Results of Proficiency Test
Liquefied Butane Analysis
May 2010

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis10S02B

July 2010

CONTENTS

1 INTRODUCTION

During the last years, with increasing frequency, requests were received by iis from laboratories that participated in the iis PT program, to organize also a proficiency test for the Liquefied Butane Analysis. Beginning 2008, iis started an investigation for the feasibility of such a PT. Because iis has limited gas-handling facilities in place to prepare gas samples, Scott Specialty Gases (Breda, the Netherlands) was contacted. This company is fully equipped and has a broad experience in the preparation of synthetic Liquefied 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 interlaboratory study was repeated in 2010, in which now 24 laboratories from 16 different countries have participated. See appendix 3 for a list of participants in alphabetical country order.
In this report the results of the 2010 proficiency test on Liquefied Butane are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organizer of this proficiency test.
To optimise the costs for the participating laboratories, it was decided to prepare one Liquefied Butane mixture. The mixture was divided over a batch of 30 cylinders.
The cylinder size is a cost-effective two-litre cylinder with dip tube device.
Each cylinder was uniquely numbered. The limited cylinder size is chosen to optimise transport and handling costs.

Participants were requested to report rounded and unrounded results. 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 30 cylinders was prepared (lot 79022) on April 29, 2010. Each cylinder was uniquely numbered. One cylinder was tested 10 times, on 10 following days, to check the stability. The remaining 29 cylinders were all tested in threefold to check the homogeneity of the batch. By ANOVA analysis on the test results in accordance with ISO 6143 the in-between bottle standard deviation was calculated. The repeatability values (r) were calculated per component by multiplication of the respective standard deviation by 2.8. Subsequently the calculated repeatabilities were compared with 0.3 times the corresponding target reproducibilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

Parameter	conc. in %mol/mol	r(observed) in %mol/mol	0.3 X R(D2163) in %mol/mol
Propane	1.947	0.056	0.068
Propylene	1.449	0.050	0.054
n-Butane	8.134	0.192	0.285
1,3-Butadiene	0.897	0.009	0.032
iso-Butylene	4.026	0.036	0.136
1-Butene	4.991	0.057	0.170
trans-2-Butene	2.005	0.029	0.075
cis-2-Butene	1.017	0.020	0.037
iso-Pentane	0.710	0.017	0.025
iso-Butane	74.824	0.264	0.360

Table 1: homogeneity test results

The calculated repeatabilities are each less than 0.3 times the corresponding reproducibility of the reference method ASTM D2163. Therefore, homogeneity of the subsamples #10BU was assumed.

To each of the participating laboratories one 2L cylinder was sent on May 10, 2010.

2.5 STABILITY OF THE SAMPLES

The ten test results of the cylinder tested at ten subsequent days were identical. Also, 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 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 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 317 appeared to be statistical outliers and because all test results of one laboratory are correlated, the remaining four test results of lab 317 were excluded manually from the statistical analysis.
Because 14 laboratories reported both results in %mol/mol as well as in %M/M, it has been possible to check the calculations of these 14 laboratories. A good correclation between the results reported in %mol/mol and the results reported in %M/M is to be expected.
Thus it was noticed that laboratory 1117 obviously had made a calculation error. Fortunately the laboratory was able to locate and correct the error and to report revised test results.
Also, one test result of laboratory 1634 (for iso-Butylene) was deviating, see page 16. Some other (small) deviations may be explained by the reporting of test results too far rounded.

In total 22 participants reported 263 numerical results. Observed were 20 outlying results, which is 7.6% 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 Molar Mass and Absolute Vapour Pressure 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 in the last version ASTM D2163:07 only repeatabilities, but no reproducibilies are mentioned. Therefore the precision data from the previous version ASTM D2163:96 were used.

Propane: The determination of this component may be problematic. Two statistical outliers were detected and the calculated reproducibility, after exclusion of the statistical outliers, is not 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).

Propylene: The determination of this component may be problematic. Two statistical outliers were detected and the calculated reproducibility, after exclusion of the statistical outliers, is not at all in agreement with the requirements of ASTM D2163:96. However, the calculated reproducibility is in agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).

n-Butane: No large 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 with the reproducibility of EN27941 (identical to IP 405 and ISO 7941).

1,3-Butadiene: The determination of this component may be problematic. The calculated reproducibility, after exclusion of the statistical outlier, is not in agreement with the requirements of ASTM D2163:96 nor with the reproducibility of EN27941 (identical to IP 405 and ISO 7941).

iso-Butylene: No large 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 with the reproducibility of EN27941 (identical to IP 405 and ISO 7941).

1-Butene: No analytical problems were observed. No statistical outliers were detected. And the calculated reproducibility is in agreement with the requirements of ASTM D2163:96 and with the reproducibility of EN27941 (identical to IP 405 and ISO 7941).

trans-2-Butene: The determination of this component may be problematic. The calculated reproducibility, after exclusion of the statistical outlier, is not in agreement with the requirements of ASTM D2163:96. However, the calculated reproducibility is in agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).

cis-2-Butene: The determination of this component may be problematic. The calculated reproducibility, after exclusion of the statistical outlier, is not in agreement with the requirements of ASTM D2163:96. However, the calculated reproducibility is in agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).

iso-Pentane: 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 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 component may be problematic. No statistical outliers were detected. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D2163:96, nor with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941). The data set seems to be bimodally divided with one maximum around 74.5 %V/V (13 test results) and another maximum around 75.8 %V/V (9 test results).

Molar Mass: This calculated parameter may be problematic. The results vary over a large range from 56.69 - 57.73 and two statistically significant outliers were present (in 9 test results). See also the discussion in 4.3.

Relative Density: This calculated parameter may be problematic. The results vary over a range from 0.5674 - 0.5700 and one statistically significant outlier was present (in 14 test results). See also the discussion in 4.3.

Abs. Vapour Pres.: This calculated parameter may be problematic. The results vary over a large range (72.0 – 76.4 psi) and one statistically significant outlier was observed. See also the discussion in 4.3.

Rel. Vapour Pres.: This calculated parameter may be problematic. The results vary over a large range (58.0 – 60.6 psi) and one statistically significant outlier was observed. See also the discussion in 4.3.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM D2163 and EN27941/ISO7941/IP405) are compared in the next table.

Parameter	unit	n	cons. value	2.8 * sd	R(D2163) in **%mol**	R(EN27941) liq.-inj. in **%mol**	R(EN27941) liq.-inj. in **%M/M**
Propane	%mol/mol	20	1.973	0.342	0.227	0.767	1
Propylene	%mol/mol	19	1.279	0.281	0.147	0.732	1
n-Butane	%mol/mol	20	8.245	0.811	0.949	1.011	1
1,3-Butadiene	%mol/mol	19	0.941	0.150	0.108	0.941	1
iso-Butylene	%mol/mol	19	3.951	0.329	0.454	0.976	1
1-Butene	%mol/mol	21	4.950	0.606	0.569	0.976	1
trans-2-Butene	%mol/mol	21	2.177	0.286	0.251	0.976	1
cis-2-Butene	%mol/mol	21	1.072	0.159	0.123	0.976	1
iso-Pentane	%mol/mol	19	0.715	0.106	0.082	1.255	1
iso-Butane	%mol/mol	20	74.904	1.907	1.200	1.516	1.5
Molar Mass	g/mol	7	57.41	0.29	n/a	n/a	n/a
Relative Density		13	0.5692	0.010	n/a	n/a	n/a
Abs. Vapour pres.	psi	10	73.19	1.74	n/a	n/a	n/a
Rel. Vapour pres.	psi	10	58.28	1.47	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 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 Propylene (z-score in bold). No explanation can be given for this observation.

