

Results of Proficiency Test
Gas condensate
November 2012

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2008, the Institute for Interlaboratory Studies organizes a proficiency test for Gas condensate every year. During the annual proficiency testing program 2012/2013, it was decided to continue the round robin for the analysis of Gas condensate. In this interlaboratory study, 51 laboratories from 18 different countries have participated. See appendix 3 for the number of participating laboratories per country. In this report, the results of the 2012 Gas condensate proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkensisse, 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 2 samples of Gas condensate (1* 0.5 L bottle labelled #12137 and 1* 0.25 L bottle labelled #12138, especially for DVPE purpose). 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 Spijkensisse, the Netherlands, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO17043:2010. 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 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 for the samples #12137 and #12138, approximately 100 litre was obtained from a local supplier. After homogenisation, the Gas condensate was transferred to 88 amber glass bottles of 0.5 litre and to 88 amber glass bottles of 0.25 litre that were labelled #12137 and #12138 respectively.

The homogeneity of the subsamples #12137 was checked by determination of Density @15°C in accordance with ASTM D4052:02e1 on 8 stratified randomly selected samples.

The homogeneity of the subsamples #12138 was checked by determination of DVPE in accordance with ASTM D5191:12 on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/L
Sample #12137-1	0.74558
Sample #12137-2	0.74552
Sample #12137-3	0.74555
Sample #12137-4	0.74552
Sample #12137-5	0.74557
Sample #12137-6	0.74557
Sample #12137-7	0.74552
Sample #12137-8	0.74553

Table 1: homogeneity test results of subsamples #12137

	DVPE in kPa
Sample #12138-1	71.5
Sample #12138-2	71.8
Sample #12138-3	71.9
Sample #12138-4	71.6
Sample #12138-5	71.2
Sample #12138-6	71.6
Sample #12138-7	71.5
Sample #12138-8	71.8

Table 2: homogeneity test results of subsamples #12138

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 °C in kg/L	DVPE in kPa
r sample	0.00007	0.62
reference method	ASTM D4052:02e1	ASTM D5191:10b
0.3xR(reference method)	0.00062	0.83

Table 3: repeatabilities of subsamples #12137 and #12138

The calculated repeatabilities were both less than 0.3 times the reproducibility of the respective reference method. Therefore, homogeneity of the subsamples #12137 and subsamples #12138 was assumed.

To each of the participating laboratories 1 * 0.5 L bottle (labelled #12137) and 1 * 0.25 L bottle (labelled #12138) were sent on October 17, 2012.

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 #12137: Density @ 15°C, total Sulphur, Distillation (IBP, 10%, 50%, 90% evaporated and FBP), Colour Saybolt, Water by KF and Mercury. Sample #12138 was for Total Vapour Pressure and DVPE only.

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:

$$Z_{(\text{target})} = (\text{result} - \text{average of PT}) / \text{target standard deviation}$$

The $Z_{(\text{target})}$ 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$	good
$1 < z < 2$	satisfactory
$2 < z < 3$	questionable
$3 < z $	unsatisfactory

4 EVALUATION

In this proficiency test, some problems were encountered during the transport of the samples to the laboratories in Australia, Malaysia, Mozambique, Negara Brunei Darussalam, Nigeria, Oman and Russia. The samples took an unexpected long time to reach a number of laboratories due to problems with custom clearance.

Only 38 of the 51 (!) participants reported test results. The 38 laboratories reported 234 numerical results. Observed were 25 outlying results, which is 10.7%. 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. All data sets proved to have a normal distribution, except for the determination of Density. In this case the statistical evaluation should be used with care.

Density @15°C: This determination was problematic for a number of laboratories. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D4052:02e1. The current version of this method 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 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 may be problematic. Eleven statistical outliers were observed. After rejection of the statistical outliers none of the calculated reproducibilities are in agreement with the requirements for automated or manual mode of ASTM D86:11b, except for 10% and 50% 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) and conversion and extrapolation up to 560 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 concentration of 163 µg Hg/kg. Two statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers, is not at all in agreement with the estimated reproducibility calculated using the Horwitz equation.

The low number of test results and the variety of test methods used may (partly) explain the large spread. Another (partial) explanation may be that some laboratories may have reported the mercury concentration in µg/L instead of in µg/kg as requested.

Saybolt Color: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D156:12.

Sulphur: This determination was problematic for a number of laboratories. The low sulphur concentration, near or below the lower detection limit of some of the test methods used may explain for the observed problems. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5453:09.

Water: This determination was problematic at this low level (0.0027%M/M). Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outliers is not in agreement with the

requirements of ASTM D4928:11. The use of ASTM D6304 may be more appropriate at this low water level.

TVP & DVPE: This determination may be problematic. In total four statistical outliers and two calculation errors were observed. Seven other results were excluded from statistical evaluation as the used test method is technically not equivalent with ASTM D5191.

