

Institute for **Interlaboratory Studies**

Results of Proficiency Test Transformer Oil (fresh) November 2022

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

Mrs. E.R. Montenij-Bos Author:

ing. R.J. Starink & ing. G.A. Oosterlaken-Buijs ing. A.S. Noordman-de Neef **Correctors:**

Approved by:

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CONTENTS

1	INTRODUCTION	3
2	SET UP	3
2.1	ACCREDITATION	3
2.2	PROTOCOL	3
2.3	CONFIDENTIALITY STATEMENT	3
2.4	SAMPLES	4
2.5	STABILITY OF THE SAMPLES	4
2.6	ANALYZES	5
3	RESULTS	5
3.1	STATISTICS	5
3.2	GRAPHICS	6
3.3	Z-SCORES	7
4	EVALUATION	7
4.1	EVALUATION PER TEST	8
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	10
4.3	COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2022 WITH PREVIOUS PTS	10

Appendices:

1.	Data, statistical and graphic results	12
2.	Other reported test results	37
3.	Number of participants per country	38
4.	Abbreviations and literature	39

1 Introduction

Since 2001 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Transformer Oil (fresh) based on the latest version of IEC60296 and ASTM D3487 every year. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of Transformer Oil (fresh).

In this interlaboratory study 61 laboratories in 34 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the 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 organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample Transformer Oil (fresh) in a 1-liter amber glass bottle labelled #22225.

The 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/IEC17043:2010 (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 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

A batch of approximately 90 liters of Transformer Oil (fresh) was obtained from a local supplier. After homogenization 80 amber glass bottles of 1 L were filled and labelled #22225. The homogeneity of the subsamples was checked by the determination of Density at 20 °C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Density at 20 °C in kg/m³
sample #22225-1	873.49
sample #22225-2	873.50
sample #22225-3	873.49
sample #22225-4	873.49
sample #22225-5	873.50
sample #22225-6	873.49
sample #22225-7	873.49
sample #22225-8	873.50

Table 1: homogeneity test results of subsamples #22225

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 20 °C in kg/m³		
r (observed)	0.01		
reference test method	ISO12185:96		
0.3 x R (reference test method)	0.15		

Table 2: evaluation of the repeatability of subsamples #22225

The calculated repeatability was in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one 1 L bottle of Transformer Oil (fresh) labelled #22225 was sent on October 26, 2022. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

The participants were requested to determine: Total Acidity (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 (C.O.C. and PMcc), Interfacial Surface Tension, Kinematic Viscosity at 40 °C, Water and Additives (2,6-Ditertiary-butyl phenol (DBP), 2,6-Ditertiary-butyl paracresol (DBPC), Dibenzyl disulfide (DBDS), Benzotriazole (BTA) and Irgamet 39). Also, an extra question regarding stirring during the determination of the Breakdown Voltage was 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' test 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 reference test methods (when applicable) 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 appendices 1 and 2 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. 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 June 2018 (iis-protocol, 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 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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 F(0.01) for the Rosner's test. Stragglers are marked by F(0.01) for the Dixon's test, by F(0.01) 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. 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 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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(target) = (test result - average of PT) / 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. Therefore, 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 with the dispatch of the samples. Six participants reported the test results after the final reporting date and four other participants did not report any test results. Not all participants were able to report all tests requested.

In total 57 participants reported 437 numerical test results. Observed were 19 outlying test results, which is 4.3%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all 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 test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D1500) and an added designation for the year that the test method was adopted or revised (e.g. D1500:12). When a method has been reapproved an "R" will be added and the year of approval (e.g. D1500:12R17).

- <u>Total Acidity (Potentiometric)</u>: This determination may not be problematic. All reporting participants agreed on a value near or below the quantification limit of test method EN62021-1:03 and the application range of ASTM D664-A:18e2. Therefore, no z-sores are calculated.
- <u>Total Acidity (Colorimetric)</u>: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D974:21.
- <u>Appearance</u>: The determination was not problematic. Almost all reporting laboratories agreed on the appearance of the sample which was 'Clear and Bright' (Pass).
- Breakdown Voltage: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of IEC60156:18.

 When evaluated separately over stirring and no stirring no effect is observed.
- Color ASTM: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO2049:96 and ASTM D1500:12R17. Please note: the test values reported as "text" (e.g. L0.5) were converted to a numerical value before calculating z-scores, see also appendix 1.
- <u>Density at 20 °C</u>: This determination was problematic for a number of participants. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96, ASTM D4052:18a, ISO3675:98 and ASTM D1298:12bR17.
- <u>DD-Factor at 90 °C</u>: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN60247:04.

<u>Specific Resistance at 90 °C</u>: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not 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. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D92:18 and ISO2592:17.

<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 and ASTM D93-A:20.

Interfacial Surface Tension: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility is in full agreement with the requirements of ASTM D971:20.

Kinematic Viscosity at 40 °C: This determination was problematic for a number of participants. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:21e2, ISO3104:20 and ASTM D7042:21a.

<u>Water</u>: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN60814:97 and ASTM

D1533:20.

Additives: The majority of the participants agreed that 2,6-Ditertiary-butyl phenol

(DBP), 2,6-Ditertiary-butyl paracresol (DBPC), Dibenzyl disulfide (DBDS), Benzotriazole (BTA) and Irgamet 39 were below the level of quantification. Therefore, these components were not further evaluated. The reported test

results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference methods are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acidity (Potentiometric)	mg KOH/g	26	0.008	0.013	(0.002)
Total Acidity (Colorimetric)	mg KOH/g	26	0.007	0.013	0.04
Appearance		37	C&B(Pass)	n.a.	n.a.
Breakdown Voltage	kV/2.5 mm	52	56.8	35.4	47.7
Color ASTM		43	0.23	0.14	1
Density at 20 °C	kg/m³	36	873.6	0.5	0.5
Di-electric Dissipation Factor		45	0.0013	0.0013	0.0024
Specific Resistance at 90 °C	GΩm	36	499	653	524
Flash Point C.O.C.	°C	19	155.4	17.8	18
Flash Point PMcc	°C	28	145.7	8.4	10.3
Interfacial Surface Tension	mN/m	37	47.5	5.1	4.7
Kinematic Viscosity at 40 °C	mm²/s	32	9.97	0.13	0.12
Water	mg/kg	47	16.3	5.3	6.1

Table 3: reproducibilities of tests on sample #22225

For results between brackets no z-scores are calculated

C&B = Clear and Bright

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2022 WITH PREVIOUS PTS

	November 2022	November 2021	November 2020	November 2019	November 2018
Number of reporting laboratories	57	64	59	48	50
Number of test results	437	436	404	377	371
Number of statistical outliers	19	20	28	24	24
Percentage of statistical outliers	4.3%	4.6%	6.9%	6.4%	6.5%

Table 4: 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 to the requirements of the reference test methods. The conclusions are given in the following table.

