Results of Proficiency Test
Gascondensate
November 2011

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 2008, the Institute for Interlaboratory Studies organizes a proficiency test for Gascondensate every year. During the annual proficiency testing program 2011/2012, it was decided to continue the round robin for the analysis of Gascondensate. In this interlaboratory study, 54 laboratories from 20 different countries have participated. See appendix 3 for the number of participating laboratories per country. In this report, the results of the Gascondensate proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (i.i.s.) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. It was decided to send 2 samples of Gascondensate (1* 0.5 L bottle labelled #11100 and 1* 0.25 L bottle labelled #11101, 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 Spijkenisse, 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% 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 present 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 #11100 and #11101, approximately 100 litre was obtained from a local refinery. After homogenisation, the Gascondensate was transferred to 105 amber glass bottles of 0.5 litre and to 110 amber glass bottles of 0.25 litre that were labelled #11100 and #11101 respectively.

The homogeneity of the subsamples #11100 was checked by determination of Density @ 15°C in accordance with ASTM D4052:09 on 8 stratified randomly selected samples. The homogeneity of the subsamples #11101 was checked by determination of DVPE in accordance with ASTM D5191:10b on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/L
Sample #11100-1	0.74332
Sample #11100-2	0.74330
Sample #11100-3	0.74333
Sample #11100-4	0.74338
Sample #11100-5	0.74341
Sample #11100-6	0.74349
Sample #11100-7	0.74347
Sample #11100-8	0.74339

Table 1: homogeneity test results of subsamples #11100

	DVPE in kPa
Sample #11101-1	67.3
Sample #11101-2	67.8
Sample #11101-3	68.0
Sample #11101-4	67.9
Sample #11101-5	67.9
Sample #11101-6	67.9
Sample #11101-7	67.3
Sample #11101-8	67.1

Table 2: homogeneity test results of subsamples #11101

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	0.00017	1.0
reference method	ASTM D4052:09	ASTM D5191:10b
0.3xR(reference method)	0.00064	0.7
r (reference method)	0.00010	1.4

Table 3: repeatabilities of subsamples #11100 and #11101

The calculated repeatabilities were less than 0.3 times the reproducibility or less than the repeatability of the respective reference method. Therefore, homogeneity of the subsamples #11100 and subsamples #11101 was assumed.

To each of the participating laboratories 1 * 0.5 L bottle (labelled #11100) and 1 * 0.25 L bottle (labelled #11101) were sent on October 19, 2011.

2.5 STABILITY OF THE SAMPLES

The stability of Gascondensate, 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 #11100: Density @ 15°C, total Sulphur, Distillation (IBP, 10%, 50%, 90% evaporated and FBP), Colour Saybolt, Water by KF and Mercury. Sample #11101 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, was sent together with each set of samples. In addition, a letter of instructions and a SDS were added to the 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; nr.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	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 Malaysia, Mozambique, Nigeria, Norway, Oman, Philippines, Qatar and U.A.E. The samples took an unexpected long time to reach a number of laboratories due to problems with custom clearance.
Only 41 (!) participants reported test results. The 41 laboratories reported 283 numerical results. Observed were 29 outlying results, which is 10.2%. 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 Colour Saybolt. In this case the statistical evaluation should be used with care.

Density @15°C: This determination was problematic for a number of laboratories. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D4052:09.

Distillation: This determination may be problematic. Nine statistical outliers were observed. Three of the five reported test results are outliers for laboratory 9100 and 9109. As the five test results are not independent, it was decided not to use the other two reported test results of both laboratories for the statistical evaluation.
After rejection of the statistical outliers and the excluded data, none of the calculated reproducibilities is in agreement with the requirements for automated or manual mode of ASTM D86:11a, except for 50% recovered for manual mode. However, as the scope of ASTM D86 does not include gascondensates, but only products with a limited boiling range like distillate fuels, the target reproducibilities as used in this report may not be applicable.

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 upto 160 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.
Thus, 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 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 in stead of in the requested mg/kg.

Saybolt Color: This determination was very problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D156:07a.

Sulphur: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D5453:09. When the ASTM D5453 data was evaluated separately, the calculated reproducibility is smaller but still not at all in agreement with the requirements of the standard.

Water: This determination was problematic. Five statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D4928:11.

TVP & DVPE: This determination may be problematic. In total five statistical outliers and two calculation errors were observed. The calculated reproducibility for TVP is, after rejection of the statistical outliers, not in agreement with the requirements of ASTM D5191:10b. However, for DVPE calculated reproducibility, after exclusion of the calculations errors and two statistical outliers, is in good agreement.

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 #11100 and #11101, 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	743.9	1.4	2.1
Initial Boiling Point	°C	20	32.8	7.9	5.0 / 5.6
10%-recovered	°C	22	64.6	5.6	3.2 / 5.1
50%-recovered	°C	22	122.0	4.8	1.9 / 6.3
90%-recovered	°C	20	263.9	19.1	6.2 / 7.5
Final Boiling Point	°C	21	315.6	20.9	6.8 / 7.2
Mercury as Hg	µg/kg	11	163.1	129.4	96.0
Saybolt Color		21	16.3	4.7	2.0
Sulphur	mg/kg	21	75.7	32.2	14.9
Water content by KF	%M/M	26	0.0041	0.0035	0.0027
Total vapour pressure	kPa	14	73.2	3.2	2.8
DVPE acc. to ASTM D5191	kPa	15	67.2	2.2	2.8

Table 4: performance evaluation sample #11100 and #11101

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 2011 WITH THE PREVIOUS PTS

	November 2011	*November 2010*	*November 2009*	*November 2008*
Number of reporting participants	41	25	32	17
Number of results reported	283	215	167	121
Number of statistical outliers	29	25	13	7
Percentage of statistical outliers	10.2%	11.6%	7.8%	5.8%

