Institute for
Interlaboratory Studies

Results of Proficiency Test
Gascondensate
November 2022

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 the analysis of Gascondensate every year. During the annual proficiency test program 2022/2023 it was decided to continue the round robin for the analysis of Gascondensate.

In this interlaboratory study 34 laboratories in 16 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the Gascondensate proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one sample of Gascondensate in a 0.5-liter amber glass bottle labelled #22220.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

A batch of approximately 60 liters of Gascondensate was selected from retain materials from earlier iis PTs on Gascondensate. After homogenization 70 amber glass bottles of 0.5 L were filled and labelled #22220.
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 #22220-1	0.74159
sample #22220-2	0.74158
sample #22220-3	0.74159
sample #22220-4	0.74161
sample #22220-5	0.74159
sample #22220-6	0.74159
sample #22220-7	0.74157
sample #22220-8	0.74157

Table 1: homogeneity test results of subsamples #22220

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 D4052:22
0.3 x R (reference test method)	0.00066

Table 2: evaluation of the repeatability of subsamples #22220

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 0.5 L bottle of Gascondensate labelled #22220 was sent on October 19, 2022. 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 and Water.

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 appendices 1 and 2 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 appendices 1 and 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

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

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

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

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

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

The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples and with the reporting of the test results. Therefore, the reporting time on the data entry portal was extended with another five weeks. Still seven participants reported test results after the extended reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 31 participants reported 204 numerical test results. Observed were 7 outlying test results, which is 3.4%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

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

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

Color Saybolt: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D6045:20.

Density at 15 °C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D4052:22.
It should be taken into account that the reproducibility from ASTM D4052:22 is applicable to petroleum distillates and viscous oils only. Therefore, no precision data are stated in the 2022 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. Four statistical outliers were observed over seven parameters. After rejection of the statistical outliers the calculated reproducibilities of Initial Boiling Point, 5%, 10%, 50% and 95% recovered are in agreement with the requirements of the manual mode of ASTM D86:20b. The calculated reproducibilities of 90% recovered and Final Boiling Point are not in agreement. For Final Boiling Point 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: Only two participants reported a test result. No evaluation could be done.

Total Mercury: The precision requirements of Table B3 in test method UOP938 is approximately 6 times stricter than the Horwitz estimate. This means that these requirements will not be met easily. Furthermore, the reproducibility of UOP938 is only available for very low concentrations (0.28 and 12.14 µg/**L**) and conversion and extrapolation will lead to extra uncertainty. Therefore, it was decided to use the estimated reproducibility calculated with the Horwitz equation for evaluation of the test results.
This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility calculated with the Horwitz equation.

Total Sulfur: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D5453:19a.

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D6304-A:20.
A new version of ASTM D6304 was published in 2020 with major changes. In the 2016 version one precision statement was mentioned for test results based on mass with a broad application range and one based on volume. In the 2020 version all precision statements are based on mass with three different procedures (A - direct injection, B - oven accessory and C - evaporation accessory) each with a different application range. In ASTM D6304:20 the reproducibilities for all three procedures A, B and C are much stricter compared to ASTM D6304:16e1. Although the latest version of ASTM D6304 is published in 2020, two participants mentioned to have used the 2016 version.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference methods are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Color Saybolt		17	17.1	2.0	1.2
Density at 15 ˚C	kg/L	30	0.7419	0.0012	0.0022
Initial Boiling Point	°C	15	33.4	7.3	7.2
5% recovered	°C	15	57.2	5.3	6.4
10% recovered	°C	12	67.9	1.6	3.7
50% recovered	°C	15	122.6	3.9	4.8
90% recovered	°C	15	250.1	14.0	6.8
95% recovered	°C	8	292.9	14.9	13.2

Parameter	unit	n	average	2.8 * sd	R(lit)
Final Boiling Point	°C	14	303.6	14.3	(4.0)
Methanol	mg/kg	2	n.e.	n.e.	n.e.
Total Mercury	µg/kg	12	340.0	189.8	179.2
Total Sulfur	mg/kg	18	33.6	11.9	8.1
Water	mg/kg	24	41.7	19.8	32.5

Table 3: reproducibilities of tests on sample #22220

For results between brackets no z-scores are calculated

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2022 WITH PREVIOUS PTS

	November 2022	November 2021	November 2020	November 2019	November 2018
Number of reporting laboratories	31	35	33	32	32
Number of test results	204	257	229	236	263
Number of statistical outliers	7	14	10	15	18
Percentage of statistical outliers	3.4%	5.4%	4.4%	6.4%	6.8%

Table 4: comparison with previous proficiency tests

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

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

Determination	November 2022	November 2021	November 2020	November 2019	November 2018
Color Saybolt	-	-	-	--	-
Density at 15 °C	+	+	++	+	+
Distillation at 760 mmHg	+/-	+/-	-	-	+/-
Methanol	n.e.	(--)	n.e.	n.e.	n.e.
Total Mercury	+/-	+	-	+/-	--
Total Sulfur	-	+	-	-	+/-
Water	+	+/-	++	++	++

Table 5: comparison determinations to the reference test methods

For results between brackets no z-scores are calculated.