Only the calculated reproducibility for TVP is, after rejection of the suspected data, not in agreement with the requirements of ASTM D5191:12. The calculated reproducibility for DVPE is, after rejection of the suspected data, in full agreement with the requirements of ASTM D5191:12.

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 #12137 and #12138, 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)
Density @ 15°C	kg/m ³	34	745.9	2.3	2.0
Initial Boiling Point	°C	14	33.3	7.9	5.0
10%-evaporated	°C	13	75.0	3.6	3.2
50%-evaporated	°C	14	131.4	3.6	1.9
90%-evaporated	°C	14	255.9	11.7	6.0
Final Boiling Point	°C	14	304.4	11.0	6.8
Mercury as Hg	µg/kg	10	560	581	274
Saybolt Color		14	23.4	11.8	2.0
Sulphur	mg/kg	12	13.75	3.40	4.14
Water content by KF	%M/M	26	0.0027	0.0025	0.0021
Total vapour pressure	kPa	10	78.3	3.1	2.8
DVPE acc. to ASTM D5191	kPa	13	71.7	2.7	2.8

Table 4: performance evaluation sample #12137 and #12138

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 2012 WITH THE PREVIOUS PTS

	<i>November 2012</i>	<i>November 2011</i>	<i>November 2010</i>	<i>November 2009</i>
Number of reporting participants	38	41	25	32
Number of results reported	234	283	215	167
Number of statistical outliers	25	29	25	13
Percentage of statistical outliers	10.7%	10.2%	11.6%	7.8%

Table 5: 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	<i>November 2012</i>	<i>November 2011</i>	<i>November 2010</i>	<i>November 2009</i>
Density @ 15 °C	-	++	-	--
Distillation (ASTM D86)	--	--	--	-
Mercury as Hg	--	-	--	++
Saybolt Color	--	--	--	n.e.
Sulphur	++	--	+	--
Water content by KF	-	-	-	--
Total vapour pressure	-	-	--	n.e.
DVPE acc. to ASTM D5191	+/-	+	--	n.e.

Table 6: 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:

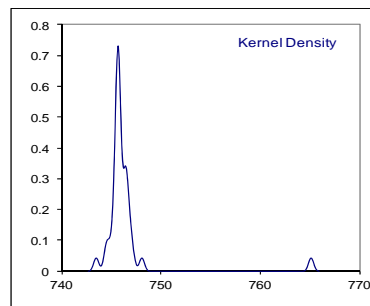
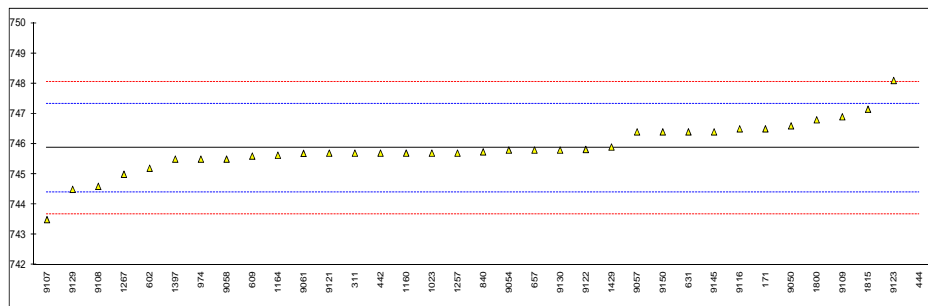
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

APPENDIX 1

Determination of Density @ 15°C on sample #12137; results in kg/m³

lab	method	value	mark	z(targ)	remarks
171	D4052	746.5		0.87	
258		-----		-----	
311	D4052	745.7		-0.23	
442	IP365	745.7		-0.23	
444	D4052	765.15	G(0.01)	26.44	
602	D1298	745.2		-0.91	
608		-----		-----	
609	D4052	745.6		-0.36	
631	D4052	746.4		0.73	
657	D4052	745.8		-0.09	
840	D4052	745.74		-0.17	
974	D4052	745.5		-0.50	
1023	D4052	745.7		-0.23	
1160	D4052	745.7		-0.23	
1164	D4052	745.63		-0.32	
1214		-----		-----	
1257	D4052	745.7		-0.23	
1267	D4052	745		-1.19	
1397	D4052	745.5		-0.50	
1429	D4052	745.9		0.05	
1800	in house	746.8		1.28	
1815	ISO12185	747.15		1.76	
1842		-----		-----	
2124		-----		-----	
9050	INH-21	746.6		1.01	
9054	D4052	745.8		-0.09	
9055		-----		-----	
9056		-----		-----	
9057	D4052	746.4		0.73	
9058	D4052	745.5		-0.50	
9061	D5002	745.69		-0.24	
9101		-----		-----	
9102		-----		-----	
9104		-----		-----	
9107	D4052	743.5		-3.24	
9108	D4052	744.6		-1.74	
9109	D4052	746.9		1.42	
9116	D4052	746.5		0.87	
9117		-----		-----	
9121	D5002	745.7		-0.23	
9122	D4052	745.82		-0.06	
9123	D4052	748.1		3.06	
9126		-----		-----	
9129	in house	744.5		-1.87	
9130	D4052	745.8		-0.09	
9132		-----		-----	
9141		-----		-----	
9144		-----		-----	
9145	D4052	746.4		0.73	
9150	D4052	746.4		0.73	
9153		-----		-----	

