Results of Proficiency Test Transformer Oil (used) November 2020

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

Author: ing. G.A. Oosterlaken-Buijs

Correctors: ing. A.S. Noordman-de Neef & ing. R.J. Starink

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CONTENTS

1	INTRODUCTION	3
2	SET UP	3
2.1	ACCREDITATION	3
2.2	PROTOCOL	3
2.3	CONFIDENTIALITY STATEMENT	4
2.4	SAMPLES	4
2.5	STABILITY OF THE SAMPLES	5
2.6	ANALYZES	6
3	RESULTS	6
3.1	STATISTICS	7
3.2	GRAPHICS	7
3.3	Z-SCORES	8
4	EVALUATION	8
4.1	EVALUATION PER SAMPLE AND PER TEST	9
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	11
4.3	COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2020 WITH PREVIOUS PTS	12

Appendices:

1.	Data, statistical and graphic results	13
2.	Other reported test results	37
3.	Number of participants per country	38
4.	Abbreviations and literature	39

1 Introduction

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Transformer Oil (used) and a proficiency scheme for Furanics in Transformer Oil (used), both in accordance with the latest version of IEC60296 and ASTM D3487, every year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of Transformer Oil (used) and the round robin for the analysis of Furanics in Transformer Oil (used).

In this interlaboratory study 73 laboratories in 36 different countries registered for participation for the PT on Transformer Oil (used) and 48 laboratories in 27 different countries registered for participation for the PT on Furanics in Transformer Oil (used). In total 74 laboratories in 36 different countries registered for participation. See appendix 3 for the number of participants per PT and per country.

In this report the results of proficiency tests on Transformer Oil (used) and Furanics in Transformer Oil (used) are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send for the PT on Transformer Oil (used) 1 bottle of 1L labelled #20226 and for the PT on Furanics in Transformer Oil (used) 1 bottle of 100mL labelled #20227. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

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

For the preparation of the sample for the regular PT Transformer Oil (used) a batch of approximately 110 liters of Transformer Oil (used) was obtained from a third party. After homogenization 100 amber glass bottles of 1L were filled and labelled #20226. The homogeneity of the subsamples was checked by determination of Density at 20 °C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Density at 20°C in kg/m³
Sample #20226-1	874.50
Sample #20226-2	874.55
Sample #20226-3	874.54
Sample #20226-4	874.56
Sample #20226-5	874.56
Sample #20226-6	874.56
Sample #20226-7	874.56
Sample #20226-8	874.55

Table 1: homogeneity test results of subsamples #20226

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 20°C in kg/m³
r (observed)	0.06
reference test method	ISO3675:98
0.3 x R (reference test method)	0.36

Table 2: evaluation of the repeatability of subsamples #20226

The calculated repeatability was less than 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

For the preparation of the sample for the PT Furanics in Transformer Oil (used) a batch of approximately 400mL of Transformer Oil, highly positive on Furanics was mixed with approximately 7L of Transformer Oil (fresh). After homogenization 78 amber glass bottles of 100mL were filled and labelled #20227.

The homogeneity of the subsamples was checked by determination of Density at 20 °C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Density at 20°C in kg/m³
Sample #20227-1	872.43
Sample #20227-2	872.41
Sample #20227-3	872.44
Sample #20227-4	872.43
Sample #20227-5	872.43
Sample #20227-6	872.41
Sample #20227-7	872.43
Sample #20227-8	872.44

Table 3: homogeneity test results of subsamples #20227

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 20°C in kg/m³
r (observed)	0.03
reference test method	ISO3675:98
0.3 x R (reference test method)	0.36

Table 4: evaluation of the repeatability of subsamples #20227

The calculated repeatability was less than 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

Depending on the registration of the participants the appropriate set of PT samples was sent on October 28, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

The participants were requested to determine on sample #20226: Total Acidity (Potentiometric and Colorimetric), Breakdown Voltage, Density at 20°C, Di-electric Dissipation Factor (DDF) at 90°C and Specific Resistance at 90°C, Flash Point (C.O.C. and PMcc), Interfacial Surface Tension, Kinematic Viscosity at 40°C and Water. Also, some analytical details were asked for the Total Acidity (Potentiometric) and for the Breakdown Voltage determination.

The participants were requested to determine on sample #20227: Total Furanic Compounds, 2-Acetyl Furan, 2-Furfural, 2-Furfurylalcohol, 5-Hydroxy Methyl-2-Furfural and 5-Methyl-2-Furfural.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and 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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

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

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. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former its proficiency tests.

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 according to:

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

The $z_{\text{(target)}}$ scores are listed in the test 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**

In this proficiency test no major problems were encountered with the dispatch of the samples. For the PT on Transformer Oil (used) four participants did not report any test result at all and none of the reporting participants reported test results after the final reporting date. For the PT on Furanics in Transformer Oil (used) three participants reported the test results after the final reporting date and four participants did not report any test results at all. Not all laboratories were able to report all analyses requested.

In total 70 participants reported 581 numerical test results. Observed were 24 outlying test results, which is 4.1% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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.

4.1 EVALUATION PER SAMPLE AND PER TEST

In this section the reported test results are discussed per sample and per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are explained in appendix 4.

For the Furanics the observed reproducibilities were compared against the (strict) estimated reproducibilities calculated with the Horwitz equation. It is remarkable that the precision requirements of IEC61198:93 are stricter than the estimated reproducibilities calculated with the Horwitz equation.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D1298) and an added designation for the year that the test method was adopted or revised (e.g. D1298:12b). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1298:12b(2017)). In the results tables of appendix 1 only the method number and the year of adoption or revision (e.g. D1298:12b) will be used.

Sample #20226

Total Acidity (Potentiometric): 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 ASTM D664-A:18e2 (BEP- 60mL/125mL and IP- 60mL/ 125mL) nor with the requirements of EN62021-1:03.

<u>Total Acidity (Colorimetric)</u>: This determination was problematic depending on the test method used. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D974:14e2, but not at all in agreement with the very strict requirements of EN62021-2:07.

Breakdown Voltage: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN60156:95. There was no significant difference between the test results with stirring and the test results without stirring.

The reproducibility of EN60156:95 was determined from Figure 3 of method EN60156:95, according to the iis memo 1702 (see lit. 18).

<u>Density at 20°C</u>: This determination may be problematic for a number of laboratories. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO3675:98 and D1298:12b(2017), but is not in agreement with the requirements of ASTM D4052:18a and ISO12185:96.

<u>DD-Factor</u>: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN60247:04.

- <u>Specific Resistance</u>: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN60247:04.
- <u>Flash Point C.O.C.</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D92:18.
- Flash Point PMcc: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ISO2719-A:16, ASTM D93-A:20 and ASTM D93-B:20. The calculated reproducibility is in agreement with the requirements of ISO2719-B:16.
- Interfacial Surface Tension: This determination was problematic depending on the test method used. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D971:20, but is in agreement with the requirements of IEC62961:18.
- Kinematic Viscosity: Till 2017 a precision statement for used oils was not present in ASTM D445. In the 2019 version of ASTM D445 a precision statement is given for used (in-service) formulated oils. However, it appears that this reproducibility is very strict. Therefore, iis has estimated a reproducibility on used oils for Kinematic Viscosity at 40°C based from previous PTs (see iis memo 1401, lit. 17). This reproducibility has been used for the evaluations. This determination may be problematic for a number of laboratories. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of iis memo 1401 and ASTM D7042:16e3, but is not in agreement with ISO3104-A:20 nor with the strict requirements of ASTM D445:19a.

Water:

This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of EN60814:98 and is in agreement with the requirements of ASTM D1533:12 and ASTM D6304-C:20.

Sample #20227

<u>Total Furanic Compounds</u>: 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 estimated reproducibility calculated with the Horwitz equation, but is not in agreement with the strict requirements of IEC61198:93.

2-Furfural:

This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility calculated with the Horwitz equation, but is not in agreement with the strict requirements of IEC61198:93.

