Results of Proficiency Test Gascondensate November 2021

Organized by:	Institute for Interlaboratory Studies Spijkenisse, the Netherlands
Author:	ing. A. Ouwerkerk
Correctors:	ing. R.J. Starink & ing. G.A. Oosterlaken-Buijs
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#### **1** INTRODUCTION

Since 2008 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Gascondensate every year. During the annual proficiency test program 2021/2022 it was decided to continue the round robin for the analysis of Gascondensate.

In this interlaboratory study 43 laboratories in 16 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the Gascondensate proficiency test 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 one sample of Gascondensate in a 0.5L amber glass bottle labelled #21230.

The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

### 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. 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

A batch of approximately 60 liters of Gascondensate was made available from the retain materials from earlier PTs on Gascondensate. After homogenization 88 amber glass bottles of 0.5L were filled and labelled #21230.

The homogeneity of the subsamples was checked by determination of Density at 15°C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/L
sample #21230-1	0.74679
sample #21230-2	0.74688
sample #21230-3	0.74677
sample #21230-4	0.74683
sample #21230-5	0.74681
sample #21230-6	0.74682
sample #21230-7	0.74682
sample #21230-8	0.74680

Table 1: homogeneity test results of subsamples #21230

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 15°C in kg/L
r (observed)	0.00009
reference test method	ASTM D4052:18a
0.3 x R (reference test method)	0.00062

Table 2: evaluation of the repeatability of subsamples #21230

The calculated repeatability is 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 sample Gascondensate labelled #21230 was sent on October 13, 2021. An SDS was added to the sample package.

#### 2.5 STABILITY OF THE SAMPLES

The stability of Gascondensate 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: Color Saybolt, Density at 15°C, Distillation at 760 mmHg (IBP, Temperature at 5%, 10%, 50%, 90%, 95% recovered, FBP, Distillation Residue and Loss), Methanol, Total Mercury, Total Sulfur, Water and Simulated Distillation (IBP, Temperature at 5%, 10%, 50%, 90%, 95% recovered and FBP).

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 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 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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, 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

