Results of Proficiency Test Toluene February 2020

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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1 Introduction

Since 1999, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Toluene based on the scope of the latest version of ASTM D841 every year. During the annual proficiency testing program 2019/2020, it was decided to continue the round robin for the analysis of Toluene.

In this interlaboratory study 36 laboratories in 18 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of this proficiency test for Toluene 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 tests (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample of one liter of Toluene labelled #20011. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/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.

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2.4 SAMPLES

A batch of approximately 75 liters of Toluene was obtained from a local chemical supplier. After homogenization 69 amber glass bottles of 1 liter were filled and labelled #20011. 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/L
sample #20011-1	0.86677
sample #20011-2	0.86678
sample #20011-3	0.86680
sample #20011-4	0.86679
sample #20011-5	0.86680
sample #20011-6	0.86683
sample #20011-7	0.86685
sample #20011-8	0.86677

Table 1: homogeneity test results of subsamples #20011

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/L
r (observed)	0.00008
reference test method	ISO12185:96
0.3 * R(reference test method)	0.00015

Table 2: evaluation of repeatability of subsamples #20011

The calculated repeatability was in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one subsample with Toluene labelled #20011 was sent on January 29, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Toluene packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

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2.6 ANALYZES

The participants were requested to determine on the Toluene sample #20011: Acid Wash Color, Appearance, Color Pt/Co, Copper Corrosion, Density at 20°C, Distillation (IBP, 50% recovered, DP and range), Purity by GC, Benzene, Nonaromatics, Total Impurities, Refractive Index at 25°C and Water.

It was explicitly requested to treat the sample as if it was a routine sample 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 appropriate reference test methods 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 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 reanalysis). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the test 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.

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

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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. In some cases, a reproducibility based on former iis proficiency tests could be used. 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(target) = (test result - average of PT) / target standard deviation
```

The z(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. 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

During the execution of this proficiency test some serious problems occurred. Due to COVID-19 outbreak 10 participants were not able to report because of restricted dispatch to some countries. It was decided that for those 10 participants the deadline was extended and an additional data entry portal round was created. Not all laboratories were able to perform all analyzes requested. Finally, 26 participants reported 253 numerical test results. Observed were 16 statistically outlying test results, which is 6.3%. 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, see also paragraph 3.1.

4.1 EVALUATION PER TEST

In this section the test results are discussed 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 methods are also in the tables together with the reported test results. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports, ASTM methods are referred to with a number e.g. D1218 and an added designation for the year that the method was adopted or revised e.g. D1218:12. If applicable, a designation in parentheses is added to designate the year of reapproval e.g. D1218:12(2016). In the results tables of appendix 1 only the method number and year of adoption or revision e.g. D1218:12 will be used.

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Unfortunately, a suitable reference test method providing the precision data is not available for all determinations. For these the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

Sample #20011

Acid Wash Color: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the

requirements of ASTM D848:18.

Appearance: This determination was not problematic. All participants agreed about the

appearance of the sample which was bright, clear and free of suspended

matter (Pass).

Color Pt/Co: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the

requirements of ASTM D5386:16 or ASTM D1209:05(2019).

<u>Copper Corrosion:</u> This determination was not problematic. All participants agreed on a result

of 1a/b (Pass).

<u>Density at 20°C:</u> This determination was not problematic. One statistical outlier was

observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO12185:96.

Distillation: This determination may be problematic for a number of laboratories. In total

seven statistical outliers were observed and two other test results were excluded. However, all calculated reproducibilities after rejection of the suspect data are in agreement with the requirements of automated or

manual modes of ASTM D850:18.

<u>Purity:</u> This determination was problematic. Two statistical outliers were observed.

The calculated reproducibility after rejection of the statistical outliers is not

in agreement with the requirements of ASTM D7504:20.

<u>Benzene:</u> This determination was not problematic. Two statistical outliers were

observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM

D7504:20.

