Results of Proficiency Test
Gascondensate
November 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION 3

2 SET UP 3

2.1 QUALITY SYSTEM 3

2.2 PROTOCOL 3

2.3 CONFIDENTIALITY STATEMENT 3

2.4 SAMPLES 4

2.5 STABILITY OF THE SAMPLES 4

2.6 ANALYSES 5

3 RESULTS 5

3.1 STATISTICS 5

3.2 GRAPHICS 6

3.3 Z-SCORES 7

4 EVALUATION 7

4.1 EVALUATION PER TEST 8

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES 10

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2018 WITH PREVIOUS PTS 11

Appendices:

1. Data and statistical results 12
2. Distillation z-scores 23
3. Number of participants per country 25
4. Abbreviations and literature 26

1 INTRODUCTION

Since 2008, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Gascondensate every year. During the annual proficiency testing program 2018/2019, it was decided to continue the round robin for the analysis of Gascondensate.
In this interlaboratory study 37 laboratories in 18 countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2018 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 analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send 1 sample of Gascondensate (0.5 L bottle, labelled #18220). 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 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 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

The necessary bulk material, approximately 35 litre Gascondensate, was obtained from a participating laboratory. After homogenisation, 58 amber glass bottles of 0.5 litre were filled and labelled #18220.
The homogeneity of the subsamples #18220 was checked by determination of Density at 15°C in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density at 15 °C in kg/L
Sample #18220-1	0.74318
Sample #18220-2	0.74320
Sample #18220-3	0.74319
Sample #18220-4	0.74320
Sample #18220-5	0.74323
Sample #18220-6	0.74319
Sample #18220-7	0.74317
Sample #18220-8	0.74318

Table 1: homogeneity test results of subsamples #18220

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

	Density at 15 °C in kg/L
r (observed)	0.00005
reference test method	ASTM D4052:18
0.3 * R (ref. test method)	0.00064

Table 2: evaluation of the repeatability of subsamples #18220

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

To each of the participating laboratories, one 0.5 L amber bottle, labelled #18220 was sent on October 17, 2018. 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 ANALYSES

The participants were requested to determine on sample #18220: Color Saybolt (Automated and Manual), Density at 15°C, Distillation (IBP, temperature at 5%, 10%, 50%, 90%, 95% recovered, FBP, distillation Residue and Loss), Methanol, Total Mercury, Sulphur, Water by KF and Simulated Distillation (IBP, temperature at 5%, 10%, 50%, 90%, 95% recovered and FBP).

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

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the 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 reanalyses). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

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

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

According to ISO 5725 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 reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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. 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

In this proficiency test problems with sample dispatch were encountered due to several reasons. One participant reported the test results after the final reporting date and five other participants did not report any test results at all. Not all laboratories were able to report all analyses requested. In total, 32 participants reported 263 numerical test results. Observed were 18 outlying test results, which is 6.8%. 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 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 methods are also in the table together with the original data. The abbreviations, used in these tables, are listed in appendix 4.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D6045) and an added designation for the year that the method was adopted or revised (e.g. D6045:12). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D6045:12(2017)). In the test results tables of appendix 1 only the method number and year of adoption or revision (e.g. D6045:12) will be used.

Unfortunately, a suitable reference test method providing the precision data is not available for all determinations. For the tests that have no precision data the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

Color Saybolt (automated): 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 D6045:12(2017).

Color Saybolt (manual): 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 D156:15.

Density at 15°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4052:18.
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 Gascondensates. However, Gascondensates 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 may be problematic. In total seven statistical outliers were observed and one other test result was excluded. After rejection of the suspect data, the calculated reproducibilities of temperature at 5%, 10%, 50% and 95% recovered were in agreement with the requirements of the manual mode of ASTM D86:18. However, the temperatures at 90 % recovered, Initial Boiling Point and Final Boiling Point were not in agreement with the requirements of the manual mode of ASTM D86:18.
It should be noted that the scope of ASTM D86 does not include Gascondensates, 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.

Methanol: Only three laboratories reported a test result. Therefore, no z-scores were calculated.

Mercury: The precision requirements of table 3b in test method UOP938 are extremely strict and approx. 6 times stricter than the Horwitz estimate. Thus, these requirements will not be met easily. Also, the reproducibility of UOP938 is only available for very low concentrations (0.28 and 12.14 µg/**L**) and conversion and extrapolation up to 113 µg/kg will lead to extra uncertainty. Therefore, it was decided to use the reproducibility based on the Horwitz estimate for evaluation of the test results in this report.
This determination was problematic at a concentration of 113 µg Hg per kg. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

Sulphur: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D5453:16e1.

Water: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D6304:16e1 (mass injection).

Simulated Distillation: This determination may be problematic. In total three statistical outliers were observed and three other test results were excluded. Only the calculated reproducibility of Final Boiling Point after rejection of the suspect data was in agreement with the requirements of ASTM D2887:18. The calculated reproducibility of the 50%, 90% and 95% recovered after rejection of the suspect data are not in agreement with the requirements of ASTM D2887:18. For 10% recovered no z-scores were calculated as the calculated reproducibility was too large compared to the requirements of ASTM D2887:18. The test results reported for Initial Boiling Point and 5% recovered were not evaluated as the temperature was below the measuring limit of 36°C.The very low number of reported test results may (partly) explain the large variation.

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 results, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM methods) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Color Saybolt (Automated)		12	17.6	1.8	1.2
Color Saybolt (Manual)		6	17.3	2.3	2
Density at 15°C	kg/L	29	0.7435	0.0015	0.0022
Distillation					
Initial Boiling Point	°C	15	32.4	10.2	7.2
5%-recovered	°C	15	55.9	4.3	6.5
10%-recovered	°C	15	67.6	3.9	3.7
50%-recovered	°C	15	121.5	2.6	4.8
90%-recovered	°C	15	245.0	16.1	7.0
95%-recovered	°C	7	296.2	14.8	15.4
Final Boiling Point	°C	14	301.7	4.1	3.6
Methanol	mg/kg	3	>120	n.a.	n.a.
Mercury as Hg	µg/kg	16	113	147	70
Sulphur	mg/kg	22	12.2	3.8	3.8
Water content by KF	mg/kg	26	51.9	49.8	180.6
Simulated Distillation					
Initial Boiling Point	°C	5	<36	n.a.	n.a.
5%-recovered	°C	5	<36	n.a.	n.a.
10%-recovered	°C	4	40.2	10.5	(2.1)
50%-recovered	°C	5	118.5	9.8	4.3
90%-recovered	°C	5	244.4	10.0	4.3
95%-recovered	°C	5	283.9	7.1	5
Final Boiling Point	°C	5	385.7	11.7	11.8

Table 3: reproducibilities of tests on sample #18220

Without further statistical calculations it can be concluded that for a number of tests there is not a good compliance of the group of participants with the relevant test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2018 WITH THE PREVIOUS PTS

	November 2018	*November 2017*	*November 2016*	*November 2015*	*November 2014*
Number of reporting participants	32	42	42	38	36
Number of results reported	263	333	297	248	251
Number of statistical outliers	18	19	23	8	8
Percentage of statistical outliers	6.8%	5.7%	7.7%	3.2%	3.2%

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 test methods. The conclusions are given the following table:

Determination	*November 2018*	*November 2017*	*November 2016*	*November 2015*	*November 2014*
Color Saybolt	-	--	--	--	--
Density at 15°C	+	++	++	+	+
Distillation (ASTM D86)	+/-	-	-	-	--
Methanol	n.e.	-	--	n.e.	n.e.
Mercury as Hg	--	+	-	-	-
Sulphur	+/-	-	+	-	--
Water content by KF	++	++	++	++	++
SimDist	--	+/-	--	n.e.	n.e.

