Results of Proficiency Test Gas Condensate November 2010

Organised by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

Authors: ing. M. Jafar

Correctors: dr. R.G. Visser and ing. R. Starink

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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 2010/2011, it was decided to continue the round robin for the analysis of Gas Condensate. In this Interlaboratory study, 56 laboratories from 19 different countries have participated. See appendix 3 for the number of participating laboratories per country. In this report, the results of the Gas Condensate 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 condensate (1* 0.5 L bottle labelled #1081 and 1* 0.25 L bottle labelled #1082, 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 100% confidentiality of participant's data. In addition, customer's satisfaction is measured on a 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 'i.i.s. 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.

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2.4 SAMPLES

The necessary bulk material for the samples #1081 and #1082 was obtained from a local refinery in the Netherlands. The bulk material was split for preparation for both samples. After homogenisation, the Gas Condensate was transferred to 90 amber glass bottles of 0.5 litre and to 90 amber glass bottles of 0.25 litre that were labelled #1081 and #1082 respectively.

The homogeneity of the subsamples #1081 was checked by determination of Density @ 15°C in accordance with ASTM D4052:02e1 on 6 stratified randomly selected samples. The homogeneity of the subsamples #1082 was checked by determination of DVPE in accordance with ASTM D5191:10 on 5 stratified randomly selected samples. The homogeneity testing was performed by a subcontracted ISO17025 accredited laboratory.

	Density @ 15 °C in kg/L (#1081)		DVPE in psi (#1082)
Sample #1081-1	0.74345	Sample #1082-1	9.56
Sample #1081-2	0.74352	Sample #1082-2	9.59
Sample #1081-3	0.74351	Sample #1082-3	9.59
Sample #1081-4	0.74352	Sample #1082-4	9.51
Sample #1081-5	0.74351	Sample #1082-5	9.50
Sample #1081-6	0.74342		

Table 1: homogeneity test of subsamples #1081 and subsamples #1082

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 (#1081)	DVPE in psi (#1082)
r sample	0.00012	0.1134
reference method	ASTM D4052:02e1	ASTM D5191:10
0.3xR(reference method)	0.00015	0.1197

Table 2: repeatability of subsamples #1081 and #1082

Each of the calculated repeatabilities was less than 0.3 times the reproducibility of the respective reference method. Therefore, homogeneity of the subsamples #1081 and subsamples #1082 was assumed.

To each of the participating laboratories 1 * 0.5 L bottle (labelled #1081) and 1 * 0.25 L bottle (labelled #1082) were sent on October 13, 2010.

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.

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2.6 ANALYSES

The participants were requested to determine on sample #1081: Density @ 15°C, total Sulphur, Distillation (IBP, 10%, 50%, 90% evaporated and FBP), Colour Saybolt, Water by KF and Mercury. Sample #1082 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.

