# **Results of Proficiency Test** Natural Gas Analysis March 2015

Organised by: Institute for Interlaboratory Studies Spijkenisse, the Netherlands

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Report: iis15S01M

May 2015

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#### 1 Introduction

Since 2009, the Institute organizes a proficiency scheme for Natural Gas (composition only). During the annual proficiency testing program 2014/2015, it was decided to continue the proficiency test for the analysis of Natural Gas.

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

In this interlaboratory study 50 laboratories in 30 different countries have participated. See appendix 3 for the number of participants per country. In this report, the results of the 2015 Natural Gas proficiency test are presented and discussed. This report is also electronically available through the iis internet site <a href="https://www.iisnl.com">www.iisnl.com</a>.

#### 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. To optimise the costs for the participating laboratories, it was decided to prepare one natural gas mixture. Samples were divided over a batch of 53 cylinders. The cylinder size is a cost-effective one-litre cylinder. Each cylinder was uniquely numbered. The limited cylinder size is chosen to optimise transport and handling costs. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

# 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010 (R007). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Also customer's satisfaction is measured on regular basis by the distribution of questionnaires.

EffecTech is an accredited provider of proficiency testing schemes under the requirements of ISO/IEC17043:2010 by UKAS (no. 4719).

#### 2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol is also electronically available through the iis internet site <a href="www.iisnl.com">www.iisnl.com</a>.

## 2.3 CONFIDENTIALITY STATEMENT

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

#### 2.4 SAMPLES

In this proficiency test one gas sample was used. The necessary one litre cylinders with artificial natural gas mixture was prepared and tested for homogeneity by EffecTech (Uttoxeter, United Kingdom) in conformance with ISO Guide 35: 2006 and ISO/IEC17043:2010.

One batch of 53 cylinders was prepared (job 15/0057) starting in January, 2015. Each cylinder was uniquely numbered. Every cylinder in the batch was analysed using eight replicate measurements. The within bottle and between bottle variations were then assessed in accordance with ISO Guide 35:2006 (Annex A.1). This procedure showed that the between bottle variations were all small compared to the uncertainties on the reference values on each component. Hence, a single reference value could be safely assigned to the entire batch of samples.

The repeatability values (r) were calculated per component by multiplication of the respective standard deviation by 2.8. Subsequently, the calculated repeatabilities were compared with 0.3 times the reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

| Parameter      | r (abs, observed)<br>in %mol/mol | 0.3xR (abs, ISO6974-3)<br>in %mol/lmol |
|----------------|----------------------------------|--|
| Methane        | 0.0046                           | 0.0553                                 |
| Ethane         | 0.0031                           | 0.0271                                 |
| Propane        | 0.0012                           | 0.0136                                 |
| iso-Butane     | 0.0003                           | 0.0027                                 |
| n-Butane       | 0.0004                           | 0.0036                                 |
| Carbon dioxide | 0.0008                           | 0.0072                                 |
| Nitrogen       | 0.0012                           | 0.0226                                 |

Table 1: evaluation of homogeneity test results against ISO6974-3 requirements

From the above table it is clear that all observed repeatability values are far less than 0.3 times the respective reproducibility of the reference method ISO6974-3.

Therefore, the homogeneity of the prepared cylinders was assumed.

To each of the participating laboratories one 1L gas cylinder was sent on March 11, 2015.

#### 2.5 STABILITY OF THE SAMPLES

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

#### 2.6 ANALYSES

The participants were asked to determine: Methane, Ethane, Propane, n-Butane, iso-Butane, Carbon dioxide, Nitrogen, Caloric Value (sup), Density, Relative Density and Wobbe index. To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The detailed report form and the letter of instructions were also made available for download on the iis website www.iisnl.com. A SDS and a form to confirm receipt of the sample was added to the sample package.

#### 3 RESULTS

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

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

#### 3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 (1986 and 1994, lit.5 and 6) the original results per determination were submitted to Dixon's and/or 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 (ref. 16). 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

#### 3.2 GRAPHICS

In order to 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 analysis results are plotted. The corresponding laboratory numbers are under the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a 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. (see appendix 3; nos.13 and 14). 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. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8.

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare.

Therefore the usual interpretation of z-scores is as follows:

```
|z| < 1 good

1 < |z| < 2 satisfactory

2 < |z| < 3 questionable

3 < |z| unsatisfactory
```

## 4 EVALUATION

In this proficiency test several problems were encountered with customs clearance. In total sixteen laboratories reported results after the final reporting date and four participants were not able to report any test results. In total 47 participants reported 532 numerical results. Observed were 33 outlying results, which is 6.2% of the numerical results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

#### 4.1 EVALUATION PER TEST/COMPONENT

In this section the results are discussed per component. The methods that were used by the participating laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

Not all original data sets proved to have a normal Gaussian distribution (see Appendix 1, normality). When the normality is "not OK" or "suspect", the statistical evaluation of these data sets should be used with due care.

Three laboratories reported deviating results on the gas composition values. At least four of the seven test results were statistical outliers for each of the laboratories 1069, 1106 and 1580. As the seven test results are not independent, it was decided not to use any of the reported results of these laboratories for the statistical evaluation. Also the reported results of these three laboratories were excluded for the statistical evaluation of the Caloric Value (sup), Density, Relative Density and Wobbe index, since these values were calculated from the measured gas composition.

All laboratories reported normalized test results as requested.

#### Methane:

The determination of this component was very problematic. Two statistical outliers were detected and two test results were excluded. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ISO6974-3:2000, nor at all in agreement with ASTM D1945:2014.

# Ethane:

The determination of this component was problematic for a number of laboratories. Four statistical outliers were detected. However, the calculated reproducibility after rejection of statistical outliers is in agreement with the requirements of ISO6974-3:2000 and ASTM D1945:2014.

## Propane:

The determination of this component may be problematic for a number of laboratories, depending on the test method used by the laboratory. Four statistical outliers were detected. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the strict requirements of ISO6974-3:2000. However the calculated reproducibility is in agreement with the requirements of ASTM D1945:2014.

# i-Butane:

The determination of this component was problematic for a number of laboratories. Four statistical outliers were detected. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of both ISO6974-3:2000 and ASTM D1945:2014.

# n-Butane:

The determination of this component may be problematic for a number of laboratories, depending on the test method used by the laboratory. Five statistical outliers were detected and one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ISO6974-3:2000, but in good agreement with the requirements of ASTM D1945:2014.

# Carbon Dioxide:

The determination of this component may be problematic for a number of laboratories, depending on the test method used by the laboratory. Three statistical outliers were detected and two test results were excluded. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ISO6974-3:2000, but in agreement with the requirements of ASTM D1945:2014.

## Nitrogen:

The determination of this component was very problematic. Five statistical outliers were detected and two test results were excluded. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ISO6974-3:2000 and also not at all in agreement with the requirements of ASTM D1945:2014.

# Calculated parameters, general remark:

In this PT, the calculated parameters were reported for two combustion temperatures (15 and 25°C) for real gas. The number of participants with results for 15°C and 25°C varied between 22 and 29.

## Caloric Value:

The calculation at combustion temperature 25°C/metering temperature 0°C may be problematic for a number of laboratories. Two statistical outliers were found and two results were excluded. Eight results were marked as calculation errors. The spread for real gas was exactly the same to the previously observed spread in iis14S01M.

The calculation at combustion temperature 15°C/metering temperature 15°C may be problematic for a number of laboratories. Three statistical outliers were found and two results were excluded. Nine results were marked as calculation errors. However, the spread for real gas was smaller than the previously observed spread in iis14S01M.

## Density:

The calculation at combustion temperature 25°C/metering temperature 0°C may be problematic for a number of laboratories. One statistical outlier was found and two results were excluded. Four results were marked as calculation errors. However, the spread for real gas was smaller than the previously observed spread in iis14S01M.

The calculation at combustion temperature 15°C/metering temperature 15°C may be problematic for a number of laboratories. Two statistical outliers were found and two results were excluded. Four results were marked as calculation errors. However, the spread for real gas was smaller than the previously observed spread in iis14S01M.

#### Relative density:

The calculation at combustion temperature 25°C/metering temperature 0°C appeared not to be problematic. No statistical outliers were found, but two results were excluded. Two calculation errors were observed. The spread for real gas was somewhat larger than the previously observed spread in iis14S01M.

The calculation at combustion temperature 15°C/metering temperature 15°C appeared not to be problematic. No statistical outliers were found, but two results were excluded. Three calculation errors were observed. The spread for real gas is the somewhat larger than the previously observed spread in iis14S01M.

## Wobbe index:

The calculation at combustion temperature 25°C/metering temperature 0°C may be problematic for a number of laboratories. Two statistical outliers were found and two results were excluded. Seven results were marked as calculation errors. However, the spread for real gas was smaller than the previously observed spread in iis14S01M.

The calculation at combustion temperature 15°C/metering temperature 15°C may be problematic for a number of laboratories. One statistical outlier was found and two results were excluded. Two results were marked as calculation errors. However, the spread for real gas smaller than the previously observed spread in iis14S01M.

# 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

The average results per component, observed reproducibilities and target reproducibilities, derived from the standard methods ISO6974-3 and ASTM D1945 are compared in the next table.

|                | unit     | n  | cons. value | 2.8 * sd | R(ISO6974-3) | R(D1945) |
|----------------|----------|----|-------------|----------|--------------|----------|
| Methane        | %mol/mol | 42 | 92.224      | 0.404    | 0.184        | 0.150    |
| Ethane         | %mol/mol | 43 | 3.026       | 0.093    | 0.091        | 0.100    |
| Propane        | %mol/mol | 43 | 1.513       | 0.055    | 0.045        | 0.100    |
| iso-Butane     | %mol/mol | 43 | 0.151       | 0.010    | 0.009        | 0.070    |
| n-Butane       | %mol/mol | 41 | 0.202       | 0.014    | 0.012        | 0.070    |
| Carbon dioxide | %mol/mol | 41 | 0.399       | 0.038    | 0.024        | 0.070    |
| Nitrogen       | %mol/mol | 40 | 2.506       | 0.194    | 0.075        | 0.100    |

Table 2: Performance of the group in comparison with the target reproducibilities

Without further statistical calculations it can be concluded that for several components there is not a good compliance of the group of participating laboratories with the relevant standard. The problematic components have been discussed in paragraph 4.1.