Table 5: comparison of the performance per determination against the requirements of the reference test methods

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

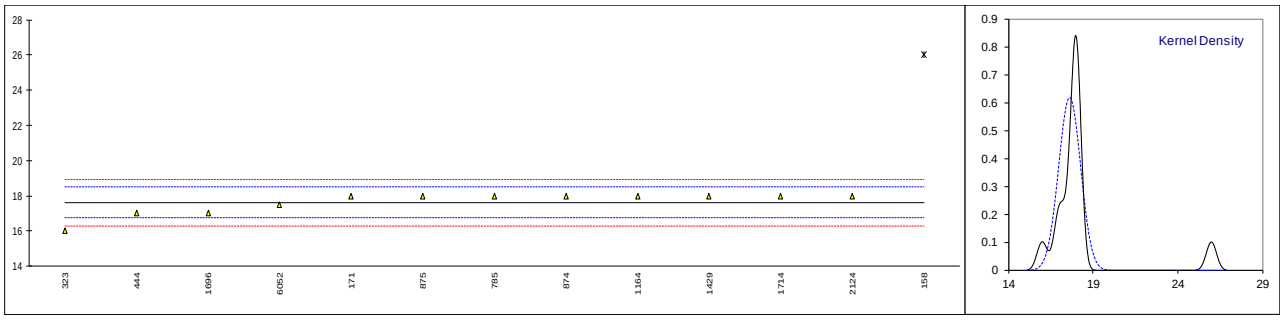
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

APPENDIX 1

Determination of Color Saybolt (automated) on sample #18220;

lab	method	value	mark	z(targ)	remarks
140		-----		-----	
158	D6045	26	G(0.01)	18.91	
171	D6045	18		0.85	
311		-----		-----	
323	D6045	16		-3.67	
442		-----		-----	
444	D6045	17		-1.41	
608		-----		-----	
609		-----		-----	
657		-----		-----	
785	D6045	18		0.85	
840		-----		-----	
874	D6045	18		0.85	
875	D6045	18		0.85	
922		-----		-----	
998		-----		-----	
1164	D6045	18		0.85	
1214		-----		-----	
1397		-----		-----	
1429	D6045	18		0.85	
1696	D6045	17		-1.41	
1714	D6045	18		0.85	
1815		-----		-----	
1957		-----		-----	
1960		-----		-----	
2124	D6045	18	C	0.85	First reported 21
6052	D6045	17.5		-0.28	
6087		-----		-----	
6218		-----		-----	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9061		-----		-----	
9099		-----		-----	
9101		-----		-----	
9107		-----		-----	
9143		-----		-----	

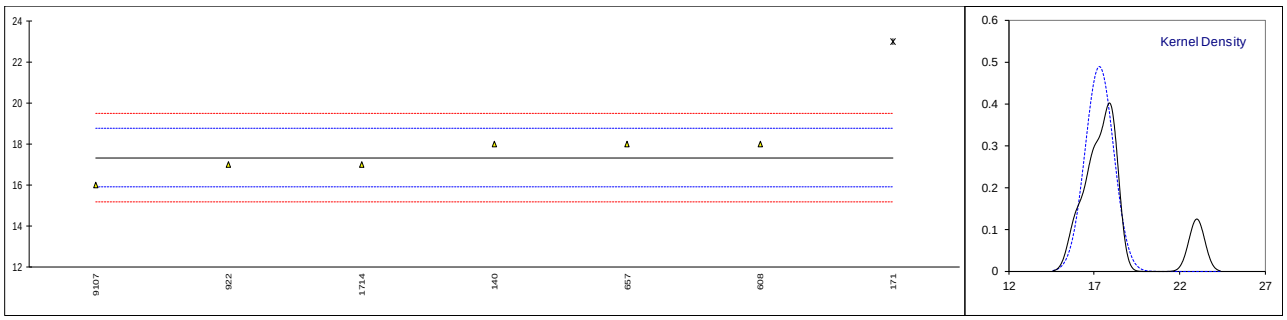
normality	not OK
n	12
outliers	1
mean (n)	17.63
st.dev. (n)	0.644
R(calc.)	1.80
st.dev.(D6045:12)	0.443
R(D6045:12)	1.24



Determination of Color Saybolt (manual) on sample #18220;

lab	method	value	mark	z(targ)	remarks
140	D156	18		0.93	
158		-----		-----	
171	D156	23	G(0.01)	7.93	
311		-----		-----	
323		-----		-----	
442		-----		-----	
444		-----		-----	
608	D156	18	C	0.93	First reported 1
609		-----		-----	
657	D156	18		0.93	
785		-----		-----	
840		-----		-----	
874		-----		-----	
875		-----		-----	
922	D156	17		-0.47	
998		-----		-----	
1164		-----		-----	
1214		-----		-----	
1397		-----		-----	
1429		-----		-----	
1696		-----		-----	
1714	D156	17		-0.47	
1815		-----		-----	
1957		-----		-----	
1960		-----		-----	
2124		-----		-----	
6052		-----		-----	
6087		-----		-----	
6218		-----		-----	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9061		-----		-----	
9099		-----		-----	
9101		-----		-----	
9107	D156	16		-1.87	
9143		-----		-----	

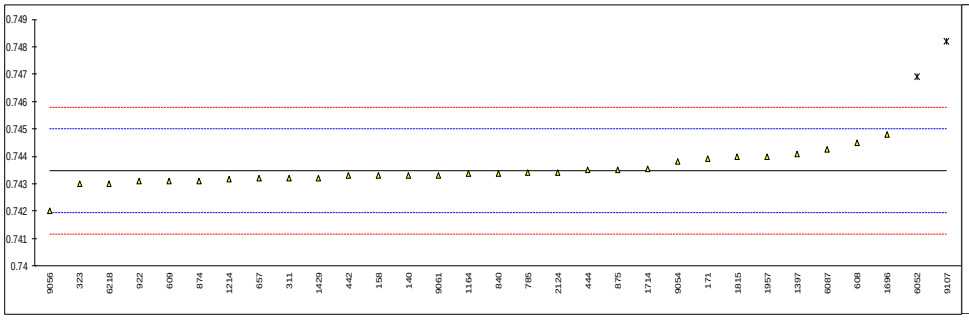
normality	unknown
n	6
outliers	1
mean (n)	17.33
st.dev. (n)	0.816
R(calc.)	2.29
st.dev.(D156:15)	0.714
R(D156:15)	2

