

Results of Proficiency Test  
Liquefied Butane Analysis  
June 2012

Organised by: Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

Author: dr. R.G. Visser & ing. L.Dijkstra  
Corrector: ing. R. Starink  
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## 1 INTRODUCTION

A first proficiency study for Liquefied Butane (composition only) was organised by iis in 2009. Afterwards the opinion of the participating laboratories was inventorised. 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 co-operation 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 32 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 Butane 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 Butane 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 butane 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/IEC17043:2010 and ILAC-G13:2007. This ensures 100% confidentiality 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).

## 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 Butane mixture was prepared and tested for homogeneity by EffectTech (Uttoxeter, United Kingdom) in conformance with ISO Guide 35: 2006 and ISO/IEC17043:2010 (job 12/124) starting May 1, 2012. Each cylinder was uniquely numbered. Every cylinder in the batch was analysed using 4 - 8 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
iso-Butane	0.081	0.333
Propane	0.027	0.076
Propylene	0.025	0.048
n-Butane	0.027	0.145
1,3-Butadiene	0.004	0.040
iso-Butylene	0.010	0.071
1-Butene	0.026	0.187
trans-2-Butene	0.014	0.082
cis-2-Butene	0.019	0.114
iso-Pentane	0.008	0.023

Table 1: homogeneity test results of samples #12074

Each calculated repeatability is far 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 May 24, 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: Propane, Propylene, n-Butane, 1,3-Butadiene, iso-Butylene, 1-Butene, trans-2-Butene, cis-2-Butene, iso-Pentane, iso-Butane, 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 by e-mail. 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 Pentane.

## 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 visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 4; nos.13 and 14).

### 3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM, 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:

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

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 customs problems two cylinders did not reach the laboratory in time to test the cylinder and to report results to be included in the final report. In total six laboratories reported test results after the final reporting date.

Not all laboratories were able to report all test results requested.

Because 20 laboratories reported both results in %mol/mol as well as in %M/M, it has been possible to check the calculations of these 20 laboratories. A good correlation between the results reported in %mol/mol and the results reported in %M/M is to be expected.

All observed (small) deviations may be explained by the reporting of test results too far rounded.

In total 30 participants reported 373 numerical results. Observed were 39 outlying results, which is 10.5% of the 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, that 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 4.

All original data sets proved to have a normal distribution, except for Molar Mass results.

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. Regrettably the last version ASTM D2163:07 contains only provisional repeatability standard deviations, determined by statistical examination of limited interlaboratory results and no reproducibilities are mentioned. Therefore the precision data from the previous version ASTM D2163:96 were used.

Two laboratories appeared to have some problems. Six test results (=60%) reported by laboratory 357 and seven test results (=70%) reported by laboratory 1011 appeared to be statistical outliers and because all test results of one laboratory are correlated, the remaining test results of laboratories 357 and 1011 were excluded manually prior to the statistical analysis.

- iso-Butane: The determination of this main component may be problematic. Three statistical outliers were detected. The calculated reproducibility is not at all in agreement with the requirements of ASTM D2163:96. However, the calculated reproducibility is in agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).
- Propane: The determination of this component may be problematic. Four statistical outliers were detected and 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 good agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).
- Propylene: The determination of this component may be problematic. Five statistical outliers were detected and 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 good agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).
- n-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).
- 1,3-Butadiene: 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-Butylene: No analytical problems were observed. Three statistical outliers were detected. 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 of EN27941 (identical to IP 405 and ISO 7941).



- 1-Butene: No analytical problems were observed. Three statistical outliers were detected. 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 of EN27941 (identical to IP 405 and ISO 7941).
- trans-2-Butene: No analytical problems were observed. Two statistical outliers were detected. 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 of EN27941 (identical to IP 405 and ISO 7941).
- cis-2-Butene: No analytical problems were observed. Two statistical outliers were detected. 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 of EN27941 (identical to IP 405 and ISO 7941).
- iso-Pentane: No analytical problems were observed. Four statistical outliers were detected. 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 of EN27941 (identical to IP 405 and ISO 7941).
- Molar Mass: This calculated parameter may be not problematic. The results vary over a range from 57.36 - 57.73 g/mol and only one statistically significant outlier was observed (in 17 test results). See also the discussion in 4.4.
- Relative Density: This calculated parameter may be problematic. The results vary over a large range from 0.5645 - 0.57258 and four statistically significant outliers were observed (in 19 test results). See also the discussion in 4.4.
- Abs. Vapour Pres.: This calculated parameter may be problematic. The results vary over a large range (56.295 – 74.30 psi) and three statistically significant outliers were observed (in 17 test results). See also the discussion in 4.4.
- Rel. Vapour Pres.: This calculated parameter may be problematic. The results vary over a large range (41.603 – 63.0565 psi) and two statistically significant outliers were observed (in 20 test results). See also the discussion in 4.4.

## 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 %mol	R(EN27941) liq.-inj. in %mol	R(EN27941) liq.-inj. in %M/M
iso-Butane	%mol/mol	27	71.729	1.467	0.717	1.479	1.5
Propane	%mol/mol	26	1.743	0.254	0.201	1.300	1
Propylene	%mol/mol	25	1.175	0.160	0.135	1.362	1
n-Butane	%mol/mol	26	5.294	0.483	0.609	0.986	1
1,3-Butadiene	%mol/mol	26	0.957	0.110	0.110	1.060	1
iso-Butylene	%mol/mol	27	3.702	0.237	0.426	1.022	1
1-Butene	%mol/mol	26	7.509	0.572	0.864	1.022	1
trans-2-Butene	%mol/mol	28	3.057	0.273	0.352	1.022	1
cis-2-Butene	%mol/mol	28	3.883	0.347	0.447	1.022	1
iso-Pentane	%mol/mol	25	0.811	0.066	0.093	0.794	1
Molar Mass	g/mol	16	57.428	0.164	n/a	n/a	n/a
Relative Density		15	0.5715	0.0004	n/a	n/a	n/a
Abs. Vapour pres.	psi	14	71.81	1.54	n/a	n/a	n/a
Rel. Vapour pres.	psi	18	57.46	2.54	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 many components there is not 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 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2012 WITH PREVIOUS PTS

	June 2012	May 2011	May 2010	July 2009
Number of reporting labs	30	27	22	25
Number of results reported	373	333	263	291
Statistical outliers	39	29	20	16
Percentage outliers	10.5%	8.7%	7.6%	5.5%

table 3: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of ASTM D2163:96. The conclusions (some slight improvements) are given the following table:

	June 2012	May 2011	May 2010	July 2009
iso-Butane	--	--	--	--
Propane	-	--	--	--
Propylene	-	--	--	--
n-Butane	++	++	+	-
1,3-Butadiene	+/-	-	--	+/-
iso-Butylene	++	++	++	++
1-Butene	++	++	-	++
trans-2-Butene	+	+	-	--
cis-2-Butene	+	++	-	--
iso-Pentane	+	-	-	--

table 4: comparison determinations against the requirements of ASTM D2163:96

The performance of the determinations against the requirements of ASTM D2163:96 is listed in the above table. The following performance categories were used:

- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard

#### 4.4 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 evaluations 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 (Uttoxeter, United Kingdom) in the following table. From this comparison it is clear that most consensus values as determined in this PT are very well in line with the values as determined during the preparation of the gas cylinders.

