

Results of Proficiency Test
Crude Oil Assay
October 2013

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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Report: iis13R02

March 2014

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1 INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies organizes a proficiency test for Crude Oil TBP / Assay. During the annual proficiency test program of 2013/2014, it was decided to continue the proficiency test for the analysis for Crude Oil TBP / Assay. In this interlaboratory study 24 laboratories in 19 different countries have participated. See appendix 6 for the number of participants per country.

In this report, the results of the 2013 proficiency test for Crude Oil TBP / Assay are presented and discussed. This report is electronically available through the iis internet site <http://www.iisnl.com>.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkensisse, the Netherlands, was the organizer of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkensisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010, since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2). This protocol may be downloaded from the iis website <http://www.iisnl.com>.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and are 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

The necessary bulk material was obtained from a local crude oil storage facility. The approx. 200 litre of Crude Oil was homogenised in a metal drum. After homogenisation, the bulk material was divided over 40 metal cans of 5 liter and labelled #13200.

The homogeneity of the subsamples was checked by determination of Density in accordance with ASTM D5002:13 of 8 stratified randomly selected samples.

	<i>Density @ 15 °C in kg/L</i>
sample #13200-1	0.88012
sample #13200-2	0.88015
sample #13200-3	0.88006
sample #13200-4	0.88010
sample #13200-5	0.88011
sample #13200-6	0.88013
sample #13200-7	0.88020
sample #13200-8	0.88028

table 1: homogeneity test results of subsamples #13200

From the above results of the homogeneity tests, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	<i>sample #13200</i>
Repeatability density kg/L	0.00019
Reference method	ASTM D5002:13
$0.3 \times R_{(\text{Reference method})}$	0.00109

table 2: evaluation of the density repeatability on subsamples #13200

The repeatability of the density results was in good agreement with the requirement based on the reference method. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories one or more cans of 5 L (as required) were sent on October 16, 2013.

2.5 ANALYSES

The participants were requested to determine a Crude Oil Assay consisting of a True Boiling Point Distillation in accordance with ASTM D2892 and the collection of 8 fractions and subsequently a distillation in accordance with ASTM D5236 and the collection of 5 fractions. On the original sample and on all fractions Density, Sulphur and Nitrogen content should be determined where possible. Furthermore on the light and heavy naphtha fractions a PIONA or PONA analysis was requested; on the combined fractions 4 +5 (kerosene and light gasoil) a D86-distillation and on the individual fractions 4 and 5 also a simdist determination. To get comparable results a detailed report form, on which the units and the standard methods were printed, was sent together with each set of samples. Also a letter of instructions and a SDS were added to the package.

3 RESULTS

During eight weeks after sample despatch the results of the individual laboratories were gathered. The original data are tabulated in the appendices of this report. The laboratories are presented by their code numbers.

After the planned deadline, a reminder fax was sent to those laboratories that had not yet reported any results at that moment.

Also 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 raw data of these tests (no reanalysis). Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

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

First the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers this check was repeated. In case a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs 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 them with a factor of 2.8.

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported 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 method is producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 7; nos.12 and 13).

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The 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 the fit-for-useness of the reported test result.

The z-scores were calculated in accordance with:

$$Z_{(\text{target})} = (\text{result} - \text{average of PT}) / \text{target standard deviation}$$

The $Z_{(\text{target})}$ 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 the proficiency test some problems were encountered with sample despatch to Greece Malaysia, Qatar and Russia. After the period of 4 weeks after sample despatch - originally set to report results - only seven participants had reported results. Therefore the deadline was extended to February 3, 2014. Evaluation of results and preparation of the final report were delayed significantly by these problems.

For objective evaluation iis uses standard reproducibilities to calculate target z-scores. Regretfully this is not possible for most density, sulphur and nitrogen data in this proficiency test, due to the fact that these results were determined on a distillation fraction produced by the laboratory itself and therefore also the distillation uncertainty is included in these test results.

Therefore other ways were sought to enable objective evaluation of the results gathered. From the masses of the collected fractions and the respective reported density, sulphur and nitrogen results, theoretical density, theoretical sulphur and theoretical nitrogen contents of the original crude sample were calculated by iis using below formulae. These theoretical values were compared with the measured values.

$$\textit{theoretical density} = \frac{\textit{original weight of dry sample}}{\sum_{i=1}^n \frac{\textit{weight of fraction } i}{\textit{density of fraction } i}}$$

$$\textit{theoretical sulfur content} = \frac{\sum_{i=1}^n (\textit{weight of fraction } i) \times (\textit{sulfur content of fraction } i)}{\textit{original weight of dry sample}}$$

$$\textit{theoretical nitrogen content} = \frac{\sum_{i=1}^n (\textit{weight of fraction } i) \times (\textit{nitrogen content of fraction } i)}{\textit{original weight of dry sample}}$$

4.1 EVALUATION PER TEST

- Density: The density determination on the original crude was not problematic. One statistical outlier was detected and the observed reproducibility is, after rejection of the statistical outlier in good agreement with the requirements of ASTM D5002:13.
- The density results on the 8 collected distillation fractions show relatively large differences between the reported test results of the participating laboratories, although in total only eleven statistical outliers (=5%) were detected. The relatively large observed spreads are most probably caused by differences in the distillations and not by differences in the performance of the density determinations. In each case the observed reproducibility will be the sum of the (small) spread in the density result and the (large) spread caused by the distillation. Therefore the observed reproducibilities were not compared with the literature requirements and consequently no z-scores were calculated.
- The average density for the first fraction (gas <30°C), after rejection of the statistical outlier is 0.57704 kg/L, which is in good agreement with the density of a mixture of C4 hydrocarbons and isopentane.
- Sulphur: The sulphur determination on the original crude sample showed no analytical problems. One statistical outlier was detected and the observed reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D4294:10.
- The sulphur results on the 8 collected distillation fractions show problems for several participating laboratories. In total 15 statistical outliers (=7%) were detected (from 8 laboratories).
- The observed reproducibility per fraction will be the sum of the spread in the sulphur result and the spread caused by the distillation. Therefore the observed reproducibilities were not compared with the literature requirements and consequently no z-scores were calculated.
- The gradual decrease of the relative standard deviation from the first to the last distillation fraction is in agreement with the expectations.
- Nitrogen: The nitrogen determination on the original crude sample was problematic. No statistical outliers were detected. However, the observed reproducibility is not at all in agreement with the requirements of ASTM D5762:12. It should be noticed that methods ASTM D4629 and ASTM D6069 are not applicable for Crude Oil, but limited to 'liquid hydrocarbons boiling in the range from approximately 50°C to 400°C'.
- The nitrogen results on the 8 collected distillation fractions show problems for several participating laboratories. In total 5 statistical outliers (=5%) from 3 laboratories were detected.
- The observed reproducibility per fraction will be the sum of the spread in the nitrogen result and the spread caused by the distillation. Therefore the observed reproducibilities were not compared with the requirements and consequently no z-scores were calculated.
- The gradual decrease of the relative standard deviation from the first to the last distillation fraction is in agreement with the expectations.

- Water: The water determination on the original crude was not problematic. Two statistical outliers were detected. The observed reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4377:11.
- P(iP)NA: This determination was performed on fractions 2 (light naphtha) and 3 (heavy naphtha) only. Serious analytical problems were observed on fraction 2 (light naphtha). In total 23 statistical outliers were detected (=14%). On the heavier fraction 3, 'only' eight statistical outliers were detected (=5%).
One laboratory used ASTM D5134, a method that is applicable up to C9. Therefore the test results of this laboratory on fraction 3 were excluded. For laboratory 1095, 8 of the 12 test results on fraction 2 turned out to be a statistical outlier. Because the 12 test results are not independent from each other, it was decided to exclude the other 4 test results of this laboratory.
Each observed reproducibility will be the sum of the spread in the analytical method and the spread caused by the D2892. Therefore the observed reproducibilities were not compared with the requirements and consequently no z-scores were calculated. The variety of test methods used may partly explain the large spreads observed during this PT. The set-up of the correct integration window is most critical in the case of testing high naphthenic distillation fractions.
- D86 distillation: This determination was performed on the combined fractions 4+5 only. No analytical problems were observed. No statistical outliers were detected. Each observed reproducibility will be the sum of the spread in the D86 result and the spread caused by the D2892. Therefore the observed reproducibilities were not compared with the requirements and consequently no z-scores were calculated.
- Simdist: This determination was performed on fractions 4 and 5 (kerosene and light gasoil) only. Most reported results showed a close resemblance. The goal was to enable evaluation of the column efficiency in accordance with appendix X2 of ASTM D2892:13.
From the reported results the ECP (effective cut point) and the standard efficiency N_{minimum} were calculated. A number of results were not in agreement with the ASTM D2892:13 requirements.
The strength of this quality control method becomes clear when the results of this round are compared with the results of the previous round iis11R02. A number of laboratories participated in both rounds and it is remarkable to see that some participants (like lab. 445) improved the distillation, while for other participants (e.g. laboratory 1065, 1066, 1095 and 1714) the performance of the distillation was less than in the previous proficiency test, based on the calculation of the ECP and the Standard Efficiency.
- D2892+ D5236: The distillation gave problems for a number of laboratories. For ASTM D2892 in total 5 statistical outliers were detected (=2.9%) and one result was excluded from the statistical evaluation. After exclusion of the

statistical outliers, all calculated reproducibilities, except for the fraction '<30°C', are in good agreement with requirements of ASTM D2892:13. For ASTM D5236 in total 2 statistical outliers were detected (=2.3%). After exclusion of the statistical outliers, none of the calculated reproducibilities is in agreement with requirements of ASTM D5236:13.

The start of the true boiling point curves (cum%M/M vs temp AET) of the laboratories show a high resemblance. The curve of laboratory 574 showed a positive deviation at the upper part of the curve (370°C-565°C). The curves for laboratories 862 and 1720 showed a negative deviation at the upper part of the curve (370°C-530°C). Laboratory 862 reported a relatively small fraction 520-565°C and laboratory 1720 reported a very small fraction 370-420°C. These observations are in agreement with the z-scores calculated.

The true boiling point curves (S in %M/M vs temp AET) of most laboratories show a high resemblance. The curves for laboratories 445 and 1264 show a negative deviation at the upper part of the curve (230°C-310°C). The deviations are probably due to analytical problems with the sulphur determination (on several fractions) and not with the distillation.

The true boiling point curves (N in %M/M vs temp AET) of most laboratories show a low resemblance. This is probably due to problems with the nitrogen determination and not with the distillation.

The evaluation of the Total Mass balance showed only five (!) recoveries that do meet the ASTM D2892 (paragraph 11.2) requirement of 0.4% max loss and the ASTM D5236 requirement of a recovery between 99.6 and 100.1%.

For the individual recoveries only eight recoveries could be taken into account due to the lack of available densities (mainly for fraction <30°C). After calculation of the Total Volume recovery, one of the eight recoveries was smaller than the Total Mass recovery. This is not to be expected as due to volume expansion a volume gain is to be expected rather than a volume loss, see paragraph 11.6 of ASTM D2892:13.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sd_R</i>	<i>R (lit)</i>
Density of #13200	kg/m ³	21	880.63	1.97	3.63
Sulphur of #13200	%M/M	19	2.030	0.078	0.115
Nitrogen of #13200	mg/kg	11	1318	624	351
Water	%V/V	16	0.0322	0.0329	0.0299
D2892 distillation	True boiling point curve				
LPG fraction < 30°C	%M/M	21	1.3	1.7	1.3
light naphtha 30 - 90°C	%M/M	21	4.3	1.0	1.3
heavy naphtha 90 - 180°C	%M/M	21	11.5	1.3	1.3
kerosene 180 - 215°C	%M/M	21	4.9	0.7	1.3
LGO 215 - 250°C	%M/M	21	5.7	0.8	1.5
MGO 250 - 310°C	%M/M	22	10.7	1.5	1.5
HGO 310 - 370°C	%M/M	21	10.4	1.1	1.5
residue > 370°C	%M/M	22	50.5	4.7	n.a.
D5236 distillation	True boiling point curve				
VGO 370 - 420°C	%M/M	17	5.3	5.3	2.6
VGO 420 - 470°C	%M/M	18	9.7	4.4	2.2
VGO 470 - 520°C	%M/M	17	7.1	2.1	1.8
VGO 520 - 565°C	%M/M	18	5.7	5.5	2.5
residue > 565°C	%M/M	18	22.4	9.5	n.a.

table 3: reproducibilities of original crude sample #13200

Without further statistical calculations it can be concluded that for almost all tests there is a good compliance of the group of participating laboratories with the relevant standards.

The tests that are problematic have been discussed in paragraph 4.1.

For the analytical test performed on the individual distillation fractions it was not possible to evaluate the reproducibilities against literature values as the observed reproducibilities include the distillation step that has a significant effect on these reproducibilities.

