Results of Proficiency Test Liquefied Propane November 2011

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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 2011, in which now 30 laboratories from 20 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 31 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.

A batch of 31 cylinders (lot 86114) was prepared on October 10, 2011. Regretfully this batch was rejected as the between cylinders spread was too large to allow use in the PT. Therefore a new batch of 31 cylinders (lot 86114R) was prepared on October 27, 2011. Each cylinder was labelled #11095 and also uniquely numbered. The cylinders were all tested in fivefold 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	0.1183	0.0039	0.0041
Propane	95.4488	0.0667	0.2863
Propylene	0.7782	0.0042	0.0269
n-Butane	1.0491	0.0404	0.0711
iso-Butane	2.0574	0.0173	0.0362
n-Pentane	0.5482	0.0163	0.0191
1-Butene	n/a	n/a	n/a
iso-Butene	n/a	n/a	n/a

Table 1: homogeneity test results

Each of the calculated repeatabilities is less than 0.3 times the corresponding reproducibility of the reference method ASTM D2163.

Therefore, homogeneity of the subsamples #11095 was assumed.

To each of the participating laboratories one 2L cylinder was sent on November 10, 2011.

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 compostion: Molar Mass, Relative Density @60F and Absolute and Relative Vapour pressure @100F. 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 seven laboratories reported the test results after the final reporting date. Not all laboratories did report all test results requested.

In total 28 participating laboratories reported 231 numerical test results. Observed were 19 outlying test results, which is 8.2% 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 absolute vapor pressure 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 (estimated from figure 3) were used.

Because 21 laboratories reported both results in %mol/mol as well as in %M/M, it has been possible to check the calculations of these 21 laboratories. A good correlation between the results reported in %mol/mol and the results reported in %M/M was found, as 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. However, the calculated reproducibility is in agreement with the much less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).
- <u>Propane:</u> Analytical problems were observed for a small number of laboratories. Two statistical outliers were detected. However, the calculated reproducibility, after exclusion of the two 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>Propylene:</u>	Analytical problems were observed for a number of laboratories. Three statistical outliers were detected. However, the calculated reproducibility, after exclusion of the statistical outliers, is in 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>	Analytical problems were observed for a number of laboratories. Five 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>iso-Butane:</u>	The determination of this component may be problematic. Two statistical outliers were detected. The calculated reproducibility, after exclusion of the statistical outliers, is not in agreement with the requirements of ASTM D2163:96. However, the calculated reproducibility is in agreement with the much less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).
<u>n-Pentane:</u>	The determination of this component was problematic. Four statistical outliers were detected. The calculated reproducibility, after exclusion of the statistical outliers, is not at all in agreement with the requirements of ASTM D2163:96. It is however almost 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>lso-Butene:</u>	This component was not present in a detectable concentration. Therefore no conclusions could be drawn.
<u>Molar Mass:</u>	This calculated parameter may be not problematic. The results vary over a range from 44.6 – 44.796. No statistically significant outliers were present. See also the discussion in 4.3.
<u>Rel. Density @60F:</u>	This calculated parameter may be problematic. The results vary over a range from 0.50805 – 0.511 and one statistically significant outlier was present (in 20 test results). Probably 5 laboratories reported the relative density @15°C, as IP432 or ISO8973 were used, methods that use 15°C in stead of 60F. However, the difference in relative density between 15°C and 60F is less than 0.0001 and therefore this cannot explain for the observed spread.

