

Results of Proficiency Test  
Gas condensate  
November 2014

Organised by: Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## **1 INTRODUCTION**

Since 2008, the Institute for Interlaboratory Studies organizes a proficiency test for Gas condensate every year. During the annual proficiency testing program 2014/2015, it was decided to continue the round robin for the analysis of Gas condensate. In this interlaboratory study, 41 laboratories from 17 different countries have participated. See appendix 3 for the number of participating laboratories per country. In this report, the results of the 2014 Gas condensate proficiency test are presented and discussed. This report is also electronically available through the iis internet site [www.iisnl.com](http://www.iisnl.com).

## **2 SET UP**

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to send 1 sample of Gas condensate (1\* 0.5 L bottle labelled #14216). Participants were requested to report rounded and unrounded results. The unrounded 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

### **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**

The necessary bulk material, approximately 100 litre was obtained from a participating laboratory. After homogenisation, 98 amber glass bottles of 0.5 litre were filled and labelled as sample #14216

The homogeneity of the subsamples #14216 was checked by determination of Density @15°C in accordance with ASTM D4052:11 on 8 stratified randomly selected samples.

	Density at 15 °C in kg/m <sup>3</sup>
Sample #14216-1	731.84
Sample #14216-2	731.89
Sample #14216-3	731.91
Sample #14216-4	731.89
Sample #14216-5	731.87
Sample #14216-6	731.97
Sample #14216-7	731.91
Sample #14216-8	731.86

Table 1: homogeneity test results of subsamples #14216

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 15 °C in kg/m <sup>3</sup>
r sample	0.11
reference method	ASTM D4052:11
0.3xR(reference method)	0.76

Table 2: repeatability of subsamples #14216

The calculated repeatability was less than 0.3 times the reproducibility of the respective reference method. Therefore, homogeneity of the subsamples #14216 was assumed.

To each of the participating laboratories, 1 \* 0.5 L bottle (labelled #14216) was sent on October 22, 2014.

## 2.5 STABILITY OF THE SAMPLES

The stability of Gas condensate, packed in the brown glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

## 2.6 ANALYSES

The participants were requested to determine on sample #14216: Color Saybolt (Automated and Manual), Density @ 15°C, Distillation (IBP, 5%, 10%, 50%, 90%, 95% recovered and FBP), Mercury, total Sulphur and Water by KF.

To get maximum information for the statistical calculations, the participants were requested to report unrounded results and results below the usual lower reporting limits, where possible.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal [www.kpmd.co.uk/sgs-iis/](http://www.kpmd.co.uk/sgs-iis/). The detailed report form was also made available for download on the iis website [www.iisnl.com](http://www.iisnl.com).

### 3 RESULTS

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

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported results at that moment. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

#### 3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or 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 (ref. 14). 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. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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 analysis results are plotted. The corresponding laboratory numbers are under the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. 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 reproducibilities, the z-scores were calculated using a target standard deviation. The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

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

The  $z_{(\text{target})}$  scores are listed in the result tables in appendix 1.

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 the fit-for-useness of the reported test result.

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, problems were encountered during the transport of the samples to the laboratories in Malaysia and Nigeria. Some samples took an unexpected long time to reach the laboratory due to several problems with custom clearance and/or transport companies. Five participants reported test results after the final reporting date and five laboratories did not report any test results at all. In total 36 laboratories reported 251 numerical results. Observed were 8 outlying results, which is 3.2%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

### 4.1 EVALUATION PER TEST

In this section, the results are discussed per test.

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 4.4.

Color Saybolt: Both the automated version (ASTM D6045) and the manual version were evaluated. Around 10 participants returned a test result for either or both tests. This limited number of results may have some effect on the statistical evaluation.

The automated determination was very problematic. 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 D6045. The manual determination was also very problematic. No statistical outliers were observed. However the calculated reproducibility is not at all in agreement with the requirements of ASTM D6045.

Density @15°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4052:11. It should be taken into account that the reproducibility from ASTM D4052:11 is applicable to petroleum distillates and viscous oils only. Therefore no precision data are stated in the 2011 version for Gas condensates. However, Gas condensates 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.

Distillation: This determination was very problematic. In total three statistical outliers were observed. After rejection of the statistical outliers none of the calculated reproducibilities is in agreement with the requirements for automated or manual mode of ASTM D86:12, except for temperatures at 5% and 50% recovered (manual mode). The mean values for the temperature at 95% recovered and final boiling point are outside of the range for which the reproducibility is valid according to the method. For these two data sets, no z-scores were calculated.

It should be noted that the scope of ASTM D86 does not include Gas condensates, 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.

Mercury: The precision requirements of UOP938 (table 3b) are extremely strict and as they are 6 – 7 times more strict than the Horwitz estimate, these requirements will not be met easily. Also, the reproducibility of UOP938 is only available for very low concentrations (0.28 and 12.14  $\mu\text{g/L}$ , table B3) and conversion and extrapolation up to 24  $\mu\text{g/kg}$  will lead to extra uncertainty. Therefore, it was decided to use the Horwitz estimates for evaluation of the test results in this report.