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R (lit)</i>
Density @15°C of cut 1: gas <30°C	kg/L	9	0.5770	0.0511	n.r.
Density @15°C of cut 2: 30-90°C	kg/L	19	0.6712	0.0110	n.r.
Density @15°C of cut 3: 90-180°C	kg/L	20	0.7539	0.0050	n.r.
Density @15°C of cut 4: 180-215°C	kg/L	21	0.8017	0.0051	n.r.
Density @15°C of cut 5: 215-250°C	kg/L	20	0.8223	0.0046	n.r.
Density @15°C of cut 6: 250-310°C	kg/L	22	0.8547	0.0065	n.r.
Density @15°C of cut 7: 310-370°C	kg/L	20	0.8829	0.0067	n.r.
Density @15°C of residue: >370°C	kg/L	19	0.9788	0.0066	n.r.
Density @15°C of cut 8: 370-420°C	kg/L	16	0.9098	0.0133	n.r.
Density @15°C of cut 9: 420-470°C	kg/L	16	0.9254	0.0104	n.r.
Density @15°C of cut 10: 470-520°C	kg/L	16	0.9442	0.0056	n.r.
Density @15°C of cut 11: 520-565°C	kg/L	16	0.9585	0.0082	n.r.
Density @15°C of residue: >565°C	kg/L	12	1.0378	0.0193	n.r.

table 4: reproducibilities of density determinations on distillation fractions

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sdR</i>	<i>R (lit)</i>
Sulphur on cut 1: gas <30°C	%M/M	3	0.018	n.a.	n.r.
Sulphur on cut 2: 30-90°C	%M/M	16	0.009	0.012	n.r.
Sulphur on cut 3: 90-180°C	%M/M	15	0.035	0.012	n.r.
Sulphur on cut 4: 180-215°C	%M/M	16	0.126	0.022	n.r.
Sulphur on cut 5: 215-250°C	%M/M	15	0.263	0.046	n.r.
Sulphur on cut 6: 250-310°C	%M/M	17	0.865	0.145	n.r.
Sulphur on cut 7: 310-370°C	%M/M	17	1.568	0.175	n.r.
Sulphur on residue: >370°C	%M/M	15	3.401	0.189	n.r.
Sulphur on cut 8: 370-420°C	%M/M	16	1.854	0.256	n.r.
Sulphur on cut 9: 420-470°C	%M/M	16	2.065	0.255	n.r.
Sulphur on cut 10: 470-520°C	%M/M	15	2.524	0.252	n.r.
Sulphur on cut 11: 520-565°C	%M/M	16	3.038	0.399	n.r.
Sulphur on residue: >565°C	%M/M	12	4.633	0.680	n.r.

table 5: reproducibilities of sulphur determinations on distillation fractions

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sdR</i>	<i>R (lit)</i>
Nitrogen on cut 1: gas <30°C	mg/kg	2	n.a.	n.a.	n.r.
Nitrogen on cut 2: 30-90°C	mg/kg	7	0.6	1.9	n.r.
Nitrogen on cut 3: 90-180°C	mg/kg	9	0.5	0.9	n.r.
Nitrogen on cut 4: 180-215°C	mg/kg	8	1.0	1.1	n.r.
Nitrogen on cut 5: 215-250°C	mg/kg	9	3.0	3.7	n.r.
Nitrogen on cut 6: 250-310°C	mg/kg	12	18.8	23.5	n.r.
Nitrogen on cut 7: 310-370°C	mg/kg	10	190.5	86.5	n.r.
Nitrogen on residue: >370°C	mg/kg	9	2612	1483	n.r.
Nitrogen on cut 8: 370-420°C	mg/kg	8	601	373	n.r.
Nitrogen on cut 9: 420-470°C	mg/kg	8	875	469	n.r.
Nitrogen on cut 10: 470-520°C	mg/kg	8	1441	523	n.r.
Nitrogen on cut 11: 520-565°C	mg/kg	8	1958	448	n.r.
Nitrogen on residue: >565°C	mg/kg	6	4610	989	n.r.

table 6: reproducibilities of nitrogen determinations on distillation fractions

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sdR</i>	<i>R (lit)</i>
Total Paraffins	%V/V	13	86.05	1.93	n.r.
C1-C4	%V/V	9	1.51	2.13	n.r.
n-paraffins	%V/V	11	46.79	1.40	n.r.
i-paraffins	%V/V	11	39.39	1.55	n.r.
naphthenes	%V/V	13	12.57	2.03	n.r.
aromatics	%V/V	13	1.38	0.49	n.r.
Total Paraffins	%M/M	14	84.09	2.97	n.r.
C1-C4	%M/M	9	1.30	1.86	n.r.
n-paraffins	%M/M	11	45.32	1.35	n.r.
i-paraffins	%M/M	11	38.70	1.32	n.r.
naphthenes	%M/M	14	14.12	2.95	n.r.
aromatics	%M/M	14	1.79	0.64	n.r.

table 7: reproducibilities of P(iP)NA determination on distillation fraction 2 (light naphtha)

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sdR</i>	<i>R (lit)</i>
Total Paraffins	%V/V	13	60.65	2.94	n.r.
C1-C4	%V/V	11	0.08	0.22	n.r.
n-paraffins	%V/V	12	27.42	2.24	n.r.
i-paraffins	%V/V	11	32.97	2.42	n.r.
naphthenes	%V/V	12	26.49	1.40	n.r.
aromatics	%V/V	13	12.49	1.18	n.r.
Total Paraffins	%M/M	15	58.01	4.60	n.r.
C1-C4	%M/M	12	0.06	0.16	n.r.
n-paraffins	%M/M	12	25.69	2.15	n.r.
i-paraffins	%M/M	11	31.56	2.43	n.r.
naphthenes	%M/M	15	27.02	3.66	n.r.
aromatics	%M/M	14	14.36	1.50	n.r.

table 8: reproducibilities of P(iP)NA determination on distillation fraction 3 (heavy naphtha)

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sdR</i>	<i>R (lit)</i>
ibp	°C	20	189.8	11.8	n.r.
5% evaporated	°C	15	198.8	11.8	n.r.
10% evaporated	°C	15	201.2	10.7	n.r.
50% evaporated	°C	15	213.7	9.7	n.r.
90% evaporated	°C	15	233.0	10.9	n.r.
95% evaporated	°C	15	237.7	10.9	n.r.
fbp	°C	20	245.2	19.0	n.r.
5% recovered	°C	19	198.6	12.1	n.r.
10% recovered	°C	19	201.1	9.4	n.r.
50% recovered	°C	19	213.3	9.3	n.r.
90% recovered	°C	19	232.4	10.0	n.r.
95% recovered	°C	19	237.2	9.7	n.r.

table 9: reproducibilities of D86 determination on combined distillation fraction 4+5

4.3 DISCUSSION

Obviously the normal time schedule is not sufficient for the completion of a round robin on Crude Oil Assay. Due to the limited resources of several participating laboratories, it was impossible to gather all results in the period of 4 weeks as set. Many laboratories have only one apparatus available and several apparatus were unforeseen not operational during the start of the round robin.

Nevertheless, in spite of the practical problems and the differences between the methods used, the distillation curves of most participating laboratories show a remarkable resemblance.

The density, sulphur and nitrogen results do show more aberrant results. These deviations may be (partly) explained by the cumulative effect of spread caused by distillation and by subsequent analytical determinations. The large range of density results on the light naphtha fraction may be caused by not or insufficient debutanization before the start of the distillation. Several densities reported on this fraction are very low, indicating the presence of C3 and C4 components. Also, twelve laboratories did report not to have collected any gasfraction, which may mean that these components did get in the light naphtha fraction unwantedly.

Still, the calculated averages for the theoretical density, sulphur and nitrogen content do show a good resemblance with the averages of the originally measured density and sulphur content (see below table).

Parameter	unit	average measured result	average theoretical result	average difference	average recovery
Density	kg/L	0.8806	0.8858	-0.0052	>100%
Sulphur	%M/M	2.0302	1.9633	+0.0629	97%
Nitrogen	mg/kg	1318	1322	-4	100%

table 10: comparison of actual measured values and theoretical values calculated from the fractions

The total mass balance varies from 95.92% up to 100.53%. ASTM D2892 does not give criteria for recovery, except that is stated that weight loss in excess of 0.4 % is a reason to discard the distillation data (see paragraph 11.2). It is remarkable to find that a large number of participating laboratories did not fulfil this latter requirement in this proficiency test (see page 36). For comparison, in ASTM D5236 is stated that the total recovery must be between 99.6 % and 100.1 % of the weight of the charge to be acceptable.

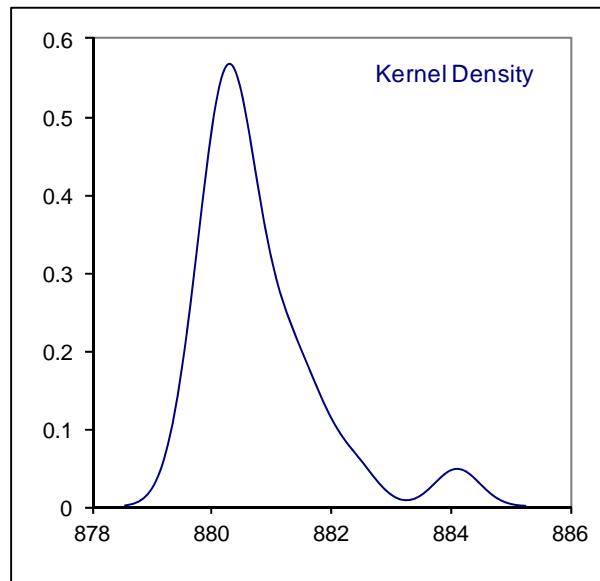
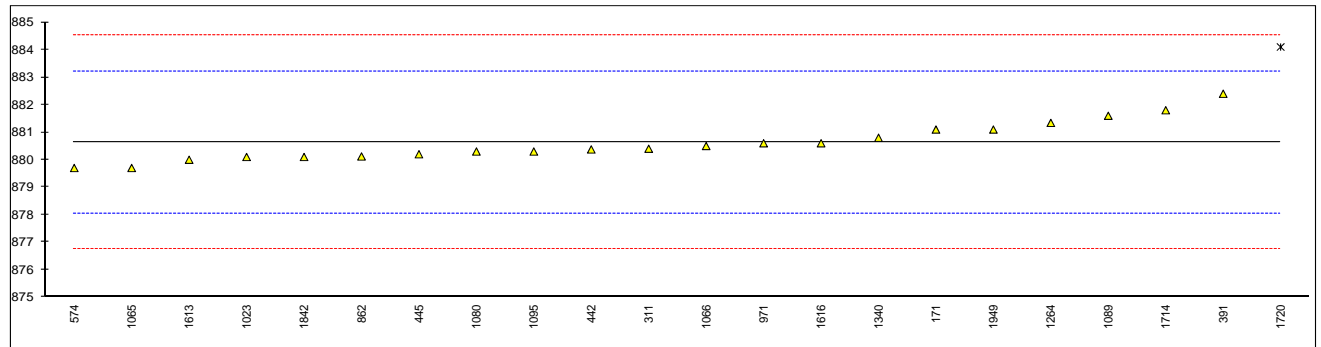
The average theoretical sulphur content shows an excellent resemblance to the average measured sulphur content and also the individual differences are rather small (0.003 - 0.136 %M/M). The calculated reproducibility of the calculated theoretical sulphur content is almost in agreement with the requirement of ASTM D4294:10. The sulphur determination obviously is much less sensitive than the density determination. This will be caused by the fact that the sulphur is present as a series of homologs.

The average theoretical nitrogen content shows a very good resemblance to the average measured nitrogen content (1322 vs. 1318 mg/kg). However, the individual values vary over a large range (1.5 – 419.15 mg/kg). The calculated reproducibility of the calculated theoretical nitrogen content is not at all in agreement with the requirement of ASTM D5762:10. Most probably the problematic nitrogen determination and the relatively high limit of detection of this determination are the main causes for the large spread rather than the distillation.

