Results of Proficiency Test
Liquefied Butane Analysis
June 2013

Organised by: Institute for Interlaboratory Studies
                      Spijkenisse, the Netherlands

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1   INTRODUCTION

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

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

In the 2013 proficiency test 33 laboratories in 20 different countries have participated. See appendix 3 for the number of participants per country. In this report the results of the 2013 proficiency test on Liquefied Butane are presented and discussed. This report is also electronically available through the iis internet site http://www.iisnl.com.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test.
To optimise the costs for the participating laboratories, it was decided to prepare one Liquefied Butane mixture. The mixture was divided over a batch of 40 cylinders.
The cylinder size is a cost-effective one-litre cylinder with dip tube device. Each cylinder, filled with approx 200 grams of liquefied butane mixture, was uniquely numbered. The limited cylinder size is chosen to optimise sample stability, cylinder costs, transport and handling costs.

Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1   QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010 and ILAC-G13:2007 (R007). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Also customer's satisfaction is measured on regular basis by the distribution of questionnaires.
EffecTech is an accredited provider of proficiency testing schemes under the requirements of ISO/IEC17043:2010 by UKAS (no. 4719).

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

In this proficiency test only one sample was used. One batch of 40 one litre cylinders with artificial Liquefied Butane mixture was prepared and tested for homogeneity by EffecTech (Uttoxeter, United Kingdom) in conformance with ISO Guide 35: 2006 and ISO/IEC17043:2010 (job 13/296) starting May 1, 2013. Each cylinder was uniquely numbered. Every cylinder in the batch was analysed using 6 replicate measurements. The within bottle and between bottle variations were then assessed in accordance with ISO Guide 35:2006 (Annex A.1). This procedure showed that the between bottle variations were all small compared to the uncertainties on the reference values on each component. Hence, a single reference value could be safely assigned to the entire batch of samples.
The repeatability values (r) were calculated per component by multiplication of the respective standard deviation by 2.8. Subsequently, the calculated repeatabilities were compared with 0.3 times the reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

Parameter	r(observed) in %mol/mol	0.3 X R(D2163) in %mol/mol
iso-Butane	0.081	0.226
Propane	0.027	0.056
Propylene	0.025	0.035
n-Butane	0.027	0.194
1,3-Butadiene	0.004	0.032
iso-Butylene	0.010	0.104
1-Butene	0.026	0.206
trans-2-Butene	0.014	0.086
cis-2-Butene	0.019	0.101
iso-Pentane	0.008	0.032

Table 1: homogeneity test results of samples #13074

Each calculated repeatability is far less than 0.3 times the corresponding reproducibility of the reference method ASTM D2163:96.
Therefore, homogeneity of the subsamples #13074 was assumed.

To each of the participating laboratories one 1L cylinder was sent on May 15, 2013.

2.5 STABILITY OF THE SAMPLES

EffecTech (Uttoxeter, United Kingdom) declares that the prepared gas cylinders have a shelf life of at least 6 months. This is sufficient for the proficiency testing purposes.

2.6 ANALYSES

The participants were asked to determine: Propane, Propylene, n-Butane, 1,3-Butadiene, iso-Butylene, 1-Butene, trans-2-Butene, cis-2-Butene, iso-Pentane, iso-Butane, Molar Mass, Relative Density and Absolute and Relative Vapour pressure. Also some method details were requested to be reported.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website (www.iisnl.com).

A SDS and a form to confirm receipt of the samples were added to the sample package Participants are also requested to send a remark if other components were found e.g. Helium or/and Pentane.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original results are tabulated per determination in the appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected data are put under 'Remarks' in the result tables in appendix 1. Results that came in after deadline were not taken into account in the screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<…' or '>…' were not used in the statistical evaluation.

First the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers this check was repeated. In case a

data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

Finally the reproducibilities were calculated from the standard deviations by multiplying these with a factor of 2.8.

3.2   GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 4; nos.13 and 14).

3.3   Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM, EN-, ISO-, IP reproducibilities, the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated according to:

z(target) = (result - average of PT) / target standard deviation

The z(target) scores are listed in the result tables in appendix 1.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate the fit-for-useness of the reported test result.

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

$$| z | < 1 \quad \text{good}$$
$$1 < | z | < 2 \quad \text{satisfactory}$$
$$2 < | z | < 3 \quad \text{questionable}$$
$$3 < | z | \qquad \text{unsatisfactory}$$

4   EVALUATION

In this proficiency test several problems were encountered with sample transport. Due to customs problems one cylinder did not reach the laboratory in time to test the cylinder and to report results to be included in the final report. In total four laboratories reported test results after the final reporting date and three laboratories did not report any test results at all. Not all laboratories were able to report all test results requested.

In total 30 participants reported 358 numerical results. Observed were 15 outlying results, which is 4.2% 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 test methods that are used by the various laboratories are taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 4.
Not all original data sets proved to have a normal distribution. Not normal distributions were found for n-Butane, 1,3-Butadiene and Relative Density@60F.

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 reproducibilities are mentioned. Therefore the precision data from the previous version ASTM D2163:96 were used.

One laboratory appeared to have some problems. Nine test results (=64%) reported by laboratory 1065 appeared to be statistical outliers and because all test results of one

laboratory are correlated, the remaining test results of laboratory 1065 were excluded manually prior to the statistical analysis.

iso-Butane: The determination of this main component may be problematic, depending on the test method used by the laboratory. Two statistical outliers were detected. The calculated reproducibility after rejection of the suspect data, is not in agreement with the requirements of ASTM D2163:96. However, the calculated reproducibility is in good agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).

Propane: The determination of this component may be problematic, depending on the test method used by the laboratory. Only one statistical outlier was detected. However, 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 good agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).

Propylene: The determination of this component may be problematic, depending on the test method used by the laboratory. Only one statistical outlier was detected. The calculated reproducibility, after exclusion of the statistical outlier, 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).

n-Butane: No analytical problems were observed. Only one statistical outlier was detected and the calculated reproducibility, after rejection of the statistical outlier, is in full agreement with the requirements of ASTM D2163:96 and also with the reproducibility of EN27941 (identical to IP 405 and ISO 7941).

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

iso-Butylene: 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).

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. 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).

cis-2-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).

iso-Pentane: The determination of this component may be problematic, depending on the test method used by the laboratory. Two statistical outliers were detected. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D2163:96. However, the calculated reproducibility is in good agreement with the less strict reproducibility of EN27941 (identical to IP 405 and ISO 7941).

Molar Mass: This calculated parameter may be problematic. The results vary over a range from 56.6 - 57.52 g/mol and only one statistical outlier was observed (in 14 test results). The calculated reproducibility after rejection of the statistical outlier is large in comparison with the theoretical reproducibility (0.17 *vs* 0.08). See also the discussion in 4.4.

Relative Density: This calculated parameter may not be problematic. The results vary over a range from 0.5699 - 0.5708 and one statistical outlier was observed (in 17 test results). The calculated reproducibility after rejection of the statistical outlier is in good agreement with the theore-tical reproducibility (0.0007 *vs* 0.0006). See also the discussion in 4.4.

Abs. Vapour Pres.:   This calculated parameter may be problematic. The results vary over a large range (61.4 – 81.22 psi). One statistical outlier was observed (in 16 test results). The calculated reproducibility after rejection of the statistical outlier is large in comparison with the theoretical reproducibility (3.95 *vs* 1.00). See also the discussion in 4.4.

