Results of Proficiency Test Transformer Oil (fresh) November 2016

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 program 2016/2017, it was decided to continue the round robin for Transformer Oil (fresh) in accordance with the latest applicable version of the specification IEC60296 and of ASTM D3487. In this interlaboratory study, 55 laboratories from 30 different countries have registered for participation. See appendix 2 for a list of number of participants per country. In this report, the results of the 2016 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. 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 #16250. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in 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 April 2014 (iis-protocol, version 3.3). This protocol is electronically available from the FAQ-page of the iis website www.iisnl.com.

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 200 litre bulk material was homogenised in a pre-cleaned drum. After homogenisation, 60 subsamples were transferred to 1 litre amber glass bottles and labelled #16250. The homogeneity of the subsamples #16250 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 #16250-1	876.38
Sample #16250-2	876.37
Sample #16250-3	876.38
Sample #16250-4	876.38
Sample #16250-5	876.37
Sample #16250-6	876.38
Sample #16250-7	876.38
Sample #16250-8	876.37

Table 1: homogeneity test results of subsamples #16250

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the target 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 #16250)	0.01
reference test method	ISO3675:98
0.3 x R _(reference test method)	0.36

Table 2: evaluation of the repeatability of subsamples #16250

The calculated repeatability of sample #16250 was less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples #16250 was assumed.

To each of the participating laboratories, 1*1 litre bottle (labelled #16250) was sent on November 2, 2016.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were asked to determine tests mentioned in either ASTM D3487 or IEC 60296 on sample #16250: 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 DBPC Additive.

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

3 RESULTS

During five weeks after sample dispatch, the 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.

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, April 2014 version 3.3). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for each determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

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

3.3 Z-SCORES

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

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

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

The z-scores were calculated in accordance with:

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

The $z_{\text{(target)}}$ 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. Therefore the usual interpretation of z-scores maybe 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 despatch of the samples to the Middle East and South Africa. One participant reported the results after the final reporting date and four participants did not report at all. Not all participants were able to report results for all tests.

In total 51 participants reported 383 numerical results. Observed were 29 outlying results, which is 7.6% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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 reported data. The abbreviations, used in these tables, are listed in appendix 3.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care.

Acidity, Potentiometric: This determination may be problematic for a number of laboratories.

Three laboratories reported a possible false positive result. These

three test results were statistical outliers.

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. The reproducibility stated in ASTM D664:11ae1 can be used from a value of 0.1 g KOH/kg, and is thus not applicable for this sample.

Acidity, Colorimetric: 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

D974:2014e2.

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 participants 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 good agreement with

the requirements of EN60156:95.

The reproducibility of EN60156:95 was determined from Figure 3 of method EN60156:95. The black line in Figure 3 shows the relative standard deviation (=SD/mean or RSDr) as a function of the value of the mean based on six breakdown measurements. To calculate the

repeatability RSDr was multiplied with a factor 2.8. The reproducibility can be estimated from the repeatability by multiplication with a factor 3, which is an empirical factor.

<u>Density at 20°C</u>: This determination was problematic for a number of laboratories.

Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full

agreement with the requirements of 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 good agreement with the

requirements of EN60247:04.

Spec. Resistance: This determination was very problematic. The reported test results

vary over a large range: 5.5 - 5900 G Ω m. Two statistical outliers were observed. The calculated reproducibility after the rejection of

the statistical outliers is 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 amount 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.

Flash Point (COC):

This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D92:2016a.

Flash Point (PMcc):

This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ISO2719:2016-procedure A.

Interf. Surf. Tension: This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D971:2012. One should be aware that ISO6295 is obsolete since February 2005.

Kinematic Viscosity: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D445:15a, but not in agreement with the requirements of ISO3104:94.

> When the test results, performed according to ISO3104 and ASTM D445, were evaluated separately, the calculated reproducibility was smaller, but still not in agreement with the requirements of ISO3104:94. Also test results were reported according to ASTM D7042 (Stabinger viscosity) and ASTM D7279 (Houillon viscosity).

Water:

This determination was problematic for a number of laboratories. Seven statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of EN60814:98.

DBPC anti-oxidant:

The group of participants agreed that DBPC (2.6-di-tert-butyl-pcresol) is not present above the limit of quantification of 100 mg/kg.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity, Total (Potentiometric Titration)	g KOH/kg	27	0.005	0.009	(0.002)
Acidity, Total (Colorimetric Titration)	g KOH/kg	17	0.005	0.009	0.040
Appearance		32	B&C	n.a.	n.a.
Color ASTM		39	L0.5	n.a.	n.a.
Breakdown Voltage	kV/2.5 mm	47	70.8	30.9	59.4
Density at 20°C	kg/m ³	34	876.40	0.62	1.20
Di-electric Dissipation Factor at 90°C		35	0.0006	0.0012	0.0017
Specific Resistance at 90°C	GΩm	30	906	2312	951
Flash Point (COC)	°C	12	159.5	19.7	18.0
Flash Point (PMcc)	°C	29	152.1	7.8	10.8
Interfacial Surface Tension	mN/m	29	48.3	5.0	4.8
Kinematic Viscosity at 40°C	mm²/s	30	9.85	0.14	0.14
Water	mg/kg	36	7.2	4.3	4.0
DBPC Antioxidant Additive	mg/kg	13	<100	n.a.	n.a.