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

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

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

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

```
| z | < 1good

1 < | z | < 2satisfactory

2 < | z | < 3questionable

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, Nigeria, Norway, Philippines, Poland, U.S.A, U.A.E, UK and Vietnam. The samples took an unexpected long time to reach a number of laboratories due to problems with custom clearance.

Only 25 (!) participants reported results. The 25 reporting laboratories did send in 215 numerical results. Observed were 25 outlying results, which is 11.6%. 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.

<u>Density @ 15°C:</u> This determination was problematic. Five statistical outliers were observed.

Also the calculated reproducibility, after rejection of the statistical outliers, is

not in agreement with the requirements of ASTM D4052:02e1.

The reproducibility requirement, mentioned in the latest version ASTM D4052:09 is surprisingly large and consequently very easy to meet.

<u>Sulphur</u>: This determination was problematic for a number of laboratories. Four

statistical outliers were observed. However, the calculated reproducibility is, after rejection of the statistical outliers, is in agreement with the requirements

of ASTM D5453:09.

<u>Distillation</u>: This determination was problematic. Eight statistical outliers were observed.

Four of the five reported test results of laboratory 1160 appeared to be statistical outliers. As the five test results are not independent, it was decided

not to use also the test results for 10% evaporated for the statistical

evaluation.

After rejection of the statistical outliers, none of the calculated reproducibilities is in agreement with the requirements for manual mode or automated

mode of ASTM D86:10a, except for 50% evaporated for manual mode.

<u>Saybolt Color:</u> 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 D156:07a.

Water: This determination was problematic. Three statistical outliers were observed

and the calculated reproducibility is not in agreement with the requirements

of ASTM D4928:10.

Mercury: Regretfully, there are no precision requirements in UOP938 available for

evaluation. Therefore, the Horwitz equation was used. This determination was problematic at this concentration level (149 μ g/kg). One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is not in agreement with the strict estimated reproducibility calculated using the Horwitz equation. The low number of test results may (partly)

