Results of Proficiency Test Liquefied Propane October 2012

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Report:

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1 INTRODUCTION

A first proficiency study for Liquefied Propane (composition only) was organized by its in 2009. Afterwards the opinion of the participating laboratories was inventarized. Most participants were very positive and therefore it was decided to repeat the PT annually.

Because iis has limited gas-handling facilities in place to prepare gas samples, a cooperation with EffecTech (Uttoxeter, United Kingdom) was set up. This company is fully equipped and has experience in the preparation of synthetic natural gas samples for PT purposes. EffecTech maintains an ISO17043 accreditation for the preparation of PT samples in homogeneous and stable batches and an ISO17025 accreditation for the calibration and assignment of reference values for these samples.

This year 30 laboratories in 20 different countries have participated. See appendix 3 for the number of participants per country. In this report the results of the 2012 proficiency test on Liquefied Propane are presented and discussed. This report is also electronically available through the iis internet site http://www.iisnl.com.

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 40 cylinders. The cylinder size is a cost-effective one-litre cylinder with dip tube device. Each cylinder, filled with approx 200 grams of liquefied propane mixture, was uniquely numbered. The limited cylinder size is chosen to optimise sample stability, cylinder costs, transport and handling costs.

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/IEC 17043:2010 and ILAC-G13:2007 (R007). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Also customer's satisfaction is measured on regular basis by the distribution of questionnaires.

EffecTech is an accredited provider of proficiency testing schemes under the requirements of ISO/IEC17043:2010 by UKAS (no. 4719).

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 presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

In this proficiency test only one sample was used. One batch of 40 one litre cylinders with artificial Liquefied Propane mixture was prepared and tested for homogeneity by EffecTech (Uttoxeter, United Kingdom) in conformance with ISO Guide 35: 2006 and ISO/IEC17043:2010 (job 12/527) starting October 1, 2012. Each cylinder was uniquely numbered. Every cylinder in the batch was analysed using 6 replicate measurements. The within bottle and between bottle variations were then assessed in accordance with ISO Guide 35:2006 (Annex A.1). This procedure showed that the between bottle variations were all small compared to the uncertainties on the reference values on each component. Hence, a single reference value could be safely assigned to the entire batch of samples. 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 reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

Parameter	r(observed) in %mol/mol	0.3 X R(D2163) in %mol/mol
Ethane	0.079	0.077
Propane	0.071	0.273
Propylene	0.008	0.033
Iso-Butane	0.037	0.094
n-Butane	0.031	0.067
1-Butene	0.002	0.004
iso-Butylene	0.004	0.009
n-Pentane	0.015	0.028

Table 1: homogeneity test results of samples #12074

Each calculated repeatability is equal or less than 0.3 times the corresponding reproducibility of the reference method ASTM D2163:96.

Therefore, homogeneity of the subsamples #12074 was assumed.

To each of the participating laboratories one 1L cylinder was sent on October 10, 2012.

2.5 STABILITY OF THE SAMPLES

EffecTech (Uttoxeter, United Kingdom) declares that the prepared gas 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 @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 as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website.

A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original 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.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally the reproducibilities were calculated from the standard deviations by multiplying 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; nos.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.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised

to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate the fit-for-useness of the reported test result.

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

|z| < 1 good 1 < |z| < 2 satisfactory 2 < |z| < 3 questionable 3 < |z| unsatisfactory

4 EVALUATION

In this proficiency test several problems were encountered with sample transport. Due to these problems seven cylinders did reach the laboratory near or after the final reporting date and were unable to test the cylinder and to report results before the deadline of reporting. In total eleven laboratories reported the test results after the final reporting date and three other laboratories did not report any test results due to several circumstances. Not all laboratories did report all test results requested.

