Results of Proficiency Test
Gascondensate
November 2016

CONTENTS

Appendices:

1   INTRODUCTION

Since 2008, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Gascondensate. During the annual proficiency testing program 2016/2017, it was decided to continue the round robin for the analysis of Gascondensate.

In this interlaboratory study 49 laboratories from 22 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2016 Gascondensate proficiency test are presented and discussed. This report is also available as PDF file from the iis website www.iisnl.com.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. In this proficiency test the participants received one sample of Gascondensate (0.5L bottle labelled #16235). 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 (R007). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2   PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: 'Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from 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 85 kg, was obtained from a participating laboratory and was spiked with Methanol (approx. 200 mg/kg). After homogenisation, 90 amber glass bottles of 0.5 litre were filled and labelled as sample #16235.

The homogeneity of the subsamples #16235 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 7 stratified randomly selected samples.

	Density at 15 °C in kg/m^3	Methanol in mg/kg
Sample #16235-1	743.06	201.9
Sample #16235-2	742.11	194.2
Sample #16235-3	743.16	194.6
Sample #16235-4	743.13	195.7
Sample #16235-5	743.10	197.7
Sample #16235-6	743.10	195.3
Sample #16235-7	743.13	195.8

Table 1: homogeneity test results of subsamples #16235

From the above test results, the repeatabilities (r) were calculated and compared with 0.3 times the reproducibility (R) of the corresponding reference test method 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	0.09	7.4
reference target method	ASTM D4052:15	Horwitz
0.3xR(ref. target method)	0.64	11.9

Table 2: repeatabilities of subsamples #16235

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the respective reference target method. Therefore, homogeneity of the subsamples of #16235 was assumed

To each of the participating laboratories, 1 * 0.5 L bottle (labelled #16235) was sent on October 19, 2016.

2.5   STABILITY OF THE SAMPLES

The stability of Gas condensate, packed in the brown glass bottles, was checked.
The material was found sufficiently stable for the period of the proficiency test.

2.6　ANALYSES

The participants were requested to determine on sample #16235: Color Saybolt (Automated and Manual), Density at 15°C, Distillation (IBP, temperature at 5%, 10%, 50%, 90%, 95% recovered and FBP), 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 to 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 results cannot be used for meaningful statistical calculations.

To get comparable test results a detailed report form, on which the units were prescribed as well as the required reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The laboratories were also requested to confirm the sample receipt on the same data entry portal. A SDS was added to the sample.

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 test 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 April 2014 (iis-protocol, version 3.3).
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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results 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 visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.
Absolute values for z<2 are very common and absolute values for z>3 are very rare.
The usual interpretation of z-scores is as follows:

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

4   EVALUATION

In this proficiency test, problems were encountered with the dispatch of the samples.
Participants in Afghanistan, Australia, Malaysia, Kazakhstan, Nigeria and Oman received the samples late or not at all.
Seven participants did not report any test results at all. Six other participants reported the test results after the final reporting date.
In total, 42 participants reported in total 297 numerical test results. Observed were 23 outlying test results, which is 7.7%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1   EVALUATION PER TEST

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

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. No statistical outliers were observed. Both calculated reproducibilities are not in agreement with the requirements of respective test methods ASTM D6045:12 and ASTM D156:15. For the automated method 11 participants returned a test result and 10 participants returned a result for the manual method. This 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 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 D4052:15.

It should be taken into account that the reproducibility from ASTM D4052:15 is applicable to petroleum distillates and viscous oils only. Therefore no precision data are stated in the 2015 version for Gas condensates. However, Gas condensates 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 eight statistical outliers were observed. After rejection of the statistical outliers, the calculated reproducibilities of IBP, temperature at 5% and 50% recovered were in agreement with the requirements of the manual mode of ASTM D86:16a. However, the temperatures at 10%, 90%, 95% recovered and Final Boiling Point were not in agreement with the requirements of the manual mode of ASTM D86:16a. 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: Serious analytical problems have been observed. The batch was spiked with methanol, therefore the minimal methanol concentration to be found was known (added amount = 199 mg/kg). The laboratories should be able to find at least 159 mg/kg [199 mg/kg$_{(added\ amount)}$ − 40 mg/kg$_{(R\ Horwitz)}$].
As the Horwitz reproducibility may be very strict, the minimum amount is set on 140 mg/kg. Two of six laboratories reported a clearly lower amount than 140 mg/kg and therefore these test results were rejected prior to data analysis.
The calculated reproducibility is not at all in agreement with the estimated reproducibility calculated using the Horwitz equation. The average recovery of Methanol (theoretical increment of 199.5 mg/kg) may be good: "less than 99%". The actual blank concentration for Methanol is unknown.

Mercury: The precision requirements of UOP938 (table 3b) are extremely strict and as they are 6 – 7 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 97 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 may be problematic at a concentration of 96.9 µg Hg per kg. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

Sulphur: This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5453:16e1.

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier 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 seven statistical outliers were observed. After rejection of the statistical outliers, the calculated reproducibilities of 10%, 50%, 90% and 95% recovered were not in agreement with the requirements of ASTM D2887:16. However, the calculated reproducibility of the Final Boiling Point after rejection of the statistical outliers is in agreement with the requirements of ASTM D2887:16. The test results reported for Initial Boiling Point and 5% recovered were not valuated as the temperature was below the measuring limit of 36°C.The 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 #16235, calculated reproducibilities and 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)		11	20.1	2.6	1.2
Color Saybolt (Manual)		10	18.6	3.8	2.0
Density at 15°C	kg/m^3	39	0.7434	0.0011	0.0021
Distillation					
Initial Boiling Point	°C	15	31.4	6.5	7.4
5%-recovered	°C	14	56.0	5.6	6.5
10%-recovered	°C	14	67.0	4.3	3.7
50%-recovered	°C	15	121.4	3.2	4.7
90%-recovered	°C	15	243.8	16.0	6.6
95%-recovered	°C	7	285.5	24.0	12.9
Final Boiling Point	°C	13	300.4	8.6	4.4
Methanol	mg/kg	4	198	139	40
Mercury as Hg	µg/kg	21	97	78	62
Sulphur	mg/kg	22	12.4	2.9	3.8
Water content by KF	mg/kg	35	52.6	29.2	182.1
Simulated Distillation					
Initial Boiling Point	°C	4	n.a.	n.a.	n.a.
5%-recovered	°C	4	n.a.	n.a.	n.a.
10%-recovered	°C	5	36.7	2.9	2.1
50%-recovered	°C	6	115.3	6.6	4.3
90%-recovered	°C	6	239.7	11.3	4.3
95%-recovered	°C	5	281.2	6.9	5.0
Final Boiling Point	°C	5	380.2	9.7	11.8

Table 3: performance evaluation sample #16235

Without further statistical calculations it can be concluded from the overview given in table 3 that for almost all 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 2016 WITH THE PREVIOUS PTS

	November 2016	November 2015	November 2014	November 2013	November 2012
Number of reporting participants	42	38	36	36	38
Number of results reported	297	248	251	216	234
Number of statistical outliers	23	8	8	15	25
Percentage of statistical outliers	7.7%	3.2%	3.2%	6.9%	10.7%

Table 4: comparison with previous proficiency tests

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 2016	November 2015	November 2014	November 2013	November 2012
Color Saybolt	--	--	--	-	--
Density at 15 ^{o}C	++	+	+	++	-
Distillation (ASTM D86)	-	-	--	--	--
Methanol	--	n.e.	n.e.	n.e.	n.e.
Mercury as Hg	-	-	-	--	--
Sulphur	+	-	--	--	++
Water content by KF	++	++	++	++	-
SimDist	--	n.e.	n.e.	n.e.	n.e.