APPENDIX 1

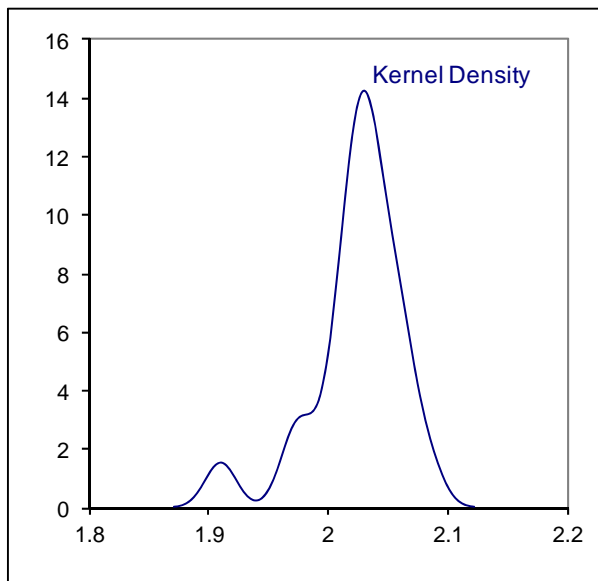
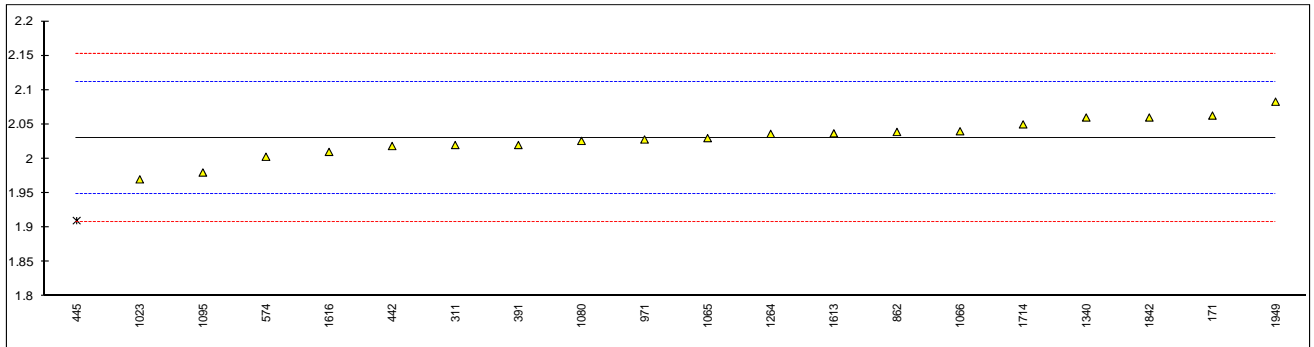
Determination of Density @15°C on original sample #13200; results in kg/m³

lab	method	value	mark	z(targ)	remarks
171	D5002	881.1		0.37	
311	D5002	880.4		-0.17	
391	D5002	882.4		1.37	
442	IP365	880.375		-0.19	
445	D5002	880.2		-0.33	
574	D7042	879.7		-0.71	
608		----		----	
862	D5002	880.12		-0.39	
971	D5002	880.6		-0.02	
1023	D5002	880.1	C	-0.41	Reported 0.8801
1065	D1298	879.7		-0.71	
1066	D5002	880.5	C	-0.10	Reported 0.8805
1080	D4052	880.3		-0.25	
1089	D5002	881.6	C	0.75	Reported 0.8816
1095	D5002	880.3		-0.25	
1108		----		----	
1264	D4052	881.34	C	0.55	Reported 0.88134
1340	ISO12185	880.8		0.13	
1613	D5002	880.0		-0.48	
1616	D4052	880.6		-0.02	
1714	D5002	881.8	C	0.91	Reported 0.8818
1720	D5002	884.1	G(0.01)	2.68	
1842	IP365	880.1	C	-0.41	Reported 0.8801
1949	D5002	881.1		0.37	
normality		OK			
n		21			
outliers		1			
mean (n)		880.63			
st.dev. (n)		0.704			
R(calc.)		1.97			
R(D5002:13)		3.63			



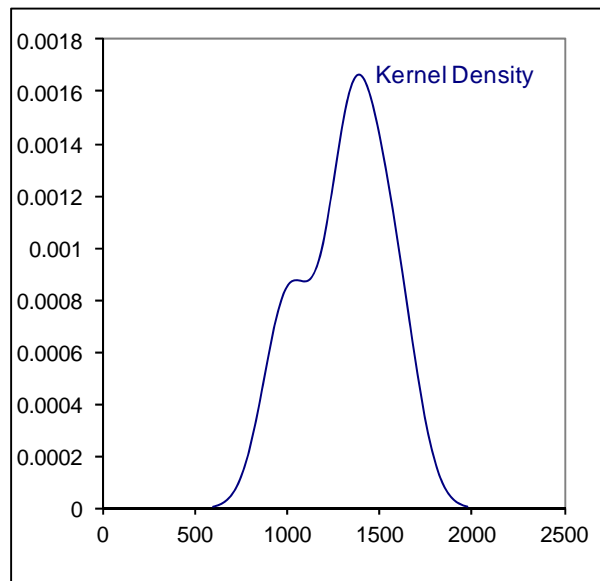
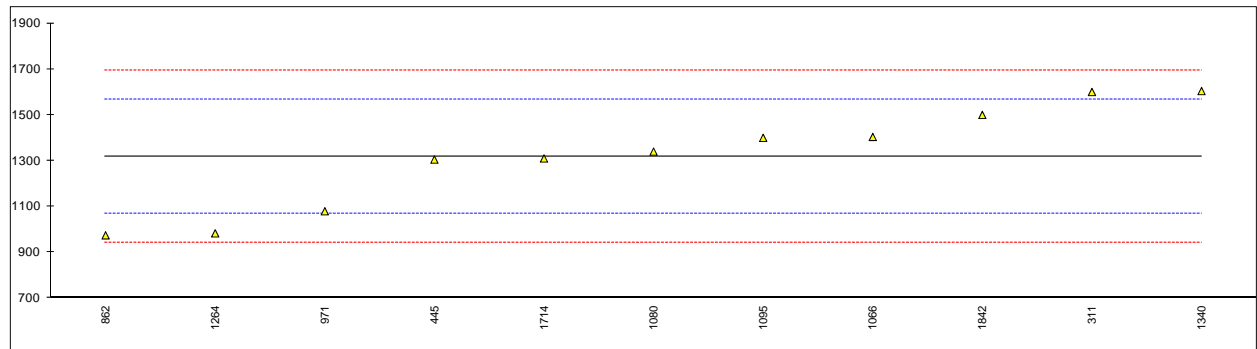
Determination of Sulphur on original sample #13200; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D4294	2.0628		0.80	
311	D4294	2.02		-0.25	
391	D4294	2.02		-0.25	
442	D4294	2.0185		-0.29	
445	IP336	1.91	G(0.05)	-2.93	
574	D4294	2.00292		-0.67	
608		-----		-----	
862	D2622	2.039		0.22	
971	D4294	2.028		-0.05	
1023	D4294	1.97		-1.47	
1065	IP336	2.03		0.00	
1066	D2622	2.04		0.24	
1080	D4294	2.026		-0.10	
1089		-----		-----	
1095	D4294	1.98		-1.23	
1108		-----		-----	
1264	D4294	2.0362		0.15	
1340	ISO8754	2.06	C	0.73	First reported 0.68
1613	D4294	2.037		0.17	
1616	D4294	2.01		-0.49	
1714	D2622	2.05		0.48	
1720		-----		-----	
1842	INH-05	2.06		0.73	
1949	D4294	2.083		1.29	
normality		OK			
n		19			
outliers		1			
mean (n)		2.0302			
st.dev. (n)		0.02783			
R(calc.)		0.0779			
R(D4294:10)		0.1147			



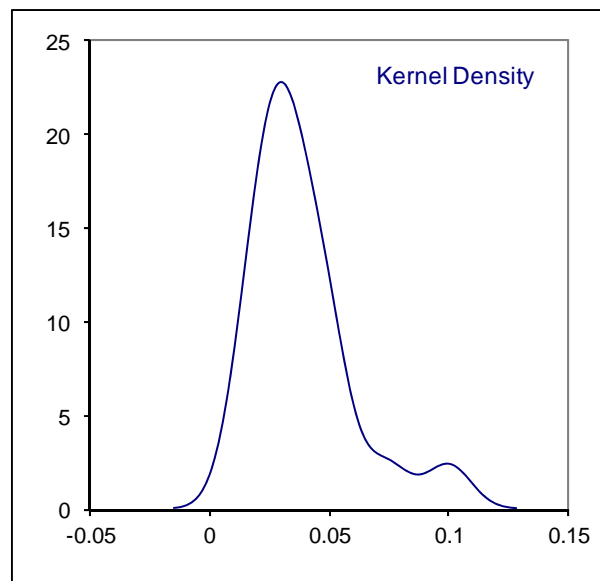
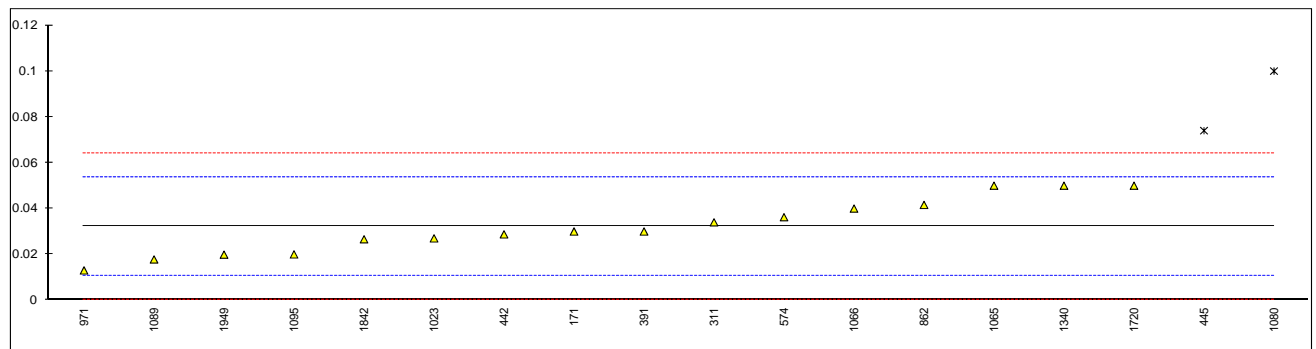
Determination of Nitrogen on original sample #13200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		----		----	
311	D5762	1600		2.25	
391		----		----	
442		----		----	
445	D5762	1305.1		-0.10	
574		----		----	
608		----		----	
862	D4629	974.5		-2.74	See §4.1
971	D5762	1079.54		-1.91	
1023		----		----	
1065		----		----	
1066	D5762	1404		0.69	
1080	D4629	1340		0.17	See §4.1
1089		----		----	
1095	D5762	1400		0.65	
1108		----		----	
1264	D6069	983		-2.68	See §4.1
1340	D5762	1604.15		2.28	
1613		----		----	
1616		----		----	
1714	D5762	1310		-0.07	
1720		----		----	
1842	INH-12	1500		1.45	
1949		----		----	
	normality	OK		OK	<u>Only ASTM D5762 data:</u>
	n	11		7	
	outliers	0		0	
	mean (n)	1318.21		1386.11	
	st.dev. (n)	222.810		182.640	
	R(calc.)	623.87		511.39	
	R(D5762:12)	350.64		368.71	



Determination of Water on original sample #13200; results in %V/V

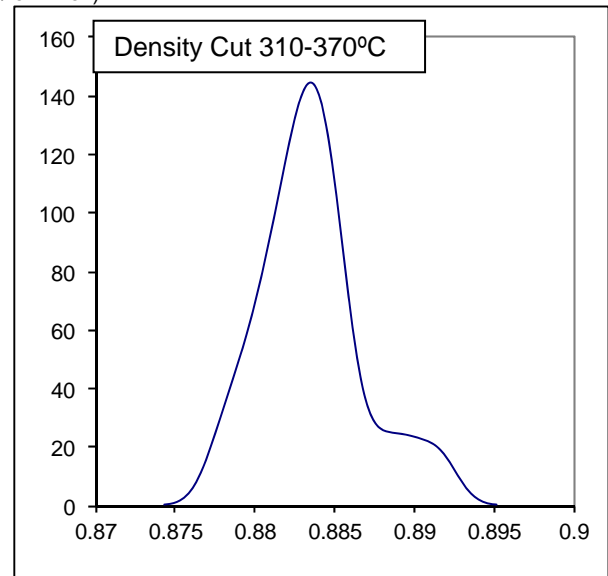
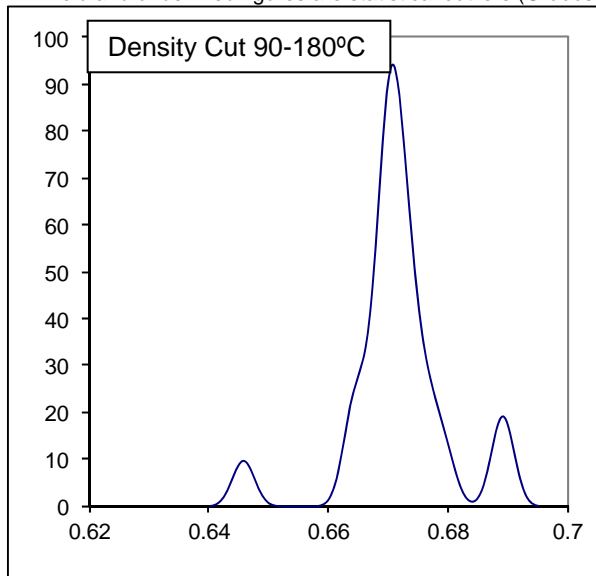
lab	method	value	mark	z(targ)	remarks
171	D4377	0.03		-0.20	
311	D4928	0.034		0.17	
391	D4377	0.03		-0.20	
442	IP386	0.02875		-0.32	
445	D4377	0.074	G(0.05)	3.92	
574	D4377	0.03624		0.38	
608		----		----	
862	D4377	0.0416		0.88	
971	D4377	0.013		-1.80	
1023	D4377	0.027		-0.49	
1065	D95	0.05		1.67	
1066	D4377	0.04		0.73	
1080	D4377	0.1	G(0.05)	6.35	
1089	D4377	0.0178	C	-1.35	Reported 178
1095	D6304	0.02		-1.14	
1108		----		----	
1264	D95	<0.05		----	
1340	ISO9029	0.05		1.67	
1613		----		----	
1616	D4006	<0.05		----	
1714	D4006	<0.05		----	
1720	D4006	0.05		1.67	
1842	IP356	0.0266		-0.52	
1949	D4377	0.0199		-1.15	
normality		OK			
n		16			
outliers		2			
mean (n)		0.03218			
st.dev. (n)		0.011763			
R(calc.)		0.03294			
R(D4377:11)		0.02990			