Parameter	Average values by EffecTech in %mol/mol	Consensus values from participants results in %mol/mol	Absolute differences in %mol/mol	z-score
iso-Butane	71.812	71.729	+0.083	+0.32
Propane	1.812	1.743	+0.069	+0.97
Propylene	1.247	1.175	+0.072	+1.50
n-Butane	5.166	5.294	-0.128	-0.59
1,3-Butadiene	0.970	0.957	+0.013	+0.34
iso-Butylene	3.740	3.702	+0.038	+0.25
1-Butene	7.504	7.509	-0.005	-0.02
trans-2-Butene	3.065	3.057	+0.008	+0.07
cis-2-Butene	3.894	3.883	+0.011	+0.07
iso-Pentane	0.790	0.811	-0.021	-0.65

Table 5: comparison of consensus values with values determined by EffecTech (Uttoxeter, United Kingdom)

In total nine laboratories reported the presence of some n-pentane, a component probably present as impurity in one or more of the pure components that were used to prepare the iso-Butane 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. 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 mole fraction per component and a Vapour Pressure factor of that component (given for all components). In ASTM D2598 the Vapour Pressure is calculated from the liquid volume percentage per component and a Vapour Pressure factor of that component (given for only several components). Also the selection of the tables to be used for the calculations may cause additional uncertainty.

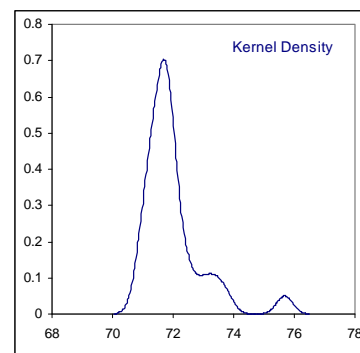
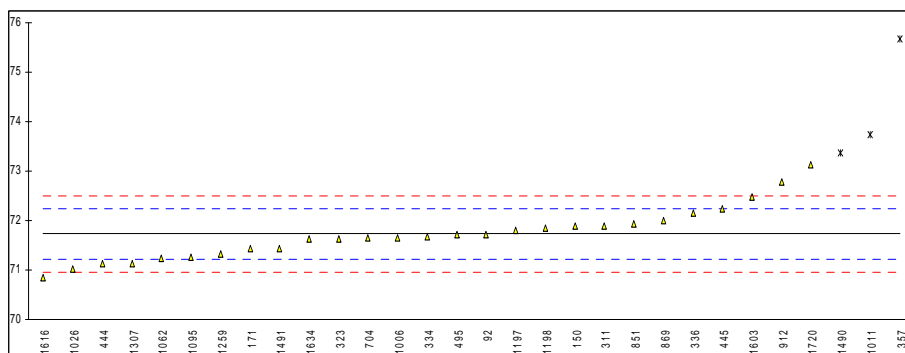
### APPENDIX 1

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

lab	method	value	mark	z(targ)	remarks
92	D2163	71.721		-0.03	
150	D2163	71.888	C	0.62	first reported 73.663
171		71.424		-1.19	
311	D2163	71.899		0.66	
317		-----		-----	
323	D2163	71.64		-0.35	
334	EN27941	71.667		-0.24	
336	EN27941	72.16		1.68	
357	D2163	75.681	G(0.01)	15.43	
444	IP405	71.123		-2.37	
445	D2163	72.25		2.03	
495	D2163	71.709		-0.08	
511		-----		-----	
704	D2163	71.648		-0.32	
851	D2163	71.942		0.83	
869	D2163	72.001		1.06	
912	D2163	72.79		4.14	
1006	D2163	71.656		-0.29	
1011	ISO7941	73.74	DG(0.05)	7.85	
1026	ISO7941	71.021		-2.77	
1062		71.243		-1.90	
1095	EN27941	71.271		-1.79	
1197	D2163	71.810		0.31	
1198	D2163	71.847		0.46	
1259	EN27941	71.321		-1.59	
1307	LHA	71.136		-2.32	
1490	EN27941	73.380	C,DG(0.05)	6.44	first reported 70.380
1491	ISO7941	71.443		-1.12	
1603	DIN51666	72.468		2.88	
1616	D2163	70.844		-3.46	
1634	ISO7941	71.630		-0.39	
1720	D2163	73.14	C	5.51	first reported 73.44

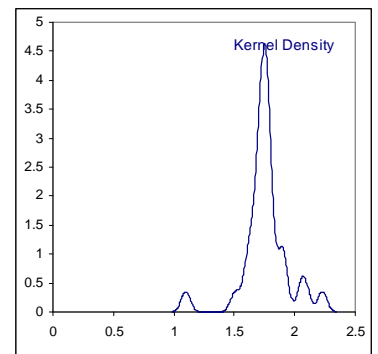
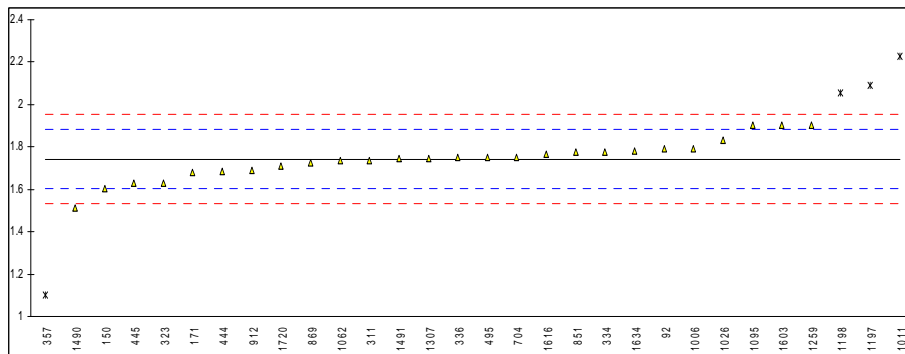
normality OK  
n 27  
outliers 3  
mean (n) 71.7293  
st.dev. (n) 0.52398  
R(calc.) 1.4672  
R(D2163:96) 0.7173