Nonaromatics: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the

requirements of ASTM D7504:20.

<u>Total Impurities:</u> This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in full agreement with the

estimated reproducibility calculated using the Horwitz equation.

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Refractive Index: 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 D1218:12(2016).

<u>Water:</u> 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 ASTM D1064:16.

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 using 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 literature reference test methods (in casu ASTM test methods) or the estimated target reproducibility are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit.)
Acid Wash Color (acid layer)		21	0.5	0.9	1.9
Appearance		23	Pass (B&C)	n.a.	n.a.
Color Pt/Co		21	3.4	3.0	5.1
Copper Corrosion		18	1 (1a,1b)	n.a.	n.a.
Density at 20°C	kg/L	23	0.86679	0.0002	0.0005
Distillation, IBP	°C	15	110.2	0.3	0.6
Distillation, 50% recovered	°C	16	110.6	0.2	0.2
Distillation, DP	°C	17	110.9	0.3	0.5
Purity	%M/M	23	99.783	0.032	0.013
Benzene	mg/kg	24	430.4	63.0	59.6
Nonaromatics	mg/kg	25	625.8	275.5	565.6
Total Impurities	mg/kg	16	2170	430	433
Refractive Index at 25°C		17	1.4940	0.0008	0.0005
Water	mg/kg	17	80.4	19.3	12.8

Table 3: performance evaluation sample #20011

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

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

	February 2020	February 2019	March 2018	March 2017	March 2016
Number of reporting laboratories	26	35	36 *)	67	59
Number of test results	253	284	267	743	793
Number of statistical outliers	16	14	10	32	19
Percentage of statistical outliers	6.3%	4.9%	3.8%	4.3%	2.4%

Table 4: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the respective reference test methods. The conclusions are given the following table.

	February 2020	February 2019	March 2018	March 2017	March 2016
Acid Wash Color (acid layer)	++	++	++	+	++
Color Pt/Co	+	++	++	+	++
Copper Corrosion	n.e.	n.e.	n.e.	n.e.	n.e.
Density at 20°C	++	++	++	++	++
Distillation	+	+	+/-	+/-	+
Purity		-	+	n.e.	+
Benzene	+/-	()	n.e.		+/-
Nonaromatics	++	++	+/-	+	++
Total Impurities	+/-	n.e.	n.e.	n.e.	n.e.
Refractive Index at 25°C	-	+	+	+	-
Water	-	n.e.	n.e.	n.e.	n.e.

Table 5: comparison determinations against the reference test methods

In the table above 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

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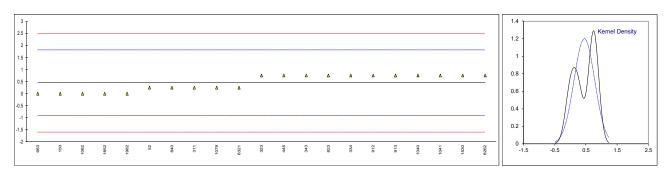
^{*)} from March 2018 the Toluene results are reported separately from Benzene, hence the lower number of reporting laboratories.

