Results of Proficiency Test
Transformer Oil (fresh)
November 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis15L08

February 2016

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1 INTRODUCTION

Since 2001, the Institute for Interlaboratory Studies organized a proficiency test for the analysis of Transformer Oil (fresh) every year. It was decided to continue this interlaboratory study during the annual program 2015/2016. In this interlaboratory study, 52 laboratories from 28 different countries have participated. See appendix 2 for a list of number of participants per country order. In this report, the results of the 2015 interlaboratory study on Transformer Oil (fresh) are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. In this proficiency test, the participants received a bottle of 1 litre of Transformer Oil (fresh), labelled #15222. 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 ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. This PT falls under the accredited scope. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded 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 (DIALA S4 Electrical Insulation Oil) was obtained from a local supplier. The approximately 125 litre bulk material was homogenised in a pre-cleaned drum. After homogenisation, 60 subsamples were transferred to 1 litre amber glass bottles and labelled #15222. The homogeneity of the subsamples #15222 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 #15222-1	804.81
Sample #15222-2	804.82
Sample #15222-3	804.82
Sample #15222-4	804.82
Sample #15222-5	804.83
Sample #15222-6	804.82
Sample #15222-7	804.81
Sample #15222-8	804.82

Table 1: homogeneity test results of subsamples #15222

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

	Density at 20°C in kg/m^3
r (sample #15222)	0.02
reference method	ISO3675:98
$0.3xR_{(reference)}$	0.36

Table 2: repeatability of subsamples #15222

The calculated repeatability of sample #15222 was less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples #15222 was assumed.

To each of the participating laboratories, 1*1 litre bottle (labelled #15222) was sent on November 4, 2015.

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 #15222: Acid Number (Neutralization Number), Breakdown Voltage, Density at 20°C, Di-electric loss at 90°C (Di-electric Dissipation Factor and Specific Resistance), Flash Point, Interfacial Surface Tension, Kinematic Viscosity at 40°C and Water.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/.
A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The original reported 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 yet reported. Shortly after the deadline, the available results were screened for suspect data. A 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 (raw data of the) reported results.
Additional or corrected results have been used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described 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 results. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.
In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and Rosner outlier tests. Outliers are marked

by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 3, no.16). Stragglers are marked by D(0.05) for the Dixon 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 under 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 (see appendix 3; nos.14 and 15). 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:

$z_{(target)}$ = (result - average of PT) / target standard deviation

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. 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, no problems were encountered with the despatch of the samples. In total 7 participants reported the results after the final reporting date and 3 participants did not report at all. Not all participants were able to report results for all tests.

In total 49 participants reported 330 numerical results. Observed were 26 outlying results, which is 7.9% 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 results are discussed per sample and per test. The specified test methods and requirements were taken into account for explaining the observed differences when possible and applicable. These 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.

Acid Number: This determination may be problematic for a number of laboratories. Six laboratories reported possibly a false positive result. No significant conclusions were drawn as the Acid Number was below the quantification limit (0.014 g KOH/kg) of the test method EN62021-1:03.

Breakdown Voltage: 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. The black line in Figure 3 of EN60156:95 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.

Density at 20°C: This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ISO3675:98.

DD-Factor: This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of EN60247:04.

Spec. Resistance: This determination was very problematic. The reported test results vary over a large range: 5.491 - 20880 GΩm. Four statistical outliers were observed and one test result was excluded, for the test temperature used was 20°C instead of 90°C. The calculated reproducibility after the rejection of the suspect data is not at all in agreement with the requirements of EN60247:04.

One participant remarked that it is well known that specific resistance of new oils can vary in a big 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: This determination was not problematic. Three laboratories were excluded as the test results were reported according to ASTM D92 which is not equivalent to ISO2719/ASTM D93/IP34 method A. No statistical outliers were observed. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ISO2719:02-A.

Interf. Surf. Tension: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D971:12. One should be aware that ISO6295 is obsolete since February 2005.

Kinematic Viscosity: This determination was problematic. Five statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers is not in agreement with the requirements of ISO3104:96.

Water: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN60814:98.

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)
Acid Number (EN62021-1)	g KOH/kg	31	0.005	0.007	(0.002)
Breakdown Voltage	kV/2.5 mm	46	50.0	40.3	79.8
Density at 20°C	kg/m^3	33	804.88	1.00	1.20
Di-electric Dissipation Factor at 90°C		34	0.0002	0.0007	0.0014
Specific Resistance at 90°C	GΩm	22	2793	7613	2933
Flash Point	°C	27	192	15	14
Interfacial Surface Tension	mN/m	31	50.7	10.9	5.1
Kinematic Viscosity at 40°C	mm^2/s	32	10.00	0.12	0.08
Water	mg/kg	45	18	8	6

Table 3: Performance of the group on sample #15222

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

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

4.3 COMPARISON OF THE NOVEMBER 2015 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2015	November 2014	November 2013	October 2012	November 2011
Number of reporting labs	49	52	60	59	56
Number of results reported	330	340	491	427	378
Statistical outliers	26	13	32	30	27
Percentage outliers	7.9%	3.8%	6.5%	7.0%	7.1%

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 2015	November 2014	November 2013	October 2012	November 2011
Acid number (EN62021-1)	(--)	(--)	(--)	(--)	n.e.
Breakdown Voltage	++	++	--	--	--
Density at 20°C	+	+/-	+/-	+	-
Di-electric Dissipation Factor	++	++	++	++	++
Specific Resistance	--	--	--	-	--
Flash Point	+/-	-	+/-	n.e.	n.e.
Interfacial Surface Tension	--	+/-	--	--	--
Kinematic Viscosity at 40°C	-	--	--	n.e.	n.e.
Water	-	-	-	-	+

Table 5: comparison determinations against the standard

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

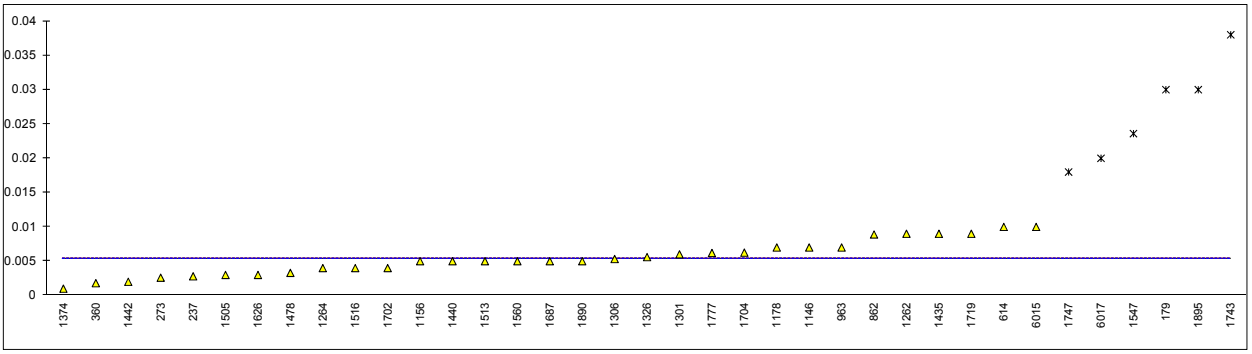
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

