Results of Proficiency Test
Liquefied Butane Analysis
May 2011

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis11S02B

August 2011

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1 INTRODUCTION

Beginning 2008, iis started an investigation for the feasibility of a PT on Liquefied Butane Analysis. 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 Butane samples for PT purposes. Together with this company, it was decided to organize a first proficiency study for Liquefied Butane (composition only) in 2009. This PT was repeated in 2010 and 2011.
This year 29 laboratories in 19 different countries have participated. See appendix 3 for the number of participants per country. In this report the results of the 2011 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 35 cylinders.
The cylinder size is a cost-effective two-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 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 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).

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 artificial Liquefied Butane mixture was prepared and tested for homogeneity by Scott Specialty Gases (Breda, the Netherlands) in conformance with ISO 6143 and ISO Guide 35.
In total one batch of 35 cylinders was prepared (lot 11048) on May 03, 2011. Each cylinder was uniquely numbered. The 35 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
Propane	1.930	0.055	0.067
Propylene	1.310	0.050	0.045
n-Butane	5.245	0.038	0.181
1,3-Butadiene	0.963	0.002	0.033
iso-Butylene	2.990	0.007	0.103
1-Butene	5.906	0.023	0.204
trans-2-Butene	2.393	0.016	0.083
cis-2-Butene	3.065	0.021	0.106
iso-Pentane	0.834	0.014	0.029
iso-Butane	75.363	0.038	0.226

Table 1: homogeneity test results of samples #11048

The calculated repeatabilities are each less than 0.3 times the corresponding reproducibility of the reference method ASTM D2163:96, except for propylene.
Therefore, homogeneity of the subsamples #11048 was assumed.

To each of the participating laboratories one 2L cylinder was sent on May 14, 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: 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 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 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.

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 3; nr.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(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 customs problems three 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 the test results after the final reporting date. Not all laboratories were able to report all test results requested.
Two laboratories appeared to have some problems. Six test results (=60%) reported by laboratory 1284 and five test results (=50%) 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 1011 and 1284 were excluded manually from the statistical analysis.
Because 17 laboratories reported both results in %mol/mol as well as in %M/M, it has been possible to check the calculations of these 17 laboratories. A good correclation 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 27 participants reported 333 numerical results. Observed were 29 outlying results, which is 8.7% 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, 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.
All original data sets proved to have a normal distribution, except for the n-Butane and n-Pentane 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. Regretfully the last version ASTM D2163:07 contains only provisional repeatability standard deviations, determined by statistical examination of limited interlaboratory results and no reproducibilies are mentioned. Therefore the precision data from the previous version ASTM D2163:96 were used.

Propane: The determination of this component may be problematic. 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 of EN27941 (identical to IP 405 and ISO 7941).

Propylene: The determination of this component was problematic. 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. And the calculated reproducibility is also not in agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).

n-Butane: No large analytical problems were observed. Three 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 less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).

1,3-Butadiene: The determination of this component may be problematic. No statistical outliers were detected. However, the calculated reproducibility is not in agreement with the requirements of ASTM D2163:96. But the calculated reproducibility is in good agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).

iso-Butylene: No analytical problems were observed. No statistical outliers were detected. Also, the calculated reproducibility 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-Butene: No analytical problems were observed. Only one statistical outlier was detected. 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 of EN27941 (identical to IP 405 and ISO 7941).

trans-2-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).

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: 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 agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).

iso-Butane: The determination of this main component may be problematic. Only one statistical outlier was detected. However, the calculated reproducibility is not in agreement with the requirements of ASTM D2163:96. But the calculated reproducibility is in agreement with the less strict 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 57.49 - 57.66 g/mol and only one statistically significant outlier was observed (in 11 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 0.5699 - 0.5871 and two statistically significant outliers were observed (in 16 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 (59 – 130.6 psi) and three statistically significant outliers were observed. See also the discussion in 4.4.

Rel. Vapour Pres.: This calculated parameter may be problematic. The results vary over a large range (44 – 61.59 psi) and two statistically significant outliers were observed. 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**
Propane	%mol/mol	23	1.635	0.363	0.183	1.295	1
Propylene	%mol/mol	23	1.033	0.306	0.116	1.357	1
n-Butane	%mol/mol	24	5.567	0.575	0.641	0.982	1
1,3-Butadiene	%mol/mol	27	0.972	0.132	0.112	1.056	1
iso-Butylene	%mol/mol	25	3.002	0.274	0.345	1.018	1
1-Butene	%mol/mol	24	5.956	0.590	0.684	1.018	1
trans-2-Butene	%mol/mol	23	2.501	0.261	0.287	1.018	1
cis-2-Butene	%mol/mol	25	2.954	0.308	0.340	1.018	1
iso-Pentane	%mol/mol	22	0.949	0.130	0.109	0.791	1
iso-Butane	%mol/mol	25	75.353	1.035	0.754	1.474	1.5
Molar Mass	g/mol	10	57.53	0.089	n/a	n/a	n/a
Relative Density		14	0.5703	0.0006	n/a	n/a	n/a
Abs. Vapour pres.	psi	10	72.03	1.71	n/a	n/a	n/a
Rel. Vapour pres.	psi	11	57.47	1.51	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 MAY 2011 WITH PREVIOUS PTS

	May 2011	May 2010	July 2009
Number of reporting labs	27	22	25
Number of results reported	333	263	291
Statistical outliers	29	20	16
Percentage outliers	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:

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

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 Scott Specialty Gases in the following table. From this comparison it is clear that most consensus values as determined in this PT are well in line with the values as determined during the preparation of the gas cylinders except for Propane and propylene (z-scores in bold). No explanation can be given for these observations.

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
Propane	1.930	1.635	-0.295	**-4.50**
Propylene	1.310	1.033	-0.227	**-6.68**
n-Butane	5.245	5.567	+0.322	+1.41
1,3-Butadiene	0.964	0.972	+0.008	+0.24
iso-Butylene	2.990	3.002	+0.012	-0.10
1-Butene	5.906	5.956	+0.050	-0.20
trans-2-Butene	2.393	2.501	+0.108	+1.05
cis-2-Butene	3.066	2.954	-0.112	-0.92
iso-Pentane	0.834	0.949	+0.115	+2.94
iso-Butane	75.363	75.353	-0.010	-0.04

Table 5: comparison of consensus values with values determined by Scott Specialty Gases

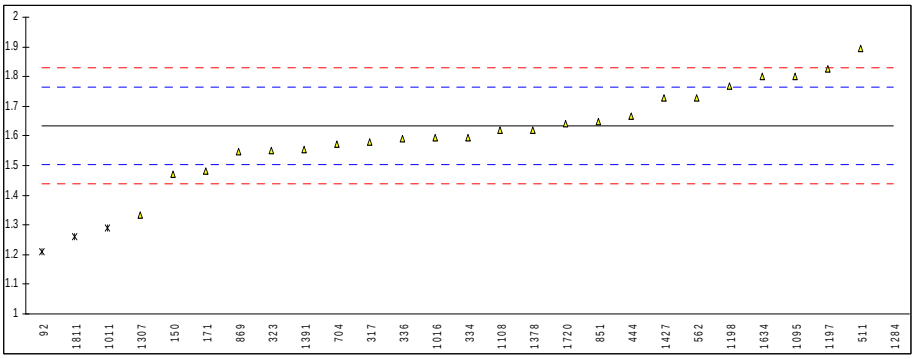
In total eleven laboratories reported the presence of some n-pentane (0.005 %mol/mol with std. dev. = 0.002 %mol/mol), a component probably present as impurity in one or more of the pure components that were used to prepare the iso-Butane mixture.