- Abs. Vapour Press.:This calculated parameter may be problematic. The results vary over a
large range (183 187.27 psi). No statistically significant outliers were
observed. Two participants may have made a calculation error.
The data appear to be bimodally distributed. This suggests that two
diffent calculation procedures were used by the participants. All
ISO8973/IP432 results except one are higher than all ASTM D2598
results. See also the discussion in 4.3.
- Rel. Vapour Press.:This calculated parameter may be problematic. The results vary over a
large range (168 172.57 psi). No statistically significant outliers were
observed. Two participants may have made a calculation error.
The data appear to be bimodally distributed. This suggests that two
diffent calculation procedures were used by the participants. All
ISO8973/IP432 results except one are higher than all ASTM D2598
results. 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. 2.8 * sd value		R(D2163) in %mol	R(EN27941) liqinj. in %mol	R(EN27941) liqinj. in %M/M
Ethane	%mol/mol	26	0.120	0.043	0.014	0.296	0.2
Propane	%mol/mol	26	95.368	0.668	0.954	1.013	1
Propylene	%mol/mol	25	0.788	<u>0.102</u>	0.090	0.213	0.2
n-Butane	%mol/mol	23	2.065	0.149	0.239	0.382	0.5
iso-Butane	%mol/mol	26	1.089	<u>0.145</u>	0.126	0.385	0.5
n-Pentane	%mol/mol	24	0.538	<u>0.122</u>	0.062	0.115	0.5
Molar Mass	g/mol	15	44.67	0.14	n/a	n/a	n/a
Rel. Density @60F		19	0.5106	0.0008	n/a	n/a	n/a
Abs. Vapour press.	psi	14	see	§4.3	n/a	n/a	n/a
Rel. Vapour press.	psi	15	see	§4.3	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	0.118	0.120	+0.002	+0.37
Propane	95.449	95.368	-0.081	-0.24
Propylene	0.778	0.788	+0.009	+0.29
n-Butane	2.057	2.065	+0.007	+0.09
iso-Butane	1.049	1.089	+0.040	+0.90
n-Pentane	0.548	0.538	-0.010	-0.46

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 seven laboratories reported the presence of one or more additional components (isopentane (0.0003-0.02 %mol/mol, cyclopropane 0.03%M/M). Probably these components were present as impurity in one or more of the pure components that were used to prepare the propane mixture.

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 striking differences between the test results as reported by the participating laboratories for absolute and relative vapour pressure.

For the calculation of the Vapour Pressure two different methods were used. Nine participants used ISO8973/IP432 and also nine participants used ASTM D2598. In ISO 8973 (identical to IP432) the <u>Absolute</u> Vapour Pressure is calculated from the <u>mole fraction</u> per component and a Vapour Pressure factor of that component (given for all components). From the Absolute Vapour Pressure the Relative Vapour Pressure is calculated.

In ASTM D2598 the Gage pressure (identical to the <u>Relative</u> Vapour Pressure) is calculated from the <u>liquid volume percentage</u> per component and a Vapour Pressure factor of that component (not given for n-pentane). From the Relative Vapour Pressure the Absolute Vapour Pressure is calculated.

As one would expect to find identical values from both calculation methods, it is remarkable to see that the results from the ASTM D2598 calculation are approx 3.3 psi lower than the results from the ISO8973/IP432 calculation. The reason is yet unknown.

APPENDIX 1

Deteri	mination of Et	hane; res	ults in %	mol/mo	
lab	method	value	mark	z(targ)	remarks
92	D2163	0.11		-2.04	
150	D2598	0.123		0.59	
171	D2163	0.359	G(0.01)	48.30	
317	D2163	0.12		-0.02	
323	D2163	0.12		-0.02	
334	EN27941	0.09		-6.09	
347	D2163	0.128		1.60	
444	IP405	0.101		-3.86	
445	IP405	0.09		-6.09	
447	EN107044				
496	EN27941	0.125		0.99	
56Z	D2163	0.10		-4.06	
704	D2163	0.115		-1.03	
1011	D2103 EN27044	0.114	C(0.01)	-1.20	
1011	LINZ/941	0.30	G(0.01)	0 00	
1010	DIN51610	0.123		0.33	
1040	DINGTOTS	0.122	W	0.50	results withdrawn
1108	D2163	0 111	••	-1 84	
1191	IP473	0.120		-0.02	
1197	D2163	0.127		1.40	
1198	D2163	0.121		0.18	
1259	EN27941	0.117		-0.63	
1284					
1335	D2163	0.1202		0.02	
1378	ISO7941	0.16		8.07	
1391	D2163	0.127		1.40	
1427	ISO7941	0.122		0.38	
1469	D2163	0.1234		0.67	
1634	ISO7941	0.15		6.05	
1810	EN27941	0.141	С	4.23	reported 0.095 %V/V
	normality	not OK			
	n autliana	26			
	outliers	2			
	mean (n)	0.120			
	P(colc)	0.0100			
	R(D2163.96)	0.043			Compare R/EN/27941(lig))-0.296
	N(D2100.00)	0.014			