The average results per calculation for the real gas, observed reproducibilities are compared in table 3 and 4.

| Combustion temperature 25°C, metering temperature 0°C, real gas |                   |    |             |          |  |  |  |
|---|-------------------|----|-------------|----------|--|--|--|
| Property  | unit              | n  | cons. value | 2.8 * sd |  |  |  |
| Caloric Value   | MJ/m <sup>3</sup> | 18 | 40.826      | 0.186    |  |  |  |
| Density   | kg/m <sup>3</sup> | 20 | 0.781       | 0.005    |  |  |  |
| Relative Density  |                   | 20 | 0.604       | 0.004    |  |  |  |
| Wobbe Index   | MJ/m <sup>3</sup> | 18 | 52.527      | 0.218    |  |  |  |

Table 3: Performance of the group for combustion temperature of 25°C, real gas

| Combustion temperature 15°C, metering temperature 15°C, real gas |                   |    |             |          |  |  |  |
|--|-------------------|----|-------------|----------|--|--|--|
| Property   | unit              | n  | cons. value | 2.8 * sd |  |  |  |
| Caloric Value  | MJ/m <sup>3</sup> | 24 | 38.709      | 0.102    |  |  |  |
| Density  | kg/m³             | 25 | 0.740       | 0.003    |  |  |  |
| Relative Density   |                   | 28 | 0.604       | 0.003    |  |  |  |
| Wobbe Index  | MJ/m <sup>3</sup> | 25 | 49.838      | 0.216    |  |  |  |

Table 4: Performance of the group for combustion temperature of 15°C, real gas

# 4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2015 WITH PREVIOUS PTS

|                            | 2015 | 2014 | 2013 | 2012 |
|----------------------------|------|------|------|------|
| Number of reporting labs   | 47   | 38   | 33   | 34   |
| Number of results reported | 532  | 600  | 466  | 475  |
| Statistical outliers       | 33   | 38   | 29   | 23   |
| Percentage outliers        | 6.2% | 6.5% | 6.2% | 4.8% |

Table 5: Comparison with previous proficiency tests

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

The performances of the determinations in the proficiency tests for Natural Gas were compared against the requirements of the two often used standard test methods. See the overview in the following table:

|                | 2015<br>ISO6974-3 | 2015<br>D1945 | 2014<br>ISO6974-3 | 2014<br>D1945 | 2013<br>ISO6974-3 | 2013<br>D1945 | 2012<br>ISO6974-3 | 2012<br>D1945 |
|----------------|-------------------|---------------|-------------------|---------------|-------------------|---------------|-------------------|---------------|
| Methane        |                   |               |                   |               |                   |               |                   |               |
| Ethane         | +/-               | +             | ++                | +             | +                 | -             | -                 | -             |
| Propane        | -                 | ++            | -                 | ++            | -                 | ++            | -                 | ++            |
| iso-Butane     | +/-               | ++            | ++                | ++            | -                 | ++            | -                 | ++            |
| n-Butane       | +/-               | ++            | +/-               | ++            |                   | ++            | -                 | ++            |
| Carbon dioxide |                   | ++            |                   |               |                   | +/-           |                   | ++            |
| Nitrogen       |                   |               |                   |               |                   |               |                   |               |

Table 6: comparison of observed precision with precision of ISO6974-3 / ASTM D1945

From the above table it is clear that the performance of the group of participating laboratories did not improve.

The following performance categories were used:

- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- -- : group performed much worse than the standard

#### 4.4 DISCUSSION

Many of the observed reproducibilities are larger than the reproducibility requirements of ISO6974-3 and therefore it had to be concluded that no improvement was observed since the 2010 PT for Natural Gas and that the determination of the composition of Natural Gas was still problematic for a significant number of participating laboratories.

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

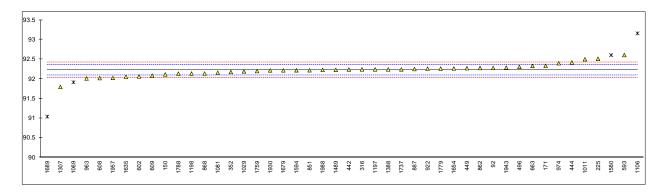
| Parameter             | Average values by<br>EffecTech<br>in %mol/mol | Consensus values from participants results in %mol/mol | Absolute differences<br>in %mol/mol |
|-----------------------|---|--|-------------------------------------|
| Methane               | 92.2166                                       | 92.2239  | 0.0073                              |
| Ethane                | 3.0134  | 3.0256   | 0.0122                              |
| Propane               | 1.5118  | 1.5130   | 0.0012                              |
| iso-Butane            | 0.1503  | 0.1512   | 0.0009                              |
| n-Butane              | n-Butane 0.2005                               |  | 0.0019                              |
| Carbon dioxide 0.4006 |   | 0.3992   | 0.0014                              |
| Nitrogen              | 2.5068  | 2.5057   | 0.0011                              |

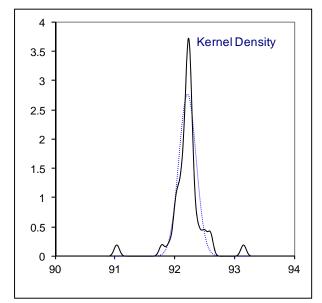
Table 7: comparison of consensus values with values determined by the supplier EffecTech

From the comparison in table 7 it is clear that the consensus values as determined in this PT are all very well in line with the values as determined during the preparation of the gas cylinders.

**APPENDIX 1**Determination of Methane on sample #15030; results in %mol/mol

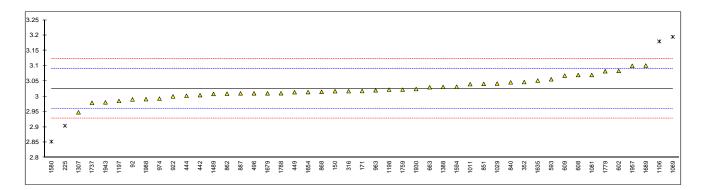
|      | •               | •           | •         |         | suits in %moi/moi                 |
|------|-----------------|-------------|-----------|---------|-----------------------------------|
| lab  | method          | value       | mark      | z(targ) | remarks                           |
| 92   | GPA2286         | 92.28       |           | 0.85    |                                   |
| 150  | D1945           | 92.114      |           | -1.67   |                                   |
| 171  | D1945           | 92.33398    |           | 1.67    |                                   |
| 225  | D1945           | 92.512      |           | 4.37    |                                   |
| 316  | ISO6974-3       | 92.242      |           | 0.28    |                                   |
| 352  | ISO6974-3       | 92.1745     |           | -0.75   |                                   |
| 442  | D1945           | 92.2404     | _         | 0.25    |                                   |
| 444  | D1945           | 92.419      | С         | 2.96    | First reported 92.380             |
| 449  | D1945           | 92.2722     |           | 0.73    |                                   |
| 496  | DIN15984        | 92.306      |           | 1.25    |                                   |
| 593  | D1945           | 92.607      |           | 5.82    |                                   |
| 602  | GPA2261         | 92.0625     |           | -2.45   |                                   |
| 608  | GPA2261         | 92.02       |           | -3.09   |                                   |
| 609  | GPA2261         | 92.0835     |           | -2.13   |                                   |
| 663  | D1945           | 92.330      |           | 1.61    |                                   |
| 840  |                 |             |           |         |                                   |
| 851  | GPA2261         | 92.219      |           | -0.07   |                                   |
| 862  | GPA2261         | 92.277      |           | 0.81    |                                   |
| 868  | GPA2261         | 92.139      |           | -1.29   |                                   |
| 887  | D1945           | 92.25       |           | 0.40    |                                   |
| 922  | D1945           | 92.26       |           | 0.55    |                                   |
| 963  | D1945           | 92.010      |           | -3.25   |                                   |
| 974  | ISO6974-3       | 92.3992     |           | 2.66    |                                   |
| 1011 | UOP539          | 92.50       |           | 4.19    |                                   |
| 1029 | D1945           | 92.192      |           | -0.48   |                                   |
| 1069 | UOP539          | 91.915      | ex        | -4.69   | See §4.1                          |
| 1081 |                 | 92.158      |           | -1.00   |                                   |
| 1095 |                 |             |           |         |                                   |
| 1106 | ISO6976         | 93.160      | R(0.01)   | 14.21   |                                   |
| 1197 | D1945           | 92.242      |           | 0.28    |                                   |
| 1198 | D1945           | 92.137      |           | -1.32   |                                   |
| 1200 |                 |             |           |         |                                   |
| 1307 | in house        | 91.796      |           | -6.50   |                                   |
| 1388 | GPA2261         | 92.2421     |           | 0.28    |                                   |
| 1489 | ISO6974-3       | 92.231      |           | 0.11    |                                   |
| 1580 | GB/T13610       | 92.605      | ex        | 5.79    | See §4.1                          |
| 1594 | GPA2261         | 92.2182     |           | -0.09   |                                   |
| 1635 | D1945           | 92.054      |           | -2.58   |                                   |
| 1654 | D1945           | 92.266      |           | 0.64    |                                   |
| 1679 | ISO6974-3       | 92.217      |           | -0.10   |                                   |
| 1689 | GB/T13610       | 91.040      | C,R(0.01) | -17.97  | First reported 90.907             |
| 1737 | in house        | 92.243      |           | 0.29    |                                   |
| 1759 | ISO6974-5       | 92.199      |           | -0.38   |                                   |
| 1779 | GPA2261         | 92.2635     |           | 0.60    |                                   |
| 1788 | D7833           | 92.1349     |           | -1.35   |                                   |
| 1892 | 1000074.0       | 00.0407     |           | 0.44    |                                   |
| 1930 | ISO6974-6       | 92.2167     |           | -0.11   |                                   |
| 1943 | ISO6974-3       | 92.2876     |           | 0.97    |                                   |
| 1957 | GPA2261         | 92.0250     |           | -3.02   |                                   |
| 1960 | 1000074.0       | 00.0076     |           | 0.06    |                                   |
| 1988 | ISO6974-6       | 92.2276     |           | 0.06    |                                   |
|      | normality       | suspect     |           |         |                                   |
|      | n               | 42          |           |         |                                   |
|      | outliers        | 2 (+ 2excl) |           |         |                                   |
|      | mean (n)        | 92.2239     |           |         |                                   |
|      | st.dev. (n)     | 0.14417     |           |         |                                   |
|      | R(calc.)        | 0.4037      |           |         |                                   |
|      | R(ISO6974-3:00) | 0.1844      |           |         | Compare R(ASTM D1945:14) = 0.1500 |
|      |                 |             |           |         |                                   |

