

Results of Proficiency Test
Transformer Oil (fresh)
November 2014

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 2014/2015. In this interlaboratory study, 53 laboratories from 29 different countries have participated. See appendix 2 for a list of number of participants per country order. In this report, the results of the 2014 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. In this proficiency test, the participants received a bottle of 1 litre of Transformer Oil (fresh), (labelled #14222). 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 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 S2 ZU-I) was obtained from a local supplier. The approximately 100 litre bulk material was homogenised in a pre-cleaned drum. After homogenisation, 99 subsamples were transferred to 1 litre amber glass bottles and labelled #14222. The homogeneity of the subsamples #14222 was checked by determination of Density in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density @ 20°C in kg/m ³
Sample #14222-1	877.92
Sample #14222-2	877.93
Sample #14222-3	877.93
Sample #14222-4	877.93
Sample #14222-5	877.93
Sample #14222-6	877.92
Sample #14222-7	877.93
Sample #14222-8	877.93

Table 1: homogeneity test results of subsamples #14222

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 @ 20°C in kg/m ³
r (sample #14222)	0.01
reference method	ISO3675:98
0.3xR _(reference)	0.36

Table 2: repeatabilities of subsamples #14222

The calculated repeatability of sample #14222 was less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples #14222 was assumed.

To each of the participating laboratories, 1*1 litre bottle (labelled #14222) was sent on November 5, 2014.

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 on sample #14222: Acid Number (Neutralization Number), Breakdown Voltage, Density @ 20°C, Di-electric loss @ 90°C (Di-electric Dissipation Factor and Specific Resistance), Flash Point, Interfacial Surface Tension, Kinematic Viscosity @ 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/. The detailed report form was also made available for download on the iis website www.iisnl.com.

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 received. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after deadline, a reminder fax 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:

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

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, no problems were encountered with the despatch of the samples. In total 7 participants reported the results after the final reporting date and 1 participant did not report at all. Not all participants were able to report results for all tests.

In total 52 participants reported 340 numerical results. Observed were 13 outlying results, which is 3.8% 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. In the iis PT reports, ASTM methods are referred to with a number (e.g. D2086) and an added designation for the year that the method was adopted or revised (e.g. D2086-08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086-08 (2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

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: 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 @ 20°C: This determination was problematic for a number of laboratories. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ISO3675:98.
- DD-Factor: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier 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: 7.51- 902.5 GΩm. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of EN60247:04.
- Flash Point: This determination was problematic. Four 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 not in agreement with the requirements of ISO2719:02.
- Interf. Surf. Tension: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility, after rejection of the statistical outliers is in agreement with the requirements of ASTM D971:12 and/or ISO6295:83. One should be aware that ISO6295 is obsolete since February 2005.
- Kinematic Viscosity: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility, after rejection of the of the statistical outliers is not at all 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	38	0.006	0.011	(0.002)
Breakdown Voltage	kV/2.5 mm	45	53.6	38.9	81.0
Density @ 20°C	kg/m ³	36	877.9	1.2	1.2
Di-electric Dissipation Factor		35	0.00175	0.00148	0.00272
Specific Resistance	GΩm	29	395.7	646.1	415.5
Flash Point	°C	30	148.9	14.4	10.6
Interfacial Surface Tension	mN/m	31	47.08	5.02	4.71
Kinematic Viscosity @ 40°C	mm ² /s	30	10.92	0.16	0.08
Water	mg/kg	48	16.79	7.64	6.15

table 3: Performance of the group on sample #14222

() = 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 2014 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2014	November 2013	October 2012	November 2011
Number of reporting labs	52	60	59	56
Number of results reported	340	491	427	378
Statistical outliers	13	32	30	27
Percentage outliers	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 2014	November 2013	October 2012	November 2011
Acid number (EN62021-1)	(--)	(--)	(--)	n.e.
Breakdown Voltage	++	--	--	--
Density @ 20°C	+/-	+/-	+	-
Di-electric Dissipation Factor	++	++	++	++
Specific Resistance	--	--	-	--
Flash Point	-	+/-	n.e.	n.e.
Interfacial Surface Tension	+/-	--	--	--
Kinematic Viscosity @ 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

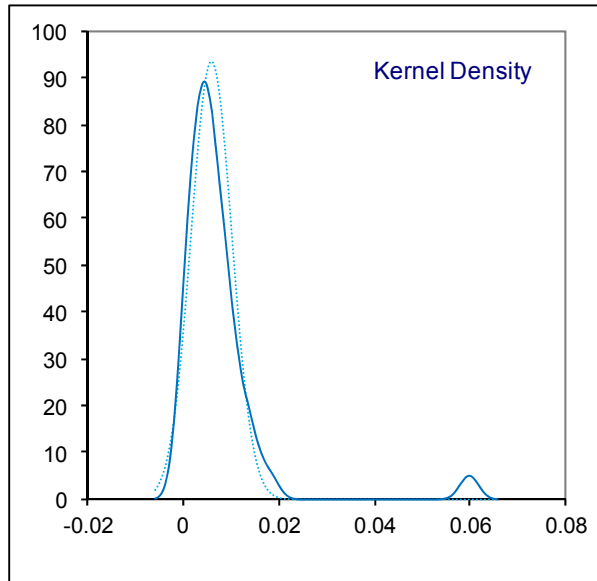
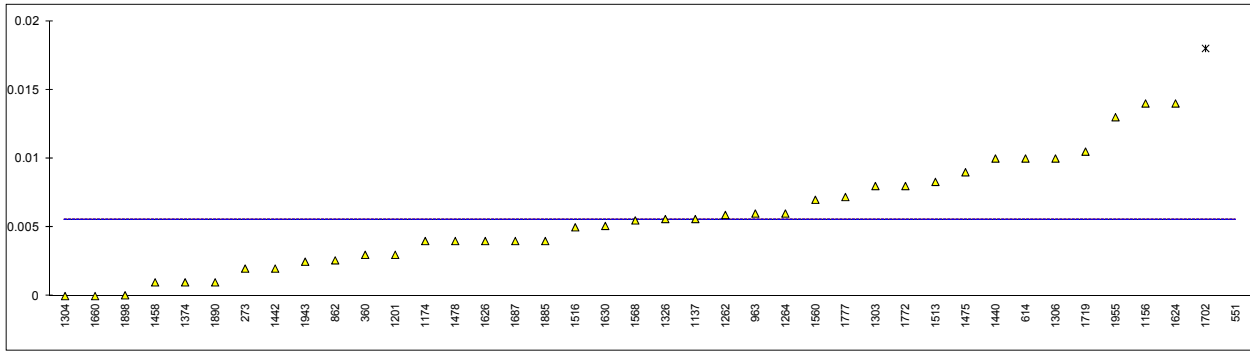
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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

