

**Results of Proficiency Test
Transformer Oil
November 2009**

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: Ing. R.J. Starink
Corrector: Dr. R.G. Visser
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1 INTRODUCTION

Since 2001, the Institute for Interlaboratory Studies organizes a proficiency test for Transformer Oil every year. During the annual proficiency testing program 2009/2010, it was decided to continue the round robin for the analysis of Transformer Oil. In this international Interlaboratory study, 38 laboratories from 19 different countries have participated. See appendix 2 for a list of participants in alphabetical country order. In this report, the results of the Transformer Oil proficiency test are presented and discussed.

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, two different samples were used. The participants received 1 litre of an unused Transformer Oil (labelled #0988) and 0.5 litre of used Transformer Oil (labelled #0989). 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 accordance with ISO guide 43 and ILAC-G13:2007, (R007), since January 2000, by the Dutch Accreditation Council: RvA (Raad voor Accreditatie). This ensures 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 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' of November 2008 (iis-protocol, version 3.1).

2.3 CONFIDENTIALITY STATEMENT

All data present 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 for 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

In this proficiency test two different samples were used.

The necessary bulk material for the unused oil sample (DIALA B dried) was obtained from a local supplier. The approximately 60 litre bulk material was homogenised. After homogenisation, 53 subsamples were transferred to 1 litre amber glass bottles and labelled #0988. The homogeneity of the subsamples #0988 was checked by determination Density in accordance with ASTM D 4052:02e1 and Water in accordance with ASTM D6304:07 on 6 stratified random selected samples.

	Water in mg/kg	Density @ 20°C in kg/L
Sample #0988-1	13	0.88227
Sample #0988-2	12	0.88227
Sample #0988-3	13	0.88227
Sample #0988-4	11	0.88227
Sample #0988-5	10	0.88227
Sample #0988-6	12	0.88228

Table 1: homogeneity test of subsamples #0988

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

	Water in mg/kg	Density @ 20°C in kg/L
r (Observed)	3	0.01
reference method	ASTM D1533:05	ASTM D 4052:02e1
0.3 * R (ref. method)	4	0.15

Table 2: repeatabilities of subsamples #0988

The necessary bulk material for the second sample (used transformer oil) was obtained from a participating laboratory. After homogenisation, 48 subsamples were transferred to 0.5 litre amber glass bottles and labelled #0989. The homogeneity of the subsamples #0989 was checked by determination of the Density in accordance with ASTM D 4052:02e1 and Water in accordance with ASTM D6304:07 on 6 stratified random selected samples:

	Water in mg/kg	Density @ 20°C in kg/L
Sample #0989-1	95	0.87817
Sample #0989-2	85	0.87818
Sample #0989-3	88	0.87817
Sample #0989-4	82	0.87817
Sample #0989-5	77	0.87818
Sample #0989-6	107	0.87817

Table 3: homogeneities test of subsamples #0989

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

	Water in mg/kg	Density @ 15 °C in kg/L
r (Observed)	25	0.01
reference method	ASTM D6304:07	ASTM D 4052:02e1
0.3 * R (ref. method)	72	0.15

table 4: repeatability of subsamples #0989

Each calculated repeatability was equal or less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 1* 1 litre (labelled #0988) and 1* 0.5 litre (labelled #0989) were sent on October 21, 2009.

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 #0988: Density @ 20 °C, Di-electric loss 90 °C (Di-electric Dissipation Factor and Specific Resistance), Breakdown Voltage, Interfacial Surface Tension, Colour, Water and Acid Number (Neutralization Number).

On sample #0989 was asked to determine: Di-electric loss 90 °C (Di-electric Dissipation Factor and Specific Resistance), Interfacial Surface Tension, Water and Acid Number (Neutralization Number), Cadmium and Furanic Compounds (2-acetyl furan, 2-furfural, 2-furfuryl alcohol, 5-hydroxymethyl-2-furfural and 5-methyl-2-furfural). To get comparable results, a detailed report form on which the units and the preferred test methods were printed, was sent together with each set of samples. Also a letter of instructions and a SDS were added to the package.

3 RESULTS

During four weeks after sample despatch the results of the individual laboratories were gathered. The original data are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported results at that moment. 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 results. Additional or corrected results are 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' of November 2008 (iis-protocol, version 3.1).

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. 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 and Grubbs 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. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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; nr.14 and 15).

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. The target standard deviation was calculated from the literature reproducibility by division with 2.8. 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 is as follows:

$ z < 1$	good
$1 < z < 2$	satisfactory
$2 < z < 3$	questionable
$3 < z $	unsatisfactory

4 EVALUATION

No problems were encountered during the dispatch of the samples. All laboratories did receive the samples in time. Four participants reported results after the final reporting date and two participants did not report any results at all. Not all participants were able to report results for all tests. In total 36 participants reported 348 numerical results. Observed were 21 outlying results, which is 6.0% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

Not all original data sets proved to have a normal distribution. Not normal distribution were found for the following determinations on sample #0988: Colour, Density and Di-electric Dissipation Factor and Water and for sample #0989 only for Di-electric Dissipation Factor. In these cases the statistical evaluations should be used with due care. In this section, the results are discussed per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

Acid Number: This determination is not problematic. In total three statistical outliers were observed and one zero result was excluded from statistical calculation. The calculated reproducibilities of both samples #0988 and #0989 are, after rejection of the statistical outliers, in full agreement with the requirements of ASTM D974:08.

- Breakdown Voltage: This determination, only performed on sample #0988 (unused oil), is problematic. One statistical outlier was noticed. The calculated reproducibility is, after rejection the statistical outlier, not at all in agreement with the requirements of IEC60156:95.
- Colour: No analytical problems are observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1500:08.
- Density @ 20°C: This determination is problematic for three individual laboratories. Three statistical outliers were observed. However, the calculated reproducibility is, after rejection of the statistical outliers, in full agreement with the requirements of ISO3675:98.
- DD-Factor: This determination is not problematic. In total four statistical outliers were observed. The calculated reproducibilities of both samples #0988 and #0989 are, after rejection of the statistical outliers, in good agreement with the requirements of IEC60247:78.
- Spec. Resistance: This determination is problematic for sample #0988 only (data bimodally divided). In total two statistical outliers were observed. The calculated reproducibility of sample #0988 (high value) is, after rejection of the statistical outlier, not at all in agreement with the requirements of IEC60247. The calculated reproducibility of samples #0989 (low value) is, after rejection of the statistical outlier, in good agreement with the requirements of IEC60247:78.
- Interf. Surf. Tension: This determination is problematic for both samples. In total three statistical outliers were observed. The calculated reproducibilities of both samples #0988 and #0989 are, after rejection of the statistical outliers, not at all in agreement with the requirements of ASTM D971:04 (ISO6295:83 is obsolete since Feb-2005).
- Water: For sample #0988, three statistical outliers were observed. The calculated reproducibility is, after rejection of statistical outliers, in good agreement with the requirements of ASTM D1533:05, but not with the requirements of IEC60814:97.
For sample #0989, only one statistical outlier was observed. The calculated reproducibility is, after rejection of statistical outlier, in good agreement with the requirements of ASTM D6305:07.
As ASTM D1533 and IEC60814 are not applicable for used insulating oils, test method ASTM D6305 was chosen for evaluation for sample #0988.