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	*November 2011*	*November 2010*	*November 2009*	*November 2008*
Density @ 15 ^{o}C	++	-	--	-
Distillation (ASTM D86)	--	--	-	+/-
Mercury as Hg	-	--	++	+
Saybolt Color	--	--	n.e.	n.e.
Sulphur	--	+	--	--
Water content by KF	-	-	--	+
Total vapour pressure	-	--	n.e.	n.e.
DVPE acc. to ASTM D5191	+	--	n.e.	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:

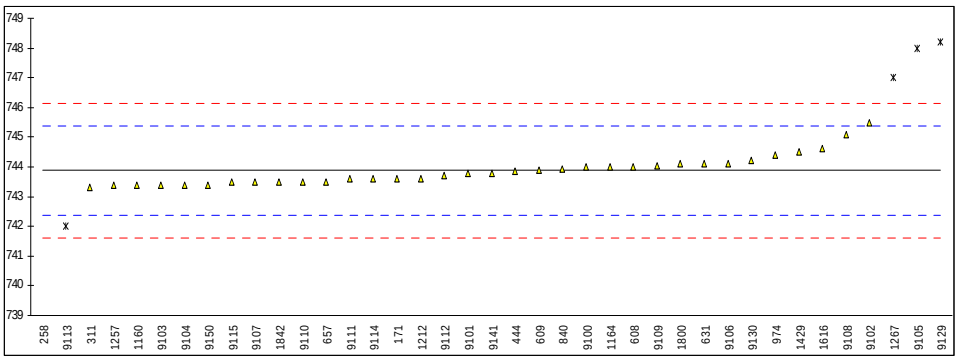
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

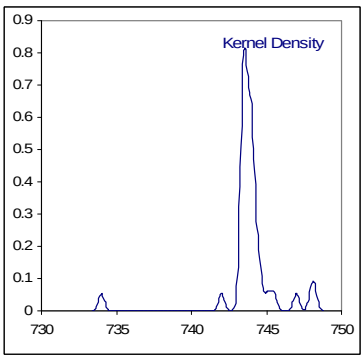
APPENDIX 1

Determination of Density @ 15°C on sample #11100; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
171	D4052	743.6		-0.37	
258	D1298	734.0	G(0.01)	-13.10	
311	D4052	743.3		-0.76	
442		-----		-----	
444	D4052	743.84		-0.05	
449		-----		-----	
602		-----		-----	
608	D4052	744.0		0.16	
609	D4052	743.9	U	0.03	Reported 0.7439 (probably an unit error)
631	D4052	744.1		0.30	
657	D4052	743.5		-0.50	
840	D4052	743.92		0.06	
974	D4052	744.4		0.70	
1023		-----		-----	
1160	D4052	743.4	C	-0.63	First reported 0.7434 (unit error)
1164	D4052	744.00		0.16	
1212	D4052	743.60		-0.37	
1214		-----		-----	
1257	D4052	743.4		-0.63	
1267	D4052	747	G(0.01)	4.14	
1429	D4052	744.5		0.83	
1616	D4052	744.6		0.96	
1800	D4052	744.1		0.30	
1815		-----		-----	
1842	D4052	743.5		-0.50	
1929		-----		-----	
9100	D4052	744.0		0.16	
9101	D5002	743.8		-0.10	
9102	In house	745.5		2.15	
9103	In house	743.4		-0.63	
9104	D4052	743.4		-0.63	
9105	D4052	748.0	G(0.01)	5.47	
9106	D5002	744.1		0.30	
9107	D5002	743.5		-0.50	
9108	D4052	745.1		1.62	
9109	D4052	744.0		0.19	
9110	D4052	743.5		-0.50	
9111	D4052	743.6		-0.37	
9112	D4052	743.7		-0.23	
9113	D4052	742.0	G(0.05)	-2.49	
9114	D4052	743.6		-0.37	
9115	D4052	743.5		-0.50	
9129	D4052	748.2	G(0.05)	5.74	
9130	D4052	744.2		0.43	
9141	D4052	743.8		-0.10	
9150	D4052	743.4		-0.63	

normality	OK
n	34
outliers	5
mean (n)	743.88
st.dev. (n)	0.498
R(calc.)	1.39
R(D4052:09)	2.11