In total 27 participating laboratories reported 267 numerical test results. Observed were 19 outlying test results, which is 7.1% 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 were 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 n-pentane and for absolute and relative vapour pressure @100F 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 IP405 and ISO7941) for reference only. Regretfully in the last version ASTM D2163:07 only repeatabilities, but no reproducibilities are mentioned. Therefore the precision data from the previous version ASTM D2163:96 (estimated from figure 3) were used.

Ethane: The determination of this component may be problematic, depending on the test method used by the laboratory. Three 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 good agreement with the less strict reproducibility requirements of EN27941 (identical to IP405 and ISO7941).

<u>Propane:</u> No analytical problems were observed. Only one statistical outlier was observed and the calculated reproducibility, after exclusion of the statistical outlier, is in good agreement with the requirements of ASTM D2163:96 and also with the reproducibility requirements of EN27941 (identical to IP405 and ISO7941).

- <u>Propylene:</u> No analytical problems were observed. Three statistical outliers were observed. However, the calculated reproducibility, after exclusion of the statistical outliers, is in good agreement with the requirements of ASTM D2163:96 and also with the reproducibility requirements of EN27941 (identical to IP405 and ISO7941).
- iso-Butane: No analytical problems were observed. Only one statistical outlier was observed. 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 requirements of EN27941 (identical to IP405 and ISO7941).
- n-Butane: The determination of this component may be problematic, depending on the test method used by the laboratory. Two statistical outliers were observed. 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 less strict reproducibility requirements of EN27941 (identical to IP405 and ISO7941).
- <u>1-Butene:</u> The determination of this component may be problematic, depending on the test method used by the laboratory. One statistical outlier was observed. The calculated reproducibility, after exclusion of the statistical outlier, is not in agreement with the requirements of ASTM D2163:96. However, the calculated reproducibility is in agreement with the less strict reproducibility requirements of EN27941 (identical to IP405 and ISO7941).
- <u>Iso-Butene:</u> The determination of this component may be problematic, depending on the test method used by the laboratory. One statistical outlier was observed. The calculated reproducibility, after exclusion of the statistical outlier, is not in agreement with the requirements of ASTM D2163:96. However, the calculated reproducibility is in agreement with the less strict reproducibility requirements of EN27941 (identical to IP405 and ISO7941).

- <u>n-Pentane:</u> The determination of this component may be problematic, depending on the test method used by the laboratory. Only one statistical outlier was 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 less strict reproducibility requirements of EN27941 (identical to IP405 and ISO7941).
- Molar Mass: This calculated parameter may be problematic. The results vary over a range from 43.368 44.9. One statistical outlier was present. The calculated reproducibility after rejection of the statistical outlier is large in comparison with the calculated reproducibility of iis11S03P (0.22 vs. 0.14). See also the discussion in 4.3.
- Rel. Density @60F: This calculated parameter may be problematic. The results vary over a range from 0.5033 0.5122. One statistically significant outlier was present. Possibly 6 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 fully explain for the observed spread.
- Abs. Vapour Press.: As the reported results calculated via ASTM D2598 and ISO8973 are not identical. It was decided to calculate the absolute vapour pressure for each laboratory according both test methods by using the reported contents of the components. It is noticed that the mean and spread when using the calculation method of ASTM D2598 are both small in comparison with the mean and spread when using the calculation method of ISO8973. See also the discussion in 4.3.
- Rel. Vapour Press.: As the reported results calculated via ASTM D2598 and ISO8973 are not identical. It was decided to calculate the relative vapour pressure for each laboratory according both test methods by using the reported contents of the components. It is noticed that the mean and spread when using the calculation method of ASTM D2598 are both small in comparison with the mean and spread when using the calculation method of ISO8973. 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)	R(EN27941) liqinj.
					in %mol	in %mol
Ethane	%mol/mol	23	1.963	0.421	0.226	0.739
Propane	%mol/mol	25	91.013	0.693	0.910	1.015
Propylene	%mol/mol	23	0.952	0.068	0.110	0.213
iso-Butane	%mol/mol	25	2.823	0.344	0.325	0.384
n-Butane	%mol/mol	24	2.053	0.309	0.236	0.386
1-Butene	%mol/mol	24	0.116	0.022	0.013	0.168
Iso-Butene	%mol/mol	24	0.254	0.049	0.029	0.167
n-Pentane	%mol/mol	25	0.832	0.219	0.096	0.305
Molar Mass	g/mol	12	44.746	0.221	n/a	n/a
Rel. Density @60F		17	0.5100	0.0029	n/a	n/a
Abs. Vapour press.	psi		see	§4.3	n/a	n/a
Rel. Vapour press.	psi		see	§4.3	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 some 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 EffecTech, United Kingdom in the following table.

