Results of Proficiency Test LPG Composition & Sulphur October 2018

Organised by: Institute for Interlaboratory Studies Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2009, the Institute for Interlaboratory Studies organizes a proficiency test for the analysis of Liquefied Propane every year. It was decided to continue the interlaboratory study for Liquefied Propane during the annual program 2018/2019. At the request of several participants, the Institute of Interlaboratory Studies decided to organise an interlaboratory study for Sulphur (total) in LPG in the 2018/2019 PT program for the second time. Because iis has limited gas-handling facilities in place to prepare gas samples, a cooperation with EffecTech (Uttoxeter, United Kingdom) was set up for the Liquefied Propane PT (iis18S03P) and a co-operation with Praxair NV (Belgium) was set up for the Sulphur in LPG PT (iis18S03S). Both EffecTech and Praxair are fully equipped and have experience in the preparation of gas mixtures.

In the interlaboratory studies for Liquefied Propane 48 laboratories in 23 different countries and for Sulphur (total) in LPG 19 laboratories in 11 different countries registered for participation. In this report, the results of the 2018 proficiency tests Liquefied Propane and Sulphur (total) in LPG are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT).

To optimise the costs for the participants for the Liquefied Propane PT (iis18S03P), it was decided to prepare one Liquefied Propane mixture for composition. The mixture was divided over a batch of 54 cylinders-(1L cylinder with dip tube device). Each cylinder, filled with approx. 250 grams of liquefied propane mixture, was labelled #18210 and uniquely coded. For the Sulphur in LPG PT (iis18S03S) it was decided to use a batch of 20 cylinders, filled with approximately 1500 grams of LPG, each spiked with Dimethyl Sulfide (DMS). Each cylinder (5L cylinder with dip tube device), was labelled #18211 and uniquely coded. The limited cylinder sizes (1L and 5L) are chosen to optimise sample stability, cylinder costs, transport and handling costs.

The preparation and testing of the sample cylinders was subcontracted to ISO 17025 accredited laboratories. Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires. EffecTech is accredited in conformance with ISO/IEC17043:2010 by UKAS (no. 4719), ISO guide 35:2006 and ISO 17025:2005. Praxair is accredited in conformance with ISO 9001-2008, ISO 14001-2004, ISO 17025-No 159 Cal and ISO TS 16949.

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 June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

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

LIQUEFIED PROPANE #18210

One batch of 54 one litre cylinders with artificial Liquefied Propane mixture was prepared and tested for homogeneity by EffecTech (Uttoxeter, United Kingdom) in conformance with ISO guide 35:2006 and ISO 17025:2005 (job 18/1030, September 2018). Each cylinder was labelled #18210 and uniquely coded. Every cylinder in the batch was analysed using 5 replicate measurements. The within bottle and between bottle variations were assessed in accordance with ISO Guide 35:2006 (Annex A.1). This procedure showed that the between bottle variations were 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 corresponding reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

Component	r(observed) in %mol/mol	0.3 * R(D2163:14e1) in %mol/mol
Ethane	0.0019	0.0447
Propane	0.0235	1.2560
Propene	0.0032	0.0603
Iso-Butane	0.0068	0.0627
n-Butane	0.0118	0.0601
1-Butene	0.0014	0.0197
iso-Butene	0.0014	0.0188
n-Pentane	0.0043	0.0242

Table 1: homogeneity test results of samples #18210

Each calculated repeatability is less than 0.3 times the corresponding reproducibility of the reference method ASTM D2163:14e1. Therefore, homogeneity of the subsamples #18210 was assumed.

Sulphur

In this proficiency test, one batch of twenty 5L cylinders with artificial LPG mixture with Dimethylsulfide in Propane/n-Butane was prepared and tested for homogeneity by Praxair NV (Belgium) in conformance with ISO 9001-2008, ISO 14001-2004, ISO 17025-No 159 Cal and ISO TS 16949 in September 2018. Each cylinder was labelled #18211 and uniquely coded.

	Sulphur in mg/kg		Sulphur in mg/kg
Sample #18211-1	47.8	Sample #18211-11	49.3
Sample #18211-2	49.6	Sample #18211-12	49.5
Sample #18211-3	49.3	Sample #18211-13	49.1
Sample #18211-4	49.7	Sample #18211-14	49.3
Sample #18211-5	49.4	Sample #18211-15	49.4
Sample #18211-6	49.7	Sample #18211-16	48.7
Sample #18211-7	49.6	Sample #18211-17	49.3
Sample #18211-8	48.7	Sample #18211-18	48.9
Sample #18211-9	49.3	Sample #18211-19	49.5
Sample #18211-10	48.9	Sample #18211-20	49.0

Table 2: homogeneity test results of subsamples #18211

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference test method and in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Sulphur in mg/kg
r (observed)	1.3
reference test method	ASTM D6667:14
0.3 * R (ref. test method)	4.6

Table 3: evaluation of the repeatability of subsamples #18211

The calculated repeatability is less than 0.3 times the corresponding reproducibility of the reference test method ASTM D6667:14. Therefore, homogeneity of the subsamples #18211 was assumed.

Depending on their registration to each of the participating laboratories one 1L cylinder of Liquefied Propane labelled #18210 and/or one 5L cylinder of Sulphur in LPG labelled #18211 was sent on October 3, 2018. Per cylinder one SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The shelf life time of the prepared gas cylinders is sufficient for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine on the Liquefied Propane sample #18210 the composition: Ethane, Propane, Propene, iso-Butane, n-Butane, 1-Butene, iso-Butene, n-Pentane and to calculate several physical parameters from the composition: Molar Mass, Relative Density at 60F, Absolute and Relative Vapor pressure at 100F, Absolute and Relative Vapor pressure at 40°C, MON, Ideal Gross Heating Value and Ideal Net Heating Value at 14.696 psia and 60F.

On the LPG sample #18211 the total Sulphur content was requested.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

Shortly after the deadline, the available test results were screened for suspect data. A test 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 reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization 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 June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test 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, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and 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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on 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 reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

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 reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 whether the reported test result is fit-for-use.