This determination appeared to be problematic at a consensus of 24.46  $\mu\text{g Hg/kg}$ . No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

Sulphur: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D5453:12.

Water: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D6304:07. It must be noted that the precision data of ASTM D4928 are not applicable at this low concentration (valid between 0.02 – 5.00%M/M).



## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results of sample #14216, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM standards) are compared in the next table.

Parameter	unit	n	mean	2.8 * sd	R (lit)
Color Saybolt (Automated)		9	18.1	4.3	1.2
Color Saybolt (Manual)		9	17.9	4.9	2.0
Density @ 15°C	kg/m <sup>3</sup>	32	732.3	1.8	2.5
Initial Boiling Point	°C	17	32.5	7.1	5.0
5%-recovered	°C	16	54.6	8.2	3.2
10%-recovered	°C	16	63.6	6.8	3.2
50%-recovered	°C	16	113.0	3.1	1.9
90%-recovered	°C	17	225.3	24.1	5.4
95%-recovered	°C	14	267.9	45.7	(6.2)*
Final Boiling Point	°C	16	289.1	14.8	(6.8)*
Mercury as Hg	µg/kg	18	24	25	19
Sulphur	mg/kg	14	38.1	16.1	8.9
Water content by KF	mg/kg	26	43	24	162

Table 3: performance evaluation sample #14216

\*) mean of test was outside reproducibility range

Without further statistical calculations it can be concluded from the overview given in table 3 that for almost all tests there is not a good compliance of the group of participants with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2014 WITH THE PREVIOUS PTS

	<i>November 2014</i>	<i>November 2013</i>	<i>November 2012</i>	<i>November 2011</i>	<i>November 2010</i>
Number of reporting participants	36	36	38	41	25
Number of results reported	251	216	234	283	215
Number of statistical outliers	8	15	25	29	25
Percentage of statistical outliers	3.2%	6.9%	10.7%	10.2%	11.6%

Table 4: comparison with previous proficiency tests

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

Determination	<i>November 2014</i>	<i>November 2013</i>	<i>November 2012</i>	<i>November 2011</i>	<i>November 2010</i>
Color Saybolt	--	-	--	--	--
Density @ 15 °C	+	++	-	++	-
Distillation (ASTM D86)	--	--	--	--	--
Mercury as Hg	-	--	--	-	--
Sulphur	--	--	++	--	+
Water content by KF	++	++	-	-	-
Total vapour pressure	n.e.	n.e.	-	-	--
DVPE acc. to ASTM D5191	n.e.	n.e.	+/-	+	--

Table 5: comparison of the performance per determination against the target requirements

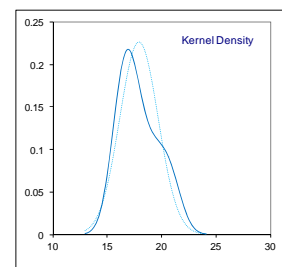
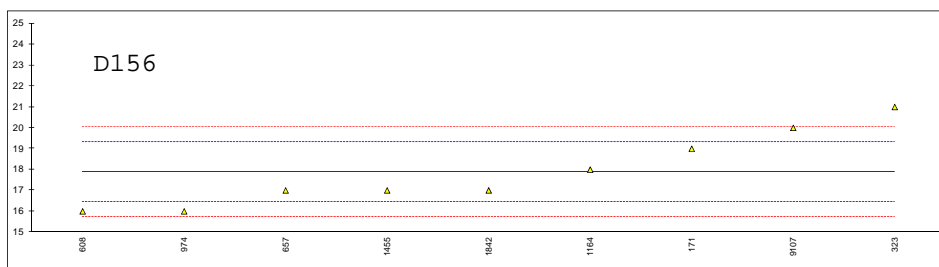
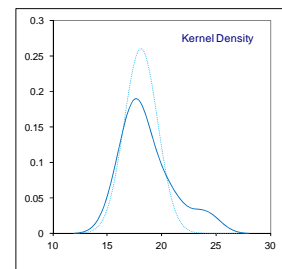
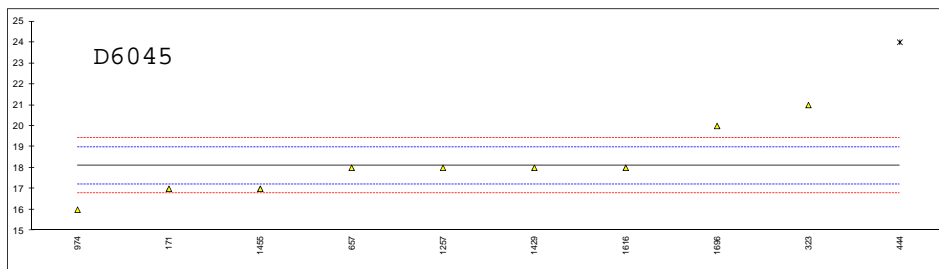
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