## 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 (derived from e.g. ISO or ASTM test methods), 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 iis 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_{(target)} = (test result - average of PT) / target standard deviation
```

The  $z_{(target)}$  scores are listed in the test result tables in appendices 1 and 2.

Absolute values for z < 2 are very common and absolute values for z > 3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

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

## 4 EVALUATION

Some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another three weeks. Six participants reported test results after the final reporting date and eight other participants did not report any test results. Not all participants were able to report all tests requested.

In total 35 participants reported 257 numerical test results. Observed were 14 outlying test results, which is 5.4%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all 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 reported 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 test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

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

In the iis PT reports ASTM test methods are referred to with a number and if appropriate an indication of sub test method (e.g. D6304-A) and an added designation for the year that the test method was adopted or revised (e.g. D6304-A:20).

- <u>Color Saybolt</u>: 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 D6045:20.
- <u>Density at 15°C</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 ASTM D4052:18a. It should be taken into account that the reproducibility from ASTM D4052:18 is applicable to petroleum distillates and viscous oils only. Therefore, no precision data are stated in the 2018 version for Gascondensate. However, Gascondensate may contain relatively high concentrations of light ends and therefore should be treated as Gasoline, i.e. cooling the sample prior to analysis to prevent loss of light ends.

<u>Distillation at 760 mmHg</u>: This determination may be problematic. Seven statistical outliers were observed and six other test results were excluded over seven parameters. After rejection of the suspect data the calculated reproducibilities of Initial Boiling Point, 5%, 10% and 50% recovered are in agreement with the requirements of the manual mode of ASTM D86:20b. The calculated reproducibilities of 90% recovered and Final Boiling Point are not in agreement. For 95% recovered no z-scores were calculated as the calculated reproducibility was too large compared to the requirements of the manual mode of ASTM D86:20b.

It should be noted that the scope of ASTM D86 does not include Gascondensate, but only products with a limited boiling range like distillate fuels, so the target reproducibilities as used in this report may not be applicable. The use of a simulated distillation determination may be more appropriate.

- <u>Methanol</u>: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the estimated reproducibility calculated with the Horwitz equation. Due to the limited number of test results and the large observed reproducibility it was decided not to calculate z-scores.