Table 3: Reproducibilities of tests on sample #16250

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

^{() =} Results between brackets were near or below detection limit, these results should be used with care B&C = Bright and Clear

4.3 COMPARISON OF THE NOVEMBER 2016 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2016	November 2015	November 2014	November 2013	October 2012
Number of reporting labs	51	49	52	60	59
Number of results reported	383	330	340	491	427
Statistical outliers	29	26	13	32	30
Percentage outliers	7.6%	7.9%	3.8%	6.5%	7.0%

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 against the target requirements. The conclusions are given the following table:

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

Table 5: comparison determinations against the standard

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

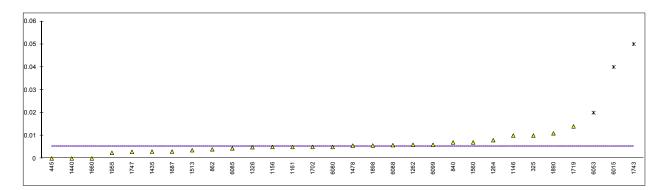
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- group performed worse than the standard
- -- : group performed much worse than the standard
- n.e: not evaluated

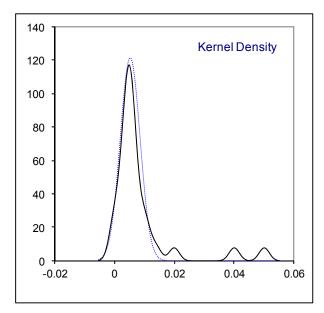
^{() =} Results between brackets were near or below detection limit, these results should be used with care

APPENDIX 1

Determination of Acidity, Total (Potentiometric Titration) on sample #16250; results in a KOH/kg

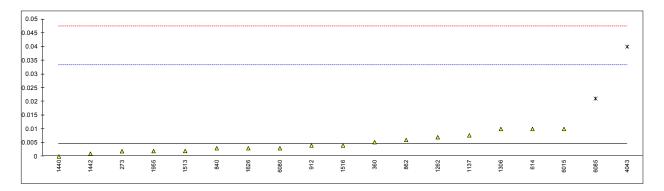
Deteri	mination of Aci	<u>dity, Total</u>	(Potentio	metric '	Titration) on sample #16250; results in g KOH/kg
lab	method	value	mark	z(targ)	remarks
179	D664-A	<0.01			
237	D664-A	<0.1			
273	D004.4				
325	D664-A	0.01			
360	D664 A	0.00			
445 446	D664-A	0.00			
614					
840	IEC62021-1	0.007			
862	IEC62021-1	0.004			
912					
962					
963					
1137					
1146	D664-A	0.010			
1156	IEC62021-1	0.005			
1161	D664-A	0.005			
1262	EN62021-1	0.006			
1264 1304	D664-A	0.008			
1304					
1326	IEC62021-1	0.0049			
1435	IEC62021-1	0.003			
1440	EN62021-1	0.00			
1442					
1444					
1461					
1478	IEC62021-1	0.0056			
1513	IEC62021-1	0.0036			
1516 1560	IEC62021-1	0.007			
1568	ILC02021-1	0.007			
1626					
1628					
1660	IEC62021-1	0.00			
1687	D664-A	0.003			
1702	IEC62021-1	0.005			
1719	D664-A	0.014			
1743	IEC62021-1	0.05	R(0.01)		possible false positive result?
1747	EN62021-1	0.0029			
1760	IEC62021-1	<0.01			
1777 1890	ISO6619	0.011			
1898	EN62021-1	0.005685			
1947	LINOZOZIII				
1955	D664-A	0.0025			
2122					
4043					
6015	D664-A	0.04	R(0.01)		possible false positive result?
6048	IEC62021-1	0.02	C B(0.01)		first reported: 0.03, possible false positive result?
6053 6080	D664-A	0.02	C,R(0.01)		ilist reported. 0.05, possible raise positive result?
6085	D664-A	0.00442			
6088	IEC62021-1	0.0058			
6099	IEC62021-1	0.006	С		first reported: 0.018
	normality	OK			
	n	27			
	outliers	3			
	mean (n) st.dev. (n)	0.0053 0.00329			Compare R(D664:11ae1) = (0.1418, n.a. with value <0.1))
	R(calc.)	0.0092			
	R(EN62021-1:03)	(0.0015)			quantification limit > 0.014 g KOH/kg

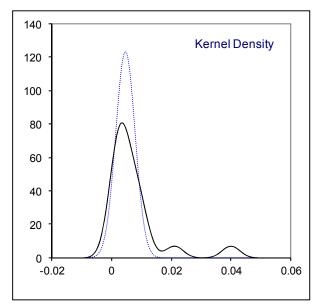




Determination of Acidity, Total (Colorimetric Titration) on sample #16250; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks
179	D974	<0.01			
237	-				
273	D974	0.0019		-0.20	
325					
360	D974	0.0052		0.04	
445	D074				
446	D974	<0.01		0.27	
614	D974 D974	0.01 0.003		0.37 -0.12	
840 862	D974 D974	0.003		0.09	
912	D974	0.0040		-0.05	
962	5011				
963					
1137	D974	0.0077		0.21	
1146					
1156					
1161	1000010				
1262	ISO6618	0.007		0.16	
1264 1304	INILI 100	<0.01			
1304	INH-122 D974	0.01		0.37	
1326	D314				
1435					
1440	ISO6618	0.00		-0.33	
1442	IEC62021-2	0.001		-0.26	
1444					
1461					
1478	1500001.0				
1513	IEC62021-2	0.002		-0.19	
1516	D974	0.004		-0.05 	
1560 1568					
1626	D974	0.003		-0.12	
1628	D014				
1660					
1687					
1702					
1719					
1743					
1747					
1760	D974	<0.01			
1777 1890	D974	<0.01 			
1898					
1947					
1955	IEC62021-2	0.00195		-0.19	
2122	EN62021-2	<0.01			
4043	ISO660	0.04	G(0.01)	2.47	
6015	D974	0.01		0.37	
6048					
6053 6080	D974	0.003		-0.12	
6085	D974 D974	0.003	G(0.01)	1.15	
6088	D314	0.021099	G(0.01)		
6099					
	normality	OK			
	n	17			
	outliers	2			
	mean (n)	0.0047			
	st.dev. (n)	0.00325 0.0091			
	R(calc.) R(D974:14e2)	0.0091			Compare R(IEC62021-2:07) = 0.0164, quantification limit 0.014 g KOH/kg
	14(0017.1702)	0.0-100			compare refreeded to 2.01) 0.0104, quantinoation innit 0.014 g NOT/kg