The z-scores were calculated according to:

```
z_{\text{(target)}} = \text{(test result - average of PT)} / \text{target standard deviation}
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The $z_{\text{(target)}}$ scores are listed in the test result tables in appendix 1. Absolute values for z<2 are very common and absolute values for z>3 are very rare. 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 interlaboratory study, some problems were encountered with the dispatch of the samples.

For the Liquefied Propane PT (iis18S03P), not all laboratories did report all test results requested. Three participants reported test results after the deadline and four participants did not report any test result at all. In total 44 participants reported 495 numerical test results. Observed were 20 outlying test results, which is 4.0%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

For the Sulphur in LPG PT (iis18S03S), four participants did not report any result at all. In total 15 participants reported 15 numerical test results. Observed was 1 outlying test result, which is 6.7%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE AND PER TEST

In this section, the reported test results are discussed per sample and per test. The test methods, which were used by the various laboratories, were 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 3.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D3588) and an added designation for the year that the method was adopted or revised (e.g. D3588:98). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D3588:98(2017)). In the test results tables of appendix 1 only the method number and year of adoption or revision (e.g. D3588:98) will be used.

Because the majority of the participating laboratories used ASTM D2163 as test method for the determination of the Propane Composition, 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. In ASTM D2163 no reproducibilities of 1-butene and iso-butene are mentioned, the reproducibilities of n-butane were used to calculate the reproducibilities of these two components.

Liquefied Propane: sample #18210

Ethane: The determination of this component was not problematic. Three

statistical outliers were observed. However, the calculated reproducibility

after rejection of the statistical outliers is in agreement with the

requirements of ASTM D2163:14e1 and also with the reproducibility

requirements of EN27941:93 (identical to IP405 and ISO7941).

<u>Propane:</u> The determination of this component was not problematic. One

statistical outlier was observed. However, the calculated reproducibility

after rejection of the statistical outlier is in agreement with the

requirements of ASTM D2163:14e1 and also in agreement with the reproducibility requirements of EN27941:93 (identical to IP405 and

ISO7941).

Propene:

The determination of this component was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2163:14e1 and also with the reproducibility requirements of EN27941:93 (identical to IP405 and ISO7941).

iso-Butane:

The determination of this component may be problematic, depending on the test method used by the laboratory. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D2163:14e1, but is in agreement with the reproducibility requirements of EN27941:93 (identical to IP405 and ISO7941).

n-Butane:

The determination of this component may be problematic, depending on the test method used by the laboratory. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D2163:14e1, but is in in full agreement with the reproducibility requirements of EN27941:93 (identical to IP405 and ISO7941).

1-Butene:

The determination of this component was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2163:14e1 and also with the reproducibility requirements of EN27941:93 (identical to IP405 and ISO7941).

Iso-Butene:

The determination of this component was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D2163:14e1 and also with the reproducibility requirements of EN27941:93 (identical to IP405 and ISO7941).

n-Pentane:

The determination of this component may be problematic, depending on the test method used by the laboratory. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D2163:14e1, but is in agreement with the reproducibility requirements of EN27941:93 (identical to IP405 and ISO7941).

Molar Mass:

This calculated parameter may not be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in line with the calculated reproducibility using the published molar mass factors obtained from ASTM D2421:18 over all reported component concentrations (0.10 vs. 0.17 g/mol).

Rel. Density at 60F: This calculated parameter may not be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in line with the calculated reproducibility using the published relative density factors obtained from ASTM D2598:16 over all reported component concentrations (0.001 vs. 0.001).

Abs. VP at 100F:

As the reported results calculated via ISO8973 and ASTM D2598 are not identical, it was decided to evaluate the test results for both methods separately.

Results reported with ISO8973: This calculated parameter may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of statistical outlier is not in line with the calculated reproducibility using the published vapour pressure factors obtained from ISO8973:97 over all reported component concentrations (2.8 vs 1.5 psi).

Results reported with ASTM D2598: This calculated parameter may not be problematic. No statistical outliers were observed. The calculated reproducibility is more in line with the calculated reproducibility using the published vapour pressure factors obtained from ASTM D2598:16 over all reported component concentrations (2.2 vs. 1.6 psi). See also the discussion in paragraph 5.

Rel. VP at 100F:

As the reported results calculated via ISO8973 and ASTM D2598 are not identical, it was decided to evaluate the test results for both methods separately.

Results reported with ISO8973: This calculated parameter may not be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of statistical outlier is in line with the calculated reproducibility using the published vapour pressure factors obtained from ISO8973:97 over all reported component concentrations (2.1 vs 1.5 psi).