explain the large spread.

TVP & DVPE: This determination was problematic. Two statistical outliers were observed

and one participating laboratory probably made a calculation error. The calculated reproducibility is after rejection of the statistical outliers not in

agreement with the requirements of ASTM D5191:07.

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 #1081 and #1082, 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	18	743.51	0.67	0.50 (2.12)
Sulphur	mg/kg	10	11.61	3.56	3.65
Initial Boiling Point	°C	14	33.23	8.71	5.02(A)-5.60(M)
10%-evaporated	°C	15	63.03	11.55	3.20(A)-4.94(M)
50%-evaporated	°C	14	117.75	5.58	1.88(A)-5.67(M)
90%-evaporated	°C	13	231.85	25.22	5.54(A)-7.06(M)
Final Boiling Point	°C	14	297.57	11.35	6.78(A)-7.20(M)
Saybolt Color		14	20.9	6.1	2.0
Water content by KF	mg/kg	18	50.76	47.24	31.01
Mercury as Hg	μg/kg	5	149.5	104.2	76.9
Total vapour pressure	psi	13	10.44	0.61	0.40
DVPE acc. to ASTM D5191	psi	12	9.49	0.63	0.40

Table 3: performance evaluation sample #1081 and #1082

Without further statistical calculations it can be concluded from the overview given in table 3 that for several 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.

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4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2010 WITH THE PREVIOUS PTS

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

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

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

The performance of the determinations against the requirements of the respective 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

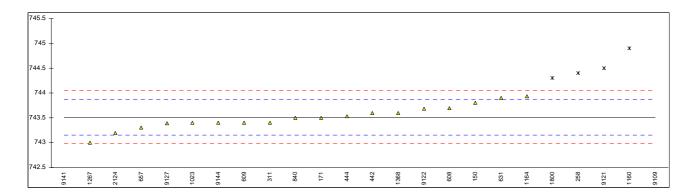
n.e.: not evaluated

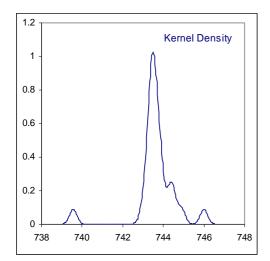
APPENDIX 1

Determination of Density @ 15°C on sample #1081; results in kg/m³

					081; results in kg/m ³
lab	method	value	mark	z(targ)	remarks
150	D4052	743.8		1.61	
171	D4052	743.5	C C(0.05)	-0.07	First reported 745 0
258 311	D1298 D4052	744.4 743.4	C, G(0.05)	4.97 -0.63	First reported 745.0
442	D4052 D4052	743.4		0.49	
444	D4052	743.53		0.49	
449	D-1002				
602					
608	D4052	743.7		1.05	
609	D4052	743.4		-0.63	
631	D4052	743.9		2.17	
657	D4052	743.3		-1.19	
840	D4052	743.50		-0.07	
974	D.1050	7.40.4			
1023	D4052	743.4	C DC(0.05)	-0.63	First reported 0.7440
1160	IP365 D4052	744.9 743.93	C, DG(0.05)	7.77 2.33	First reported 0.7449
1164 1214	D4032	743.93		2.33	
1267	D4052	743		-2.87	
1368	D4052	743.6		0.49	
1616	2 .002				
1800	in house	744.3	ex	4.41	Measured at 15.6°C
1815					
1842					
2124	D4052	743.2		-1.75	
9050					
9054					
9055					
9056 9057					
9058					
9061					
9100					
9101					
9102					
9103					
9104					