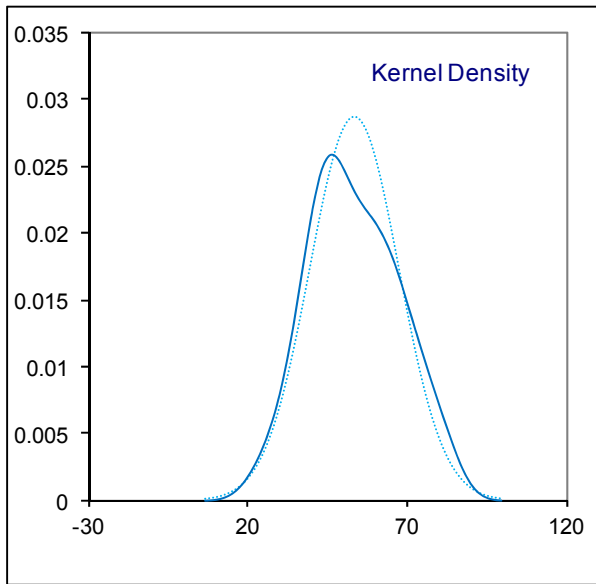
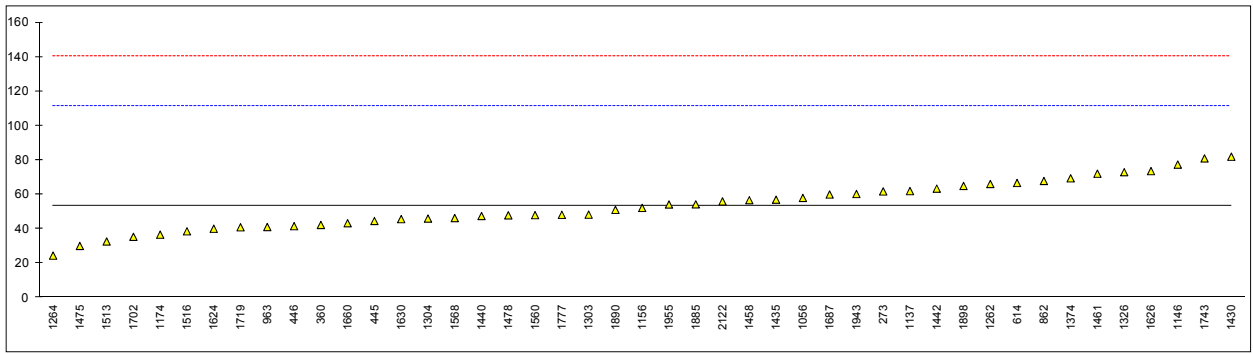
Determination of Acid Number on sample #14222; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks
173		----		----	
273	D974	0.0020		----	
360	EN62021-2	0.003		----	
445	EN62021-1	<0.01		----	
446		----		----	
541		----		----	
551	D664	0.06	R(0.01)	----	false positive test result?
614	EN62021-1	0.01		----	
862	IEC62021-1	0.0026	C	----	first reported: 0.0809
963	D974	0.006		----	
1056		----		----	
1137	D974	0.0056		----	
1146		----		----	
1156	EN62021-1	0.014		----	
1174	INH-1752	0.004		----	
1201	D976	0.003		----	
1262	EN62021-1	0.0059		----	
1264	D664	0.006		----	
1303	D974	0.008		----	
1304	INH-122	0.00		----	
1306	D974	0.01		----	
1326	EN62021-1	0.0056		----	
1374	IEC62021-1	0.001		----	
1417		----		----	
1430	EN62021-1	<0.01		----	
1435	IEC62021-1	<0.01		----	
1440	EN62021-1	0.01		----	
1442	IEC62021-2	0.002		----	
1458	D974	0.001	C	----	first reported:0.082
1461		----		----	
1475	D664	0.009		----	
1478	EN62021-1	0.0040		----	
1513	IEC62021-1	0.0083		----	
1516	D974	0.005		----	
1560	IEC62021-1	0.007		----	
1568	D974	0.0055		----	
1624	IEC62021-1	0.014	C	----	first reported:0.024
1626	D974	0.004		----	
1630	D974	0.0051		----	
1660	EN62021-1	0.000		----	
1687	D664	0.004		----	
1702	IEC62021	0.018	G(0.05)	----	false positive test result?
1719	D664	0.0105		----	
1743	IEC62021-1	<0.05	C	----	first reported:0.060
1772	EN62021-2	0.008		----	
1777	EN62021-1	0.0072		----	
1885	D974	0.004		----	
1890	ISO6619	0.001		----	
1898	EN62021-1	0.00006084	C	----	first reported:0.0684
1943	ISO6618	0.0025		----	
1955	D664	0.013		----	
1959		----		----	
2122	EN62021-1	<0.01		----	
	normality	OK			
	n	38			
	outliers	2			
	mean (n)	0.0056			
	st.dev. (n)	0.00383			
	R(calc.)	0.0107			
	R(EN62021-1:03)	(0.0016)			Quantification limit >0.014 g KOH/kg