Results reported with ASTM D2598: This calculated parameter may not be problematic. No statistical outliers were observed. The calculated reproducibility is also in line with the calculated reproducibility using the published vapour pressure factors obtained from ASTM D2598:16 over all reported component concentrations (2.0 vs. 1.6 psi). See also the discussion in paragraph 5.

Abs. VP at 40°C:

This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in line with the calculated reproducibility using the published vapour pressure factors obtained from ISO8973:97 over all reported component concentrations (8.6 vs. 10.4 psi).

Rel. VP at 40°C:

This determination may be problematic. One statistical outlier was observed and two other test results were excluded. The calculated reproducibility after rejection of the suspect data is in line with the calculated reproducibility using the published vapour pressure factors obtained from ISO8973:97 over all reported component concentrations (10.8 vs. 10.4 psi).

MON:

As the reported results calculated via EN589-A1 and ASTM D2598 are not identical, it was decided to evaluate the test results for both methods separately.

Results reported with EN589-A1: This calculated parameter may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of statistical outlier is not in line with the calculated reproducibility using the published vapour pressure factors obtained from EN589:08-A1:12 over all reported component concentrations (0.45 vs 0.05).

For D2598 only three test results were reported, so no conclusions could be drawn.

See also the discussion in paragraph 5.

Ideal Gross Heating Value at 14.696 psia / 60F:

All laboratories reported to have calculated according to ASTM D3588. This calculated parameter may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is less in line with the calculated reproducibility using the published Ideal Gross Heating Values obtained from EN3588:98(2017) over all reported component concentrations (12 vs 8). This may be caused by the small number of reported rest results.

Ideal Net Heating Value at 14.696 psia / 60F:

All laboratories reported to have calculated according to ASTM D3588. This calculated parameter may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is less in line with the calculated reproducibility using the published Ideal Net Heating Values obtained from EN3588:98(2017) over all reported component concentrations (11 *vs* 8). This may be caused by the small number of reported rest results.

Sulphur in LPG: sample #18211

Sulphur, total:

The determination of this component was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D6667:14.

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, ISO standards) are compared in the next tables.

Component	unit	n	average	2.8 * sd	R(D2163:14 ^e 1) in %mol	R(EN27941) liqinj. in %mol
Ethane	%mol/mol	41	0.279	0.052	0.140	0.299
Propane	%mol/mol	43	93.82	0.79	4.18	1.02
Propene	%mol/mol	44	0.583	0.076	0.200	0.213
iso-Butane	%mol/mol	43	1.86	0.25	0.21	0.39
n-Butane	%mol/mol	43	2.36	0.40	0.20	0.39
1-Butene	%mol/mol	44	0.197	0.039	0.066	0.160
Iso-Butene	%mol/mol	43	0.181	0.041	0.064	0.160
n-Pentane	%mol/mol	41	0.727	0.146	0.082	0.311

Table 4: reproducibilities of composition tests on sample #18210

Parameter	unit	n	average	2.8 * sd	R (iis calc.)	n (all calc.)
Molar Mass	g/mol	19	44.89	0.10	0.17	44
Rel. Density at 60F		24	0.512	0.001	0.001	43
Abs. VP at 100F-ISO	psi	7	185.2	2.8	1.5	44
Abs. VP at 100F-ASTM	psi	4	182.2	2.2	1.6	44
Rel. VP at 100F-ISO	psi	7	170.6	2.1	1.5	44
Rel. VP at 100F-ASTM	psi	8	167.2	2.0	1.6	44
Abs. VP at 40°C	kPa	16	1315	8.6	10.4	44
Rel. VP at 40°C	kPa	17	1214	10.9	10.4	44
MON – EN589		11	95.17	0.45	0.05	40
MON – D2598		3	96.15	(1.23)*	0.14	43
Ideal Gross HV	kJ/mol	5	2260	12	8	44
Net Gross HV	kJ/mol	5	2080	11	8	44

Table 5: reproducibilities of calculated parameters on sample #18210

^{*)} results in brackets based on only three test results.

Component	unit	n	average	2.8 * sd	R(lit)
Sulphur, total	mg/kg	15	51.6	30.2	16.2

Table 6: reproducibility of test on sample #18211

Without further statistical calculations it can be concluded that for a large number of parameters there is a good compliance of the group of participating laboratories with the relevant reference test methods for the component determination. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2018 WITH PREVIOUS PTS

	Oct. 2018	Oct. 2017	Oct. 2016	Oct. 2015	Oct. 2014
Number of reporting labs	44	47	43	41	44
Number of test results reported	495	536	472	468	395
Statistical outliers	20	30	34	24	27
Percentage outliers	4.0%	5.6%	7.2%	5.1%	6.8%

Table 7: comparison with previous proficiency tests on Liquefied Propane (excluded Sulphur in LPG)

	Oct. 2018	Oct. 2017
Number of reporting labs	15	8
Number of test results reported	15	8
Statistical outliers	1	1
Percentage outliers	6.7%	12.5%

Table 8: comparison with previous proficiency tests on Sulphur in LPG only

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 the respective reference test methods. The conclusions are given the following tables:

Component	Oct. 2018	Oct. 2017	Oct. 2016	Oct. 2015	Oct. 2014
Ethane	++	++	++	++	-
Propane	++	++	++	++	+/-
Propene	++	++	++	+	+/-
iso-Butane	-	+/-	•	+	+/-
n-Butane		-	-	-	-
1-Butene	+	++	++	++	
Iso-Butene	+	++	+	++	
n-Pentane	-	-	-	-	

Table 9: comparison determinations on Liquefied Propane against the requirements of the reference standards

Component	Oct. 2018	Oct. 2017
Sulphur, total	-	+

Table 10: comparison determinations on Sulphur in LPG against the requirements of the reference standard

The performance of the determinations against the requirements of the respective reference test methods is listed in the above tables. The following performance categories were used:

- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- group performed worse than the reference test method
- -- : group performed much worse than the reference test method

5 DISCUSSION

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

The consensus values as determined in this PT are compared with the average values from the homogeneity testing by EffecTech, United Kingdom in the following table.

Parameter	Average values by EffecTech in %mol/mol	Consensus values from participants in %mol/mol	Differences in %mol/mol	calc. z-score