Parameter	Average values by EffecTech in %mol/mol	Consensus values from participants results in %mol/mol	Absolute differences in %mol/mol	z-score
Ethane	2.230	1.963	+0.267	+3.30
Propane	90.980	91.013	-0.033	-0.10
Propylene	0.964	0.952	+0.012	+0.32
iso-Butane	2.722	2.823	-0.101	-0.87
n-Butane	1.945	2.053	-0.108	-1.28
1-Butene	0.111	0.116	-0.005	-0.96
Iso-Butene	0.250	0.254	-0.004	-0.37
n-Pentane	0.798	0.832	-0.034	-0.99

Table 3: comparison of consensus values with values determined by EffecTech

From this comparison it is clear that not <u>all</u> consensus values as determined in this PT are in line with the values as determined by EffecTech during the preparation of the cylinders.

For the calculation of the Vapour Pressure (VP), ten participants used ISO8973/IP432 and four participants used ASTM D2598. In ISO 8973 (identical to IP432) the Absolute VP is calculated from the mole fraction per component and a VP factor of that component (given for all components). From the Absolute VP, the Relative VP is calculated. In ASTM D2598 the Gage pressure (identical to the Relative VP) is calculated from the liquid volume percentage per component and a VP factor of that component. Regretfully in the 2002 (2007) version of D2598 no factors are given for n-butene, 1-butene and n-pentane. However, in the draft 2012 version, factors are mentioned for these and other components. 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 significantly lower than the results from the ISO8973/IP432 calculation. The observed difference is caused by a difference in the VP factor of Ethane. ASTM (Subcommittee D02.H) commented: "The vapor pressure of ethane in D2598 was revised a few times prior to 2002. The current value, 611 psi, has remained the same for the last ten years. The revision of ethane was done because components in LPG blends do not necessarily behave as ideal gases. In particular, properties of ethane and ethylene appear to differ from ideality. Factors for these two components have been modified from 'ideal gas' values to make the calculated vapor pressure results more closely approximate actual measured vapor pressures of LPG blends. (i.e. D1267). Chapter 2 of Fuels and Lubricants Handbook (George Totten, © 2003), states that calculated vapor pressure were found to be biased high relative to experimental vapor pressure measured by D1267 for high ethane samples in earlier versions of D2598".

APPENDIX 1

Determination of Ethane on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	2.08		1.44	
150	D2163	2.185		2.75	
171	D2163	1.672		-3.61	
311	D2163	2.005		0.52	
317	D2163	1.98		0.21	
323	D2163	1.65		-3.88	
334	EN27941	1.964		0.01	
347	D2163	2.494	DG(0.05)	6.57	
444	IP405	1.998		0.43	
445	D2163	1.99		0.33	
511	D2163	1.830		-1.65	
704	D2163	1.893		-0.87	
706	D2163	1.953		-0.13	
868	D2163	1.952		-0.14	
912					
1006	D2163	2.261		3.69	
1026	ISO7941	2.467	DG(0.05)	6.24	
1095	EN27941	1.862		-1.26	
1197	D2163	2.089		1.56	
1198	D2163	1.940		-0.29	
1200	Dotoo				
1257	D2163	1.///	0.0(0.04)	-2.31	
1259	EN27941	4.968	C,G(0.01)	37.22	First reported 5.704
1272	EN27941	1.86		-1.28	
1419	EN27941	2.015		0.64	
1490	EN27941	1.929		-0.43	
1491	1507941	2.138		2.16	
1497	1007044	 0 105		2 1 2	
1034	1307941	2.155		2.13	
	normality	OK			
	n	23			
	outliers	3			
	mean (n)	1.963			
	st.dev. (n)	0.1503			
	R(calc.)	0.421			
	R(D2163:96)	0.226			Compare R(EN27941(liq)) = 0.739