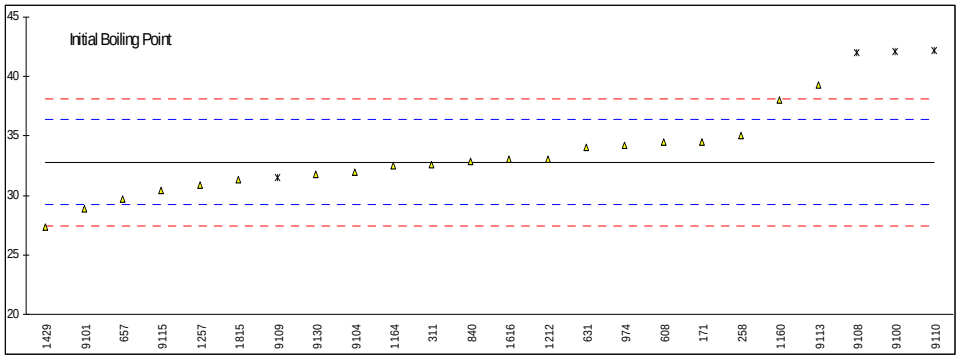




Determination of Distillation on sample #11100; results in °C

lab	method	IBP	mark	10%	mark	50%	mark	90%	mark	FBP	mark	remarks
171	D86-A	34.5		68.5		124.0		265.6		326.7		
258	D86-A	35.0		65.8		122.6		267.2		308.5		
311	D86-A	32.6		65.3		121.2		260.6		307.3		
442		-----		-----		-----		-----		-----		
444		-----		-----		-----		-----		-----		
449		-----		-----		-----		-----		-----		
602		-----		-----		-----		-----		-----		
608	D86-M	34.5		61.0		124.0		271.0		314.5		
609		-----		-----		-----		-----		-----		
631	D86	34.0		63.0		119.5		264.0		-----		
657	D86-A	29.7		66.3		125.0		262.0		322.8		
840	D86-A	32.84		60.16		119.85		254.07		317.36		
974	D86-A	34.2		65.0		122.7		273.9		312.1		
1023		-----		-----		-----		-----		-----		
1160	D86-M	38.0		67.5		124.5		-----	W	305.0		
1164	D86-A	32.5		66.0		122.0		259.0		313.5		
1212	D86-A	33.0		62.0		119.7		261.1		319.3		
1214		-----		-----		-----		-----		-----		
1257	D86-A	30.9		64.4		121.5		261.5		307.6		
1267		-----		-----		-----		-----		-----		
1429	D86	27.3		64.4		121.5		261.4		318.0		
1616	D86-M	33.0		66.0		122.0		257.0		312.0		
1800		-----		-----		-----		-----		-----		
1815	ISO3405-A	31.35		63.30		121.30		265.10		302.65		
1842		-----		-----		-----		-----		-----		
1929		-----		-----		-----		-----		-----		
9100	D86	42.1	G(0.01)	69.4	ex	127.0	ex	291.6	G(0.05)	356.2	G(0.01)	
9101	D86	28.9		63.2		121.0		267.3		317.5		
9102		-----		-----		-----		-----		-----		
9103		-----		-----		-----		-----		-----		
9104	D86	32.0		65.0		123.0		274.0		316.0		
9105		-----		-----		-----		-----		-----		
9106		-----		-----		-----		-----		-----		
9107		-----		-----		-----		-----		-----		
9108	D86	42.0	G(0.01)	63.0		125.0		280.5		318.5		
9109	D86	31.5	ex	56.0	G(0.05)	116.0	G(0.01)	242.0	G(0.05)	301.5	ex	
9110	D86	42.2	G(0.01)	65.6		122.2		260.6		335.6		
9111		-----		-----		-----		-----		-----		
9112		-----		-----		-----		-----		-----		
9113	D86	39.3		65.5		119.6		245.7	G(0.05)	318.3		
9114		-----		-----		-----		-----		-----		
9115	D86	30.4		65.6		121.3		257.3		318.0		
9129		-----		-----		-----		-----		-----		
9130	D86	31.8		65.0		120.8		255.4		317.0		
9141		-----		-----		-----		-----		-----		
9150		-----		-----		-----		-----		-----		
	normality	OK		OK		OK		OK		OK		
	n	20		22		22		20		21		
	outliers	3		1		1		3		1		
	mean (n)	32.79		64.62		122.01		263.93		315.63		
	st.dev. (n)	2.826		1.999		1.699		6.821		7.480		
	R(calc.)	7.91		5.60		4.76		19.10		20.94		
	R(D86:11a-A)	5.00		3.20		1.88		6.15		6.78		
	R(D86:11a-M)	5.60		5.10		6.34		7.54		7.20		