Ethane	0.306	0.279	0.027	+0.54
Propane	93.92	93.82	0.100	-0.07
Propene	0.589	0.583	0.006	+0.08
iso-Butane	1.806	1.86	-0.054	-0.71
n-Butane	2.303	2.36	-0.057	-0.79
1-Butene	0.194	0.197	-0.003	-0.13
Iso-Butene	0.175	0.181	-0.006	-0.26
n-Pentane	0.704	0.727	-0.023	-0.79

Table 11: comparison of consensus values with values determined by EffecTech

From this comparison it is clear that <u>all</u> consensus values as determined in this PT are in line with the values as determined by EffecTech during the preparation of the cylinders.

In principle no additional variation 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 test 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, Vapor Pressure, Motor Octane Number and Heating Value several standardized test methods are available, e.g. ASTM D2421 for the interconversion of the units to gas-volume, liquid-volume or mass basis. Also, different test methods for the calculation of the Vapor Pressure do exist. In ISO 8973 (identical to IP432) the Vapor Pressure is calculated from the mole fraction per component and a Vapor Pressure factor of that component (given for all components). In ASTM D2598 the Vapor Pressure is calculated from the liquid volume percentage per component and a Vapor Pressure factor of that component (given for only several components). The selection of the tables to be used for the calculations may cause additional uncertainty.

It is remarkable to see that the results for Vapour Pressure from the ASTM D2598 calculation are significantly lower than the results from the ISO8973/IP432 calculation.

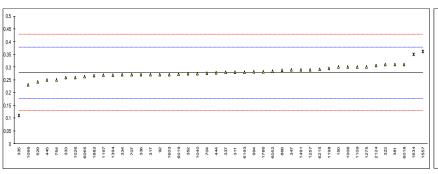
The observed difference is caused by a difference in the VP factor of Ethane. ASTM (Subcommittee D02.H) commented (see also Appendix 3, literature: 20):

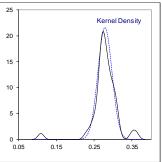
"The vapor pressure of ethane in D2598 was revised a few times prior to 2002. The current value, 611 psi, has remained the same for the last ten years. The revision of ethane was done because components in LPG blends do not necessarily behave as ideal gases. In particular, properties of ethane and ethylene appear to differ from ideality. Factors for these two components have been modified from 'ideal gas' values to make the calculated vapor pressure results more closely approximate actual measured vapor pressures of LPG blends. (i.e. D1267). Chapter 2 of Fuels and Lubricants Handbook (George Totten, © 2003), states that calculated vapor pressure were found to be biased high relative to experimental vapor pressure measured by D1267 for high ethane samples in earlier versions of D2598'.

APPENDIX 1

Determination of Ethane on sample #18210; results in %mol/mol

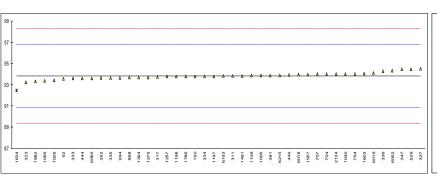
lab	method	value	mark	z(targ)	remarks
92		0.271		-0.15	
150	D2163	0.30	С	0.43	first reported: 0.442
158					
171	D2162	0.20		0.02	
311 317	D2163 D2163	0.28 0.27		0.03 -0.17	
323	D2163	0.27		0.63	
333	D2163	0.26		-0.37	
334	D2163	0.27		-0.17	
335	D2163	0.11	R(0.01)	-3.36	
336	EN27941	0.27	(/	-0.17	
337	D2163	0.28		0.03	
347	D2163	0.289		0.21	
352	EN27941	0.2735		-0.10	
381	EN27941	0.31		0.63	
444	D2163	0.278		-0.01	
445 511	D2163	0.25		-0.57 	
529		0.242		-0.73	
704	EN27941	0.276		-0.75	
707	EN27941	0.270		-0.17	
754	D2163	0.25		-0.57	
868	D2163	0.287		0.17	
994	D2163	0.2816		0.06	
1006	D2163	0.300		0.43	
1026	ISO7941	0.26		-0.37	('
1040 1095	D2163 ISO7941	0.274 0.23	С	-0.09 -0.97	first reported: 0.183%M/M
11093	IP405	0.23		0.43	
1197	D2163	0.268		-0.21	
1198	D2163	0.295		0.33	
1257	D2163	0.2901		0.23	
1275	EN27941	0.301		0.45	
1394		0.269		-0.19	
1491	ISO7941	0.290		0.23	
1557	EN27941	0.361	R(0.05)	1.64	
1603	In house	0.2714	D(0.05)	-0.14	
1634 1786	ISO7941 D2163	0.35 0.282	R(0.05)	1.42 0.07	
1882	EN27941	0.2668		-0.24	
2124	LINETOTI	0.307		0.57	
6018	EN27941	0.310		0.63	
6019	EN27941	0.272		-0.13	
6052	D2163	0.2842		0.11	
6065	D2163	0.2632		-0.31	
6193	D2163	0.28		0.03	
6215	D2163	0.2909970)4	0.25	
7014					
	normality	OK			
	n	41			
	outliers	3			
	mean (n)	0.2786			
	st.dev. (n)	0.01849			
	R(calc.)	0.0518			
	st.dev.(D2163:14e1)	0.05011			Compare P/EN/27041:02/lig/\ = 0.2096
	R(D2163:14e1)	0.1403			Compare R(EN27941:93(liq)) = 0.2986

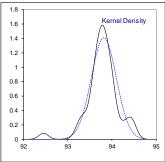




Determination of Propane on sample #18210; results in %mol/mol

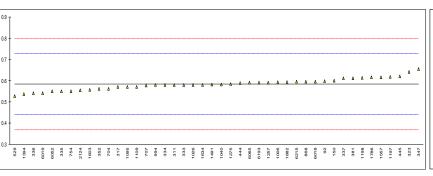
lab	method	value	mark	z(targ)	remarks
92		93.537		-0.19	
150	D2163	93.78	С	-0.03	first reported: 91.932
158					
171	Ba. 100				
311	D2163	93.80		-0.02	
317	D2163	93.71		-0.08	
323	D2163	93.23		-0.40	
333	D2163	93.56		-0.18	
334	D2163	93.78		-0.03	
335 336	D2163 EN27941	93.63 94.24		-0.13 0.28	
337	D2163	94.24		0.26	
347	D2163	94.47		0.43	
352	EN27941	93.614		-0.14	
381	EN27941	93.86		0.03	
444	D2163	93.583		-0.16	
445	D2163	93.89		0.05	
511					
529		94.465		0.43	
704	EN27941	93.980		0.11	
707	EN27941	93.972		0.10	
754	D2163	94.01		0.13	
868	D2163	93.668		-0.10	
994	D2163	93.6472		-0.12	
1006	D2163	93.850		0.02	
1026	ISO7941	93.38	_	-0.30	4
1040	D2163	94.009	С	0.12	first reported: 92.068%M/M
1095	ISO7941	93.35		-0.32	
1109	IP405	93.84		0.01	
1197 1198	D2163	93.780		-0.03	
1257	D2163 D2163	93.768 93.7634		-0.04 -0.04	
1275	EN27941	93.691		-0.04	
1394	LINZISTI	93.675		-0.10	
1491	ISO7941	93.801		-0.01	
1557	EN27941	93.947		0.08	
1603	In house	94.0132		0.13	
1634	ISO7941	92.46	R(0.01)	-0.91	
1786	D2163	93.771	, ,	-0.03	
1882	EN27941	93.3175		-0.34	
2124		93.997		0.12	
6018	EN27941	93.924		0.07	
6019	EN27941	94.081		0.17	
6052	D2163	94.3084		0.33	
6065	D2163	93.6018		-0.15	
6193	D2163	93.79		-0.02	
6215	D2163	93.861795		0.03	
7014					
	normality	OK			
	n	43			
	outliers	1			
	mean (n)	93.8226			
	st.dev. (n)	0.28340			
	R(calc.)	0.7935			
	st.dev.(D2163:14e1)	1.49410			
	R(D2163:14e1)	4.1835			Compare R(EN27941:93(liq)) = 1.018

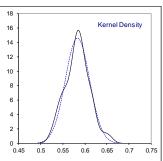




Determination of Propene on sample #18210; results in %mol/mol

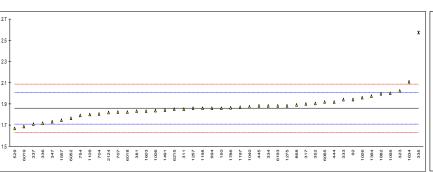
lab	method	value	mark	z(targ)	remarks
92		0.598		0.21	
150	D2163	0.60	С	0.23	first reported: 0.632
158					
171					
311	D2163	0.58		-0.05	
317	D2163	0.57		-0.19	
323	D2163	0.64		0.79	
333	D2163	0.58		-0.05	
334	D2163	0.58		-0.05	
335	D2163	0.55		-0.47	
336	EN27941	0.54		-0.61	