Determination of Density @15°C on distillation fractions; results in kg/L

lab	method	Gas LPG <30°C	L.Naphtha 30-90°C	H.Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C
171	D4052	0.5933	0.6705	0.7516	0.8013	0.8194	0.8494	0.8779	0.974
311	D4052	0.5822	0.6759	0.7546	0.8014	0.8239	0.8554	0.8804	0.9764
391	D4052	0.5939	0.6713	0.7553	0.8009	0.8260	0.8540	0.8836	0.9822
442	IP365	----	0.6693	0.7581	0.8024	0.8218	0.8554	0.8834	0.9804
445	D4052	0.6011	0.6734	0.7539	0.8032	0.8223	0.8509	0.8790	0.9790
574	D7042	----	0.6796	0.7548	0.8014	0.8231	0.8556	0.8846	----
608		----	----	----	----	----	----	----	----
862	D4052	0.5666	0.6696	0.7524	0.8045	0.8250	0.8566	0.8835	0.9802
971	D4052	----	0.6694	0.7600	0.8015	0.8225	0.8547	0.8857	0.9808
1023	D4052	----	0.6737	0.7551	0.8010	0.8224	0.8553	0.8852	0.9787
1065	D4052	----	0.6738	0.7537	0.8013	0.8212	0.8564	0.8836	0.9793
1066	D4052	0.5576	0.6708	0.7541	0.8023	0.8215	0.8552	0.8820	0.9769
1080	D4052	----	0.6459	0.7536	0.8011	0.8203	0.855	0.8878	0.983
1089	D4052	----	0.6645	0.7517	0.7997	0.8208	0.8539	0.8827	0.9779
1095	D4052	0.5674	0.6895	0.7600	0.8061	0.8292	0.8603	0.8896	0.9806
1108		----	----	----	----	----	----	----	----
1264	D4052	0.5840	0.6889	0.7520	----	0.8210	0.8531	0.8800	----
1340	ISO12185	0.5473	0.6668	0.7534	0.8009	0.8220	0.8549	0.8833	0.9773
1613	D4052	----	0.6718	0.7569	0.8041	0.8278	0.8573	0.8916	0.9812
1616	D4052	----	0.6688	0.7508	0.8014	0.8229	0.8546	0.8811	0.9791
1714	D4052	----	0.6714	0.7526	0.7991	0.8208	0.8513	0.8848	----
1720	D4052	0.654	0.6642	0.7535	0.7978	0.8219	0.8525	0.8848	0.9756
1842	IP365	----	0.6712	0.7547	0.8009	0.8219	0.8545	0.8818	0.9786
1949	D4052	----	0.6772	0.7544	0.8029	0.8244	0.8565	0.8819	0.9762
normality		OK	OK	OK	not OK	OK	OK	OK	OK
n		9	19	20	21	20	22	20	19
outliers		1	3	2	0	2	0	2	0
mean (n)		0.57704	0.67122	0.75386	0.80168	0.82226	0.85467	0.88286	0.97881
st.dev. (n)		0.018258	0.003916	0.001778	0.001828	0.001623	0.002320	0.002408	0.002353
R(calc.)		0.05112	0.01096	0.00498	0.00512	0.00455	0.00650	0.00674	0.00659

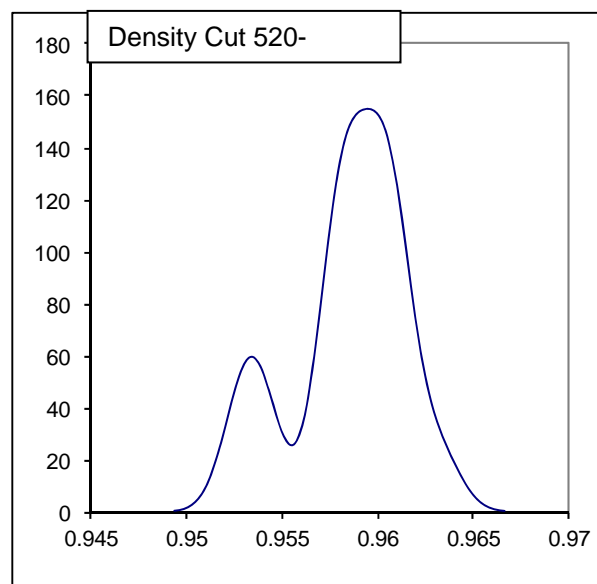
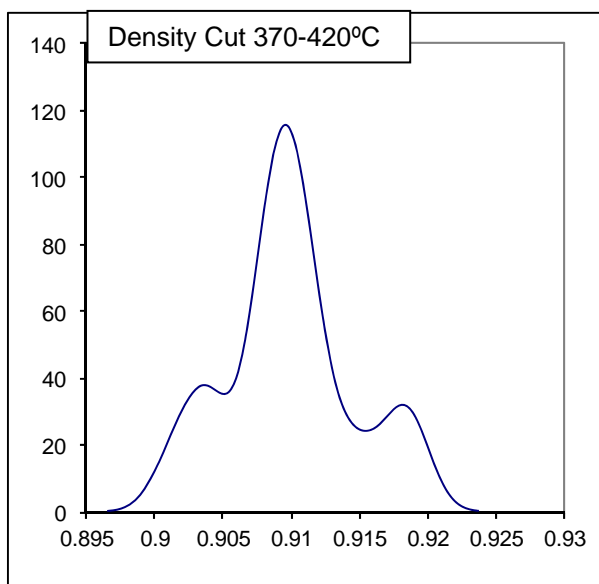
NB: Bold and underlined figures are statistical outliers (Grubbs and / or Dixon)



Determination of Density @15°C on distillation fractions (continued); results in kg/L

lab	Method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	VGO 520-565°C	Residue >565°C	remarks
171	D4052	0.9110	0.9279	0.9464	0.9606	1.041	D163 / D70
311	D4052	0.9097	0.9269	0.9461	0.9632	1.0361	
391	D4052	0.9080	0.9289	0.9451	0.9609	1.052	
442		----	----	----	----	----	
445	D4052	0.9180	0.9306	0.9440	0.9528	1.0270	D70
574	D7042	0.9150	0.9298	0.9478	0.9580	----	
608		----	----	----	----	----	
862	D4052	0.9188	0.9281	0.9410	0.9534	1.0345	GB/T 13377
971	IP365	0.9082	0.9224	0.9445	0.9610	1.035	
1023	IP336	----	----	----	----	----	
1065	D7777	0.904	0.920	0.9417	0.9607	----	
1066	D4052	0.9014	0.9223	0.9427	0.9580	1.0357	
1080	D4052	0.9095	0.9247	0.9455	0.9593	----	
1089		----	----	----	----	----	
1095	D4052	0.9107	0.9266	0.9466	0.9594	1.0456	
1108		----	----	----	----	----	
1264	D4052	0.9077	0.9255	0.9423	0.9586	----	
1340		----	----	----	----	----	
1613	D4052	0.9123	0.9261	0.944	0.9576	1.0392	
1616	D4052	0.9037	0.9168	0.9415	0.9540	1.040	
1714	D70	----	----	----	----	(*)	(*) Reported relative density only
1720	D4052	----	----	----	----	<u>1.0108</u>	
1842	IP365	0.9094	0.9265	0.9431	0.9580	1.0394	
1949	D4052	0.9100	0.9231	0.9446	0.9603	1.0278	
normality		OK	OK	OK	OK	OK	
n		16	16	16	16	12	
outliers		0	0	0	0	1	
mean (n)		0.90984	0.92539	0.94418	0.95849	1.03777	
st.dev. (n)		0.004731	0.003698	0.002014	0.002925	0.006907	
R(calc.)		0.01325	0.01035	0.00564	0.00819	0.01934	

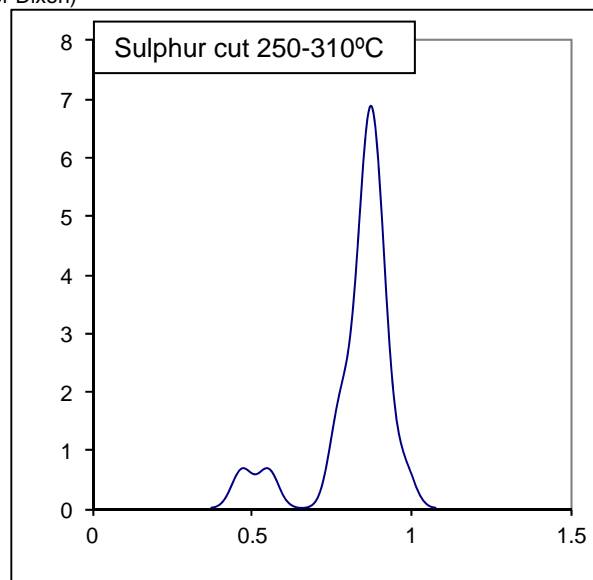
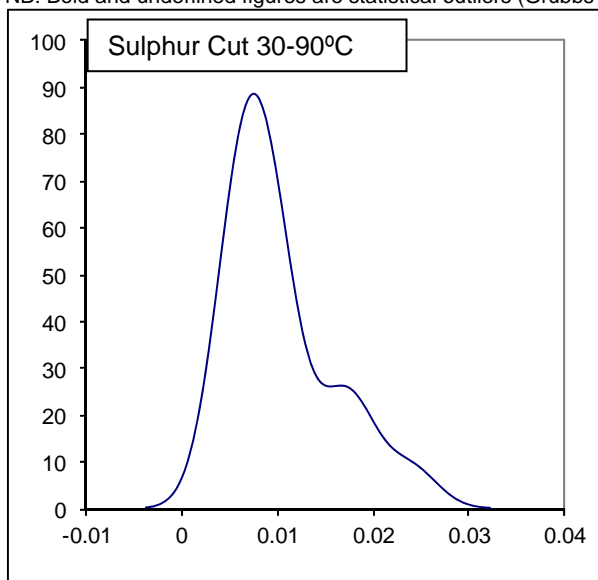
NB: Bold and underlined figures are statistical outliers (Grubbs and / or Dixon)



Determination of Sulphur on distillation fractions; results in %M/M

lab	Method	Gas LPG <30°C	L. Naphtha 30-90°C	H. Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C
171	D2622	----	0.00958	0.0354	0.124	0.226	0.765	1.452	3.48
311	D2622	----	0.0080	0.0365	0.122	0.280	0.873	1.54	----
391	D4294	----	0.006	0.033	0.130	0.261	0.843	1.52	3.40
442		----	----	----	----	----	----	----	----
445	D5453	----	0.00968	0.04	0.12	0.25	0.55	0.88	3.35
574	D4294	----	0.00644	0.03632	0.12226	0.28265	0.89563	1.57820	----
608		----	----	----	----	----	----	----	----
862	D5453	0.0480	0.0065	0.0264	0.126	0.282	0.878	1.58	3.30
971	D4294	----	0.0056	0.0317	0.110	0.269	0.844	1.607	3.437
1023	IP336	----	----	----	----	0.195	0.871	1.628	1.802
1065	IP336	----	0.0109	0.0358	0.13	0.27	0.91	1.59	3.38
1066	D2622	----	0.0178	0.0540	0.125	0.250	0.849	1.531	3.31
1080		----	----	----	----	----	----	----	----
1089		----	----	----	----	----	----	----	----
1095	D4294	0.0034	0.0102	0.0453	0.140	0.336	0.979	1.65	3.48
1108		----	----	----	----	----	----	----	----
1264	D4294	----	----	----	0.0908	0.2521	0.4680	1.3141	3.31
1340	ISO20847	----	0.0082	0.0318	0.13	0.26	0.87	1.61	3.39
1613	D4294	----	0.0077	0.039	0.133	0.323	0.919	1.708	3.499
1616	D4294	----	0.016	0.037	0.127	0.280	0.887	1.498	3.350
1714	D5453	----	0.0058	0.0328	0.116	0.248	0.801	1.56	3.41
1720	D5453	0.0022	0.0045	0.0317	----	----	0.777	1.498	2.891
1842	INH-05	----	0.018	0.036	0.119	0.256	0.855	1.54	3.44
1949		----	0.0240	0.0594	0.138	0.278	0.884	1.57	3.48
normality		n.a.	OK	OK	OK	OK	OK	OK	OK
n		3	16	15	16	15	17	17	15
outliers		n.a.	1	2	1	3	2	2	2
mean (n)		0.01787	0.00943	0.03525	0.12577	0.26298	0.86474	1.56825	3.40107
st.dev. (n)		n.a.	0.004301	0.004398	0.007802	0.016362	0.051922	0.062502	0.067482
R(calc.)		n.a.	0.01204	0.01231	0.02185	0.04581	0.14538	0.17501	0.18895
RSD in %		n.a.	45.6%	12.5%	6.2%	6.2%	6.0%	4.0%	2.0%