Determination of Propane; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	95.28		-0.26	
150	D2598	95.281		-0.26	
171	D2163	97.406	G(0.05)	5.98	
317	D2163	95.48	· · · ·	0.33	
323	D2163	95.36		-0.02	
334	EN27941	96.00		1.86	
347	D2163	95.457		0.26	
444	IP405	94.688		-2.00	
445	IP405	95.36		-0.02	
447					
496	EN27941	95.555		0.55	
562	D2163	94.94		-1.26	
704	D2163	95.456		0.26	
868	D2163	95.212		-0.46	
1011	EN27941	97.32	G(0.01)	5.73	
1016	ISO7941	95.055	-()	-0.92	
1040	DIN51619	95.556		0.55	
1095			W		results withdrawn
1108	D2163	95.437		0.20	
1191	IP473	95.501		0.39	
1197	D2163	95.392		0.07	
1198	D2163	95.340		-0.08	
1259	FN27941	95.650		0.83	
1284					
1335	D2163	95,4355		0.20	
1378	ISO7941	95.41		0.12	
1391	D2163	95 244		-0.36	
1427	ISO7941	95.328		-0.12	
1469	D2163	95,2989		-0.20	
1634	ISO7941	95.39		0.06	
1810	EN27941	95.460	С	0.27	reported 94.289 %V/V
	-		-	-	
	normality	OK			
	n	26			
	outliers	2			
	mean (n)	95.368			
	st.dev. (n)	0.238			
	R(calc.)	0.668			
	R(D2163:96)	0.954			Compare R(EN27941(lig))=1.013
	· · · · ·				
98 _T					
97.5					
07					x ^x
9/					
96.5 + -					
96 + -					ζ





Determination of Propylene; results in %mol/mol

lab	method	value	mark	z(targ)	rei	mark	s													
92	D2163	0.73		-1.78																
150	D2598	0.863		2.34																
171	D2163	0.941	DG(0.05)	4.75																
317	D2163	0.75		-1.16																
323	D2163	0.82		1.01																
334	EN27941	0.64	G(0.05)	-4.57																
347	D2163	0.783		-0.14																
444	IP405	0.812		0.76																
445	IP405	0.76		-0.85																
447																				
496	EN27941	0.804		0.51																
562	D2163	0.82		1.01																
704	D2163	0.754		-1.04																
868	D2163	0.774		-0.42																
1011	EN27941	0.94	DG(0.05)	4.72																
1016	ISO7941	0.801		0.42																
1040	DIN51619	0.787		-0.02																
1095			W		res	sults	with	draw	n											
1108	D2163	0.767		-0.64																
1191	IP473	0.755		-1.01																
1197	D2163	0.787		-0.02																
1198	D2163	0.815		0.85																
1259	EN27941	0.713		-2.31																
1204	D0160	0 7700		0.00																
1333	D2103	0.7762		-0.29																
1301	D2163	0.02		-0.17																
1/27	15070/1	0.782		-0.17																
1427	D2163	0.762		0.17																
1634	1907941	0.7502		2 55																
1810	FN27941	0.07	C	-0.51	rer	orte	d 0 7	727 9	<u>~</u> //\	/										
1010		0.771	Ũ	0.01	101	50110	u 0.1	21 /	0070											
	normality	ОК																		
	n	25																		
	outliers	3																		
	mean (n)	0.788																		
	st.dev. (n)	0.0364																		
	R(calc.)	0.102																		
	R(D2163:96)	0.090			Co	mpa	re R	(EN2	2794	1(liq))=0.2	213								
1 T																				
0.95 -																			*	*
0.9 -																				
-																		4		
0.85																				
0.8 -										۵	۵	Δ	Δ	-						
0.75	•	Δ Δ Δ	<u>م</u> م	ΔΔ	Δ	Δ	-	-												
0.7 + -																				
0.65																				
	259 92 317	704 191 445	108 310 368	335 427	391	347	197	040	469	J16	496	444	198	378	562	323	150	334	111	171
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Determination of n-Butane; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	2.05		-0.17	
150	D2598	2.090		0.30	
171	D2163	0.725	G(0.01)	-15.72	
317	D2163	2.04		-0.29	
323	D2163	2.07		0.06	
334	EN27941	1.82	D(0.05)	-2.87	
347	D2163	2.028		-0.43	
444	IP405	2.419	G(0.05)	4.16	
445	IP405	2.15		1.00	
447					
496	EN27941	2.000		-0.76	
562	D2163	2.33	D(0.05)	3.11	
704	D2163	2.053		-0.14	
868	D2163	2.164		1.16	
1011	EN27941	0.82	G(0.01)	-14.61	
1016	ISO7941	2.142		0.91	
1040	DIN51619	1.966		-1.16	
1095	B a <i>i</i> a a		VV		results withdrawn
1108	D2163	2.091		0.31	
1191	IP473	2.024		-0.48	
1197	D2163	2.042		-0.27	
1198	D2163	2.040		-0.29	
1259	EN27941	1.994		-0.83	
1204	D0160	2.0492	6	0.00	first reported 4 0000, result mixed up with i hutans
1220	1907041	2.0402	C	-0.20	nist reported 1.0090, result mixed up with i-butane
1301	D2163	2.05		1 03	
1427	1907941	2.100		0.46	
1469	D2163	2 1125		0.40	
1634	IS07941	2.04		-0.29	
1810	EN27941	2.060	С	-0.06	reported 2.681 %V/V
			-		
	normality	OK			
	n	23			
	outliers	5			
	mean (n)	2.0649			
	st.dev. (n)	0.05318			
	R(calc.)	0.1489			
	R(D2163:96)	0.2386			Compare R(EN27941(liq))=0.3815
^{2.5} T					
24					×
					×.