Rel. Vapour Pres.: This calculated parameter may be problematic. The results vary over a large range (57.246 – 62.7205 psi). One statistical outlier was observed (in 15 test results). The calculated reproducibility after rejection of the statistical outlier is large in comparison with the theoretical reproducibility (1.90 *vs* 1.00). See also the discussion in 4.4.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	cons. value	2.8 * sd	R(D2163) in **%mol**	R(EN27941) liq.-inj. in **%mol**	R(EN27941) liq.-inj. in **%M/M**
iso-Butane	%mol/mol	27	77.189	1.033	0.772	1.479	1.5
Propane	%mol/mol	28	1.003	0.178	0.116	1.300	1
Propylene	%mol/mol	28	1.889	0.441	0.217	1.362	1
n-Butane	%mol/mol	29	2.104	0.257	0.242	0.986	1
1,3-Butadiene	%mol/mol	29	1.193	0.229	0.137	1.060	1
iso-Butylene	%mol/mol	27	4.999	0.330	0.576	1.022	1
1-Butene	%mol/mol	28	3.006	0.249	0.346	1.022	1
trans-2-Butene	%mol/mol	29	4.079	0.278	0.470	1.022	1
cis-2-Butene	%mol/mol	29	3.989	0.359	0.459	1.022	1
iso-Pentane	%mol/mol	28	0.596	0.116	0.069	0.794	1
Molar Mass	g/mol	13	57.419	0.166	n/a	n/a	n/a
Rel. Density @60F		16	0.5703	0.0007	n/a	n/a	n/a
Abs. Vapour pres.	psi	14	73.25	3.95	n/a	n/a	n/a
Rel. Vapour pres.	psi	14	58.39	1.90	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 a good compliance of the group of participating laboratories with the relevant standard. The problematic components/tests have been discussed in paragraph 4.1.

4.3   COMPARISON OF THE PROFICIENCY TEST OF JUNE 2013 WITH PREVIOUS PTS

	June 2013	June 2012	May 2011	May 2010	July 2009
Number of reporting labs	30	30	27	22	25
Number of results reported	358	373	333	263	291
Statistical outliers	15	39	29	20	16
Percentage outliers	4.2%	10.5%	8.7%	7.6%	5.5%

table 3: comparison with previous proficiency tests

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

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

	June 2013	June 2012	May 2011	May 2010	July 2009
iso-Butane	-	--	--	--	--
Propane	--	-	--	--	--
Propylene	--	-	--	--	--
n-Butane	+/-	++	++	+	-
1,3-Butadiene	--	+/-	-	--	+/-
iso-Butylene	++	++	++	++	++
1-Butene	++	++	++	-	++
trans-2-Butene	++	+	+	-	--
cis-2-Butene	++	+	++	-	--
iso-Pentane	--	+	-	-	--

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

The following performance categories were used in the above table:

  ++:   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 the majority of the reproducibility requirements of ASTM D2163 differ significantly from the reproducibility requirements of EN27941 (for liquid injection), the outcome of the evaluations will be strongly dependent on the target test method selected for the evaluation.

The consensus values as determined in this PT are compared with the average values from the homogeneity testing by EffecTech (Uttoxeter, United Kingdom) in the following table. From this comparison it is clear that most consensus values as determined in this PT are very well in line with the values as determined during the preparation of the gas cylinders.

Parameter	Average values by EffecTech in %mol/mol	Consensus values from participants results in %mol/mol	Absolute differences in %mol/mol	z-score
iso-Butane	77.062	77.189	-0.127	+0.46
Propane	0.987	1.003	-0.016	-0.39
Propylene	1.963	1.889	+0.074	+0.95
n-Butane	2.039	2.104	-0.065	-0.75
1,3-Butadiene	1.249	1.193	+0.056	+1.14
iso-Butylene	5.027	4.999	+0.028	+0.14
1-Butene	3.005	3.006	-0.001	-0.01
trans-2-Butene	4.046	4.079	-0.033	-0.20
cis-2-Butene	4.024	3.989	+0.035	+0.21
iso-Pentane	0.598	0.596	+0.002	+0.08

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

In total nine laboratories reported the presence of some impurities, components 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 in most cases. See the differences between the values from the results as reported by the participating laboratories (each using its own calculation procedure) and the values as calculated by iis using one calculation procedure for each set of laboratory test results. For the calculation of the Molar Mass, Relative Density and Vapour Pressure several standardized methods are available, e.g. ASTM D2421 for the interconversion of the units to gas-volume, liquid-volume or mass basis. Also different methods for the calculation of the Vapour Pressure do exist. In ISO 8973 (identical to IP432) the Vapour Pressure is calculated from the mole fraction per component and a Vapour Pressure factor of that component (given for all components). In ASTM D2598 the Vapour Pressure is calculated from the liquid volume percentage per component and a Vapour Pressure factor of that component (given for only several components). Also the selection of the tables to be used for the calculations may cause additional uncertainty.

APPENDIX 1

Determination of iso-Butane on sample #13074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	77.257		0.25	
150	D2163	77.261		0.26	
171	D2163	76.583		-2.20	
311	INH-407	77.320		0.48	
323	D2163	76.04	G(0.05)	-4.17	
334	EN27941	77.600		1.49	
336	EN27941	77.68		1.78	
337	EN27941	77.042		-0.53	
357	D2163	77.110	C	-0.29	First reported 77.930
444	IP405	76.539		-2.36	
445	IP405/D2163	77.61		1.53	
495	D2163	77.49		1.09	
704	D2163	77.324		0.49	
706		-----		-----	
851	D2163	77.044		-0.52	
869	D2163	77.246		0.21	
912		-----		-----	
1006	D2163	76.998		-0.69	
1016	ISO7941	76.572		-2.24	
1040	DIN51619	77.21		0.08	
1062	D2163	77.7970		2.21	
1065	D2163	76.28	ex	-3.30	Result excluded see §4.1
1095	EN27941	77.617		1.55	
1197	D2163	77.300		0.40	
1198	D2163	76.663		-1.91	
1257		-----		-----	
1259	EN27941	77.242		0.19	
1307	INH-LHA	76.662		-1.91	
1491	ISO7941	76.948		-0.87	
1616	D2163	77.421	C	0.84	First reported 78.017
1634	ISO7941	76.89		-1.08	
1689	INH-230	77.668		1.74	
1720	D2163	78.319	G(0.05)	4.10	