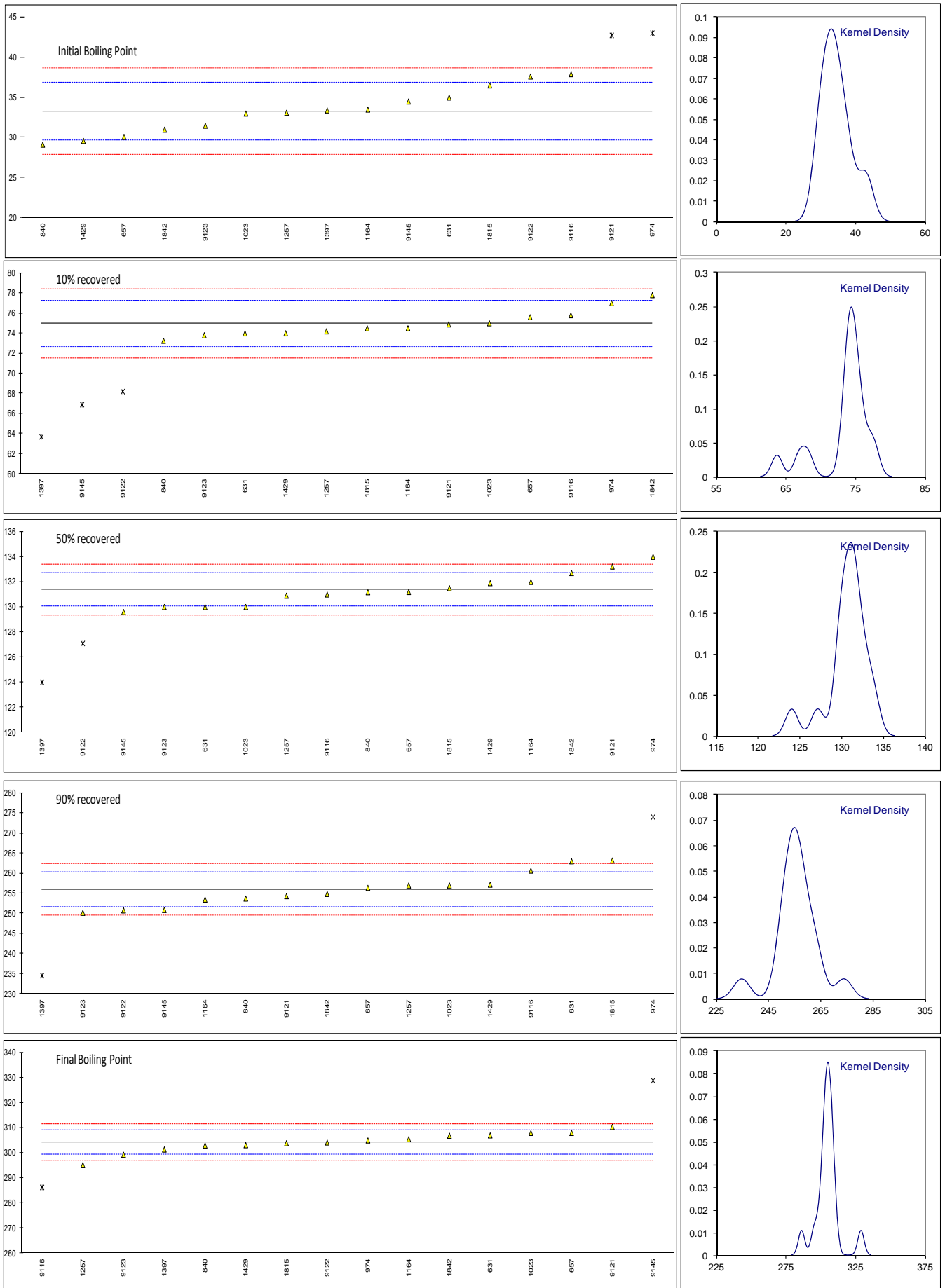
normality not OK
n 34
outliers 1
mean (n) 745.87
st.dev. (n) 0.818
R(calc.) 2.29
R(D4052:02e1) 2.04



