Results of Proficiency Test
Gas condensate
November 2013

Organised by: Institute for Interlaboratory Studies
              Spijkenisse, the Netherlands

Authors: ing. R.J. Starink
Correctors: dr. R.G. Visser and ing. L. Sweere
Report: iis13R03

January 2014

CONTENTS

Appendices:

1   INTRODUCTION

Since 2008, the Institute for Interlaboratory Studies organizes a proficiency test for Gas condensate every year. During the annual proficiency testing program 2013/2014, it was decided to continue the round robin for the analysis of Gas condensate. In this interlaboratory study, 44 laboratories from 17 different countries have participated. See appendix 3 for the number of participating laboratories per country. In this report, the results of the 2013 Gas condensate proficiency test are presented and discussed.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to send 1 sample of Gas condensate (1* 0.5 L bottle labelled #13202). Participants were requested to report rounded and unrounded results. The unrounded 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% confidentially 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 January 2010 (iis-protocol, version 3.2).

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

The necessary bulk material, approximately 50 litre was obtained from a local supplier. After homogenisation, 98 amber glass bottles of 0.5 litre were filled and labelled as sample #13202

The homogeneity of the subsamples #13202 was checked by determination of Density @15°C in accordance with ASTM D4052:11 on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/L
Sample #13202-1	743.52
Sample #13202-2	743.57
Sample #13202-3	743.55
Sample #13202-4	743.55
Sample #13202-5	743.53
Sample #13202-6	743.53
Sample #13202-7	743.50
Sample #13202-8	743.60

Table 1: homogeneity test results of subsamples #13202

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density @ 15 ^{o}C in kg/L
r sample	0.09
reference method	ASTM D4052:11
0.3xR(reference method)	0.64

Table 2: repeatability of subsamples #13202

The calculated repeatability was less than 0.3 times the reproducibility of the respective reference method. Therefore, homogeneity of the subsamples #13202 was assumed.

To each of the participating laboratories, 1 * 0.5 L bottle (labelled #13202) were sent on October 16, 2013.

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 #13202: Density @ 15°C, total Sulphur, Distillation (IBP, 5%, 10%, 50%, 90%, 95% recovered and FBP), Colour Saybolt, Water by KF and Mercury.

To get maximum information for the statistical calculations, the participants were requested to report unrounded results and results below the usual lower reporting limits, where possible.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website.
A SDS and a form to confirm receipt of the samples were added to the sample package.

3   RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported results at that moment. Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1   STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. 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 results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs 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 under 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 standard. 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 (see appendix 4; nos.12 and 13)

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. The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

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

The $z_{(target)}$ scores are listed in the result tables in appendix 1.

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 the fit-for-useness of the reported test result.

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:

$$| z | < 1 \quad \text{good}$$
$$1 < | z | < 2 \quad \text{satisfactory}$$
$$2 < | z | < 3 \quad \text{questionable}$$
$$3 < | z | \quad \text{unsatisfactory}$$

4   EVALUATION

In this proficiency test, some problems were encountered during the transport of the samples to the laboratories in Indonesia, Malaysia, Nigeria, Norway, Oman, Qatar, Russia, United Arab Emirates and United Kingdom. The samples took an unexpected long time to reach a number of laboratories due to several problems with custom clearance and/or transport companies.

Eleven participants reported test results after the final reporting date and eight laboratories did not report any test results at all due to several reasons. In total 36 laboratories reported 216 numerical results. Observed were 15 outlying results, which is 6.9%. 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. Not all original data sets proved to have a normal distribution. Not normal distributions were found for the following determinations: Colour Saybolt, Density, Distillation 10%rec, Mercury and Water. In these cases the statistical evaluation should be used with due care.

Colour Saybolt: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D156:12.

Density @15°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4052:11. It should be noticed that ASTM D4052:11 gives reproducibilities for the density range 0.71 g/ml to 0.88 g/ml, being valid only for petroleum distillates and viscous oils. Therefore this 2011 version is in principle not applicable for Gas condensates. It should be noted that 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 was very problematic. In total eight statistical outliers were observed. After rejection of the statistical outliers none of the calculated reproducibilities is in agreement with the requirements for automated or manual mode of ASTM D86:12b, except for 5%, 50% and 95% recovered (manual mode). However, 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 simdist determination may be more appropriate.

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 104 µg/**kg** will lead to extra uncertainty. Therefore, it was decided to use the Horwitz estimates for evaluation of the test results in this report.
             This determination appeared to be problematic at a consensus of 104 µg Hg/kg. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the estimated reproducibility calculated using the Horwitz equation. One false negative was observed.
             The large spread may (partly) explained by the low number of test results, the variety of test methods used, reporting in a different unit µg/L instead of in µg/kg as requested and/or the presents of particle bound mercury.

