

Institute for Interlaboratory Studies

Results of Proficiency Test Refinery Gas February 2022





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## **1** INTRODUCTION

During the last years several participants requested iis to set up a proficiency test (PT) for Refinery Gas Analysis. In 2021 iis started an investigation for the feasibility of such a PT. Because iis has limited gas-handling facilities in place to prepare gas samples, EffecTech (Uttoxeter, United Kingdom) was contacted. This company is fully equipped and has a broad experience in the preparation of synthetic Refinery Gas samples for PT purposes. It was decided to organize a proficiency study for Refinery Gas in 2022 for the first time.

In this interlaboratory study 19 laboratories in 13 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the Refinery Gas proficiency test 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 optimize the costs for the participating laboratories it was decided to prepare one Refinery Gas mixture. The mixture was divided over a batch of 21 cylinders. The cylinder size is a cost-effective one-liter cylinder. Each cylinder was uniquely numbered and labelled #22015. The limited cylinder size is chosen to optimize transport and handling costs.

Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.

The 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/IEC17043:2010. 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 regular basis by sending out questionnaires. EffecTech is accredited in conformance with ISO/IEC17043:2010 by UKAS (no. 4719) and ISO17025:2017 by UKAS (no. 0590).

## 2.2 PROTOCOL

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). This protocol is also 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

One batch of 21 one-liter cylinders with an artificial Refinery Gas mixture was prepared and tested for homogeneity by EffecTech (Uttoxeter, United Kingdom) in conformance with ISO Guide 35 and ISO/IEC17025 (job 21/0601). Each cylinder was uniquely numbered and labelled #22015. Every cylinder in the batch was analyzed using replicate measurements. The within bottle and between bottle variations were assessed in accordance with ISO Guide 35. This evaluation showed that all between bottle variations were small compared to the uncertainties on the reference values on each component.

The calculated repeatabilities were calculated per component and compared with 0.3 times the corresponding reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

Component	r (observed) in %mol/mol	0.3 * R (target) in %mol/mol	reference method		
Hydrogen	0.0989	1.0180	Horwitz		
Oxygen/Argon	0.0003	0.0311	EN15984:22		
Nitrogen	0.0094	0.1739	EN15984:22		
Carbon Monoxide	0.0034	0.0696	EN15984:22		
Carbon Dioxide	0.0028	0.0236	EN15984:22		
Methane	0.0589	0.2729	EN15984:22		
Ethane	0.0196	0.0942	EN15984:22		
Ethene	0.0051	0.0374	EN15984:22		
Propane	0.0739	0.0715	EN15984:22		
Propene	0.0205	0.0287	EN15984:22		
iso-Butane	0.0203	0.0345	EN15984:22		
n-Butane	0.0169	0.0364	EN15984:22		
trans-2-Butene	0.0018	0.0068	Horwitz		
1-Butene	0.0021	0.0068	Horwitz		
iso-Butene	0.0017	0.0048	Horwitz		
cis-2-Butene	0.0017	0.0048	Horwitz		
iso-Pentane	0.0024	0.0034	EN15984:22		
n-Pentane	0.0024	0.0088	EN15984:22		

Table 1: evaluation of the repeatabilities of subsamples #22015

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed. For more details about choice of reference method see paragraph 4.1.

To each of the participating laboratories one 1L cylinder labelled #22015 was sent on January 26, 2022. An SDS was added to the sample package.

## 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 ANALYZES

The participants were requested to determine: Hydrogen, Argon, Oxygen, Nitrogen, Carbon Monoxide, Carbon Dioxide, Hydrogen Sulfide, Methane, Ethane, Ethene, Ethyne, Propane, Propene, Propyne, Propadiene, iso-Butane, n-Butane, trans-2-Butene, 1-Butene, iso-Butene, cis-2-Butene, 1,3-Butadiene, iso-Pentane, n-Pentane, Other components with C5 or more C atoms (except iso-Butane and Pentane), Carbon content and Lower Calorific Value.

It was explicitly requested to treat the sample as if it was a routine sample 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' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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 reference test methods (when applicable) 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 appendices 1 and 2 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 reanalyzes). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the result tables in appendices 1 and 2.

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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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, 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 requirements 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 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 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

## 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 derived from e.g. ASTM or EN methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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, like Horwitz or an estimated reproducibility based on former is proficiency tests.

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:

 $z_{(target)}$  = (test result - average of PT) / target standard deviation

The  $z_{(target)}$  scores are listed in the result tables of 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 proficiency test some problems were encountered with the dispatch of the samples. Therefore, the reporting time on the data entry portal was extended with another week. One participant reported test results after the extended final reporting date and two other participants did not report any test results. Not all participants were able to report all tests requested.

In total 17 participants reported 357 numerical test results. Observed were 21 outlying test results, which is 5.9%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a 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 COMPONENT

In this section the reported test results are discussed per component. 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 in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

Test method EN15984 is used to evaluate the performance of the test results for Refinery Gas. The method version of 2017 describes only precision data for Carbon content and Lower Calorific Value. In February of 2022 a new version of this method was published. The precision data for the parameters Carbon content and Lower Calorific Value remained the same. Precision data for most of the components appeared to have been added to the test method in an informative annex. It was decided to use these published precision data for the evaluation of the components, except for Hydrogen. The mean of Hydrogen is far out of the application range mentioned in test method EN15984.

For components trans-2-Butene, iso-Butene and cis-2-Butene no precision data was given in test method EN15984. Therefore, the estimated reproducibility calculated by the Horwitz equation was used for the evaluation of these three components and also for Hydrogen.

One laboratory reported deviating test results for many of the gas composition test results. In total 10 test results of the 17 reported test results were statistical outliers. As the test results are not obtained independently, it was decided not to use any of the reported results of this laboratory for the statistical evaluation. This means that the remaining reported test results were excluded.

<u>Total of the composition results</u>: The total of the test results of the composition per laboratory was calculated by iis. Since the composition is requested as normalized the total should be 100%. Two calculated results were found to be lower than 100%. Both laboratories did not report test results for all components.

<u>Hydrogen</u>: The determination of this component was not problematic. No statistical outliers were observed but one test result was excluded. The calculated

reproducibility after rejection of the suspect data is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Oxygen/Argon: Test method EN15984:22 describes Argon and Oxygen combined as the second Refinery heating gas component. In this PT both components were requested separately. Many participants commented that these two gases co-elute and that the total of the two gases was submitted either as Argon or as Oxygen. Therefore, it was decided to evaluate these components as the sum of Oxygen and Argon.

