Results of Proficiency Test Liquefied Propane October 2010

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## 1 INTRODUCTION

During the last years, with increasing frequency, requests were received by iis from laboratories that participated in the iis PT program, to organize also a proficiency test for the Liquefied Propane Analysis. Beginning 2008, iis started an investigation for the feasibility of such a PT. Because iis has limited gas-handling facilities in place to prepare gas samples, Scott Specialty Gases (Breda, the Netherlands) was contacted. This company is fully equipped and has a broad experience in the preparation of synthetic Liquefied Propane samples for PT purposes. Together with this company, it was decided to organize a first proficiency study for Liquefied Propane (composition only) in 2009.

This interlaboratory study was repeated in 2010, in which now 20 laboratories from 14 different countries have participated. See appendix 3 for the number of participants per country.

In this report the results of the proficiency test on Liquefied Propane are presented and discussed.

## 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test.

To optimise the costs for the participating laboratories, it was decided to prepare one Liquefied Propane mixture. The mixture was divided over a batch of 21 cylinders.

The cylinder size is a cost-effective two-litre cylinder with dip tube device.

Each cylinder was uniquely numbered. The limited cylinder size is chosen to optimise transport and handling costs.

Participants were requested to report rounded and unrounded results, both in %M/M and in %mol/mol. 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, ISO 17043:2010 and ILAC-G13:2007. This ensures 100% confidentially of participant's data. Also customer's satisfaction is measured on regular basis by the distribution of 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), that can be downloaded from the iis web site http://www.iisnl.com.

### 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

In this proficiency test only one sample was used. A batch of two-litre cylinders with dip-tube containing artificial Liquefied Propane mixture was prepared and tested for homogeneity by Scott Specialty Gases (Breda, the Netherlands) in conformance with ISO 6143 and ISO Guide 35.

In total one batch of 21 cylinders (lot 81110) was prepared on October 4, 2010. Each cylinder was labelled #1075 and also uniquely numbered. The cylinders were all tested in threefold to check the homogeneity of the batch. By ANOVA analysis on the test results in accordance with ISO 6143 the in-between bottle standard deviation was calculated. The repeatability values (r) were calculated per component by multiplication of the respective standard deviation by 2.8.

Subsequently the calculated repeatabilities were compared with 0.3 times the corresponding target reproducibilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

Parameter	conc. in %mol/mol	r (observed) in %mol/mol	0.3 X R(D2163) in %mol/mol
Ethane	0.2454	0.0108	0.0120
Propane	93.2000	0.1137	3.210
Propylene	0.9759	0.0084	0.0330
n-Butane	3.2139	0.0762	0.1110
iso-Butane	1.5958	0.0324	0.0540
n-Pentane	0.7689	0.0226	0.0264
1-Butene	n/a	n/a	n/a
iso-Butene	n/a	n/a	n/a

Table 1: homogeneity test results

The calculated repeatabilities are each less than 0.3 times the corresponding reproducibility of the reference method ASTM D2163.

Therefore, homogeneity of the subsamples #1075 was assumed.

To each of the participating laboratories one 2L cylinder was sent on October 6, 2010.

### 2.5 STABILITY OF THE SAMPLES

Scott Specialty Gases (Breda, the Netherlands) declares that the prepared sample cylinders have a shelf life of at least 6 months. This is sufficient for the proficiency testing purposes.

### 2.6 ANALYSES

The participants were asked to determine the composition: Ethane, Propane, Propylene, n-Butane, iso-Butane, n-Pentane, 1-Butene, iso-Butene and some physical parameters calculated from the composition: Molar Mass, Relative Density and Absolute and Relative Vapour pressure. Also some method details were requested to be reported.

To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. Also a letter of instructions and a SDS were added to the package.

Participants are also requested to send a remark if other components were found e.g. Helium or/and iso-Pentane or some other impurity.

### 3 RESULTS

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

Directly 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 data are put under 'Remarks' in the result tables in appendix 1. Results that came in after deadline were not taken into account in the screening for suspect data and thus these participants were not requested for checks.

### 3.1 STATISTICS

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).

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. In case a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care. In accordance with 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 and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

# 3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported 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 method is producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nr.14 and 15).

## 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. EN-, ISO-, IP reproducibilities, the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated according to:

z(target) = (result - average of PT) / 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| < 1 good 1 < |z| < 2 satisfactory 2 < |z| < 3 questionable 3 < |z| unsatisfactory

## 4 EVALUATION

In this proficiency test several problems were encountered with sample transport. Due to these problems three cylinders did not reach the laboratory in time to test the cylinder and to report results before the deadline of reporting.

In total eight laboratories reported the test results after the final reporting date. Not all laboratories did report all test results requested.

