Results of Proficiency Test
Transformer Oil
November 2010

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 2001, the Institute for Interlaboratory Studies organized a proficiency test for the analysis of Transformer Oil every year. It was decided to continue this interlaboratory study during the annual program 2010/2011. In this interlaboratory study, 50 laboratories from 24 different countries have participated. See appendix 2 for a list of number of participants per country order. In this report, the results of the interlaboratory study on unused transformer oil 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 a bottle of 1 litre of an unused Transformer Oil (labelled #1085) and a bottle of 0.5 litre of used Transformer Oil (labelled #1086). After receipt of the samples several participants reported that sample #1086 was cloudy and contained free water. Regretfully, this was not noticed during filling and analyzing of the samples #1086. It was decided to cancel sample #1086. However, to fulfil the needs of the participants that test for analyze Furanic compounds, it was decide to send an additional positive sample #1086FC especially for the analysis of Furanic compounds.
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% confidentially 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 January 2010 (iis-protocol, version 3.2).

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

The necessary bulk material (DIALA B dried) for the unused oil sample #1085 was obtained from a local supplier. The approximately 60 litre bulk material was homogenised. After homogenisation, 60 subsamples were transferred to 1 litre amber glass bottles and labelled #1085. The homogeneity of the subsamples #1085 was checked by determination Density and Water on 8 stratified randomly selected samples.

	Water in mg/kg	Density @ 15°C in kg/m^3
Sample #1085-1	23	881.47
Sample #1085-2	22	881.46
Sample #1085-3	21	881.45
Sample #1085-4	21	881.45
Sample #1085-5	23	881.45
Sample #1085-6	23	881.44
Sample #1085-7	23	881.44
Sample #1085-8	22	881.40

Table 1: homogeneity test results of subsamples #1085

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/m^3
r (Observed)	2	0.01
reference method	IEC60814:97	ISO3675:98
0.3 * R (ref. method)	2	0.36

Table 2: repeatabilities of subsamples #1085

The necessary bulk material for additional sample #1086FC, specifically for Furanic compounds was obtained from a participating laboratory. This material was also used in a previous proficiency test iis07L03. The spread found in on the samples in 2007 was acceptable and therefore it was decided to use this material again.
After homogenisation, the bulk material was transferred to 50 subsamples, 100 mL amber glass bottles and labelled #1086FC.

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

To each of the participating laboratories, 1*1 litre bottle (labelled #1085) and 1*0.5 litre bottle (labelled #1086) was sent on October 20, 2010. On request, the additional sample #1086FC a 1*100mL bottle (labelled #1086FC) was sent on November 10, 2010.

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 #1085: Acid Number (Neutralization Number), Breakdown Voltage, Colour, Density @ 20 ^{o}C, Di-electric loss 90 ^{o}C (Di-electric Dissipation Factor and Specific Resistance), Interfacial Surface Tension and Water.
Initially on sample #1086 was asked to determine: Acid Number (Neutralization Number), Di-electric loss 90 ^{o}C (Di-electric Dissipation Factor and Specific Resistance), Interfacial Surface Tension, Water and Furanic Compounds. However, this sample was cancelled after several remarks from participants about presence of (free) water and cloudiness.
On sample #1086FC was asked to determine: Furanic Compounds (2-acetylfuran, 2-furfural, 2-furfurylalcohol, 5-hydroxymethyl-2-furfural and 5-methyl-2-furfural). To get comparable results, detailed report forms on which the units and the preferred test methods were printed, were 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 January 2010 (iis-protocol, version 3.2).

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:

$z_{(target)}$ = (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 is as follows:

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

4 EVALUATION

Some serious problems were encountered after dispatch with the samples. Several participants complained about the present of small water drops on the bottom and cloudy appearance of the sample #1086.
After investigation, it turned out that the water content of sample #1086 was above the maximum solubility in transformer oil. Therefore, it was decided by iis to cancel sample #1086 for all analyses.
To compensate the participants for the loss of sample #1086 for the determination of Furanic compounds, it was decided to send another sample transformer oil that is positive on Furanic compounds, to those participants that wanted to receive and test a sample for Furanic compounds.

Some problems were encountered during the dispatch of the unused transformer oil samples #1085 to Serbia and Tanzania due to custom clearance. One participant did not receive the Transformer Oil positive on Furanic compounds, sample #1086FC.
In total eight participants, reported results after the final reporting date and four participants did not report any results at all. Not all participants were able to report results for all tests.
In total 46 participants reported 289 numerical results. Observed were 18 outlying results, which is 6.2% 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 was not problematic. Three statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers, is in full agreement with the requirements of ASTM D974:08.

Breakdown Voltage: This determination was very problematic, as the results seem to be divided bimodally. One statistical outlier was noticed. When the two groups are evaluated separately, both calculated reproducibilities are almost in agreement with the requirements of IEC60156:95.

Colour: No analytical problems were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1500:08.

Density @ 20°C: This determination was problematic for a number of laboratories. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is almost in agreement with the requirements of ISO3675:98.

DD-Factor: 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 agreement with the requirements of IEC60247:78.

Interf. Surf. Tension: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D971:04 and/or ISO6295:83. One should be aware that ISO6295 is obsolete since Feb-2005).

Spec. Resistance: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of IEC60247:78.

Water: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of IEC60814:97.