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	1.947	1.973	+0.026	+0.32
Propylene	1.449	1.279	-0.170	**-3.16**
n-Butane	8.134	8.245	+0.111	+0.32
1,3-Butadiene	0.897	0.941	+0.044	+1.11
iso-Butylene	4.026	3.951	-0.075	-0.46
1-Butene	4.991	4.950	-0.041	-0.20
trans-2-Butene	2.005	2.177	+0.172	+1.92
cis-2-Butene	1.017	1.072	+0.055	+1.28
iso-Pentane	0.710	0.715	+0.005	+0.18
iso-Butane	74.824	74.904	+0.080	+0.19

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

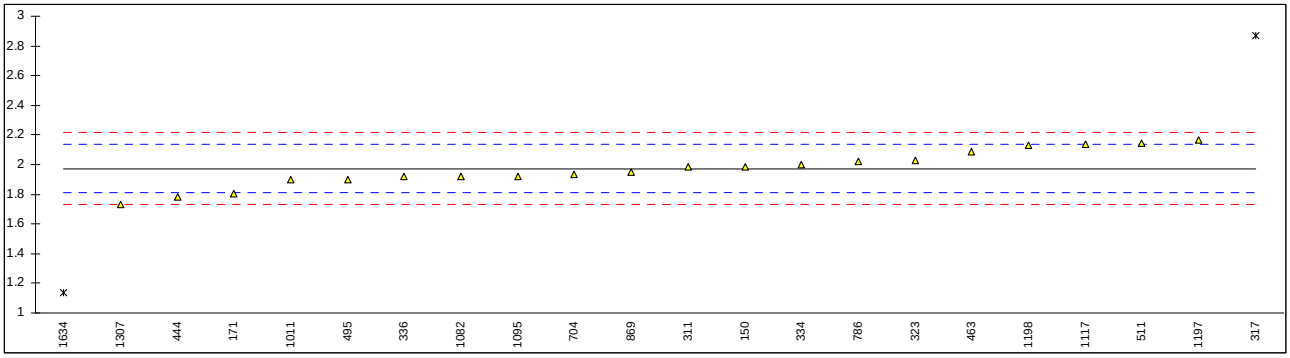
In total 8 laboratories reported the presence of some n-pentane (0.0035 %mol/mol with sd = 0.0004 %mol/mol), a component probably present as impurity in one or more of the pure components that were used to prepare the iso-Butane mixture. Also two laboratories reported the presence of some helium (0.45 – 1.4 %mol/mol). See appendix 2.

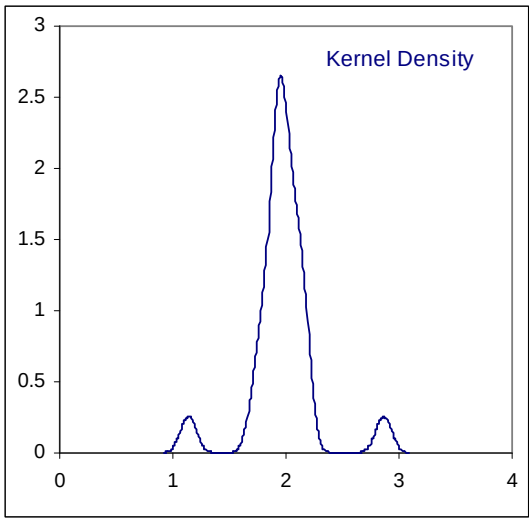
In principle no additional spread should be introduced when applying a calculation on the reported component concentrations. However, in practice a significant additional uncertainty is added. See the differences between the values from the results as reported by the participating laboratories (each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

APPENDIX 1

Determination of Propane; results in %mol/mol

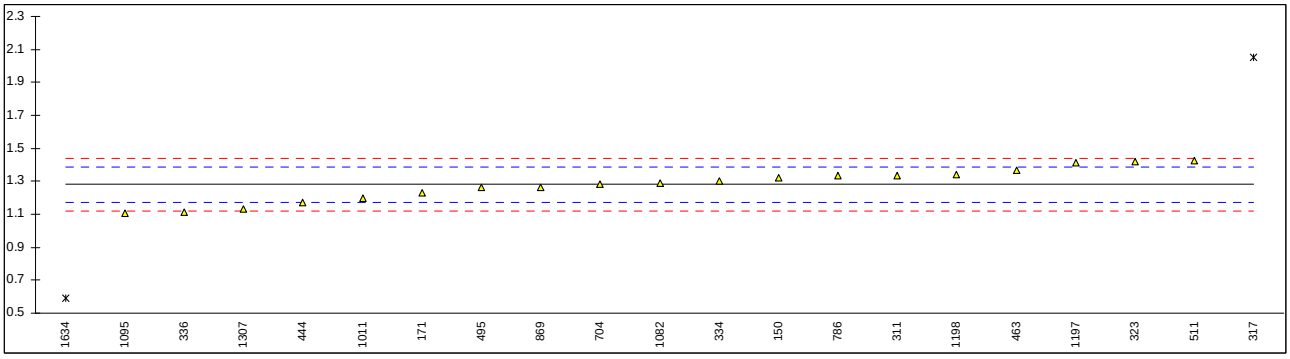
lab	method	value	mark	z(targ)	remarks
150	D2163	1.989		0.20	
171	D2163	1.8036		-2.09	
225		-----		-----	
311	D2163	1.982		0.11	
317	D2163	2.867	G(0.01)	11.03	
323	D2163	2.03		0.70	
334	EN27941	2.0		0.33	
336	EN27941	1.92		-0.65	
444	IP405	1.78		-2.38	
463	EN27941	2.09		1.44	
495	D2163	1.902		-0.87	
511	D2163	2.145		2.12	
704	D2163	1.933		-0.49	
786	D2163	2.024		0.63	
869	D2163	1.951		-0.27	
912		-----		-----	
1011	EN27941	1.9		-0.90	
1082	IP473	1.921		-0.64	
1095	EN27941	1.922		-0.63	
1117	in house	2.14	C	2.06	first reported 1.25
1197	D2163	2.166		2.38	
1198	D2163	2.127		1.90	
1307	GC	1.732		-2.97	
1634	ISO7941	1.14	G(0.05)	-10.27	
	normality	OK			
	n	20			
	outliers	2			
	mean (n)	1.973			
	st.dev. (n)	0.1222			
	R(calc.)	0.342			
	R(ASTM D2163)	0.227			Compare R(EN27941(liq))=0.767

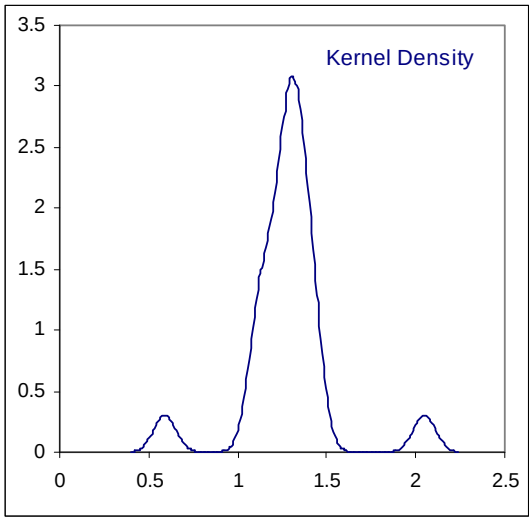




Determination of Propylene; results in %mol/mol

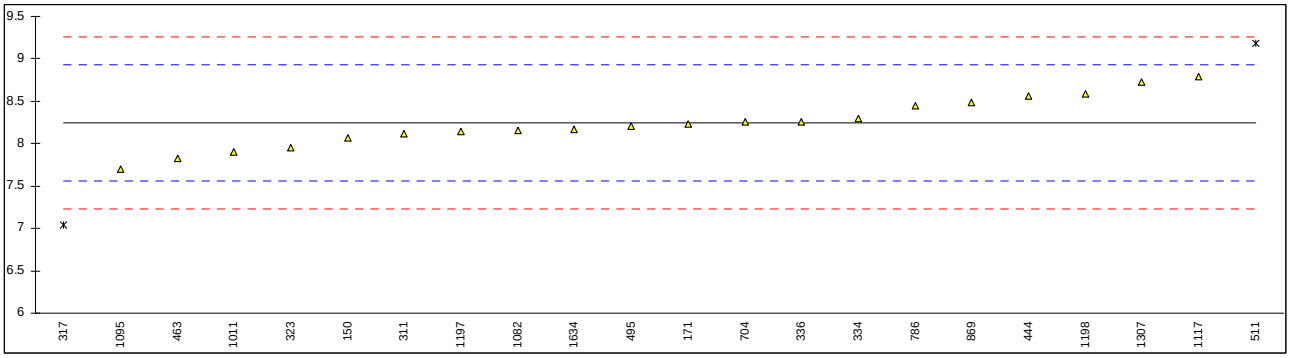
lab	method	value	mark	z(targ)	remarks
150	D2163	1.319		0.74	
171	D2163	1.2319		-0.89	
225		-----		-----	
311	D2163	1.337		1.07	
317	D2163	2.049	G(0.01)	14.37	
323	D2163	1.42		2.62	
334	EN27941	1.3		0.38	
336	EN27941	1.11		-3.16	
444	IP405	1.17		-2.04	
463	EN27941	1.37		1.69	
495	D2163	1.262		-0.33	
511	D2163	1.428		2.77	
704	D2163	1.284		0.09	
786	D2163	1.336		1.06	
869	D2163	1.265		-0.27	
912		-----		-----	
1011	EN27941	1.2		-1.48	
1082	IP473	1.286		0.12	
1095	EN27941	1.104		-3.27	
1117		-----		-----	co-elution with iso-Butane
1197	D2163	1.411		2.46	
1198	D2163	1.341		1.15	
1307	GC	1.134		-2.71	
1634	ISO7941	0.59	G(0.05)	-12.87	
	normality	OK			
	n	19			
	outliers	2			
	mean (n)	1.279			
	st.dev. (n)	0.1004			
	R(calc.)	0.281			
	R(ASTM D2163)	0.147			Compare R(EN27941(liq))=0.732