**APPENDIX 1**

**Determination of Color Saybolt (automated and manual) on sample #14216;**

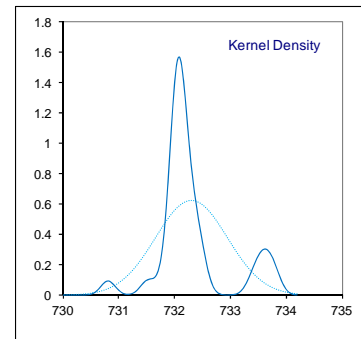
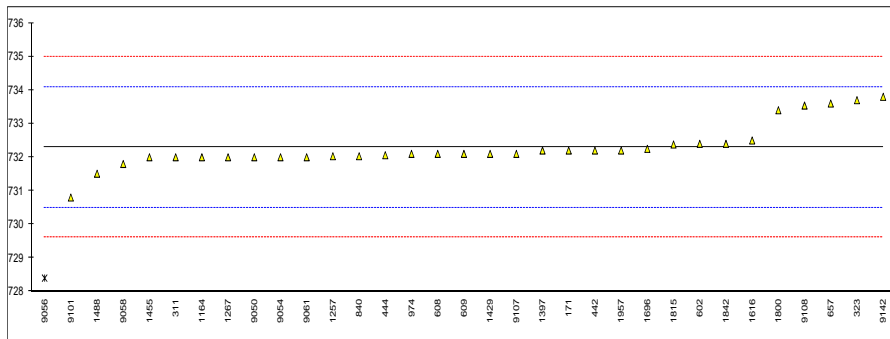
lab	method	value	mark	z(targ)	method	value	mark	z(targ)	remarks
171	D6045	17		-2.51	D156	19		1.56	
311		----		----		----		----	
323	D6045	21		6.52	D156	21		4.36	
442		----		----		----		----	
444	D6045	24.0	G(0.05)	13.30		----		----	
602		----		----		----		----	
608		----		----	D156	16		-2.64	
609		----		----		----		----	
657	D6045	18		-0.25	D156	17		-1.24	
840		----		----		----		----	
974	D6045	16		-4.77	D156	16		-2.64	
1164		----		----	D156	18		0.16	
1257	D6045	18		-0.25		----		----	
1267		----		----		----		----	
1397		----		----		----		----	
1429	D6045	18		-0.25		----		----	
1455	D6045	17		-2.51	D156	17		-1.24	
1488		----		----		----		----	
1616	D6045	18		-0.25		----		----	
1696	D6045	20		4.27		----		----	
1780		----		----		----		----	
1800		----		----		----		----	
1815		----		----		----		----	
1842		----		----	D156	17		-1.24	
1879		----		----		----		----	
1957		----		----		----		----	
1960		----		----		----		----	
9050		----		----		----		----	
9054		----		----		----		----	
9055		----		----		----		----	
9056		----		----		----		----	
9057		----		----		----		----	
9058		----		----		----		----	
9061		----		----		----		----	
9101		----		----		----		----	
9107		----		----	D156	20		2.96	
9108		----		----		----		----	
9142		----		----		----		----	
9143		----		----		----		----	
9150		----		----		----		----	
9153		----		----		----		----	
normality		OK			normality	OK			
n		9			n	9			
outliers		1			outliers	0			
mean (n)		18.11			mean (n)	17.89			
st.dev. (n)		1.537			st.dev. (n)	1.764			
R(calc.)		4.30			R(calc.)	4.94			
R(D6045:12)		1.24			R(D156:12)	2.00			



Determination of Density @ 15°C on sample #14216; results in kg/m<sup>3</sup>

lab	method	value	mark	z(targ)	remarks
171	D4052	732.2		-0.11	
311	D4052	732.0		-0.33	
323	D4052	733.7		1.57	
442	IP365	732.2		-0.11	
444	D4052	732.06		-0.26	
602	D1298	732.4		0.12	
608	D4052	732.1		-0.22	
609	D5002	732.1		-0.22	
657	D4052	733.6		1.45	
840	D4052	732.03		-0.30	
974	D4052	732.1		-0.22	
1164	D4052	732.0		-0.33	
1257	D4052	732.03	C	-0.30	First reported: 0.73203
1267	D4052	732		-0.33	
1397	D4052	732.2		-0.11	
1429	D4052	732.1		-0.22	
1455	D4052	732.0		-0.33	
1488	D1298	731.51		-0.88	
1616	D4052	732.5		0.23	
1696	D1298	732.25		-0.05	
1780		-----		-----	
1800	D4052	733.4	C	1.23	First reported: 0.7349 kg/L
1815	ISO12185	732.38		0.09	
1842	D4052	732.4		0.12	
1879		-----		-----	
1957	D5002	732.2		-0.11	
1960		-----		-----	
9050	ISO12185	732.0		-0.33	
9054	D4052	732.0		-0.33	
9055		-----		-----	
9056	in house	728.4	G(0.01)	-4.35	Comment of lab: "weighed in wt/ml, no suitable hydrometer available"
9057		-----		-----	
9058	D4052	731.8		-0.55	
9061	D5002	732.0		-0.33	
9101	D1298	730.8		-1.67	
9107	D4052	732.1		-0.22	Reported 0.7321 kg/L
9108	D4052	733.54		1.39	Reported 0.73354 kg/L
9142	D1298	733.8		1.68	
9143		-----		-----	
9150		-----		-----	
9153		-----		-----	

normality suspect  
n 32  
outliers 1  
mean (n) 732.30  
st.dev. (n) 0.645  
R(calc.) 1.81  
R(D4052:11) 2.51