normality	OK		
n	27		
outliers	2	(+1 ex)	
mean (n)	77.1887		
st.dev. (n)	0.36876		
R(calc.)	1.0325		
R(D2163:96)	0.7719		Compare R(EN27941(liq)) = 1.4792
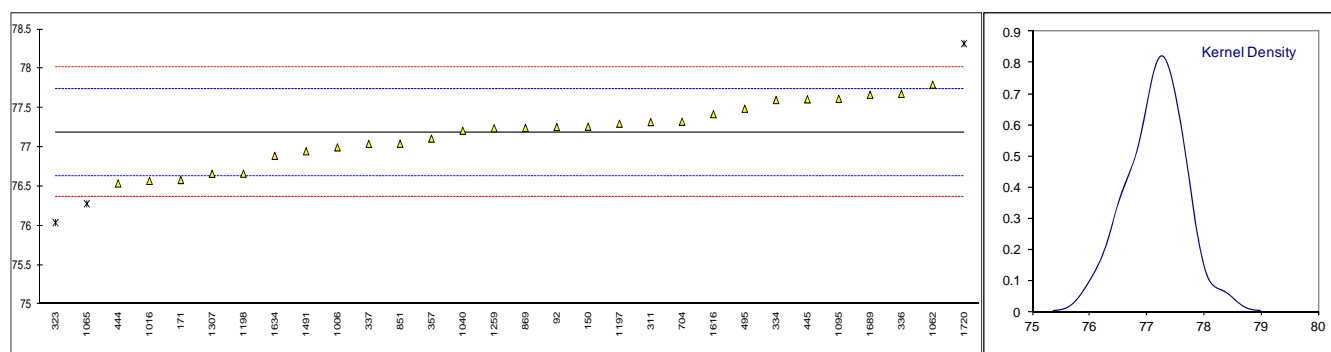
Determination of Propane on sample #13074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	1.004		0.02	
150	D2163	1.019		0.38	
171	D2163	1.063		1.45	
311	INH-407	0.997		-0.15	
323	D2163	0.99		-0.32	
334	EN27941	0.944		-1.44	
336	EN27941	0.97		-0.81	
337	EN27941	0.983		-0.49	
357	D2163	1.030		0.65	
444	IP405	0.988		-0.37	
445	IP405/D2163	0.86		-3.48	
495	D2163	1.13		3.07	
704	D2163	1.095		2.22	
706		-----		-----	
851	D2163	1.019		0.38	
869	D2163	1.022		0.45	
912		-----		-----	
1006	D2163	1.051		1.15	
1016	ISO7941	0.992		-0.28	
1040	DIN51619	1.00		-0.08	
1062	D2163	0.9984		-0.12	
1065	D2163	2.29	G(0.01)	31.19	
1095		-----	W	-----	Result withdrawn
1197	D2163	0.955		-1.17	
1198	D2163	0.962		-1.00	
1257		-----		-----	
1259	EN27941	0.958		-1.10	
1307	INH-LHA	0.989		-0.35	
1491	ISO7941	1.093		2.17	
1616	D2163	0.990		-0.32	
1634	ISO7941	1.00		-0.08	
1689	INH-230	1.132		3.12	
1720	D2163	0.860		-3.48	

normality	OK		
n	28		
outliers	1		
mean (n)	1.0034		
st.dev. (n)	0.06373		
R(calc.)	0.1785		
R(D2163:96)	0.1155	Compare R(EN27941(liq)) = 1.2998
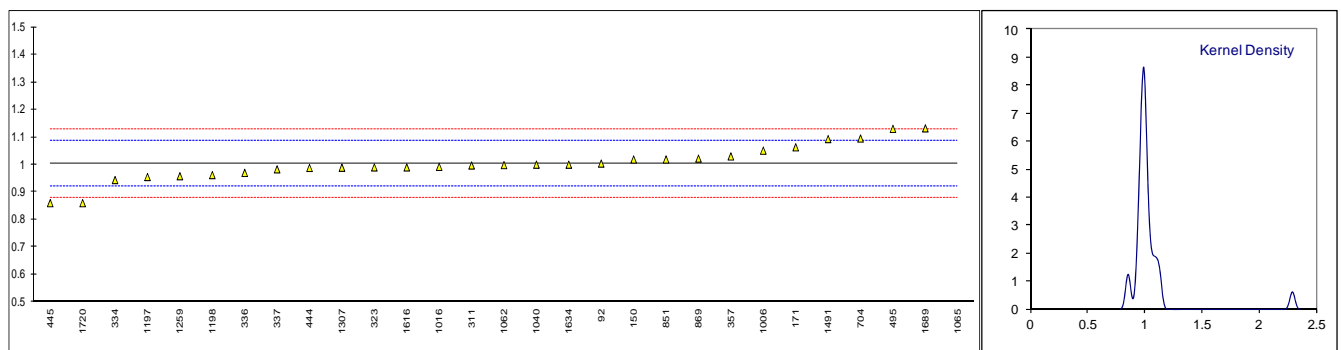
Determination of Propylene on sample #13074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	1.885		-0.05	
150	D2163	1.901		0.16	
171	D2163	2.154		3.41	
311	INH-407	1.866		-0.29	
323	D2163	1.96		0.92	
334	EN27941	1.790		-1.27	
336	EN27941	1.82		-0.89	
337	EN27941	1.842		-0.60	
357	D2163	1.961		0.93	
444	IP405	1.860		-0.37	
445	IP405/D2163	1.65		-3.08	
495	D2163	2.20		4.01	
704	D2163	2.089		2.58	
706		-----		-----	
851	D2163	1.955		0.85	
869	D2163	1.841		-0.62	
912		-----		-----	
1006	D2163	2.000		1.43	
1016	ISO7941	1.915		0.34	
1040	DIN51619	1.88		-0.11	
1062	D2163	1.8675		-0.28	
1065	D2163	5.20	G(0.01)	42.63	
1095		-----	W	-----	Result withdrawn
1197	D2163	1.783		-1.36	
1198	D2163	1.725		-2.11	
1257		-----		-----	
1259	EN27941	1.904		0.19	
1307	INH-LHA	1.800		-1.14	
1491	ISO7941	2.106		2.80	
1616	D2163	1.655		-3.01	
1634	ISO7941	1.92		0.40	
1689	INH-230	2.082		2.49	
1720	D2163	1.477		-5.30	

normality	OK	
n	28	
outliers	1	
mean (n)	1.8889	
st.dev. (n)	0.15739	
R(calc.)	0.4407	
R(D2163:96)	0.2175	Compare R(EN27941(liq)) = 1.3621
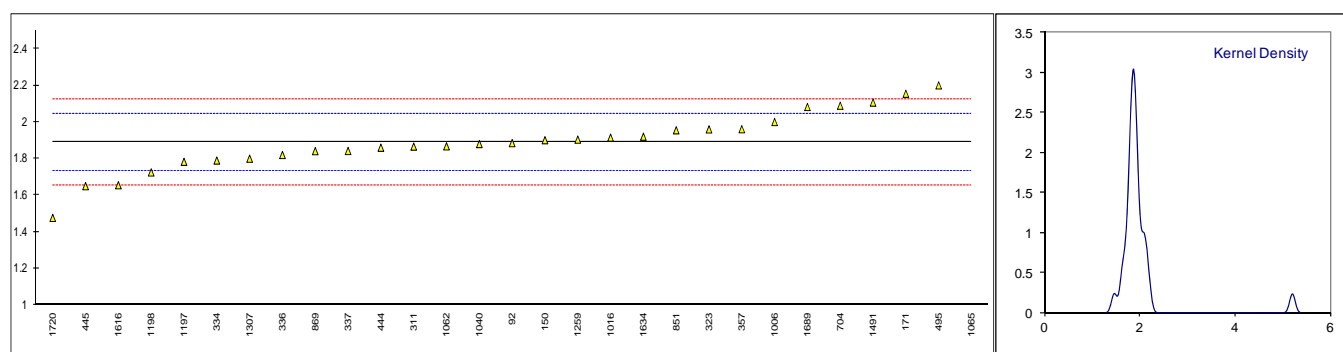
Determination of n-Butane on sample #13074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	1.955		-1.72	
150	D2163	2.133		0.34	
171	D2163	2.036		-0.78	
311	INH-407	2.076		-0.32	
323	D2163	2.11		0.07	
334	EN27941	2.041		-0.73	
336	EN27941	2.02		-0.97	
337	EN27941	2.104		0.00	
357	D2163	2.061		-0.50	
444	IP405	2.094		-0.11	
445	IP405/D2163	2.08		-0.28	
495	D2163	2.01		-1.09	
704	D2163	2.032		-0.83	
706		-----		-----	
851	D2163	2.092		-0.14	
869	D2163	2.132		0.33	
912		-----		-----	
1006	D2163	2.089		-0.17	
1016	ISO7941	2.280		2.04	
1040	DIN51619	2.09		-0.16	
1062	D2163	2.1703		0.77	
1065	D2163	1.73	G(0.05)	-4.32	
1095	EN27941	2.217		1.31	
1197	D2163	2.093		-0.13	
1198	D2163	2.369		3.06	
1257		-----		-----	
1259	EN27941	2.035		-0.80	
1307	INH-LHA	2.079		-0.29	
1491	ISO7941	2.255		1.75	
1616	D2163	2.122		0.21	
1634	ISO7941	2.22		1.34	
1689	INH-230	2.015		-1.03	
1720	D2163	2.002		-1.18	