Table 5: comparison of the performance per determination against the target requirements

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 #16235;

lab	method	automated	mark	z(targ)	method	manual	mark	z(targ)	Remarks
171	D6045	19		-2.46	D156	20		1.96	
311		-----		-----		-----		-----	
323	D6045	21		2.05		-----		-----	
334		-----		-----	D156	17		-2.24	
442		-----		-----		-----		-----	
444		-----		-----		-----		-----	
491		-----		-----		-----		-----	
492		-----		-----		-----		-----	
499		-----		-----		-----		-----	
602		-----		-----		-----		-----	
608		-----		-----	D156	18		-0.84	
609		-----		-----		-----		-----	
657		-----		-----	D156	21		3.36	
785	D6045	19		-2.46		-----		-----	
840		-----		-----	D156	18		-0.84	
873	D6045	19		-2.46		-----		-----	
875		-----		-----		-----		-----	
998		-----		-----		-----		-----	
1066		-----		-----	D156	18		-0.84	
1164	D6045	19		-2.46		-----		-----	
1214		-----		-----		-----		-----	
1257		-----		-----	D156	20		1.96	
1267		-----		-----		-----		-----	
1284	D6045	21		2.05		-----		-----	
1397		-----		-----		-----		-----	
1429	D6045	21		2.05		-----		-----	
1455		-----		-----		-----		-----	
1696		21		2.05		-----		-----	
1714		20		-0.21	D156	18		-0.84	
1800		-----		-----		-----		-----	
1815		-----		-----		-----		-----	
1957		-----		-----		-----		-----	
1960	D6045	20		-0.21		-----		-----	
1995		-----		-----		-----		-----	
2124		-----		-----	D156	19		0.56	
6016		-----		-----		-----		-----	
6052	D6045	21.0		2.05		-----		-----	
6087		-----		-----		-----		-----	
9054		-----		-----		-----		-----	
9056		-----		-----		-----		-----	
9057		-----		-----		-----		-----	
9058		-----		-----		-----		-----	
9061		-----		-----		-----		-----	
9101		-----		-----		-----		-----	
9107		-----		-----	D156	17		-2.24	
9130		-----		-----		-----		-----	
9142		-----		-----		-----		-----	
9143		-----		-----		-----		-----	
9150		-----		-----		-----		-----	
	normality	OK			normality	OK			
	n	11			n	10			
	outliers	0			outliers	0			
	mean (n)	20.09			mean (n)	18.60			
	st.dev. (n)	0.944			st.dev. (n)	1.350			
	R(calc.)	2.64			R(calc.)	3.78			
	R(D6045:12)	1.24			R(D156:15)	2.00			
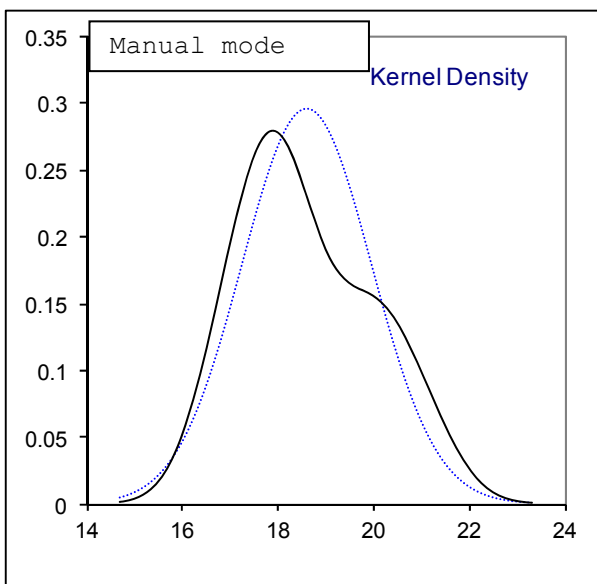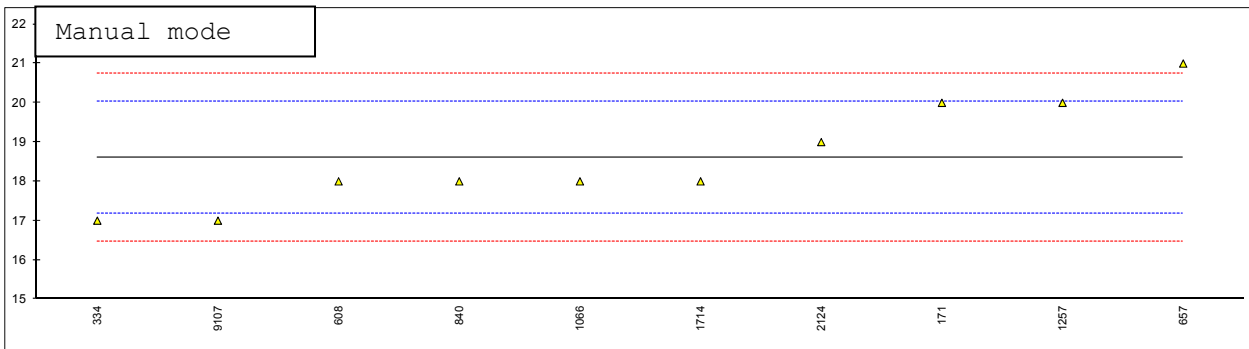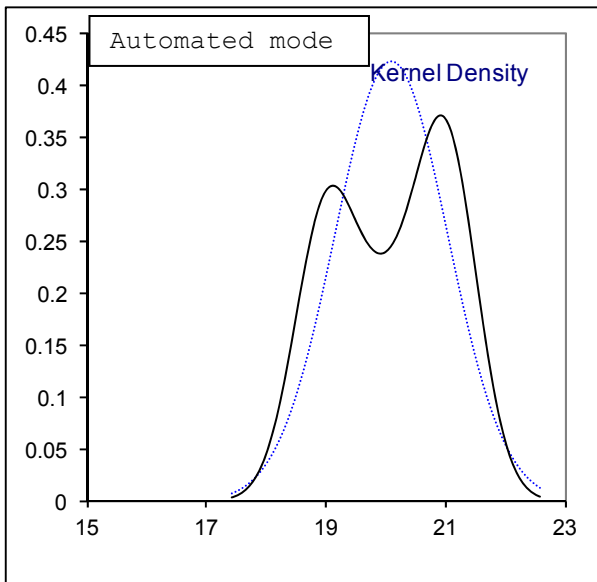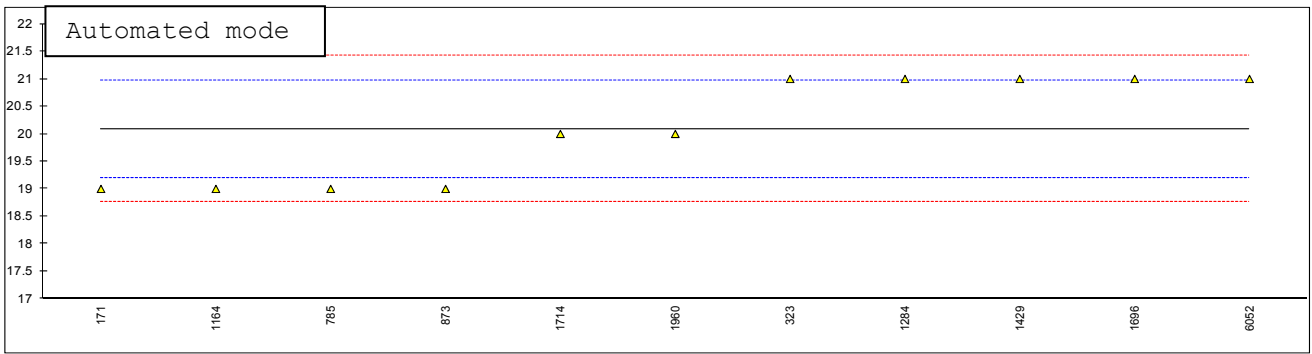
Determination of Density at 15°C on sample #16235; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7432	C	-0.21	First reported 743.2 kg/L
311	D4052	0.74316		-0.26	
323	D4052	0.7430		-0.47	
334	D4052	0.7432		-0.21	
442	D4052	0.7434		0.05	
444	D4052	0.7432		-0.21	
491	ISO12185	0.74329		-0.09	
492	ISO12185	0.74342		0.08	
499	ISO12185	0.74328		-0.10	
602	D1298	0.7429		-0.60	
608	D4052	0.7438		0.58	
609	D5002	0.7432	C	-0.21	First reported 743.2 kg/L
657	D4052	0.7432		-0.21	
785	D4052	0.7431		-0.34	
840	D4052	0.74306		-0.39	
873	D4052	0.7433		-0.08	
875		-----		-----	
998		-----		-----	
1066	D4052	0.7431		-0.34	
1164	D4052	0.7432		-0.21	
1214		-----		-----	
1257	D4052	0.7432		-0.21	
1267	IP365	0.743		-0.47	
1284	D4052	0.74328		-0.10	
1397	D4052	0.7442		1.11	
1429	D4052	0.7431		-0.34	
1455	D4052	0.7432		-0.21	
1696		0.7428		-0.74	
1714	D4052	0.74317		-0.25	
1800	D4052	0.7444		1.37	
1815	ISO12185	0.74378		0.55	
1957	D4052	0.7438	C	0.58	First reported 0.75816
1960	D4052	0.743126		-0.31	
1995		-----		-----	
2124	D4052	0.7434		0.05	
6016	D4052	0.74420		1.11	
6052	D4052	0.7445		1.50	
6087	D4052	0.743490		0.17	
9054	D4052	0.7432		-0.21	
9056	D4052	0.739	R(0.01)	-5.73	
9057	D5002	0.74355	C	0.25	First reported 743.55 kg/L
9058	D5002	0.7431		-0.34	
9061	D5002	0.74319		-0.22	
9101		-----		-----	
9107	D4052	0.74553	C,R(0.01)	2.86	First reported 737.3 kg/L
9130	D4052	0.7433		-0.08	
9142		-----		-----	
9143		-----		-----	
9150	D4052	0.7416	R(0.01)	-2.31	