- <u>Total Mercury</u>: The precision requirements of Table B3 in test method UOP938 is approximately 6 times stricter than the Horwitz estimate. This means that these requirements will not be met easily. Furthermore, the reproducibility of UOP938 is only available for very low concentrations (0.28 and 12.14  $\mu$ g/<u>L</u>) and conversion and extrapolation will lead to extra uncertainty. Therefore, it was decided to use the estimated reproducibility calculated with the Horwitz equation for evaluation of the test results. This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation.
- <u>Total Sulfur</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 ASTM D5453:19a.
- Water:This determination was not problematic. Two statistical outliers were<br/>observed. The calculated reproducibility after rejection of the statistical<br/>outliers is in full agreement with the requirements of ASTM D6304-A:20.<br/>A new version of ASTM D6304 was published in 2020 with major changes.<br/>In the 2016 version one precision statement was mentioned for test results<br/>based on mass with a broad application range and one based on volume.<br/>In the 2020 version all precision statements are based on mass with three<br/>different procedures (A direct injection, B oven accessory and<br/>C evaporation accessory) each with a different application range. In<br/>ASTM D6304:20 the reproducibilities for all three procedures A, B and C<br/>are much stricter compared to ASTM D6304:16e1.

<u>Simulated Distillation</u>: Only two participants reported test results and therefore the test results were not further evaluated.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method 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 estimated using the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Color Saybolt		18	16.4	2.1	1.2
Density at 15°C	kg/L	34	0.7470	0.0010	0.0020
Distillation at 760 mmHg					
Initial Boiling Point	°C	13	34.3	7.1	7.6
5% recovered	°C	13	60.3	3.6	6.7
10% recovered	°C	13	71.4	2.9	3.8
50% recovered	°C	13	126.7	3.5	4.8
90% recovered	°C	13	252.1	17.9	6.7
95% recovered	°C	6	292.7	38.0	(12.6)
Final Boiling Point	°C	12	308.7	10.6	4.4
Methanol	mg/kg	4	40.0	35.1	(10.3)
Total Mercury	µg/kg	16	138.4	65.4	83.5
Total Sulfur	mg/kg	16	33.8	6.9	8.1
Water	mg/kg	26	48.4	32.5	36.0
Simulated Distillation					
Initial Boiling Point	°C	2	<36	n.e.	n.e.
5% recovered	°C	2	<36	n.e.	n.e.
10% recovered	°C	2	50.5	n.e.	n.e.
50% recovered	°C	2	122.0	n.e.	n.e.
90% recovered	°C	2	258.3	n.e.	n.e.
95% recovered	°C	2	294.0	n.e.	n.e.
Final Boiling Point	°C	2	389.5	n.e.	n.e.

 Table 3: reproducibilities of tests on sample #21230

For results between brackets no z-scores are calculated

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

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2021 WITH PREVIOUS PTS

	November 2021	November 2020	November 2019	November 2018	November 2017
Number of reporting laboratories	35	33	32	32	42
Number of test results	257	229	236	263	333
Number of statistical outliers	14	10	15	18	19
Percentage of statistical outliers	5.4%	4.4%	6.4%	6.8%	5.7%