Furanic Compounds: Sample #1086FC was found to be positive only for 2-Furfural. This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in full agreement with the estimated requirements calculated using the Horwitz equation.
It is remarkable that the precision requirements of IEC61198:93 are much smaller than precision data calculated using the Horwitz equation. This very strict precision seems to be impossible to meet as only few test results would be acceptable against IEC61198:98.
This material was also used in a previous proficiency test iis07L03. When the data are compared is it remarkable to notice that the consensus values for both rounds differ only very little, while the spread in the current PT iis10L03 is smaller then in the previous PT iis07L03.

	iis07L03 (#0784)	iis10L03 (#1086FC)
number of results	13	16
mean	0.463	0.474
st.dev	0.1133	0.0834
reproducibility	0.317	0.234

table 5: Comparison of Performance for 2-Furfural

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	g KOH/kg	30	0.0086	0.0103	0.0400
Breakdown Voltage *)	kV/2.5 mm	23/19	31.0/50.2	12.7/19.5	10.2/16.6
Colour		16	0.54	0.23	1.00
Density @ 20 ^{o}C	kg/m^3	31	877.95	1.31	1.20
Di-electric Dissipation Factor		27	0.0015	0.0018	0.0056
Interfacial Surface Tension	mN/m	23	44.49	7.06	4.45
Specific Resistance	GΩm	20	260.04	466.22	273.04
Water	mg/kg	39	22.20	8.17	7.07

table 6: Performance of the group on sample #1085

*) Data appears to be bimodally distributed

Parameter	unit	n	average	2.8 * sd	R(lit)
* 2-acetylfuran	mg/kg	5	0.023	n.a.	n.a.
* 2-furfural	mg/kg	16	0.474	0.234	0.237
* 2-furfurylalcohol	mg/kg	2	0.029	n.a.	n.a.
* 5-hydroxy-2-furfural	mg/kg	5	0.005	n.a.	n.a.
* 5-methyl-2-furfural	mg/kg	7	0.019	n.a.	n.a.

table 7: Performance of the group on sample #1086FC

Without further statistical calculations, it can be concluded that for many tests there is not a 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 2010 PROFICIENCY TEST WITH PREVIOUS PTS.

	November 2010	November 2009	November 2008	November 2007
Number of reporting labs	46	36	41	46
Number of results reported	289	348	410	437
Statistical outliers	18	21	16	27
Percentage outliers	6.2%	6.0%	3.9%	6.2%

table 8: 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 2010	November 2009	November 2008	November 2007
Acid number	++	+	++	++
Breakdown Voltage	--	--	--	--
Colour	++	++	++	++
Density @ 20^{o}C	+/-	++	--	--
Di-electric Dissipation Factor	++	++	++	+/-
Interfacial Surface Tension	--	--	--	--
Specific Resistance	--	-- / ++	+/-	--
Water	--	++	--	--
Furanic Compounds	(+/-)	--	(--)	(--)

table 9: 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:

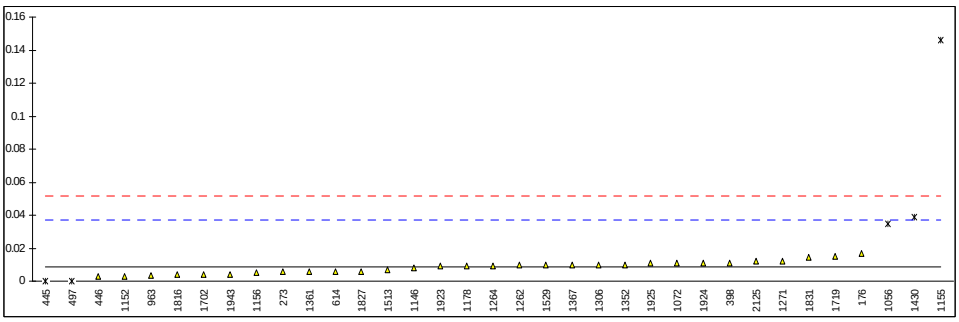
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- - : group performed worse than the standard
- -- : group performed much worse than the standard

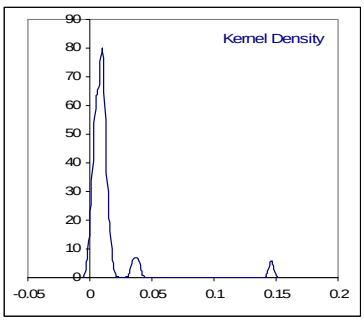
APPENDIX 1

Determination of Acid Number on sample #1085; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks
128		-----		-----	
176	ASTM D974	0.01683		0.58	
255		-----		-----	
273	ASTM D974	0.006		-0.18	
398	ASTM D974	0.011		0.17	
445	ASTM D974	0.00	ex	-0.60	Result excluded, zero not a real result
446	ASTM D974	0.0029		-0.40	
497	ASTM D974	0.000	ex	-0.60	Result excluded, zero not a real result
614	IEC60422	0.006		-0.18	
963	ASTM D974	0.0035		-0.36	
1056	ASTM D974	0.035	G(0.01)	1.85	
1066		-----		-----	
1072	INH-04	0.0110		0.17	
1146	ASTM D664	0.008		-0.04	
1152	ASTM D974	0.003		-0.39	
1155	ASTM D664	0.14615	G(0.01)	9.63	
1156	ASTM D974	0.005		-0.25	
1178	IEC62021	0.009		0.03	
1201	ASTM D974	<0.01		-----	
1262	EN62021	0.0099		0.09	
1264	ASTM D974	0.0094		0.06	
1271	ASTM D6618	0.0123		0.26	
1303	ASTM D974	<0.01		-----	
1304	INH-122	<0.01		-----	
1306	In house	0.01		0.10	
1352	IEC60621	0.01		0.10	
1361	EN62021	0.006		-0.18	
1367	ASTM D974	0.01		0.10	
1375		-----		-----	
1430	ASTM D974	0.039	G(0.01)	2.13	
1435	IEC62021	<0.01		-----	
1513	IEC62021	0.0070		-0.11	
1516	ASTM D974	<0.005		-----	
1526		-----		-----	
1529	ASTM D974	0.01		0.10	
1628		-----		-----	
1660	IEC62021	<0.01		-----	
1702	IEC62021	0.0042		-0.31	
1719	ASTM D664	0.015		0.45	
1801		-----		-----	
1816	ASTM D664	0.0041		-0.32	
1826		-----		-----	
1827	ASTM D664	0.006		-0.18	
1831	IEC60296	0.0147		0.43	
1863	ASTM D974	<0.01		-----	
1923	IEC62021	0.009		0.03	
1924	IEC62021	0.011		0.17	
1925	IEC62021	0.011		0.17	
1943	ISO6618	0.0043		-0.30	
2125	ISO6615	0.012		0.24	