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

lab	method	value	mark	z(targ)	remarks	
92	D2163	1.13		0.91		
150	D2598	1.087		-0.05		
171	D2163	0.493	G(0.01)	-13.30		
317	D2163	1.06	. /	-0.65		
323	D2163	1.08		-0.21		
334	EN27941	0.93		-3.55		
347	D2163	1.062		-0.61		
444	IP405	1.217		2.85		
445	IP405	1.13		0.91		
447						
496	EN27941	1.061		-0.63		
562	D2163	1.17		1.80		
704	D2163	1.063		-0.59		
868	D2163	1.133		0.97		
1011	EN27941	0.52	G(0.01)	-12.70		
1016	ISO7941	1.109		0.44		
1040	DIN51619	1.084		-0.12		
1095			W		results withdrawn	
1108	D2163	1.078		-0.25		
1191	IP473	1.048		-0.92		
1197	D2163	1.088		-0.03		
1198	D2163	1.123		0.75		
1259	EN27941	1.022		-1.50		
1284						
1335	D2163	1.0690	С	-0.45	first reported 2.0482, result mixed up with n-butane	
1378	ISO7941	1.07		-0.43		
1391	D2163	1.112		0.51		
1427	ISO7941	1.104		0.33		
1469	D2163	1.0993		0.22		
1634	ISO7941	1.08		-0.21		
1810	EN27941	1.112	С	0.51	reported 1.448 %V/V	
	normality/	OK				
	normality	26				
	outliere	20				
	mean (n)	1 0803				
	st dev (n)	0.05167				
	R(calc.)	0.03107				
	R(D2163.96)	0.1256			Compare R/EN27941/lig))=0 3849	
	N(B2100.00)	0.1200				
4.0						
I.3 T						
1.2					X	. – – –
1.1						
	<u>م</u>	ΔΔ		Δ Δ		
0.9 -	-					
0.8 -						
0.7						
0.6 -						
0.5 - *	*					
0.4						
171	1011 334 1259 1191	317	347 704 1335	1378	322 1634 1640 1040 1197 1197 1196 1196 1196 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1198 1016 1197 1016 1197 1016 1197 1016 1197 1016 1016 1016 1016 1016 1016 1016 101	444