Determination of Propane on sample #12074 ; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	91.35		1.04	
150	D2163	90.721		-0.90	
171	D2163	90.528		-1.49	
311	D2163	91.007		-0.02	
317	D2163	91.59	С	1.78	First reported 91.41
323	D2163	91.11		0.30	
334	EN27941	90.891		-0.38	
347	D2163	91.095		0.25	
444	IP405	90.783		-0.71	
445	D2163	91.13		0.36	
511	D2163	90.900		-0.35	
704	D2163	91.133		0.37	
706	D2163	91.330		0.98	
868	D2163	90.948		-0.20	
912					
1006	D2163	90.979		-0.10	
1026	ISO7941	90.069	G(0.05)	-2.90	
1095	EN27941	91.221		0.64	
1197	D2163	90.874		-0.43	
1198	D2163	91.106		0.29	
1200					
1257	D2163	91.060		0.14	
1259	EN27941	90.767	С	-0.76	First reported 87.061
1272	EN27941	90.82		-0.59	
1419	EN27941	90.698		-0.97	
1490	EN27941	90.806		-0.64	
1491	ISO7941	91.069		0.17	
1497					
1634	ISO7941	91.407		1.21	
	normality	ОК			
	n	25			
	outliers	1			
	mean (n)	91.013			
	st.dev. (n)	0.2474			
	R(calc.)	0.693			
	R(D2163:96)	0.910			Compare R(EN27941(liq)) = 1.015



1.6 Kernel Density 1.4 1.2 1 0.8 0.6 0.4 0.2 0 89.5 90 90.5 91 91.5 92 92.5

Determination of Propylene on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	0.92		-0.81	
150	D2163	0.976		0.63	
171	D2163	0.968		0.42	
311	D2163	0.943		-0.22	
317	D2163	0.89	С	-1.57	First reported 0.86
323	D2163	0.93		-0.55	
334	EN27941	0.955		0.09	
347	D2163	0.982		0.78	
444	IP405	0.967		0.40	
445	D2163	0.97		0.47	
511	D2163	0.990		0.98	
704	D2163	0.923		-0.73	
706	D2163	0.930		-0.55	
868	D2163	0.940		-0.29	
912					
1006	D2163	0.977		0.65	
1026	ISO7941	1.087	DG(0.01)	3.46	
1095	EN27941	0.966		0.37	
1197	D2163	0.958		0.17	
1198	D2163	0.943		-0.22	
1200					
1257	D2163	0.959		0.19	
1259	EN27941	1.080	C,DG(0.01)	3.28	First reported 1.037
1272	EN27941	0.92		-0.81	
1419	EN27941	0.965		0.34	
1490	EN27941	0.944		-0.19	
1491	ISO7941	0.969		0.45	
1497					
1634	ISO7941	1.048	G(0.05)	2.47	
	normality	OK			
	n	23			
	outliers	3			
	mean (n)	0.952			
	st.dev. (n)	0.0245			
	R(calc.)	0.068			
	R(D2163:96)	0.110			Compare $R(EN27941(lig)) = 0.213$
		5			