Cadmium: Regretfully, no numerical results were reported, therefore no significant conclusions were drawn.

Furanic Compounds: For sample #0989 only a positive result was found for 2-Furfural.. Only one statistical outlier was observed. The calculated reproducibility is, after rejection of the statistical outlier, not at all in agreement with the estimated requirements calculated using the Horwitz equation. It is remarkable that the precision data of IEC61198:93 are much smaller than precision data calculated using the Horwitz equation. This very strict precision seems to be impossible to meet.

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 standards) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acid Number (#0988)	g KOH/kg	24	0.009	0.017	0.040
Acid Number (#0989)	g KOH/kg	30	0.048	0.040	0.040
Breakdown Voltage (#0988)	kV/2.5 mm	31	56.0	32.6	18.5
Colour (#0988)		13	0.3	0.7	1.0
Density @ 20 °C (#0988)	kg/m ³	23	878.8	1.0	1.2
Di-electric loss 90 °C:					
* Di-electric Dissipation Factor (#0988)		23	0.0012	0.0017	0.0043
* Di-electric Dissipation Factor (#0989)		24	0.0137	0.0139	0.0328
* Specific Resistance (#0988)	GΩm	15	520.8	705.9	546.8
* Specific Resistance (#0989)	GΩm	14	16.8	9.2	17.7
Interfacial Surface Tension (#0988)	mN/m	21	43.8	5.5	4.4
Interfacial Surface Tension (#0988)	mN/m	20	21.8	5.6	2.2
Water (#0988)	mg/kg	32	14.4	6.5	14.0
Water (#0989)	mg/kg	33	66.5	23.4	209.7
Cadmium (#0989)	mg/kg	0	n.a.	n.a.	n.a
Furanic Compounds (#0989):					
* 2-acetylfuran	mg/kg	4	n.a.	n.a.	n.a
* 2-furfural	mg/kg	12	0.0348	0.0518	0.0259*
* 2-furfuryl alcohol	mg/kg	1	n.a.	n.a.	n.a
* 5-hydroxy-2-furfural	mg/kg	4	n.a.	n.a.	n.a
* 5-methyl-2-furfural	mg/kg	5	n.a.	n.a.	n.a

table 6: Performance of the group on sample #0988 and #0989

* R(Horwitz) was used for calculating the z-score

Without further statistical calculations it can be concluded that for many tests there is not a good compliance of the group of participating laboratories with the relevant standards or the

rather strict calculated estimates using Horwitz. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE NOVEMBER 2009 PROFICIENCY TEST WITH PREVIOUS PT'S.

	November 2009	November 2008	November 2007	November 2006
Number of reporting labs	36	41	46	38
Number of results reported	348	410	437	285
Statistical outliers	21	16	27	30
Percentage outliers	6.0%	3.9%	6.2%	10.5%

table 7: 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 requirements of the respective standards. The conclusions are given the following table:

Determination	November 2009	November 2008	November 2007	November 2006
Acid number	+	++	++	++
Breakdown Voltage	--	--	--	--
Colour	++	++	++	++
Density @ 20 °C	++	--	--	++
Di-electric Dissipation Factor	++	++	+/-	--
Specific Resistance	-- / ++	+/-	--	--
Interfacial Surface Tension	--	--	--	--
Water	++	--	--	+/-
Furanic Compounds	--	(--)	(--)	n.d.

table 8: comparison determinations against the standards

Comparison between brackets is evaluated against the Horwitz equation

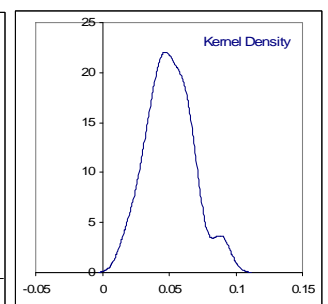
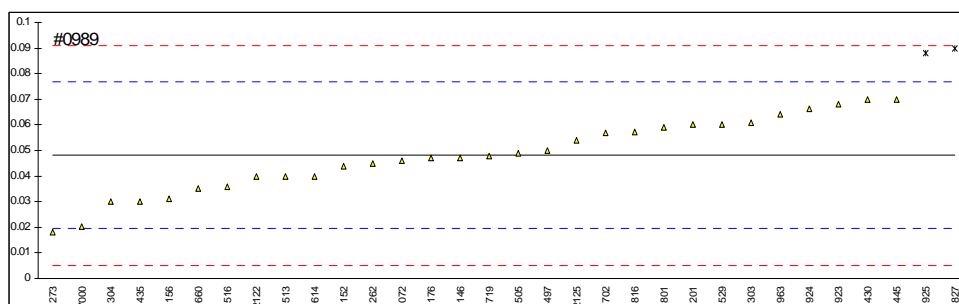
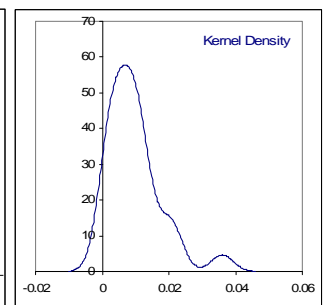
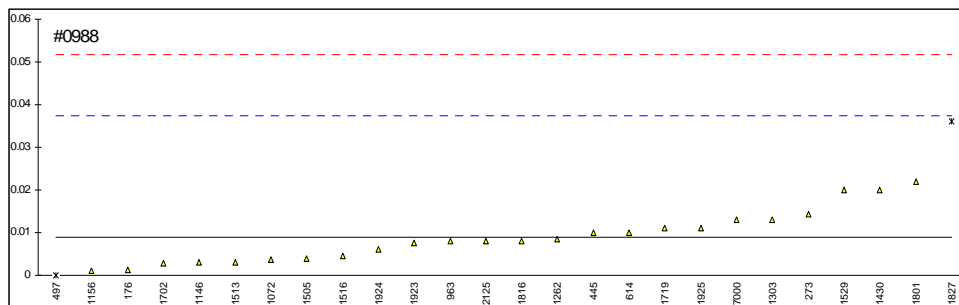
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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
- n.d.: not determined