Sulphur: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D5453:12.

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 D6304:07. It must be noted that the precision data of ASTM D4928 are not applicable at this low concentration (valid between 0.02 – 5.00%M/M).

4.2   PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant
standard and the reproducibility as found for the group of participating laboratories. The
average results of sample #13202, calculated reproducibilities and reproducibilities, derived
from literature standards (in casu ASTM standards) are compared in the next table.

Parameter	unit	n	mean	2.8 * sd	R (lit)
Colour Saybolt		15	15.0	3.0	2.0
Density @ 15°C	kg/m^3	33	743.7	1.4	2.1
Initial Boiling Point	°C	15	32.5	9.7	5.0
5%-recovered	°C	15	54.9	7.0	3.2
10%-recovered	°C	15	65.6	6.4	3.2
50%-recovered	°C	15	122.8	4.1	1.9
90%-recovered	°C	13	263.2	10.2	6.1
90%-recovered	°C	7	301.0	11.5	6.9
Final Boiling Point	°C	14	317.5	10.8	6.8
Mercury as Hg	µg/kg	15	104.3	147.3	65.7
Sulphur	mg/kg	19	80.0	29.2	15.5
Water content by KF	mg/kg	24	36.5	17.9	146.3

Table 3: performance evaluation sample #13202

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 standards. The problematic tests have been discussed in paragraph 4.1.

4.3   COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2013 WITH THE PREVIOUS PTS

	November 2013	*November 2012*	*November 2011*	*November 2010*
Number of reporting participants	36	38	41	25
Number of results reported	216	234	283	215
Number of statistical outliers	15	25	29	25
Percentage of statistical outliers	6.9%	10.7%	10.2%	11.6%

Table 4: comparison with previous proficiency tests

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

Determination	*November 2013*	*November 2012*	*November 2011*	*November 2010*
Colour Saybolt	-	--	--	--
Density @ 15 ^{o}C	++	-	++	-
Distillation (ASTM D86)	--	--	--	--
Mercury as Hg	--	--	-	--
Sulphur	--	++	--	+
Water content by KF	++	-	-	-
Total vapour pressure	n.e.	-	-	--
DVPE acc. to ASTM D5191	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 standards is listed in the above table. The following performance categories were used:

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

APPENDIX 1

Determination of Colour Saybolt on sample #13202;

lab	method	value	mark	z(targ)	remarks
171	D156	16		1.40	
311		-----		-----	
323	D156	15		0.00	
442		-----		-----	
444	D6045	14		-1.40	
602		-----		-----	
608	D156	13		-2.80	
609		-----		-----	
657	D156	13		-2.80	
840		-----		-----	
974	D156	22	G(0.01)	9.80	
1023		-----		-----	
1164	D156	14	C	-1.40	First reported 23
1214		-----		-----	
1257	D156	16		1.40	
1267		-----		-----	
1397		-----		-----	
1429	D6045	16		1.40	
1455	D6045	15		0.00	
1585		-----		-----	
1616	D6045	15		0.00	
1696	D6045	15		0.00	
1800		-----		-----	
1815		-----		-----	
1842		-----		-----	
1857		-----		-----	
1949		-----		-----	
2124	D156	16		1.40	
2513		-----		-----	
9050		-----		-----	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9090	D6045	16		1.40	
9107	D156	16		1.40	
9108		-----		-----	
9117		-----		-----	
9127	D156	15		0.00	
9129		-----		-----	
9130		-----		-----	
9150		-----		-----	

