Results of Proficiency Test
Gascondensate
November 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2008 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Gascondensate every year. During the annual proficiency test program 2020/2021 it was decided to continue the round robin for the analysis of Gascondensate.

In this interlaboratory study 36 laboratories in 16 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the Gascondensate proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample of 0.5L with Gascondensate labelled #20220.
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 100 liters of Gascondensate was made available from the retain materials from earlier PTs on Gascondensate. After homogenization 70 amber glass bottles of 0.5 liter were filled and labelled #20220.
The homogeneity of the subsamples was checked by determination of Density at 15°C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/L
Sample #20220-1	0.74147
Sample #20220-2	0.74148
Sample #20220-3	0.74149
Sample #20220-4	0.74149
Sample #20220-5	0.74158
Sample #20220-6	0.74149
Sample #20220-7	0.74149
Sample #20220-8	0.74157

Table 1: homogeneity test results of subsamples #20220

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)	0.00012
reference test method	ASTM D4052:18a
0.3 * R (reference test method)	0.00067

Table 2: evaluation of the repeatability of subsamples #20220

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

To each of the participating laboratories one sample Gascondensate labelled #20220 was sent on October 14, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

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

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

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

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

According to ISO5725 the original test results per determination were submitted to Dixon’s, Grubbs’ 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 in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.
The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

Some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with two weeks. Four participants reported test results after the final reporting date and three other participants did not report any test results. Not all participants were able to perform all analyzes requested.
In total 33 participants reported 229 numerical test results. Observed were 10 outlying test results, which is 4.4%. In proficiency tests 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 test methods are also in the tables together with the reported test results in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

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

In the iis PT reports ASTM test methods are referred to with a number and if appropriate an indication of sub test method (e.g. D2887-A) and an added designation for the year that the test method was adopted or revised (e.g. D2887-A:19ae2).

Color Saybolt: 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 D6045:20.

Density at 15°C: This determination was not problematic. Three statistical outliers were observed. 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 was problematic. Four statistical outliers were observed and four other test result were excluded over seven parameters. After rejection of the suspect data only the calculated reproducibility at 50% recovered is in agreement with the requirements of the manual mode of ASTM D86:20b. The calculated reproducibilities of Initial Boiling Point, 5%, 10%, 90% recovered and Final Boiling Point were not in agreement. For 95% recovered no z-scores were calculated as the calculated reproducibility was too large compared to the requirements of the manual mode of ASTM D86:20b.
It should be noted that the scope of ASTM D86 does not include Gascondensate, but only products with a limited boiling range like distillate fuels, so the target reproducibilities as used in this report may not be applicable. The use of a simulated distillation determination may be more appropriate.

Methanol: This determination may not be problematic. The three reporting laboratories agreed on a level <50mg/kg. Therefore, no z-scores were calculated.

Total Mercury: The precision requirements of table 3b in test method UOP938 is approximately 6 times stricter than the Horwitz estimate. This means that these requirements will not be met easily. Furthermore, the reproducibility of UOP938 is only available for very low concentrations (0.28 and 12.14 µg/**L**) and conversion and extrapolation 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. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Total Sulfur: 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 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. No statistical outliers were observed over seven parameters. Only the calculated reproducibility for 90% recovered was in agreement with the requirements of procedure A of ASTM D2887:19ae2. The calculated reproducibility for 50% and 95% recovered were not in agreement. For Final Boiling Point no z-scores were calculated as the calculated reproducibility was too large compared to the requirements of procedure A of ASTM D2887:19ae2. The test results reported for Initial Boiling Point, 5% and 10% recovered were not evaluated as the temperature was below the measuring limit of 36°C and/or the number of reported test results was too low.
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 reference test method or as declared by the estimated target reproducibility calculated with the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference test methods (in casu ASTM test methods) or the estimated reproducibility calculated with the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Color Saybolt		16	17.5	1.6	1.2
Density at 15˚C	kg/L	30	0.7419	0.0011	0.0022
Distillation at 760 mmHg					
Initial Boiling Point	°C	14	33.0	11.5	7.3
5% recovered	°C	14	57.2	7.3	6.5
10% recovered	°C	14	68.1	5.0	3.7
50% recovered	°C	14	122.5	4.9	4.8
90% recovered	°C	14	247.0	15.4	6.5
95% recovered	°C	11	280.1	48.2	(10.6)
Final Boiling Point	°C	12	302.0	10.6	4.9
Methanol	mg/kg	3	<50	n.a.	n.a.
Total Mercury	µg/kg	15	336	207	178
Total Sulfur	mg/kg	18	33.2	12.4	8.0
Water	mg/kg	24	43.5	31.6	162.5

Parameter	unit	n	average	2.8 * sd	R(lit)
Simulated Distillation					
Initial Boiling Point	°C	3	<36	n.a.	n.a.
5% recovered	°C	3	<36	n.a.	n.a.
10% recovered	°C	2	40	n.a.	n.a.
50% recovered	°C	3	119.5	9.2	4.3
90% recovered	°C	3	251.2	4.3	4.3
95% recovered	°C	3	289.2	8.1	5
Final Boiling Point	°C	3	379.7	36.5	(11.8)

Table 3: reproducibilities of tests on sample #20220

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 reference test methods. The problematic tests have been discussed in paragraph 4.1.