APPENDIX 1

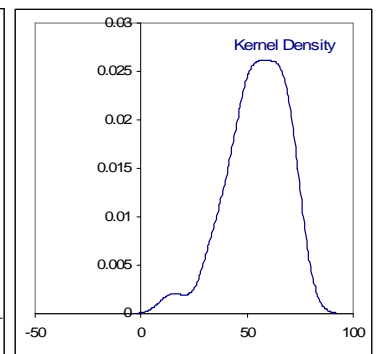
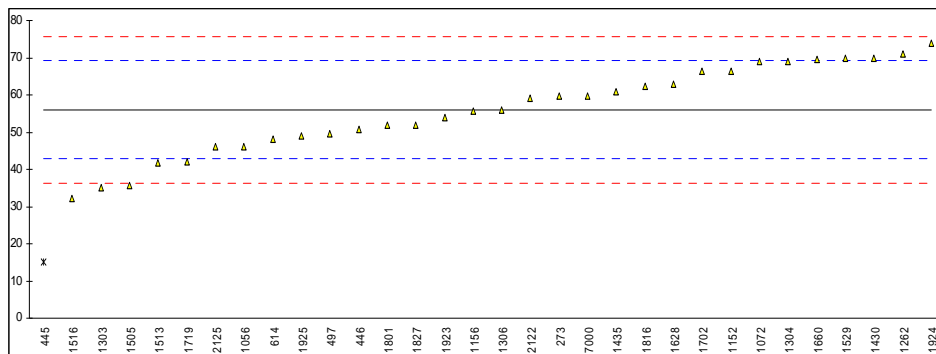
Determination of Acid Number on samples #0988 and #0989; results in g KOH/kg

lab	method	#0988	mark	z(targ)	#0989	mark	z(targ)	remarks
176	D974	0.001267		-0.53	0.046945		-0.08	
273	D974	0.0143		0.38	0.0182	C	-2.09	First reported 0.0903
445	IEC62021	0.01		0.08	0.07		1.53	
446	D974	<0.01		----	----		----	
497	D974	0	ex	-0.62	0.050		0.13	Zero not a real value
614	IEC60296	0.01		0.08	0.04		-0.57	
963	D974	0.008		-0.06	0.064		1.11	
1056		----		----	----		----	
1072	INH-04	0.0037		-0.36	0.0459		-0.15	
1146	D664	0.003		-0.41	0.047		-0.08	
1152	D974	<0.001		----	0.044		-0.29	
1156	D974	0.001		-0.55	0.031		-1.20	
1178		----		----	----		----	
1201	D974	<0.01		----	0.06		0.83	
1262	EN62021	0.0084		-0.04	0.045		-0.22	
1303	D974	0.013		0.29	0.061		0.90	
1304	INH-122	<0.01		----	0.03		-1.27	
1306		----		----	----		----	
1430	D974	0.02		0.78	0.07		1.53	
1435	IEC62021	<0.01		----	0.03		-1.27	
1505	D974	0.004		-0.34	0.049		0.06	
1513	IEC62021	0.0031		-0.41	0.04		-0.57	
1516	D974	0.0045		-0.31	0.036		-0.85	
1526		----		----	----		----	
1529	D974	0.02		0.78	0.06		0.83	
1628		----		----	----		----	
1660	IEC62021	<0.01		----	0.035		-0.92	
1702	IEC62021	0.00285		-0.42	0.05684		0.61	
1719	D664	0.011		0.15	0.048		-0.01	
1801	IEC62021	0.022		0.92	0.059		0.76	
1816	D664	0.0081		-0.06	0.0574		0.65	
1827	D664	0.036	G(0.01)	1.90	0.090	DG(0.05)	2.93	
1923	IEC62021	0.0076		-0.09	0.068		1.39	
1924	IEC62021	0.00599		-0.20	0.0662		1.27	
1925	IEC62021	0.011		0.15	0.088	DG(0.05)	2.79	
2122	IP1A	<0.01		----	0.04		-0.57	
2125	ISO6615	0.008		-0.06	0.054		0.41	
7000	ICE62021	0.013		0.29	0.0202		-1.95	
	normality	OK			OK			
	n	24			30			
	outliers	1			2			
	mean (n)	0.0089			0.0481			
	st.dev. (n)	0.00590			0.01423			
	R(calc.)	0.0165			0.0399			
	R(D974:08)	0.0400			0.0400			



Determination of Breakdown Voltage on sample #0988, results in kV/2.5 mm

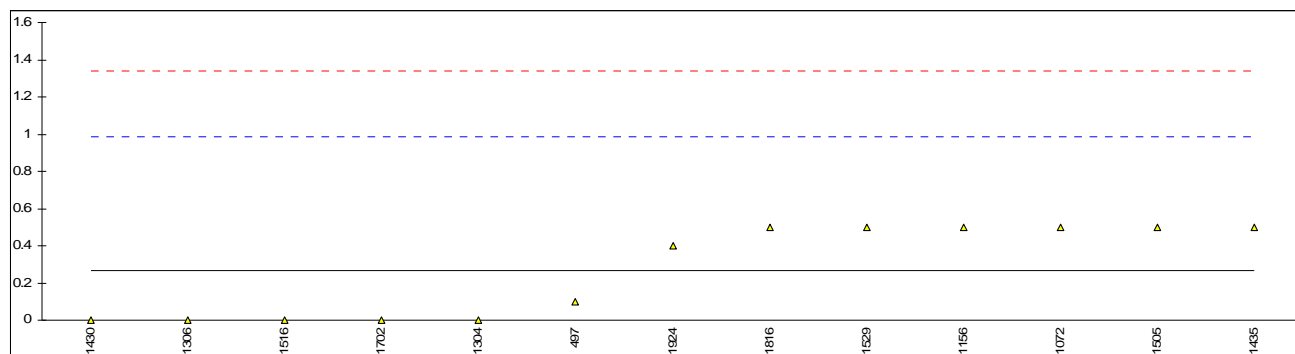
lab	method	value	mark	z(targ)	remarks
176		----		----	
273	IEC60156	59.6		0.55	
445	IEC60156	15	G(0.05)	-6.21	
446	IEC60156	50.7		-0.80	
497	IEC60156	49.7		-0.95	
614	IEC60156	48.1		-1.19	
963		----		----	
1056	IEC60156	46		-1.51	
1072	EN60156	69		1.97	
1146	IEC156	>80		>3.64	False positive?
1152	IEC60156	66.43		1.58	
1156	IEC60156	55.6		-0.06	
1178		----		----	
1201		----		----	
1262	IEC60156	71.1		2.29	
1303	IEC60156	35.0		-3.18	
1304	INH-124	69		1.97	
1306	IEC60247	56		0.00	
1430	IEC60156	70.0		2.13	
1435	IEC60156	61		0.76	
1505	IEC60156	35.7		-3.07	
1513	IEC60156	41.8		-2.15	
1516	IEC60156	32.3		-3.59	
1526		----		----	
1529	IEC60156	70		2.13	
1628	IEC156	62.8		1.03	
1660	IEC60156	69.6		2.06	
1702	IEC60156	66.40		1.58	
1719	IEC60156	41.9		-2.13	
1801	IEC60156	51.9		-0.62	
1816	IEC60156	62.3		0.96	
1827	EN60156	52		-0.60	
1923	IEC60156	53.8		-0.33	
1924	IEC60156	74.00		2.73	
1925	IEC60156	48.9		-1.07	
2122	IP295	59		0.46	
2125	IEC60156	46		-1.51	
7000	IEC60156	59.725		0.57	
normality		OK			
n		31			
outliers		1			
mean (n)		55.98			
st.dev. (n)		11.639			
R(calc.)		32.59			
R(IEC60156:95)		18.47			