Parameter	November 2022	November 2021	November 2020	November 2019	November 2018
Total Acidity (Potentiometric)	()	()	()	()	()
Total Acidity (Colorimetric)	++	++	++	++	++
Breakdown Voltage	+	+	+	+	+/-
Color ASTM	++	++	++	++	n.e.
Density at 20 °C	+/-	+	+	+	++
Di-electric Dissipation Factor	+	+	+	+	+
Specific Resistance	-	-		-	-
Flash Point C.O.C.	+/-	+/-	+	+	+
Flash Point PMcc	+	+/-	+/-	+	+
Interfacial Surface Tension	+/-	-	-	+/-	+/-
Kinematic Viscosity at 40 °C	+/-	-	-	-	+/-
Water	+	+	+/-	-	+/-
DBPC Antioxidant Additive	n.e.	n.e.	n.e.	n.e.	+

Table 5. comparison of group performances to the reference test methods

For results between brackets no z-scores are calculated.

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

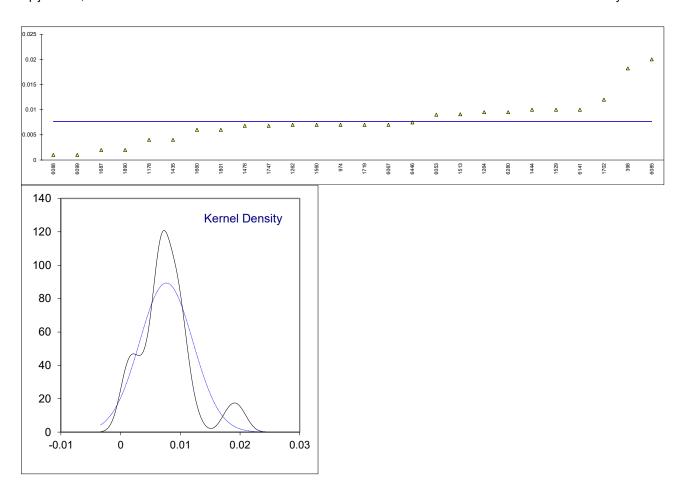
-- : group performed much worse than the reference test method

n.e. : not evaluated

APPENDIX 1

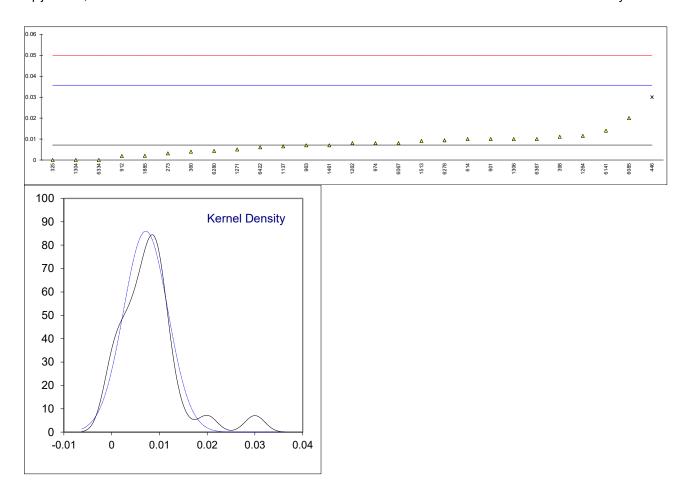
Determination of Total Acidity (Potentiometric) on sample #22225; results in mg KOH/g

lab	method	value	mark	z(targ)	ple #22225; results in mg KOH/g remarks
173	ouiou		murk	<u> </u>	10110110
179					
273					
325					
360	ENCO004 4	0.0400			
398 446	EN62021-1	0.0182			
614					
657	D664-A	<0.1			
862	200171				
901					
912					
963	D004 A	0.007			
974 1137	D664-A	0.007			
1146	D664-A	<0.10			
1178	IEC62021-1	0.004			
1262	EN62021-1	0.007			
1264	D664-A	0.0095			
1271					
1304					
1306					
1326 1381					
1435	IEC62021-1	0.004			
1442					
1444	EN62021-1	0.01			
1458					
1461	15000004				
1478	IEC62021-1	0.0068			
1513 1529	IEC62021-1 IEC62021-1	0.0091 0.01			
1560	IEC62021-1	0.007			
1660	IEC62021-1	0.006			
1687	D664-A	0.002			
1702	IEC62021-1	0.012			
1719	D664-A	0.007			
1747	IEC62021-1	0.0068			
1801 1885	EN62021-1	0.006			
1890	ISO6619	0.002			
6015					
6048					
6053	IEC62021-1	0.009			
6067	EN62021-1	0.007			
6071 6085	IEC62021-1 D8045	<0.01 0.02			
6088	IEC62021-1	0.02			
6099	IEC62021-1	0.001			
6141	D664-A	0.01			
6167					
6278	IE000004 4				
6280	IEC62021-1	0.0095			
6322 6334					
6367					
6382	IEC62021-1	<0,01			
6391					
6402					
6422	IE000004 4	0.00740			
6446	IEC62021-1	0.00746			
norma	litv	not OK			
n	,	26			
outliers	S	0			
mean	(n)	0.0077			
st.dev.		0.00446			
R(calc		0.0125			
	(EN62021-1:03)	(0.00077)			Quantification limit EN62021 1:02 >0.014 mg KOH/c
compa	32021-1:03) are	(0.0021)			Quantification limit EN62021-1:03 >0.014 mg KOH/g
	4-A:18e2,IP 60 mL)	(0.0074)			Application range D664-A:18e2: 0.1 – 150 mg KOH/g
,	, ,	, ,			5



Determination of Total Acidity (Colorimetric) on sample #22225; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
173	metriou		IIIQIK		Temarks
179					
273	D974	0.0031		-0.28	
325	D974	0.00		-0.50	
360	D974	0.004		-0.22	
398	D974	0.0111		0.28	
446	D974	0.03	R(0.01)	1.60	
614	D974	0.01		0.20	
657	D974	<0.02			
862	D074				
901	D974	0.01		0.20	
912	D974	0.0019		-0.37	
963 974	D974 D974	0.007 0.008		-0.01 0.06	
1137	D974	0.0065		-0.05	
1146	D07-4				
1178					
1262	ISO6618	0.008		0.06	
1264	D974	0.0115		0.30	
1271	ISO6618	0.005		-0.15	
1304	In house	0.000		-0.50	
1306	D974	0.01		0.20	
1326					
1381					
1435	IEC62024 2	 <0.01			
1442	IEC62021-2	<0,01 			
1444 1458	D974	<0.01			
1461	D314	0.007		-0.01	
1478				-0.01	
1513	IEC62021-2	0.0091		0.14	
1529					
1560					
1660					
1687					
1702					
1719					
1747					
1801	D074				
1885	D974	0.002		-0.36	
1890					
6015 6048	D974	<0,02			
6053	D974				
6067	D974	0.008		0.06	
6071	5011				
6085	D974	0.02		0.90	
6088					
6099					
6141	D974	0.014		0.48	
6167					
6278	D974	0.0093398		0.15	
6280	IEC62021-2	0.0043		-0.20	
6322	IEC62024 2	0.000		0.50	
6334 6367	IEC62021-2	0.000		-0.50 0.20	
6382	IEC62021-2	0.01		0.20	
6391					
6402					
6422	IEC62021-2	0.006		-0.08	
6446					
	normality	suspect			
	n	26			
	outliers	1			
	mean (n)	0.0071			
	st.dev. (n)	0.00464			
	R(calc.)	0.0130			
	st.dev.(D974:21) R(D974:21)	0.01429 0.04			
	11(00/4.21)	0.04			