In total 19 participanting laboratories reported 154 numerical test results. Observed were 14 outlying test results, which is 9.1% of all numerical results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

## 4.1 EVALUATION PER TEST/COMPONENT

In this section the results are discussed per component. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

Not all original data sets proved to have a normal distribution. For ethane and for molar mass a non Gaussian data distribution was found and the statistical evaluation should be used with due care.

Because the majority of the participating laboratories used ASTM D2163 as test method, it was decided to use the reproducibilities of this test method as target reproducibilities, and to mention the reproducibilities of EN27941 (identical to IP 405 and ISO 7941) for reference only. Regretfully in the last version ASTM D2163:07 only repeatabilities, but no reproducibilies are mentioned. Therefore the precision data from the previous version ASTM D2163:96 were used.

Because 13 laboratories reported both results in %mol/mol as well as in %M/M, it has been possible to check the calculations of these 13 laboratories. A good correlation between the results reported in %mol/mol and the results reported in %M/M was found, as was to be expected.

- Ethane: The determination of this component may be problematic. Two statistical outliers were detected and the calculated reproducibility, after exclusion of the statistical outliers, is not at all in agreement with the requirements of ASTM D2163:96 (estimated from figure 3). However, the calculated reproducibility is in agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).
- <u>Propane:</u> Analytical problems were observed for a number of laboratories. Two statistical outliers were detected. However, the calculated reproducibility, after exclusion of two statistical outliers and two other test results, is in full agreement with the requirements of ASTM D2163:96 and also with the reproducibility of EN27941 (identical to IP 405 and ISO 7941).

- <u>Propylene:</u> No analytical problems were observed. Two statistical outliers were detected. However, the calculated reproducibility, after exclusion of the statistical outliers, is in full agreement with the requirements of ASTM D2163:96 and also with the reproducibility of EN27941 (identical to IP 405 and ISO 7941).
- <u>n-Butane:</u> No analytical problems were observed. Two statistical outliers were detected. However, the calculated reproducibility, after exclusion of the statistical outliers, is in full agreement with the requirements of ASTM D2163:96 and also with the reproducibility of EN27941 (identical to IP 405 and ISO 7941).
- iso-Butane: No analytical problems were observed. Two statistical outliers were detected. However, the calculated reproducibility, after exclusion of the statistical outliers, is in full agreement with the requirements of ASTM D2163:96 and also with the reproducibility of EN27941 (identical to IP 405 and ISO 7941).
- <u>n-Pentane:</u> The determination of this component may be problematic. One statistical outlier was detected. The calculated reproducibility, after exclusion of the statistical outlier, is not at all in agreement with the requirements of ASTM D2163:96. It is however in agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).
- <u>1-Butene:</u> This component was not present in a detectable concentration. Therefore no conclusions could be drawn.
- <u>Iso-Butene:</u> This component was not present in a detectable concentration. Therefore no conclusions could be drawn.
- Molar Mass: This calculated parameter may be problematic. The results vary over a large range from 44.752 49.041 and one statistically significant outlier was present (in 8 test results). Also three calculation errors may be present. See also the discussion in 4.3.
- <u>Relative Density:</u> This calculated parameter may be problematic. The results vary over a range from 0.5068 0.513 and one statistically significant outlier was present (in 12 test results). Also two calculation errors may be present. See also the discussion in 4.3.
- <u>Abs. Vapour Press.</u>: This calculated parameter may be problematic. The results vary over a large range (150.4 185.9 psi) and one statistically significant outlier was observed. Also two calculation errors may be present. See also the discussion in 4.3.

<u>Rel. Vapour Press.</u>: This calculated parameter may be problematic. The results vary over a large range (165.52 - 170 psi). See also the discussion in 4.3.

### 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 per sample, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM D2163 and EN27941/ISO7941/IP405) are compared in the next table.

Parameter	unit	n	cons. value	2.8 * sd	R(D2163) in <b>%mol</b>	R(EN27941) liqinj. in <b>%mol</b>	R(EN27941) liqinj. in <b>%M/M</b>
Ethane	%mol/mol	17	0.243	<u>0.097</u>	0.040	0.3	0.2
Propane	%mol/mol	15	92.983	0.587	0.930	1	1
Propylene	%mol/mol	15	0.993	0.078	0.114	0.2	0.2
n-Butane	%mol/mol	17	3.334	0.235	0.384	0.4	0.5
iso-Butane	%mol/mol	17	1.675	0.147	0.193	0.4	0.5
n-Pentane	%mol/mol	16	0.817	<u>0.171</u>	0.094	0.3	0.5
Molar Mass	g/mol	6	44.91	0.33	n/a	n/a	n/a
Relative Density		10	0.5121	0.0015	n/a	n/a	n/a
Abs. Vapour pres.	psi	7	182.89	6.18	n/a	n/a	n/a
Rel. Vapour pres.	psi	12	167.59	3.96	n/a	n/a	n/a

Table 2: Performance of the group in comparison with the target reproducibilities

Without further statistical calculations it can be concluded that for most components/tests there is a good compliance of the group of participating laboratories with the relevant standard. The problematic components/tests have been discussed in paragraph 4.1.