^{*)} Reproducibility between brackets is based on a much higher level than present in sample

APPENDIX 1 Determination of Acid Wash Color (acid layer) on sample #20011;

lab	method	reported test value	iis conversion*	mark	z(targ)	remarks
52	D848	0+	0.25		-0.30	
150	D848	0	0		-0.66	
171	D848	Pass				
311	D848	0+	0.25		-0.30	
323	D848	-1	0.75		0.44	
333						
334	D848	1-	0.75		0.44	
343	D848	1-	0.75		0.44	
445	D848	1-	0.75		0.44	
551						
555						
663	D848	No. 0	0		-0.66	
823	D848	1-	0.75		0.44	
840	D848	0+	0.25		-0.30	
855						
862						
864						
865						
866						
870						
912	D848	-1	0.75	С	0.44	first reported I
913	D848	1-	0.75		0.44	•
1040	D848	<1	0.75		0.44	
1041	D848	1-	0.75		0.44	
1062	D848	0	0		-0.66	
1065						
1079	D848	0+	0.25		-0.30	
1151						
1530	D848	< 1	0.75		0.44	
1741						
1852	D848	0	0		-0.66	
1982	D848	0	0		-0.66	
6203						
6262	D848	1-	0.75		0.44	
6315						
6321	D848	0+	0.25		-0.30	
	normality	OK				
	n	21				
	outliers	0				
	mean (n)	0.45				
	st.dev. (n)	0.332				
	R(calc.)	0.93				
	st.dev.(D848:18)	0.682				
	R(D848:18)	1.91				

^{*)} In the calculation of the mean, standard deviation, reproducibility and for the graphs, a reported value of 'y-', '-y' or '<y' is changed into y-0.25 (for example 1- into 0.75) and 'y+' is changed into y+0.25 (for example 0+ into 0.25).
**) The precision data of Benzene is used



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Determination of Appearance on sample #20011;

lab ı	method	value	mark	z(targ)	remarks
52 I	D4176	Pass			
150 I	E2680	Pass			
171 I	E2680	Pass			
311 I	E2680	pass			
	E2680	clear and bright			
333					
334 I	EN15769	Clear and colourless			
343 I	E2680	PASS			
	D4176	C&B			
551					
555					
	Visual	Bright & Clear			
823 I	E2680	Pass			
840 I	E2680	Pass			
855					
862					
864					
865					
866					
870					
	E2680	PASS			
913 I	E2680	Pass			
1040	Visual	clear & bright visual free			
1041 \	Visual	CBFSM			
1062 \	Visual	pass			
1065					
	Visual	Bright&Clear			
1151					
1530 \	Visual	clear & bright			
1741					
	Visual	clear & colourless			
	Visual	waterclear			
6203					
6262	Visual	Clear and Bright			
6315	Visual	clear,bright			
6321 I	D4176	Pass - Clear and Bright			
1	n	23			
	mean (n)	Pass (B&C)			

Abbreviations:

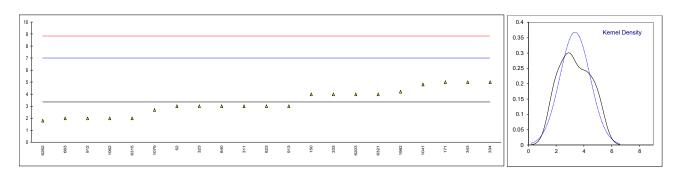
C&B = clear and bright

CBFSM = clear and bright and free from suspended matter

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Determination of Color Pt/Co on sample #20011;

lab	method	value	mark z(targ)	remarks