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

lab	method	value	mark	z(targ)	remarks
92	D2163	0.57		1.44	
150	D2598	0.555		0.76	
171	D2163	0.076	G(0.05)	-20.74	
317	D2163	0.55		0.54	
323	D2163	0.55		0.54	
334	EN27941	0.52		-0.81	
347	D2163	0.543		0.22	
444	IP405	0.759	G(0.01)	9.92	
445	IP405	0.50	. ,	-1.71	
447					
496	EN27941	0.453		-3.82	
562	D2163	0.63		4.13	
704	D2163	0.556		0.81	
868	D2163	0.602		2.87	
1011	EN27941	0.096	G(0.01)	-19.84	
1016	ISO7941	0.767	G(0.05)	10.28	
1040	DIN51619	0.485	- ()	-2.38	
1095			W		results withdrawn
1108	D2163	0.514		-1.08	
1191	IP473	0.551		0.58	
1197	D2163	0.561		1.03	
1198	D2163	0.558		0.90	
1259	FN27941	0.502		-1.62	
1284					
1335	D2163	0.5485		0.47	
1378	ISO7941	0.51		-1.26	
1391	D2163	0.582		1.97	
1427	ISO7941	0.558		0.90	
1469	D2163	0.5738		1.61	
1634	ISO7941	0.49		-2.16	
1810	EN27941	0.450	С	-3.95	reported 0.757 %V/V
	normality	OK			
	n	24			
	outliers	4			
	mean (n)	0.5380			
	st.dev. (n)	0.04366			
	R(calc.)	0.1222			
	R(D2163:96)	0.0624			Compare R(EN27941(liq))=0.1153
0.9 T					
0.8 -					v X
0.7 -					m.
0.6					Δ
0.5			· · · ·	A	
0.3 =	= = = = = = = = = = = = = =				
0.4					
0.3 -					
0.2					





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

lah	method	value	mark	z(tard)	romarks
92	methou		Παικ	2(laig)	Temarka
150					
171	D2163	<0.01			
317	D2163	< 0.01			
323	D2163	<0.01			
334	EN27941	<0.1			
347					
444	IP405	<0.01			
445	IP405	<0.01			
447					
496	EN27941	<0.001			
562	D2163	0.00			
704	D2163	< 0.01			
808	D2163	<0.01			
1011	EN27941	<0.1			
1010	ISU7941 DINE1610	<0.01			
1040	DINSTOTS	0	۱۸/		results withdrawn
1108	D2163	n d	vv		
1100	IP473	0.000			
1197	11 470				
1198					
1259	EN27941	0.0			
1284					
1335					
1378					
1391	D2163	<0.01			
1427					
1469	D2163	0.0000			
1634	ISO7941	0.00			
1810	EN27941	0.0001	С		reported 0.0002 %V/V
	normality	na			
	n	7			
	outliers	0			
	mean (n)	0.00001			
	st.dev. (n)	0.00004			
	R(calc.)	0.00011			
	R(D2163:96)	n.a.			

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

				4.	
lab	method	value	mark	z(targ)	remarks
92					
150	50/00				
171	D2163	<0.01			
317	D2163	<0.01			
323	D2163	<0.01			
334	EN27941	<0.1			
347					
444	IP405	<0.01			
445	IP405	<0.01			
447					
496	EN27941	<0.001			
562	D2163	0.00			
704	D2163	<0.01			
868	D2163	<0.01			
1011	EN27941	<0.1			
1016	ISO7941	<0.01			
1040	DIN51619	0			
1095			W		results withdrawn
1108	D2163	n.d.			
1191	IP473	0.000			
1197					
1198					
1259	EN27941	0.0			
1284					
1335					
1378	_				
1391	D2163	<0.01			
1427	_				
1469	D2163	0.0000			
1634	ISO7941	0.00	_		
1810	EN27941	0.0001	С		reported 0.0001 %V/V
	normality	n.a.			
	n	7			
	outliers	0			
	mean (n)	0.00001			
	st.dev. (n)	0.00004			
	R(calc.)	0.00011			
	R(D2163:96)	n.a.			
	. ,				