### 4.3 DISCUSSION

Because several of the reproducibility requirements of ASTM D2163 differ significantly from the reproducibility requirements of EN27941 (for liquid injection), the outcome of the evaluation will be strongly dependent on the target test method selected for the evaluation.

The consensus values as determined in this PT are compared with the average values from the homogeneity testing by Scott Specialty Gases in the following table.

Parameter	Average values by Scott Specialty Gases in %mol/mol	Consensus values from participants results in %mol/mol	Absolute differences in %mol/mol	z-score
Ethane	0.245	0.243	-0.002	-0.12
Propane	93.200	92.983	-0.217	-0.61
Propylene	0.976	0.993	+0.017	+0.40
n-Butane	3.214	3.334	+0.120	+0.88
iso-Butane	1.596	1.675	+0.079	+1.15
n-Pentane	0.769	0.817	+0.048	+1.44

Table 3: comparison of consensus values with values determined by Scott Specialty Gases

From this comparison it is clear that <u>all</u> consensus values as determined in this PT are very well in line with the values as determined by Scott during the preparation of the cylinders.

In total 5 laboratories reported the presence of some additional components (iso-pentane, neo-pentane, 1,3-butadiene, cyclopentane, cis-2-butene and trans-2-butene). Probably these components were present as impurity in one or more of the pure components that were used to prepare the propane mixture. Also one laboratory reported the presence of some helium (0.645 %mol/mol). See appendix 2.

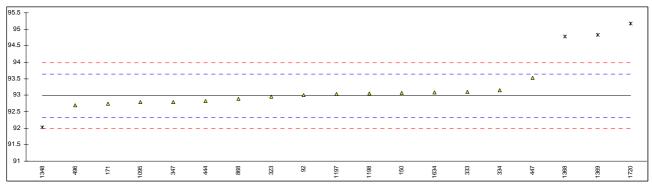
In principle no additional spread should be introduced when applying a calculation on the reported component concentrations. However, in practice a significant additional uncertainty is added. This cannot be caused only by rounding of test results before use in the calculations. See the differences between the values from the results as reported by the participating laboratories (each using its own calculation procedure) and the values as calculated by iis using one calculation procedure for each set of laboratory test results. For the calculation of the Molar Mass, Relative Density and Vapour Pressure several standardized methods are available, e.g. ASTM D2421 for the interconversion of the units to gas-volume, liquid-volume or mass basis. Also different methods for the calculation of the Vapour Pressure do exist. In ISO 8973 (identical to IP432) the Vapour Pressure is calculated from the <u>mole fraction</u> per component and a Vapour Pressure factor of that component (given for all components). In ASTM D2598 the Vapour Pressure is calculated from the <u>liquid</u> volume percentage per component and a Vapour Pressure factor of that component (given for only several components).

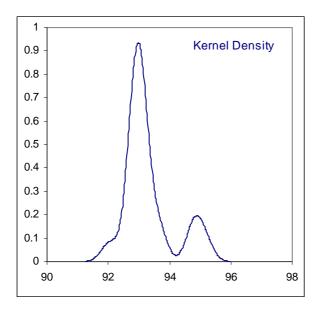
Determination of Ethane; results in %mol/mol