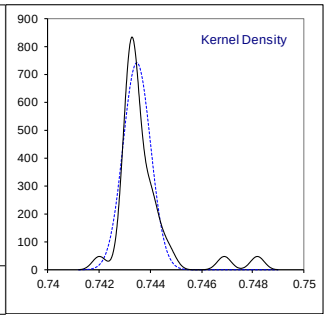


Determination of Density at 15°C on sample #18220; results in kg/L

lab	method	value	mark	z(targ)	remarks	original reported
140	D4052	0.7433		-0.22		0.7433
158	D4052	0.7433	C	-0.22	Reported 743.3 kg/l	743.3 kg/l
171	D4052	0.7439		0.56		743.9 kg/m^3
311	D4052	0.7432		-0.35		0.7432 kg/l
323	D4052	0.7430		-0.61		0.7430 kg/l
442	IP365	0.7433		-0.22		0.7433 kg/l
444	D4052	0.7435		0.04		0.7435 kg/l
608	D4052	0.7445		1.34		0.7445 kg/l
609	D4052	0.7431		-0.48		743.1 kg/m^3
657	D4052	0.7432		-0.35		0.7432 kg/l
785	D4052	0.7434		-0.09		0.7434 kg/l
840	D4052	0.74336		-0.15		0.74336 kg/l
874	D4052	0.7431		-0.48		743.1 kg/m^3
875	D4052	0.7435		0.04		0.7435 kg/l
922	D4052	0.7431		-0.48		0.7431 kg/l
998		-----		-----		
1164	D4052	0.74336		-0.15		0.74336 kg/l
1214	D7042	0.74318		-0.38		0.74318 kg/l
1397	D4052	0.7441		0.82		
1429	D4052	0.7432		-0.35		0.7432 kg/l
1696	D1298	0.7448		1.72		744.8 kg/m^3
1714	D4052	0.74353		0.08		743.53 kg/m^3
1815	ISO3675	0.7440		0.69		744.00 kg/m^3
1957	D4052	0.744		0.69		744 kg/m^3
1960		-----		-----		
2124	D5002	0.7434		-0.09		0.7434 kg/l
6052	D4052	0.7469	C,R(0.01)	4.45	First reported 0.7469 kg/m^3	0.7469 kg/m^3
6087	D4052	0.744241		1.00		744.241 kg/m^3
6218	GOST R51069	0.7430		-0.61		743.0 kg/m^3
9054	D4052	0.7438		0.43		743.8 kg/m^3
9055		-----		-----		
9056	ISO12185	0.742	C	-1.91	Reported 742 kg/l	742 kg/l
9061	ISO12185	0.74332		-0.20		0.74332 kg/l
9099		-----		-----		
9101		-----		-----		
9107	D4052	0.7482	R(0.01)	6.14		0.7482 kg/l
9143		-----		-----		

normality	suspect
n	29
outliers	2
mean (n)	0.74347
st.dev. (n)	0.000539
R(calc.)	0.00151
st.dev.(D4052:18)	0.000770
R(D4052:18)	0.00216

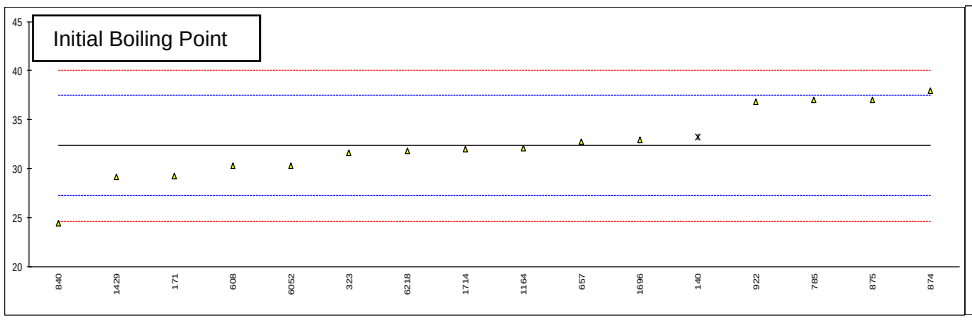


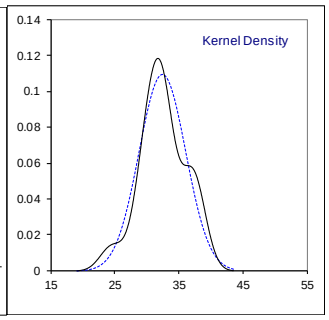


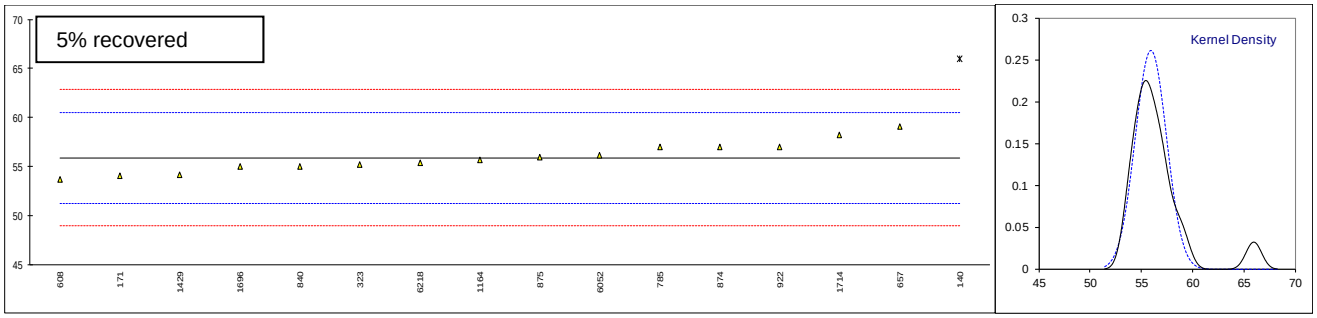
Determination of Distillation on sample #18220; results in °C