The determination of the sum of both components was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN15984:22.

- <u>Nitrogen</u>: The determination of this component was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN15984:22.
- <u>Carbon Monoxide</u>: The determination of this component was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN15984:22.
- <u>Carbon Dioxide</u>: The determination of this component was not problematic. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN15984:22.
- <u>Methane</u>: The determination of this component was not problematic. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN15984:22.
- <u>Ethane</u>: The determination of this component was not problematic. One statistical outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN15984:22.
- <u>Ethene</u>: The determination of this component was not problematic. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN15984:22.
- <u>Propane</u>: The determination of this component was problematic. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN15984:22.

- <u>Propene</u>: The determination of this component was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN15984:22.
- <u>iso-Butane</u>: The determination of this component was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN15984:22.
- <u>n-Butane</u>: The determination of this component was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN15984:22.
- <u>trans-2-Butene</u>: The determination of this component was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.
- <u>1-Butene</u>: The determination of this component was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement the requirements of EN15984:22.
- <u>iso-Butene</u>: The determination of this component was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.
- <u>cis-2-Butene</u>: The determination of this component was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.
- iso-Pentane: The determination of this component was problematic. Three statistical outliers were observed and one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN15984:22.
- <u>n-Pentane</u>: The determination of this component may be problematic for a number of laboratories. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN15984:22.
- <u>Carbon content</u>: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN15984:22.
- <u>Lower Calorific Value</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN15984:22.

The majority of the participants agreed on a concentration near or below the limit of detection for all other requested components mentioned in paragraph 2.5. Therefore, no z-scores were calculated. The reported test results of these components are given in appendix 2.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 \* standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM and EN standards) or estimated using the Horwitz equation are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
Hydrogen	%mol/mol	16	55.58	1.68	3.40
Oxygen/Argon	%mol/mol	13	0.125	0.125	0.109
Nitrogen	%mol/mol	15	2.617	0.553	0.581
Carbon Monoxide	%mol/mol	15	0.820	0.180	0.235
Carbon Dioxide	%mol/mol	16	0.503	0.084	0.079
Methane	%mol/mol	16	19.85	0.90	0.91
Ethane	%mol/mol	15	7.913	0.267	0.311
Ethene	%mol/mol	15	1.985	0.116	0.124
Propane	%mol/mol	16	5.032	0.360	0.236
Propene	%mol/mol	15	1.601	0.089	0.095
iso-Butane	%mol/mol	15	1.752	0.133	0.112
n-Butane	%mol/mol	15	1.361	0.109	0.118
trans-2-Butene	%mol/mol	15	0.146	0.020	0.022
1-Butene	%mol/mol	15	0.146	0.021	0.015
iso-Butene	%mol/mol	13	0.098	0.015	0.016
cis-2-Butene	%mol/mol	14	0.097	0.015	0.015
iso-Pentane	%mol/mol	12	0.176	0.015	0.009
n-Pentane	%mol/mol	13	0.156	0.025	0.027
Carbon content	g/100g	11	67.61	0.97	2.16
Lower Calorific Value	kJ/100g	11	4929	118	120

Table 2: reproducibilities of tests of sample #22015

Without further statistical calculations it can be concluded that for many components and the calculated parameters there is a good compliance of the group of participating laboratories with the relevant reference test method. The problematic components have been discussed in paragraph 4.1.

#### 4.3 OVERVIEW OF THE PROFICIENCY TEST OF FEBRUARY 2022

	February 2022
Number of reporting laboratories	17
Number of test results	357
Number of statistical outliers	21
Percentage of statistical outliers	5.9%

Table 3: overview of this proficiency test

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 reference methods. The conclusions are given the following table.

Component	February 2022
Hydrogen	++
Oxygen/Argon	-
Nitrogen	+/-
Carbon Monoxide	+
Carbon Dioxide	+/-
Methane	+/-
Ethane	+
Ethene	+/-
Propane	-
Propene	+
iso-Butane	-
n-Butane	+/-
trans-2-Butene	+/-
1-Butene	-
iso-Butene	+/-
cis-2-Butene	+/-
iso-Pentane	-
n-Pentane	+/-
Carbon content	++
Lower Calorific Value	+/-

Table 4: comparison determinations against the reference method

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
- n.e. : not evaluated

## 5 DISCUSSION

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 the average values as determined in this PT are very well in line with the values as determined during the preparation of the gas cylinders.

Component	EffecTech in %mol/mol	Average PT in %mol/mol	Differences in %mol/mol	z-score
Hydrogen	55.448	55.583	-0.135	-0.11
Oxygen/Argon	0.100	0.125	-0.025	-0.64
Nitrogen	2.510	2.617	-0.107	-0.52
Carbon Monoxide	0.808	0.820	-0.011	-0.14
Carbon Dioxide	0.499	0.503	-0.004	-0.14
Methane	19.907	19.846	0.061	0.19
Ethane	7.989	7.913	0.076	0.68
Ethene	1.997	1.985	0.011	0.25
Propane	5.087	5.032	0.055	0.65
Propene	1.612	1.601	0.011	0.32
iso-Butane	1.796	1.752	0.044	1.11
n-Butane	1.398	1.361	0.037	0.89
trans-2-Butene	0.152	0.146	0.006	0.73
1-Butene	0.152	0.146	0.006	1.15
iso-Butene	0.100	0.098	0.002	0.33
cis-2-Butene	0.102	0.097	0.004	0.81
iso-Pentane	0.180	0.175	0.005	1.37
n-Pentane	0.163	0.156	0.007	0.70

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

lab	method	iis calculated	remarks
444	EN15984	100	
446			
1026	EN15984	99.9871	
1040	EN15984	100.04	
1062	EN15984	99.9999	
1069	UOP539	99.9996	
1081		99.908	
1140	D7833	99.999	
1528	UOP539	99.981	
1635	UOP539	94.1	not 100%, reported zero for some components
1737	EN15984	100	
1741	UOP539	100	
1961	EN15984	100.001	
1964	In house	99.9953	did not report individual C4 compounds, only the sum of C4: 3.92%
6142	EN15984	96.3255	not 100%, did not report all components
6203	UOP539	100	
6369	ISO17025	100	
6404	EN15984	100.001	
9008			