normality	not OK
n	15
outliers	1
mean (n)	15.0
st.dev. (n)	1.07
R(calc.)	3.0
R(D156:12)	2.0
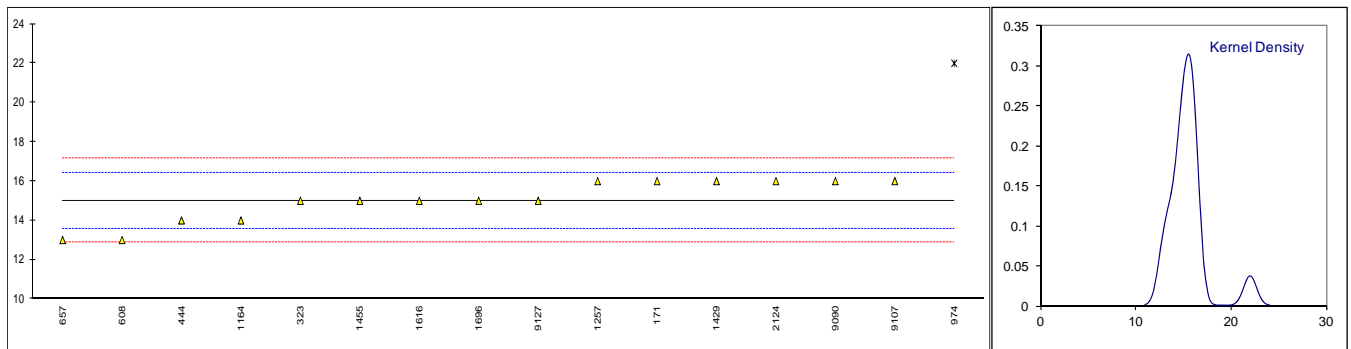
Determination of Density @ 15°C on sample #13202; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
171	D4052	747.5	G(0.01)	4.98	
311	D4052	743.6		-0.19	
323	D4052	745.0		1.67	
442	D4052	743.6		-0.19	
444	D4052	743.61		-0.17	
602	D1298	742.9		-1.11	
608	D4052	744.1		0.48	
609	D5002	743.60		-0.19	
657	D4052	743.5		-0.32	
840	D4052	743.77		0.04	
974	D4052	743.4		-0.45	
1023		-----		-----	
1164	D4052	743.49		-0.33	
1214		-----		-----	
1257	D4052	743.9		0.21	
1267	D4052	743		-0.98	
1397	D4052	744.2		0.61	
1429	D4052	744.2		0.61	
1455	D4052	743.7	C	-0.05	First reported 0.7437
1585		-----		-----	
1616	D4052	743.65		-0.12	
1696	D1298	744.50		1.01	
1800	D4052	747.4	G(0.01)	4.84	
1815	ISO12185	744.94		1.59	
1842	D4052	743.5	C	-0.32	First reported 0.7435
1857		-----		-----	
1949		-----		-----	
2124	D4052	743.6		-0.19	
2513	D4052	743.7		-0.05	
9050		-----		-----	
9054	D4052	743.8		0.08	
9055		-----		-----	
9056	INH-21	743.6		-0.19	
9057	D5002	744.02		0.37	
9058	D5002	743.3		-0.58	
9061	D5002	743.5		-0.32	
9090	D4052	743.5		-0.32	
9107	D4052	743.8		0.08	
9108		-----		-----	
9117		-----		-----	
9127	D4052	743.46		-0.37	
9129	in house	744.5		1.01	
9130	D4052	743.5		-0.32	
9150	D4052	743.0		-0.98	

normality	not OK	
n	33	
outliers	2	
mean (n)	743.74	
st.dev. (n)	0.485	
R(calc.)	1.36	
R(D4052:11)	2.11	
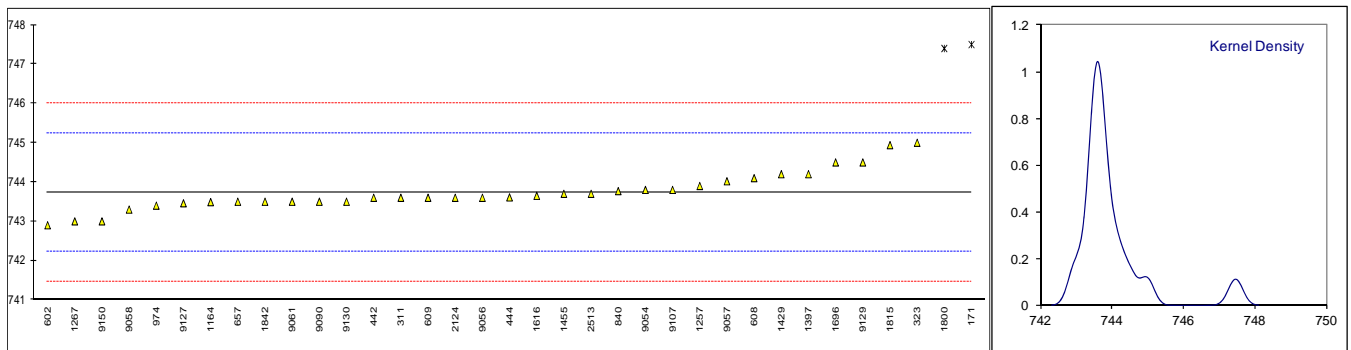
Determination of Distillation on sample #13202; results in °C