Determination of Colour on samples #0988;

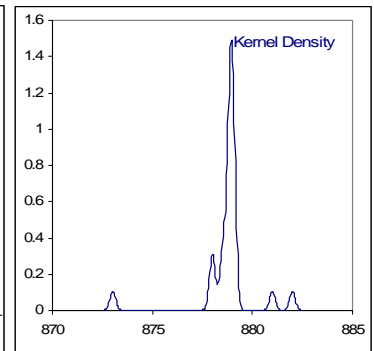
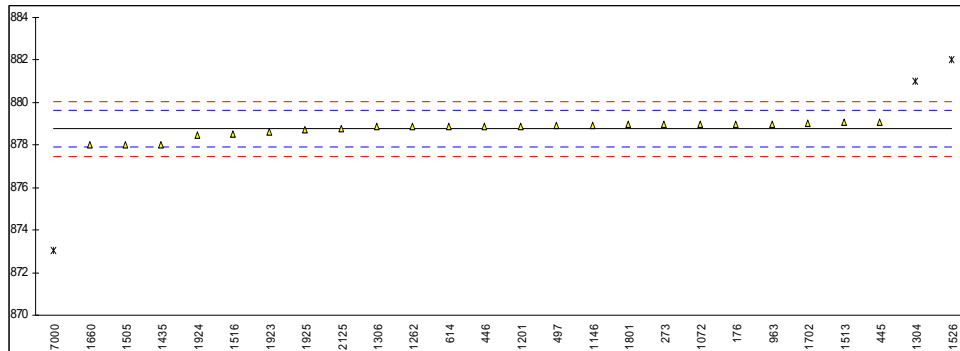
lab	method	value	mark	z(targ)	remarks
176	D1500	L0.5		----	
273	D1500	L0.5		----	
445	D1500	L0.5		----	
446	D1500	L0.5		----	
497	D1500	0.1		-0.47	
614	INH-830	Straw		----	
963	D1500	L0.5		----	
1056		----		----	
1072	UNE21320	0.5		0.65	
1146		----		----	
1152	D1500	L0.5		----	
1156	D1500	0.5		0.65	
1178		----		----	
1201	D1500	L0.5		----	
1262	D1500	L0.5		----	
1303	D1500	L0.5		----	
1304	D1500	0		-0.75	
1306	D1500	0		-0.75	
1430	D1500	0		-0.75	
1435	ISO2049	0.5		0.65	
1505	D1500	0.5		0.65	
1513	ISO2049	L0.5		----	
1516	D1500	0		-0.75	
1526		----		----	
1529	D1500	0.5		0.65	
1628		----		----	
1660	D1500	L0.5		----	
1702	D1500	0.0		-0.75	
1719	D1524	L0.5		----	
1801	UNE21320-3	L0.5		----	
1816	D1500	0.5		0.65	
1827		----		----	
1923	D1500	L0.5		----	
1924	D6045	0.4		0.37	
1925	D1500	L0.5		----	
2122	D1500	Pale Straw		----	
2125		----		----	
7000		----		----	

normality not OK
n 13
outliers 0
mean (n) 0.3
st.dev. (n) 0.25
R(calc.) 0.7
R(D1500:08) 1.0



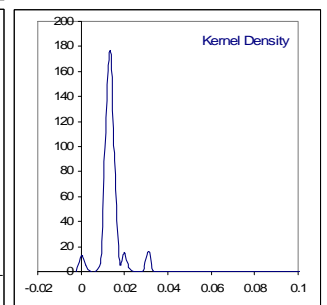
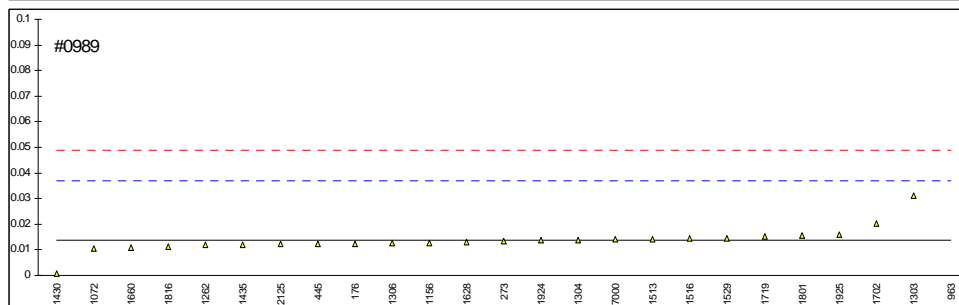
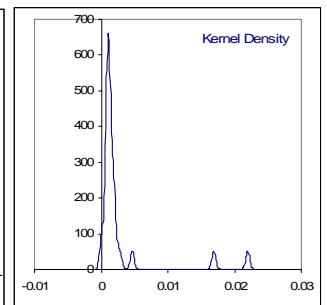
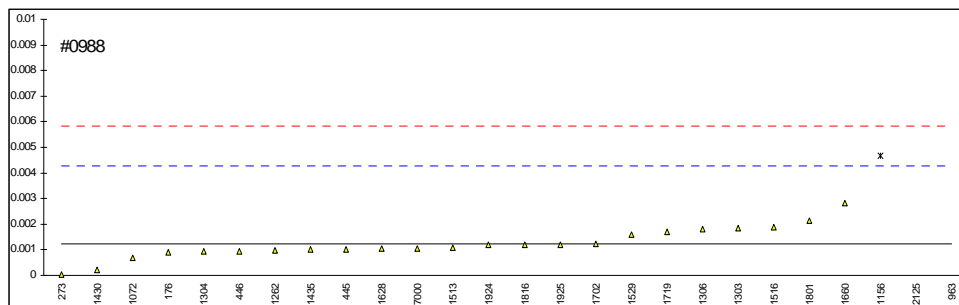
Determination of Density @ 20°C on sample #0988; results in kg/m³

