Results of Proficiency Test
Gascondensate
November 2019

CONTENTS

Appendices:

1   INTRODUCTION

Since 2008 the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the analyses of Gascondensate every year. During the annual program 2019/2020, it was decided to continue the round robin for the analyzes of Gascondensate.

In this interlaboratory study 36 laboratories in 17 countries registered for participation. See appendix 3 for the number of participants per country.
In this report, the results of the 2019 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/IEC17025 accredited laboratory. It was decided to send one 0.5-liter bottle with Gascondensate, labelled #19230. 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 30 liters of Gascondensate was obtained from a local supplier. After homogenization 58 amber glass bottles of 0.5 liter were filled and labelled #19230. The homogeneity of the subsamples #19230 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 #19230-1	0.74735
Sample #19230-2	0.74733
Sample #19230-3	0.74737
Sample #19230-4	0.74738
Sample #19230-5	0.74734
Sample #19230-6	0.74736
Sample #19230-7	0.74735
Sample #19230-8	0.74736

Table 1: homogeneity test results of subsamples #19230

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)	0.00004
reference test method	ASTM D4052:18a
0.3 * R (ref. test method)	0.00060

Table 2: evaluation of the repeatability of subsamples #19230

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 #19230, was sent on October 30, 2019. 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 on sample #19230: Color Saybolt (automated and manual), 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 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 reanalyzes). Additional or corrected test results are used for data analyzes 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. instead of the rounded test results.

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

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2　GRAPHICS

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

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

3.3　Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM 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 some problems were encountered with the dispatch of the samples. Participants in Australia, Malaysia, Russia, United States of America and Vietnam received the samples late or not at all. Finally, six participants reported the test results after the final reporting date and four other participants did not report any test results at all. Not all laboratories were able to report all analyzes requested.

In total, 32 participants reported in total 236 numerical test results. Observed were 15 outlying test results, which is 6.4%. 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 explained 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 and manual): The reported test results for the automated and manual tests were combined as the number of reported test results for the manual mode was small.
This Color Saybolt 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) and 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 D4052:18a.
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.

Distillation at 760 mmHg: This determination may be problematic. In total eight statistical outliers were observed and one other test result was excluded. After rejection of the suspect data, the calculated reproducibilities at Initial Boiling Point, 10%, 90%, 95% recovered and Final Boiling Point were not in agreement with the requirements of the manual mode of ASTM D86:19. The calculated reproducibilities for 5% and 50% recovered after rejection of the suspect data are in agreement with the requirements of ASTM D86:19. 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.

Methanol: Only three laboratories reported a test result close to the detection limit. Therefore, no z-scores were calculated.

Mercury, Total: The precision requirements of table 3b in test method UOP938 are extremely strict and 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 µg/$\underline{L}$) and conversion and extrapolation up to 159 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 not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the estimated reproducibility calculated using the Horwitz equation.

Sulfur, Total: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5453:19a.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6304:16e1 (mass injection).

Simulated Distillation: This determination may be problematic. In total one statistical outlier was observed. The calculated reproducibility for 95% recovered and Final Boiling Point after rejection of the statistical outlier were not in agreement with the requirements of ASTM D2887:19a. For 10%, 50% and 90% recovered no z-scores were calculated as the calculated reproducibility was too large compared to the requirements of ASTM D2887:19a. 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 result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Color Saybolt		15	14.5	3.3	1.2
Density at 15°C	kg/L	28	0.7476	0.0014	0.0020
Distillation at 760 mmHg					
Initial Boiling Point	°C	13	33.2	9.9	7.8
5% recovered	°C	13	60.0	5.4	6.9
10% recovered	°C	13	71.3	4.5	3.8
50% recovered	°C	13	128.5	4.9	5.0
90% recovered	°C	13	262.0	16.1	6.8
95% recovered	°C	6	302.2	32.6	12.5
Final Boiling Point	°C	10	318.1	5.5	4.4
Methanol	mg/kg	3	<2	n.a.	n.a.
Mercury, Total	µg/kg	14	159	96	94
Sulfur, Total	mg/kg	16	59.4	20.5	12.4
Water	mg/kg	23	51.8	48.5	180.5
Simulated Distillation					
Initial Boiling Point	°C	6	<36	n.a.	n.a.
5% recovered	°C	6	<36	n.a.	n.a.
10% recovered	°C	6	46.5	23.3	(2.2)
50% recovered	°C	6	123.3	14.9	(4.3)
90% recovered	°C	6	264.4	17.5	(4.3)
95% recovered	°C	5	300.7	9.4	5
Final Boiling Point	°C	6	401.9	60.4	11.8

Table 3: reproducibilities of tests on sample #19230

Results between brackets should be used with due care because the calculated reproducibility was too large

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 2019 WITH THE PREVIOUS PTS

	November 2019	November 2018	November 2017	November 2016	November 2015
Number of reporting participants	32	32	42	42	38
Number of results reported	236	263	333	297	248
Number of statistical outliers	15	18	19	23	8
Percentage of statistical outliers	6.4%	6.8%	5.7%	7.7%	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 2019	November 2018	November 2017	November 2016	November 2015
Color Saybolt	--	-	--	--	--
Density at 15°C	+	+	++	++	+
Distillation at 760 mmHg	-	+/-	-	-	-
Methanol	n.e.	n.e.	-	--	n.e.
Mercury, Total	+/-	--	+	-	-
Sulfur, Total	-	+/-	-	+	-
Water	++	++	++	++	++
Simulated Distillation	--	--	+/-	--	n.e.