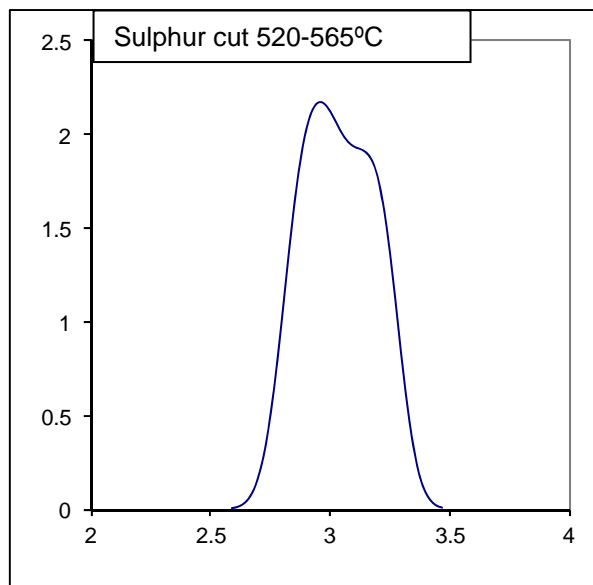
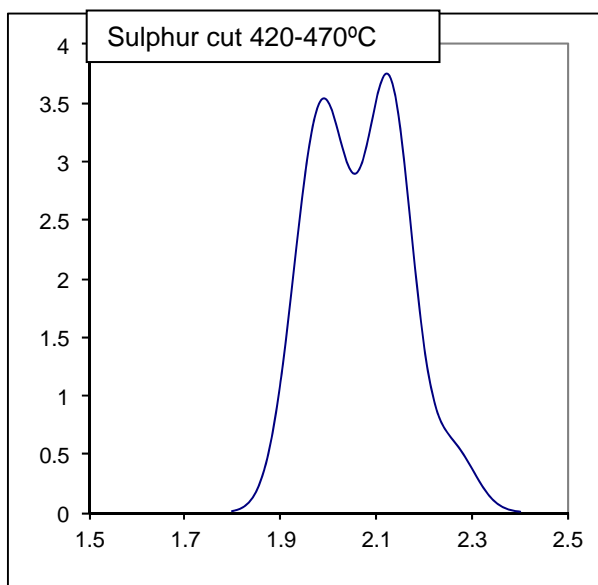
NB: Bold and underlined figures are statistical outliers (Grubbs and / or Dixon)



Determination of Sulphur on distillation fractions (continued); results in %M/M

lab	Method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	VGO 520-565°C	Residue >565°C	remarks
171	D4294	1.91	2.12	2.63	3.20	5.06	
311	D4294	1.90	2.12	2.62	3.12	4.18	
391	D4294	1.98	2.26	2.61	3.10	4.57	
442		----	----	----	----	----	
445	IP336	1.99	2.15	2.50	2.81	4.44	
574	D4294	1.89209	2.13007	2.57801	3.24345	----	
608		----	----	----	----	----	
862	D2622	2.01	2.12	2.52	2.93	4.31	
971	D4294	1.754	2.003	2.548	3.251	4.761	
1023		----	----	----	----	----	
1065	IP336	1.75	1.97	2.38	3.12	4.57	
1066	D2622	1.71	1.99	2.37	2.89	4.61	
1080		----	----	----	----	----	
1089		----	----	----	----	----	
1095	D4294	1.78	2.04	2.65	3.20	>4.6	ISO20847
1108		----	----	----	----	----	
1264	D4294	1.7754	1.9386	<u>2.2358</u>	2.8757	----	
1340		----	----	----	----	----	
1613	D4294	1.858	2.142	2.547	2.976	4.796	
1616	D4294	1.777	1.946	2.472	3.032	4.815	
1714	D2622	1.86	2.11	2.52	3.00	4.66	
1720	D4294	----	----	----	----	<u>3.303</u>	
1842	INH-05	1.88	1.99	2.39	2.87	----	
1949		1.83	2.01	2.52	2.99	4.82	
normality		OK	OK	OK	OK	OK	
n		16	16	15	16	12	
outliers		0	0	1	0	1	
mean (n)		1.85353	2.06498	2.52367	3.03801	4.63267	
st.dev. (n)		0.091596	0.091075	0.090032	0.142619	0.242966	
R(calc.)		0.25647	0.25501	0.25209	0.39933	0.68030	
RSD in %		4.9%	4.4%	3.6%	4.7%	5.2%	

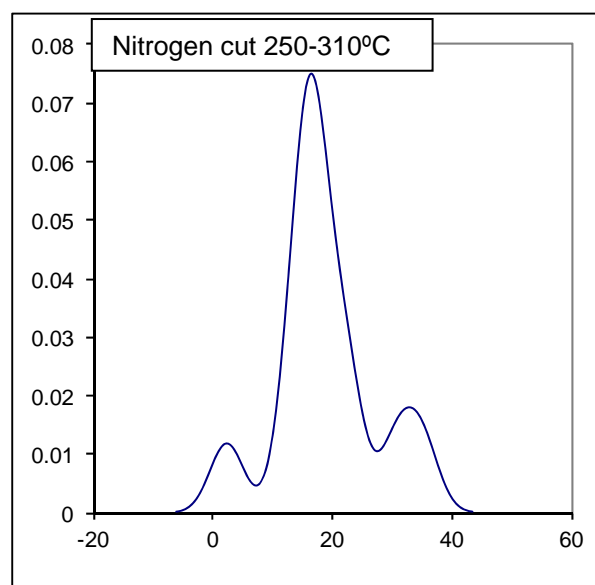
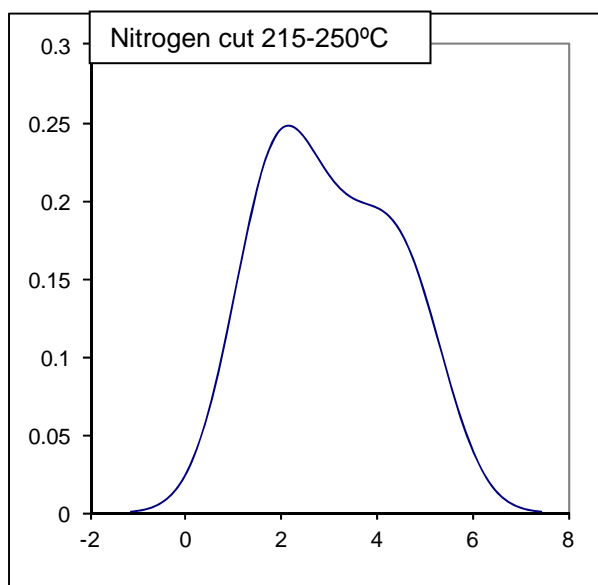
NB: Bold and underlined figures are statistical outliers (Grubbs and / or Dixon)



Determination of Nitrogen on distillation fractions; results in mg/kg

lab	method	Gas LPG <30°C	L.Naphtha 30-90°C	H. Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C
171	D4629	----	<1	<1	<1	<1	2.4	----	----
311	D4629	----	0.3	0.6	1.6	3.3	22.6	196.1	2900
391		----	----	----	----	----	----	----	----
442		----	----	----	----	----	----	----	----
445	D5762	----	----	----	----	----	----	213	3003
574		----	----	----	----	----	----	----	----
608		----	----	----	----	----	----	----	----
862	D4629	1	2	1	1	3	22	230	1789
971	D4629	----	0.402	0.438	0.756	2.014	16.64	219	2544
1023		----	----	----	----	----	----	----	----
1065		----	----	----	----	----	----	----	----
1066	D5762	----	1	1	1	2	16	203	2870
1080		----	----	----	----	----	----	----	----
1089		----	----	----	----	----	----	----	----
1095	D4629	<0.1	<0.3	0.3	1.4	4.3	30.9	----	----
1108		----	----	----	----	----	----	----	----
1264	D6069	----	0.1381	0.297	0.789	1.955	17.30	152	1856
1340	D4629	----	<u>4.40</u>	<u>4.62</u>	<u>4.73</u>	5.11	18.38	216.0	2313.7
1613	D4629	----	0.290	0.281	0.909	4.313	35.06	170.54	----
1616		----	----	----	----	----	----	----	----
1714	D5762	----	<1	<2	<5	<5	15.4	145	3330
1720	D4629	----	----	0.33	----	----	13.0	<u>68.0</u>	----
1842	INH-12	----	0.2	0.2	0.4	1.2	16	160	2900
1949		----	----	----	----	----	----	----	----
normality		n.a.	not OK	OK	OK	OK	OK	OK	OK
n		2	7	9	8	9	12	10	9
outliers		n.a.	1	1	1	0	0	1	0
mean (n)		n.a.	0.619	0.494	0.982	3.021	18.807	190.46	2611.7
st.dev. (n)		n.a.	0.6728	0.3085	0.3760	1.3349	8.3797	30.908	529.54
R(calc.)		n.a.	1.884	0.864	1.053	3.738	23.463	86.54	1482.7
RSD in %		n.a.	109%	62.4%	38.3%	44.2%	44.6%	16.2%	20.3%

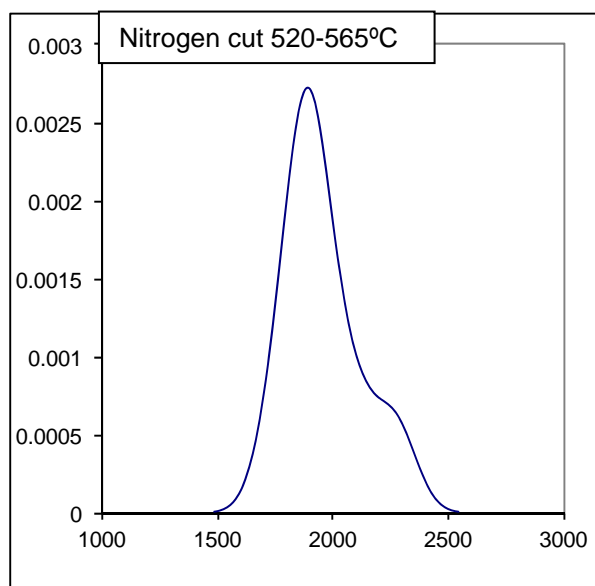
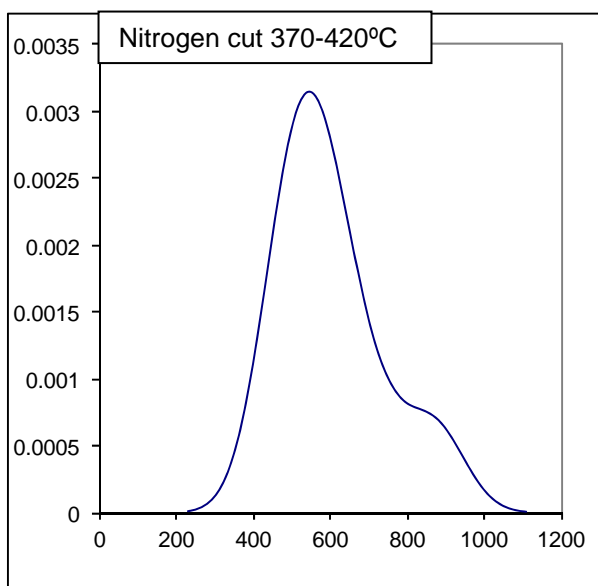
NB: Bold and underlined figures are statistical outliers (Grubbs and / or Dixon)



Determination of Nitrogen on distillation fractions (Continued); results in mg/kg

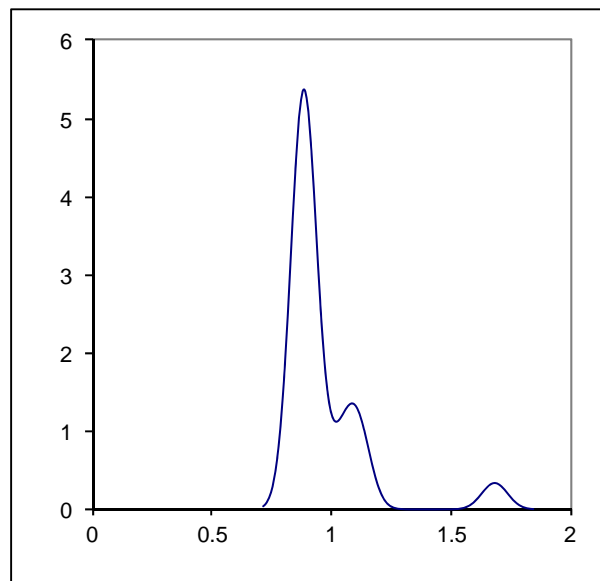
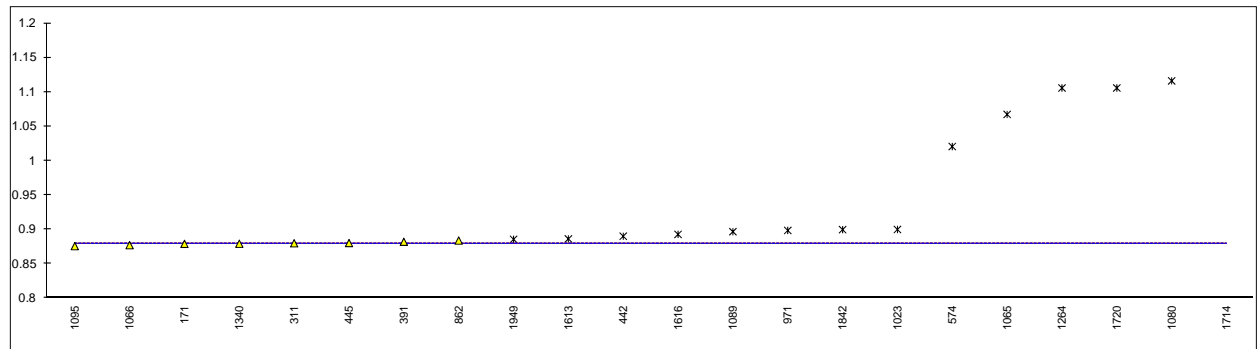
lab	method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	VGO 520-565°C	Residue >565°C	remarks
171		----	----	----	----	----	
311	D5762	600	900	1500	2100	4500	
391		----	----	----	----	----	
442		----	----	----	----	----	
445	D5762	870	1226	1770	2272	4982	
574		----	----	----	----	----	
608		----	----	----	----	----	
862	D4629	711	982	1433	1914	<u>2451</u>	
971	D5762	590	865	1425	1864	4019	
1023		----	----	----	----	----	
1065		----	----	----	----	----	
1066	D5762	505	800	1330	1980	4770	
1080		----	----	----	----	----	
1089		----	----	----	----	----	
1095		----	----	----	----	----	
1108		----	----	----	----	----	
1264	D6069	467	705	1167	1762	----	
1340		----	----	----	----	----	
1613		----	----	----	----	----	
1616		----	----	----	----	----	
1714	D5762	493	770	1600	1870	4490	
1720		----	----	----	----	----	
1842	INH-12	570	750	1300	1900	4900	
1949		----	----	----	----	----	
normality		OK	OK	OK	OK	OK	
n		8	8	8	8	6	
outliers		0	0	0	0	1	
mean (n)		600.75	874.75	1440.6	1957.8	4610.2	
st.dev. (n)		133.299	167.566	186.93	160.15	353.23	
R(calc.)		373.24	469.19	523.4	448.4	989.1	
RSD in %		22.2%	19.2%	13.0%	8.2%	7.7%	