52	D5386	3	-0.20	
150	D5386	4	0.35	
171	D1209	5	0.90	
311	D5386	3	-0.20	
323	D5386	3	-0.20	
333	D5386	4	0.35	
334	D1209	5	0.90	
343	D5386	5	0.90	
445		- - 5	0.90	
551	D6045			
555	D.F.0.0.0			
663	D5386	2	-0.74	
823	D5386	3	-0.20	
840	D1209	3	-0.20	
855				
862				
864				
865				
866				
870				
912	D5386	2	-0.74	
913	D5386	3	-0.20	
1040	D5386	<5		
1041	ISO6271	4.8	0.79	
1062	D5386	2	-0.74	
1065				
1079	D5386	2.7	-0.36	
1151				
1530	D1209	< 3		
1741				
1852				
1982	ISO6271	4.2	0.46	
6203	D1209	4.0	0.35	
6262	D5386	1.8	-0.85	
6315	ISO6271	2	-0.74	
6321	D1209	4	0.35	
0021	B 1200	•	0.00	
	normality	OK		
	n	21		
	outliers	0		
	mean (n)	3.36		
	st.dev. (n)	1.085		
		3.04		
	R(calc.)	3.0 4 1.828		
	st.dev.(D5386:16)			
Comr	R(D5386:16)	5.12		
Compa		7		
	R(D1209:05)	7		



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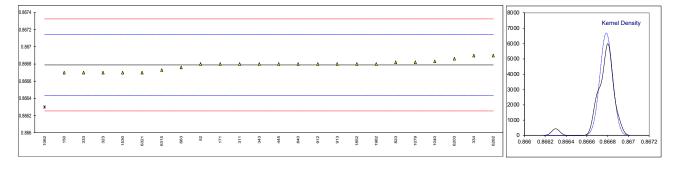
Determination of Copper Corrosion on sample #20011;

lab	method	value	mark	z(targ)	remarks
52	D849	1a			
150	D849	1a			
171	D849	1a			
311	D849	pass			
323	D849	1A			
333					
334	D849	Pass			
343					
445	D849	1a			
551					
555					
663	D849	1a			
823	D849	1a			
840	D849	1a			
855					
862					
864					
865					
866					
870					
912	D849	1A			
913	D849	1a			
1040					
1041					
1062	D849	1B			
1065	5040				
1079	D849	1A			
1151	D0.40				
1530	D849	1a			
1741					
1852	D040	4.0			
1982	D849	1A			
6203	D040	1.0			
6262	D849	1A			
6315	DIN EN ISO 2160	1			
6321					
	n	18			
	mean (n)	1a/b (Pass)			
	` '	` '			

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Determination of Density at 20°C on sample #20011: results in kg/L

lab	method	value	mark	z(targ)	remarks
52	D4052	0.8668		0.07	
150	D4052	0.8667		-0.49	
171	D4052	0.8668		0.07	
311	D4052	0.8668		0.07	
323	D4052	0.8667		-0.49	
333	ISO12185	0.8667		-0.49	
334	ISO12185	0.8669		0.63	
343	ISO12185	0.8668		0.07	
445	D4052	0.8668		0.07	
551					
555					
663	D4052	0.86676		-0.16	
823	ISO12185	0.86682		0.18	
840	D4052	0.86680		0.07	
855					
862					
864					
865					
866					
870					
912	D4052	0.8668		0.07	
913	D4052	0.8668		0.07	
1040	ISO12185	0.86683		0.24	
1041					
1062	D4052	0.8663	R(0.01)	-2.73	
1065					
1079	ISO12185	0.86682		0.18	
1151					
1530	ISO12185	0.86670		-0.49	
1741					
1852	ISO12185	0.8668		0.07	
1982	D4052	0.8668		0.07	
6203	ISO12185	0.86686		0.40	
6262	ISO12185	0.8669		0.63	
6315	ISO12185	0.86673		-0.32	
6321	ISO12185	0.8667		-0.49	
	normality	OK			
	n	23			
	outliers	1			
	mean (n)	0.86679			
	st.dev. (n)	0.000060			