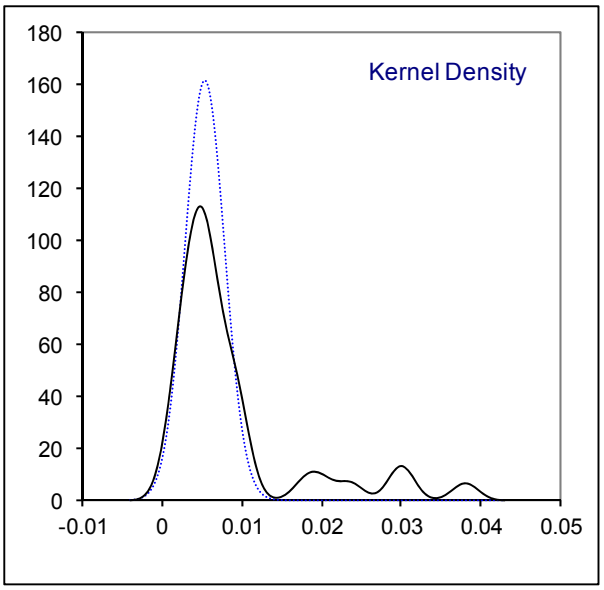
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e: not evaluated

APPENDIX 1

Determination of Acid Number on sample #15222; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks
179	D664	0.03	R(0.01)	-----	possible false positive result?
237	D974	0.0028		-----	
273	D974	0.0026		-----	
360	EN62021-2	0.0018		-----	
445	EN62021-1	<0.01		-----	
446		-----		-----	
541	D664	< 0.1		-----	
614	EN62021-1	0.01		-----	
862	D664	0.0089		-----	
963	D974	0.007		-----	
1146	D664	0.007		-----	
1156	IEC62021-1	0.005		-----	
1178	IEC62021-1	0.007		-----	
1262	EN62021-1	0.009		-----	
1264	D664	0.004		-----	
1301	D974	0.006		-----	
1303		-----		-----	
1304	INH-122	<0.01		-----	
1306	D974	0.00532		-----	
1326	IEC62021-1	0.0056		-----	
1374	IEC62021-1	0.001		-----	
1417	D664	<0.05		-----	
1435	IEC62021-1	0.009		-----	
1440	EN62021-1	0.005		-----	
1442	IEC62021-2	0.002		-----	
1461		-----		-----	
1478	IEC62021-1	0.0033		-----	
1505	D974	0.003		-----	
1513	IEC62021-1	0.005		-----	
1516	D974	0.004		-----	
1543		-----		-----	
1547	D974	0.0236	R(0.01)	-----	possible false positive result?
1560	EN62021-1	0.005		-----	
1626	D974	0.003		-----	
1628		-----		-----	
1660	EN62021-1	<0.01		-----	
1687	D664	0.005		-----	
1702	IEC62021-1	0.004		-----	
1704	IEC62021-1	0.00625		-----	
1719	D664	0.009		-----	
1720		-----		-----	
1743	IEC62021-1	0.038	C,R(0.01)	-----	first reported: 0.057, possible false positive result?
1747	IEC62021-1	0.0180	R(0.01)	-----	possible false positive result?
1760		-----		-----	
1777	EN62021-1	0.0062		-----	
1890	ISO6619	0.005		-----	
1895	D664	0.03	R(0.01)	-----	possible false positive result?
1947		-----		-----	
2122	EN62021-1	<0.01		-----	
6000		-----		-----	
6015	ISO6618	0.01		-----	
6017	EN62021-1	0.02	R(0.01)	-----	possible false positive result?
	normality	OK			
	n	31			
	outliers	6			
	mean (n)	0.0054			
	st.dev. (n)	0.00248			
	R(calc.)	0.0069			
	R(EN62021-1:03)	(0.0015)			quantification limit >0.014 g KOH / kg

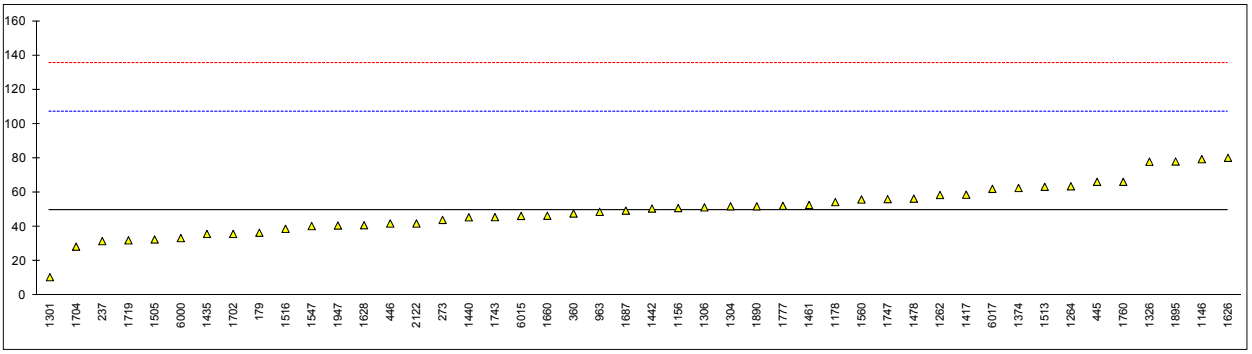

0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
1374 360 1442 273 237 1505 1626 1478 1264 1516 1702 1156 1440 1513 1560 1687 1890 1306 1326 1301 1777 1704 1178 1146 963 862 1262 1435 1719 614 6015 1747 6017 1547 179 1895 1743

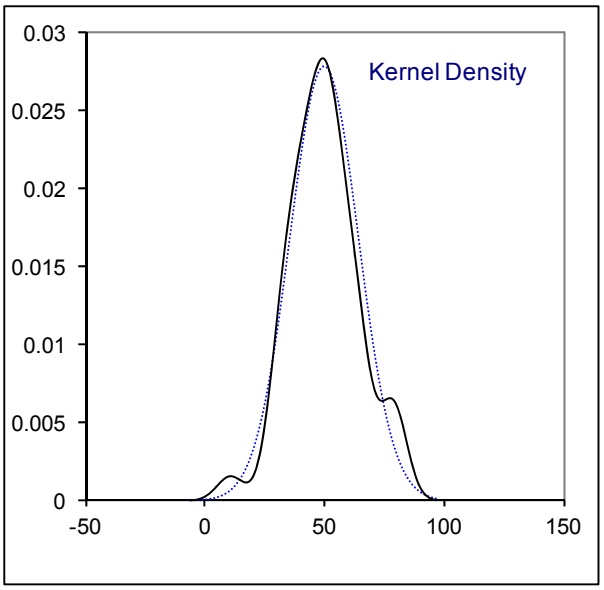

Kernel Density
180
160
140
120
100
80
60
40
20
0
-0.01 0 0.01 0.02 0.03 0.04 0.05