Determination of Molar Mass; results in g/mol

lah	method	value	mark	z(taro) rema	rke							
02	CDA214F	116	mark	2(10)9		ana							
9Z 150	GFAZ145	44.0			-								
150					-								
217	D2509	44.66			-								
202	D2590	44.00			-								
323	D3300	44.7			-								
247	D2424	 11 GEE			-								
347	DZ4Z1	44.000			-								
444	ID422	44 670			-								
445	11 432	44.070			_								
496	D2421	44 623			-								
562	DZHZI	44 73			-								
704		44,6593			-								
868	calc.	44.70			-								
1011					-								
1016					-								
1040					-								
1095			W		- resul	t withdra	wn						
1108	D2421	44.62			-								
1191	ISO6976	44.65	С		 first r 	eported	36.52						
1197					-								
1198					-								
1259	ISO8973	44.630			-								
1284					-								
1335	D2598	44.655			-								
1378	ISO8973	44.63			-								
1391					-								
1427	_				-								
1469	D2421	44.796			-								
1634					-								
1810					-								
					Calci	ulated by	us fron	n all repoi	ted test	results:			
	normality	UK 45			OK								
	n outliere	15			23								
	outliers	0			C 2	26							
	mean (n)	44.665			44.60	00							
	SLOEV. (II)	0.0499			0.023	53							
	R(Calc.)	0.140			0.000								
						OWN S (for oor	nnoriaa	n)					
	R(11510503P)	0.329			0.090		npanso	(1)					
44.85 T													
14.9													
44.0 -													Δ
44.75 -													
												Δ	
44.7 -										۵	Δ		
14.6E					A	•	Δ	Δ	4				
44.05 -		Δ	۵	Δ	-	-							
44.6 -	Δ	4											
44.55 -													
44.5													
44.5	08 92	20 00	78	91	35	47	04	17	45	89	23	62	69
		4 5	13	7	13	e	7	e	4	æ	e	a.	4
10			14				1						
10	reporte	d test results	14		calc h	oviis							
9 -	٨		12 -		λ	Jy 113							
8 -	{	{											
7 -		{	10 -										
6 -			8										
5 -		}											
4		$\left\{ \right\}$	6 -										
	}												
3-	l I		4 -										
		1 1											
2 -			2										
2 - 1 -		\backslash	2 -	\cap		\wedge							
2 - 1 - 0		4	2 - 0	<u> </u>	Jh	\wedge							

Determination of Relative Density @60F; unitless results

lab	mothod			mark		z(tara)		marke										
	D2509	<u> </u>		mark		z(targ)	re	marks										
92 150	D2598		5100															
171	D2421	(0.50805	G(0.0	1)													
317	D2421	Ċ).5103	0(0.0	• /													
323	D2598	C).5107															
334		-																
347	D2598	C).5103															
444	10 400	-		•														
445	IP432	C	0.5107	C														
447 496	D2598	-	51047															
562	D2598	0).511															
704	D2598	Ċ).5107															
868	D2598	C).5109															
1011		-																
1016		-																
1040		-																
11095	D2598	-	5106															
1191	D2598	(0.5106															
1197		_																
1198		-																
1259	ISO8973	C).510	С														
1284	B	-																
1335	D2598	(0.5107															
13/0	1506973 D2598		0.5109															
1427	ISO8973	().511															
1469	D2598	Ċ	0.5108															
1634	ISO8973	C).5105															
1810		-																
							<u>c</u>	alculate	d by	iis from	all rep	orted t	est res	ults:	Ide	m for '	<u>15°C</u>	
	normality						0	ĸ							0K			
	outliers	1	9				2. 5	3							23 5			
	mean (n)	Ċ	.51062				0	51065							0.5	1059		
	st.dev. (n)	Ċ	0.000273	5			0.	000129)						0.0	00129		
	R(calc.)	C	0.00076				0.	00036							0.0	0036		
	R(lit)	ι	Inknown				u	nknown							unł	known		
	R(iis10S03P)) (0.00150				0.	00070	(for (compar	ison)				0.0	0070		
[
0.5111																	Δ	Δ
0.5109 -															Δ	Δ		
0.5107 -									Δ	Δ	_	Δ	Δ	۵				
						Δ	Δ	Δ										
0.5105 -				Δ	Δ													
0.5103 -		. ▲	۵															
0.5101 -	4	7																
0.5000	۵																	
0.5035 -																		
0.5097 -																		
0.5095					-										~			
	171	347	317	496	1634	1191	1108	62	1335	445	704	323	1391	1469	868	1378	562	1427
1400				2500														
	repo	orted test	results					calc. by ii	s									
1200 -		()		2000 -				Ν										
1000 -																		
				1500 -				Λ										
800 -		- / \																
600 -				1000 -				$ \rangle$										
400		1																
		1		500 -														
200 -	2	1						h										
0	\land	,	\mathbf{k}	0	\mathcal{M}			Ŋ										
0.507	0.508 0.509 0	0.51 0.5	11 0.512	0.507	0.508	0.509	0.51	0.511	0.512									