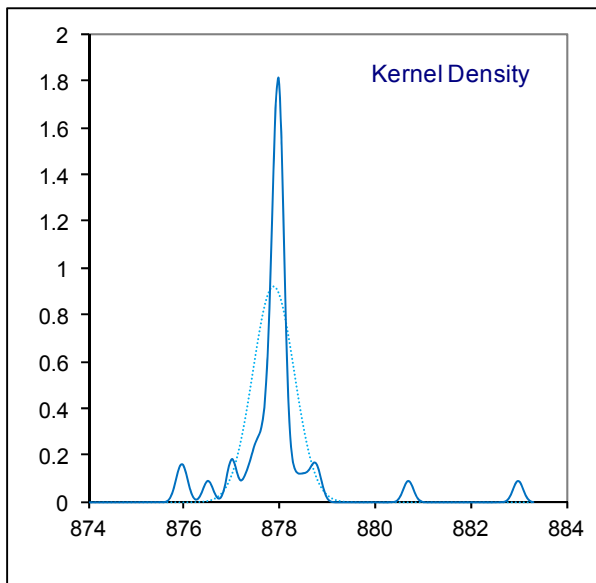
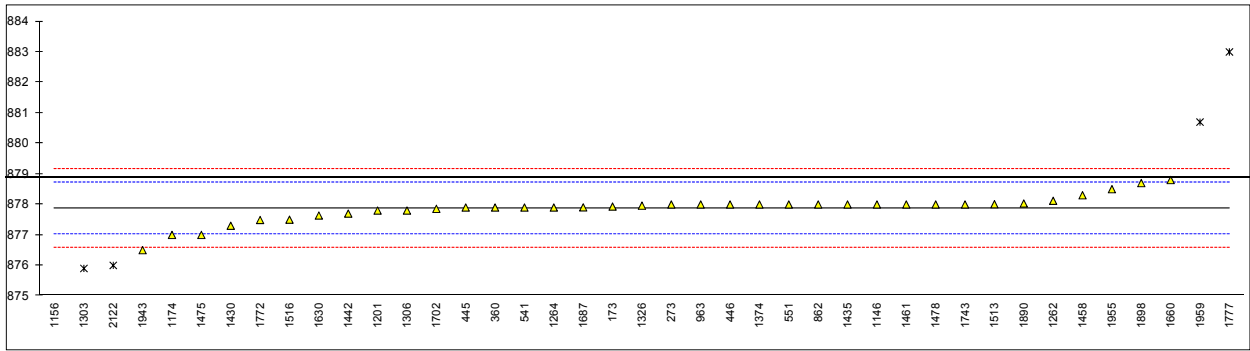
Determination of Breakdown Voltage on sample #14222, results in kV/2.5 mm

lab	method	value	mark	z(targ)	remarks
173		----		----	
273	IEC60156	61.8		0.28	
360	EN60156	42.3		-0.39	
445	EN60156	44.6		-0.31	
446	EN60156	41.6		-0.41	
541		----		----	
551		----		----	
614	IEC60156	66.7		0.45	
862	IEC60156	67.87		0.49	
963	D877	41.1		-0.43	
1056	IP295	58		0.15	
1137	IEC60156	62.0		0.29	
1146	IEC60156	77.4		0.82	
1156	EN60156	52.2		-0.05	
1174	EN60156	36.66		-0.58	
1201		----		----	
1262	EN60156	66.1		0.43	
1264	EN60156	24.5		-1.01	
1303	IEC60156	48.3		-0.18	
1304	INH-124	46		-0.26	
1306		----		----	
1326	IEC60156	73.0		0.67	
1374	IEC60156	69.4		0.55	
1417		----		----	
1430	EN60156	82		0.98	
1435	IEC60156	57		0.12	
1440	EN60156	47.5		-0.21	
1442	IEC60156	63.43		0.34	
1458	IEC60156	56.7		0.11	
1461	EN60156	72.1		0.64	
1475	IP295	30.1		-0.81	
1478	EN60156	47.9		-0.20	
1513	IEC60156	32.7		-0.72	
1516	IEC60156	38.6		-0.52	
1560	IEC60156	48		-0.19	
1568	D877	46.32		-0.25	
1624	IEC60156	40.1		-0.47	
1626	IEC60156	73.6		0.69	
1630	IS6792	45.7		-0.27	
1660	EN60156	43.3		-0.36	
1687	EN60156	60		0.22	
1702	IEC60156	35.4		-0.63	
1719	IEC60156	40.97		-0.44	
1743	IEC60156	81		0.95	
1772		----		----	
1777	IEC60156	48.2		-0.19	
1885	IEC60156	54.3		0.02	
1890	IEC60156	51.1		-0.09	
1898	EN60156	65		0.39	
1943	EN60156	60.3		0.23	
1955	IEC156	54.2		0.02	
1959		----		----	
2122	EN60156	56		0.08	
	normality	OK			
	n	45			
	outliers	0			
	mean (n)	53.58			
	st.dev. (n)	13.896			
	R(calc.)	38.91			
	R(EN60156:95)	81.01			



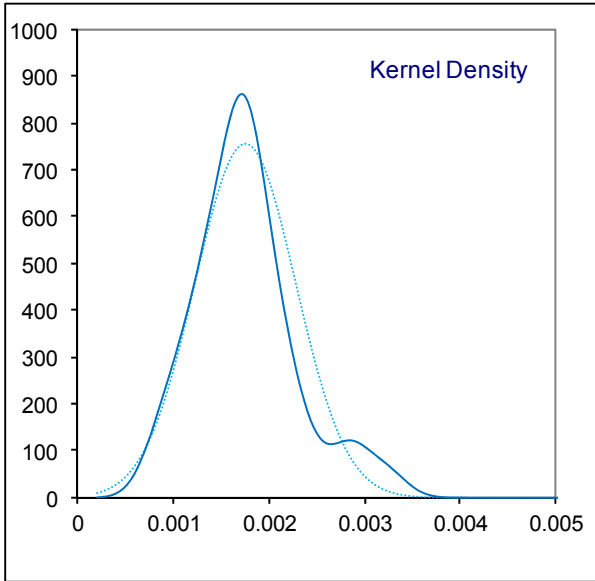
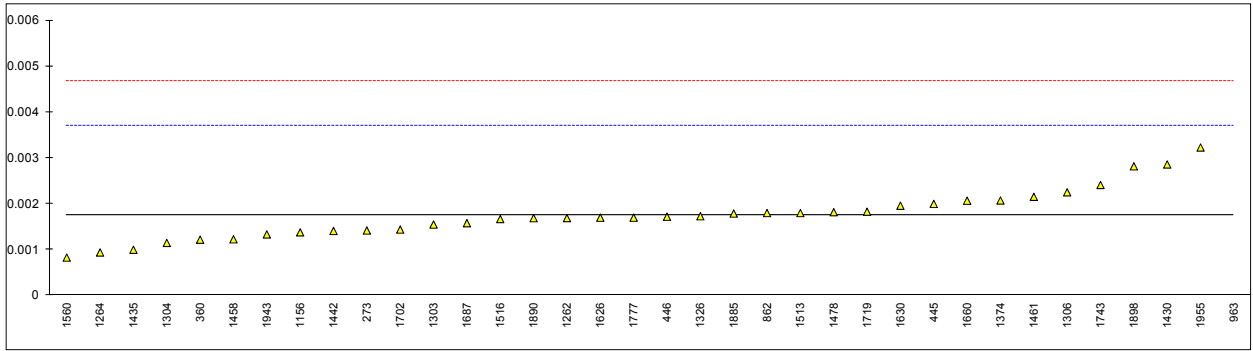
Determination of Density @ 20°C on sample #14222; results in kg/m³