|	
normality	not OK
n	39
outliers	3
mean (n)	0.74336
st.dev. (n)	0.000397
R(calc.)	0.00111
R(D4052:15)	0.00213
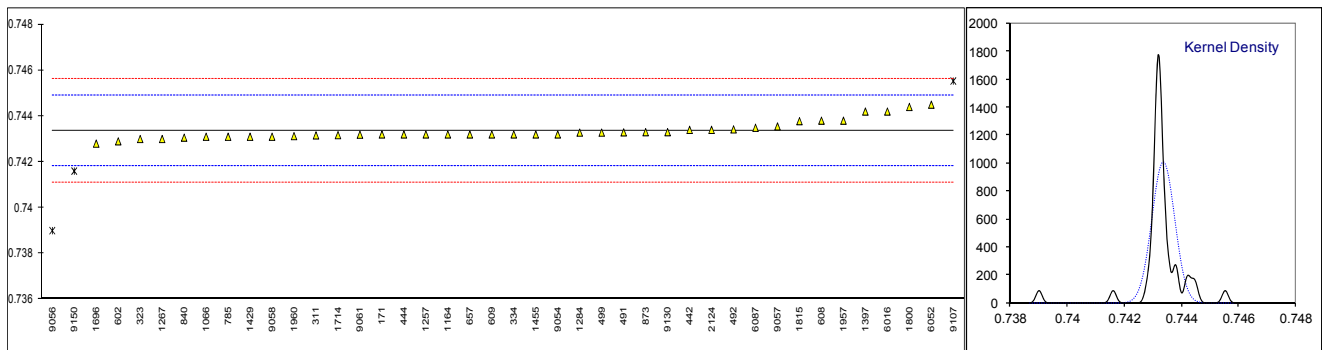
Determination of Distillation on sample #16235; results in °C and %V/V

lab	method	IBP	5%rec	10%rec	50%rec	90%rec	95%rec	FBP	res	loss
171	D86-Automated	35.5	57.5	68.5	122.8	256.5	279.0	306.0	<0.1	6.4
311		-----	-----	-----	-----	-----	-----	-----	-----	-----
323	D86-Automated	29.4	54.7	66.5	121.6	249.3	-----	-----	-----	-----
334	D86-Automated	29.8	53.1	64.6	118.9	234.4	**267.9**	**271.3**	1.7	1.9
442		-----	-----	-----	-----	-----	-----	-----	-----	-----
444		-----	-----	-----	-----	-----	-----	-----	-----	-----
491		-----	-----	-----	-----	-----	-----	-----	-----	-----
492		-----	-----	-----	-----	-----	-----	-----	-----	-----
499		-----	-----	-----	-----	-----	-----	-----	-----	-----
602		-----	-----	-----	-----	-----	-----	-----	-----	-----
608	D86-Automated	30.4	55.1	66.5	121.3	246.7	-----	301.3	1.3	4.5
609		-----	-----	-----	-----	-----	-----	-----	-----	-----
657	D86-Automated	33.3	60.6	70.7	122.3	237.2	276.6	300.7	1.4	1.3
785		-----	-----	-----	-----	-----	-----	-----	-----	-----
840	D86-Automated	31.29	55.92	66.78	119.99	243.17	-----	298.78	1.3	4.4
873	D86-Manual	33.0	56.5	67.0	120.5	242.5	294.0	298.0	1.5	3.0
875		-----	-----	-----	-----	-----	-----	-----	-----	-----
998		-----	-----	-----	-----	-----	-----	-----	-----	-----
1066		-----	-----	-----	-----	-----	-----	-----	-----	-----
1164	D86-Automated	29.2	54.6	65.9	121.2	236.7	274.7	293.3	3.0	0.2
1214		-----	-----	-----	-----	-----	-----	-----	-----	-----
1257	D86-Automated	31.1	55.5	66.7	121.9	242.0	287.1	301.5	-----	-----
1267		-----	-----	-----	-----	-----	-----	-----	-----	-----
1284	D86-Automated	32.9	57.4	67.9	122.9	243.2	293.8	303.8	1.7	1.4
1397		-----	-----	-----	-----	-----	-----	-----	-----	-----
1429	D86-Automated	28.1	54.7	66.4	121.5	241.8	-----	301.4	1.4	3.7
1455	D86-Automated	29.1	53.5	65.2	121.1	240.9	-----	299.8	1.1	4.7
1696		**37.8**	**64.1**	**73.7**	120	244.25	293.2	297.8	0.9	3.7
1714	D86-Automated	35.8	58.3	68.6	122.6	249.8	-----	**322**	0.7	4.5
1800		-----	-----	-----	-----	-----	-----	-----	-----	-----
1815		-----	-----	-----	-----	-----	-----	-----	-----	-----
1957		-----	-----	-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----	-----	-----
1995		-----	-----	-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----	-----	-----
6016		30.2	**63.8**	**74.2**	**127.6**	**276.9**	-----	301.6	1.4	5.7
6052	D86-Automated	31.9	56.4	67.0	121.6	248.4	-----	301.1	1.3	4.9
6087		-----	-----	-----	-----	-----	-----	-----	-----	-----
9054		-----	-----	-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----	-----	-----
9057		-----	-----	-----	-----	-----	-----	-----	-----	-----
9058		-----	-----	-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----	-----	-----
9101		-----	-----	-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----	-----	-----
9130		-----	-----	-----	-----	-----	-----	-----	-----	-----
9142		-----	-----	-----	-----	-----	-----	-----	-----	-----
9143		-----	-----	-----	-----	-----	-----	-----	-----	-----
9150		-----	-----	-----	-----	-----	-----	-----	-----	-----
	normality	OK	OK	suspect	OK	OK	unknown	suspect		
	n	15	14	14	15	15	7	13		
	outliers	0 (+1excl)	2	2	1	1	0 (+1excl)	2		
	mean (n)	31.40	55.99	67.02	121.35	243.79	285.49	300.39		
	st.dev. (n)	2.314	2.003	1.532	1.131	5.718	8.574	3.080		
	R(calc.)	6.48	5.61	4.29	3.17	16.01	24.01	8.63		
	R(D86:16a-M)	7.37	6.54	3.73	4.74	6.59	12.89	4.35		

NB. Results in bold and underlined are statistical outliers or are excluded for statistical evaluation