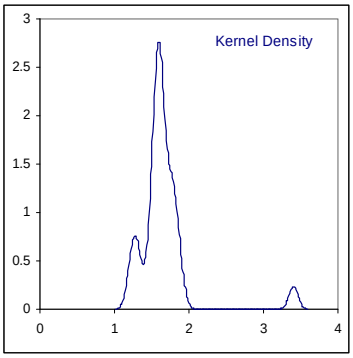
In principle no additional spread should be introduced when applying a calculation on the reported component concentrations. However, in practice a significant additional uncertainty is added. See the differences between the values from the results as reported by the participating laboratories (each 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 Propane on sample #11048; results in %mol/mol

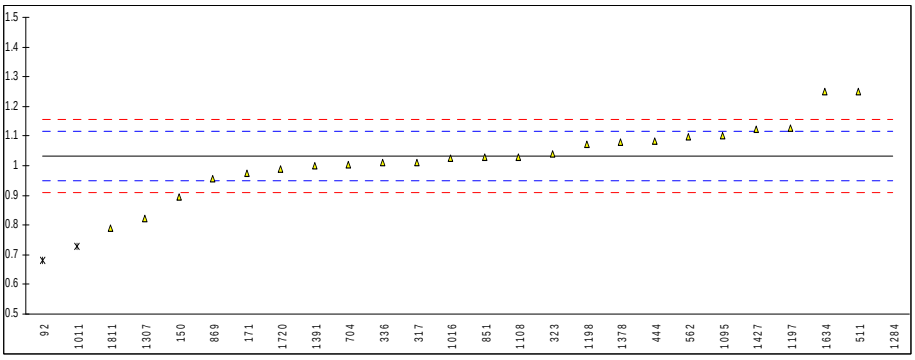
lab	method	value	mark	z(targ)	remarks
92	D2163	1.21	C,DG(0.05)	-6.50	First reported 1.90
150	D2598	1.472		-2.49	
171	D2163	1.481		-2.36	
311		-----		-----	
317	D2163	1.58		-0.84	
323	D2163	1.55		-1.30	
334	ISO 8973	1.596		-0.60	
336	EN27941	1.59		-0.69	
444	IP405	1.665		0.46	
511	D2163	1.895		3.97	
562	D2163	1.727		1.40	
704	D2163	1.572		-0.97	
786		-----		-----	
851		1.649		0.21	
869	D2163	1.548		-1.33	
912		-----		-----	
1011	EN 27941	1.29	C, ex	-5.28	See §4.1; First reported 1.71
1016	ISO7941	1.595		-0.61	
1095	EN 27941	1.80		2.52	
1108	EN27941	1.62		-0.23	
1197	D2163	1.827		2.93	
1198	D2163	1.769		2.04	
1284	D2163	3.404	G(0.01)	27.03	
1307	LHA-GC	1.333		-4.62	
1368		-----		-----	
1369		-----		-----	
1378	ISO7941	1.62		-0.23	
1391	D2163	1.554		-1.24	
1427	EN27941	1.727		1.40	
1634	ISO8973	1.80		2.52	
1720	D2163	1.64		0.07	
1811		1.260	C,DG(0.05)	-5.73	First reported 2.630
	normality	OK			
	n	23			
	outliers	3			
	mean (n)	1.6352			
	st.dev. (n)	0.12975			
	R(calc.)	0.3633			
	R(D2163:96)	0.1832			Compare R(EN27941(liq))=0.6473

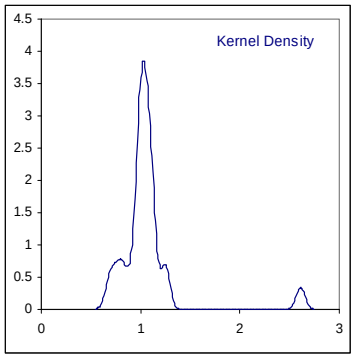




Determination of Propylene on sample #11048; results in %mol/mol

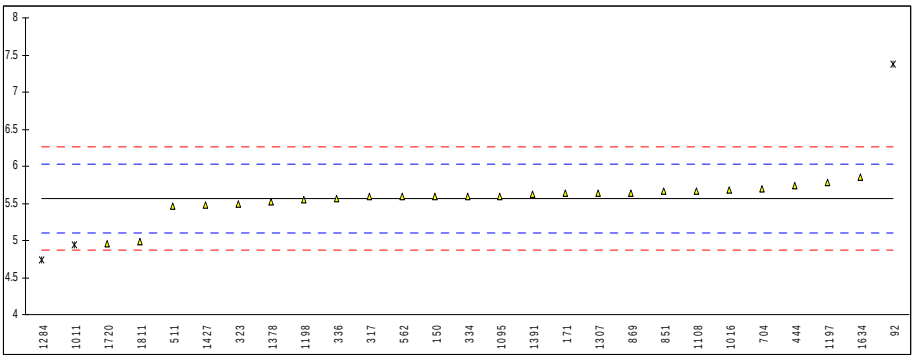
lab	method	value	mark	z(targ)	remarks
92	D2163	0.68	C,DG(0.05)	-8.53	First reported 1.05
150	D2598	0.896		-3.31	
171	D2163	0.974		-1.43	
311		-----		-----	
317	D2163	1.01		-0.56	
323	D2163	1.04		0.17	
334		-----		-----	
336	EN27941	1.01		-0.56	
444	IP405	1.082		1.18	
511	D2163	1.25		5.24	
562	D2163	1.099		1.59	
704	D2163	1.002		-0.75	
786		-----		-----	
851		1.028		-0.12	
869	D2163	0.956		-1.86	
912		-----		-----	
1011	EN 27941	0.73	C,DG(0.05)	-7.32	First reported 1.06
1016	ISO7941	1.024		-0.22	
1095	EN 27941	1.10		1.62	
1108	EN27941	1.03		-0.07	
1197	D2163	1.126		2.25	
1198	D2163	1.074		0.99	
1284	D2163	2.612	G(0.01)	38.15	
1307	LHA-GC	0.823		-5.07	
1368		-----		-----	
1369		-----		-----	
1378	ISO7941	1.08		1.13	
1391	D2163	1.001		-0.77	
1427	EN27941	1.125		2.22	
1634	ISO8973	1.25		5.24	
1720	D2163	0.99		-1.04	
1811		0.79	C	-5.87	First reported 1.930
	normality	OK			
	n	23			
	outliers	3			
	mean (n)	1.0330			
	st.dev. (n)	0.10930			
	R(calc.)	0.3060			
	R(D2163:96)	0.1159			Compare R(EN27941(liq))=0.2713