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

In the table above the following performance categories were used:

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

APPENDIX 1

Determination of Color Saybolt (automated and manual) on sample #19230;

lab	method	value	mark	z(targ)	remarks
140	D156	12		-5.57	
171	D6045	15		1.20	
311		-----		-----	
323	D6045	15		1.20	
442		-----		-----	
444	D6045	14		-1.05	
608	D156	15		1.20	
609		-----		-----	
657	D6045	14		-1.05	Reported Color Saybolt (manual): 14
785		-----		-----	
840		-----		-----	
874		-----		-----	
875	D6045	15		1.20	
922	D156	16		3.46	
998		-----		-----	
1164	D6045	14		-1.05	
1397		-----		-----	
1429	D6045	13		-3.31	
1696	D6045	16.0		3.46	
1714	D6045	21	G(0.01)	14.75	
1815		-----		-----	
1957		-----		-----	
1960	D6045	14		-1.05	
2124	D6045	16		3.46	
6052	D6045	15		1.20	
6087		-----		-----	
6234		-----		-----	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9061		-----		-----	
9099		-----		-----	
9101		-----		-----	
9107	D156	13		-3.31	
9141		-----		-----	
9143		-----		-----	

	normality	OK
	n	15
	outliers	1
	mean (n)	14.47
	st.dev. (n)	1.187
	R(calc.)	3.32
	st.dev.(D6045:12)	0.443
	R(D6045:12)	1.24
Compare		
	R(D156:15)	2
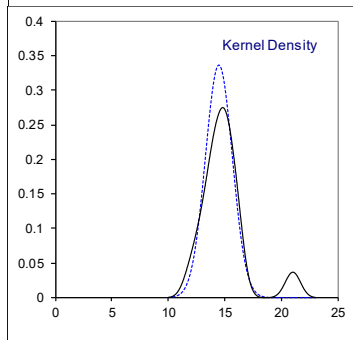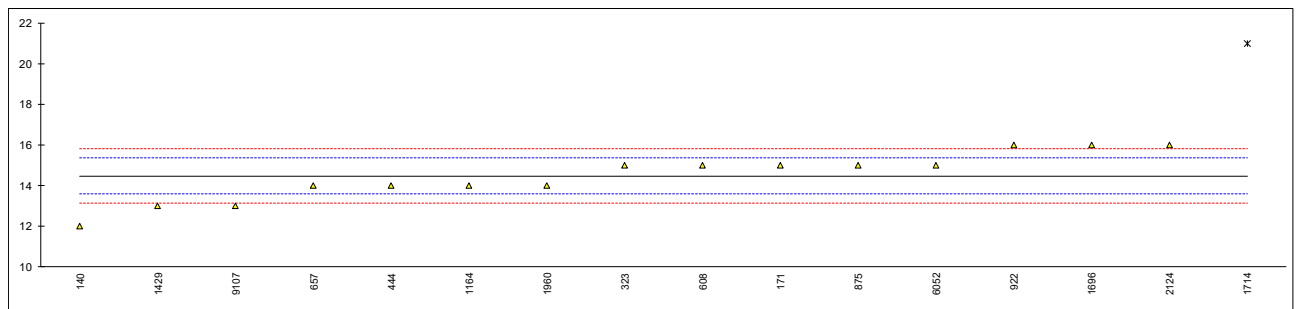
Determination of Density at 15°C on sample #19230; results in kg/L

lab	method	value	mark	z(targ)	remarks
140	D4052	0.7475		-0.15	
171	D4052	0.74772	C	0.15	Reported 747.72 kg/L
311	D4052	0.7474		-0.29	
323	D4052	0.7472		-0.56	
442	IP365	0.7475		-0.15	
444	D4052	0.7476		-0.01	
608	D4052	0.7476		-0.01	
609	D4052	0.7478		0.27	
657	D4052	0.7473		-0.43	
785		-----		-----	
840	D4052	0.74708		-0.73	
874		-----		-----	
875	D4052	0.7476		-0.01	
922	D4052	0.7480		0.54	
998	D4052	0.74729		-0.44	
1164	D4052	0.74734		-0.37	
1397	ISO12185	0.7483		0.96	
1429	D4052	0.7474		-0.29	
1696	D1298	0.7475		-0.15	
1714	D4052	0.74739		-0.30	
1815	ISO12185	0.74755		-0.08	
1957	D4052	0.8737	R(0.01)	174.32	
1960	D4052	0.747420	C	-0.26	Reported 747.420 kg/L
2124		0.7473		-0.43	
6052	D4052	0.7474		-0.29	
6087	D4052	0.747412		-0.27	
6234		-----		-----	
9054	D4052	0.7481		0.68	
9055		-----		-----	
9056		0.743	C,R(0.01)	-6.37	Reported 743 kg/L
9061	ISO12185	0.74736		-0.34	
9099		-----		-----	
9101	D1298	0.74702		-0.81	
9107	D4052	0.74868		1.48	
9141	D1298	0.749262	C	2.29	First reported 750.0 kg/m^3
9143		-----		-----	