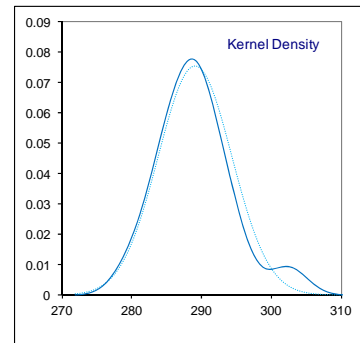
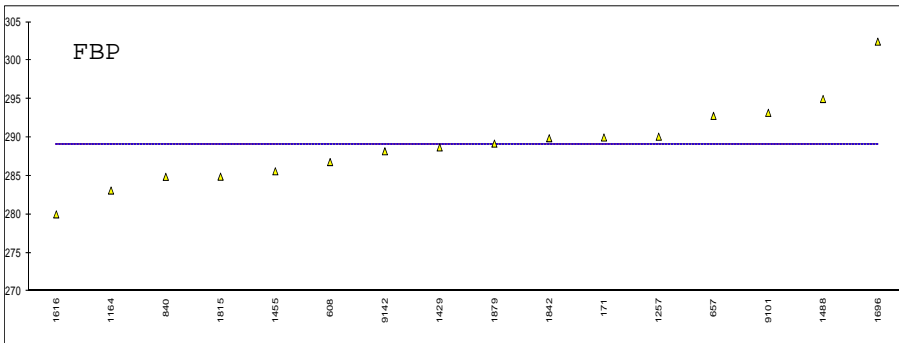
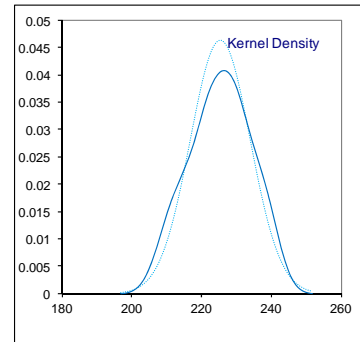
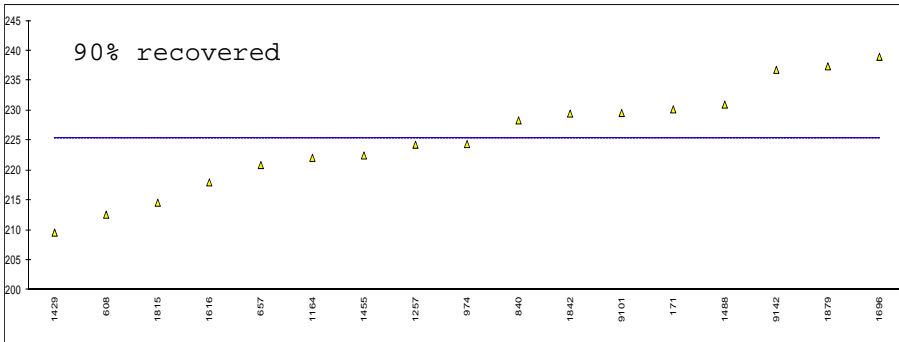
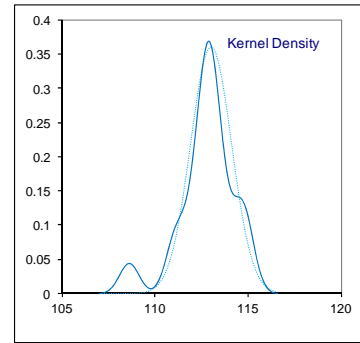
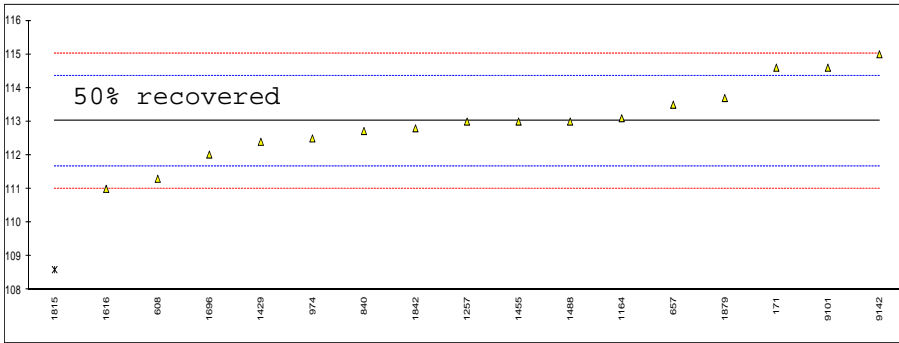
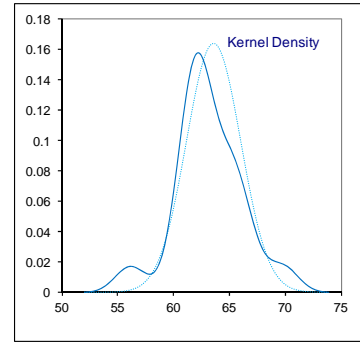
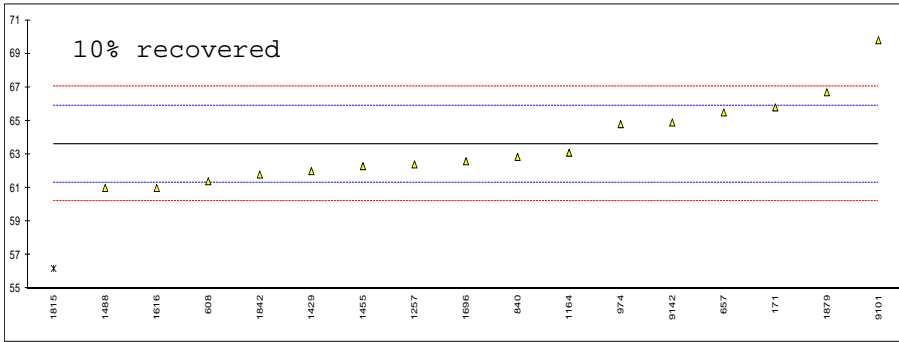
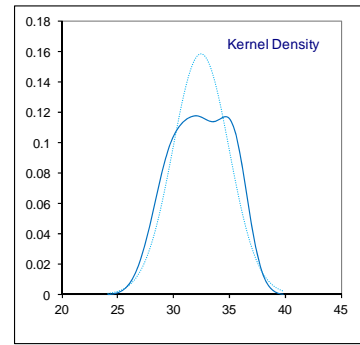
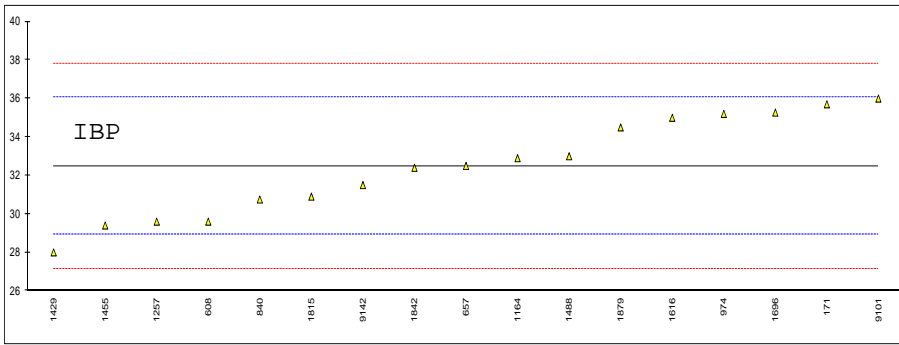
## Determination of Distillation on sample #14216; results in °C and %V/V