Determination of iso-Butane on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	2.86		0.32	
150	D2163	2.860		0.32	
171	D2163	3.136		2.70	
311	D2163	2.805		-0.15	
317	D2163	2.59		-2.01	
323	D2163	2.91		0.75	
334	EN27941	2.861		0.33	
347	D2163	2.575		-2.14	
444	IP405	2.899		0.66	
445	D2163	2.80		-0.20	
511	D2163	2.750		-0.63	
704	D2163	2.797		-0.22	
706	D2163	2.733		-0.77	
868	D2163	2.838		0.13	
912					
1006	D2163	2.717		-0.91	
1026	ISO7941	2.965		1.23	
1095	EN27941	2.709		-0.98	
1197	D2163	2.832		0.08	
1198	D2163	2.793		-0.26	
1200					
1257	D2163	2.889		0.57	
1259	EN27941	1.301	C,G(0.01)	-13.11	First reported 1.137
1272	EN27941	2.98		1.35	
1419	EN27941	2.915		0.79	
1490	EN27941	2.911		0.76	
1491	ISO7941	2.770		-0.45	
1497					
1634	ISO7941	2.675		-1.27	
	normality	ОК			
	n	25			
	outliers	1			
	mean (n)	2.823			
	st.dev. (n)	0.1228			
	R(calc.)	0.344			
	R(D2163:96)	0.325			Compare R(EN27941(liq)) = 0.384
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Determination of n-Butane on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	1.95		-1.22	
150	D2163	2.039		-0.17	
171	D2163	2.307		3.01	
311	D2163	2.030		-0.28	
317	D2163	1.86		-2.29	
323	D2163	2.13		0.91	
334	EN27941	2.077		0.28	
347	D2163	1.826		-2.69	
444	IP405	2.097		0.52	
445	D2163	1.98		-0.87	
511	D2163	2.140		1.03	
704	D2163	2.040		-0.16	
706	D2163	1.960		-1.10	
868	D2163	2.099		0.54	
912					
1006	D2163	1.932		-1.44	
1026	ISO7941	2.148		1.12	
1095	EN27941	2.106		0.62	
1197	D2163	2.008		-0.54	
1198	D2163	1.991		-0.74	
1200	_				
1257	D2163	2.225		2.03	
1259	EN27941	0.753	C,G(0.01)	-15.40	First reported 0.629
1272	EN27941	2.11		0.67	
1419	EN27941	2.136		0.98	
1490	EN27941	2.119		0.78	
1491	ISO7941	1.968		-1.01	
1497			- ()		
1634	ISO7941	1.696	G(0.05)	-4.23	
	normality	ОК			
	n	24			
	outliers	2			
	mean (n)	2.053			
	st.dev. (n)	0.1104			
	R(calc.)	0.309			
	R(D2163:96)	0.236			Compare R(EN27941(liq)) = 0.386
					· · · · · ·





Determination of 1-Butene on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92					
150	D2163	0.111		-0.96	
171	D2163	0.130		3.03	
311	D2163	0.110		-1.17	
317	D2163	0.10		-3.28	
323	D2163	0.12		0.93	
334	EN27941	0.117		0.30	
347	D2163	0.103		-2.65	
444	IP405	0.119		0.72	
445	D2163	0.11		-1.17	
511	D2163	0.120		0.93	
704	D2163	0.113		-0.54	
706	D2163	0.107		-1.81	
868	D2163	0.115		-0.12	
912					
1006	D2163	0.110		-1.17	
1026	ISO7941	0.123		1.56	
1095	EN27941	0.111		-0.96	
1197	D2163	0.118		0.51	
1198	D2163	0.119		0.72	
1200					
1257	D2163	0.123		1.56	
1259	EN27941	0.059	C.G(0.01)	-11.91	First reported 0.039
1272	EN27941	0.13		3.03	•
1419	EN27941	0.122		1.35	
1490	EN27941	0.118		0.51	
1491	ISO7941	0.122		1.35	
1497					
1634	ISO7941	0.103		-2.65	
	normality	ОК			
	n	24			
	outliers	1			
	mean (n)	0.116			
	st.dev. (n)	0.0080			
	R(calc.)	0.022			
	R(D2163:96)	0.013			Compare R(EN27941(liq)) = 0.168
J.14					