The following performance categories were used:

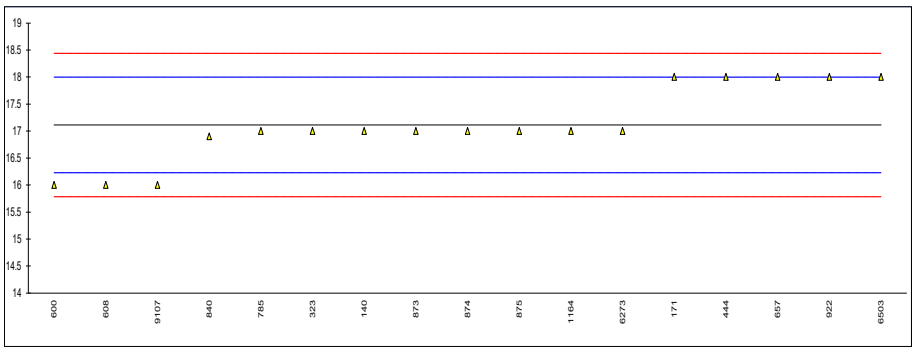
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

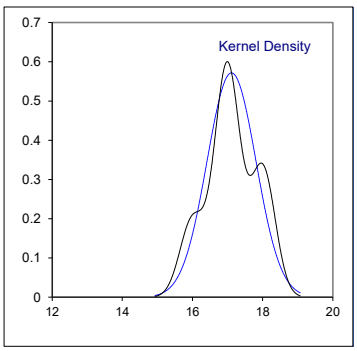
APPENDIX 1

Determination of Color Saybolt on sample #22220;

lab	method	value	mark	z(targ)	remarks
140	D6045	17	C	-0.25	first reported 0.1
171	D6045	18		2.01	
311		-----		-----	
323	D6045	17		-0.25	
442		-----		-----	
444	D6045	18		2.01	
600		16		-2.51	
608	D156	16		-2.51	
609		-----		-----	
657	D6045	18		2.01	
785	D6045	17		-0.25	
840	D6045	16.9		-0.48	
873	D6045	17		-0.25	
874	D6045	17		-0.25	
875	D6045	17		-0.25	
922	D6045	18		2.01	
1164	D6045	17		-0.25	
1488		-----		-----	
1696		-----		-----	
1815		-----		-----	
1960		-----		-----	
6087		-----		-----	
6273	D6045	17		-0.25	
6379		-----		-----	
6447		-----		-----	
6486		-----		-----	
6503	D6045	18		2.01	
9055		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9107	D156	16		-2.51	
9130		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	17.11
st.dev. (n)	0.698
R(calc.)	1.95
st.dev.(D6045:20)	0.443
R(D6045:20)	1.24