normality	not OK		
n	29		
outliers	1		
mean (n)	2.1039		
st.dev. (n)	0.09184		
R(calc.)	0.2572		
R(D2163:96)	0.2422	Compare R(EN27941(liq)) = 0.9861	
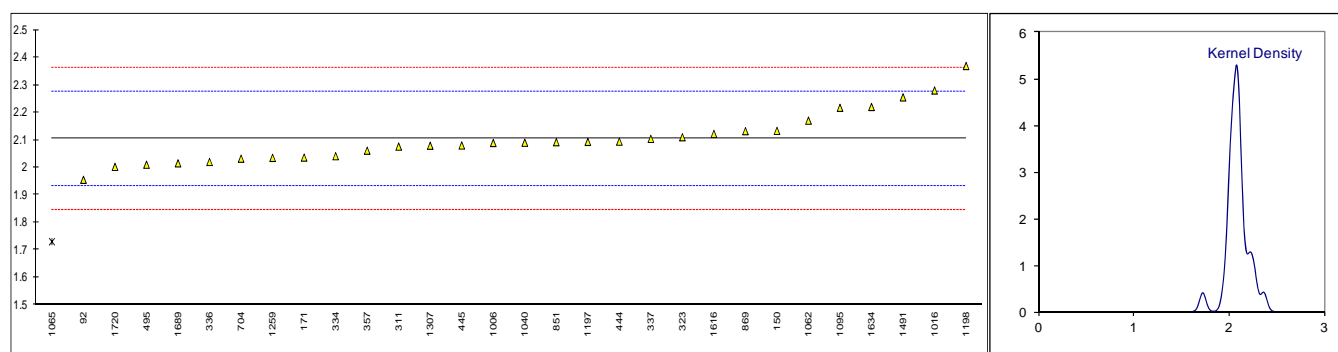
Determination of 1,3-Butadiene on sample #13074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	1.228		0.70	
150	D2163	1.237		0.89	
171	D2163	1.212		0.38	
311	INH-407	1.200		0.13	
323	D2163	1.33		2.78	
334	EN27941	1.131		-1.27	
336	EN27941	1.16		-0.68	
337	EN27941	1.218		0.50	
357	D2163	1.209		0.32	
444	IP405	1.315		2.48	
445	IP405/D2163	1.17		-0.48	
495	D2163	1.18		-0.27	
704	D2163	1.198		0.09	
706		-----		-----	
851	D2163	1.226		0.66	
869	D2163	1.192		-0.03	
912		-----		-----	
1006	D2163	1.222		0.58	
1016	ISO7941	1.228		0.70	
1040	DIN51619	1.21		0.34	
1062	D2163	1.0170		-3.60	
1065	D2163	1.37	ex	3.60	Result excluded see §4.1
1095	EN27941	1.316		2.50	
1197	D2163	1.183		-0.21	
1198	D2163	1.244		1.03	
1257		-----		-----	
1259	EN27941	1.160		-0.68	
1307	INH-LHA	1.210		0.34	
1491	ISO7941	1.227		0.68	
1616	D2163	0.995	C	-4.04	First reported 1.060
1634	ISO7941	1.26		1.36	
1689	INH-230	1.145		-0.99	
1720	D2163	0.988		-4.19	
	normality	not OK			
	n	29			
	outliers	0	(+1 ex)		
	mean (n)	1.1935			
	st.dev. (n)	0.08163			
	R(calc.)	0.2286			
	R(D2163:96)	0.1374			Compare R(EN27941(liq)) = 1.0596
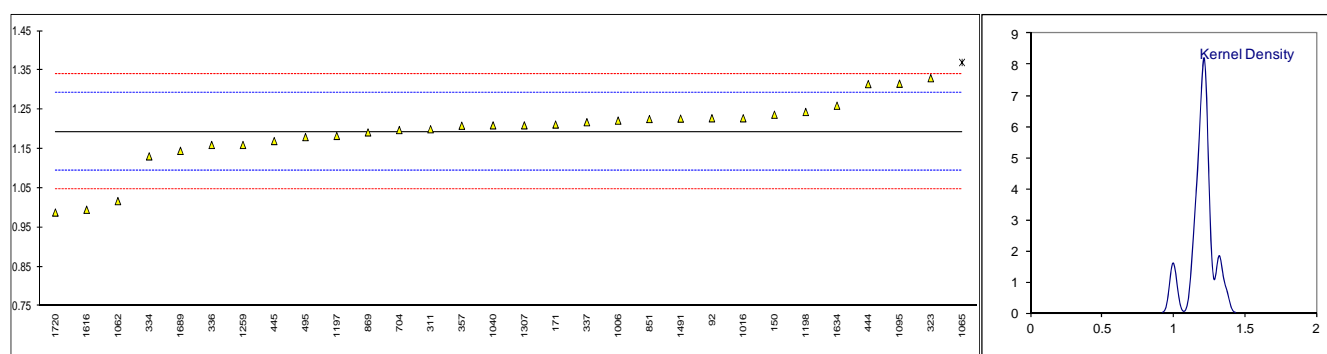
Determination of iso-Butylene on sample #13074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	4.878		-0.59	
150	D2163	4.905		-0.46	
171	D2163	5.05		0.25	
311	INH-407	4.919	C	-0.39	First reported 3.993
323	D2163	5.22		1.07	
334	EN27941	4.969		-0.15	
336	EN27941	4.89		-0.53	
337	EN27941	4.989		-0.05	
357	D2163	4.977		-0.11	
444	IP405	5.091		0.45	
445	IP405/D2163	5.08		0.39	
495	D2163	4.93		-0.34	
704	D2163	4.926		-0.36	
706		-----		-----	
851	D2163	5.200		0.98	
869	D2163	4.903		-0.47	
912		-----		-----	
1006	D2163	5.023		0.12	
1016	ISO7941	5.067		0.33	
1040	DIN51619	4.97		-0.14	
1062	D2163	4.1722	G(0.01)	-4.02	
1065	D2163	4.63	ex	-1.80	Result excluded see §4.1
1095	EN27941	5.242		1.18	
1197	D2163	4.941		-0.28	
1198	D2163	4.984		-0.07	
1257		-----		-----	
1259	EN27941	5.050		0.25	
1307	INH-LHA	5.188		0.92	
1491	ISO7941	4.942		-0.28	
1616	D2163	4.757		-1.18	
1634	ISO7941	5.04		0.20	
1689		-----		-----	
1720	D2163	4.850		-0.73	
	normality	OK			
	n	27			
	outliers	1	(+1 ex)		
	mean (n)	4.9993			
	st.dev. (n)	0.11801			
	R(calc.)	0.3304			
	R(D2163:96)	0.5756			Compare R(EN27941(liq)) = 1.0216
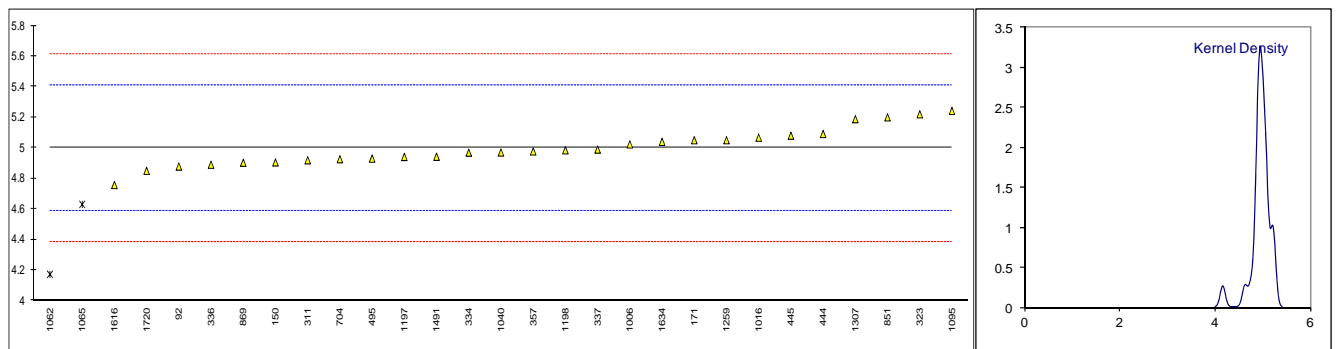
Determination of 1-Butene on sample #13074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	3.011		0.04	
150	D2163	2.877		-1.05	
171	D2163	3.16		1.24	
311	INH-407	2.980		-0.21	
323	D2163	3.20		1.57	
334	EN27941	2.934		-0.58	
336	EN27941	2.93		-0.62	
337	EN27941	3.013		0.05	
357	D2163	3.030		0.19	
444	IP405	3.081		0.60	
445	IP405/D2163	3.00		-0.05	
495	D2163	2.96		-0.37	
704	D2163	2.984		-0.18	
706		-----		-----	
851	D2163	2.968		-0.31	
869	D2163	2.897		-0.88	
912		-----		-----	
1006	D2163	3.008		0.01	
1016	ISO7941	3.063		0.46	
1040	DIN51619	2.99		-0.13	
1062	D2163	3.0464		0.32	
1065	D2163	2.55	G(0.01)	-3.69	
1095	EN27941	3.132		1.02	
1197	D2163	2.987		-0.16	
1198	D2163	3.143		1.11	
1257		-----		-----	
1259	EN27941	2.909		-0.79	
1307	INH-LHA	3.100		0.76	
1491	ISO7941	3.020		0.11	
1616	D2163	2.848		-1.28	
1634	ISO7941	3.03		0.19	
1689		-----		-----	
1720	D2163	2.874		-1.07	

