Results of Proficiency Test
Gascondensate
November 2017

January 2018

CONTENTS

Appendices:

1   INTRODUCTION

Since 2008, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Gascondensate. During the annual proficiency testing program 2017/2018, it was decided to continue the round robin for the analysis of Gascondensate.
In this interlaboratory study 45 laboratories in 20 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2017 Gascondensate proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send 1 sample of Gascondensate (0.5L bottle, labelled #17220). 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 March 2017 (iis-protocol, version 3.4). 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 are for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary bulk material, approximately 35 liter, was obtained from a participating laboratory. This batch was spiked with Methanol (approx. 100 mg/kg). After homogenisation, 70 amber glass bottles of 0.5 litre were filled and labelled as sample #17220.
The homogeneity of the subsamples #17220 was checked by determination of Density at 15°C in accordance with ASTM D4052 and Methanol in accordance with an in house test method on 8 stratified randomly selected samples.

	Density at 15 °C in kg/m^3	Methanol in mg/kg
Sample #17220-1	741.95	120.1
Sample #17220-2	741.96	121.2
Sample #17220-3	742.06	116.2
Sample #17220-4	741.95	120.6
Sample #17220-5	742.09	115.8
Sample #17220-6	742.00	117.7
Sample #17220-7	742.41	115.0
Sample #17220-8	742.11	116.9

Table 1: homogeneity test results of subsamples #17220

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

	Density at 15 °C in kg/m^3	Methanol in mg/kg
r observed	0.43	6.6
reference (test method)	ASTM D4052:15	Horwitz
0.3xR(ref. test method)	0.65	7.7

Table 2: repeatabilities of subsamples #17220

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference test method and the estimated reproducibility calculated using the Horwitz equation. Therefore, homogeneity of the subsamples was assumed

To each of the participating laboratories, 1 * 0.5 L bottle (labelled #17220) was sent on October 18, 2017. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were requested to determine on sample #17220: Color Saybolt (Automated and Manual), Density at 15°C, Distillation (IBP, temperature at 5%, 10%, 50%, 90%, 95% recovered, FBP, distillation Residue and Loss), Methanol, Mercury, Sulphur, Water by KF and Simulated Distillation.

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

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 required reference test methods that will be used during 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 reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).
For the statistical evaluation, *unrounded* (when available) figures were used instead of rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are 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. The Kernel Density Graph 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. When no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency test 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 result tables in appendix 1.

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

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

4 EVALUATION

In this proficiency test, problems with sample dispatch were encountered due to several reasons. Nine participants reported the test results after the final reporting date and three other participants did not report any test results at all. Not all laboratories were able to report all analyses requested. In total, 42 participants reported 333 numerical test results. Observed were 19 outlying test results, which is 5.7%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1   EVALUATION PER TEST

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

In the iis PT reports, ASTM methods are referred to with a number (e.g. D3588) and an added designation for the year that the method was adopted or revised (e.g. D3588:98). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D3588:98(2017)). In the test results tables of appendix 1 only the method number and year of adoption or revision (e.g. D3588:98) will be used.

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

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.

Color Saybolt: Both the automated method (ASTM D6045) and the manual method (ASTM D156) were evaluated and were problematic. In total two statistical outliers were observed. Both calculated reproducibilities after rejection of the statistical outliers are not in agreement with the requirements of respective test methods ASTM D6045:12(2017) and ASTM D156:15. The limited number of test results and the rounding of the reported test results may (partly) explain the large variation.

Density at 15°C: This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D4052:16.
It should be taken into account that the reproducibility from ASTM D4052:16 is applicable to petroleum distillates and viscous oils only. Therefore, no precision data are stated in the 2016 version for Gascondensates. However, Gascondensates may contain relatively high concentrations of light ends and therefore should be treated as gasoline, i.e. cooling the sample prior to analysis to prevent loss of light ends.

Distillation: This determination may be problematic. In total five statistical outliers were observed. After rejection of the statistical outliers, the calculated reproducibilities of IBP, temperature at 5%, 10% and 50% recovered were in agreement with the requirements of the manual mode of ASTM D86:17. However, the temperatures at 90%, 95% recovered and Final Boiling Point were not in agreement with the requirements of the manual mode of ASTM D86:17. It should be noted that the scope of ASTM D86 does not include

Gas condensates, but only products with a limited boiling range like distillate fuels, so the target reproducibilities as used in this report may not be applicable. The use of a simulated distillation determination may be more appropriate.

Methanol: This determination may be problematic. Only four test results were reported. The calculated reproducibility is not in agreement with the estimated reproducibility calculated using the Horwitz equation.
The average recovery of Methanol (theoretical increment of 103.6 mg/kg) may be good: "less than 92%". The actual blank concentration for Methanol is unknown.

Mercury: The precision requirements of UOP938 (table 3b) are extremely strict and as they are approx. 6 times more strict than the Horwitz estimate, these requirements will not be met easily. Also, the reproducibility of UOP938 is only available for very low concentrations (0.28 and 12.14 µg/**L**, table B3) and conversion and extrapolation up to 320 µg/**kg** will lead to extra uncertainty. Therefore, it was decided to use the Horwitz estimate for evaluation of the test results in this report.
This determination was not problematic at a concentration of 320 µg Hg per kg. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the estimated reproducibility calculated using the Horwitz equation.

Sulphur: 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 D5453:16e1.

Water: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D6304:16e1.
It must be noted that the precision data of ASTM D4928 is not applicable at this low concentration (valid between 0.02 – 5.00%M/M).

Simulated Distillation: This determination may be problematic. In total two statistical outliers were observed. After rejection of the statistical outliers, the calculated reproducibilities of 10%, 95% recovered and Final Boiling Point were in agreement with the requirements of ASTM D2887:16a. However, the calculated reproducibility of the 50% and 90% recovered are not in agreement with the requirements of ASTM D2887:16a. 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 and the reproducibility as found for the group of participating laboratories. The average results of sample #17220, calculated reproducibilities and target reproducibilities, derived from literature reference test methods (in casu ASTM methods) are compared in the next table.

Parameter	unit	n	Mean	2.8 * sd	R (lit)
Color Saybolt (Automated)		8	18.3	2.0	1.2
Color Saybolt (Manual)		9	18.2	4.6	2
Density at 15°C	kg/m^3	37	0.7422	0.0010	0.0022
Distillation					
Initial Boiling Point	°C	19	33.9	7.0	7.3
5%-recovered	°C	18	58.1	5.9	6.4
10%-recovered	°C	19	68.5	3.7	3.7
50%-recovered	°C	19	123.1	4.7	4.8
90%-recovered	°C	17	248.9	13.0	6.8
95%-recovered	°C	11	292.4	23.1	13.4
Final Boiling Point	°C	17	305.4	12.4	4.2
Methanol	mg/kg	4	95.0	34.6	21.5
Mercury as Hg	µg/kg	16	322	126	171
Sulphur	mg/kg	26	37.2	12.4	8.7
Water content by KF	mg/kg	32	42.3	36.3	159.8
Simulated Distillation					
Initial Boiling Point	°C	5	<36	n.a.	n.a.
5%-recovered	°C	5	<36	n.a.	n.a.
10%-recovered	°C	4	36.1	0.3	2.0
50%-recovered	°C	5	117.2	7.8	4.3
90%-recovered	°C	5	248.3	10.2	4.3
95%-recovered	°C	4	285.8	1.1	5
Final Boiling Point	°C	5	383.3	6.4	11.8

Table 3: reproducibilities of tests on sample #17220

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 2017 WITH THE PREVIOUS PTS

	November 2017	November 2016	November 2015	November 2014	November 2013
Number of reporting participants	42	42	38	36	36
Number of results reported	333	297	248	251	216
Number of statistical outliers	19	23	8	8	15
Percentage of statistical outliers	5.7%	7.7%	3.2%	3.2%	6.9%

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 2017	November 2016	November 2015	November 2014	November 2013
Color Saybolt	--	--	--	--	-
Density at 15°C	++	++	+	+	++
Distillation (ASTM D86)	-	-	-	--	--
Methanol	-	--	n.e.	n.e.	n.e.
Mercury as Hg	+	-	-	-	--
Sulphur	-	+	-	--	--
Water content by KF	++	++	++	++	++
SimDist	+/-	--	n.e.	n.e.	n.e.