NB: Bold and underlined figures are statistical outliers (Grubbs and / or Dixon)



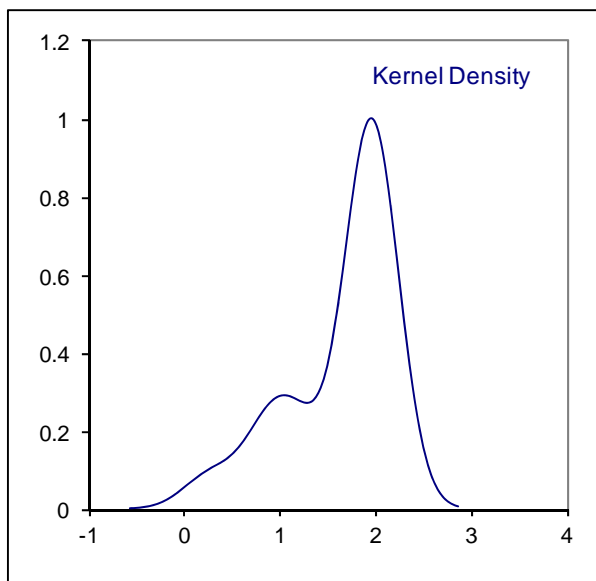
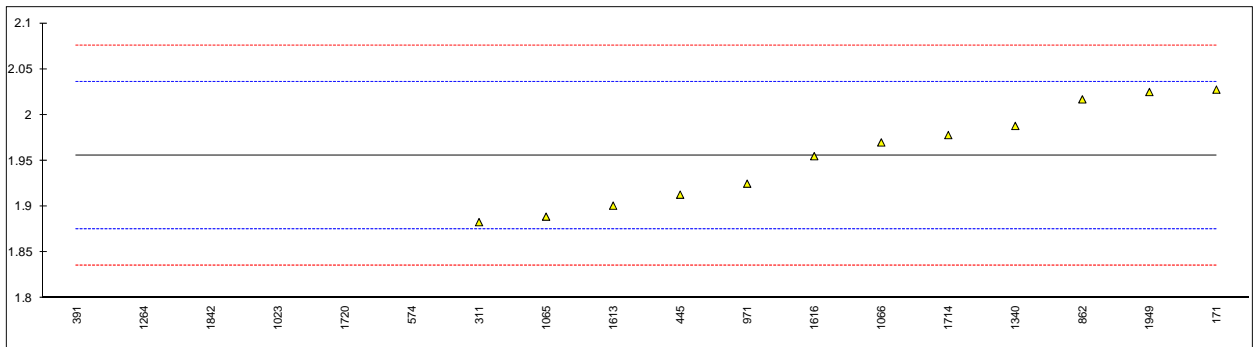
Determination of calculated theoretical Density @15°C of sample #13200; results in kg/L

lab	method	value	mark	z(targ)	remarks	Meas. density	difference
171	calc.	0.8791		----		0.8811	-0.0020
311	calc.	0.8799		----		0.8804	-0.0005
391	calc.	0.8821		----		0.8824	-0.0003
442	calc.	0.8902	ex	----	density cut 1 not reported	0.880375	----
445	calc.	0.8803		----		0.8802	+0.0001
574	calc.	1.0207	ex	----	Not enough data available	0.8797	----
608	calc.	----		----		----	----
862	calc.	0.8839		----		0.88012	+0.0038
971	calc.	0.8986	ex	----	density cut 1 not reported	0.8806	----
1023	calc.	0.9000	ex	----	density cut 1 not reported	0.8801	----
1065	calc.	1.0674	ex	----	Not enough data available	0.8797	----
1066	calc.	0.8772		----		0.8805	-0.0033
1080	calc.	1.1162	ex	----	Not enough data available	0.8803	----
1089	calc.	0.8968	ex	----	density cut 1 not reported	0.8816	----
1095	calc.	0.8758		----		0.8803	-0.0045
1108	calc.	----		----		----	----
1264	calc.	1.1059	ex	----	density residue not reported	0.88134	----
1340	calc.	0.8793		----		0.8808	-0.0015
1613	calc.	0.8863	ex	----	density cut 1 not reported	0.8800	----
1616	calc.	0.8929	ex	----	density cut 1 not reported	0.8806	----
1714	calc.	1.6821	ex	----	Not enough data available	0.8818	----
1720	calc.	1.1059	ex	----	Not enough data available	0.8841	----
1842	calc.	0.8997	ex	----	density cut 1 not reported	0.8801	----
1949	calc.	0.8857	ex	----	density cut 1 not reported	0.8881	----
normality		OK					
n		8					
outliers		0	+ 14 excl.				
mean (n)		0.87970			Average:	0.88063	+0.00093
st.dev. (n)		0.002557					
R(calc.)		0.00716					
R(D5002:13)		0.00365					



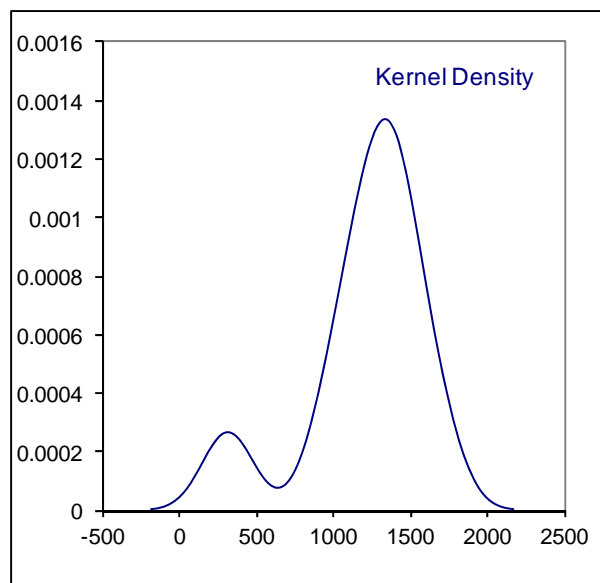
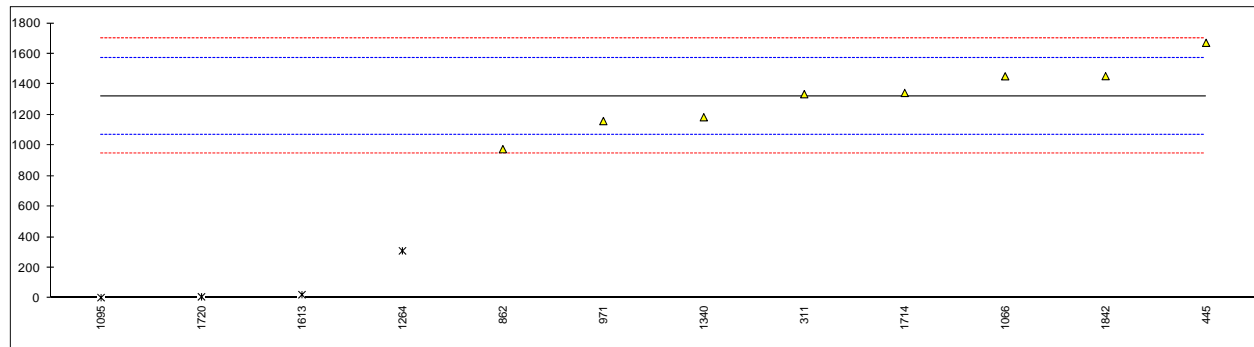
Determination of calculated theoretical sulphur content on sample #13200; results in %M/M

lab	method	Value	mark	z(targ)	remarks	Meas. sulphur	Δ absolute	rel%
171	calc.	2.0276		----		2.0628	-0.0352	-1.7
311	calc.	1.883		----		2.02	-0.137	-7.3
391	calc.	0.269	ex	----	Not enough data available	2.02	----	
442	calc.	----		----		2.0185	----	
445	calc.	1.913		----		1.91	+0.003	0.2
574	calc.	1.19520	ex	----	Not enough data available	2.00292	----	
608	calc.	----		----		----	----	
862	calc.	2.017		----		2.039	-0.022	-1.1
971	calc.	1.925		----		2.028	-0.103	-5.4
1023	calc.	1.184	ex	----	Not enough data available	1.97	----	
1065	calc.	1.889		----		2.03	-0.141	-7.5
1066	calc.	1.970		----		2.04	-0.070	-3.6
1080	calc.	----		----		2.026	----	
1089	calc.	----		----		----	----	
1095	calc.	>1.905		----		1.98	<0.075	>-3.9
1108	calc.	----		----		----	----	
1264	calc.	0.848	ex	----	Not enough data available	2.0362	----	
1340	calc.	1.988		----		2.06	-0.072	-3.6
1613	calc.	1.901		----		2.037	-0.136	-7.2
1616	calc.	1.955		----		2.01	-0.055	-2.8
1714	calc.	1.978		----		2.05	-0.072	-3.6
1720	calc	1.193	ex	----	Not enough data available	----	----	
1842	calc	0.879	ex	----	Not enough data available	2.06	----	
1949	calc	2.025		----		2.083	-0.058	-2.9
normality		OK						
n		12						
outliers		0	+ 6 excl					
mean (n)		1.9560			Average:	2.0302	+0.0742	
st.dev. (n)		0.05306						
R(calc.)		0.1486						
R(D4294:10)		0.1122						



Determination of calculated theoretical nitrogen content on sample #13200; results in mg/kg

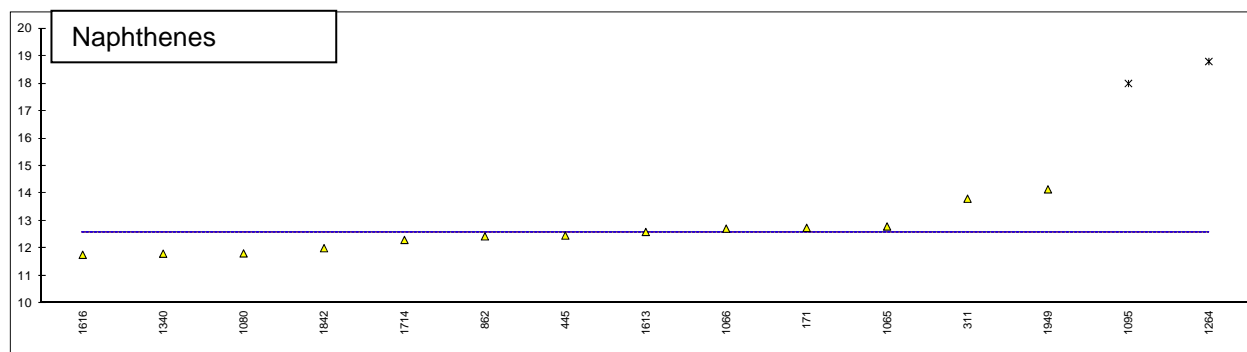
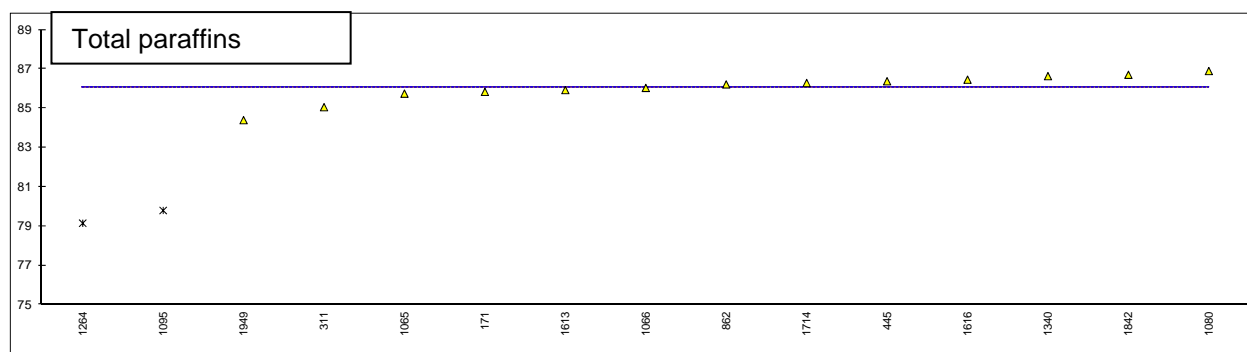
lab	method	value	mark	z(targ)	remarks	Meas. nitrogen	Δ absolute	rel%
171	calc.	----				----	----	
311	calc.	1336				1600	-264	-19.8
391	calc.	----				----	----	
442	calc.	----				----	----	
445	calc.	1672.4				1305.1	367.3	22.0
574	calc.	----				----	----	
608	calc.	----				----	----	
862	calc.	976.2				974.5	1.7	0.2
971	calc.	1160.27				1079.54	80.73	7.0
1023	calc.	----				----	----	
1065	calc.	----				----	----	
1066	calc.	1453				1404	49	3.4
1080	calc.	----				1340	----	
1089	calc.	----				----	----	
1095	calc.	4	ex		Not enough data available	1400	----	
1108	calc.	----				----	----	
1264	calc.	310.44	ex		Not enough data available	983.00	----	
1340	calc.	1185.11				1604.15	-419.04	-35.4
1613	calc.	23	ex		Not enough data available	----	----	
1616	calc.	----				----	----	
1714	calc.	1344				1310	34	2.5
1720	calc.	9	ex		Not enough data available	----	----	
1842	calc.	1454				1500	-46	-3.2
1949	calc.	----				----	----	
normality		OK						
n		8						
outliers		0	+4 excl.					
mean (n)		1322.6				Average	1318.2	-4.4
st.dev. (n)		214.65						
R(calc.)		601.0						
R(D5762:10)		351.8						