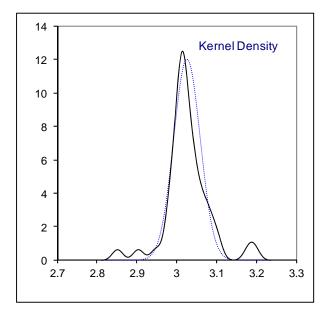




# Determination of Ethane on sample #15030; results in %mol/mol

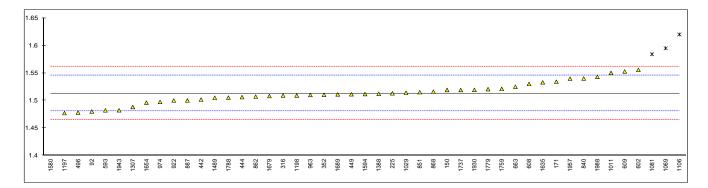
| loh          | mothod                  | value            | mark       | 7(tora)        | romarke                           |
|--------------|-------------------------|------------------|------------|----------------|-----------------------------------|
| lab          | method<br>GRA2286       | value            | mark       | z(targ)        | remarks                           |
| 92<br>150    | GPA2286<br>D1945        | 2.99<br>3.017    |            | -1.10<br>-0.27 |                                   |
| 171          | D1945                   | 3.017515         |            | -0.27          |                                   |
| 225          | D1945                   | 2.904            | C,R(0.05)  | -3.75          | First reported 2.915              |
| 316          | ISO6974-3               | 3.017            | 0,11(0.00) | -0.27          | That reported 2.515               |
| 352          | ISO6974-3               | 3.0468           |            | 0.65           |                                   |
| 442          | D1945                   | 3.0040           |            | -0.67          |                                   |
| 444          | D1945                   | 3.002            | С          | -0.73          | First reported 3.017              |
| 449          | D1945                   | 3.0137           | _          | -0.37          |                                   |
| 496          | DIN15984                | 3.010            |            | -0.48          |                                   |
| 593          | D1945                   | 3.056            |            | 0.94           |                                   |
| 602          | GPA2261                 | 3.0834           |            | 1.78           |                                   |
| 608          | GPA2261                 | 3.07             |            | 1.37           |                                   |
| 609          | GPA2261                 | 3.0680           |            | 1.31           |                                   |
| 663          | D1945                   | 3.030            |            | 0.13           |                                   |
| 840          | D1945                   | 3.046            |            | 0.63           |                                   |
| 851          | GPA2261                 | 3.041            |            | 0.47           |                                   |
| 862          | GPA2261                 | 3.009            |            | -0.51          |                                   |
| 868          | GPA2261                 | 3.015            |            | -0.33          |                                   |
| 887          | D1945                   | 3.01             |            | -0.48          |                                   |
| 922          | D1945                   | 3.00             |            | -0.79          |                                   |
| 963          | D1945                   | 3.020            |            | -0.17          |                                   |
| 974          | ISO6974-3               | 2.9926           |            | -1.02          |                                   |
| 1011<br>1029 | UOP539<br>D1945         | 3.04<br>3.042    |            | 0.44<br>0.50   |                                   |
| 1029         | UOP539                  | 3.195            | R(0.01)    | 5.22           |                                   |
| 1081         | 001339                  | 3.070            | K(0.01)    | 1.37           |                                   |
| 1095         |                         |                  |            |                |                                   |
| 1106         | ISO6976                 | 3.180            | R(0.01)    | 4.76           |                                   |
| 1197         | D1945                   | 2.985            | (0.0 . )   | -1.25          |                                   |
| 1198         | D1945                   | 3.022            |            | -0.11          |                                   |
| 1200         |                         |                  |            |                |                                   |
| 1307         | in house                | 2.948            |            | -2.40          |                                   |
| 1388         | GPA2261                 | 3.0310           |            | 0.17           |                                   |
| 1489         | ISO6974-3               | 3.008            |            | -0.54          |                                   |
| 1580         | GB/T13610               | 2.852            | R(0.01)    | -5.36          |                                   |
| 1594         | GPA2261                 | 3.0316           |            | 0.18           |                                   |
| 1635         | D1945                   | 3.052            |            | 0.81           |                                   |
| 1654         | D1945                   | 3.014            |            | -0.36          |                                   |
| 1679         | ISO6974-3               | 3.010            |            | -0.48          |                                   |
| 1689         | GB/T13610               | 3.101            |            | 2.32           |                                   |
| 1737         | in house                | 2.979            |            | -1.44          |                                   |
| 1759         | ISO6974-5               | 3.022            |            | -0.11          |                                   |
| 1779<br>1788 | GPA2261<br>D7833        | 3.0824<br>3.0103 |            | 1.75<br>-0.47  |                                   |
| 1892         | D1033                   | 3.0103           |            | -0.47          |                                   |
| 1930         | ISO6974-6               | 3.0240           |            | -0.05          |                                   |
| 1943         | ISO6974-3               | 2.9805           |            | -1.39          |                                   |
| 1957         | GPA2261                 | 3.1000           |            | 2.29           |                                   |
| 1960         | - · · · <del> ·</del> · |                  |            |                |                                   |
| 1988         | ISO6974-6               | 2.9908           |            | -1.07          |                                   |
|              |                         |                  |            |                |                                   |
|              | normality               | OK               |            |                |                                   |
|              | n                       | 43               |            |                |                                   |
|              | outliers                | 4                |            |                |                                   |
|              | mean (n)                | 3.0256           |            |                |                                   |
|              | st.dev. (n)             | 0.03315          |            |                |                                   |
|              | R(calc.)                | 0.0928           |            |                | Compare D(ACTM D4045:44) 0 4000   |
|              | R(ISO6974-3:00)         | 0.0908           |            |                | Compare R(ASTM D1945:14) = 0.1000 |

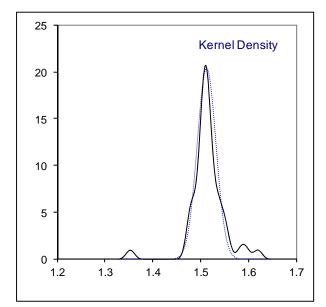




# Determination of Propane on sample #15030; results in %mol/mol

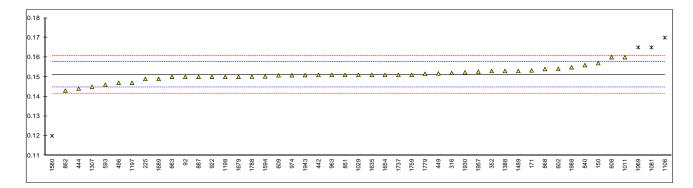
| lab          | method            | value           | mark     | z(targ)        | remarks                           |
|--------------|-------------------|-----------------|----------|----------------|-----------------------------------|
| 92           | GPA2286           | 1.48            |          | -2.03          |                                   |
| 150          | D1945             | 1.519           | С        | 0.37           | First reported 1.589              |
| 171          | D1945             | 1.53406         |          | 1.30           |                                   |
| 225          | D1945             | 1.513           |          | 0.00           |                                   |
| 316          | ISO6974-3         | 1.509           |          | -0.24          |                                   |
| 352          | ISO6974-3         | 1.5103          |          | -0.16          |                                   |
| 442          | D1945             | 1.5016          |          | -0.70          |                                   |
| 444          | D1945             | 1.506           |          | -0.43          |                                   |
| 449<br>496   | D1945             | 1.5112<br>1.478 |          | -0.11          |                                   |
| 593          | DIN15984<br>D1945 | 1.476           |          | -2.16<br>-1.91 |                                   |
| 602          | GPA2261           | 1.5556          |          | 2.63           |                                   |
| 608          | GPA2261           | 1.53            |          | 1.05           |                                   |
| 609          | GPA2261           | 1.5528          |          | 2.46           |                                   |
| 663          | D1945             | 1.525           |          | 0.74           |                                   |
| 840          | D1945             | 1.540           |          | 1.67           |                                   |
| 851          | GPA2261           | 1.515           |          | 0.13           |                                   |
| 862          | GPA2261           | 1.507           |          | -0.37          |                                   |
| 868          | GPA2261           | 1.516           |          | 0.19           |                                   |
| 887          | D1945             | 1.50            |          | -0.80          |                                   |
| 922          | D1945             | 1.50            |          | -0.80          |                                   |
| 963          | D1945             | 1.510           |          | -0.18          |                                   |
| 974          | ISO6974-3         | 1.4975          |          | -0.95          |                                   |
| 1011         | UOP539            | 1.55            |          | 2.28           |                                   |
| 1029         | D1945             | 1.514           | D(0.05)  | 0.06           |                                   |
| 1069         | UOP539            | 1.595           | R(0.05)  | 5.06           |                                   |
| 1081<br>1095 |                   | 1.584           | R(0.05)  | 4.38           |                                   |
| 11095        | ISO6976           | 1.620           | R(0.01)  | 6.60           |                                   |
| 1197         | D1945             | 1.477           | 11(0.01) | -2.22          |                                   |
| 1198         | D1945             | 1.509           |          | -0.24          |                                   |
| 1200         | 51010             |                 |          |                |                                   |
| 1307         | in house          | 1.488           |          | -1.54          |                                   |
| 1388         | GPA2261           | 1.5122          |          | -0.05          |                                   |
| 1489         | ISO6974-3         | 1.505           |          | -0.49          |                                   |
| 1580         | GB/T13610         | 1.354           | R(0.01)  | -9.81          |                                   |
| 1594         | GPA2261           | 1.5116          |          | -0.08          |                                   |
| 1635         | D1945             | 1.533           |          | 1.24           |                                   |
| 1654         | D1945             | 1.496           |          | -1.05          |                                   |
| 1679         | ISO6974-3         | 1.508           |          | -0.31          |                                   |
| 1689         | GB/T13610         | 1.511           |          | -0.12          |                                   |
| 1737         | in house          | 1.519           |          | 0.37           |                                   |
| 1759         | ISO6974-5         | 1.521           |          | 0.50           |                                   |
| 1779         | GPA2261           | 1.5205          |          | 0.46           |                                   |
| 1788         | D7833             | 1.5052          |          | -0.48          |                                   |
| 1892<br>1930 | ISO6974-6         | 1.5193          |          | 0.39           |                                   |
| 1943         | ISO6974-3         | 1.4821          |          | -1.90          |                                   |
| 1957         | GPA2261           | 1.5396          |          | 1.64           |                                   |
| 1960         | J. / LEU !        |                 |          |                |                                   |
| 1988         | ISO6974-6         | 1.5429          |          | 1.85           |                                   |
|              | -                 | -               |          |                |                                   |
|              | normality         | OK              |          |                |                                   |
|              | n                 | 43              |          |                |                                   |
|              | outliers          | 4               |          |                |                                   |
|              | mean (n)          | 1.5130          |          |                |                                   |
|              | st.dev. (n)       | 0.01956         |          |                |                                   |
|              | R(calc.)          | 0.0548          |          |                | O P(ACTIA BAGAS AA)               |
|              | R(ISO6974-3:00)   | 0.0454          |          |                | Compare R(ASTM D1945:14) = 0.1000 |

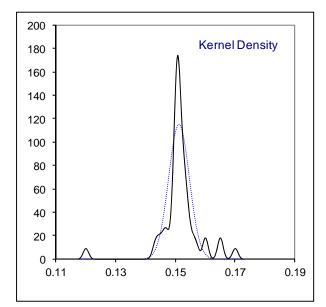




# Determination of iso-Butane on sample #15030; results in %mol/mol

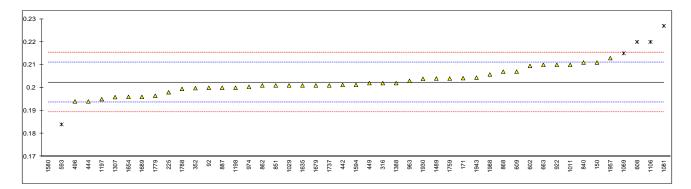
| lab          | method               | value           | mark     | z(targ)       | remarks  |
|--------------|----------------------|-----------------|----------|---------------|--|
| 92           | GPA2286              | 0.15            |          | -0.36         |  |
| 150          | D1945                | 0.157           |          | 1.81          |  |
| 171          | D1945                | 0.153321        |          | 0.67          |  |
| 225          | D1945                | 0.149           |          | -0.66         |  |
| 316          | ISO6974-3            | 0.1520          |          | 0.26          |  |
| 352          | ISO6974-3            | 0.1530          |          | 0.57          |  |
| 442          | D1945                | 0.1510          |          | -0.05         |  |
| 444          | D1945                | 0.144           |          | -2.21         |  |
| 449          | D1945                | 0.1518          |          | 0.20          |  |
| 496          | DIN15984             | 0.147           |          | -1.28         |  |
| 593          | D1945                | 0.146           |          | -1.59         |  |
| 602          | GPA2261              | 0.1541          |          | 0.91          |  |
| 608          | GPA2261              | 0.16            |          | 2.73          |  |
| 609          | GPA2261              | 0.1508          |          | -0.11         |  |
| 663<br>840   | D1945<br>D1945       | 0.150<br>0.156  |          | -0.36<br>1.50 |  |
| 851          | GPA2261              | 0.150           |          | -0.05         |  |
| 862          | GPA2261              | 0.143           |          | -2.52         |  |
| 868          | GPA2261              | 0.154           |          | 0.88          |  |
| 887          | D1945                | 0.15            |          | -0.36         |  |
| 922          | D1945                | 0.15            |          | -0.36         |  |
| 963          | D1945                | 0.151           |          | -0.05         |  |
| 974          | ISO6974-3            | 0.1508          |          | -0.11         |  |
| 1011         | UOP539               | 0.16            |          | 2.73          |  |
| 1029         | D1945                | 0.151           |          | -0.05         |  |
| 1069         | UOP539               | 0.165           | R(0.05)  | 4.28          |  |
| 1081         |                      | 0.165           | R(0.05)  | 4.28          |  |
| 1095         |                      |                 |          |               |  |
| 1106         | ISO6976              | 0.170           | R(0.05)  | 5.82          |  |
| 1197         | D1945                | 0.147           |          | -1.28         |  |
| 1198         | D1945                | 0.150           |          | -0.36         |  |
| 1200         | to become            | 0.445           |          | 4.00          |  |
| 1307         | in house             | 0.145           |          | -1.90         |  |
| 1388<br>1489 | GPA2261<br>ISO6974-3 | 0.1530<br>0.153 |          | 0.57<br>0.57  |  |
| 1580         | GB/T13610            | 0.133           | R(0.01)  | -9.62         |  |
| 1594         | GPA2261              | 0.1502          | C (0.01) | -0.29         | Result first reported as n-butane. Result was mixed-up |
| 1635         | D1945                | 0.151           | J        | -0.05         | Result mot reported do il butane. Result was mixed ap  |
| 1654         | D1945                | 0.151           |          | -0.05         |  |
| 1679         | ISO6974-3            | 0.150           |          | -0.36         |  |
| 1689         | GB/T13610            | 0.149           |          | -0.66         |  |
| 1737         | in house             | 0.151           |          | -0.05         |  |
| 1759         | ISO6974-5            | 0.151           |          | -0.05         |  |
| 1779         | GPA2261              | 0.1516          |          | 0.14          |  |
| 1788         | D7833                | 0.1501          |          | -0.32         |  |
| 1892         |                      |                 |          |               |  |
| 1930         | ISO6974-6            | 0.1523          |          | 0.35          |  |
| 1943         | ISO6974-3            | 0.1509          | _        | -0.08         |  |
| 1957         | GPA2261              | 0.1527          | С        | 0.48          | First reported 0.1671                                  |
| 1960         | 1000074.0            | 0.4540          |          |               |  |
| 1988         | ISO6974-6            | 0.1549          |          | 1.16          |  |
|              | normality            | suspect         |          |               |  |
|              | n                    | 43              |          |               |  |
|              | outliers             | 43              |          |               |  |
|              | mean (n)             | 0.1512          |          |               |  |
|              | st.dev. (n)          | 0.00345         |          |               |  |
|              | R(calc.)             | 0.0097          |          |               |  |
|              | R(ISO6974-3:00)      | 0.0091          |          |               | Compare R(ASTM D1945:14) = 0.0700                      |
|              |                      |                 |          |               | ,  |