Compare R(EN27941(liq)) = 1.4792



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

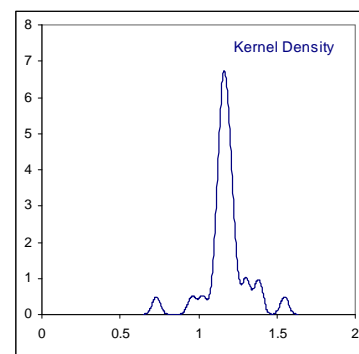
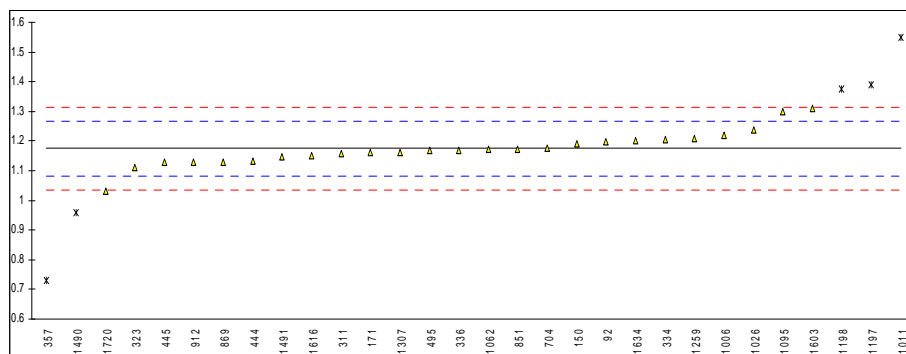
lab	method	value	mark	z(targ)	remarks
92	D2163	1.793		0.70	
150	D2163	1.603		-1.95	
171		1.682		-0.85	
311	D2163	1.737		-0.08	
317		-----		-----	
323	D2163	1.63		-1.57	
334	EN27941	1.778		0.49	
336	EN27941	1.75		0.10	
357	D2163	1.100	G(0.01)	-8.97	
444	IP405	1.687		-0.78	
445	D2163	1.63		-1.57	
495	D2163	1.750		0.10	
511		-----		-----	
704	D2163	1.752		0.13	
851	D2163	1.775		0.45	
869	D2163	1.727		-0.22	
912	D2163	1.69		-0.73	
1006	D2163	1.793		0.70	
1011	ISO7941	2.23	G(0.05)	6.80	
1026	ISO7941	1.831		1.23	
1062		1.733		-0.13	
1095	EN27941	1.902		2.22	
1197	D2163	2.091	DG(0.05)	4.86	
1198	D2163	2.053	DG(0.05)	4.33	
1259	EN27941	1.905		2.27	
1307	LHA	1.745		0.03	
1490	EN27941	1.510		-3.25	
1491	ISO7941	1.744		0.02	
1603	DIN51666	1.905		2.27	
1616	D2163	1.767		0.34	
1634	ISO7941	1.780		0.52	
1720	D2163	1.71	C	-0.46	first reported 1.53
normality		OK			
n		26			
outliers		4			
mean (n)		1.7427			
st.dev. (n)		0.09066			
R(calc.)		0.2538			
R(D2163:96)		0.2006			
				Compare R(EN27941(liq)) = 1.2998	



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

lab	method	value	mark	z(targ)	remarks
92	D2163	1.199		0.51	
150	D2163	1.189	C	0.30	first reported 1.032
171		1.160		-0.30	
311	D2163	1.157		-0.36	
317		-----		-----	
323	D2163	1.11		-1.34	
334	EN27941	1.204		0.61	
336	EN27941	1.17		-0.10	
357	D2163	0.731	G(0.01)	-9.19	
444	IP405	1.132		-0.88	
445	D2163	1.13		-0.92	
495	D2163	1.169		-0.12	
511		-----		-----	
704	D2163	1.176		0.03	
851	D2163	1.173		-0.03	
869	D2163	1.130		-0.92	
912	D2163	1.13		-0.92	
1006	D2163	1.219		0.92	
1011	ISO7941	1.55	G(0.01)	7.77	
1026	ISO7941	1.237		1.29	
1062		1.173		-0.03	
1095	EN27941	1.301		2.62	
1197	D2163	1.391	DG(0.05)	4.48	
1198	D2163	1.377	DG(0.05)	4.19	
1259	EN27941	1.208		0.69	
1307	LHA	1.163		-0.24	
1490	EN27941	0.960	C,D(0.05)	-4.44	first reported 0.790
1491	ISO7941	1.147		-0.57	
1603	DIN51666	1.309		2.78	
1616	D2163	1.149		-0.53	
1634	ISO7941	1.200		0.53	
1720	D2163	1.03	C	-2.99	first reported 0.91
normality		OK			
n		25			
outliers		5			
mean (n)		1.1746			
st.dev. (n)		0.05713			
R(calc.)		0.1600			
R(D2163:96)		0.1352			

Compare R(EN27941(liq)) = 1.3621

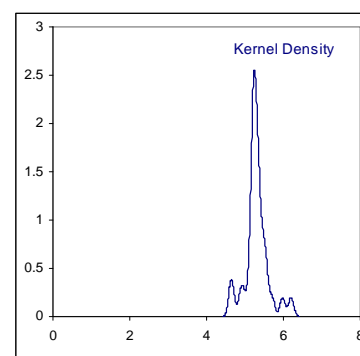
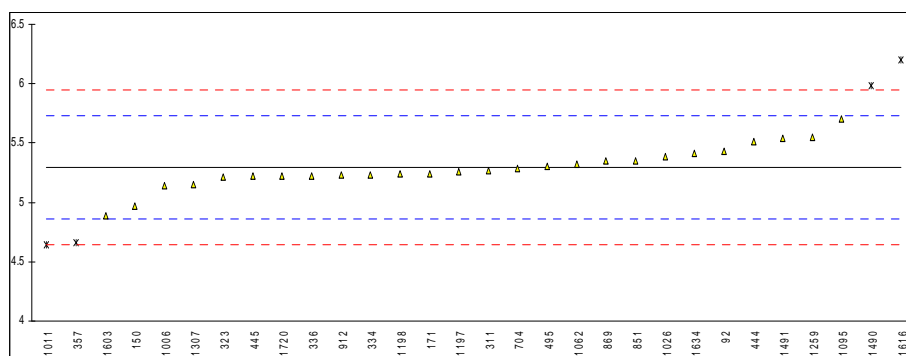