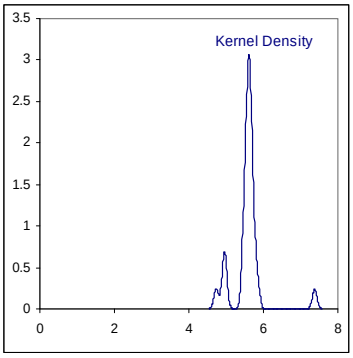




Determination of n-Butane on sample #11048; results in %mol/mol

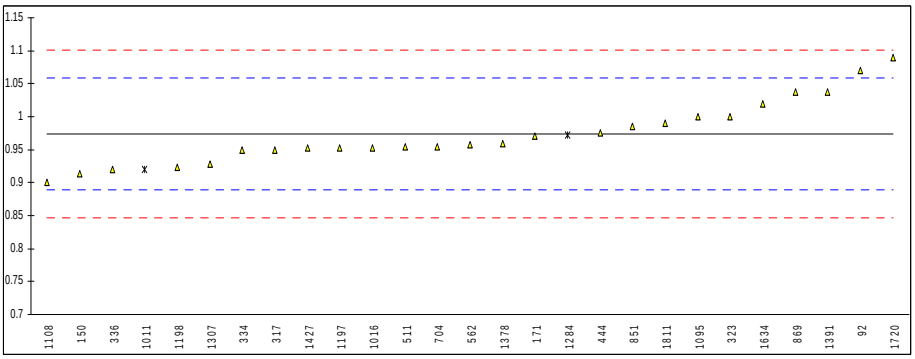
lab	method	value	mark	z(targ)	remarks
92	D2163	7.38	C,G(0.01)	7.92	First reported 9.20
150	D2598	5.593		0.11	
171	D2163	5.635		0.30	
311		-----		-----	
317	D2163	5.59		0.10	
323	D2163	5.49		-0.34	
334	ISO 8973	5.599		0.14	
336	EN27941	5.57		0.01	
444	IP405	5.746		0.78	
511	D2163	5.465		-0.45	
562	D2163	5.590		0.10	
704	D2163	5.692		0.54	
786		-----		-----	
851		5.665		0.43	
869	D2163	5.642		0.33	
912		-----		-----	
1011	EN 27941	4.94	C,DG(0.05)	-2.74	First reported 4.78
1016	ISO7941	5.686		0.52	
1095	EN 27941	5.60		0.14	
1108	EN27941	5.67		0.45	
1197	D2163	5.778		0.92	
1198	D2163	5.551		-0.07	
1284	D2163	4.734	DG(0.05)	-3.64	
1307	LHA-GC	5.640		0.32	
1368		-----		-----	
1369		-----		-----	
1378	ISO7941	5.52		-0.21	
1391	D2163	5.628		0.26	
1427	EN27941	5.480		-0.38	
1634	ISO8973	5.85		1.23	
1720	D2163	4.96		-2.65	
1811		4.979		-2.57	
	normality	not OK			
	n	24			
	outliers	3			
	mean (n)	5.5674			
	st.dev. (n)	0.20540			
	R(calc.)	0.5751			
	R(D2163:96)	0.6409			Compare R(EN27941(liq))=0.9822

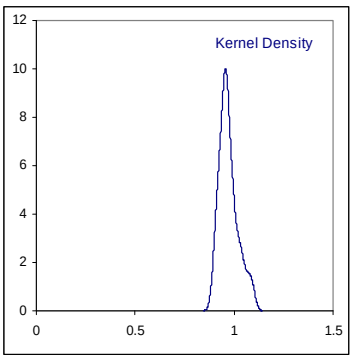




Determination of 1,3-Butadiene on sample #11048; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	1.07		2.26	
150	D2598	0.914		-1.41	
171	D2163	0.971		-0.07	
311		-----		-----	
317	D2163	0.95		-0.56	
323	D2163	1.00		0.62	
334	ISO 8973	0.949		-0.58	
336	EN27941	0.92		-1.27	
444	IP405	0.975		0.03	
511	D2163	0.95		-0.47	
562	D2163	0.958		-0.37	
704	D2163	0.955		-0.44	
786		-----		-----	
851		0.985		0.26	
869	D2163	1.037		1.49	
912		-----		-----	
1011	EN 27941	0.92	C, ex	-1.27	See §4.1; First reported 0.94
1016	ISO7941	0.953		-0.49	
1095	EN 27941	1.00		0.62	
1108	EN27941	0.90		-1.74	
1197	D2163	0.952		-0.51	
1198	D2163	0.924		-1.17	
1284	D2163	0.973	ex	-0.02	See §4.1
1307	LHA-GC	0.929		-1.06	
1368		-----		-----	
1369		-----		-----	
1378	ISO7941	0.96		-0.33	
1391	D2163	1.038		1.51	
1427	EN27941	0.952		-0.51	
1634	ISO8973	1.02		1.09	
1720	D2163	1.09	C	2.73	First reported 0.74
1811		0.990		0.38	
	normality	OK			
	n	27			
	outliers	0			
	mean (n)	0.9718			
	st.dev. (n)	0.04712			
	R(calc.)	0.1319			
	R(D2163:96)	0.1119			Compare R(EN27941(liq))=0.2111



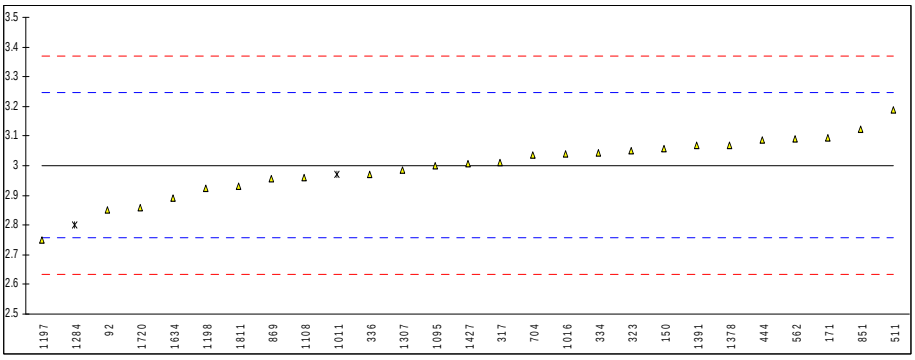


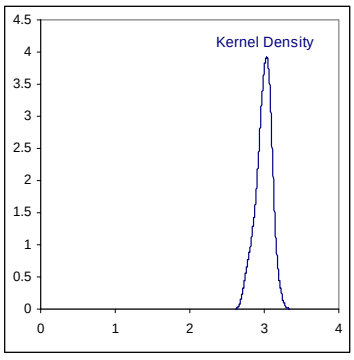
Determination of iso-Butylene on sample #11048; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	2.85		-1.23	
150	D2598	3.058		0.46	
171	D2163	3.095		0.76	
311		-----		-----	
317	D2163	3.01		0.07	
323	D2163	3.05		0.39	
334	ISO 8973	3.043		0.34	
336	EN27941	2.97		-0.26	
444	IP405	3.087		0.69	
511	D2163	3.190		1.53	
562	D2163	3.091		0.73	
704	D2163	3.037		0.29	
786		-----		-----	
851		3.123		0.99	
869	D2163	2.955		-0.38	
912		-----		-----	
1011	EN 27941	2.97	C, ex	-0.26	See §4.1; First reported 2.95
1016	ISO7941	3.039		0.30	
1095	EN 27941	3.00		-0.01	
1108	EN27941	2.96		-0.34	
1197	D2163	2.749		-2.05	
1198	D2163	2.924		-0.63	
1284	D2163	2.801	ex	-1.63	See §4.1
1307	LHA-GC	2.986		-0.13	
1368		-----		-----	
1369		-----		-----	
1378	ISO7941	3.07		0.55	
1391	D2163	3.069		0.55	
1427	EN27941	3.007		0.04	
1634	ISO8973	2.89		-0.91	
1720	D2163	2.86		-1.15	
1811		2.930		-0.58	

	normality	OK
	n	25
	outliers	0
	mean (n)	3.0017
	st.dev. (n)	0.09769
	R(calc.)	0.2735
	R(D2163:96)	0.3446