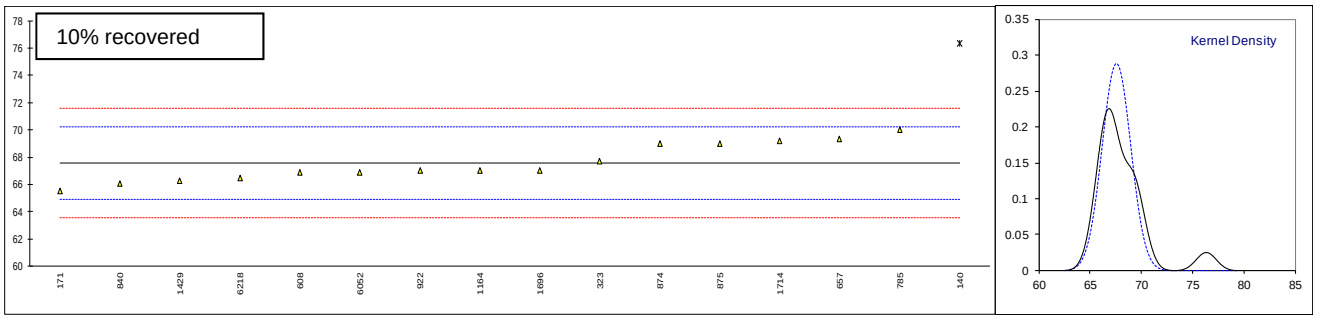
lab	method	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP	residue	loss
140	D86-automated	33.2 ex	**66.0**	**76.3**	**131.5**	**302.2**	-----	-----	-----	-----
158	D86-automated	-----	-----	-----	-----	-----	-----	-----	-----	-----
171	D86-automated	29.3	54.1	65.5	120.7	241.7	299.9	302.2	0.5	4.4
311		-----	-----	-----	-----	-----	-----	-----	-----	-----
323	IP123-automated	31.6	55.2	67.7	121.5	246.3	297.8	302.2	-----	-----
442		-----	-----	-----	-----	-----	-----	-----	-----	-----
444		-----	-----	-----	-----	-----	-----	-----	-----	-----
608	D86-automated	30.3	53.7	66.9	121.2	245.1	-----	301.3	-----	-----
609		-----	-----	-----	-----	-----	-----	-----	-----	-----
657	D86-automated	32.8	59.1	69.3	122.7	242.8	288.1	**308.3**	1.5	1.3
785	GOST2177:B	37.0	57.0	70.0	121.0	247.5	298.0	302.0	1.3	2.2
840	D86-automated	24.49	55.02	66.09	121.52	243.37	-----	300.70	1.3	4.0
874	D86-manual	38.0	57.0	69.0	122.0	251.5	300.5	304.0	1.5	2.0
875	D86-manual	37.0	56.0	69.0	121.0	250.0	300.0	302.0	1.3	2.2
922	D86-automated	36.8	57.0 C	67.0 C	121.6	248.5	-----	302.2	1.3	-----
998		-----	-----	-----	-----	-----	-----	-----	-----	-----
1164	D86-automated	32.1	55.7	67.0	122.3	242.8	289.2	301.9	3.0	0.9
1214		-----	-----	-----	-----	-----	-----	-----	-----	-----
1397		-----	-----	-----	-----	-----	-----	-----	-----	-----
1429	D86-automated	29.2	54.2	66.3	121.7	245.9	-----	299.2	1.3	4.6
1696	D86-manual	33	55	67	121	246	-----	299	1.8	4.2
1714	D86-automated	32.0	58.2	69.2	123.6	255.0	-----	302.1	1.0	5.6
1815		-----	-----	-----	-----	-----	-----	-----	-----	-----
1957		-----	-----	-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----	-----	-----
6052	D86-automated	30.3	56.2	66.9	119.7	231.2	**266.9**	300.8	1.2	1.6
6087		-----	-----	-----	-----	-----	-----	-----	-----	-----
6218	GOST2177- aut.	31.8	55.4	66.5	120.8	237.1	**279.9**	304.0	1.3	2.6
9054		-----	-----	-----	-----	-----	-----	-----	-----	-----
9055		-----	-----	-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----	-----	-----
9099		-----	-----	-----	-----	-----	-----	-----	-----	-----
9101		-----	-----	-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----	-----	-----
9143		-----	-----	-----	-----	-----	-----	-----	-----	-----
	normality	OK	OK	OK	suspect	suspect	OK	OK		
	n	15	15	15	15	15	7	14		
	outliers	0+1ex	1	1	1	1	2	1		
	mean (n)	32.38	55.92	67.56	121.49	244.99	296.21	301.69		
	st.dev. (n)	3.645	1.526	1.383	0.926	5.765	5.276	1.447		
	R(calc.)	10.21	4.27	3.87	2.59	16.14	14.77	4.05		
	st.dev.(D86-M:18)	2.564	2.314	1.335	1.696	2.487	5.510	1.271		
	R(D86-M:18)	7.18	6.48	3.74	4.75	6.96	15.43	3.56		
Compare										
	R(D86-A:18)	1.78	n.a.	1.49	3.0	3.67	6.20	7.1		

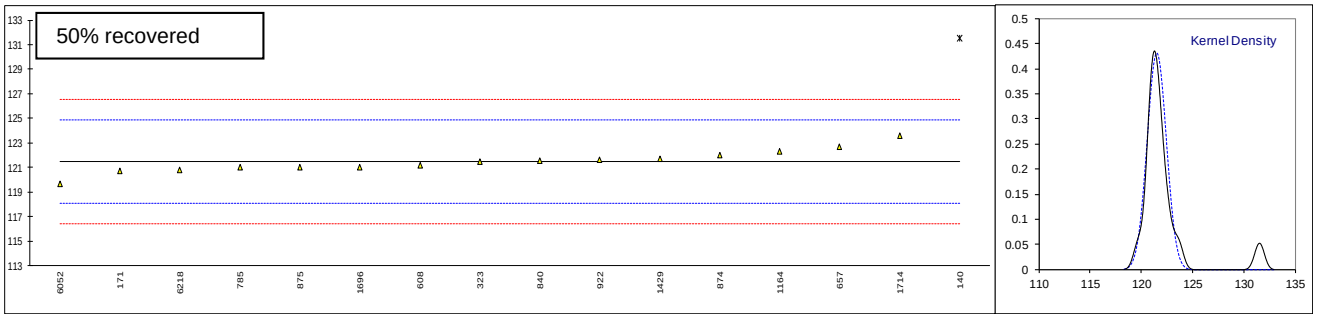
Results in Bold and Underlined are statistical outliers
Ex = excluded due to observed outliers in other Distillation parameters
Lab 922 first reported 65.1 and 75.5