lab	method	IBP	mark	5%rec	mark	10%rec	mark	50%rec	mark	90%rec	mark	95%rec	mark	FBP	mark
171	D86-A	33.2		56.3		67.2		123.7		259.5		298.8		314.8	
311		-----		-----		-----		-----		-----		-----		-----	
323	D86-A	33.3		55.2		65.5		123.3		266.4		-----		320.9	
442		-----		-----		-----		-----		-----		-----		-----	
444		-----		-----		-----		-----		-----		-----		-----	
602		-----		-----		-----		-----		-----		-----		-----	
608	D86-A	29.4		53.7		64.4		121.5		260.4		-----		-----	
609		-----		-----		-----		-----		-----		-----		-----	
657	D86-A	34.9		58.5		68.5		123.7		261.0		304.7		323.0	
840	D86-A	31.67		55.10		65.46		121.71		261.68		303.12		317.46	
974	D86-M	38.0		55.0		68.0		124.0		283.0	DG(1)	-----		308.0	
1023		-----		-----		-----		-----		-----		-----		-----	
1164	D86-A	31.0		54.5		65.5		123.0		259.5		300.5		315.0	
1214		-----		-----		-----		-----		-----		-----		-----	
1257	D86-A	31.0		53.9		64.1		121.6		260.5		303		313.5	
1267		-----		-----		-----		-----		-----		-----		-----	
1397		-----		-----		-----		-----		-----		-----		-----	
1429	D86-A	27.1		53.5		64.7		123.6		271.2		-----		317.9	
1455	D86-A	28.2		51.9		63.5		121.9		265.4		-----		317.7	
1585		-----		-----		-----		-----		-----		-----		-----	
1616	D86-M	35.0		56.0		64.0		123.0		266.0		304.0		322.0	
1696	D86-M	33.00		53.00		64.00		121.00		267.00		293.00		318.00	
1800		-----		-----		-----		-----		-----		-----		-----	
1815	ISO3405-A	31.8		51.0		62.3		120.9		260.7		-----		296.8	G(1)
1842	D86-A	39.8		61.1		71.1		126.5		281.0	DG(1)	-----		318.9	
1857		-----		-----		-----		-----		-----		-----		-----	
1949		-----		-----		-----		-----		-----		-----		-----	
2124		-----		-----		-----		-----		-----		-----		-----	
2513	D86-A	29.4		54.0		65.1		122.3		262.5		-----		317.3	
9050		-----		-----		-----		-----		-----		-----		-----	
9054		-----		-----		-----		-----		-----		-----		-----	
9055		-----		-----		-----		-----		-----		-----		-----	
9056		-----		-----		-----		-----		-----		-----		-----	
9057		-----		-----		-----		-----		-----		-----		-----	
9058		-----		-----		-----		-----		-----		-----		-----	
9061		-----		-----		-----		-----		-----		-----		-----	
9090		-----		-----		-----		-----		-----		-----		-----	
9107		-----		-----		-----		-----		-----		-----		-----	
9108		-----		-----		-----		-----		-----		-----		-----	
9117		-----		-----		-----		-----		-----		-----		-----	
9127	D86-A	29.4	ex	37.9	G(1)	54.2	G(1)	115.0	G(1)	236.1	G(5)	266.0	G(1)	320.3	ex
9129		-----		-----		-----		-----		-----		-----		-----	
9130		-----		-----		-----		-----		-----		-----		-----	
9150		-----		-----		-----		-----		-----		-----		-----	
	normality	OK		OK		not OK		OK		OK		OK		OK	
	n	15		15		15		15		13		7		14	
	outliers	0		1		1		1		3		1		1	
	mean (n)	32.45		54.85		65.56		122.78		263.21		301.02		317.48	
	st.dev. (n)	3.479		2.505		2.270		1.462		3.626		4.088		3.844	
	R(calc.)	9.74		7.01		6.36		4.09		10.15		11.45		10.76	
Auto	R(D86:12)	4.98		3.20		3.20		1.88		6.14		6.85		6.78	
Manu	R(D86:12)	5.60		8.86		4.63		6.30		7.69		10.51		7.20	