normality	not OK	
n	28	
outliers	2	
mean (n)	0.74761	
st.dev. (n)	0.000484	
R(calc.)	0.00135	
st.dev.(D4052:18a)	0.000723	
R(D4052:18a)	0.00203	
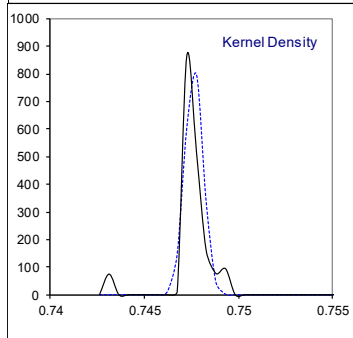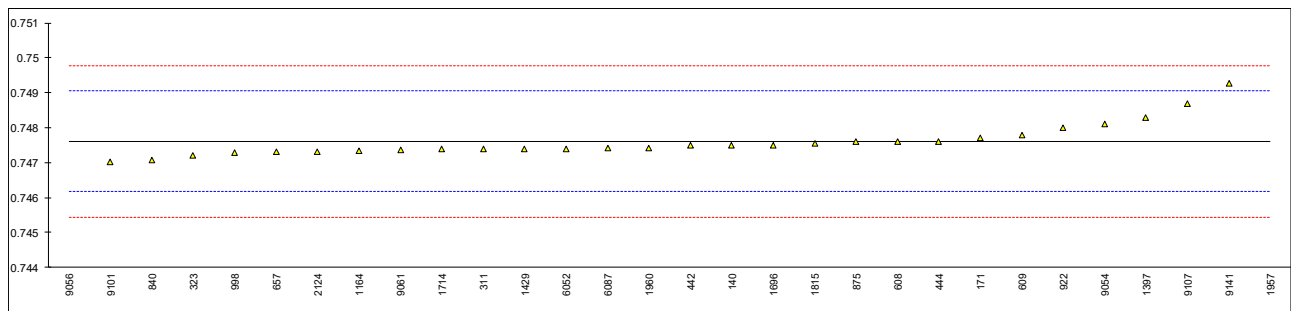
Determination of Distillation at 760 mmHg on sample #19230; results in °C

| lab	method	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP	residue	loss
140		-----	-----	-----	-----	-----	-----	-----	-----	-----
171	D86	30.611	56.778	68.389	125.278	253.00	291.889	**306.000**	1.0	0.9
311		-----	-----	-----	-----	-----	-----	-----	-----	-----
323	D86-automated	30.8	60.4	72.2	129.3	267.0	316.3	318.3	1.3	2.5
442		-----	-----	-----	-----	-----	-----	-----	-----	-----
444		-----	-----	-----	-----	-----	-----	-----	-----	-----
608	D86-automated	26.9	59.6	70.9	128.4	262.6	-----	318.2	1.3	-----
609		-----	-----	-----	-----	-----	-----	-----	-----	-----
657	D86-automated	31.0	63.4	73.7	130.9	263.6	309.5	320.0	1.4	1.2
785		-----	-----	-----	-----	-----	-----	-----	-----	-----
840	D86-automated	30.79	59.79	71.42	129.01	261.92	-----	314.42	1.3	4.3
874		-----	-----	-----	-----	-----	-----	-----	-----	-----
875	D86-manual	41.0	60.5	71.0	127.5	264.0	311.5	319.5	1.6	1.9
922	D86-manual	33.3	59.5	71.5	130.3	268.9	-----	318.0	-----	-----
998	D86-manual	33.5	57.0	68.5	125.0	255.0	296.0	>300.0	-----	-----
1164	D86-automated	36.8	61.3	72.2	128.8	252.5	288.2	**303.1**	3.0	0.2
1397		-----	-----	-----	-----	-----	-----	-----	-----	-----
1429	D86-automated	31.6	58.1	70.0	129.1	265.7	-----	316.5	1.3	4.4
1696		35.0	60.0	71.0	129.0	266.0	-----	317.0 C	-----	-----
1714	D86-automated	34.4	61.4	73.1	129.9	268.4	-----	317.2	1.2	5.8
1815		-----	-----	-----	-----	-----	-----	-----	-----	-----
1957		-----	-----	-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----	-----	-----
6052	D86-automated	35.6	62.3	72.9	127.7	257.0	**263.0**	321.4	-----	-----
6087		-----	-----	-----	-----	-----	-----	-----	-----	-----
6234		-----	-----	-----	-----	-----	-----	-----	-----	-----
9054		-----	-----	-----	-----	-----	-----	-----	-----	-----
9055		-----	-----	-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----	-----	-----
9099		-----	-----	-----	-----	-----	-----	-----	-----	-----
9101		-----	-----	-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----	-----	-----
9141		-----	-----	-----	-----	-----	-----	-----	-----	-----
9143	D1078	**23.5**	**35.5**	**43.10**	**95.40**	259.70 ex	-----	**291.30**	1.30	5.30

							unknown			
	normality	suspect	OK	OK	OK	OK	n	OK		
	n	13	13	13	13	13	6	10		
	outliers	1	1	1	1	0 (+1ex)	1	3		
	mean (n)	33.18	60.01	71.29	128.48	261.97	302.23	318.05		
	st.dev. (n)	3.532	1.923	1.622	1.752	5.740	11.656	1.961		
	R(calc.)	9.89	5.38	4.54	4.91	16.07	32.64	5.49		
	st.dev.(D86-M:19)	2.782	2.453	1.366	1.774	2.418	4.468	1.582		
	R(D86-M:19)	7.79	6.87	3.82	4.97	6.77	12.51	4.43		
Compare										
	R(D86-A:19)	1.83	n.a.	1.57	3.0	3.93	6.66	7.10		

Results in bold and underlined are statistical outliers
Ex = excluded due to observed outliers in other distillation parameters
Lab 1696 first reported 310.0