The majority of the participants agreed on a concentration near or below the limit of detection for all other Furanic compounds mentioned in paragraph 2.6. Therefore, no z-scores were calculated for these elements. The test results of these components are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method or as declared by the estimated target reproducibility calculated with the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference test methods (in casu ASTM standards) or based on previous proficiency tests or estimated using the Horwitz equation are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acidity (Potentiometric)	g KOH/kg	39	0.024	0.025	0.015
Total Acidity (Colorimetric)	g KOH/kg	30	0.021	0.020	0.04
Breakdown Voltage	kV/2.5 mm	65	22.4	14.7	11.5
Density at 20°C	kg/m³	48	874.6	0.7	1.2
Di-electric Dissipation Factor at 90°C		54	0.012	0.006	0.013
Specific Resistance at 90°C	GΩm	44	27.0	11.1	28.3
Flash Point C.O.C.	°C	21	157.6	18.9	18
Flash Point PMcc	°C	35	148.8	9.7	10.6
Interfacial Surface Tension	mN/m	52	27.1	6.0	2.7
Kinematic Viscosity at 40°C	mm²/s	37	11.136	0.162	0.201
Water	mg/kg	62	28.60	7.97	8.02

Table 5: reproducibilities of tests on sample #20226

Component	unit	n	average	2.8 * sd	R(target)
Total Furanic Compounds	mg/kg	29	5.46	1.77	1.89
2-Furfural	mg/kg	41	5.50	2.05	1.91

Table 6: reproducibilities of tests on sample #20227

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2020 WITH PREVIOUS PTS

	November 2020	November 2019	November 2018	November 2017	November 2016
Number of reporting laboratories	70	70	75	74	76
Number of test results	581	678	805	667	660
Number of statistical outliers	24	24	41	35	31
Percentage of statistical outliers	4.1%	3.5%	5.1%	5.2%	4.7%

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 reference test methods. The conclusions are given the following table.

Parameter	November 2020	November 2019	November 2018	November 2017	November 2016
Total Acidity (Potentiometric)	-	-			-
Total Acidity (Colorimetric)	++	+	+/-	++	++
Breakdown Voltage	-	-		+/-	+/-
Density at 20°C	+	-	+/-	+	+/-
Di-electric Dissipation Factor at 90°C	++	+	++	++	++
Specific Resistance at 90°C	++	+	+	++	++
Flash Point C.O.C.	+/-	+	+	++	+/-
Flash Point PMcc	+/-	+	+	++	+
Interfacial Surface Tension					
Kinematic Viscosity at 40°C	+	-	-		+
Water	+/-			-	+/-
Total Furanic Compunds	+/-	++	++	++	++
2-Acetyl Furan	n.e.	n.e.	+	n.e.	n.e.
2-Furfural	+/-	++	+	++	+/-
2-Furfurylalcohol	n.e.	-	+/-	n.e.	+/-
5-Hydroxymethyl-2-Furfural	n.e.	n.e.	+	n.e.	n.e.
5-Methyl-2-Furfural	n.e.	+	+	n.e.	+

Table 8: comparison determinations against the reference test methods

The following performance categories were used:

++ : group performed much better than the reference test method

+ : group performed better than the reference test method

+/- : group performance equals the reference test method

- : group performed worse than the reference test method

-- : group performed much worse than the reference test method

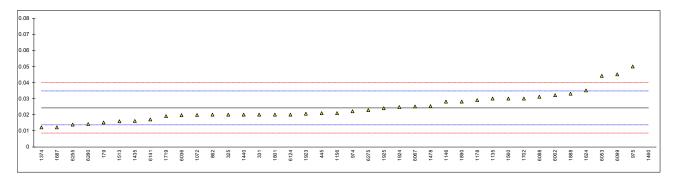
n.e. : not evaluated

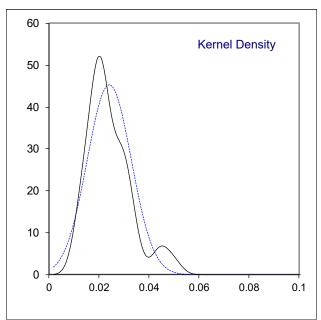
APPENDIX 1

Determination of Total Acidity (Potentiometric) on sample #20226; results in g KOH/kg

	•		_		1 sample #20226; res	•	**
lab	method	value	mark	z(targ)	End Point	Vol. solv	r. remarks
179	D664-A	0.015	С	-1.76	Inflection Point	60 mL	first reported <0.01
237							
	DCC4 A				Duffer Fiel Deint (all 44)		
325	D664-A	0.02		-0.81	Buffer End Point (pH 11)	125 mL	
331	D664Mod.	0.02		-0.81			
398							
445	EN62021-1	0.021		-0.62		60 mL	
511	LINOZOZ 1-1						
614							
657	D664-A	< 0.1			Inflection Point	60 mL	
862	D664-A	0.02		-0.81			
902							
912							
913							
963							
974	D664-A	0.022		-0.43	Inflection Point	125 mL	
975	D664-A	0.05		4.91	Inflection Point	60 mL	
1072		0.0197	,	-0.86	Inflection Point	60 mL	
1135	D664-A					125 mL	
	D004-A	0.03		1.10	Inflection Point		
1137							
1146	D664-A	0.028		0.72	Buffer End Point (pH 11)	125 mL	
1156	EN62021-1	0.021		-0.62			
1178	EN62021-1	0.029		0.91			
1264							
					Puffer End Daint (all 10)		
1304			_		Buffer End Point (pH 10)	125 mL	_ , ,
1306			С				see Total Acidity (Colorim.)
1352							
1367							
1374	D664-A	0.012		-2.33	Buffer End Point (pH 10)	60 mL	
	D004-74				" ,		
1430	.=						
1435	IEC62021-1	0.016		-1.57			
1440	IEC62021-1	0.02		-0.81			
1442							
1444							
1458							
	D004 A		D(0.04)		Doffee Food Defeat (call 44)		
1460	D664-A	0.294	R(0.01)		Buffer End Point (pH 11)	60 mL	
1478	IEC62021-1	0.0252		0.18	Buffer End Point (pH 11)		
1505							
1513	IEC62021-1	0.0158	1	-1.61			
1545							
	IECC0004 4						
1560	IEC62021-1	0.03	_	1.10			
1624	IEC62021-1	0.035	С	2.05	Buffer End Point (pH 11)	60 mL	first reported 0.075
1660	IEC62021-1	< 0,01			Buffer End Point (pH 11)	60 mL	
1687	D664-A	0.012		-2.33	Buffer End Point (pH 10)	60 mL	
1702	IEC62021-1	0.030		1.10			
1719				-1.00		60 mL	
	D664-A	0.019			Inflection Point		
1743	IEC62021-1	<0.02			Buffer End Point (pH 11)	60 mL	
1801	EN62021-1	0.020		-0.81	Buffer End Point (pH 11)	125 mL	
1885							
1888	EN62021-1	0.033		1.67	Buffer End Point (pH 11)	60 mL	
1890	ISO6619	0.028		0.72			
1891	1000013	0.020					
	EN100004 4				Inflection Point	60 mL	
1923	EN62021-1	0.0206		-0.69	Inflection Point	60 mL	
1924	EN62021-1	0.0246	i	0.07	Buffer End Point (pH 11)	60 mL	
1925	EN62021-1	0.024		-0.04	Buffer End Point (pH 11)	60 mL	
6002	D664-A	0.032		1.48	Inflection Point "	60 mL	
6015	200.71						
6031							
6036	IEC62021-1	0.0195	i	-0.90			
6053	IEC62021-1	0.044		3.77	Buffer End Point (pH 10)	60 mL	
6067	IEC62021-1	0.025		0.15	Inflection Point	125 mL	
6085							
	IEC62024 4						
6088	IEC62021-1	0.0310	•	1.29		60 mL	
6099	IEC62021-1	0.045		3.96			
6124	IEC62021-1	0.02		-0.81		60 mL	
6140							
6141	D664-A	0.017		-1.38	Buffer End Point (pH 10)	60 mL	
6155	200171	0.017			" ,		
6167			-				
6255	IEC62021-1	0.0137		-2.01	Buffer End Point (pH 11)	60 mL	
6275	IEC62021-1	0.0228	}	-0.27	Buffer End Point (pH 11.5)	100 mL	
6278						60 mL	
6280	IEC62021-1	0.0141	С	-1.93			first reported 0.5569
6334	1_002021-1		9				31 10001104 0.0000
0554							

		only D664	only EN/IEC 62021-1
normality	not OK	not OK	OK
n	39	13	24
outliers	1	1	0
mean (n)	0.0242	0.0228	0.0250
st.dev. (n)	0.00880	0.01029	0.00835
R(calc.)	0.0247	0.0288	0.0234
st.dev.(D664-A:18e2, BEP 60mL)	0.00525	0.00495	
R(D664-A:18e2, BEP 60mL)	0.0147	0.0139	
compare			
R(EN62021-1:03)	0.0085		0.0088
R(D664-A:18e2, IP 125mL)	0.0045	0.0059	
R(D664-A:18e2, IP 60mL)	0.0190	0.0181	
R(D664-A:18e2, BEP 125mL)	0.0063	0.0059	