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

	November 2020	November 2019	November 2018	November 2017	November 2016
Number of reporting laboratories	33	32	32	42	42
Number of test results	229	236	263	333	297
Number of statistical outliers	10	15	18	19	23
Percentage of statistical outliers	4.4%	6.4%	6.8%	5.7%	7.7%

Table 4: comparison with previous proficiency tests

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

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

Determination	November 2020	November 2019	November 2018	November 2017	November 2016
Color Saybolt	-	--	-	--	--
Density at 15°C	++	+	+	++	++
Distillation at 760 mmHg	-	-	+/-	-	-
Methanol	n.e.	n.e.	n.e.	-	--
Total Mercury	-	+/-	--	+	-
Total Sulfur	-	-	+/-	-	+
Water	++	++	++	++	++
Simulated Distillation	--	--	--	+/-	--

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:

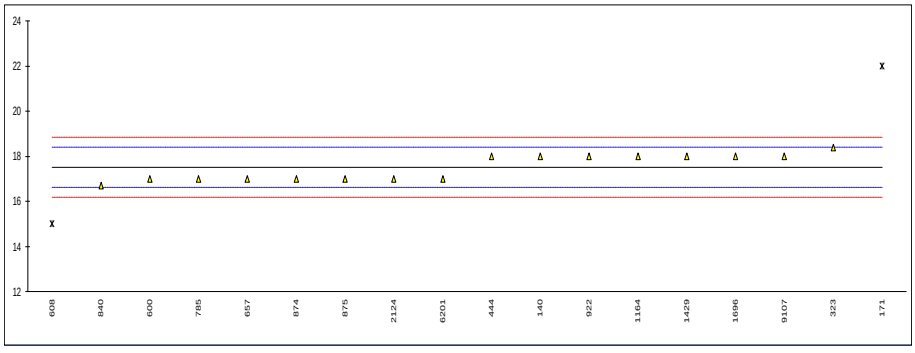
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.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

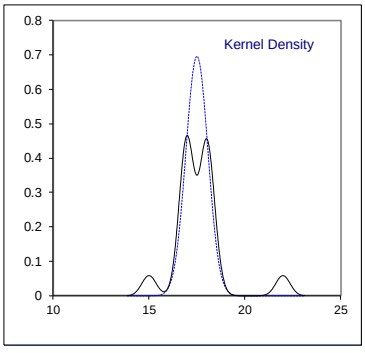
APPENDIX 1

Determination of Color Saybolt on sample #20220;

lab	method	value	mark	z(targ)	remarks
140	D6045	18		1.11	
171	D6045	22	G(0.01)	10.15	
311		-----		-----	
323	D6045	18.4		2.02	
442		-----		-----	
444	D6045	18.0		1.11	
600		17		-1.14	
608	D156	15	C,G(0.05)	-5.66	first reported 16
609		-----		-----	
657	D6045	17	C	-1.14	first reported 15
785	D6045	17		-1.14	
840	D6045	16.7		-1.82	
874	D6045	17		-1.14	
875	D6047	17		-1.14	
922	D6045	18		1.11	
1164	D6045	18		1.11	
1397		-----		-----	
1429	D6045	18		1.11	
1696	D6045	18		1.11	
1815		-----		-----	
1960		-----		-----	
2124	D6045	17		-1.14	
6052		-----		-----	
6087		-----		-----	
6201	D6045	17		-1.14	
9054		-----		-----	
9055		-----	C	-----	first reported 735.7 (see density test results)
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9101		-----		-----	
9107	D156	18		1.11	
9130		-----		-----	
9141		-----		-----	
9143		-----		-----	

normality	OK
n	16
outliers	2
mean (n)	17.51
st.dev. (n)	0.574
R(calc.)	1.61
st.dev.(D6045:20)	0.443
R(D6045:20)	1.24

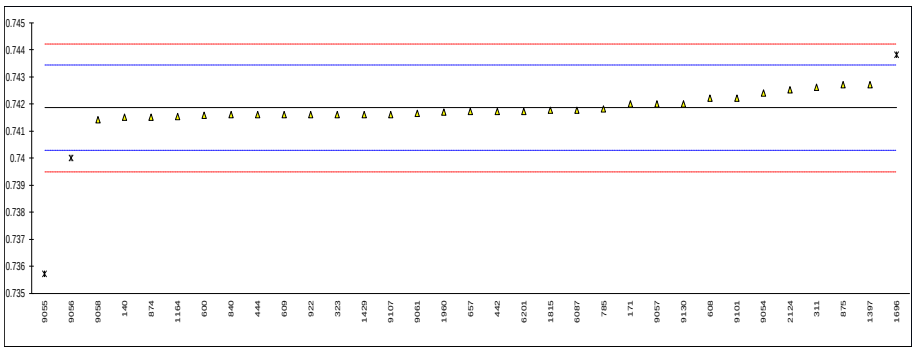


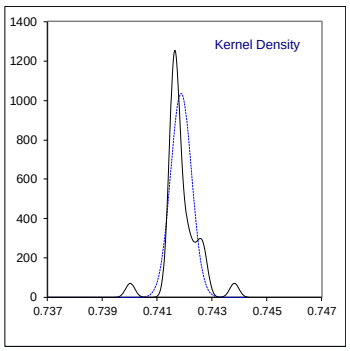


Determination of Density at 15°C on sample #20220; results in kg/L

lab	method	value	mark	z(targ)	remarks
140	D4052	0.7415		-0.45	
171	D4052	0.7420		0.18	
311	D4052	0.7426		0.94	
323	D4052	0.7416		-0.33	
442	IP365	0.7417		-0.20	
444	D4052	0.7416		-0.33	
600	D4052	0.74156		-0.38	
608	D4052	0.7422		0.44	
609	D4052	0.7416		-0.33	
657	D4052	0.7417		-0.20	
785	D5002	0.7418		-0.07	
840	D4052	0.74158		-0.35	
874	D4052	0.7415		-0.45	
875	D4052	0.7427	C	1.07	reported 742.7 kg/L
922	D4052	0.7416		-0.33	
1164	D4052	0.74152		-0.43	
1397	ISO12185	0.7427		1.07	
1429	D4052	0.7416	C	-0.33	first reported 741.6 kg/L
1696	D4052	0.7438	R(0.01)	2.47	
1815	ISO12185	0.74175		-0.13	
1960	D4052	0.741675		-0.23	
2124	D4052	0.7425	C	0.82	first reported 0.7452 kg/m^3
6052		-----		-----	
6087	D4052	0.741753		-0.13	
6201	D4052	0.7417		-0.20	
9054	D4052	0.7424		0.69	
9055	IP365	0.7357	C,R(0.01)	-7.81	first reported as Color Saybolt
9056	In house	0.740	R(0.01)	-2.36	
9057		0.7420	C	0.18	first reported 742.0 without unit
9058		0.7414		-0.58	
9061	D5002	0.74165		-0.26	
9101	D1298	0.74220		0.44	
9107	D4052	0.7416	C	-0.33	first reported 0.7486 kg/L
9130	D4052	0.742		0.18	
9141		-----		-----	
9143		-----		-----	

normality	suspect
n	30
outliers	3
mean (n)	0.74186
st.dev. (n)	0.000385
R(calc.)	0.00108
st.dev.(D4052:18a)	0.000788
R(D4052:18a)	0.00221