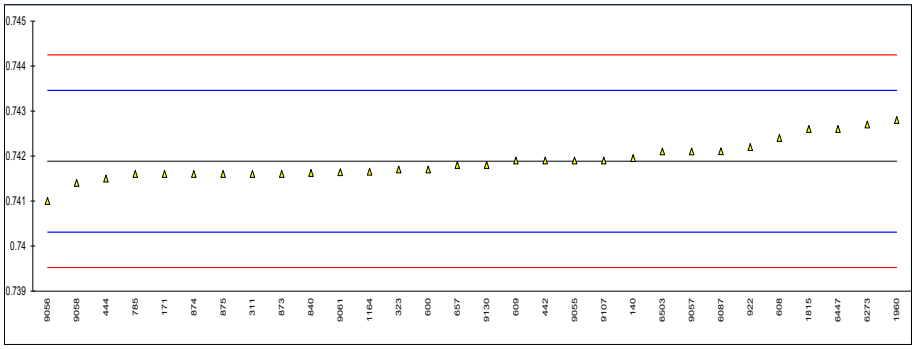


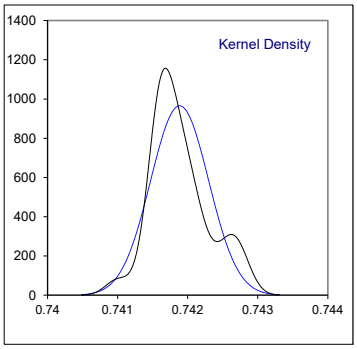


Determination of Density at 15 °C on sample #22220; results in kg/L

lab	method	value	mark	z(targ)	remarks
140	D4052	0.74195		0.08	
171	D4052	0.7416		-0.36	
311	D1298	0.7416		-0.36	
323	D4052	0.7417		-0.24	
442	IP365	0.7419		0.02	
444	D4052	0.7415		-0.49	
600	D4052	0.7417		-0.24	
608	D4052	0.7424		0.65	
609	D4052	0.7419		0.02	
657	D4052	0.7418		-0.11	
785	D4052	0.7416		-0.36	
840	D4052	0.74162		-0.34	
873	D4052	0.7416	C	-0.36	first reported 741.6 kg/L
874	D4052	0.7416		-0.36	
875	D4052	0.7416		-0.36	
922	D4052	0.7422		0.40	
1164	D4052	0.74165		-0.30	
1488		-----		-----	
1696		-----		-----	
1815	ISO12185	0.7426		0.91	
1960	D4052	0.7428		1.16	
6087	D4052	0.742104		0.28	
6273	D4052	0.7427		1.03	
6379		-----		-----	
6447	D4052	0.7426		0.91	
6486		-----		-----	
6503	D4052	0.7421		0.27	
9055	D4052	0.7419		0.02	
9056	In house	0.7410		-1.12	
9057	D5002	0.7421		0.27	
9058	D5002	0.7414		-0.62	
9061	D4052	0.74164		-0.31	
9107	D4052	0.74190		0.02	
9130	D4052	0.7418		-0.11	

normality	OK
n	30
outliers	0
mean (n)	0.74189
st.dev. (n)	0.000413
R(calc.)	0.00116
st.dev.(D4052:22)	0.000788
R(D4052:22)	0.00221