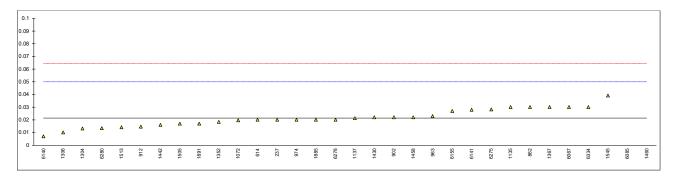


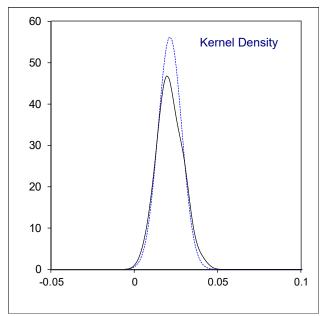


Determination of Total Acidity (Colorimetric) on sample #20226; results in g KOH/kg

lab	method	value	mark	z(targ)	remarks
179	D974	<0.02			
237	D974	0.020		-0.10	
325 331					
398					
445					
511					
614	D974	0.02		-0.10	
657 862	D974 D974	< 0.02		0.60	
902	D974 D974	0.03 0.022		0.00	
912	D974	0.0146		-0.48	
913					
963	D974	0.023		0.11	
974 975	D974	0.020		-0.10 	
1072	IEC62021-2	0.0196		-0.13	
1135	D974	0.03		0.60	
1137	D1534	0.0212		-0.01	
1146 1156					
1178					
1264					
1304	In house	0.013		-0.59	
1306	D974	0.010131	С	-0.79	reported as Total Acidity (Potentiometric)
1352 1367	IEC62021-2 IP139	0.0183 0.03		-0.22 0.60	
1374	IF 139	0.03			
1430	EN62021-2	0.022		0.04	
1435					
1440	IEC62024 2	0.016		0.20	
1442 1444	IEC62021-2	0.016		-0.38	
1458	D974	0.022		0.04	
1460	D974	0.405	R(0.01)	26.85	
1478	D074	0.017		0.24	
1505 1513	D974 IEC62021-2	0.017 0.0141		-0.31 -0.51	
1545	D974	0.039		1.23	
1560					
1624					
1660 1687					
1702					
1719					
1743	ISO6618	<0.04			
1801	D974	0.020		-0.10	
1885 1888	ם פופע	0.020		-0.10	
1890					
1891	IEC62021-2	0.017		-0.31	
1923 1924					
1924					
6002					
6015					
6031					
6036 6053					
6067	IEC62021-2	0.03		0.60	
6085	D974	0.1241	R(0.01)	7.19	
6088					
6099 6124					
6140	D974	0.007		-1.01	
6141	D974	0.028		0.46	
6155	D974	0.027		0.39	
6167 6255					
6275	D974	0.0280675		0.47	
6278	D974	0.020		-0.10	
6280	IEC62021-2	0.0133		-0.57	
6334	IEC62021-2	0.03		0.60	

		only ASTM D974/ISO6618/IP139	Only EN/IEC 62021-2
normality	OK	OK	OK
n	30	19	9
outliers	2	2	0
mean (n)	0.0214	0.0225	0.0200
st.dev. (n)	0.00710	0.00760	0.00624
R(calc.)	0.0199	0.0213	0.0175
st.dev.(D974:14e2)	0.01429	0.0143	
R(D974:14e2)	0.04	0.04	
compare			
R(EN62021-2:07)	0.0043		0.0040

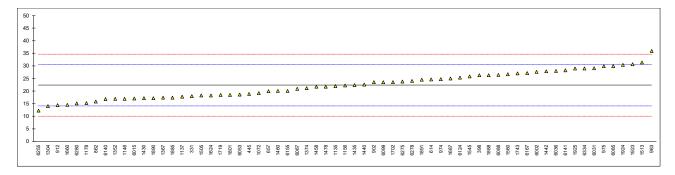


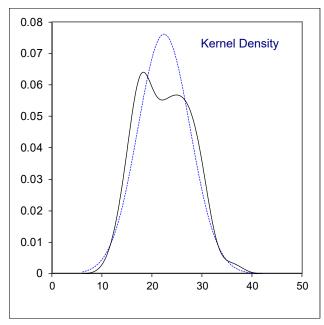


Determination of Breakdown Voltage on sample #20226, results in kV/2.5 mm

lab	method	value	mark	z(targ)	stirred	remarks
179						
237						
325	=1100.1=0					
331	EN60156	18		-1.07	 V	
398 445	EN60156 IEC60156	26.3 18.9		0.95 -0.85	Yes Yes	
511	12000130	10.9		-0.03		
614	IEC60156	24.6		0.54	Yes	
657	EN60156	20.0		-0.58	No	
862	IEC60156	15.9		-1.58		
902	EN60156	23.5	_	0.27		
912	IEC60156	14.4	С	-1.94		reported 43.4 kV, first reported 36
913 963	EN60156	36.0		3.31		
974	EN60156	24.8		0.59	Yes	
975	IEC60156	29.9		1.83	Yes	
1072	EN60156	19.2		-0.77	Yes	
1135	IEC60156	22		-0.09	No	
1137	IEC60156	17.8		-1.11	No	
1146 1156	IEC60156	17 22.2		-1.31	No No	
1178	EN60156 EN60156	15.2		-0.04 -1.75	No Yes	
1264	L1400130			-1.75		
1304	IEC60156	14.0		-2.04	Yes	
1306						
1352	IEC60156	17.0		-1.31	No	
1367	EN60156	17.4		-1.21	 \/	
1374 1430	IEC60156 EN60156	21.2 17.2		-0.29 -1.26	Yes	
1435	IEC60156	22.3		-0.02		
1440	IEC60156	22.55		0.02	Yes	
1442	IEC60156	27.9		1.34	Yes	
1444						
1458	IEC60156	21.7		-0.17	Yes	
1460	IEC60156	20.1		-0.56	Yes	
1478 1505	IEC60156 IEC60156	21.7 18.3		-0.17 -0.99	Yes Yes	
1513	IEC60156	31.4		2.19	Yes	
1545	IEC60156	25.9		0.86		
1560	IEC60156	26.7		1.05	Yes	
1624	IEC60156	18.3		-0.99	Yes	
1660	IEC60156	14.5		-1.92	Yes	
1687 1702	EN60156 IEC60156	25.0 23.6		0.64 0.30	No Yes	
1719	IEC60156	18.5		-0.94	Yes	
1743	IEC60156	27		1.12	No	
1801	EN60156	18.5		-0.94	Yes	
1885	IEC60156	17.4	С	-1.21	No	reported 36 kV
1888	EN60156	26.3		0.95	Yes	
1890 1891	IEC60156 IEC60156	17.2 24.5		-1.26 0.52	Yes Yes	
1923	EN60156	30.6		2.00	No	
1924	EN60156	30.4		1.95	No	
1925	EN60156	29.0		1.61	No	
6002	EN60156	27.6		1.27	Yes	
6015	EN60156	17.10		-1.28	Yes	
6031 6036	IEC60156 IEC60156	29.1 28.05		1.63 1.38	No No	
6053	IEC60156	18.6		-0.92	Yes	
6067	IEC60156	20.9		-0.36	Yes	
6085	IEC60156	29.9		1.83	Yes	
6088	IEC60156	26.5		1.00	Yes	
6099	IEC60156	23.5		0.27	No	
6124 6140	IEC60156	25.35 16.8		0.72 -1.36	Yes	
6141	EN60156 IEC60156	16.8 28.2		-1.36 1.41	 Yes	
6155	IEC60156	20.2		-0.56	Yes	
6167	IEC60156	27.2		1.17	Yes	
6255	IEC60156	12.2		-2.48	Yes	
6275	IEC60156	23.8		0.34	Yes	
6278 6280	IEC60156 IEC60156	24 15.1		0.39 -1.77	No Yes	
6334	IEC60156	29		1.61	Yes	
550 -1					,	