	-	•			-									
lab	method	value	mark	z(targ)	remarks									
92	D2163	0.23		-0.93										
150	D2163	0.231		-0.86										
171 323	D2163 D2163	0.229		-1.00										
323 333	EN27941	0.23 0.2		-0.93 -3.03										
334	EN27941	0.221		-1.56										
347	D2163	0.326		5.79										
444	IP405	0.30		3.97										
447	D2163	0.309		4.60										
496	EN27941	0.227		-1.14										
704														
868	D2163	0.218		-1.77										
095	EN27941	0.230		-0.93										
197	D2163	0.243107		-0.02										
198	D2163	0.223588	0(0.04)	-1.38										
348	D2163	0.763	G(0.01)	36.38										
368	D2163	0.236		-0.51										
369 634	D2163 ISO7941	0.243 0.24		-0.02 -0.23										
720	D2163	0.24 1.479	G(0.01)	-0.23 86.50										
120	D2103	1.479	G(0.01)	00.00										
	normality	not OK												
	n	17												
	outliers	2												
	mean (n)	0.2433												
	st.dev. (n)	0.03445												
	R(calc.)	0.0965			_									
		0 0400			Compare		941(lia	))=0.30	0					
4 - 5 - 3 -	R(ASTM D2163)	0.0400				R(EN27				<u> </u>	Δ	Δ		
5 - 3 -	R(ASTM D2163)										Δ	<b>A</b>		
5 - 3 - 5	K(ASTM D2163)		Δ								<u>م</u>	Δ		
5 - 3 - 5 - 2 - <u>A</u> 5 -						Δ	Δ 			44 			348	720
					Δ Δ		5		1197	44 	447	347	1348	1720
5 - 3 - 5 - 2 - 5 - 1 - 8		986 996 •	12	22 Z6	Δ Δ	Δ	Δ 			45 			1348	1720
5 - 3 - 5 - 2 - 5 - 1		986 996 •		22 Z6	Δ Δ	Δ	Δ 			44 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4			1348	1720
5 - 3 - 5 - 1 - 12 -		986 996 •	12	22 Z6	Δ Δ	Δ	Δ 			44 44 			846	1720
5		986 996 •	12	22 Z6	Δ Δ	Δ	Δ 			4 <del>4</del>			84CE	1720
5		986 996 •	12	22 Z6	Δ Δ	Δ	Δ 			49 10 10 10 10 10 10 10 10 10 10 10 10 10			865 1334	11220
5 3 5 2 2 5 5 1 1 8 - 6 - 6 - - - - - - - - - - - - -		986 996 •	12	22 Z6	Δ Δ	Δ	Δ 			45 45 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4			1348	1720

# Determination of Propane; results in %mol/mol

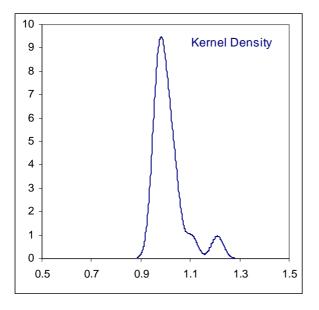
92 [ 150 [ 171 [	method D2163 D2163	value 93.00	mark	z(targ)	
171 [	D2163			0.05	remarks
		93.067		0.25	
323 [	D2163	92.739		-0.73	
JZJ I	D2163	92.96		-0.07	
333 E	EN27941	93.1		0.35	
334 E	EN27941	93.145		0.49	
347 E	D2163	92.801		-0.55	
444 I	IP405	92.83		-0.46	
447 [	D2163	93.535		1.66	
496 E	EN27941	92.689	С	-0.89	first reported 92.091 (not normalised)
704					,
868 E	D2163	92.899		-0.25	
1095 E	EN27941	92.798		-0.56	
1197 E	D2163	93.03868		0.17	
1198 E	D2163	93.05452		0.22	
1348 [	D2163	92.034	G(0.05)	-2.86	
1368 E	D2163	94.775	ex	5.40	did not report quantify and report propylene and n-pentane
1369 E	D2163	94.835	ex	5.58	did not report quantify and report propylene and n-pentane
1634 l	ISO7941	93.09		0.32	
1720 E	D2163	95.172	G(0.01)	6.59	
r	normality	ОК			
	n	15			
(	outliers	2			
r	mean (n)	92.983			
ę	st.dev. (n)	0.2098			
F	R(calc.)	0.587			
F	R(ASTM D2163)	0.930			Compare R(EN27941(liq))=1.009





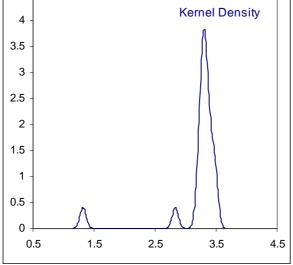
# Determination of Propylene; results in %mol/mol

lab	method	value	mark	z(targ)	remarks	6							
92	D2163	0.98		-0.31									
150	D2163	0.969		-0.57									
171	D2163	0.982		-0.26									
323	D2163	1.01		0.43									
333	EN27941	1.0		0.18									
334	EN27941	0.956		-0.89									
347	D2163	1.210	C,G(0.01)	5.33	first repo	orted 1.150	)						
444	IP405	0.96		-0.80									
447	D2163	1.031		0.94									
496	EN27941	0.973		-0.48									
704	D0100												
868	D2163	0.969		-0.57									
1095	EN27941	0.975		-0.43									
1197 1198	D2163 D2163	1.002546 1.000261		0.25 0.19									
1348	D2163 D2163	1.105	G(0.05)	2.76									
1368	D2103	1.105	G(0.05)	2.70									
1369													
1634	ISO7941	1.04		1.16									
1720	D2163	1.039		1.10									
1720	02100	1.000		1.14									
	normality	OK											
	n	15											
	outliers	2											
	mean (n)	0.9925											
	st.dev. (n)	0.02787											
	R(calc.)	0.0780			-	_ /							
	R(ASTM D2163)	0.1143			Compar	e R(EN27	941(liq))	=0.2020	)				
<sup>1.25</sup> T													
1.2 -													*
1.15													
1.1 +												×	
1.05 -									Δ	۵	Δ		
1					۵	Δ	۵	۵	4				
0.95 -	Δ Δ Δ	Δ 4	<u>م</u>	Δ	Δ								
0.9 +													
0.85 -													
0.8	+ + ~			01	_ ~	~	~	~				~	
	334 444 868	150	1095	92	171 333	1198	1197	323	447	1720	1634	1348	347