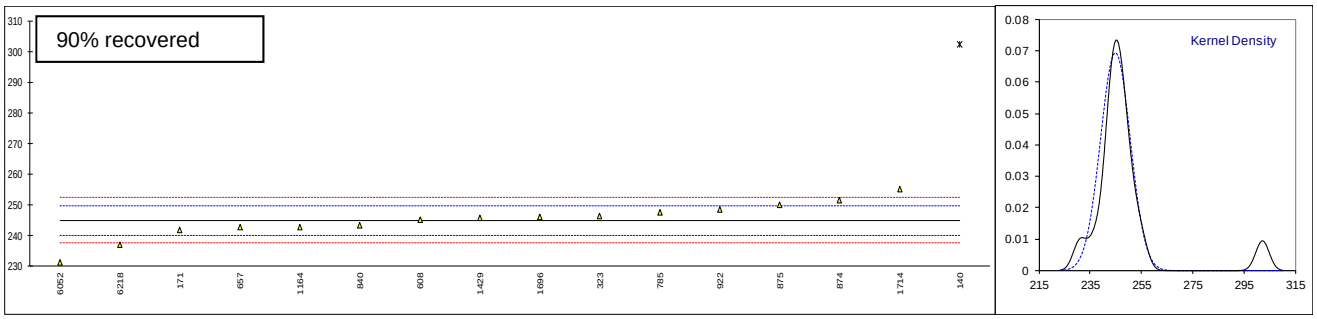


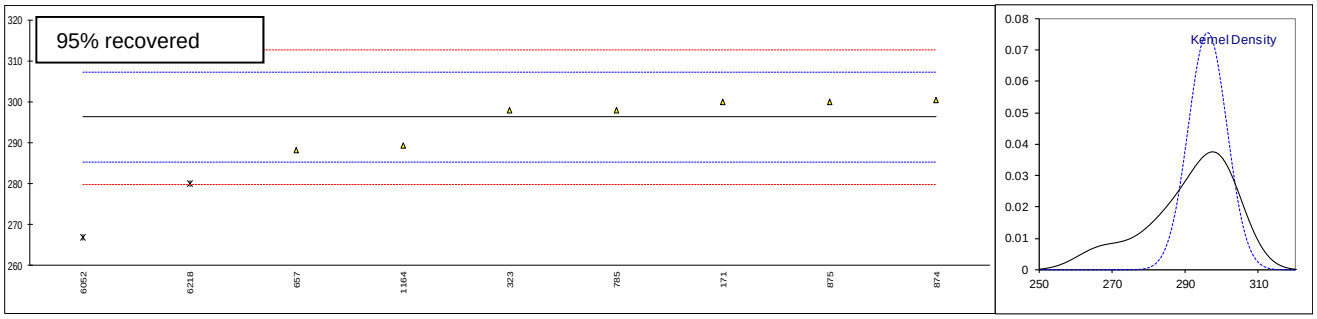


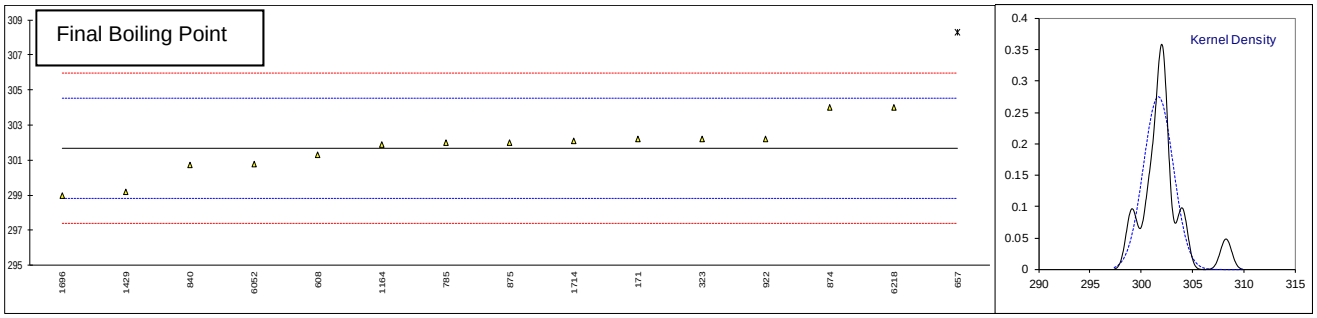

5% recovered
Kernel Density


10% recovered
Kernel Density


50% recovered
Kernel Density


90% recovered
Kernel Density


95% recovered
Kernel Density


Final Boiling Point
Kernel Density

Determination of Methanol on sample #18220; results in mg/kg

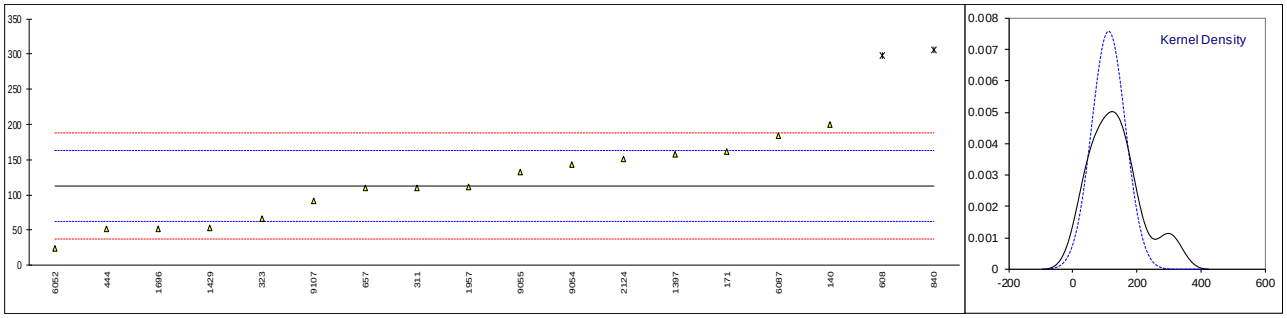
lab	method	value	mark	z(targ)	remarks
140		-----		-----	
158		-----		-----	
171		-----		-----	
311		-----		-----	
323	INH-304	133		-----	
442		-----		-----	
444		-----		-----	
608		-----		-----	
609		-----		-----	
657	INH-0130	228.6		-----	
785		-----		-----	
840		-----		-----	
874		-----		-----	
875		-----		-----	
922		-----		-----	
998		-----		-----	
1164		-----		-----	
1214		-----		-----	
1397		-----		-----	
1429	INH-97	120		-----	
1696		-----		-----	
1714		-----		-----	
1815		-----		-----	
1957		-----		-----	
1960		-----		-----	
2124		-----		-----	
6052		-----		-----	
6087		-----		-----	
6218		-----		-----	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9061		-----		-----	
9099		-----		-----	
9101		-----		-----	
9107		-----		-----	
9143		-----		-----	

normality	unknown
n	3
outliers	0
mean (n)	>120
st.dev. (n)	n.a.
R(calc.)	n.a.
st.dev.(Horwitz)	n.a.
R(Horwitz)	n.a.

Determination of total Mercury as Hg on sample #18220; results in µg/kg

lab	method	value	mark	z(targ)	remarks
140	UOP938	200		3.50	
158		-----		-----	
171	UOP938	162		1.98	
311	UOP938	110		-0.10	
323	UOP938	66		-1.86	
442		-----		-----	
444	UOP938	51.59		-2.44	
608	D7622	298.33	C,DG(0.05)	7.43	First reported 0.29833
609		-----		-----	
657	UOP938	109.8		-0.11	
785		-----		-----	
840	EPA7470A	306.0	DG(0.05)	7.74	
874		-----		-----	
875		-----		-----	
922		-----		-----	
998		-----		-----	
1164		-----		-----	
1214		-----		-----	
1397	In house	158		1.82	
1429	INH-91	53		-2.38	
1696	UOP938	51.7		-2.43	
1714		-----		-----	
1815		-----		-----	
1957	UOP938	111.4		-0.04	
1960		-----		-----	
2124	INH-210	151.32		1.55	
6052	UOP938	24.33		-3.53	
6087	UOP938	183.4867		2.84	
6218		-----		-----	
9054	UOP938	142.7481		1.21	
9055	In house	133		0.82	
9056		-----		-----	
9061		-----		-----	
9099		-----		-----	
9101		-----		-----	
9107	UOP938	91.702		-0.83	
9143		-----		-----	