		Only 'stirred'	Only 'not stirred'
normality	OK	OK	OK
n	65	39	16
outliers	0	0	0
mean (n)	22.382	22.241	23.753
st.dev. (n)	5.2419	5.0168	4.9291
R(calc.)	14.677	14.047	13.802
st.dev.(EN60156:95)	4.1119	4.0859	4.3637
R(EN60156:95)	11.513	11.441	12.218

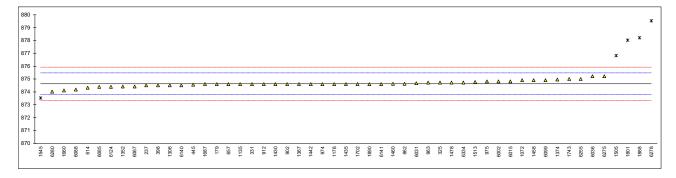


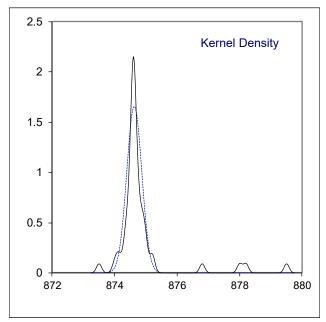


Determination of Density at 20°C on sample #20226; results in kg/m³

lab	method	value	mark	z(targ)	remarks
179	D4052	874.6		-0.07	
237	D4052 D4052	874.5		-0.30	
325	D4052 D4052	874.7		0.30	
331	ISO12185	874.7 874.6		-0.07	
398	ISO12185	874.5		-0.07	
396 445	D4052	674.5 874.55		-0.30 -0.18	
511	D7002	0/4.55		-0.16	
614	D4052	874.3		-0.77	
657	D4052 D4052	674.3 874.6		-0.77 -0.07	
862	D4052 D4052	874.62		-0.07	
902					
	D4052 ISO3675	874.6		-0.07	
912	1503075	874.6		-0.07	
913	D4052	 874.7		0.17	
963 974	D4052 D4052			0.17	
975	D4052 D4052	874.6	C	-0.07 0.40	first reported 0.9749 kg/m ³
1072	ISO3675	874.8 874.9	С	0.40	first reported 0.8748 kg/m ³
1135		874.9 874.6	С	-0.03	first reported 0.9746 kg/m ³
1137	ISO12185	074.0	C		first reported 0.8746 kg/m ³
1137					
1156					
1178	ISO12195	874 G		-0.07	
1264	ISO12185	874.6		-0.07	
1304					
	D4052			0.30	
1306 1352	D4052	874.5 874.4		-0.30 -0.53	
1352 1367	D7042	874.4 874.6	C	-0.53 -0.07	first reported 876.2
	ISO12185 D4052	874.6 874.05	C C	-0.07 0.75	,
1374 1430	D4052 D4052	874.95 874.6	U	-0.75	first reported 0.87495 kg/m ³
	D4052 D4052	874.6		-0.07 -0.07	
1435	D4032			-0.07	
1440	ISO1218E	 074 G			
1442	ISO12185	874.6		-0.07	
1444 1458	D4052	874.9		0.63	
1460 1478	D4052 ISO12185	874.61 874.7		-0.04 0.17	
			D(0.04)		
1505	D7042	876.8 974.764	R(0.01)	5.07	
1513 1545	ISO12185	874.764 873.5	C D(0.04)	0.32	first reported 0.8735 kg/m ³
1545 1560	ISO3675	873.5	C,R(0.01)	-2.63	first reported 0.8735 kg/m ³
1560 1624					
1624	D7042	Q7/1 1		1 22	
1660 1687	D7042	874.1 874.58		-1.23	
1687	ISO12185	874.58 874.6		-0.11 0.07	
1702	ISO3675	874.6		-0.07	
1719 1743	In house	975 O		0.87	
1743	In house	875.0 878.0	D(0.01)	0.87	
1801	ISO3675	878.0	R(0.01)	7.87	
1885	1002675	070 2	C B/0 04\	0 22	first reported 960 0
1888	ISO3675	878.2 874.6	C,R(0.01)	8.33	first reported 868.8
1890	ISO12185	874.6		-0.07	
1891					
1923					
1924					
1925	10012105	074.0	<u> </u>	0.40	first reported 977
6002	ISO12185	874.8	C	0.40	first reported 864.10
6015	ISO12185	874.80 974.67	С	0.40	first reported 864.10
6031	D1298	874.67		0.10	
6036	ISO3675	875.2		1.33	
6053	ISO1210E	97 <i>1</i> /	C	0.53	first reported 976.2
6067	ISO12185	874.4 874.37	С	-0.53	first reported 876.2
6085	D7042	874.37 874.16	C	-0.60 1.00	first reported 976.07
6088	ISO3675	874.16	С	-1.09	first reported 876.07
6099	ISO12185	874.9 874.37		0.63	
6124	D7042	874.37 874.5		-0.60	
6140	ISO3675	874.5		-0.30	
6141	D4052	874.6		-0.07	
6155					
6167	1002675	075	0	0.07	first reported 072
6255	ISO3675	875 875 24	С	0.87	first reported 873
6275	D1298	875.21	D(0.04)	1.36	
6278	D1298	879.5	R(0.01)	11.37	
6280	ISO12185	874	0	-1.47	first reported 977.0
6334	ISO12185	874.7	С	0.17	first reported 877.8