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

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

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

APPENDIX 1

Determination of Color Saybolt (automated and manual) on sample #17220;

lab	method	automated	mark	z(targ)	method	manual	mark	z(targ)	Remarks
140		-----		-----		-----		-----	
158	D6045	26.0	G(0.01)	17.50		-----		-----	
171		-----		-----	D156	16		-3.11	
311		-----		-----		-----		-----	
323	D6045	20		3.95		-----		-----	
442		-----		-----		-----		-----	
444	D6045	18.0		-0.56		-----		-----	
608		-----		-----	D156	18		-0.31	
609		-----		-----		-----		-----	
657		-----		-----	D156	20		2.49	
785	D6045	18		-0.56		-----		-----	
840		-----		-----		-----		-----	
873	D6045	18		-0.56		-----		-----	
874		-----		-----		-----		-----	
875	D6045	18		-0.56		-----		-----	
998		-----		-----	D156	21		3.89	
1164	D6045	18		-0.56		-----		-----	
1214		-----		-----		-----		-----	
1257		-----		-----	D156	18		-0.31	
1267		-----		-----		-----		-----	
1397		-----		-----		-----		-----	
1429	D6045	18		-0.56		-----		-----	
1455		-----		-----		-----		-----	
1696		-----		-----		-----		-----	
1714		-----		-----	D156	18		-0.31	
1815		-----		-----		-----		-----	
1957		-----		-----		-----		-----	
1960	D6045	18		-0.56		-----		-----	
1995		-----		-----		-----		-----	
2124		-----		-----	D156	19		1.09	
6011		-----		-----	D156	30	G(0.01)	16.49	
6016		-----		-----		-----		-----	
6087		-----		-----		-----		-----	
6159		-----		-----		-----		-----	
6160		-----		-----		-----		-----	
6161		-----		-----	D156	16		-3.11	
9054		-----		-----		-----		-----	
9056		-----		-----		-----		-----	
9057		-----		-----		-----		-----	
9058		-----		-----		-----		-----	
9061		-----		-----		-----		-----	
9107		-----		-----	D156	18		-0.31	
9130		-----		-----		-----		-----	
9131		-----		-----		-----		-----	
9150		-----		-----		-----		-----	

	normality	not OK		normality	OK
	n	8		n	9
	outliers	1		outliers	1
	mean (n)	18.25		mean (n)	18.22
	st.dev. (n)	0.707		st.dev. (n)	1.641
	R(calc.)	1.98		R(calc.)	4.60
	st.dev.(D6045:12)	0.443		st.dev.(D156:15)	0.714
	R(D6045:12)	1.24		R(D156:15)	2
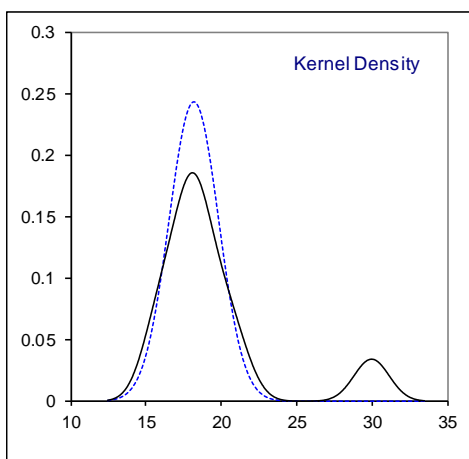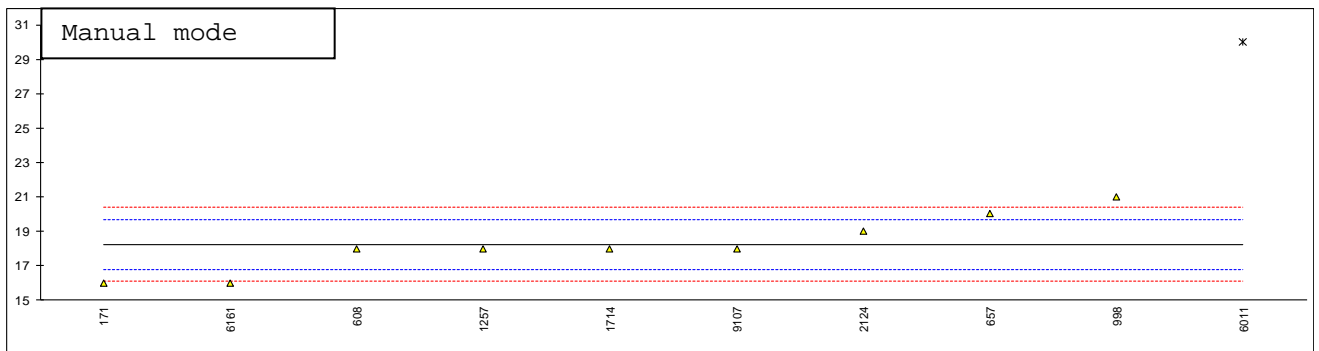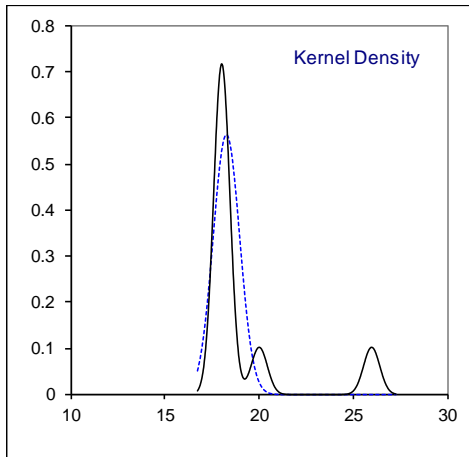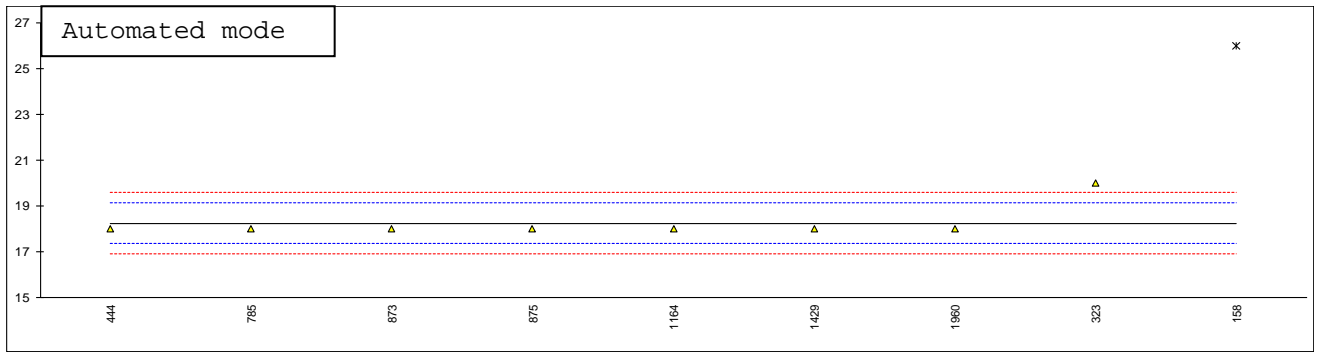
Determination of Density at 15°C on sample #17220; results in kg/L