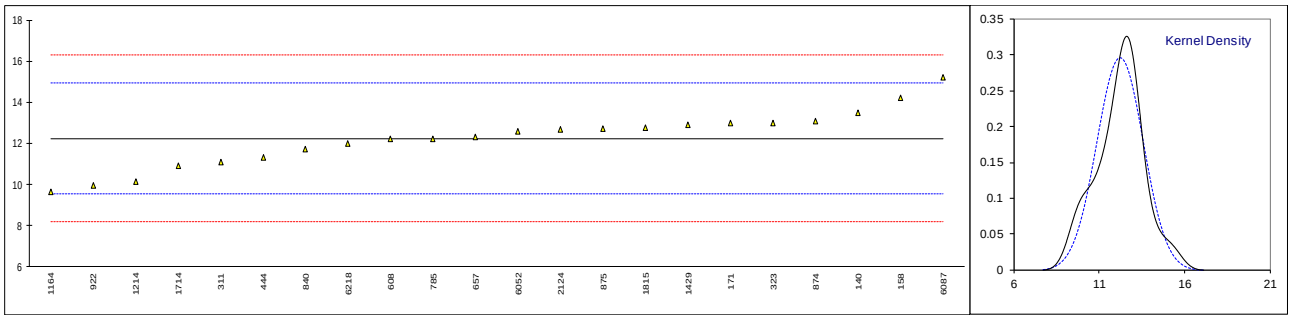
	normality	OK
	n	16
	outliers	2
	mean (n)	112.505
	st.dev. (n)	52.5284
	R(calc.)	147.079
	st.dev.(Horwitz)	25.0094
	R(Horwitz)	70.026
Compare		
	R(UOP938)	10.565



Determination of Sulphur on sample #18220; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140	D2622	13.5		0.94	
158	D2622	14.2		1.45	
171	D5453	13		0.57	
311	D5453	11.1		-0.84	
323	D5453	13		0.57	
442		-----		-----	
444	D5453	11.3		-0.69	
608	D5453	12.20		-0.02	
609		-----		-----	
657	D5453	12.3		0.05	
785	ISO20884	12.2		-0.02	
840	D5453	11.7		-0.39	
874	D2622	13.1		0.64	
875	ISO20846	12.7		0.35	
922	D5453	9.94		-1.69	
998		-----		-----	
1164	D5453	9.63		-1.92	
1214	D5453	10.145		-1.54	
1397		-----		-----	
1429	D5453	12.9		0.49	
1696		-----		-----	
1714	D5453	10.90		-0.98	
1815	D5453	12.78		0.41	
1957		-----		-----	
1960		-----		-----	
2124	D5453	12.66		0.32	
6052	D5453	12.6	C	0.27	First reported 6.8
6087	D5453	15.219		2.21	
6218	GOST32139	12		-0.17	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9061		-----		-----	
9099		-----		-----	
9101		-----		-----	
9107		-----		-----	
9143		-----		-----	

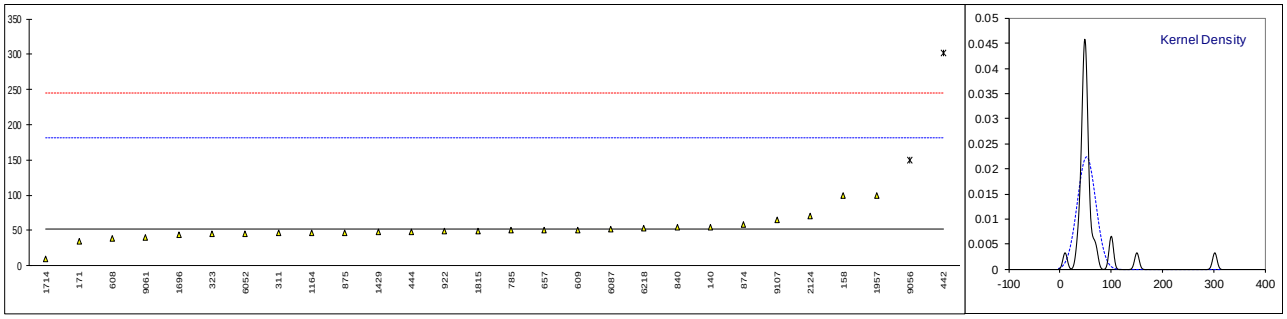
normality	OK
n	22
outliers	0
mean (n)	12.231
st.dev. (n)	1.3499
R(calc.)	3.780
st.dev.(D5453:16e1)	1.3540
R(D5453:16e1)	3.791



Determination of Water content by KF on sample #18220; results in mg/kg

lab	method	value	mark	z(targ)	remarks	original reported
140	D6304-A	54		0.03		54 mg/kg
158	D4928	100		0.75		0.01 %M/M
171	D6304-A	34		-0.28		34 mg/kg
311	D6304-A	46		-0.09		46 mg/kg
323	D6304-A	45		-0.11		45 mg/kg
442	IP438	302	R(0.01)	3.88		0.0302 %M/M
444	D6304-A	48.5		-0.05		48.5 mg/kg
608	D6304-A	39		-0.20		39 mg/kg
609	D4928	50.1		-0.03		50.1 mg/kg
657	D6304-A	50		-0.03		50 mg/kg
785	ISO12937	50.0		-0.03		50.0 mg/kg
840	D6304	54.0		0.03		54.0 mg/kg
874	D6304-A	59		0.11		59 mg/kg
875	D6304-A	47		-0.08		47 mg/kg
922	D6304-A	49		-0.04		49 mg/kg
998		-----		-----		
1164	D6304-A	46		-0.09		46 mg/kg
1214		-----		-----		
1397		-----		-----		
1429	IP438	48		-0.06		48 mg/kg
1696	D6304-A	43.9		-0.12		43.9 mg/kg
1714	D6304-C	10		-0.65		0.001 %M/M
1815	ISO12937	49.14		-0.04		0.004914 %M/M
1957	D6304-A	100		0.75		0.01 %M/M
1960		-----		-----		
2124	D4928	70.8		0.29		0.00708 %M/M
6052	D6304-A	45.02		-0.11		45.02 mg/kg
6087	D6304-A	52.4		0.01		52.4 mg/kg
6218	D4928	53		0.02		0.0053 %M/M
9054		-----		-----		
9055		-----		-----		
9056	IP356	150	R(0.01)	1.52		150 mg/kg
9061	D4928	40		-0.18		0.004 %M/M
9099		-----		-----		
9101		-----		-----		
9107	D6304-A	65		0.20		65 mg/kg
9143		-----		-----		

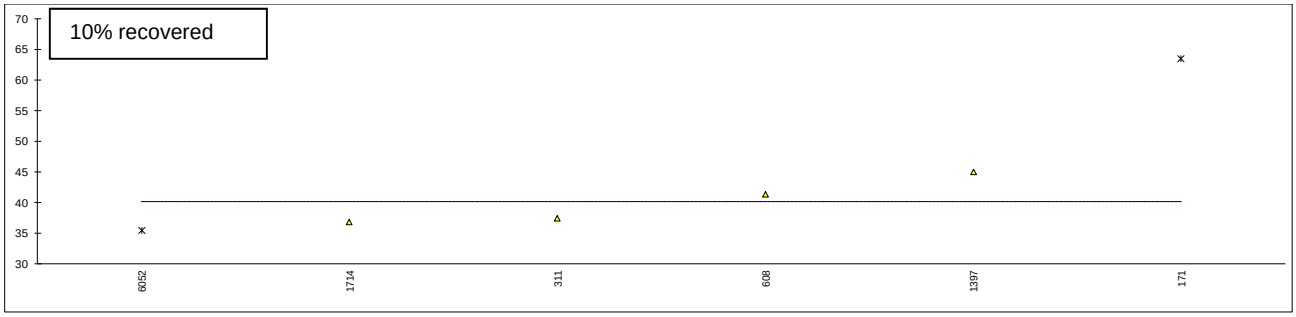
normality	not OK
n	26
outliers	2
mean (n)	51.88
st.dev. (n)	17.784
R(calc.)	49.80
st.dev.(D6304:16e1 (mass inj))	64.493
R(D6304:16e1 (mass inj))	180.58