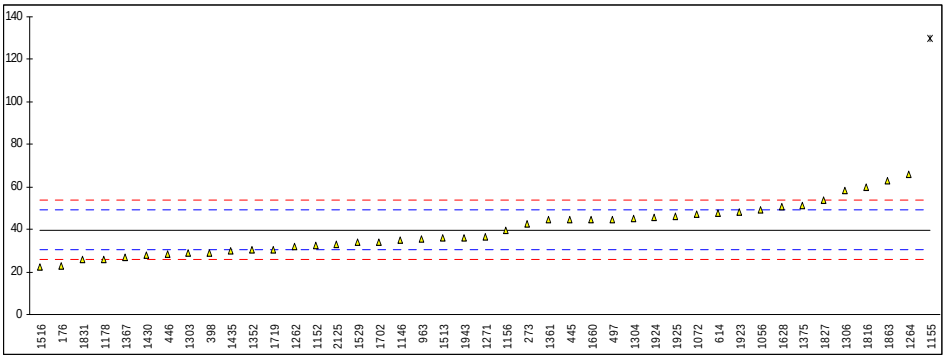
normality	OK
n	30
outliers	3
mean (n)	0.0086
st.dev. (n)	0.00369
R(calc.)	0.0103
R(D974:08)	0.0400

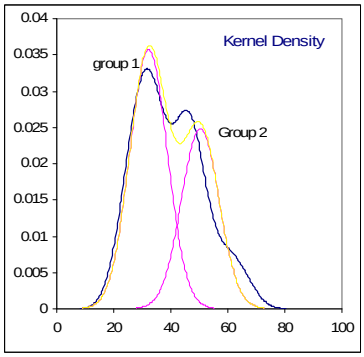




Determination of Breakdown Voltage on sample #1085, results in kV/2.5 mm

lab	method	value	mark	z(targ)	remarks
128		-----		-----	
176	ASTM D1816	22.80		-----	
255		-----		-----	
273	IEC60156	42.4		-----	
398	IEC60156	29.0		-----	
445	IEC60156	44.5		-----	
446	IEC60156	28.4		-----	
497	IEC60156	44.72		-----	
614	IEC60156	47.5		-----	
963	ASTM D877	35.5		-----	
1056	IEC60156	49		-----	
1066		-----		-----	
1072	IEC60156	47.3		-----	
1146	IEC60156	35.0		-----	
1152	IEC60156	32.50		-----	
1155	IEC60156	130	G(0.01)	-----	
1156	IEC60156	39.6		-----	
1178	IEC60156	25.8		-----	
1201		-----		-----	
1262	IEC60156	31.8		-----	
1264	IEC60156	66.1	C	-----	First reported 72
1271	IEC60156	36.3		-----	
1303	IEC60156	28.8		-----	
1304	INH-1767	45		-----	
1306	IEC60156	58.5		-----	
1352	IEC60156	30.2		-----	
1361	IEC60156	44.48333		-----	
1367	IEC60156	27		-----	
1375	IEC60156	51.2		-----	
1430	IEC60156	28		-----	
1435	IEC60156	30		-----	
1513	IEC60156	35.9		-----	
1516	IEC60156	22.4		-----	
1526		-----		-----	
1529	IEC60156	34		-----	
1628	IEC60156	50.6		-----	
1660	IEC60156	44.5		-----	
1702	IEC60156	34.2		-----	
1719	IEC60156	30.4		-----	
1801		-----		-----	
1816	IEC60156	60.0		-----	
1826		-----		-----	
1827	IEC60156	54		-----	
1831	IEC60156	25.73		-----	
1863	IEC60156	63		-----	
1923	IEC60156	48.3		-----	
1924	IEC60156	45.6		-----	
1925	IEC60156	46.4		-----	
1943	IEC60156	36.0		-----	
2125	IEC60156	33.0		-----	
		Group 1	Group 2		
	normality	OK	not OK		
	n	23	19		
	outliers	0	1		
	mean (n)	30.97	50.16		
	st.dev. (n)	4.533	6.949		
	R(calc.)	12.69	19.46		
	R(IEC60156:95)	10.22	16.55		

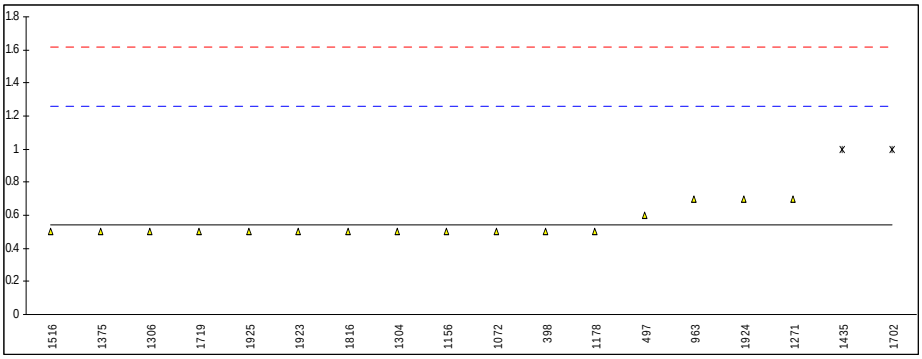


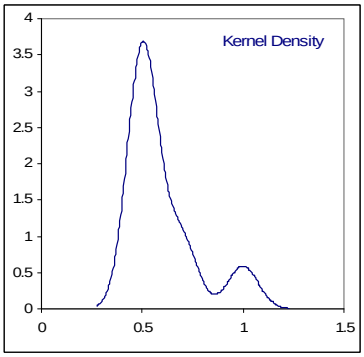


Determination of Colour on samples #1085;