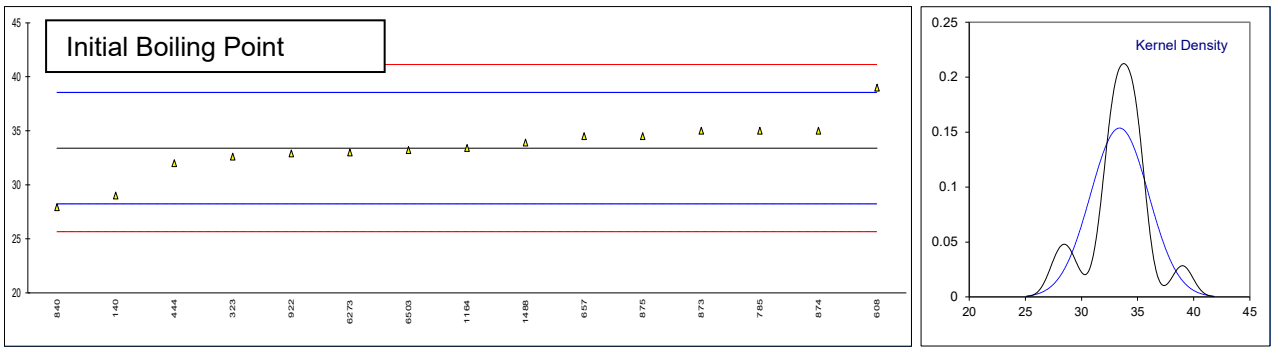


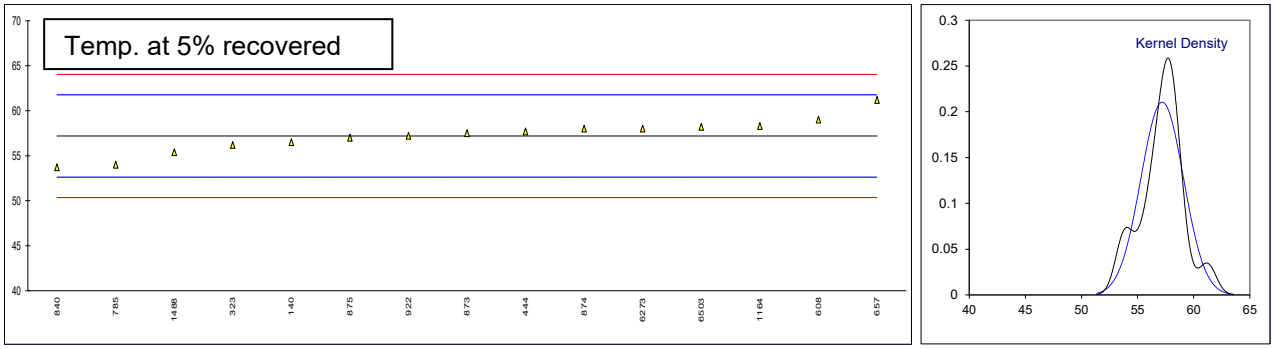


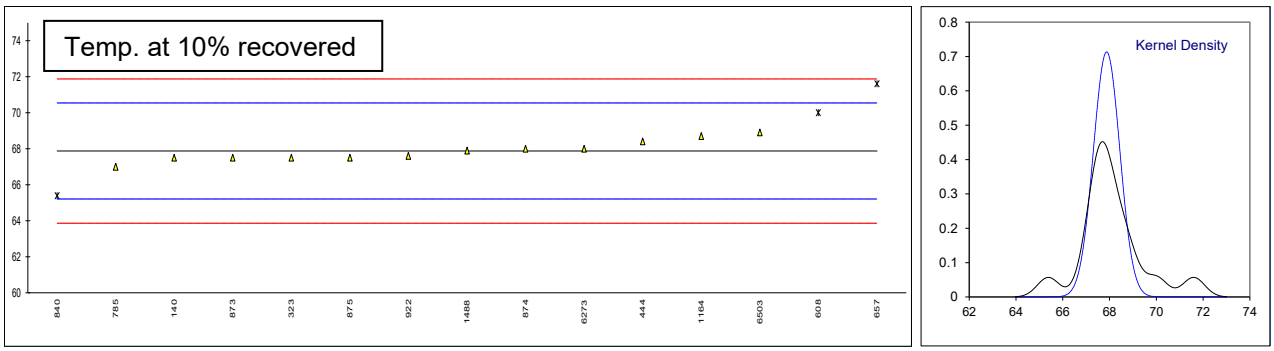
Determination of Distillation at 760 mmHg on sample #22220; results in °C

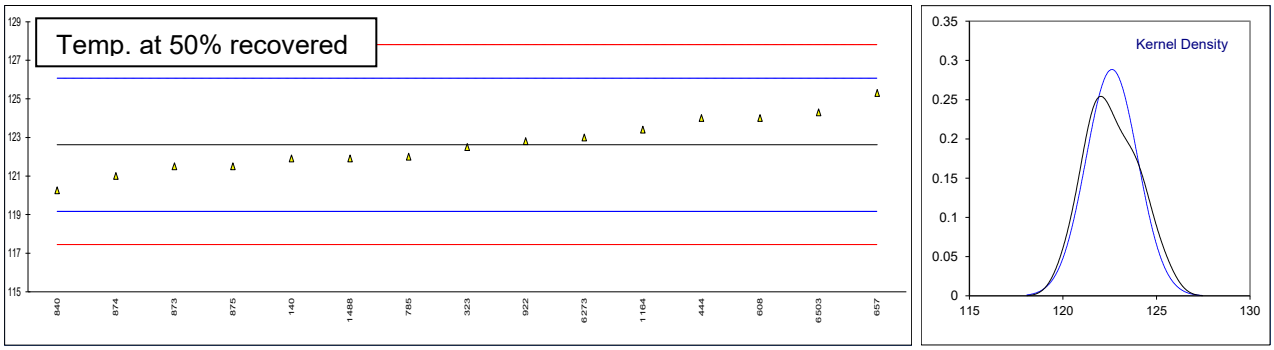
lab	method	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP	residue (%V/V)	loss (%V/V)
140	D86-automated	29.0	56.5	67.5	121.9	247.2	-----	-----	-----	-----
171		-----	-----	-----	-----	-----	-----	-----	-----	-----
311		-----	-----	-----	-----	-----	-----	-----	-----	-----
323	D86-automated	32.6	56.2	67.5	122.5	247.1	-----	305.7	1.3	3.2
442		-----	-----	-----	-----	-----	-----	-----	-----	-----
444	D86-automated	32.0	57.7	68.4	124.0	257.6	258.4 G1	303.1	1.3	4.9
600		-----	-----	-----	-----	-----	-----	-----	-----	-----
608	D86-manual	39.0	59.0	70.0 DG5	124.0	261.0	-----	300.0	1.4	4.6
609		-----	-----	-----	-----	-----	-----	-----	-----	-----
657	D86-automated	34.5 C	61.2 C	71.6 C,DG5	125.3 C	248.7 C	294.7 C	305.1 C	1.5 C	2.4 C
785	D86	35.0	54.0	67.0	122.0	247.0	286.0	300.0	1.5	1.5
840	D86-automated	27.91	53.71	65.38 G5	120.25	244.67	-----	298.81	1.3	3.4
873	D86-manual	35.0	57.5	67.5	121.5	250.5	299.5	308.5	1.5	0.7
874	D86-manual	35.0	58.0	68.0	121.0	249.5	298.0	308.0	1.5	0.7
875	D86-manual	34.5	57.0	67.5	121.5	250.5	292.5	302.0	1.2	0.8
922	D86-automated	32.9	57.2	67.6	122.8	247.9	-----	304.1	1.3	4.2
1164	D86-automated	33.4	58.3	68.7	123.4	244.7	284.6	290.9	3.0	0.8
1488	ISO3405-manual	33.9	55.4	67.9	121.9	248.9	295.9	311.4 C	1.3	2.2
1696		-----	-----	-----	-----	-----	-----	-----	-----	-----
1815		-----	-----	-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----	-----	-----
6087		-----	-----	-----	-----	-----	-----	-----	-----	-----
6273	D86-manual	33.0	58.0	68.0	123.0	248.0	292.0	305.0	1	1
6379		-----	-----	-----	-----	-----	-----	-----	-----	-----
6447		-----	-----	-----	-----	-----	-----	-----	-----	-----
6486		-----	-----	-----	-----	-----	-----	-----	-----	-----
6503	D86-automated	33.2	58.2	68.9	124.3	258.9	-----	307.4	1.4	5.4
9055		-----	-----	-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----	-----	-----
9057		-----	-----	-----	-----	-----	-----	-----	-----	-----
9058		-----	-----	-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----	-----	-----
9130		-----	-----	-----	-----	-----	-----	-----	-----	-----
	normality	suspect	OK	OK	OK	suspect	OK	suspect		
	n	15	15	12	15	15	8	14		
	outliers	0	0	3	0	0	1	0		
	mean (n)	33.39	57.19	67.88	122.62	250.14	292.90	303.57		
	st.dev. (n)	2.595	1.895	0.559	1.383	5.010	5.336	5.118		
	R(calc.)	7.27	5.31	1.57	3.87	14.03	14.94	14.33		
	st.dev.(D86-M:20b)	2.581	2.281	1.334	1.725	2.440	4.705	(1.427)		
	R(D86-M:20b)	7.23	6.39	3.73	4.83	6.83	13.17	(4.00)		
Compare										
	R(D86-A:20b)	1.84	1.72	1.49	3.0	3.75	6.28	(7.1)		