Determination of Distillation continued on sample #13202; result in %V/V
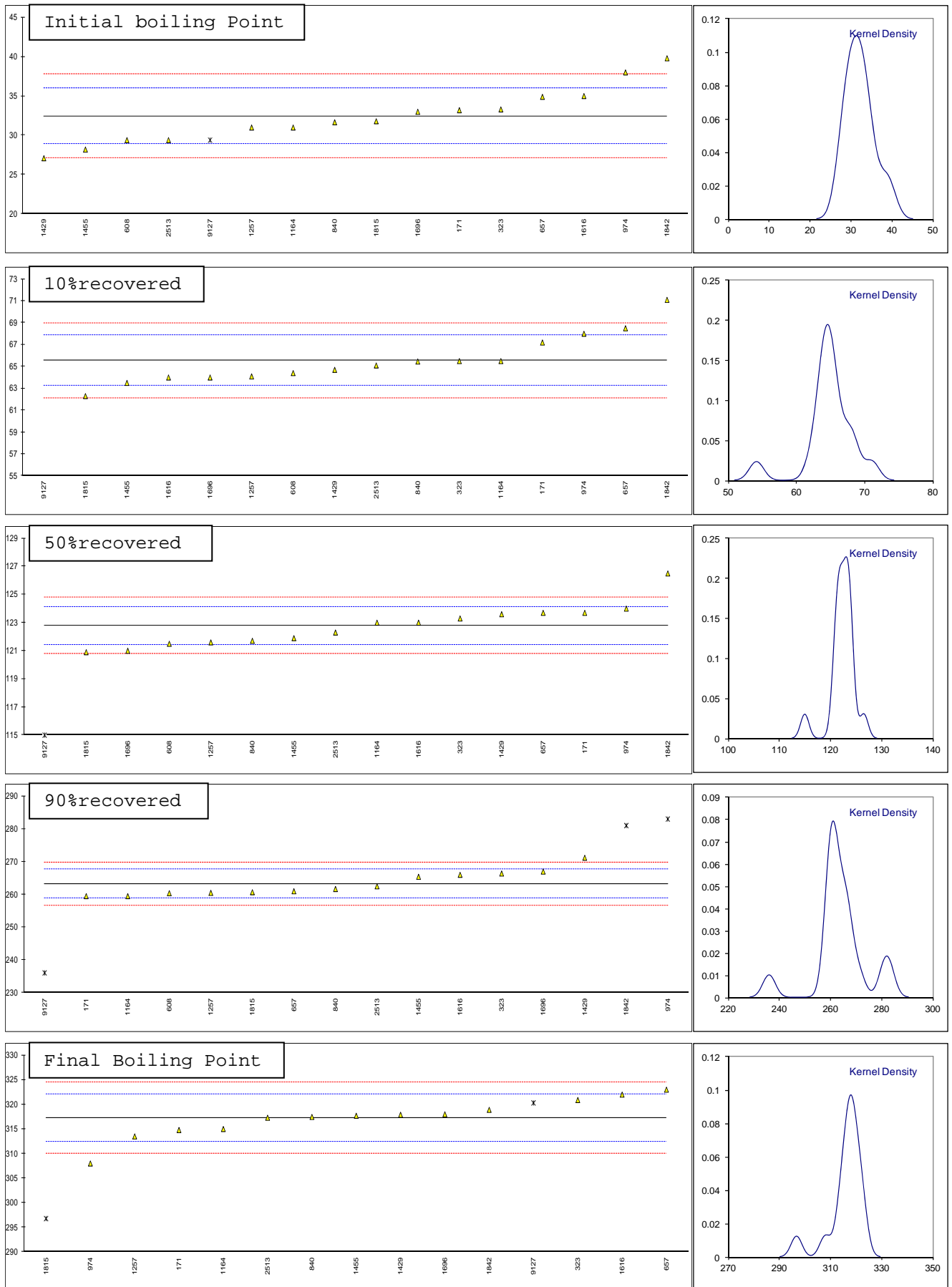
Determination of Mercury as Hg on sample #13202; results in µg/kg

lab	method	value	mark	z(targ)	remarks
171	UOP938	58		-1.98	
311	INH-001	210		4.50	Also reported 220 µg/kg (UOP938) and 290 µg/kg (ASTM D7623)
323		-----		-----	
442		-----		-----	
444	UOP938	85.0		-0.82	
602		-----		-----	
608		-----		-----	
609	UOP938	94.0465		-0.44	
657	UOP938	54		-2.15	
840		-----		-----	
974	UOP938	175		3.01	
1023		-----		-----	
1164		-----		-----	
1214		-----		-----	
1257		-----		-----	
1267		-----		-----	
1397	in house	72.26		-1.37	
1429	in house	139.25		1.49	
1455		-----		-----	
1585		-----		-----	
1616		-----		-----	
1696	D7623	96.97		-0.31	
1800	UOP938	144.46		1.71	
1815		-----		-----	
1842		-----		-----	
1857		-----		-----	
1949		-----		-----	
2124		-----		-----	
2513		-----		-----	
9050		-----		-----	
9054	UOP938	43		-2.61	
9055	INH-245	65.43		-1.66	
9056		-----		-----	
9057	UOP938	181.5		3.29	
9058		-----		-----	
9061	D4928	60		-1.89	
9090		-----		-----	
9107	UOP938	86.0067		-0.78	
9108		-----		-----	
9117		-----		-----	
9127	UOP938	nil		-----	False negative?
9129		-----		-----	
9130		-----		-----	
9150		-----		-----	