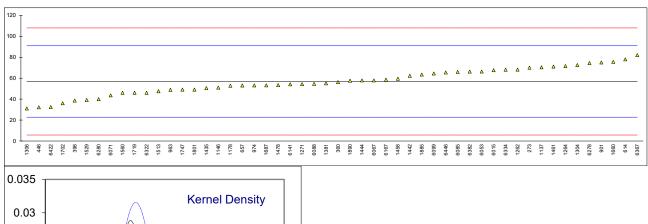


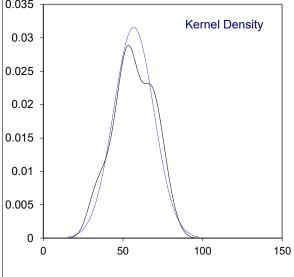
Determination of Appearance on sample #22225;

lab	method	value	mark z(targ)	remarks
173	Visual	Clear & Bright		
179				
273	Visual	Bright & Clear		
325	Visual	water white		
360	Visual	Clear and Bright		
398	Visual	C&B		
446	D4176	Pass		
614				
657	Visual	Clear		
862 901				
912				
963	Visual	Bright and Clear		
974	Visual	Clear and Bright		
1137				
1146	Visual	clear		
1178	Visual	clear, fssm		
1262	IEC60296	bright and clear		
1264	Visual	Clear		
1271	Visual	BRIGHT & CLEAR		
1304	Vioual	CLEAD		
1306 1326	Visual	CLEAR 		
1326				
1435	Visual	Clear		
1442	Visual	clear		
1444	D1524	Clear & Bright		
1458	Visual	Clear Bright		
1461				
1478	Visual	clear		
1513	Visual	Clear		
1529	IEC60422/ISO2049	clear / no deposits		
1560	Visual	Clear & Bright		
1660 1687	Visual	Clear		
1702	Visual	Clear		
1719	Vioudi			
1747				
1801				
1885	Visual	C&B		
1890	Visual	clear		
6015				
6048	Visual	bright and clear		
6053	Visual	Clear		
6067 6071	Visual	C&B 		
6085				
6088	Visual	clear and bright		
6099	Visual	clear		
6141	Visual	Clear and bright		
6167				
6278	Visual	Bright and clear		
6280				
6322	Visual	slightly yellow		
6334	Visual	Clear Pright		
6367 6382	Visual Visual	Clean&Bright		
6391	vioual	Clear, free from sediment		
6402				
6422				
6446	Visual	clear & bright		
		Ü		
	n	37		
	mean (n)	Clear and Bright (Pass)		

Determination of Breakdown Voltage on sample #22225, results in kV/2.5 mm

lab	method	value	mark	z(targ)	stirred or not stirred	remarks
173						
179						
273	IEC60156	70		0.77	Stirred continuously including breakdowns	
325	ENGO156	 56 1		0.04	Stirred continuously including breakdows	
360 398	EN60156 IEC60156	56.1 38.6		-0.04 -1.07	Stirred continuously including breakdowns Stirred continuously including breakdowns	
446	IEC60156	32.1		-1.45	Stirred continuously excluding breakdowns	
614	IEC60156	78		1.24	Stirred continuously including breakdowns	
657	IEC60156	52.94		-0.23	Not stirred	
862						
901	IEC60156	75.0		1.07	Stirred continuously including breakdowns	
912 963	IEC60156	48.8		-0.47	Not stirred	
974	EN60156	53		-0.47		
1137	IEC60156	70.3		0.79	Not stirred	
1146	IEC-156	51.0		-0.34	Stirred continuously including breakdowns	
1178	IEC60156	52.8		-0.24		
1262	EN60156	68.2		0.67	Stirred continuously including breakdowns	
1264	IEC60156	71.5		0.86	Not stirred	
1271 1304	IEC60156 IEC60156	54.25 72.55		-0.15 0.92	 Stirred continuously including breakdowns	
1304	12000100	31		-1.51		
1326						
1381	IEC60156	55.0		-0.11	Stirred continuously excluding breakdowns	
1435	IEC60156	50.5		-0.37		
1442	IEC60156	62.2		0.32	Stirred continuously excluding breakdowns	
1444	IEC60156	57.8		0.06	Stirred continuously excluding breakdowns	
1458 1461	IEC60156 EN60156	59.5 71.1		0.16 0.84	Stirred continuously including breakdowns	
1478	IEC60156	53.3		-0.21	Stirred continuously excluding breakdowns	
1513	IEC60156	47.4		-0.55	Stirred continuously including breakdowns	
1529	IEC60156	39.1		-1.04	Stirred continuously excluding breakdowns	
1560	IEC60156	46		-0.63	Stirred continuously including breakdowns	
1660	IEC60156	75.5		1.10	Stirred continuously including breakdowns	
1687 1702	IEC60156 IEC60156	53.1 36.0		-0.22 -1.22	Stirred continuously excluding breakdowns Stirred continuously including breakdowns	
1719	IEC60156	46		-0.63	Stirred continuously excluding breakdowns	
1747	IEC60156	49		-0.46	Stirred continuously including breakdowns	
1801	EN60156	49.0		-0.46	Stirred continuously including breakdowns	
1885	IEC60156	63.4		0.39	Not stirred	
1890	IEC60156	57.5		0.04	Stirred continuously including breakdowns	
6015 6048	EN60156	67.55 		0.63	Stirred continuously excluding breakdowns	
6053	IEC60156	66.3		0.56	Stirred continuously including breakdowns	
6067	IEC60156	57.8		0.06	Stirred continuously excluding breakdowns	
6071	IEC60156	43.5		-0.78	Stirred continuously excluding breakdowns	
6085	IEC60156	65.9		0.53	Stirred continuously including breakdowns	
6088	IEC60156	54.5		-0.14	Stirred continuously including breakdowns	
6099	IEC60156	64.4		0.44	Not stirred	
6141 6167	IEC60156 IEC60156	54.1 58.5		-0.16 0.10	Stirred continuously including breakdowns Stirred continuously including breakdowns	
6278	IEC60156	74.4		1.03	Not stirred	
6280	IEC60156	39.9		-0.99		
6322	IEC60156	46.0		-0.63		
6334	IEC60156	68.1		0.66	Stirred continuously excluding breakdowns	
6367	IEC60156	82 66.2		1.48	Stirred continuously including breakdowns	
6382 6391	IEC60156	66.2		0.55		
6402						
6422	IEC60156	32.4		-1.43	Stirred continuously including breakdowns	
6446	IEC60156	65.4		0.50	Stirred continuously including breakdowns	
					-	
		OK			<u>'stirred' only</u>	
	normality	OK 52			OK	
	n outliers	52 0			36 0	
	mean (n)	56.82			56.78	
	st.dev. (n)	12.645			12.905	
	R(calc.) ´	35.40			36.13	
	st.dev.(IEC60156:18)	17.045			17.033	
	R(IEC60156:18)	47.73			47.69	