ex = result excluded, see paragraph 4.1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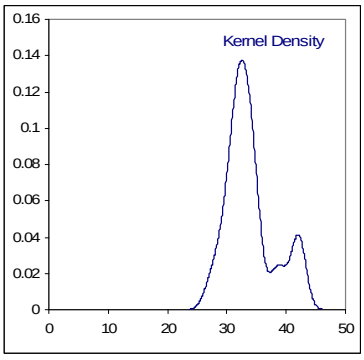
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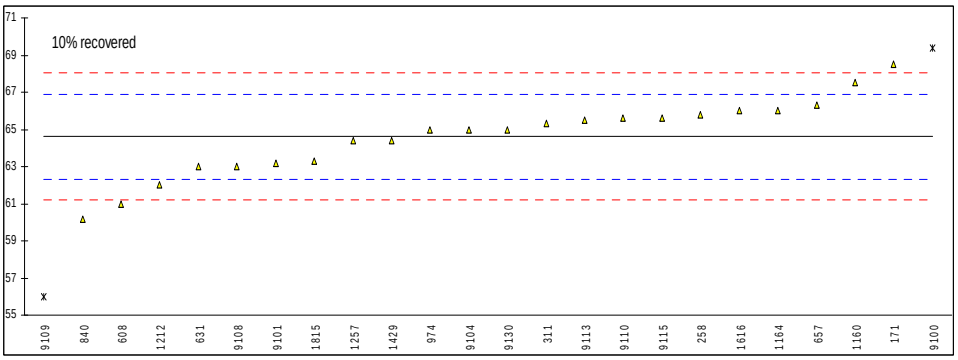
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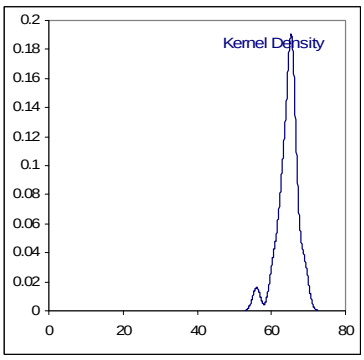
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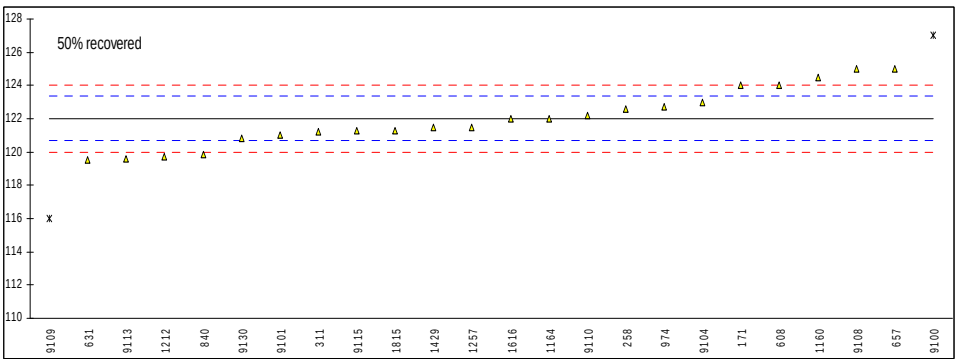
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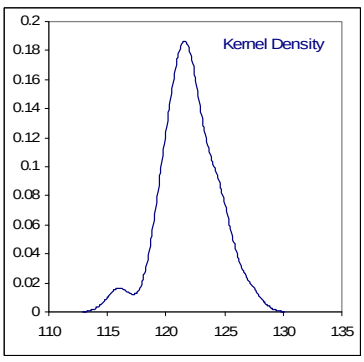
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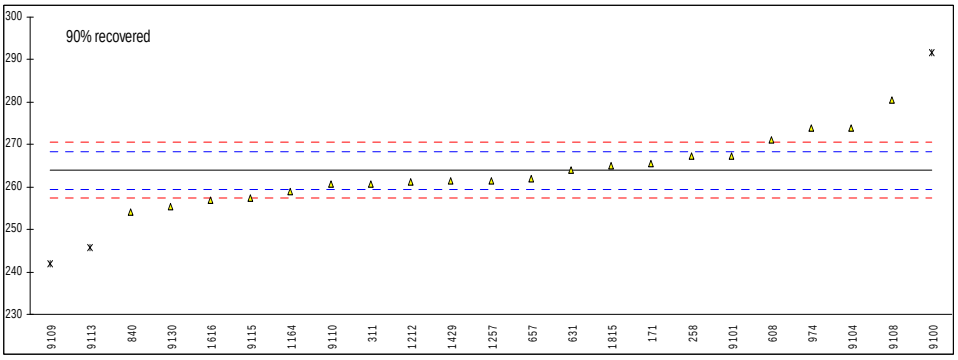
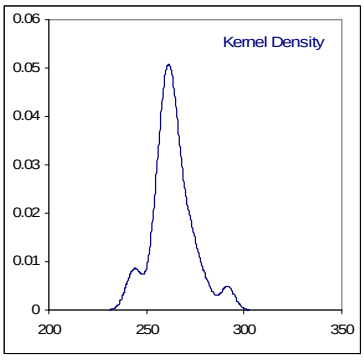
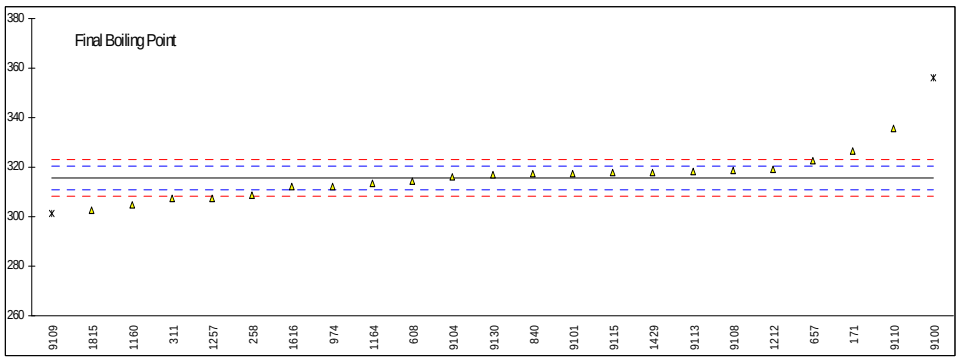
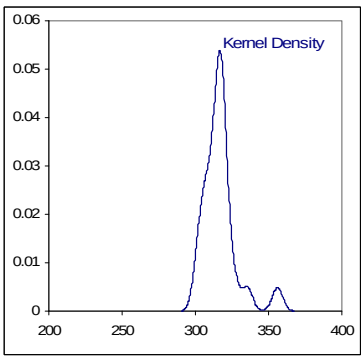

90% recovered
9109 9113 840 9130 1616 9115 1164 9110 311 1212 1429 1257 657 631 1815 171 258 9101 608 974 9104 9108 9100

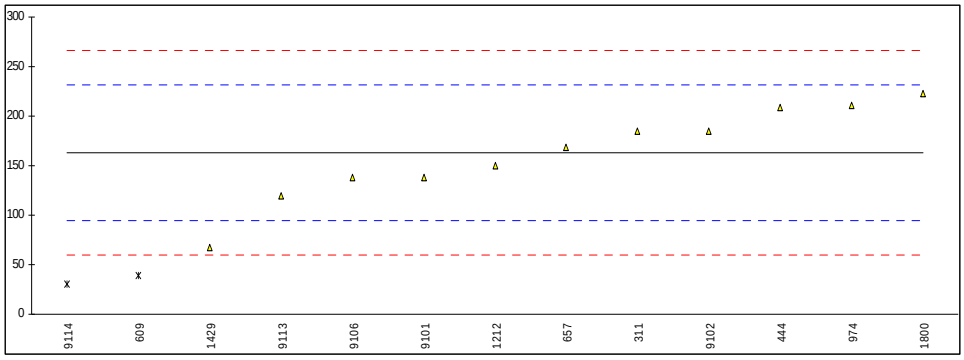

Kernel Density

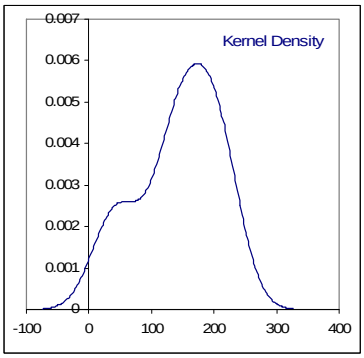

Final Boiling Point
9109 1815 1160 311 1257 258 1616 974 1164 608 9104 9130 840 9101 9115 1429 9113 9108 1212 657 171 9110 9100