lab	method	value	mark	z(targ)	remarks
176	D4052	879.0	C	0.55	First reported 0.8790
273	D4052	879.0		0.55	
445	IP365	879.1		0.79	
446	D4052	878.9		0.32	
497	ISO3675	878.92		0.37	
614	ISO3675	878.9		0.32	
963	D4052	879.0		0.55	
1056		----		----	
1072	UNE21320part4	879	C	0.55	First reported 884
1146	ISO12185	878.92		0.37	
1152		----		----	
1156		----		----	
1178		----		----	
1201	ISO3675	878.9		0.32	
1262	ISO3675	878.9		0.32	
1303		----		----	
1304	INH-102	881.0	G(0.01)	5.22	
1306	IEC60256	878.9		0.32	
1430		----		----	
1435	ISO3675	878		-1.78	
1505	D1298	878.0		-1.78	
1513	ISO12185	879.073		0.72	
1516	ISO3675	878.5		-0.61	
1526	INH-D5002	882	G(0.01)	7.55	
1529		----		----	
1628		----		----	
1660	ISO3675	878.0		-1.78	
1702	ISO3675	879.004		0.56	
1719		----		----	
1801	ISO3675	879	C	0.55	First reported 0.8794
1816		----		----	
1827		----		----	
1923	ISO3675	878.6	C	-0.38	First reported 0.8786
1924	ISO3675	878.46		-0.71	
1925	ISO3675	878.7	C	-0.15	First reported 0.8787
2122		----		----	
2125	ISO3675	878.77		0.02	
7000	ISO3675	873.0202	C,G(0.01)	-13.40	First reported 0.87302
normality		not OK			
n		23			
outliers		3			
mean (n)		878.76			
st.dev. (n)		0.346			
R(calc.)		0.97			
R(ISO3675:98)		1.20			



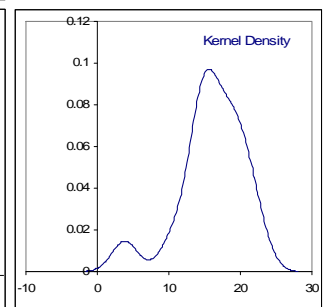
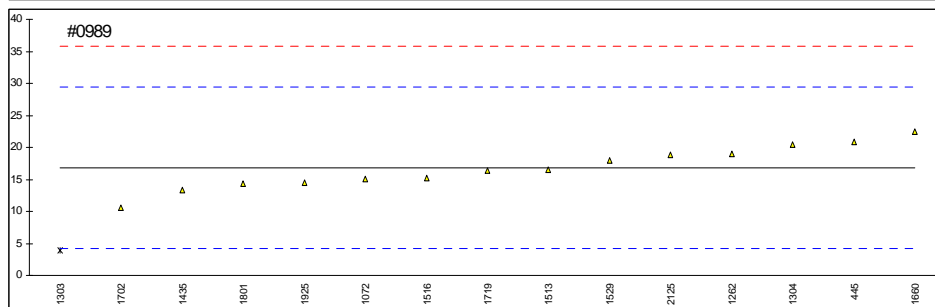
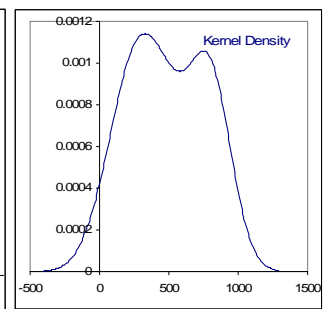
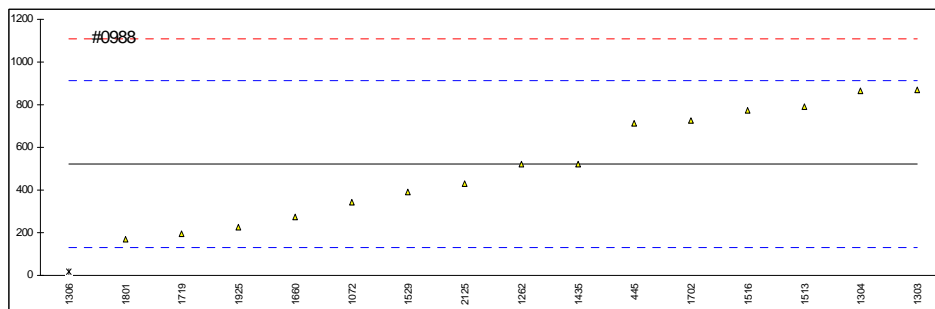
Determination of Di-electric Dissipation Factor on samples #0988 and #0989;

lab	method	#0988	mark	z(targ)	#0989	mark	z(targ)	remarks
176	IEC247	0.00092		-0.21	0.01243		-0.11	
273	IEC60247	0.00003		-0.80	0.01338		-0.03	
445	IEC60247	0.00103		-0.14	0.01232		-0.12	
446	IEC60247	0.00096		-0.19	----		----	
497		----		----	----		----	
614		----		----	----		----	
963	D924	0.022	G(0.01)	13.62	0.560	G(0.01)	46.67	
1056		----		----	----		----	
1072	EN60247	0.000680		-0.37	0.010500		-0.27	
1146		----		----	----		----	
1152		----		----	----		----	
1156	IEC60247	0.00467	CG(0.01)	2.25	0.01285		-0.07	First reported 0.0096
1178		----		----	----		----	
1201		----		----	----		----	
1262	IEC60247	0.00099		-0.17	0.01182		-0.16	
1303	IEC60247	0.00185		0.40	0.03102		1.48	
1304	INH-125	0.00094		-0.20	0.01389		0.01	
1306	IEC60247	0.00182		0.38	0.01269		-0.09	
1430	IEC60247	0.00022		-0.67	0.000685		-1.11	
1435	IEC60247	0.001		-0.16	0.012		-0.15	
1505		----		----	----		----	
1513	IEC60247	0.00107		-0.11	0.01421		0.04	
1516	IEC60247	0.001875		0.41	0.01439		0.06	
1526		----		----	----		----	
1529	IEC60247	0.0016		0.23	0.0145		0.07	
1628	IEC60247	0.00105		-0.13	0.01313		-0.05	
1660	IEC60247	0.00282		1.03	0.01080		-0.25	
1702	IEC60247	0.001235		-0.01	0.0202385		0.56	
1719	IEC60247	0.00172		0.31	0.01531		0.14	
1801	IEC60247	0.00215		0.59	0.01555		0.16	
1816	IEC61620	0.00121		-0.02	0.0114		-0.20	
1827		----		----	----		----	
1923		----		----	----		----	
1924	IEC60247	0.001200		-0.03	0.01385		0.01	
1925	IEC60247	0.00121		-0.02	0.01595		0.19	
2122		----		----	----		----	
2125	IEC60247	0.0169	G(0.01)	10.27	0.01229		-0.12	
7000	IEC60247	0.00106		-0.12	0.01403		0.03	
	normality	not OK			not OK			
	n	23			24			
	outliers	3			1			
	mean (n)	0.0012			0.0137			
	st.dev. (n)	0.00061			0.00495			
	R(calc.)	0.0017			0.0139			
	R(IEC60247:78)	0.0043			0.0328			