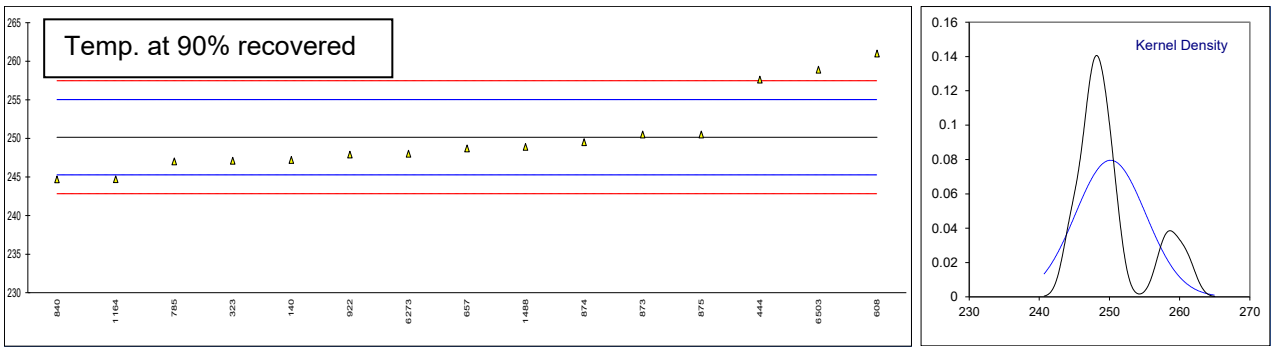
Lab 657 first reported respectively 33.2; 58.9; 69.2; 122.0; 232.8; 263.6; 303.9; 0.8; 0.4
Lab 1488 first reported 312.4