# Determination of Hydrogen on sample #22015; results in %mol/mol

la	ab	metho	bd		value	mar	'k	z(targ)	rem	arks							
44	14 16	EN159	984		55.761	С		0.15	first	reported	1: 55.65	8					
102 104 106 106 108 114 152 163 173 174 196 614 620 638 640 900	446 1026 1040 1062 1069 1081 1140 1528 1635 1737 1741 1961 1964 6142 6203 6369 6404 9008		444       EN15984       55.761       C       0.15       Inst reported: 55.558         446         first reported: 54.41833333         1026       EN15984       55.80       0.18         1062       EN15984       55.7340       0.12         1069       UOP539       55.32       -0.22         1081       55.997       0.34         1140       D7833       55.538       C       -0.04         1528       UOP539       56.10       C       0.43         1635       UOP539       55.04       ex       -0.45         1635       UOP539       55.75       0.14         1737       55.57       -0.01         1741       UOP539       55.75       0.14         1961       EN15984       55.875       0.24         1964       In house       54.953       -0.52         3142       EN15984       55.557       -0.02         3203       UOP539       54.02       -1.29         3369       ISO17025       56.3179       0.61         3404       EN15984       56.325       0.61         3008     <								4.1						
		norma	lity		suspect												
compa	re	n outlier mean st.dev R(calc st.dev R(Hor	s (n) . (n) 2.) .(Horwit witz)	tz)	16 0 (+1ex) 55.5830 0.60100 1.6828 1.21440 3.4003												
oompu		R(EN1	15984:2	2)	1.5684				appl	ication r	ange: 3	.60 - 4.6	60				
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$		Δ	۵	×	Δ		<u>A</u>	<u>.</u>	A	<u>A</u>	<b>A</b>	Δ	Δ	Δ	۵	Δ	
50		1026	1964	1635	1069	1140	6142	1737	1062	1741	444	1040	1961	1081	1528	6369	6404
0.8 - 0.7 - 0.6 - 0.5 - 0.4 - 0.3 - 0.2 - 0.1 - 5	2	53	54		55 56		nsity	58									

# Determination of Oxygen/Argon on sample #22015; results in %mol/mol

$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$		lab	method			Ar	<b>O</b> <sub>2</sub>	Ar+O <sub>2</sub>	mark	z(targ)	remarks			
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	2	444	EN15984			0.096		0.096		-0.75				
1026 EN15984 0.3393 0.3393 G(0.01) 5.48 $1040 EN15984 0.000 0.1017 0.1017 - 0.60$ $1069$	2	446												
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	10	026	EN15984				0.3393	0.3393	G(0.01)	5.48				
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	10	040	EN15984			0.13	0.00	0.13		0.12				
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	10	062	EN15984			0.000	0.1017	0.1017		-0.60				
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	10	069												
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	10	180	D7000				0.127	0.127		0.05				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	10	140	D7833				0.130	0.13		0.12				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	16	020 635	00F559			0.105		0.105		-0.51				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	17	737												
$     \begin{array}{ccccccccccccccccccccccccccccccccc$	17	741	LIOP530				0.11	0.11		-0.30				
1964 In house 0.047 0 0.047 -2.00 6142 EN15984 0.176 0.176 1.30 6203 UOP539 0 0 .15 0.15 0.64 6369 ISO17025 0.1229 0.1229 -0.06 6404 EN15984 0.097 0.097 -0.72 9008 normality n ot OKn 0.1251 0.15 st.dev. (n) 0.04476 R(calc.) 0.1253 st.dev. (EN15984:22) 0.1253 st.dev. (EN15984:22) 0.1094 application range: 0.20 - 2.30 $R(EN15984:22) 0.1094 application range: 0.20 - 2.30$	19	961	EN15984				0.234	0.234		2 79				
	19	964	In house			0 047	0	0.047		-2 00				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	61	142	EN15984			0.176		0.176		1.30				
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	62	203	UOP539			0	0.15	0.15		0.64				
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	63	369	ISO17025				0.1229	0.1229		-0.06				
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	64	404	EN15984				0.097	0.097		-0.72				
$normality \\ n \\ outliers \\ mean (n) \\ st.dev. (n) \\ R(calc.) \\ st.dev. (EN15984:22) \\ R(EN15984:22) \\ 0.1094 \\ application range: 0.20 - 2.30 \\ \\ n $	90	800												
$normality \\ n \\ outliers \\ st.dev. (n) \\ R(calc.) \\ st.dev. (EN15984:22) \\ R(EN15984:22) \\ 0.1094 \\ application range: 0.20 - 2.30 \\ x \\ $														
n = 13 = 13 = 13 = 13 = 13 = 13 = 13 = 1			normality					not OK						
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$			n					13						
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$			outliers					1						
$\begin{array}{cccccccccccccccccccccccccccccccccccc$			mean (n)					0.1251						
$\begin{array}{c} \begin{array}{c} (car.) \\ st.dev.(EN15984:22) \\ R(EN15984:22) \end{array} \\ \begin{array}{c} 0.1253 \\ 0.03909 \\ 0.1094 \end{array} \\ \begin{array}{c} application range: 0.20 - 2.30 \end{array} \\ \begin{array}{c} x \\ x \\ \hline \\$			st.dev. (n)					0.04476						
$\begin{array}{c} \text{Sidev.(EN15984:22)} \\ \text{R(EN15984:22)} \\ \text$			R(calc.)	45004.00	<b>)</b> \			0.1253						
$\begin{array}{cccccccccccccccccccccccccccccccccccc$			R/EN1508	10904.22 (1·22)	<u>-</u> )			0.03909			applicatio	n range	· 0 20 - 2	30
104 35 13 13 13 14 14 15 15 15 15 15 15 15 15 15 15				H.22)				0.1034			applicatio	manye	5. 0.20 - 2	.50
35 <td>04 -</td> <td></td>	04 -													
35     4     60     60     4     60     60     4     60       25     2     2     2     2     2     2     2       0     0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     0     0     0       0     0     0     <	0.4													
103 100 100 100 100 100 100 100 100 100	0.35 -													*
25 - 22 - 22 - 22 - 22 - 22 - 22 - 22 -	0.3 -													
	0.25 -													
	0.2 -												4	
	0.15											۵		
0 1 1 1 1 1 1 1 1 1 1 1 1 1	J. 10 -						•	Δ Δ	A	A	Δ			
136 136 137 138 138 138 138 138 138 138 138	0.1 -		Δ	۵	Δ	Δ	-							
1986 1122.66 6.60.4 1124.1 1040 6.638.9 6.638.9 6.638.9 6.638.9 1144.0 6.638.9 6.638.9 1144.0	0.05 -	Δ												
8 7 7 8 8 8 7 7 8 8 8 7 7 8 8 8 7 7 8 8 8 8 7 7 8 8 8 8 7 7 7 8 8 8 8 7 7 7 8 8 8 8 7 7 7 8 8 8 8 7 7 7 8 8 8 8 7 7 7 8 8 8 8 8 7 7 7 8 8 8 8 8 7 7 7 8	o ــــــــــــــــــــــــــــــــــــ	4	4	4	5	80	<del>.</del>	6 -	0	0	e	7	<u>~</u>	9
		196	4	640	106	152	174	636	101	114	620	614	196	102