# Determination of n-Butane; results in %mol/mol

lab	method	value	mark	z(targ)	rem	arks									
92	D2163	3.32		-0.10											
150	D2163	3.320		-0.10											
171	D2163	3.487		1.11											
323	D2163	3.34		0.04											
333	EN27941	3.3		-0.25											
334	EN27941	3.297		-0.27											
347	D2163	3.244		-0.66											
444	IP405	3.40		0.48											
447	D2163	2.831	G(0.01)	-3.67											
496	EN27941	3.457	-()	0.90											
704	-														
868	D2163	3.386		0.38											
1095	EN27941	3.298		-0.26											
1197	D2163	3.206798		-0.93											
1198	D2163	3.232980		-0.74											
1348	D2163	3.481	С	1.07	first	reporte	ed 1.78	5 (resu	lt mixe	d up wit	h i-hut	ane)			
1368	D2163	3.355	U	0.15	mot	ropond	5a 1.7 0	0 (1000		a ap m		uno)			
1369	D2163	3.298		-0.26											
1634	ISO7941	3.26		-0.54											
1720	D2163	1.318	G(0.01)	-14.71											
1120	52100	1.010	0(0.01)												
	normality	OK													
	n	17													
	outliers	2													
	mean (n)	3.334													
	st.dev. (n)	0.0841													
	R(calc.)	0.235													
	R(ASTM D2163				Con	npare F	R(EN27	941(lia	))=0.39	2					
	,	,				•			,,						
3.8 T															
3.7 -															
3.6															
3.5 -													۵	Δ	Δ
3.4 -											_	Δ			
-							Δ	Δ	Δ	۵	-				
3.3 -			۵	۵	Δ	۵	-	-							
3.2 -	۵	▲ ▲													
3.1 -															
3															
1720	447	347	1634 334	1095	1369	333	150	92	323	1368	868	444	496	1348	171
					٦										
4.5 T															
		l l	Kernel Dei	nsity											
4 -				í											
1			Л		1										



# Determination of iso-Butane; results in %mol/mol

lab	method	value	mark	z(targ)	rem	arks									
92	D2163	1.65	mark	-0.36	- Cill	uno									
150	D2163	1.640		-0.51											
171	D2163	1.708		0.48											
323	D2163	1.65		-0.36											
333	EN27941	1.6		-1.09											
334	EN27941	1.620		-0.80											
347	D2163	1.718		0.62											
444	IP405	1.70		0.36											
447	D2163	1.389	G(0.01)	-4.15											
447	EN27941	1.674	G(0.01)	-4.15											
704	LIN2/941			-0.01											
868	D2163	1.682		0.10											
1095	EN27941	1.766		1.32											
1197	D2163	1.717403		0.62											
1197	D2163 D2163	1.686213		0.82											
1348	D2163 D2163	1.785	С	1.60	firet	roporte	42.40	1 (rooul	t mivo	duowi	th n hu	topo)			
		1.634	C	-0.60	mst	reporte	u 3.40	i (iesu	t mixe	d up wi	un n-bu	lane)			
1368	D2163														
1369	D2163	1.624		-0.74											
1634	ISO7941	1.62	0(0.04)	-0.80											
1720	D2163	0.881	G(0.01)	-11.53											
	normality	OK													
	n	17													
	outliers	2													
	mean (n)	1.675													
	st.dev. (n)	0.0526													
	R(calc.)	0.147			-	_									
	R(ASTM D2163)	0.193			Con	npare F	R(EN27	941 (IIQ	))=0.39	96					
1.8 1.7 1.6 -	Δ	Δ Δ	<u>م</u> ۵	Δ	Δ	Δ				<u>A</u>	<u> </u>	<u> </u>	Δ	Δ	
1.5 -															
1.4	*														
1720	447 333	334 1634	1369	150	323	92	496	868	1198	<del>4</del> 4 4	171	1197	347	1095	1348
<u> </u>												•			
					-										
6															
		L	kernel/De	ncity											
		r	remeiline	nsity											
5 -															
			11												
4 -															
4 1															
3 -															
2 -															
1 -															
			1	1 1	1										