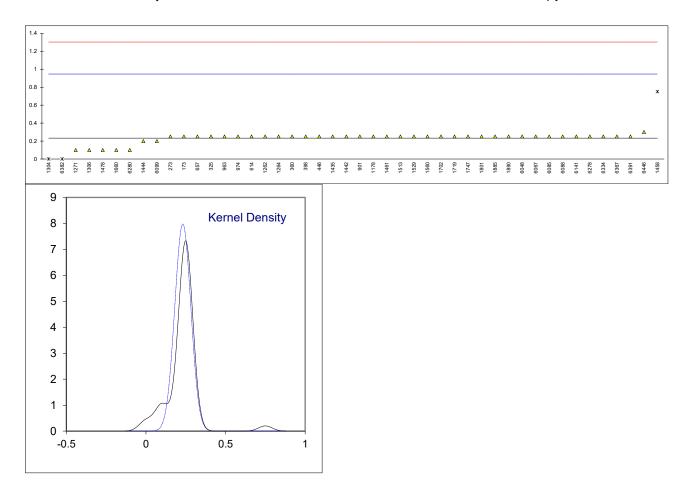




Determination of Color ASTM on sample #22225;

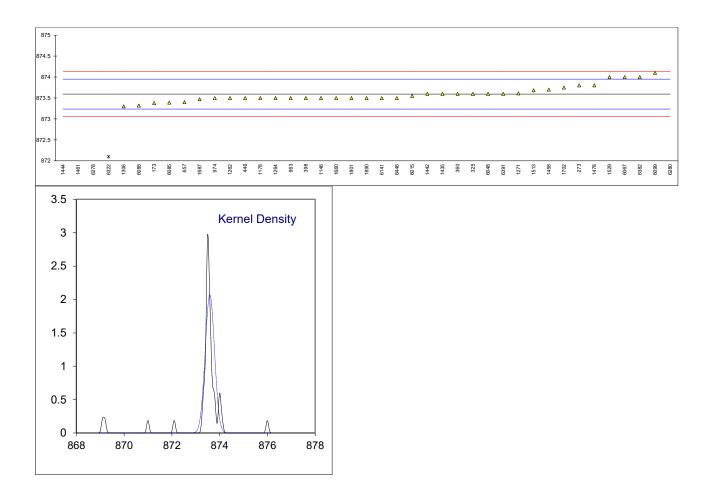
lab	method	value	iis conversion*	mark	z(targ)	remarks
173	D1500	L0.5	0.25	mark	0.05	i Gilial N3
173	D 1000	LU.3	0.25		0.05	
273	D1500	L0.5	0.25		0.05	
325	D6045	L0.5	0.25		0.05	
360	D1500	< 0.5	0.25		0.05	
398	ISO2049	L0.5	0.25		0.05	
446	D1500	L0.5	0.25		0.05	
614	D1500	<0.5	0.25		0.05	
657	D1500	L 0.5	0.25		0.05	
862	10.000.40		0.05			
901	ISO2049	L0.5	0.25		0.05	
912 963	D1500	L0.5	0.25		0.05	
974	D1500	L0.5	0.25		0.05	
1137	D 1000					
1146						
1178	ISO2049	L0,5	0.25		0.05	
1262	ISO2049	L 0.5	0.25		0.05	
1264	D1500	L0.5	0.25		0.05	
1271	D6045	0.1	0.1	D(0.04)	-0.37	
1304	In house	0.0	0.0	R(0.01)	-0.65	
1306	D1500	0.1	0.1		-0.37	
1326 1381						
1435	D1500	0.5	0.25		0.05	
1442	ISO2049	L0,5	0.25		0.05	
1444	ISO2049	0.2	0.2		-0.09	
1458	D1500	L1.0	0.75	R(0.01)	1.45	
1461		L0,5	0.25	, ,	0.05	
1478	ISO2049	0.1	0.1		-0.37	
1513	ISO2049	L0,5	0.25		0.05	
1529	IEC60422/ISO2049	0-0.5	0.25		0.05	
1560	ISO2049	L0.5	0.25		0.05	
1660 1687	D1500	0.1	0.1		-0.37 	
1702	D1500	L 0.5	0.25		0.05	
1719	D1524	<0.5	0.25		0.05	
1747	ISO2049	<0.5	0.25		0.05	
1801	ISO2049	<0,5	0.25		0.05	
1885	D1500	< 0.5	0.25		0.05	
1890	D1500	<0.5	0.25		0.05	
6015						
6048	D1500	L 0,5	0.25		0.05	
6053	ISO2049	Clear	Clear		0.05	
6067 6071	ISO2049	L0.5	0.25		0.05	
6085	D1500	< 0.5	0.25		0.05	
6088	D1500	L0.5	0.25		0.05	
6099	D1500	0.2	0.2		-0.09	
6141	D1500	L0.5	0.25		0.05	
6167						
6278	D1500	<0.5	0.25		0.05	
6280	D1500	0.1	0.1		-0.37	
6322	D1500		0.25		0.05	
6334 6367	D1500	<0.5	0.25		0.05 0.05	
6367 6382	D1500 ISO2049	0.5 0.0	0.25 0.0	R(0.01)	-0.65	
6391	ISO2049	L0.5	0.25	11(0.01)	0.05	
6402						
6422						
6446	D1500	0.3	0.3		0.19	
	e					
	normality		not OK			
	n		43			
	outliers		3			
	mean (n) st.dev. (n)		0.23 0.050			
	R(calc.)		0.030			
	st.dev.(ISO2049:96)		0.14			
	R(ISO2049:96)		1		compare	R(D1500:12R17) = 1
					,	` '

^{*)} In the calculation of the mean, standard deviation and the reproducibility in this column, a reported value of 'L y' or '<y' is changed to y-0.25 (for example L0.5 is changed into 0.25).



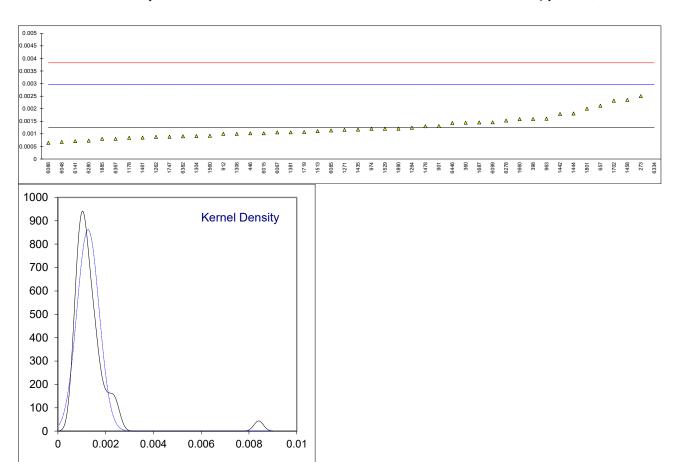
Determination of Density at 20 $^{\circ}\text{C}$ on sample #22225; results in kg/m 3