Determination of Appearance on sample #16250;

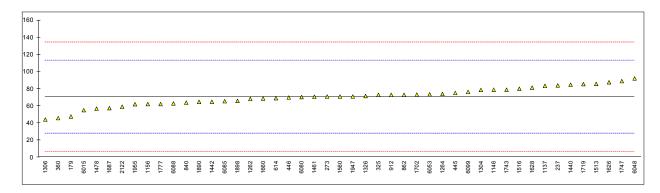
lab	method	value	remarks
179			
237	Visual	Clear and Bright	
273		Bright & Clear	
325	Visual	Bright & Clear	
360	Visual	Clear and Bright	
445	Visual	C & B	
446	D4176	Pass	
614	\ r		
840	Visual Visual	Clear, free from sediment and suspended matter	
862 912	Visual	Colorless Transparent Liquid clear	
962	visuai		
963			
1137			
1146			
1156	Visual	CLEAR	
1161	Visual	Clear and Bright	
1262	Visual	bright and clear	
1264	Visual	Clear & Bright	
1304			
1306			
1326 1435	Visual	clear	
1440	Visual	Limpide	
1442	Visual	clear	
1444	D1524	clear	
1461			
1478	IEC60296	clear	
1513	IEC60296	Limpid	
1516	Visual	Clean	
1560	Visual	Clear, free from sediment and suspended matter	
1568 1626	Visual	clear	
1628	visuai		
1660	Visual	Clear, free from sediments	
1687			
1702	IEC60296	CLEAR	
1719			
1743	Visual	limpide	
1747			
1760	Viewel	Class	
1777 1890	Visual Visual	Clear clear	
1898	Visual	Clear	
1947	Visual		
1955			
2122			
4043			
6015	Visual	as expected	
6048	Visual	bright & clear	
6053	\/:	alan and Dright	
6080 6085	Visual	clear and Bright	
6088	Visual	CLEAR AND CLEAN	
6099	visuai		
0000			
	n	32	
	mean (n)	Bright and clear	

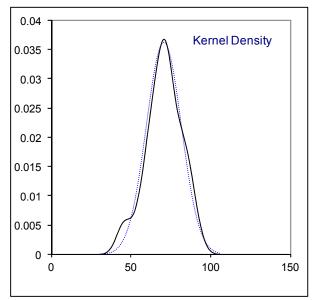
Determination of Color ASTM on sample #16250;

lab	method	value	mark	z(targ)	remarks
179	D1500	L0.5			
237	D1500	L0.5			
273	ISO2049	L0.5			
325	D6045	L0.5			
360	ISO2049	L0.5			
445	ISO2049	<0.5			
446	D1500	<0.5			
614	D1500	<0.5			
840	ISO2049	L0.5			
862	D1500	L0.5			
912					
962					
963					
1137					
1146					
1156	ISO2049	0			
1161	D1500	<0,5			
1262	ISO2049	L 0.5			
1264	D1500	<1			
1304	INH-132	0.0			
1306	D1500	0			
1326					
1435	ISO2049	0.5			
1440	ISO2049	0.00			
1442	ISO2049	L0,5			
1444	ISO2049	0.3			
1461					
1478	ISO2049	0.1			
1513	ISO2049	L0,5			
1516	ISO2049	0			
1560	ISO2049	L0.5			
1568					
1626	D1500	<0.5			
1628					
1660	D1500	0.0			
1687					
1702	D1500	L0.5			
1719	D1524	<0.5			
1743	ISO2049	L0.5			
1747	ISO2049	0			
1760	D.1500				
1777	D1500	<0.5			
1890	D4500				
1898	D1500	< 0,5			
1947					
1955					
2122					
4043	1000040				
6015	ISO2049	0.5			
6048 6053	D1500 ISO2049	0.0			
6080		0.5			
6085	D1500 D1500	<0.5 <0.5			
6088					
6099	D1500 D1500	0.5 0.5			
0099	טוטטו ט	0.0			
	n	39			
	n mean (n)	lower than 0.5			
	mean (n)	iowei liiaii 0.3	,		