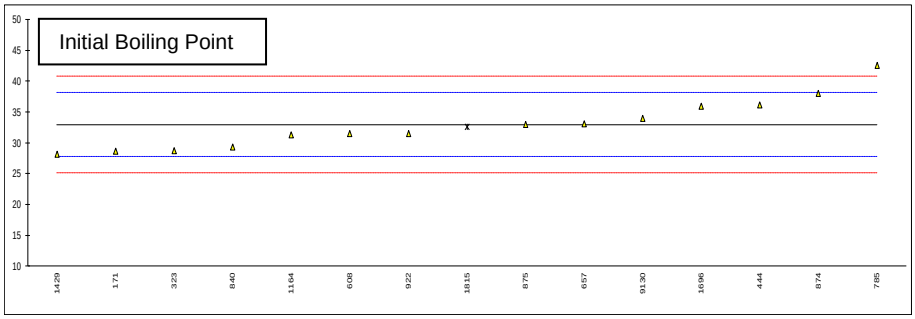


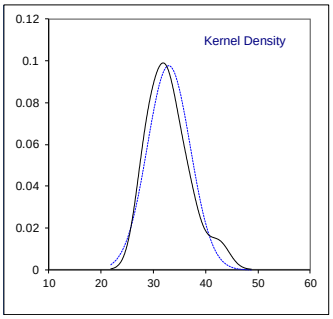


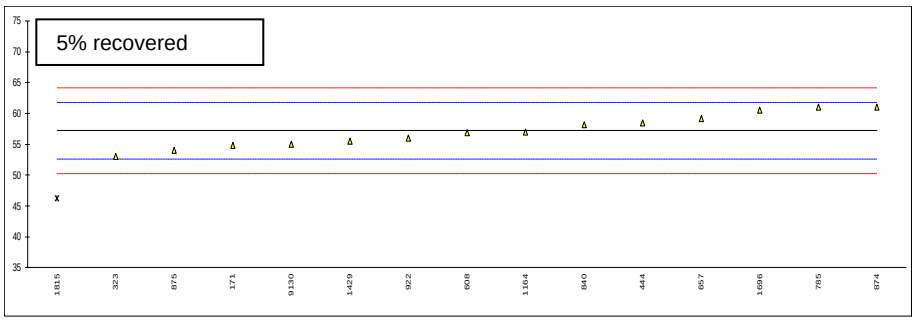
Determination of Distillation at 760 mmHg on sample #20220; results in °C

lab	method	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP	residue (%V/V)	loss (%V/V)
140		-----	-----	-----	-----	-----	-----	-----	-----	-----
171	D86-A	28.6	54.8	66.0	120.4	238.4	278.1	294.1	1.2	1.2
311		-----	-----	-----	-----	-----	-----	-----	-----	-----
323	D86-A	28.7	53.0	65.5	120.0	246.0 C	263.0	299.3	2.0 C	3.7 C
442		-----	-----	-----	-----	-----	-----	-----	-----	-----
444	D86-A	36.1	58.5	68.1	120.8	239.7	279	301.3	1.3	2.2
600		-----	-----	-----	-----	-----	-----	-----	-----	-----
608	D86-A	31.5	56.9	68.6	123.7	253.9	254.9	302.8	1.3	4.9
609		-----	-----	-----	-----	-----	-----	-----	-----	-----
657	D86-A	33.1	59.2	69.5	123.8	251.0	299.9	303.6	1.8	2.5
785	D86-M	42.5	61.0	70.0	125.5	248.5	290.0	291.0 DG5	2.2	1.8
840	D86-A	29.30	58.21	68.91	121.48	241.16	-----	301.72	1.3	2.9
874	D86-M	38.0	61.0	71.0	123.0	251.0	295.0	299.0	2.0	2.0
875	D86-M	33.0	54.0	66.0	120.0	242.5	283.5	304.0	-----	-----
922	D86-M	31.5	56.0	67.5	123.0	252.0	-----	304.5	-----	-----
1164	D86-A	31.3	57.0	68.1	123.0	240.6	278.7	300.2	2.1	0.8
1397		-----	-----	-----	-----	-----	-----	-----	-----	-----
1429	D86-A	28.1	55.5	67.0	123.4	252.7	253.7	303.5	1.3	4.8
1696	D86-A	35.9	60.5	70.9	124.8	252.6	-----	-----	-----	-----
1815	ISO3405-A	32.60 ex	46.20 G5	59.95 G5	117.10 ex	233.20 ex	267.10 ex	283.90 DG5	2.30	2.60
1960		-----	-----	-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----	-----	-----
6052		-----	-----	-----	-----	-----	-----	-----	-----	-----
6087		-----	-----	-----	-----	-----	-----	-----	-----	-----
6201		-----	-----	-----	-----	-----	-----	-----	-----	-----
9054		-----	-----	-----	-----	-----	-----	-----	-----	-----
9055		-----	-----	-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----	-----	-----
9057		-----	-----	-----	-----	-----	-----	-----	-----	-----
9058		-----	-----	-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----	-----	-----
9101		-----	-----	-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----	-----	-----
9130	D86-A	34	55	66.8	122.1	248.1	304.7	309.7	1	3.41
9141		-----	-----	-----	-----	-----	-----	-----	-----	-----
9143		-----	-----	-----	-----	-----	-----	-----	-----	-----
normality		OK	OK	OK	OK	OK	OK	suspect		
n		14	14	14	14	14	11	12		
outliers		0 (+1 ex)	1	1	0 (+1 ex)	0 (+1 ex)	0 (+1 ex)	2		
mean (n)		32.97	57.19	68.14	122.50	247.01	280.05	301.98		
st.dev. (n)		4.088	2.623	1.788	1.754	5.516	17.206	3.784		
R(calc.)		11.45	7.34	5.01	4.91	15.44	48.18	10.59		
st.dev.(D86-M:20b)		2.609	2.313	1.332	1.705	2.306	(3.781)	1.765		
R(D86-M:20b)		7.30	6.48	3.73	4.77	6.46	(10.59)	4.94		
compare										
R(D86-A:20b)		1.81	1.72	1.50	3.0	3.71	(5.75)	7.1		

ex = excluded due to observed outliers in other Distillation at 760 mmHg parameters
lab 323 first reported 230.0, 95.8, 4.2 respectively