Determination of Distillation (automated and manual) on sample #12137; results in °C

lab	method	IBP	mark	10%	mark	50%	mark	90%	mark	FBP	mark	loss %
171		----		----		----		----		----		----
258		----		----		----		----		----		----
311		----		----		----		----		----		----
442		----		----		----		----		----		----
444		----		----		----		----		----		----
602		----		----		----		----		----		----
608		----		----		----		----		----		----
609		----		----		----		----		----		----
631	D86-M	35.0		74.0		130.0		263.0		307.0		4.1
657	D86-A	30.1		75.6		131.2		256.4		308.0		2.8
840	D86-A	29.14		73.26		131.18		253.74		303.01		2.2
974	D86-M	43.0	DG(0.05)	77.0		134.0		274.0	G(0.05)	305.0		6.4
1023	D86-M	33	C	75	C	130		257		308		4
1160		----		----		----		----		----		----
1164	D86-A	33.5		74.5		132.0		253.5		305.5		2.2
1214		----		----		----		----		----		----
1257	D86	33.1		74.2		130.9		257.0		295.2		----
1267		----		----		----		----		----		----
1397	D86	33.4		63.7	DG(0.05)	124.0	G(0.05)	234.6	G(0.01)	301.4		3.7
1429	D86-A	29.6		74.0		131.9		257.2		303.1		5.1
1800		----		----		----		----		----		----
1815	ISO3405-A	36.5		74.5		131.5		263.2		303.9		4.9
1842	D86-A	31.0		77.8		132.7		254.9		306.9		3.5
2124		----		----		----		----		----		----
9050		----		----		----		----		----		----
9054		----		----		----		----		----		----
9055		----		----		----		----		----		----
9056		----		----		----		----		----		----
9057		----		----		----		----		----		----
9058		----		----		----		----		----		----
9061		----		----		----		----		----		----
9101		----		----		----		----		----		----
9102		----		----		----		----		----		----
9104		----		----		----		----		----		----
9107		----		----		----		----		----		----
9108		----		----		----		----		----		----
9109		----		----		----		----		----		----
9116	D86-A	37.9		75.8		131.0		260.7		286.3	G(0.01)	1.0
9117		----		----		----		----		----		----
9121	D86-M	42.72	DG(0.05)	74.89		133.22		254.33		310.39		1.5
9122	D86-M	37.6		68.2	G(0.01)	127.1	G(0.05)	250.8		304.2		2.64
9123	D86-A	31.5		73.8		130.0		250.2		299.3		3.9
9126		----		----		----		----		----		----
9129		----		----		----		----		----		----
9130		----		----		----		----		----		----
9132		----		----		----		----		----		----
9141		----		----		----		----		----		----
9144		----		----		----		----		----		----
9145	D86-M	34.5		66.9	DG(0.05)	129.6		250.9		328.8	G(0.05)	2
9150		----		----		----		----		----		----
9153		----		----		----		----		----		----
	normality	OK		OK		OK		OK		OK		
	n	14		13		14		14		14		
	outliers	2		3		2		2		2		
	mean (n)	33.27		74.95		131.37		255.92		304.35		
	st.dev. (n)	2.831		1.305		1.301		4.192		3.921		
	R(calc.)	7.93		3.65		3.64		11.74		10.98		
Auto	R(D86:11b)	5.03		3.20		1.88		6.00		6.78		
Manu	R(D86:11b)	5.60		5.41		5.94		6.82		7.20		

Lab 1023 first reported; IBP: 24 and 10% rec.: 55

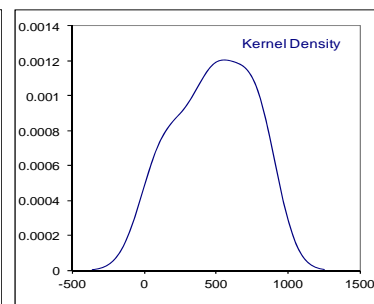
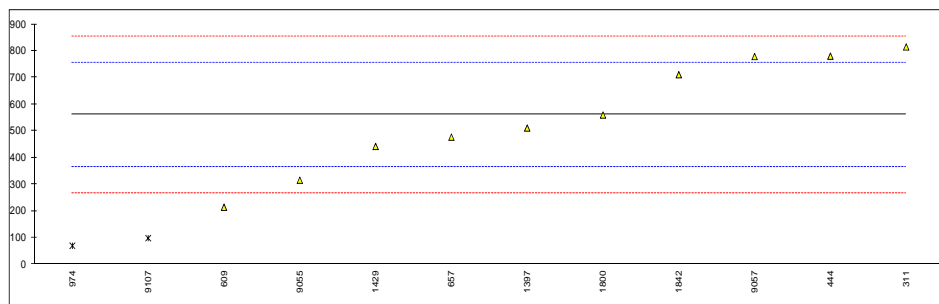