	R(calc.)	0.00017			
	st.dev.(ISO12185:96)	0.000179			
	R(ISO12185:96)	0.0005			
	` '				



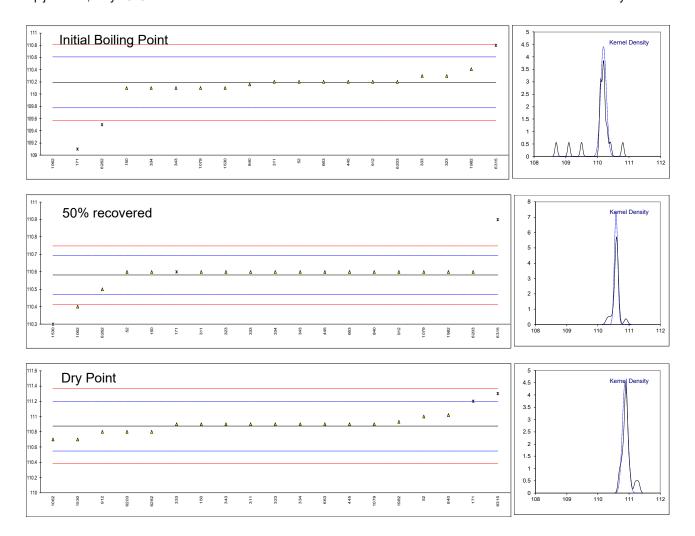
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Determination of Distillation on sample #20011; results in °C

lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)	range
52	D850-automated	110.2		0.04	110.6		0.34	111.0		0.78	0.8
150	D850-automated	110.1		-0.44	110.6		0.34	110.9		0.16	0.8
171	D850	109.1	G(0.01)	-5.26	110.6	ex	0.34	111.2	DG(0.01)	2.00	
311	D850-automated	110.2	,	0.04	110.6		0.34	110.9	,	0.16	0.7
323	D850-manual	110.3		0.52	110.6		0.34	110.9		0.16	0.6
333	D850	110.3		0.52	110.6		0.34	110.9		0.16	0.6
334	D850-automated	110.1		-0.44	110.6		0.34	110.9		0.16	0.8
343	D850-automated	110.1		-0.44	110.6		0.34	110.9		0.16	8.0
445	D850-automated	110.2		0.04	110.6		0.34	110.9		0.16	0.7
551											
555											
663	D850-automated	110.2		0.04	110.6		0.34	110.9		0.16	0.7
823											
840	D850-automated	110.16		-0.15	110.60		0.34	111.02		0.90	0.86
855											
862											
864											
865											
866											
870											
912	D1078-manual	110.2		0.04	110.6		0.34	110.8		-0.45	0.6
913											
1040											
1041											
1062	D850-automated	108.7	G(0.05)	-7.19	110.4		-3.25	110.7		-1.07	2.0
1065			, ,								
1079	D850-automated	110.1		-0.44	110.6		0.34	110.9		0.16	8.0
1151											
1530	D850-automated	110.10		-0.44	110.30	G(0.01)	-5.05	110.70		-1.07	0.6
1741											
1852											
1982	D850-automated	110.41		1.05	110.60		0.34	110.93		0.35	0.52
6203	D850-manual	110.2		0.04	110.6		0.34	110.8		-0.45	0.6
6262	D850-automated	109.5	C,G(0.01)	-3.33	110.5	С	-1.46	110.8	С	-0.45	1.3 C
6315	D850-automated	110.8	ex	2.93	110.9	G(0.05)	5.72	111.3	DG(0.01)	2.62	0.5
6321											
	normality	not OK			not OK			OK			
	n	15			16			17			
	outliers	3 +1ex			2 +1ex			2			
	mean (n)	110.19			110.58			110.87			
	st.dev. (n)	0.090			0.054			0.088			
	R(calc.)	0.25			0.15			0.25			
	st.dev.(D850-A:18)	0.208			0.056			0.163			
_	R(D850-A:18)	0.58			0.16			0.46			
Compa											
	R(D850-M:18)	0.41			0.65			0.65			

Lab 171 test result excluded for 50% recovered as statistical outliers in other related distillation parameters Lab 6262 first reported 110.4 for IBP / 111.0 for 50% rec / 111.3 for DP / 0.9 for range Lab 6315 test result excluded for IBP as statistical outliers in other related distillation parameters

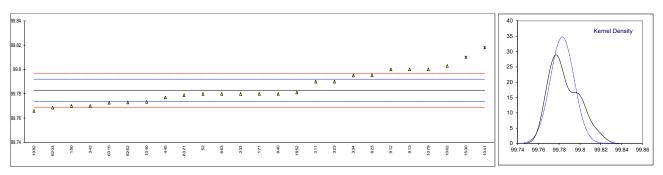