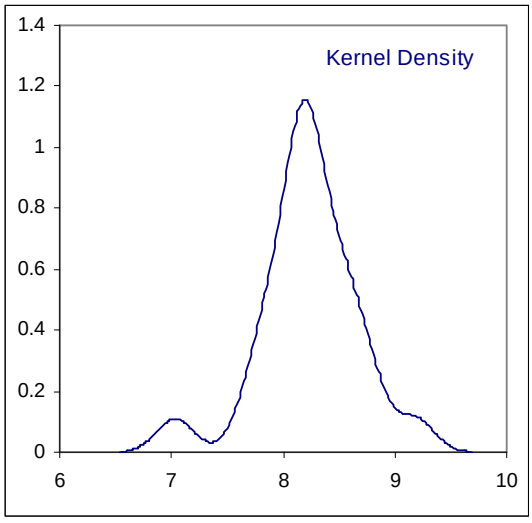




Determination of n-Butane; results in %mol/mol

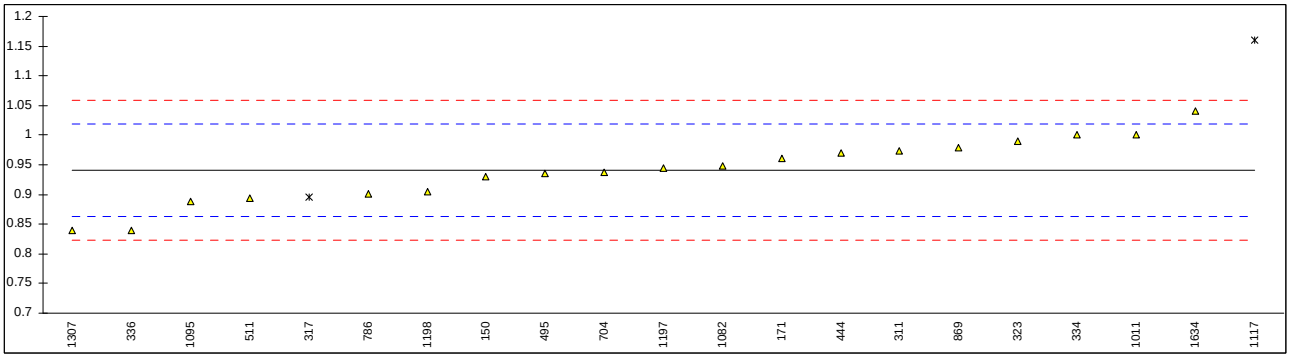
lab	method	value	mark	z(targ)	remarks
150	D2163	8.072		-0.51	
171	D2163	8.2332		-0.03	
225		-----		-----	
311	D2163	8.121		-0.36	
317	D2163	7.034	DG(0.05)	-3.57	
323	D2163	7.95		-0.87	
334	EN27941	8.3		0.16	
336	EN27941	8.26		0.05	
444	IP405	8.56		0.93	
463	EN27941	7.82		-1.25	
495	D2163	8.202		-0.13	
511	D2163	9.186	DG(0.05)	2.77	
704	D2163	8.255		0.03	
786	D2163	8.453		0.61	
869	D2163	8.480		0.69	
912		-----		-----	
1011	EN27941	7.9		-1.02	
1082	IP473	8.162		-0.24	
1095	EN27941	7.703		-1.60	
1117	in house	8.79	C	1.61	first reported 8.89
1197	D2163	8.149		-0.28	
1198	D2163	8.581		0.99	
1307	GC	8.730		1.43	
1634	ISO7941	8.17		-0.22	
	normality	OK			
	n	20			
	outliers	2			
	mean (n)	8.245			
	st.dev. (n)	0.2898			
	R(calc.)	0.811			
	R(ASTM D2163)	0.949			Compare R(EN27941(liq))=1.011

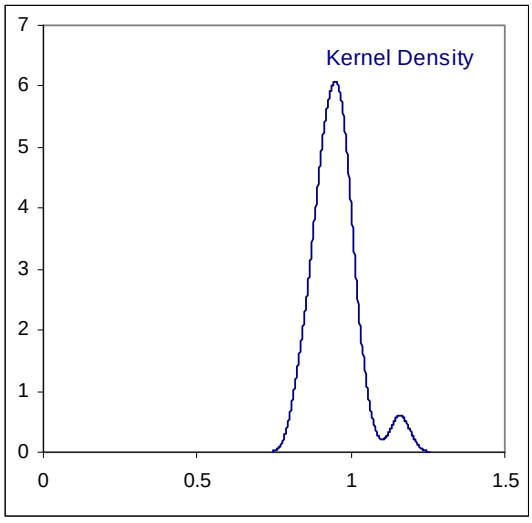




Determination of 1,3-Butadiene; results in %mol/mol

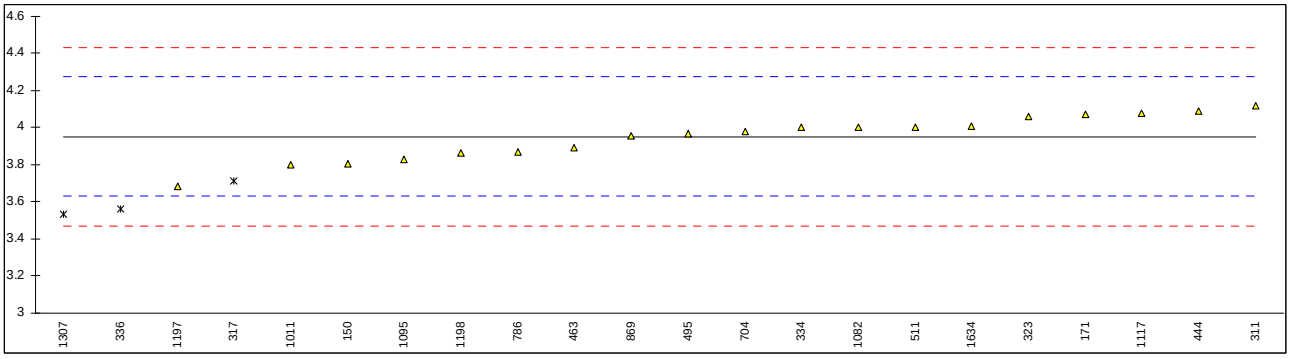
lab	method	value	mark	z(targ)	remarks
150	D2163	0.930		-0.28	
171	D2163	0.9612		0.52	
225		-----		-----	
311	D2163	0.973		0.82	
317	D2163	0.895	ex	-1.17	see §4
323	D2163	0.99		1.25	
334	EN27941	1.0		1.50	
336	EN27941	0.84		-2.57	
444	IP405	0.97		0.74	
463	EN27941	<0.1		<-21.40	false negative?
495	D2163	0.936		-0.12	
511	D2163	0.894		-1.19	
704	D2163	0.937		-0.10	
786	D2163	0.901		-1.02	
869	D2163	0.979		0.97	
912		-----		-----	
1011	EN27941	1.0		1.50	
1082	IP473	0.948		0.18	
1095	EN27941	0.889		-1.32	
1117	in house	1.16	C,G(0.05)	5.58	first reported 1.02
1197	D2163	0.945		0.10	
1198	D2163	0.905		-0.91	
1307	GC	0.839		-2.59	
1634	ISO7941	1.04		2.52	
	normality	OK			
	n	19			
	outliers	1			
	mean (n)	0.941			
	st.dev. (n)	0.0535			
	R(calc.)	0.150			
	R(ASTM D2163)	0.108			Compare R(EN27941(liq))=0.941