lab	method	value	mark	z(targ)	remarks
173	D4052	873.38	III MIII	-1.19	
179					
273	D4052	873.8		1.16	5
325 360	D4052 D4052	873.6 873.6	С	0.04 0.04	first reported 0.8736 kg/m ³
398	ISO12185	873.5		-0.52	
446	D4052	873.5		-0.52	
614					
657	D4052	873.4		-1.08	
862 901					
912					
963	D4052	873.5		-0.52	
974	D4052	873.5		-0.52	
1137 1146	D4052	873.5	С	-0.52	first reported 0.8735 kg/m ³
1178	ISO12185	873.5	J	-0.52	motroported 0.0700 kg/m
1262	ISO3675	873.5	С	-0.52	first reported 873.0
1264	D4052	873.5		-0.52	
1271 1304	D5002	873.61 		0.10	
1304	D4052	873.3		-1.64	
1326					
1381	D4050	070.0			
1435 1442	D4052 ISO12185	873.6 873.60		0.04 0.04	
1444	ISO12185	869.10	R(0.01)	-25.16	
1458	D4052	873.7		0.60	
1461	ISO3675	869.2	C,R(0.01)	-24.60	first reported 873.0
1478 1513	ISO12185 ISO12185	873.8 873.682		1.16 0.50	
1529	10012100	874.0	С	2.28	reported 0.8740 kg/m ³
1560					
1660	D7042	873.5		-0.52	
1687 1702	ISO12185 ISO12185	873.47 873.75		-0.69 0.88	
1719					
1747	10000==				6
1801 1885	ISO3675	873.5 	С	-0.52 	first reported 876.2
1890	ISO12185	873.5		-0.52	
6015	ISO12185	873.55		-0.24	
6048	ISO12185	873.6		0.04	
6053 6067	ISO12185	 874.0		2.28	
6071	10012100				
6085	D7042	873.39		-1.14	
6088	ISO3675	873.32	С	-1.53	first reported 873.02
6099 6141	ISO12185 D4052	874.1 873.5		2.84 -0.52	
6167	2 1002				
6278	D1298	871.0	C,R(0.01)	-14.52	first reported 874.5
6280 6322	ISO12185 ISO12185	876 872.1	R(0.01) C,R(0.01)	13.48 -8.36	reported 0.8721 kg/m ³
6334	13012103		C,IX(0.01)	-0.30	reported 0.8721 kg/iii
6367					
6382	DIN51757	874		2.28	
6391 6402	ISO3675	873.6 		0.04	
6422					
6446	ISO12185	873.5		-0.52	
	normality	suspect			
	normality n	36			
	outliers	5			
	mean (n)	873.593			
	st.dev. (n) R(calc.)	0.1926 0.539			
	st.dev.(ISO12185:96)	0.1786			
	R(ISO12185:96)	0.5			
	compare	0.50			
	R(D4052:18a) R(ISO3675:98)	0.50 1.2			
	R(D1298:12bR17)	1.2			



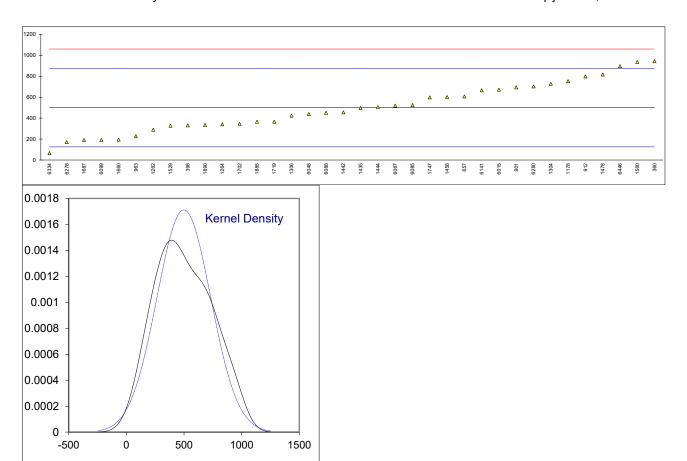
Determination of Di-electric Dissipation Factor (DDF) at 90 °C on sample #22225

lab	method	value	mark	z(targ)	frequency	remarks
173			-			
179						
273	IEC60247	0.00250		1.45		
325 360	EN60247	0.00144		0.21	50	
398	EN60247	0.001590		0.21	50	
446	EN60247	0.00102		-0.28	60	
614						
657	IEC60247	0.002119		1.00	50	
862 901	EN60247	0.001307		0.05		
912	EN60247	0.0010		-0.31		
963	IEC60247	0.0016		0.40	60	
974	EN60247	0.0012		-0.07	50	
1137						
1146 1178	IEC60247	0.00084		-0.49	50	
1262	EN60247	0.00088		-0.45	50	
1264	IEC60247	0.001243	С	-0.02	60	first reported 0.00354
1271	EN60247	0.001156		-0.12		
1304	IEC60247	0.000912		-0.41	50	
1306 1326	IEC60247	0.001		-0.31 		
1381	IEC60247	0.00106		-0.24		
1435	IEC60247	0.001169		-0.11	50	
1442	IEC60247	0.00179		0.62	55	
1444	EN60247	0.001807		0.64	50	
1458 1461	IEC60247 EN60247	0.002352 0.000849		1.28 -0.48	50	
1478	IEC60247	0.000349		0.05		
1513	IEC60247	0.00111		-0.18		
1529	IEC60247	0.0012		-0.07		
1560	IEC60247	0.000922		-0.40	50	
1660 1687	IEC60247 IEC60247	0.001587 0.001451		0.38 0.22	55 50	
1702	IEC60247	0.002318		1.24	50	
1719	IEC60247	0.001071		-0.22		
1747	IEC60247	0.000883		-0.44		
1801 1885	EN60247 IEC60247	0.002002 0.00080		0.87 -0.54	60	
1890	IEC60247	0.00080		-0.54 -0.07	50Hz	
6015	EN60247	0.0010220		-0.28	50	
6048	IEC60247	0.000678		-0.68		
6053	IE000047	0.004050		0.04		
6067 6071	IEC60247	0.001056		-0.24	50	
6085	IEC60247	0.001132		-0.15	55	
6088	IEC60247	0.00064		-0.73	50	
6099	IEC60247	0.001455		0.23		
6141	IEC60247	0.000710		-0.65	60	
6167 6278	IEC60247	0.00153		0.31		
6280	IEC60247	0.000729		-0.62		
6322						
6334	IEC60247	0.008405	R(0.01)	8.36		
6367 6382	IEC60247 IEC60247	0.0008 0.0009		-0.54 -0.42		
6391	12000247	0.0009		-0.42		
6402						
6422						
6446	IEC60247	0.001432	С	0.20	50	first reported as frequency
	normality	suspect				
	n	45				
	outliers	1				
	mean (n)	0.001262				
	st.dev. (n) R(calc.)	0.0004628 0.001296				
	st.dev.(EN60247:04)	0.001290				
	R(EN60247:04)	0.002393				