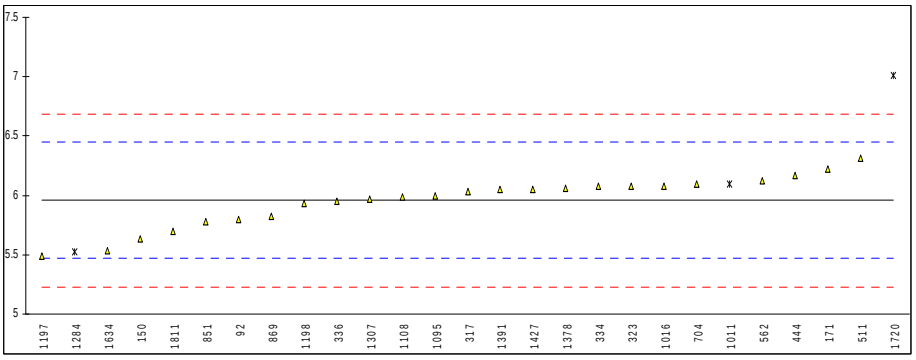
Compare R(EN27941(liq))=0.5088

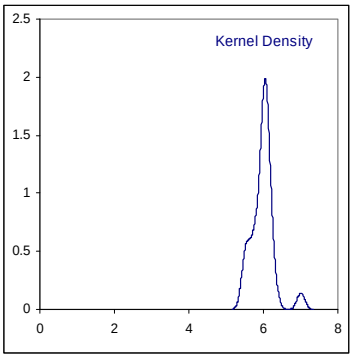




Determination of 1-Butene on sample #11048; results in %mol/mol

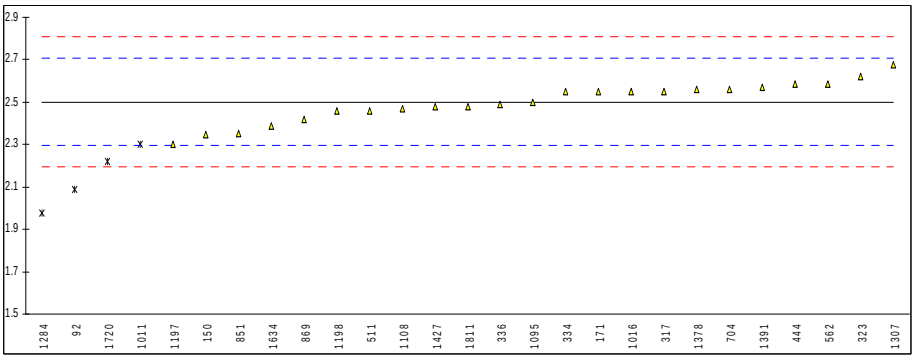
lab	method	value	mark	z(targ)	remarks
92	D2163	5.80		-0.64	
150	D2598	5.633		-1.32	
171	D2163	6.224		1.10	
311		-----		-----	
317	D2163	6.03		0.30	
323	D2163	6.08		0.51	
334	ISO 8973	6.079		0.50	
336	EN27941	5.95		-0.02	
444	IP405	6.170		0.88	
511	D2163	6.31		1.45	
562	D2163	6.122		0.68	
704	D2163	6.100		0.59	
786		-----		-----	
851		5.783		-0.71	
869	D2163	5.821		-0.55	
912		-----		-----	
1011	EN 27941	6.10	C, ex	0.59	See §4.1; First reported 6.04
1016	ISO7941	6.080		0.51	
1095	EN 27941	6.00		0.18	
1108	EN27941	5.99		0.14	
1197	D2163	5.487		-1.92	
1198	D2163	5.930		-0.11	
1284	D2163	5.529	ex	-1.75	See §4.1
1307	LHA-GC	5.970		0.06	
1368		-----		-----	
1369		-----		-----	
1378	ISO7941	6.06		0.43	
1391	D2163	6.047		0.37	
1427	EN27941	6.051		0.39	
1634	ISO8973	5.53		-1.74	
1720	D2163	7.01	C,G(0.01)	4.31	First reported 6.64
1811		5.696		-1.06	
	normality	OK			
	n	24			
	outliers	1			
	mean (n)	5.9560			
	st.dev. (n)	0.21065			
	R(calc.)	0.5898			
	R(D2163:96)	0.6844			Compare R(EN27941(liq))=1.0177

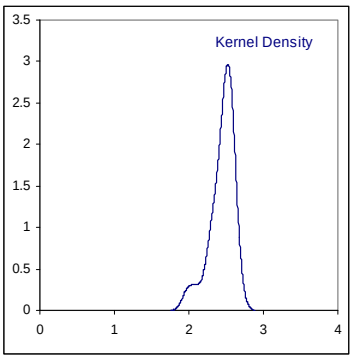




Determination of trans-2-Butene on sample #11048; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	2.09	DG(0.05)	-4.01	
150	D2598	2.345		-1.52	
171	D2163	2.550		0.48	
311		-----		-----	
317	D2163	2.55		0.48	
323	D2163	2.62		1.17	
334	ISO 8973	2.550		0.48	
336	EN27941	2.49		-0.10	
444	IP405	2.588		0.85	
511	D2163	2.46		-0.40	
562	D2163	2.588		0.85	
704	D2163	2.562		0.60	
786		-----		-----	
851		2.354		-1.43	
869	D2163	2.420		-0.79	
912		-----		-----	
1011	EN 27941	2.30	C, ex	-1.96	See §4.1; First reported 2.23
1016	ISO7941	2.550		0.48	
1095	EN 27941	2.50		-0.01	
1108	EN27941	2.47		-0.30	
1197	D2163	2.303		-1.93	
1198	D2163	2.459		-0.41	
1284	D2163	1.976	G(0.05)	-5.12	
1307	LHA-GC	2.678		1.73	
1368		-----		-----	
1369		-----		-----	
1378	ISO7941	2.56		0.58	
1391	D2163	2.568		0.66	
1427	EN27941	2.479		-0.21	
1634	ISO8973	2.39		-1.08	
1720	D2163	2.22	DG(0.05)	-2.74	
1811		2.48	C	-0.20	First reported 2.206
	normality	OK			
	n	23			
	outliers	3			
	mean (n)	2.5006			
	st.dev. (n)	0.09328			
	R(calc.)	0.2612			
	R(D2163:96)	0.2869			Compare R(EN27941(liq))=0.5089