lab	method	value	mark	z(targ)	remarks
128		-----		-----	
176	ASTM D1500	L0.5		-----	
255		-----		-----	
273	ASTM D1500	L0.5		-----	
398	ASTM D1500	0.5		-0.12	
445	ASTM D1500	L0.5		-----	
446	ASTM D1500	L1.0		-----	
497	ASTM D1500	0.6		0.16	
614	TM830	Pale Yellow		-----	
963	ASTM D1500	0.7		0.44	
1056		-----		-----	
1066		-----		-----	
1072	UNE 21320-3	0.5		-0.12	
1146		-----		-----	
1152	ASTM D1500	L1.0		-----	
1155	ASTM D1500	L1.0		-----	
1156	ASTM D1500	0.5		-0.12	
1178	ISO2049	0.5		-0.12	
1201	ASTM D1500	L0.5		-----	
1262	ASTM D1500	L1.0		-----	
1264	ASTM D1500	L1.0		-----	
1271	ASTM D6045	0.7		0.44	
1303	ASTM D1500	L1.0		-----	
1304	ISO2049	0.5		-0.12	
1306	ASTM D1500	0.5		-0.12	
1352		-----		-----	
1361		-----		-----	
1367	ASTM D1500	Amber		-----	
1375	ISO2049	0.5		-0.12	
1430	ASTM D1500	L1.0		-----	
1435	ISO2049	1.0	DG(0.01)	1.28	
1513	ISO2049	L1.0		-----	
1516	ASTM D1500	0.5		-0.12	
1526		-----		-----	
1529	ASTM D1500	0.5-1.0		-----	
1628		-----		-----	
1660	ASTM D1500	L0.5		-----	
1702	ASTM D1500	1.0	DG(0.01)	1.28	
1719	ASTM D1524	0.5		-0.12	
1801		-----		-----	
1816	ASTM D1500	0.5		-0.12	
1826		-----		-----	
1827		-----		-----	
1831		-----		-----	
1863	ASTM D1500	L0.5		-----	
1923	ISO2049	0.5		-0.12	
1924	ASTM D6045	0.7		0.44	
1925	ISO2049	0.5		-0.12	
1943		-----		-----	
2125		-----		-----	

normality	not OK
n	16
outliers	2
mean (n)	0.54
st.dev. (n)	0.081
R(calc.)	0.23
R(D1500:08)	1.00

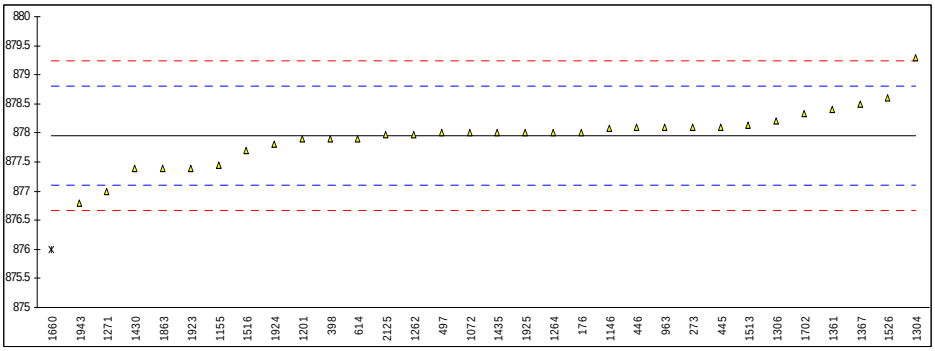


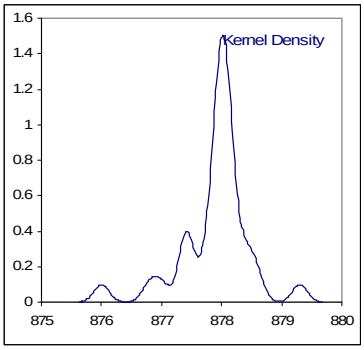


Determination of Density @ 20°C on sample #1085; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
128		-----		-----	
176	ASTM D4052	878.0		0.11	
255		-----		-----	
273	ASTM D4052	878.1		0.34	
398	ISO3675	877.9		-0.12	
445	ISO3675	878.1		0.34	
446	ASTM D4052	878.1		0.34	
497	ISO3675	878.0		0.11	
614	ISO3675	877.9		-0.12	
963	ASTM D4052	878.1		0.34	
1056		-----		-----	
1066		-----		-----	
1072	UNE 21320-4	878		0.11	
1146	ISO12185	878.08		0.30	
1152		-----		-----	
1155	ISO3675	877.45		-1.17	
1156		-----		-----	
1178		-----		-----	
1201	ISO3675	877.9		-0.12	
1262	ISO3675	877.98	U	0.06	Reported 0.87798 (probably unit error)
1264	ASTM D4052	878.0		0.11	
1271	ISO3675	877.0		-2.22	
1303		-----		-----	
1304	INH-102	879.3		3.14	
1306	IEC60256	878.2		0.58	
1352		-----		-----	
1361	ISO3675	878.4	U	1.04	Reported 0.8784 (probably unit error)
1367	INH-ISO3675	878.5	U	1.28	Reported 0.8785 (probably unit error)
1375		-----		-----	
1430	ISO3675	877.4		-1.29	
1435	ASTM D4052	878.0	U	0.11	Reported 0.8780 (probably unit error)
1513	ISO12185	878.13		0.41	
1516	ISO3675	877.7		-0.59	
1526	INH-D5002	878.6		1.51	
1529		-----		-----	
1628		-----		-----	
1660	ISO3675	876.0	C,G(0.01)	-4.56	
1702	ISO12185	878.331		0.88	
1719		-----		-----	
1801		-----		-----	
1816		-----		-----	
1826		-----		-----	
1827		-----		-----	
1831		-----		-----	
1863	ASTM D4052	877.4	U	-1.29	Reported 0.8774 (probably unit error)
1923	ISO3675	877.4		-1.29	
1924	ISO3675	877.80		-0.36	
1925	ISO3675	878.0		0.11	
1943	ISO3675	876.8		-2.69	
2125	ISO12185	877.97		0.04	