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

92 150 D2598 184.68 E 171 317 ISO8973 186.9 323 334 347 D2598 183.7 444 445 IP432 187 447 496 D2598 183.9 562 D2598 183 1011 1016 1046 1055 W result withdrawn 1095 W 1191 ISO8973 186.955 1197	lab	method	value	mark	z(targ)	remarks	;						
150 D2598 184.68 E calculation by iis gave 183.52 171 317 ISO8973 186.9 323 334 347 D2598 183.7 444 444 444 444 444 447 446 D2598 183.9 562 D2598 183 1011 1040 result withdrawn 1040	92												
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	150	D2598	184.68	E		calculati	on by iis	gave 183	.52				
317 ISO8973 186.9 323 334 347 D2598 183.7 444 444 444 447 496 D2598 183.9 562 D2598 183.9 562 D2598 183.3 704 ISO8973 186.86 1011 1016 1040 W 1095 W 1108 D2598 186.955 1197 1109	171							•					
323 334 347 D2598 183.7 444 444 444 447 496 D2598 183.9 562 D2598 183 704 ISO8973 186.86 1011 1016 1095 W 1095 W 1108 D2598 186.9 1191 ISO8973 186.955 1197 1197 1108 D2598 186.955	317	ISO8973	186.9										
334 347 D2598 183.7 444 444 445 IP432 187 447 496 D2598 183.9 562 D2598 183 562 D2598 183 704 ISO8973 186.86 1011 1016 1040 1095 W 1095 W 1108 D2598 186.9 1197 1197 1108 D25973 186.955	323												
347 D2598 183.7 444 445 IP432 187 447 496 D2598 183.9 562 D2598 183 562 D2598 183 704 ISO8973 186.86 1011 1016 1040 1095 W 1095 W 1108 D2598 186.95 1197 1197	334	B a a a a											
444 445 IP432 187 447 496 D2598 183.9 562 D2598 183 704 ISO8973 186.86 868 D2598 183 1011 1016 1040 1095 W 1095 W 1108 D2598 186.9 1191 ISO8973 186.955 1197	347	D2598	183.7										
445 IP432 187 447 496 D2598 183.9 562 D2598 183 704 ISO8973 186.86 868 D2598 183 1011 1016 1040 1095 W 1095 W 1108 D2598 186.9 1191 ISO8973 186.955 1197 1108	444	10400	407										
447 183.9 562 D2598 183.9 704 ISO8973 186.86 868 D2598 183 1011 1016 1040 1095 1095 1108 D2598 186.9 1191 ISO8973 186.955 1197	445	IP432	187										
560 D2598 183 704 ISO8973 186.86 868 D2598 183 1011 1016 1040 1095 W 1018 D2598 186.9 1108 D2598 186.95 1197 1197	447	D2598	183.0										
704 ISO8973 186.86 868 D2598 183 1011 1016 1040 1095 1095 1108 D2598 186.9 1191 ISO8973 186.955 1197 1198	562	D2598	183										
868 D2598 183 1011 1016 1040 1095 W 1108 D2598 186.9 1191 ISO8973 186.955 1197 1198	704	ISO8973	186.86										
1011 1016 1040 1095 W 1018 D2598 186.9 1191 ISO8973 186.955 1197	868	D2598	183										
1016 1040 1095 W 1108 D2598 186.9 1191 ISO8973 186.955 1197	1011												
1040 1095 W 1108 D2598 186.9 1191 ISO8973 186.955 1197 1198	1016												
1095 W result withdrawn 1108 D2598 186.9 1191 ISO8973 186.955 1197 1198	1040												
1108 D2598 186.9 1191 ISO8973 186.955 1197 1197	1095	_		W		result wi	thdrawn						
1191 ISO8973 186.955 1197	1108	D2598	186.9										
1197 1109	1191	ISO8973	186.955										
	1197												
1190 1250 ICO2072 127.00	1798	1909072	197.00										
1284	1239	1300973	187.09										
1335 C first reported 169, result mixed up with relative vapour pressure	1335			С		first repo	orted 169	result m	ixed up y	with relative	vapour	pressure	
1378 ISO8973 187.27	1378	ISO8973	187.27	Ũ		motrope		, 10001111	nou up	inter rolative	rapour	procedure	
1391 ISO8973 185.8 E calculation by iis gave 186.7	1391	ISO8973	185.8	Е		calculati	on by iis	qave 186	.7				
1427	1427							0					
1469 D2598 186.762	1469	D2598	186.762										
1634	1634												
	1810												
Calculated by lis from all reported test results, see §4.3:						Calculate	ed by iis	from all re	eported t	est results,	<u>see §4.3</u>	<u>3:</u>	
ISUB97371P432: ASTM D2398:		normality (not OK			1508973	5 / IP432:			ASTM D	2598:		
n 14 26 26		n	101 OK			26				26			
14 20 20		outliers	0			20				20			
mean (n) 185.701 186.822 183.493		mean (n)	185.701			186.822				183.493			
st.dev. (n) 1.6615 0.3519 0.3853		st.dev. (n)	1.6615			0.3519				0.3853			
R(calc.) 4.652 0.985 1.079		R(calc.)	4.652			0.985				1.079			
R(lit) unknown unknown unknown		R(lit)	unknown			unknowr	n			unknowr	า		
188 -	188 _T												
	407											•	۵
	187 +					Δ	Δ	Δ	Δ	Δ	Δ	-	
186	186 -				A								
185 -	185												
				4									
	184 -		Δ Δ										
	183 -	Δ Δ											
182	182												
	102												
181 +	181 +												
180	180	_											-
868 562 562 347 496 150 150 150 1191 1108 11108 11191 1191 1191 1191 1		562	347	150	1391	1469	704	317	1108	1191	445	1259	1378