normality	OK	
n	28	
outliers	1	
mean (n)	3.0063	
st.dev. (n)	0.08891	
R(calc.)	0.2489	
R(D2163:96)	0.3461	Compare R(EN27941(liq)) = 1.0216
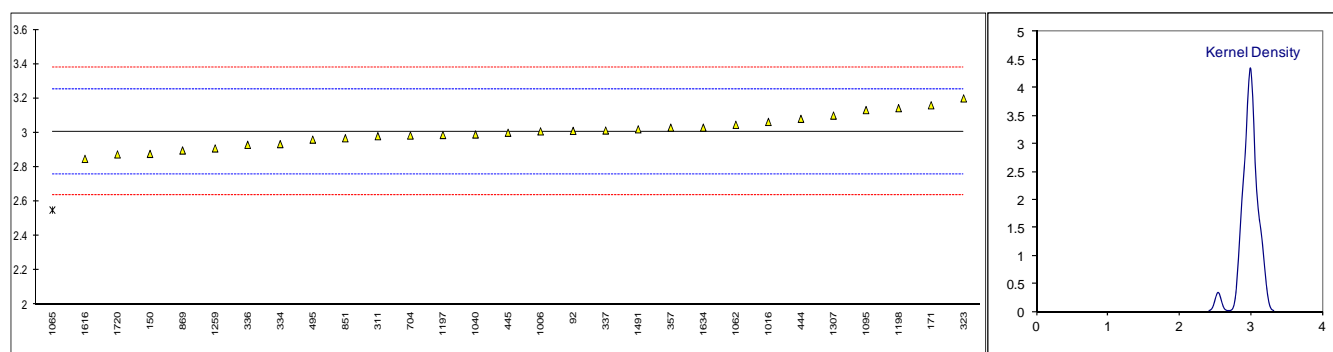
Determination of trans-2-Butene on sample #13074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	4.215		0.81	
150	D2163	4.032		-0.28	
171	D2163	4.118		0.23	
311	INH-407	4.043		-0.21	
323	D2163	4.27		1.14	
334	EN27941	4.050		-0.17	
336	EN27941	4.01		-0.41	
337	EN27941	4.116		0.22	
357	D2163	4.028		-0.30	
444	IP405	4.213		0.80	
445	IP405/D2163	4.03		-0.29	
495	D2163	3.85		-1.37	
704	D2163	3.945		-0.80	
706		-----		-----	
851	D2163	4.046		-0.20	
869	D2163	4.041		-0.23	
912		-----		-----	
1006	D2163	4.053		-0.15	
1016	ISO7941	4.155		0.45	
1040	DIN51619	4.06		-0.11	
1062	D2163	4.2248		0.87	
1065	D2163	2.98	G(0.01)	-6.55	
1095	EN27941	4.163		0.50	
1197	D2163	4.077		-0.01	
1198	D2163	4.059		-0.12	
1257		-----		-----	
1259	EN27941	4.070		-0.05	
1307	INH-LHA	4.246		1.00	
1491	ISO7941	3.978		-0.60	
1616	D2163	4.147		0.41	
1634	ISO7941	4.13		0.30	
1689	INH-230	3.914		-0.98	
1720	D2163	4.006		-0.44	

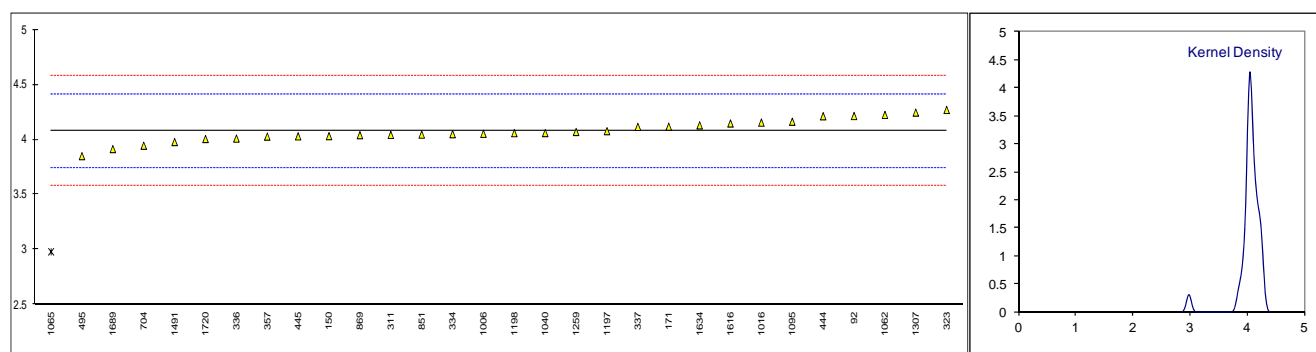|	
normality	OK
n	29
outliers	1
mean (n)	4.0790
st.dev. (n)	0.09934
R(calc.)	0.2782
R(D2163:96)	0.4696