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 to the requirements of the reference test methods. The conclusions are given the following table.

Determination	November 2021	November 2020	November 2019	November 2018	November 2017
Color Saybolt	-	-		-	
Density at 15°C	+	++	+	+	++
Distillation at 760 mmHg	+/-	-	-	+/-	-
Methanol	()	n.e.	n.e.	n.e.	-
Total Mercury	+	-	+/-		+
Total Sulfur	+	-	-	+/-	-
Water	+/-	++	++	++	++
Simulated Distillation	n.e.				+/-

Table 5: comparison determinations against the reference test methods

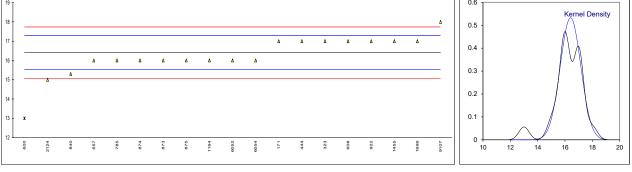
Results between brackets should be used with due care

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

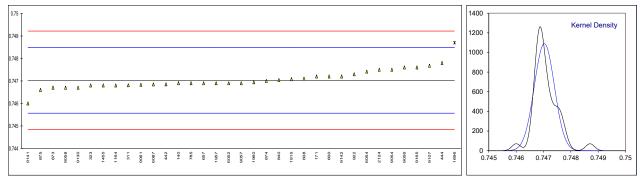
Determination	of Color	Covhalt on	aamala	#01000.
Determination		Saybull UII	Sample	#ZIZ30

	mination of Color				
lab	method	value	mark	z(targ)	remarks
140	D				
171	D6045	17		1.34	
311	500/5				
323	D6045	17		1.34	
442	500/5				
444		17	0(0.04)	1.34	
600		13	G(0.01)	-7.69	
608		17		1.34	
657		16		-0.92	
785	D6045	16	0	-0.92	First reported 14.1
840		15.3	С	-2.50	First reported 14.1
873	D6045 D6045	16 16		-0.92 -0.92	
	D6045 D6045	16 17		-0.92 1.34	
1164				-0.92	
		16 17			
1455 1696		17 17		1.34 1.34	
	D0045				
1815 1957					
1960					
2124	D6045	 15		-3.17	
	D6045	16		-0.92	
6052		16		-0.92	
6087	00040			-0.52	
9054					
9055					
9056					
9057					
9058					
9061					
9100					
9107	D156	18		3.60	
9130					
9141					
9142					
9143					
9160					
9161					
9162					
9163					
9164					
9165					
	normality	OK			
	n	18			
	outliers	1			
	mean (n)	16.41			
	st.dev. (n)	0.749			
	R(calc.)	2.10			
	st.dev.(D6045:20)	0.443			
	R(D6045:20)	1.24			
<sup>19</sup> T					0.6
					Kernel Density



# Determination of Density at 15°C on sample #21230; results in kg/L

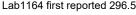
lak	moth od	value	monte	-(40	
		value	mark	z(targ)	remarks
140	D4052	0.7469		-0.18	
171		0.7472		0.23	
	D4052	0.74681	0	-0.30	Einstein eiter d. 7400 Jun (m.3
	D4052	0.7468	С	-0.31	First reported 7468 kg/m <sup>3</sup>
	IP365	0.74685		-0.25	
	D4052	0.7478		1.06	
	D4052	0.7472		0.23	
	D4052	0.7471		0.10	
	D4052	0.7469		-0.18	
	D4052	0.7469		-0.18	
	D4052	0.74704		0.01	
	D4052	0.7467		-0.45	
	D4052	0.7470		-0.04	
	D4052	0.7466		-0.59	
	D4052	0.7473		0.37	
	D4052	0.7468		-0.31	
	D4052	0.7468	D(0.01)	-0.31	
	D4052	0.7487	R(0.01)	2.29	
	ISO12185	0.74708		0.07	
	D4928	0.7469		-0.18	
	D4052	0.746941		-0.12	
	D4052	0.7475		0.65	
	D4052	0.7469	0	-0.18	First reported 747.40 without unit
	D4052 D4052	0.74742 0.746844	С	0.54	First reported 747.42 without unit
	D4052 D4052	0.746644		-0.25 0.65	
9054 9055	D4052	0.7475			
	In house	0.7476		0.78	
9057		0.7469		-0.18	
9058	D3002	0.7469		-0.18	
9061	D5002	0.74683		-0.43	
9100	D3002			-0.27	
	D4052	0.74768		0.89	
		0.7467		-0.45	
	D1298	0.7460		-1.41	
	D1298	0.7472	С	0.23	First reported 750.0 kg/m <sup>3</sup>
9143	01200		U U		
9160					
9161					
9162					
9163					
9164					
	D1298	0.7476		0.78	
	normality	ОК			
	n	34			
	outliers	1			
	mean (n)	0.74703			
	st.dev. (n)	0.000366			
	R(calc.)	0.00103			
	st.dev.(D4052:18a)	0.000730			
	R(D4052:18a)	0.00204			
	· · · · /				

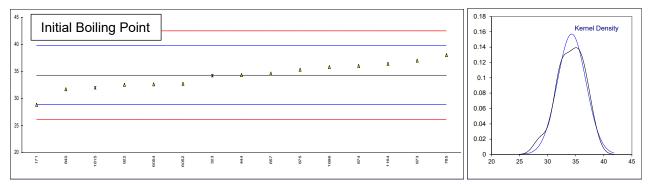