# Determination of Nitrogen on sample #22015; results in %mol/mol

lab	method		value	1	mark	z(targ)	r	remarks							
444	EN15984		2.499	(	C	-0.57	f	irst report	ed: 2.4	95					
446															
1026	EN15984		2.9783	(	С	1.74	f	irst reporte	ed: 3.3	039					
1040	EN15984		2.55			-0.32									
1062	EN15984		2.4709			-0.70									
1069	UOP539		2.561			-0.27									
1081			2.563			-0.26									
1140	D7833		2.688	(	С	0.34	f	irst report	ed: 2.6	75					
1528	UOP539		2.445			-0.83									
1635	UOP539		0.61	(	G(0.01)	-9.66									
1737			2.54		. ,	-0.37									
1741	UOP539		2.47			-0.71									
1961	EN15984		2.548			-0.33									
1964	In house		2.723			0.51									
6142	EN15984		3.4035	(	G(0.05)	3.79									
6203	UOP539		3.14		. ,	2.52									
6369	ISO17025		2.5896			-0.13									
6404	EN15984		2.487			-0.63									
9008															
	normality		not OK												
	n		15												
	outliers		2												
	mean (n)		2.6169												
	st.dev. (n)		0.19743	3											
	R(calc.)		0.5528												
	st.dev.(EN1598	4:22)	0.20767	7											
	R(EN15984:22)	)	0.5815				6	application	n range	: 5.00 - 5	57.00				
3.6 T															
34 -															×
3.2 -														Δ	
3 -													Δ		
2.8 -															
26											۵	Δ			
2.0 -	<u>م</u>	▲	۵	Δ	4	Δ	Δ	Δ	4	Δ					
2.4 -	<b>4</b> –														
2.2															
2			-											~	
1635	1526	1062	6404	444	1737	1961	1040	1069	1081	6369	1140	1964	1026	6203	6142
L															



# Determination of Carbon Monoxide on sample #22015; results in %mol/mol

lab	method	value	mark	z(targ)	remarks						
444	EN15984	0.771	С	-0.58	first reported:	0.770					
446			0		Constant of 1	0 70770					
1026	EN15984	0.8099	C	-0.12	first reported;	0.78773	3333				
1040	EN15904 EN15984	0.03		-0.12							
1069	UOP539	0 789		-0.20							
1081		0.721		-1.18							
1140	D7833	0.131	C,G(0.01)	-8.22	first reported:	0.040					
1528	UOP539	0.851	. ,	0.37							
1635	UOP539	0.0	G(0.05)	-9.79							
1737		0.85		0.36							
1/41	UOP539	0.81		-0.12							
1901	EN 15984	1.018		2.37							
6142	FN15984	0.774		-0.55							
6203	UOP539	0.80		-0.24							
6369	ISO17025	0.8324		0.15							
6404	EN15984	0.821		0.01							
9008											
	normality	not OK									
	n	15									
	mean (n)	∠ 0 8198									
	st.dev. (n)	0.06418									
	R(calc.)	0.1797									
	st.dev.(EN15984:2	22) 0.08376									
	R(EN15984:22)	0.2345			application ra	nge: 0.50	) – 3.10				
<sup>1.1</sup> T											
1											Δ
09-											
0.9 T					•		<b>A</b>	Δ	Δ	Δ	
0.8	Δ	<u>م</u> ۵	Δ	Δ Δ	<u> </u>						
0.7	۵										
0.6											
0.5 <u>-</u> 32 2	1140 1081 444	1964	1062	6203	1741	6142	1040	6369	1737	1528	1961



## Determination of Carbon Dioxide on sample #22015; results in %mol/mol

la	b	method		value	Э	mark	z(tar	·g)	remarks							
44	14	EN15984		0.489	)	С	-0.	51	first repor	ted: 0.4	88					
44	16															
102	26	EN15984		0.485	59	С	-0.	62	first repor	ted: 0.4	9223333	33				
104	10	EN15984		0.48			-0.	83								
106	62	EN15984		0.496	69		-0.	23								
106	59	UOP539		0.504	1		0.	02								
108	31			0.503	3		-0.	01								
114	10	D7833		0.544	1		1.	45								
152	28	UOP539		0.500	)		-0.	12								
163	35	UOP539		0.5		ex	-0.	12	test result	t exclud	ed, see	paragra	ph 4.1			
173	37			0.51			0.	24								
174	11	UOP539		0.50			-0.	12								
196	51	EN15984		0.506	j N		0.	09								
196	54	In house		0.426	5		-2.	75								
614	12	EN15984		0.561	1		2.	05								
620	)3	UOP539		0.51	-		0.	24								
636	59	ISO17025		0.537	6		1.	22								
640	)4	EN15984		0.500	)		-0.	12								
900	18															
		n ormality		not O												
		normality		16	'n											
		outliore		0 (±1	<b>0</b> Y)											
		mean (n)		0 (+1	57) 52											
		et dev (n)		0.000	284											
		R(calc.)		0.023	26 26											
		st dev (EN1508	4.22)	0.000	312											
		R(FN15984-22)	)	0.020	37				applicatio	n range	0 40 -	10.00				
			/	0.070					apprioutio	in range	. 0. 10	10.00				
06-																
0.58																
0.56																
0.54 -															۵	
0.52 -														4		
0.5					¥				Δ	Δ	Δ	Δ	Δ			
0.48 -		<u>م</u>	Δ	Δ		-	-	-								
0.46 -		-														
0.44																
0.42																
0.4																
1964		1040	444	1062	1635	1528	1741	6404	1081	1069	1961	1737	6203	6369	1140	6142