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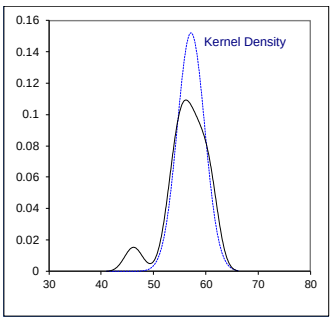
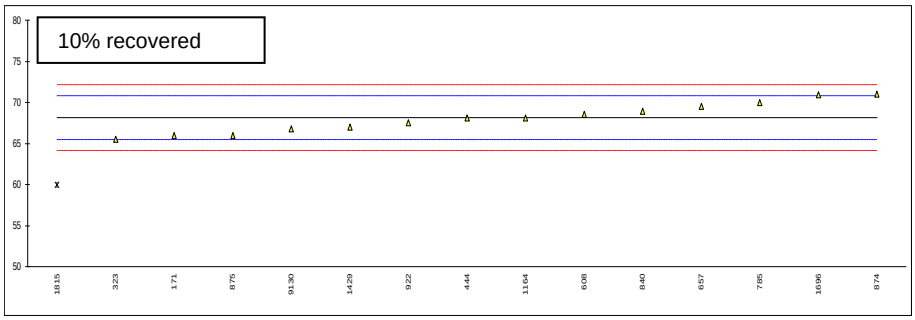

Kernel Density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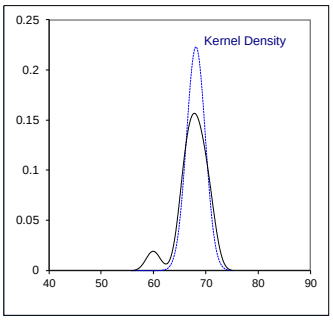
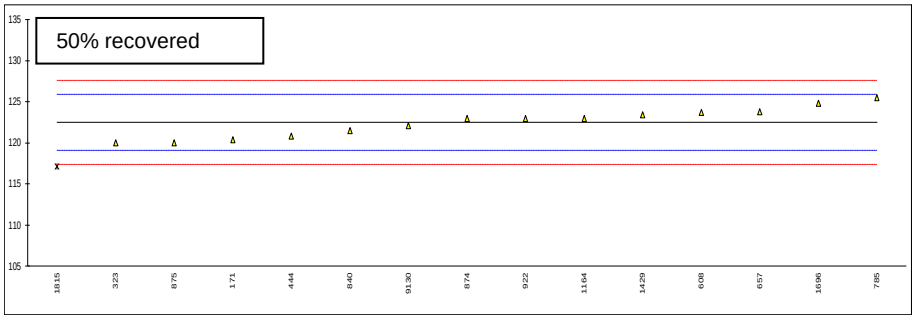

Kernel Density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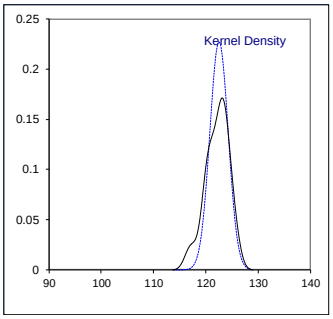
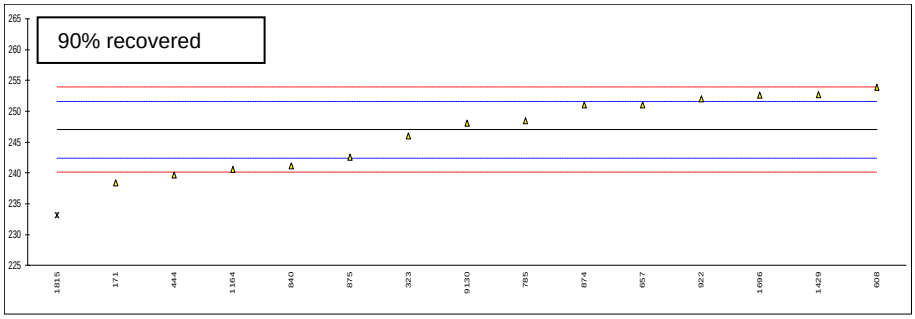