Determination of Breakdown Voltage on sample #16250, results in kV/2.5 mm

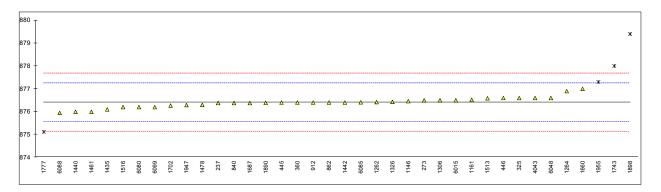
lab	method	value	mark	z(targ)	remarks
179	D877	47.8	mark	-1.08	- Containe
237	IEC60156	84.0		0.62	
273	EN60156	71		0.02	
325	D1816	73		0.11	
360	EN60156	45.9		-1.17	
445	IEC60156	75.2		0.21	
446	EN60156	69.9		-0.04	
614	IEC60156	69.1		-0.04	
840	IEC60156	63.9		-0.32	
862	IEC60156	73		0.11	
912	IEC60156	73		0.11	
962	12000130				
963					
1137	IEC60156	83.5		0.60	
1146	IEC60156	79		0.39	
1156	IEC60156	62.3		-0.40	
1161	12000130				
1262	EN60156	68.2		-0.12	
1264	IEC60156	74		0.15	
1304	INH-124	78.8		0.38	
1306	IEC60156	44.2		-1.25	
1326	IEC60156	71.6		0.04	
1435	12000100				
1440	EN60156	84.8		0.66	
1442	IEC60156	64.971		-0.27	
1444	12000100				
1461	EN60156	70.8		0.00	
1478	IEC60156	56.9		-0.65	
1513	IEC60156	85.8		0.71	
1516	IEC60156	80.2		0.44	
1560	IEC60156	71		0.01	
1568					
1626	IEC60156	87.66		0.80	
1628	EN60156	81.4		0.50	
1660	IEC60156	68.7		-0.10	
1687	IEC60156	57.5		-0.62	
1702	IEC60156	73.3		0.12	
1719	IEC60156	85.5		0.69	
1743	IEC60156	79		0.39	
1747	IEC60156	89		0.86	
1760					
1777	IEC60156	62.3		-0.40	
1890	IEC60156	64.9		-0.28	
1898	EN60156	66		-0.22	
1947	IEC60156	71.1		0.02	
1955	IEC60156	62.1		-0.41	
2122	EN60156	59.1666667		-0.55	
4043					
6015		55.2		-0.73	
6048	IEC60156	92.1		1.01	
6053	IEC60156	73.6		0.13	
6080	IEC60156	70.4		-0.02	
6085	EN60156	65.6		-0.24	
6088	IEC60156	62.9		-0.37	
6099	IEC60156	76.5		0.27	
	normalit.	OK			
	normality	OK			
	n outliers	47 0			
		70.76			
	mean (n) st.dev. (n)	11.035			
	R(calc.)	30.90			Compare R(D877:13) = 21.44
	R(EN60156:95)	59.44			Compare R(D816:12) = 28.30
	1 ([1400 100.00)	55.77			Compare (10 10 10.12) - 20.00

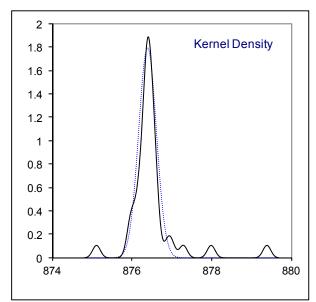




Determination of Density at 20°C on sample #16250; results in kg/m³

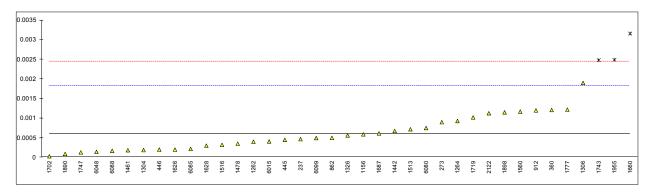
lab	method	value	mark	z(targ)	remarks
179					
237	D4052	876.38		-0.05	
273	ISO3675	876.5		0.23	
325	D4052	876.6	С	0.46	first reported: 0.8766 kg/m ³
360	ISO12185	876.4		0.00	
445	D4052	876.4		0.00	
446	D4052	876.6		0.46	
614					
840	ISO12185	876.38		-0.05	
862	D4052	876.4		0.00	
912	ISO3675	876.4		0.00	
962					
963					
1137	In house	076.46		0.14	
1146 1156	In house	876.46 		0.14	
	ISO12185	876.52		0.28	
1161 1262	ISO3675	876.42		0.28	
1264	D4052	876.9		1.16	
1304	D4032				
1304	D1298	876.5		0.23	
1326	D4052	876.43		0.07	
1435	D4052	876.1		-0.70	
1440	In house	876	С	-0.94	first reported: 0.876 kg/m ³
1442	D7042	876.4		0.00	merrepense read ng m
1444					
1461	ISO3675	876.0		-0.94	
1478	ISO12185	876.3		-0.24	
1513	ISO12185	876.586	С	0.43	first reported: 0.876586 kg/m ³
1516	ISO3675	876.2		-0.47	
1560					
1568					
1626					
1628	5-0.40		_		5
1660	D7042	877.0	С	1.40	first reported: 0.8770 kg/m ³
1687	ISO12185	876.38		-0.05	
1702	ISO12185	876.269		-0.31	
1719 1743		070	R(0.01)	3.73	
1743		878	K(0.01)	3.73	
1747					
1777	D4052	875.12	C,R(0.01)	-2.99	first reported: 0.87512 kg/m ³
1890	ISO12185	876.39	0,11(0.01)	-0.03	mat reported. 0.07312 kg/m
1898	ISO12185	879.4	R(0.01)	7.00	
1947	ISO12185	876.29	11(0.01)	-0.26	
1955	D7042	877.3	R(0.05)	2.10	
2122			/		
4043	ISO6883	876.6		0.46	
6015	ISO12185	876.50		0.23	
6048	ISO12185	876.6		0.46	
6053					
6080	D4052	876.2	_	-0.47	
6085	D7042	876.41	C	0.02	first reported: 0.87641 kg/m ³
6088	ISO3675	875.95	С	-1.05	first reported: 0.87595 kg/m ³
6099	ISO12185	876.2	С	-0.47	first reported: 879.2
	normality	suspect			
	n	34			
	outliers	4			
	mean (n)	876.402			
	st.dev. (n)	0.2220			
	R(calc.)	0.622			Compare $R(D4052:16) = R(ISO12185:96) = 0.500$
	R(ISO3675:98)	1.200			Compare R(D7042:16e3) = 1.300