lab	method	value	mark	z(targ)	remarks
140		-----		-----	
158	D4052	0.7421		-0.18	
171	D4052	0.7419		-0.44	
311	D4052	0.7420		-0.31	
323	D4052	0.7424	C	0.20	First reported 0.7424 kg/m3
442	IP365	0.7424		0.20	
444	D4052	0.7427		0.58	
608	D4052	0.7422		-0.05	
609	D4052	0.7421		-0.18	
657	D4052	0.7420		-0.31	
785	D4052	0.7420		-0.31	
840	D4052	0.74210		-0.18	
873	D4052	0.7421		-0.18	
874	D4052	0.7421		-0.18	
875	D4052	0.7424		0.20	
998	D4052	0.7479	R(0.01)	7.22	
1164	D4052	0.74203	C	-0.27	First reported 0.74203 kg/m3
1214	D4052	0.74216		-0.10	
1257	D4052	0.7424		0.20	
1267	IP365	0.742		-0.31	
1397	D4052	0.7433		1.35	
1429	D4052	0.7422		-0.05	
1455	D4052	0.7421		-0.18	
1696		-----		-----	
1714	D4052	0.74265		0.52	
1815	ISO12185	0.74240		0.20	
1957	D4052	0.7432		1.22	
1960	D4052	0.742428		0.24	
1995		-----		-----	
2124	D4052	0.7423		0.07	
6011	D4052	0.7420		-0.31	
6016	D4052	0.7425		0.33	
6087	D4052	0.744252	R(0.01)	2.56	
6159	D4052	0.74191		-0.42	
6160		-----		-----	
6161	D4052	0.74166		-0.74	
9054	D4052	0.7421		-0.18	
9056		0.738	R(0.01)	-5.41	
9057		0.74249		0.32	
9058		0.7417		-0.69	
9061	ISO12185	0.74204		-0.26	
9107	D4052	0.7422		-0.05	
9130	D4052	0.7424		0.20	
9131	D4052	0.7423		0.07	
9150	D4052	0.740	R(0.01)	-2.86	

|	
normality	not OK
n	37
outliers	4
mean (n)	0.74224
st.dev. (n)	0.000338
R(calc.)	0.00095
st.dev.(D4052:16)	0.000784
R(D4052:16)	0.00219
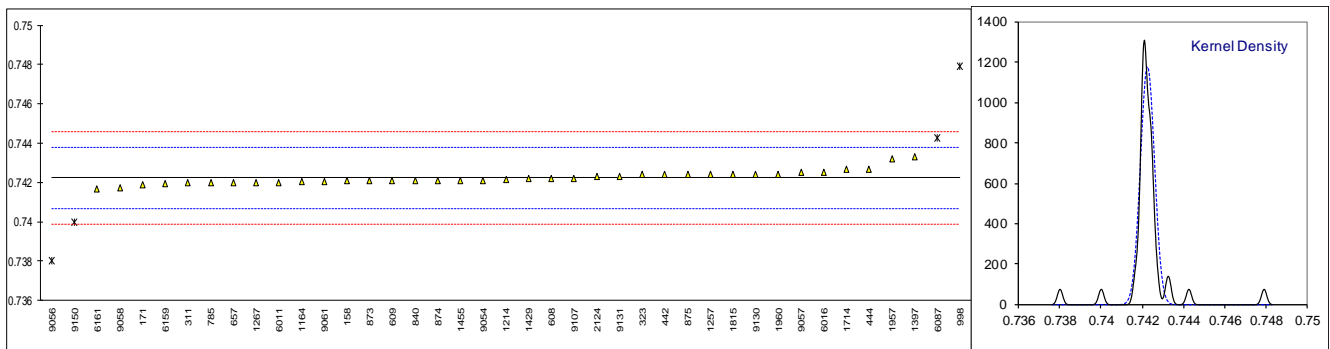
Determination of Distillation on sample #17220; results in °C

lab	method	IBP	5% rec	10% rec	50% rec	90% rec	95% rec	FBP	residue	loss
140		-----	-----	-----	-----	-----	-----	-----	-----	-----
158	D86-automated	30.2	56.4	67.3	121.5	246.3	287.9	**292.3**	1.5	3.0
171	D86-automated	34.5	57.5	69.3	122.2	238.9	275.6	304.6	1.2	1.0
311		-----	-----	-----	-----	-----	-----	-----	-----	-----
323	D86-automated	35.5	57.8	68.8	123.1	247.3	296.0	308.2	1.5	2.4
442		-----	-----	-----	-----	-----	-----	-----	-----	-----
444		-----	-----	-----	-----	-----	-----	-----	-----	-----
608	D86-automated	35.6	**40.5** C	69.5 C	127.5 C	**270.5** C	-----	310.0	-----	-----
609		-----	-----	-----	-----	-----	-----	-----	-----	-----
657	D86-automated	34.1	59.9	70.3	122.3	242.1	280.2	306.3	1.5	1.5
785	D86-manual	32.4	55.4	66.9	123.0	250.1	300.5	308.2	1.5	2.0
840	D86-automated	31.28	57.37	68.46	122.99	248.54	-----	305.59	1.3	3.6
873	D86-manual	33.0	56.0	67.0	122.0	251.5	295.0	313.0	1.5	2.0
874	D86-manual	33.0	57.0	67.0	122.0	253.0	296.0	313.0	2	1.5
875	D86-manual	34.0	58.0	67.0	123.0	252.0	300.0	309.0	1.4	1.6
998	D86-manual	39.6	63.6	70.6	120.7	245.9	294.0	300.0	2.0	2.0
1164		33.7	59.0	69.4	124.1	248.2	291.0	296.9	3.0	1.1
1214		-----	-----	-----	-----	-----	-----	-----	-----	-----
1257		34.2	57.5	68.6	125	256	-----	305	-----	-----
1267		-----	-----	-----	-----	-----	-----	-----	-----	-----
1397		-----	-----	-----	-----	-----	-----	-----	-----	-----
1429	D86-automated	30.6	55.6	67.3	122.6	248.9	-----	302.0	1.5	3.7
1455	D86-automated	29.4	55.6	66.7	120.5	242.9	300.7	301.4	1.3	3.1
1696		-----	-----	-----	-----	-----	-----	-----	-----	-----
1714	D86-automated	35.1	58.9	70.3	125.4	**267.0**	-----	-----	-----	-----
1815		-----	-----	-----	-----	-----	-----	-----	-----	-----
1957		-----	-----	-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----	-----	-----
1995		-----	-----	-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----	-----	-----
6011		-----	-----	-----	-----	-----	-----	-----	-----	-----
6016		-----	-----	-----	-----	-----	-----	-----	-----	-----
6087		-----	-----	-----	-----	-----	-----	-----	-----	-----
6159	D86-automated	35.4	58.6	68.7	122.7	253.6	**270.3**	304.2	1.9	1.3
6160		36.5	60.1	69.1	124.0	253.5	-----	302.1	1.82	1.24
6161	D86-automated	36.9	60.9	70.0	124.3	252.6	-----	303.0	2.0	1.0
9054		-----	-----	-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----	-----	-----
9057		-----	-----	-----	-----	-----	-----	-----	-----	-----
9058		-----	-----	-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----	-----	-----
9130		-----	-----	-----	-----	-----	-----	-----	-----	-----
9131		-----	-----	-----	-----	-----	-----	-----	-----	-----
9150		-----	-----	-----	-----	-----	-----	-----	-----	-----
	normality	OK	suspect	OK	suspect	OK	OK	OK		
	n	19	18	19	19	17	11	17		
	outliers	0	1	0	0	2	1	1		
	mean (n)	33.946	58.065	68.540	123.099	248.902	292.445	305.441		
	st.dev. (n)	2.5106	2.1235	1.3227	1.6816	4.6311	8.2509	4.4300		
	R(calc.)	7.030	5.946	3.704	4.709	12.967	23.102	12.404		
	st.dev.(D86-M:17)	2.6022	2.2861	1.3293	1.7138	2.4304	4.7795	1.4970		
	R(D86-M:17)	7.286	6.401	3.722	4.799	6.805	13.382	4.192		