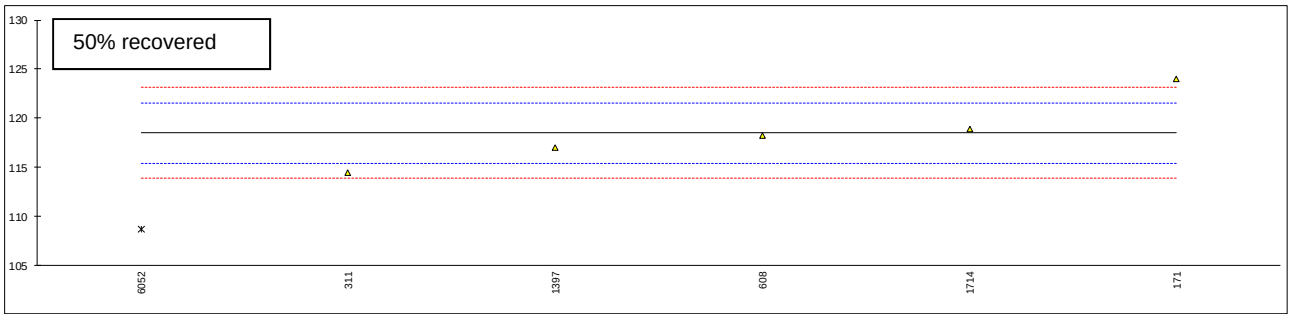


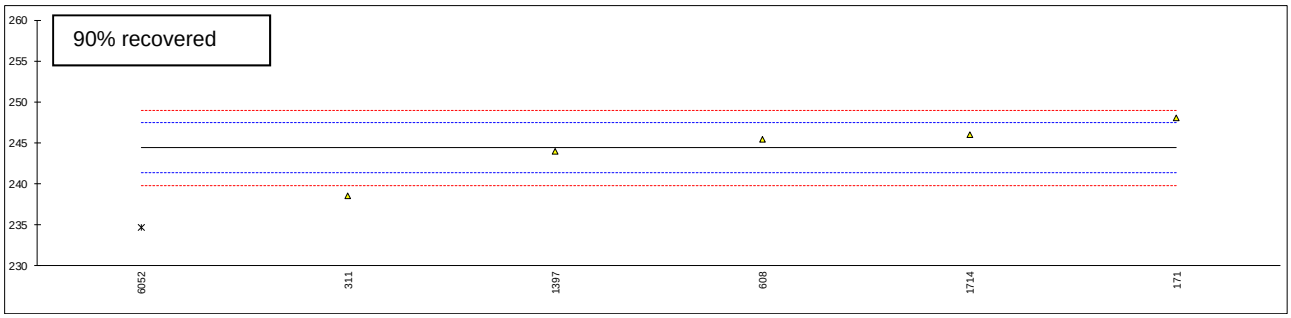
Determination of Simulated Distillation on sample #18220; results in °C

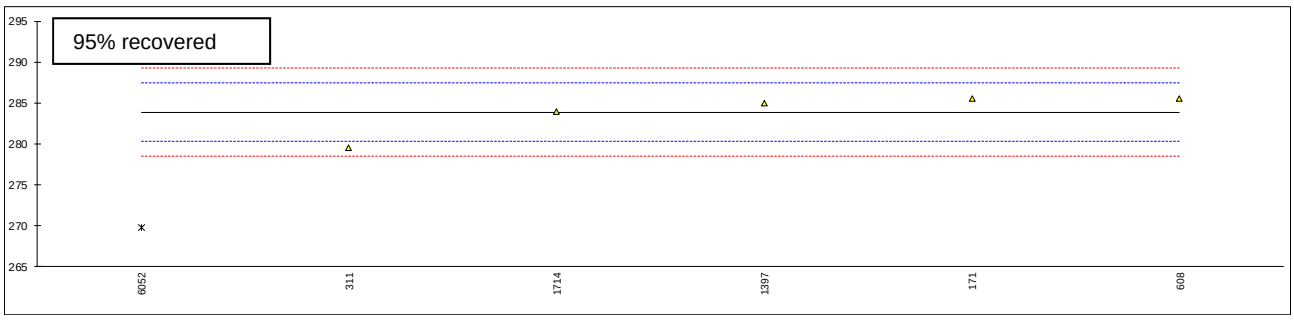
lab	method	IBP	5%rec	10%rec	50%rec	90%rec	95%rec	FBP
140		-----	-----	-----	-----	-----	-----	-----
158		-----	-----	-----	-----	-----	-----	-----
171	D2887	20.5	34.0	**63.5**	124.0	248.0	285.5	388.5
311	D2887	<36.0	<36.0	37.5	114.5	238.5	279.5	380.5
323		-----	-----	-----	-----	-----	-----	-----
442		-----	-----	-----	-----	-----	-----	-----
444		-----	-----	-----	-----	-----	-----	-----
608	D2887	-12.2	19.7	41.3	118.2	245.4	285.5	389.9
609		-----	-----	-----	-----	-----	-----	-----
657		-----	-----	-----	-----	-----	-----	-----
785		-----	-----	-----	-----	-----	-----	-----
840		-----	-----	-----	-----	-----	-----	-----
874		-----	-----	-----	-----	-----	-----	-----
875		-----	-----	-----	-----	-----	-----	-----
922		-----	-----	-----	-----	-----	-----	-----
998		-----	-----	-----	-----	-----	-----	-----
1164		-----	-----	-----	-----	-----	-----	-----
1214		-----	-----	-----	-----	-----	-----	-----
1397	D2887	13	31	45	117	244	285	382
1429		-----	-----	-----	-----	-----	-----	-----
1696		-----	-----	-----	-----	-----	-----	-----
1714	D2887	<-0.5	12.15	36.90	118.90	246.00	283.95	387.70
1815		-----	-----	-----	-----	-----	-----	-----
1957		-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----
6052	D2887	n/a	n/a	35.4 ex	108.7 ex	234.7 ex	**269.8**	**342.9**
6087		-----	-----	-----	-----	-----	-----	-----
6218		-----	-----	-----	-----	-----	-----	-----
9054		-----	-----	-----	-----	-----	-----	-----
9055		-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----
9099		-----	-----	-----	-----	-----	-----	-----
9101		-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----
9143		-----	-----	-----	-----	-----	-----	-----
	normality	unknown	unknown	unknown	unknown	unknown	unknown	unknown
	n	5	5	4	5	5	5	5
	outliers	0	0	1+1ex	0+1ex	0+1ex	1	1
	mean (n)	<36	<36	40.18	118.52	244.38	283.90	385.72
	st.dev. (n)	n.a.	n.a.	3.761	3.491	3.588	2.534	4.190
	R(calc.)	n.a.	n.a.	10.530	9.775	10.046	7.096	11.731
	st.dev.(D2887:18)	n.a.	n.a.	(0.751)	1.536	1.536	1.786	4.214
	R(D2887:18)	n.a.	n.a.	(2.103)	4.3	4.3	5	11.8