suspect
48
5
874.628
0.2406
0.674
0.4286
1.2
0.5
0.5
1.2

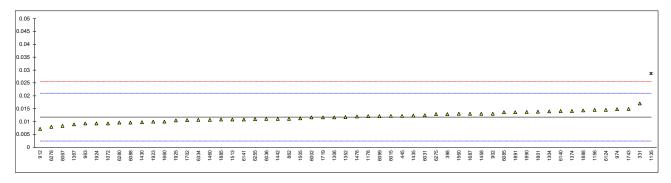


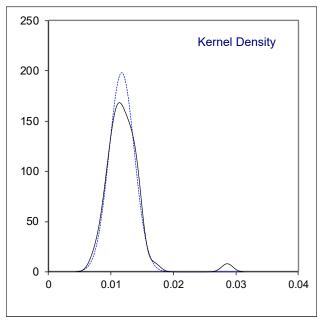


Determination of Di-electric Dissipation Factor (DDF) at 90°C on sample #20226

lah	method	value	mark	7/toral	fraguency	remarks
lab 179	method	value	mark	z(targ)	frequency	I GIII di KS
237						
325						
331	D924	0.01695		1.13	50	
398	EN60247	0.012853		0.25	 EE	
445 511	IEC60247	0.0121635		0.10	55	
614						
657						
862	IEC60247	0.0110		-0.15	50	
902	EN60247	0.013		0.28	50	
912 913	IEC60247	0.0071		-0.99		
963	IEC60247	0.0092		-0.54	60	
974	EN60247	0.0147		0.65	50	
975						
1072	EN60247	0.00924	D(0.04)	-0.53	50	
1135 1137	IEC60247	0.0286	R(0.01)	3.64	50	
1146						
1156	EN60247	0.01445		0.59		
1178	EN60247	0.01199		0.06	50	
1264	IEC60247	0.012026		0.46	 50	
1304 1306	IEC60247 IEC60247	0.013836 0.011556		0.46 -0.03	50	
1352	IEC60247	0.01171		0.00		
1367	EN60247	0.008796		-0.62	50	
1374	IEC60247	0.014		0.50		
1430	EN60247	0.009616		-0.45		
1435 1440	IEC60247	0.012253		0.12		
1442	IEC60247	0.010939		-0.16		
1444						
1458	IEC60247	0.012962		0.27		
1460	IEC60247	0.010582		-0.24	60	
1478 1505	IEC60247 IEC60247	0.011885 0.011240		0.04 -0.10		
1513	IEC60247	0.011240		-0.10		
1545						
1560	IEC60247	0.01290		0.26		
1624	15000047					
1660 1687	IEC60247 EN60247	0.009896 0.01294		-0.39 0.27	50 50	
1702	IEC60247	0.01254		-0.26		
1719	IEC60247	0.01152		-0.04		
1743	IEC60247	0.01480		0.67	50	
1801	EN60247	0.01372		0.44		
1885 1888	IEC60247 EN60247	0.010679 0.014270		-0.22 0.55		
1890	IEC60247	0.014270		0.55	50	
1891	IEC60247	0.0135		0.39		
1923	EN60247	0.00987		-0.39		
1924	EN60247	0.009215		-0.53		
1925	EN60247	0.01038 0.0115		-0.28	50	
6002 6015	EN60247 EN60247	0.0115 0.0121535		-0.04 0.10	50 50	
6031	IEC60247	0.012354		0.14	50	
6036	IEC60247	0.010906		-0.17	50	
6053	150000.47					
6067	IEC60247	0.0081955		-0.75	50.00	
6085 6088	IEC60247 IEC60247	0.013490 0.0095		0.39 -0.47	50	
6099	IEC60247	0.012020		0.07		
6124	EN60247	0.014484		0.60	50	
6140	EN60247	0.013966		0.49		
6141	IEC60247	0.010758		-0.20	60	
6155 6167						
6255	IEC60247	0.0108		-0.19		
6275	IEC60247	0.0127885		0.24	55	
6278	IEC60247	0.00786		-0.82		
6280	IEC60247	0.009488		-0.47	50	
6334	IEC60247	0.01056		-0.24		

normality	OK
n	54
outliers	1
mean (n)	0.011694
st.dev. (n)	0.0020142
R(calc.)	0.005640
st.dev.(EN60247:04)	0.0046473
R(EN60247:04)	0.013013

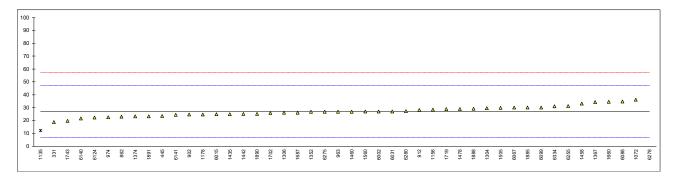


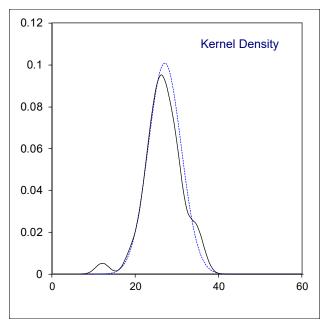


Determination of Specific Resistance at 90°C on sample #20226; results in $G\Omega m$

lab	method	value	mark	z(targ)	remarks
179	mourou		arı		Tomatic
237					
325 331	D924	18.66		-0.82	
398	D924	10.00		-0.02	
445	IEC60247	23.415		-0.35	
511					
614					
657 862	IEC60247	22.82		-0.41	
902	EN60247	24.5		-0.41	
912	IEC60247	28.1	С	0.11	first reported 281
913			_		
963	IEC60247	26.7	С	-0.03	first reported 2.67
974 975	EN60247	22.42		-0.45 	
1072	EN60247	36.10		0.90	
1135	IEC60247	12	R(0.05)	-1.48	
1137					
1146 1156	EN60247	28.2		0.12	
1178	EN60247	24.6		-0.23	
1264					
1304	IEC60247	29.535		0.25	
1306 1352	IEC60247 IEC60247	25.82 26.51		-0.11	
1367	EN60247	34.11		-0.05 0.71	
1374	IEC60247	23		-0.39	
1430					
1435	IEC60247	24.82		-0.21	
1440 1442	IEC60247	24.86		-0.21	
1444	12000247			-0.21	
1458	IEC60247	33.02		0.60	
1460	IEC60247	26.70		-0.03	
1478 1505	IEC60247 IEC60247	28.84 29.61		0.19 0.26	
1513	12000247				
1545					
1560	IEC60247	26.77		-0.02	
1624	IEC60247	24.4	0	0.72	first reported 47.20
1660 1687	EN60247	34.4 25.94	С	0.73 -0.10	first reported 47.20
1702	IEC60247	25.68		-0.13	
1719	IEC60247	28.71		0.17	
1743	IEC60247	19.5		-0.74	
1801 1885	IEC60247	29.92	С	0.29	first reported 663.43
1888	EN60247	29.03	· ·	0.20	mot reported 600.40
1890	IEC60247	25.05		-0.19	
1891	IEC60247	23.0		-0.39	
1923 1924					
1925					
6002	EN60247	26.87		-0.01	
6015	EN60247	24.785		-0.22	
6031 6036	IEC60247	26.94		0.00	
6053					
6067	IEC60247	29.865	С	0.29	first reported 47.255
6085					
6088	IEC60247	34.51		0.75	
6099 6124	IEC60247 IEC60247	29.95 22.08		0.29 -0.48	
6140	EN60247	21.36		-0.55	
6141	IEC60247	24.15		-0.28	
6155					
6167 6255	IEC60247	31.1		0.41	
6275	IEC60247	26.65		-0.03	
6278	IEC60247	271.6	R(0.01)	24.19	
6280	IEC60247	27.1		0.01	
6334	IEC60247	30.9		0.39	

normality	OK
n	44
outliers	2
mean (n)	26.968
st.dev. (n)	3.9552
R(calc.)	11.075
st.dev.(EN60247:04)	10.1131
R(EN60247:04)	28.317

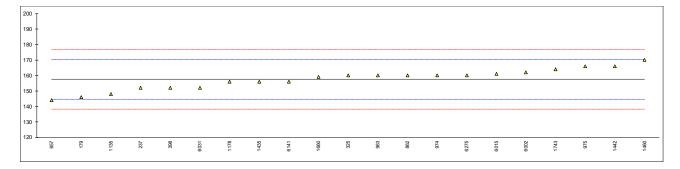


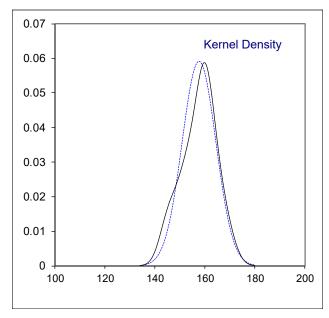


Determination of Flash Point C.O.C. on sample #20226; results in °C

lab	method	value	mark z(targ)	remarks
179	D92	146	-1.81	
237	D92	152	-0.87	
325	D92	160	0.37	
331				
398	ISO2592	152	-0.87	
445				
511 614				
657	D92	 144.0	-2.12	
862	D92	160	0.37	
902				
912				
913				
963	D92	160.0	0.37	
974 975	D92	160	0.37	
1072	D92	166 	1.30	
1135	D92	148.0	-1.50	
1137				
1146				
1156				
1178	ISO2592	156.0	-0.25	
1264 1304				
1304				
1352				
1367				
1374				
1430	D 00			
1435	D92	156.0	-0.25	
1440 1442	ISO2592	 166	1.30	
1444	1302392		1.30	
1458				
1460	D92	170.0	1.93	
1478				
1505				
1513				
1545 1560				
1624				
1660	D92	159	0.21	
1687				
1702				
1719	1000500	404		
1743 1801	ISO2592	164 	0.99	
1885				
1888				
1890				
1891				
1923				
1924 1925				
6002	ISO2592	162.0	0.68	
6015	ISO2592	161.0	0.53	
6031	D92	152.0	-0.87	
6036				
6053				
6067 6085				
6088				
6099				
6124				
6140				
6141	D92	156	-0.25	
6155				
6167 6255				
6275	D92	160	0.37	
6278	J02		0.57	
6280				
6334				

normality	OK
n	21
outliers	0
mean (n)	157.62
st.dev. (n)	6.749
R(calc.)	18.90
st.dev.(D92:18)	6.429
R(D92:18)	18