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Determination of Purity by GC on sample #20011; results in %M/M

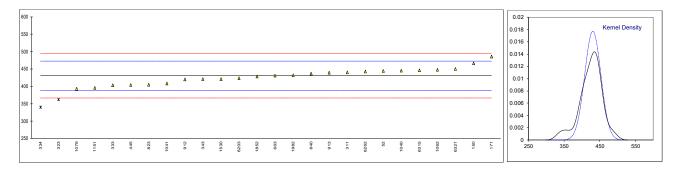
lab	method	value	mark	z(targ)	remarks
52	D7504	99.78		-0.60	
150	D7504	99.77	С	-2.74	first reported 99.75
171	D7504	99.78		-0.60	
311	D7504	99.79		1.54	
323	D7504	99.79		1.54	
333	D7504	99.78		-0.60	
334	D2360	99.795		2.61	
343	D2360	99.77	С	-2.74	first reported 99.81
445	D6526	99.7770		-1.24	
551					
555					
663	D7504	99.780		-0.60	
823	D2360	99.7952		2.65	
840	D7504	99.780		-0.60	
855					
862					
864					
865					
866					
870	D7504	00.00	С	2.00	first name at al 00 00
912 913	D7504 D7504	99.80	C	3.68 3.68	first reported 99.82
1040	D6526	99.80 99.7733		-2.04	
1040	D0320	99.818	R(0.05)	7.53	
1062	D2360	99.8030	11(0.00)	4.32	
1065	D2300				
1079	D7504	99.8002		3.72	
1151	D1001				
1530	D7504	99.810	R(0.05)	5.82	
1741			` ,		
1852	D7504	99.7812		-0.35	
1982	D2360	99.766		-3.60	
6203	D7504	99.7687		-3.02	
6262	D7504	99.7729		-2.12	
6315	D7504	99.7727		-2.17	
6321	D2360	99.779		-0.82	
	normality	OK			
	n	23			
	outliers	2			
	mean (n)	99.7828			
	st.dev. (n)	0.01148			
	R(calc.)	0.0321			
	st.dev.(D7504:20)	0.00467			
	R(D7504:20)	0.0131			



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Determination of Benzene on sample #20011; results in mg/kg

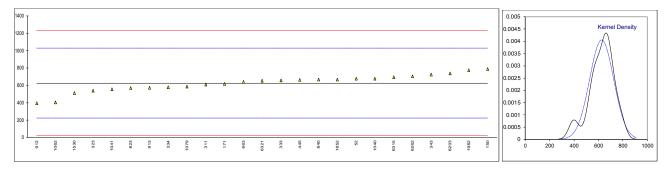
lab	method	value	mark	z(targ)	remarks
52	D7504	444		0.64	
150	D7504	466		1.68	
171	D7504	486		2.62	
311	D7504	440		0.45	
323	D7504	362	DG(0.05)	-3.21	
333	D7504	403	, ,	-1.29	
334	D2360	340	DG(0.05)	-4.25	
343	D2360	421	C ()	-0.44	first reported 410
445	D6526	404		-1.24	•
551					
555					
663	D7504	431		0.03	
823	D2360	405		-1.19	
840	D7504	436.6		0.29	
855					
862					
864					
865					
866					
870					
912	D7504	420		-0.49	
913	D7504	439		0.41	
1040	D6526	445		0.69	
1041		408		-1.05	
1062	D2360	448		0.83	
1065					
1079	D7504	393		-1.76	
1151	In-house	395.38		-1.64	
1530	D7504	421		-0.44	
1741					
1852	D7504	428	С	-0.11	reported 0.0428 mg/kg
1982	D2360	432	С	0.08	first reported 0.0432 mg/kg
6203	D7504	423		-0.35	
6262	D7504	443		0.59	
6315	D7504	446		0.74	
6321	D2360	450.5		0.95	
	normality	OK			
	n	24			
	outliers	2			
	mean (n)	430.35			
	st.dev. (n)	22.502			
	R(calc.)	63.00			
	st.dev.(D7504:20)	21.266			
	R(D7504:20)	59.55			
Compa	are	77.00			
	R(Horwitz)	77.39			



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Determination of Nonaromatics on sample #20011; results in mg/kg

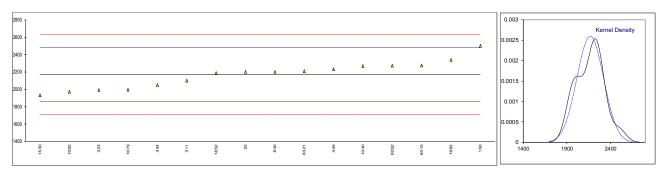
lab	method	value	mark	z(targ)	remarks