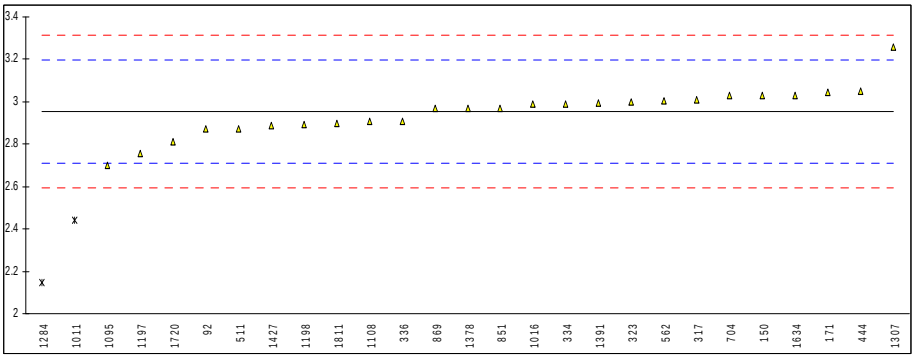


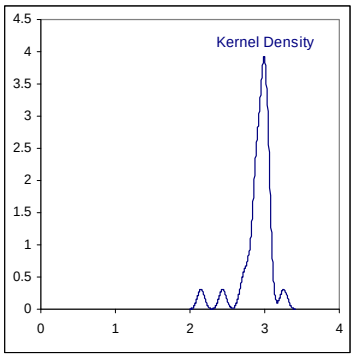


Determination of cis-2-Butene on sample #11048; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	2.87		-0.69	
150	D2598	3.029		0.62	
171	D2163	3.044		0.74	
311		-----		-----	
317	D2163	3.01		0.46	
323	D2163	3.00		0.38	
334	ISO 8973	2.991		0.31	
336	EN27941	2.91		-0.36	
444	IP405	3.050		0.79	
511	D2163	2.87		-0.69	
562	D2163	3.002		0.40	
704	D2163	3.028		0.61	
786		-----		-----	
851		2.971		0.14	
869	D2163	2.967		0.11	
912		-----		-----	
1011	EN 27941	2.44	C,G(0.01)	-4.23	First reported 2.38
1016	ISO7941	2.988		0.28	
1095	EN 27941	2.70		-2.09	
1108	EN27941	2.91		-0.36	
1197	D2163	2.758		-1.61	
1198	D2163	2.895		-0.48	
1284	D2163	2.148	G(0.01)	-6.63	
1307	LHA-GC	3.257		2.50	
1368		-----		-----	
1369		-----		-----	
1378	ISO7941	2.97		0.13	
1391	D2163	2.996		0.35	
1427	EN27941	2.890		-0.53	
1634	ISO8973	3.03		0.63	
1720	D2163	2.81	C	-1.18	First reported 2.59
1811		2.90	C	-0.44	First reported 2.516

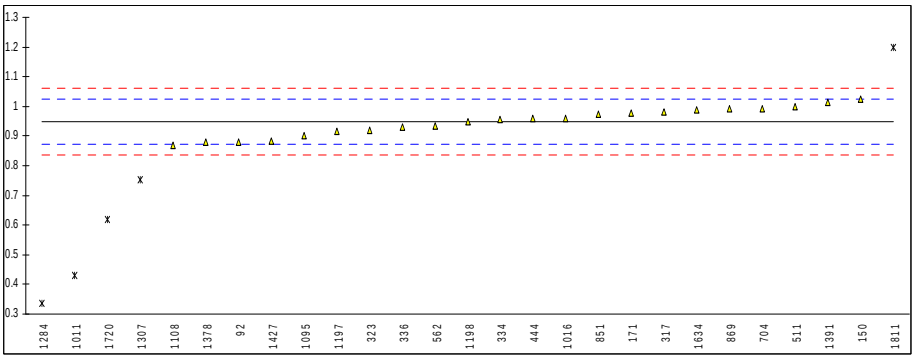
normality	OK	
n	25	
outliers	2	
mean (n)	2.9538	
st.dev. (n)	0.11006	
R(calc.)	0.3082	
R(D2163:96)	0.3401	Compare R(EN27941(liq))=0.5089

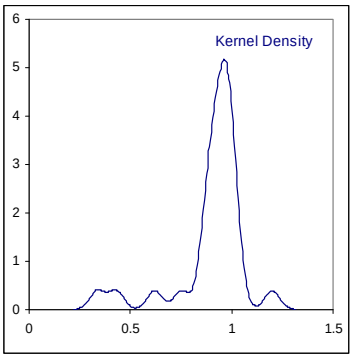




Determination of iso-Pentane on sample #11048; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	0.88	C	-1.76	First reported 1.14
150	D2598	1.023		1.90	
171	D2163	0.976		0.70	
311		-----		-----	
317	D2163	0.98		0.80	
323	D2163	0.92		-0.74	
334	ISO 8973	0.955		0.16	
336	EN27941	0.93		-0.48	
444	IP405	0.958		0.24	
511	D2163	1.00		1.31	
562	D2163	0.934		-0.38	
704	D2163	0.991		1.08	
786		-----		-----	
851		0.973		0.62	
869	D2163	0.991		1.08	
912		-----		-----	
1011	EN 27941	0.43	C,G(0.01)	-13.30	First reported 0.42
1016	ISO7941	0.959		0.26	
1095	EN 27941	0.90		-1.25	
1108	EN27941	0.87		-2.02	
1197	D2163	0.916		-0.84	
1198	D2163	0.949		0.01	
1284	D2163	0.336	G(0.05)	-15.71	
1307	LHA-GC	0.752	G(0.05)	-5.04	
1368		-----		-----	
1369		-----		-----	
1378	ISO7941	0.88		-1.76	
1391	D2163	1.015		1.70	
1427	EN27941	0.882		-1.71	
1634	ISO8973	0.99		1.06	
1720	D2163	0.62	C,G(0.05)	-8.43	First reported 0.75
1811		1.20	C,G(0.01)	6.44	First reported 0.480
	normality	OK			
	n	22			
	outliers	5			
	mean (n)	0.9487			
	st.dev. (n)	0.04644			
	R(calc.)	0.1300			
	R(D2163:96)	0.1092			Compare R(EN27941(liq))=0.3957