9106					
9107					
9108 9109	D4052	746.0	G(0.01)	13.93	
9110	D4032	740.0	G(0.01)		
9111					
9116					
9117					
9121	D4052	744.5	U, DG(0.05)	5.53	Reported 0.7445
9122	D4052	743.69	U	0.99	Reported 0.74369
9123					
9125					
9126 9127	D4052	743.39		-0.69	
9129	D4032	743.39		-0.03	
9132					
9141	D5002	739.56	U, G(0.01)	-22.14	Reported 0.73956
9144	D4052	743.4	-, -(,	-0.63	Transfer and the second
9145					
	normality	OK			
	n	18			
	outliers	5			
	mean (n)	743.513			
	st.dev. (n) R(calc.)	0.2377 0.666			
	R(D4052:02e1)	0.500			Compare (D4052:09) = 2.123
	(5.1002.0201)	5.555			- 1120

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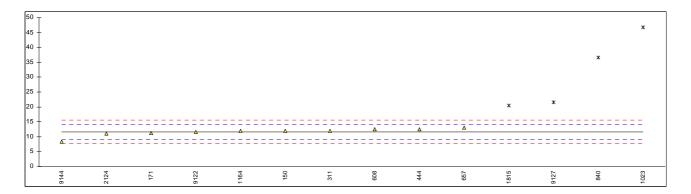


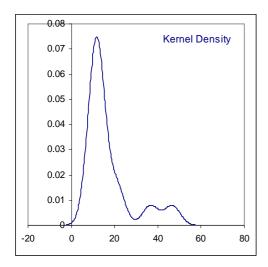


Determination of Sulphur on sample #1081; results in mg/kg

					results in mg/kg
lab	method	value	mark	z(targ)	remarks
150	D5453	12		0.30	
171 258	D5453	11.3 		-0.24 	
311	D5453	12		0.30	
442	D0400				
444	D5453	12.47		0.66	
449					
602					
608	D5453	12.44 		0.64	
609 631					
657	D5453	13.0		1.07	
840	D4294	36.6	G(0.01)	19.19	
974					
1023	D2622	46.7	G(0.05)	26.94	
1160	DE 152	11.9		0.22	
1164 1214	D5453			0.22	
1267					
1368					
1616					
1800 1815	D5453mod.	20.52	DG(0.01)	6.84	
1842	D545311100.	20.52	DG(0.01)	0.64	
2124	D5453	11		-0.47	
9050					
9054					
9055					
9056 9057					
9058					
9061					
9100					
9101					
9102 9103					
9104					
9106					
9107					
9108 9109					
9110					
9111					
9116					
9117					
9121 9122	D5453	11.6		-0.01	
9123	D0400				
9125					
9126			50(55)		
9127	D5453	21.5	DG(0.01)	7.59	
9129 9132					
9141					
9144	D5453	8.4		-2.47	
9145					
	normality	OK 10			
	n outliers	10 4			
	mean (n)	11.611			
	st.dev. (n)	1.2702			
	R(calc.)	3.557			
	R(D5453:09)	3.646			

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Determination of Distillation on sample #1081; results in °C

Deter	mination of D	istillatio	n on sa	mple #1	1081; r	esults ir	า °C					
lab	method	IBP	mark	10%	mark	50%	mark	90%	mark	FBP	Mark	remarks
150	D86-A	33.9		61.1		116.0		223.4		297.2		
171	D86	30.9		67.2		120.4		244.7		295.1		
258	D86-A	36.8		60.8		120.1		235.5		278.0	G(0.01)	
311	D86-A	32.4		69.8		116.6		229.4		300.4	- (/	
442	20071											
444												
449												
602												
608	D86-M	36.0		62.5		117.0		220.5		293.0		
609	D00-IVI											
631	D86-M	38.0		66.0		120.0				290.0	С	fr. 280.0
657	D86-A	33.5		60.9		116.5		228.2		294.6	C	11. 200.0
840	D86-A	28.75		62.45		118.85		231.35		304.40		
974	D00-A	20.73		02.43				231.33		304.40		
	D86-M			70.0		125.0	DG(0.05)	260.0	DG(0.05)	295.0		
1023		32.0	C(0.05)		01/						C(0.05)	
1160	IP123-M	48.0	G(0.05)	72.0	ex	125.5	DG(0.05)	270.0	DG(0.05)	270.0	G(0.05)	