Initial Boiling Point
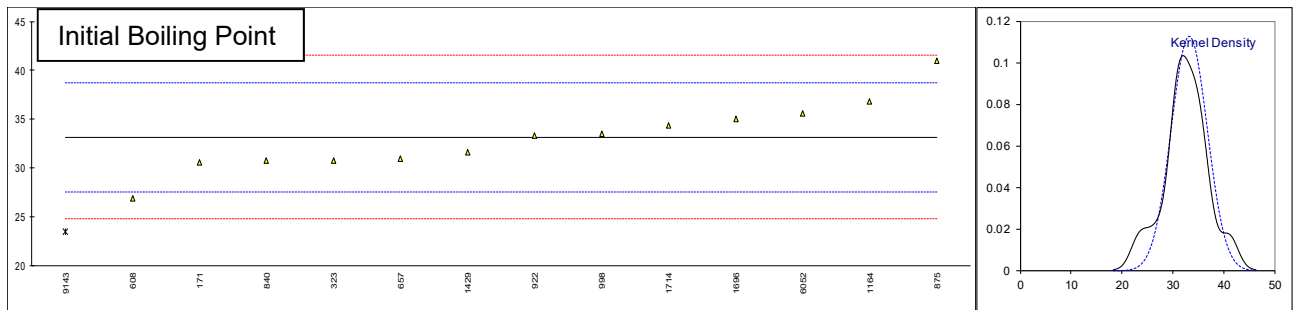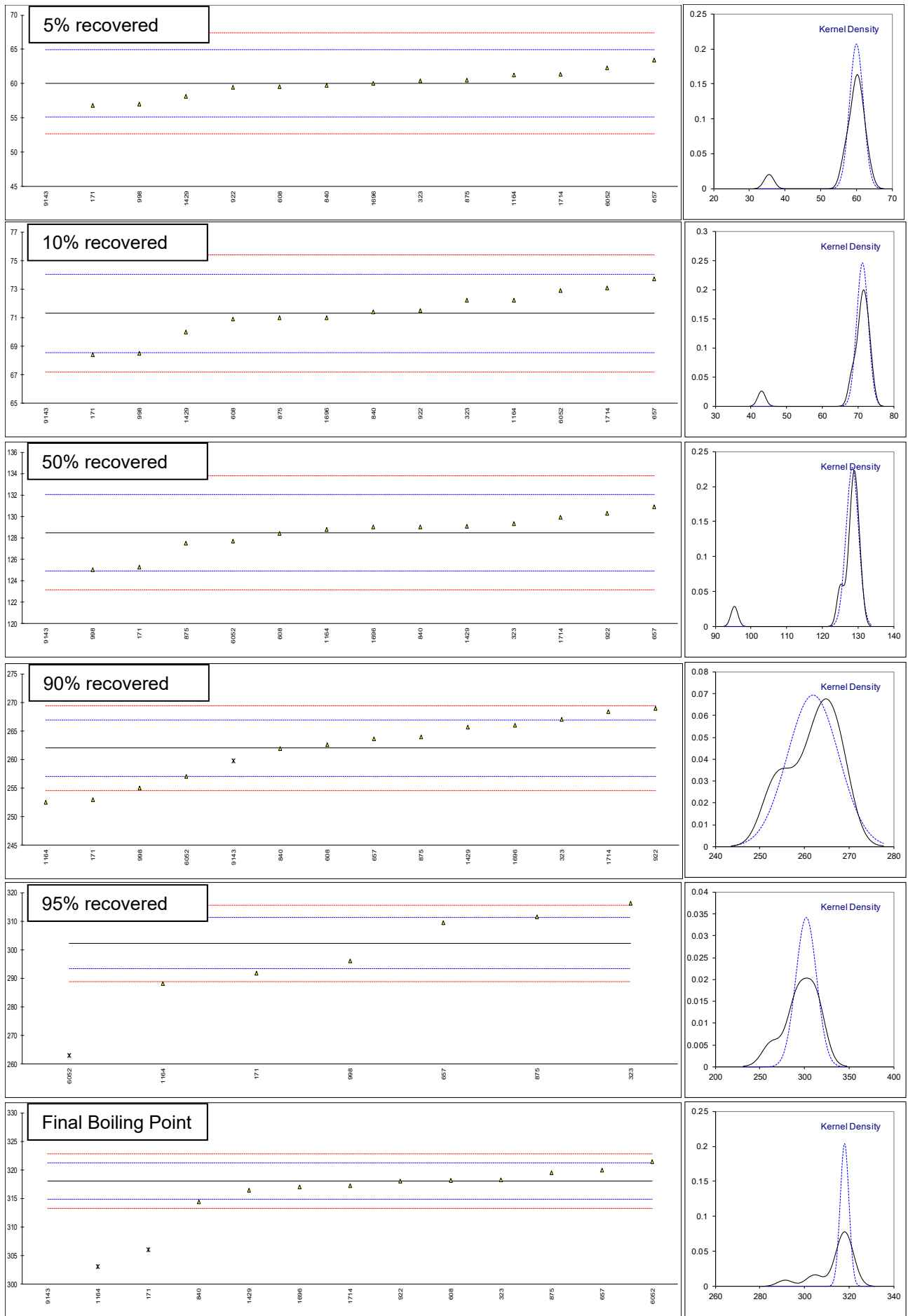
Determination of Methanol on sample #19230; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140		-----		-----	
171		-----		-----	
311		-----		-----	
323	INH-304	<2		-----	
442		-----		-----	
444		-----		-----	
608		-----		-----	
609		-----		-----	
657	INH-0130	0.4		-----	
785		-----		-----	
840		-----		-----	
874		-----		-----	
875		-----		-----	
922		-----		-----	
998		-----		-----	
1164		-----		-----	
1397		-----		-----	
1429	In house	0.5		-----	
1696		-----		-----	
1714		-----		-----	
1815		-----		-----	
1957		-----		-----	
1960		-----		-----	
2124		-----		-----	
6052		-----		-----	
6087		-----		-----	
6234		-----		-----	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9061		-----		-----	
9099		-----		-----	
9101		-----		-----	
9107		-----		-----	
9141		-----		-----	
9143		-----		-----	