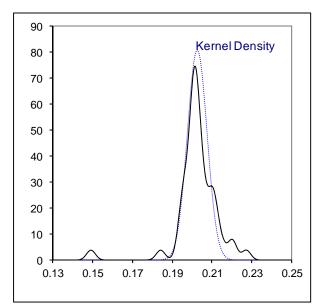




# Determination of n-Butane on sample #15030; results in %mol/mol

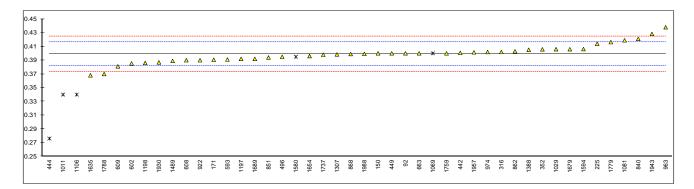
| lab          | method            | value          | mark     | z(targ)        | remarks  |
|--------------|-------------------|----------------|----------|----------------|--|
| 92           | GPA2286           | 0.20           |          | -0.55          |  |
| 150          | D1945             | 0.211          |          | 1.99           |  |
| 171          | D1945             | 0.204166       |          | 0.41           |  |
| 225          | D1945             | 0.198          |          | -1.01          |  |
| 316          | ISO6974-3         | 0.2020         |          | -0.09          |  |
| 352          | ISO6974-3         | 0.1998         |          | -0.59          |  |
| 442          | D1945             | 0.2012         |          | -0.27          |  |
| 444          | D1945             | 0.194          |          | -1.93          |  |
| 449          | D1945             | 0.2020         |          | -0.09          |  |
| 496<br>593   | DIN15984<br>D1945 | 0.194<br>0.184 | R(0.05)  | -1.93<br>-4.24 |  |
| 602          | GPA2261           | 0.2095         | K(0.03)  | 1.64           |  |
| 608          | GPA2261           | 0.22           | R(0.05)  | 4.07           |  |
| 609          | GPA2261           | 0.2070         | 11(0.00) | 1.07           |  |
| 663          | D1945             | 0.210          |          | 1.76           |  |
| 840          | D1945             | 0.211          |          | 1.99           |  |
| 851          | GPA2261           | 0.201          |          | -0.32          |  |
| 862          | GPA2261           | 0.201          | С        | -0.32          | First reported 0.172                                     |
| 868          | GPA2261           | 0.207          |          | 1.07           |  |
| 887          | D1945             | 0.20           |          | -0.55          |  |
| 922          | D1945             | 0.21           |          | 1.76           |  |
| 963          | D1945             | 0.203          |          | 0.15           |  |
| 974          | ISO6974-3         | 0.2004         |          | -0.45          |  |
| 1011         | UOP539            | 0.21           |          | 1.76           |  |
| 1029<br>1069 | D1945<br>UOP539   | 0.201<br>0.215 | R(0.05)  | -0.32<br>2.91  |  |
| 1089         | 005339            | 0.215          | R(0.05)  | 5.68           |  |
| 1095         |                   | 0.221          | 11(0.03) |                |  |
| 1106         | ISO6976           | 0.220          | ex       | 4.07           | See §4.1   |
| 1197         | D1945             | 0.195          |          | -1.70          |  |
| 1198         | D1945             | 0.200          |          | -0.55          |  |
| 1200         |                   |                |          |                |  |
| 1307         | in house          | 0.1959         |          | -1.49          |  |
| 1388         | GPA2261           | 0.2020         |          | -0.09          |  |
| 1489         | ISO6974-3         | 0.204          |          | 0.38           |  |
| 1580         | GB/T13610         | 0.149          | R(0.01)  | -12.31         | Decell Cost assessed as included a Decell cost of an     |
| 1594         | GPA2261           | 0.2012         | С        | -0.27          | Result first reported as iso-butane. Result was mixed-up |
| 1635<br>1654 | D1945<br>D1945    | 0.201<br>0.196 |          | -0.32<br>-1.47 |  |
| 1679         | ISO6974-3         | 0.190          |          | -0.32          |  |
| 1689         | GB/T13610         | 0.196          |          | -1.47          |  |
| 1737         | in house          | 0.201          |          | -0.32          |  |
| 1759         | ISO6974-5         | 0.204          |          | 0.38           |  |
| 1779         | GPA2261           | 0.1965         |          | -1.35          |  |
| 1788         | D7833             | 0.1995         |          | -0.66          |  |
| 1892         |                   |                |          |                |  |
| 1930         | ISO6974-6         | 0.2039         |          | 0.35           |  |
| 1943         | ISO6974-3         | 0.2044         |          | 0.47           |  |
| 1957         | GPA2261           | 0.2129         |          | 2.43           |  |
| 1960<br>1988 | 1000074.6         | 0.2059         |          | 0.79           |  |
| 1900         | ISO6974-6         | 0.2058         |          | 0.79           |  |
|              | normality         | OK             |          |                |  |
|              | n                 | 41             |          |                |  |
|              | outliers          | 5 (+ 1 excl)   |          |                |  |
|              | mean (n)          | 0.2024         |          |                |  |
|              | st.dev. (n)       | 0.00494        |          |                |  |
|              | R(calc.)          | 0.0138         |          |                |  |
|              | R(ISO6974-3:00)   | 0.0121         |          |                | Compare R(ASTM D1945:14) = 0.0700                        |

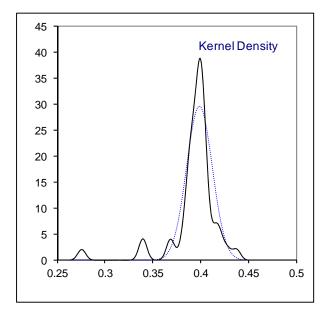




# Determination of Carbon Dioxide on sample #15030; results in %mol/mol

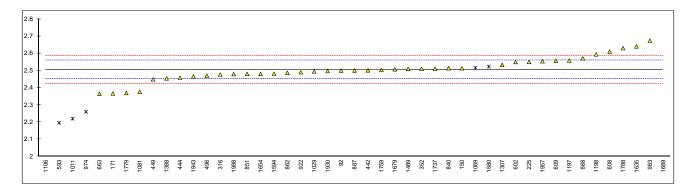
| lab          | method                  | value             | mark      | z(targ)        | remarks                                  |
|--------------|-------------------------|-------------------|-----------|----------------|--|
| 92           | GPA2286                 | 0.40              | mark      | 0.10           | Tollario                                 |
| 150          | D1945                   | 0.400             |           | 0.10           |  |
| 171          | D1945                   | 0.390838          |           | -0.98          |  |
| 225          | D1945                   | 0.414             |           | 1.73           |  |
| 316          | ISO6974-3               | 0.402             |           | 0.33           |  |
| 352          | ISO6974-3               | 0.4059            |           | 0.79           |  |
| 442          | D1945                   | 0.4007            |           | 0.18           |  |
| 444          | D1945                   | 0.276             | C,R(0.01) | -14.40         | First reported 0.300                     |
| 449          | D1945                   | 0.4000            |           | 0.10           |  |
| 496          | DIN15984                | 0.395             |           | -0.49          |  |
| 593          | D1945                   | 0.391             |           | -0.96          |  |
| 602<br>608   | GPA2261<br>GPA2261      | 0.3854<br>0.39    |           | -1.61<br>-1.07 |  |
| 609          | GPA2261<br>GPA2261      | 0.3811            |           | -1.07          |  |
| 663          | D1945                   | 0.400             |           | 0.10           |  |
| 840          | D1945                   | 0.421             |           | 2.55           |  |
| 851          | GPA2261                 | 0.394             |           | -0.61          |  |
| 862          | GPA2261                 | 0.403             |           | 0.45           |  |
| 868          | GPA2261                 | 0.399             |           | -0.02          |  |
| 887          |                         |                   |           |                |  |
| 922          | D1945                   | 0.39              |           | -1.07          |  |
| 963          | D1945                   | 0.438             |           | 4.54           |  |
| 974          | ISO6974-3               | 0.4019            | D(0.04)   | 0.32           |  |
| 1011         | UOP539                  | 0.34              | R(0.01)   | -6.92          |  |
| 1029<br>1069 | D1945<br>UOP539         | 0.406<br>0.4      | 0.4       | 0.80           | Son \$4.1                                |
| 1089         | 005339                  | 0.419             | ex        | 0.10<br>2.32   | See §4.1                                 |
| 1095         |                         |                   |           |                |  |
| 1106         | ISO6976                 | 0.340             | R(0.01)   | -6.92          |  |
| 1197         | D1945                   | 0.392             | ( /       | -0.84          |  |
| 1198         | D1945                   | 0.386             |           | -1.54          |  |
| 1200         |                         |                   |           |                |  |
| 1307         | in house                | 0.3984            |           | -0.09          |  |
| 1388         | GPA2261                 | 0.4052            |           | 0.70           |  |
| 1489         | ISO6974-3               | 0.389             |           | -1.19          | 0 044                                    |
| 1580<br>1594 | GB/T13610<br>GPA2261    | 0.395<br>0.4062   | ex        | -0.49<br>0.82  | See §4.1                                 |
| 1635         | D1945                   | 0.368             |           | -3.65          |  |
| 1654         | D1945                   | 0.396             |           | -0.37          |  |
| 1679         | ISO6974-3               | 0.406             |           | 0.80           |  |
| 1689         | GB/T13610               | 0.392             |           | -0.84          |  |
| 1737         | in house                | 0.398             |           | -0.14          |  |
| 1759         | ISO6974-5               | 0.400             |           | 0.10           |  |
| 1779         | GPA2261                 | 0.4163            |           | 2.00           |  |
| 1788         | D7833                   | 0.3699            |           | -3.42          |  |
| 1892         | 1000074.0               | 0.0000            |           | 4.45           |  |
| 1930<br>1943 | ISO6974-6<br>ISO6974-3  | 0.3868<br>0.4282  |           | -1.45<br>3.39  |  |
| 1943         | GPA2261                 | 0.4262            |           | 0.27           |  |
| 1960         | OI AZZOI                |                   |           |                |  |
| 1988         | ISO6974-6               | 0.3992            |           | 0.00           |  |
|              | -                       |                   |           |                |  |
|              | normality               | suspect           |           |                |  |
|              | n                       | 41                |           |                |  |
|              | outliers                | 3 (+2 excl)       |           |                |  |
|              | mean (n)                | 0.3992            |           |                |  |
|              | st.dev. (n)<br>R(calc.) | 0.01345<br>0.0377 |           |                |  |
|              | R(ISO6974-3:00)         | 0.0377            |           |                | Compare R(ASTM D1945:14) = 0.0700        |
|              | 11(1000014-0.00)        | 0.0270            |           |                | σοπραίο ((//ο (1)) D (στο. (τ)) = 0.0700 |