Kernel Density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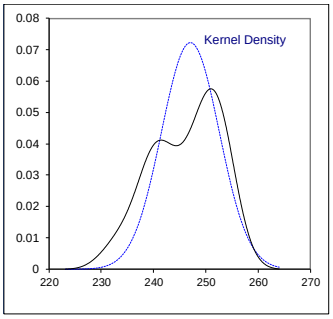
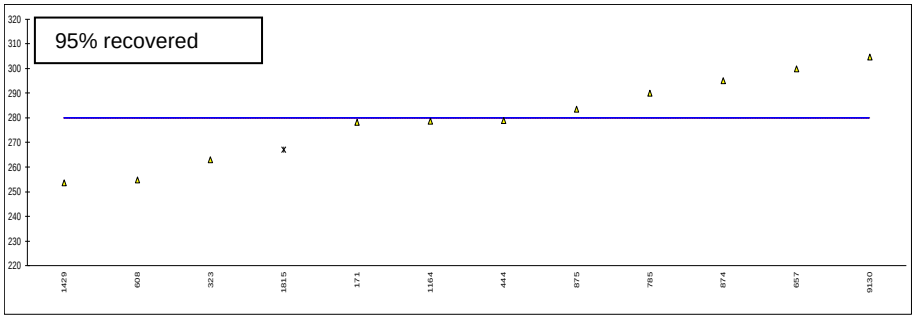
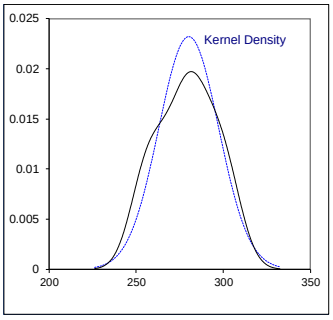
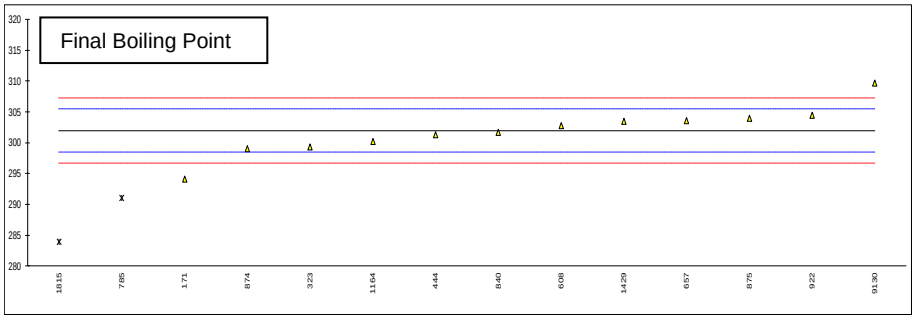
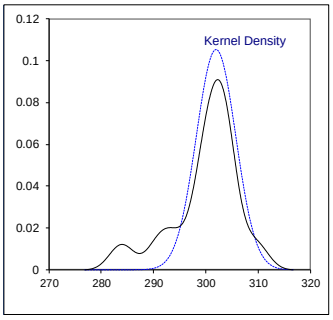

Kernel Density


95% recovered


Kernel Density


Final Boiling Point


Kernel Density

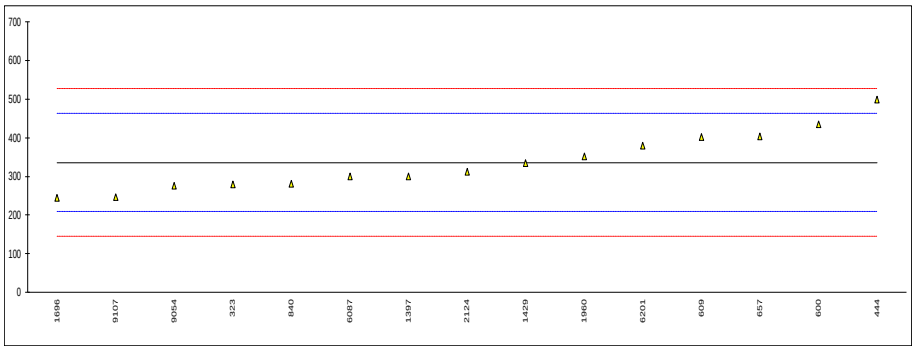
Determination of Methanol on sample #20220; results in mg/kg

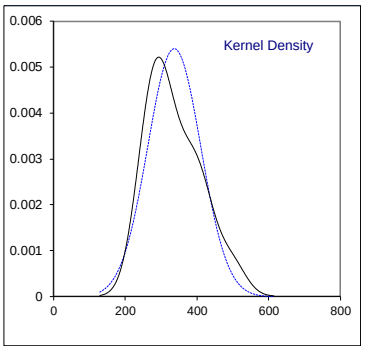
lab	method	value	mark	z(targ)	remarks
140		-----		-----	
171		-----		-----	
311		-----		-----	
323	INH-304	23		-----	
442		-----		-----	
444		-----		-----	
600		-----		-----	
608		-----		-----	
609		-----		-----	
657	INH-0130	44		-----	
785		-----		-----	
840		-----		-----	
874		-----		-----	
875		-----		-----	
922		-----		-----	
1164		-----		-----	
1397		-----		-----	
1429		-----		-----	
1696		-----		-----	
1815		-----		-----	
1960		-----		-----	
2124		-----		-----	
6052		-----		-----	
6087		-----		-----	
6201	D7900	30.2		-----	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9101		-----		-----	
9107		-----		-----	
9130		-----		-----	
9141		-----		-----	
9143		-----		-----	
	n	3			
	mean (n)	<50			

Determination of Total Mercury on sample #20220; results in µg/kg

lab	method	value	mark	z(targ)	remarks
140		-----		-----	
171		-----		-----	
311		-----		-----	
323	UOP938	280		-0.89	
442		-----		-----	
444	UOP938	497.94		2.55	
600	D7622	434.77		1.55	
608		-----		-----	
609	D7622	402		1.04	
657	UOP938	403		1.05	
785		-----		-----	
840	EPA7470A	281.2		-0.87	
874		-----		-----	
875		-----		-----	
922		-----		-----	
1164		-----		-----	
1397	In house	301		-0.56	
1429	In house	334		-0.04	
1696	UOP938	244.566		-1.45	
1815		-----		-----	
1960	UOP938	352.79		0.26	
2124		312.46		-0.38	
6052		-----		-----	
6087	UOP938	299.2910		-0.58	
6201	UOP938	380		0.69	
9054	UOP938	275.77		-0.96	
9055		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9101		-----		-----	
9107	UOP938	246.40		-1.42	
9130		-----		-----	
9141		-----		-----	
9143		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	336.346
st.dev. (n)	73.8371
R(calc.)	206.744
st.dev.(Horwitz)	63.4062
R(Horwitz)	177.537
compare	
R(UOP938:10)	31.584