n	3	
mean (n)	<2	

Determination of Mercury, Total on sample #19230; results in µg/kg

lab	method	value	mark	z(targ)	remarks
140		-----		-----	
171	UOP938	163.632		0.13	
311	UOP938	153		-0.19	
323	UOP938	198		1.15	
442		-----		-----	
444	UOP938	126.47		-0.98	
608		-----		-----	
609	In House	150.7		-0.25	
657		-----		-----	
785		-----		-----	
840	EPA7470	145.5		-0.41	
874		-----		-----	
875		-----		-----	
922		-----		-----	
998		-----		-----	
1164		-----		-----	
1397		179		0.59	
1429	In house	320	G(0.01)	4.79	
1696	UOP938	148.86		-0.31	
1714		-----		-----	
1815		-----		-----	
1957	UOP938	81.0		-2.33	
1960	UOP938	219.34		1.79	
2124		-----		-----	
6052	UOP938	ND		-----	
6087	UOP938	179.821		0.61	
6234		-----		-----	
9054	UOP938	174.45		0.45	
9055	D6722	183		0.71	
9056		-----		-----	
9061		-----		-----	
9099		-----		-----	
9101		-----		-----	
9107	D7622	126.5		-0.97	
9141		-----		-----	
9143		-----		-----	

normality	suspect
n	14
outliers	1
mean (n)	159.234
st.dev. (n)	34.4397
R(calc.)	96.431
st.dev.(Horwitz)	33.5939
R(Horwitz)	94.063
Compare	
R(UOP938:10)	14.953
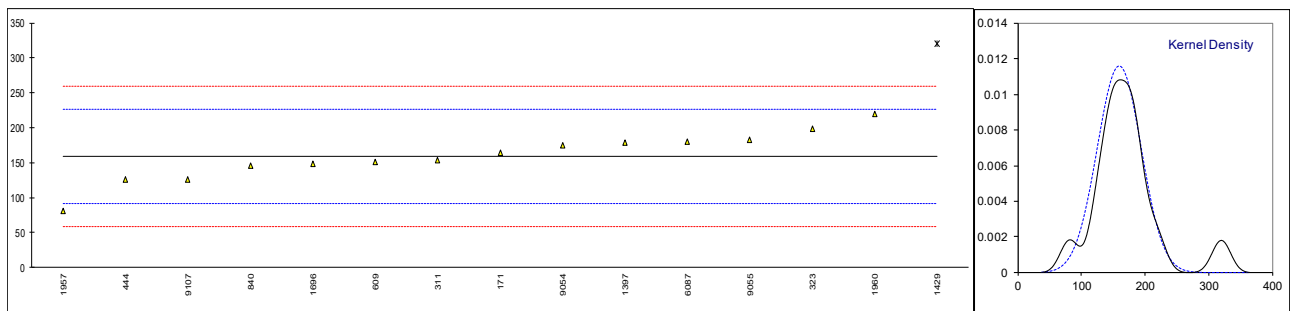
Determination of Sulfur, Total on sample #19230; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140	D2622	71.2		2.66	
171	D5453	53.81		-1.27	
311	D5453	57.0		-0.55	
323	D5453	68		1.93	
442		-----		-----	
444	D5453	43.47		-3.60	
608		-----		-----	
609		-----		-----	
657	D5453	64		1.03	
785		-----		-----	
840	D5453	55.9		-0.80	
874		-----		-----	
875	ISO20846	63		0.81	
922	D5453	62.1		0.60	
998		-----		-----	
1164	D5453	35.4	DG(0.05)	-5.42	
1397	D2622	63.0		0.81	
1429	D5453	54.7		-1.07	
1696		-----		-----	
1714		-----		-----	
1815	D5453	60.31		0.20	
1957		-----		-----	
1960	D5453	64.69		1.19	
2124	D5453	63.59		0.94	
6052	D5453	46.5		-2.92	
6087	D5453	59.5508		0.03	
6234		-----		-----	
9054		-----		-----	
9055		-----		-----	
9056		30	C,DG(0.05)	-6.64	First reported 10
9061		-----		-----	
9099		-----		-----	
9101		-----		-----	
9107		-----		-----	
9141		-----		-----	
9143		-----		-----	

normality	OK
n	16
outliers	2
mean (n)	59.426
st.dev. (n)	7.3279
R(calc.)	20.518
st.dev.(D5453:19a)	4.4313
R(D5453:19a)	12.408
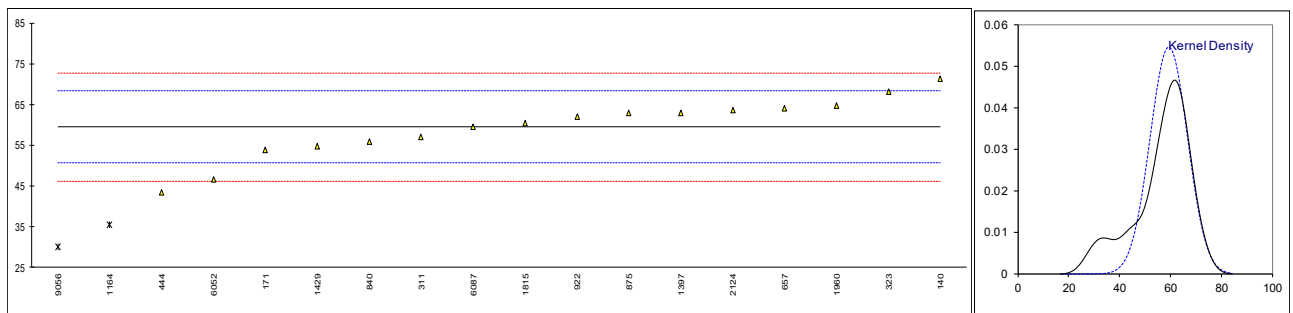
Determination of Water on sample #19230; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140	D4928	73.8		0.34	
171	D6304	42.1038		-0.15	
311		-----		-----	
323	D6304-A	40		-0.18	
442	IP438	45.5		-0.10	
444	D6304-A	41		-0.17	
608	D4928	58		0.10	
609	D4928	92.4		0.63	
657	D6304-A	43		-0.14	
785		-----		-----	
840	D6304	63.8		0.19	
874		-----		-----	
875		-----		-----	
922	D6304-A	38		-0.21	
998		-----		-----	
1164	D6304-A	38		-0.21	
1397	ISO12937	45		-0.11	
1429	IP438	35.9		-0.25	
1696	D6304-A	58.53		0.10	
1714	D6304-C	56		0.06	
1815	ISO12937	46.22		-0.09	
1957	D6304-A	100		0.75	
1960	D4928	56		0.06	
2124	D4928	40	C	-0.18	First reported 0.01550 %M/M
6052	D6304-A	32.87		-0.29	
6087	D6304	47.15		-0.07	
6234		-----		-----	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9061	D4928	40		-0.18	
9099		-----		-----	
9101		-----		-----	
9107	D6304	59		0.11	
9141		-----		-----	
9143		-----		-----	