lab	method	mode	IBP	5% <sub>rec</sub>	10% <sub>rec</sub>	50% <sub>rec</sub>	90% <sub>rec</sub>	95% <sub>rec</sub>	FBP	loss	res
171	D86	Automated	35.7	57.1	65.8	114.6	230.2	277.2	290.0	1.5	2.4
311			----	----	----	----	----	----	----	----	----
323			----	----	----	----	----	----	----	----	----
442			----	----	----	----	----	----	----	----	----
444			----	----	----	----	----	----	----	----	----
602			----	----	----	----	----	----	----	----	----
608	D86	Automated	29.6	52.3	61.4	111.3	212.6	248.5	286.8	1.2	0.4
609			----	----	----	----	----	----	----	----	----
657	D86	Automated	32.5	56.9	65.5	113.5	220.9	257.7	292.8	1.0	1.1
840	D86	Automated	30.75	53.78	62.85	112.72	228.36	----	284.88	3.0	1.2
974	D86	Automated	35.2	56.4	64.8	112.5	224.4	----	----	----	----
1164	D86	Automated	32.9	54.3	63.1	113.1	222.1	260.8	283.1	0.0	3.0
1257	D86	Automated	29.6	53.1	62.4	113.0	224.3	265.5	290.1	1.3	0.8
1267			----	----	----	----	----	----	----	----	----
1397			----	----	----	----	----	----	----	----	----
1429	D86	Automated	28.0	52.5	62.0	112.4	209.6	244.8	288.7	1.2	1.4
1455	D86	Automated	29.4	52.5	62.3	113.0	222.5	267.0	285.6	2.4	1.2
1488	D86	Manual	33	52	61	113	231	274	295	0.6	1.4
1616	D86	Manual	35.0	52.0	61.0	111.0 <i>fr</i>	218.0	254.0	280.0	1.0	1.0
1696	D86		35.27	51.46	62.59	112.02	238.99	300.13	302.45	4.99	0.90
1780			----	----	----	----	----	----	----	----	----
1800			----	----	----	----	----	----	----	----	----
1815	ISO3405	Automated	30.9	<b><u>43.1</u></b>	<b><u>56.2</u></b>	<b><u>108.6</u></b>	214.6	251.6	284.9	3.1	1.6
1842	D86	Automated	32.4	52.5	61.8	112.8	229.5	281.6	289.9	3.0	1.2
1879	INH-2177	Automated	34.5	58.0	66.7	113.7	237.4	290.1	289.2	4.3	1.2
1957			----	----	----	----	----	----	----	----	----
1960			----	----	----	----	----	----	----	----	----
9050			----	----	----	----	----	----	----	----	----
9054			----	----	----	----	----	----	----	----	----
9055			----	----	----	----	----	----	----	----	----
9056			----	----	----	----	----	----	----	----	----
9057			----	----	----	----	----	----	----	----	----
9058			----	----	----	----	----	----	----	----	----
9061			----	----	----	----	----	----	----	----	----
9101	D86	Automated	36.0	62.0	69.8	114.6	229.6	277.4	293.2	2.4	1.3
9107			----	----	----	----	----	----	----	----	----
9108			----	----	----	----	----	----	----	----	----
9142	D86	Automated	31.5	55.9	64.9	115.0	236.8	----	288.2	3.1	1.3
9143			----	----	----	----	----	----	----	----	----
9150			----	----	----	----	----	----	----	----	----
9153			----	----	----	----	----	----	----	----	----
	normality		OK	not OK	not OK	OK	OK	OK	suspect		
	n		17	16	16	16	17	14	16		
	outliers		0	1	1	1	0	0	0		
	mean (n)		32.48	54.55	63.62	113.01	225.34	267.88	289.05		
	st.dev. (n)		2.518	2.917	2.432	1.109	8.598	16.322	5.283		
	R(calc.)		7.05	8.17	6.81	3.10	24.07	45.70	14.79		
	R(D86:12-A)		4.98	3.20	3.20	1.88	5.42	(6.23)*	(6.78)*		
	R(D86:12-B)		5.60	8.52	4.26	5.52	6.79	(12.19)	(7.20)		