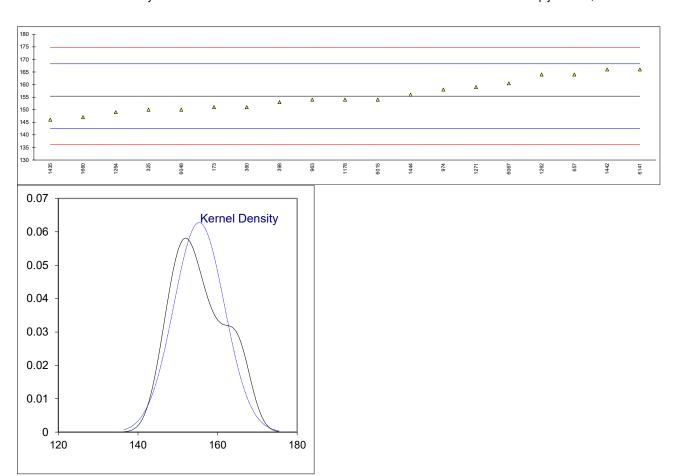
Determination of Specific Resistance at 90 °C on sample #22225; results in $G\Omega m$

lab	method	value	mark	z(targ)	remarks
173	momou		man		Tomano
179					
273					
325	ENC0047	040.0		0.07	
360	EN60247	942.6 330.02		2.37 -0.90	
398 446	EN60247	330.02		-0.90	
614					
657	IEC60247	606		0.57	
862					
901	EN60247	693		1.04	
912 963	EN60247 IEC60247	797.6 226.0		1.60 -1.46	
974	IEC00247			-1.40	
1137					
1146					
1178	IEC60247	752.7		1.36	
1262	EN60247	288.9	0	-1.12	first new auto-d 444 40
1264 1271	IEC60247	340.71	С	-0.84 	first reported 111.18
1304	IEC60247	727.60		1.22	
1306	IEC60247	423.76		-0.40	
1326					
1381					
1435	EN60247	495.76		-0.02	
1442 1444	IEC60247 EN60247	454.47 505.92		-0.24 0.04	
1458	IEC60247	601.0		0.55	
1461					
1478	IEC60247	815.96		1.70	
1513					
1529	IEC60247	326.8		-0.92	
1560 1660	IEC60247 IEC60247	935.99 195		2.34 -1.62	
1687	IEC60247	190.44		-1.65	
1702	IEC60247	344.86		-0.82	
1719	IEC60247	366.12		-0.71	
1747	IEC60247	597.69		0.53	
1801 1885	IEC60247	364.87		-0.72	
1890	IEC60247	333.4		-0.72	
6015	EN60247	671.295		0.92	
6048	IEC60247	438.75		-0.32	
6053	15000015				
6067	IEC60247	518.46		0.11	
6071 6085	IEC60247	524.26		0.14	
6088	IEC60247	450		-0.26	
6099	IEC60247	190.62		-1.65	
6141	IEC60247	665.16		0.89	
6167	IE000047	470.55		4.75	
6278 6280	IEC60247	170.55 702.19		-1.75 1.09	
6280 6322	IEC60247	702.19		1.09	
6334	IEC60247	65.45		-2.32	
6367					
6382					
6391					
6402 6422					
6446	IEC60247	895.89		2.12	
	normality	OK			
	n tl:	36			
	outliers mean (n)	0 498.61			
	st.dev. (n)	233.079			
	R(calc.)	652.62			
	st.dev.(EN60247:04)	186.977			
	R(EN60247:04)	523.54			



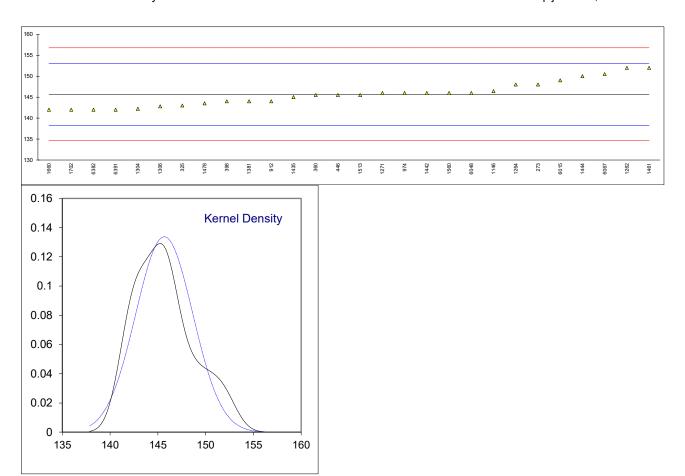
Determination of Flash Point C.O.C. on sample #22225; results in °C

lab	method	value	mark z(targ)	remarks
173	D92	151	-0.68	Tomano
179	202		-0.00	
273				
325	D92	150	-0.84	
360	D92	151	-0.68	
398	D92	153	-0.37	
446				
614	D00	404	4.24	
657 862	D92	164 	1.34	
901				
912				
963	D92	154	-0.22	
974	D92	158	0.41	
1137				
1146				
1178	ISO2592	154.0	-0.22	
1262	ISO2592	164	1.34	
1264	D92 ISO2592	149	-0.99	
1271 1304	1502592	159 	0.56	
1304				
1326				
1381				
1435	D92	146.0	-1.46	
1442	D92	166	1.65	
1444	D92	156.0	0.09	
1458				
1461				
1478 1513				
1529				
1560				
1660	D92	147	-1.31	
1687				
1702				
1719				
1747				
1801				
1885 1890				
6015	ISO2592	154.0	-0.22	
6048	D92	150	-0.84	
6053				
6067	D92	160.5	0.79	
6071				
6085				
6088				
6099 6141	D92	166	 1.65	
6167	D32		1.05	
6278				
6280				
6322				
6334				
6367				
6382				
6391 6402				
6422				
6446				
00				
	normality	OK		
	n	19		
	outliers	0		
	mean (n)	155.39		
	st.dev. (n)	6.356		
	R(calc.) st.dev.(D92:18)	17.80 6.429		
	R(D92:18)	18		
	compare	. •		
	R(ISO2592:17)	18		
	•			



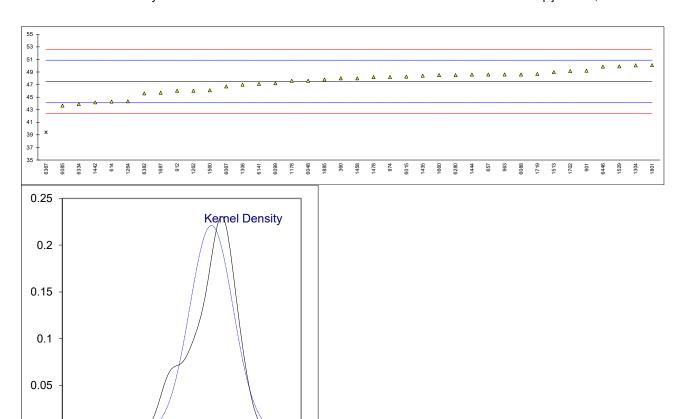
Determination of Flash Point PMcc on sample #22225; results in °C

lab	method	value	mark z(tai	arg) remarks
173	momou			
179				
273	D93-A	148.0	0.	0.63
325	D93-A	143.0).72
360	D93-A	145.5		0.05
398	ISO2719-A	144		0.45
446 614	ISO2719-A	145.5		0.05
614 657			-	
862				
901				
912	ISO2719	144	-0.	0.45
963				
974	D93-A	146		0.09
1137				
1146	D93-B	146.4		0.20
1178 1262	ISO2719-A	152.0		 1.71
1262	D93-A	148		0.63
1271	ISO2719-A	146		0.09
1304	In house	142.2		0.94
1306		142.8).78
1326				
1381	ISO2719-A	144.0		0.45
1435	D93-A	145.0		0.18
1442	ISO2719-A	146.0		0.09 1.17
1444 1458	ISO2719	150.0 		l.17
1461	ISO2719-A	152		 I.71
1478	ISO2719-A	143.5		0.59
1513	ISO2719-A	145.5		0.05
1529				
1560	ISO2719-A	146		0.09
1660	D93-A	142		0.99
1687	1500710 A	142.0		
1702 1719	ISO2719-A	142.0		0.99
1747				
1801				
1885				
1890				
6015	ISO2719-A	149.0		0.90
6048	D93-A	146.0		0.09
6053 6067	D93-A	 150.5		 .31
6071	D93-A	150.5		
6085				
6088				
6099				
6141				
6167				
6278				
6280				
6322 6334				
6367				
6382	ISO2719-A	142		0.99
6391	ISO2719-A	142.0		0.99
6402				
6422				
6446				
	normality	OK		
	normality n	OK 28		
	outliers	0		
	mean (n)	145.68		
	st.dev. (n)	2.982		
	R(calc.)	8.35		
	st.dev.(ISO2719-A:16)	3.694		
	R(ISO2719-A:16)	10.34		
	compare	10.24		
	R(D93-A:20)	10.34		