Determination of Breakdown Voltage on sample #15222, results in kV/2.5 mm

lab	method	value	mark	z(targ)	remarks
179	D877	36.6		-0.47	
237	IEC60156	31.7		-0.64	
273	IEC60156	44.1		-0.21	
360	EN60156	47.8		-0.08	
445	IEC60156	66.3		0.57	
446	IEC60156	42		-0.28	
541		-----		-----	
614		-----		-----	
862		-----		-----	
963	D877	48.8		-0.04	
1146	IEC156	79.5		1.03	
1156	IEC60156	51.0		0.03	
1178	IEC60156	54.5		0.16	
1262	EN60156	58.6		0.30	
1264	IEC60156	63.7		0.48	
1301	IEC60156	10.70		-1.38	
1303		-----		-----	
1304	INH-124	52		0.07	
1306	IEC60156	51.4		0.05	
1326	IEC60156	78.0		0.98	
1374	IEC60156	62.7		0.45	
1417	EN60156	58.8		0.31	
1435	IEC60156	35.9		-0.50	
1440	EN60156	45.6		-0.15	
1442	IEC60156	50.7		0.02	
1461	EN60156	52.8		0.10	
1478	IEC60156	56.5		0.23	
1505	IEC60156	32.7		-0.61	
1513	IEC60156	63.4		0.47	
1516	IEC60156	38.9		-0.39	
1543		-----		-----	
1547	IEC60156	40.5		-0.33	
1560	IEC60156	56		0.21	
1626	IEC60156	80.3		1.06	
1628	EN60156	41.0		-0.32	
1660	EN60156	46.5		-0.12	
1687	EN60156	49.5		-0.02	
1702	IEC60156	35.9		-0.50	
1704	IEC60156	28.5		-0.75	
1719	IEC60156	32.2		-0.62	
1720		-----		-----	
1743	IEC60156	45.7		-0.15	
1747	IEC60156	56.2		0.22	
1760	IEC60156	66.3		0.57	
1777	IEC60156	52.3		0.08	
1890	EN60156	52		0.07	
1895	IEC156	78.125		0.99	
1947	IEC60156	40.8		-0.32	
2122	EN60156	42		-0.28	
6000	EN60156	33.475		-0.58	
6015	EN60156	46.4		-0.13	
6017	IEC60156	62.2		0.43	

normality	OK
n	46
outliers	0
mean (n)	50.01
st.dev. (n)	14.379
R(calc.)	40.26
R(EN60156:95)	79.82

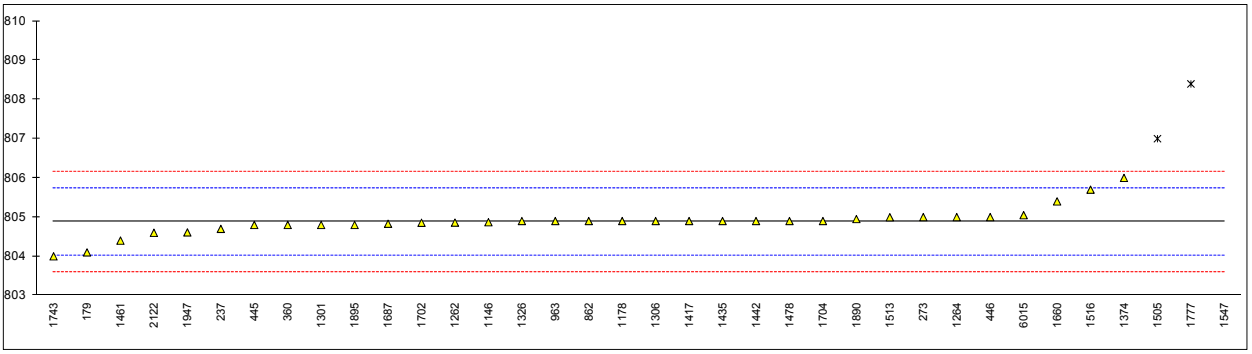

160
140
120
100
80
60
40
20
0
1301 1704 237 1719 1505 6000 1435 1702 179 1516 1547 1947 1628 446 2122 273 1440 1743 6015 1660 360 963 1687 1442 1156 1306 1304 1890 1777 1461 1178 1560 1747 1478 1262 1417 6017 1374 1513 1264 445 1760 1326 1895 1146 1626

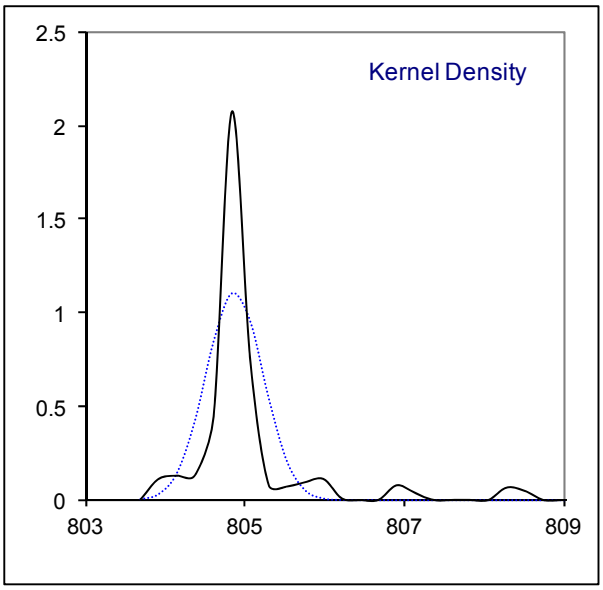

Kernel Density
0.03
0.025
0.02
0.015
0.01
0.005
0
-50
0
50
100
150

Determination of Density at 20°C on sample #15222; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
179	D4052	804.1		-1.83	
237	D4052	804.7		-0.43	
273	D4052	805.0		0.27	
360	D4052	804.8		-0.19	
445	D4052	804.8		-0.19	
446	D4052	805.0		0.27	
541		-----		-----	
614		-----		-----	
862	D4052	804.9		0.04	
963	D4052	804.9		0.04	
1146	ISO12185	804.87		-0.03	
1156		-----		-----	
1178	ISO12185	804.90		0.04	
1262	D4052	804.86		-0.05	
1264	D4052	805.0		0.27	
1301	D4052	804.8	C	-0.19	first reported: 0.8048 kg/m^3
1303		-----		-----	
1304		-----		-----	
1306	D4052	804.9		0.04	
1326	D4052	804.90		0.04	
1374	D7777	806		2.61	
1417	D4052	804.9		0.04	
1435	D4052	804.9	C	0.04	reported: 0.8049 kg/m^3
1440		-----		-----	
1442	D7042	804.9		0.04	
1461	ISO3675	804.4		-1.13	
1478	ISO12185	804.9		0.04	
1505	D7042	807.0	R(0.01)	4.94	
1513	ISO12185	804.997		0.27	
1516	ISO3675	805.7		1.91	
1543		-----		-----	
1547	D1298	862	C,R(0.01)	133.27	reported: 0.862 kg/m^3
1560		-----		-----	
1626		-----		-----	
1628		-----		-----	
1660	D7042	805.4		1.21	
1687	ISO12185	804.83		-0.12	
1702	ISO3675	804.856		-0.06	
1704	ISO3675	804.9	C	0.04	first reported: 802.5
1719		-----		-----	
1720		-----		-----	
1743		804		-2.06	
1747		-----		-----	
1760		-----		-----	
1777	D4052	808.397	C,R(0.01)	8.20	first reported: 877.4
1890	ISO12185	804.95		0.16	
1895	D4052	804.8	C	-0.19	reported: 0.8048 kg/m^3
1947	ISO12185	804.61		-0.64	
2122	INH-12185	804.6		-0.66	
6000		-----		-----	
6015	ISO12185	805.05		0.39	
6017		-----		-----	