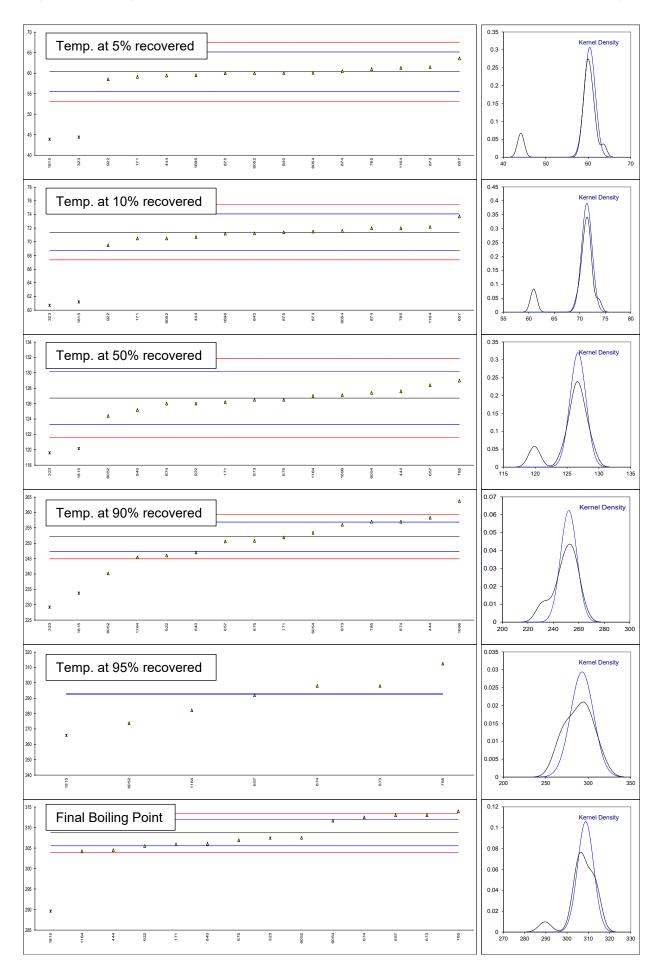
#### Determination of Distillation at 760 mmHg on sample #21230; results in °C

	method	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP	residue (%V/V)	loss (%V/V)
140 171 311	D86-A	28.8	 59.1	70.5	 126.2	 251.9	 	305.9	1.3	1.2
	D86-A	34.2 ex		60.7 DG1	119.6 DG1	229.2 ex		307.4 ex	1.3	4.7
	D86-A	34.3	59.4 	70.7	127.6 	258.3 		304.5	1.3	4.8
	D86-A	 34.6	 63.6	 73.7	 128.4	 250.6	 291.9	 313.0	 1.3	 1.2
840	D86-M D86-A	38.0 31.68	61.0 60.03	72.0 71.24	129.0 125.17	257.0 247.01	312.5	314.0 306.03	1.4 1.3	1.7 3.2
874	D86-M D86-M D86-A	37.0 36.0 35.3	61.5 60.5 60.0	71.5 72.0 71.4	126.5 126.0 126.5	256.0 257.0 250.8	298.0 298.0	313.0 312.5 306.9	1.6 1.6 C 1.3	2.2 1.6 2.7
922 1164	D86-M D86-A	32.5 36.4	58.5 61.3	69.5 72.2	126.0 127.0	246.0 245.4	 282.2	305.5 304.2 C	1.5 3.0	3.5 0.3
1455 1696 1815	D86-A	 35.8 31.95 ex	 59.5 43.85 DG1	 71.2 61.20 DG1	 127.1 120.15 DG1	 263.7 233.75 ex	  265.85 ex	  289.60 G1	  2.20	  3.40
1957 1960										
	D86-A	 32.7	60 60 1	 70.5	 124.4	 240.2	 273.7	 307.5	 1.3	 1.9
6054 6087 9054	D86-A	32.6 	60.1 	71.6 	127.4 	253.4 	 	311.7 	1.3 	4.0 
9055 9056										
9057 9058						 				
9061 9100 9107				 						 
9130 9141										
9142 9143										
9160 9161 9162					 					
9163 9164										
9165										
	normality n	OK 13	not OK 13	suspect 13	OK 13 2	OK 13	unknown 6	OK 12		
	outliers mean (n) st.dev. (n)	0 (+2ex) 34.28 2.540	2 60.35 1.299	2 71.39 1.020	2 126.71 1.247	0 (+2ex) 252.10 6.400	0 (+1ex) 292.72 13.565	1 (+1ex) 308.73 3.769		
	R(calc.) st.dev.(D86-M:20b) R(D86-M:20b)	7.11 2.731 7.65	3.64 2.405 6.73	2.86 1.343 3.76	3.49 1.716 4.80	17.92 2.395 6.71	37.98 (4.501) (12.60)	10.55 1.587 4.44		
	Compare R(D86-A:20b)	1.89	1.81	1.57	3.0	3.78	(6.27)	7.1		

Ex = excluded due to observed outliers in other Distillation at 760 mmHg parameters Lab 874 first reported 96.5 Lab1164 first reported 296.5







# Determination of Methanol on sample #21230; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140					
171 311	D7423	30.0			
323	INH-304	 30			
442					
444					
600 608					
	INH-0103	43.9			
785					
840					
873 874					
875					
922					
1164 1455	D7423	 56			
1696	D7423				
1815					
1957					
1960 2124					
6052					
6054					
6087 9054					
9054 9055					
9056					
9057					
9058 9061					
9100					
9107					
9130 9141					
9142					
9143					
9160					
9161 9162					
9163					
9164					
9165					
	normality	unknown			
	n	4			
	outliers	0			
	mean (n) st.dev. (n)	39.97 12.533			
	R(calc.)	35.09			
	st.dev.(Horwitz)	(3.671)			
	R(Horwitz)	(10.28)			
60					]
<sup>60</sup> T					۵
50 -					
40 -					Δ
30 -	۵		۵		
20 -					
10 -					
۰ L	171		323		667
	÷-		e4		۵ <del>۲</del>

# Determination of Total Mercury on sample #21230; results in $\mu$ g/kg

				_//		
	method	value	mark	z(targ)	remarks	
140 171 311	UOP938 UOP938	 111 175		-0.92 1.23		
323 442 444	UOP938	135  230.6	G(0.05)	-0.11  3.09		
600 608 657		151.81  114		0.45  -0.82		
785 840 873	UOP938	 118.12 160		 -0.68 0.72		
874 875 922	UOP938	163 		0.82 		
1164 1455	In house	162		0.79		
1696 1815		114.77  25.067	C C(0.05)	-0.79  2.77	First reported 19 775	