Kernel Density

Determination of Mercury as Hg on sample #11100; results in µg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
258		-----		-----	
311	INH-001	185		0.64	
442		-----		-----	
444	UOP938	208.4		1.32	
449		-----		-----	
602		-----		-----	
608		-----		-----	
609	UOP938	39.1465	G(0.05)	-3.62	
631		-----		-----	
657	UOP938	168.8		0.17	
840		-----		-----	
974	UOP938	210.69		1.39	
1023		-----		-----	
1160		-----		-----	
1164		-----		-----	
1212	INH-80	150		-0.38	
1214		-----		-----	
1257		-----		-----	
1267		-----		-----	
1429	in house	67.84		-2.78	
1616		-----		-----	
1800	D7623Mod.	222.9706		1.75	
1815		-----		-----	
1842		-----		-----	
1929		-----		-----	
9100		-----		-----	
9101	in house	138		-0.73	
9102	in house	185		0.65	
9103		-----		-----	
9104		-----		-----	
9105		-----		-----	
9106	in house	138		-0.73	
9107		-----		-----	
9108		-----		-----	
9109		-----		-----	
9110		-----		-----	
9111		-----		-----	
9112		-----		-----	
9113	UOP938	119.097		-1.28	
9114	UOP938	30.02	G(0.05)	-3.88	
9115		-----		-----	
9129		-----		-----	
9130		-----		-----	
9141		-----		-----	
9150		-----		-----	
					UOP938 data only:
	normality	OK			n.a.
	n	11			4
	outliers	2			2
	mean (n)	163.10			176.75
	st.dev. (n)	46.205			42.976
	R(calc.)	129.37			120.33
	R(Horwitz)	96.00	R(UOP938) = 15.32		102.78 compare R(UOP938) = 16.60

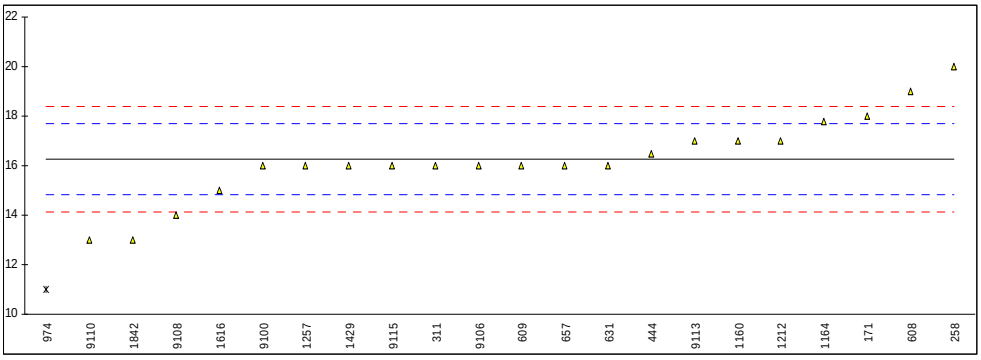


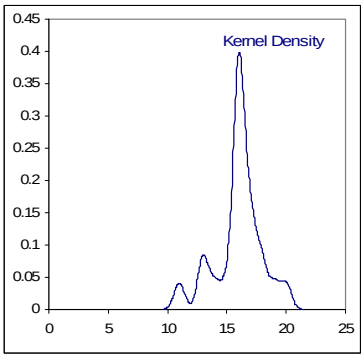


Determination of Saybolt Colour on sample #11100;

lab	method	value	mark	z(targ)	remarks
171	D156	18		2.45	
258	D156	20		5.25	
311	D5386	16		-0.35	
442		-----		-----	
444	D6045	16.5		0.35	
449		-----		-----	
602		-----		-----	
608	D156	19		3.85	
609	D156	16		-0.35	
631	D156	16		-0.35	
657	D156	16		-0.35	
840		-----		-----	
974	D156	11	G(0.05)	-7.35	
1023		-----		-----	
1160	D156	17		1.05	
1164	D156	17.8		2.17	
1212	D156	17		1.05	
1214		-----		-----	
1257	D156	16		-0.35	
1267		-----		-----	
1429	D6045	16		-0.35	
1616	D6045	15		-1.75	
1800		-----		-----	
1815		-----		-----	
1842	D156	13		-4.55	
1929		-----		-----	
9100	D6045	16		-0.35	
9101		-----		-----	
9102		-----		-----	
9103		-----		-----	
9104		-----		-----	
9105		-----		-----	
9106	D6045	16		-0.35	
9107		-----		-----	
9108	D156	14		-3.15	
9109		-----		-----	
9110	D156	13		-4.55	
9111		-----		-----	
9112		-----		-----	
9113	D156	17		1.05	
9114		-----		-----	
9115	D156	16		-0.35	
9129		-----		-----	
9130		-----		-----	
9141		-----		-----	
9150		-----		-----	