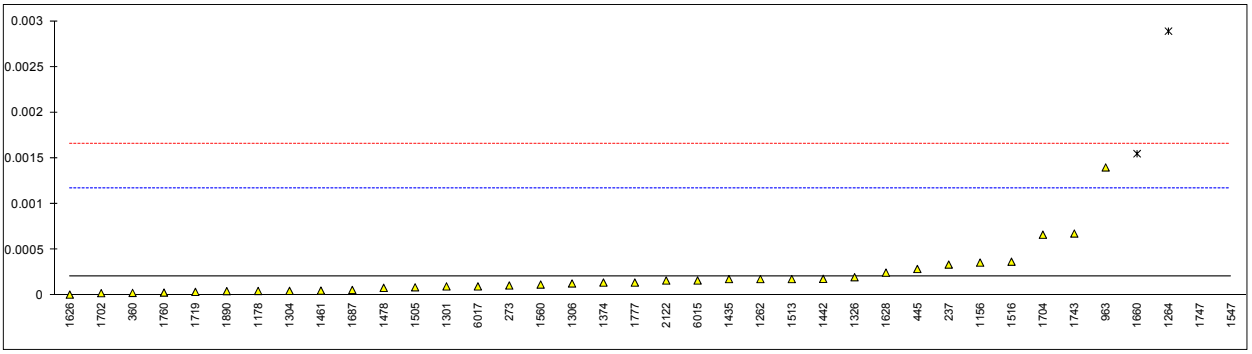
normality	not OK
n	33
outliers	3
mean (n)	804.883
st.dev. (n)	0.3587
R(calc.)	1.004
R(ISO3675:98)	1.200

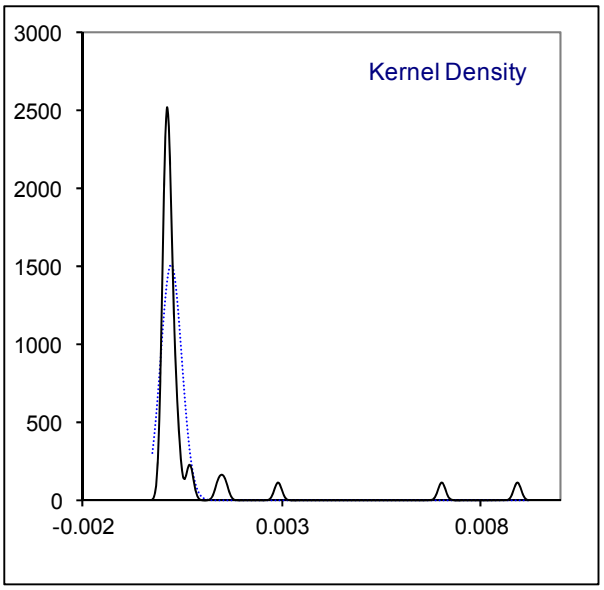

810
809
808
807
806
805
804
803
1743
179
1461
2122
1947
237
445
360
1301
1895
1687
1702
1262
1146
1326
963
862
1178
1306
1417
1435
1442
1478
1704
1890
1513
273
1264
446
6015
1660
1516
1374
1505
1777
1547


Kernel Density
2.5
2
1.5
1
0.5
0
803
805
807
809

Determination of Di-electric Dissipation Factor at 90°C on sample #15222

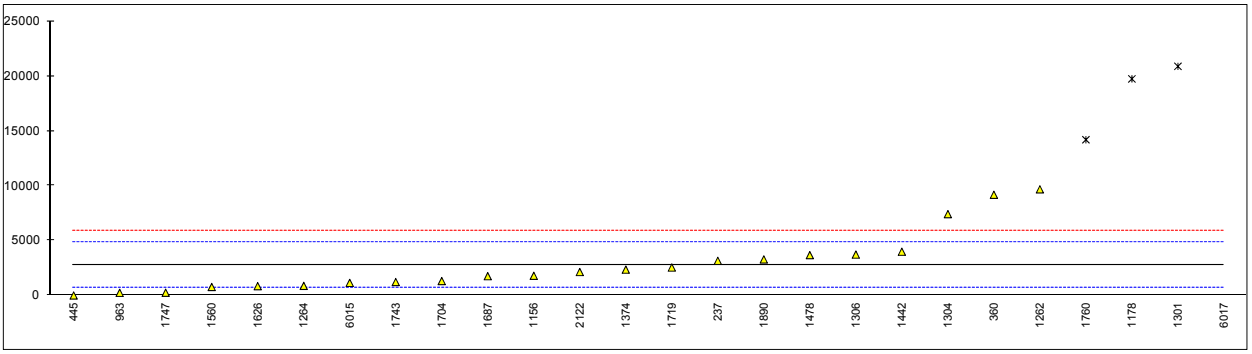
lab	method	value	mark	z(targ)	remarks
179		-----		-----	
237	IEC60247	0.000338		0.27	
273	IEC60247	0.00011		-0.20	
360	EN60247	0.000029		-0.37	
445	IEC60247	0.00029		0.17	
446	IEC60247	<0.0010		-----	
541		-----		-----	
614		-----		-----	
862		-----		-----	
963	IEC60247	0.0014		2.48	
1146		-----		-----	
1156	IEC60247	0.00036		0.32	
1178	IEC60247	0.00005		-0.32	
1262	EN60247	0.00018		-0.06	
1264	IEC60247	0.00289	R(0.01)	5.57	
1301	EN60247	0.00010		-0.22	
1303		-----		-----	
1304	INH-125	0.000051		-0.32	
1306	IEC60247	0.000132		-0.15	
1326	IEC60247	0.0002		-0.01	
1374	IEC60247	0.000142		-0.13	
1417		-----		-----	
1435	EN60247	0.00018		-0.06	
1440		-----		-----	
1442	IEC60247	0.000182		-0.05	
1461	EN60247	0.000054		-0.32	
1478	IEC60247	0.000084		-0.25	
1505	IEC60247	0.00009		-0.24	
1513	IEC60247	0.00018		-0.06	
1516	IEC60247	0.00037		0.34	
1543		-----		-----	
1547	D924	0.0089	R(0.01)	18.03	
1560	IEC60247	0.00012		-0.18	
1626	IEC60247	0.00001		-0.41	
1628	EN60247	0.00025		0.09	
1660	EN60247	0.00155	R(0.01)	2.79	
1687	EN60247	0.000061		-0.30	
1702	IEC60247	0.000026		-0.37	
1704	IEC60247	0.000665		0.95	
1719	IEC60247	0.00004		-0.35	
1720		-----		-----	
1743	IEC60247	0.000678		0.98	
1747	IEC60247	0.0070	R(0.01)	14.09	
1760	IEC60247	0.000032		-0.36	
1777	IEC60247	0.000142		-0.13	
1890	IEC60247	0.000048		-0.33	
1895		-----		-----	
1947		-----		-----	
2122	EN60247	0.000164		-0.09	
6000		-----		-----	
6015	EN60247	0.0001645		-0.09	
6017	EN60247	0.0001		-0.22	
	normality	not OK			
	n	34			
	outliers	4			
	mean (n)	0.000207			
	st.dev. (n)	0.0002641			
	R(calc.)	0.000739			
	R(EN60247:04)	0.001350			