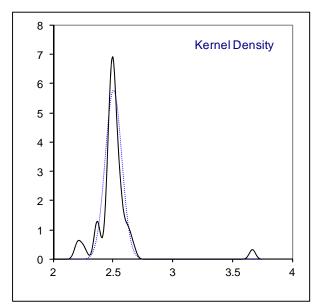




# Determination of Nitrogen on sample #15030; results in %mol/mol

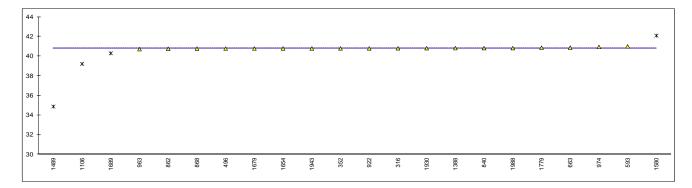
| lab          | method               | value           | mark      | z(targ)         | remarks                           |
|--------------|----------------------|-----------------|-----------|-----------------|-----------------------------------|
| 92           | GPA2286              | 2.50            |           | -0.21           |                                   |
| 150          | D1945                | 2.513           |           | 0.27            |                                   |
| 171          | D1945                | 2.366125        |           | -5.20           |                                   |
| 225          | D1945                | 2.550           | С         | 1.65            | First reported 2.295              |
| 316          | ISO6974-3            | 2.477           |           | -1.07           |                                   |
| 352          | ISO6974-3            | 2.5097          |           | 0.15            |                                   |
| 442          | D1945                | 2.5010          |           | -0.17           |                                   |
| 444          | D1945                | 2.458           |           | -1.78           |                                   |
| 449          | D1945                | 2.4488          |           | -2.12           |                                   |
| 496<br>503   | DIN15984<br>D1945    | 2.470<br>2.196  | D(0.05)   | -1.33<br>-11.54 |                                   |
| 593<br>602   | GPA2261              | 2.5495          | R(0.05)   | 1.63            |                                   |
| 608          | GPA2261              | 2.61            |           | 3.89            |                                   |
| 609          | GPA2261              | 2.5569          |           | 1.91            |                                   |
| 663          | D1945                | 2.365           |           | -5.24           |                                   |
| 840          | D1945                | 2.513           |           | 0.27            |                                   |
| 851          | GPA2261              | 2.480           |           | -0.96           |                                   |
| 862          | GPA2261              | 2.487           |           | -0.70           |                                   |
| 868          | GPA2261              | 2.571           |           | 2.43            |                                   |
| 887          | D1945                | 2.50            |           | -0.21           |                                   |
| 922          | D1945                | 2.49            |           | -0.58           |                                   |
| 963          | D1945                | 2.674           | O D(0 05) | 6.27            | First remarked 0.0570             |
| 974          | ISO6974-3            | 2.2599          | C,R(0.05) | -9.16           | First reported 2.3576             |
| 1011<br>1029 | UOP539<br>D1945      | 2.22<br>2.495   | R(0.05)   | -10.64<br>-0.40 |                                   |
| 1029         | UOP539               | 2.516           | ex        | 0.38            | See §4.1                          |
| 1081         | 001 000              | 2.376           | CX        | -4.83           | 000 g4.1                          |
| 1095         |                      |                 |           |                 |                                   |
| 1106         | ISO6976              | 1.300           | R(0.01)   | -44.91          |                                   |
| 1197         | D1945                | 2.558           | ,         | 1.95            |                                   |
| 1198         | D1945                | 2.594           |           | 3.29            |                                   |
| 1200         |                      |                 |           |                 |                                   |
| 1307         | in house             | 2.5327          |           | 1.01            |                                   |
| 1388         | GPA2261              | 2.4545          |           | -1.91           |                                   |
| 1489         | ISO6974-3            | 2.509           |           | 0.12            | 0 044                             |
| 1580<br>1594 | GB/T13610<br>GPA2261 | 2.523<br>2.4812 | ex        | 0.65<br>-0.91   | See §4.1                          |
| 1635         | D1945                | 2.640           |           | 5.00            |                                   |
| 1654         | D1945                | 2.481           |           | -0.92           |                                   |
| 1679         | ISO6974-3            | 2.507           |           | 0.05            |                                   |
| 1689         | GB/T13610            | 3.671           | C,R(0.01) | 43.41           | First reported 3.371              |
| 1737         | in house             | 2.510           | -, (-, -, | 0.16            |                                   |
| 1759         | ISO6974-5            | 2.503           |           | -0.10           |                                   |
| 1779         | GPA2261              | 2.3693          |           | -5.08           |                                   |
| 1788         | D7833                | 2.6302          |           | 4.64            |                                   |
| 1892         |                      |                 |           |                 |                                   |
| 1930         | ISO6974-6            | 2.4970          |           | -0.32           |                                   |
| 1943         | ISO6974-3            | 2.4663          |           | -1.47           |                                   |
| 1957         | GPA2261              | 2.5539          |           | 1.80            |                                   |
| 1960<br>1988 | ISO6974-6            | <br>2.4791      |           | -0.99           |                                   |
| 1900         | 1000314-0            | 2.4131          |           | -0.99           |                                   |
|              | normality            | OK              |           |                 |                                   |
|              | n                    | 40              |           |                 |                                   |
|              | outliers             | 5 (+2 excl)     |           |                 |                                   |
|              | mean (n)             | 2.5057          |           |                 |                                   |
|              | st.dev. (n)          | 0.06928         |           |                 |                                   |
|              | R(calc.)             | 0.1940          |           |                 |                                   |
|              | R(ISO6974-3:00)      | 0.0752          |           |                 | Compare R(ASTM D1945:14) = 0.1000 |

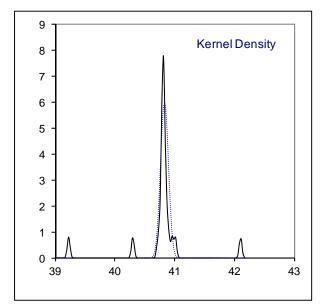




# Determination of Caloric Value Superior (101.325 kPa, comb. temp. 25°C, metering temp 0°C) on sample #15030; results in $MJ/m^3$ (real gas)

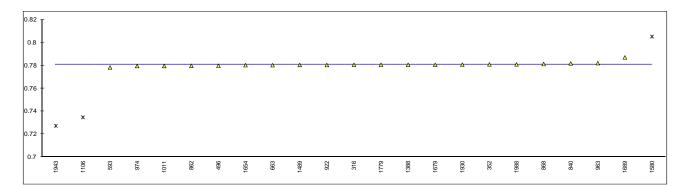
| lab          | method               | value            | mark               | z(targ) | remarks  |
|--------------|----------------------|------------------|--------------------|---------|--|
| 92           |                      |                  |                    |         |  |
| 150          |                      |                  |                    |         |  |
| 171          |                      |                  |                    |         |  |
| 225          | 1000075              | 40.040           |                    |         |  |
| 316          | ISO6976              | 40.812           |                    |         |  |
| 352          | ISO6976              | 40.805           |                    |         |  |
| 442<br>444   |                      |                  |                    |         |  |
| 449          |                      |                  |                    |         |  |
| 496          | DIN51857             | 40.7847          |                    |         |  |
| 593          | ISO6976              | 41.006           | E                  |         | lis calculated 40.927                                    |
| 602          |                      |                  |                    |         |  |
| 608          |                      |                  |                    |         |  |
| 609          | 1000000              |                  |                    |         |  |
| 663          | ISO6976              | 40.880           |                    |         |  |
| 840<br>851   | ISO6976              | 40.828           |                    |         |  |
| 862          | ISO6976              | 40.766           | Е                  |         | lis calculated 40.805                                    |
| 868          | ISO6976              | 40.78            | _                  |         | no calculated 40.000                                     |
| 887          | .0000.0              |                  |                    |         |  |
| 922          | ISO6976              | 40.806           |                    |         |  |
| 963          | ISO6976              | 40.72            |                    |         |  |
| 974          | GPA2172              | 40.948           | E                  |         | iis calculated 40.842                                    |
| 1011         |                      |                  |                    |         |  |
| 1029         |                      |                  |                    |         |  |
| 1069<br>1081 |                      |                  |                    |         |  |
| 1095         |                      |                  |                    |         |  |
| 1106         | GPA2286              | 39.22402         | ex, E              |         | See §4.1, iis calculated 41.450                          |
| 1197         |                      |                  | , -                |         | 3,   |
| 1198         |                      |                  |                    |         |  |
| 1200         |                      |                  |                    |         |  |
| 1307         | 1000070              | 40.007           |                    |         |  |
| 1388         | ISO6976              | 40.827           | D(0.04) F          |         | iio polaulated 40 004                                    |
| 1489<br>1580 | ISO6976<br>GB/T11062 | 34.875<br>42.098 | R(0.01),E<br>ex, E |         | iis calculated 40.801<br>See §4.1, iis calculated 40.576 |
| 1594         | OD/111002            |                  | CX, L              |         | occ 94.1, iis calculated 40.570                          |
| 1635         |                      |                  |                    |         |  |
| 1654         | ISO6976              | 40.797           |                    |         |  |
| 1679         | ISO6976              | 40.792           |                    |         |  |
| 1689         | GB/T11062            | 40.297           | C,R(0.01),E        |         | First reported 38.250, iis calculated 40.380             |
| 1737         |                      |                  |                    |         |  |
| 1759<br>1779 | 1006076              | 40.9702          |                    |         |  |
| 1778         | ISO6976              | 40.8703          |                    |         |  |
| 1892         |                      |                  |                    |         |  |
| 1930         | DIN51857             | 40.8197          |                    |         |  |
| 1943         | ISO6976              | 40.7988          | E                  |         | lis calculated 40.779                                    |
| 1957         |                      |                  |                    |         |  |
| 1960         | 1000070              | 40.0004          |                    |         |  |
| 1988         | ISO6976              | 40.8284          |                    |         |  |
|              | normality            | not OK           |                    |         |  |
|              | n                    | 18               |                    |         |  |
|              | outliers             | 2 (+2 excl)      |                    |         |  |
|              | mean (n)             | 40.8260          |                    |         |  |
|              | st.dev. (n)          | 0.06626          |                    |         |  |
|              | R(calc.)             | 0.1855           |                    |         | Compare P(iio14904M) = 0.1955                            |
|              | R(lit)               | unknown          |                    |         | Compare R(iis14S01M) = 0.1855                            |