## Determination of Methane on sample #22015; results in %mol/mol

	lab	method		value		mark	z(targ)	rem	arks							
	444	EN15984		19.99	6	С	0.46	first	reported	1: 19.959	9					
	446															
	1026	EN15984		20.12	241	С	0.86	first	reported	1: 20.044	468333					
	1040	EN15984		19.97			0.38									
	1062	EN15984		20.03	96		0.60									
	1069	UOP539		19.89	82		0.16									
	1081			19.83	6	-	-0.03									
	1140	D7833		20.20	)7	C	1.11	first	reported	1: 20.229	9					
	1528	UOP539		19.61		C	-0.73	first	reported	1: 20.09	(					
	1635	UOP539		19.23	5	ex	-1.90	test	result ex	cluded,	see pa	ragraph	4.1			
	1/3/			20.19	)		1.06									
	1/41	UUP539		19.41	2		-1.35									
	1064			10.07	34		-1.40									
	6142	EN1508/		19.07	1		-1.25									
	6203			20 44	• 1		-1.20									
	6360	19017025		10.65	20		-0.60									
	6404	EN15984		19.03	23 '5		-0.00									
	9008	LINISSOF			0		-1.14									
	0000															
		normality		OK												
		n		16												
		outliers		0 (+1	ex)											
		mean (n)		19.84	-6Ó											
		st.dev. (n)		0.323	13											
		R(calc.)		0.904	8											
		st.dev.(EN1	5984:22)	0.324	13											
		R(EN15984:	22)	0.907	6			appl	ication r	ange: 4	.00 – 20	0.00				
<sup>21</sup>																
20.5 -																Δ
													۵	۵	Δ	
20 -							Δ	4	4	Δ	Δ	Δ				
10.5					۵	۵										
19.5 -		Δ Δ	Δ	Δ												
	×															
19 -																
18.5																
10.0	1635	1961	8142	6404	1528	6369	1081	1964	1069	1040	444	1062	1026	1737	1140	6203
			-	-		-										-



## Determination of Ethane on sample #22015; results in %mol/mol

li	ab	method			value	)	mark	z(ta	rg)	remarks							
4	44	EN1598	34		7.939	)	С	0.	23	first repor	ted: 7.9	27					
4	46									-							
10	26	EN1598	84		7.976	6	С	0.	57	first repor	ted: 7.9	478166	67				
10	40	EN1598	84		7.86			-0.	48	•							
10	62	EN1598	34		7.998	33		0.	76								
10	69	UOP53	9		8.025	5		1.	.00								
10	81				7.920	)		0.	.06								
11	40	D7833			8.061		С	1.	.33	first repor	ted:8.07	70					
15	28	UOP53	9		7.948	3		0.	.31								
16	35	UOP53	9		7.66		ex	-2.	28	test resul	t exclud	ed, see	paragra	ph 4.1			
17	37				7.95			0.	.33					•			
17	41	UOP53	9		7.85			-0.	57								
19	61	EN1598	34		7.756	6		-1.	42								
19	64	In house	е		8.003	37		0.	.81								
61	42	EN1598	34		7.761			-1.	37								
62	03	UOP53	9		8.38		G(0.05)	4.	.20								
63	69	ISO170	25		7.831	1	. ,	-0.	74								
64	04	EN1598	34		7.821			-0.	83								
90	08																
		normalit	ty		OK												
		n	•		15												
		outliers			1 (+1	ex)											
		mean (r	ı)		7.913	34											
		st.dev. (	(n)		0.095	525											
		R(calc.)			0.266	67											
		st.dev.(	EN1598	34:22)	0.111	15											
		R(EN15	984:22	)	0.311	2				applicatio	n range	: 3.90 –	10.00				
<sup>8.5</sup> T																	
8.4 -																	*
8.3 -																	
8.2																	
8.1 -																•	
8 -												▲	Δ	۵	Δ	-	
7.9							•		Δ	Δ	Δ						
7.8			•	۵	4	Δ	-										
7.7		4	-														
7.6																	
7.5			2	*		2	9		Z	88	28	98	22	*	g	9	
163		196	614	640	636	174	104	108	44	152	173	102	106	196	106	114	620
L																	



# Determination of Ethene on sample #22015; results in %mol/mol

lab	method	value	mark	z(targ)	remarks							
444	EN15984	1.971	С	-0.32	first repo	rted: 1.9	62					
446												
1026	EN15984	1.965	С	-0.45	first repo	rted: 1.9	6225					
1040	EN15984	1.97		-0.34								
1062	EN15984	1.9917		0.15								
1069	UOP539	2.01		0.56								
1081		1.975		-0.23								
1140	D7833	2.016	С	0.69	first repo	rted: 2.0	18					
1528	UOP539	1.877		-2.43								
1635	UOP539	1.92	ex	-1.47	test resul	t exclud	ed, see p	baragrap	h 4.1			
1737		2.01		0.56								
1741	UOP539	1.98		-0.12								
1961	EN15984	2.005		0.45								
1964	In house	2.0006		0.35								
6142												
6203	UOP539	2.07		1.91								
6369	ISO17025	1.9872		0.05								
6404	EN15984	1.949		-0.81								
9008												
	normality	not OK										
	n 	15										
	outliers	0 (+1ex)										
	mean (n)	1.9852										
	st.dev. (n)	0.04143										
	R(Calc.)	0.1100										
	D/EN15084-22)	0.04440			applicatio	n rango	.1.00	1 00				
	N(LN15504.22)	0.1245			applicatio	mange	. 1.00 – .	4.00				
0.45												
2.15												
2.1 -												
2.05 -												Δ
									•	•	Δ	
2				<u>Λ Δ</u>		Δ	Δ	Δ	4	Δ		
1.95 -	<u>م</u>	<b>A</b>	Δ	Δ –								
	*											
1.9 -												
1.85												
18												
1528	1635 6404 1026	1040	444	1081	6369	1062	1964	1961	1069	1737	1140	6203