Determination of n-Butane on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	5.433		0.64	
150	D2163	4.969		-1.49	
171		5.241		-0.24	
311	D2163	5.267		-0.12	
317		-----		-----	
323	D2163	5.21		-0.38	
334	EN27941	5.234		-0.27	
336	EN27941	5.22		-0.34	
357	D2163	4.657	ex	-2.93	see §4.1
444	IP405	5.511		1.00	
445	D2163	5.22		-0.34	
495	D2163	5.303		0.04	
511		-----		-----	
704	D2163	5.282		-0.05	
851	D2163	5.348		0.25	
869	D2163	5.347		0.24	
912	D2163	5.23		-0.29	
1006	D2163	5.145		-0.68	
1011	ISO7941	4.64	ex	-3.00	see §4.1
1026	ISO7941	5.388		0.43	
1062		5.326		0.15	
1095	EN27941	5.706		1.89	
1197	D2163	5.263		-0.14	
1198	D2163	5.238		-0.26	
1259	EN27941	5.550		1.18	
1307	LHA	5.149		-0.67	
1490	EN27941	5.980	DG(0.05)	3.15	
1491	ISO7941	5.536		1.11	
1603	DIN51666	4.887		-1.87	
1616	D2163	6.203	DG(0.05)	4.18	
1634	ISO7941	5.415		0.56	
1720	D2163	5.22		-0.34	

normality OK  
n 26  
outliers 2  
mean (n) 5.2938  
st.dev. (n) 0.17251  
R(calc.) 0.4830  
R(D2163:96) 0.6095

Compare R(EN27941(liq)) = 0.9861



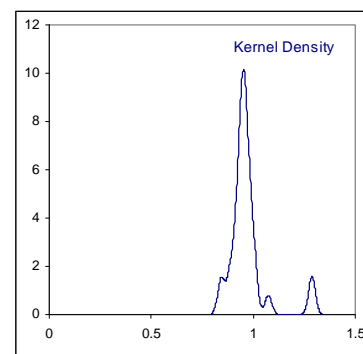
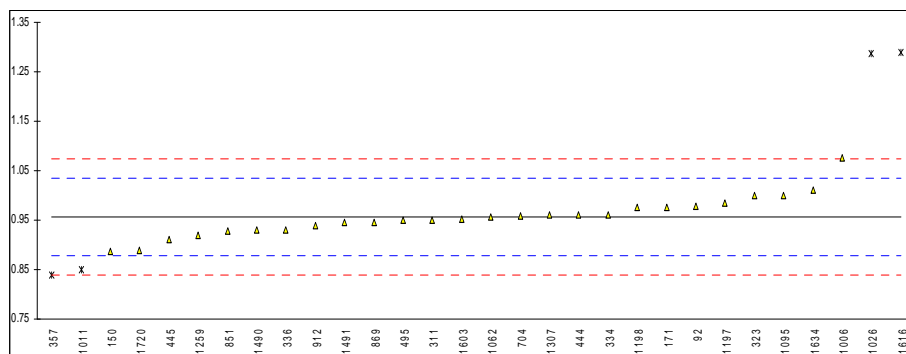


Determination of 1,3-Butadiene on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	0.978		0.54	
150	D2163	0.887		-1.77	
171		0.976		0.49	
311	D2163	0.950		-0.17	
317		-----		-----	
323	D2163	1.00		1.10	
334	EN27941	0.961		0.11	
336	EN27941	0.93		-0.68	
357	D2163	0.839	ex	-2.99	see §4.1
444	IP405	0.961		0.11	
445	D2163	0.91		-1.19	
495	D2163	0.950		-0.17	
511		-----		-----	
704	D2163	0.958		0.03	
851	D2163	0.929		-0.70	
869	D2163	0.946		-0.27	
912	D2163	0.94		-0.43	
1006	D2163	1.075		3.01	
1011	ISO7941	0.85	ex	-2.71	see §4.1
1026	ISO7941	1.288	G(0.01)	8.42	
1062		0.956		-0.02	
1095	EN27941	1.001		1.13	
1197	D2163	0.984		0.69	
1198	D2163	0.975		0.46	
1259	EN27941	0.920		-0.93	
1307	LHA	0.960		0.08	
1490	EN27941	0.930	C	-0.68	first reported 0.730
1491	ISO7941	0.945		-0.30	
1603	DIN51666	0.953		-0.09	
1616	D2163	1.290	G(0.01)	8.47	
1634	ISO7941	1.010		1.35	
1720	D2163	0.89		-1.70	

normality OK  
n 26  
outliers 2  
mean (n) 0.9567  
st.dev. (n) 0.03914  
R(calc.) 0.1096  
R(D2163:96) 0.1101

Compare R(EN27941(liq)) = 1.0596

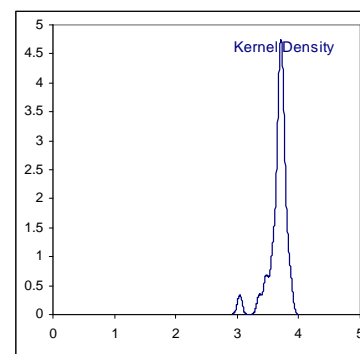
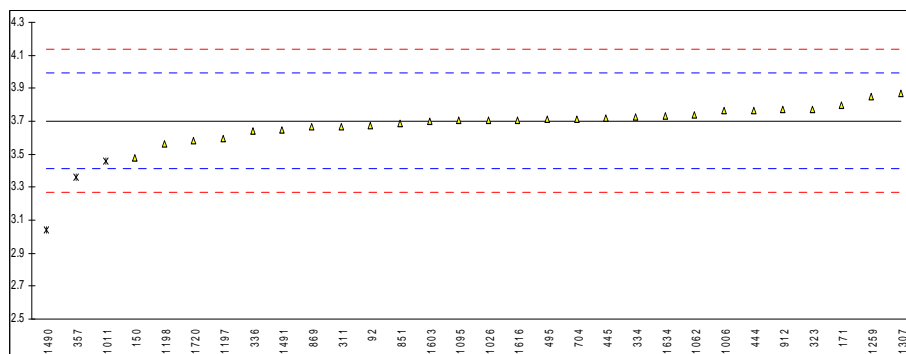


Determination of iso-Butylene on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	3.675		-0.18	
150	D2163	3.477		-1.48	
171		3.800		0.64	
311	D2163	3.670		-0.21	
317		-----		-----	
323	D2163	3.77		0.45	
334	EN27941	3.723		0.14	
336	EN27941	3.64		-0.41	
357	D2163	3.358	DG(0.05)	-2.26	
444	IP405	3.764		0.41	
445	D2163	3.72		0.12	
495	D2163	3.711		0.06	
511		-----		-----	
704	D2163	3.715		0.09	
851	D2163	3.684		-0.12	
869	D2163	3.669		-0.22	
912	D2163	3.77		0.45	
1006	D2163	3.762		0.40	
1011	ISO7941	3.46	DG(0.05)	-1.59	
1026	ISO7941	3.705		0.02	
1062		3.741		0.26	
1095	EN27941	3.704		0.01	
1197	D2163	3.594		-0.71	
1198	D2163	3.561		-0.93	
1259	EN27941	3.853		0.99	
1307	LHA	3.868		1.09	
1490	EN27941	3.040	C,G(0.01)	-4.35	first reported 0.000
1491	ISO7941	3.651		-0.33	
1603	DIN51666	3.703		0.01	
1616	D2163	3.705		0.02	
1634	ISO7941	3.735		0.22	
1720	D2163	3.58		-0.80	

normality OK  
n 27  
outliers 3  
mean (n) 3.7019  
st.dev. (n) 0.08480  
R(calc.) 0.2374  
R(D2163:96) 0.4262