lab	method	value	mark	z(targ)	remarks
173	D4052	877.93		0.13	
273	D4052	878.0		0.30	
360	ISO12185	877.9		0.06	
445	D4052	877.9		0.06	
446	D4052	878.0		0.30	
541	ISO12185	877.9		0.06	
551	D4052	878.0		0.30	
614		----		----	
862	D4052	878.0		0.30	
963	D4052	878.0		0.30	
1056		----		----	
1137		----		----	
1146	ISO12185	878.00		0.30	
1156	in house	869	R(0.01)	-20.70	
1174	ISO3675	877.0		-2.04	
1201	ISO3675	877.8		-0.17	
1262	ISO3675	878.12		0.58	
1264	D4052	877.9		0.06	
1303	D4052	875.9	R(0.05)	-4.60	
1304		----		----	
1306	D4052	877.8		-0.17	
1326	D4052	877.96		0.20	
1374	D7777	878		0.30	
1417		----		----	
1430	ISO3675	877.3		-1.34	
1435	D4052	878		0.30	
1440		----		----	
1442	D7042	877.7		-0.40	
1458	D4052	878.3		1.00	
1461	ISO3675	878.0	C	0.30	first reported:0.8780
1475	D1298	877.0		-2.04	
1478	ISO12185	878.0		0.30	
1513	ISO12185	878.006		0.31	
1516	ISO3675	877.5		-0.87	
1560		----		----	
1568		----		----	
1624		----		----	
1626		----		----	
1630	IS1448	877.636		-0.55	
1660	ISO3675	878.8	C	2.16	first reported:0.8788
1687	ISO12185	877.905		0.07	
1702	ISO12185	877.856		-0.04	
1719		----		----	
1743	in house	878		0.30	
1772	ISO3675	877.49	C	-0.89	first reported:0.87749
1777	D4052	883	R(0.01)	11.96	
1885		----		----	
1890	ISO12185	878.03		0.37	
1898	ISO12185	878.7	C	1.93	first reported:0.8787
1943	ISO3675	876.5		-3.20	
1955	D7042	878.5		1.46	
1959	GB/T1884	880.7	C,R(0.01)	6.60	first reported:879.8
2122	INH-12185	876.0	R(0.01)	-4.37	
	normality	not OK			
	n	36			
	outliers	5			
	mean (n)	877.873			
	st.dev. (n)	0.4321			
	R(calc.)	1.210			
	R(ISO3675:98)	1.200			



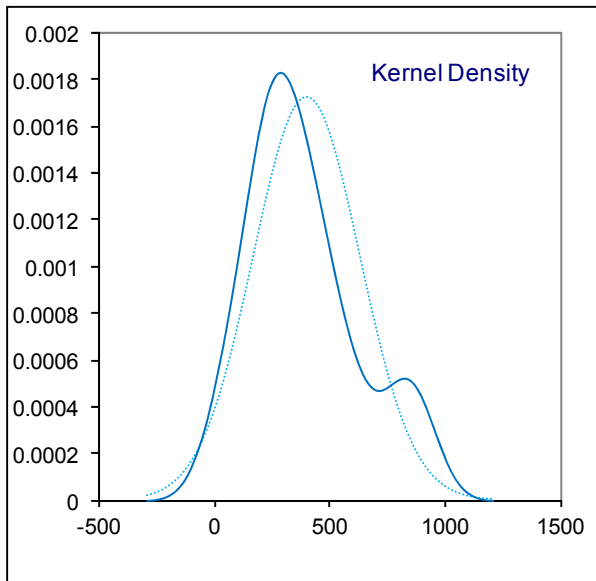
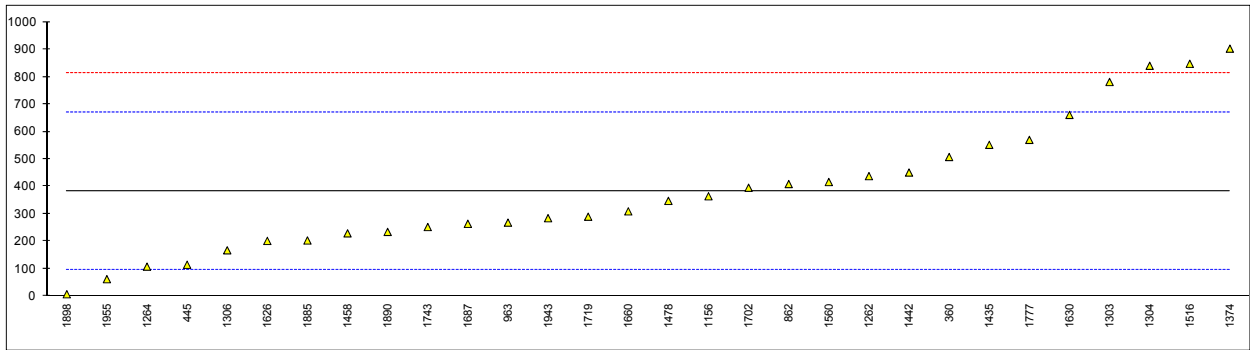
Determination of Di-electric Dissipation Factor at 90°C on sample #14222