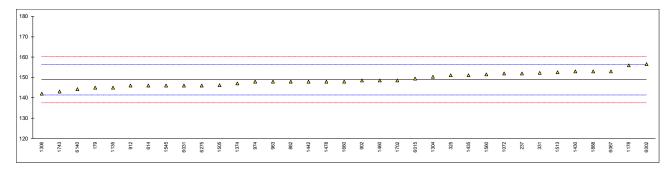


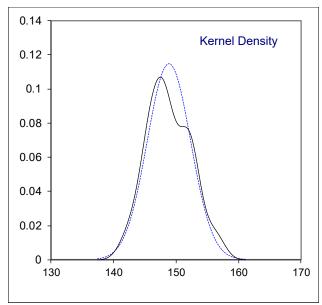


Determination of Flash Point PMcc on sample #20226; results in °C

lab	method	value	mark z(targ)	remarks
179	D93-A	145	-1.02	
237	D93-B	152.0	0.84	
325	D93-A	151.0	0.57	
331	D93-A	152.2	0.89	
398	20071			
445				
511				
614	D93-A	146	-0.75	
657	D00 / (
862	D93-A	148	-0.22	
902	D93-A	148.5	-0.09	
912	ISO2719-B	146	-0.75	
913	.00202			
963	D93-A	148.0	-0.22	
974	D93-A	148	-0.22	
975				
1072	ISO2719-A	152	0.84	
1135	ISO2719-A	145.0	-1.02	
1137				
1146				
1156				
1178	ISO2719-A	155.9	1.87	
1264				
1304	In house	150.3	0.39	
1306	D93-A	142	-1.81	
1352				
1367				
1374	D93-A	147.1	-0.46	
1430	D93-A	153	1.10	
1435	D93-A	151.0	0.57	
1440	1000740 4			
1442	ISO2719-A	148.0	-0.22	
1444				
1458	D93-A	110 E	-0.09	
1460 1478	ISO2719-A	148.5 148.0	-0.22	
1505	D93-A	146.2	-0.70	
1513	ISO2719-A	152.5	0.97	
1545	ISO2719-A	146	-0.75	
1560	ISO2719-A	151.5	0.70	
1624				
1660	D93-A	148	-0.22	
1687				
1702	ISO2719-A	148.5	-0.09	
1719				
1743	ISO2719-A	143	-1.55	
1801				
1885	D00 D	450		
1888	D93-B	153	1.10	
1890				
1891 1923				
1923				
1924				
6002	ISO2719-A	156.5	2.03	
6015	ISO2719-A	149.5	0.17	
6031	D93-A	146.0	-0.75	
6036	200.1		-0.75	
6053				
6067	D93-B	153	1.10	
6085				
6088				
6099				
6124				
6140	ISO2719-A	144.3	-1.20	
6141				
6155				
6167				
6255	D03 A	146.0	0.75	
6275	D93-A	146.0	-0.75	
6278 6280				
6334				
0004				

normality	OK
n	35
outliers	0
mean (n)	148.84
st.dev. (n)	3.472
R(calc.)	9.72
st.dev.(ISO2719-A:16)	3.774
R(ISO2719-A:16)	10.57
compare	
R(ISO2719-B:16)	16
R(D93-A:20)	10.57
R(D93-B:20)	10

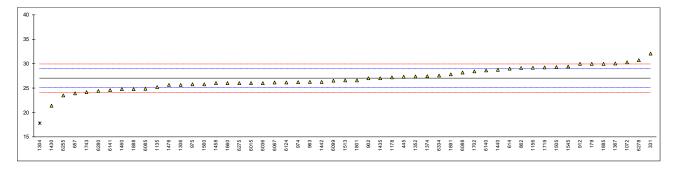


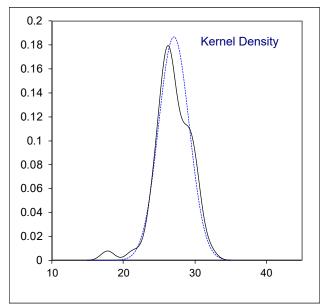


Determination of Interfacial Surface Tension on sample #20226; results in mN/m

lab	method	value	mark	7(tara)	remarks
179	D971	30	mark	z(targ) 3.05	IGHIQINƏ
237	וופּע	30 		3.05	
325					
331	EN14210	32.09		5.22	
398	LIVITZIO				
445	D971	27.3		0.26	
511	5071				
614	D971	29		2.02	
657	D971	23.95		-3.21	
862	D971	29.1		2.12	
902	D971	26.99		-0.06	
912	D971	30		3.05	
913					
963	D971	26.3		-0.78	
974	D971	26.2		-0.88	
975		25.8		-1.29	
1072	D971	30.30		3.36	
1135	D971	25.2		-1.91	
1137					
1146	D071	20.2		2.23	
1156 1178	D971	29.2			
1264	D971	27.19 		0.15 	
1304	D971	17.80	R(0.01)	-9.57	
1304	D971	25.67	(0.01)	-1.43	
1352	D971	27.4		0.36	
1367	D971	30.03		3.09	
1374	D971	27.45		0.41	
1430	D971	21.4		- 5.85	
1435	D971	27.01		-0.04	
1440	D971	28.735		1.74	
1442	IEC62961	26.3		-0.78	
1444					
1458	D971	26.0		-1.09	
1460	D971	24.8		-2.33	
1478	D971	25.66		-1.44	
1505	D971	29.3		2.33	
1513	D971	26.59		-0.48	
1545	D971	29.44		2.47	
1560 1624	D971	25.8		-1.29 	
1660	D971	26.0	С	-1.09	first reported 47.2
1687	D37 1		C	-1.03	ilist reported 47.2
1702	D971	28.455		1.45	
1719	D2285	29.24		2.27	
1743	D971	24.2		-2.95	
1801	D971	26.6		-0.47	
1885	D971	30.0		3.05	
1888	ISO6295	24.8	С	-2.33	first reported 21.8
1890					
1891	D971	27.8		0.78	
1923					
1924					
1925					
6002	D074			4.07	
6015	D971	26.020		-1.07	
6031 6036	IEC62061	26.0256		 -1.06	
6053	IEC62961	26.0256 		-1.06	
6067	D971	26.15		-0.93	
6085	D971 D971	24.868		-0.93 -2.26	
6088	ISO6295	28.2		1.19	
6099	ISO6295	26.5		-0.57	
6124	D971	26.15		-0.93	
6140	D971	28.6		1.60	
6141	D971	24.59		-2.55	
6155					
6167					
6255	ISO6295	23.5		-3.67	
6275	D971	26		-1.09	
6278	D971	30.7		3.78	
6280	D971	24.43		-2.71	
6334	D971	27.55		0.52	

normality	OK
n	52
outliers	1
mean (n)	27.050
st.dev. (n)	2.1339
R(calc.)	5.975
st.dev.(D971:20)	0.9661
R(D971:20)	2.705
compare	
R(IEC62961:18)	10