5% recovered

10% recovered

50% recovered

90% recovered

95% recovered

Final Boiling Point
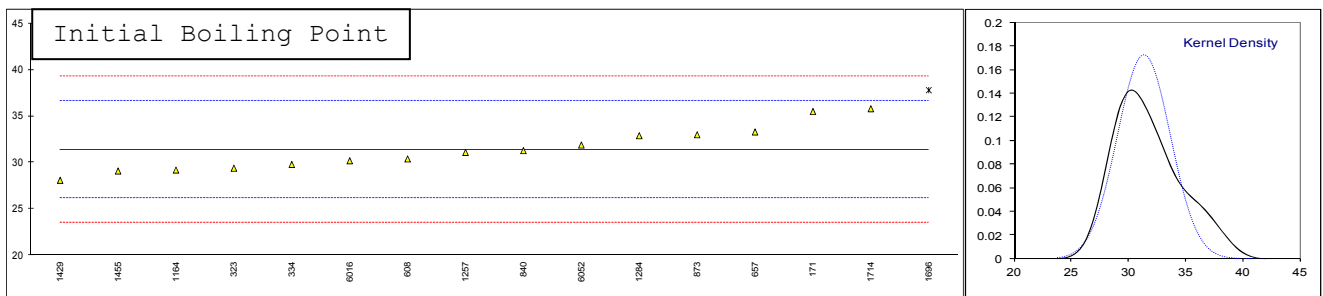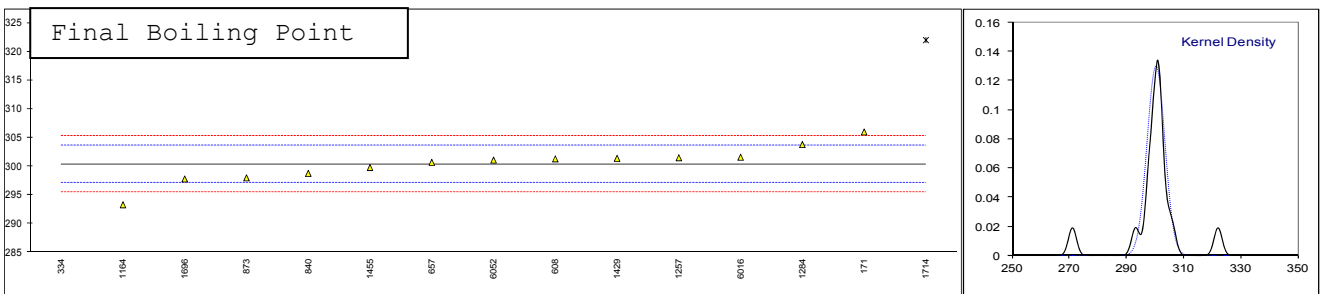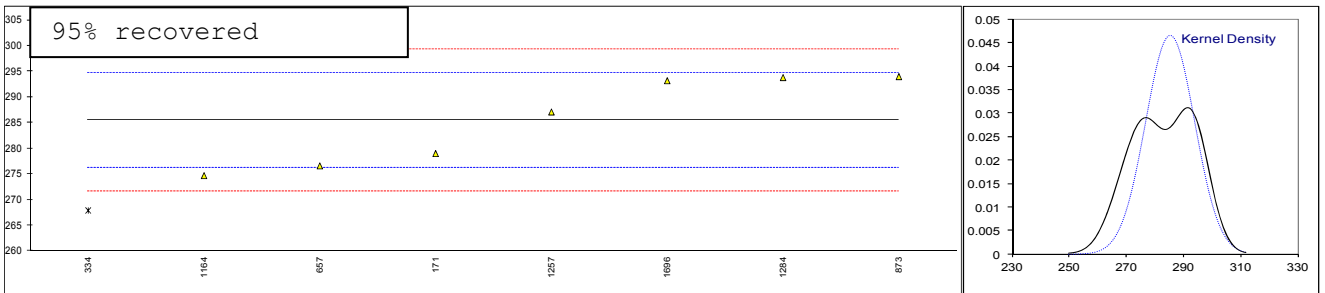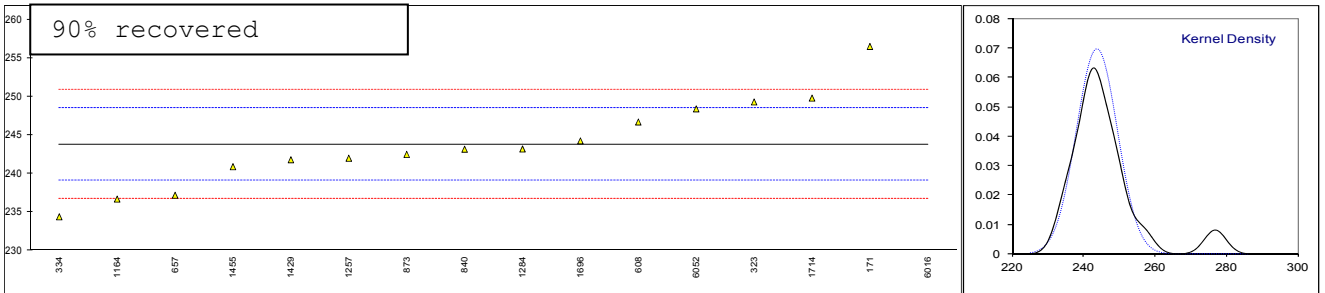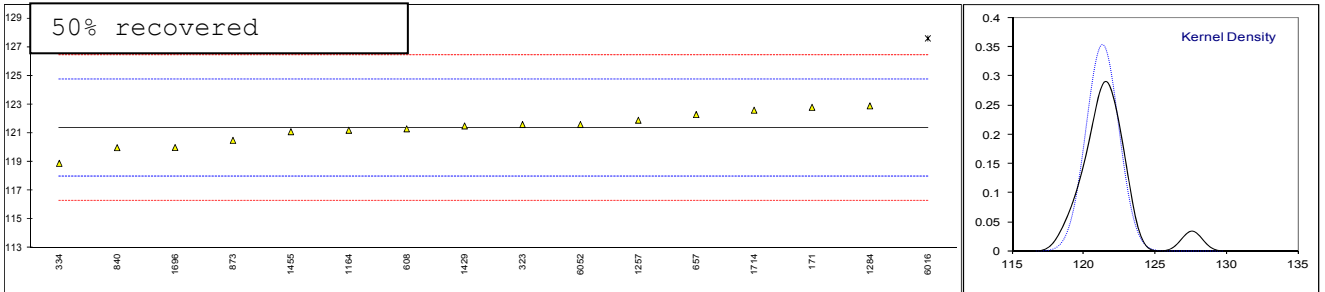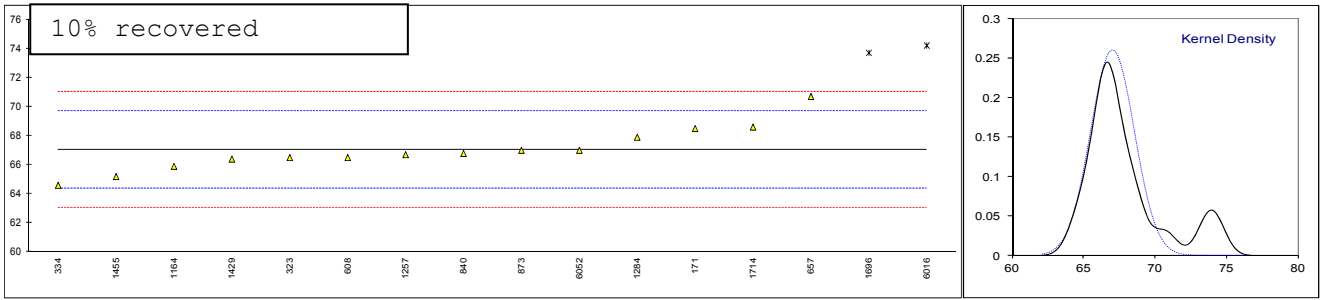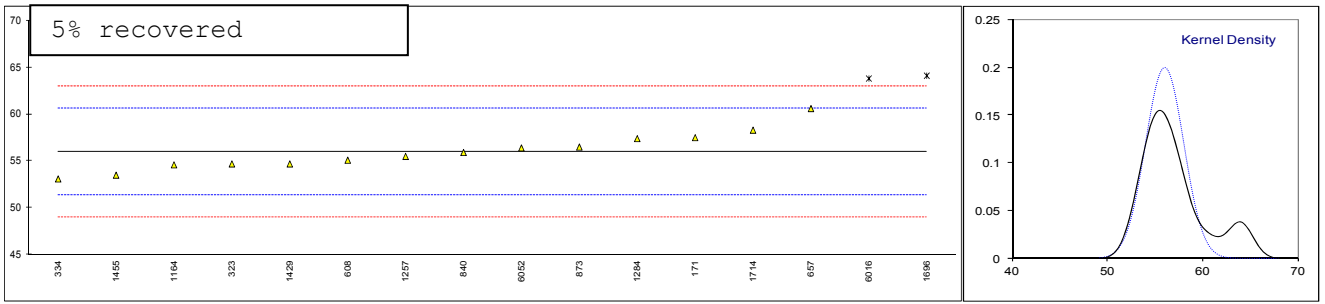
Determination of Methanol on sample #16235; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311		-----		-----	
323	INH-304	210		0.82	
334		-----		-----	
442		-----		-----	
444	INH-008	260.0		4.32	
491		-----		-----	
492		-----		-----	
499		-----		-----	
602		-----		-----	
608		-----		-----	
609		-----		-----	
657	INH-130	20.3	ex	-12.44	Result excluded, see §4.1
785		-----		-----	
840		-----		-----	
873		-----		-----	
875		-----		-----	
998		-----		-----	
1066	In house	10	ex	-13.16	Result excluded, see §4.1
1164		-----		-----	
1214		-----		-----	
1257		-----		-----	
1267		-----		-----	
1284		-----		-----	
1397		-----		-----	
1429	In house	142		-3.93	
1455	D7423	181		-1.21	
1696		-----		-----	
1714		-----		-----	
1800		-----		-----	
1815		-----		-----	
1957		-----		-----	
1960		-----		-----	
1995		-----		-----	
2124		-----		-----	
6016		-----		-----	
6052		-----		-----	
6087		-----		-----	
9054		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9101		-----		-----	
9107		-----		-----	
9130		-----		-----	
9142		-----		-----	
9143		-----		-----	
9150		-----		-----	