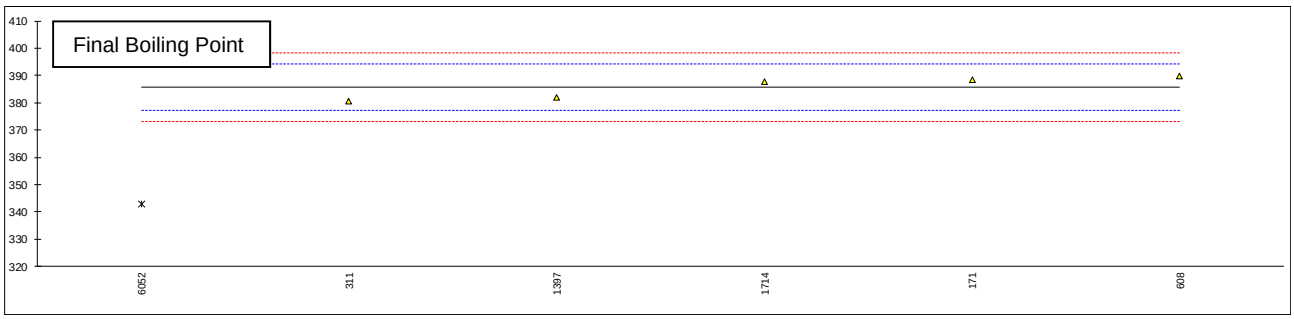
Results in Bold and Underlined are statistical outliers.
Ex = excluded due to observed outliers in other Simulated Distillation parameters


10% recovered
70
65
60
55
50
45
40
35
30
6052
1714
311
608
1397
171


50% recovered
130
125
120
115
110
105
6052
311
1397
608
1714
171


90% recovered
260
255
250
245
240
235
230
6052
311
1397
608
1714
171


95% recovered
295
290
285
280
275
270
265
6052
311
1714
1397
171
608


Final Boiling Point
410
400
390
380
370
360
350
340
330
320
6052
311
1397
1714
171
608

APPENDIX 2:

Atmospheric Distillation z-scores

lab	IBP	5%	10%	50%	90%	95%	FBP
140	0.32	4.36	6.55	5.90	23.00	-----	-----
158	-----	-----	-----	-----	-----	-----	-----
171	-1.20	-0.79	-1.54	-0.46	-1.32	0.67	0.40
311	-----	-----	-----	-----	-----	-----	-----
323	-0.30	-0.31	0.11	0.01	0.53	0.29	0.40
442	-----	-----	-----	-----	-----	-----	-----
444	-----	-----	-----	-----	-----	-----	-----
608	-0.81	-0.96	-0.49	-0.17	0.05	-----	-0.30
609	-----	-----	-----	-----	-----	-----	-----
657	0.16	1.37	1.30	0.71	-0.88	-1.47	5.20
785	1.80	0.47	1.83	-0.29	1.01	0.32	0.25
840	-3.08	-0.39	-1.10	0.02	-0.65	-----	-0.78
874	2.19	0.47	1.08	0.30	2.62	0.78	1.82
875	1.80	0.03	1.08	-0.29	2.02	0.69	0.25
922	1.72	0.47	-0.42	0.07	1.41	-----	0.40
998	-----	-----	-----	-----	-----	-----	-----
1164	-0.11	-0.10	-0.42	0.48	-0.88	-1.27	0.17
1214	-----	-----	-----	-----	-----	-----	-----
1397	-----	-----	-----	-----	-----	-----	-----
1429	-1.24	-0.74	-0.94	0.12	0.37	-----	-1.96
1696	0.24	-0.40	-0.42	-0.29	0.41	-----	-2.11
1714	-0.15	0.98	1.23	1.25	4.03	-----	0.33
1815	-----	-----	-----	-----	-----	-----	-----
1957	-----	-----	-----	-----	-----	-----	-----
1960	-----	-----	-----	-----	-----	-----	-----
2124	-----	-----	-----	-----	-----	-----	-----
6052	-0.81	0.12	-0.49	-1.05	-5.54	-5.32	-0.70
6087	-----	-----	-----	-----	-----	-----	-----
6218	-0.23	-0.23	-0.79	-0.41	-3.17	-2.96	1.82
9054	-----	-----	-----	-----	-----	-----	-----
9055	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9099	-----	-----	-----	-----	-----	-----	-----
9101	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9143	-----	-----	-----	-----	-----	-----	-----

Simulated Distillation z-scores

lab	IBP	5%	10%	50%	90%	95%	FBP
140	-----	-----	-----	-----	-----	-----	-----
158	-----	-----	-----	-----	-----	-----	-----
171	-----	-----	-----	3.57	2.36	0.90	0.66
311	-----	-----	-----	-2.62	-3.83	-2.46	-1.24
323	-----	-----	-----	-----	-----	-----	-----
442	-----	-----	-----	-----	-----	-----	-----
444	-----	-----	-----	-----	-----	-----	-----
608	-----	-----	-----	-0.21	0.66	0.90	0.99
609	-----	-----	-----	-----	-----	-----	-----
657	-----	-----	-----	-----	-----	-----	-----
785	-----	-----	-----	-----	-----	-----	-----
840	-----	-----	-----	-----	-----	-----	-----
874	-----	-----	-----	-----	-----	-----	-----
875	-----	-----	-----	-----	-----	-----	-----
922	-----	-----	-----	-----	-----	-----	-----
998	-----	-----	-----	-----	-----	-----	-----
1164	-----	-----	-----	-----	-----	-----	-----
1214	-----	-----	-----	-----	-----	-----	-----
1397	-----	-----	-----	-0.99	-0.25	0.62	-0.88
1429	-----	-----	-----	-----	-----	-----	-----
1696	-----	-----	-----	-----	-----	-----	-----
1714	-----	-----	-----	0.25	1.05	0.03	0.47
1815	-----	-----	-----	-----	-----	-----	-----
1957	-----	-----	-----	-----	-----	-----	-----
1960	-----	-----	-----	-----	-----	-----	-----
2124	-----	-----	-----	-----	-----	-----	-----
6052	-----	-----	-----	-6.39	-6.30	-7.89	-10.16
6087	-----	-----	-----	-----	-----	-----	-----
6218	-----	-----	-----	-----	-----	-----	-----
9054	-----	-----	-----	-----	-----	-----	-----
9055	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9099	-----	-----	-----	-----	-----	-----	-----
9101	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9143	-----	-----	-----	-----	-----	-----	-----

APPENDIX 3:

Number of participating laboratories per country

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

APPENDIX 4

Abbreviations:

C	= final test result after checking of first reported suspect test 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	= probably an error in calculations
U	= test result probably reported in a different unit
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:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ASTM E178:16
3 ASTM E1301:95(2003)
4 ISO 5725:86 (1994)
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 IP 367:84
10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee Technical Brief, No 4 January 2001
14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst 2002, 127, 1359-1364, (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)
16 Horwitz, W and Albert, R, J. AOAC Int, 79, 3, 589, (1996)