Determination of iso-Butene on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92					
150	D2163	0.251		-0.28	
171	D2163	0.287		3.17	
311	D2163	0.250		-0.37	
317	D2163	0.22		-3.25	
323	D2163	0.26		0.59	
334	EN27941	0.261		0.68	
347	D2163	0.229		-2.38	
444	IP405	0.263		0.87	
445	D2163	0.24		-1.33	
511	D2163	0.280		2.50	
704	D2163	0.250		-0.37	
706	D2163	0.230		-2.29	
868	D2163	0.254		0.01	
912					
1006	D2163	0.248		-0.56	
1026	ISO7941	0.266		1.16	
1095	EN27941	0.247		-0.66	
1197	D2163	0.259		0.49	
1198	D2163	0.257		0.30	
1200					
1257	D2163	0.264		0.97	
1259	EN27941	0.123	C,G(0.01)	-12.54	First reported 0.102
1272	EN27941	0.28		2.50	
1419	EN27941	0.272		1.74	
1490	EN27941	0.262		0.78	
1491	ISO7941	0.236		-1.71	
1497					
1634	ISO7941	0.227		-2.57	
	normality	ОК			
	n	24			
	outliers	1			
	mean (n)	0.254			
	st.dev. (n)	0.0175			
	R(calc.)	0.049			
	R(D2163:96)	0.029			Compare R(EN27941(liq)) = 0.167





Determination of n-Pentane on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	0.84		0.24	
150	D2163	0.854		0.65	
171	D2163	0.973		4.13	
311	D2163	0.850		0.53	
317	D2163	0.77	С	-1.81	First reported 0.99
323	D2163	0.89		1.70	
334	EN27941	0.874		1.23	
347	D2163	0.696		-3.97	
444	IP405	0.872		1.17	
445	D2163	0.78		-1.52	
511	D2163	0.990		4.62	
704	D2163	0.850		0.53	
706	D2163	0.757		-2.19	
868	D2163	0.853		0.62	
912					
1006	D2163	0.777		-1.60	
1026	ISO7941	0.861		0.85	
1095	EN27941	0.778		-1.58	
1197	D2163	0.859		0.79	
1198	D2163	0.846		0.41	
1200					
1257	D2163	0.703		-3.77	
1259	EN27941	0.121	C,G(0.01)	-20.78	First reported 0.092
1272	EN27941	0.90		1.99	
1419	EN27941	0.877		1.32	
1490	EN27941	0.910		2.28	
1491	ISO7941	0.728		-3.04	
1497					
1634	ISO7941	0.709		-3.59	
	normality	not OK			
	n	25			
	outliers	1			
	mean (n)	0.832			
	st.dev. (n)	0.0782			
	R(calc.)	0.219			
	R(D2163:96)	0.096			Compare $R(EN27941(lig)) = 0.305$
		0.000			