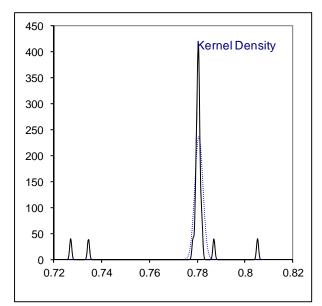




Determination of Density (101.325 kPa, comb. 25°C, metering temp. 0°C) on sample #15030; results in kg/m³ (real gas)

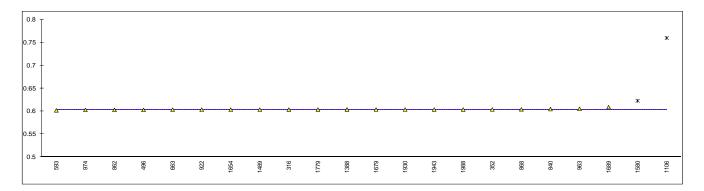
|              | s in kg/m³ (rea      | l gas)           |           |         |                                  |
|--------------|----------------------|------------------|-----------|---------|----------------------------------|
| lab          | method               | value            | mark      | z(targ) | remarks                          |
| 92           |                      |                  |           |         |                                  |
| 150          |                      |                  |           |         |                                  |
| 171          |                      |                  |           |         |                                  |
| 225          | 1000076              | 0.7005           |           |         |                                  |
| 316<br>352   | ISO6976<br>ISO6976   | 0.7805<br>0.7809 |           |         |                                  |
| 442          | 1300970              | 0.7809           |           |         |                                  |
| 444          |                      |                  |           |         |                                  |
| 449          |                      |                  |           |         |                                  |
| 496          | DIN51857             | 0.779720         |           |         |                                  |
| 593          | ISO6976              | 0.7783           |           |         |                                  |
| 602          |                      |                  |           |         |                                  |
| 608          |                      |                  |           |         |                                  |
| 609          | 1000070              | 0.70004          | •         |         | Fi                               |
| 663          | ISO6976              | 0.78021          | С         |         | First reported 0.60347           |
| 840          | ISO6976              | 0.78178<br>      |           |         |                                  |
| 851<br>862   | ISO6976              | 0.77970          |           |         |                                  |
| 868          | ISO6976              | 0.7812           |           |         |                                  |
| 887          | 1000370              | 0.7012           |           |         |                                  |
| 922          | ISO6976              | 0.7804           |           |         |                                  |
| 963          | ISO6976              | 0.7820           |           |         |                                  |
| 974          | GPA2172              | 0.7795           | E         |         | lis calculated 0.7783            |
| 1011         | D3588                | 0.7795           |           |         |                                  |
| 1029         |                      |                  |           |         |                                  |
| 1069         |                      |                  |           |         |                                  |
| 1081         |                      |                  |           |         |                                  |
| 1095         | GPA2286              | 0.724742         | ov. F     |         | Con S4.4. iin coloulated 0.77654 |
| 1106         | GPA2200              | 0.734713         | ex, E     |         | See §4.1, iis calculated 0.77651 |
| 1197<br>1198 |                      |                  |           |         |                                  |
| 1200         |                      |                  |           |         |                                  |
| 1307         |                      |                  |           |         |                                  |
| 1388         | ISO6976              | 0.78059          |           |         |                                  |
| 1489         | ISO6976              | 0.78039          |           |         |                                  |
| 1580         | GB/T11062            | 0.8052           | ex, E     |         | See §4.1, iis calculated 0.77606 |
| 1594         |                      |                  |           |         |                                  |
| 1635         | 1000070              |                  |           |         |                                  |
| 1654         | ISO6976              | 0.7802           |           |         |                                  |
| 1679<br>1689 | ISO6976<br>GB/T11062 | 0.78061<br>0.787 | С         |         | First reported 0.639             |
| 1737         | GB/111002            | 0.767            | C         |         | riist reported 0.009             |
| 1759         |                      |                  |           |         |                                  |
| 1779         | ISO6976              | 0.78058          |           |         |                                  |
| 1788         |                      |                  |           |         |                                  |
| 1892         |                      |                  |           |         |                                  |
| 1930         | DIN51857             | 0.7807           |           |         |                                  |
| 1943         | ISO6976              | 0.7273           | R(0.01),E |         | iis calculated 0.78025           |
| 1957         |                      |                  |           |         |                                  |
| 1960         | 1000076              | 0.7000           |           |         |                                  |
| 1988         | ISO6976              | 0.7809           |           |         |                                  |
|              | normality            | not OK           |           |         |                                  |
|              | n                    | 20               |           |         |                                  |
|              | outliers             | 1 (+2 excl)      |           |         |                                  |
|              | mean (n)             | 0.7807           |           |         |                                  |
|              | st.dev. (n)          | 0.00169          |           |         |                                  |
|              | R(calc.)             | 0.0047           |           |         |                                  |
|              | R(lit)               | unknown          |           |         | Compare R(iis14S01M) = 0.0058    |

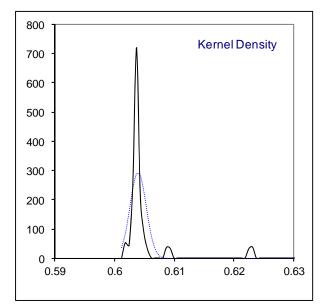




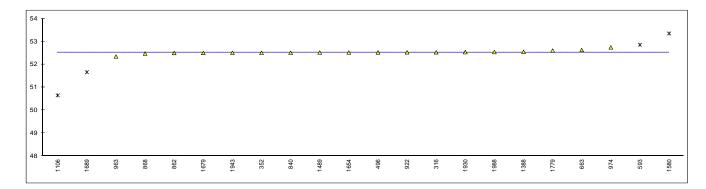
# Determination of Relative Density (101.325 kPa, comb. temp. 25°C, metering temp. 0°C) on sample #15030; results have no unit (real gas)

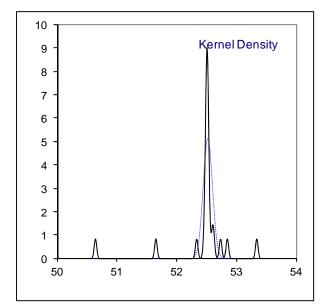
| lab          | method             | value              | mark  | z(targ) | remarks   |
|--------------|--------------------|--------------------|-------|---------|---|
| 92           |                    |                    |       |         |   |
| 150          |                    |                    |       |         |   |
| 171          |                    |                    |       |         |   |
| 225          | 1000070            | 0.0007             |       |         |   |
| 316<br>352   | ISO6976<br>ISO6976 | 0.6037<br>0.6040   |       |         |   |
| 442          | 1300970            | 0.0040             |       |         |   |
| 444          |                    |                    |       |         |   |
| 449          |                    |                    |       |         |   |
| 496          | DIN51857           | 0.603068           |       |         |   |
| 593          | ISO6976            | 0.6020             |       |         |   |
| 602          |                    |                    |       |         |   |
| 608<br>609   |                    |                    |       |         |   |
| 663          | ISO6976            | 0.60347            | С     |         | First reported 0.60219                              |
| 840          | ISO6976            | 0.60466            | -     |         |   |
| 851          |                    |                    |       |         |   |
| 862          | ISO6976            | 0.60306            |       |         |   |
| 868          | ISO6976            | 0.6042             |       |         |   |
| 887<br>922   | ISO6976            | 0.6036             |       |         |   |
| 963          | ISO6976            | 0.6052             |       |         |   |
| 974          | GPA2172            | 0.6029             |       |         |   |
| 1011         |                    |                    |       |         |   |
| 1029         |                    |                    |       |         |   |
| 1069         |                    |                    |       |         |   |
| 1081<br>1095 |                    |                    |       |         |   |
| 1106         | GPA2286            | 0.759981           | ex, E |         | See §4.1, iis calculated 0.60059                    |
| 1197         |                    |                    | , -   |         | <b>3</b> ··· , ··· • ··· • ··· • ··· • ·· • · · · · |
| 1198         |                    |                    |       |         |   |
| 1200         |                    |                    |       |         |   |
| 1307         | ISO6976            | 0.60274            |       |         |   |
| 1388<br>1489 | ISO6976            | 0.60374<br>0.60362 |       |         |   |
| 1580         | GB/T11062          | 0.6228             | ex, E |         | See §4.1, iis calculated 0.60024                    |
| 1594         |                    |                    | - ,   |         | ,             |
| 1635         |                    |                    |       |         |   |
| 1654         | ISO6976            | 0.6036             |       |         |   |
| 1679         | ISO6976            | 0.60376<br>0.609   | С     |         | First reported 0.742                                |
| 1689<br>1737 | GB/T11062          | 0.609              | C     |         | First reported 0.742                                |
| 1759         |                    |                    |       |         |   |
| 1779         | ISO6976            | 0.60373            |       |         |   |
| 1788         |                    |                    |       |         |   |
| 1892         | DINE 4057          |                    |       |         |   |
| 1930         | DIN51857           | 0.6038             |       |         |   |
| 1943<br>1957 | ISO6976            | 0.6039             |       |         |   |
| 1960         |                    |                    |       |         |   |
| 1988         | ISO6976            | 0.6039             |       |         |   |
|              |                    |                    |       |         |   |
|              | normality          | not OK             |       |         |   |
|              | n<br>outliers      | 20<br>0 (+2 excl)  |       |         |   |
|              | mean (n)           | 0.6039             |       |         |   |
|              | st.dev. (n)        | 0.0033             |       |         |   |
|              | R(calc.)           | 0.0038             |       |         |   |
|              | R(lit)             | unknown            |       |         | Compare $R(iis14S01M) = 0.0025$                     |
|              |                    |                    |       |         |   |