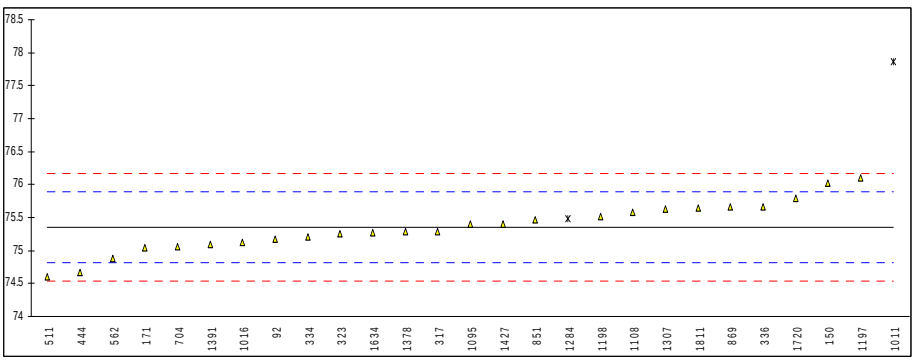


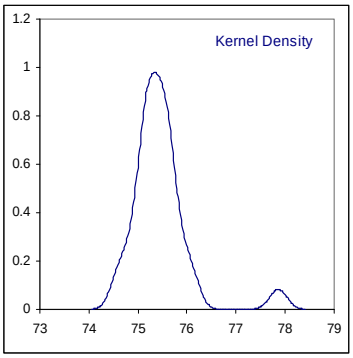
Determination of iso-Butane on sample #11048; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	75.17	C	-0.68	First reported 85.61
150	D2598	76.019		2.47	
171	D2163	75.046		-1.14	
311		-----		-----	
317	D2163	75.29		-0.23	
323	D2163	75.25		-0.38	
334	ISO 8973	75.208		-0.54	
336	EN27941	75.66		1.14	
444	IP405	74.673		-2.53	
511	D2163	74.60		-2.80	
562	D2163	74.887		-1.73	
704	D2163	75.056		-1.10	
786		-----		-----	
851		75.470		0.43	
869	D2163	75.659		1.14	
912		-----		-----	
1011	EN 27941	77.86	C,G(0.01)	9.31	First reported 77.50
1016	ISO7941	75.125		-0.85	
1095	EN 27941	75.40		0.17	
1108	EN27941	75.58		0.84	
1197	D2163	76.098		2.77	
1198	D2163	75.521		0.62	
1284	D2163	75.487	ex	0.50	See §4.1
1307	LHA-GC	75.630		1.03	
1368		-----		-----	
1369		-----		-----	
1378	ISO7941	75.28		-0.27	
1391	D2163	75.085		-1.00	
1427	EN27941	75.404		0.19	
1634	ISO8973	75.27		-0.31	
1720	D2163	75.80	C	1.66	First reported 76.79
1811		75.643		1.08	

normality	OK
n	25
outliers	1
mean (n)	75.3530
st.dev. (n)	0.36966
R(calc.)	1.0350
R(D2163:96)	0.7540

Compare R(EN27941(liq))=1.4737

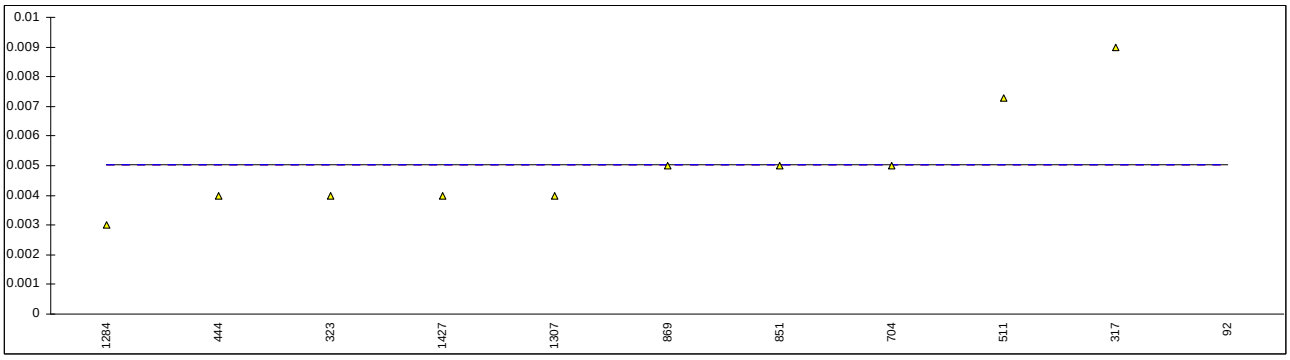


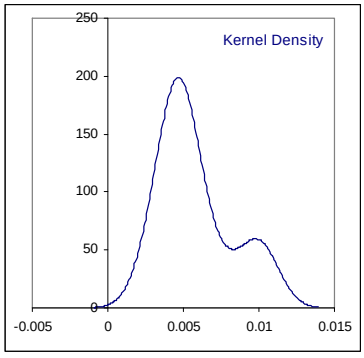


Determination of n-Pentane on sample #11048; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2598	0.9	G(0.01)	-----	reported 1.1 %M/M
150		-----		-----	
171		-----		-----	
311		-----		-----	
317	D2598	0.009		-----	reported 0.01 %M/M
323	D2598	0.004		-----	reported 0.005 %M/M
334		-----		-----	
336		-----		-----	
444	D2598	0.004		-----	reported 0.005 %M/M
511	D2598	0.0073		-----	
562		-----		-----	
704	D2598	0.005		-----	
786		-----		-----	
851	D2598	0.005		-----	
869	D2598	0.005		-----	
912		-----		-----	
1011		-----		-----	
1016		-----		-----	
1095		-----		-----	
1108		-----		-----	
1197		-----		-----	
1198		-----		-----	
1284	D2598	0.003		-----	
1307	D2598	0.004		-----	
1368		-----		-----	
1369		-----		-----	
1378		-----		-----	
1391		-----		-----	
1427	D2598	0.004		-----	reported 0.005 %M/M
1634		-----		-----	
1720		-----		-----	
1811		-----		-----	

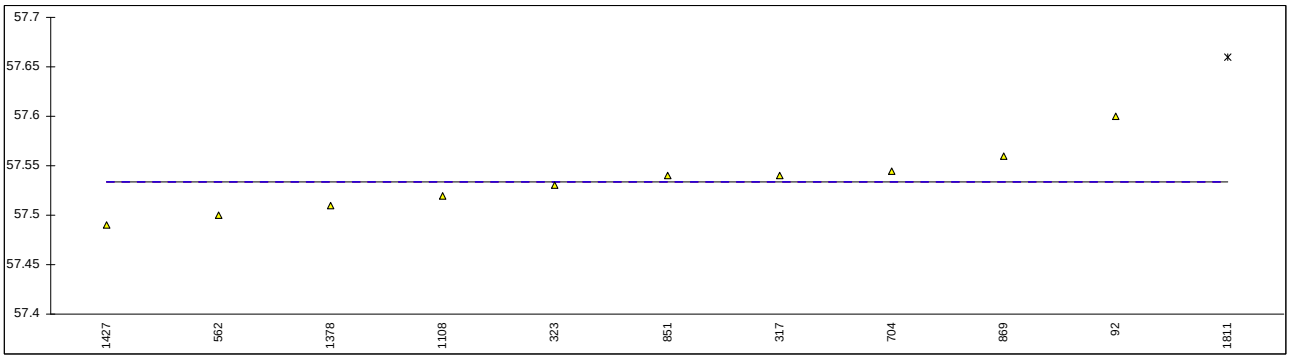
normality	not OK
n	10
outliers	1
mean (n)	0.005
st.dev. (n)	0.0018
R(calc.)	0.005
R(D2163:96)	(0.001)

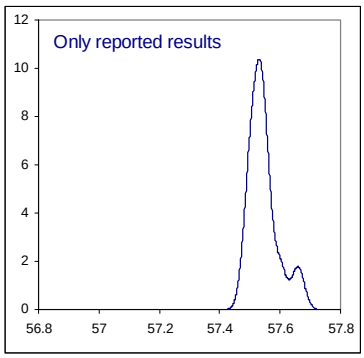


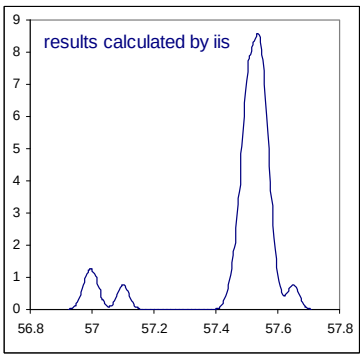


Determination of Molar Mass on sample #11048; results in g/mol

lab	method	value	mark	z(targ)	remarks
92	D2598	57.6		-----	
150		-----		-----	
171		-----		-----	
311		-----		-----	
317	D2598	57.54		-----	
323	D3588	57.53		-----	
334		-----		-----	
336		-----		-----	
444		-----		-----	
511		-----		-----	
562	D2163/D2598	57.50		-----	
704	D2421	57.5446		-----	
786		-----		-----	
851	calc.	57.54		-----	
869	calc.	57.56		-----	
912		-----		-----	
1011		-----		-----	
1016		-----		-----	
1095		-----		-----	
1108	D2421	57.52		-----	
1197		-----		-----	
1198		-----		-----	
1284		-----		-----	
1307		-----		-----	
1368		-----		-----	
1369		-----		-----	
1378	ISO8973	57.51		-----	
1391		-----		-----	
1427	ISO8973	57.49		-----	
1634		-----		-----	
1720		-----		-----	
1811		57.66	G(0.05)	-----	Calc. by iis from the reported test results: 57.01
					Calculated by iis from all reported test results:
	normality	OK			OK
	n	10			23
	outliers	1			4
	mean (n)	57.533			57.526
	st.dev. (n)	0.0318			0.0320
	R(calc.)	0.089			0.090 Compare R(iis10S02B) = 0.107
	R(lit.)	unknown			unknown