normality	not OK
n	31
Outliers	1
mean (n)	877.95
st.dev. (n)	0.469
R(calc.)	1.31
R(ISO3675:98)	1.20

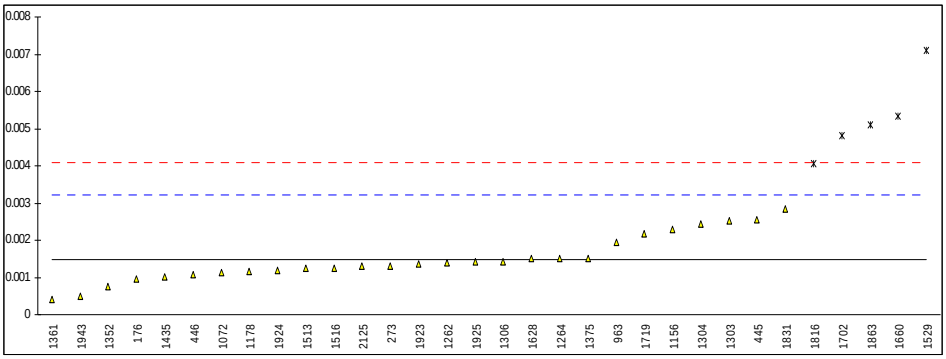


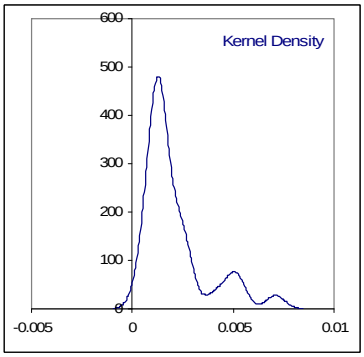


Determination of Di-electric Dissipation Factor on sample #1085;

lab	method	value	mark	z(targ)	Remarks
128		-----		-----	
176	ASTM D924	0.00097		-0.60	
255		-----		-----	
273	IEC60247	0.00130		-0.21	
398		-----		-----	
445	IEC60247	0.00256		1.24	
446	IEC60247	0.001083		-0.47	
497		-----		-----	
614		-----		-----	
963	ASTM D924	0.001935	U	0.52	Reported 0.1935 (probably in %)
1056		-----		-----	
1066		-----		-----	
1072	IEC60247	0.00112		-0.42	
1146		-----		-----	
1152		-----		-----	
1155		-----		-----	
1156	IEC60247	0.00228		0.92	
1178	IEC60247	0.00115		-0.39	
1201		-----		-----	
1262	IEC60247	0.0014		-0.10	
1264	IEC60247	0.00152		0.04	
1271		-----		-----	
1303	IEC60247	0.00253		1.21	
1304	INH-1767	0.00244	U	1.10	Reported 2.44 (probably in %)
1306	IEC60247	0.00143		-0.06	
1352	IEC60247	0.00074		-0.86	
1361	IEC60247	0.0004		-1.25	
1367		-----		-----	
1375	IEC60247	0.00152		0.04	
1430		-----		-----	
1435	IEC60247	0.001		-0.56	
1513	IEC60247	0.00124		-0.28	
1516	IEC60247	0.001245		-0.28	
1526		-----		-----	
1529	IEC60247	0.0071	G(0.05)	6.49	
1628	IEC60247	0.00150		0.02	
1660	IEC60247	0.00534	DG(0.05)	4.45	
1702	IEC60247	0.004809	G(0.05)	3.84	
1719	IEC60247	0.00217		0.79	
1801		-----		-----	
1816	IEC61620	0.00407	G(0.05)	2.99	
1826		-----		-----	
1827		-----		-----	
1831	IEC60247	0.00283		1.55	
1863	IEC60247	0.005110	DG(0.05)	4.19	
1923	IEC60247	0.00135		-0.16	
1924	IEC60247	0.001200		-0.33	
1925	IEC60247	0.00141		-0.09	
1943	IEC60247	0.0005		-1.14	
2125	IEC60247	0.00129		-0.23	

normality	not OK
n	27
outliers	5
mean (n)	0.0015
st.dev. (n)	0.00063
R(calc.)	0.0018
R(IEC60247:78)	0.0056