NB Results in Bold and Underlined are statistical outliers or are excluded for statistical evaluation.

Lab 608: first reported 65.8, 75.4, 130.8, 269

5% recovered

10% recovered

50% recovered

90% recovered

95% recovered

Final Boiling Point
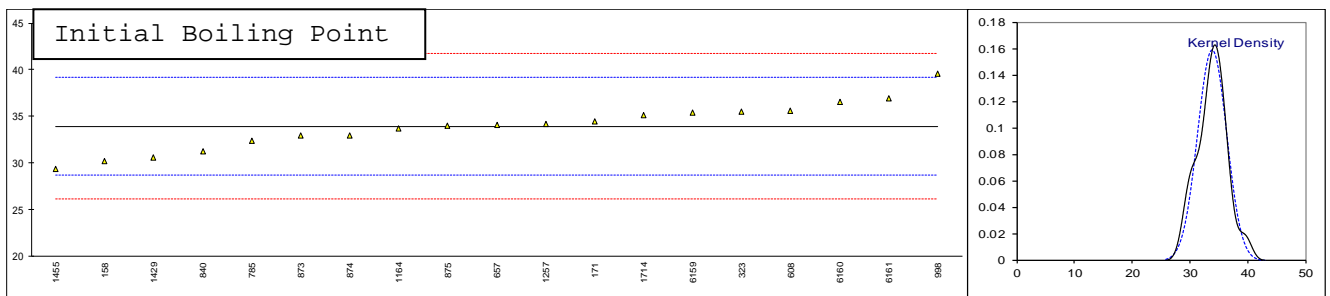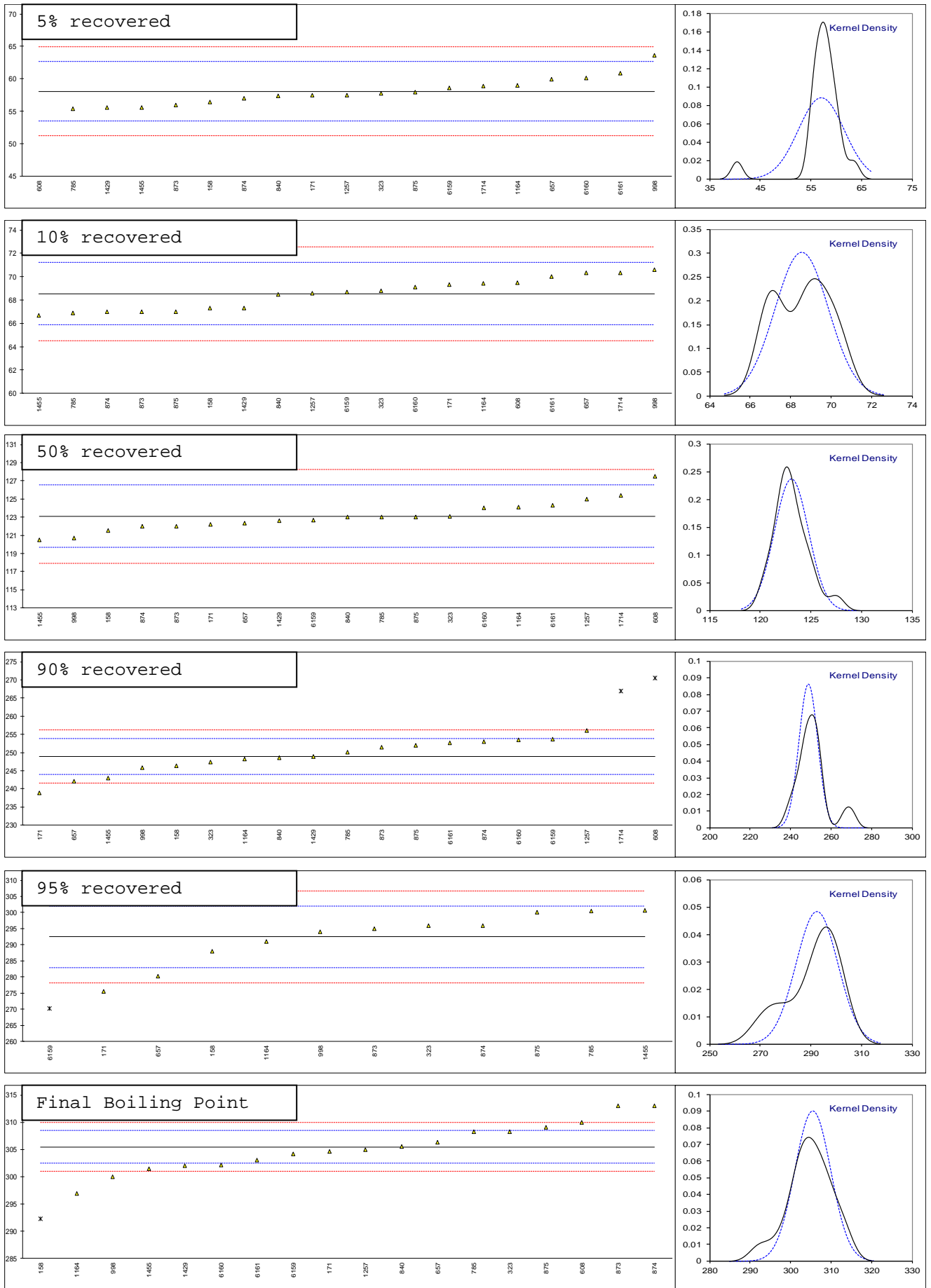
Determination of Methanol on sample #17220; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140		-----		-----	
158		-----		-----	
171		-----		-----	
311		-----		-----	
323	INH-304	80		-1.96	
442		-----		-----	
444	INH-008	97		0.26	
608		-----		-----	
609		-----		-----	
657	INH-0130	93.1		-0.25	
785		-----		-----	
840		-----		-----	
873		-----		-----	
874		-----		-----	
875		-----		-----	
998		-----		-----	
1164		-----		-----	
1214		-----		-----	
1257		-----		-----	
1267		-----		-----	
1397		-----		-----	
1429		-----		-----	
1455		110		1.95	
1696		-----		-----	
1714		-----		-----	
1815		-----		-----	
1957		-----		-----	
1960		-----		-----	
1995		-----		-----	
2124		-----		-----	
6011		-----		-----	
6016		-----		-----	
6087		-----		-----	
6159		-----		-----	
6160		-----		-----	
6161		-----		-----	
9054		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9107		-----		-----	
9130		-----		-----	
9131		-----		-----	
9150		-----		-----	