normality	unknown			
n	4			
outliers	0 (+2 excl)	Spike:		
mean (n)	198.25	199.5		Recovery% : <99%
st.dev. (n)	49.708			
R(calc.)	139.18			
R(Horwitz)	40.06			
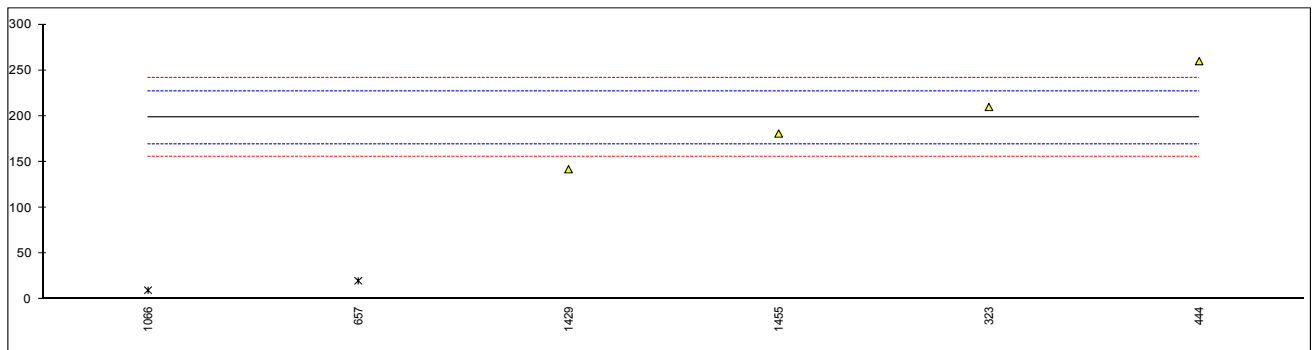
Determination of Mercury as Hg on sample #16235; results in µg/kg

lab	method	value	mark	z(targ)	remarks
171	UOP938	52.7		-2.01	
311	INH-001	120		1.05	
323	UOP938	68		-1.31	
334	INH-09003	133		1.64	
442		-----		-----	
444	UOP938	103.0		0.28	
491		-----		-----	
492		-----		-----	
499		-----		-----	
602		-----		-----	
608		-----		-----	
609		-----		-----	
657	UOP938	90		-0.31	
785		-----		-----	
840		-----		-----	
873	UOP938	99.35		0.11	
875		-----		-----	
998		-----		-----	
1066	INH-DMA80	118		0.96	
1164	UOP938	72		-1.13	
1214		-----		-----	
1257		-----		-----	
1267		-----		-----	
1284		-----		-----	
1397	In house	94.41		-0.11	
1429	INH-91	55.8659		-1.86	
1455		-----		-----	
1696	UOP938	45.83		-2.32	
1714		-----		-----	
1800	UOP938	97.5		0.03	
1815		-----		-----	
1957		-----		-----	
1960	ISO6978-2	128.50		1.44	
1995		-----		-----	
2124	INH-210	137		1.82	
6016	D7622	90.755		-0.28	
6052	UOP938	140	C	1.96	First reported 140000
6087	UOP938	111.972		0.69	
9054	UOP938	97.7124		0.04	
9056		-----		-----	
9057	In house	107.2		0.47	
9058		-----		-----	
9061		-----		-----	
9101		-----		-----	
9107	UOP938	71.17		-1.17	
9130		-----		-----	
9142		-----		-----	
9143		-----		-----	
9150		-----		-----	

				Only UOP938 data:
normality	OK			OK
n	21			12
outliers	0			0
mean (n)	96.855			87.436
st.dev. (n)	27.9088			26.5961
R(calc.)	78.145			74.469
R(Horwitz)	61.660			56.528
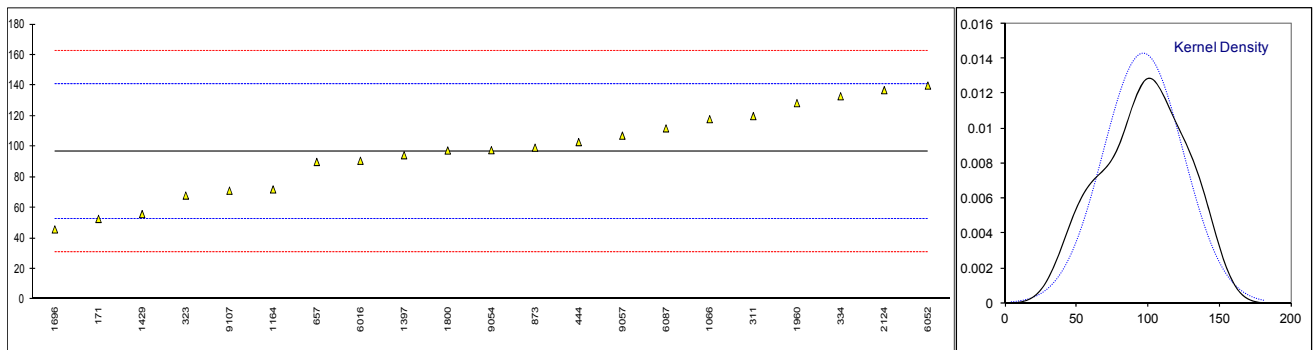
Determination of Sulphur on sample #16235; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D5453	12		-0.26	
311	D5453	11		-0.99	
323	D5453	13		0.47	
334	D5453	10.3		-1.51	
442		-----		-----	
444	D5453	7.27	R(0.05)	-3.73	
491		-----		-----	
492		-----		-----	
499		-----		-----	
602		-----		-----	
608	D5453	12.56		0.15	
609		-----		-----	
657	D5453	13.1		0.55	
785	ISO20884	12.3		-0.04	
840		-----		-----	
873	ISO20846	12.59		0.17	
875		-----		-----	
998		-----		-----	
1066	D5453	14		1.21	
1164	D5453	10.76		-1.17	
1214		-----		-----	
1257	D4294	13		0.47	
1267		-----		-----	
1284	D2622	12.8		0.33	
1397	ISO20846	11.4		-0.70	
1429	ISO20846	16.4	DG(0.05)	2.96	
1455	D2622	15.6	DG(0.05)	2.38	
1696	D5453	13.16		0.59	
1714	D5453	11.20		-0.85	
1800		< 50		-----	
1815	D5453	12.45		0.07	
1957		-----		-----	
1960	D5453	13.2		0.62	
1995		-----		-----	
2124	D5453	12.31		-0.03	
6016	D5453	6.6805	R(0.05)	-4.16	
6052	D5453	14.477		1.56	
6087	D5453	11.31		-0.77	
9054		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9101		-----		-----	
9107		-----		-----	
9130	D5453	11.94		-0.30	
9142		-----		-----	
9143		-----		-----	
9150	D5453	12.95		0.44	