**Bold and underlined test results are outliers according to Dixon/Grubbs/Rosner**

\*) Mean value of the group is outside the temperature range for which this reproducibility is valid, therefore no z-scores were calculated.

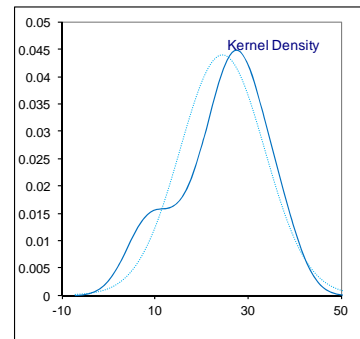
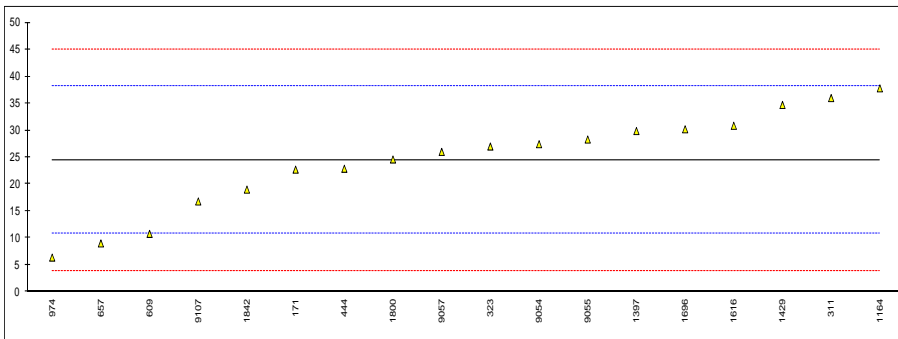
Lab 1616 first reported for 50% recovered 109.0



Determination of Mercury as Hg on sample #14216; results in µg/kg

lab	method	value	mark	z(targ)	remarks
171	UOP938	22.7		-0.26	
311	D7623	36		1.69	
323	UOP938	27		0.37	
442		----		----	
444	UOP938	22.867		-0.23	
602		----		----	
608		----		----	
609	UOP938	10.7740		-2.00	
657	UOP938	9		-2.26	
840		----		----	
974	UOP938	6.37		-2.64	
1164	UOP938	37.8		1.95	
1257		----		----	
1267		----		----	
1397	in house	29.87		0.79	
1429	in house	34.7157		1.50	
1455		----		----	
1488		----		----	
1616	UOP938	30.85		0.93	
1696	UOP938	30.2		0.84	
1780		----		----	
1800	D7623	24.59		0.02	
1815		----		----	
1842	UOP938	19.0		-0.80	
1879		----		----	
1957		----		----	
1960		----		----	
9050		----		----	
9054	UOP938	27.4299		0.43	
9055	D6722	28.3		0.56	
9056		----		----	
9057	in house	26		0.23	
9058		----		----	
9061		----		----	
9101		----		----	
9107	INH-915	16.8		-1.12	Reported 0.0168 (probably unit error, mg/kg instead of µg/kg)
9108		----		----	
9142		----		----	
9143		----		----	
9150		----		----	
9153		----		----	