normality	unknown			
n	4			
outliers	0	Spike		
mean (n)	95.025	103.6	Recovery <92%	
st.dev. (n)	12.3505			
R(calc.)	34.582			
st.dev.(Horwitz)	7.6606			
R(Horwitz)	21.450			
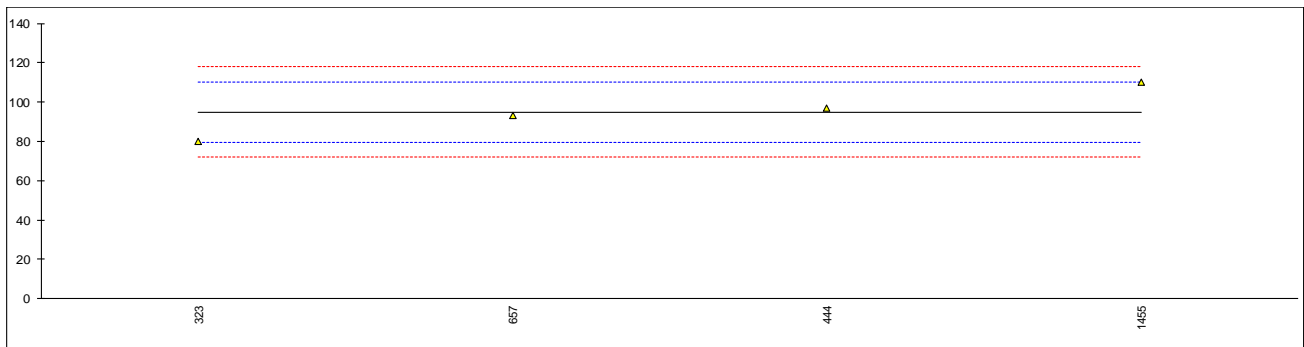
Determination of Mercury as Hg, total on sample #17220; results in µg/kg

lab	method	value	mark	z(targ)	remarks
140		-----		-----	
158		-----		-----	
171	UOP938	319		-0.05	
311	INH-001	340		0.30	
323	UOP938	350		0.46	
442		-----		-----	
444	UOP938	160.9	G(0.05)	-2.64	
608		-----		-----	
609		-----		-----	
657	UOP938	327.5385		0.09	
785		-----		-----	
840	EPA7470A	373.4		0.84	
873	UOP938	332.20		0.17	
874		-----		-----	
875		-----		-----	
998		-----		-----	
1164	UOP938	294		-0.46	
1214		-----		-----	
1257		-----		-----	
1267		-----		-----	
1397	In house	258		-1.05	
1429	In house	375		0.87	
1455		-----		-----	
1696		-----		-----	
1714	UOP938	348		0.43	
1815		-----		-----	
1957		-----		-----	
1960	UOP938	338.45		0.27	
1995		-----		-----	
2124		-----		-----	
6011	UOP938	300.81		-0.35	
6016		-----		-----	
6087	UOP938	371.290		0.81	
6159		-----		-----	
6160		-----		-----	
6161		-----		-----	
9054	UOP938	229.5463		-1.51	
9056		-----		-----	
9057		347.7		0.42	
9058		-----		-----	
9061		-----		-----	
9107	UOP938	245.4		-1.25	
9130		-----		-----	
9131		-----		-----	
9150		-----		-----	

normality	OK
n	16
outliers	1
mean (n)	321.896
st.dev. (n)	45.0802
R(calc.)	126.225
st.dev.(Horwitz)	61.0846
R(Horwitz)	171.037

Compare R(UOP938) = 30.227
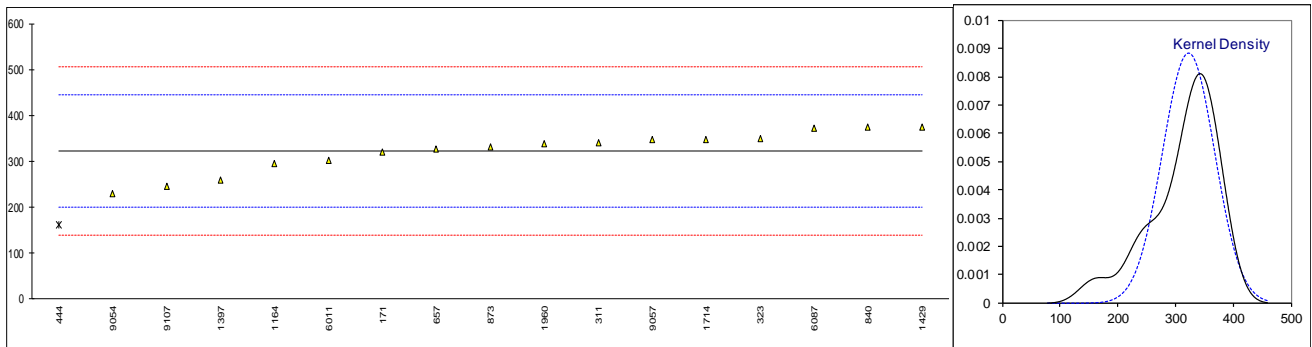
Determination of Sulphur on sample #17220; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140		-----		-----	
158	D2622	41.33		1.32	
171	D5453	37		-0.07	
311	D5453	34		-1.03	
323	D5453	39		0.57	
442		-----		-----	
444	D5453	39.3		0.67	
608	D5453	36.87		-0.11	
609		-----		-----	
657	D5453	34		-1.03	
785	ISO20884	41.9		1.50	
840	D5453	29.2		-2.57	
873	ISO20846	38.90		0.54	
874	ISO20846	39.4		0.70	
875	ISO20846	43.2		1.92	
998	D4294	33		-1.35	
1164	D5453	30.2		-2.25	
1214	D5453	30.92		-2.02	
1257	D3120	42		1.53	
1267		-----		-----	
1397		-----		-----	
1429	D5453	39.5		0.73	
1455	D2622	39.5		0.73	
1696		-----		-----	
1714	D5453	35.70		-0.49	
1815	D5453	33.41		-1.22	
1957		-----		-----	
1960	D5453	41.38		1.33	
1995		-----		-----	
2124	D5453	35.39		-0.59	
6011	D5453	48.06		3.48	
6016		-----		-----	
6087	D5453	34.962		-0.72	
6159	D4294	74	R(0.01)	11.79	
6160		-----		-----	
6161	D4294	82	R(0.01)	14.35	
9054		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9107		-----		-----	
9130	D5453	36.247		-0.31	
9131		-----		-----	
9150	D5453	33.3		-1.26	