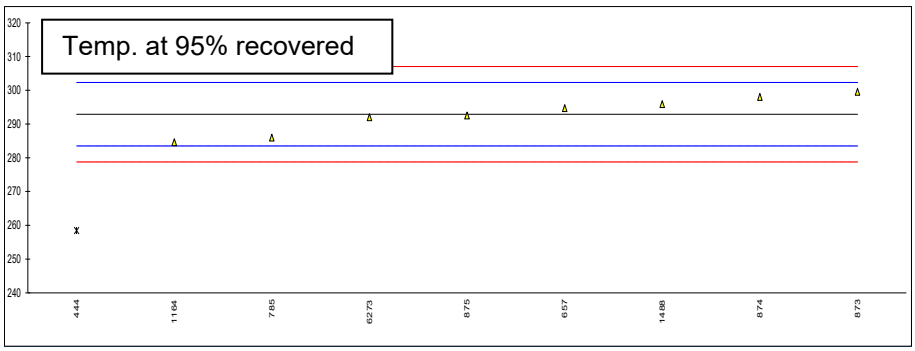

Initial Boiling Point
Kernel Density

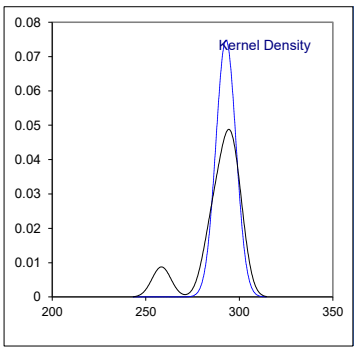

Temp. at 5% recovered
Kernel Density

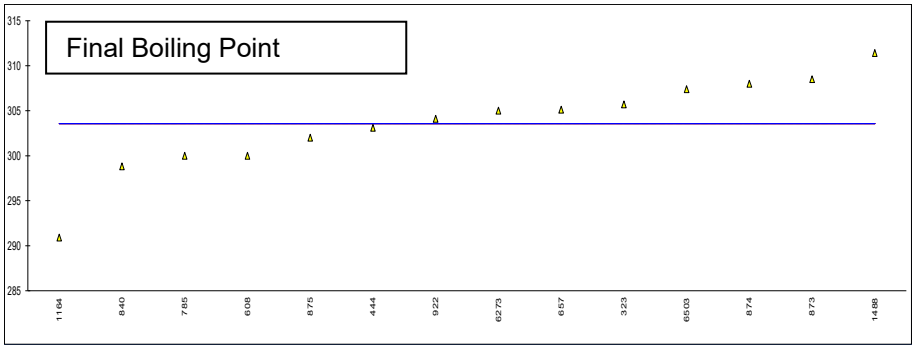

Temp. at 10% recovered
Kernel Density

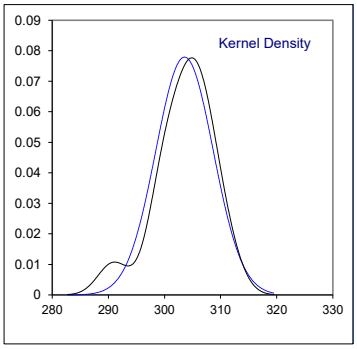

Temp. at 50% recovered
Kernel Density


Temp. at 90% recovered
Kernel Density


Temp. at 95% recovered
320
310
300
290
280
270
260
250
240
444
1164
785
6273
875
657
1488
874
873


Kernel Density
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
200
250
300
350


Final Boiling Point
315
310
305
300
295
290
285
1164
840
785
608
875
444
922
6273
657
323
6503
874
873
1488


Kernel Density
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
280
290
300
310
320
330

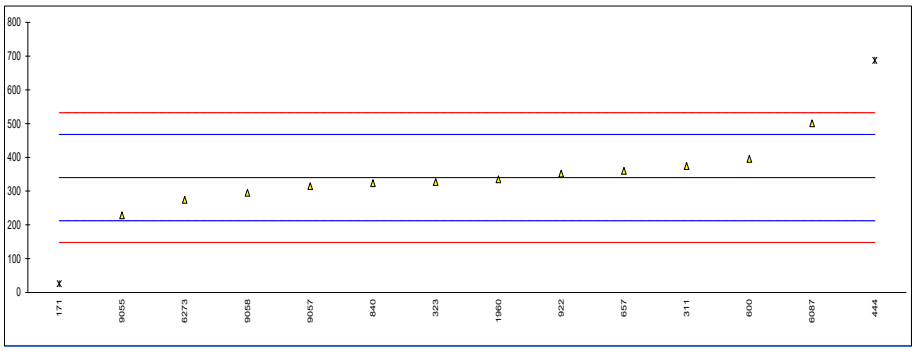
Determination of Methanol on sample #22220; results in mg/kg

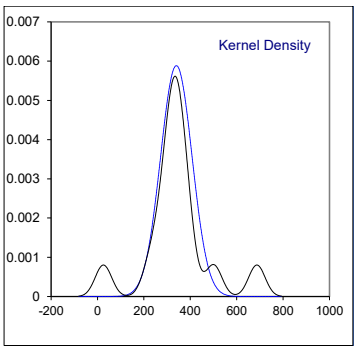
lab	method	value	mark	z(targ)	remarks
140		-----		-----	
171		-----		-----	
311		-----		-----	
323	INH-304	25		-----	
442		-----		-----	
444		-----		-----	
600		-----		-----	
608		-----		-----	
609		-----		-----	
657	INH-0130	29		-----	
785		-----		-----	
840		-----		-----	
873		-----		-----	
874		-----		-----	
875		-----		-----	
922		-----		-----	
1164		-----		-----	
1488		-----		-----	
1696		-----		-----	
1815		-----		-----	
1960		-----		-----	
6087		-----		-----	
6273		-----		-----	
6379		-----		-----	
6447		-----		-----	
6486		-----		-----	
6503		-----		-----	
9055		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9107		-----		-----	
9130		-----		-----	