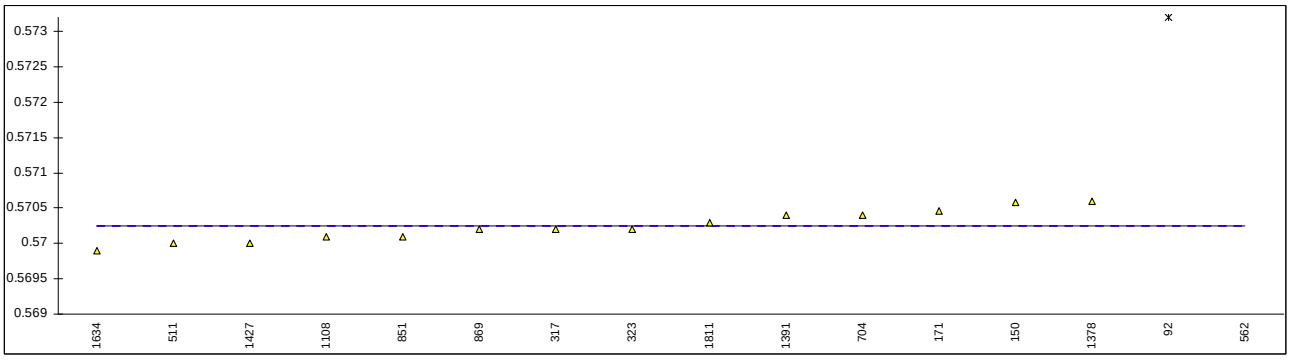


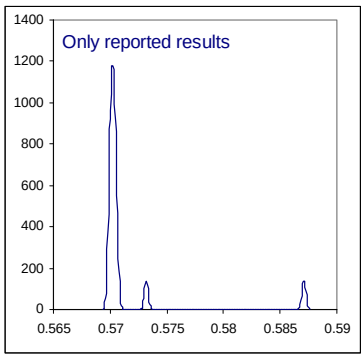


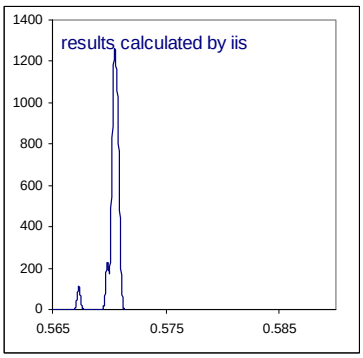


Determination of Relative Density @ 60F on sample #11048; unitless results

lab	method	value	mark	z(targ)	remarks
92	D2598 (@ 15°C)	0.5732	G(0.01)	-----	Calc. by iis @60F from the reported test results: 0.5709
150	D2598	0.57059		-----	
171	D2598	0.57046		-----	
311		-----		-----	
317	D2421	0.5702		-----	
323	D2598	0.5702		-----	
334		-----		-----	
336		-----		-----	
444		-----		-----	
511	D2598	0.570		-----	
562	D2598	0.5871	G(0.01)	-----	Calc. by iis from the reported test results: 0.5706
704	D2598	0.5704		-----	
786		-----		-----	
851	D2598	0.5701		-----	
869	D2598	0.5702		-----	
912		-----		-----	
1011		-----		-----	
1016		-----		-----	
1095		-----		-----	
1108	D2598	0.5701		-----	
1197		-----		-----	
1198		-----		-----	
1284		-----		-----	
1307		-----		-----	
1368		-----		-----	
1369		-----		-----	
1378	ISO8973 (@ 15°C)	0.5706		-----	
1391	D2598	0.5704		-----	
1427	ISO8973	0.570		-----	
1634	ISO8973	0.5699		-----	
1720		-----		-----	
1811		0.5703		-----	
					Calculated by iis from all reported test results:
	normality	OK			OK
	n	14			22
	outliers	2			4
	mean (n)	0.57025			0.57057
	st.dev. (n)	0.000220			0.000205
	R(calc.)	0.00062			0.00057 Compare R(iis10S02B) = 0.00120
	R(lit.)	unknown			unknown