337 347	D2163	0.61		0.37	
347 352	D2163 EN27941	0.654		0.99	
381	EN27941	0.5602 0.61		-0.32 0.37	
444	D2163	0.588		0.07	
445	D2163	0.62		0.51	
511	D2103				
529		0.526		-0.80	
704	EN27941	0.562		-0.30	
707	EN27941	0.577		-0.09	
754	D2163	0.55		-0.47	
868	D2163	0.596		0.18	
994	D2163	0.5797		-0.05	
1006	D2163	0.592		0.12	
1026	ISO7941	0.58		-0.05	
1040	D2163	0.582	С	-0.02	first reported: 0.544%M/M
1095	ISO7941	0.57		-0.19	
1109	IP405	0.57		-0.19	
1197	D2163	0.618		0.49	
1198	D2163	0.613		0.42	
1257	D2163	0.5907		0.10	
1275	EN27941	0.584		0.01	
1394 1491	ISO7941	0.537		-0.65 -0.03	
1557	EN27941	0.581 0.616		0.46	
1603	In house	0.5577		-0.36	
1634	ISO7941	0.58		-0.05	
1786	D2163	0.615		0.44	
1882	EN27941	0.5930		0.14	
2124		0.555		-0.40	
6018	EN27941	0.596		0.18	
6019	EN27941	0.541		-0.59	
6052	D2163	0.5487		-0.49	
6065	D2163	0.5900		0.09	
6193	D2163	0.59		0.09	
6215	D2163	0.595792		0.17	
7014					
	normality	OK			
	n 	44			
	outliers	0			
	mean (n)	0.5833			
	st.dev. (n)	0.02730			
	R(calc.)	0.0764			
	st.dev.(D2163:14e1) R(D2163:14e1)	0.07132 0.1997			Compare R(EN27941:93(liq)) = 0.2133
	11(02100.1461)	0.1331			Outipare IN(LINZ/341.30(IIY)) - 0.2133

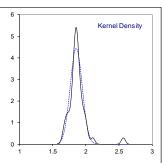




Determination of iso-Butane on sample #18210; results in %mol/mol

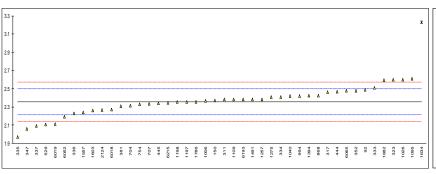
lab	method	value	mark	z(targ)	remarks
92		1.941		1.08	
150	D2163	1.86	С	0.00	first reported: 1.432
158					
171					
311	D2163	1.85		-0.13	
317	D2163	1.90		0.53	
323	D2163	2.02		2.12	
333	D2163	1.94		1.06	
334	D2163	1.88	D(0.04)	0.27	
335	D2163	2.57	R(0.01)	9.40	
336 337	EN27941 D2163	1.72 1.71		-1.85 -1.98	
347	D2163	1.733		-1.68	
352	EN27941	1.9065		0.62	
381	EN27941	1.83		-0.39	
444	D2163	1.920		0.80	
445	D2163	1.88		0.27	
511					
529		1.669		-2.53	
704	EN27941	1.804		-0.74	
707	EN27941	1.825		-0.46	
754	D2163	1.79		-0.92	
868	D2163	1.891		0.41	
994	D2163	1.8593		-0.01	
1006	D2163	1.835		-0.33	
1026	ISO7941	1.96	^	1.33	first man ant als O 44F0/N/N
1040	D2163	1.871	С	0.15	first reported: 2.415%M/M
1095 1109	ISO7941 IP405	2.00 1.80		1.86 -0.79	
1109	D2163	1.869		0.19	
1198	D2163	1.858		-0.02	
1257	D2163	1.8571		-0.03	
1275	EN27941	1.883		0.31	
1394		1.974		1.51	
1491	ISO7941	1.842		-0.23	
1557	EN27941	1.747		-1.49	
1603	In house	1.8328		-0.36	
1634	ISO7941	2.11		3.31	
1786	D2163	1.865		0.07	
1882	EN27941	1.9961		1.81	
2124	EN07044	1.816		-0.58	
6018	EN27941	1.825		-0.46	
6019	EN27941	1.685		-2.31	
6052 6065	D2163 D2163	1.7647 1.9199		-1.26 0.80	
6193	D2163 D2163	1.88		0.80	
6215	D2163	1.8488884		-0.14	
7014	52100			-0.14	
, , , ,					
	normality	OK			
	n	43			
	outliers	1			
	mean (n)	1.8597			
	st.dev. (n)	0.09025			
	R(calc.)	0.2527			
	st.dev.(D2163:14e1)	0.07553			- (- 1) - 1
	R(D2163:14e1)	0.2115			Compare R(EN27941:93(liq)) = 0.3861

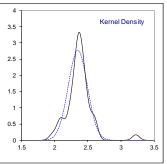




Determination of n-Butane on sample #18210; results in %mol/mol

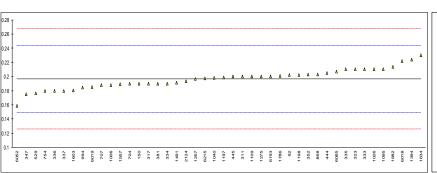
lab	method	value	mark	z(targ)	remarks
92		2.489		1.85	
150	D2163	2.37	С	0.21	first reported: 1.822
158					
171					
311	D2163	2.38		0.34	
317	D2163	2.46		1.45	
323	D2163	2.60		3.39	
333	D2163	2.51		2.14	
334	D2163	2.41		0.76	
335	D2163	1.97		-5.33	
336	EN27941	2.23		-1.73	
337	D2163	2.09		-3.67	
347	D2163	2.06		-4.08	
352	EN27941	2.4771		1.69	
381	EN27941	2.31		-0.62	
444	D2163	2.468		1.56	
445	D2163	2.34		-0.21	
511					
529		2.106		-3.45	
704	EN27941	2.316		-0.54	
707	EN27941	2.334		-0.29	
754	D2163	2.33		-0.35	
868	D2163	2.426		0.98	
994	D2163	2.4195		0.89	
1006	D2163	2.365		0.14	
1026	ISO7941	2.60	_	3.39	
1040	D2163	2.419	С	0.88	first reported: 3.122%M/M
1095	ISO7941	2.61		3.53	
1109	IP405	2.38		0.34	
1197	D2163	2.355		0.00	
1198	D2163	2.354		-0.02	
1257	D2163	2.3841		0.40	
1275	EN27941	2.407		0.72	
1394	1007044	2.423		0.94	
1491	ISO7941	2.381		0.36	
1557 1603	EN27941 In house	2.239 2.2620		-1.61 -1.29	
1634	ISO7941	3.23	R(0.01)	12.11	
1786	D2163	2.355	K(0.01)	0.00	
1882	EN27941	2.5957		3.33	
2124	LINCIUTI	2.267		-1.22	
6018	EN27941	2.272		-1.15	
6019	EN27941	2.112		-3.36	
6052	D2163	2.112		-2.28	
6065	D2163	2.4769		1.69	
6193	D2163	2.38		0.34	
6215	D2163	2.3473865		-0.11	
7014					
*··					
	normality	OK			
	n	43			
	outliers	1			
	mean (n)	2.3551			
	st.dev. (n)	0.14449			
	R(calc.)	0.4046			
	st.dev.(D2163:14e1)	0.07225			
	R(D2163:14e1)	0.2023			Compare R(EN27941:93(liq)) = 0.3861

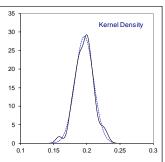




Determination of 1-Butene on sample #18210; results in %mol/mol

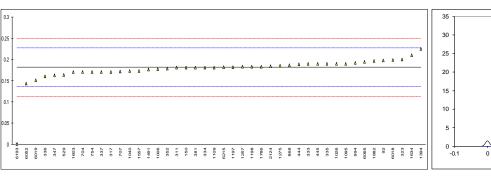
lab	method	value	mark	z(targ)	remarks
92		0.202		0.23	
150	D2163	0.19	С	-0.28	first reported: 0.155
158					
171	D0160	0.20		0.14	
311 317	D2163 D2163	0.20		0.14 -0.28	
323	D2163	0.19 0.21		0.57	
333	D2163	0.21		0.57	
334	D2163	0.19		-0.28	
335	D2163	0.21		0.57	
336	EN27941	0.18		-0.70	
337	D2163	0.18		-0.70	
347	D2163	0.175		-0.92	
352	EN27941	0.2027		0.26	
381	EN27941	0.19		-0.28	
444	D2163	0.205		0.35	
445	D2163	0.20		0.14	
511					
529	EN107044	0.176		-0.87	
704	EN27941	0.190		-0.28	
707 754	EN27941 D2163	0.188 0.18		-0.37 -0.70	
868	D2163	0.16		0.27	
994	D2163	0.203		-0.50	
1006	D2163	0.188		-0.37	
1026	ISO7941	0.21		0.57	
1040	D2163	0.198	С	0.06	first reported: 0.247%M/M
1095	ISO7941	0.21		0.57	·
1109	IP405	0.20		0.14	
1197	D2163	0.199		0.10	
1198	D2163	0.202		0.23	
1257	D2163	0.1967		0.00	
1275	EN27941	0.200		0.14	
1394 1491	ISO7941	0.224 0.191		1.16 -0.24	
1557	EN27941	0.191		-0.24	
1603	In house	0.1897		-0.52	
1634	ISO7941	0.23		1.41	
1786	D2163	0.201		0.18	
1882	EN27941	0.2136		0.72	
2124		0.193		-0.15	
6018	EN27941	0.222		1.07	
6019	EN27941	0.185		-0.49	
6052	D2163	0.1587		-1.60	
6065	D2163	0.2067		0.43	
6193	D2163	0.20		0.14	
6215	D2163	0.1970809		0.02	
7014					
	normality	OK			
	n	44			
	outliers	0			
	mean (n)	0.1966			
	st.dev. (n)	0.01385			
	R(calc.)	0.0388			
	st.dev.(D2163:14e1)	0.02364			- /
	R(D2163:14e1)	0.0662			Compare R(EN27941:93(liq)) = 0.1600

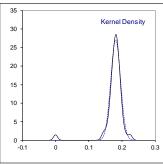




Determination of iso-Butene on sample #18210; results in %mol/mol

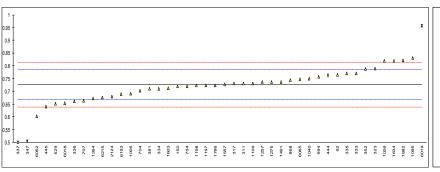
lab	method	value	mark	z(targ)	remarks
92		0.197		0.69	
150	D2163	0.18	С	-0.05	first reported: 0.142
158					
171					
311	D2163	0.18		-0.05	
317	D2163	0.17		-0.49	
323	D2163	0.20		0.82	
333	D2163	0.19		0.38	
334	D2163	0.18		-0.05	