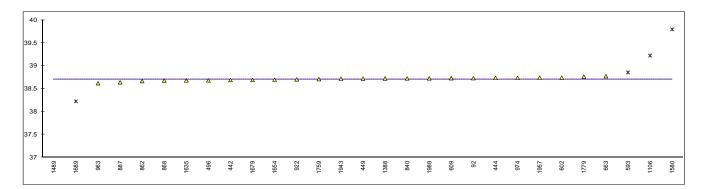


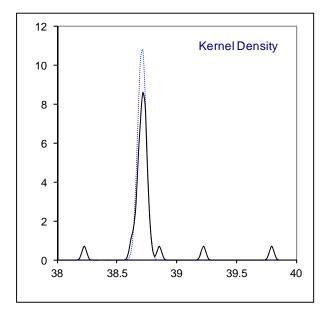
| lab          | method        | value        | m <sup>3</sup> (real gas) | z(targ)  | remarks                                      |
|--------------|---------------|--------------|---------------------------|----------|--|
| 92           | metriou       |              | IIIQIK                    | <u> </u> | Tentario                                     |
| 150          |               |              |                           |          |  |
| 171          |               |              |                           |          |  |
| 225          |               |              |                           |          |  |
| 316          | ISO6976       | 52.526       |                           |          |  |
| 352          | ISO6976       | 52.504       |                           |          |  |
| 142          |               |              |                           |          |  |
| 144          |               |              |                           |          |  |
| 149          |               |              |                           |          |  |
| 196          | DIN51857      | 52.5187      |                           |          |  |
| 593          | ISO6976       | 52.853       | R(0.05), E                |          | iis calculated 52.733                        |
| 502          |               |              |                           |          |  |
| 808          |               |              |                           |          |  |
| 609          |               |              |                           |          |  |
| 663          | ISO6976       | 52.624       |                           |          |  |
| 340          | ISO6976       | 52.506       |                           |          |  |
| 351          |               |              |                           |          |  |
| 362          | ISO6976       | 52.494       | E                         |          | iis calculated 52.518                        |
| 368          | ISO6976       | 52.47        |                           |          |  |
| 387          |               |              |                           |          |  |
| 922          | ISO6976       | 52.524       | _                         |          | W  |
| 963          | ISO6976       | 52.34        | Ē                         |          | iis calculated 52.358                        |
| 974          | GPA2172       | 52.74        | E                         |          | iis calculated 52.640                        |
| 1011         |               |              |                           |          |  |
| 1029         |               |              |                           |          |  |
| 1069         |               |              |                           |          |  |
| 1081         |               |              |                           |          |  |
| 1095<br>1106 | GPA2286       | 50.6464      | ex, E                     |          | See §4.1, iis calculated 53.485              |
| 1197         | GFA2200       | 50.6464      | ex, L                     |          | See §4.1, iis calculated 55.465              |
| 1198         |               |              |                           |          |  |
| 1200         |               |              |                           |          |  |
| 1307         |               |              |                           |          |  |
| 1388         | ISO6976       | 52.542       |                           |          |  |
| 1489         | ISO6976       | 52.513       |                           |          |  |
| 1580         | GB/T11062     | 53.346       | ex, E                     |          | See §4.1, iis calculated 52.373              |
| 1594         | 02,002        |              | ολ, <b>–</b>              |          | 500 3, 110 00100101010                       |
| 1635         |               |              |                           |          |  |
| 1654         | ISO6976       | 52.514       |                           |          |  |
| 1679         | ISO6976       | 52.498       |                           |          |  |
| 1689         | GB/T11062     | 51.658       | C,R(0.01),E               |          | First reported 51.821, iis calculated 51.738 |
| 1737         |               |              |                           |          |  |
| 1759         |               |              |                           |          |  |
| 1779         | ISO6976       | 52.6000      |                           |          |  |
| 1788         |               |              |                           |          |  |
| 1892         |               |              |                           |          |  |
| 1930         | DIN51857      | 52.5320      |                           |          |  |
| 1943         | ISO6976       | 52.5027      |                           |          |  |
| 1957         |               |              |                           |          |  |
| 1960         | 1000070       |              |                           |          |  |
| 1988         | ISO6976       | 52.5352      |                           |          |  |
|              | normality     | not OV       |                           |          |  |
|              | normality     | not OK<br>18 |                           |          |  |
|              | n<br>outliers | 2 (+2 excl)  |                           |          |  |
|              | mean (n)      | 52.5269      |                           |          |  |
|              | st.dev. (n)   | 0.07768      |                           |          |  |
|              |               |              |                           |          |  |
|              | R(calc.)      | 0.2175       |                           |          |  |





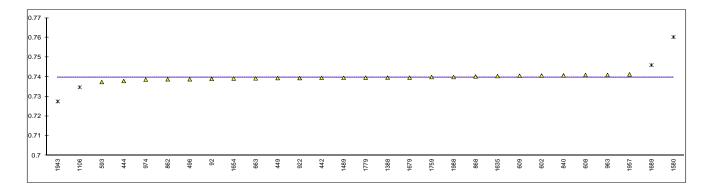
| lab        | method                  | value              | m <sup>3</sup> (real gas) | z(targ) | remarks                                      |
|------------|-------------------------|--------------------|---------------------------|---------|--|
| 2          |                         | 38.73              | E                         |         | iis calculated 38.673                        |
| 50         |                         |                    |                           |         |  |
| 71         |                         |                    |                           |         |  |
| 25         |                         |                    |                           |         |  |
| 16         |                         |                    |                           |         |  |
| 52         |                         |                    |                           |         |  |
| 42         | ISO6974                 | 38.69              |                           |         |  |
| 44         | ISO6976                 | 38.738             |                           |         |  |
| 49         | ISO6976                 | 38.720             |                           |         |  |
| 96         | DIN51857                | 38.6836            |                           |         |  |
| 93         | ISO6976                 | 38.854             | R(0.05),E                 |         | iis calculated 38.818                        |
| 02         | ISO6976                 | 38.7411            |                           |         |  |
| 80         |                         |                    |                           |         |  |
| 09         | ISO6976                 | 38.7290            |                           |         |  |
| 63         | ISO6976                 | 38.773             |                           |         |  |
| 40         | ISO6976                 | 38.725             |                           |         |  |
| 51         | 1000070                 |                    | _                         |         | "  |
| 62         | ISO6976                 | 38.666             | E                         |         | iis calculated 38.703                        |
| 68         | ISO6976                 | 38.68              | 0                         |         | First year arts dist 0500/000                |
| 87         | D3588                   | 38.63997           | С                         |         | First reported at 25°C/ 0°C                  |
| 22         | ISO6976                 | 38.704             |                           |         |  |
| 63<br>74   | ISO6976<br>GPA2172      | 38.62              |                           |         |  |
| 74<br>011  | GFAZ17Z                 | 38.738             |                           |         |  |
| 011<br>029 |                         |                    |                           |         |  |
| 069        |                         |                    |                           |         |  |
| 081        |                         |                    |                           |         |  |
| 095        |                         |                    |                           |         |  |
| 106        | GPA2286                 | 39.22402           | ex, E                     |         | See §4.1, iis calculated 39.314              |
| 197        | O. 7.2200               |                    | ολ, <u>L</u>              |         | 200 3 1.1, 110 0010010100 00.011             |
| 198        |                         |                    |                           |         |  |
| 200        |                         |                    |                           |         |  |
| 307        |                         |                    |                           |         |  |
| 388        | ISO6976                 | 38.722             |                           |         |  |
| 489        | ISO6976                 | 34.912             | R(0.01), E                |         | iis calculated 38.699                        |
| 580        | GB/T11062               | 39.795             | ex, E                     |         | See §4.1, iis calculated 38.486              |
| 594        |                         |                    | ·                         |         |  |
| 635        | ISO6976                 | 38.681             |                           |         |  |
| 654        | ISO6976                 | 38.696             |                           |         |  |
| 679        | ISO6976                 | 38.690             |                           |         |  |
| 689        | GB/T11062               | 38.226             | C,R(0.01),E               |         | First reported 38.165, iis calculated 38.300 |
| 737        |                         |                    |                           |         |  |
| 759        | ISO6976                 | 38.7088            |                           |         |  |
| 779        | ISO6976                 | 38.7651            |                           |         |  |
| 788        |                         |                    |                           |         |  |
| 892        |                         |                    |                           |         |  |
| 930        | 1000000                 |                    | _                         |         | W  |
| 943        | ISO6976                 | 38.7187            | E                         |         | iis calculated 38.678                        |
| 957        | ISO6976                 | 38.74              | E                         |         | iis calculated 38.725                        |
| 960        | 1000070                 |                    |                           |         |  |
| 988        | ISO6976                 | 38.7258            |                           |         |  |
|            | normality               | OK                 |                           |         |  |
|            | normality               |                    |                           |         |  |
|            | n<br>outliers           | 24<br>3 (+2 excl)  |                           |         |  |
|            |                         | 38.7094            |                           |         |  |
|            | mean (n)<br>st.dev. (n) | 38.7094<br>0.03635 |                           |         |  |
|            | JI.UCV. (11)            | 0.00000            |                           |         |  |
|            | R(calc.)                | 0.1018             |                           |         |  |

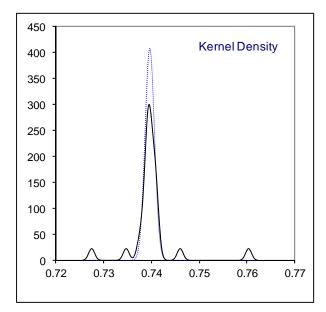




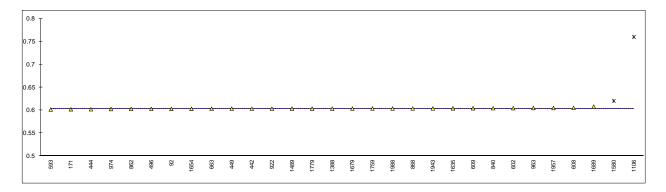
Determination of Density (101.325 kPa, combustion temp. 15°C, metering temp. 15°C) on sample #15030; results in kg/m³ (real gas)

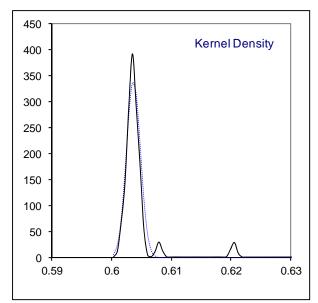
|              | 0; results in k    | kg/m³ (real        | -            | -       |                                  |
|--------------|--------------------|--------------------|--------------|---------|----------------------------------|
| lab          | method             | value              | mark         | z(targ) | remarks                          |
| 92           | GPA2286            | 0.739              |              |         |                                  |
| 150          |                    |                    |              |         |                                  |
| 171          |                    |                    |              |         |                                  |
| 225          |                    |                    |              |         |                                  |
| 316<br>352   |                    |                    |              |         |                                  |
| 442          | ISO6974            | 0.7395             |              |         |                                  |
| 444          | ISO6976            | 0.7379             |              |         |                                  |
| 449          | ISO6976            | 0.7394             |              |         |                                  |
| 496          | DIN51857           | 0.738792           |              |         |                                  |
| 593          | ISO6976            | 0.7374             |              |         |                                  |
| 602          | ISO6976            | 0.7407             |              |         |                                  |
| 608          | ISO6976            | 0.741              |              |         |                                  |
| 609          | ISO6976            | 0.74061            |              |         |                                  |
| 663<br>840   | ISO6976<br>ISO6976 | 0.73926<br>0.74074 |              |         |                                  |
| 851          | 1500970            |                    |              |         |                                  |
| 862          | ISO6976            | 0.73878            |              |         |                                  |
| 868          | ISO6976            | 0.7402             |              |         |                                  |
| 887          |                    |                    |              |         |                                  |
| 922          | ISO6976            | 0.7394             |              |         |                                  |
| 963          | ISO6976            | 0.7410             | _            |         | W                                |
| 974          | GPA2172            | 0.7386             | E            |         | iis calculated 0.73745           |
| 1011<br>1029 |                    |                    |              |         |                                  |
| 1029         |                    |                    |              |         |                                  |
| 1081         |                    |                    |              |         |                                  |
| 1095         |                    |                    |              |         |                                  |
| 1106         | GPA2286            | 0.734713           | ex, E        |         | See §4.1, iis calculated 0.73575 |
| 1197         |                    |                    |              |         |                                  |
| 1198         |                    |                    |              |         |                                  |
| 1200         |                    |                    |              |         |                                  |
| 1307         | 1000076            | 0.72062            |              |         |                                  |
| 1388<br>1489 | ISO6976<br>ISO6976 | 0.73962<br>0.73954 |              |         |                                  |
| 1580         | GB/T11062          | 0.7603             | ex, E        |         | See §4.1, iis calculated 0.73533 |
| 1594         | 05/11/002          |                    | ολ, <b>L</b> |         | 000 3 1.11, 110 00100101000      |
| 1635         | ISO6976            | 0.7404             |              |         |                                  |
| 1654         | ISO6976            | 0.7392             |              |         |                                  |
| 1679         | ISO6976            | 0.73964            |              |         |                                  |
| 1689         | GB/T11062          | 0.746              | C,R(0.01)    |         | First reported 0.605             |
| 1737<br>1759 | ISO6976            | 0.7200             |              |         |                                  |
| 1739         | ISO6976            | 0.7399<br>0.73961  |              |         |                                  |
| 1778         | 1300370            | 0.73301            |              |         |                                  |
| 1892         |                    |                    |              |         |                                  |
| 1930         |                    |                    |              |         |                                  |
| 1943         | ISO6976            | 0.7275             | R(0.01), E   |         | iis calculated 0.73929           |
| 1957         | ISO6976            | 0.7413             |              |         |                                  |
| 1960         | 1000070            | . 7000             |              |         |                                  |
| 1988         | ISO6976            | 0.7399             |              |         |                                  |
|              | normality          | OK                 |              |         |                                  |
|              | n                  | 25                 |              |         |                                  |
|              | outliers           | 2 (+2 excl)        |              |         |                                  |
|              | mean (n)           | 0.7397             |              |         |                                  |
|              | st.dev. (n)        | 0.00096            |              |         |                                  |
|              | R(calc.)           | 0.0027             |              |         | O P(": 440044)                   |
|              | R(lit)             | unknown            |              |         | Compare R(iis14S01M) = 0.0043    |