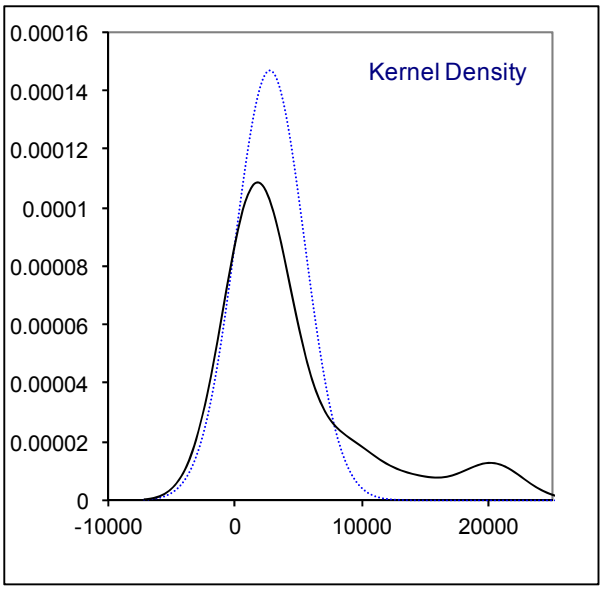

0.003
0.0025
0.002
0.0015
0.001
0.0005
0
1626
1702
360
1760
1719
1890
1178
1304
1461
1687
1478
1505
1301
6017
273
1560
1306
1374
1777
2122
6015
1435
1262
1513
1442
1326
1628
445
237
1156
1516
1704
1743
963
1660
1264
1747
1547


Kernel Density
3000
2500
2000
1500
1000
500
0
-0.002
0.003
0.008

Determination of Specific Resistance at 90°C on sample #15222; results in GΩm

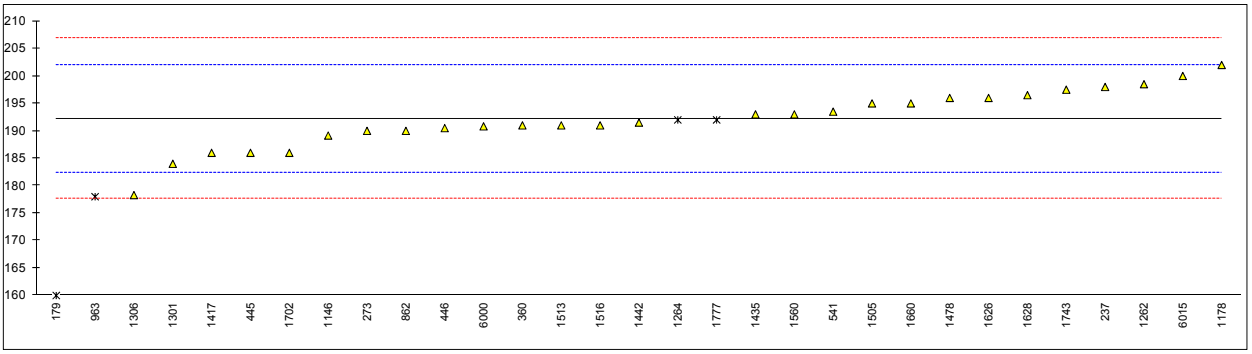
lab	method	value	mark	z(targ)	remarks
179		-----		-----	
237	IEC60247	3160	C	0.35	first reported: 316
273		-----		-----	
360	EN60247	9180		6.10	
445	IEC60247	5.491		-2.66	
446		-----		-----	
541		-----		-----	
614		-----		-----	
862		-----		-----	
963	D1169	244.0		-2.43	
1146		-----		-----	
1156	IEC60247	1800		-0.95	
1178	IEC60247	19736.6	C,R(0.01)	16.18	first reported: 20000.0
1262	EN60247	9675.2		6.57	
1264	IEC60247	880.03		-1.83	
1301	EN60247	20880	R(0.01)	17.27	
1303		-----		-----	
1304	INH-125	7415		4.41	
1306	IEC60247	3730	C	0.89	first reported: 3.73
1326		-----		-----	
1374	IEC60247	2360		-0.41	
1417		-----		-----	
1435	IEC60247	>20000.0		-----	
1440		-----		-----	
1442	IEC60247	3990		1.14	
1461		-----		-----	
1478	IEC60247	3680		0.85	
1505		-----		-----	
1513		-----		-----	
1516		-----		-----	
1543		-----		-----	
1547		-----		-----	
1560	IEC60247	776		-1.93	
1626	IEC60247	854		-1.85	
1628		-----		-----	
1660		-----		-----	
1687	EN60247	1760		-0.99	
1702	IEC60247	50000	C,R(0.01)	45.07	first reported: 100000
1704	IEC60247	1320		-1.41	
1719	IEC60247	2550	C	-0.23	reported: 2550E9 GΩm (possible unit error?)
1720		-----		-----	
1743	IEC60247	1230		-1.49	
1747	IEC60247	257.3	C	-2.42	first reported: 25730.0
1760	IEC60247	14190	R(0.05)	10.88	
1777		-----		-----	
1890	IEC60247	3290		0.47	
1895		-----		-----	
1947		-----		-----	
2122	EN60247	2140		-0.62	
6000		-----		-----	
6015	EN60247	1150		-1.57	
6017	EN60247	$7.98*10^{14}$	ex	-----	excluded, testtemp. used was 20°C
	normality	not OK			
	n	22			
	outliers	4 (+1ex)			
	mean (n)	2793.05			
	st.dev. (n)	2718.750			
	R(calc.)	7612.50			
	R(EN60247:04)	2932.70			