Compare R(EN27941(liq)) = 1.0216

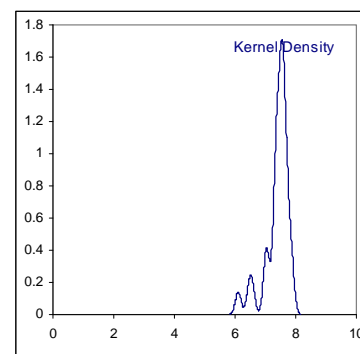
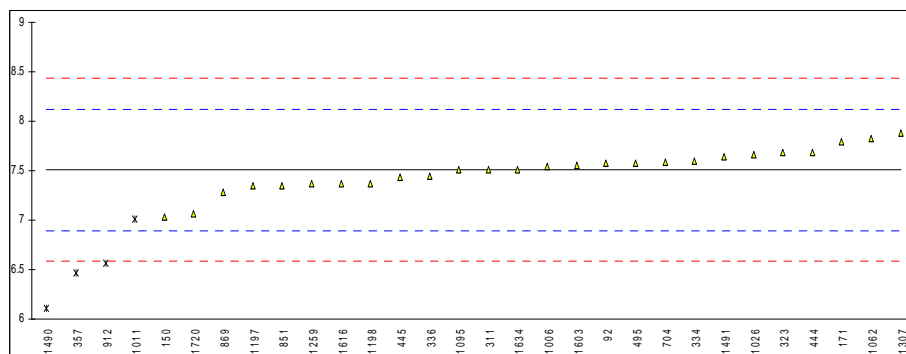


Determination of 1-Butene on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	7.572		0.20	
150	D2163	7.033		-1.54	
171		7.796		0.93	
311	D2163	7.510		0.00	
317		-----		-----	
323	D2163	7.68		0.55	
334	EN27941	7.596		0.28	
336	EN27941	7.45		-0.19	
357	D2163	6.470	G(0.05)	-3.36	
444	IP405	7.689		0.58	
445	D2163	7.44		-0.22	
495	D2163	7.574		0.21	
511		-----		-----	
704	D2163	7.585		0.25	
851	D2163	7.346		-0.53	
869	D2163	7.278		-0.75	
912	D2163	6.57	G(0.05)	-3.04	
1006	D2163	7.548		0.13	
1011	ISO7941	7.01	ex	-1.62	see §4.1
1026	ISO7941	7.664		0.50	
1062		7.829		1.04	
1095	EN27941	7.508		0.00	
1197	D2163	7.345		-0.53	
1198	D2163	7.375		-0.43	
1259	EN27941	7.368		-0.46	
1307	LHA	7.883		1.21	
1490	EN27941	6.105	C,G(0.05)	-4.55	first reported 11.660
1491	ISO7941	7.645		0.44	
1603	DIN51666	7.558		0.16	
1616	D2163	7.374		-0.44	
1634	ISO7941	7.515		0.02	
1720	D2163	7.07		-1.42	

normality OK  
n 26  
outliers 3  
mean (n) 7.5089  
st.dev. (n) 0.20442  
R(calc.) 0.5724  
R(D2163:96) 0.8645

Compare R(EN27941(liq)) = 1.0216

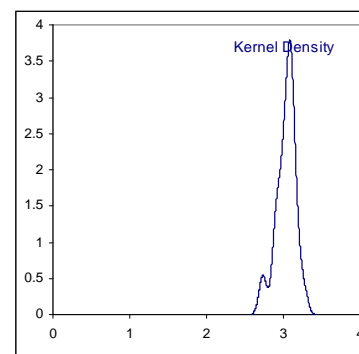
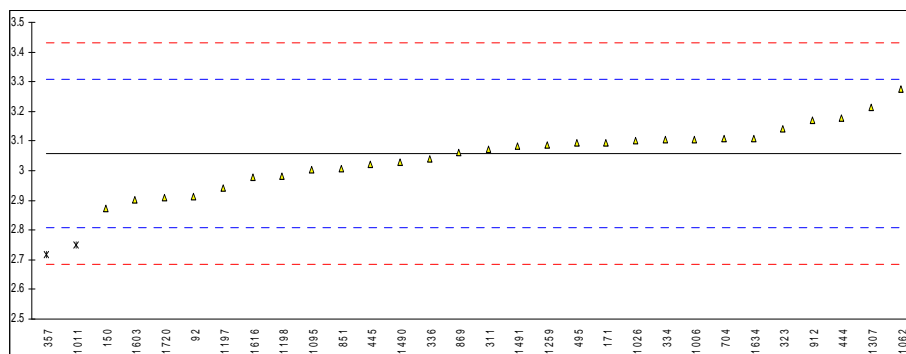


Determination of trans-2-Butene on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	2.914		-1.14	
150	D2163	2.872		-1.47	
171		3.094		0.29	
311	D2163	3.073		0.13	
317		-----		-----	
323	D2163	3.14		0.66	
334	EN27941	3.104		0.37	
336	EN27941	3.04		-0.13	
357	D2163	2.718	DG(0.05)	-2.70	
444	IP405	3.178		0.96	
445	D2163	3.02		-0.29	
495	D2163	3.093		0.29	
511		-----		-----	
704	D2163	3.109		0.41	
851	D2163	3.006		-0.41	
869	D2163	3.060		0.02	
912	D2163	3.17		0.90	
1006	D2163	3.105		0.38	
1011	ISO7941	2.75	DG(0.05)	-2.44	
1026	ISO7941	3.100		0.34	
1062		3.277		1.75	
1095	EN27941	3.003		-0.43	
1197	D2163	2.942		-0.91	
1198	D2163	2.982		-0.60	
1259	EN27941	3.087		0.24	
1307	LHA	3.212		1.23	
1490	EN27941	3.030		-0.21	
1491	ISO7941	3.084		0.22	
1603	DIN51666	2.902		-1.23	
1616	D2163	2.978		-0.63	
1634	ISO7941	3.110		0.42	
1720	D2163	2.91		-1.17	

normality OK  
n 28  
outliers 2  
mean (n) 3.0570  
st.dev. (n) 0.09746  
R(calc.) 0.2729  
R(D2163:96) 0.3519