1164	D86-A	33.0		63.5		119.0		237.5		297.0		
1214												
1267												
1368												
1616												
1800	100010-1											
1815	ISO3405-A	36.0		57.3		115.6		223.3		299.5		
1842												
2124												
9050												
9054												
9055												
9056												
9057												
9058												
9061												
9100												
9101												
9102												
9103												
9104												
9106												
9107												
9108												
9109												
9110												
9111												
9116												
9117												
	D86-M	28.7		59.1		115.6		240.9		304.4		
9122	D86-M	36.2		61.7		119.4		245.9		299.2		
9123												
9125												
9126												
9127	D86-A	29.1		56.6		114.4		218.4		299.6		
9129												
9132												
9141												
9144	D86-M	42.0	G(0.05)	66.5		119.0		235.0		296.5		
9145												
	normality	ОК		ОК		ОК		OK		OK		
	n	14		15		14		13		14		
	outliers	2		0		2		2		2		
	mean (n)	33.23		63.03		117.75		231.85		297.57		
	st.dev. (n)	3.111		4.124		1.993		9.006		4.054		
	R(calc.)	8.71		11.55		5.58		25.22		11.35		
	R(D86:10a) - A	5.02		3.20		1.88		5.54		6.78		
	R(D86:10a) - M			4.94		5.67		7.06		7.20		
	. ((DOO. 10a) - W	1 3.00		1 7.0-7		1 3.07		1.00		1.20		

Automated R (D86:10a) (Paragraph 13.1.2)

IBP: 5.023 =0.0595 (mean + 51.19)

10%: 3.20

50%: 1.88

90%: 5.54 = 0.019 (mean + 59.77)

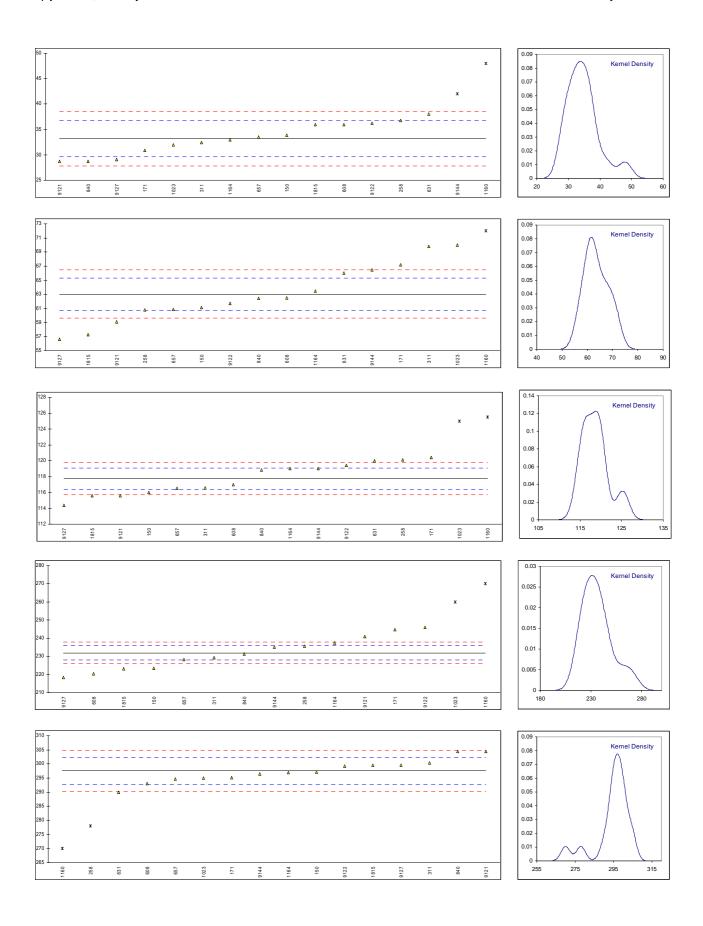
FBP: 6.780

Manual R (D86:10a) (Table A4.4)

IBP: 5.6

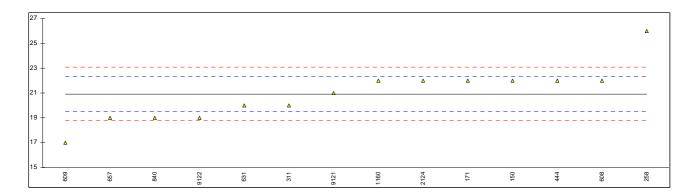
10%: 4.94 = $2.0 + 1.74 \times C/\%V$ and C/%V = 1.6950%: 5.67 = $2.0 + 1.74 \times C/\%V$ and C/%V = 2.1190%: 7.06 = $0.8 + 1.74 \times C/\%V$ and $0.80 \times C/\%V = 3.60$

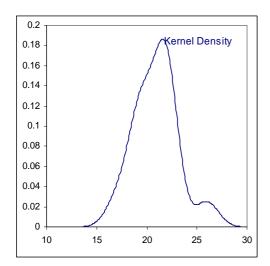
FBP: 7.2



Determination of Saybolt Color on sample #1081;

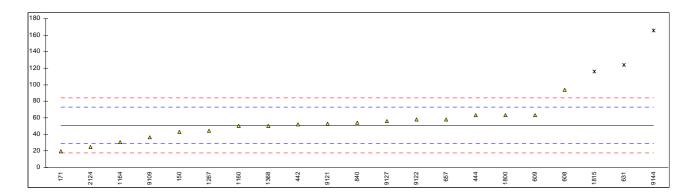
Deten	mination of S				
lab	method	value	mark	z(targ)	remarks
150	D156	22		1.50	
171	D156	22		1.50	
258	D156	26		7.10	
311	D156	20		-1.30	
442					
444	D156	22		1.50	
449					
602					
608	D156	22		1.50	
609	D156	17		-5.50	
631	D156	20		-1.30	
657	D156	19		-2.70	
840	D156	19		-2.70	
974					
1023	5				
1160	D156	22		1.50	
1164					
1214					
1267					
1368					
1616 1800					
1815					
1842					
2124	D156	22		1.50	
9050	D130				
9054					
9055					
9056					
9057					
9058					
9061					
9100					
9101					
9102					
9103					
9104					
9106					
9107					
9108					
9109					
9110					
9111					
9116 9117					
9121	D156	21		0.10	
9122	D156	19		-2.70	
9123	D100				
9125					
9126					
9127					
9129					
9132					
9141					
9144					
9145					
	normality	not OK			
	n	14			
	outliers	0			