Determination of Mercury as Hg on sample #12137; results in µg/kg

lab	method	value	mark	z(targ)	Remarks
171		----		----	
258		----		----	
311	INH-001	815	C	2.60	First reported 1100
442		----		----	
444	UOP938	780.38		2.25	
602		----		----	
608		----		----	
609	UOP938	214		-3.54	
631		----		----	
657	UOP938	477		-0.85	
840		----		----	
974	UOP938	69.695	G(0.05)	-5.02	
1023		----		----	
1160		----		----	
1164		----		----	
1214		----		----	
1257		----		----	
1267		----		----	
1397	in house	510.97		-0.51	
1429	in house	442		-1.21	
1800	in house	559.81		-0.01	
1815		----		----	
1842	UOP938	711		1.54	
2124		----		----	
9050		----		----	
9054		----		----	
9055	in house	315.5		-2.50	
9056		----		----	
9057	UOP938	779		2.23	
9058		----		----	
9061		----		----	
9101		----		----	
9102		----		----	
9104		----		----	
9107	UOP938	98.1	G(0.05)	-4.73	
9108		----		----	
9109		----		----	
9116		----		----	
9117		----		----	
9121		----		----	
9122		----		----	
9123		----		----	
9126		----		----	
9129		----		----	
9130		----		----	
9132		----		----	
9141		----		----	
9144		----		----	
9145		----		----	
9150		----		----	
9153		----		----	

normality OK
n 10
outliers 2
mean (n) 560.47
st.dev. (n) 207.382
R(calc.) 580.67
R(Horwitz) 273.95

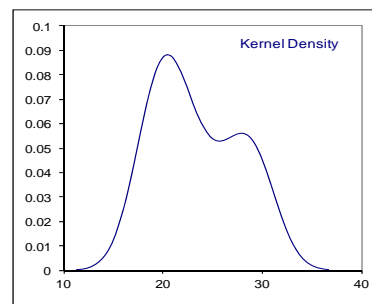
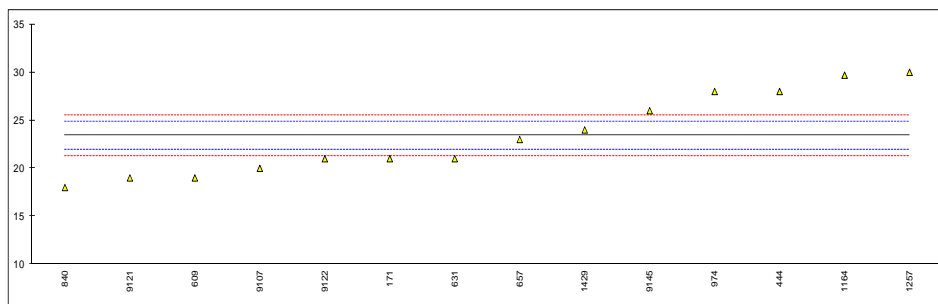
(R(UOP938) = 52.63



Determination of Saybolt Colour on sample #12137;

lab	method	value	mark	z(targ)	remarks
171	D156	21		-3.37	
258		----		----	
311		----		----	
442		----		----	
444	D6045	28		6.43	
602		----		----	
608		----		----	
609	D156	19		-6.17	
631	D156	21		-3.37	
657	D156	23		-0.57	
840	D156	18		-7.57	
974	D156	28		6.43	
1023		----		----	
1160		----		----	
1164	D156	29.7		8.81	
1214		----		----	
1257	D156	30		9.23	
1267		----		----	
1397		----		----	
1429	D6045	24		0.83	
1800		----		----	
1815		----		----	
1842		----		----	
2124		----		----	
9050		----		----	
9054		----		----	
9055		----		----	
9056		----		----	
9057		----		----	
9058		----		----	
9061		----		----	
9101		----		----	
9102		----		----	
9104		----		----	
9107	D156	20		-4.77	
9108		----		----	
9109		----		----	
9116		----		----	
9117		----		----	
9121	D156	19		-6.17	
9122	D156	21		-3.37	
9123		----		----	
9126		----		----	
9129		----		----	
9130		----		----	
9132		----		----	
9141		----		----	
9144		----		----	
9145	D156	26		3.63	
9150		----		----	
9153		----		----	

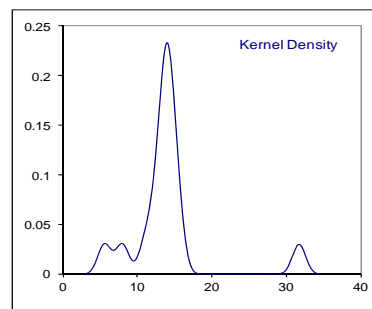
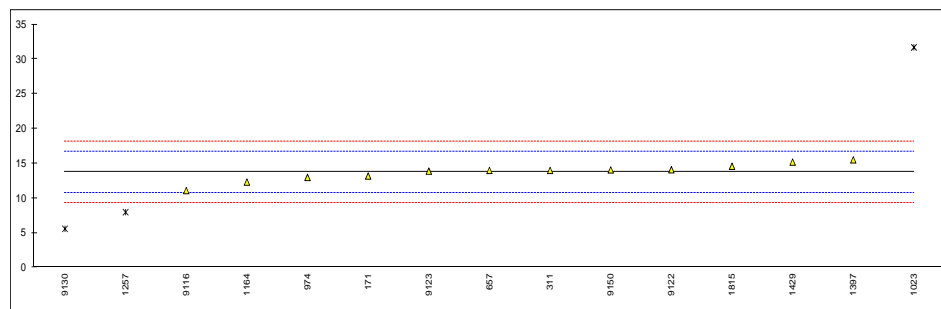
normality OK
n 14
outliers 0
mean (n) 23.41
st.dev. (n) 4.202
R(calc.) 11.77
R(D156:12) 2.00