1

1.5

2

0 ↓ 0.5

# Determination of n-Pentane; results in %mol/mol

lab	method	value	mark	z(targ)	remarks								
92	D2163	0.80	IIIdi K	-0.52	Telliars								
150	D2163	0.773		-1.32									
171	D2163	0.847		0.88									
323	D2163	0.81		-0.22									
333	EN27941	0.8		-0.52									
334 347	EN27941 D2163	0.761 0.705		-1.68 -3.34									
444	IP405	0.705		-0.22									
447	D2163	0.906		2.64									
496	EN27941	0.914		2.88									
704													
868	D2163	0.845		0.82									
1095 1197	EN27941 D2163	0.932 0.791465		3.41 -0.77									
1198	D2163	0.802439		-0.44									
1348	D2163	0.831		0.41									
1368													
1369													
1634 1720	ISO7941 D2163	0.75 0.110	G(0.01)	-2.00 -21.05									
	normality	OK 16											
	n outliers	10											
	mean (n)	0.8174											
	st.dev. (n)	0.06114											
	R(calc.) R(ASTM D2163)	0.1712 0.0941			Compare I		(11/lia)	0 222					
	R(ASTIN D2103)	0.0941			Compare i		/4 I (IIQ))=	=0.323					
0.85 -									Δ	▲			
0.8			Δ	۵	Δ Δ	۵	۵	۵	-				
0.8 - 0.75 -	<b>_</b>	Δ	۵	<b>Δ</b>	Δ Δ	<b>A</b>		<u> </u>					
0.75 -		<b>A</b>	<u>م</u> 	Δ	Δ Δ								
0.75 - 0.7 - 0.65 -	<u>_</u>	<u>A</u>	<u>م</u> م	<b>A</b>	Δ Δ	Δ	Δ 	<u> </u>					
0.75 - 0.7 - 0.65 - 0.6		45 	۵ 	<b>۵</b>	333	<b>۵</b> 	444  	348		171	447	8 9 9 9	1095
0.75 - 0.7 - 0.65 - 0.6		4 4 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7	۵ 							171	447	64 66 6	1085
0.75 - 0.7 - 0.65 - 0.6			001 2011 2011	8						121	447	0 9 9	
0.75 - 0.7 - 0.65 - 0.6			۵ 	8						171	447		1085
0.75 - 0.7 - 0.65 - 0.6			001 2011 2011	8						12	447	964	1095
0.75 - 0.7 - 0.65 - 0.6			001 2011 2011	8						121	447	66	1095
0.75 - 0.7 - 0.65 - 0.6 -			001 2011 2011	8						121	447	66	1035
0.75 - 0.7 - 0.65 - 0.6			001 2011 2011	8						121	447	69	1085
0.75 - 0.7 - 0.65 - 0.6 7 - 6 - 5 -			001 2011 2011	8						12	447	6	1085
0.75 - 0.7 - 0.65 - 0.6 -			001 2011 2011	8							447	64 66	1095
0.75 - 0.7 - 0.65 - 0.6 7 - 6 - 5 -			001 2011 2011	8						12	447	9 9 9	1095
0.75 - 0.7 - 0.65 - 0.6 7 - 6 - 5 -			001 2011 2011	8						121	447	<u>0</u>	1085
0.75 - 0.7 - 0.65 - 0.6 - 6 - 5 - 4 -			001 2011 2011	8							447	0 9	1085
0.75 - 0.7 - 0.65 - 0.6 - 6 - 5 - 4 -			001 2011 2011	8						121	447		1085
0.75 - 0.7 - 0.65 - 0.6 - 5 - 4 - 3 -			001 2011 2011	8						121	447		1085
0.75 - 0.7 - 0.65 - 0.6 7 - 6 - 5 - 4 - 3 - 2 -			001 2011 2011	8						121	447	90	1085
0.75 - 0.7 - 0.65 - 0.6 - 5 - 4 - 3 -			001 2011 2011	8						121	447	90	1085
0.75 - 0.7 - 0.65 - 0.6 7 - 6 - 5 - 4 - 3 - 2 -			001 2011 2011	8						131	447	98	

0.7

0.5

0.9

1.1

# Determination of 1-Butene; results in %mol/mol

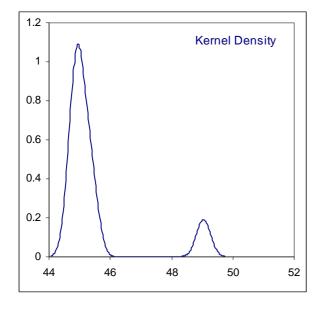
					-	
lab	method	value	mark	z(targ)	remarks	
92						
150	D2163	<0.001				
171	D2163	0.001				
323	D2163	<0.01				
333	EN27941	<0.1				
334	EN27941	0				
347						
444	IP405	<0.0010				
447	D2163	<0.01				
496	EN27941	0.001				
704						
868	D2163	<0.01				
1095	EN27941	0.000				
1197						
1198						
1348	D2163	0				
1368						
1369						
1634	ISO7941	0.00				
1720	D2163	0.00				
	normality	n.a.				
	n	7				
	outliers	0				
	mean (n)	<0.01				
	st.dev. (n)	n.a.				
	R(calc.)	n.a.				
	R(ASTM D2163)	n.a.				