	mean (n)	20.93			
	st.dev. (n)	2.165			
	R(calc.)	6.06			
	R(D156:07a)	2.00			

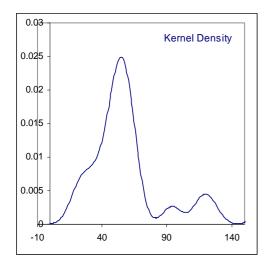




Determination of Water on sample #1081; results in mg/kg

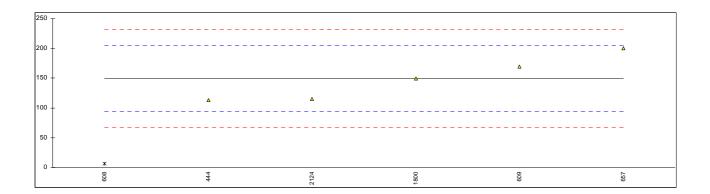
Determination of V		•		
lab method	value	mark		remarks
150 D4928	43		-0.70	
171 D4928	19.7		-2.80	
258 311 D4028	<0.02	U		Probably reported in different unit
311 D4928		U		Probably reported in different unit
442 IP386	51.89		0.10	
444 E203	63 		1.11	
449 602				
608 D4928	94		3.90	
609 D4928	63.5		1.15	
631 D6304	124	C, DG(0.05)		First reported 694
657 D4928	58.25	C, DG(0.03)	0.68	Tilist reported 094
840 D4928	54.0		0.29	
974				
1023				
1160 IP386	50	С		First reported 0.005
1164 D6304	30.4	· ·	-1.84	
1214				
1267 D4928	44.20		-0.59	
1368 D1744	50.146		-0.06	
1616				
1800 in house	63.2		1.12	
1815 ISO12937	115.8	DG(0.05)	5.87	
1842		, ,		
2124 in house	24.99		-2.33	
9050				
9054				
9055				
9056				
9057				
9058				
9061				
9100				
9101				
9102				
9103				
9104				
9106				
9107				
9108			1.20	
9109 D4928	36.5		-1.29	
9110				
9111				
9116 9117				
9121 D4928	53		0.20	
9121 D4928 9122 D4928	58		0.20	
9123			0.05	
9125				
9126				
9127 D6304	55.9		0.46	
9129				
9132				
9141				
9144 D4928	165.5	G(0.05)	10.36	
9145		. ,		
normality	OK			
n	18			
outliers	3			
mean (n)	50.76			
st.dev. (n)	16.872			
R(calc.)	47.24			
R(D4928:10)	31.01			





Determination of Mercury as Hg on sample #1081; results in µg/kg

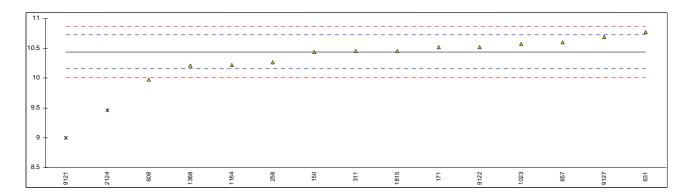
					1081; results in µg/kg
lab	method	value	mark	z(targ)	remarks
150					
171					
258					
311					
442 444	UOP938	112.0		 -1.15	
444	007936	112.8 		-1.15	
602					
608	INH-010	6.049	G(0.05)	-4.51	
609	UOP938	169.7230	- (,	0.64	
631					
657	UOP938	200.47	С	1.60	First reported 22.3
840					
974					
1023					
1160 1164					
1214					
1267					
1368					
1616					
1800	in house	149.5		0.00	
1815					
1842					
2124	in house	115		-1.08	
9050					
9054 9055					
9056					
9057					
9058					
9061					
9100					
9101					
9102					
9103					
9104 9106					
9107					
9108					
9109					
9110					
9111					
9116					
9117					
9121 9122					
9122					
9125					
9126					
9127					
9129					
9132					
9141					
9144 9145					
3140					
	normality	n/a			
	n	5			
	outliers	1			
	mean (n)	149.5			
	st.dev. (n)	37.23			
	R(calc.)	104.2			
	R(Horwitz)	76.9			

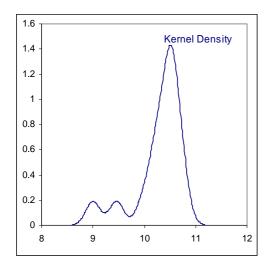


Determination of Total Vapour Pressure (TVP) on sample #1082; results in psi

					VP) on sample #1082; results in psi
lab	method	value	mark	z(targ)	remarks
150	D5191	10.44		0.01	
171	D5191	10.52		0.57	
258	D5191	10.268		-1.20	
311	D5191	10.46		0.15	
442					
