom	0	Yes	25	Breakdown Voltage at the first.
		brass polished, partially				The sample container is gently agitated and
		spherical of				turn over without causing the formation of air
		shape(13mm diameter)	5	No	20	bubbles.
902	Baur	-		Yes	90	
912						
						Sample was inspected for presence of
040	M/a da sa C	Diala Flanton da			07	moisture, sludge, metallic particles and
913 962	Wadegati	Disk Electrode	2	Yes	27	evidence of free water
902		2.5 mm Spherical				agitated and turned over several times to make
963	Baur DTA 100 C	electrode	2	No	21	homogeneous
- 000	Baar B 177 100 0	olection de	_	140		Gently agitated and turned over the bottle
						several times without causing formation of air
974		Spherical Electrode	5	Yes	24.0	bubbles
	megger foster OTS					
1146	80 AF/2	Bol electrodes	circa 15	Yes	20	15 minutes shaking gently, then 2 hrs resting
						TEMPERED AT ROOM TEMPERATURE AND
1262	BAUR DPA 75C	IEC 60156 FIG.II	5	Yes	25	FILLED IN THE CELL
1071	EI UI 300 NIÅ , SRBIJA	Cu (anhariaal alaatrada)	_	No	22	The sample container gently agitated and
1271	SKDIJA	Cu (spherical electrode)	5	No	23	turned over several times. Gently rotate the bottle to mix without
1304	Baur	Round balls	5	Yes	20-23	introducing bubbles
1326	Megger	Ball cap electrode.	5	Yes	18	I mile data ing 2 data ing
1435	- 33 -					
1 100		spherical electrodes				gently agitated and turn the bottle, fill with no
1442	baur	according IEC 60156	5	Yes	21,0	bubbles - according IEC 60156
						shake well before the test and Electrode and
1444	BAuR	Mushroom	5	Yes	23	Vessel are cleaned
1460						
1461						
		partially spherical				
1478	BAUR DTA 100	electrodes	10	Yes	25	we turn sample bottle over 3 times
1513	B2 HV	Spherical electrodes	5	Yes	20	Gently agitated and turned over several times
		Mushroom electrodes				
1516	Baur 100 DTA	according to IEC 60156 figure 2	5	Yes	20	No preparation
1529	Bauer	Sperical electrodes	10	Yes	20	homogenize without introducing air bubbles.
1560	Megger OTS100AF	IEC 60156 Fig. II	5	Yes	24	Gently Shaking to homogenize the sample
			-			Homogenized the sample by cautious turning
1626	Megger OTS100AF	Mushroom	5	No	20	upside down
1660	B2	VDE		Yes	24	gentle shaking
		half spherical				Smooth shaking and rotating for
1687	Baur DTA 100	electrodes	0	No	22	homogenization, then pouring out with care.
1702			-		04	
1719 1743	Megger	round	5	Yes	21	no The comple is mixed before filling the test cell
1/43	BAUR	Spherical electrodes		No	<u> </u>	The sample is mixed before filling the test cell Drain and rinse the test cell with the test liquid
1747	BAUR DTA 100C	IEC60156 FIG.11	5	Yes	20.0 +- 5	and check electrodes distance before test.
1801	BAUR	SEMISPHERIC	5	Yes	22	NO
1885	Megger	Hemi spherical	1	No	22	Sample shaken
	33-	Fig II: partially spherical		-		,
		electrodes 25mm				
	_	radius and diameter				
1890	Baur	36mm	30	Yes	21	homogenisation on rolling equipment

			1	1		
			Test	Ctiveina	Tomp	
	Brand of test		started after	Stirring during	Temp.	
lab	2.4.14 6. 1661	Type of electrode	min	test	sample in °C	Preparation of the sample
lab	equipment	Type of electrode		เษรเ	C	Preparation of the sample
6007	not tested	not tested	not tested		not tested	not tested
0007	not tested	polished sperical disk	lested		not tested	not tested
6015	Baur	electrodes	5	Yes	25	moderate shaking
6048	Baur	Partially spherical	5	Yes	22	No special preparation
0040	Daui	Faitially Sprietical	5	163	22	Remove all residues of the previous liquid with
						an appropriate solvent, rinse the assembly with
6053	Megger	Spherical	3	Yes	25	clean, dry wiper then refill with test specimen.
0000	HV Diagnostics	Oprierical	3	163	20	Sample is gently agitated and turned several
6067	BA100	Mushroom type	10	Yes	23	times without causing any air bubbles
0007	DATOU	Musiliooni type	10	163	25	The sample container was gently agitated &
6085	BAUR DTA E	ROGOWSKI	5	Yes	22	turned over several times.
0000	DAORDIAL	partially spherical - IEC	3	103	22	turred over several times.
6088	BAUR DTA 100	60156 Fig. II	5	Yes	24	Only cold the bottle to laboratory temperature
0000	2710112771100	Spherical (figure 1				Strily solid till bottle to laboratory temporature
6099	BAUR DTA 100 C	IEC60156:1995-07)	5	No	21	shake gently by turning the bottle over
		,				Rinsing twice the test cell before final filling for
6141	Megger OTS100AF	Mushroom	1	No	25	the test.
6165	Megger	Spherical	1	Yes	22	shake manually
	FOSTER (AVO)					Clean dried cell is rinsed with a portion of the
	MEGGER UK - OTS					sample before filling the sample into the test
6167	100AF/2	MUSHROOM	5	Yes	23	cell.
	AVO MEGGER	Partially spherical				
	FOSTER OTS	shape. Made with				The sample bottle was agitated, turned over
6199	100AF/2	brass.	5	Yes	24	several times to ensure homogeneity

APPENDIX 3

Number of participants per country

- 3 labs in AUSTRALIA
- 3 labs in BELGIUM
- 1 lab in BOSNIA and HERZEGOVINA
- 3 labs in BULGARIA
- 2 labs in CHINA, People's Republic
- 1 lab in FRANCE
- 5 labs in GERMANY
- 1 lab in GREECE
- 2 labs in INDIA
- 2 labs in ITALY
- 1 lab in KUWAIT
- 2 labs in MALAYSIA
- 2 labs in NETHERLANDS
- 1 lab in NEW ZEALAND
- 2 labs in PORTUGAL
- 1 lab in QATAR
- 4 labs in SAUDI ARABIA
- 2 labs in SINGAPORE
- 1 lab in SLOVENIA
- 2 labs in SPAIN
- 1 lab in SWEDEN
- 1 lab in SWITZERLAND
- 1 lab in TURKEY
- 3 labs in UNITED ARAB EMIRATES
- 2 labs in UNITED KINGDOM
- 2 labs in UNITED STATES OF AMERICA
- 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 D(0.05) = outlier in Double Grubbs' outlier

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 = possibly an error in calculations

U = test result possibly 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

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation, June 2018
- 2 prNEN 12766-2:00
- 3 ASTM E178:02
- 4 ASTM E1301:03
- 5 ISO 5725:86
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- 7 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367:84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 13 Analytical Methods Committee Technical Brief, No 4, January 2001
- 14 P.J. Lowthian and M. Thompson, the Royal Society of Chemistry, Analyst, <u>127</u>, 1359-1364 (2002)
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, <u>25(2)</u>, 165-172, (1983)
- iis memo 'Evaluation of the reproducibility of the Breakdown Voltage in Transformer Oils (fresh and used) as per EN60156:1998 based on Proficiency Tests performed from 2001 till 2016, December 2017