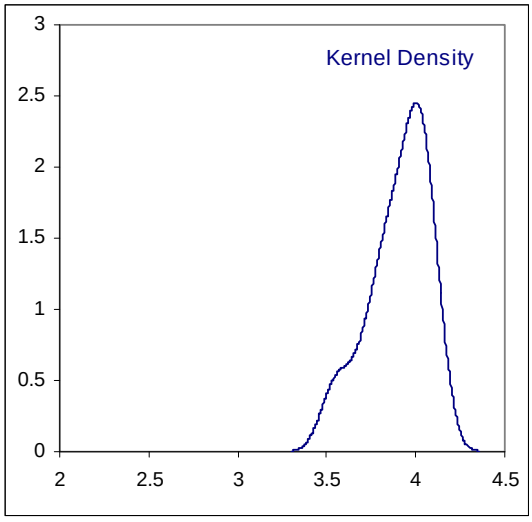


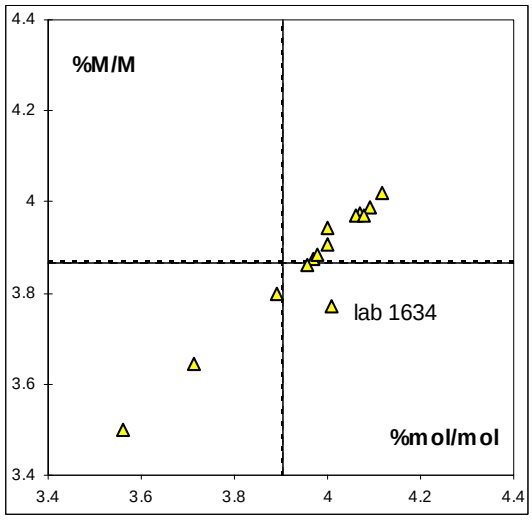


Determination of iso-Butylene; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
150	D2163	3.805		-0.91	
171	D2163	4.0701		0.74	
225		-----		-----	
311	D2163	4.116		1.02	
317	D2163	3.714	ex	-1.48	see §4
323	D2163	4.06		0.68	
334	EN27941	4.0		0.30	
336	EN27941	3.56	DG(0.05)	-2.44	
444	IP405	4.09		0.86	
463	EN27941	3.89		-0.38	
495	D2163	3.969		0.11	
511	D2163	4.004		0.33	
704	D2163	3.978		0.17	
786	D2163	3.872		-0.49	
869	D2163	3.956		0.03	
912		-----		-----	
1011	EN27941	3.8		-0.94	
1082	IP473	4.000		0.30	
1095	EN27941	3.829		-0.76	
1117	in house	4.08	C	0.80	first reported 3.84
1197	D2163	3.684		-1.66	
1198	D2163	3.864		-0.54	
1307	GC	3.534	DG(0.05)	-2.60	
1634	ISO7941	4.01		0.36	reported 3.77%M/M while
	normality	OK			
	n	19			
	outliers	2			
	mean (n)	3.951			
	st.dev. (n)	0.1177			
	R(calc.)	0.329			
	R(ASTM D2163)	0.454			Compare R(EN27941(liq))=0.976

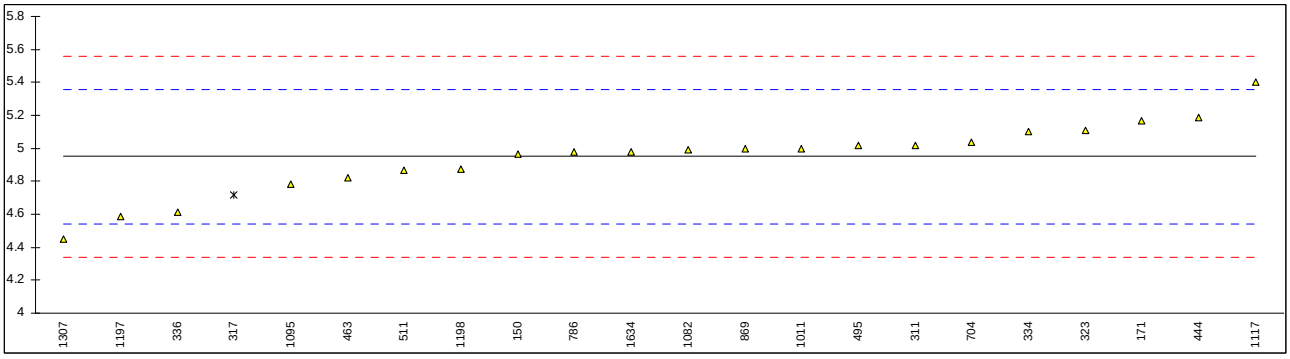


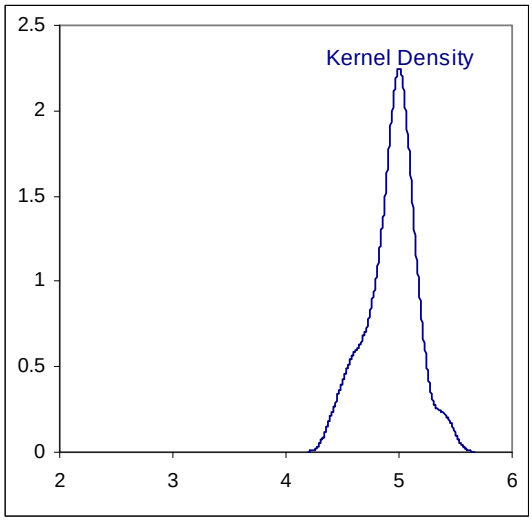




Determination of 1-Butene; results in %mol/mol

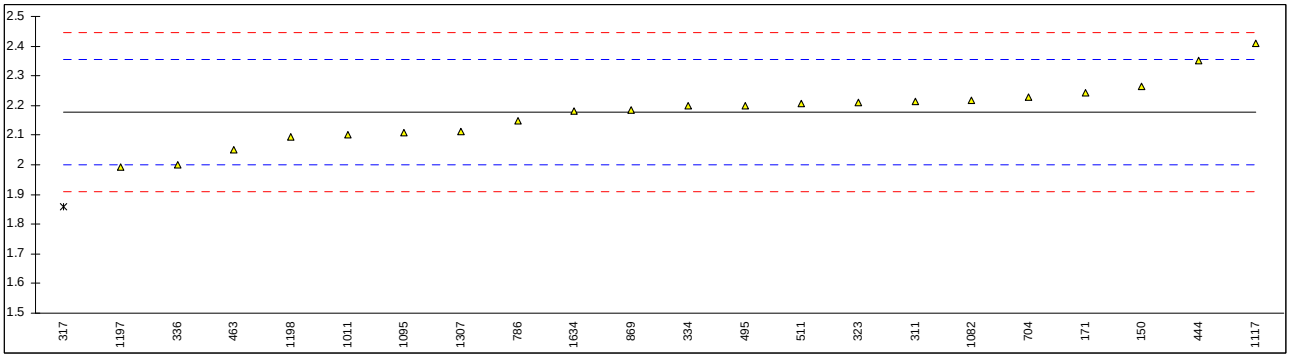
lab	method	value	mark	z(targ)	remarks
150	D2163	4.962		0.06	
171	D2163	5.1696		1.08	
225		-----		-----	
311	D2163	5.020		0.34	
317	D2163	4.720	ex	-1.13	see §4
323	D2163	5.11		0.79	
334	EN27941	5.1		0.74	
336	EN27941	4.61		-1.67	
444	IP405	5.19		1.18	
463	EN27941	4.82		-0.64	
495	D2163	5.017		0.33	
511	D2163	4.868		-0.40	
704	D2163	5.036		0.42	
786	D2163	4.979		0.14	
869	D2163	4.997		0.23	
912		-----		-----	
1011	EN27941	5.0		0.25	
1082	IP473	4.993		0.21	
1095	EN27941	4.782		-0.82	
1117	in house	5.40	C	2.21	first reported 5.09
1197	D2163	4.586		-1.79	
1198	D2163	4.877		-0.36	
1307	GC	4.449		-2.46	
1634	ISO7941	4.98		0.15	
	normality	OK			
	n	21			
	outliers	0			
	mean (n)	4.950			
	st.dev. (n)	0.2166			
	R(calc.)	0.606			
	R(ASTM D2163)	0.569			Compare R(EN27941(liq))=0.976