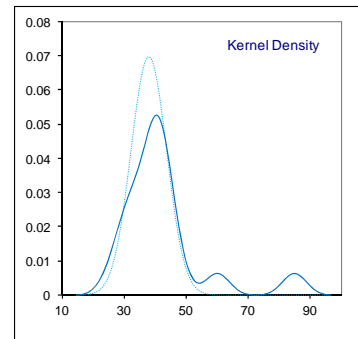
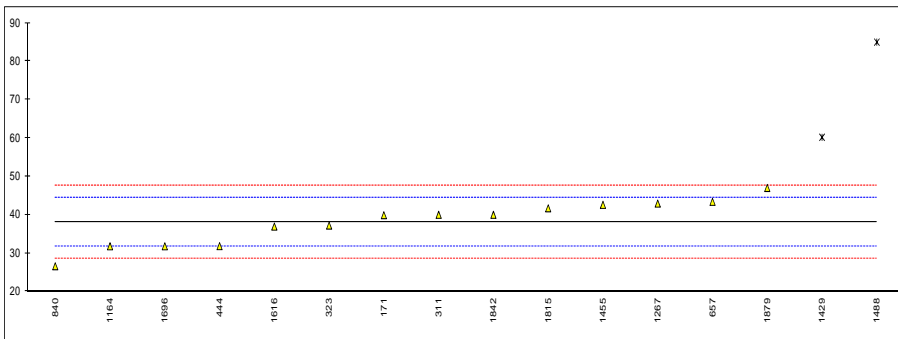
normality OK  
n 18  
outliers 0  
mean (n) 24.459  
st.dev. (n) 9.0731  
R(calc.) 25.405  
R(Horwitz) 19.155



Determination of Sulphur on sample #14216; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D5453	39.9		0.56	
311	D5453	40		0.59	
323	D5453	37.2		-0.29	
442		----		----	
444	D5453	31.83		-1.98	
602		----		----	
608		----		----	
609		----		----	
657	D5453	43.39		1.66	
840	D4294	26.6		-3.63	
974		----		----	
1164	D5453	31.8		-1.99	
1257		----		----	
1267	D4928	42.92		1.51	
1397		----		----	
1429	IP490	60.2	G(0.05)	6.95	
1455	D2622	42.6		1.41	
1488	D4294	85	C,G(0.01)	14.76	First reported 146
1616	D5453	36.93		-0.37	
1696	D5453	31.8		-1.99	
1780		----		----	
1800		----		----	
1815	D5453	41.7		1.13	
1842	D5453	40		0.59	
1879	INH-19121	47		2.80	
1957		----		----	
1960		----		----	
9050		----		----	
9054		----		----	
9055		----		----	
9056		----		----	
9057		----		----	
9058		----		----	
9061		----		----	
9101		----		----	
9107		----		----	
9108		----		----	
9142		----		----	
9143		----		----	
9150		----		----	
9153		----		----	

normality OK  
n 14  
outliers 2  
mean (n) 38.12  
st.dev. (n) 5.733  
R(calc.) 16.05  
R(D5453:12) 8.89

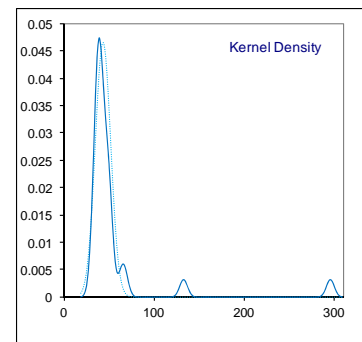
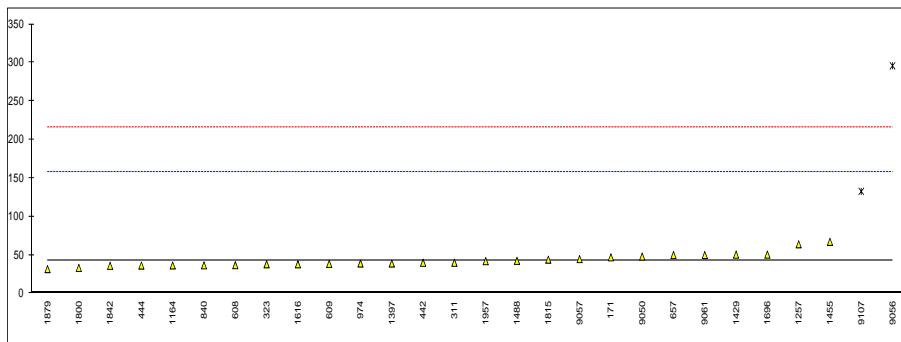