## Determination of Propane on sample #22015; results in %mol/mol

	lab	method		value	e	mark	z(targ)	rem	arks							
	444	EN15984		4.993	3	С	-0.46	first	reported	1: 4.984						
	446								·							
1	026	EN15984		5.005	5	С	-0.32	first	reported	l: 4.9855	83333					
1	040	EN15984		5.00			-0.38									
1	062	EN15984		4.964	46		-0.79									
1	069	UOP539		5.12	1		1.06									
1	1081			4.973	3		-0.69									
1	140	D7833		5.092	2	С	0.71	first	reported	1: 5.098						
1	1528	UOP539		5.035	5		0.04									
1	1635	UOP539		4.86		ex	-2.03	test	result ex	cluded,	see par	agraph	4.1			
1	1737			4.99			-0.49									
1	1741	UOP539		5.24			2.47									
1	961	EN15984		5.058	3		0.31									
1	964	In house		5.29	19		3.08									
6	6142	EN15984		4.908	35		-1.46									
6	6203	UOP539		5.12			1.05									
6	6369	ISO17025		4.738	38		-3.47									
6	6404	EN15984		4.976	5		-0.66									
g	8006															
		normality		susp	ect											
		n		16												
		outliers		0 (+1	ex)											
		mean (n)		5.03	17											
		st.dev. (n)		0.128	363											
		R(calc.)		0.360	)2											
		st.dev.(EN1	5984:22)	0.084	143							_				
		R(EN15984	:22)	0.236	54			appl	ication r	ange 2.0	00 - 6.00	J				
<sup>5.4</sup>																
5.3 -																∆
5.2															۵	
													Δ	Δ		
5.1 +											Δ	Δ				
5 -			4	Δ	Δ	Δ	Δ	Δ	Δ	<u> </u>						
4.9 -		۵	-													
48		*														
0 T	Δ															
4.7 -																
4.6	8	42 B5	8	5	ß	37	4	6	8	8	5	4	8	68	14	25
	63(	16:	106	106	64(	173	42	102	102	152	196	114	62(	106	174	196
L																



# Determination of Propene on sample #22015; results in %mol/mol

la	ıb	method	value	mark	z(targ)	remai	'ks						
44	14	EN15984	1.574	С	-0.80	first re	ported:	1.639					
44	16						•						
102	26	EN15984	1.6118	С	0.30	first re	ported:	1.603533	3333				
104	10	EN15984	1.58		-0.63		•						
106	62	EN15984	1.5781		-0.68								
106	69	UOP539	1.613		0.34								
108	31		1.569		-0.95								
114	10	D7833	1.608	С	0.19	first re	ported:	1.610					
152	28	UOP539	1.59	С	-0.34	first re	ported:	1.337					
163	35	UOP539	2.30	G(0.01)	20.50								
173	37		1.58		-0.63								
174	1	UOP539	1.68		2.31								
196	61	EN15984	1.606		0.13								
196	64	In house	1.6269		0.75								
614	12												
620	)3	UOP539	1.65		1.43								
636	69	ISO17025	1.5816		-0.58								
640	)4	EN15984	1.573		-0.83								
900	)8												
			1.01/										
		normality	not OK										
		n	15										
		outliers	1 6014										
		mean (n)	1.0014										
		SLOEV. (II)	0.03172										
		st day (EN15081.2	2) 0.03407										
		R(FN15984.22)	0.00407			annlic	ation rar	nde: 0.50	-6.00				
			0.0001			appilo	adonnai	.go. 0.00	0.00				
18 -													
4.75													
1.75 -													
1.7												Δ	
1.65 -											Δ		
1.6						Δ	Δ	Δ	Δ	Δ			
1 55 A		Δ Δ 4	▲ ▲	Δ Δ	4								
1.5													
1.45 -													
1.4		4 4 5	4 9	25 Q	8		9	98	g	X	g	=	
106		6641 42 106	<u> </u>	17:	152	196	114	102	106	196	62(	174	160
r													



## Determination of iso-Butane on sample #22015; results in %mol/mol

lab	method	value	mark	z(targ)	rema	rks						
444	EN15984	1.768	С	0.41	first re	eported:	1.764					
446												
1026	EN15984	1.7703	С	0.46	first re	eported:	1.7641					
1040	EN15984	1.75		-0.04								
1062	EN15984	1.7175		-0.86								
1069	UOP539	1.8066		1.37								
1081	D7000	1./1/	0	-0.87	<b>C</b>		4 700					
1140	D7833	1.794	C	1.06	TIPST P	eported:	1.796					
1528	UOP539	1.70		-1.30	TIPST P	eportea:	1.498					
1033	00F339	1.30	G(0.01)	-9.0Z								
1737		1.70		1.06								
1061	EN15084	1.05		0.03								
1964	LINIUUU			0.00								
6142	EN15984	1 7255		-0 66								
6203	UOP539	1.65		-2.55								
6369	ISO17025	1.7134		-0.96								
6404	EN15984	1.765		0.33								
9008												
	normality	OK										
	n	15										
	outliers	1										
	mean (n)	1.7518										
	st.dev. (n)	0.04740										
	R(Calc.)	0.1327										
	R/EN15984-22)	0.03991			annlir	ation rar	nde: 1.00	- 2 50				
	N(LN15504.22)	0.1110			applic	allon la	ige. 1.00	- 2.50				
10												
1.9 -												
1.85 -												Δ
1.8 -									▲	۵	۵	
1.75				Δ	۵	۵	۵	Δ				
17		۵	۵ ۵									
1.7 -	Δ											
1.65 -	۵											
1.6 -												
1.55	69 KS Q3	18	42 62	64	04	44	56	37	61	40	69	41
162	152 624	106	100	2	640	4	102	21	196	14	106	174



# Determination of n-Butane on sample #22015; results in %mol/mol

lab	method	value	mark	z(targ)	remar	ks						
444	EN15984	1.369	С	0.19	first re	ported:	1.366					
446												
1026	EN15984	1.3775	С	0.39	first re	ported:	1.374					
1040	EN15984	1.34		-0.49								
1062	EN15984	1.3166		-1.05								
1069	UOP539	1.3995		0.91								
1081		1.312		-1.15								
1140	D7833	1.376	С	0.36	first re	ported:	1.378					
1528	UOP539	1.38	С	0.45	first re	ported:	1.216					
1635	UOP539	0.14	G(0.01)	-28.86								
1737		1.37		0.22								
1741	UOP539	1.42		1.40								
1961	EN15984	1.391		0.71								
1964												
6142	EN15984	1.3445		-0.39								
6203	UOP539	1.37		0.22								
6369	ISO17025	1.2673		-2.21								
6404	EN15984	1.379		0.43								
9008												
	normality	suspect										
	n	15										
	outliers	1										
	mean (n)	1.3608										
	st.dev. (n)	0.03886										
	R(calc.)	0.1088										
	D/EN15084-22)	0.04231			applier	ation rar	ngo 1 00	4 00				
	N(LN15504.22)	0.1105			applica	allon fai	ige 1.00	- 4.00				
1.55												
1.5 -												
1.45 -												
												Δ
1.4 -								•	•	Δ	Δ	
1.35 -			<u> </u>	Δ	Δ	Δ	4	-	-			
	۵	Δ	-									
1.3 -	-											
1.25 -	Δ											
1.2												
1635	6369	1062	6142	1737	6203	1140	1026	6404	1528	1961	1069	1741