Compare R(EN27941(liq)) = 1.0216

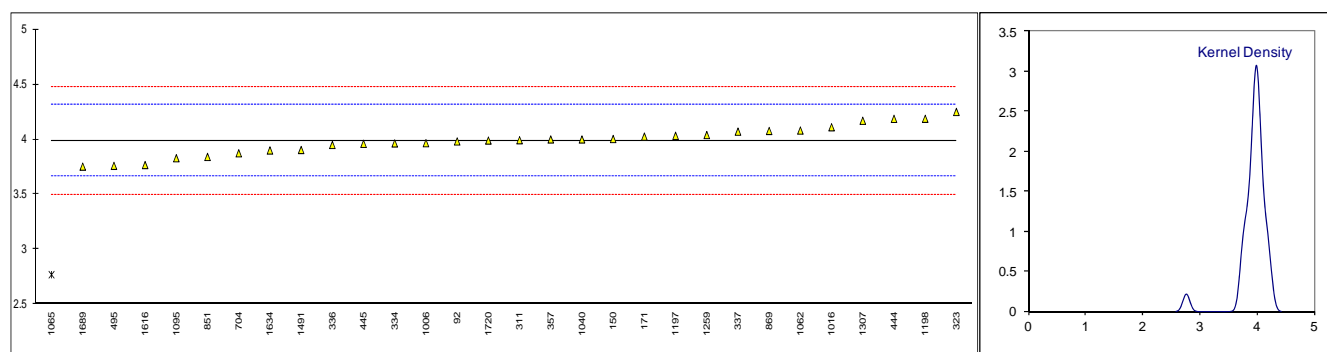## Determination of cis-2-Butene on sample #13074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	3.982		-0.04	
150	D2163	4.004		0.09	
171	D2163	4.026		0.23	
311	INH-407	3.993	C	0.03	First reported 4.919
323	D2163	4.25		1.59	
334	EN27941	3.964		-0.15	
336	EN27941	3.95		-0.24	
337	EN27941	4.071		0.50	
357	D2163	4.000		0.07	
444	IP405	4.187		1.21	
445	IP405/D2163	3.96		-0.17	
495	D2163	3.76		-1.39	
704	D2163	3.874		-0.70	
706		-----		-----	
851	D2163	3.841		-0.90	
869	D2163	4.077		0.54	
912		-----		-----	
1006	D2163	3.966		-0.14	
1016	ISO7941	4.111		0.75	
1040	DIN51619	4.00		0.07	
1062	D2163	4.0808		0.56	
1065	D2163	2.77	G(0.01)	-7.43	
1095	EN27941	3.829		-0.97	
1197	D2163	4.033		0.27	
1198	D2163	4.188		1.22	
1257		-----		-----	
1259	EN27941	4.040		0.31	
1307	INH-LHA	4.170		1.11	
1491	ISO7941	3.904		-0.52	
1616	D2163	3.767		-1.35	
1634	ISO7941	3.90		-0.54	
1689	INH-230	3.753		-1.44	
1720	D2163	3.990		0.01	

normality	OK	
n	29	
outliers	1	
mean (n)	3.9886	
st.dev. (n)	0.12827	
R(calc.)	0.3592	
R(D2163:96)	0.4592	Compare R(EN27941(liq)) = 1.0216

Determination of iso-Pentane on sample #13074; results in %mol/mol

lab	method	value	mark	z(targ)	remarks
92	D2163	0.585		-0.45	
150	D2163	0.602		0.25	
171	D2163	0.582		-0.57	
311	INH-407	0.607		0.45	
323	D2163	0.62		0.98	
334	EN27941	0.578		-0.73	
336	EN27941	0.57		-1.06	
337	EN27941	0.622		1.06	
357	D2163	0.590	C	-0.24	First reported 0.935
444	IP405	0.630		1.39	
445	IP405/D2163	0.54		-2.28	
495	D2163	0.50		-3.92	
704	D2163	0.533		-2.57	
706		-----		-----	
851	D2163	0.610		0.57	
869	D2163	0.647		2.08	
912		-----		-----	
1006	D2163	0.591		-0.20	
1016	ISO7941	0.618		0.90	
1040	DIN51619	0.60		0.17	
1062	D2163	0.6256		1.21	
1065	D2163	0.18	G(0.01)	-16.98	
1095	EN27941	0.527		-2.81	
1197	D2163	0.644		1.96	
1198	D2163	0.658		2.53	
1257		-----		-----	
1259	EN27941	0.631		1.43	
1307	INH-LHA	0.555		-1.67	
1491	ISO7941	0.529		-2.73	
1616	D2163	0.638		1.72	
1634	ISO7941	0.62		0.98	
1689	INH-230	0.421	G(0.01)	-7.14	
1720	D2163	0.634		1.55	

normality	OK	
n	28	
outliers	2	
mean (n)	0.5960	
st.dev. (n)	0.04151	
R(calc.)	0.1162	
R(D2163:96)	0.0686	Compare R(EN27941(liq)) = 0.7944
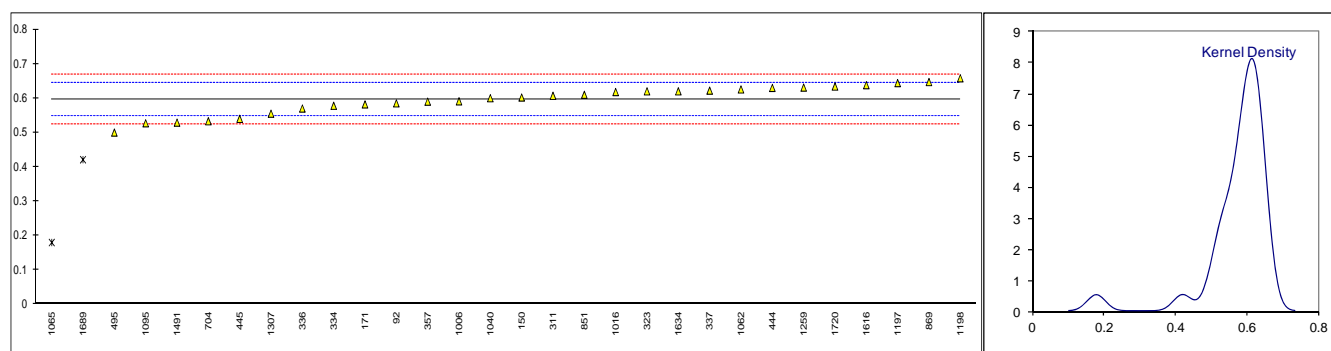
Determination of Molar Mass on sample #13074; results in g/mol

lab	method	value	mark	z(targ)	remarks
92		-----		-----	
150		-----		-----	
171	D2598	57.5003	E	-----	Calculated by iis from the reported test results: 57.322
311	INH- 407	57.4		-----	
323	D3588	57.36		-----	
334		-----		-----	
336		-----		-----	
337		-----		-----	
357	ISO8973	57.397		-----	
444		-----		-----	
445	IP432	57.442		-----	
495	ISO8973	57.48	E	-----	Calculated by iis from the reported test results: 57.328
704	D2163/D2421	57.3441		-----	
706		-----		-----	
851	D2598	57.38		-----	
869	Calc.	57.41		-----	
912		-----		-----	
1006		-----		-----	
1016		-----		-----	
1040		-----		-----	
1062		-----		-----	
1065	D3588	56.6	G(0.01)	-----	
1095	D2421	57.52		-----	
1197		-----		-----	
1198		-----		-----	
1257		-----		-----	
1259	ISO8973	57.40		-----	
1307		-----		-----	
1491	ISO8973	57.34		-----	
1616	D2421	57.474	E	-----	Calculated by iis from the reported test results: 57.072
1634		-----		-----	
1689		-----		-----	
1720		-----		-----	