normality	OK
n	22
outliers	4
mean (n)	12.355
st.dev. (n)	1.0434
R(calc.)	2.921
R(D5453:16e1)	3.820
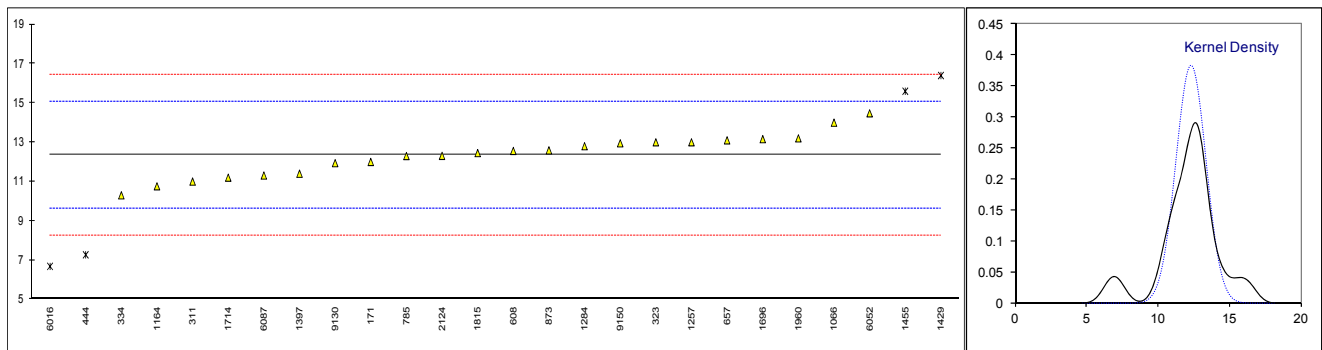
Determination of Water content by KF on sample #16235; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D6304-A	60		0.11	
311	D6304-A	65		0.19	
323	D6304-A	49		-0.06	
334	D6304	60		0.11	
442	IP438	39		-0.21	
444	IP438	38		-0.22	
491		-----		-----	
492		-----		-----	
499		-----		-----	
602		-----		-----	
608	D4928	54.2		0.02	
609	D4928	46.996		-0.09	
657	D6304-A	36.6		-0.25	
785	ISO12937	50		-0.04	
840	D6304-A	45.8		-0.10	
873	D6304-A	59.5		0.11	
875		-----		-----	
998		-----		-----	
1066	IP439	78		0.39	
1164	D6304-A	51.3		-0.02	
1214		-----		-----	
1257	D6304-A	60.2		0.12	
1267	D4928	46.10		-0.10	
1284		-----		-----	
1397	ISO12937	36		-0.26	
1429	IP438	45.2		-0.11	
1455	ISO12937	56		0.05	
1696		65.61		0.20	
1714		45		-0.12	
1800	D6304-A	42.9		-0.15	
1815	ISO12937	46.49		-0.09	
1957	D6304-A	51		-0.02	
1960	D4928	51		-0.02	
1995		-----		-----	
2124	D4928	57.02		0.07	
6016	D6304-A	36.05		-0.25	
6052	D6304-A	70.9		0.28	
6087	D4928	49.333		-0.05	
9054		-----		-----	
9056	D6304	200	R(0.01)	2.27	
9057	In house	52.4		0.00	
9058	In house	54.5		0.03	
9061	D4928	50		-0.04	
9101		-----		-----	
9107	D6304-A	74		0.33	
9130	D6304-A	58.94		0.10	
9142		-----		-----	
9143		-----		-----	
9150	D6304-A	59.05		0.10	

normality	OK
n	35
outliers	1
mean (n)	52.603
st.dev. (n)	10.4409
R(calc.)	29.234
R(D6304:16e1)	182.086
(mass injection)	
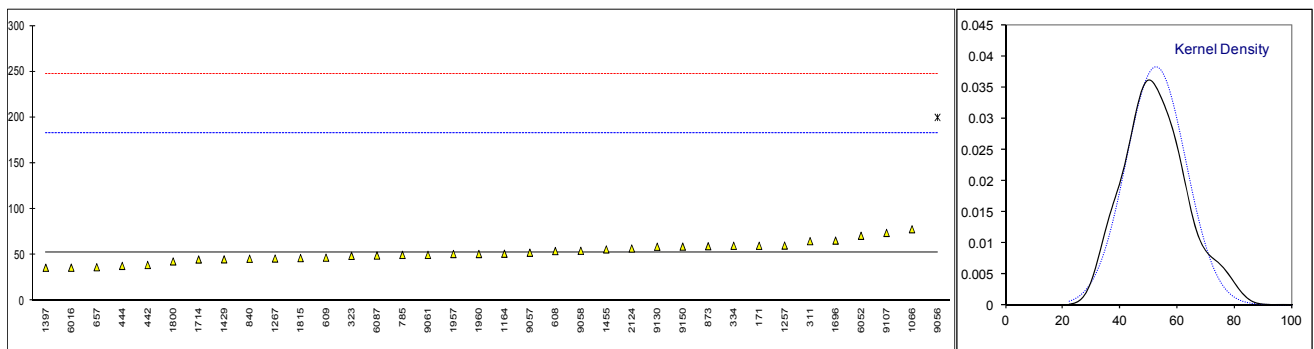
Determination of Simulated Distillation on sample #16235; results in °C

lab	method	IBP	5%rec	10%rec	50%rec	90%rec	95%rec	FBP
171	D2887	18.0	24.0	36.0	113.0	239.0	279.0	379.0
311	D2887	<36	<36	38.5	115.0	240.5	281.0	382.5
323	D2887	-----	-----	-----	**135.7**	**256.7**	**300.4**	**404.7**
334		-----	-----	-----	-----	-----	-----	-----
442		-----	-----	-----	-----	-----	-----	-----
444		-----	-----	-----	-----	-----	-----	-----
491		-----	-----	-----	-----	-----	-----	-----
492		-----	-----	-----	-----	-----	-----	-----
499		-----	-----	-----	-----	-----	-----	-----
602		-----	-----	-----	-----	-----	-----	-----
608	D2887	-7.29	22.81	**42.61**	117.46	238.44	279.19	374.70
609		-----	-----	-----	-----	-----	-----	-----
657		-----	-----	-----	-----	-----	-----	-----
785		-----	-----	-----	-----	-----	-----	-----
840		-----	-----	-----	-----	-----	-----	-----
873		-----	-----	-----	-----	-----	-----	-----
875		-----	-----	-----	-----	-----	-----	-----
998		-----	-----	-----	-----	-----	-----	-----
1066	D2887	-10	14.5	36.5	116.5	243.0	282.0	381.5
1164		-----	-----	-----	-----	-----	-----	-----
1214		-----	-----	-----	-----	-----	-----	-----
1257		-----	-----	-----	-----	-----	-----	-----
1267		-----	-----	-----	-----	-----	-----	-----
1284		-----	-----	-----	-----	-----	-----	-----
1397	D2887	16.00	26.00	36.00	112.00	233.00	**269.00**	**345.00**
1429		-----	-----	-----	-----	-----	-----	-----
1455		-----	-----	-----	-----	-----	-----	-----
1696		-----	-----	-----	-----	-----	-----	-----
1714	D2887	<0.5	<0.5	36.4	117.7	244.5	285.0	383.3
1800		-----	-----	-----	-----	-----	-----	-----
1815		-----	-----	-----	-----	-----	-----	-----
1957		-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----
1995		-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----
6016		-----	-----	-----	-----	-----	-----	-----
6052		-----	-----	-----	-----	-----	-----	-----
6087		-----	-----	-----	-----	-----	-----	-----
9054		-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----
9057		-----	-----	-----	-----	-----	-----	-----
9058		-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----
9101		-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----
9130		-----	-----	-----	-----	-----	-----	-----
9142		-----	-----	-----	-----	-----	-----	-----
9143		-----	-----	-----	-----	-----	-----	-----
9150		-----	-----	-----	-----	-----	-----	-----
	normality	unknown	unknown	unknown	unknown	unknown	unknown	unknown
	n	4	4	5	6	6	5	5
	outliers	n.a.	n.a.	1	1	1	2	2
	mean (n)	unknown	unknown	36.68	115.28	239.74	281.24	380.20
	st.dev. (n)	n.a.	n.a.	1.043	2.372	4.038	2.449	3.474
	R(calc.)	n.a.	n.a.	2.92	6.64	11.31	6.86	9.73
	R(D2887:16)	n.a.	n.a.	2.05	4.30	4.30	5.00	11.80