Compare R(EN27941(liq)) = 1.0216

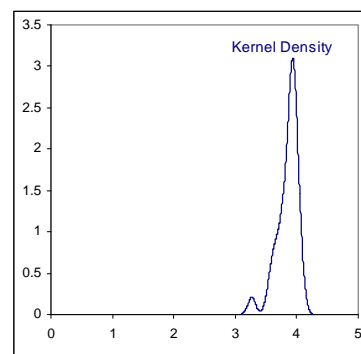
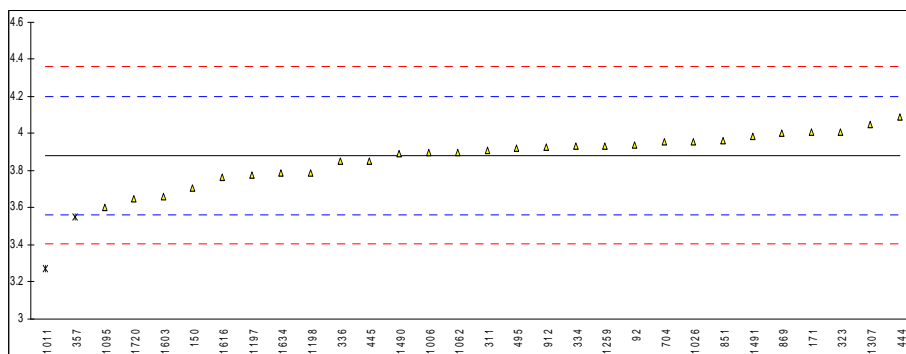


Determination of cis-2-Butene on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	3.937		0.34	
150	D2163	3.709		-1.09	
171		4.007		0.78	
311	D2163	3.910		0.17	
317		-----		-----	
323	D2163	4.01		0.80	
334	EN27941	3.932		0.31	
336	EN27941	3.85		-0.21	
357	D2163	3.551	ex	-2.08	see §4.1
444	IP405	4.091		1.30	
445	D2163	3.85		-0.21	
495	D2163	3.924		0.26	
511		-----		-----	
704	D2163	3.957		0.46	
851	D2163	3.963		0.50	
869	D2163	4.001		0.74	
912	D2163	3.93		0.29	
1006	D2163	3.896		0.08	
1011	ISO7941	3.27	G(0.01)	-3.84	
1026	ISO7941	3.958		0.47	
1062		3.898		0.09	
1095	EN27941	3.604		-1.75	
1197	D2163	3.776		-0.67	
1198	D2163	3.791		-0.58	
1259	EN27941	3.936		0.33	
1307	LHA	4.052		1.06	
1490	EN27941	3.890	C	0.04	first reported 4.990
1491	ISO7941	3.984		0.63	
1603	DIN51666	3.662		-1.38	
1616	D2163	3.767		-0.73	
1634	ISO7941	3.790		-0.58	
1720	D2163	3.65		-1.46	

normality OK  
n 28  
outliers 1  
mean (n) 3.8830  
st.dev. (n) 0.12377  
R(calc.) 0.3465  
R(D2163:96) 0.4470

Compare R(EN27941(liq)) = 1.0216

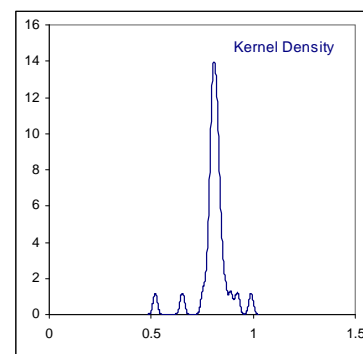
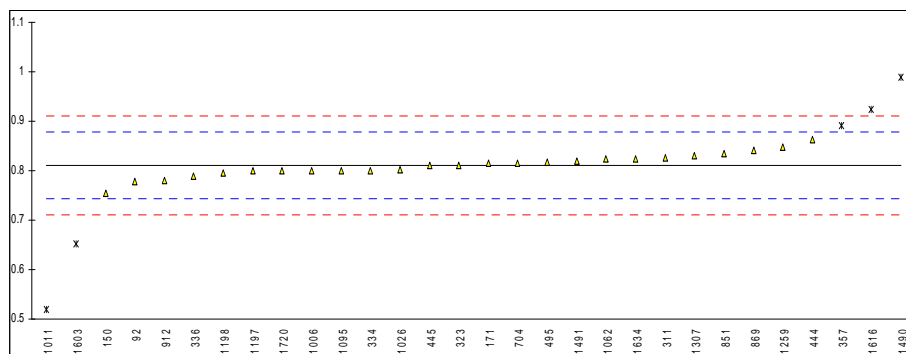


Determination of iso-Pentane on sample #12074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	0.778		-1.00	
150	D2163	0.755		-1.69	
171		0.815		0.11	
311	D2163	0.827		0.47	
317		-----		-----	
323	D2163	0.81		-0.04	
334	EN27941	0.801		-0.31	
336	EN27941	0.79		-0.64	
357	D2163	0.891	ex	2.38	see §4.1
444	IP405	0.863		1.55	
445	D2163	0.81		-0.04	
495	D2163	0.817		0.17	
511		-----		-----	
704	D2163	0.816		0.14	
851	D2163	0.834		0.68	
869	D2163	0.842		0.92	
912	D2163	0.78		-0.94	
1006	D2163	0.801		-0.31	
1011	ISO7941	0.52	G(0.01)	-8.74	
1026	ISO7941	0.802		-0.28	
1062		0.824		0.38	
1095	EN27941	0.801		-0.31	
1197	D2163	0.800		-0.34	
1198	D2163	0.796		-0.46	
1259	EN27941	0.848		1.10	
1307	LHA	0.831		0.59	
1490	EN27941	0.990	G(0.05)	5.35	
1491	ISO7941	0.820		0.26	
1603	DIN51666	0.653	G(0.05)	-4.75	
1616	D2163	0.923	G(0.05)	3.34	
1634	ISO7941	0.825		0.41	
1720	D2163	0.80		-0.34	

normality OK  
n 25  
outliers 4  
mean (n) 0.8114  
st.dev. (n) 0.02347  
R(calc.) 0.0657  
R(D2163:96) 0.0934

Compare R(EN27941(liq)) = 0.7944

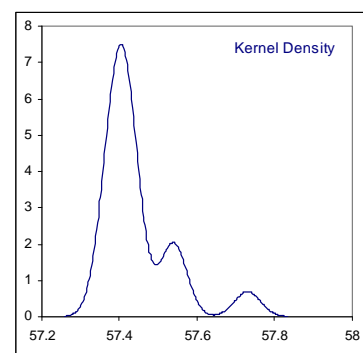
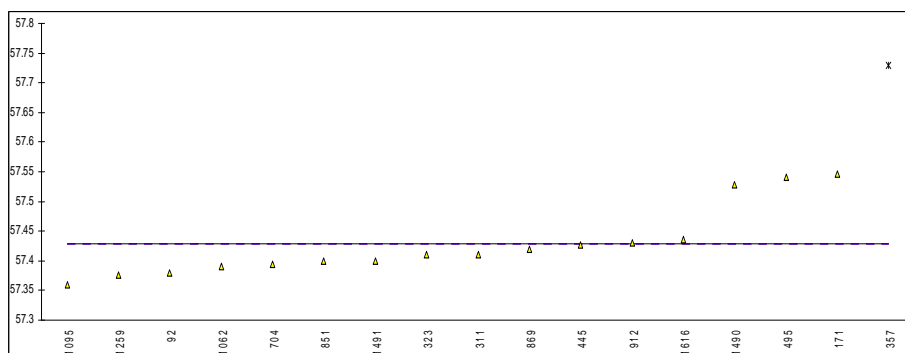