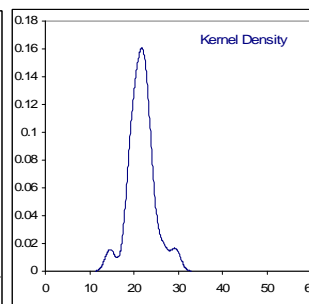
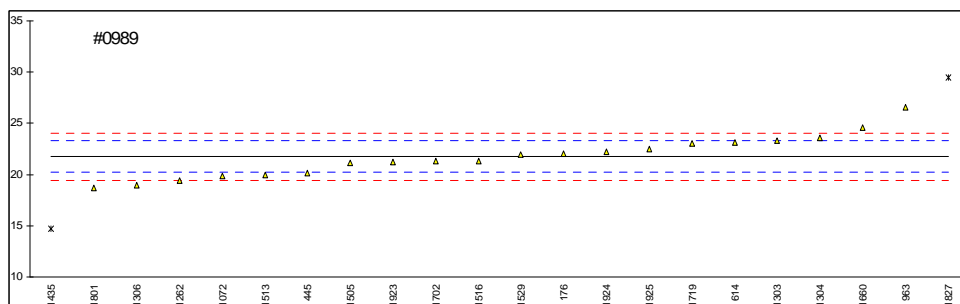
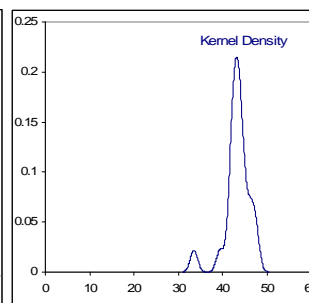
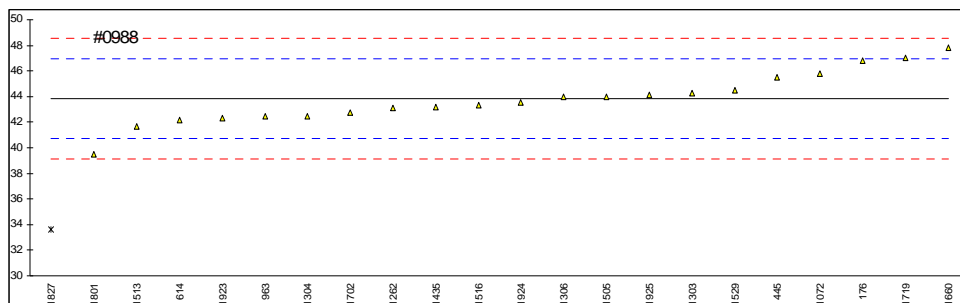
Determination of Specific Resistance on sample #0988 and #0989; results in GΩm

lab	method	#0988	mark	z(targ)	#0989	mark	z(targ)	remarks
176		----		----	----		----	
273		----		----	----		----	
445	IEC60247	714.0		0.99	20.8		0.63	
446		----		----	----		----	
497		----		----	----		----	
614		----		----	----		----	
963		----		----	----		----	
1056		----		----	----		----	
1072	EN60247	344.8		-0.90	15.05		-0.28	
1146		----		----	----		----	
1152		----		----	----		----	
1156		----		----	----		----	
1178		----		----	----		----	
1201		----		----	----		----	
1262	IEC60247	521.5		0.00	19.0		0.35	
1303	IEC60247	869		1.78	3.852	G(0.05)	-2.06	
1304	INH-125	863.95		1.76	20.4		0.57	
1306	IEC60247	18.9	G(0.05)	-2.57	----		----	
1430		----		----	----		----	
1435	IEC60247	523.9		0.02	13.4		-0.54	
1505		----		----	----		----	
1513	IEC60247	789.8		1.38	16.5		-0.05	
1516	IEC60247	776.0		1.31	15.2		-0.26	
1526		----		----	----		----	
1529	IEC60247	390		-0.67	18		0.19	
1628		----		----	----		----	
1660	IEC60247	273.9		-1.26	22.5		0.90	
1702	IEC60247	725.15		1.05	10.55		-0.99	
1719	IEC60247	194.7		-1.67	16.4		-0.07	
1801	IEC60247	168.0		-1.81	14.3		-0.40	
1816		----		----	----		----	
1827		----		----	----		----	
1923		----		----	----		----	
1924		----		----	----		----	
1925	IEC60247	227.6		-1.50	14.5		-0.37	
2122		----		----	----		----	
2125	IEC60247	429.0		-0.47	18.9		0.33	
7000		----		----	----		----	
normality		OK			OK			
n		15			14			
outliers		1			1			
mean (n)		520.75			16.82			
st.dev. (n)		252.109			3.284			
R(calc.)		705.91			9.19			
R(IEC60247:78)		546.79			17.66			



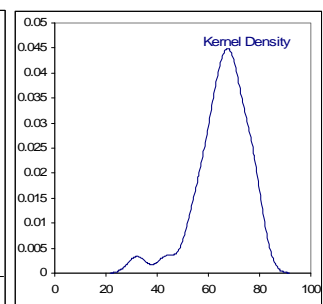
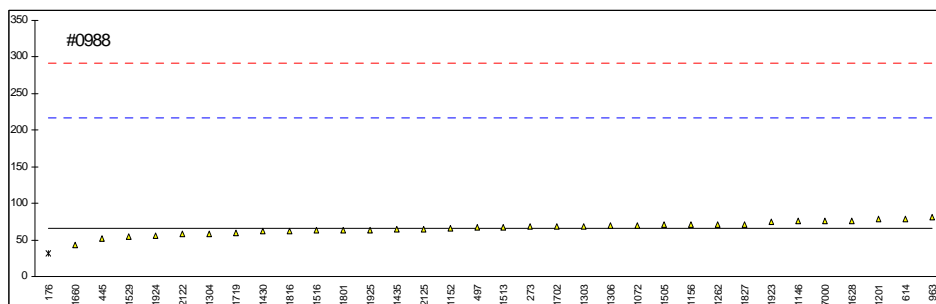
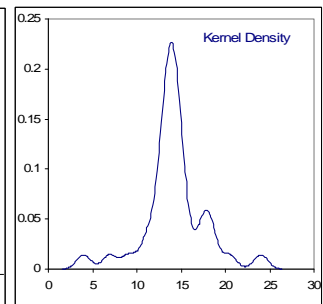
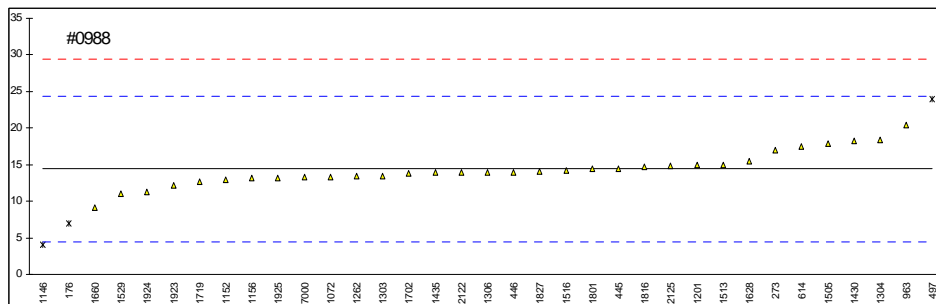
Determination of Interfacial Surface Tension on samples #0988 and #0989; results in mN/m