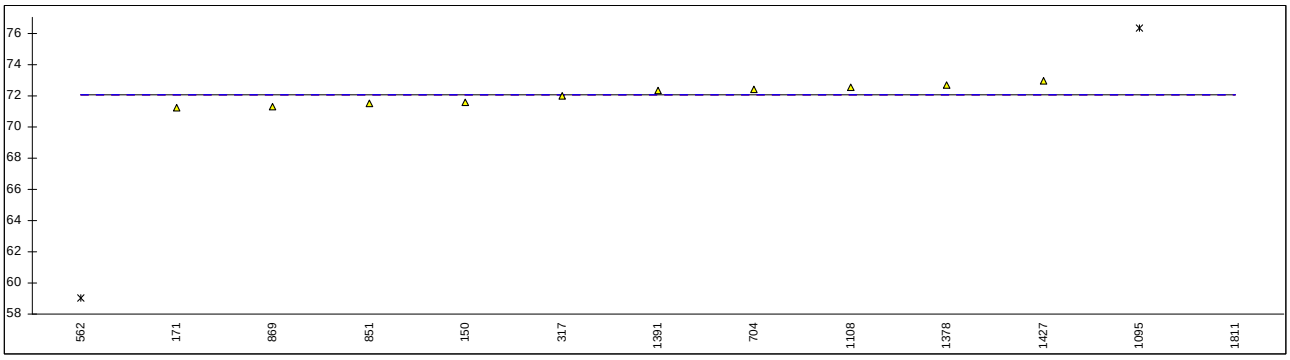


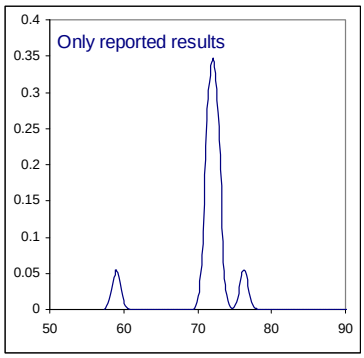


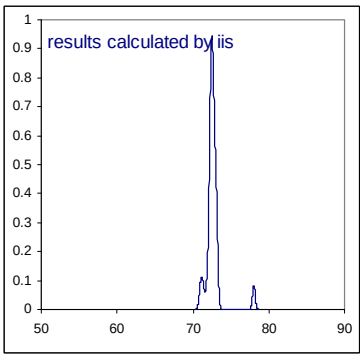


Determination of Abs. Vapour Pressure on sample #11048; results in psi

lab	method	value	mark	z(targ)	remarks
92		-----		-----	
150	D2598	71.5489		-----	
171	D2598	71.215		-----	Calc. by iis from the reported test results: 72.21
311		-----		-----	
317	ISO8973	71.98		-----	
323		-----		-----	
334		-----		-----	
336		-----		-----	
444		-----		-----	
511		-----		-----	
562	D2598	59	G(0.01)	-----	Calc. by iis from the reported test results: 72.7
704	ISO8973	72.37		-----	
786		-----		-----	
851	D2598	71.5		-----	Calc. by iis from the reported test results: 72.6
869	D2598	71.3		-----	Calc. by iis from the reported test results: 72.4
912		-----		-----	
1011		-----		-----	
1016		-----		-----	
1095		76.29	G(0.01)	-----	Calc. by iis from the reported test results: 72.97
1108	D2598	72.5		-----	
1197		-----		-----	
1198		-----		-----	
1284		-----		-----	
1307		-----		-----	
1368		-----		-----	
1369		-----		-----	
1378	ISO8973	72.64		-----	
1391	ISO8973	72.3		-----	
1427	ISO8973	72.95		-----	
1634		-----		-----	
1720		-----		-----	
1811		130.6	G(0.01)	-----	Different unit? Calc. by iis from the reported test results: 71.0
					Calculated by iis from all reported test results:
	normality	OK			OK
	n	10			23
	outliers	3			3
	mean (n)	72.03			72.60
	st.dev. (n)	0.609			0.333
	R(calc.)	1.71			0.93 Compare R(iis10S02B) = 0.83
	R(lit.)	unknown			unknown

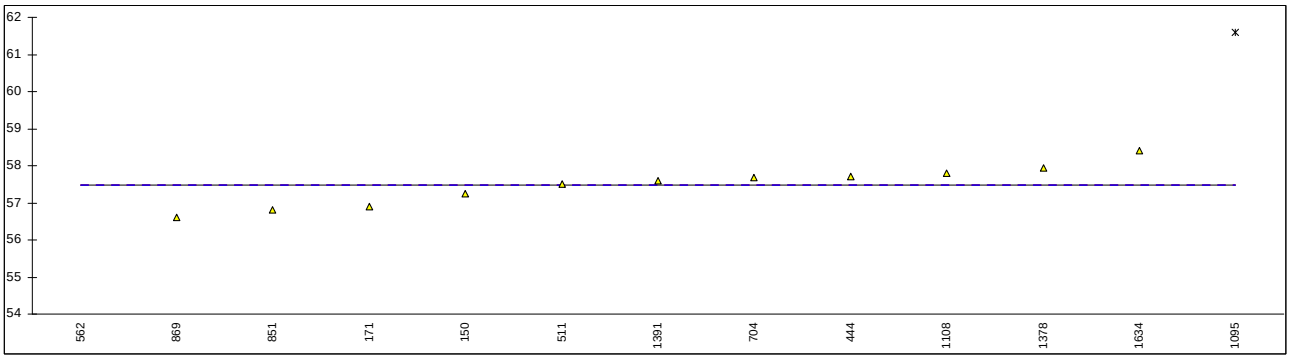


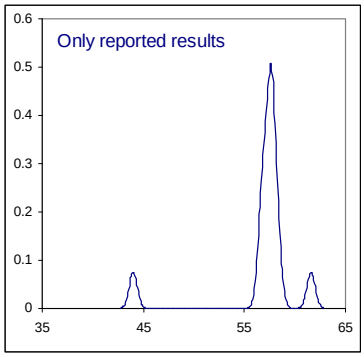


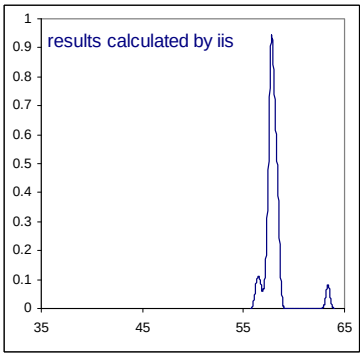


Determination of Rel. Vapour Pressure on sample #11048; results in psi

lab	method	value	mark	z(targ)	remarks
92		-----		-----	
150	D2598	57.244		-----	
171	D2598	56.911		-----	
311		-----		-----	
317		-----		-----	
323		-----		-----	
334		-----		-----	
336		-----		-----	
444	IP432	57.7		-----	
511	D2598	57.5		-----	
562	D2598	44	G(0.01)	-----	Calc. by iis from the reported test results: 58.0
704	ISO8973	57.68		-----	
786		-----		-----	
851	D2598	56.8		-----	
869	D2598	56.6		-----	
912		-----		-----	
1011		-----		-----	
1016		-----		-----	
1095		61.59	G(0.01)	-----	Calc. by iis from the reported test results: 58.28
1108	D2598	57.8		-----	
1197		-----		-----	
1198		-----		-----	
1284		-----		-----	
1307		-----		-----	
1368		-----		-----	
1369		-----		-----	
1378	ISO8973	57.95		-----	
1391	ISO8973	57.6		-----	
1427		-----		-----	
1634	ISO8973	58.417		-----	
1720		-----		-----	
1811		-----		-----	
					Calculated by iis from all reported test results:
	normality	OK			OK
	n	11			23
	outliers	2			3
	mean (n)	57.47			57.90
	st.dev. (n)	0.540			0.333
	R(calc.)	1.51			0.93 Compare R(iis10S02B) = 0.83
	R(lit.)	unknown			unknown







APPENDIX 2

Additional details

	Sample Volume	Type of vaporizer	Remarks
92	---	---	1.10 %M/M n-pentane
150	---	---	none
171	---	---	---
311	---	---	---
317	88	liquid injection	0.01 %M/M n-pentane
323	---	none, liquid injection	0.005 %M/M n-pentane
334	---	---	---
336	---	---	none
444	---	none, liquid injection	0.005 %M/M n-pentane
511	---	---	0.0073 %mol/mol n-pentane
562	52.8	---	none
704	0.0005	SPL	0.005 %mol/mol n-pentane
786	---	---	---
851	70	Flash Evaporator	0.005 %mol/mol n-pentane
869	10	Flash Evaporator	0.005 %mol/mol n-pentane
912	---	---	---
1011	---	---	none
1016	---	LSV	---
1095	40.8 g	---	---
1108	0.1 µl	Liquid injection	---
1197	---	---	n-pentane present
1198	---	---	n-pentane present
1284	---	---	0.003 %mol/mol n-pentane
1307	---	---	0.004 %mol/mol n-pentane
1368	---	---	---
1369	---	---	---
1378	---	---	none
1391	---	---	none
1427	---	---	0.005 %M/M n-pentane
1634	---	---	none
1720	---	---	none
1811	25	Water bath	---

APPENDIX 3

Number of participants per country

2 labs in BELGIUM
1 lab in CANADA
1 lab in CHILE
1 lab in CZECH REPUBLIC
2 labs in FRANCE
2 labs in GREECE
1 lab in HONG KONG
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 RUSSIA
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
1 lab 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.
3 ASTM E178-89
4 ASTM E1301-89
5 ISO 5725-86
6 ISO 5725, parts 1-6, 1994
7 M. Thompson and R. Wood, J. AOAC Int, 76, 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, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 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