normality	not OK		
n	23		
outliers	0		
mean (n)	51.838		
st.dev. (n)	17.3296		
R(calc.)	48.523		
st.dev.(D6304:16e1)	64.4620	mass inj	
R(D6304:16e1)	180.494	mass inj	
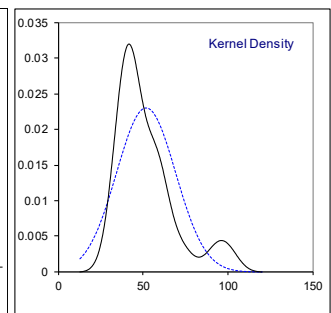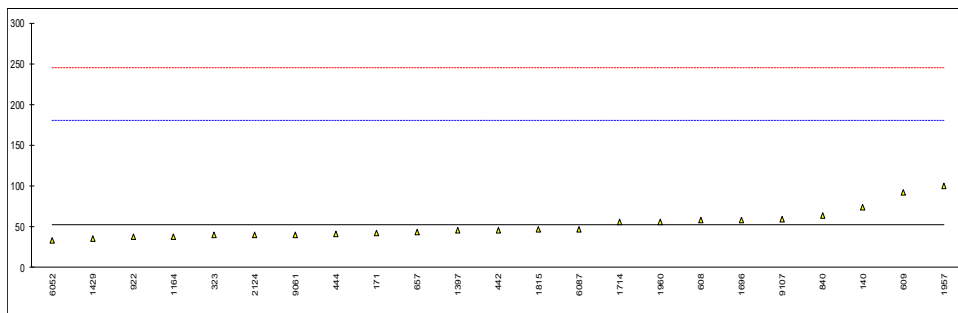
Determination of Simulated Distillation on sample #19230; results in °C