Determination of Total Mercury on sample #22220; results in µg/kg

lab	method	value	mark	z(targ)	remarks
140		-----		-----	
171	UOP938	25.7	DG(0.05)	-4.91	
311	UOP938	374		0.53	
323	UOP938	327		-0.20	
442		-----		-----	
444	UOP938	687	DG(0.05)	5.42	
600	D7622	395.32		0.87	
608		-----		-----	
609		-----		-----	
657	UOP938	360		0.31	
785		-----		-----	
840	UOP938	323.61		-0.26	
873		-----		-----	
874		-----		-----	
875		-----		-----	
922	UOP938	352		0.19	
1164		-----		-----	
1488		-----		-----	
1696		-----		-----	
1815		-----		-----	
1960	UOP938	335		-0.08	
6087	UOP938	500.7384		2.51	
6273	UOP938	273.9		-1.03	
6379		-----		-----	
6447		-----		-----	
6486		-----		-----	
6503		-----		-----	
9055	UOP938	228.4		-1.74	
9056		-----		-----	
9057	UOP938	314.80		-0.39	
9058	UOP938	294.85		-0.71	
9061		-----		-----	
9107		-----		-----	
9130		-----		-----	

	normality	not OK
	n	12
	outliers	2
	mean (n)	339.968
	st.dev. (n)	67.7945
	R(calc.)	189.825
	st.dev.(Horwitz)	63.9858
	R(Horwitz)	179.160
Compare		
	R(UOP938:20)	31.925

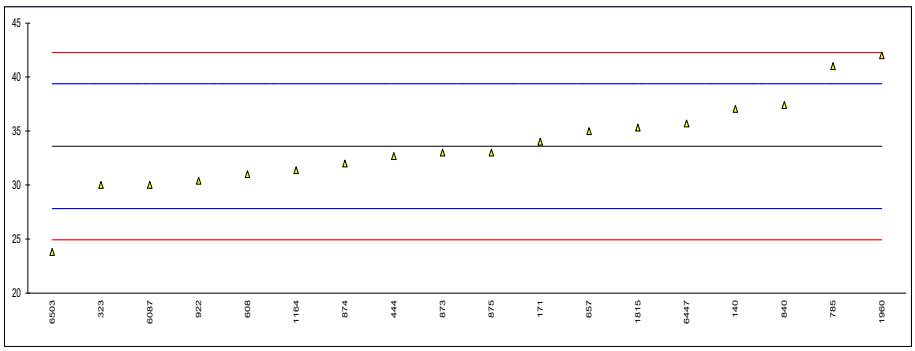


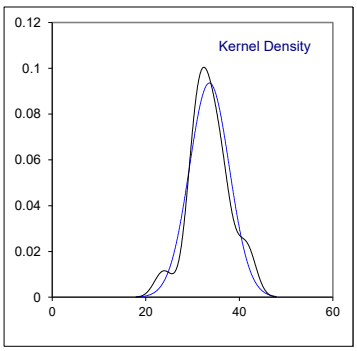


Determination of Total Sulfur on sample #22220; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140	D5453	37.04	C	1.19	first reported 43.19
171	D5453	34		0.14	
311		-----		-----	
323	D5453	30		-1.24	
442		-----		-----	
444	D5453	32.68		-0.32	
600		-----		-----	
608	D5453	31		-0.90	
609		-----		-----	
657	D5453	34.993		0.48	
785	D4294	41		2.56	
840	D5453	37.4		1.32	
873	ISO20846	33		-0.21	
874	ISO20846	32		-0.55	
875	ISO20846	33.0		-0.21	
922	D5453	30.4		-1.11	
1164	D5453	31.37		-0.77	
1488		-----		-----	
1696		-----		-----	
1815	D5453	35.3		0.59	
1960	D5453	42		2.91	
6087	D5453	30.0		-1.24	
6273		-----		-----	
6379		-----		-----	
6447	D5453	35.70		0.73	
6486		-----		-----	
6503	D5453	23.81		-3.39	
9055		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9107		-----		-----	
9130		-----		-----	

normality	OK
n	18
outliers	0
mean (n)	33.594
st.dev. (n)	4.2624
R(calc.)	11.935
st.dev.(D5453:19a)	2.8890
R(D5453:19a)	8.089