Determination of Interfacial Surface Tension on sample #22225; results in mN/m

lab	method	value	mark	z(targ)	remarks
173					
179					
273					
325					
360	D971	48.0		0.30	
398					
446 614	D971	44.3	С	-1.88	first reported 39.2
657	D971	48.595	C	0.65	ilist reported 55.2
862	5071				
901	D971	49.21		1.01	
912	D971	46		-0.88	
963	D971	48.6		0.65	
974	D971	48.20		0.42	
1137					
1146 1178	D971	47.60		0.07	
1262	D971	46	С	-0.88	first reported 43.0
1264	D971	44.33	Č	-1.86	first reported 41.5
1271					•
1304	In house	50.05		1.51	
1306	D971	46.98		-0.30	
1326					
1381	D071	 10 11		0.54	
1435 1442	D971 IEC62961	48.41 44.160		0.54 -1.96	
1444	D971	48.59		0.65	
1458	D971	48.0		0.30	
1461					
1478	D971	48.18		0.41	
1513	D971	48.99		0.88	
1529	D971	49.9		1.42	
1560 1660	D971 D971	46.1 48.5		-0.82 0.60	
1687	D971	45.7		-1.05	
1702	D971	49.15		0.98	
1719	D2285	48.7		0.71	
1747					
1801	D971	50.08		1.53	
1885	D971	47.8		0.18	
1890 6015	D971	 10 275		0.46	
6048	D971	48.275 47.6		0.40	
6053	5071				
6067	D971	46.7		-0.47	
6071					
6085	D971	43.659		-2.26	
6088	ISO6295	48.6		0.65	
6099 6141	ISO6295 D971	47.2 47.1		-0.17 -0.23	
6167	D971	47.1		-0.23	
6278					
6280	D971	48.5		0.60	
6322					
6334	D971	43.89	6 5 / 6 5 · 1	-2.12	
6367	D971	39.4	C,R(0.01)	-4.77	first reported 48
6382 6391	ISO6295	45.6 		-1.11 	
6402					
6422					
6446	D971	49.85		1.39	
	normality	OK			
	n outliers	37			
	outliers mean (n)	1 47.489			
	st.dev. (n)	1.8046			
	R(calc.)	5.053			
	st.dev.(D971:20)	1.6960			
	R(D971:20)	4.749			



55

50

0

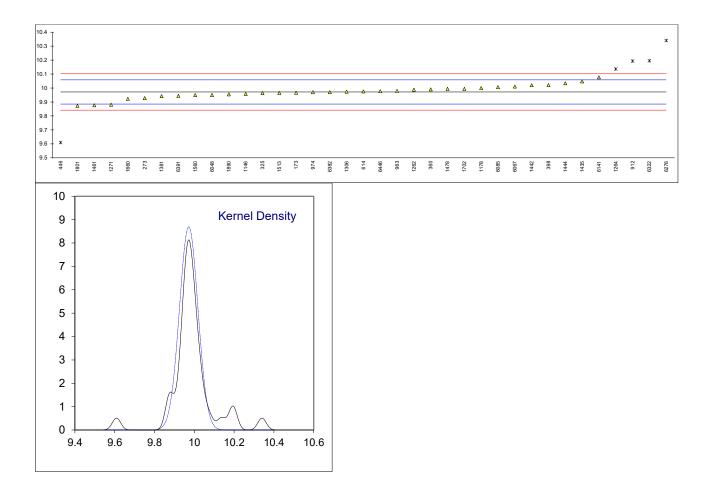
35

40

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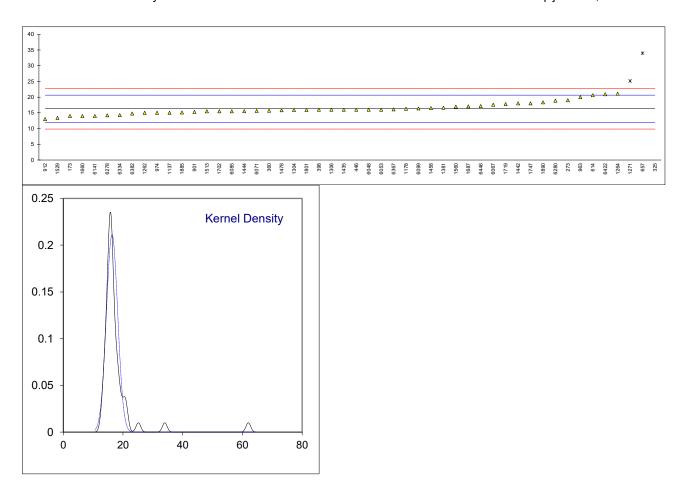
Determination of Kinematic Viscosity at 40 °C on sample #22225; results in mm²/s

lab	method	value	mark	z(targ)	remarks
173	D445	9.966	man	-0.13	Tomario
179	D.445				
273 325	D445 D445	9.928 9.964		-1.01 -0.18	
360	D445	9.9900		0.42	
398	D445	10.021		1.13	
446	D445	9.609	R(0.01)	-8.35	
614 657	D7042	9.9752		0.08	
862					
901					
912	D445	10.193	R(0.05)	5.09	
963	D445	9.979		0.17	
974 1137	D445	9.970 		-0.04 	
1146	D445	9.9573		-0.33	
1178	D7042	10.000		0.65	
1262	ISO3104 D7042	9.988	C,R(0.05)	0.37	first reported 10 175
1264 1271	ISO3104	10.136 9.88	C,R(0.05)	3.78 - 2.11	first reported 10.175
1304					
1306	D445	9.974		0.05	
1326 1381	ISO3104	 9.9415		-0.70	
1435	D7042	10.0470		1.73	
1442	D7042	10.02		1.11	
1444	D445	10.034		1.43	
1458 1461	ISO3104	 9.8765		 -2.19	
1478	D7042	9.0703	С	0.51	first reported 10.157
1513	ISO3104	9.9646	· ·	-0.17	metropolica romo.
1529					
1560 1660	ISO3104 D7042	9.95 9.9219		-0.50 -1.15	
1687	D7042	9.9219		-1.13	
1702	D7042	9.9941		0.51	
1719					
1747 1801	ISO3104	9.8731	С	-2.27	first reported 9.8532
1885	1000101		· ·		mot reported 0.0002
1890	ISO3104	9.954		-0.41	
6015 6048	D445	9.950		-0.50	
6053	D440			-0.50	
6067	D445	10.01		0.88	
6071 6085	D7042	10.006		0.79	
6088	D7042				
6099					
6141	D445	10.0768		2.42	
6167 6278	D445	10.34	C,R(0.01)	8.47	first reported 10.2213
6280					
6322	D7042	10.195	C,R(0.05)	5.14	first reported 10.137
6334 6367					
6382	DIN51562-1	9.971		-0.02	
6391	ISO3104	9.944		-0.64	
6402 6422					
6446	D445	9.977		0.12	
	normality	OK 32			
	n outliers	52 5			
	mean (n)	9.972			
	st.dev. (n)	0.0459			
	R(calc.)	0.129 0.0434			
	st.dev.(D445:21e2) R(D445:21e2)	0.0434			
	compare				
	R(ISO3104:20)	0.122			
	R(D7042:21a)	0.224			