lab	method	IBP	5%rec	10%rec	50%rec	90%rec	95%rec	FBP
140		-----	-----	-----	-----	-----	-----	-----
171	D2887	19.1	30.6	51.2	125.3	265.8	302.6	403.2
311	D2887	<36.0	<36.0	40.5	119.5	264.5	302.5	401.5
323		-----	-----	-----	-----	-----	-----	-----
442		-----	-----	-----	-----	-----	-----	-----
444		-----	-----	-----	-----	-----	-----	-----
608	D2887	-7.5	24.62	58.1	126.15	268.97	303.9	405.72
609		-----	-----	-----	-----	-----	-----	-----
657		-----	-----	-----	-----	-----	-----	-----
785		-----	-----	-----	-----	-----	-----	-----
840		-----	-----	-----	-----	-----	-----	-----
874		-----	-----	-----	-----	-----	-----	-----
875		-----	-----	-----	-----	-----	-----	-----
922		-----	-----	-----	-----	-----	-----	-----
998		-----	-----	-----	-----	-----	-----	-----
1164		-----	-----	-----	-----	-----	-----	-----
1397	D2887	16	23	43	124	260	299	390
1429		-----	-----	-----	-----	-----	-----	-----
1696		-----	-----	-----	-----	-----	-----	-----
1714	In house	5.6	33.30	50.9	130.0	272.2	327.6 G(0.05)	438.2
1815		-----	-----	-----	-----	-----	-----	-----
1957		-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----
6052	D2887	-17.0	30.6	35.5	115.0	254.8	295.7	372.7
6087		-----	-----	-----	-----	-----	-----	-----
6234		-----	-----	-----	-----	-----	-----	-----
9054		-----	-----	-----	-----	-----	-----	-----
9055		-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----
9099		-----	-----	-----	-----	-----	-----	-----
9101		-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----
9141		-----	-----	-----	-----	-----	-----	-----
9143		-----	-----	-----	-----	-----	-----	-----
	normality	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	n	6	6	6	6	6	5	6
	outliers	0	0	0	0	0	1	0
	mean (n)	<36	<36	46.53	123.33	264.38	300.74	401.89
	st.dev. (n)	n.a.	n.a.	8.310	5.307	6.248	3.353	21.580
	R(calc.)	n.a.	n.a.	23.27	14.86	17.50	9.39	60.42
	st.dev.(D2887:19a)	n.a.	n.a.	(0.785)	(1.536)	(1.536)	1.786	4.214
	R(D2887:19a)	n.a.	n.a.	(2.20)	(4.3)	(4.3)	5	11.8
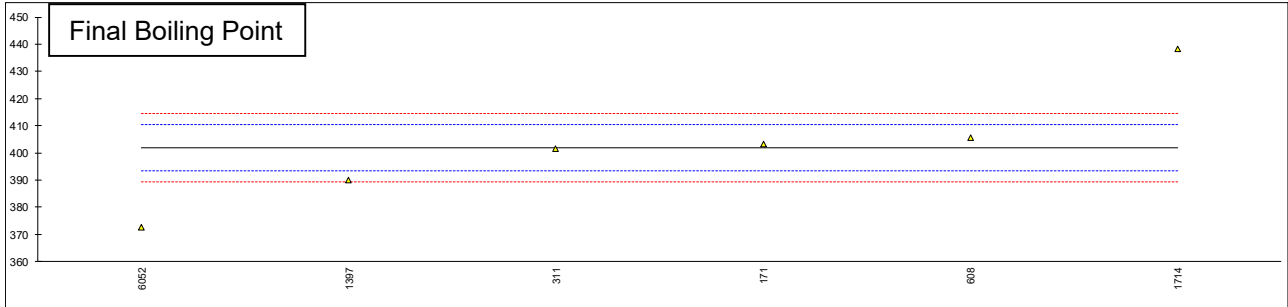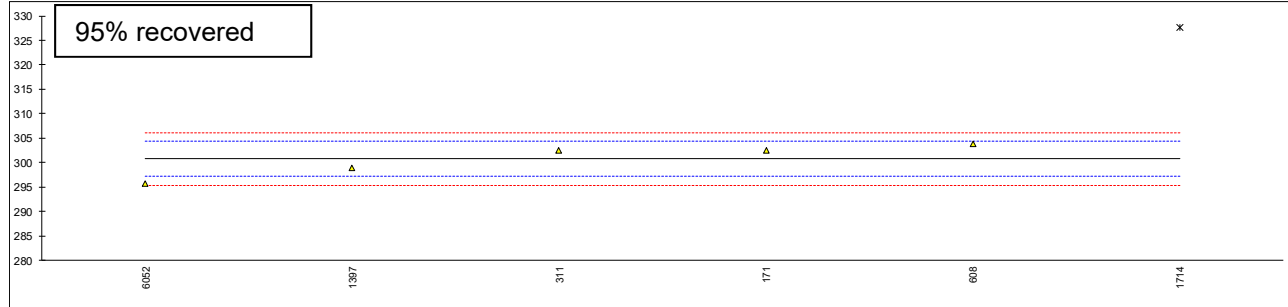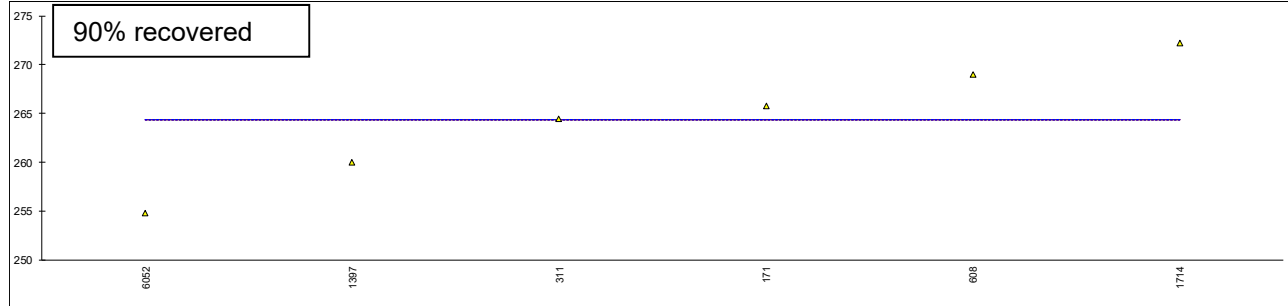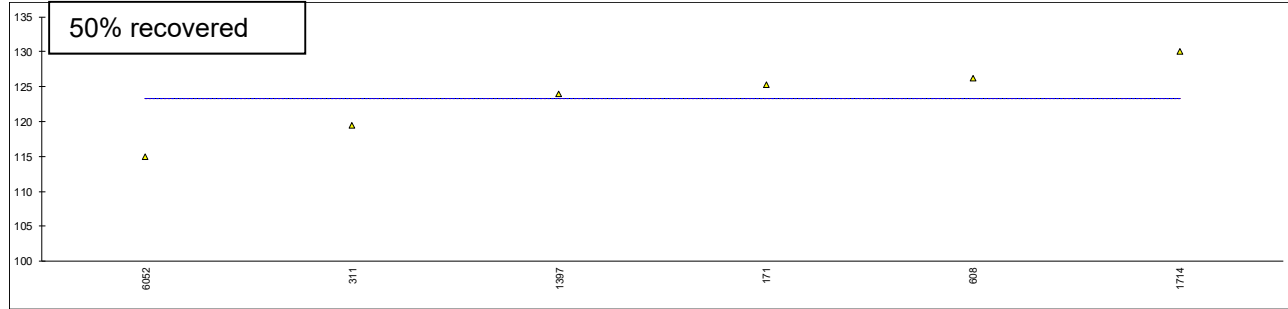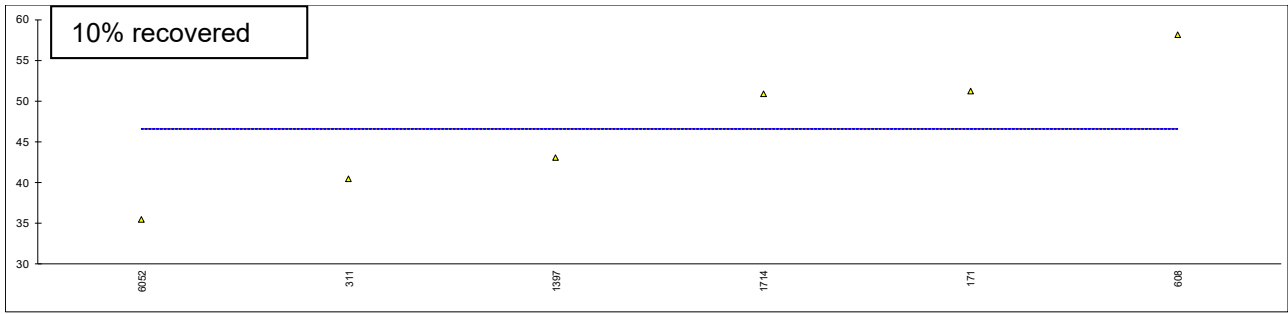
APPENDIX 2