lab	method	#0988	mark	z(targ)	#0989	mark	z(targ)	remarks
176	D971	46.80		1.90	22.02		0.35	
273		----		----	----		----	
445	D971	45.5		1.07	20.1	C	-2.12	First reported 16.2
446		----		----	----		----	
497		----		----	----		----	
614	ISO6295	42.2		-1.04	23.1		1.74	
963	D971	42.46		-0.87	26.6	C	6.25	First reported 26.6
1056		----		----	----		----	
1072	UNE21320part6	45.8	C	1.26	19.9		-2.38	First reported 37.6
1146		----		----	----		----	
1152		----		----	----		----	
1156		----		----	----		----	
1178		----		----	----		----	
1201		----		----	----		----	
1262	ISO6295	43.1		-0.46	19.4		-3.02	
1303	D971	44.3		0.30	23.3		2.00	
1304	INH-123	42.5		-0.85	23.6		2.39	
1306	D971	44		0.11	19		-3.54	
1430		----		----	----		----	
1435	D971	43.2		-0.40	14.7	G(0.05)	-9.07	
1505	D971	44.0		0.11	21.1		-0.83	
1513	D971	41.65		-1.39	19.96		-2.30	
1516	D971	43.3		-0.33	21.3		-0.58	
1526		----		----	----		----	
1529	ISO6295	44.5		0.43	22.0		0.33	
1628		----		----	----		----	
1660	D971	47.8		2.54	24.6		3.67	
1702	ISO6295	42.728		-0.70	21.284		-0.60	
1719	D2285	47		2.03	23		1.61	
1801	ISO6295	39.5		-2.76	18.7		-3.92	
1816		----		----	----		----	
1827	D971	33.60	G(0.01)	-6.53	29.45	G(0.05)	9.92	
1923	D971	42.3		-0.97	21.27		-0.61	
1924	D971	43.56		-0.17	22.20		0.58	
1925	D971	44.1		0.18	22.5		0.97	
2122		----		----	----		----	
2125		----		----	----		----	
7000		----		----	----		----	
normality		OK			OK			
n		21			20			
outliers		1			2			
mean (n)		43.82			21.75			
st.dev. (n)		1.959			1.981			
R(calc.)		5.48			5.55			
R(ISO6295:83)		4.38			2.17			ISO 6295 is obsolete since feb 2005
R(D971:04)		4.38			2.17			



Determination of Water on samples #0988 and #0989; results in mg/kg

lab	method	#0988	mark	z(targ)	#0989	mark	z(targ)	remarks
176	D1533	6.93	DG(0.05)	-1.49	32.17	G(0.05)	-0.46	
273	IEC60814	17.0		0.52	68		0.02	
445	IEC60814	14.5		0.02	52.5		-0.19	
446	D1533	14		-0.08	-----		-----	
497	D1364	24	G(0.01)	1.92	67		0.01	
614	IEC60814	17.5		0.62	78.2		0.16	
963	D1533	20.46	C	1.21	81.02		0.19	First reported 23.84
1056		-----		-----	-----		-----	
1072	EN60814	13.3		-0.22	70.0		0.05	
1146	D6304-C	4	DG(0.05)	-2.08	76		0.13	
1152	IEC60814	12.90		-0.30	65.74		-0.01	
1156	IEC60814	13.2		-0.24	71.4		0.06	
1178		-----		-----	-----		-----	
1201	IEC60814	15		0.12	78		0.15	
1262	IEC60814	13.4		-0.20	71.58		0.07	
1303	IEC60814	13.5		-0.18	69		0.03	
1304	INH-121	18.4		0.80	58.6		-0.11	
1306	IEC60814	14		-0.08	70		0.05	
1430	IEC60814	18.3		0.78	61.6		-0.07	
1435	IEC60814	14		-0.08	64.3		-0.03	
1505	D1533	17.9		0.70	70.4		0.05	
1513	IEC60814	15.02		0.12	67.40		0.01	
1516	IEC60814	14.2		-0.04	62.8		-0.05	
1526	INH-D4377	<5000		-----	<5000		-----	
1529	IEC60814	11		-0.68	54		-0.17	
1628	IEC60814	15.5		0.22	76.5		0.13	
1660	IEC60814	9.1		-1.06	43.7		-0.31	
1702	IEC60814	13.785		-0.12	68.130		0.02	
1719	IEC60814	12.7		-0.34	59.2		-0.10	
1801	IEC60814	14.5		0.02	63.0		-0.05	
1816	IEC60814	14.7		0.06	62.6		-0.05	
1827	D6304	14.1		-0.06	71.6		0.07	
1923	IEC60814	12.2		-0.44	75.1		0.11	
1924	IEC60814	11.34		-0.61	55.56		-0.15	
1925	IEC60814	13.2		-0.24	63.8		-0.04	
2122	IEC733	14		-0.08	58		-0.11	
2125	IEC60814	14.8		0.08	65.0		-0.02	
7000	IEC60814	13.3		-0.22	76.15		0.13	
	normality	not OK			OK			
	n	32			33			
	outliers	3			1			
	mean (n)	14.40			66.54			
	st.dev. (n)	2.314			8.338			
	R(calc.)	6.48			23.35			
	R(D1533:05)	14.00			209.67	R(D6305:07)		
	R(IEC60814:97)	5.69			(12.25)	R(IEC60814:97)	not applicable for used oils	



Determination of Cadmium on samples #0989; results in mg/kg

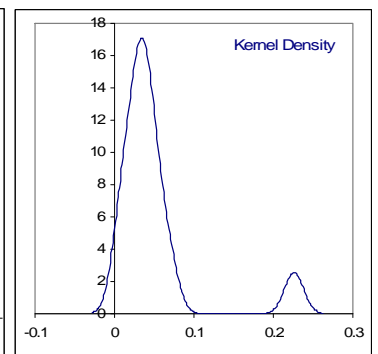
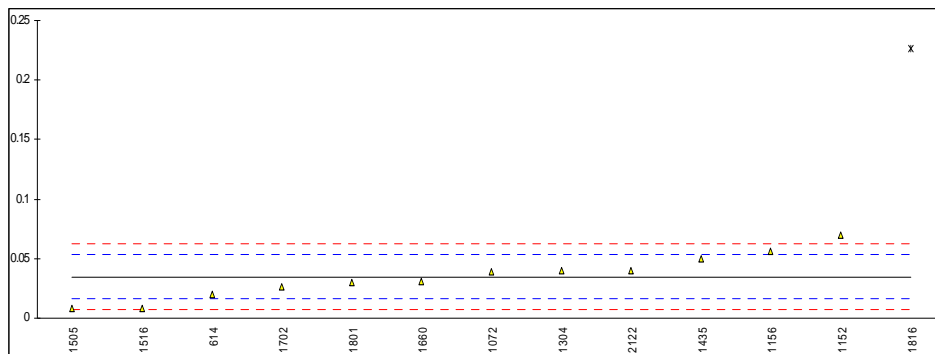
lab	method	value	mark	z(targ)	remarks
176		----		----	
273		----		----	
445		----		----	
446		----		----	
497		----		----	
614		----		----	
963		----		----	
1056		----		----	
1072		----		----	
1146		----		----	
1152		----		----	
1156		----		----	
1178		----		----	
1201		----		----	
1262	D4951	0.0		----	
1303	INHOUSE	ND		----	
1304		----		----	
1306		----		----	
1430		----		----	
1435	D5184	<1		----	
1505	AAS	<0.1		----	
1513		----		----	
1516		----		----	
1526	INH-D5185	<0.25		----	
1529		----		----	
1628		----		----	
1660	D7151	<0.1		----	
1702		----		----	
1719		----		----	
1801		----		----	
1816		----		----	
1827		----		----	
1923		----		----	
1924		----		----	
1925		----		----	
2122		----		----	
2125		----		----	
7000		----		----	
	normality	n.a.			
	n	0			
	outliers	0			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	unknown			