## Determination of trans-2-Butene on sample #22015; results in %mol/mol

lah	mothed	value	mork	=/torg)	Pamarka
	ENI45004			Z(targ)	
444	EN15984	0.137	C	-1.14	first reported: 0.178
446			•		
1026	EN15984	0.1483	C	0.31	first reported: 0.14795
1040	EN15984	0.14		-0.75	
1062	EN15984	0.1422		-0.47	
1069	UOP539	0.1515		0.72	
1081		0.143		-0.37	
1140	D7833	0.144		-0.24	
1528	UOP539	0.15	С	0.53	first reported: 0.114
1635	UOP539	0.10	G(0.01)	-5.88	
1737		0.15		0.53	
1741	UOP539	0.16		1.81	
1961	EN15984	0.150		0.53	
1964					
6142	EN15984	0.143		-0.37	
6203	UOP539	0.13		-2.04	
6369	ISO17025	0.1500		0.53	
6404	EN15984	0.149		0.40	
9008					
	normality	suspect			
	n	15			
	outliers	1			
	mean (n)	0.1459			
	st.dev. (n)	0.00714			
	R(calc.)	0.0200			
	st.dev.(Horwitz)	0.00780			
	R(Horwitz)	0.0218			
	. ,				





# Determination of 1-Butene on sample #22015; results in %mol/mol

lab	method	value	mark	z(targ)	remar	ks						
444	EN15984	0.135	С	-2.06	first re	ported: (	).167					
446												
1026	EN15984	0.1482	С	0.39	first re	ported: (	).148333	333				
1040	EN15984	0.14		-1.13								
1062	EN15984	0.1442		-0.35								
1069	UOP539	0.1513		0.96								
1081		0.141		-0.95								
1140	D7833	0.148		0.35								
1528	UOP539	0.15	С	0.72	first re	ported: 0	).119					
1635	UOP539	0	G(0.01)	-27.05								
1737		0.15		0.72								
1741	UOP539	0.16		2.57								
1961	EN15984	0.151		0.90								
1964												
6142	EN15984	0.144		-0.39								
6203	UOP539	0.13		-2.98								
6369	ISO17025	0.1500		0.72								
6404	EN15984	0.149		0.53								
9008												
	normality	OK										
	n	15										
	outliers	1										
	mean (n)	0.1461										
	st.dev. (n)	0.00737										
	R(calc.)	0.0206										
	st.dev.(EN15984:22	) 0.00540										
	R(EN15984:22)	0.0151			applica	ation ran	ge: 0.50-	-2.00				
0.165 T												
0.16 -												Δ
0.155												
0.15						•	۵	Δ	۵	۵	Δ	
0.145			A A	Δ	Δ	_						
0.14	۵	۵	-									
0.135	Δ											
0.13	δ											
0.125 -												
0.12												
1635	6203 444 1040	1081	6142	1140	1026	6404	1528	1737	6369	1961	1069	1741



## Determination of iso-Butene on sample #22015; results in %mol/mol

			-		
lab	method	value	mark	z(targ)	remarks
444	EN15984	0.088	С	-1.87	first reported: 0.109
446					
1026	EN15984	0.0956	С	-0.51	first reported: 0.097
1040	EN15984	0.10		0.28	
1062	EN15984	0.0955		-0.53	
1069	UOP539	0.1014		0.53	
1081					
1140	D7833	0.098	С	-0.08	first reported: 0.099
1528	UOP539	0.10	С	0.28	first reported: 0.082
1635	UOP539	0	G(0.01)	-17.64	
1737		0.10		0.28	
1741	UOP539	0.11		2.07	
1961	EN15984	0.101		0.46	
1964					
6142					
6203	UOP539	0.09		-1.51	
6369	ISO17025	0.1014		0.53	
6404	EN15984	0.099		0.10	
9008					
	normality	suspect			
	n	13			
	outliers	1			
	mean (n)	0.0985			
	st.dev. (n)	0.00550			
	R(calc.)	0.0154			
	st.dev.(Horwitz)	0.00558			
	R(Horwitz)	0.0156			
<sup>0.12</sup> T					





# Determination of cis-2-Butene on sample #22015; results in %mol/mol

	•	-			-
lab	method	value	mark	z(targ)	remarks
444	EN15984	0.09	С	-1.33	first reported: 0.119
446					
1026	EN15984	0.0967	С	-0.12	first reported: 0.09795
1040	EN15984	0.09		-1.33	
1062	EN15984	0.0936		-0.68	
1069	UOP539	0.1006		0.58	
1081		0.097		-0.07	
1140	D7833	0.095	С	-0.43	first reported: 0.096
1528	UOP539	0.10	С	0.47	first reported: 0.076
1635	UOP539	0.22	G(0.01)	22.17	
1737		0.10	. ,	0.47	
1741	UOP539	0.11		2.28	
1961	EN15984	0.100		0.47	
1964					
6142					
6203	UOP539	0.09		-1.33	
6369	ISO17025	0.1014		0.73	
6404	EN15984	0.099		0.29	
9008					
	normality	OK			
	n	14			
	outliers	1			
	mean (n)	0.0974			
	st.dev. (n)	0.00550			
	R(calc.)	0.0154			
	st.dev.(Horwitz)	0.00553			
	R(Horwitz)	0.0155			
	. ,				





## Determination of iso-Pentane on sample #22015; results in %mol/mol

lah	method	valuo	mark	z(tara)	romarks
444	EN15084	0 172	main	_1 04	Tomuno
444				-1.04	
1026	EN15984	0 1826	C	2 15	first reported: 0 18385
1040	EN15984	0.17	U	-1.64	
1062	EN15984	0.1686		-2.07	
1069	UOP539	0.2335	G(0.01)	17.48	
1081		0.166	( )	-2.85	
1140	D7833	0.178		0.77	
1528	UOP539	0.18	С	1.37	first reported: 0.140
1635	UOP539	0.16	ex	-4.66	test result excluded, see paragraph 4.1
1737					
1741	UOP539	0.21	G(0.01)	10.40	
1961	EN15984	0.179		1.07	
1964	In house	0.1799		1.34	
6142	EN15984	0.179		1.07	
6203	UOP539	0.14	G(0.01)	-10.68	
6369	ISO17025	0.1724		-0.92	
6404	EN15984	0.178		0.77	
9008					
	normality	OK			
	normality	12			
	n outliers	12 3 (+1ev)			
	mean (n)	0 1755			
	st dev (n)	0.00537			
	R(calc.)	0.00007			
	st dev (FN15984·22)	0.00332			
	R(EN15984:22)	0.0093			application range: 0.20-2.10
	( ····,				
0.24 -					
0.24					x
0.20					