NB. Results in bold and underlined are statistical outliers
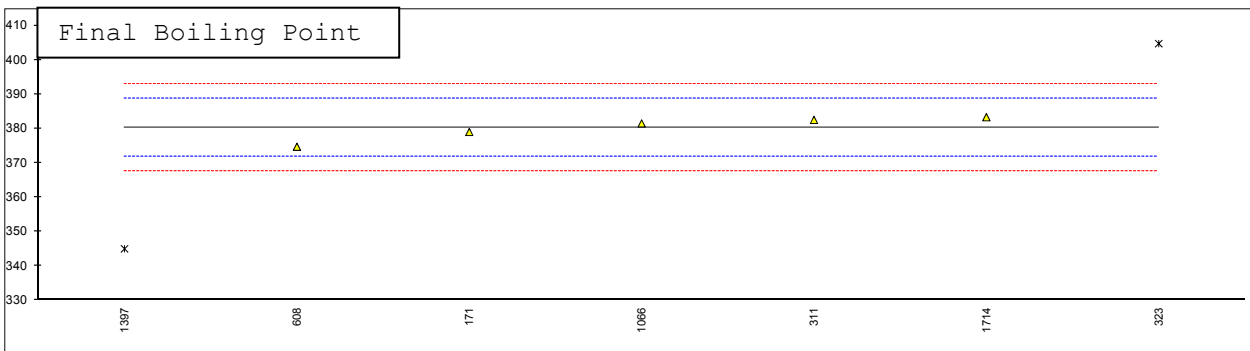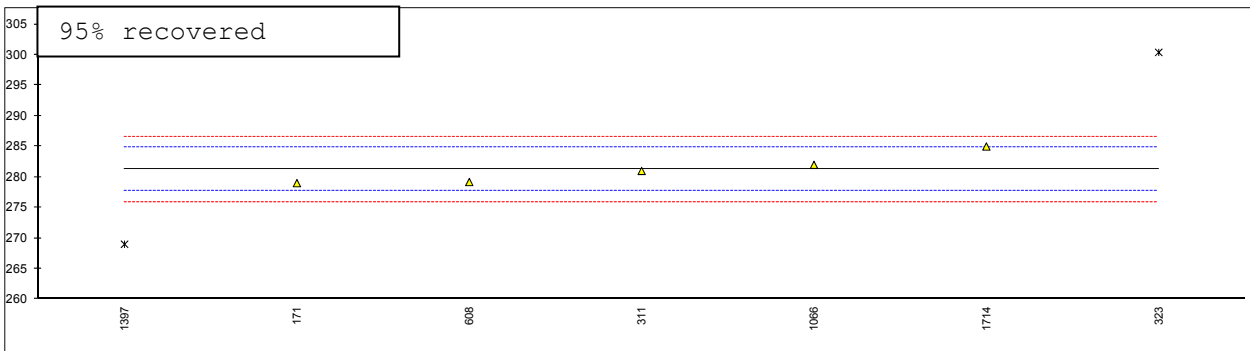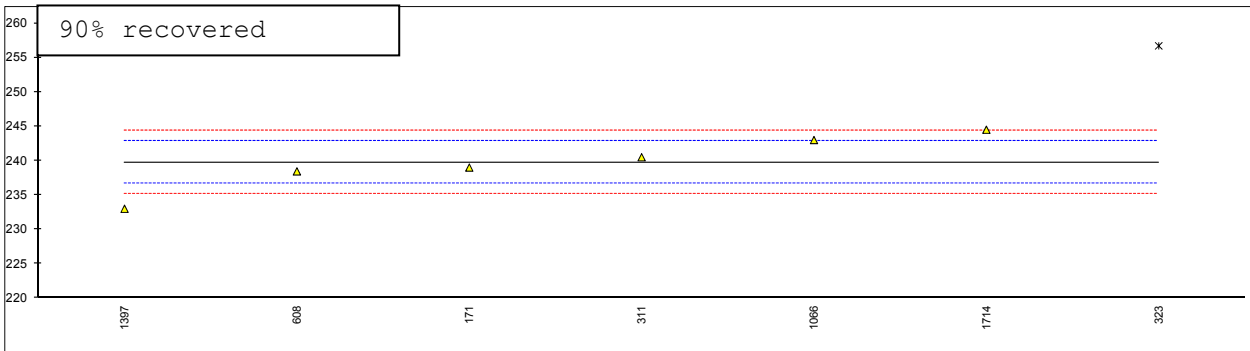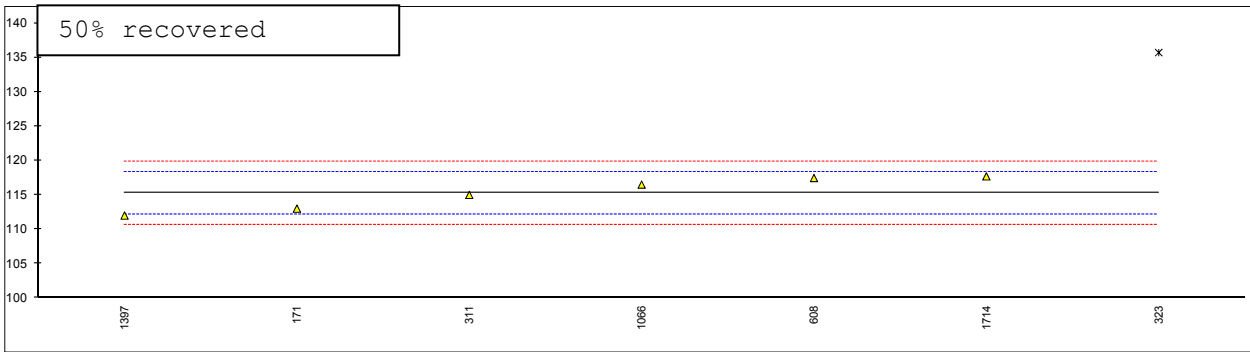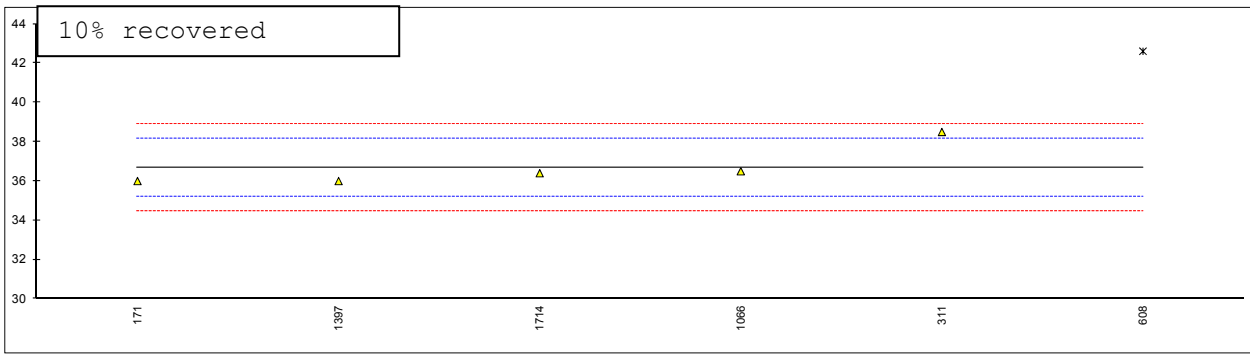
APPENDIX 2:

Atmospheric Distillation z-scores

lab		IBP	5%	10%	50%	90%	95%	FBP
171	D86	1.56	0.65	1.11	0.86	5.35	-1.41	3.61
311		-----	-----	-----	-----	-----	-----	-----
323	D86	-0.76	-0.55	-0.39	0.15	2.32	-----	-----
334	D86	-0.61	-1.24	-1.82	-1.45	-3.95	**-3.82**	**-18.72**
442		-----	-----	-----	-----	-----	-----	-----
444		-----	-----	-----	-----	-----	-----	-----
491		-----	-----	-----	-----	-----	-----	-----
492		-----	-----	-----	-----	-----	-----	-----
499		-----	-----	-----	-----	-----	-----	-----
602		-----	-----	-----	-----	-----	-----	-----
608	D86	-0.38	-0.38	-0.39	-0.03	1.23	-----	0.58
609		-----	-----	-----	-----	-----	-----	-----
657	D86	0.72	1.98	2.76	0.56	-2.77	-1.93	0.20
785		-----	-----	-----	-----	-----	-----	-----
840	D86	-0.04	-0.03	-0.18	-0.80	-0.26	-----	-1.04
873	D86	0.61	0.22	-0.02	-0.50	-0.54	1.85	-1.54
875		-----	-----	-----	-----	-----	-----	-----
998		-----	-----	-----	-----	-----	-----	-----
1066		-----	-----	-----	-----	-----	-----	-----
1164	D86	-0.84	-0.59	-0.84	-0.09	-2.98	-2.34	-4.56
1214		-----	-----	-----	-----	-----	-----	-----
1257	D86	-0.11	-0.21	-0.24	0.33	-0.75	0.35	0.71
1267		-----	-----	-----	-----	-----	-----	-----
1284	D86	0.57	0.61	0.66	0.92	-0.25	1.81	2.19
1397		-----	-----	-----	-----	-----	-----	-----
1429	D86	-1.25	-0.55	-0.47	0.09	-0.84	-----	0.65
1455	D86	-0.87	-1.07	-1.37	-0.15	-1.22	-----	-0.38
1696		**2.43**	**3.47**	**5.01**	-0.80	0.19	1.68	-1.67
1714	D86	1.67	0.99	1.19	0.74	2.53	-----	**13.90**
1800		-----	-----	-----	-----	-----	-----	-----
1815		-----	-----	-----	-----	-----	-----	-----
1957		-----	-----	-----	-----	-----	-----	-----
1960		-----	-----	-----	-----	-----	-----	-----
1995		-----	-----	-----	-----	-----	-----	-----
2124		-----	-----	-----	-----	-----	-----	-----
6016		-0.46	**3.35**	**5.39**	**3.70**	13.94	-----	0.78
6052	D86	0.19	0.18	-0.02	0.15	1.94	-----	0.46
6087		-----	-----	-----	-----	-----	-----	-----
9054		-----	-----	-----	-----	-----	-----	-----
9056		-----	-----	-----	-----	-----	-----	-----
9057		-----	-----	-----	-----	-----	-----	-----
9058		-----	-----	-----	-----	-----	-----	-----
9061		-----	-----	-----	-----	-----	-----	-----
9101		-----	-----	-----	-----	-----	-----	-----
9107		-----	-----	-----	-----	-----	-----	-----
9130		-----	-----	-----	-----	-----	-----	-----
9142		-----	-----	-----	-----	-----	-----	-----
9143		-----	-----	-----	-----	-----	-----	-----
9150		-----	-----	-----	-----	-----	-----	-----

Bold and underlined test results are outliers according to Dixon/Grubbs/Rosner

Simulated Distillation z-scores

lab	IBP	5%	10%	50%	90%	95%	FBP
171	-----	-----	-0.93	-1.48	-0.48	-1.25	-0.28
311	-----	-----	2.49	-0.18	0.49	-0.13	0.55
323	-----	-----	-----	**13.30**	**11.04**	**10.73**	**5.81**
334	-----	-----	-----	-----	-----	-----	-----
442	-----	-----	-----	-----	-----	-----	-----
444	-----	-----	-----	-----	-----	-----	-----
491	-----	-----	-----	-----	-----	-----	-----
492	-----	-----	-----	-----	-----	-----	-----
499	-----	-----	-----	-----	-----	-----	-----
602	-----	-----	-----	-----	-----	-----	-----
608	-----	-----	**8.10**	1.42	-0.85	-1.15	-1.31
609	-----	-----	-----	-----	-----	-----	-----
657	-----	-----	-----	-----	-----	-----	-----
785	-----	-----	-----	-----	-----	-----	-----
840	-----	-----	-----	-----	-----	-----	-----
873	-----	-----	-----	-----	-----	-----	-----
875	-----	-----	-----	-----	-----	-----	-----
998	-----	-----	-----	-----	-----	-----	-----
1066	-----	-----	-0.25	0.80	2.12	0.43	0.31
1164	-----	-----	-----	-----	-----	-----	-----
1214	-----	-----	-----	-----	-----	-----	-----
1257	-----	-----	-----	-----	-----	-----	-----
1267	-----	-----	-----	-----	-----	-----	-----
1284	-----	-----	-----	-----	-----	-----	-----
1397	-----	-----	-0.93	-2.13	-4.39	**-6.85**	**-8.35**
1429	-----	-----	-----	-----	-----	-----	-----
1455	-----	-----	-----	-----	-----	-----	-----
1696	-----	-----	-----	-----	-----	-----	-----
1714	-----	-----	-0.38	1.58	3.10	2.11	0.74
1800	-----	-----	-----	-----	-----	-----	-----
1815	-----	-----	-----	-----	-----	-----	-----
1957	-----	-----	-----	-----	-----	-----	-----
1960	-----	-----	-----	-----	-----	-----	-----
1995	-----	-----	-----	-----	-----	-----	-----
2124	-----	-----	-----	-----	-----	-----	-----
6016	-----	-----	-----	-----	-----	-----	-----
6052	-----	-----	-----	-----	-----	-----	-----
6087	-----	-----	-----	-----	-----	-----	-----
9054	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9057	-----	-----	-----	-----	-----	-----	-----
9058	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9101	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9130	-----	-----	-----	-----	-----	-----	-----
9142	-----	-----	-----	-----	-----	-----	-----
9143	-----	-----	-----	-----	-----	-----	-----
9150	-----	-----	-----	-----	-----	-----	-----

Bold and underlined test results are outliers according to Dixon/Grubbs/Rosner

APPENDIX 3:

Number of participating laboratories per country

1 lab in AFGHANISTAN
3 labs in AUSTRALIA
1 lab in BELGIUM
1 lab in CROATIA
1 lab in EGYPT
1 lab in FRANCE
3 labs in GERMANY
1 lab in INDONESIA
1 lab in KAZAKHSTAN
5 labs in MALAYSIA
6 labs in NETHERLANDS
3 labs in NIGERIA
2 labs in NORWAY
1 lab in OMAN
1 lab in POLAND
1 lab in QATAR
3 labs in RUSSIAN FEDERATION
1 lab in SINGAPORE
4 labs in UNITED ARAB EMIRATES
7 labs in UNITED KINGDOM
1 lab in UNITED STATES OF AMERICA
1 lab in VIETNAM

APPENDIX 4

Abbreviations:

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

Literature:

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