lab	method	value	mark	z(targ)	remarks
173		----		----	
273	IEC60247	0.00142		-0.34	
360	EN60247	0.00122		-0.55	
445	EN60247	0.0020		0.26	
446	EN60247	0.00172		-0.03	
541		----		----	
551		----		----	
614		----		----	
862	IEC60247	0.0018		0.05	
963	IEC60247	0.011	R(0.01)	9.51	
1056		----		----	
1137		----		----	
1146		----		----	
1156	EN60247	0.00138		-0.38	
1174		----		----	
1201		----		----	
1262	IEC60247	0.00169		-0.06	
1264	EN60247	0.000941		-0.83	
1303	IEC60247	0.00155		-0.21	
1304	INH-125	0.001150		-0.62	
1306	IEC60247	0.002252		0.52	
1326	IEC60247	0.0017305		-0.02	
1374	IEC60247	0.002074		0.33	
1417		----		----	
1430	EN60247	0.00286		1.14	
1435	IEC60247	0.001		-0.77	
1440		----		----	
1442	IEC60247	0.00141		-0.35	
1458	IEC60247	0.001228		-0.54	
1461	EN60247	0.002155		0.42	
1475		----		----	
1478	EN60247	0.001820		0.07	
1513	IEC60247	0.00180		0.05	
1516	IEC60247	0.00167		-0.08	
1560	IEC60247	0.00083		-0.95	
1568		----		----	
1624		----		----	
1626	IEC60247	0.0017		-0.05	
1630	IS6262	0.00196		0.22	
1660	EN60247	0.00207		0.33	
1687	EN60247	0.001580		-0.18	
1702	IEC60247	0.001441		-0.32	
1719	IEC60247	0.00183		0.08	
1743	IEC60247	0.002413		0.68	
1772		----		----	
1777	IEC60247	0.0017		-0.05	
1885	IEC60247	0.00179	C	0.04	first reported:0.179
1890	IEC60247	0.001689		-0.06	
1898	IEC60247	0.00282	C	1.10	first reported:0.282
1943	EN60247	0.001335		-0.43	
1955	IEC60247	0.00323		1.52	
1959		----		----	
2122		----		----	
	normality	suspect			
	n	35			
	outliers	1			
	mean (n)	0.001750			
	st.dev. (n)	0.0005272			
	R(calc.)	0.001476			
	R(EN60247:04)	0.002723			



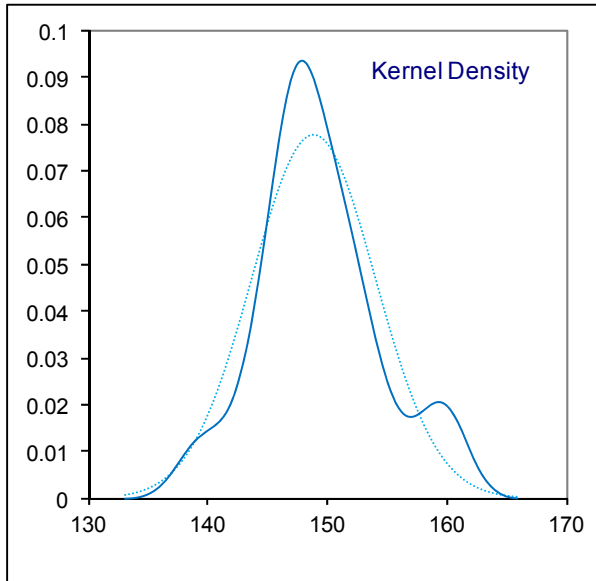
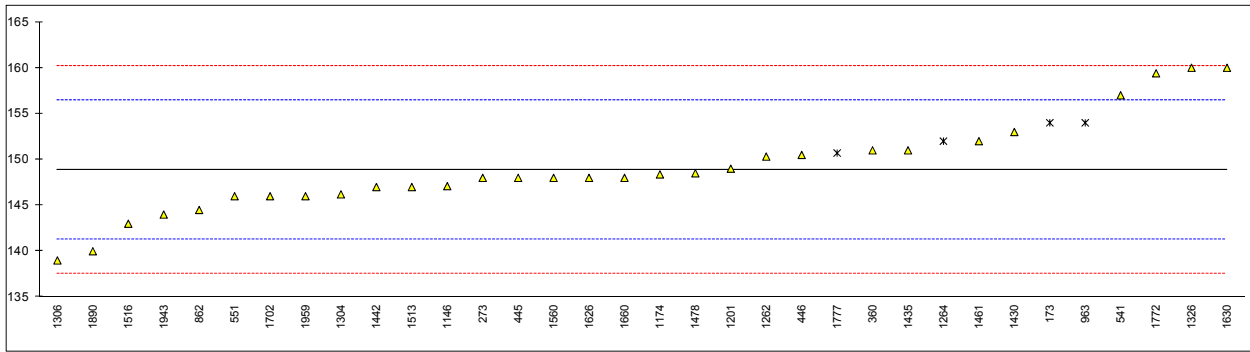
Determination of Specific Resistance at 90°C on sample #14222; results in GΩm

lab	method	value	mark	z(targ)	remarks
173		----		----	
273		----		----	
360	EN60247	507.64		0.87	
445	EN60247	114.971		-1.87	
446		----		----	
541		----		----	
551		----		----	
614		----		----	
862	IEC60247	409.00		0.18	
963	D1169	268.6		-0.80	
1056		----		----	
1137		----		----	
1146		----		----	
1156	EN60247	364.6		-0.13	
1174		----		----	
1201		----		----	
1262	IEC60247	437.9		0.38	
1264	EN60247	108		-1.91	
1303	IEC60247	781		2.77	
1304	INH-125	840.06		3.19	
1306	IEC60247	167.64		-1.50	
1326		----		----	
1374	IEC60247	902.5		3.62	
1417		----		----	
1430		----		----	
1435	IEC60247	552.0		1.18	
1440		----		----	
1442	IEC60247	450.97		0.48	
1458	IEC60247	229.34		-1.07	
1461		----		----	
1475		----		----	
1478	EN60247	347.65		-0.24	
1513		----		----	
1516	IEC60247	847.2		3.24	
1560	IEC60247	416.5		0.24	
1568		----		----	
1624		----		----	
1626	IEC60247	201.8		-1.26	
1630	IS6103	660.9		1.94	
1660	EN60247	309.6		-0.51	
1687	EN60247	264.24		-0.83	
1702	IEC60247	395.57		0.09	
1719	IEC60247	290	C	-0.65	probably unit error, reported: 290 E9 GΩm
1743	IEC60247	252.34		-0.91	
1772		----		----	
1777	IEC60247	570		1.30	
1885	IEC60247	203.4		-1.25	
1890	IEC60247	234.68		-1.03	
1898	IEC60247	7.51	C	-2.61	first reported: 0.0751 GΩm
1943	EN60247	284.67		-0.68	
1955	IEC60247	62.500		-2.23	
1959		----		----	
2122		----		----	
	normality	OK			
	n	30			
	outliers	0			
	mean (n)	382.76			
	st.dev. (n)	237.543			
	R(calc.)	665.12			
	R(EN60247:04)	401.90			