335	D2163	0.19		0.38	
336	EN27941	0.16		-0.93	
337 347	D2163 D2163	0.17		-0.49 -0.84	
352	EN27941	0.162 0.1780		-0.64 -0.14	
381	EN27941	0.1760		-0.14	
444	D2163	0.188		0.30	
445	D2163	0.100		0.38	
511	D2100				
529		0.163		-0.80	
704	EN27941	0.170		-0.49	
707	EN27941	0.171		-0.45	
754	D2163	0.17		-0.49	
868	D2163	0.186		0.21	
994	D2163	0.1913		0.44	
1006	D2163	0.177		-0.19	
1026	ISO7941	0.19		0.38	
1040	D2163	0.172	С	-0.41	first reported: 0.215%M/M
1095	ISO7941	0.19		0.38	
1109	IP405	0.18		-0.05	
1197	D2163	0.182		0.03	
1198	D2163	0.183		0.08	
1257	D2163	0.1822		0.04	
1275	EN27941	0.185		0.17	
1394 1491	ISO7941	0.225 0.176		1.92 -0.23	
1557	EN27941	0.176		-0.23	
1603	In house	0.173		-0.50	
1634	ISO7941	0.1033		1.26	
1786	D2163	0.183		0.08	
1882	EN27941	0.1963		0.66	
2124		0.184		0.12	
6018	EN27941	0.199		0.78	
6019	EN27941	0.151		-1.33	
6052	D2163	0.1433		-1.66	
6065	D2163	0.1941		0.56	
6193	D2163	0.00	R(0.01)	-7.95	
6215	D2163	0.1811388		0.00	
7014					
	normality	suspect			
	n 	43			
	outliers	1			
	mean (n)	0.1812			
	st.dev. (n)	0.01472			
	R(calc.) st.dev.(D2163:14e1)	0.0412 0.02279			
	R(D2163:14e1)	0.02279			Compare R(EN27941:93(liq)) = 0.1600
	11(02100.1461)	0.0000			σοπράτο Ιλ(ΕΙΝΣΙ 371.30(IIq)) = 0.1000

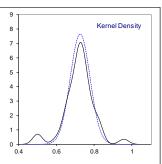




Determination of n-Pentane on sample #18210; results in %mol/mol

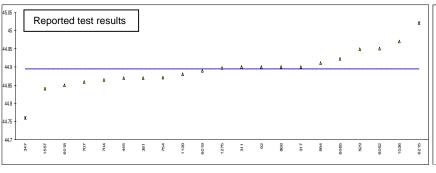
lab	method	value	mark	z(targ)	remarks
92		0.765		1.31	
150	D2163	0.72	С	-0.22	first reported: 0.443
158					
171	D0400	0.70		0.40	
311	D2163	0.73		0.12	
317 323	D2163	0.73 0.79		0.12	
333	D2163 D2163	0.79		2.17 1.49	
334	D2163	0.71		-0.57	
335	D2163	0.77		1.49	
336	EN27941	0.66		-2.28	
337	D2163	0.50	R(0.01)	-7.75	
347	D2163	0.504	R(0.01)	-7.61	
352	EN27941	0.7883		2.11	
381	EN27941	0.71		-0.57	
444	D2163	0.765		1.31	
445	D2163	0.64		-2.96	
511					
529	EN107044	0.652		-2.55	
704	EN27941	0.702		-0.84	
707 754	EN27941	0.662		-2.21	
754 868	D2163 D2163	0.72 0.744		-0.22 0.60	
994	D2163	0.7580		1.08	
1006	D2163	0.692		-1.18	
1026	ISO7941	0.82		3.20	
1040	D2163	0.750	С	0.80	first reported: 1.202%M/M
1095	ISO7941	0.83		3.54	•
1109	IP405	0.73		0.12	
1197	D2163	0.724		-0.09	
1198	D2163	0.723		-0.12	
1257	D2163	0.7357		0.31	
1275	EN27941	0.736		0.32	
1394	1007044	0.673		-1.83	
1491 1557	ISO7941 EN27941	0.737 0.727		0.36 0.01	
1603	In house	0.727		-0.49	
1634	ISO7941	0.82		3.20	
1786	D2163	0.724		-0.09	
1882	EN27941	0.8210		3.23	
2124		0.679		-1.63	
6018	EN27941	0.653		-2.52	
6019	EN27941	0.957	R(0.01)	7.88	
6052	D2163	0.6018		-4.27	
6065	D2163	0.7475		0.72	
6193	D2163	0.69		-1.25	
6215	D2163	0.6769479		-1.70	
7014					
	normality	OK			
	n	41			
	outliers	3			
	mean (n)	0.7266			
	st.dev. (n)	0.05207			
	R(calc.)	0.1458			
	st.dev.(D2163:14e1)	0.02923			
	R(D2163:14e1)	0.0818			Compare $R(EN27941:93(liq)) = 0.3111$

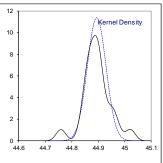




Determination of Molar Mass on sample #18210; results in g/mol

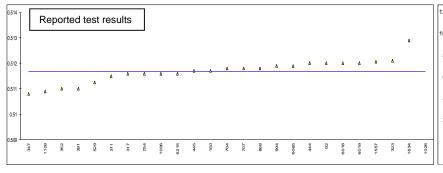
lab	method	value	mark	z(targ)	remarks
92	D2163	44.9			
150					
158					
171					
311	INH-407	44.90			
317	INH-001	44.9			
323					
333					
334					
335					
336					
337					
347	D2598	44.76	R(0.05)		
352			` ,		
381	ISO8973	44.87			
444					
445	D2163	44.869			
511					
529	D2421	44.9495			
704	D2421	44.8636			
707	D2421	44.8589			
754	D2421	44.871			
868	D2598	44.90			
994	D2598	44.91			
1006					
1026	ISO8973	44.97			
1040					
1095					
1109	ISO8973	44.88			
1197					
1198					
1257					
1275	EN589	44.897			
1394					
1491					
1557	ISO8973	44.84			
1603					
1634					
1786					
1882					
2124					
6018	ISO8973	44.85			
6019	ISO8973	44.89			
6052	D3588	44.95			
6065		44.923			
6193					
6215	D2598	45.0209	R(0.05)		
7014					
					Calc. by iis from ALL rep. composition results (acc. to D2421:18)
	normality	OK			suspect
	n	19			44
	outliers	2			0
	mean (n)	44.894			44.888
	st.dev. (n)	0.0351			0.0617
	R(calc.)	0.098			0.173
comp	R(iis17S03P)	0.085			0.082
•	•				
45.05 T					12
I Re	eported test results				V Accord Density

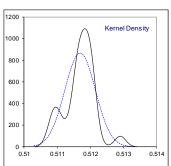




Determination of Relative Density at 60/60F on sample #18210; unitless results

lab	method	value	mark	z(targ)	remarks
92	D2598	0.512			
150	D2598	0.5117	С		first reported: 0.5102
158					
171					
311	INH-407	0.5115			
317	INH-001	0.5116			
323	D2598	0.5121			
333					
334					
335					
336					
337	D				
347	D2598	0.5108			
352	ISO8973	0.511			
381	D2598	0.511			
444	D2598	0.512			
445		0.5117			
511 529	D2424	0 F1126			
	D2421	0.51126			
704 707	D2598 D2598	0.5118 0.5118			
757 754	D2598	0.5116			
868	D2598	0.5118			
994	D2598	0.5119			
1006	D2598	0.5116			
1026	ISO8973	511.1	R(0.01), E		calculation error: iis calculated: 0.5122
1040	1000010		11(0.01), =		Salediation offer. No salediated. 5.5 122
1095					
1109	D2598	0.5109	E		calculation error: iis calculated 0.5117
1197					
1198					
1257					
1275					
1394					
1491					
1557	ISO8973	0.51206	E		calculation error: iis calculated 0.5114
1603					
1634	ISO8973	0.5129			
1786					
1882					
2124					
6018	ISO8973	0.512			
6019	ISO8973	0.512			
6052	Doron				
6065	D2598	0.5119	14/		first negative of E44 O leader3
6193	DOCOO	0.5440	W		first reported: 511.9 kg/m ³
6215	D2598	0.5116			
7014					Calc. by iis from ALL rep. composition results (acc. to D2598:16)
	normality	suspect			OK
	n	24			43
	outliers	1			1
	mean (n)	0.51169			0.51169
	st.dev. (n)	0.000462			0.000289
	R(calc.)	0.00129			0.00081
comp	_ 1 '	0.00101			0.00063
	, ,				

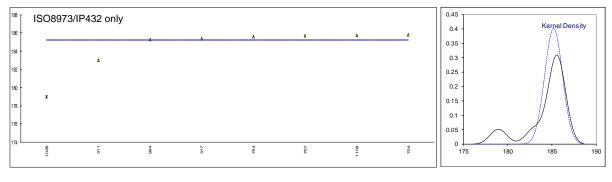


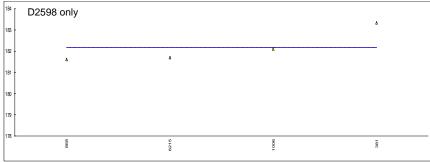


Determination of Absolute Vapor Pressure at 100F on sample #18210; results in psi

lab	method	value	mark	z(targ)	remarks
92					
150					
158					
171					
311	ISO8973	183	E		calculation error: iis calculated: 185.6 (ISO8973)
317	ISO8973	185.4			
323					
333					
334					
335					
336					
337					
347					
352 381	D2598	183.34	Е		calculation error: iis calculated: 182.0 (D2598)
444	D2390	103.54	_		calculation end. ils calculated. 102.0 (D2090)
445					
511					
529					
704	ISO8973	185.8			
707	ISO8973	185.7			
754	ISO8973	185.6			
868	D2598	181.6			
994	IP432	185.29			
1006	D2598	182.1			
1026	ISO8973	178.98	G(0.01),E		calculation error: iis calculated: 184.8 (ISO8973)
1040					
1095	10000=0				
1109	ISO8973	185.73			
1197					