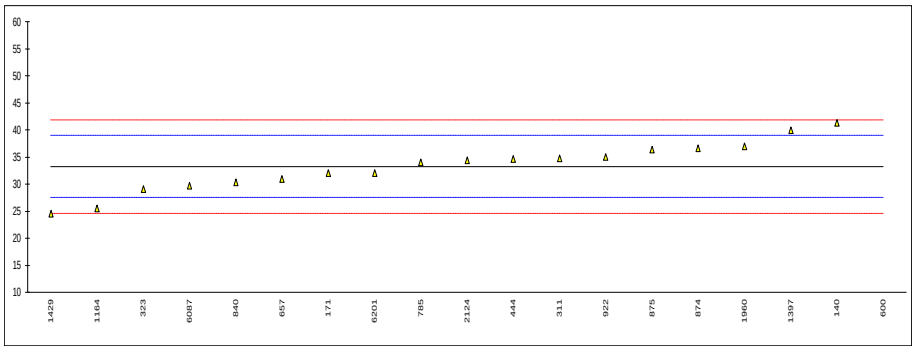


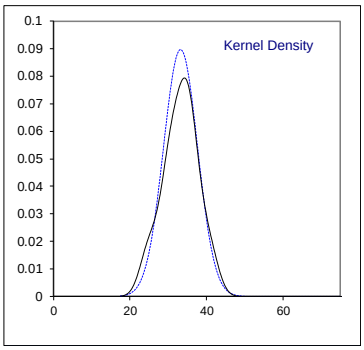


Determination of Total Sulfur on sample #20220; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140	D2622	41.28		2.80	
171	D5453	32		-0.43	
311	D5453	34.8		0.54	
323	D5453	29.1		-1.45	
442		-----		-----	
444	D5453	34.7		0.51	
600	D4294	146	G(0.01)	39.34	
608		-----		-----	
609		-----		-----	
657	D5453	31		-0.78	
785	ISO20884	34.0		0.26	
840	D5453	30.3		-1.03	
874	D4294	36.6	C	1.17	reported in a different unit
875	ISO20846	36.4		1.10	
922	D4294	35		0.61	
1164	D5453	25.56		-2.68	
1397	D2622	40		2.36	
1429		24.58		-3.02	
1696		-----		-----	
1815		-----		-----	
1960	D5453	36.94		1.29	
2124	D5453	34.44		0.42	
6052		-----		-----	
6087	D5453	29.7		-1.24	
6201	D5453	32.0		-0.43	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9101		-----		-----	
9107		-----		-----	
9130		-----		-----	
9141		-----		-----	
9143		-----		-----	

normality	OK
n	18
outliers	1
mean (n)	33.244
st.dev. (n)	4.4416
R(calc.)	12.436
st.dev.(D5453:19a)	2.8664
R(D5453:19a)	8.026

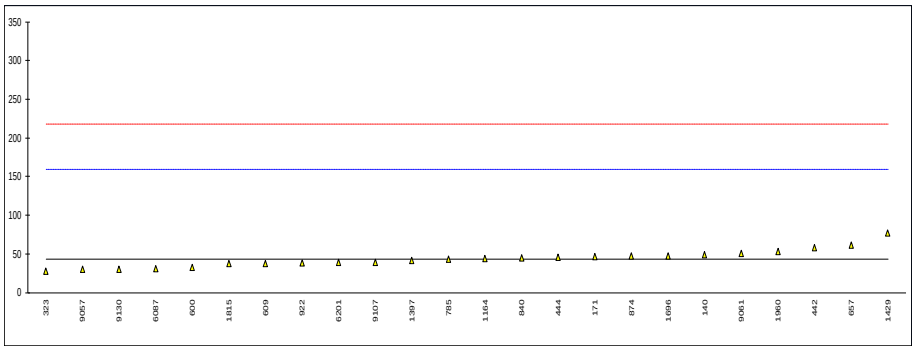


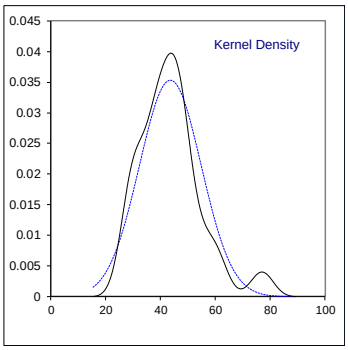


Determination of Water on sample #20220; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140	D6304-A	48.8		0.09	
171	D6304-A	46		0.04	
311		-----		-----	
323	D6304-A	27.6		-0.27	
442	IP438	58		0.25	
444	D6304-A	45		0.03	
600	D6304-A	32		-0.20	
608		-----		-----	
609	D4928	37		-0.11	
657	D6304-A	61		0.30	
785	D6304	43		-0.01	
840	D6304-A	44.2		0.01	
874	D6304	47		0.06	
875		-----		-----	
922	D6304-A	38		-0.10	
1164	D6304-A	44		0.01	
1397	ISO12937	41		-0.04	
1429	IP438	77.0		0.58	
1696	D6304-A	47.04		0.06	
1815	ISO12937	36.78		-0.12	
1960	D4928	53		0.16	
2124		-----		-----	
6052		-----		-----	
6087	D4928	30.2		-0.23	
6201	D6304-A	38.85		-0.08	
9054		-----		-----	
9055		-----		-----	
9056	In house	-----		-----	reported 900 %V/V
9057		30		-0.23	
9058		-----		-----	
9061	D4928	50		0.11	
9101		-----		-----	
9107	D6304-A	39	C	-0.08	first reported 0.0389 mg/kg
9130	D6304-A	30		-0.23	
9141		-----		-----	
9143		-----		-----	