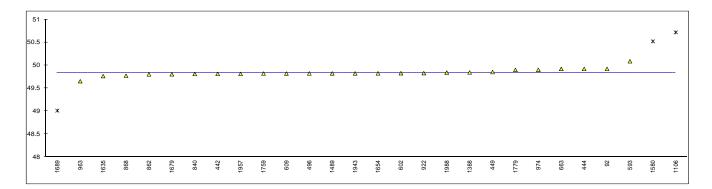
| lab        | e #15030; re       | value              | mark  | z(targ) | remarks                                 |
|------------|--------------------|--------------------|-------|---------|---|
|            | GPA2286            | 0.603              |       |         |   |
| 0          |                    |                    |       |         |   |
| 1          | D3588              | 0.6021             | Е     |         | lis calculated 0.60325                  |
| .5         |                    |                    |       |         |   |
| 6          |                    |                    |       |         |   |
| 52<br> 2   | ISO6974            | 0.6034             |       |         |   |
| 4          | ISO6974            | 0.6022             |       |         |   |
| 9          | ISO6976            | 0.6034             |       |         |   |
| 6          | DIN51857           | 0.602894           |       |         |   |
| 3          | ISO6976            | 0.6018             |       |         |   |
| )2         | ISO6976            | 0.6046             |       |         |   |
| 8          | ISO6976            | 0.605              |       |         |   |
| )9         | ISO6976            | 0.60437            |       |         |   |
| 33<br>10   | ISO6976<br>ISO6976 | 0.60329<br>0.60448 |       |         |   |
| 51         | 1300370            | 0.00440            |       |         |   |
| 62         | ISO6976            | 0.60288            |       |         |   |
| 88         | ISO6976            | 0.6040             |       |         |   |
| 37         |                    |                    |       |         |   |
| 22         | ISO6976            | 0.6034             |       |         |   |
| 33         | ISO6976            | 0.6049             |       |         |   |
| 74         | GPA2172            | 0.6027             |       |         |   |
| )11<br>)29 |                    |                    |       |         |   |
| )69        |                    |                    |       |         |   |
| )81        |                    |                    |       |         |   |
| 95         |                    |                    |       |         |   |
| 106        | GPA2286            | 0.759981           | ex, E |         | See §4.1, iis calculated 0.60041        |
| 197        |                    |                    |       |         |   |
| 198        |                    |                    |       |         |   |
| 200        |                    |                    |       |         |   |
| 307<br>388 | ISO6976            | 0.60357            |       |         |   |
| 189        | ISO6976            | 0.60352            |       |         |   |
| 580        | GB/T11062          | 0.6205             | ex, E |         | See §4.1, iis calculated 0.60007        |
| 594        |                    |                    | , -   |         | 3 · · · , · · · · · · · · · · · · · · · |
| 35         | ISO6976            | 0.6042             |       |         |   |
| 654        | ISO6976            | 0.6032             |       |         |   |
| 379        | ISO6976            | 0.60359            | 0     |         | First are arted 0.740                   |
| 889        | GB/T11062          | 0.608              | С     |         | First reported 0.742                    |
| 737<br>759 | ISO6976            | 0.6038             |       |         |   |
| 779        | ISO6976            | 0.60356            |       |         |   |
| 788        | .0000.0            |                    |       |         |   |
| 392        |                    |                    |       |         |   |
| 930        |                    |                    |       |         |   |
| 943        | ISO6976            | 0.6040             |       |         |   |
| 957        | ISO6976            | 0.6049             |       |         |   |
| 960<br>988 | ISO6976            | 0.6038             |       |         |   |
|            | normality          | not OK             |       |         |   |
|            | n                  | 28                 |       |         |   |
|            | outliers           | 0 (+2 excl)        |       |         |   |
|            | mean (n)           | 0.6037             |       |         |   |
|            | st.dev. (n)        | 0.00118            |       |         |   |
|            | R(calc.)<br>R(lit) | 0.0033             |       |         | Comprae R(iis14S01M) = 0.0029           |

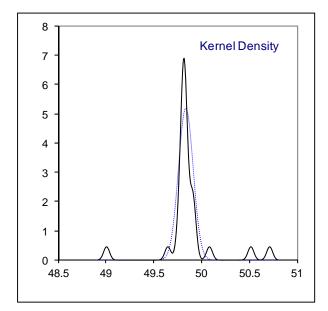




Determination of Wobbe Index (101.325 kPa, combustion temp. 15 $^{\circ}$ C, metering temp. 15 $^{\circ}$ C) on sample #15030; results in MJ/m<sup>3</sup>

|              | e #15030; re | •           |            | =/tora) | romarko                         |
|--------------|--------------|-------------|------------|---------|---------------------------------|
| lab          | method       | value       | mark       | z(targ) | remarks                         |
| 92<br>150    | ISO6976      | 49.92<br>   | E          |         | lis calculated 49.80            |
| 171          |              |             |            |         |                                 |
| 225          |              |             |            |         |                                 |
| 316          |              |             |            |         |                                 |
| 352          |              |             |            |         |                                 |
| 442          | ISO6974      | 49.8100     |            |         |                                 |
| 444          | ISO6976      | 49.920      |            |         |                                 |
| 449          | ISO6976      | 49.8500     |            |         |                                 |
| 496          | DIN51857     | 49.8203     |            |         |                                 |
| 593          | ISO6976      | 50.086      |            |         |                                 |
| 602          | ISO6976      | 49.8250     |            |         |                                 |
| 608          |              |             |            |         |                                 |
| 609          | ISO6976      | 49.8177     |            |         |                                 |
| 663          | ISO6976      | 49.919      |            |         |                                 |
| 840          | ISO6976      | 49.808      |            |         |                                 |
| 851          | 1000070      |             |            |         |                                 |
| 862          | ISO6976      | 49.798      |            |         |                                 |
| 868          | ISO6976      | 49.77       |            |         |                                 |
| 887<br>922   | ISO6976      | 49.826      |            |         |                                 |
| 963          | ISO6976      | 49.65       |            |         |                                 |
| 974          | GPA2172      | 49.90       |            |         |                                 |
| 1011         | 0.7.2.7.2    |             |            |         |                                 |
| 1029         |              |             |            |         |                                 |
| 1069         |              |             |            |         |                                 |
| 1081         |              |             |            |         |                                 |
| 1095         |              |             |            |         |                                 |
| 1106         | GPA2286      | 50.7166     | ex         |         | See §4.1                        |
| 1197         |              |             |            |         |                                 |
| 1198         |              |             |            |         |                                 |
| 1200         |              |             |            |         |                                 |
| 1307         |              |             |            |         |                                 |
| 1388         | ISO6976      | 49.843      |            |         |                                 |
| 1489         | ISO6976      | 49.821      | <b>-</b>   |         | Caa C4.4. "a salaulated 40.000  |
| 1580         | GB/T11062    | 50.520      | ex, E      |         | See §4.1, iis calculated 49.683 |
| 1594<br>1635 | ISO6976      | 49.762      |            |         |                                 |
| 1654         | ISO6976      | 49.702      |            |         |                                 |
| 1679         | ISO6976      | 49.801      |            |         |                                 |
| 1689         | GB/T11062    | 49.008      | C,R(0.01)  |         | First reported 49.162           |
| 1737         |              |             | -, ( ( ) ) |         |                                 |
| 1759         | ISO6976      | 49.8170     |            |         |                                 |
| 1779         | ISO6976      | 49.8978     |            |         |                                 |
| 1788         |              |             |            |         |                                 |
| 1892         |              |             |            |         |                                 |
| 1930         |              |             |            |         |                                 |
| 1943         | ISO6976      | 49.8210     |            |         |                                 |
| 1957         | ISO6976      | 49.81       |            |         |                                 |
| 1960<br>1988 | 1000076      | 40.0200     |            |         |                                 |
| 1988         | ISO6976      | 49.8390     |            |         |                                 |
|              | normality    | not OK      |            |         |                                 |
|              | n            | 25          |            |         |                                 |
|              | outliers     | 1 (+2 excl) |            |         |                                 |
|              | mean (n)     | 49.8382     |            |         |                                 |
|              | st.dev. (n)  | 0.07708     |            |         |                                 |
|              | R(calc.)     | 0.2158      |            |         |                                 |
|              | R(lit)       | unknown     |            |         | Compare R(iis14S01M) = 0.2417   |
|              |              |             |            |         |                                 |





## **APPENDIX 2**

# Number of participants per country

- 1 lab in AUSTRALIA
- 1 lab in BELGIUM
- 1 lab in BRUNEI
- 1 lab in CANADA
- 7 labs in CHINA, People's Republic
- 1 lab in COTE D'IVOIRE
- 1 lab in CROATIA
- 1 lab in ECUADOR
- 1 lab in FINLAND
- 1 lab in FRANCE
- 2 labs in GERMANY
- 1 lab in HONG KONG
- 1 lab in IRAQ
- 1 lab in LATVIA
- 1 lab in LITHUANIA
- 7 labs in MALAYSIA
- 2 labs in NETHERLANDS
- 1 lab in PAKISTAN
- 1 lab in PERU
- 4 labs in PORTUGAL
- 2 labs in SAUDI ARABIA
- 1 lab in SERBIA
- 1 lab in SLOVAKIA
- 1 lab in TAIWAN
- 1 lab in THAILAND
- 1 lab in TURKEY
- 1 lab in UNITED ARAB EMIRATES
- 3 labs in UNITED KINGDOM
- 2 labs in UNITED STATES OF AMERICA
- 1 lab in VIETNAM

#### **APPENDIX 3**

#### Abbreviations:

C = final result after checking of first reported suspect result

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \end{array}$ 

DG(0.01) = outlier in Double Grubbs' outlier test DG(0.05) = straggler in Double Grubbs' outlier test

R(0.01) = outlier in Rosner outlier test R(0.05) = straggler in Rosner outlier test ex = excluded from calculations

n/a = not applicable

W = withdrawn on request participant

U = reported in wrong unit
E = error in calculations
SDS = Safety Data Sheet

#### Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, April 2014
- 2 ISO 6974, Natural Gas Determination of composition with defined uncertainty by GC
- 3 ASTM E178-89
- 4 ASTM E1301-89
- 5 ISO 5725-86
- 6 ISO 5725, parts 1-6, 1994
- 7 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, First reported Z. Anal. Chem, <u>331</u>, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical Brief, No4 January 2001
- The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson.
- 15 ASTM D1945, Standard test method for Analysis of Natural Gas by GC
- Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)