### Determination of iso-Butene; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	method		mark		Tomarko
150	D2163	<0.001			
171	D2163	0.001			
323	D2163	<0.01			
333	EN27941	<0.1			
334	EN27941	0			
347					
444	IP405	<0.0010			
447	D2163	<0.01			
496	EN27941	0.001			
704	_				
868	D2163	<0.01			
1095	EN27941	0.000			
1197					
1198	D0400				
1348	D2163	0			
1368					
1369 1634	ISO7941	0.00			
1720	D2163	0.00			
1720	D2103	0.00			
	normality	n.a.			
	n	7			
	outliers	0			
	mean (n)	<0.01			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(ASTM D2163)	n.a.			
	(				

# Determination of Molar Mass; results in g/mol

lab	method	value	mark	z(targ)	remarks
92	INH-2145	45.0			
150	0410		-		colo huijo novo 45.00
171 323	CALC D3588	45.38 45	E		calc. by iis gave 45.03
333	D3366	45			
334					
347	D2421	44.9246			
444					
447					
496	CALC	45.381	E		calc. by iis gave 45.020
704	Docoo				
868	D2598	44.99			
1095 1197					
1197					
1348	CALC	49.041	E,G(0.01)		calc. by iis gave 44.964
1368	D2598	44.763	_,0(0.01)		
1369	D2598	44.752			
1634					
1720					
					Calculated by iis from all reported test results:
	normality	not OK			OK
	n outlioro	6 1			15 2
	outliers mean (n)	ı 44.905			2 44.984
	st.dev. (n)	0.1177			0.03444
	R(calc.)	0.329			0.0964
	R(lit)	unknown			unknown
	R(iis09S03P)	0.417			0.0397 (for comparison)
<sup>50</sup> T					
49 -					*
48 -					
47 -					
47 -					
46 -					
					ж ж
45 —	A		Δ	Δ	<u> </u>
	4	Δ			
44	1369	1368	347	868	92 171 1348 1348
	ţ <u></u>	ā.	ń	8	



# Determination of Relative Density @ 60F; unitless results

lab	method	value	mark	z(targ)	remarks						
92	D2598	0.512	iiidi K	z(targ)	Temarks						
150	D2598 D2598	0.512									
171	D2598	0.51267									
323	D2598	0.513	E		calc. by ii	s gave 0	.512 (0.51	240)			
333						U	,	,			
334											
347	D2598	0.5118									
444											
447	Docoo										
496	D2598	0.5128									
704 868	D2598	0.5126									
1095	D2530										
1197											
1198											
1348	D2598	0.512									
1368	D2598	0.51131									
1369	D2598	0.51125									
1634	ISO8973	0.5123				_					
1720	D2598	0.5068	E,G(0.01)		calc. by ii	s gave 0	0.5072				
	normality	ОК			Calculate OK	a by lis f	from all repo	orted test re	esuits:		
	n	10			16						
	outliers	1			1						
	mean (n)	0.5121			0.5123						
	st.dev. (n)	0.00054			0.00024						
	R(calc.)	0.0015			0.0007						
	R(lit)	unknown			unknown						
	R(iis09S03P)	0.0005			0.0003	(for con	nparison)				
0.514 T											
0.513 -									۵	۵	*
0.512 -				Δ	Δ	Δ	Δ	Δ			
	۵	۵	۵								
0.511 -											
0.51 -											
0.509 -											
0.508 -											
0.507 -	ж										
0.506											
	1720	1368	347	1348	32	1634	150	868	171	496	323
					1						
500 -											
450 -	-	ł	Kernel D <mark>/e</mark> n:	sity							
400 -	-										
350 -											
350 -											
300 -	-										
			<pre>{</pre>								
250 -	-										
200 -											
200 -											
150 -	-										
				1							
100 -	1										