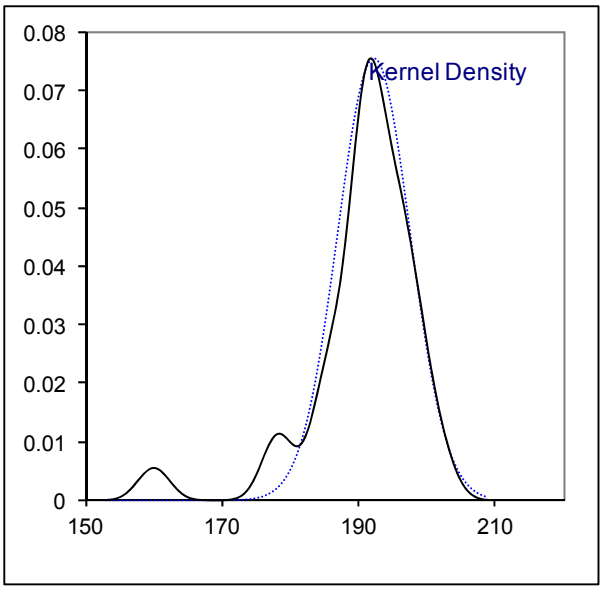

25000
20000
15000
10000
5000
0
445
963
1747
1560
1626
1264
6015
1743
1704
1687
1156
2122
1374
1719
237
1890
1478
1306
1442
1304
360
1262
1760
1178
1301
6017


Kernel Density
0.00016
0.00014
0.00012
0.0001
0.00008
0.00006
0.00004
0.00002
0
-10000
0
10000
20000

Determination of Flash Point PMcc on sample #15222; results in °C

lab	method	value	mark	z(targ)	remarks
179	D93	160.0	R(0.01)	-6.61	
237	D93	198		1.19	
273	D93	190	C	-0.45	first reported: 216
360	ISO2719	191.0		-0.25	
445	ISO2719	186.0		-1.27	
446	D93	190.5		-0.35	
541	ISO2719	193.5		0.27	
614		-----		-----	
862	D93	190.0		-0.45	
963	D92	178	ex	-2.91	excluded for method is not equivalent to Flash Point PMcc
1146	in house	189.15		-0.62	
1156		-----		-----	
1178	ISO2719	202.0		2.01	
1262	ISO2719	198.5		1.29	
1264	D92	192	ex	-0.04	excluded for method is not equivalent to Flash Point PMcc
1301	D93	184		-1.68	
1303		-----		-----	
1304		-----		-----	
1306	D93	178.3		-2.85	
1326		-----		-----	
1374		-----		-----	
1417	IP34	186.0		-1.27	
1435	D93	193		0.17	
1440		-----		-----	
1442	ISO2719	191.5		-0.14	
1461		-----		-----	
1478	ISO2719	196.0		0.78	
1505	D93	195		0.58	
1513	ISO2719	191.0		-0.25	
1516	ISO2719	191		-0.25	
1543		-----		-----	
1547	D92	180 <	ex	-----	excluded for method is not equivalent to Flash Point PMcc
1560	ISO2719	193		0.17	
1626	D93	196		0.78	
1628	ISO2719	196.5		0.88	
1660	D93	195		0.58	
1687		-----		-----	
1702	ISO2719	186.0		-1.27	
1704		-----		-----	
1719		-----		-----	
1720		-----		-----	
1743	ISO2719	197.5		1.09	
1747		-----		-----	
1760		-----		-----	
1777	D92	192	ex,C	-0.04	excluded for method is not equivalent to Flash Point PMcc
1890		-----		-----	
1895		-----		-----	
1947		-----		-----	
2122		-----		-----	
6000	ISO2719	190.825		-0.28	
6015	D7236	200.0		1.60	
6017		-----		-----	
	normality	OK			
	n	27			
	outliers	1 (+3ex)			
	mean (n)	192.20			
	st.dev. (n)	5.294			
	R(calc.)	14.823			
	R(ISO2719:02-A)	13.646			R(ISO2719:02-A) = R(D93:15a-A) = R(IP34:03-A)

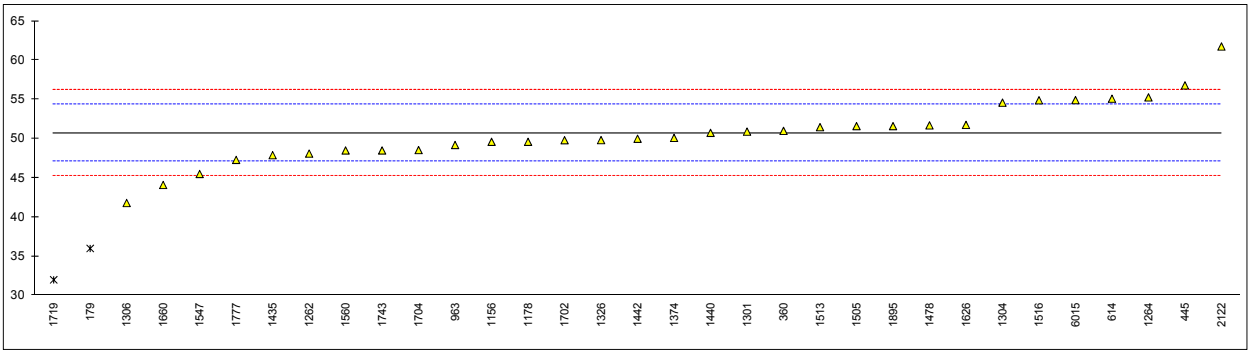

210
205
200
195
190
185
180
175
170
165
160
179
963
1306
1301
1417
445
1702
1146
273
862
446
6000
360
1513
1516
1442
1264
1777
1435
1560
541
1505
1660
1478
1626
1628
1743
237
1262
6015
1178

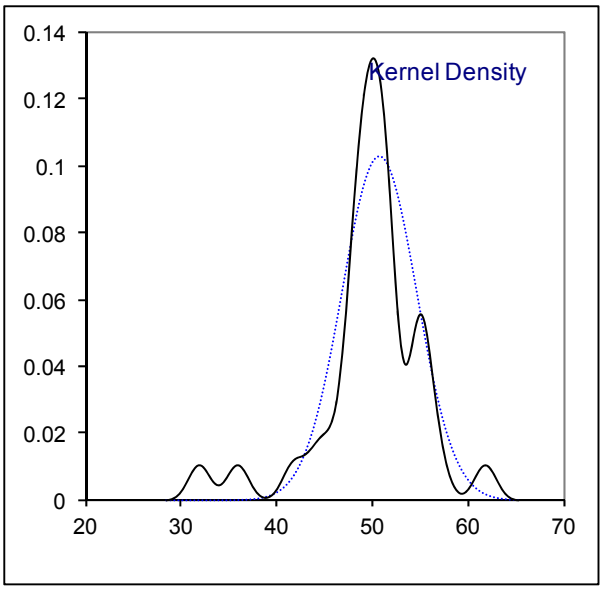

Kernel Density
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
150
170
190
210