Determination of P(iP)NA on distillation fraction 2 (light naphtha); results in %V/V

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D5134	85.84	4.79	46.72	39.11	12.75	1.42	
311	D5443	85.06	1.63	45.92	39.14	13.81	1.11	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D5443Mod	86.38	1.86	46.86	39.52	12.47	1.14	
574		-----	-----	-----	-----	-----	-----	
608		-----	-----	-----	-----	-----	-----	
862	D6839	86.22	2.96	47.02	39.20	12.44	1.31	
971		-----	-----	-----	-----	-----	-----	
1023		-----	-----	-----	-----	-----	-----	
1065		85.75	0.64	47.13	38.62	12.80	1.45	
1066		86.04	1.85	47.28	38.76	12.72	1.24	
1080	in house	86.9	0.54	46.44	40.14	11.82	1.28	
1089		-----	-----	-----	-----	-----	-----	
1095		79.8	0.1 ex	41.6	38.2 ex	18.0	2.2	
1108		-----	-----	-----	-----	-----	-----	
1264	D5443	79.15	-----	44.41	34.74	18.80	2.04	
1340	ISO22854	86.64	-----	47.49	39.15	11.81	1.55	
1613	D6839	85.93	-----	-----	-----	12.60	1.46	
1616	D6839	86.46	1.31	45.99	40.47	11.77	1.77	
1714	in house	86.29	1.91	46.84	39.45	12.31	1.40	
1720		-----	-----	-----	-----	-----	-----	
1842		86.71	0.0	47.04	39.67	12.01	1.29	
1949	D5443	84.40	0.91	-----	-----	14.15	1.45	
	normality	OK	OK	OK	OK	OK	OK	
	n	13	9	11	11	13	13	
	outliers	2	2 + 1 ex.	2	1 + 1 ex	2	2	
	mean (n)	86.048	1.512	46.794	39.385	12.574	1.375	
	st.dev. (n)	0.6905	0.7593	0.4993	0.5524	0.7263	0.1760	
	R(calc.)	1.933	2.126	1.398	1.547	2.034	0.493	

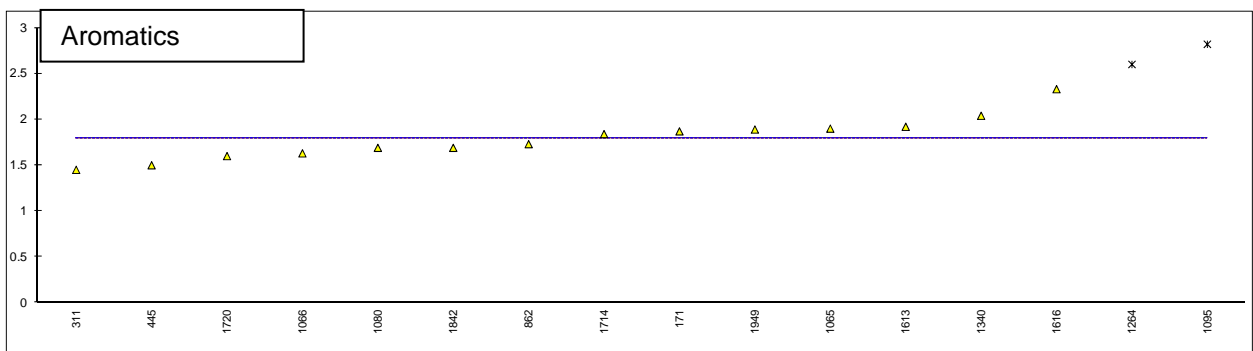
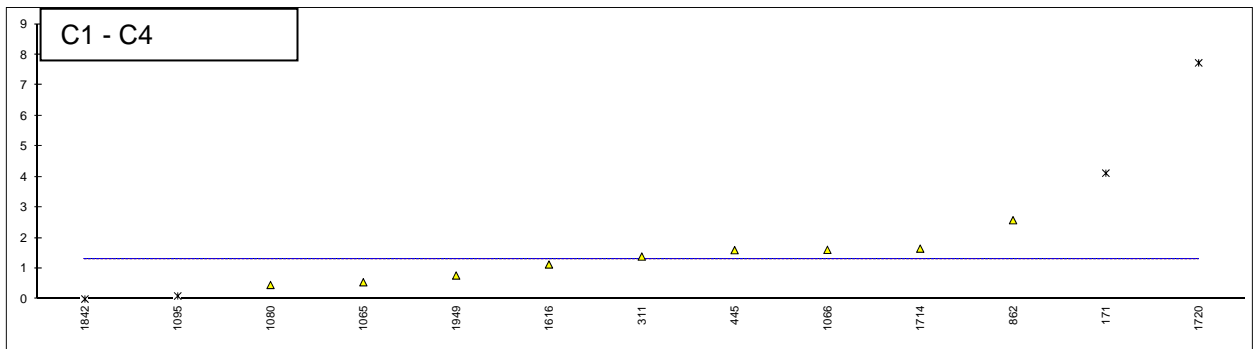
NB: Bold and underlined figures are statistical outliers (Grubbs and / or Dixon)



Determination of P(iP)NA on distillation fraction 2 (light naphtha); results in %M/M

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D5134	83.60	4.12	45.12	38.48	14.53	1.87	
311	D5443	82.82	1.39	44.36	38.46	15.71	1.45	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D5443Mod	84.26	1.60	45.35	38.91	14.25	1.50	
574		-----	-----	-----	-----	-----	-----	
608		-----	-----	-----	-----	-----	-----	
862	D6839	84.11	2.58	45.51	38.60	14.12	1.73	
971		-----	-----	-----	-----	-----	-----	
1023		-----	-----	-----	-----	-----	-----	
1065		83.61	0.55	45.62	37.99	14.49	1.90	
1066		83.95	1.61	45.74	38.21	14.42	1.63	
1080	in house	84.7	0.46	45.1	39.42	13.61	1.69	
1089		-----	-----	-----	-----	-----	-----	
1095		77.3	0.1 ex	40.0	37.3 ex	19.9	2.82	
1108		-----	-----	-----	-----	-----	-----	
1264	D5443	76.44	-----	42.66	33.78	20.94	2.60	
1340	ISO22854	84.51	-----	45.97	38.54	13.46	2.04	
1613	D6839	83.76	-----	-----	-----	14.32	1.92	
1616	D6839	84.21	1.13	44.65	39.56	13.46	2.33	
1714	in house	84.08	1.65	45.44	38.64	14.08	1.84	
1720		86.91	7.73	-----	-----	11.49	1.60	
1842		84.56	0.0	45.65	38.91	13.74	1.69	
1949	D5443	82.18	0.77	-----	-----	15.93	1.89	
	normality	OK	OK	OK	OK	OK	OK	
	n	14	9	11	11	14	14	
	outliers	2	3 + 1 ex	2	1 + 1 ex	2	2	
	mean (n)	84.090	1.304	45.319	38.702	14.115	1.791	
	st.dev. (n)	1.0607	0.6645	0.4804	0.4728	1.0543	0.2299	
	R(calc.)	2.970	1.861	1.345	1.324	2.952	0.644	

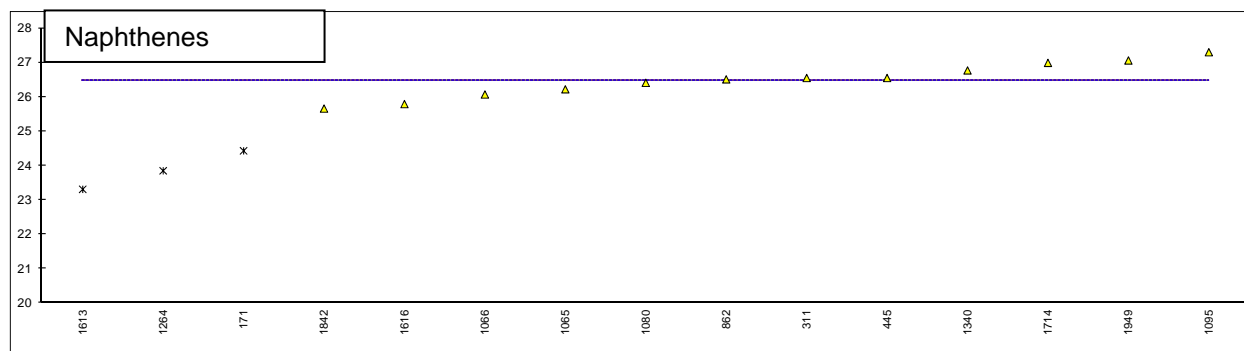
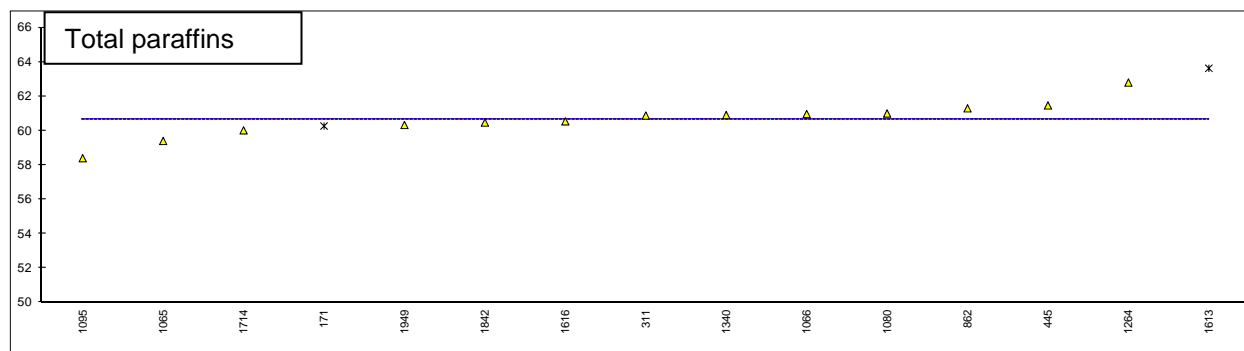
NB: Bold and underlined figures are statistical outliers (Grubbs and / or Dixon)



Determination of P(iP)NA on distillation fraction 3 (heavy naphtha); results in %V/V

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D5134	60.27 ex	0.50 ex	30.98 ex	29.29 ex	24.43 ex	15.29 ex	Method not applicable, see §4.1
311	D5443	60.88	0.16	27.43	33.45	26.55	12.50	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D5443Mod	61.47	0.12	28.93	32.32	26.55	11.98	
574		-----	-----	-----	-----	-----	-----	
608		-----	-----	-----	-----	-----	-----	
862	D6839	61.30	0	26.87	34.43	26.51	12.11	
971		-----	-----	-----	-----	-----	-----	
1023		-----	-----	-----	-----	-----	-----	
1065		59.40	0.10	28.12	31.28	26.22	14.37	
1066		60.97	0.07	28.33	32.64	26.07	12.57	
1080	in house	61.0	0.05	27.12	33.93	26.41	12.59	
1089		-----	-----	-----	-----	-----	-----	
1095		58.4	0	26.0	32.4	27.3	13.4	
1108		-----	-----	-----	-----	-----	-----	
1264	D5443	62.80	-----	26.49	36.31	23.85	12.12	
1340	ISO22854	60.91	-----	27.44	33.47	26.77	12.11	
1613	D6839	63.63	-----	-----	-----	23.31	13.05	
1616	D6839	60.54	0.26	27.70	32.84	25.79	12.41	
1714	in house	60.02	0.04	27.40	32.62	26.99	12.10	
1720		-----	-----	-----	-----	-----	-----	
1842		60.47	0.00	27.18	33.29	25.66	12.83	
1949	D5443	60.33	0.10	-----	-----	27.06	12.61	
	normality	OK	OK	OK	OK	OK	OK	
	n	13	11	12	11	12	13	
	outliers	1 + 1 ex	0 + 1 ex	0 + 1 ex	1 + 1 ex	2 + 1 ex	1 + 1 ex	
	mean (n)	60.653	0.0818	27.417	32.970	26.490	12.491	
	st.dev. (n)	1.0501	0.07936	0.7982	0.8653	0.5001	0.4216	
	R(calc.)	2.940	0.2222	2.235	2.423	1.400	1.180	