Determination of Water on sample #22225; results in mg/kg

173 179	method DC204 C-20	value	mark	z(targ)	remarks
	D6304-C:20	14		-1.07	
273	IEC60814	19		1.24	
325	D6304-C:20	62	R(0.01)	21.10	
360	EN60814	15.7		-0.29	
398	EN60814	16.0		-0.15	
446	EN60814	16		-0.15	
614	IEC60814	20.6		1.97	
657	D6304-A:20	34	C,R(0.01)	8.17	first reported 29.42
862	D.1500				
901	D1533	15.3		-0.47	
912	EN60814	13		-1.54	
963	D1533	20.0		1.70	
974 1137	D1533 ISO10337	15 15		-0.61	
1146	D6304-B:20	<100		-0.61 	
1178	IEC60814	16.3		-0.01	
1262	EN60814	15		-0.61	
1264	D1533	21.07		2.19	
1271	ISO12937	25.15	C,R(0.01)	4.08	first reported 51.25
1304	In house	15.9	-,: (-:- : /	-0.20	
1306	D1533	16		-0.15	
1326					
1381	IEC60814	16.6		0.13	
1435	IEC60814	16		-0.15	
1442	EN60814	18.00		0.77	
1444	EN60814	15.5307		-0.37	
1458	IEC60814	16.5		0.08	
1461					
1478	IEC60814	15.8		-0.24	
1513	IEC60814	15.5		-0.38	
1529	IEC60814	13.4		-1.35	
1560	IEC60814	17		0.31	
1660	IEC60814	14		-1.07	
1687 1702	EN60814 IEC60814	17.069 15.5		0.34 -0.38	
1719	IEC60814	17.8		0.68	
1747	IEC60814	18		0.00	
1801	EN60814	15.9		-0.20	
1885	D1533	15.1		-0.57	
1890	IEC60814	18.33		0.93	
6015					
6048	ISO12937	16		-0.15	
6053	IEC60814	16		-0.15	
6067	IEC60814	17.6		0.59	
6071	IEC60814	15.61		-0.33	
6085	D6304-C:16e1	15.5		-0.38	
6088			_		
6099	IEC60814	16.4	С	0.03	first reported 10.9
6141	D1533	14		-1.07	
6167	D4500	44.045			
6278	D1533	14.215		-0.98	
6280	IEC60814	18.87		1.18	
6322 6334	IEC60814-Mod.	 14.28		-0.95	
6334 6367	IEC60814-Mod.	14.28 16.1		-0.95 -0.10	
6382	IEC60814	14.77		-0.10 -0.72	
6391	1200017	14.77		-0.72	
6402					
6422	IEC60814	20.92	С	2.12	first reported 26.62
6446	ISO12937	17.16	_	0.39	
-		-			
	normality	OK			
	n	47			
	outliers	3			
	mean (n)	16.326			
	st.dev. (n)	1.8875			
	R(calc.)	5.285			
	st.dev.(EN60814:97)	2.1646			
	R(EN60814:97)	6.061			
	compare	14			Application range D1533:20: 0 -50 mg/kg
	R(D1533:20)				



APPENDIX 2 Other reported test results on sample #22225

2,6-Ditertiary-butyl phenol (DBP) in %M/M, 2,6-Ditertiary-butyl paracresol (DBPC) in %M/M, Dibenzyl disulfide (DBDS) in mg/kg, Benzotriazole (BTA) in mg/kg and Irgamet 39 in mg/kg

lab	DBP	DBPC	DBDS	ВТА	Irgamet 39	remarks
173						
179						
273						
325		 < 0.01				
360 398		< 0.01				
446						
614						
657	0.17616	0.15754				
862						
901		<0.01				
912			 -F			
963 974	<0.02 	<0.02 	<5 	<1 	<1 	
1137						
1146						
1178	0.001					
1262		0.0				
1264	<0.008		1	0	0	
1271		<0,02				
1304 1306		0	0		0	
1326						
1381						
1435		<0.024		<1.00	<5.0	
1442		<0,03	0	<0,04	<5	
1444			ND			
1458		<0.02				
1461 1478		0.00				
1513		<0,01	<5		<5	
1529						
1560		Not Detected				
1660	0.00	0.00	0	0	0	
1687						
1702		Not Detected	<5		<1	
1719 1747						
1801			0.122			
1885	0.002					
1890						
6015		0.0018				
6048						
6053 6067	0.0	0.0	0.0	0.0	0.0	
6071						
6085		0.0				
6088						
6099						
6141		0	0	0	0	
6167						
6278 6280						
6322						
6334		<0.01	<5		<5	
6367						
6382						
6391						
6402 6422						
6446	0.0	0.0				
•	- · -					

APPENDIX 3

Number of participants per country

- 4 labs in AUSTRALIA
- 3 labs in BELGIUM
- 1 lab in BOSNIA and HERZEGOVINA
- 1 lab in BOTSWANA
- 3 labs in BULGARIA
- 2 labs in CHINA, People's Republic
- 1 lab in CROATIA
- 1 lab in ESTONIA
- 7 labs in GERMANY
- 1 lab in GREECE
- 1 lab in HONG KONG
- 1 lab in INDIA
- 2 labs in ITALY
- 1 lab in KOREA, Republic of
- 1 lab in KUWAIT
- 1 lab in MALAYSIA
- 1 lab in MOROCCO
- 2 labs in NETHERLANDS
- 1 lab in NEW ZEALAND
- 1 lab in NORWAY
- 1 lab in PHILIPPINES
- 1 lab in POLAND
- 1 lab in PORTUGAL
- 1 lab in QATAR
- 3 labs in SAUDI ARABIA
- 3 labs in SINGAPORE
- 1 lab in SLOVENIA
- 2 labs in SOUTH AFRICA
- 2 labs in SPAIN
- 1 lab in SWITZERLAND
- 2 labs in TURKEY
- 3 labs in UNITED ARAB EMIRATES
- 2 labs in UNITED KINGDOM
- 2 labs in UNITED STATES OF AMERICA

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 G(0.01) = outlier in Grubbs' outlier test G(0.05) = straggler in Grubbs' outlier test

DG(0.01) = outlier in Double Grubbs' outlier test DG(0.05) = straggler in Double Grubbs' outlier test

R(0.01) = outlier in Rosner's outlier test R(0.05) = straggler in Rosner's outlier test

E = calculation difference between reported test result and result calculated by iis

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

f+? = possibly a false positive test result? f-? = possibly a false negative test result?

SDS = Safety Data Sheet

Literature

- iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
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- 8 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
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- Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)