Determination of Interfacial Surface Tension on sample #15222; results in mN/m

lab	method	value	mark	z(targ)	remarks
179	D971	36	R(0.05)	-8.13	
237		-----		-----	
273		-----		-----	
360	D971	51.0		0.15	
445	D971	56.8		3.35	
446		-----		-----	
541		-----		-----	
614	ISO6295	55.1		2.41	
862		-----		-----	
963	D971	49.2		-0.84	
1146		-----		-----	
1156	D971	49.6		-0.62	
1178	D971	49.6		-0.62	
1262	D971	48.1		-1.45	
1264	D971	55.27		2.51	
1301	D971	50.9		0.10	
1303		-----		-----	
1304	INH-123	54.6		2.14	
1306	D971	41.8		-4.93	
1326	ISO6295	49.82		-0.50	
1374	D971	50.1		-0.35	
1417		-----		-----	
1435	ISO6295	47.9		-1.56	
1440	D971	50.75		0.01	
1442	EN14210	49.995		-0.40	
1461		-----		-----	
1478	D971	51.7		0.54	
1505	D971	51.6		0.48	
1513	D971	51.47		0.41	
1516	D971	54.9		2.30	
1543		-----		-----	
1547	D971	45.5		-2.88	
1560	D971	48.5		-1.23	
1626	ISO6295	51.76		0.57	
1628		-----		-----	
1660	D971	44.1		-3.66	
1687		-----		-----	
1702	D971	49.811		-0.51	
1704	ISO6295	48.55		-1.20	
1719	in house	32	R(0.05)	-10.34	
1720		-----		-----	
1743	D971	48.5		-1.23	
1747		-----		-----	
1760		-----		-----	
1777	D971	47.3		-1.89	
1890		-----		-----	
1895	IEC971	51.6		0.48	
1947		-----		-----	
2122	ISO6295	61.76		6.09	
6000		-----		-----	
6015	D971	54.925		2.32	
6017		-----		-----	

normality	suspect
n	31
outliers	2
mean (n)	50.726
st.dev. (n)	3.8828
R(calc.)	10.872
R(D971:12)	5.073

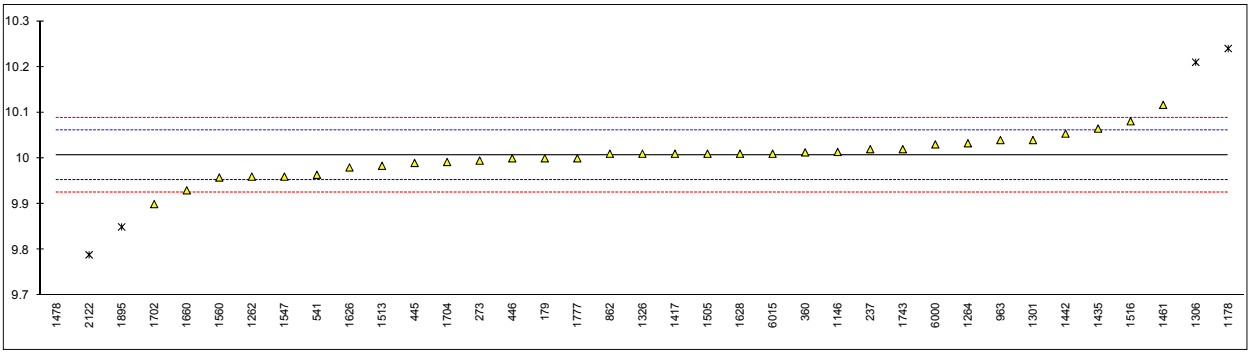

65
60
55
50
45
40
35
30
1719
179
1306
1660
1547
1777
1435
1262
1560
1743
1704
963
1156
1178
1702
1326
1442
1374
1440
1301
360
1513
1505
1895
1478
1626
1304
1516
6015
614
1264
445
2122

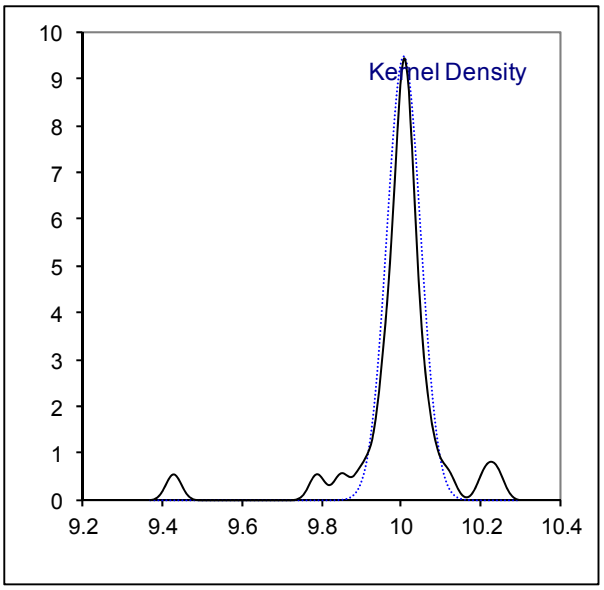

Kernel Density
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
20
30
40
50
60
70

Determination of Kinematic Viscosity at 40°C on sample #15222; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
179	D445	10.0		-0.23	
237	D445	10.02		0.51	
273	D445	9.995		-0.41	
360	ISO3104	10.013		0.25	
445	ISO3104	9.9899		-0.60	
446	D445	10.00		-0.23	
541	ISO3104	9.964		-1.56	
614		-----		-----	
862	D445	10.01		0.14	
963	D445	10.04		1.24	
1146	D445	10.014		0.29	
1156		-----		-----	
1178	DIN53015	10.24	R(0.01)	8.61	
1262	D7042	9.960		-1.70	
1264	D7042	10.033		0.99	
1301	D445	10.04	C	1.24	first reported: 9.782
1303		-----		-----	
1304		-----		-----	
1306	D445	10.21	R(0.01)	7.50	
1326	D445	10.01		0.14	
1374		-----		-----	
1417	D7279	10.01		0.14	
1435	D7042	10.065		2.16	
1440		-----		-----	
1442	D7042	10.054		1.76	
1461	ISO3104	10.1169		4.07	
1478	ISO3104	9.43	C,R(0.01)	-21.22	first reported: 10.43
1505	D7042	10.010		0.14	
1513	ISO3104	9.98359		-0.83	
1516	ISO3104	10.081		2.75	
1543		-----		-----	
1547	D445	9.96		-1.70	
1560	ISO3104	9.958		-1.78	
1626	D445	9.98		-0.97	
1628	ISO3104	10.010		0.14	
1660	D7042	9.93		-2.81	
1687		-----		-----	
1702	ISO3104	9.9		-3.91	
1704	ISO3104	9.992	C	-0.52	first reported: 10.23
1719		-----		-----	
1720		-----		-----	
1743	ISO3104	10.02	C	0.51	first reported: 10.4
1747		-----		-----	
1760		-----		-----	
1777	D88	10.00	C	-0.23	first reported: 10.91
1890		-----		-----	
1895	D445	9.85	R(0.05)	-5.75	
1947		-----		-----	
2122	INH-445	9.789	R(0.01)	-8.00	
6000	ISO3104	10.03042		0.89	
6015	D7279	10.01		0.14	
6017		-----		-----	

normality	suspect
n	32
outliers	5
mean (n)	10.006
st.dev. (n)	0.0422
R(calc.)	0.118
R(ISO3104:96)	0.076