				Calculated by iis from all reported test results
normality	OK			OK
n	13			25
outliers	1			3
mean (n)	57.419			57.386
st.dev. (n)	0.0593			0.02990
R(calc.)	0.166			0.0837
R(lit)	unknown			unknown
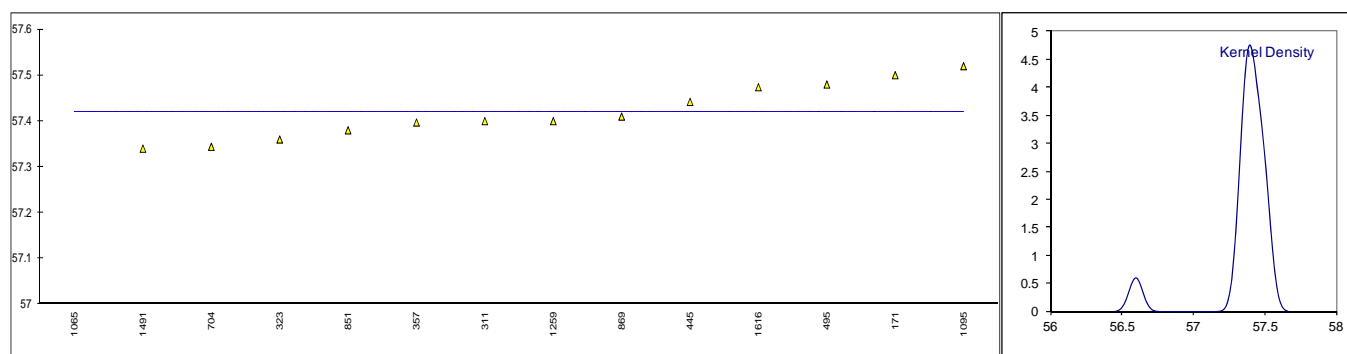
Determination of Relative Density @ 60F on sample #13074; unitless results

lab	method	value	mark	z(targ)	remarks
92	D2598	0.5703		-----	
150	D2598	0.5708	C	-----	First reported 0.56915
171	D2598	0.57033		-----	
311	INH- 407	0.570		-----	
323		0.5706		-----	
334		-----		-----	
336		-----		-----	
337		-----		-----	
357	D2598	0.5708		-----	
444		-----		-----	
445	IP432	0.5703	C	-----	First reported 570.3 (unit error)
495	D2598	0.57052	C	-----	
704	D2598	0.5699		-----	
706		-----		-----	
851	D2598	0.5703		-----	
869	D2158	0.5703		-----	
912		-----		-----	
1006	D2598	0.5703		-----	
1016		-----		-----	
1040		-----		-----	
1062		-----		-----	
1065	D3588	1.956	G(0.01)	-----	
1095		-----		-----	
1197		-----		-----	
1198		-----		-----	
1257		-----		-----	
1259	ISO8973	0.57		-----	
1307		-----		-----	
1491		-----		-----	
1616	D2421	0.5701		-----	
1634	ISO8973	0.5703		-----	
1689	GB/T12576	0.5701		-----	
1720		-----		-----	
					Calculated by iis from all reported test results
	normality	not OK			OK
	n	16			26
	outliers	1			2
	mean (n)	0.57031			0.57083
	st.dev. (n)	0.000266			0.000228
	R(calc.)	0.00074			0.00064
	R(lit)	unknown			unknown
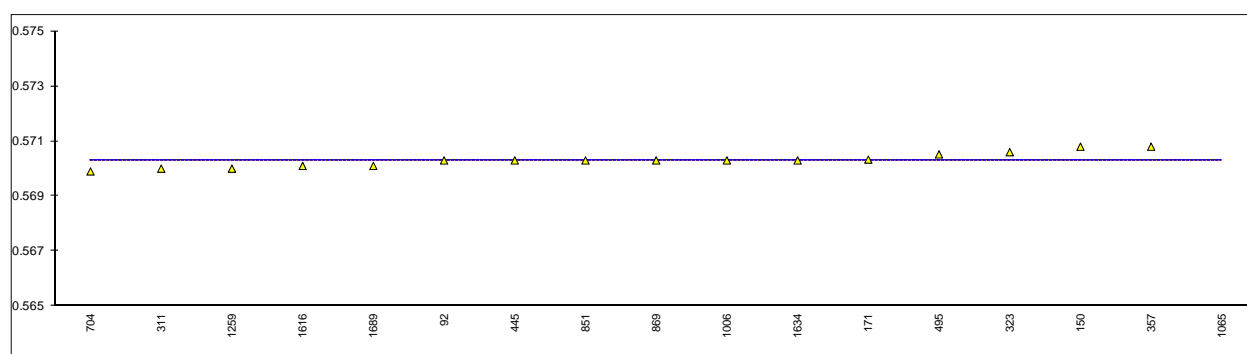
Determination of Abs. Vapour Pressure on sample #13074; results in psi

lab	method	value	mark	z(targ)	remarks
92		-----		-----	
150	D2598	72.9428	C,E	-----	First reported 73.0053; Calc by iis from the reported test results: 73.61
171	D2598	72.7691	E	-----	Calculated by iis from the reported test results: 74.02
311	ISO8973	73	E	-----	Calculated by iis from the reported test results: 73.55
323		-----		-----	
334		-----		-----	
336		-----		-----	
337		-----		-----	
357	ISO8973	73.66		-----	
444		-----		-----	
445		-----		-----	
495	D2598	73.04	E	-----	Calculated by iis from the reported test results: 74.42
704	ISO8973	74.12		-----	
706		-----		-----	
851	D2598	72	E	-----	Calculated by iis from the reported test results: 73.72
869	D2158	72	E	-----	Calculated by iis from the reported test results: 73.50
912		-----		-----	
1006	ISO8973	73.40		-----	
1016		-----		-----	
1040		-----		-----	
1062		-----		-----	
1065	IP432	81.22	ex	-----	Result exclude, see §4.1
1095	ISO8973	71.939		-----	
1197		-----		-----	
1198		-----		-----	
1257		-----		-----	
1259	ISO8973	77.4165	C,E	-----	First reported 62.66: Calc by iis from the reported test results: 73.54
1307		-----		-----	
1491	ISO8973	74.05		-----	
1616	in house	71.951	E	-----	Calculated by iis from the reported test results: 72.81
1634	ISO8973	73.23		-----	
1689	GB/T12576	61.4	G(0.01)	-----	
1720		-----		-----	