Determination of Molar Mass on sample #12074; results in g/mol

lab	method	value	mark	z(targ)	remarks						
92	inh-2145	44.7	mark		Tomarito						
150											
171											
311	INH-407	44.760									
317	D2598	44.7									
323	D3588	44.9									
334 347	D2421	44 583									
444											
445	IP432	44.731									
511											
704	D2163	44.7735									
706	D2163	44.7157									
868	CALC	44.78									
1006											
1026											
1095		44.8									
1197											
1198											
1200											
1257	1000070		0.0(0.04)		—						
1259	1508973	43.3680036	C,G(0.01)		First reported	141.71	55				
1419											
1490	CALC	44.813									
1491	ISO8973	44.69									
1497											
1634											
	n ormolity (OK			Calculated by	y iis fror	n all repo	rted test r	<u>esults:</u>		
	normality	12			25						
	outliers	1			1						
	mean (n)	44.746			44.754						
	st.dev. (n)	0.0788			0.0738						
	R(calc.)	0.221			0.207						
	R(lit)	unknown			unknown						
	R(IISTTS03P)	0.140			0.065 (for col	mparisc	n)				
44.95											
44.9 -											▲
44.85 -											
44.8 -									Δ	Δ	
44.75 -					· · · · · · · · · · · · · · · · · · ·	Δ	Δ	4			
44.7 -		Δ Δ	Δ	Δ	Δ						
44.65 -		4									
116											
44.55	Δ										
44.55 -											
44.5	347	491 92	317	206	445	311	704	368	395	490	323
	ę .,	÷			•	.,		-	7	÷	
						٦					
6	Kern	el Density	6		Kernel Density						
5.5	Rein	erbensity	5 A 1	,	Kenter Density						
3 Re	eported test		° Calo	2 DV 11							
4 re	esults		4								
3 -			3 -								
						1					
2			2			1					
			1			1					
	Λ ſ		^			1					
0						1					
43	43.0 44 44.5	40 45.5	43 43.5	44 4	4.0 40 45.5						

Determination of Relative Density @60F; unitless results

lah	mothod		voluo		mor	,	T(torg)	ron	orko								
	Decoe		value		mari	(z(targ)	ren	iarks								
92	D2598		0.509														
150	D2598		0.5097														
1/1	D2598		0.5122														
311	INH-407		0.5096														
317	1508973		0.5091														
323	D2598		0.5107														
334	DOCOD																
347	D2598		0.5082														
444																	
445	IP432		0.5103														
204	D2596		0.5100														
704	D2596		0.5101														
700	D2596		0.5097														
000	D2390		0.5101														
1006	D2508		0 5002														
1000	1508073		0.5092														
1020	1300973		0.5091		۱۸/												
1107					vv												
1108																	
1200																	
1200																	
1250			0 5033	143	CG	0.01)		Fire	t renort	ed 0 50	180						
1233	1508073		0.5055	145	0,0(0.01)		1 113	stiepon	eu 0.50	103						
1/10	1000375		0.5110		U												
1410	1508073		0 51070	14													
1400	1000373			7													
1/107																	
1634	IS08973		0.5096														
1004	1000070		0.0000					Cal	culated	hv iis fr	om all	renorte	d test re	esulte.	Ide	m for 1	5°C:
	normality		OK						oulutou	<u>by 10 11</u>	onnan	reported		<u></u>			<u>0 0.</u>
	n		17					25							25		
	outliers		1					1							1		
	mean (n)		0.50998	3				0.5	0990						0.5	1020	
	st.dev. (n)		0.0010	16				0.0	00530						0.0	00498	
	R(calc.)		0.0028	5				0.0	0148						0.0	0139	
	R(lit)		unknow	'n				unk	nown						unł	nown	
	R(iis11S03	P)	0.00076	5				0.0	0036						0.0	0036	
0.514																	
0.011																	
0.512 -																Δ	Δ
												•	Δ	Δ	Δ		
0.51 -		•		^	Δ	Δ	Δ	Δ	Δ	∆	Δ						
0.508 -	۵	-	-	_													
0.506 -																	
0.504																	
0.504 -	ж																
0.502 -																	
0.5	65 14	8	2	9	9	Ξ	*	ø	02	88	4	5	Ξ	8	8	22	2
	ő <u>1</u>		'n	10:	100	'n	16:	7	4	8	Ř	4	à	69	14	12	÷
400			10 11		1200												
350 -		∧ Kern	lei Density					Kerner	Density								
Re	eported	test	:		¹⁰⁰⁰ C	alc	by iis	۰ ۱									
300 re	esults																
250					000 -												
200					600												
200					000			-									
150 -					400 -												
100 -								$ \rangle$									
		1			200 -												