normality	not OK			
n	15			
outliers	0			
mean (n)	104.33			
st.dev. (n)	52.619			
R(calc.)	147.33			
R(Horwitz)	65.68		Compare R(UOP938) = 9.80	
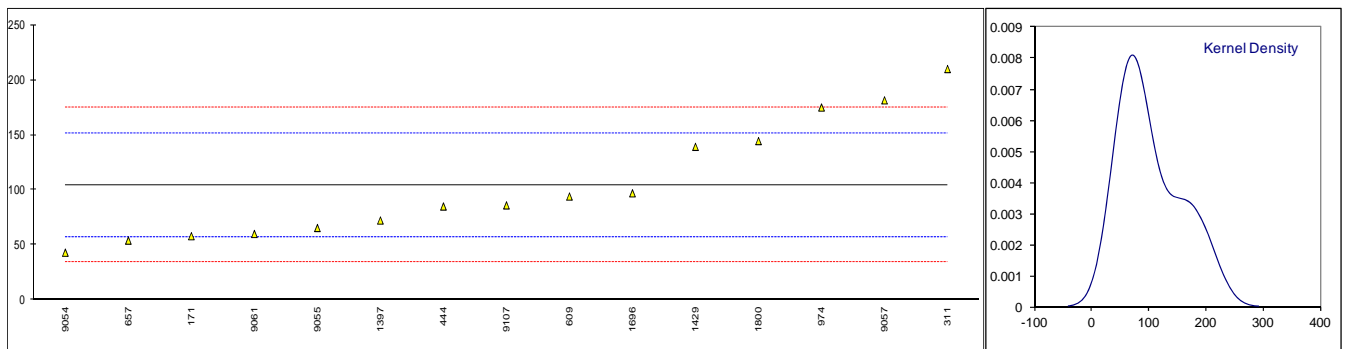
Determination of Sulphur on sample #13202; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D5453	90		1.81	
311	D5453	73		-1.26	
323	D5453	68		-2.16	
442		-----		-----	
444	D5453	91.4		2.06	
602		-----		-----	
608		-----		-----	
609		-----		-----	
657	D5453	73.3		-1.21	
840	D4294	73.3		-1.21	
974	D4294	105		4.52	
1023		-----		-----	
1164	D5453	70.1		-1.78	
1214		-----		-----	
1257	D4294	78		-0.36	
1267		-----		-----	
1397		-----		-----	
1429	D5453	97.39		3.14	
1455	D2622	85.0		0.91	
1585		-----		-----	
1616	D4294	89.0		1.63	
1696	D5453	30.7	C,G(0.01)	-8.90	First reported 57.5
1800		-----		-----	
1815	D5453Mod.	75.3		-0.85	
1842		-----		-----	
1857		-----		-----	
1949		-----		-----	
2124	D5453	81.3		0.24	
2513	D5453	79.6		-0.07	
9050		-----		-----	
9054		-----		-----	
9055		-----		-----	
9056		-----		-----	
9057		-----		-----	
9058		-----		-----	
9061		-----		-----	
9090	D5453	78.2		-0.32	
9107		-----		-----	
9108		-----		-----	
9117		-----		-----	
9127	D5453	67.15		-2.32	
9129		-----		-----	
9130	D5453	73		-1.26	
9150	D5453	71.58		-1.52	