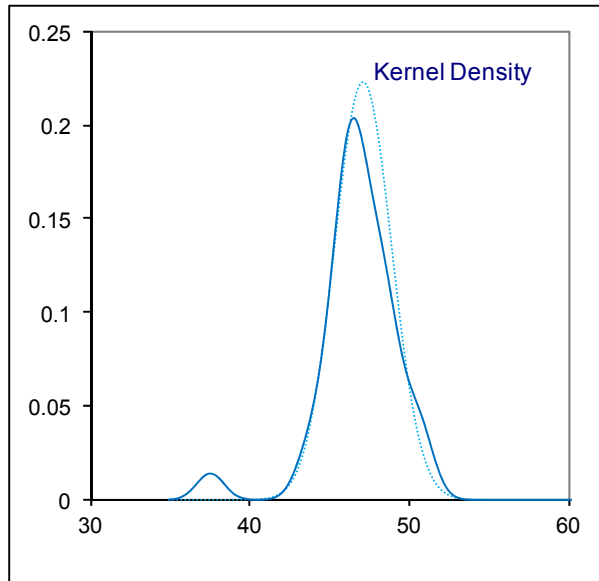
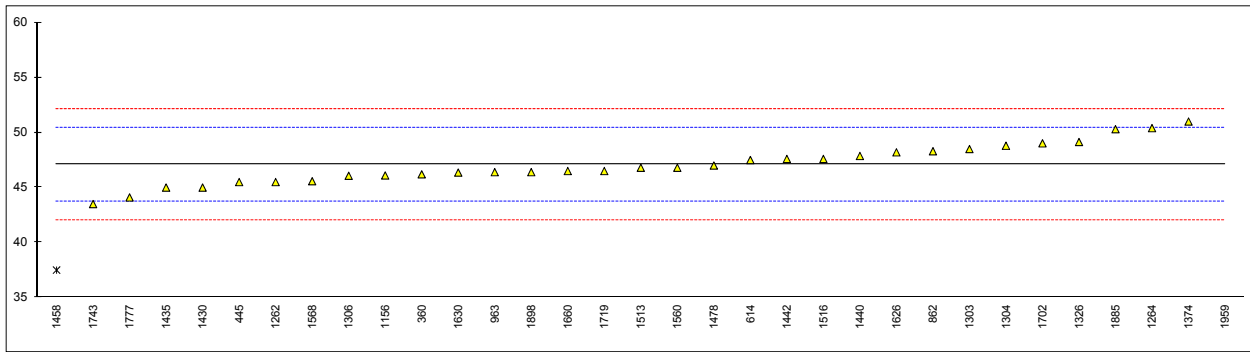
Determination of Flash Point PMcc on sample #14222; results in °C

lab	method	value	mark	z(targ)	remarks
173	D92	154	ex	1.36	result excluded, method is not equivalent to Flash Point PMcc
273	D93	148.0		-0.23	
360	ISO2719	151.0		0.57	
445	D93	148.0		-0.23	
446	D93	150.5		0.43	
541	ISO2719	157.0		2.16	
551	D93	146.0		-0.76	
614		----		----	
862	D93	144.5		-1.16	
963	D92	154	ex	1.36	result excluded, method is not equivalent to Flash Point PMcc
1056		----		----	
1137		----		----	
1146	in house	147.1		-0.47	
1156		----		----	
1174	ISO2719	148.375		-0.13	
1201	ISO2719	149.0		0.04	
1262	ISO2719	150.32		0.39	
1264	D92	152	ex	0.83	result excluded, method is not equivalent to Flash Point PMcc
1303		----		----	
1304	IP34	146.2		-0.71	
1306	D93	139.0		-2.61	
1326	D93	160.0		2.95	
1374		----		----	
1417		----		----	
1430	ISO2719	153		1.10	
1435	D93	151		0.57	
1440		----		----	
1442	ISO2719	147		-0.49	
1458		----		----	
1461	ISO2719	152		0.83	
1475		----		----	
1478	ISO2719	148.5		-0.10	
1513	ISO2719	147.0		-0.49	
1516	ISO2719	143		-1.55	
1560	ISO2719	148		-0.23	
1568		----		----	
1624		----		----	
1626	D93	148.0		-0.23	
1630	D93	160		2.95	
1660	ISO2719	148.0		-0.23	
1687		----		----	
1702	ISO2719	146.0		-0.76	
1719		----		----	
1743		----		----	
1772	ISO2719	159.4		2.79	
1777	D92	150.7	ex	0.49	result excluded, method is not equivalent to Flashpoint PMcc
1885		----		----	
1890	ISO2719	140		-2.35	
1898		----		----	
1943	ISO2719	144		-1.29	
1955		----		----	
1959	GB/T261	146.0		-0.76	
2122		----		----	
	normality	OK			
	n	30			
	outliers	0 (+4 excl)			
	mean (n)	148.86			
	st.dev. (n)	5.136			
	R(calc.)	14.38			
	R(ISO2719:02-A)	10.57			R(ISO2719:02-A)=R(D93:02-A)=R(IP34:03-A)