Determination of 2-furfural on sample #0989; results in mg/kg

lab	method	value	mark	z(targ)	remarks
176		----		----	
273		----		----	
445	IEC61198	<0.05		----	
446		----		----	
497		----		----	
614	IEC61198	0.02		-1.61	
963		----		----	
1056		----		----	
1072	EN61198	0.039		0.45	
1146		----		----	
1152	IEC61198	0.07		3.81	
1156	IEC61198	0.056		2.29	
1178		----		----	
1201		----		----	
1262		----		----	
1303		----		----	
1304	INH-126	0.04		0.56	
1306	IEC61198	<0.061		----	
1430	IEC61198	<0.03		----	
1435	IEC61198	0.05		1.64	
1505	D5837	0.008		-2.90	
1513	IEC61198	<0.05		----	
1516	IEC61198	0.008		-2.90	
1526		----		----	
1529	IEC61198	<0.10		----	
1628		----		----	
1660	IEC61198	0.031		-0.41	
1702	IEC61198	0.025957	C	-0.96	First reported 25.957
1719		----		----	
1801	IEC61198	0.03		-0.52	
1816	IEC61198	0.226	G(0.01)	20.70	
1827		----		----	
1923		----		----	
1924		----		----	
1925		----		----	
2122	IEC61198	0.04		0.56	
2125	IEC61198	<0.1		----	
7000		----		----	

normality OK
n 12
outliers 1
mean (n) 0.0348
st.dev. (n) 0.01849
R(calc.) 0.0518
R(Horwitz) 0.0259

Compare R(IEC61198) = 0.0052



Determination of other Furanic compounds on sample #0989; results in mg/kg

lab	method	2-af	z(targ)	2-fa	z(targ)	5-hm-2-f	z(targ)	5-m-2-f	z(targ)
176		----	----	----	----	----	----	----	----
273		----	----	----	----	----	----	----	----
445	IEC61198	<0.05	----	<0.05	----	<0.05	----	<0.05	----
446		----	----	----	----	----	----	----	----
497		----	----	----	----	----	----	----	----
614	IEC61198	<0.01	----	<0.01	----	<0.01	----	<0.01	----
963		----	----	----	----	----	----	----	----
1056		----	----	----	----	----	----	----	----
1072	EN61198	<0.01	----	----	----	<0.01	----	0.007	----
1146		----	----	----	----	----	----	----	----
1152	IEC61198	<0.01	----	<0.01	----	<0.01	----	<0.01	----
1156	IEC61198	0.00	----	----	----	0.00	----	0.00	----
1178		----	----	----	----	----	----	----	----
1201		----	----	----	----	----	----	----	----
1262		----	----	----	----	----	----	----	----
1303		----	----	----	----	----	----	----	----
1304	INH-126	<0.01	----	<0.01	----	<0.01	----	<0.01	----
1306	IEC61198	<0.01	----	<0.01	----	<0.01	----	<0.01	----
1430	IEC61198	<0.01	----	<0.04	----	<0.02	----	<0.03	----
1435	IEC61198	<0.03	----	<0.03	----	<0.03	----	<0.03	----
1505	D5837	0.007	----	<0.001	----	<u>0.046</u>	----	<u>0.093</u>	----
1513	IEC61198	<0.05	----	<0.05	----	<0.05	----	<0.05	----
1516	IEC61198	<0.05	----	<0.05	----	<0.05	----	<0.05	----
1526		----	----	----	----	----	----	----	----
1529	IEC61198	<0.10	----	<0.10	----	<0.10	----	<0.10	----
1628		----	----	----	----	----	----	----	----
1660	IEC61198	<0.01	----	<0.01	----	<0.01	----	<0.01	----
1702	IEC61198	nd.	----	nd.	----	nd.	----	nd.	----
1719		----	----	----	----	----	----	----	----
1801	IEC61198	0.01	----	----	----	<u>0.12</u>	----	0.01	----
1816	IEC61198	0.00	----	<u>0.310</u>	----	0.00	----	0.00	----
1827		----	----	----	----	----	----	----	----
1923		----	----	----	----	----	----	----	----
1924		----	----	----	----	----	----	----	----
1925		----	----	----	----	----	----	----	----
2122	IEC61198	----	----	----	----	----	----	----	----
2125	IEC61198	<0.1	----	<0.1	----	<0.1	----	<0.1	----
7000		----	----	----	----	----	----	----	----
	normality	n.a.		n.a.		n.a.		n.a.	
	n	4		1		4		5	
	outliers	0		0		0		0	
	mean (n)	n.a.		n.a.		n.a.		n.a.	
	st.dev. (n)	n.a.		n.a.		n.a.		n.a.	
	R(calc.)	n.a.		n.a.		n.a.		n.a.	
	R(lit)	n.a.		n.a.		n.a.		n.a.	

(results in italic, bold and underlined are marked as "false positive")

Abbreviations:

2-af	= 2-acetylfuran
2-fa	= 2-furfurylalcohol
5-hm-2-f	= 5-hydroxymethyl-2-furfural
5-m-2-f	= 5-methyl-2-furfural

APPENDIX 2

List of participants

3 laboratories in	AUSTRALIA
2 laboratories in	BELGIUM
4 laboratories in	BULGARIA
1 laboratory in	CROATIA
1 laboratory in	ESTONIA
1 laboratory in	GERMANY
1 laboratory in	GREECE
1 laboratory in	IRAN
1 laboratory in	ITALY
3 laboratories in	MALAYSIA
1 laboratory in	NEW ZEALAND
1 laboratory in	NORWAY
2 laboratories in	PORTUGAL
1 laboratory in	SAUDI ARABIA
2 laboratories in	SOUTH AFRICA
6 laboratories in	SPAIN
3 laboratories in	THE NETHERLANDS
1 laboratory in	U.S.A.
3 laboratories in	UNITED KINGDOM

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
ex	= excluded from calculations
n.a.	= not applicable
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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