444					
449					
602	D5404				
608	D5191	9.98		-3.21	
609	DE404	40.77			
631 657	D5191	10.77		2.32	
657 840	D5191	10.60		1.13	
974					
1023	D5191	10.57		0.92	
1160	D3131				
1164	D6378	10.22		-1.53	
1214	200.0				
1267					
1368	D5191	10.2107		-1.60	
1616					
1800					
1815	D6378mod.	10.46		0.15	
1842					
2124	D5191	9.46	G(0.05)	-6.85	
9050					
9054					
9055					
9056					
9057					
9058					
9061					
9100					
9101					
9102					
9103 9104					
9104					
9107					
9108					
9109					
9110					
9111					
9116					
9117					
9121	D323	9.0	G(0.05)	-10.07	
9122	D5191	10.52		0.57	
9123					
9125					
9126					
9127	D5191	10.689		1.75	
9129					
9132					
9141					
9144					
9145					
	p.	014			
	normality	OK			
	n outliere	13			
	outliers	2			
	mean (n)	10.439			
	st.dev. (n) R(calc.)	0.2178 0.610			
	R(D5191:07)	0.400			
	. ((20101.01)	0.400			

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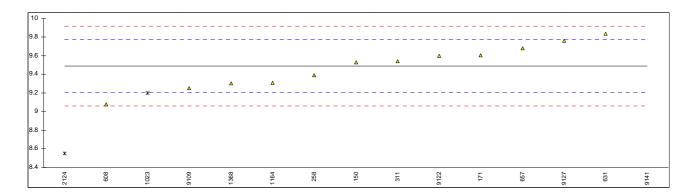


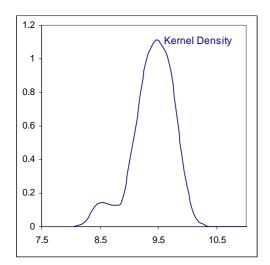


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Determination of DVPE (ASTM D5191 calculation) on sample #1082; results in psi

					ulation) on sample #1082; results in psi
lab 150	method DE101	value	mark	z(targ)	remarks
150 171	D5191 D5191	9.53 9.604		0.27 0.79	
258	D5191 D5191	9.804		-0.79	
311	D5191	9.54		0.34	
442	20101				
444					
449					
602					
608	D5191	9.08		-2.88	
609	D5404				
631	D5191	9.84		2.44	
657 840	D5191	9.68		1.32	
974					
1023	D5191	9.20	ex, E	-2.04	Recalculate by iis 9.65 ($z = 7.55$)
1160			, -		
1164	D6378	9.31		-1.27	
1214					
1267	_		_		
1368	D5191	9.30504	С	-1.30	First Reported 8.8908
1616					
1800 1815					
1842					
2124	D5191	8.55	G(0.05)	-6.59	
9050			,		
9054					
9055					
9056					
9057 9058					
9061					
9100					
9101					
9102					
9103					
9104 9106					
9107					
9108					
9109	D5191	9.25		-1.69	
9110					
9111					
9116					
9117 9121					
9121	D5191	9.60		0.76	
9123					
9125					
9126	B=15:				
9127	D5191	9.761		1.89	
9129 9132					
9141	D5191	59.9500	G(0.01)	353.21	Probably reported in Kpa
9144	20101		O (0.01)		Trobably reported in Trea
9145					
	normality	OK			
	n	12			
	outliers	2			
	mean (n) st.dev. (n)	9.491 0.2263			
	R(calc.)	0.634			
	R(D5191:07)	0.400			





APPENDIX 2:

Z-scores distillation ASTM D86 (automated and manual mode used for calculation)

	Automated mode							Manual mode				
lab	method	IBP	10%	50%	90%	FBP	IBP	10%	50%	90%	FBP	
150	D86-A	0.37	-1.69	-2.60	-4.27	-0.15	0.34	-1.09	-0.86	-3.35	-0.14	
171	D86	-1.30	3.65	3.95	6.49	-1.02	-1.16	2.36	1.31	5.10	-0.96	
258	D86-A	1.99	-1.95	3.51	1.84	-8.08	1.79	-1.26	1.16	1.45	-7.61	
311	D86-A	-0.46	5.92	-1.71	-1.24	1.17	-0.41	3.84	-0.57	-0.97	1.10	