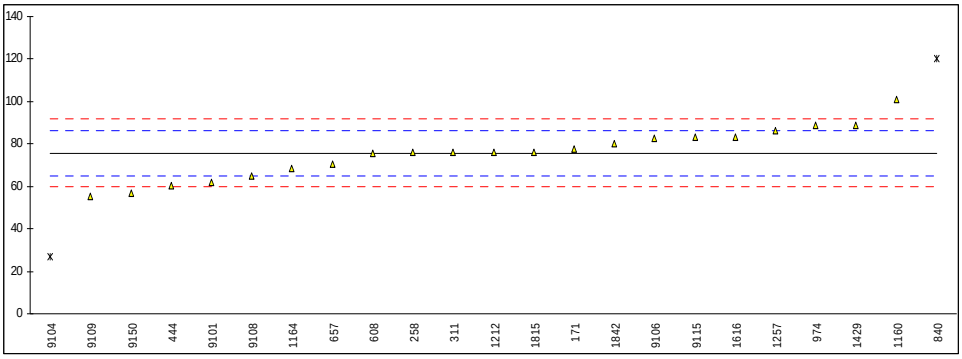
			Only ASTM D156 data:	Only ASTM D6045 data:
	normality	not OK	not OK	n.a.
	n	21	15	5
	outliers	1	1	0
	mean (n)	16.25	16.39	15.90
	st.dev. (n)	1.690	1.982	0.548
	R(calc.)	4.73	5.55	1.53
	R(D156:07a)	2.00	2.00	1.24

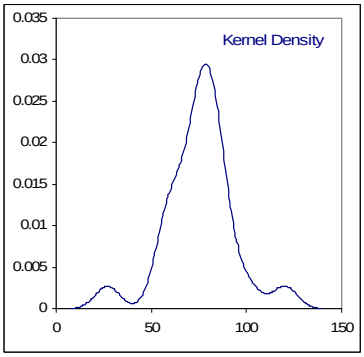




Determination of Sulphur on sample #11100; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D5453	77.81		0.40	
258	D5453	75.9		0.04	
311	D5453	76		0.06	
442		-----		-----	
444	D5453	60.2		-2.91	
449		-----		-----	
602		-----		-----	
608	D5453	75.4		-0.05	
609		-----		-----	
631		-----		-----	
657	D5453	70.6		-0.96	
840	D4294	120.2	G(0.05)	8.38	
974	D4294	88.6		2.42	
1023		-----		-----	
1160	D4294	101		4.77	
1164	D5453	68.4		-1.37	
1212	D5453	76.1		0.08	
1214		-----		-----	
1257	D4294	86		1.94	
1267		-----		-----	
1429	IP490	88.93		2.49	
1616	D5453	83.0		1.38	
1800		-----		-----	
1815	D5453Mod.	76.2		0.10	
1842	in house	80.0		0.81	
1929		-----		-----	
9100		-----		-----	
9101	D5453	62.0		-2.58	
9102		-----		-----	
9103		-----		-----	
9104	D5453	27.0	G(0.05)	-9.16	
9105		-----		-----	
9106	D5453	82.8		1.34	
9107		-----		-----	
9108	D5453	65.0		-2.01	
9109	D5453	55.40		-3.82	
9110		-----		-----	
9111		-----		-----	
9112		-----		-----	
9113		-----		-----	
9114		-----		-----	
9115	D5453	83.0		1.38	
9129		-----		-----	
9130		-----		-----	
9141		-----		-----	
9150	D5453	57.0		-3.52	
					Only ASTM D5453 data
	normality	OK			not OK
	n	21			16
	outliers	2			1
	mean (n)	75.68			71.55
	st.dev. (n)	11.512			9.202
	R(calc.)	32.23			25.77
	R(D5453:09)	14.87			14.26

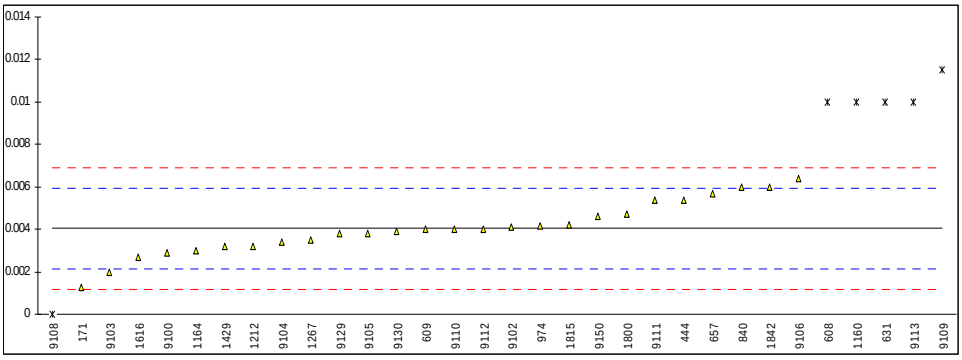


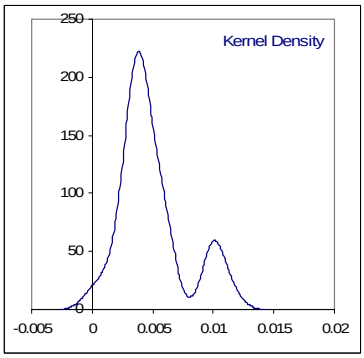


Determination of Water on sample #11100; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D4928	0.00126		-2.93	
258		-----		-----	
311	D4928	<0.02		-----	
442		-----		-----	
444	E203	0.0054		1.42	
449		-----		-----	
602		-----		-----	
608	D4928	0.01	G(0.05)	6.25	
609	D4928	0.004		-0.05	
631	D6304	0.01	G(0.05)	6.25	
657	D4928	0.0057	C	1.73	First reported 57.15 (unit error)
840	D6304	0.006		2.05	
974	D4928	0.00415		0.11	
1023		-----		-----	
1160	IP386	0.010	G(0.05)	6.25	
1164	D4928	0.00297	C	-1.13	First reported 29.7 (unit error)
1212	D4928	0.00322	C	-0.87	First reported 32.2 (unit error)
1214		-----		-----	
1257		-----		-----	
1267	D4928	0.003487		-0.59	
1429	IP386	0.0032		-0.89	
1616	UOP481	0.0027		-1.42	
1800	D4928	0.0047		0.68	
1815	ISO12937	0.0042		0.16	
1842	IP386	0.006		2.05	
1929		-----		-----	
9100	D4928	0.0029		-1.21	
9101	D4928	<0.01		-----	
9102	In house	0.0041		0.05	
9103	In house	0.002		-2.15	
9104	D4928	0.0034		-0.68	
9105	D4928	0.0038		-0.26	
9106	D4928	0.0064		2.47	
9107	D4928	<0.02		-----	
9108	D4928	0.0000	ex	-4.25	Result excluded, zero not a real result
9109	D4928	0.0115	G(0.05)	7.82	
9110	D4928	0.004		-0.05	
9111	In house	0.0054		1.42	
9112	IP438	0.0040		-0.05	
9113	D4928	0.01	G(0.05)	6.25	
9114		-----		-----	
9115	D4928	<0.02		-----	
9129	D4928	0.0038		-0.26	
9130	D4928	0.0039		-0.16	
9141		-----		-----	
9150	D6304	0.0046		0.58	