Determination of Sulphur on sample #12137; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D5453	13.2		-0.37	
258		----		----	
311	D5453	14		0.17	
442		----		----	
444		----		----	
602		----		----	
608		----		----	
609		----		----	
631		----		----	
657	D5453	14		0.17	
840	D4294	<16		----	
974	D4294	13.0		-0.51	
1023	IP336	31.7	C,G(0.01)	12.14	First reported 21.1
1160		----		----	
1164	D5453	12.33	C	-0.96	First reported 9.19 mg/l
1214		----		----	
1257	D4294	8	C,G(0.05)	-3.89	First reported 24
1267		----		----	
1397	D5453	15.52		1.20	
1429	IP490	15.2		0.98	
1800		----		----	
1815	D5453Mod.	14.6		0.57	
1842		----		----	
2124		----		----	
9050		----		----	
9054		----		----	
9055		----		----	
9056		----		----	
9057		----		----	
9058		----		----	
9061		----		----	
9101		----		----	
9102		----		----	
9104		----		----	
9107		----		----	
9108		----		----	
9109		----		----	
9116	D5453	11.1		-1.79	
9117		----		----	
9121		----		----	
9122	D5453	14.1		0.24	
9123	D5453	13.9		0.10	
9126		----		----	
9129		----		----	
9130	D5453	5.6	G(0.05)	-5.51	
9132		----		----	
9141		----		----	
9144		----		----	
9145		----		----	
9150	D5453	14.07		0.22	
9153		----		----	

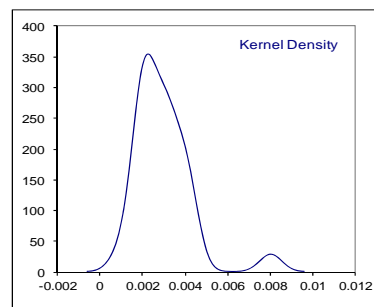
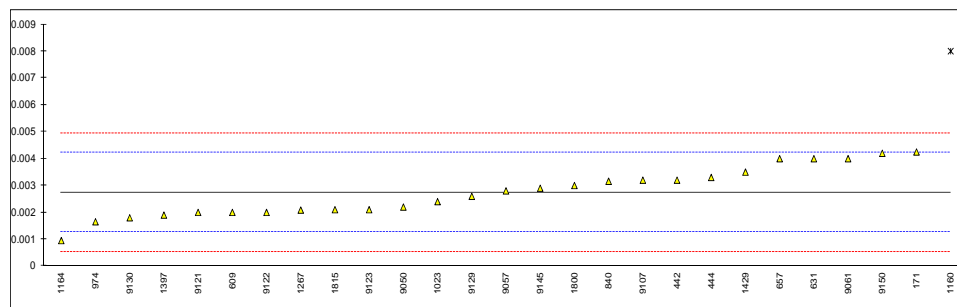
normality OK
n 12
outliers 3
mean (n) 13.752
st.dev. (n) 1.2148
R(calc.) 3.402
R(D5453:09) 4.140



Determination of Water on sample #12137; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D4928	0.00425		2.05	
258		----		----	
311	D4928	<0.02		----	
442	IP438	0.0032		0.62	
444	E203	0.0033		0.76	
602		----		----	
608		----		----	
609	D4928	0.002		-1.01	
631	E203	0.004		1.71	
657	D6304	0.004		1.71	
840	D6304	0.00316		0.57	
974	D6304	0.00165		-1.49	
1023	D4928	0.0024		-0.47	
1160	D4928	0.008	C,G(0.01)	7.16	First reported 0.041
1164	D6305	0.00095		-2.44	
1214		----		----	
1257		----		----	
1267	D4928	0.002087		-0.89	
1397	D6304	0.0019		-1.15	
1429	D4928	0.0035		1.03	
1800	in house	0.003		0.35	
1815	ISO12937	0.0021		-0.87	
1842		----		----	
2124		----		----	
9050	INH-256	0.0022		-0.74	
9054		----		----	
9055		----		----	
9056		----		----	
9057	D4928	0.0028		0.08	
9058		----		----	
9061	D4928	0.004		1.71	
9101		----		----	
9102		----		----	
9104		----		----	
9107	D4928	0.0032		0.62	
9108		----		----	
9109		----		----	
9116	D4928	<0.0100		----	
9117		----		----	
9121	D4928	0.002		-1.01	
9122	D4928	0.002		-1.01	
9123	D4928	0.0021		-0.87	
9126		----		----	
9129	in house	0.0026		-0.19	
9130	D6304	0.0018		-1.28	
9132		----		----	
9141		----		----	
9144		----		----	
9145	D4928	0.0029		0.21	
9150	D6304	0.0042		1.98	
9153		----		----	