Determination of Relative Vapour Pressure @100F (Gage pressure); results in psi

lab	method	value	mark	z(targ)	remarks	
92	D 0500		_			
150	D2598	169.84	E		calculation by its gave 172.17	
317						
323	D2598	168.9				
334						
347	D2598	169				
444	IP432	171.1				
445	IP432	172				
447 496	D2598	169.2				
562	D2598	168				
704	ISO8973	172.16				
868	D2598	168				
1011						
1016						
1040			14/		and the Minderson	
1095	D2508	172.2	VV		result withdrawn	
1100	D2390	172.2				
1197						
1198						
1259		172.45				
1284						
1335	D2598	169	~			
1378	1508973	172.57	C E		first reported n.a., result mixed up with absolute vapour pressure	
1427	1300973		L		Calculation by its gave 172.00	
1469						
1634	ISO8973	172.497				
1810						
					Calculated by iis from all reported test results, see §4.3:	
	n o rm olitr /				<u>ISO897371P432:</u> not OK	
	normality	15			101 OK 101 OK	
	outliers	0			20 20	
	mean (n)	170.541			172.126 168.797	
	st.dev. (n)	1.74195			0.3519 0.3853	
	R(calc.)	4.8774			0.985 1.079	
	R(lit)	unknown			unknown unknown	
¹⁷³ T						
172						4
					-	
171 -						
170						-
					Δ	
169 -		▲ ▲	۵	Δ		
168 -						
167 +						
166						
	868 868	323	347	496	150 444 445 445 704 1108 1259 1259 1634	1378



APPENDIX 2

Number of participants per country

1 lab in BELGIUM

- 1 lab in CANADA
- 1 lab in CHILE
- 1 lab in CROATIA
- 1 lab in CZECH REPUBLIC
- 1 lab in EGYPT
- 1 lab in FINLAND
- 1 lab in FRANCE
- 2 labs in GERMANY
- 2 labs in GREECE
- 2 labs in MALAYSIA
- 2 labs in P.R. of CHINA
- 4 labs in PORTUGAL
- 1 lab in QATAR
- 1 lab in SPAIN
- 1 lab in TAIWAN R.O.C.
- 1 lab in THE NETHERLANDS
- 2 labs in U.S.A.
- 1 lab in UKRAINE
- 3 labs in UNITED KINGDOM

APPENDIX 3

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
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- 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