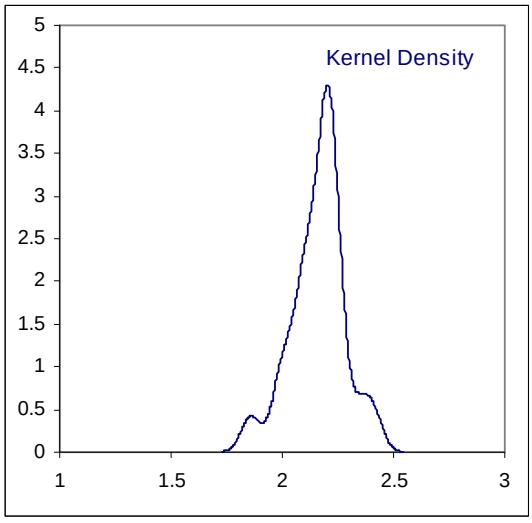




Determination of trans-2-Butene; results in %mol/mol

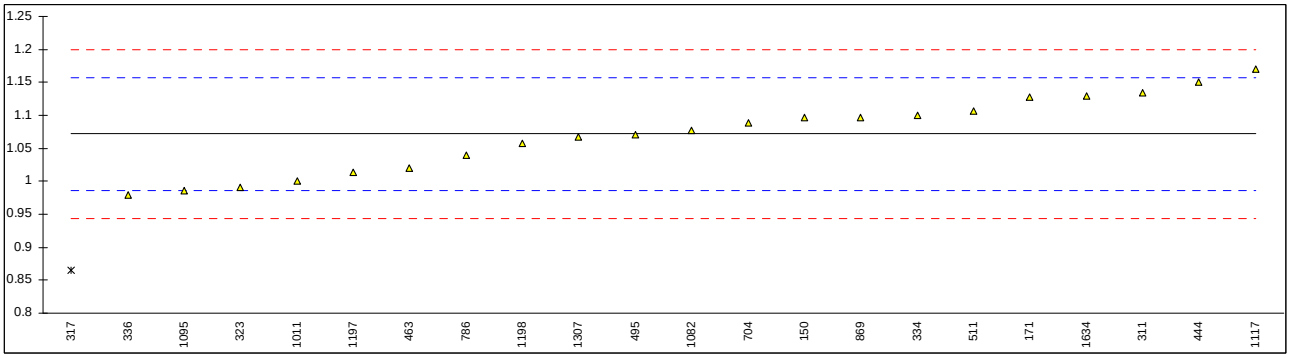
lab	method	value	mark	z(targ)	remarks
150	D2163	2.265		0.98	
171	D2163	2.2443		0.75	
225		-----		-----	
311	D2163	2.212		0.39	
317	D2163	1.860	D(0.05)	-3.55	
323	D2163	2.21		0.37	
334	EN27941	2.2		0.26	
336	EN27941	2.00		-1.98	
444	IP405	2.35		1.94	
463	EN27941	2.05		-1.42	
495	D2163	2.201		0.27	
511	D2163	2.208		0.35	
704	D2163	2.227		0.56	
786	D2163	2.148		-0.33	
869	D2163	2.185		0.09	
912		-----		-----	
1011	EN27941	2.1		-0.86	
1082	IP473	2.217		0.45	
1095	EN27941	2.109		-0.76	
1117	in house	2.41	C	2.61	first reported 2.27
1197	D2163	1.994		-2.05	
1198	D2163	2.095		-0.92	
1307	GC	2.114		-0.71	
1634	ISO7941	2.18		0.03	
	normality	OK			
	n	21			
	outliers	1			
	mean (n)	2.177			
	st.dev. (n)	0.1020			
	R(calc.)	0.286			
	R(ASTM D2163)	0.251			Compare R(EN27941(liq))=0.976

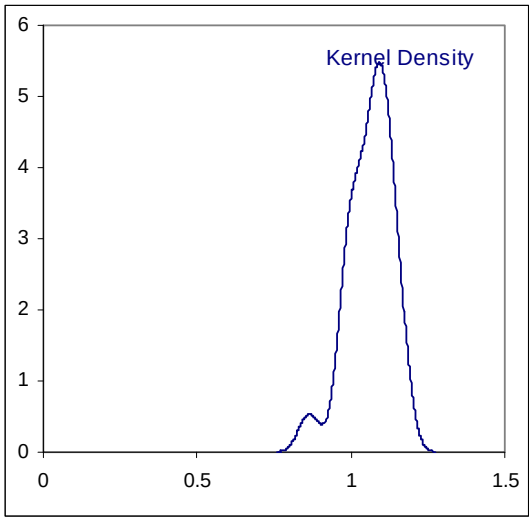




Determination of cis-2-Butene; results in %mol/mol

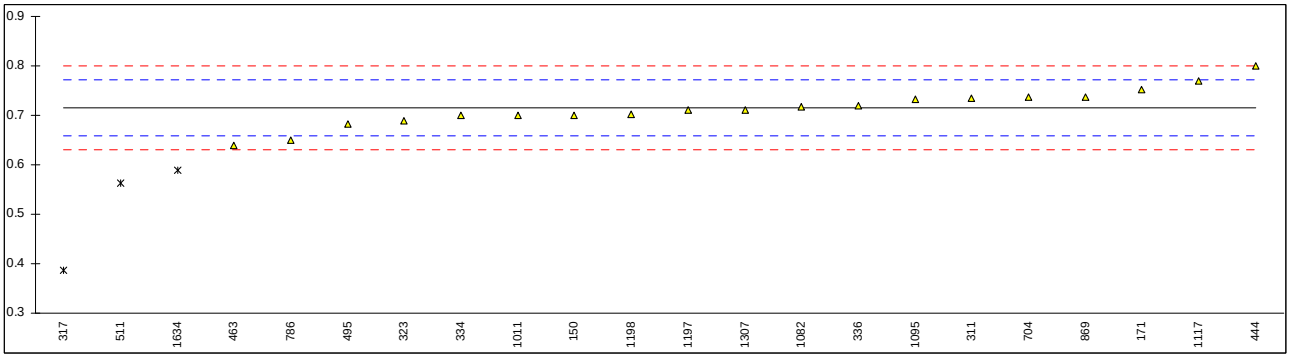
lab	method	value	mark	z(targ)	remarks
150	D2163	1.097		0.59	
171	D2163	1.1279		1.32	
225		-----		-----	
311	D2163	1.135		1.48	
317	D2163	0.865	G(0.05)	-4.82	
323	D2163	0.99		-1.90	
334	EN27941	1.1		0.66	
336	EN27941	0.98		-2.14	
444	IP405	1.15		1.83	
463	EN27941	1.02		-1.20	
495	D2163	1.070		-0.04	
511	D2163	1.107		0.83	
704	D2163	1.088		0.38	
786	D2163	1.039		-0.76	
869	D2163	1.097		0.59	
912		-----		-----	
1011	EN27941	1.0		-1.67	
1082	IP473	1.077		0.13	
1095	EN27941	0.986		-2.00	
1117	in house	1.17	C	2.30	first reported 1.10
1197	D2163	1.014		-1.34	
1198	D2163	1.057		-0.34	
1307	GC	1.067		-0.11	
1634	ISO7941	1.13		1.36	
	normality	OK			
	n	21			
	outliers	1			
	mean (n)	1.072			
	st.dev. (n)	0.0569			
	R(calc.)	0.159			
	R(ASTM D2163)	0.123			Compare R(EN27941(liq))=0.976

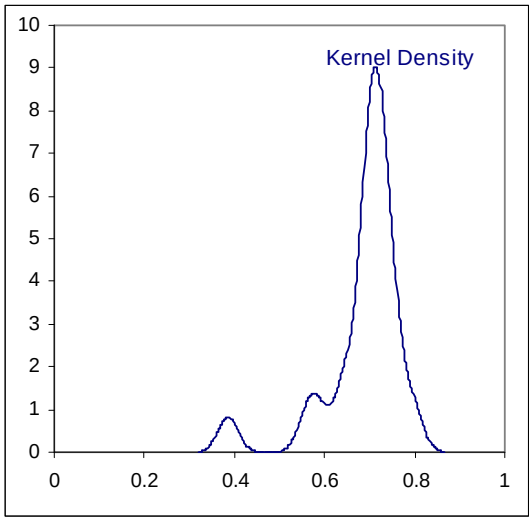




Determination of iso-Pentane; results in %mol/mol

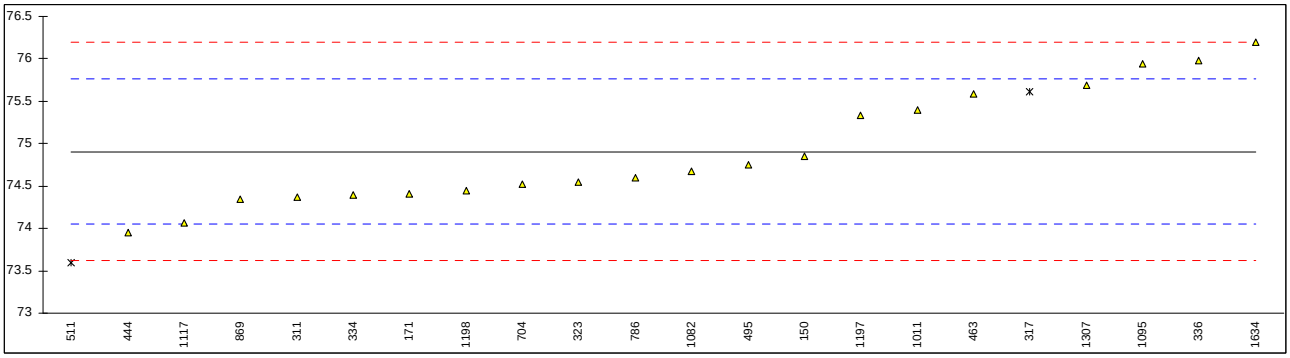
lab	method	value	mark	z(targ)	remarks
150	D2163	0.701		-0.50	
171	D2163	0.7519		1.29	
225		-----		-----	
311	D2163	0.735		0.69	
317	D2163	0.386	G(0.01)	-11.52	
323	D2163	0.69		-0.88	
334	EN27941	0.7		-0.53	
336	EN27941	0.72		0.17	
444	IP405	0.80		2.97	
463	EN27941	0.64		-2.63	
495	D2163	0.682		-1.16	
511	D2163	0.562	DG(0.05)	-5.36	
704	D2163	0.736		0.73	
786	D2163	0.651		-2.25	
869	D2163	0.738		0.80	
912		-----		-----	
1011	EN27941	0.7		-0.53	
1082	IP473	0.717		0.06	
1095	EN27941	0.732		0.59	
1117	in house	0.77	C	1.92	first reported 1.67
1197	D2163	0.710		-0.18	
1198	D2163	0.703		-0.43	
1307	GC	0.711		-0.15	
1634	ISO7941	0.59	DG(0.05)	-4.38	
	normality	OK			
	n	19			
	outliers	3			
	mean (n)	0.715			
	st.dev. (n)	0.0377			
	R(calc.)	0.106			
	R(ASTM D2163)	0.082			Compare R(EN27941(liq))=1.255