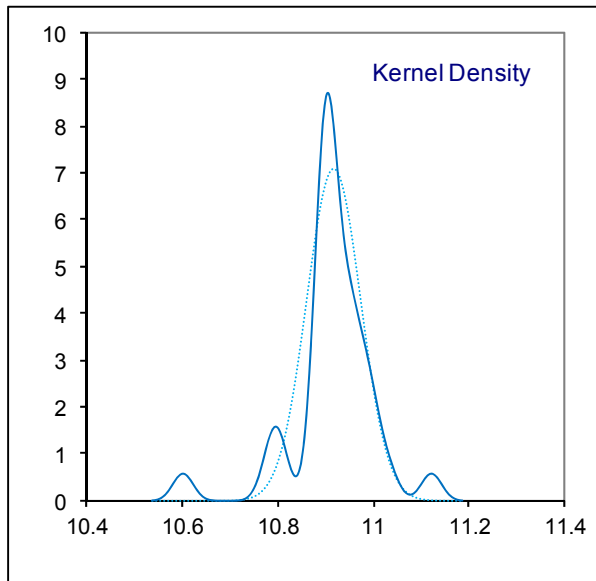
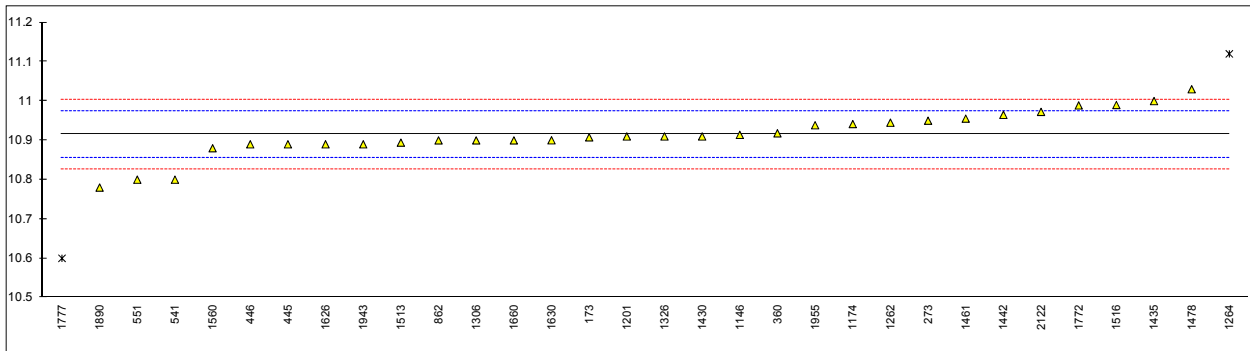
Determination of Interfacial Surface Tension on sample #14222; results in mN/m

lab	method	value	mark	z(targ)	remarks
173		----		----	
273		----		----	
360	D971	46.2		-0.52	
445	D971	45.5		-0.94	
446		----		----	
541		----		----	
551		----		----	
614	ISO6295	47.5		0.25	
862	ISO6295	48.3		0.72	
963	D971	46.4		-0.41	
1056		----		----	
1137		----		----	
1146		----		----	
1156	EN14210	46.1		-0.58	
1174		----		----	
1201		----		----	
1262	D971	45.5		-0.94	
1264	D971	50.40		1.97	
1303	D971	48.5		0.84	
1304	INH-123	48.8		1.02	
1306	D971	46.07		-0.60	
1326	ISO6295	49.14		1.22	
1374	D2285	51		2.33	
1417		----		----	
1430	ISO6295	45		-1.24	
1435	D971	45.0		-1.24	
1440	ISO6295	47.87		0.47	
1442	EN14210	47.6		0.31	
1458	D971	37.5	R(0.01)	-5.70	
1461		----		----	
1475		----		----	
1478	D971	47		-0.05	
1513	D971	46.8		-0.17	
1516	D971	47.6		0.31	
1560	D971	46.8		-0.17	
1568	D2285	45.5781		-0.89	
1624		----		----	
1626	ISO6295	48.2		0.66	
1630	D971	46.36	C	-0.43	probably unit error, reported: 0.04636 mN/m
1660	ISO6295	46.5		-0.35	
1687		----		----	
1702	D971	49.020		1.15	
1719	D2285	46.5		-0.35	
1743	ISO6295	43.5		-2.13	
1772		----		----	
1777	D971	44.1		-1.77	
1885	D971	50.3		1.91	
1890		----		----	
1898	D971	46.4		-0.41	
1943		----		----	
1955		----		----	
1959	GB/T6541	87.2	R(0.01)	23.86	
2122		----		----	
	normality	OK			
	n	31			
	outliers	2			
	mean (n)	47.082			
	st.dev. (n)	1.7931			
	R(calc.)	5.021			
	R(D971:12)	4.708			Compare R(ISO6295:83) = 4.70



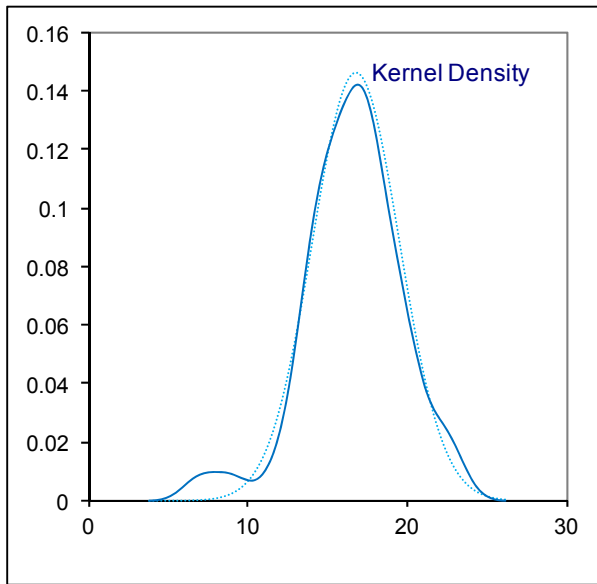
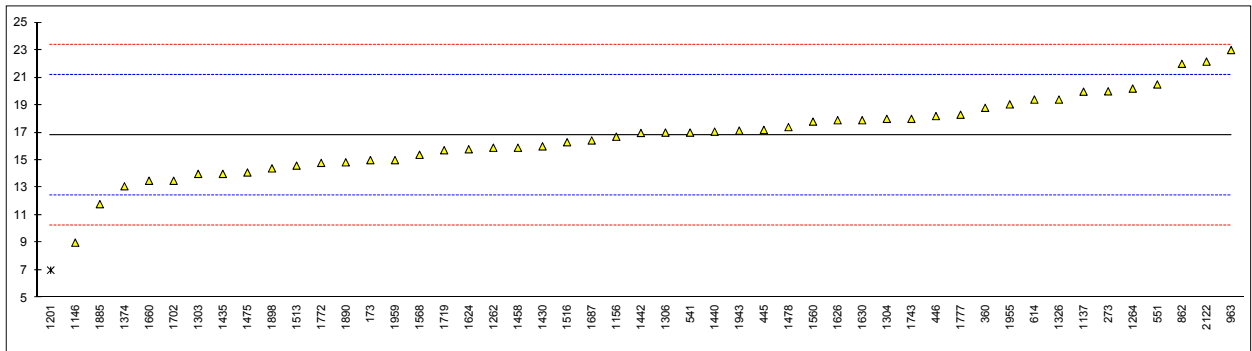
Determination of Kinematic Viscosity @ 40°C on sample #14222; results in mm²/s