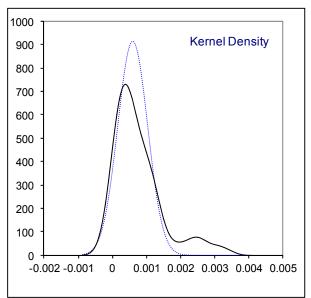




Determination of Di-electric Dissipation Factor at 90°C on sample #16250

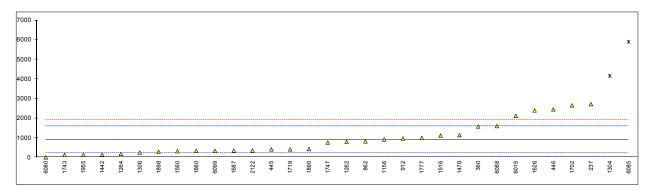
lab	method	value	mark	z(targ)	remarks
179					
237	IEC60247	0.0004675		-0.21	
273	EN60247	0.0009		0.49	
325					
360	EN60247	0.00121		0.99	
445	IEC60247	0.000448		-0.25	
446	EN60247	0.0002		-0.65	
614					
840					
862	IEC60247	0.0005		-0.16	
912	IEC60247	0.0012		0.97	
962	120002-17				
963					
1137					
1146					
1156	IEC60247	0.00059		-0.02	
1161	12000247			-0.02	
1262	EN60247	0.00040		-0.32	
1264				0.54	
1304	IEC60247 INH-125	0.000930 0.000188		-0.67	
1304	EN60247	0.000188		2.11	
1326	IEC60247	0.000562		-0.06	
1435					
1440	IECC0047	0.000070		0.40	
1442	IEC60247	0.000676		0.12	
1444	ENC0047	0.000405		0.07	
1461	EN60247	0.000185		-0.67	
1478	IEC60247	0.000351		-0.40	
1513	IEC60247	0.000719		0.19	
1516	IEC60247	0.000322		-0.45	
1560	IEC60247	0.00117		0.93	
1568	.=				
1626	IEC60247	0.00020		-0.65	
1628	EN60247	0.00030		-0.49	
1660	IEC60247	0.00316	R(0.01)	4.16	
1687	IEC60247	0.000612		0.02	
1702	IEC60247	0.00003		-0.92	
1719	IEC60247	0.00102		0.68	
1743	IEC60247	0.00248	R(0.01)	3.05	
1747	IEC60247	0.00013	С	-0.76	first reported: 0.0130
1760					
1777	IEC60247	0.00122		1.01	
1890	IEC60247	0.00009		-0.83	
1898	IEC60247	0.00115		0.89	
1947					
1955	IEC60247	0.00249	R(0.01)	3.07	
2122	EN60247	0.001128		0.86	
4043					
6015	EN60247	0.000409		-0.31	
6048	IEC60247	0.000141		-0.74	
6053					
6080	IEC60247	0.000750		0.24	
6085	EN60247	0.000216		-0.62	
6088	IEC60247	0.00017		-0.70	
6099	IEC60247	0.000495		-0.17	
	normality	OK			
	n	35			
	outliers	3			
	mean (n)	0.000599			
	st.dev. (n)	0.0004376			
	R(calc.)	0.001225			
	R(EN60247:04)	0.001725			

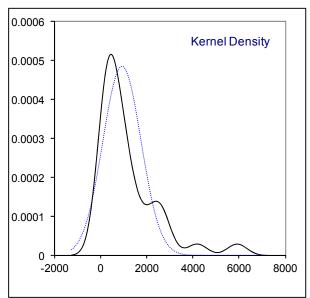




Determination of Specific Resistance at 90°C on sample #16250; results in $G\Omega m$

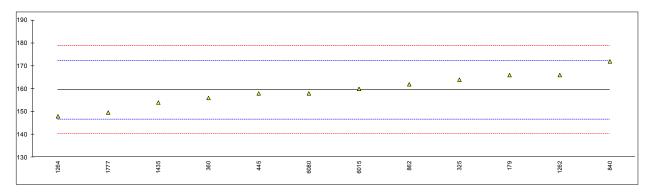
lab	method	value	mark	z(targ)	remarks
179					
237	IEC60247	2720		5.34	
273					
325 360	EN60247	1580		1.99	
445	IEC60247	412.61		-1.45	
446	EN60247	2455		4.56	
614	21100217				
840					
862	IEC60247	820.79		-0.25	
912	IEC60247	969.0		0.19	
962					
963					
1137 1146					
1156	IEC60247	920.3		0.04	
1161	12000217				
1262	EN60247	800		-0.31	
1264	IEC60247	184.53		-2.12	
1304	INH-125	4170.00	R(0.05)	9.61	
1306	EN60247	249.75		-1.93	
1326					
1435 1440					
1442	IEC60247	144.06		-2.24	
1444	12000217				
1461					
1478	IEC60247	1140		0.69	
1513					
1516	IEC60247	1115		0.62	
1560 1568	IEC60247	318.75		-1.73 	
1626	IEC60247	2400		4.40	
1628	12000247				
1660	IEC60247	336.3		-1.68	
1687	IEC60247	353.41		-1.63	
1702	IEC60247	2660		5.17	
1719	IEC60247	414.025	С	-1.45	reported: 141.025 E9 G Ω m (a possible unit error?)
1743	IEC60247	130		-2.28	C + + + + 7500 5
1747 1760	IEC60247	758.35 	С	-0.43 	first reported: 7583.5
1777	IEC60247	1000		0.28	
1890	IEC60247	429.5		-1.40	
1898	IEC60247	295.56		-1.80	
1947					
1955	IEC60247	134		-2.27	
2122	EN60247	358.56		-1.61	
4043	ENGO247	2420		2.50	
6015 6048	EN60247	2120		3.58	
6053					
6080	IEC60247	5.55		-2.65	
6085	EN60247	5900	R(0.01)	14.71	
6088	IEC60247	1600		2.04	
6099	IEC60247	342		-1.66	
	normality	euencet			
	normality n	suspect 30			
	outliers	2			
	mean (n)	905.57			
	st.dev. (n)	825.842			
	R(calc.)	2312.36			
	R(EN60247:04)	950.85			