	normality	OK	
	n	26	
	outliers	2	
	mean (n)	37.218	
	st.dev. (n)	4.4329	
	R(calc.)	12.412	
	st.dev.(D5453:16e1)	3.1197	
	R(D5453:16e1)	8.735	
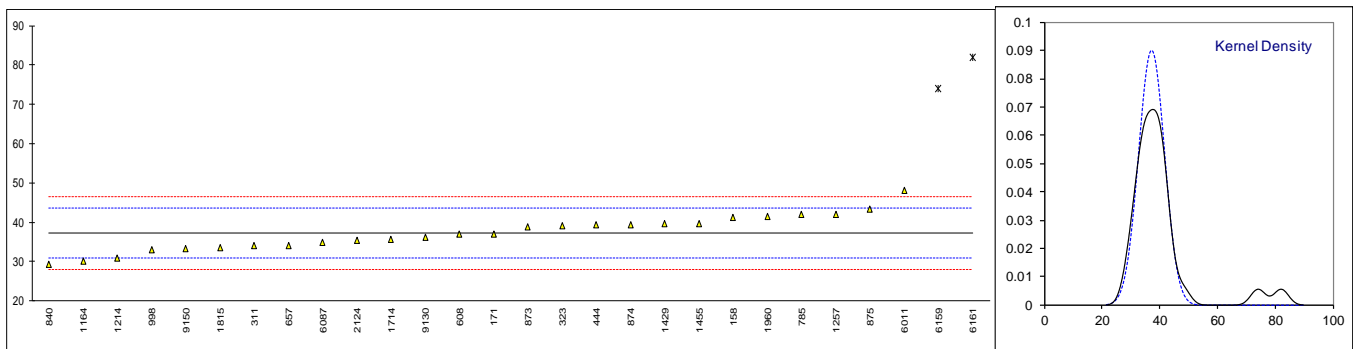
Determination of Water content by KF on sample #17220; results in mg/kg

lab	method	value	mark	z(targ)	remarks
140		-----		-----	
158		-----		-----	
171	D6304-A	40		-0.04	
311		-----		-----	
323	D1744	42		-0.01	
442	IP438	40		-0.04	
444	D6304-A	34.5		-0.14	
608	D4928	14.4	R(0.01)	-0.49	
609	D4928	39.3		-0.05	
657	D6304-A	45		0.05	
785		-----		-----	
840	D6304-A	45.5		0.06	
873	D6304-A	38.0		-0.08	
874	D6304-A	38		-0.08	
875	D6304-A	37		-0.09	
998	D6304	87.15		0.79	
1164	D6304-A	33.7		-0.15	
1214		-----		-----	
1257	D6304-A	38		-0.08	
1267	D4928	29.01		-0.23	
1397	ISO12937	38		-0.08	
1429	IP438	45.9		0.06	
1455	D4928	40		-0.04	
1696		-----		-----	
1714	D6304-C	25		-0.30	
1815	ISO12937	28.88		-0.24	
1957	D6304-A	70		0.49	
1960	D4928	44		0.03	
1995		-----		-----	
2124	D4928	38.7		-0.06	
6011	D6304-A	61		0.33	
6016		32.8		-0.17	
6087	D6304-A	34.5		-0.14	
6159	D4928	41		-0.02	
6160		-----		-----	
6161		-----		-----	
9054		-----		-----	
9056		100	R(0.01)	1.01	
9057		37.9		-0.08	
9058		540	R(0.01)	8.72	
9061	D4928	40		-0.04	
9107	D6304-A	72		0.52	
9130	D6304-A	41.25		-0.02	
9131	D6304-A	39.10		-0.06	
9150	D6304-A	36.4		-0.10	

	normality	not OK	
	n	32	
	outliers	3	
	mean (n)	42.300	
	st.dev. (n)	12.9495	
	R(calc.)	36.258	
	st.dev.(D6304:16e1 (mass inj))	57.0581	
	R(D6304:16e1 (mass inj))	159.763	
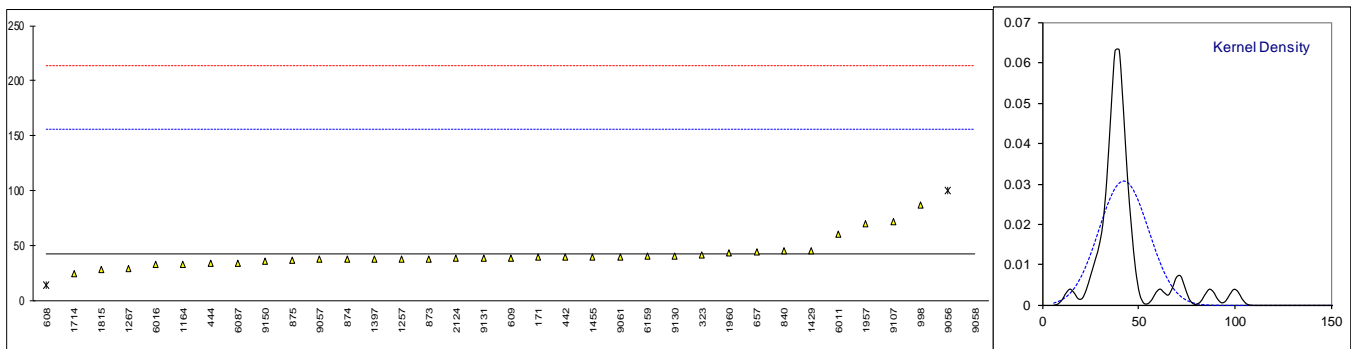
Determination of Simulated Distillation on sample #17220; results in °C

lab	method	IBP	5%rec	10%rec	50%rec	90%rec	95%rec	FBP
140		-----	-----	-----	-----	-----	-----	-----
158		-----	-----	-----	-----	-----	-----	-----
171	D2887	18.0	29.5	36.0	113.5	245.5	285.5	382.5
311	D2887	<36	<36	**41.0**	115.0	245.0	285.5	380.0
323		-----	-----	-----	-----	-----	-----	-----
442		-----	-----	-----	-----	-----	-----	-----
444		-----	-----	-----	-----	-----	-----	-----
608		-16.04	26.65	36.20	119.58	250.40	286.27	383.64
609		-----	-----	-----	-----	-----	-----	-----
657		-----	-----	-----	-----	-----	-----	-----
785		-----	-----	-----	-----	-----	-----	-----
840		-----	-----	-----	-----	-----	-----	-----
873		-----	-----	-----	-----	-----	-----	-----
874		-----	-----	-----	-----	-----	-----	-----
875		-----	-----	-----	-----	-----	-----	-----
998		-----	-----	-----	-----	-----	-----	-----
1164		-----	-----	-----	-----	-----	-----	-----
1214		-----	-----	-----	-----	-----	-----	-----
1257		-----	-----	-----	-----	-----	-----	-----
1267		-----	-----	-----	-----	-----	-----	-----
1397	D2887	11.00	22.00	36.00	119.00	247.00	286.00	384.00
1429		-----	-----	-----	-----	-----	-----	-----
1455		-----	-----	-----	-----	-----	-----	-----
1696		-----	-----	-----	-----	-----	-----	-----
1714	D2887	<-0.5	20.9	36.2	119.1	253.6	**296.2**	386.3
1815		-----	-----	-----	-----	-----	-----	-----
1957		-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----
1995		-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----
6011		-----	-----	-----	-----	-----	-----	-----
6016		-----	-----	-----	-----	-----	-----	-----
6087		-----	-----	-----	-----	-----	-----	-----
6159		-----	-----	-----	-----	-----	-----	-----
6160		-----	-----	-----	-----	-----	-----	-----
6161		-----	-----	-----	-----	-----	-----	-----
9054		-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----
9057		-----	-----	-----	-----	-----	-----	-----
9058		-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----
9130		-----	-----	-----	-----	-----	-----	-----
9131		-----	-----	-----	-----	-----	-----	-----
9150		-----	-----	-----	-----	-----	-----	-----
	normality	unknown	unknown	unknown	unknown	unknown	unknown	unknown
	n	5	5	4	5	5	4	5
	outliers	<36	<36	1	0	0	1	0
	mean (n)	unknown	unknown	36.100	117.236	248.300	285.818	383.288
	st.dev. (n)	n.a.	n.a.	0.1155	2.7856	3.6373	0.3828	2.2986
	R(calc.)	n.a.	n.a.	0.323	7.800	10.184	1.072	6.436
	st.dev.(D2887:16a)	n.a.	n.a.	0.7291	1.5357	1.5357	1.7857	4.2143
	R(D2887:16a)	n.a.	n.a.	2.041	4.3	4.3	5	11.8