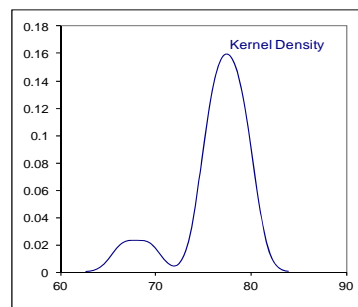
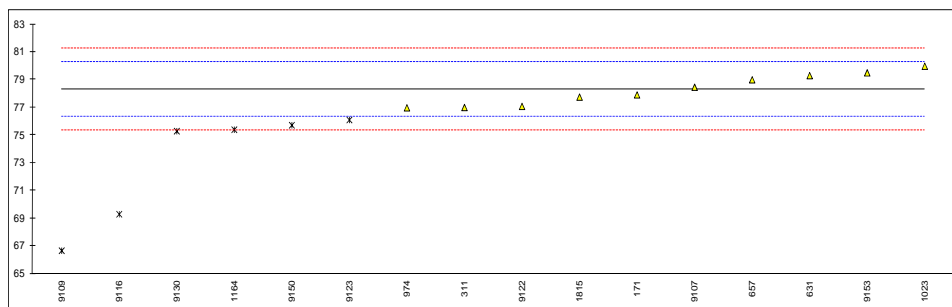
normality OK
n 26
outliers 1
mean (n) 0.00274
st.dev. (n) 0.000896
R(calc.) 0.00251
R(D4928:11) 0.00206



Determination of Total Vapour Pressure (TVP) on sample #12138; results in kPa

lab	method	value	mark	z(targ)	remarks
171	D5191	77.91		-0.39	
258		----		----	
311	D5191	77.0		-1.32	
442		----		----	
444		----		----	
602		----		----	
608		----		----	
609		----		----	
631	D5191	79.3		1.02	
657	D5191	79.0		0.72	
840		----		----	
974	D5191	76.979		-1.34	
1023	D5191	79.98		1.71	
1160		----		----	
1164	D6378	75.4	ex	-2.95	Result excluded, test method is not equivalent with D5191
1214		----		----	
1257		----		----	
1267		----		----	
1397		----		----	
1429		----		----	
1800		----		----	
1815	EN13016	77.75		-0.56	
1842		----		----	
2124		----		----	
9050		----		----	
9054		----		----	
9055		----		----	
9056		----		----	
9057		----		----	
9058		----		----	
9061		----		----	
9101		----		----	
9102		----		----	
9104		----		----	
9107	D5191	78.4623		0.17	
9108		----		----	
9109	D5191	66.67	G(0.05)	-11.84	
9116	D5191	69.3	G(0.01)	-9.16	
9117		----		----	
9121		----		----	
9122	D5191	77.08		-1.24	
9123	D5191	76.1	ex	-2.24	Result excluded, calculated result from DVPE is 76.87
9126		----		----	
9129		----		----	
9130	D6378	75.29	ex	-3.06	Result excluded, test method is not equivalent with D5191
9132		----		----	
9141		----		----	
9144		----		----	
9145		----		----	
9150	D6378	75.72	ex	-2.62	Result excluded, test method is not equivalent with D5191
9153	D5191	79.50		1.23	

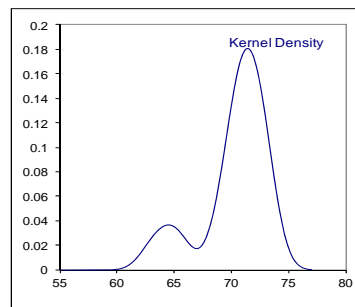
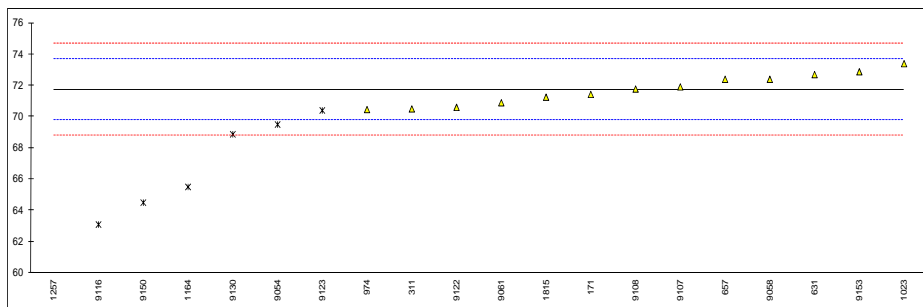
normality OK
n 10
outliers 2
mean (n) 78.269
st.dev. (n) 1.1134
R(calc.) 3.118
R(D5191:12) 2.750