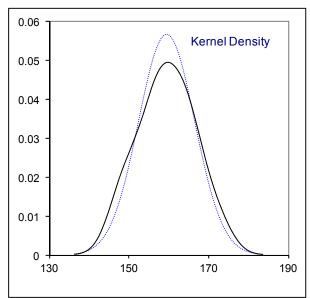




Determination of Flash Point COC on sample #16250; results in °C

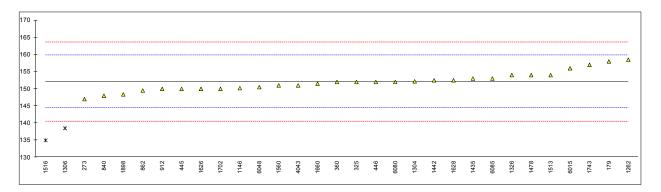
lab	method	value	mark	z(targ)	remarks
179	D92	166		1.02	
237					
273					
325	D92	164		0.70	
360	D92	156		-0.54	
445	D92	158.0		-0.23	
446					
614 840	ISO2592	172		1.95	
862	D92	162		0.39	
912	D02				
962					
963					
1137					
1146					
1156					
1161	1000500	400		4.00	
1262 1264	ISO2592 D92	166	С	1.02 -1.78	first reported as DMcs
1304	D9Z	148 	C	-1.70	first reported as PMcc
1306					
1326					
1435	D92	154		-0.85	
1440					
1442					
1444					
1461					
1478 1513					
1516					
1560					
1568					
1626					
1628					
1660					
1687					
1702 1719					
1743					
1747					
1760					
1777	D92	149.65		-1.53	
1890					
1898					
1947					
1955 2122					
4043					
6015	ISO2592	160		0.08	
6048					
6053					
6080	D92	158		-0.23	
6085					
6088					
6099					
	normality	OK			
	n	12			
	outliers	0			
	mean (n)	159.47			
	st.dev. (n)	7.031			
	R(calc.)	19.69			D/(CO2502,2000) = 47.00
	R(D92:16a)	18.00			compare R(ISO2592:2000) = 17.00

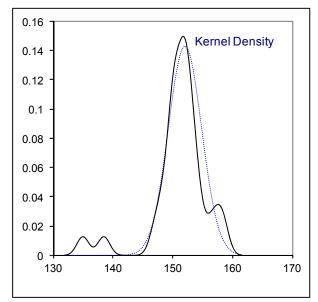




Determination of Flash Point PMcc on sample #16250; results in °C

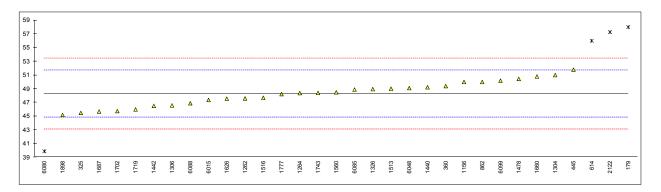
lab	method	value	mark	z(targ)	remarks
179	D93-A	158.0		1.54	
237					
273	D93	147		-1.31	
325	D93-A	152		-0.02	
360 445	ISO2719-A ISO2719-A	152.0 150.0		-0.02 -0.53	
446	D93-A	150.0		-0.53 -0.02	
614	D00 / (
840	ISO2719-A	148.0		-1.05	
862	D93-A	149.5		-0.66	
912	D93-A	150		-0.53	
962 963					
1137					
1146	In house	150.2		-0.48	
1156					
1161	1000740 4	450.5		4.07	
1262	ISO2719-A	158.5		1.67	
1264 1304	INH-115	152.2		0.04	
1304	D7094	138.5	C,R(0.01)	-3.52	first reported as an Open Cup method
1326	D93-A	154.0	-, ()	0.50	, common and the comm
1435	D93-A	153		0.24	
1440	1000740 4	450.5		0.44	
1442 1444	ISO2719-A	152.5 		0.11	
1461					
1478	ISO2719-A	154.0		0.50	
1513	ISO2719-A	154.0		0.50	
1516	D93-A	135	R(0.01)	-4.42	
1560	ISO2719	151		-0.28 	
1568 1626	D93-A	150		-0.53	
1628	ISO2719-A	152.5		0.11	
1660	D93-A	151.5		-0.15	
1687					
1702	ISO2719-B	150		-0.53	
1719 1743	ISO2719-A	 157.0		1.28	
1747	10021 19-1				
1760					
1777					
1890	1000740.0	440.075			
1898 1947	ISO2719-C	148.375		-0.96	
1955					
2122					
4043	ISO15267	151		-0.28	
6015	D7236	156.0		1.02	
6048 6053	D93-A	150.5 		-0.40 	
6080	D93-A	152		-0.02	
6085	D93-A	153		0.24	
6088					
6099					
	normality	ОК			
	n	29			
	outliers	2			
	mean (n)	152.06			
	st.dev. (n)	2.789			
	R(calc.) R(ISO2719:16-A)	7.81 10.80			R(ISO2719:16-A) = R(D93:16a-A) = R(IP34:03-A)
	14(10021 13.10-A)	10.00			$1 \times 1000 = 1 \times 1000 $