Determination of Water on sample #14216; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D6304	47.0		0.07	
311	D6304-A	40		-0.05	
323	D6304	38		-0.09	
442	IP438	40		-0.05	
444	E1064	36.3		-0.12	
602		----		----	
608	D6304	36.8		-0.11	
609	D4928	38.5		-0.08	
657	D6304-A	49.95		0.12	
840	D6304-A	36.6		-0.11	
974	D6304-A	39		-0.07	
1164	D6304-A	36.5		-0.11	
1257	D6304	64		0.36	
1267		----		----	
1397	D6304	39		-0.07	
1429	IP438	50.532		0.13	
1455	ISO12937	67.1		0.42	
1488	ISO12937	42.5		-0.01	
1616	UOP481	38		-0.09	
1696	D6304-A	50.6		0.13	
1780		----		----	
1800	D6304-A	33.27		-0.17	
1815	ISO12937	44		0.02	Reported 0.0044 (probably unit error, M/M% instead of mg/kg)
1842	D6304-A	36		-0.12	
1879	D4928	31.7		-0.20	
1957	D6304-A	42		-0.02	
1960		----		----	
9050	INH-386	48		0.09	
9054		----		----	
9055		----		----	
9056	INH-18	296	G(0.01)	4.38	
9057	in house	44.7		0.03	Reported 0.00447 (probably unit error, M/M% instead of mg/kg)
9058		----		----	
9061	D4928	50		0.12	Reported 0.005 (probably unit error, M/M% instead of mg/kg)
9101		----		----	
9107	D6304	132.7	G(0.01)	1.55	
9108		----		----	
9142		----		----	
9143		----		----	
9150		----		----	
9153		----		----	

normality not OK  
n 26  
outliers 2  
mean (n) 43.079  
st.dev. (n) 8.5738  
R(calc.) 24.007  
R(D6304:07) 161.522



## APPENDIX 2:

## Distillation z-scores

lab	IBP	5%	10%	50%	90%	95%	FBP
171	D86	1.81	2.23	1.91	2.36	2.51	----
311		----	----	----	----	----	----
323		----	----	----	----	----	----
442		----	----	----	----	----	----
444		----	----	----	----	----	----
602		----	----	----	----	----	----
608	D86	-1.62	-1.97	-1.94	-2.55	-6.59	----
609		----	----	----	----	----	----
657	D86	0.01	2.06	1.64	0.72	-2.30	----
840	D86	-0.97	-0.67	-0.67	-0.44	1.56	----
974	D86	1.53	1.62	1.03	-0.77	-0.49	----
1164	D86	0.23	-0.22	-0.46	0.13	-1.68	----
1257	D86	-1.62	-1.27	-1.07	-0.02	-0.54	----
1267		----	----	----	----	----	----
1397		----	----	----	----	----	----
1429	D86	-2.52	-1.79	-1.42	-0.92	-8.14	----
1455	D86	-1.73	-1.79	-1.16	-0.02	-1.47	----
1488	D86	0.29	-2.23	-2.29	-0.02	2.92	----
1616	D86	1.42	-2.23	-2.29	-3.00	-3.80	----
1696	D86	1.57	-2.70	-0.90	-1.48	7.05	----
1780		----	----	----	----	----	----
1800		----	----	----	----	----	----
1815	ISO3405	-0.89	<b><u>-10.02</u></b>	<b><u>-6.49</u></b>	<b><u>-6.58</u></b>	-5.55	----
1842	D86	-0.05	-1.79	-1.59	-0.32	2.15	----
1879	INH-2177	1.13	3.02	2.69	1.02	6.23	----
1957		----	----	----	----	----	----
1960		----	----	----	----	----	----
9050		----	----	----	----	----	----
9054		----	----	----	----	----	----
9055		----	----	----	----	----	----
9056		----	----	----	----	----	----
9057		----	----	----	----	----	----
9058		----	----	----	----	----	----
9061		----	----	----	----	----	----
9101	D86	1.98	6.52	5.41	2.36	2.20	----
9107		----	----	----	----	----	----
9108		----	----	----	----	----	----
9142	D86	-0.55	1.18	1.12	2.96	5.92	----
9143		----	----	----	----	----	----
9150		----	----	----	----	----	----
9153		----	----	----	----	----	----

**Bold and underlined test results are outliers according to Dixon/Grubbs/Rosner**

## **APPENDIX 3:**

### **Number of participating laboratories per country**

1 lab in ALGERIA  
1 lab in AUSTRALIA  
1 lab in BELGIUM  
1 lab in BULGARIA  
1 lab in CROATIA  
1 lab in INDONESIA  
6 labs in MALAYSIA  
4 labs in NETHERLANDS  
3 labs in NIGERIA  
2 labs in NORWAY  
1 lab in POLAND  
1 lab in QATAR  
1 lab in RUSSIAN FEDERATION  
1 lab in SINGAPORE  
3 labs in UNITED ARAB EMIRATES  
11 labs in UNITED KINGDOM  
1 lab in UNITED STATES OF AMERICA  
1 lab in VIETNAM

## APPENDIX 4

### Abbreviations:

C	= final 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 outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from calculations
E	= error in calculations
n.a.	= not applicable
W	= withdrawn on request participant
U	= reported in deviating unit
SDS	= Safety Data Sheet

### Literature:

- 1 i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
- 2 ASTM E178-02
- 3 ASTM E1301-03
- 4 ISO 5725-86
- 5 ISO 5725, parts 1-6, 1994
- 6 ISO 13528-05
- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP367/96
- 9 DIN 38402 T41/42
- 10 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 11 J.N. Miller, Analyst, 118, 455, (1993)
- 12 Analytical Methods Committee Technical Brief, No4 February 2001
- 13 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see <http://www.rsc.org/suppdata/an/b2/b205600n/>)
- 14 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)