normality	OK
n	26
outliers	5
mean (n)	0.00405
st.dev. (n)	0.001247
R(calc.)	0.00349
R(D4928:11)	0.00267

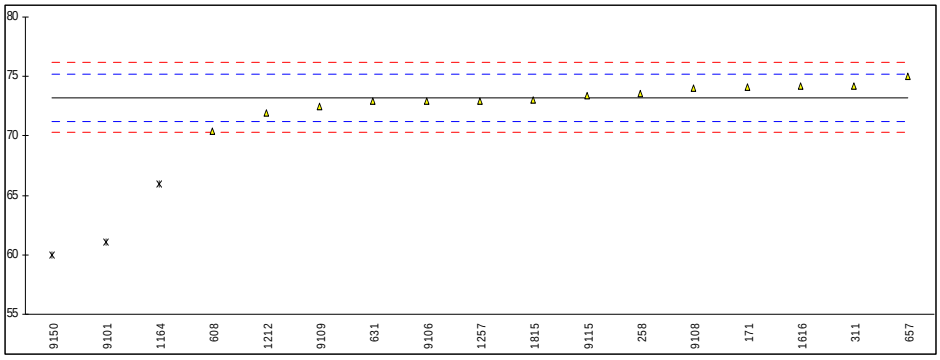


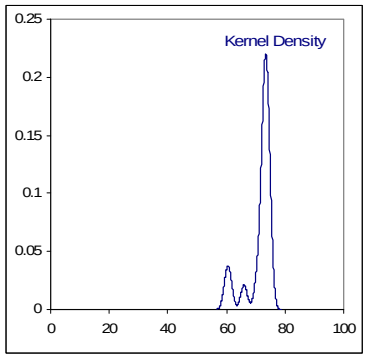


Determination of Total Vapour Pressure (TVP) on sample #11101; results in kPa

lab	method	value	mark	z(targ)	remarks
171	D5191	74.11	C	0.90	First reported 10.75 psi (unit error)
258	D5191	73.6		0.38	
311	D5191	74.2	C	0.99	Reported 67.8 (mixed up with DVPE)
442		-----		-----	
444		-----		-----	
449		-----		-----	
602		-----		-----	
608	D5191	70.42		-2.85	
609		-----		-----	
631	D5191	72.9		-0.33	
657	D5191	75.0		1.81	
840		-----		-----	
974		-----		-----	
1023		-----		-----	
1160		-----		-----	
1164	D5191	66.0	G(0.05)	-7.35	
1212	D5191	71.90		-1.35	
1214		-----		-----	
1257	D5191	72.95	C	-0.28	Reported 10.58 psi (unit error)
1267		-----		-----	
1429		-----		-----	
1616	D5191	74.19		0.98	
1800		-----		-----	
1815	EN13016	73.05		-0.18	
1842		-----		-----	
1929		-----		-----	
9100		-----		-----	
9101	D5191	61.1	DG(0.01)	-12.34	
9102		-----		-----	
9103		-----		-----	
9104		-----		-----	
9105		-----		-----	
9106	D5191	72.9		-0.33	
9107		-----		-----	
9108	D5191	74.0		0.79	
9109	D5191	72.5		-0.74	
9110		-----		-----	
9111		-----		-----	
9112		-----		-----	
9113		-----		-----	
9114		-----		-----	
9115	D5191	73.4		0.18	
9129		-----		-----	
9130		-----		-----	
9141		-----		-----	
9150	D6378	60.0	DG(0.01)	-13.48	

normality	OK
n	14
outliers	3
mean (n)	73.22
st.dev. (n)	1.145
R(calc.)	3.21
R(D5191:10b)	2.75