1198 1257					
1275					
1394					
1491					
1557					
1603					
1634					
1786					
1882					
2124					
6018					
6019					
6052 6065					
6193					
6215	D2598	181.7	С		first reported: 167
7014	22000		· ·		mot reperious rev
Evaluat	ted over ISO8973/IP432	test results of	nly		iis calculated from ALL reported composition test results
	normality	unknown			suspect
	n autliana	7			44
	outliers	1			0
	mean (n) st.dev. (n)	185.217 0.9947			185.552 0.5252
	R(calc.)	0.9947 2.785			0.5252 1.470
comp	R(iis17S03P)	2.700			0.872
	ted over D2598 test resu	ults only			iis calculated from ALL reported composition test results
	normality	unknown			suspect
	n	4			44
	outliers	0			0
	mean (n)	182.185			181.695
	st.dev. (n)	0.7997			0.5691
	R(calc.)	2.239			1.593
comp	R(iis17S03P)				1.007

Reported test results

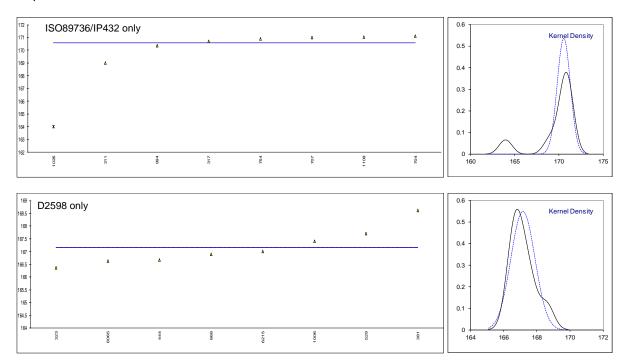




Determination of Relative Vapor Pressure at 100F on sample #18210; results in psi

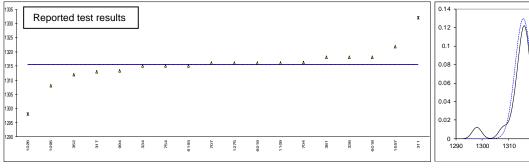
lab	method	value	mark	z(targ)	remarks
92					
150					
158					
171					
311	ISO8973	169	E		calculation error: iis calculated: 170.9 (ISO8973)
317	ISO8973	170.7			
323	D2598	166.37			
333					
334					
335					
336					
337					
347					
352					
381	D2598	168.61	E		calculation error: iis calculated: 167.3 (D2598)
444	D2598	166.66			
445		171			
511					
529	D2598	167.713			
704	ISO8973	171.1			
707	ISO8973	171.0			
754	ISO8973	170.9			
868	D2598	166.9			
994	IP432	170.34			
1006	D2598	167.4	0(0.04) =		
1026	ISO8973	164	G(0.01),E		calculation error: iis calculated: 170.1 (ISO8973)
1040					
1095	10.00070	474.04			
1109	ISO8973	171.04			
1197					
1198					
1257					
1275 1394					
1491					
1557					
1603					
1634					
1786					
1882					
2124					
6018					
6019					
6052					
6065	D2598	166.63	С		first reported as Absolute Vapor Pressure
6193					
6215	D2598	167	С		first reported as Absolute Vapor Pressure
7014					·
Evaluat	ted over ISO8973/IP432	test results of	nly		iis calculated from ALL reported composition test results
	normality	unknown		·	suspect
	n	7			44
	outliers	1			0
	mean (n)	170.583			170.856
	st.dev. (n)	0.7449			0.5252
	R(calc.)	2.086			1.470
comp	R(iis17S03P)				0.872
Evaluat	ted over D2598 test resu				iis calculated from ALL reported composition test results
	normality	unknown			suspect
	n tli	8			44
	outliers	0			0
	mean (n)	167.160			166.998
	st.dev. (n)	0.7284			0.5691
00.777	R(calc.)	2.040			1.593
comp	R(iis17S03P)				1.007

Reported test results



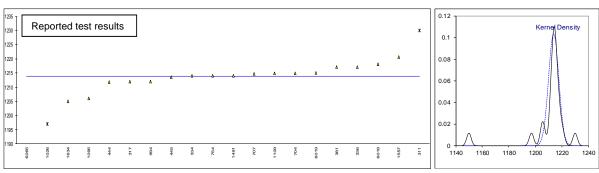
Determination of Absolute Vapor Pressure at 40°C on sample #18210; results in kPa

lab	method	value	mark	z(targ)	remarks
92					
150					
158					
171					
311	ISO8973	1332	C,G(0.01),E		first reported: 193, calculation error: iis calculated: 1315
317	ISO8973	1313	С		first reported: 190.4
323					
333					
334	ISO8973	1315			
335					
336	ISO8973	1318			
337					
347					
352	ISO8973	1312			
381	ISO8973	1318			
444					
445					
511					
529 704	ISO8973				
704	ISO8973	1316.2 1316.0			
754	ISO8973	1315.0			
868	1000373				
994	IP432	1313.33			
1006	11 102				
1026	ISO8973	1298	G(0.05),E		calculation error: iis calculated: 1310
1040			-(),-		
1095	ISO8973	1308			
1109	ISO8973	1316.1			
1197					
1198					
1257					
1275	EN589	1316.0			
1394					
1491					
1557	ISO8973	1321.9			
1603					
1634					
1786					
1882					
2124	1000070	4240			
6018	ISO8973	1318			
6019 6052	ISO8973	1316 			
6065					
6193	ISO8973	1315			
6215	1000070				
7014					
					Calc. by iis from ALL rep. composition results (ISO8973/IP432)
	normality	not OK			suspect
	n	16			44
	outliers	2			0
	mean (n)	1315.47			1314.84
	st.dev. (n)	3.071			3.712
	R(calc.)	8.605			10.39
comp	R(iis17S03P)	3.91			
1335 Re	eported test results				x 0.14
1330					0.12
1325 -					۸ _{0.1} -
1320 -					Δ Δ Δ
1315	. Δ Δ	Δ Δ Δ	A A A		0.08 -
1310 -	Δ Δ Δ				0.06 -
1 1					



Determination of Relative Vapor Pressure at 40°C on sample #18210; results in kPa

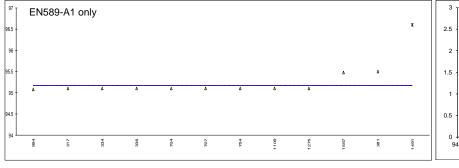
lab	method	value	mark	z(targ)	remarks
92					
150					
158					
171					
311	ISO8973	1230	ex,C,E		fr.: 178, calc. error: iis calc. 1214, excluded for outlier in AVP
317	ISO8973	1212	C		first reported: 175.8
323			_		
333					
334	ISO8973	1214			
335	1000010				
336	ISO8973	1217			
337	1000010				
347					
352					
381	ISO8973	1217			
444	ISO8973	1217.8			
445	1300913				
		1213.5			
511					
529	1000070				
704	ISO8973	1214.9			
707	ISO8973	1214.7			
754	ISO8973	1214			
868	ID 400				
994	IP432	1212			
1006	10000=0		_		
1026	ISO8973	1197	ex,E		calculation error: iis calculated: 1208, excluded for outlier in AVP
1040					
1095	ISO8973	1206			
1109	ISO8973	1214.8			
1197					
1198					
1257					
1275					
1394					
1491	ISO8973	1214	С		first reported as Absolute Vapor Pressure
1557	ISO8973	1220.6			
1603					
1634	ISO8973	1205			
1786					
1882					
2124					
6018	ISO8973	1218			
6019	ISO8973	1215			
6052					
6065	D2598	1149.72	C,G(0.01)		first reported as Absolute Vapor Pressure
6193					
6215					
7014					
					Calc. by iis from ALL rep. composition results (ISO8973/IP432)
	normality	suspect			suspect
	n	17			44
	outliers	1 (+2ex)			0
	mean (n)	1213.78			1213.51
	st.dev. (n)	3.855			3.712
	R(calc.)	10.79			10.39
comp	R(iis17S03P)	3.88			
	()				

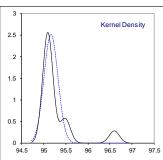


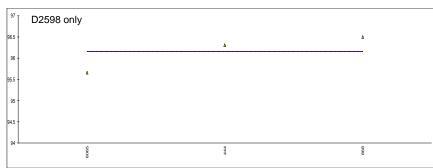
Determination of Motor Octane Number, MON on sample #18210;

lab	method	value	mark	z(targ)	remarks
92					
150					
158					
171					
311					
317	EN589	95.1			
323					
333					
334	EN589	95.1			
335					
336	EN589	95.1			
337					
347					
352					
381	EN589	95.5			
444	D2598	96.3			
445					
511					
529	=1.1=00				
704	EN589	95.10			
707	EN589	95.10			
754	EN589	95.1			
868	D2598	96.5			
994	EN589	95.08			
1006		05.07			
1026		95.07			
1040					
1095	ENEGO	05.4			
1109	EN589	95.1			
1197					
1198 1257					
1275	EN589	95.1			
1394	LNJOS				
1491	EN589	96.6	Е		method used is probably D2598 (iis calc: 96.6 / EN589: 95.1)
1557	EN589	95.48	_		metrica asca is probably become this date. 30.07 E11000. 30.1)
1603	211000				
1634					
1786					
1882					
2124					
6018					
6019					
6052					
6065	D2598	95.66			
6193					
6215					
7014					
Evaluat	ted over EN589-A1 test				iis calculated from ALL reported composition test results