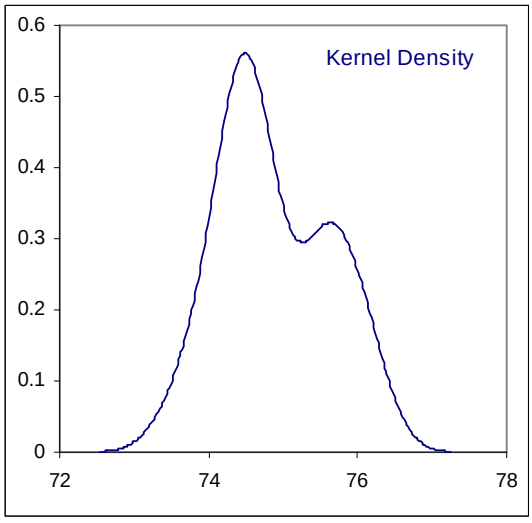




Determination of iso-Butane; results in %mol/mol

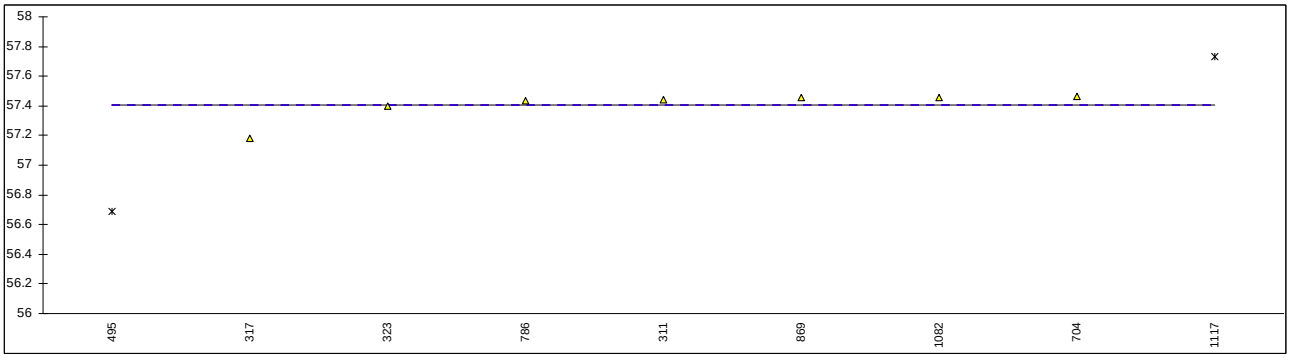
lab	method	value	mark	z(targ)	remarks
150	D2163	74.857		-0.11	
171	D2163	74.4030		-1.17	
225		-----		-----	
311	D2163	74.369		-1.25	
317	D2163	75.609	ex	1.65	see §4
323	D2163	74.55		-0.83	
334	EN27941	74.4		-1.18	
336	EN27941	75.98		2.51	
444	IP405	73.95		-2.23	
463	EN27941	75.59		1.60	
495	D2163	74.756		-0.34	
511	D2163	73.60		-3.04	manually calc. via 100%-sum of all other components
704	D2163	74.523		-0.89	
786	D2163	74.596		-0.72	
869	D2163	74.349		-1.29	
912		-----		-----	
1011	EN27941	75.4		1.16	
1082	IP473	74.678		-0.53	
1095	EN27941	75.943		2.42	
1117	in house	74.06	C	-1.97	first reported 74.87
1197	D2163	75.337		1.01	
1198	D2163	74.445		-1.07	
1307	GC	75.690		1.83	
1634	ISO7941	76.20		3.02	
	normality	OK			
	n	20			
	outliers	1			
	mean (n)	74.904			
	st.dev. (n)	0.6810			
	R(calc.)	1.907			
	R(ASTM D2163)	1.200			Compare R(EN27941(liq))=1.516

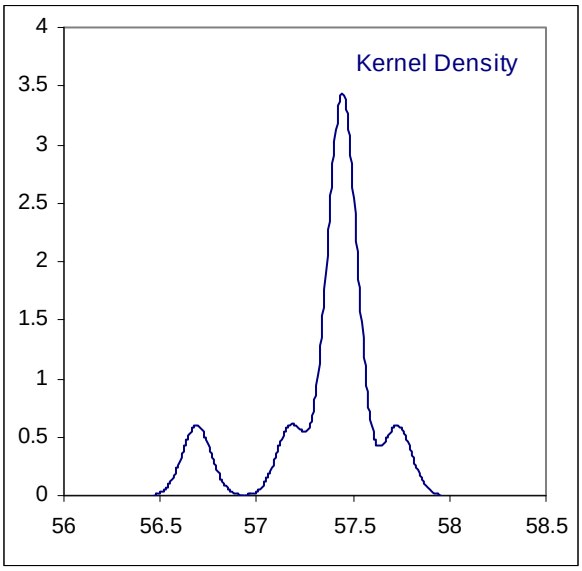


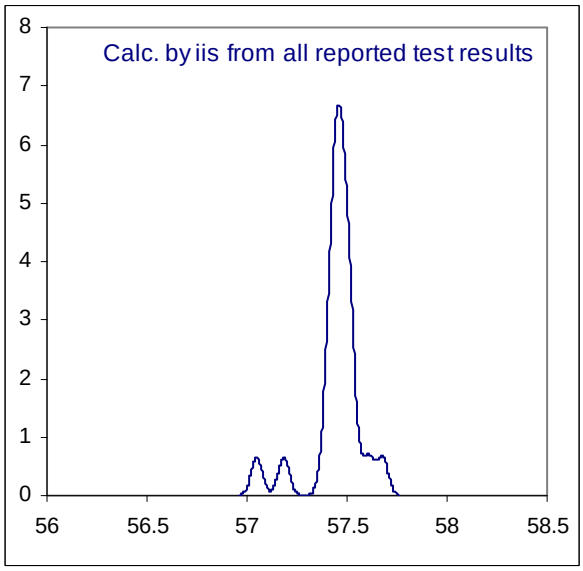


Determination of Molar Mass; results in g/mol

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
171		-----		-----	
225		-----		-----	
311	in house	57.440		-----	
317	D2598	57.48		-----	
323	D5388	57.4		-----	
334		-----		-----	
336		-----		-----	
444		-----		-----	
463		-----		-----	
495		56.69	G(0.05)	-----	Calculated by iis from the reported test results: 57.46
511		-----		-----	
704		57.4633		-----	
786	D2421	57.438		-----	
869	D2598	57.46		-----	
912		-----		-----	
1011		-----		-----	
1082	ISO6976	57.46		-----	
1095		-----		-----	
1117	in house	57.73	D(0.05)	-----	
1197		-----		-----	
1198		-----		-----	
1307		-----		-----	
1634		-----		-----	
					Calculated by iis from all reported test results:
	normality	not OK			OK
	n	7			18
	outliers	2			4
	mean (n)	57.406			57.465
	st.dev. (n)	0.1020			0.0381
	R(calc.)	0.286			0.107
	R(lit)	unknown			unknown