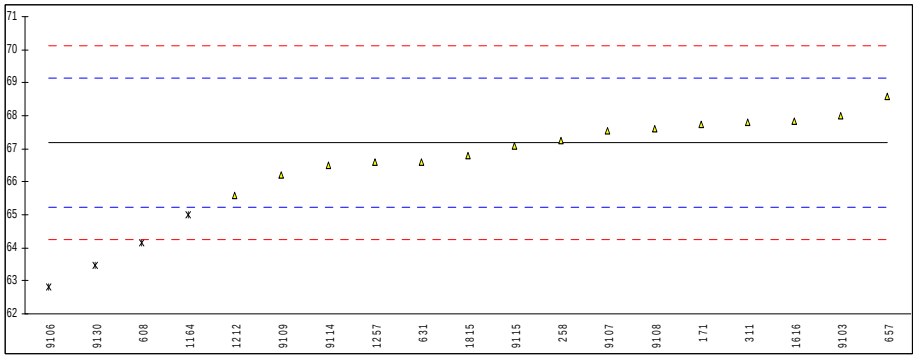


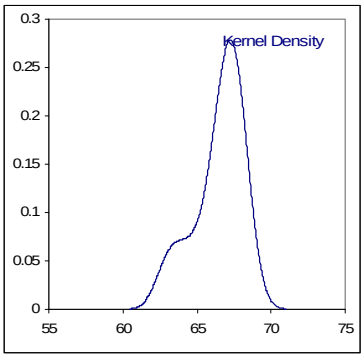


Determination of DVPE (ASTM D5191 calculation) on sample #11101; results in kPa

lab	method	value	mark	z(targ)	remarks
171	D5191	67.74	C	0.57	First reported 9.83 psi (unit error)
258	D5191	67.244		0.06	
311	D5191	67.8	C	0.63	Reported 74.2 (mixed up wiith TVP)
442		-----		-----	
444		-----		-----	
449		-----		-----	
602		-----		-----	
608	D5191	64.15	DG(0.05)	-3.09	
609		-----		-----	
631	D5191	66.6		-0.60	
657	D5191	68.6		1.44	
840		-----		-----	
974		-----		-----	
1023		-----		-----	
1160		-----		-----	
1164	D5191	65.0	E	-2.22	possibly a calculation error? DVPE from TVP = 65 should be 59.91
1212	D5191	65.60		-1.61	
1214		-----		-----	
1257	D5191	66.60	C	-0.60	Recalculated, reported 9.66 psi (unit error)
1267		-----		-----	
1429		-----		-----	
1616	D5191	67.84		0.67	
1800		-----		-----	
1815	EN13016	66.8		-0.39	
1842		-----		-----	
1929		-----		-----	
9100		-----		-----	
9101		-----		-----	
9102		-----		-----	
9103	In house	68.0		0.83	
9104		-----		-----	
9105		-----		-----	
9106	D6378	62.8	E	-4.46	possibly a calculation error? DVPE from TVP = 72.9 should be 66.57
9107	D5191	67.55		0.37	
9108	D5191	67.6		0.42	
9109	D5191	66.2		-1.00	
9110		-----		-----	
9111		-----		-----	
9112		-----		-----	
9113		-----		-----	
9114	D5191	66.5		-0.70	
9115	D5191	67.1		-0.09	
9129		-----		-----	
9130	D5191	63.5	DG(0.05)	-3.78	
9141		-----		-----	
9150		-----		-----	

normality	OK
n	15
outliers	2
mean (n)	67.18
st.dev. (n)	0.797
R(calc.)	2.23
R(D5191:10b)	2.75





APPENDIX 2:

Z-scores distillation ASTM D86

	Automated					Manual				
lab	IBP	10%	50%	90%	FBP	IBP	10%	50%	90%	FBP
171	0.96	3.40	2.96	0.76	4.57	0.86	2.13	0.88	0.62	4.31
258	1.24	1.04	0.88	1.49	-2.94	1.11	0.65	0.26	1.22	-2.77
311	-0.11	0.60	-1.21	-1.52	-3.44	-0.09	0.37	-0.36	-1.24	-3.24
442	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
444	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
449	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
602	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
608	0.96	-3.16	2.96	3.22	-0.47	0.86	-1.98	0.88	2.63	-0.44
609	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
631	0.68	-1.41	-3.74	0.03	-----	0.61	-0.89	-1.11	0.03	-----
657	-1.73	1.47	4.45	-0.88	2.96	-1.54	0.92	1.32	-0.72	2.79
840	0.03	-3.90	-3.22	-4.49	0.71	0.03	-2.44	-0.96	-3.66	0.67
974	0.79	0.34	1.03	4.54	-1.46	0.71	0.21	0.30	3.70	-1.37
1023	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1160	2.92	2.52	3.71	-----	-4.39	2.61	1.58	1.10	-----	-4.13
1164	-0.16	1.21	-0.02	-2.24	-0.88	-0.14	0.76	-0.01	-1.83	-0.83
1212	0.12	-2.29	-3.44	-1.29	1.52	0.11	-1.44	-1.02	-1.05	1.43
1214	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1257	-1.06	-0.19	-0.76	-1.11	-3.32	-0.94	-0.12	-0.23	-0.90	-3.12
1267	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1429	-3.08	-0.19	-0.76	-1.15	0.98	-2.74	-0.12	-0.23	-0.94	0.92
1616	0.12	1.21	-0.02	-3.15	-1.50	0.11	0.76	-0.01	-2.57	-1.41
1800	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1815	-0.81	-1.15	-1.06	0.53	-5.36	-0.72	-0.72	-0.31	0.44	-5.05
1842	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1929	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9100	5.22	4.19	7.43	12.60	16.75	4.66	2.62	2.20	10.28	15.78
9101	-2.18	-1.24	-1.51	1.53	0.77	-1.94	-0.78	-0.45	1.25	0.73
9102	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9103	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9104	-0.44	0.34	1.47	4.59	0.15	-0.39	0.21	0.44	3.74	0.14
9105	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9106	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9107	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9108	5.16	-1.41	4.45	7.54	1.19	4.61	-0.89	1.32	6.16	1.12
9109	-0.72	-7.54	-8.95	-9.98	-5.84	-0.64	-4.73	-2.66	-8.15	-5.49
9110	5.27	0.86	0.28	-1.52	8.25	4.71	0.54	0.08	-1.24	7.77
9111	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9112	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9113	3.65	0.77	-3.59	-8.30	1.10	3.26	0.48	-1.07	-6.77	1.04
9114	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9115	-1.34	0.86	-1.06	-3.02	0.98	-1.19	0.54	-0.31	-2.46	0.92
9129	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9130	-0.55	0.34	-1.80	-3.88	0.57	-0.49	0.21	-0.54	-3.17	0.53
9141	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
9150	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

APPENDIX 3:

Number of participating laboratories per country

1 laboratory in	AUSTRALIA
3 laboratories in	CANADA
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
2 laboratories in	QATAR
1 laboratory in	RUSSIA
1 laboratory in	SINGAPORE
1 laboratory in	SLOVAKIA
1 laboratory in	SWEDEN
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

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