z-scores of Determination of Distillation at 760 mmHg

lab	IBP	5%	10%	50%	90%	95%	FBP
140	-----	-----	-----	-----	-----	-----	-----
171	-0.92	-1.32	-2.13	-1.80	-3.62	-2.31	-7.62
311	-----	-----	-----	-----	-----	-----	-----
323	-0.85	0.16	0.66	0.46	2.03	3.15	0.16
442	-----	-----	-----	-----	-----	-----	-----
444	-----	-----	-----	-----	-----	-----	-----
608	-2.26	-0.17	-0.29	-0.04	0.25	-----	0.09
609	-----	-----	-----	-----	-----	-----	-----
657	-0.78	1.38	1.76	1.37	0.66	1.63	1.23
785	-----	-----	-----	-----	-----	-----	-----
840	-0.86	-0.09	0.09	0.30	-0.02	-----	-2.30
874	-----	-----	-----	-----	-----	-----	-----
875	2.81	0.20	-0.21	-0.55	0.82	2.07	0.92
922	0.04	-0.21	0.15	1.03	2.80	-----	-0.03
998	0.12	-1.22	-2.05	-1.96	-2.81	-1.39	-----
1164	1.30	0.53	0.66	0.18	-3.82	-3.14	-9.45
1397	-----	-----	-----	-----	-----	-----	-----
1429	-0.57	-0.78	-0.95	0.35	1.51	-----	-0.98
1696	0.66	0.00	-0.21	0.30	1.63	-----	-0.67
1714	0.44	0.57	1.32	0.80	2.60	-----	-0.54
1815	-----	-----	-----	-----	-----	-----	-----
1957	-----	-----	-----	-----	-----	-----	-----
1960	-----	-----	-----	-----	-----	-----	-----
2124	-----	-----	-----	-----	-----	-----	-----
6052	0.87	0.94	1.18	-0.44	-2.01	-8.78	2.12
6087	-----	-----	-----	-----	-----	-----	-----
6234	-----	-----	-----	-----	-----	-----	-----
9054	-----	-----	-----	-----	-----	-----	-----
9055	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9099	-----	-----	-----	-----	-----	-----	-----
9101	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9141	-----	-----	-----	-----	-----	-----	-----
9143	-3.48	-9.99	-20.65	-18.63	-0.92	-----	-16.91

z-scores of Determination of Simulated Distillation

lab	IBP	5%	10%	50%	90%	95%	FBP
140	-----	-----	-----	-----	-----	-----	-----
171	-----	-----	-----	-----	-----	1.04	0.31
311	-----	-----	-----	-----	-----	0.99	-0.09
323	-----	-----	-----	-----	-----	-----	-----
442	-----	-----	-----	-----	-----	-----	-----
444	-----	-----	-----	-----	-----	-----	-----
608	-----	-----	-----	-----	-----	1.77	0.91
609	-----	-----	-----	-----	-----	-----	-----
657	-----	-----	-----	-----	-----	-----	-----
785	-----	-----	-----	-----	-----	-----	-----
840	-----	-----	-----	-----	-----	-----	-----
874	-----	-----	-----	-----	-----	-----	-----
875	-----	-----	-----	-----	-----	-----	-----
922	-----	-----	-----	-----	-----	-----	-----
998	-----	-----	-----	-----	-----	-----	-----
1164	-----	-----	-----	-----	-----	-----	-----
1397	-----	-----	-----	-----	-----	-0.97	-2.82
1429	-----	-----	-----	-----	-----	-----	-----
1696	-----	-----	-----	-----	-----	-----	-----
1714	-----	-----	-----	-----	-----	15.04	8.62
1815	-----	-----	-----	-----	-----	-----	-----
1957	-----	-----	-----	-----	-----	-----	-----
1960	-----	-----	-----	-----	-----	-----	-----
2124	-----	-----	-----	-----	-----	-----	-----
6052	-----	-----	-----	-----	-----	-2.82	-6.93
6087	-----	-----	-----	-----	-----	-----	-----
6234	-----	-----	-----	-----	-----	-----	-----
9054	-----	-----	-----	-----	-----	-----	-----
9055	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9099	-----	-----	-----	-----	-----	-----	-----
9101	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9141	-----	-----	-----	-----	-----	-----	-----
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
　5 labs in MALAYSIA
　2 labs in NETHERLANDS
　4 labs in NIGERIA
　2 labs in NORWAY
　　1 lab in PAKISTAN
　　1 lab in POLAND
　3 labs in RUSSIAN FEDERATION
　　1 lab in SINGAPORE
　　1 lab in UNITED ARAB EMIRATES
　6 labs in UNITED KINGDOM
　2 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	= possibly an error in calculations
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature

1   iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018

2   ASTM E178:16

3   ASTM E1301:95(2003)

4   ISO5725:86 (1994)

5   ISO5725, parts 1-6, 1994

6   ISO13528:05

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   IP367:84

10   DIN38402 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)