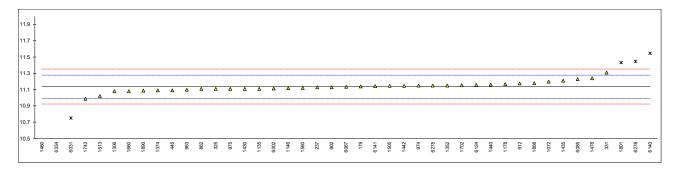


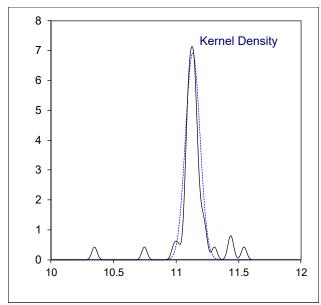


Determination of Kinematic Viscosity at 40°C on sample #20226; results in mm²/s

lab	method	value	mark	z(targ)	remarks
179	D445	11.14	mark	0.05	Tomand
237	D445	11.13		-0.09	
325	D445	11.11		-0.37	
331	D7279 corr. to D445	11.31		2.43	
398					
445	D445	11.09		-0.65	
511 614					
657					
862	D445	11.11		-0.37	
902	D445	11.13		-0.09	
912	D445	11.175		0.54	
913					
963	D445	11.10		-0.51	
974	D445	11.15		0.19	
975 1072	ISO3104	11.11 11.20		-0.37 0.89	
1135	ISO3104	11.11		-0.37	
1137					
1146	D445	11.117		-0.27	
1156					
1178	D7042	11.168		0.44	
1264					
1304 1306	D445	11.08		-0.79	
1352	D7042	11.152		0.22	
1367	· - · -				
1374	D445	11.09		-0.65	
1430	D445	11.11		-0.37	
1435	D7042	11.21		1.03	
1440 1442	D7279 corr. to D445	11.1602		0.33 0.15	
1444	D7042	11.147 		0.15	
1458					
1460	D445	9.863	R(0.01)	-17.79	
1478	D7042	11.239	, ,	1.44	
1505	D7042	11.1461		0.14	
1513	ISO3104	11.02		-1.62	
1545 1560	ISO3104	 11.12		-0.23	
1624	1303104			-0.23	
1660	D7042	11.082		-0.76	
1687					
1702	D7042	11.1540		0.25	
1719	D7070	40.000	0		First are rested 40.54
1743	D7279 corr. to D445 ISO3104	10.986	C P(0.05)	-2.10 4.14	first reported 10.51
1801 1885	1003104	11.4328 	R(0.05)	4.14	
1888	D445	11.18		0.61	
1890	ISO3104	11.089		-0.66	
1891					
1923					
1924					
1925 6002	ISO3104	 11.113		-0.32	
6015	1000104	11.113		-0.32	
6031	D445	10.75	R(0.01)	-5.40	
6036			. ,		
6053	100015:				
6067	ISO3104	11.134		-0.03	
6085 6088	D7042	11.228		1.28	
6099					
6124	D7042	11.1593		0.32	
6140	D445	11.5470	R(0.01)	5.74	
6141	D445	11.1421	. ,	0.08	
6155					
6167					
6255 6275	D445	 11.15		0.19	
6278	D445 D445	11.15	R(0.05)	4.35	
6280	D 170		11(0.00)	4.55	
6334	D445	10.35	C,R(0.01)	-10.98	first reported 10.41
			, ,		

normality	not OK
n	37
outliers	6
mean (n)	11.1363
st.dev. (n)	0.05776
R(calc.)	0.1617
st.dev.(iis memo 1401)	0.07159
R(iis memo 1401)	0.2005
compare	
R(D445:19a)	0.0377
R(ISO3104-A:20)	0.1359
R(D7042:16e3)	0.3928

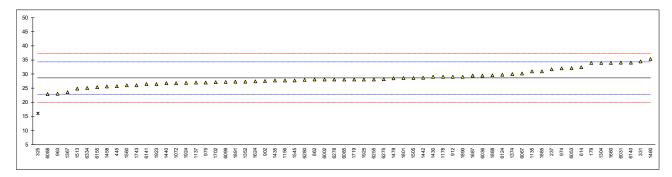


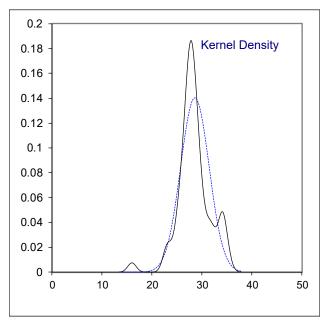


Determination of Water on sample #20226; results in mg/kg

lab	method	value	mark	z(targ)	remarks
179	D1533	34	man	1.88	
237	D6304-C	31.7	С	1.08	first reported 53.9
325	D6304-C	16	R(0.01)	-4.40	
331	D6304-A	34.5	\ - /	2.06	
398					
445	IEC60814	25.69		-1.02	
511					
614	IEC60814	32.5		1.36	
657	D6304-C	< 10		<-6.49	possibly a false negative test result?
862	D6304-A	28		-0.21	
902	D1533	27.5		-0.39	
912 913	D6304-C	29 		0.14	
963	D1533	23		-1.96	
974	D1533	32		1.19	
975	D 1000	27		-0.56	
1072	EN60814	26.8		-0.63	
1135	IEC60814	31		0.84	
1137	ISO10337	27		-0.56	
1146					
1156	EN60814	27.7		-0.32	
1178	EN60814	29.0		0.14	
1264	la la acces	24.0		4.00	
1304	In house	34.0		1.88	
1306 1352	IEC60814	 27.34		 -0.44	
1352	D1533	27.34 23.5		-0.44 -1.78	
1307	IEC60814	23.5 30		0.49	
1430	D6304-A	29		0.14	
1435	IEC60814	27.7		-0.32	
1440	EN60814	26.75		-0.65	
1442	IEC60814	28.65		0.02	
1444					
1458	IEC60814	25.6		-1.05	
1460	D1533	35.3		2.34	
1478	IEC60814	28.45		-0.05	
1505	IEC60814	28.6		0.00	
1513 1545	IEC60814 IEC60814	24.8 27.71		-1.33 -0.31	
1560	IEC60814	26		-0.51	
1624	IEC60814	27.4		-0.42	
1660	IEC60814	34		1.88	
1687	EN60814	29.4		0.28	
1702	IEC60814	27.1		-0.52	
1719	IEC60814	28.05		-0.19	
1743	IEC60814	26		-0.91	
1801	EN60814	28.55		-0.02	
1885	D1533	31		0.84	
1888	EN60814	29.56		0.33	
1890	IEC60814	29.02		0.15	
1891 1923	IEC60814 EN60814	27.3 26.45		-0.45 -0.75	
1923	EN60814 EN60814	26.45		-0.73	
1925	EN60814	28.1		-0.00	
6002	EN60814	28.0		-0.21	
6015					
6031	IEC60814	34.0175		1.89	
6036	IEC60814	29.4		0.28	
6053	IEC60814	32.1		1.22	
6067	IEC60814	30.25		0.57	
6085	IEC60814	28.0063		-0.21	
6088 6099	D1533 IEC60814	22.9 27.2		-1.99 -0.49	
6124	D1533	27.2		0.49	
6140	EN60814	34.1		1.92	
6141	D1533	26.435		-0.76	
6155	D1533	25.4		-1.12	
6167					
6255	IEC60814	28.1		-0.18	
6275	IEC60814	28.16		-0.15	
6278	D1533	28		-0.21	
6280	IEC60814	27.93		-0.24	
6334	IEC60814	25		-1.26	

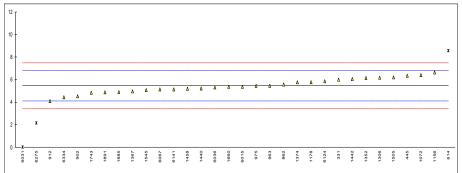
normality	OK
n	62
outliers	1
mean (n)	28.603
st.dev. (n)	2.8454
R(calc.)	7.967
st.dev.(EN60814:98)	2.8651
R(EN60814:98)	8.022
compare	
R(D1533:12)	14
R(D6304-C:20)	16.296

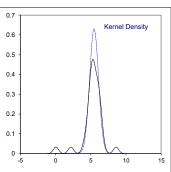




Determination of Total Furanic Compounds on sample #20227; results in mg/kg

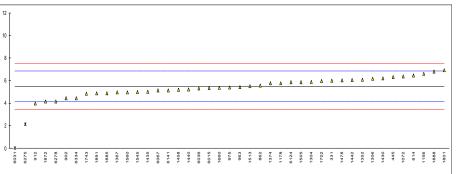
lab	method	value	mark	z(targ)	remarks
331	EN61198	6		0.80	
445	IEC61198	6.348		1.32	
614	IEC61198	8.58	R(0.01)	4.62	
862	IEC61198	5.57		0.17	
902	D5837	4.5133		-1.39	
912	IEC61198	4.085		-2.03	
963	D5837	5.451		-0.01	
975	D5837	5.4435	С	-0.02	first reported 5443.5
1072	EN61198	6.400		1.40	
1135					
1137	EN04400			4.70	
1156	EN61198	6.625		1.73	
1178	EN61198	5.78195		0.48	
1264 1304					
1304	IEC61198	6.180		1.07	
1352	IEC61198	6.14263		1.07	
1367	EN61198	4.95		-0.75	
1374	D5837	5.76		0.45	
1430	2000.				
1435					
1440	IEC61198	5.215		-0.36	
1442	IEC61198	6.05		0.88	
1444					
1458	IEC61198	5.19		-0.39	
1478					
1505	D5837	6.20		1.10	
1513	15004400				
1545	IEC61198	5.0845		-0.55	
1560 1660	IEC61100	 5.366		-0.13	
1702	IEC61198	J.300 		-0.13	
1743	IEC61198	4.84		-0.91	
1801	12001100				
1872					
1885	D5837	4.896		-0.83	
1888					
1891	IEC61198	4.873		-0.86	
6015	EN61198	5.3771		-0.12	
6031	IEC61198	0.031570	C,R(0.01)	-8.02	first reported 0.0495702
6036	IEC61198	5.32		-0.20	
6067	IEC61198	5.12630		-0.49	
6085	IEC61100	 E 0E20E		0.50	
6124	IEC61198 D5837	5.85295 5.135627		0.59	
6141 6275	IEC61198	5.135627 2.161219	R(0.01)	-0.47 -4.87	
6278	15001190	2.101219	K(0.01)	-4.07	
6334	IEC61198	4.45		-1.49	
0004	12001130	7.70		-1.40	
	normality	OK			
	n	29			
	outliers	3			
	mean (n)	5.4561			
	st.dev. (n)	0.63249			
	R(calc.)	1.7710			
	st.dev.(Horwitz)	0.67622			
	R(Horwitz)	1.8934			
	compare	0.0404			
	R(IEC61198:93)	0.8184			
12 T					0.7