lab	method	value	mark	z(targ)	remarks
173	D445	10.9077		-0.26	
273	D445	10.95		1.17	
360	ISO3104	10.918		0.09	
445	D445	10.89		-0.86	
446	D445	10.89		-0.86	
541	ISO3104	10.80		-3.89	
551	D7279	10.80		-3.89	
614		----		----	
862	ISO3104	10.90		-0.52	
963		----		----	
1056		----		----	
1137		----		----	
1146	ISO3104	10.914		-0.05	
1156		----		----	
1174	ISO3104	10.94151		0.88	
1201	ISO3104	10.91		-0.18	
1262	ISO3104	10.9450		1.00	
1264	D7042	11.120	R(0.05)	6.91	
1303		----		----	
1304		----		----	
1306	D445	10.90		-0.52	
1326	D445	10.91		-0.18	
1374		----		----	
1417		----		----	
1430	ISO3104	10.91		-0.18	
1435	D7042	11.0		2.86	
1440		----		----	
1442	D7042	10.965		1.68	
1458		----		----	
1461	ISO3104	10.9553		1.35	
1475		----		----	
1478	ISO3104	11.03		3.87	
1513	ISO3104	10.8943		-0.71	
1516	ISO3104	10.99		2.52	
1560	ISO3104	10.88		-1.19	
1568		----		----	
1624		----		----	
1626	D445	10.89		-0.86	
1630	D445	10.90025		-0.51	
1660	ISO3104	10.9		-0.52	
1687		----		----	
1702		----		----	
1719		----		----	
1743		----		----	
1772	ISO3104	10.98893		2.48	
1777	D445	10.6	C,R(0.01)	-10.64	first reported:61
1885		----		----	
1890	ISO3104	10.78		-4.57	
1898		----		----	
1943	ISO3104	10.89		-0.86	
1955	D7042	10.9385		0.78	
1959		----		----	
2122	INH-445	10.9725		1.93	
	normality	OK			
	n	30			
	outliers	2			
	mean (n)	10.915			
	st.dev. (n)	0.0564			
	R(calc.)	0.158			
	R(ISO3104:96)	0.083			



Determination of Water on sample #14222; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	D6304	15		-0.82	
273	EN60814	20		1.46	
360	EN60814	18.8		0.91	
445	EN60814	17.2		0.18	
446	EN60814	18.2		0.64	
541	D6304	17		0.09	
551	EN60814	20.5		1.69	
614	EN60814	19.4		1.19	
862	D6304	22		2.37	
963	D1533	23		2.83	
1056		----		----	
1137	ISO10337	19.975		1.45	
1146	D6304	9		-3.55	
1156	EN60814	16.7		-0.04	
1174		----		----	
1201	EN60814	7	R(0.05)	-4.46	
1262	EN60814	15.9		-0.41	
1264	D1533	20.2		1.55	
1303	EN60814	14.0		-1.27	
1304	INH-121	18.0		0.55	
1306	D1533	17		0.09	
1326	D1533	19.4		1.19	
1374	IEC60814	13.1		-1.68	
1417		----		----	
1430	EN60814	16		-0.36	
1435	IEC60814	14		-1.27	
1440	EN60814	17.07		0.13	
1442	IEC60814	16.975		0.08	
1458	IEC60814	15.9		-0.41	
1461		----		----	
1475	D6304	14.1		-1.23	
1478	EN60814	17.4		0.28	
1513	IEC60814	14.6		-1.00	
1516	IEC60814	16.3		-0.23	
1560	IEC60814	17.8		0.46	
1568	D1533	15.3895		-0.64	
1624	IEC60814	15.79		-0.46	
1626	IEC60814	17.9		0.50	
1630	IS13567	17.90		0.50	
1660	EN60814	13.5		-1.50	
1687	EN60814	16.429		-0.17	
1702	IEC60814	13.5		-1.50	
1719	IEC60814	15.72		-0.49	
1743	IEC60814	18		0.55	
1772	EN60814	14.8		-0.91	
1777	IEC60814	18.3		0.69	
1885	D1533	11.8		-2.27	
1890	IEC60814	14.84		-0.89	
1898	EN60814	14.4		-1.09	
1943	EN60814	17.14		0.16	
1955	D6304	19.05		1.03	
1959	GB/T7600	15		-0.82	
2122	EN60814	22.16		2.44	
	normality	OK			
	n	48			
	outliers	1			
	mean (n)	16.795			
	st.dev. (n)	2.7278			
	R(calc.)	7.638			
	R(EN60814:98)	6.147			



APPENDIX 2

Number of participants per country

1 lab in ARGENTINA
6 labs in AUSTRALIA
2 labs in BELGIUM
1 lab in BRAZIL
5 labs in BULGARIA
3 labs in CHINA, People's Republic
2 labs in FRANCE
2 labs in GERMANY
1 lab in INDIA
1 lab in ISRAEL
1 lab in ITALY
1 lab in LATVIA
1 lab in MALAYSIA
1 lab in MEXICO
3 labs in NETHERLANDS
1 lab in NEW ZEALAND
1 lab in NORWAY
2 labs in PORTUGAL
1 lab in SAUDI ARABIA
1 lab in SLOVENIA
2 labs in SOUTH AFRICA
1 lab in SOUTH KOREA
2 labs in SPAIN
1 lab in SWEDEN
2 labs in TURKEY
2 labs in UNITED ARAB EMIRATES
4 labs in UNITED KINGDOM
1 lab in UNITED STATES OF AMERICA
1 lab in VIETNAM

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:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
- 2 prNEN 12766-2:2001
- 3 ASTM E178-02
- 4 ASTM E1301-03
- 5 ISO 5725-86
- 6 ISO 5725, parts 1-6, 1994
- 7 ISO13528-05
- 8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 10 IP 367/96
- 11 DIN 38402 T41/42
- 12 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
- 13 J.N. Miller, Analyst, 118, 455, (1993)
- 14 Analytical Methods Committee Technical Brief, No4 January 2001
- 15 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see <http://www.rsc.org/suppdata/an/b2/b205600n/>)
- 16 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)