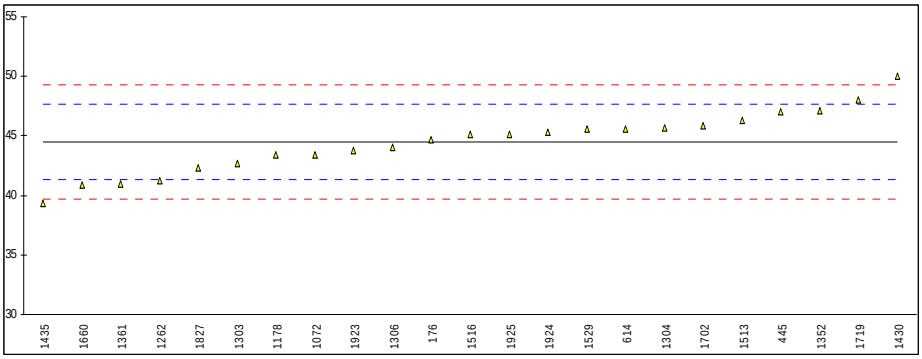


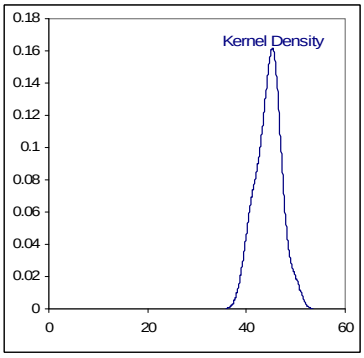


Determination of Interfacial Surface Tension on sample #1085; results in mN/m

lab	method	value	mark	z(targ)	remarks
128		-----		-----	
176	ASTM D971	44.68		0.12	
255		-----		-----	
273		-----		-----	
398		-----		-----	
445	ISO6295	47.0		1.58	
446		-----		-----	
497		-----		-----	
614	ISO6295	45.6		0.70	
963		-----		-----	
1056		-----		-----	
1066		-----		-----	
1072	UNE 21320-6	43.44		-0.66	
1146		-----		-----	
1152		-----		-----	
1155		-----		-----	
1156		-----		-----	
1178	ASTM D971	43.4		-0.69	
1201		-----		-----	
1262	ASTM D971	41.2	C	-2.07	First reported 37.2
1264		-----		-----	
1271		-----		-----	
1303	ASTM D971	42.7		-1.13	
1304	INH-123	45.7		0.76	
1306	ISO6295	44		-0.31	
1352	ASTM D971	47.1		1.64	
1361	ASTM D971	41		-2.20	
1367		-----		-----	
1375		-----		-----	
1430	ISO6295	50		3.47	
1435	ASTM D971	39.3		-3.27	
1513	ASTM D971	46.3		1.14	
1516	ASTM D971	45.1		0.38	
1526		-----		-----	
1529	ISO6295	45.6		0.70	
1628		-----		-----	
1660	ISO6295	40.9		-2.26	
1702	ISO6295	45.811		0.83	
1719	ASTM D2285	48		2.21	
1801		-----		-----	
1816		-----		-----	
1826		-----		-----	
1827	ASTM D971	42.29		-1.39	
1831		-----		-----	
1863		-----		-----	
1923	ISO6295	43.8		-0.44	
1924	ASTM D971	45.3		0.51	
1925	ISO6295	45.1		0.38	
1943		-----		-----	
2125		-----		-----	

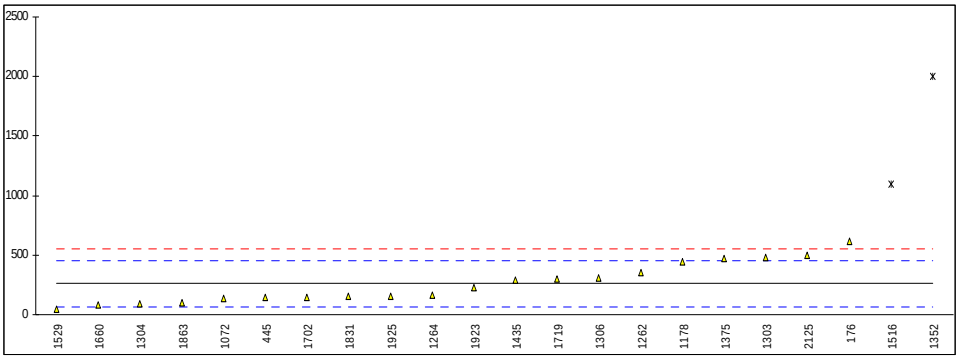
	normality	OK	
	n	23	
	outliers	0	
	mean (n)	44.49	
	st.dev. (n)	2.523	
	R(calc.)	7.06	
	R(ISO6295:83)	4.45	R(ASTM D971:04) = 4.45 (ISO6295 is obsolete since 2005)

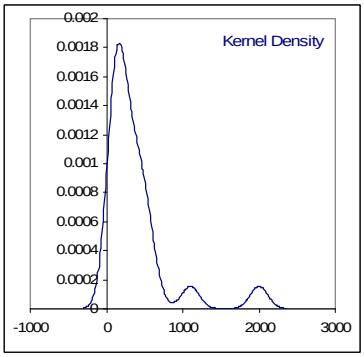




Determination of Specific Resistance on sample #1085; results in GΩm

lab	method	value	mark	z(targ)	remarks
128		-----		-----	
176	ASTM D924	613.35		3.62	
255		-----		-----	
273		-----		-----	
398		-----		-----	
445	IEC60247	147.7		-1.15	
446		-----		-----	
497		-----		-----	
614		-----		-----	
963		-----		-----	
1056		-----		-----	
1066		-----		-----	
1072	IEC60247	132		-1.31	
1146		-----		-----	
1152		-----		-----	
1155		-----		-----	
1156		-----		-----	
1178	IEC60247	448.0		1.93	
1201		-----		-----	
1262	IEC60247	351.6		0.94	
1264	IEC60247	162.5		-1.00	
1271		-----		-----	
1303	IEC60247	479.5		2.25	
1304	INH-1767	92.9		-1.71	
1306	IEC60247	310.4		0.52	
1352	IEC60247	2000	G(0.01)	17.84	
1361		-----		-----	
1367		-----		-----	
1375	IEC60247	466.7		2.12	
1430		-----		-----	
1435	IEC60247	288		0.29	
1513		-----		-----	
1516	IEC60247	1100	G(0.01)	8.61	
1526		-----		-----	
1529	IEC60247	49		-2.16	
1628		-----		-----	
1660	IEC60247	84.3		-1.80	
1702	IEC60247	148.0		-1.15	
1719	IEC60247	295.7		0.37	
1801		-----		-----	
1816		-----		-----	
1826		-----		-----	
1827		-----		-----	
1831	IEC60247	151.2		-1.12	
1863	IEC60247	96.9		-1.67	
1923	IEC60247	227.0		-0.34	
1924		-----		-----	
1925	IEC60247	155.5		-1.07	
1943		-----		-----	
2125	IEC60247	500.55		2.47	
	normality	not OK			
	n	20			
	outliers	2			
	mean (n)	260.04			
	st.dev. (n)	166.507			
	R(calc.)	466.22			
	R(IEC60247:78)	273.04			