# Determination of n-Pentane on sample #22015; results in %mol/mol

	lab	method		value		mark	z(1	targ)	remark	s						
	444	EN15984		0.152		С	-	0.42	first rep	orted: 0	.151					
	446															
	1026	EN15984		0.162		С		0.61	first rep	orted: 0	.163566	667				
	1040	EN15984		0.14			-	1.65								
	1062	EN15984		0.147	5		-	0.88								
	1069	UOP539		0.214	0	DG(0.05)		5.97								
	1081	5-000		0.148			-	0.83								
	1140	D7833		0.151		0	-	0.52	<b>c</b> .		100					
	1528	UOP539		0.16		C		0.41	first rep	orted: 0	.130					
	1635	00P539		0		G(0.01)	-1	6.08								
	1/3/			0.16				0.41								
	1/41	UUP539		0.20		DG(0.05)		4.53								
	1901	EN 10904		0.101	0			0.51								
	1904 6140	EN15094		0.1/5	9			∠.05 ∩ / 1								
	6203	LIOD520		0.100		C(0.05)		2 71								
	6360	19017025		0.12	1	G(0.03)		0.41								
	6404	EN15084		0.152	1		-	0.41								
	9008	LIN10004														
	0000															
		normality		suspe	ect											
		n		13 ່												
		outliers		4												
		mean (n)		0.156	0											
		st.dev. (n)		0.008	96											
		R(calc.)		0.025	1											
		st.dev.(EN1598	4:22)	0.009	70											
		R(EN15984:22)		0.027	2				applicat	ion rang	ge: 0.10	– 0.35				
<sup>0.22</sup> T																*
																*
0.2 -															ж	
0.18 -																
														Δ		
0.16 -								۵	۵	۵	۵	Δ	۵			
0.14			۵	Δ	۵	Δ	Δ									
0.14		Δ														
0.12		*														
0.1 -	635	040 203	062	081	140	444	369	404	528	737	142	961	026	964	741	690
	-	- v	-	-	-		9	ø	-	-	9	-	-	-	-	-



# Determination of Carbon content on sample #22015; results in g/100g

lab	method	value	mark	z(targ)	remarks
444	EN15984	67.74	С	0.16	first reported: 67.86
446			-		
1026	EN15984	67.03		-0.76	
1040	EN15984	67.6		-0.02	
1062	EN15984	67.71		0.13	
1069	EN15984	68.11		0.64	
1081					
1140					
1528	UOP539	67.79	С	0.23	first reported: 67.732
1635					
1737	EN15984	67.73		0.15	
1741					
1961	EN15984	67.33		-0.37	
1964	In house	68.024		0.53	
6142	EN15984	66.025	G(0.05)	-2.06	
6203	EN15984	67.07		-0.70	
6369					
6404	EN15984	67.61		0.00	
9008					
	hormality	UK 11			
	n autliana	11			
	mean (n)	07.013			
	St.dev. (n)	0.3466			
	r(UdlC.)	0.971			
	SI.UEV.(EIN 15984.22)	0.7714			
	R(EN13904.22)	2.10			





# Determination of Lower Calorific Value on sample #22015; results in kJ/100g

lab	method	value	mark	z(targ)	remarks
444	EN15984	4956.84	С	0.66	first reported: 4953.85
446					
1026					
1040	EN15984	4948.0	С	0.45	first reported: 494.8
1062	EN15984	4956.69		0.65	
1069	EN15984	4946	С	0.40	first reported: 494600
1081					
1140					
1528	ISO6976	4954.9	С	0.61	first reported: 49.73
1635					
1737	EN15984	4954.60		0.60	
1741					
1961	EN15984	4911.80		-0.39	
1964	In house	4930.507		0.04	
6142	EN15984	4835.58		-2.17	
6203	ISO6976	4863.31		-1.53	
6369					
6404	EN15984	4957.54		0.67	
9008					
	normality	suspect			
	n	11			
	outliers	0			
	mean (n)	4928.706			
	st.dev. (n)	42.0382			
	R(calc.)	117.707			
	st.dev.(EN15984:22)	42.8214			
	R(EN15984:22)	119.90			





#### Other reported test results

lab	Hydrogen sulfide	Ethyne	Propyne	Propadiene	1,3-Butadiene	Other *)
444	0	0	0	0	0	0
446						
1026	0					0
1040	0.00	0.00	0.00	0.00	0.00	0.00
1062	0.000	0.000	0.000	0.000	0.000	0.0000
1069		0.00	0.00	0.00	0.00	0.00
1081		0 C	0 C			
1140		0.000		0.000	0.000	0.000
1528						
1635	0	0			0	
1737		<0,01	<0,01	<0,01	<0,01	
1741						
1961	0.000	0.000		0.000	0.000	0.000
1964						
6142						
6203	0	0	0	0	0	0
6369						
6404	0.000	0.000	0.000	0.000	0.000	0.000
9008						

\*) Other components with 5 or more carbon atoms, excluding iso-Butane and Pentanes (C5+)

Lab 1081 first reported for Ethyne: 1.975, for Propyne: 1.569

#### Number of participants per country

1 lab in AUSTRIA 2 labs in BELGIUM 1 lab in CROATIA 1 lab in FINLAND 1 lab in FRANCE 1 lab in GERMANY 1 lab in IRELAND 1 lab in KUWAIT 2 labs in NETHERLANDS 2 labs in ROMANIA 1 lab in SERBIA 1 lab in SWEDEN

4 labs in UNITED KINGDOM

#### Abbreviations

С	= final test 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
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
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from the statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Material Safety Data Sheet

## Literature

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
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- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 7 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 8 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
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- 11 W. Horwitz and R. Albert, J. AOAC Int, <u>79.3</u>, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, <u>25(2)</u>, 165-172, (1983)