Determination of DVPE (ASTM D5191 calculation) on sample #12138; results in kPa

lab	method	value	mark	z(targ)	remarks
171	D5191	71.43		-0.31	
258		----		----	
311	D5191	70.5		-1.26	
442		----		----	
444		----		----	
602		----		----	
608		----		----	
609		----		----	
631	D5191	72.7		0.98	
657	D5191	72.4		0.67	
840		----		----	
974	D5191	70.46		-1.30	
1023	D5191	73.40		1.69	
1160		----		----	
1164	D6378	65.5	ex	-6.35	Result excluded, test method is not equivalent with D5191
1214		----		----	
1257	D5191	12.937	C,G(0.01)	-59.87	First reported 10.46
1267		----		----	
1397		----		----	
1429		----		----	
1800		----		----	
1815	EN13016	71.25		-0.50	
1842		----		----	
2124		----		----	
9050		----		----	
9054	D323	69.5016	ex	-2.28	Result excluded, test method is not equivalent with D5191
9055		----		----	
9056		----		----	
9057		----		----	
9058	D5191	72.4		0.67	
9061	D5191	70.895		-0.86	
9101		----		----	
9102		----		----	
9104		----		----	
9107	D5191	71.9123		0.18	
9108	D5191	71.774		0.04	
9109		----		----	
9116	D5191	63.09	G(0.05)	-8.81	
9117		----		----	
9121		----		----	
9122	D5191	70.60		-1.16	
9123	D5191	70.4	ex	-1.36	Result excluded, calculated result from TVP is 76.87
9126		----		----	
9129		----		----	
9130	D5191	68.87	ex	-2.92	TVP test method is not equivalent with D5191
9132		----		----	
9141		----		----	
9144		----		----	
9145		----		----	
9150	D6379	64.49	ex	-7.38	Result excluded, test method is not equivalent with D5191
9153	D5191	72.88		1.16	

normality OK
n 13
outliers 2
mean (n) 71.739
st.dev. (n) 0.9749
R(calc.) 2.730
R(D5191:12) 2.750



APPENDIX 2:

Z-scores distillation ASTM D86

lab	IBP	10%	50%	90%	FBP
171	----	----	----	----	----
258	----	----	----	----	----
311	----	----	----	----	----
442	----	----	----	----	----
444	----	----	----	----	----
602	----	----	----	----	----
608	----	----	----	----	----
609	----	----	----	----	----
631	0.96	-0.83	-2.04	3.31	1.09
657	-1.77	0.57	-0.26	0.22	1.51
840	-2.30	-1.48	-0.29	-1.02	-0.55
974	5.42	1.79	3.91	8.44	0.27
1023	-0.15	0.04	-2.04	0.50	1.51
1160	----	----	----	----	----
1164	0.13	-0.39	0.94	-1.13	0.47
1214	----	----	----	----	----
1257	-0.10	-0.66	-0.70	0.50	-3.78
1267	----	----	----	----	----
1397	0.07	-9.84	-10.98	-9.95	-1.22
1429	-2.05	-0.83	0.79	0.60	-0.52
1800	----	----	----	----	----
1815	1.80	-0.39	0.19	3.40	-0.19
1842	-1.27	2.49	1.98	-0.48	1.05
2124	----	----	----	----	----
9050	----	----	----	----	----
9054	----	----	----	----	----
9055	----	----	----	----	----
9056	----	----	----	----	----
9057	----	----	----	----	----
9058	----	----	----	----	----
9061	----	----	----	----	----
9101	----	----	----	----	----
9102	----	----	----	----	----
9104	----	----	----	----	----
9107	----	----	----	----	----
9108	----	----	----	----	----
9109	----	----	----	----	----
9116	2.58	0.74	-0.55	2.23	-7.45
9117	----	----	----	----	----
9121	5.26	-0.05	2.75	-0.74	2.49
9122	2.41	-5.91	-6.36	-2.39	-0.06
9123	-0.99	-1.01	-2.04	-2.67	-2.09
9126	----	----	----	----	----
9129	----	----	----	----	----
9130	----	----	----	----	----
9132	----	----	----	----	----
9141	----	----	----	----	----
9144	----	----	----	----	----
9145	0.68	-7.04	-2.64	-2.34	10.10
9150	----	----	----	----	----
9153	----	----	----	----	----

APPENDIX 3:

Number of participating laboratories per country

- 1 laboratory in AUSTRALIA
- 3 laboratories in CANADA
- 1 laboratory in CROATIA
- 6 laboratories in MALAYSIA
- 1 laboratory in MOZAMBIQUE
- 1 laboratory in NEGARA BRUNEI DARUSSALAM
- 3 laboratories in NIGERIA
- 3 laboratories in NORWAY
- 2 laboratories in OMAN
- 2 laboratories in PHILIPPINES
- 1 laboratory in POLAND
- 1 laboratory in RUSSIA
- 1 laboratory in SINGAPORE
- 3 laboratories in THE NETHERLANDS
- 4 laboratories in U.A.E.
- 5 laboratories in U.S.A.
- 12 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/>)