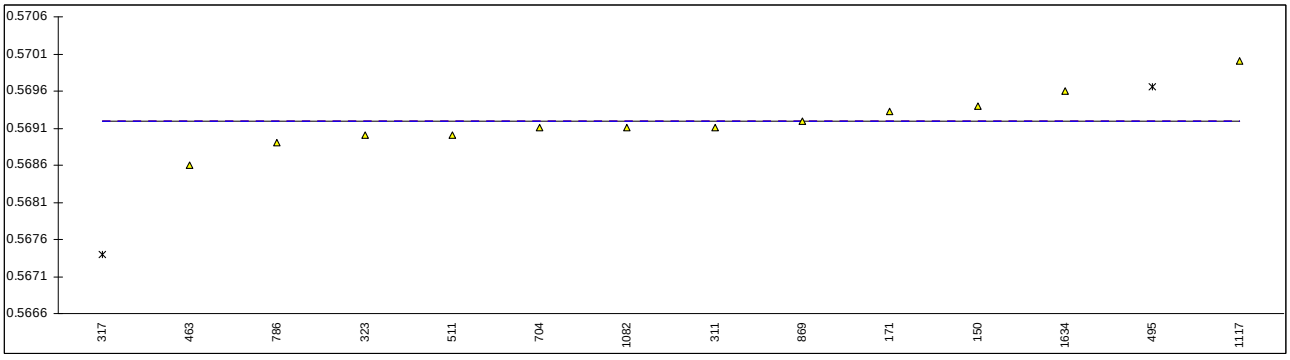


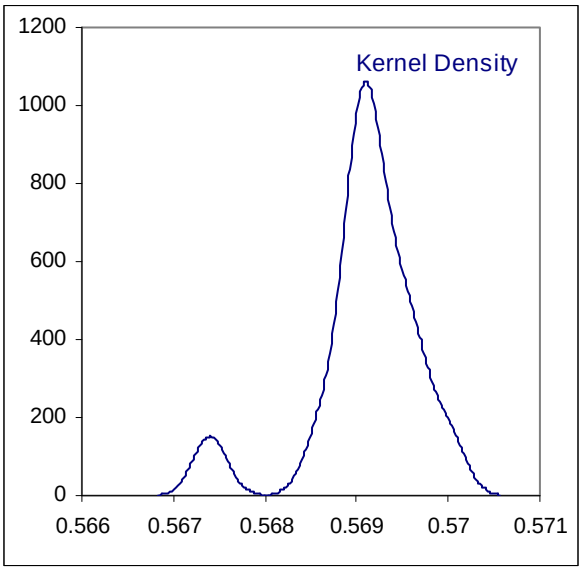


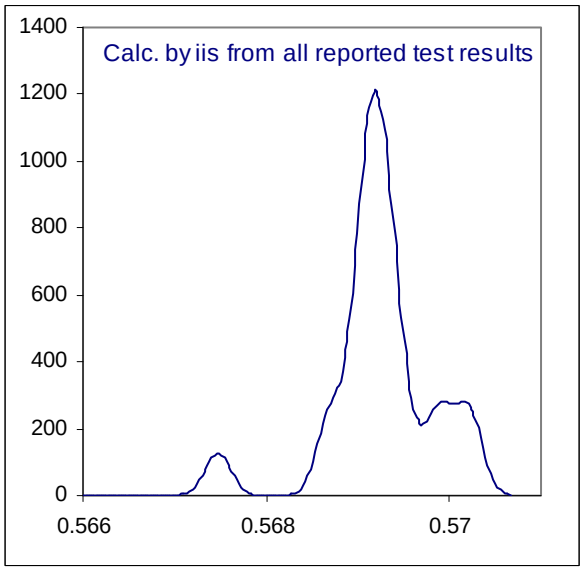


Determination of Relative Density @ 60F; unitless results

lab	method	value	mark	z(targ)	remarks
150	D2598	0.5694		-----	
171	D2598	0.56933		-----	
225		-----		-----	
311	D2598	0.5691		-----	
317	D2421	0.5674	G(0.01)	-----	
323	D2598	0.5690		-----	
334		-----		-----	
336		-----		-----	
444		-----		-----	
463	ISO8973	0.5686		-----	
495	D2598	0.569652		-----	
511	D2598	0.5690		-----	
704	D2598	0.5691		-----	
786	D2598	0.5689		-----	
869	D2163	0.5692		-----	
912		-----		-----	
1011		-----		-----	
1082	D2598	0.5691		-----	
1095		-----		-----	
1117	calc.	0.5700		-----	
1197		-----		-----	
1198		-----		-----	
1307		-----		-----	
1634	ISO8973	0.5696		-----	
					Calculated by iis from all reported test results:
	normality	OK			OK
	n	13			20
	outliers	1			2
	mean (n)	0.5692			0.5693
	st.dev. (n)	0.00037			0.00043
	R(calc.)	0.0010			0.0012
	R(lit)	unknown			unknown

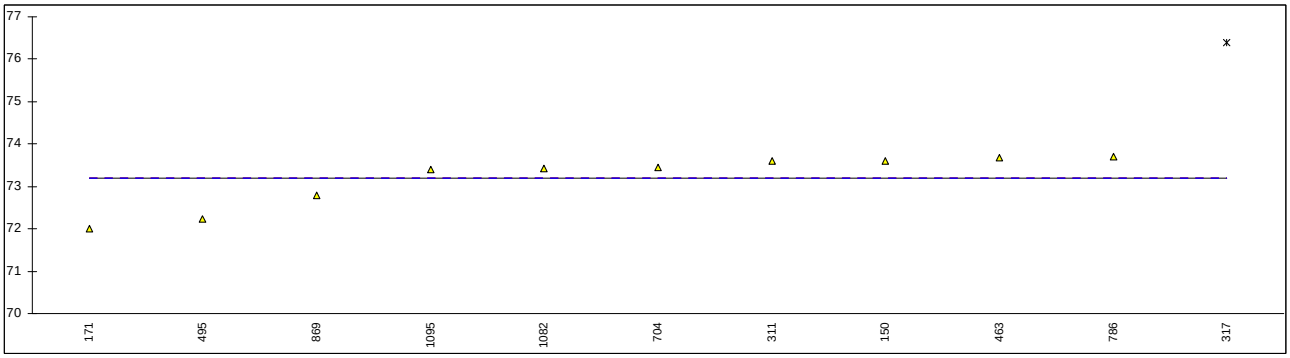


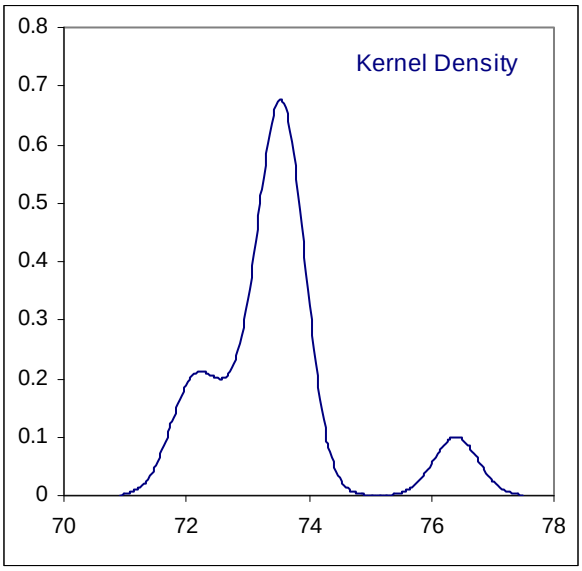


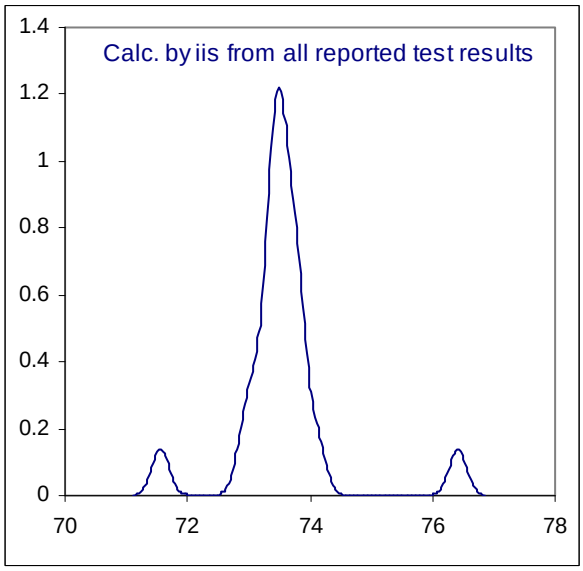


Determination of Abs. Vapour Pressure; results in psi

lab	method	value	mark	z(targ)	remarks
150	IP432	73.6	C	-----	first reported 59.6
171	D2598	72.0036		-----	Calculated by iis from the reported test results: 73.17 psi
225		-----		-----	
311	D2598	73.6		-----	
317	ISO8973	76.4	G(0.01)	-----	
323		-----		-----	
334		-----		-----	
336		-----		-----	
444		-----		-----	
463	ISO8973	73.6752		-----	
495	D2598	72.23		-----	Calculated by iis from the reported test results: 73.44 psi
511		-----		-----	
704	ISO8973	73.45		-----	
786	ISO8973	73.699		-----	
869	D2163	72.8		-----	
912		-----		-----	
1011		-----		-----	
1082	ISO8973	73.436		-----	
1095		73.4		-----	
1117		-----		-----	
1197		-----		-----	
1198		-----		-----	
1307		-----		-----	
1634		-----		-----	
					Calculated by iis from all reported test results:
	normality	not OK			OK
	n	10			18
	outliers	1			2
	mean (n)	73.19			73.52
	st.dev. (n)	0.622			0.296
	R(calc.)	1.74			0.83
	R(lit)	unknown			unknown