normality	OK
n	19
outliers	1
mean (n)	79.980
st.dev. (n)	10.4359
R(calc.)	29.221
R(D5453:12)	15.504
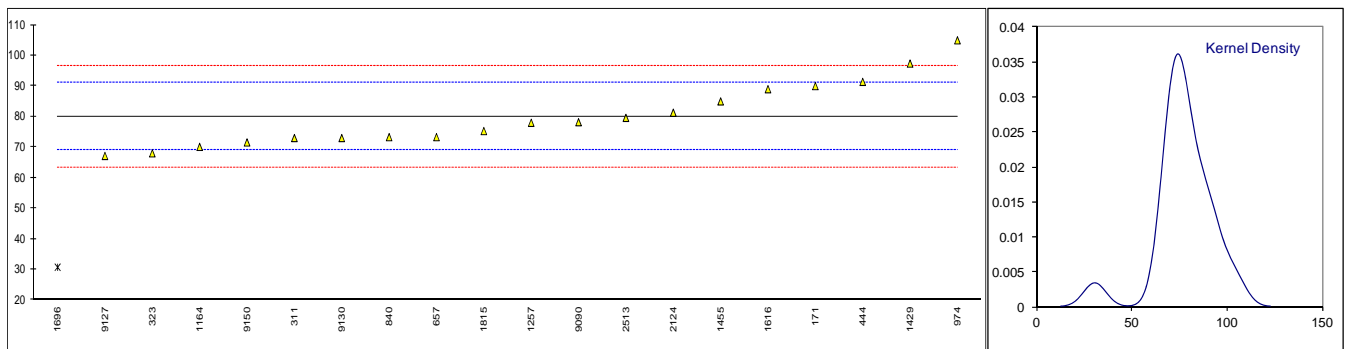
Determination of Water on sample #13202; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D6304	40		0.07	
311	D6304	32		-0.09	
323	D6304	42		0.10	
442	IP438	45		0.16	
444	D6304	29.8	C	-0.13	First reported 11.3
602		-----		-----	
608	D4928	80.1	G(0.01)	0.83	
609	D4928	367.5	G(0.01)	6.34	
657	D6304	69	C,G(0.01)	0.62	First reported 53
840	D6304	32.2		-0.08	
974	D6304	32		-0.09	
1023		-----		-----	
1164	D6304	25.9		-0.20	
1214		-----		-----	
1257	D6304	35		-0.03	
1267	D4928	31.65		-0.09	
1397		-----		-----	
1429	IP438	43.13	C	0.13	First reported 86.26
1455	D4928	<200		-----	Reported <0.02 (probably unit error, %M/M instead of mg/kg)
1585		-----		-----	
1616		-----		-----	
1696	D6304	33.862		-0.05	
1800	D6304	32.4		-0.08	
1815	ISO12937	32.7		-0.07	
1842	IP386	41		0.09	
1857		-----		-----	
1949		-----		-----	
2124	D6304	34.95		-0.03	
2513	D6304	32.7		-0.07	
9050		-----		-----	
9054		-----		-----	
9055		-----		-----	
9056	INH-256	45.305		0.17	
9057	D6304	41.02		0.09	
9058		-----		-----	
9061		-----		-----	
9090	D6304	38.65		0.04	
9107	D6304	54.8		0.35	
9108		-----		-----	
9117		-----		-----	
9127	D6304	33.6		-0.06	
9129		-----	W	-----	Result withdrawn, reported 73.64
9130	D6304	34		-0.05	
9150	D6304	32.76		-0.07	
	normality	not OK			
	n	24			
	outliers	3			
	mean (n)	36.518			
	st.dev. (n)	6.4002			
	R(calc.)	17.920			
	R(D6304:07)	146.277			Compare R(D4928:12) = 24.900
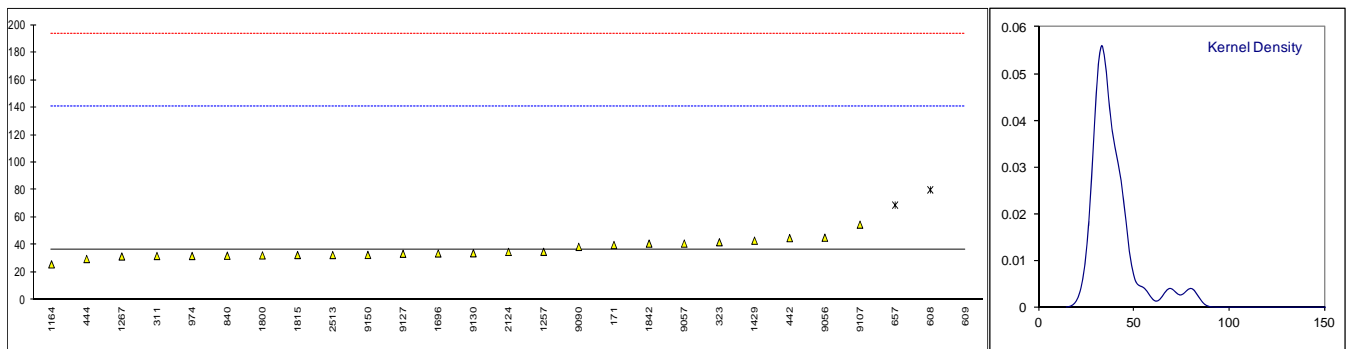
APPENDIX 2:

Z-scores distillation ASTM D86

lab	IBP	5%	10%	50%	90%	95%	FBP
171	0.42	1.27	1.44	1.37	-1.69	-0.91	-1.02
311	-----	-----	-----	-----	-----	-----	-----
323	0.48	0.31	-0.05	0.77	1.45	-----	1.50
442	-----	-----	-----	-----	-----	-----	-----
444	-----	-----	-----	-----	-----	-----	-----
602	-----	-----	-----	-----	-----	-----	-----
608	-1.72	-1.00	-1.01	-1.91	-1.28	-----	-----
609	-----	-----	-----	-----	-----	-----	-----
657	1.38	3.20	2.57	1.37	-1.01	1.50	2.37
840	-0.44	0.22	-0.09	-1.59	-0.70	0.86	0.08
974	3.12	0.13	2.14	1.82	9.03	-----	-3.83
1023	-----	-----	-----	-----	-----	-----	-----
1164	-0.82	-0.30	-0.05	0.33	-1.69	-0.21	-0.94
1214	-----	-----	-----	-----	-----	-----	-----
1257	-0.82	-0.83	-1.28	-1.76	-1.24	0.81	-1.56
1267	-----	-----	-----	-----	-----	-----	-----
1397	-----	-----	-----	-----	-----	-----	-----
1429	-3.01	-1.18	-0.75	1.22	3.64	-----	0.26
1455	-2.39	-2.58	-1.80	-1.31	1.00	-----	0.18
1585	-----	-----	-----	-----	-----	-----	-----
1616	1.43	1.01	-1.36	0.33	1.27	1.22	1.95
1696	0.31	-1.62	-1.36	-2.65	1.73	-3.27	0.30
1800	-----	-----	-----	-----	-----	-----	-----
1815	-0.37	-3.37	-2.85	-2.80	-1.15	-----	-8.45
1842	4.13	5.47	4.85	5.54	8.12	-----	0.67
1857	-----	-----	-----	-----	-----	-----	-----
1949	-----	-----	-----	-----	-----	-----	-----
2124	-----	-----	-----	-----	-----	-----	-----
2513	-1.72	-0.74	-0.40	-0.72	-0.33	-----	0.01
9050	-----	-----	-----	-----	-----	-----	-----
9054	-----	-----	-----	-----	-----	-----	-----
9055	-----	-----	-----	-----	-----	-----	-----
9056	-----	-----	-----	-----	-----	-----	-----
9057	-----	-----	-----	-----	-----	-----	-----
9058	-----	-----	-----	-----	-----	-----	-----
9061	-----	-----	-----	-----	-----	-----	-----
9090	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----
9108	-----	-----	-----	-----	-----	-----	-----
9117	-----	-----	-----	-----	-----	-----	-----
9127	-1.72	-14.83	-9.94	-11.59	-12.37	-14.30	1.25
9129	-----	-----	-----	-----	-----	-----	-----
9130	-----	-----	-----	-----	-----	-----	-----
9150	-----	-----	-----	-----	-----	-----	-----
9130	-----	-----	-----	-----	-----	-----	-----
9132	-----	-----	-----	-----	-----	-----	-----
9141	-----	-----	-----	-----	-----	-----	-----
9144	-----	-----	-----	-----	-----	-----	-----
9145	0.68	-7.04	-7.04	-2.64	-2.34	-2.34	10.10
9150	-----	-----	-----	-----	-----	-----	-----
9153	-----	-----	-----	-----	-----	-----	-----

APPENDIX 3:

Number of participating laboratories per country

    1 laboratory in AUSTRALIA
    1 laboratory in BELGIUM
    1 laboratory in CROATIA
    1 laboratory in INDONESIA
    6 laboratories in MALAYSIA
    1 laboratory in NIGERIA
    3 laboratories in NORWAY
    1 laboratory in OMAN
    1 laboratory in POLAND
    2 laboratories in QATAR
    4 laboratories in RUSSIA
    1 laboratory in SINGAPORE
    4 laboratories in THE NETHERLANDS
    4 laboratories in U.A.E.
    2 laboratories in U.S.A.
   10 laboratories in UNITED KINGDOM
    1 laboratory in VIETNAM

APPENDIX 4

Abbreviations:

C   = final result after checking of first reported suspect 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
ex   = excluded from calculations
E   = error in calculations
n.a.   = not applicable
W   = withdrawn on request participant
U   = reported in deviating unit
SDS   = Safety Data Sheet

Literature:

1   i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
2   ASTM E178-02
3   ASTM E1301-03
4   ISO 5725-86
5   ISO 5725, parts 1-6, 1994
6   ISO 13528-05
7   M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8   W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9   IP367/96
9   DIN 38402 T41/42
10   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
11   J.N. Miller, Analyst, 118, 455, (1993)
12   Analytical Methods Committee Technical Brief, No4 February 2001
13   The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M.
        Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)