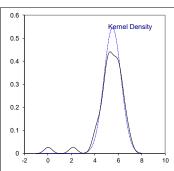




Determination of 2-Furfural on sample #20227; results in mg/kg

lab	method	value	mark	z(targ)	remarks
331	EN61198	6		0.74	
445	IEC61198	6.331		1.22	
614	IEC61198	6.47		1.43	
862	IEC61198	5.57		0.11	
902	D5837	4.4474		-1.54	
912	IEC61198	3.978		-2.23	
963	D5837	5.409		-0.13	
975	D5837	5.4015	С	-0.14	first reported 5401.5
1072	EN61198	6.387		1.31	•
1135					
1137					
1156	EN61198	6.619		1.65	
1178	EN61198	5.78195		0.42	
1264					
1304	IEC61198	5.894		0.58	
1306	IEC61198	6.180		1.00	
1352	IEC61198	6.07533		0.85	
1367	EN61198	4.95		-0.80	
1374	D5837	5.76		0.39	
1430	EN61198	6.21		1.05	
1435	IEC61198	5.009		-0.72	
1440	IEC61198	5.215		-0.42	
1442	IEC61198	6.05		0.81	
1444	.====				
1458	IEC61198	5.19		-0.45	
1478	IEC61198	6.030		0.78	
1505	D5837	5.86		0.53	
1513	IEC61198	5.502		0.01	
1545	IEC61198	4.9975		-0.74	
1560	IEC61198	4.952		-0.80	
1660	IEC61198	5.366		-0.19	
1702	IEC61198	5.960		0.68	
1743	IEC61198	4.84	С	-0.97	first reported < 0.05
1801	EN61198	6.92807	· ·	2.10	motroportou otoo
1872	EN61198	4.143		-1.99	
1885	D5837	4.874		-0.92	
1888	EN61198	6.81		1.93	
1891	IEC61198	4.863		-0.93	
6015	EN61198	5.3325		-0.24	
6031	IEC61198	0.013182	C,R(0.01)	-8.06	first reported 0.0311823
6036	IEC61198	5.32	-, (,	-0.26	
6067	IEC61198	5.12630		-0.55	
6085	.====				
6124	IEC61198	5.84295		0.51	
6141	D5837	5.12976		-0.54	
6275	IEC61198	2.13833	R(0.01)	-4.94	
6278	D5837	4.156	()	-1.97	
6334	IEC61198	4.45		-1.54	
		014			
	normality	OK			
	n	41			
	outliers	2			
	mean (n)	5.4978			
	st.dev. (n)	0.73105			
	R(calc.)	2.0469			
	st.dev.(Horwitz)	0.68062			
	R(Horwitz)	1.9057			
	compare	0.0047			
	R(IEC61198:93)	0.8247			
					0.6





APPENDIX 2

Other reported Furanics in sample #20227; results in mg/kg

lab	2-Acetyl Furan	2-Furfurylalcohol	5-Hydroxy Methyl-2-Furfural	5-Methyl-2-Furfural
331	<0.05	<0.05	< 0.05	<0.05
445	0.007	<0.001	<0.001	0.010
614	<0.01	2.11	<0.01	<0.01
862	<0.05	<0.05	<0.05	<0.05
902	0.0117	0.0205	0.0209	0.0127
912	0.023	0.048	0.026	0.010
963	<0.01	0.0246	0.0181	<0.01
975	<0.01 C	0.024 0.024 C	0.018 C	<0.01 C
1072	0.006	<0,01	<0,01	0.007
1135		~U,U I 		
1133				
	4.379286	0.00	0.00	
1156	0.000	0.00	0.00	0.006
1178	0.0001	0.0001	0.0001	0.0001
1264				
1304				
1306	<0.03	<0.03	<0.03	<0.03
1352	not detected	not detected	0.06730	not detected
1367	<0.05	<0.05	<0.05	<0.05
1374	<0.01	<0.01	<0.01	<0.01
1430	<0.1	<0.04	<0.02	<0.03
1435	<0.03	<0.03	<0.03	<0.03
1440	0	0	0	0
1442	<0,05	<0,05	<0,05	<0,05
1444				
1458	<0.01	<0.01	<0.01	<0.01
1478	0.006	0.00	0.00	0.00
1505	0.32	0.01	0.02	0.01
1513	<0.05	<0.05	<0.05	<0.05
1545	0.0061	0.0023	0.0098	0.0688
1560	Not detected	Not detected	Not detected	Not detected
1660	< 0.05	< 0.05	< 0,05	< 0,05
1702	Not Detected	Not Detected	Not Detected	Not Detected
1743	<0.05	<0.05	<0.05	<0.05
1801				
1872	< 0.05	< 0.05	0.0502	< 0.05
1885	0.001	0	0.006	0.015
1888	<0.05	<0.05	<0.05	<0.05
1891	0.01	<0.01	<0.03	<0.03
6015	0.0095	0.0068	0.0221	0.0062
6031	0.0151326	0.0001031	0.0013159	0.0002
6036	0.0131320	0.0001031	0	0
	0	0	0	0
6067		U 		
6085	0.000			0.000
6124	0.009	<0.01	<0.01	0.006
6141	0	0	0.005867	0
6275	0	0	0	0.022889
6278	0.003	0	0.009	0.008
6334	<0.01	<0.01	<0.01	<0.01

Lab 975: first reported <10, 24, 18, <10 respectively

APPENDIX 3

Number of participants per country

- 7 labs in AUSTRALIA
- 4 labs in BELGIUM
- 1 lab in BOSNIA and HERZEGOVINA
- 4 labs in BULGARIA
- 1 lab in CHINA, People's Republic
- 1 lab in CROATIA
- 3 labs in FRANCE
- 3 labs in GERMANY
- 2 labs in GREECE
- 2 labs in INDIA
- 1 lab in INDONESIA
- 2 labs in ITALY
- 1 lab in KUWAIT
- 2 labs in MALAYSIA
- 1 lab in MOROCCO
- 2 labs in NETHERLANDS
- 1 lab in NEW ZEALAND
- 1 lab in NIGERIA
- 1 lab in PAKISTAN
- 1 lab in PERU
- 1 lab in PHILIPPINES
- 2 labs in POLAND
- 1 lab in PORTUGAL
- 2 labs in QATAR
- 4 labs in SAUDI ARABIA
- 2 labs in SINGAPORE
- 1 lab in SLOVENIA
- 1 lab in SOUTH AFRICA
- 2 labs in SOUTH KOREA
- 5 labs in SPAIN
- 2 labs in SWITZERLAND
- 2 labs in TURKEY
- 3 labs in UNITED ARAB EMIRATES
- 3 labs in UNITED KINGDOM
- 1 lab in UNITED STATES OF AMERICA
- 1 lab in URUGUAY

APPENDIX 4

Abbreviations

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

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \\ DG(0.05) &= \text{straggler in Double Grubbs' outlier test} \\ \end{array}$

R(0.01) = outlier in Rosner's outlier test R(0.05) = straggler in Rosner's outlier test

E = calculation difference between reported test result and result calculated by iis

W = test result withdrawn on request of participant ex = test result excluded from statistical evaluation

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

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