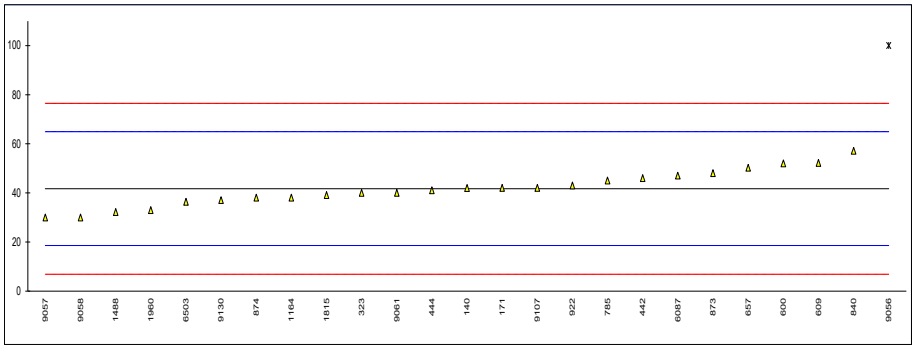


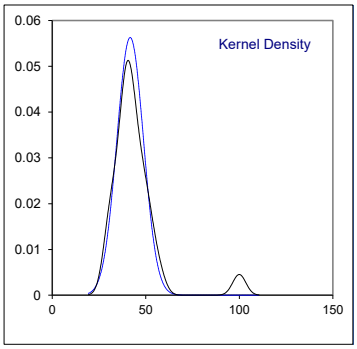


Determination of Water on sample #22220; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140	D6304-A:16e1	41.93		0.02	
171	D6304-A:20	42		0.02	
311		-----		-----	
323	D6304-A:16e1	40		-0.15	
442	IP438	46		0.37	
444	D4928	41		-0.06	
600	D6304-A:20	52		0.89	
608		-----		-----	
609	D4928	52.2		0.90	
657	D6304-A:20	50.2		0.73	
785	ISO12937	45		0.28	
840	D6304-A:20	57.1		1.33	
873	D6304-A:20	48		0.54	
874	D6304	38		-0.32	
875		-----		-----	
922	D6304-A:20	43		0.11	
1164	D6304-A:20	38		-0.32	
1488	ISO12937	32.2		-0.82	
1696		-----		-----	
1815	ISO12937	39.10		-0.23	
1960	D4928	33		-0.75	
6087	D4928	47.0		0.46	
6273		-----		-----	
6379		-----		-----	
6447		-----		-----	
6486		-----		-----	
6503	D6304-A:20	36.36		-0.46	
9055		-----		-----	
9056	In house	100	R(0.01)	5.02	
9057		30		-1.01	
9058		30		-1.01	
9061	D4928	40		-0.15	
9107	D6304-A:20	42		0.02	
9130	D6304-A:20	37		-0.41	

normality	OK
n	24
outliers	1
mean (n)	41.712
st.dev. (n)	7.0882
R(calc.)	19.847
st.dev.(D6304-A:20)	11.5997
R(D6304-A:20)	32.479





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	-1.70	-0.30	-0.28	-0.42	-1.21	-----	-----
171	-----	-----	-----	-----	-----	-----	-----
311	-----	-----	-----	-----	-----	-----	-----
323	-0.31	-0.44	-0.28	-0.07	-1.25	-----	-----
442	-----	-----	-----	-----	-----	-----	-----
444	-0.54	0.22	0.39	0.80	3.06	-7.33	-----
600	-----	-----	-----	-----	-----	-----	-----
608	2.17	0.79	1.59	0.80	4.45	-----	-----
609	-----	-----	-----	-----	-----	-----	-----
657	0.43	1.76	2.79	1.55	-0.59	0.38	-----
785	0.62	-1.40	-0.66	-0.36	-1.29	-1.47	-----
840	-2.12	-1.53	-1.87	-1.38	-2.24	-----	-----
873	0.62	0.13	-0.28	-0.65	0.15	1.40	-----
874	0.62	0.35	0.09	-0.94	-0.26	1.08	-----
875	0.43	-0.09	-0.28	-0.65	0.15	-0.09	-----
922	-0.19	0.00	-0.21	0.10	-0.92	-----	-----
1164	0.00	0.48	0.62	0.45	-2.23	-1.76	-----
1488	0.20	-0.79	0.02	-0.42	-0.51	0.64	-----
1696	-----	-----	-----	-----	-----	-----	-----
1815	-----	-----	-----	-----	-----	-----	-----
1960	-----	-----	-----	-----	-----	-----	-----
6087	-----	-----	-----	-----	-----	-----	-----
6273	-0.15	0.35	0.09	0.22	-0.88	-0.19	-----
6379	-----	-----	-----	-----	-----	-----	-----
6447	-----	-----	-----	-----	-----	-----	-----
6486	-----	-----	-----	-----	-----	-----	-----
6503	-0.08	0.44	0.77	0.97	3.59	-----	-----
9055	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9057	-----	-----	-----	-----	-----	-----	-----
9058	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9130	-----	-----	-----	-----	-----	-----	-----

APPENDIX 3

Number of participants per country

3 labs in AUSTRALIA
1 lab in BELGIUM
1 lab in BULGARIA
1 lab in FINLAND
1 lab in INDONESIA
5 labs in MALAYSIA
4 labs in NETHERLANDS
2 labs in NORWAY
1 lab in PAKISTAN
1 lab in POLAND
4 labs in RUSSIAN FEDERATION
1 lab in SINGAPORE
2 labs in UNITED ARAB EMIRATES
4 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)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

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2 ISO5725:86
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6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)