52	D7504	675		0.24	
150	D7504	786		0.79	
171	D7504	617		-0.04	
311	D7504	610		-0.08	
323	D7504	537		-0.44	
333	D7504	657		0.15	
334	D2360	580		-0.23	
343	D2360	722	С	0.48	first reported 308
445	D6526	662	O	0.18	mat reported 600
551	D0320				
555					
663	D7504	640		0.07	
823	D2360	567		-0.29	
840	D7504	666.9		0.29	
	D7504				
855					
862					
864					
865					
866					
870	5				
912	D7504	394		-1.15	
913	D7504	571		-0.27	
1040	D6526	677		0.25	
1041		554		-0.36	
1062	D2360	402		-1.11	
1065					
1079	D7504	585		-0.20	
1151					
1530	D7504	512		-0.56	
1741					
1852	D7504	667	С	0.20	first reported 0.065 mg/kg
1982	D2360	775	С	0.74	first reported 0.0775 mg/kg
6203	D7504	737		0.55	
6262	D7504	705		0.39	
6315	D7504	692		0.33	
6321	D2360	654.4		0.14	
	normality	OK			
	n	25			
	outliers	0			
	mean (n)	625.81			
	st.dev. (n)	98.381			
	R(calc.)	275.47			
	st.dev.(D7504:20) R(D7504:20)	202.013 565.64			
	N(D1304.20)	303.04			



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Determination of Total Impurities on sample #20011; results in mg/kg

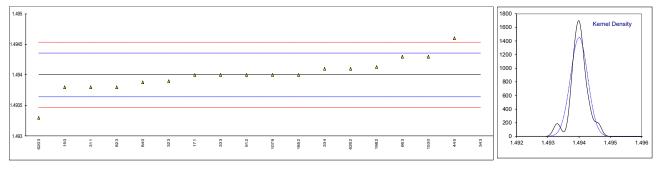
lab	method	value	mark	z(targ)	remarks
52	D7504	2200		0.19	
150	D7504	2500		2.14	
171					
311	D7504	2100		-0.45	
323	D7504	1991		-1.16	
333	B0000				
334	D2360	2050		-0.78	
343	DOFOO				
445 551	D6526	2230		0.39	
555					
663					
823					
840	D7504	2201.4		0.20	
855	B1304				
862					
864					
865					
866					
870					
912					
913					
1040	D6526	2267		0.63	
1041	B0000				
1062	D2360	1971		-1.29	
1065	D7504	4000		4.40	
1079 1151	D7504	1996 		-1.13 	
1530	D7504	1933		-1.53	
1741	D1304			-1.55	
1852	D7504	2188	С	0.12	reported 0.2188 mg/kg
1982	D2360	2340	Č	1.10	first reported 0.2340 mg/kg
6203					1
6262	D7504	2271		0.65	
6315	D7504	2275		0.68	
6321	D2360	2207.6		0.24	
	normality	OK			
	n	16			
	outliers	0			
	mean (n)	2170.06			
	st.dev. (n) R(calc.)	153.416 429.56			
	st.dev.(Horwitz 2 comp)	154.496			
	R(Horwitz 2 comp)	432.59			
	M. ISI WILE 2 GOIND)	102.00			



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Determination of Refractive Index at 25°C on sample #20011;

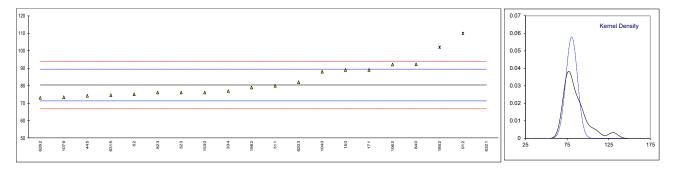
lab	method	value	mark	z(targ)	remarks
52					
150	D1218	1.4938		-1.12	
171	D1218	1.4940		0.00	
311	D1218	1.4938		-1.12	
323	D1218	1.4939		-0.56	
333	D1218	1.4940		0.00	
334	D1218	1.4941		0.56	
343	D1218	1.4968	C,G(0.01)	15.68	first reported 1.4969
445	D1218	1.4946	0,0(0.01)	3.36	mst reported 1.4909
551	D1216	1.4940		3.30	
555	D4040	4.4040		4.00	
663	D1218	1.4943		1.68	