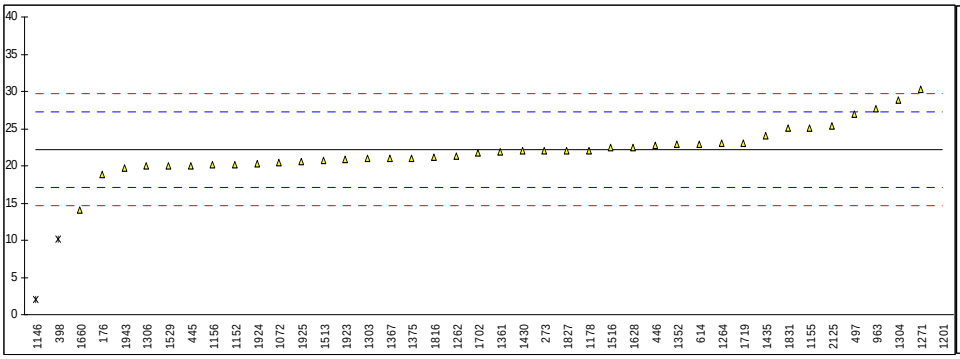


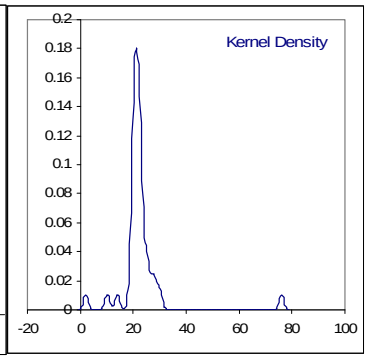


Determination of Water on sample #1085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
128		-----		-----	
176	ASTM D1533	18.78		-1.35	
255		-----		-----	
273	IEC60814	22		-0.08	
398	IEC60814	10.2	C,G(0.05)	-4.75	First reported 70.2
445	IEC60814	20.0		-0.87	
446	IEC60814	22.7		0.20	
497	ASTM D1364	27		1.90	
614	IEC60814	22.9		0.28	
963	ASTM D1533	27.683		2.17	
1056		-----		-----	
1066		-----		-----	
1072	IEC60814	20.5		-0.67	
1146	ASTM D6304	2	G(0.01)	-8.00	
1152	IEC60814	20.14		-0.82	
1155	IEC60814	25.12		1.16	
1156	IEC60814	20.1		-0.83	
1178	IEC60814	22.1		-0.04	
1201	IEC60814	76	G(0.01)	21.32	
1262	IEC60814	21.30		-0.36	
1264	ASTM D1533	23.0		0.32	
1271	ISO12937	30.25	C	3.19	First reported 34
1303	IEC60814	21		-0.47	
1304	IN HOUSE	28.8		2.62	
1306	IEC60814	20		-0.87	
1352	IEC60814	22.9		0.28	
1361	IEC60814	21.8666		-0.13	
1367	IEC60814	21		-0.47	
1375	IEC60814	21		-0.47	
1430	IEC60814	22		-0.08	
1435	IEC60814	24		0.71	
1513	IEC60814	20.79		-0.56	
1516	IEC60814	22.48		0.11	
1526	ASTM D4377	<5000		-----	
1529	IEC60814	20		-0.87	
1628	IEC60814	22.5		0.12	
1660	IEC60814	14		-3.25	
1702	IEC60814	21.73		-0.19	
1719	IEC60814	23.1		0.36	
1801		-----		-----	
1816	IEC60814	21.1		-0.44	
1826		-----		-----	
1827	ASTM D6304	22.1		-0.04	
1831	IEC60814	25.1		1.15	
1863		-----		-----	
1923	IEC60814	20.8		-0.55	
1924	IEC60814	20.25		-0.77	
1925	IEC60814	20.6		-0.63	
1943	IEC60814	19.65		-1.01	
2125	IEC60814	25.39		1.26	

normality	not OK
n	39
outliers	3
mean (n)	22.20
st.dev. (n)	2.917
R(calc.)	8.17
R(IEC60814:97)	7.07