0.505

0.51

0.515

0

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

lab	method	value	mark	z(targ)	remarks							
92 150 171 311 317 323 334 347 444 445 511 704 706 868 912 1006	D2598 ISO8973 ISO8973 D2598 D2598 ISO8973 ISO8973 D2598 ISO8973	value 188.22 195 180.2 180.2 114.4316 194.10 194.84 188 194.8	G(0.01)	<u>z(targ)</u>	First reported							
1000 1026 1095 1197 1198 1200 1257 1259 1272 1419 1490 1491 1497	ISO8973 ISO8973 ISO8973	190.4 191.74 222.5829 193.95 195.89 	190.4 191.74 222.5829 C,G(0.01) 193.95 195.89									
	normality n outliers mean (n) st.dev. (n) R(calc.) R(lit)	196.435			Calculated by ISO8973 / IP/ OK 25 1 194.273 1.4745 4.129 unknown	iis from all 132	reported	<u>ASTM D2598</u> OK 25 1 188.300 1.2060 3.377 unknown				
240 220 - 200 - 180 - 160 - 140 - 120 -	<u>۸</u>	Δ Δ	Δ Δ	۵	A A.	Δ	Δ	A	Δ	۵	ж	
0.1	347	₩ E Kernel Density	1026	1490	704	708	317	311	1491	1634	1255	



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

lab	method	·	value	mark	z(targ)	remark	s						
92													
150 171													
311	ISO8973		180										
317	ISO8973		181	I C First reported 180									
323	D2598		171.5										
334 347	D2598		165.5										
444	22000												
445	IP432		180	0(0.04)									
511 704	D2598		99.7316	G(0.01)									
704	ISO8973		180.14										
868	D2598		173										
912	1000070												
1006	1508973		180.1 175 7										
1095	1000070		177.05										
1197													
1198													
1200													
1259			207.887	C,G(0.01)		First re	ported 19	98.8105					
1272													
1419	1508073		170.26										
1490	1000373												
1497													
1634	ISO8973		181.739			Calavia				4 4		\$4.0	
						LSO897	<u>ated by lis</u> 73 / IP432	<u>s from all</u> 2	геропео	ASTN	<u>// D2598</u>	<u>94.3</u>	
	normality					OK	0 / 11 10	=		OK		•	
	n					25				25			
	outliers					1	7			1 173 6	<u>۵</u> 4		
	st.dev. (n)					1.4745	'			1.206	504 50		
	R(calc.)					4.129				3.377	,		
	R(lit)					unknov	vn			unkn	own		
²³⁰													
210 -													*
190 -						A	A	^	A	A	4	Δ	
170 -	۵	Δ	Δ	A A	_	_							
150 -													
130 -													
110 -	ĸ												
90 -													
70 -													
50	34.7	323	968)26)95	06†	704	145	311	006	206	317	334	559
				10	1,		7		7			16	1
0.09													



APPENDIX 2

Number of participants per country

1 lab in BELGIUM

- 1 lab in CANADA
- 1 lab in CROATIA
- 1 lab in EGYPT
- 1 lab in FRANCE
- 1 lab in INDIA
- 2 labs in MALAYSIA
- 1 lab in P.R. of CHINA
- 1 lab in PERU
- 4 labs in PORTUGAL
- 1 lab in SAUDI ARABIA
- 2 labs in SERBIA
- 1 lab in SLOVAKIA
- 1 lab in SPAIN
- 1 lab in TAIWAN R.O.C.
- 3 labs in THE NETHERLANDS
- 1 lab in U.A.E.
- 2 labs in U.S.A.
- 2 labs in UKRAINE
- 2 labs in UNITED KINGDOM

APPENDIX 3

Abbreviations:

- 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 ASTM D2163-96
- 3 ASTM D2163-07
- 4 ASTM D2421-07
- 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)
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- 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)
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- 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 17043:2010
- 16 EN 27941:1993
- 17 ASTM D2598-02 (reapproved 2007)
- 18 IP 432-2000 = ISO8973-1997
- 19 Work Item WK36318, proposal to revise ASTM D2598-02 (07)
- 20 Private communication ASTM Subcommittee D02.H