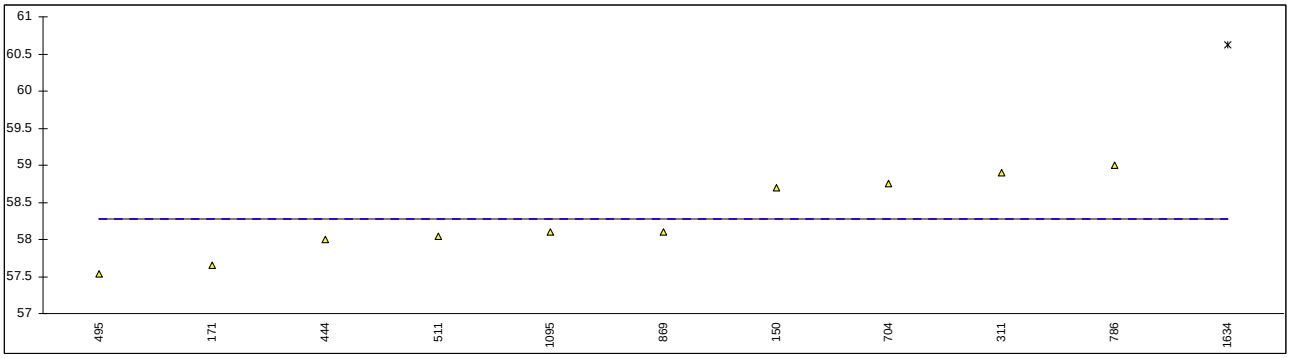


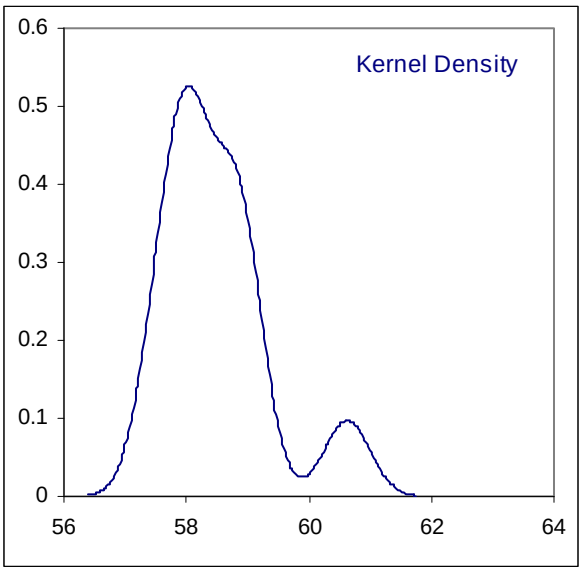


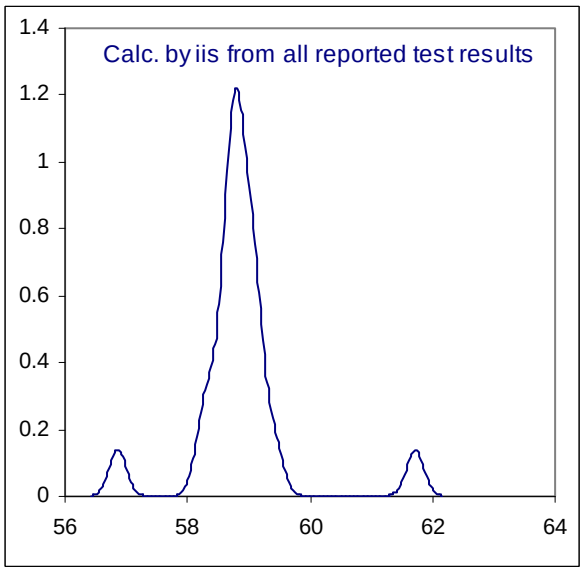


Determination of Rel. Vapour Pressure; results in psi

lab	method	value	mark	z(targ)	remarks
150	D2598	58.7		-----	
171	D2598	57.656		-----	
225		-----		-----	
311	D2598	58.9		-----	
317		-----		-----	
323		-----		-----	
334		-----		-----	
336		-----		-----	
444	IP432	58.0	C	-----	first reported Abs. Vapour Pressure 58.0
463		-----		-----	
495	D2598	57.53		-----	
511	D2598	58.038		-----	
704	ISO8973	58.76		-----	
786	ISO8973	59.004		-----	
869	D2163	58.1		-----	
912		-----		-----	
1011		-----		-----	
1082		-----		-----	
1095		58.1		-----	
1117		-----		-----	
1197		-----		-----	
1198		-----		-----	
1307		-----		-----	
1634	ISO8973	60.626	G(0.05)	-----	
					Calculated by iis from all reported test results:
	normality	OK			OK
	n	10			18
	outliers	1			2
	mean (n)	58.28			58.82
	st.dev. (n)	0.524			0.296
	R(calc.)	1.47			0.83
	R(lit)	unknown			unknown







APPENDIX 2

Additional details

	Sample Volume	Type of vaporizer	Remarks
150	---	---	C6+ 0.005 %mol/mol
171	---	---	n-pentane: 0.0037 %mol/mol (0.0044 %M/M)
225			
311	---	---	n-pentane: 0.0037 %mol/mol & helium 0.45 %mol/mol
317	0.1 mL	none, liquid injection	none
323	190 mL	none, liquid injection	none
334	---	---	none
336	---	---	---
444	---	none, liquid injection	sample for one injection only, n-pentane <0.01 %mol/mol
463	45 mL	---	---
495	202 mL	waterbath	n-pentane trace & helium 1.4 %mol/mol (0.05 %M/M)
511	---	---	---
704	0.0005 mL	SPL	n-pentane: 0.004 %M/M (0.003 %mol/mol)
786	---	---	---
869	0.2 µL	none, liquid injection	n-pentane: 0.003 %mol/mol
912			
1011	---	---	none
1082	0.1 µL	none, liquid injection	n-pentane: 0.003 %M/M
1095	20.1 grams	vaporised at RT	---
1117	18.1 mL	LSV	iso-butane and propene co-elute
1197	---	GSV with heater	n-pentane: 0.003695 %mol/mol
1198	---	GSV with heater	n-pentane: 0.003539 %mol/mol
1307	---	---	---
1634	---	---	none

APPENDIX 3

List of participants

SGS Belgium N.V. - OGC	Antwerp	BELGIUM
FINA Antwerp Olefins	Antwerp	BELGIUM
SGS Cote d'Ivoire S.A.	Abidjan	CÔTE D'IVOIRE
Neste Oil Oyj, Development and Laboratories	Porvoo	FINLAND
SGS France S.A.	Lavera	FRANCE
SGS France S.A. (Donges)	Montoir de Bretagne	FRANCE
SGS Germany GmbH	Wilhelmshaven	GERMANY
M/S SGS India Pvty Ltd.	Thane, Mumbai	INDIA
Petronas Gas Berhad - laboratory GPPA	Kerteh, Kemamn, Terengganu	MALAYSIA
Petronas Gas Berhad - laboratory GPPB	Kerteh, Kemamn, Terengganu	MALAYSIA
SGS-CSTC Standards Technical Services Ltd., Zhuhai branch	Shenzhen	P.R. of CHINA
SGS Del Peru S.A.C.	Callao	PERU
GalpEnergia / Petrogal S.A. - Refinaria do Porto – Laboratorio	Leça da Palmeira	PORTUGAL
GalpEnergia / Petrogal S.A. - Refinaria de Sines - Laboratorio	Sines	PORTUGAL
Companhia Logistica de Combustiveis S.A.	Azambuja	PORTUGAL
ZAO "SGS Vostok Ltd", Temruk branch	Temryuk, Krasnodar Region	RUSSIA
SGS Sweden AB	Göteborg	SWEDEN
SGS Nederland BV - OGC	Spijkenisse	THE NETHERLANDS
SGS Nederland BV	Vlissingen	THE NETHERLANDS
Shell Ned. Chemie B.V., Clab/3	Moerdijk	THE NETHERLANDS
SGS New Orleans (St. Rose)	New Orleans	U.S.A.
SGS Deer Park	Deer Park	U.S.A.
SGS Ukraine Ltd., Odessa branch	Odessa	UKRAINE
SGS (UK) Ltd., Billingham branch (Teesside)	Billingham, Cleveland	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:

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12 J.N. Miller, Analyst, 118, 455, (1993)
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15 ISO 17043
16 EN 27941
17 ASTM D2163
18 ASTM D2421