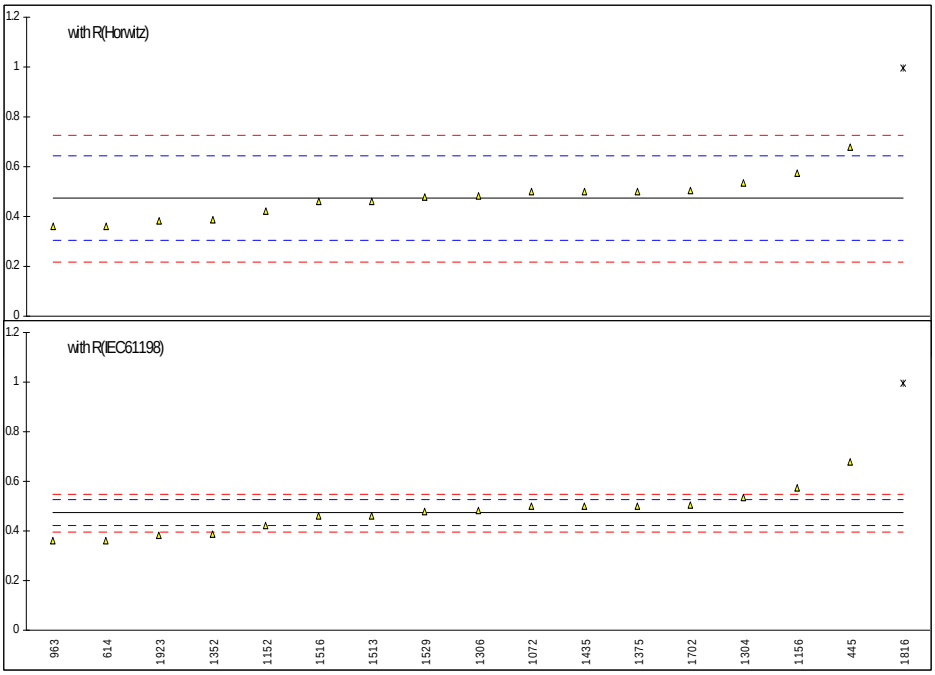


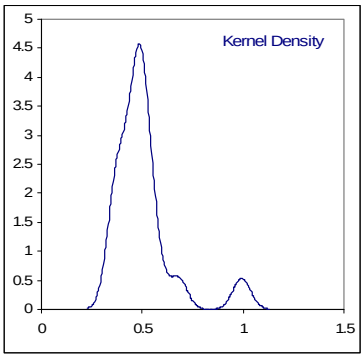
Determination of 2-Furfural on sample #1086FC; results in mg/kg

lab	method	value	mark	z(targ)	$z_{(IEC\ 61198)}$	remarks
445	IEC61198	0.68		2.43	8.12	
614	IEC61198	0.36		-1.34	-4.48	
963	D5837	0.359		-1.35	-4.52	
1072	IEC61198	0.498		0.29	0.96	
1152	IEC61198	0.42		-0.63	-2.12	
1156	IEC61198	0.573		1.17	3.91	
1304	INH-126	0.536		0.73	2.45	
1306	INH-223	0.482241		0.10	0.34	
1352	IEC61198	0.3857		-1.04	-3.47	
1375	IEC61198	0.50		0.31	1.04	
1430		-----		-----	-----	
1435	IEC61198	0.50		0.31	1.04	
1513	IEC61198	0.4595		-0.17	-0.56	
1516	IEC61198	0.459		-0.17	-0.58	
1529	IEC61198	0.48		0.07	0.25	
1660		-----		-----	-----	
1702	IEC61198	0.502896		0.34	1.15	
1801		-----		-----	-----	
1816	IEC61198	0.994	G(0.01)	6.13	20.49	
1923	IEC61198	0.384		-1.06	-3.53	

normality	OK
n	16
outliers	1
mean (n)	0.474
st.dev. (n)	0.0834
R(calc.)	0.234
R(Horwitz)	0.237

compare R(IEC61198) = 0.071





Determination of other Furanic compounds on sample #1086FC; 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)
445	IEC61198	<0.05	-----	<0.05	-----	<0.05	-----	***0.23***	-----
614	IEC61198	<0.01	-----	<0.01	-----	0.01	-----	<0.01	-----
963	D5837	nd	-----	nd	-----	nd	-----	nd	-----
1072	IEC61198	0.029	-----	***0.088***	-----	<0.001	-----	0.041	-----
1152	IEC61198	0.04	-----	***0.78***	-----	<0.01	-----	<0.01	-----
1156	IEC61198	0.00	-----	0.00	-----	0.00	-----	0.00	-----
1304	INH-126	<0.01	-----	<0.01	-----	<0.01	-----	0.014	-----
1306	INH-223	0	-----	0	-----	0	-----	0	-----
1352	IEC61198	nd	-----	nd	-----	0.0137	-----	0.0168	-----
1375	IEC61198	<0.03	-----	***0.16***	-----	<0.03	-----	<0.03	-----
1430		-----	-----	-----	-----	-----	-----	-----	-----
1435	IEC61198	<0.03	-----	<0.03	-----	<0.03	-----	0.04	-----
1513	IEC61198	<0.05	-----	<0.05	-----	<0.05	-----	<0.05	-----
1516	IEC61198	<0.05	-----	<0.05	-----	<0.05	-----	<0.05	-----
1529	IEC61198	<0.1	-----	<0.1	-----	<0.1	-----	<0.1	-----
1660		-----	-----	-----	-----	-----	-----	-----	-----
1702	IEC61198	nd	-----	nd	-----	nd	-----	nd	-----
1801		-----	-----	-----	-----	-----	-----	-----	-----
1816	IEC61198	0.045	-----	***0.366***	-----	0.00	-----	0.022	-----
1923	IEC61198	<0.01	-----	<0.01	-----	<0.01	-----	***0.176***	-----
	normality	n.a.		n.a.		n.a.		OK	
	n	5		2		5		7	
	outliers	n.a.		n.a.		n.a.		n.a.	
	mean (n)	0.0228		0.029		0.005		0.019	
	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

Number of participants per country

4 laboratories in	AUSTRALIA
2 laboratories in	BELGIUM
1 laboratory in	BOSNIA and HERZEGOVINA
5 laboratories in	BULGARIA
1 laboratory in	CROATIA
1 laboratory in	ESTONIA
1 laboratory in	FRANCE
1 laboratory in	GERMANY
2 laboratories in	ITALY
1 laboratory in	LATVIA
3 laboratories in	MALAYSIA
1 laboratory in	NEW ZEALAND
1 laboratory in	NORWAY
2 laboratories in	PORTUGAL
1 laboratory in	SAUDI ARABIA
1 laboratory in	SERBIA
1 laboratory in	SINGAPORE
2 laboratories in	SOUTH AFRICA
7 laboratories in	SPAIN
1 laboratory in	TANZANIA
5 laboratories in	THE NETHERLANDS
1 laboratory in	U.A.E.
2 laboratories 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

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