442												
444												
449												
602	D00.14					4.00				4.50	4.70	
608	D86-M	1.54	-0.46	-1.11	-5.74	-1.89	1.39	-0.30	-0.37	-4.50	-1.78	
609	DOC M	0.00	0.00	2.20		2.42	0.00	4.00	4.44			
631	D86-M D86-A	2.66	2.60	3.36	1 0 1	-3.13	2.39	1.68	1.11	1 15	-2.94 1.15	
657	D86-A	0.15	-1.86	-1.86	-1.84	-1.23	0.14	-1.21	-0.62 0.54	-1.45 -0.20	-1.15	
840 974	D00-A	-2.50 	-0.51 	1.64	-0.25 	2.82	-2.24 	-0.33 	0.54	-0.20	2.66	
1023	D86-M	-0.69	6.10	10.80	14.23	-1.06	-0.61	3.95	3.58	11.17	-1.00	
1160	IP123-M	8.23	7.85	11.55	19.28	-11.38	7.39	5.08	3.83	15.14	-10.72	
1164	D86-A	-0.13	0.41	1.87	2.86	-0.23	-0.11	0.27	0.62	2.24	-0.22	
1214	D00-A	-0.13			2.00	-0.23	-0.11			2.24	-0.22	
1267												
1368												
1616												
1800												
1815	ISO3405-A	1.54	-5.01	-3.20	-4.32	0.80	1.39	-3.25	-1.06	-3.39	0.75	
1842												
2124												
9050												
9054												
9055												
9056												
9057												
9058												
9061												
9100												
9101												
9102												
9103												
9104												
9106												
9107												
9108 9109												
9110												
9111												
9116												
9117												
9121	D86-M	-2.54	-3.43	-3.20	4.57	2.84	-2.28	-2.22	-1.06	3.59	2.67	
	D86-M	1.66	-1.16	2.46	7.10	0.67	1.49	-0.75	0.82	5.57	0.64	
9123	200											
9125												
9126												
	D86-A	-2.30	-5.63	-4.98	-6.80	0.84	-2.06	-3.64	-1.65	-5.34	0.79	
9129												
9132												
9141												
9144	D86-M	4.89	3.04	1.87	1.59	-0.44	4.39	1.97	0.62	1.25	-0.41	
9145												

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APPENDIX 3:

Number of participating laboratories per country

- 2 labs in AUSTRALIA
- 3 labs in CANADA
- 6 labs in MALAYSIA
- 1 lab in MOZAMBIQUE
- 1 lab in NEGARA BRUNEI DARUSSALAM
- 7 labs in NIGERIA
- 2 labs in NORWAY
- 2 labs in OMAN
- 1 lab in P.R. of CHINA
- 3 labs in PHILIPPINES
- 1 lab in POLAND
- 1 lab in QATAR
- 1 lab in RUSSIA
- 1 lab in SINGAPORE
- 4 labs in THE NETHERLANDS
- 1 lab in U.A.E.
- 7 labs in U.S.A.
- 11 labs in UNITED KINGDOM
 - 1 lab 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
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- 5 ISO 5725, parts 1-6, 1994
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- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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- 9 DIN 38402 T41/42
- 10 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 11 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
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- 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/)