Calculated by iis from all reported test results

normality	OK		OK	
n	14		27	
outliers	1		1	
mean (n)	73.25		73.54	
st.dev. (n)	1.412		0.358	
R(calc.)	3.95		1.00	
R(lit)	unknown		unknown	
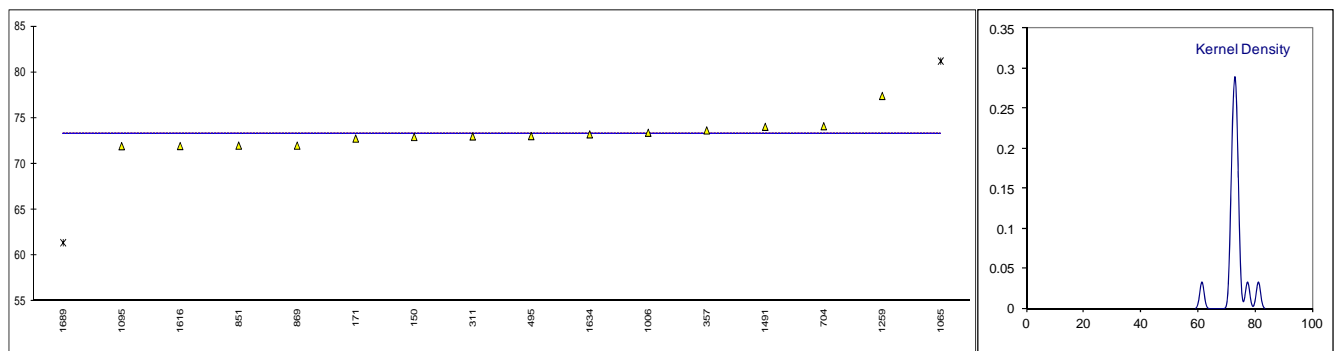
Determination of Rel. Vapour Pressure on sample #13074; results in psi

lab	method	value	mark	z(targ)	remarks
92		-----		-----	
150	D2598	58.645	C	-----	First reported 58.653
171	D2598	58.465	E	-----	Calculated by iis from the reported test results: 59.32
311	ISO8973	59		-----	
323		58.02	E	-----	Calculated by iis from the reported test results: 58.86
334		-----		-----	
336		-----		-----	
337		-----		-----	
357	ISO8973	58.96		-----	
444		-----		-----	
445	IP432	58		-----	
495	D2598	58.35	E	-----	Calculated by iis from the reported test results: 59.72
704	ISO8973	59.42		-----	
706		-----		-----	
851	D2598	58	E	-----	Calculated by iis from the reported test results: 59.02
869	D2158	58	E	-----	Calculated by iis from the reported test results: 58.80
912		-----		-----	
1006	ISO8973	58.70		-----	
1016		-----		-----	
1040		-----		-----	
1062		-----		-----	
1065		-----		-----	
1095	ISO8973	57.246		-----	
1197		-----		-----	
1198		-----		-----	
1257		-----		-----	
1259	ISO8973	62.7205	C,G(0.01),E	-----	First reported 47.96; Calc by iis from the reported test results: 58.85
1307		-----		-----	
1491		-----		-----	
1616	in house	57.261	E	-----	Calculated by iis from the reported test results: 58.12
1634	ISO8973	59.34		-----	
1689		-----		-----	
1720		-----		-----	
					Calculated by iis from all reported test results
	normality	OK			OK
	n	14			27
	outliers	1			1
	mean (n)	58.39			58.84
	st.dev. (n)	0.680			0.358
	R(calc.)	1.90			1.00
	R(lit)	unknown			unknown
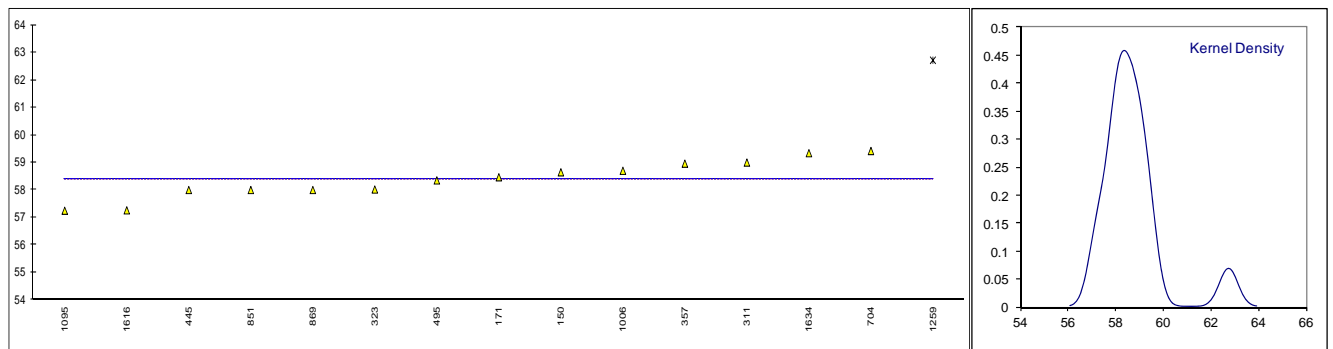
APPENDIX 2

Additional details

	Sample Volume	Type of vaporizer	Remarks
92	--	--	No
150	--	--	--
171	--	--	Ethane 0.0004 %M/M, Heptanes & Heavier 0.0275 %M/M
311	52 g	LSV	No
323	--	--	--
334	--	--	No
336	--	--	No
337	--	--	--
357	--	--	n-Pentane 0.003 mol%
444	--	not vaporized, liquid injection	n-Pentane 0.0020 %M/M
445	--	--	n-Pentane 0.01 %M/M
495	51	selfmade	No
704	0.0005	SPL	n-Pentane 0.002 %M/M
706	--	--	--
851	20	--	No
869	70	--	n-Pentane 0.002 %mol/mol
912	--	--	--
1006	--	--	No
1016	--	LSV	No
1040	--	--	No
1062	--	--	C2+ 0.001 %mol, n-C5+ 0.002 %mol, Acetylene 0.005 %mol
1065	1	--	No
1095	0.00025	--	Hexane and Heavier 0.009 %mol/mol
1197	--	--	--
1198	--	--	--
1257	--	--	--
1259	--	--	No
1307	--	--	C2+ 6 ppmmol, nC5+ 19 ppm mol, 2,2-diMeC3+ 1 ppm mol
1491	--	--	--
1616	--	--	No
1634	--	--	--
1689	245.6	60°C constant temperature bath	No
1720	--	--	No

APPENDIX 3

Number of participants per country

3 labs in BELGIUM

1 lab in CANADA

1 lab in CROATIA

1 lab in FINLAND

3 labs in FRANCE

2 labs in GERMANY

1 lab in HONG KONG

1 lab in INDIA

1 lab in ISRAEL

2 labs in MALAYSIA

2 labs in P.R. of CHINA

3 labs in PORTUGAL

1 lab in QATAR

1 lab in SUDAN

1 lab in TAIWAN R.O.C.

2 labs in THE NETHERLANDS

1 lab in U.A.E.

2 labs in U.S.A.

2 labs in UKRAINE

2 labs in UNITED KINGDOM

APPENDIX 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 Organization, Statistics and Evaluation, January 2010
2   prNEN 12766-2:2000.
3   ASTM E178-02
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5   ISO 5725-86
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7   M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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9   IP 367/84
10   DIN 38402 T41/42
11   P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
12   J.N. Miller, Analyst, 118, 455, (1993)
13   Analytical Methods Committee Technical Brief, No4 January 2001
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15   ISO17043
16   EN27941
17   ASTM D2163
18   ASTM D2421