NB Results in Bold and Underlined are statistical outliers or are excluded for statistical evaluation.
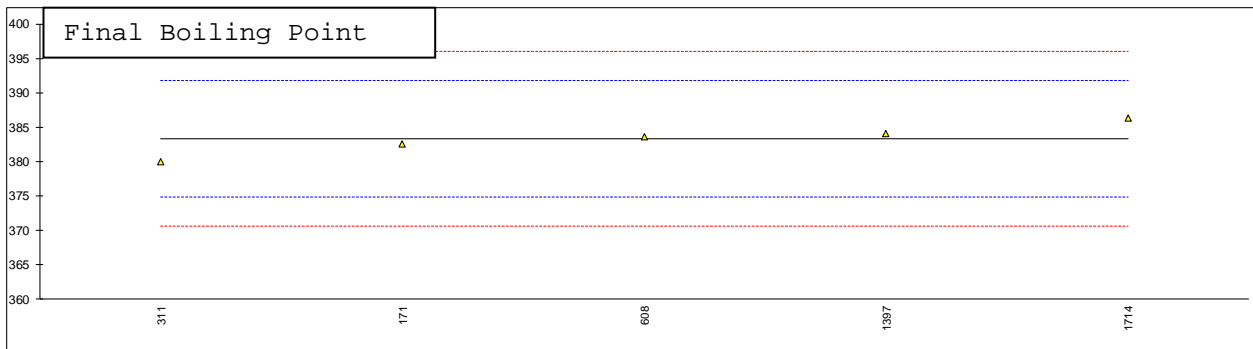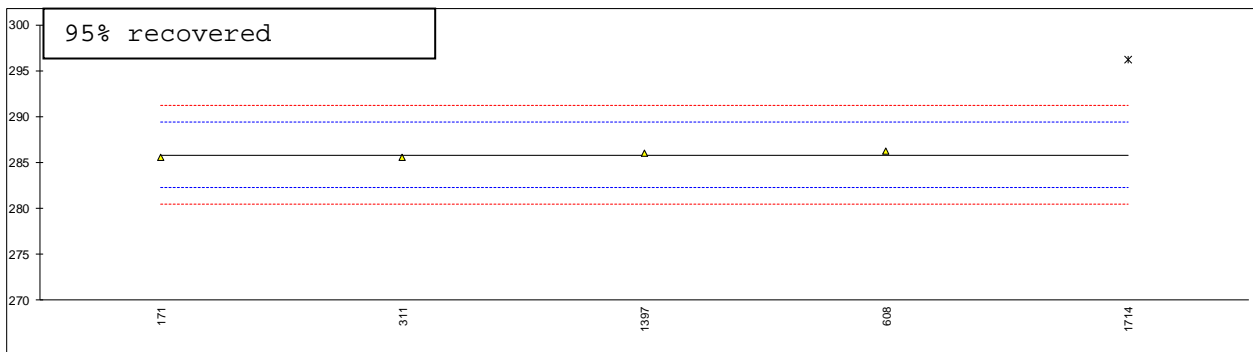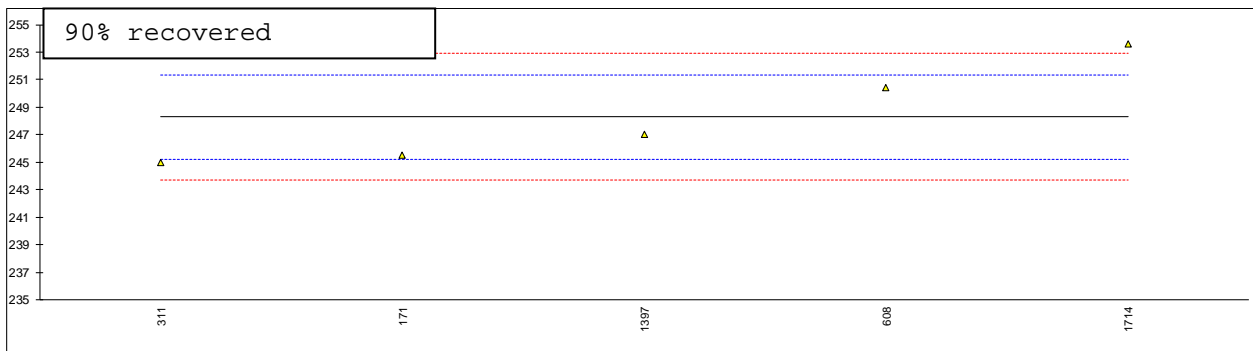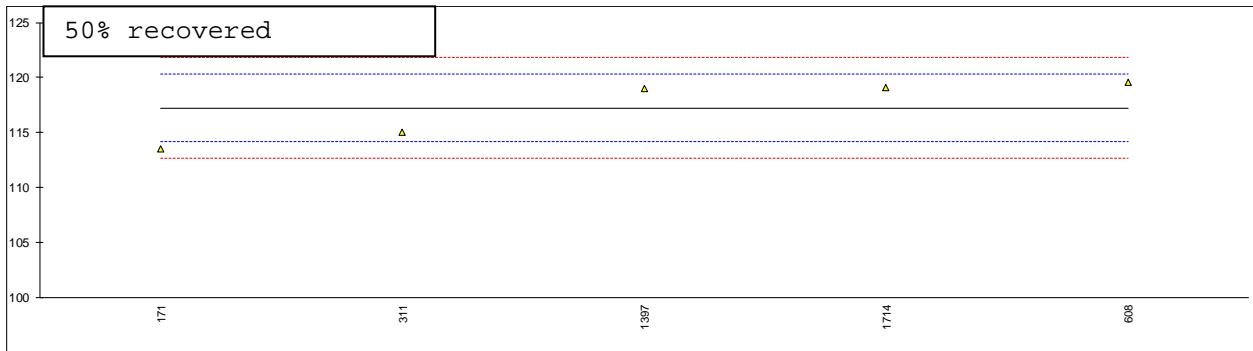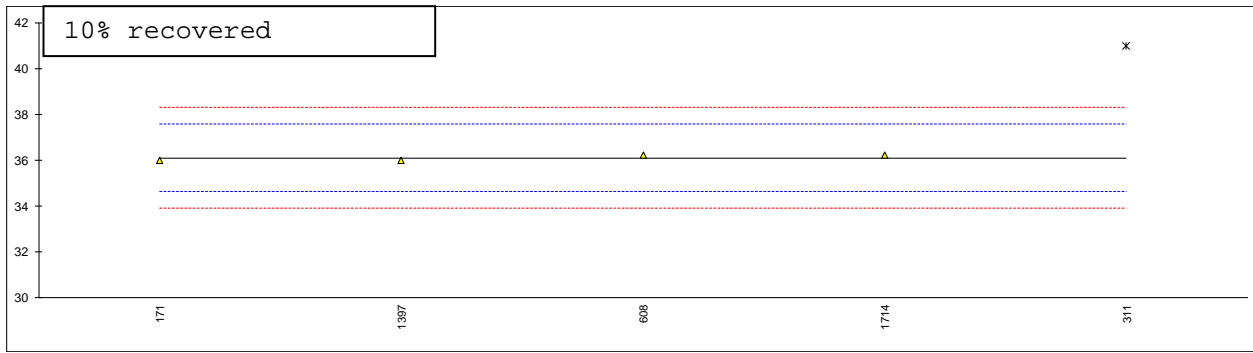
APPENDIX 2:

Atmospheric Distillation z-scores

lab	IBP	5%	10%	50%	90%	95%	FBP
140	-----	-----	-----	-----	-----	-----	-----
158	-1.44	-0.73	-0.93	-0.93	-1.07	-0.95	-8.78
171	0.21	-0.25	0.57	-0.52	-4.12	-3.52	-0.56
311	-----	-----	-----	-----	-----	-----	-----
323	0.60	-0.12	0.20	0.00	-0.66	0.74	1.84
442	-----	-----	-----	-----	-----	-----	-----
444	-----	-----	-----	-----	-----	-----	-----
608	0.64	-7.68	0.72	2.57	8.89	-----	3.05
609	-----	-----	-----	-----	-----	-----	-----
657	0.06	0.80	1.32	-0.47	-2.80	-2.56	0.57
785	-0.59	-1.17	-1.23	-0.06	0.49	1.69	1.84
840	-1.02	-0.30	-0.06	-0.06	-0.15	-----	0.10
873	-0.36	-0.90	-1.16	-0.64	1.07	0.53	5.05
874	-0.36	-0.47	-1.16	-0.64	1.69	0.74	5.05
875	0.02	-0.03	-1.16	-0.06	1.27	1.58	2.38
998	2.17	2.42	1.55	-1.40	-1.24	0.33	-3.63
1164	-0.09	0.41	0.65	0.58	-0.29	-0.30	-5.71
1214	-----	-----	-----	-----	-----	-----	-----
1257	0.10	-0.25	0.05	1.11	2.92	-----	-0.29
1267	-----	-----	-----	-----	-----	-----	-----
1397	-----	-----	-----	-----	-----	-----	-----
1429	-1.29	-1.08	-0.93	-0.29	0.00	-----	-2.30
1455	-1.75	-1.08	-1.38	-1.52	-2.47	1.73	-2.70
1696	-----	-----	-----	-----	-----	-----	-----
1714	0.44	0.37	1.32	1.34	7.45	-----	-----
1815	-----	-----	-----	-----	-----	-----	-----
1957	-----	-----	-----	-----	-----	-----	-----
1960	-----	-----	-----	-----	-----	-----	-----
1995	-----	-----	-----	-----	-----	-----	-----
2124	-----	-----	-----	-----	-----	-----	-----
6011	-----	-----	-----	-----	-----	-----	-----
6016	-----	-----	-----	-----	-----	-----	-----
6087	-----	-----	-----	-----	-----	-----	-----
6159	0.56	0.23	0.12	-0.23	1.93	-4.63	-0.83
6160	0.98	0.89	0.42	0.53	1.89	-----	-2.23
6161	1.14	1.24	1.10	0.70	1.52	-----	-1.63
9054	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9057	-----	-----	-----	-----	-----	-----	-----
9058	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9130	-----	-----	-----	-----	-----	-----	-----
9131	-----	-----	-----	-----	-----	-----	-----
9150	-----	-----	-----	-----	-----	-----	-----

Simulated Distillation z-scores

lab	IBP	5%	10%	50%	90%	95%	FBP
140	-----	-----	-----	-----	-----	-----	-----
158	-----	-----	-----	-----	-----	-----	-----
171	-----	-----	-0.14	-2.43	-1.82	-0.18	-0.19
311	-----	-----	6.72	-1.46	-2.15	-0.18	-0.78
323	-----	-----	-----	-----	-----	-----	-----
442	-----	-----	-----	-----	-----	-----	-----
444	-----	-----	-----	-----	-----	-----	-----
608	-----	-----	0.14	1.53	1.37	0.25	0.08
609	-----	-----	-----	-----	-----	-----	-----
657	-----	-----	-----	-----	-----	-----	-----
785	-----	-----	-----	-----	-----	-----	-----
840	-----	-----	-----	-----	-----	-----	-----
873	-----	-----	-----	-----	-----	-----	-----
874	-----	-----	-----	-----	-----	-----	-----
875	-----	-----	-----	-----	-----	-----	-----
998	-----	-----	-----	-----	-----	-----	-----
1164	-----	-----	-----	-----	-----	-----	-----
1214	-----	-----	-----	-----	-----	-----	-----
1257	-----	-----	-----	-----	-----	-----	-----
1267	-----	-----	-----	-----	-----	-----	-----
1397	-----	-----	-0.14	1.15	-0.85	0.10	0.17
1429	-----	-----	-----	-----	-----	-----	-----
1455	-----	-----	-----	-----	-----	-----	-----
1696	-----	-----	-----	-----	-----	-----	-----
1714	-----	-----	0.14	1.21	3.45	5.81	0.71
1815	-----	-----	-----	-----	-----	-----	-----
1957	-----	-----	-----	-----	-----	-----	-----
1960	-----	-----	-----	-----	-----	-----	-----
1995	-----	-----	-----	-----	-----	-----	-----
2124	-----	-----	-----	-----	-----	-----	-----
6011	-----	-----	-----	-----	-----	-----	-----
6016	-----	-----	-----	-----	-----	-----	-----
6087	-----	-----	-----	-----	-----	-----	-----
6159	-----	-----	-----	-----	-----	-----	-----
6160	-----	-----	-----	-----	-----	-----	-----
6161	-----	-----	-----	-----	-----	-----	-----
9054	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9057	-----	-----	-----	-----	-----	-----	-----
9058	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9130	-----	-----	-----	-----	-----	-----	-----
9131	-----	-----	-----	-----	-----	-----	-----
9150	-----	-----	-----	-----	-----	-----	-----

APPENDIX 3:

Number of participating laboratories per country

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 1 lab in AFGHANISTAN
3 labs in AUSTRALIA
 1 lab in BELGIUM
 1 lab in CROATIA
 1 lab in EGYPT
 1 lab in INDONESIA
3 labs in IRAN, Islamic Republic of
 1 lab in KAZAKHSTAN
4 labs in MALAYSIA
5 labs in NETHERLANDS
2 labs in NORWAY
 1 lab in OMAN
 1 lab in POLAND
4 labs in RUSSIAN FEDERATION
 1 lab in SINGAPORE
 1 lab in THAILAND
4 labs in UNITED ARAB EMIRATES
6 labs in UNITED KINGDOM
3 labs in UNITED STATES OF AMERICA
 1 lab in VIETNAM
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APPENDIX 4

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

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