823	D1218	1.49380		-1.12	
840	D1218	1.49388		-0.68	
855					
862					
864					
865					
866					
870					
912	D1218	1.4940		0.00	
913					
1040					
1041					
1062					
1065					
1079	D1218	1.49400		0.00	
1151	2.2.3				
1530	D1218	1.49430		1.68	
1741	21210				
1852		1.4940		0.00	
1982	DIN 51423-1	1.49413		0.72	
6203	D1218	1.49413		-3.92	
6262	D1218	1.4933		0.56	
6315	D1210				
6321					
	normality	not OK			
	n	17			
	outliers	1			
	mean (n)	1.49400			
	st.dev. (n)	0.000275			
	R(calc.)	0.000275			
	st.dev.(D1218:12)	0.000179			
	R(D1218:12)	0.0005			



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Determination of Water on sample #20011; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D6304	75		-1.18	
150	E1064	89		1.89	
171	E1064	89		1.89	
311	E1064	80		-0.08	
323	E1064	76		-0.96	
333	E4004				
334	E1064	77		-0.74	
343 445	E1064	 74.3		 -1.33	
551	□1004	74.3		-1.33	
555					
663					
823	E1064	76		-0.96	
840	E1064	92.2		2.59	
855					
862					
864					
865					
866					
870	=		0.00(0.05)		
912	E203	110	C,DG(0.05)	6.49	first reported 118
913 1040	DIN 51777	88		1.67	
1040	DINSTITI			1.07	
1062	D6304	92		2.54	
1065	D0004				
1079	E1064	73.35		-1.54	
1151					
1530	E1064	76.1		-0.94	
1741					
1852		102	DG(0.05)	4.74	
1982	E1064	79		-0.30	
6203	D6304	82		0.35	
6262	E1064	73		-1.62	
6315 6321	DIN EN ISO12937 E1064	74.6 130	G(0.01)	-1.27 10.87	
0321	□1004	130	G(0.01)	10.07	
	normality	OK			
	n	17			
	outliers	3			
	mean (n)	80.39			
	st.dev. (n)	6.880			
	R(calc.)	19.26			
	st.dev.(D1064:16)	4.565			
	R(D1064:16)	12.78			



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APPENDIX 2

Number of participants per country

- 4 labs in BELGIUM
- 2 labs in BRAZIL
- 1 lab in CANADA
- 6 labs in CHINA, People's Republic
- 2 labs in FRANCE
- 6 labs in GERMANY
- 2 labs in INDIA
- 1 lab in ISRAEL
- 1 lab in NETHERLANDS
- 1 lab in ROMANIA
- 1 lab in SAUDI ARABIA
- 1 lab in SERBIA
- 1 lab in SOUTH KOREA
- 1 lab in SPAIN
- 1 lab in THAILAND
- 2 labs in UNITED KINGDOM
- 2 labs in UNITED STATES OF AMERICA
- 1 lab in VIETNAM

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APPENDIX 3

Abbreviations

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

D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test

R(0.01) = outlier in Rosner's outlier test R(0.05) = straggler in Rosner's outlier test E = possibly an error in calculations

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

Literature

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