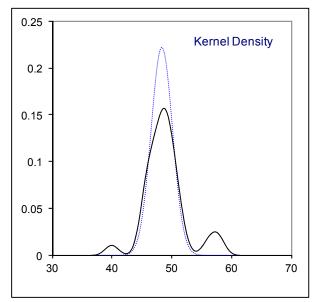




Determination of Interfacial Surface Tension on sample #16250; results in mN/m

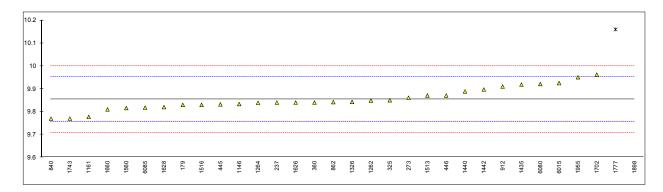
lab	method	value	mark	z(targ)	remarks
179	D971	58	C,R(0.01)	5.62	first reported: 57
237	2011		0,11(0.01)	3.02	mot reported. Of
273					
325	In house	45.5		-1.62	
360	D971	49.4		0.64	
445	D971	51.8		2.03	
446	D37 1			2.03	
614	D971	56	R(0.01)	4.46	
840	D37 1		11(0.01)		
862	ISO6295	50		0.98	
912	1000233				
962					
963					
1137					
1146					
1156	D971	50.0		0.98	
1161	D07 1				
1262	D971	47.6		-0.41	
1264	D971	48.38		0.04	
1304	INH-123	51.0		1.56	
1306	D971	46.58		-1.00	
1326	D971	48.96		0.38	
1435	2011				
1440	D971	49.23		0.54	
1442	EN14210	46.530		-1.03	
1444	21111210				
1461					
1478	D971	50.45		1.24	
1513	D971	49.02		0.42	
1516	D971	47.7		-0.35	
1560	D971	48.5		0.11	
1568					
1626	ISO6295	47.56		-0.43	
1628					
1660	D971	50.8		1.45	
1687	D971	45.7		-1.51	
1702	D971	45.759	С	-1.47	first reported: 42.354
1719	D2285	46		-1.33	
1743	D971	48.4		0.06	
1747					
1760					
1777	D971	48.23		-0.04	
1890					
1898	D971	45.2		-1.80	
1947					
1955	=111.1010		D(0.04)		
2122	EN14210	57.27	R(0.01)	5.20	
4043	D074	47.075		0.54	
6015	D971	47.375		-0.54	
6048	D971	49.11		0.47	
6053	D074		D(0.04)	4.07	
6080	D971	39.9	R(0.01)	-4.87	
6085	D971	48.89		0.34	
6088	ISO6295	46.9		-0.81	
6099	EN14210	50.2		1.10	
	normality	OK			
	normality	OK 29			
	n outliers	29 4			
	mean (n)	48.303			
	st.dev. (n)	46.303 1.7944			
	R(calc.)	5.024			
	R(D971:12)	4.830			
	11(0011.12)	4.000			

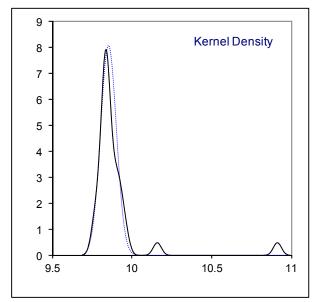




Determination of Kinematic Viscosity at 40°C on sample #16250; results in mm²/s

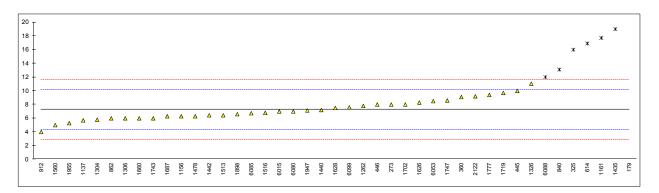
lab	method	value	mark	z(targ)	remarks		
179	D445	9.83		-0.50			
237	D445	9.840		-0.29			
273	D445	9.861		0.14			
325	D445	9.850		-0.09			
360	ISO3104	9.8401		-0.29			
445	ISO3104	9.832		-0.46			
446	D445	9.871		0.35			
614							
840	ISO3104	9.7696		-1.74			
862	ISO3104	9.842		-0.25			
912	D445	9.91		1.15			
962							
963 1137							
1146	ISO3104	9.8336		-0.42			
1156	1303104	9.6556		-0.42			
1161	ISO3104	9.777		-1.59			
1262	ISO3104	9.849		-0.11			
1264	D7042	9.8393		-0.31			
1304	2.0.2						
1306							
1326	D445	9.843		-0.23			
1435	D7042	9.9184		1.32			
1440	ISO3104	9.88891		0.72			
1442	D7042	9.8970		0.88			
1444							
1461							
1478	1000404	0.07005					
1513	ISO3104	9.87095	0	0.35	final namentals 0.00		
1516 1560	ISO3104	9.83	С	-0.50	first reported: 9.30		
1560 1568	ISO3104	9.816 		-0.79 			
1626	D445	9.84		-0.29			
1628	ISO3104	9.820		-0.70			
1660	D7042	9.81		-0.91			
1687							
1702	D7042	9.9622		2.22			
1719							
1743	D7279	9.77		-1.73			
1747							
1760	D.445		0.0(0.04)				
1777	D445	10.16	C,R(0.01)	6.30	first reported: 10.054		
1890 1898	ISO3104	10.913	R(0.01)	21.80			
1947	1003104		11(0.01)				
1955	D7042	9.9507		1.99			
2122							
4043							
6015	D7279	9.925		1.46			
6048							
6053							
6080	D445	9.921		1.38			
6085	D7042	9.8177		-0.75			
6088							
6099					Only ISO3104/D445:	Only D7042	Only D7270:
	normality	OK			OK 01119 1503 104/D445.	Only D7042: OK	Only D7279: unknown
	n	30			21	7	2
	outliers	2			2	0	n.a.
	mean (n)	9.854			9.845	9.885	9.845 (average of results)
	st.dev. (n)	0.0494			0.0362	0.0630	0.155 (difference of results)
	R(calc.)	0.138			0.101	0.176	,
	R(D445:15a)	0.136			0.136		
comp		0.075			0.075		
comp						0.222	0.440
comp	R(D7279:16)						0.149