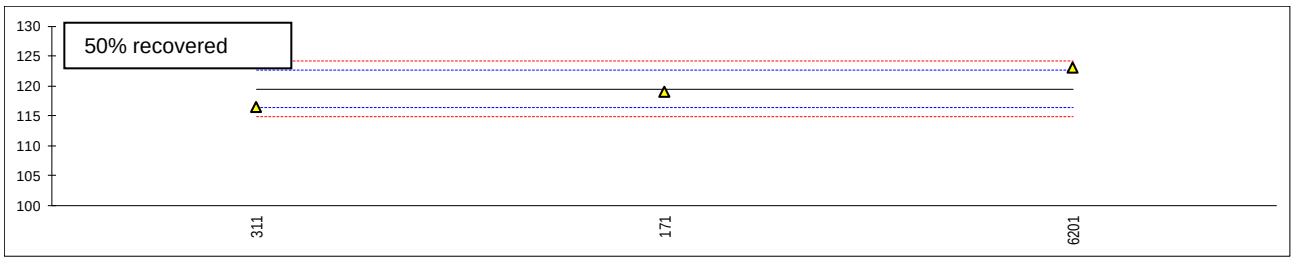
normality	not OK	
n	24	
outliers	0	
mean (n)	43.520	
st.dev. (n)	11.2787	
R(calc.)	31.580	
st.dev.(D6304:16e1)	58.0398	(mass inj)
R(D6304:16e1)	162.511	(mass inj)

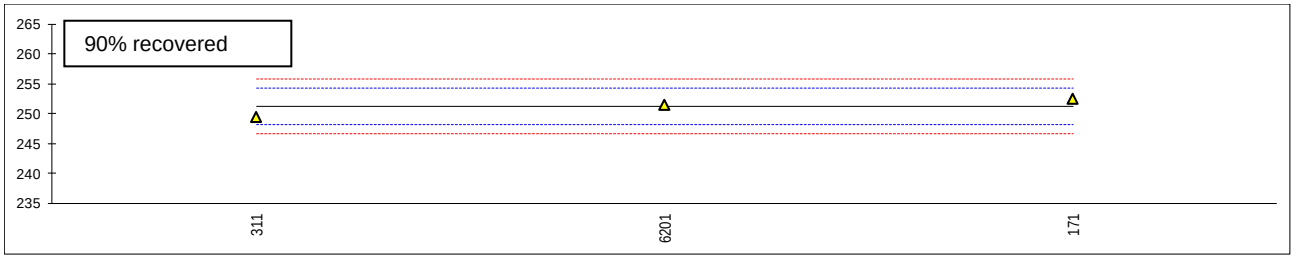


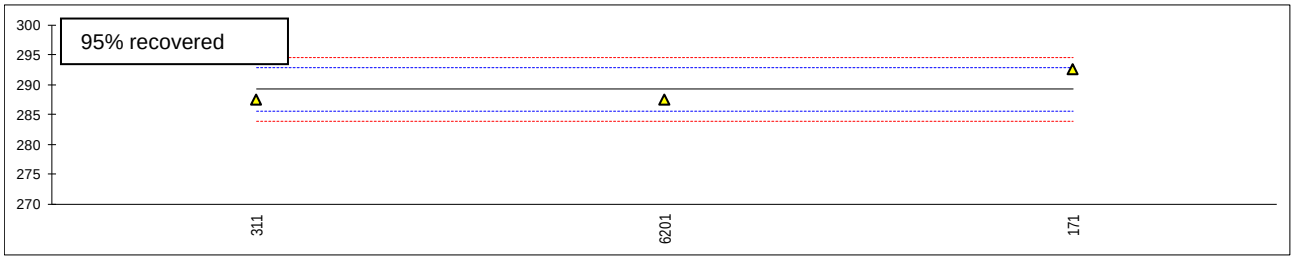


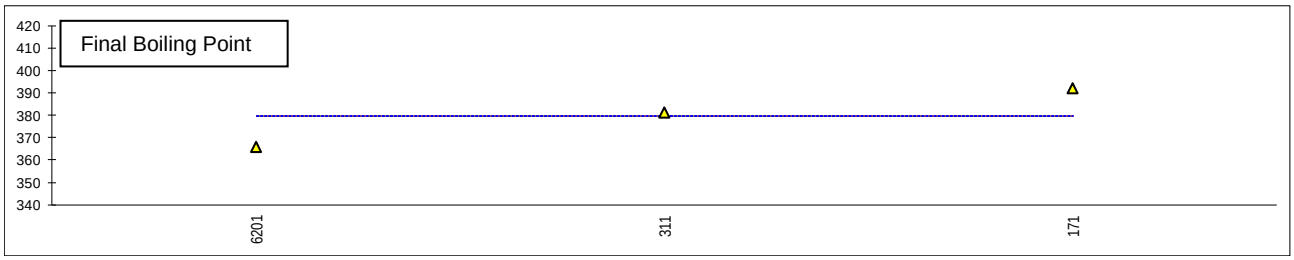
Determination of Simulated Distillation on sample #20220; results in °C

lab	method	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP
140		-----	-----	-----	-----	-----	-----	-----
171	D2887	19.5	32.0	39.0	119.0	252.5	292.5	392.0
311	D2887	<36.0	<36.0	41.0	116.5	249.5	287.5	381.0
323		-----	-----	-----	-----	-----	-----	-----
442		-----	-----	-----	-----	-----	-----	-----
444		-----	-----	-----	-----	-----	-----	-----
600		-----	-----	-----	-----	-----	-----	-----
608		-----	-----	-----	-----	-----	-----	-----
609		-----	-----	-----	-----	-----	-----	-----
657		-----	-----	-----	-----	-----	-----	-----
785		-----	-----	-----	-----	-----	-----	-----
840		-----	-----	-----	-----	-----	-----	-----
874		-----	-----	-----	-----	-----	-----	-----
875		-----	-----	-----	-----	-----	-----	-----
922		-----	-----	-----	-----	-----	-----	-----
1164		-----	-----	-----	-----	-----	-----	-----
1397		-----	-----	-----	-----	-----	-----	-----
1429		-----	-----	-----	-----	-----	-----	-----
1696		-----	-----	-----	-----	-----	-----	-----
1815		-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----
6052		-----	-----	-----	-----	-----	-----	-----
6087		-----	-----	-----	-----	-----	-----	-----
6201	D7619	<36	<36	<36	123	251.5	287.5	366.0
9054		-----	-----	-----	-----	-----	-----	-----
9055		-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----
9057		-----	-----	-----	-----	-----	-----	-----
9058		-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----
9101		-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----
9130		-----	-----	-----	-----	-----	-----	-----
9141		-----	-----	-----	-----	-----	-----	-----
9143		-----	-----	-----	-----	-----	-----	-----
	normality	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	n	3	3	2	3	3	3	3
	outliers	0	0	0	0	0	0	0
	mean (n)	<36	<36	40	119.50	251.17	289.17	379.67
	st.dev. (n)	n.a.	n.a.	n.a.	3.279	1.528	2.887	13.051
	R(calc.)	n.a.	n.a.	n.a.	9.18	4.28	8.08	36.54
	st.dev.(D2887-A:19ae2)	n.a.	n.a.	n.a.	1.536	1.536	1.786	(4.214)
	R(D2887-A:19ae2)	n.a.	n.a.	n.a.	4.3	4.3	5	(11.8)