1957 1960 2124 6052	UOP938	25.967 129.035 143.96 	C,G(0.05)	-3.77 -0.31 0.19 	First reported 18.775	
6054 6087 9054 9055	UOP938 UOP938	 171.1201 143.9 		1.10 0.18 		
9056 9057 9058 9061		 110 112.1 		-0.95 -0.88		
9100 9107 9130				 		
9141 9142 9143		 		 		
9160 9161 9162				 		
9163 9164 9165				 		
	normality n	OK 16				
	outliers mean (n) st.dev. (n) R(calc.)	2 138.426 23.3711 65.439				
	st.dev.(Horwitz) R(Horwitz) Compare R(UOP938:20)	29.8261 83.513 12.999				
<sup>250</sup> T	· · ·					0.018
200 -					x	0.016 - Kernel Density
150 -		Δ	ΔΔ	۵		0.012 - 0.01 -
100 -	Δ Δ Δ Δ	Δ Δ Ξ				0.008 -

x

0.006 0.004

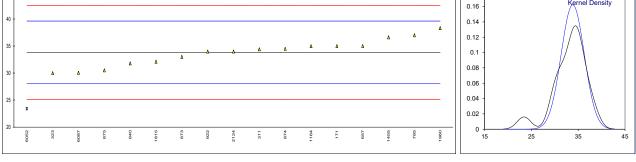
0.002

 0 <del>|</del> -100

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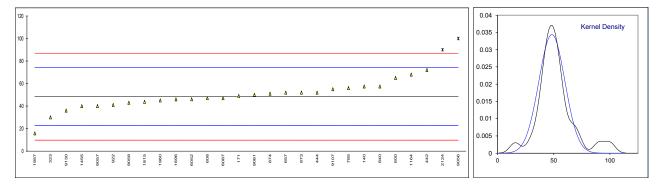
## Determination of Total Sulfur on sample #21230; results in mg/kg

	-				<u> </u>		
lab	method	value	mark	z(targ)	remarks		
140	DE 450						
171	D5453	35		0.40			
311	D5453	34.4		0.20			
	D5453	30		-1.32			
442							
444							
600							
608	DE (50						
657		35		0.40			
	D4294	37		1.09			
	D5453	31.8		-0.70			
	D4294	33		-0.29			
	D2622	34.5		0.23			
	D4294	30.5		-1.15			
	D4294	34.0	0	0.06	First way arts d 00.40		
1164		34.98	С	0.40	First reported 26.12		
	D5453	36.6		0.95			
1696	DE452						
1815	D5453	32.1		-0.60			
1957	DE4E2			 1 EE			
		38.327		1.55			
2124		34		0.06			
	D5453	23.415	G(0.05)	-3.59			
6054	DE 450						
	D5453	30.0464		-1.30			
9054							
9055							
9056							
9057							
9058							
9061							
9100							
9107							
9130							
9141 9142							
9142							
9143 9160							
9161 9162							
9162							
9163							
9165							
9105							
	normality	ОК					
	n	16					
	outliers	10					
	mean (n)	33.828					
	st.dev. (n)	2.4592					
	R(calc.)	6.886					
	st.dev.(D5453:19a)	2.9041					
	R(D5453:19a)	8.131					
	(LOTO. 104)	5.101					
<sup>45</sup>						0.18	Kernel Danalta
						0.16 -	Kernel Density
40 -						0.14 -	



# Determination of Water on sample #21230; results in mg/kg

lah	method	value	mark	z(tara)	romarks
140	method D4928	57.40	mark	z(targ) 0.70	remarks
171		49		0.05	
311	2000111				
	E1064	30		-1.43	
442	IP438	72		1.84	
444	D6304-A:20	52		0.28	
	D6304-A:20	65		1.29	
	D6304	47		-0.11	
	D6304-A:16e1	52		0.28	
	D6304-A	56 57 4		0.59	
	D6304-A:20 D6304-A:20	57.4 52		0.70 0.28	
874		52 51		0.20	
875	D0004-A.20				
	D6304-A:20	41		-0.57	
1164		68		1.53	
1455	ISO12937	40		-0.65	
1696	D6304-A:20	45.96		-0.19	
	ISO12937	43.73		-0.36	
	D4928	15.7		-2.54	
	D4928	45		-0.26	
	D6304-A:16e1	90 46.03	R(0.05)	3.23 -0.18	
6052 6054	D6304-A:16e1	40.03		-0.16	
	D4928	47.0		-0.11	
9054	D-1020				
9055					
9056	In house	100	R(0.05)	4.01	
9057		40		-0.65	
9058		43		-0.42	
	D4928	50.0		0.13	
9100	D0004 0 00				
9107 9130	D6304-C:20 D6304-A:20	55 26		0.52	
9130	D0304-A.20	36		-0.96	
	D4006				Reported 0.025 %vol/vol
9143	D-1000				
9160					
9161					
9162					
9163					
9164	<b>D</b> (000				
9165	D4006				Reported 0.025 %V/V
	normality	suspect			
	n	26			
	outliers	2			
	mean (n)	48.355			
	st.dev. (n)	11.6000			
	R(calc.)	32.480			
	st.dev.(D6304-A:20)	12.8743			
	R(D6304-A:20)	36.048			



## Determination of Simulated Distillation on sample #21230; results in °C

lab	method	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP
140								
171	D2887	18.0	30.0	52.0	119.0	253.5	294.0	391.0
311								
323								
442								
444								
600								
608								
657								
785								
840								
873								
874								
875								
922								
1164								
1455								
1696								
1815								
1957								
1960								
2124								
6052								
6054								
6087								
9054								
9055								
9056								
9057								
9058								
9061								
9100								
9107								
9130	D2887	<36	<36	49	125	263	294	388
9141	2200.							
9142								
9143								
9160								
9161								
9162								
9163								
9164								
9165								
0100								
	n	2	2	2	2	2	2	2
	mean (n)	2 <36	<36	2 50.5	122.0	258.3	294.0	2 389.5
	mean (n)	~30	<b>~</b> 50	50.5	122.0	200.0	294.0	009.0

z-scores of Determination of Distillation at 760 mmHg

lab	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP
140							
171	-2.01	-0.52	-0.66	-0.30	-0.08		-1.78
311							
323	-0.03	-6.63	-7.96	-4.15	-9.56		-0.84
442							
444	0.01	-0.39	-0.51	0.52	2.59		-2.66
600							
608							
657	0.12	1.35	1.72	0.98	-0.63		2.69
785	1.36	0.27	0.46	1.33	2.05		3.32
840	-0.95	-0.13	-0.11	-0.90	-2.13		-1.70
873	0.99	0.48	0.08	-0.12	1.63		2.69
874	0.63	0.06	0.46	-0.42	2.05		2.38
875	0.37	-0.14	0.01	-0.12	-0.54		-1.15
922	-0.65	-0.77	-1.41	-0.42	-2.55		-2.03
1164	0.78	0.40	0.60	0.17	-2.80		-2.85
1455							
1696	0.56	-0.35	-0.14	0.23	4.84		
1815	-0.85	-6.86	-7.58	-3.82	-7.66		-12.05
1957							
1960							
2124							
6052	-0.58	-0.14	-0.66	-1.35	-4.97		-0.77
6054	-0.62	-0.10	0.16	0.40	0.54		1.87
6087							
9054							
9055							
9056							
9057							
9058							
9061							
9100							
9107							
9130							
9141							
9142							
9143							
9160							
9161							
9162							
9163							
9164							
9165							
0100							

#### Number of participants per country

- 3 labs in AUSTRALIA
- 1 lab in BELGIUM
- 1 lab in COTE D'IVOIRE
- 1 lab in INDONESIA
- 4 labs in MALAYSIA
- 4 labs in NETHERLANDS
- 10 labs in NIGERIA
- 2 labs in NORWAY
- 1 lab in PAKISTAN
- 1 lab in POLAND
- 4 labs in RUSSIAN FEDERATION
- 1 lab in SINGAPORE
- 2 labs in UNITED ARAB EMIRATES
- 5 labs in UNITED KINGDOM
- 2 labs in UNITED STATES OF AMERICA
- 1 lab in VIETNAM

#### Abbreviations

С	= final test result after checking of first reported suspect test result
D(0.01), D1	= outlier in Dixon's outlier test
D(0.05), D5	= straggler in Dixon's outlier test
G(0.01), G1	= outlier in Grubbs' outlier test
G(0.05), G5	= straggler in Grubbs' outlier test
DG(0.01), DG1	= outlier in Double Grubbs' outlier test
DG(0.05), DG5	= 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	= 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
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

#### Literature

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
- 5 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
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- 8 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
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- 11 W. Horwitz and R. Albert, J. AOAC Int, <u>79.3</u>, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, <u>25(2)</u>, 165-172, (1983)