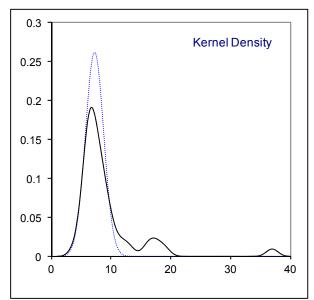




Determination of Water on sample #16250; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D6304-C	37	C,R(0.01)	20.64	first reported: 2.4
237			-,: (-:,		
273	EN60814	8		0.52	
325	D6304-C	16	R(0.01)	6.07	
360	EN60814	9.1		1.29	
445	D6304-B	10		1.91	
446	EN60814	8	D(0.04)	0.52	
614	IEC60814	16.9	R(0.01)	6.70	
840 862	IEC60814 D6304-A	13.10 6	R(0.05)	4.06 -0.86	
912	D6304-A	6 4		-2.25	
962	D0004-0			-2.20	
963					
1137	ISO12937	5.70		-1.07	
1146					
1156	IEC60814	6.3		-0.66	
1161	ISO12937	17.712	C,R(0.01)	7.26	first reported: 12.217
1262	EN60814	7.8		0.39	
1264					
1304	INH-121	5.8		-1.00	
1306	EN60814	6 11.05		-0.86	
1326 1435	D1533 IEC60814		R(0.01)	2.64 8.15	
1440	EN60814	19 7.2	K(0.01)	-0.03	
1442	IEC60814	6.44		-0.56	
1444	12000014			-0.50	
1461					
1478	EN60814	6.3		-0.66	
1513	IEC60814	6.44		-0.56	
1516	IEC60814	6.8		-0.31	
1560	IEC60814	5		-1.56	
1568					
1626	IEC60814	8.3		0.73	
1628	EN60814	7.5		0.18	
1660	EN60814	6		-0.86	
1687 1702	IEC60814	6.296		-0.66	
1702	IEC60814 IEC60814	8 9.7		0.52 1.70	
1743	IEC60814	6		-0.86	
1747	IEC60814	8.6		0.94	
1760					
1777	IEC60814	9.4		1.49	
1890					
1898	EN60814	6.6		-0.45	
1947	IEC60814	7.138		-0.07	
1955	IEC60814	5.2927		-1.35	
2122	EN60814	9.21		1.36	
4043 6015	DIN51777	7.00		-0.17	
6048	TITICHIL	7.00		-0.17	
6053	IEC60814	8.5		0.87	
6080	D1533	7		-0.17	
6085	EN60814	6.7427		-0.35	
6088	D1533	12	C,R(0.05)	3.30	first reported; 20
6099	IEC60814	7.6	, ()	0.25	• •
	normality	OK			
	n	36			
	outliers	7			
	mean (n)	7.245			
	st.dev. (n)	1.5289			Compare R(D1533:12) = 14.000
	R(calc.)	4.281			Compare R(D6304:16e1-C) = 55.422, range: 10-25000
	R(EN60814:98)	4.037			Compare R(ISO12937:00) = 18.510, range: 30-10000





Determination of DBPC Antioxidant additive on sample #16250; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179					
237					
273					
325					
360	IEC60666	<100			
445	ILC00000				
446					
614					
840	IEC60666	0.00			
862	ILC00000				
912	D4768	Not detected			
962	D4700				
963					
1137					
1146					
1156	IEC60666	0			
1161	1200000				
1262	IEC60666	none			
1264	1200000				
1304					
1304					
1326					
1435	IEC60666-B	<loq< td=""><td></td><td></td><td></td></loq<>			
1440	IEC60666	0.00			
1442	IEC60666	<0,01			
1444	1200000				
1461					
1478	IEC60666	0.00			
1513	IEC60666	< 0,01			
1516	IEC60666	0			
1560	IEC60666	Not detectable	•		
1568					
1626	IEC60666	0			
1628					
1660	IEC60666	0.000			
1687					
1702	IEC60666	not detected			
1719					
1743					
1747					
1760					
1777	IEC60666	0.001			
1890					
1898					
1947					
1955					
2122					
4043					
6015					
6048					
6053	15000000				
6080	IEC60666	4			
6085					
6088	IECGOGGG	0.00			
6099	IEC60666	0.00			
	n	13			
	n mean (n)	<100			
	mean (II)	*100			

APPENDIX 2

Number of participants per country

- 2 labs in AUSTRALIA
- 3 labs in BELGIUM
- 3 labs in BULGARIA
- 2 labs in CHINA, People's Republic
- 1 lab in ESTONIA
- 3 labs in FRANCE
- 5 labs in GERMANY
- 1 lab in GREECE
- 1 lab in INDIA
- 1 lab in ISRAEL
- 1 lab in ITALY
- 1 lab in MALAYSIA
- 1 lab in MEXICO
- 1 lab in NETHERLANDS
- 1 lab in NEW ZEALAND
- 1 lab in NIGERIA
- 2 labs in PORTUGAL
- 2 labs in SAUDI ARABIA
- 2 labs in SINGAPORE
- 1 lab in SLOVENIA
- 2 labs in SOUTH AFRICA
- 1 lab in SOUTH KOREA
- 2 labs in SPAIN
- 1 lab in SWEDEN
- 1 lab in SWITZERLAND
- 3 labs in TURKEY
- 5 labs in UNITED ARAB EMIRATES
- 3 labs in UNITED KINGDOM
- 1 lab in UNITED STATES OF AMERICA
- 1 lab in VIETNAM

APPENDIX 3

Abbreviations:

C = final test result after checking of first reported suspect test result

D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DC(0.01) = 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 = probably an error in calculations

U = test result probably reported in a different unit
W = test result withdrawn on request participant
ex = test result excluded from statistical evaluation

n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported
SDS = Safety Data Sheet

B&C = Bright & Clear

Literature:

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