Determination of Molar Mass on sample #12074; results in g/mol

lab	method	value	mark	z(targ)	remarks
92	D2598	57.38		-----	
150		-----		-----	
171	D2421	57.547	E	-----	calculated by iis from the reported test results: 57.396 (see §4.4)
311	INH-407	57.41		-----	
317		-----		-----	
323	D3588	57.41		-----	
334		-----		-----	
336		-----		-----	
357	Calc.	57.73	G(0.01), E	-----	calculated by iis from the reported test results: 57.62 (see §4.4)
444		-----		-----	
445	IP432	57.427		-----	
495		57.541	E	-----	calculated by iis from the reported test results: 57.397 (see §4.4)
511		-----		-----	
704	D2163	57.3943		-----	
851	Calc.	57.40		-----	
869	Calc.	57.42		-----	
912	D2598	57.43		-----	
1006		-----		-----	
1011		-----		-----	
1026		-----		-----	
1062		57.3912		-----	
1095	D2421	57.36		-----	
1197		-----		-----	
1198		-----		-----	
1259	ISO8973	57.3757		-----	
1307		-----		-----	
1490	Calc.	57.529	C	-----	first reported 57.60
1491	ISO8973	57.40		-----	
1603		-----		-----	
1616	D2421	57.435		-----	
1634		-----		-----	
1720		-----		-----	

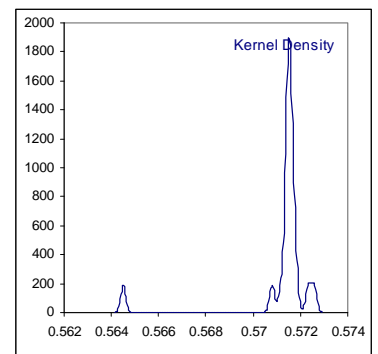
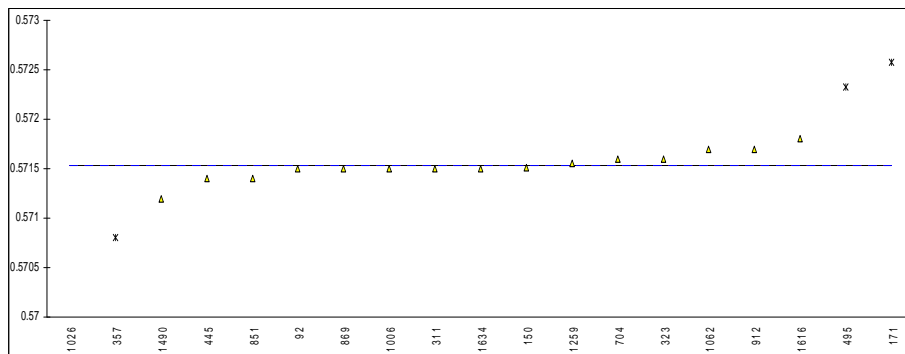
Calculated by iis from all reported test results:

normality	not OK	OK
n	16	25
outliers	1	5
mean (n)	57.428	57.392
st.dev. (n)	0.0587	0.0305
R(calc.)	0.164	0.086
R(lit.)	unknown	unknown



Determination of Relative Density @ 60F on sample #12074; unitless results

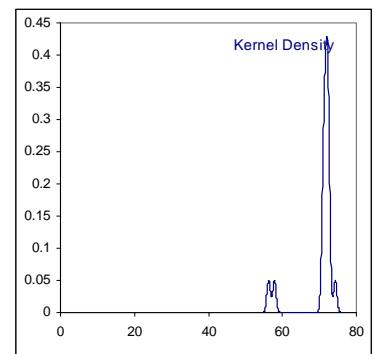
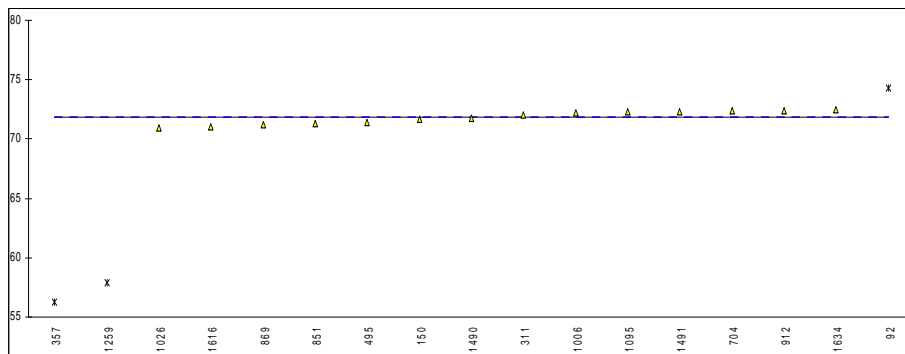
lab	method	value	mark	z(targ)	remarks
92	D2598	0.5715		-----	
150	D2598	0.57151	E	-----	calculated by iis from the reported test results: 0.5623 (see §4.4)
171	D2421	0.57258	DG(0.01)	-----	
311	INH-407	0.5715		-----	
317		-----		-----	
323	D2598	0.5716		-----	
334		-----		-----	
336		-----		-----	
357	D2598	0.5708	G(0.01)	-----	
444		-----		-----	
445	IP432	0.5714	C	-----	first reported 571.4
495	D2598	0.57233	C,DG(0.01)	-----	first reported 572.334
511		-----		-----	
704	D2598	0.5716		-----	
851	D2598	0.5714		-----	
869	D2598	0.5715		-----	
912	D2598	0.5717		-----	
1006	D2598	0.5715		-----	
1011		-----		-----	
1026	ISO8973	0.5645	C,G(0.01)	-----	first reported 564.5
1062		0.5717		-----	
1095		-----		-----	
1197		-----		-----	
1198		-----		-----	
1259	ISO8973	0.571553		-----	
1307		-----		-----	
1490	ISO8973	0.571197	C	-----	first reported 572.6
1491		-----		-----	
1603		-----		-----	
1616	D2421	0.5718		-----	
1634	ISO8973	0.5715	C	-----	first reported 571.5
1720		-----		-----	
					<u>Calculated by iis from all reported test results:</u>
normality		OK		OK	
n		15		26	
outliers		4		4	
mean (n)		0.57153		0.57197	
st.dev. (n)		0.000144		0.000292	
R(calc.)		0.00040		0.00082	
R(lit.)		unknown		unknown	