50% recovered
130
125
120
115
110
105
100
311
171
6201


90% recovered
265
260
255
250
245
240
235
311
6201
171


95% recovered
300
295
290
285
280
275
270
311
6201
171


Final Boiling Point
420
410
400
390
380
370
360
350
340
6201
311
171

APPENDIX 2

z-scores of Determination of Distillation at 760 mmHg

lab	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP
140	-----	-----	-----	-----	-----	-----	-----
171	-1.68	-1.03	-1.60	-1.23	-3.73	-----	-4.46
311	-----	-----	-----	-----	-----	-----	-----
323	-1.64	-1.81	-1.98	-1.47	-0.44	-----	-1.52
442	-----	-----	-----	-----	-----	-----	-----
444	1.20	0.57	-0.03	-1.00	-3.17	-----	-0.38
600	-----	-----	-----	-----	-----	-----	-----
608	-0.56	-0.12	0.35	0.70	2.99	-----	0.47
609	-----	-----	-----	-----	-----	-----	-----
657	0.05	0.87	1.02	0.76	1.73	-----	0.92
785	3.65	1.65	1.40	1.76	0.65	-----	-6.22
840	-1.41	0.44	0.58	-0.60	-2.54	-----	-0.15
874	1.93	1.65	2.15	0.29	1.73	-----	-1.69
875	0.01	-1.38	-1.60	-1.47	-1.96	-----	1.15
922	-0.56	-0.51	-0.48	0.29	2.16	-----	1.43
1164	-0.64	-0.08	-0.03	0.29	-2.78	-----	-1.01
1397	-----	-----	-----	-----	-----	-----	-----
1429	-1.87	-0.73	-0.85	0.53	2.47	-----	0.86
1696	1.12	1.43	2.07	1.35	2.42	-----	-----
1815	-0.14	-4.75	-6.14	-3.17	-5.99	-----	-10.24
1960	-----	-----	-----	-----	-----	-----	-----
2124	-----	-----	-----	-----	-----	-----	-----
6052	-----	-----	-----	-----	-----	-----	-----
6087	-----	-----	-----	-----	-----	-----	-----
6201	-----	-----	-----	-----	-----	-----	-----
9054	-----	-----	-----	-----	-----	-----	-----
9055	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9057	-----	-----	-----	-----	-----	-----	-----
9058	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9101	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9130	0.39	-0.95	-1.00	-0.23	0.47	-----	4.38
9141	-----	-----	-----	-----	-----	-----	-----
9143	-----	-----	-----	-----	-----	-----	-----

z-scores of Determination of Simulated Distillation

lab	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP
140	-----	-----	-----	-----	-----	-----	-----
171	-----	-----	-----	-0.33	0.87	1.87	-----
311	-----	-----	-----	-1.95	-1.09	-0.93	-----
323	-----	-----	-----	-----	-----	-----	-----
442	-----	-----	-----	-----	-----	-----	-----
444	-----	-----	-----	-----	-----	-----	-----
600	-----	-----	-----	-----	-----	-----	-----
608	-----	-----	-----	-----	-----	-----	-----
609	-----	-----	-----	-----	-----	-----	-----
657	-----	-----	-----	-----	-----	-----	-----
785	-----	-----	-----	-----	-----	-----	-----
840	-----	-----	-----	-----	-----	-----	-----
874	-----	-----	-----	-----	-----	-----	-----
875	-----	-----	-----	-----	-----	-----	-----
922	-----	-----	-----	-----	-----	-----	-----
1164	-----	-----	-----	-----	-----	-----	-----
1397	-----	-----	-----	-----	-----	-----	-----
1429	-----	-----	-----	-----	-----	-----	-----
1696	-----	-----	-----	-----	-----	-----	-----
1815	-----	-----	-----	-----	-----	-----	-----
1960	-----	-----	-----	-----	-----	-----	-----
2124	-----	-----	-----	-----	-----	-----	-----
6052	-----	-----	-----	-----	-----	-----	-----
6087	-----	-----	-----	-----	-----	-----	-----
6201	-----	-----	-----	2.28	0.22	-0.93	-----
9054	-----	-----	-----	-----	-----	-----	-----
9055	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9057	-----	-----	-----	-----	-----	-----	-----
9058	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9101	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9130	-----	-----	-----	-----	-----	-----	-----
9141	-----	-----	-----	-----	-----	-----	-----
9143	-----	-----	-----	-----	-----	-----	-----

APPENDIX 3

Number of participants per country

3 labs in	AUSTRALIA
1 lab in	BELGIUM
1 lab in	CROATIA
1 lab in	INDONESIA
4 labs in	MALAYSIA
4 labs in	NETHERLANDS
3 labs in	NIGERIA
2 labs in	NORWAY
1 lab in	PAKISTAN
1 lab in	POLAND
3 labs in	RUSSIAN FEDERATION
1 lab in	SINGAPORE
2 labs 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), D1	= outlier in Dixon's outlier test
D(0.05), D5	= straggler in Dixon's outlier test
G(0.01), G1	= outlier in Grubbs' outlier test
G(0.05), G5	= straggler in Grubbs' outlier test
DG(0.01), DG1	= outlier in Double Grubbs' outlier test
DG(0.05), DG5	= straggler in Double Grubbs' outlier test
R(0.01), R1	= outlier in Rosner's outlier test
R(0.05), R5	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

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