0.505

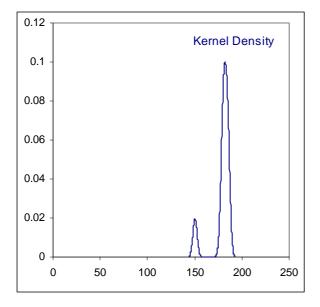
0.51

0.515

50 -0 -0.5

# Determination of Absolute Vapour Pressure @ 100F; results in psi

lab	method	value	mark	z(targ)	remarks
92	D2598	150.4	E,C,G(0.05)		calc. by iis gave 184.7; first reported 165.0
150	D2598	182.7	С		first reported 166.3
171	D2598	180.7			
323					
333	ISO8973	185			
334					
347	D2598	181.5			
444					
447					
496	D2598	180.21			
704					
868	D2598	185.9			
1095	ISO8973	184.227			
1197					
1198					
1348					
1368					
1369					
1634	B		_		
1720	D2598	178.6	E		calc. by iis gave 196.8
		<b></b>			Calculated by iis from all reported test results:
	normality	OK			OK
	n	7			13
	outliers	1			4
	mean (n)	182.891			184.756
	st.dev. (n)	2.2078			0.3180
	R(calc.)	6.181			0.891
	R(lit)	unknown			unknown
<sup>188</sup> T					
186 -					Δ
184 -					Δ Δ
					Δ
182 -				۵	Δ
180 -			Δ	•	
178 -		ж			
176 -					
174 -					
172 -					
170					
	92	1720	496	171	347 160 1095 868
L					



# Determination of Relative Vapour Pressure @ 100F; results in psi

lab	method	value	mark	z(targ)	remark	(S					
92											
150	D2598	167.8									
171	D2598	165.8									
323	D2598	166									
333	ISO8973	170									
334											
347	D2598	166.8									
444											
447											
496	D2598	165.52									
704											
868	D2598	168.2									
1095	ISO8973	169.531									
1197											
1198											
1348	D2598	167	_								
1368	D2598	167.9	С			oorted 167.9					
1369	D2598	168.1	С		first rep	orted 168.1	l as abs. va	apour press	sure		
1634	ISO8973	168.389	С		first rep	orted 175.4	196 at 40°C	C (= 104F)			
1720											
	normality	OK									
	n	12									
	outliers	0									
	mean (n)	167.587									
	st.dev. (n)	1.4134									
	R(calc.)	3.958									
	R(lit)	unknown									
172 <sub>T</sub>											
170 -											▲
										۵	
168 -					•	۵	Δ	Δ	۵		
			Δ	۵							
166 -		۵	-								
	<u>۸</u>										
164 -											
162 -											
160											
	496	323	347	1348	150	1368	1369	868	1634	1095	333
				,					,		
0.0					٦						
0.3											
		1	Kernel De	ensity							
0.25	-	$\wedge$									
0.2	-	1									
		$\int$									
0.15		1	L .								

0.2 0.15 0.15 0.1 0.05 0 160 165 170 175

# Type of sampling used

	sample volume	type of vaporizer	Other imp.	Cylinder nr.	Weight bef ore analysis
92			iso-pentane (trace amount)	7500383	
150				7500441	
171			0.0027%M/M iso-pentane 0.0008%M/M 1,3-butadiene 0.0009%M/M neo-pentane	7500443	5823
323	96 g	Liquid injection	no	7500444	5720
333			no		5722
334			no	7500411	4672
347	1 ml	on-line heated vapourizing device	yes, but <0.01% M/M	7500402	5600
444			0.0026%M/M iso-pentane	7500438	6000
447					
496			0.059%M/M Helium 0.003%M/M cyclopentane 0.002%M/M cis-2-butene 0.004%M/M trans-2-butene	7500440	5655
868	200 ml	Liquid injection	no	7500381	5674.2
1095	1 ml	liquid fase vaporized before injection		not readable	5654.6
1197		GSV	no	7500392	5690
1198		GSV	no	7500396	5670
1348			no	7500433	5715
1368			no		
1369			no		
1634			no	7500405	5684
1720	500 ml	500	no	7500404	5650

### Number of participants per country

2 labs in AUSTRALIA

- 1 lab in BELGIUM
- 1 lab in CANADA
- 2 labs in FRANCE
- 1 lab in GERMANY
- 1 lab in LEBANON
- 2 labs in MALAYSIA
- 1 lab in P.R. of CHINA
- 2 labs in PORTUGAL
- 1 lab in SPAIN
- 1 lab in SUDAN
- 2 labs in U.S.A.
- 1 lab in UKRAINE
- 2 labs in UNITED KINGDOM

#### Abbreviations:

С	= final result after checking of first reported suspect result
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- 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
- n/a = not applicable
- W = withdrawn on request participant
- U = reported in wrong unit
- E = error in calculations
- SDS = Safety Data Sheet

### Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation, January 2010
- 2 prNEN 12766-2:2000.
- 3 ASTM E178-89
- 4 ASTM E1301-89
- 5 ISO 5725-86
- 6 ISO 5725, parts 1-6, 1994
- 7 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, First reported Z. Anal. Chem, <u>331</u>, 513, (1988)
- 12 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 13 Analytical Methods Committee Technical Brief, No4 January 2001
- 14 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/)
- 15 ISO 17043
- 16 EN 27941
- 17 ASTM D2163
- 18 ASTM D2421