Determination of Abs. Vapour Pressure on sample #12074; results in psi

lab	method	value	mark	z(targ)	remarks
92	D2598	74.30	G(0.05), E	----	calculated by iis from the reported test results: 72.48 (see §4.4)
150	D2598	71.7093		----	
171		----		----	
311	ISO8973	72		----	
317		----		----	
323		----		----	
334		----		----	
336		----		----	
357	ISO8973	56.295	G(0.05), E	----	calculated by iis from the reported test results: 71.42 (see §4.4)
444		----		----	
445		----		----	
495	D2598	71.403		----	
511		----		----	
704	ISO8973	72.36		----	
851	D2598	71.3		----	
869	D2598	71.2		----	
912	IP432	72.37		----	
1006	ISO8973	72.2	C	----	first reported 75.9
1011		----		----	
1026		70.92		----	
1062		----		----	
1095	ISO8973	72.3086		----	
1197		----		----	
1198		----		----	
1259	ISO8973	57.8549	G(0.01), E	----	calculated by iis from the reported test results: 72.54 (see §4.4)
1307		----		----	
1490	ISO8973	71.78	C	----	first reported 70.92
1491	ISO8973	72.33		----	
1603		----		----	
1616	in house	71.038		----	
1634	ISO8973	72.448	C	----	first reported 57.752
1720		----		----	
	normality	OK			Calculated by iis from all reported test results:
	n	14			not OK
	outliers	3			29
	mean (n)	71.81			72.34
	st.dev. (n)	0.550			0.410
	R(calc.)	1.54			1.15
	R(lit.)	unknown			unknown

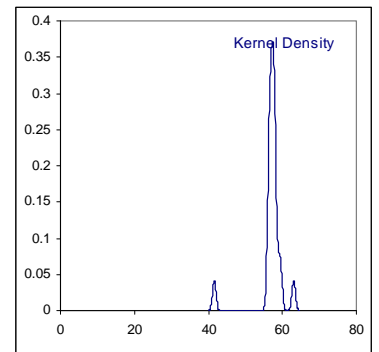
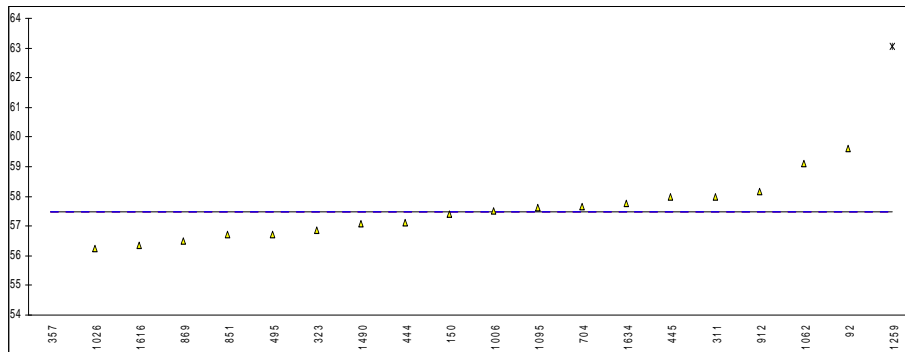


Determination of Rel. Vapour Pressure on sample #12074; results in psi

lab	method	value	mark	z(targ)	remarks
92	D2598	59.61	E	----	calculated by iis from the reported test results: 57.79 (see §4.4)
150	D2598	57.402		----	
171		----		----	
311	ISO8973	58		----	
317		----		----	
323		56.88		----	
334		----		----	
336		----		----	
357	ISO8973	41.603	G(0.01), E	----	calculated by iis from the reported test results: 56.72 (see §4.4)
444	IP432	57.1		----	
445	IP432	58		----	
495	D2598	56.707		----	
511		----		----	
704	ISO8973	57.66		----	
851	D2598	56.7		----	
869	D2598	56.5		----	
912	IP432	58.15		----	
1006	ISO8973	57.5	C	----	first reported 61.2
1011		----		----	
1026		56.23		----	
1062		59.1		----	
1095	ISO8973	57.6123		----	
1197		----		----	
1198		----		----	
1259	ISO8973	63.0565	G(0.01), E	----	calculated by iis from the reported test results: 57.84 (see §4.4)
1307		----		----	
1490	ISO8973	57.08	C	----	first reported 56.22
1491		----		----	
1603		----		----	
1616	in house	56.342		----	
1634	ISO8973	57.752		----	
1720		----		----	

Calculated by iis from all reported test results:

normality	OK	not OK
n	18	29
outliers	2	1
mean (n)	57.46	57.64
st.dev. (n)	0.906	0.410
R(calc.)	2.54	1.15
R(lit.)	unknown	unknown



**APPENDIX 2****Additional details**

	Sample Volume	Type of vaporizer	Remarks
92	---	---	none
150	---	---	none
171	108.76 g	---	0.003 %M/M n-pentane
311	31.5 ml	---	< 0.003 %M/M n-pentane
317	---	---	---
323	---	---	0.004 %M/M n-pentane
334	---	---	none
336	---	---	none
357	---	---	0.006 %M/M n-pentane
444	---	---	0.0023 %M/M n-pentane
445	---	---	none
495	41 ml	waterbath	none
511	---	---	---
704	0.0005 ml	SPL	n-pentane present, but <0.01 %mol/mol
851	50 ml	Liquid injection	none
869	20 ml	---	none
912	200 µl	GSV@60°C	none
1006	---	---	none
1011	---	---	---
1026	355 g	Tedlar bag	0.0022 %mol/mol n-pentane; 0.0016%mol/mol methane
1062	---	---	0.003 %mol/mol n-pentane; 0.008%mol/mol acetylene
1095	---	---	n-pentane present
1197	---	---	---
1198	---	---	---
1259	---	---	none
1307	---	---	0.0023 %mol/mol C5+
1490	200 ml	PTV	0.02 %mol/mol 1-pentene
1491	---	---	---
1603	---	---	0.002 %mol/mol n-pentane; 0.175 %mol/mol helium
1616	---	---	---
1634	---	---	---
1720	---	---	---

## **APPENDIX 3**

### **Number of participants per country**

3 labs in BELGIUM  
1 lab in CANADA  
1 lab in CROATIA  
1 lab in FINLAND  
2 labs in FRANCE  
2 labs in GERMANY  
1 lab in HONG KONG  
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 QATAR  
1 lab in SERBIA  
1 lab in SUDAN  
1 lab in TAIWAN R.O.C.  
3 labs in THE NETHERLANDS  
2 labs in U.S.A.  
1 lab in UKRAINE  
2 labs in UNITED KINGDOM

## APPENDIX 4

### Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
ex	= excluded from calculations
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.
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- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 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
- 16 EN 27941
- 17 ASTM D2163
- 18 ASTM D2421