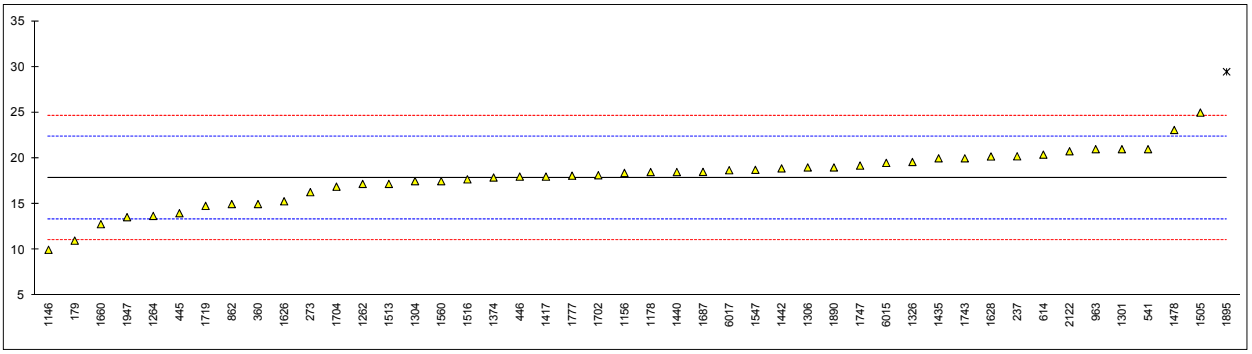

10.3
10.2
10.1
10
9.9
9.8
9.7
1478
2122
1895
1702
1660
1560
1262
1547
541
1626
1513
445
1704
273
446
179
1777
862
1326
1417
1505
1628
6015
360
1146
237
1743
6000
1264
963
1301
1442
1435
1516
1461
1306
1178

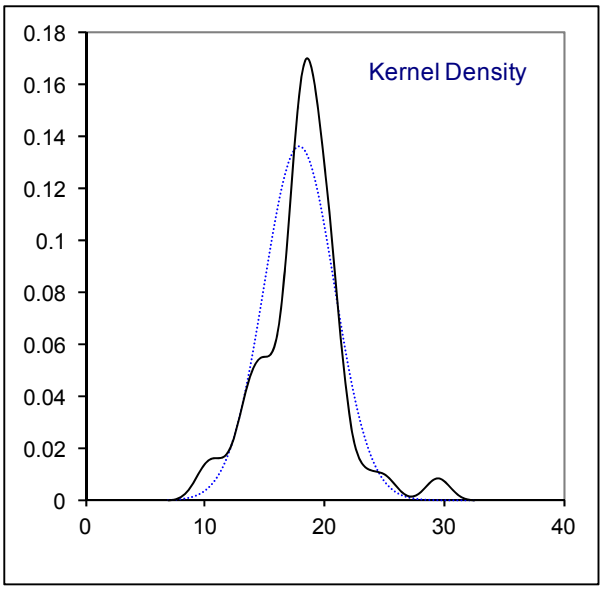

Kernel Density
10
9
8
7
6
5
4
3
2
1
0
9.2
9.4
9.6
9.8
10
10.2
10.4

Determination of Water on sample #15222; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D6304	11		-3.04	
237	D6304	20.22		1.03	
273	IEC60814	16.3		-0.70	
360	EN60814	15.0		-1.27	
445	IEC60814	14		-1.72	
446	IEC60814	18		0.05	
541	D6304	21		1.37	
614	EN60814	20.4		1.11	
862	D6304	15	C	-1.27	first reported: 7.17
963	D1533	21		1.37	
1146	D6304	10		-3.48	
1156	IEC60814	18.4		0.23	
1178	IEC60814	18.5		0.27	
1262	EN60814	17.2		-0.30	
1264	D1533	13.7		-1.85	
1301	IEC60814	21		1.37	
1303		-----		-----	
1304	INH-121	17.5		-0.17	
1306	D1533	19		0.49	
1326	D1533	19.6		0.76	
1374	IEC60814	17.9		0.01	
1417	D6304	18		0.05	
1435	IEC60814	20		0.93	
1440	EN60814	18.5		0.27	
1442	IEC60814	18.9		0.45	
1461		-----		-----	
1478	IEC60814	23.1		2.30	
1505	D1533	25.0		3.14	
1513	IEC60814	17.2		-0.30	
1516	IEC60814	17.7		-0.08	
1543		-----		-----	
1547	D1533	18.74		0.38	
1560	IEC60814	17.5		-0.17	
1626	IEC60814	15.3		-1.14	
1628	EN60814	20.2		1.02	
1660	EN60814	12.8		-2.24	
1687	IEC60814	18.518		0.28	
1702	IEC60814	18.15		0.12	
1704	IEC60814	16.9		-0.44	
1719	IEC60814	14.8		-1.36	
1720		-----		-----	
1743	IEC60814	20		0.93	
1747	IEC60814	19.205		0.58	
1760		-----		-----	
1777	IEC60814	18.1		0.09	
1890	IEC60814	19		0.49	
1895	D1533	29.46	R(0.05)	5.11	
1947	IEC60814	13.566		-1.91	
2122	EN60814	20.77		1.27	
6000		-----		-----	
6015	DIN51777	19.5		0.71	
6017	EN60814	18.7		0.36	

normality	OK
n	45
outliers	1
mean (n)	17.886
st.dev. (n)	2.9364
R(calc.)	8.222
R(EN60814:98)	6.344


35
30
25
20
15
10
5
1146 179 1660 1947 1264 445 1719 862 360 1626 273 1704 1262 1513 1304 1560 1516 1374 446 1417 1777 1702 1156 1178 1440 1687 6017 1547 1442 1306 1890 1747 6015 1326 1435 1743 1628 237 614 2122 963 1301 541 1478 1505 1895


Kernel Density
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0 10 20 30 40

APPENDIX 2

Number of participants per country

1 lab in ARGENTINA
4 labs in AUSTRALIA
2 labs in BELGIUM
4 labs in BULGARIA
2 labs in CHINA, People's Republic
1 lab in CROATIA
1 lab in ESTONIA
3 labs in FRANCE
4 labs in GERMANY
1 lab in GREECE
1 lab in INDIA
1 lab in ITALY
1 lab in MALAYSIA
1 lab in NETHERLANDS
1 lab in NEW ZEALAND
1 lab in NIGERIA
2 labs in PORTUGAL
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SLOVENIA
2 labs in SOUTH AFRICA
1 lab in SPAIN
3 labs in SUDAN
1 lab in SWEDEN
2 labs in TURKEY
4 labs in UNITED ARAB EMIRATES
4 labs in UNITED KINGDOM
1 lab in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect result
C(0.01)	= outlier in Cochran's outlier test
C(0.05)	= straggler in Cochran's outlier test
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 outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from calculations
n.a.	= not applicable
n.e	= not evaluated
W	= withdrawn on request participant
U	= reported in a deviating unit
E	= error in calculations
SDS	= Safety Data Sheet
fr.	= first reported

Literature:

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12 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
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