	normality	not OK 11			OK 40
	n outliere				
	outliers mean (n)	1 95.169			4 95.096
	mean (n) st.dev. (n)	0.1588			0.0174
	R(calc.)	0.1366			0.049
comp	R(iis17S03P)				0.038
	ted over D2598 test resu				iis calculated from ALL reported composition test results
Lvaluat	normality	unknown			suspect
	n	3			43
	outliers	0			1
	mean (n)	96.153			96.559
	st.dev. (n)	(0.4388)			0.0485
	R(calc.)	(1.229)			0.136
comp	R(iis17S03P)				0.064
· · · ·	, ,				

Reported test results







Determination of Ideal Gross Heating Value at 14.696 psia/60Fon sample #18210; in kJ/mol

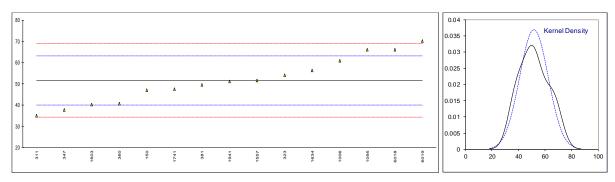
lab	method	value	mark	z(targ)	remarks
92					
150					
158 171					
311					
317	D3588	2258.2			
323					
333 334					
335					
336					
337					
347 352	D3588	2259.815			
381	D3588	2256.2			
444					
445					
511 520					
529 704					
707					
754	D3588	2189.6	G(0.01),E		calculation error: iis calculated: 2256.62 (D3588)
868					
994 1006					
1026					
1040					
1095					
1109	D3588	2256.94			
1197 1198					
1257					
1275					
1394					
1491 1557					
1603					
1634	D3588	2266.63			
1786					
1882 2124					
6018					
6019					
6052					
6065	D3588	9505520	G(0.01),E		calculation error: iis calculated: 2258.90 (D3588)
6193 6215					
7014					
					Calc. by iis from ALL reported composition results (D3588)
	normality	unknown			suspect
	n outliers	5 2			44 0
	mean (n)	2259.56			2257.36
	st.dev. (n)	4.185			2.905
	R(calc.)	11.72			8.13
	R(iis17S03P)	(0.92)			3.77
2400 T R	eported test results				0.12 Kernel Density
2350					0.1 -
2300 -					
2250 -	Δ	Å	4	4	
2200 -	×				0.06 -
2150 -					
2100 +					0.04
2050 +					0.02 -
2000	75.4	98	34.7	362	2150 2200 2250 2300

Determination of Ideal Net Heating Value at 14.696 psia/60F on sample #18210; results in kJ/mol

lab	method	value	mark	z(targ)	remarks	
92						
150						
158						
171 311						
317	D3588	2078.3				
323	D3300	2070.3				
333						
334						
335						
336						
337						
347						
352	D3588	2079.796				
381	D3588	2076.5				
444 445						
511						
529						
704						
707						
754	D3588	2014.9	G(0.01),E		calculation error: iis calcu	lated: 2076.82 (D3588)
868						
994						
1006 1026						
1026						
1040						
1109	D3588	2076.88				
1197	20000					
1198						
1257						
1275						
1394						
1491						
1557 1603						
1634	D3588	2086.19				
1786	D0000					
1882						
2124						
6018						
6019						
6052	D		0(0.04) =			
6065	D3588	8749191	G(0.01),E		calculation error: iis calcu	lated: 2078.97 (D3588)
6193 6215						
7014						
7014					Calc. by iis from ALL repo	orted composition results (D3588)
	normality	unknown			suspect	<u>, , , , , , , , , , , , , , , , , , , </u>
	n	5			44	
	outliers	2			0	
	mean (n)	2079.53			2077.53	
	st.dev. (n)	3.942			2.710	
	R(calc.) R(iis17S03P)	11.04			7.59 3.77	
	K(11817303F)	(1.65)			3.77	
2150	an auto d to attt-					0.14
	eported test results					0.12 - Kernel Density
2110 -						\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \
2090 -		Δ	Δ	Δ	Δ	0.1 -
2070 +	<u>.</u>					0.08 -
2050 -						0.06 -
2030 -	x					[0.00]
2010 +	m					0.04 -
1990 -						0.02
1970 -						
1950	88 1 2 8 4	1108	7.	362	1634	2000 2020 2040 2060 2080 2100
	٧9	-	**	**	96	

Determination of Sulphur, total on sample #18211; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150	D6667	47	•	-0.79	
158					
171					
311	D6667	35		-2.87	
323	D6667	54		0.42	
337					
347	D6667	37.8		-2.38	
360	D6667	40.6		-1.90	
381	D6667	49.4	С	-0.37	first reported: 96.7
445					
1006	D6667	60.83		1.60	
1095	D6667	66		2.50	
1557	D6667	51.6		0.01	
1603	In house	40.3		-1.95	
1634	D6667	56.37		0.83	
1741	D6667	47.4		-0.72	
1941	D6667	51.01		-0.10	
6018	D6667	66.0		2.50	
6019	D6667	70.2		3.22	
	normality	OK			
	n	15			
	outliers	0			
	mean (n)	51.57			
	st.dev. (n)	10.800			
	R(calc.)	30.24			
	st.dev.(D6667:14)	5.782			
	R(D6667:14)	16.19			
	` ,				



APPENDIX 2

Number of participants per country in the Propane PT iis18S03P

- 2 labs in AUSTRALIA
- 1 lab in AZERBAIJAN
- 1 lab in BELGIUM
- 1 lab in CANADA
- 1 lab in CHINA, People's Republic
- 1 lab in COLOMBIA
- 1 lab in DENMARK
- 5 labs in FRANCE
- 2 labs in GERMANY
- 1 lab in IRAN, Islamic Republic of
- 3 labs in MALAYSIA
- 2 labs in MEXICO
- 3 labs in NETHERLANDS
- 1 lab in PERU
- 6 labs in PORTUGAL
- 2 labs in RUSSIAN FEDERATION
- 3 labs in SERBIA
- 1 lab in SPAIN
- 1 lab in TAIWAN
- 2 labs in UKRAINE
- 2 labs in UNITED ARAB EMIRATES
- 3 labs in UNITED KINGDOM
- 3 labs in UNITED STATES OF AMERICA

Number of participants per country in the Sulphur in LPG PT iis18S03S

- 1 lab in BELGIUM
- 1 lab in BULGARIA
- 1 lab in FRANCE
- 1 lab in GERMANY
- 1 lab in NETHERLANDS
- 4 labs in PORTUGAL
- 4 labs in SERBIA
- 1 lab in SPAIN
- 1 lab in TAIWAN
- 1 lab in UNITED KINGDOM
- 3 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C = final result after checking of first reported suspect test result

D(0.01) = outlier in Dixon's outlier test D(0.05) = straggler in Dixon's outlier test D(0.01) = outlier in Grubbs' outlier test D(0.05) = straggler in Grubbs' outlier test D(0.01) = outlier in Double Grubbs' outlier test D(0.05) = straggler in Double Grubbs' outlier test

R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
E = probably an error in calculations

ex = test result excluded from the statistical evaluation

n.a. = not applicable
n.e. = not evaluated
fr. = first reported
SDS = safety data sheet

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, June 2018
- 2 ASTM D2163:14e1
- 3 ASTM D2421:13
- 4 ISO 5725:86
- 5 ISO 5725, parts 1-6, 1994
- 6 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 7 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 8 IP 367:84
- 9 DIN 38402 T41/42
- 10 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 11 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 12 Analytical Methods Committee Technical Brief, No 4 January 2001
- 13 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
- 14 ISO 17043:2010
- 15 EN 27941:1993 = ISO 7941:88 = IP 405:94
- 16 ASTM D2598:16
- 17 IP 432:2000 = ISO 8973:1997
- 18 ASTM D2598:16
- 19 EN 589:08-A1:12
- 20 Private communication ASTM Subcommittee D02.H
- 21 ASTM D3588:98(2017)
- 22 ISO 6976:95(1996)
- 23 ISO 6976:16
- Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, <u>25(2)</u>, 165-172, (1983)