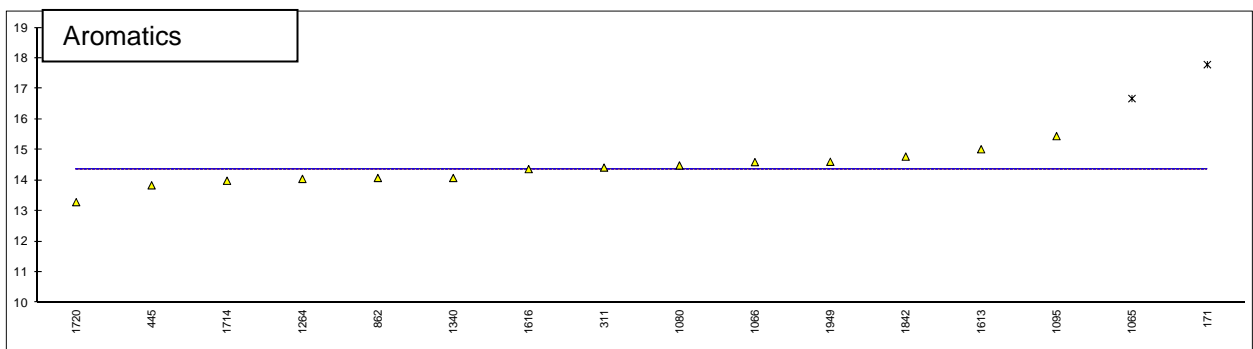
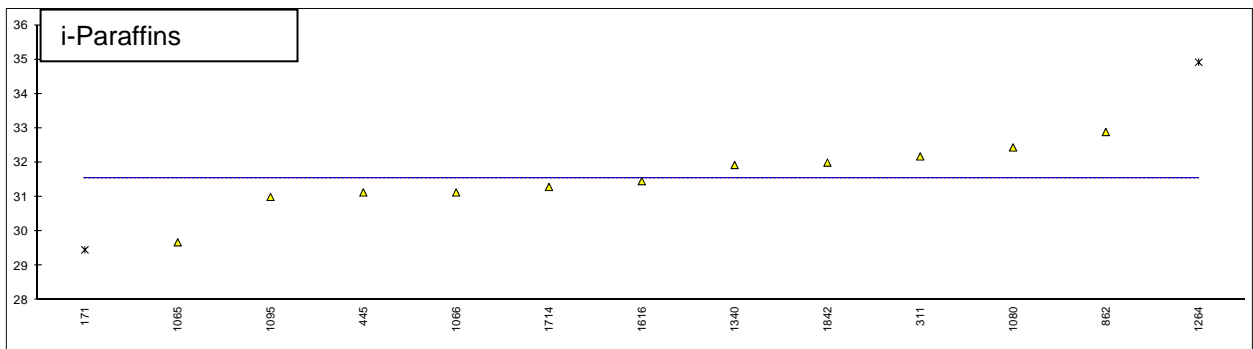
NB: Bold and underlined figures are statistical outliers (Grubbs and / or Dixon)



Determination of P(iP)NA on distillation fraction 3 (heavy naphtha); results in %M/M

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	Remarks
171	D5134	56.89 ex	0.38 ex	27.83 ex	29.46 ex	25.32 ex	17.79 ex	Method not applicable, see §4.1
311	D5443	57.83	0.12	25.65	32.18	27.68	14.42	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D5443Mod	58.48	0.08	27.11	31.13	27.67	13.84	
574		-----	-----	-----	-----	-----	-----	
608		-----	-----	-----	-----	-----	-----	
862	D6839	58.15	0	25.26	32.89	27.69	14.08	
971		-----	-----	-----	-----	-----	-----	
1023		-----	-----	-----	-----	-----	-----	
1065		56.05	0.08	26.37	29.68	27.27	16.68	
1066		57.78	0.06	26.65	31.13	27.17	14.60	
1080	in house	58.04	0.04	25.49	32.44	27.46	14.49	
1089		-----	-----	-----	-----	-----	-----	
1095		55.3	0	24.3	31	28.4	15.45	
1108		-----	-----	-----	-----	-----	-----	
1264	D5443	59.73	-----	24.81	34.92	24.85	14.05	
1340	ISO22854	57.70	-----	25.77	31.93	28.00	14.08	
1613	D6839	60.92	-----	-----	-----	24.06	15.02	
1616	D6839	57.33	0.20	25.87	31.46	26.87	14.37	
1714	in house	56.88	0.03	25.59	31.29	28.11	13.99	
1720	D5543	61.46	0.06	-----	-----	25.25	13.29	
1842		57.42	0.0	25.42	32.00	26.61	14.78	
1949	D5443	57.13	0.08	-----	-----	28.26	14.61	
	normality	OK	OK	OK	OK	OK	OK	
	n	15	12	12	11	15	14	
	outliers	0 + 1 ex	0 + 1 ex	1 + 1 ex	1 + 1 ex	0 + 1 ex	1 + 1 ex	
	mean (n)	58.013	0.0625	25.691	31.557	27.023	14.362	
	st.dev. (n)	1.6431	0.05754	0.7660	0.8694	1.3085	0.5368	
	R(calc.)	4.601	0.1611	2.145	2.434	3.664	1.503	

NB: Bold and underlined figures are statistical outliers (Grubbs and / or Dixon)



Results of D86 distillation on combined distillation fractions 4 + 5, results in °C

Lab method	IBP	5% evap.	10% evap.	50% evap.	90% evap.	95% evap.	FBP	remarks
171 D86	184	189.7	193.5	209.4	231.3	236.9	241.1	
311 D86	193.9	201.3	202.9	214.9	234.2	238.9	244.3	
391	188	-----	-----	-----	-----	-----	243	
442	-----	-----	-----	-----	-----	-----	-----	
445 IP123	186.9	-----	-----	-----	-----	-----	237.2	
574 D86	191.67	200.61	202.17	215.73	236.89	242.65	250.43	
608	-----	-----	-----	-----	-----	-----	-----	
862 D86	187.1	199.2	201.8	219.1	239.5	243.3	258.9	
971 D86	188	198	202	212	230	236	244	
1023	187	196	199	214	235	241	250	
1065 D86	190.1	-----	-----	-----	-----	-----	238.3	
1066	193.7	199.4	200.7	210.1	226.8	231.0	236.9	
1080 D86	186.9	-----	-----	-----	-----	-----	242.6	
1089	-----	-----	-----	-----	-----	-----	-----	
1095 D86	198.6	208.5	210.1	220.7	238.4	242.1	260.9	
1108	-----	-----	-----	-----	-----	-----	-----	
1264	189.8	197.8	199.0	209.7	229.1	234.0	240.8	
1340 D86	187.9	198.1	199.8	212.2	231.1	235.8	245.2	
1613 D86	196.3	203.8	205.5	218.2	238.6	243.2	250.7	
1616 in house	189.4	-----	-----	-----	-----	-----	249.0	
1714 D86	191.5	197.0	199.2	212.1	231.6	236.2	244.3	
1720 D86	181.2	194.1	197.0	210.8	230.4	235.4	249.3	
1842 D86	189.6	199.1	201.8	213.1	231.0	235.1	240.1	
1949 D86	195.00	199.50	204.00	213.00	230.50	233.89	237.00	
normality	OK	OK	OK	OK	not OK	OK	OK	
n	20	15	15	15	15	15	20	
outliers	0	0	0	0	0	0	0	
mean (n)	189.83	198.81	201.23	213.67	232.96	237.70	245.20	
st.dev. (n)	4.199	4.207	3.813	3.469	3.886	3.894	6.769	
R(calc.)	11.76	11.78	10.68	9.71	10.88	10.90	18.95	

Results of D86 distillation on combined distillation fractions 4 + 5, results in °C

Lab method	%rec	5% rec.	10% rec.	50% rec.	90% rec.	95% rec.	%res.	remarks
171 D86	98	190.7	195.4	207.8	230.0	235.0	2	
311 D86	97.6	201.9	202.9	215.2	235.1	240.1	1.4	
391	99	199	204	213	231	236	0.5	
442	-----	-----	-----	-----	-----	-----	-----	
445 IP123	98.7	195.6	197.8	210.8	228.6	233.1	1.0	
574 D86	99.0	200.71	202.00	215.69	236.66	242.28	0.30	
608	-----	-----	-----	-----	-----	-----	-----	
862 D86	98.4	199.5	201.9	219.3	239.8	243.7	1.3	
971 D86	99.0	198	202	212	230	236	1.0	
1023	98.5	-----	-----	-----	-----	-----	1.0	
1065 D86	98.2	198.4	199.8	211.3	229.6	234.2	1.8	
1066	98.2	200.2	201.1	210.5	228.3	233.3	1.1	
1080 D86	98.7	198.8	199.1	212.2	231.5	235.9	0.8	
1089	-----	-----	-----	-----	-----	-----	-----	
1095 D86	99.9	208.5	210.1	220.7	238.5	242.2	0.1	
1108	-----	-----	-----	-----	-----	-----	-----	
1264	99.0	189.8	199.0	209.7	229.1	234.0	1.0	
1340 D86	98.3	198.3	200.1	212.4	231.6	236.5	1.1	
1613 D86	99.2	203.8	205.5	218.2	238.6	243.2	0.8	
1616 in house	-----	196.8	198.6	213.8	233.0	238.6	-----	
1714 D86	98.6	197.1	199.4	212.4	232.0	236.8	0.9	
1720 D86	98.7	194.3	197.1	210.8	230.6	235.8	1.1	
1842 D86	98.7	199.4	201.8	213.2	231.3	235.4	1.0	
1949 D86	99.00	203.00	204.00	213.00	230.00	234.00	0.90	
normality		OK	OK	OK	not OK	not OK		
n		19	19	19	19	19		
outliers		0	0	0	0	0		
mean (n)		198.62	201.14	213.26	232.38	237.16		
st.dev. (n)		4.329	3.356	3.305	3.586	3.465		
R(calc.)		12.12	9.40	9.25	10.04	9.70		

Results of Simdist on distillation fraction 4 (Kerosene, 180-215°C); yields of fractions in %M/M

Lab	175-185°C	185-195°C	195-205°C	205-215°C	215-225°C	225-235°C	235-245°C	245-255°C	255-265°C
171	12.7	17.8	25.0	15.5	9.0	2.5	0	0	0
311	9.5	16.5	28.5	20.0	14.0	3.0	0	0	0
391	----	----	----	----	----	----	----	----	----
442	----	----	----	----	----	----	----	----	----
445	11.7	13.8	22.1	20.9	17.0	1.4	0.9	0	0
574	----	----	----	----	----	----	----	----	----
608	----	----	----	----	----	----	----	----	----
862	17.3	11.4	21.0	12.3	20.0	<u>7.7</u>	<u>1.3</u>	0	0
971	----	----	----	----	----	----	----	----	----
1023	----	----	----	----	----	----	----	----	----
1065	14.5	16.5	30.0	17.8	10.1	1.0	0	0	0
1066	13.0	22.2	30.3	17.9	8.4	0.7	0.5	0	0
1080	----	----	----	----	----	----	----	----	----
1089	----	----	----	----	----	----	----	----	----
1095	<u>5.0</u>	11.0	24.5	20.5	23.0	<u>9.8</u>	<u>3.1</u>	0	0
1108	----	----	----	----	----	----	----	----	----
1264	----	----	----	----	----	----	----	----	----
1340	----	----	----	----	----	----	----	----	----
1613	----	----	----	----	----	----	----	----	----
1616	----	----	----	----	----	----	----	----	----
1714	13.4	17.2	25.3	17.5	11.1	2.9	0	0	0
1720	----	----	----	----	----	----	----	----	----
1842	----	----	----	----	----	----	----	----	----
1949	----	----	----	----	----	----	----	----	----
normality	OK	OK	OK	OK	OK	OK	OK	n.a.	n.a.
n	7	8	8	8	8	6	6	n.a.	n.a.
outliers	1	0	0	0	0	2	2	n.a.	n.a.
mean (n)	13.16	15.80	25.84	17.80	14.07	1.92	0.23	n.a.	n.a.
st.dev. (n)	2.407	3.670	3.474	2.863	5.424	1.007	0.383	n.a.	n.a.
R(calc.)	6.74	10.28	9.73	8.02	15.19	2.82	1.07	n.a.	n.a.

Results of Simdist on distillation fraction 5 (light gasoil, 215-250°C); yields of fractions in %M/M

Lab	175-185°C	185-195°C	195-205°C	205-215°C	215-225°C	225-235°C	235-245°C	245-255°C	255-265°C
171	<u>1.8</u>	2.4	5.0	11.3	21.5	27.5	22.0	7.6	<u>0 ex</u>
311	0	0	2.2	5.5	14.8	20.5	26.0	17.0	12.4
391	----	----	----	----	----	----	----	----	----
442	----	----	----	----	----	----	----	----	----
445	0	0.9	2.8	7.3	20.2	24.5	25.5	16.1	2.7
574	----	----	----	----	----	----	----	----	----
608	----	----	----	----	----	----	----	----	----
862	0	0	0.4	2.3	<u>9.0</u>	<u>14.1</u>	25.0	22.2	22.8
971	----	----	----	----	----	----	----	----	----
1023	----	----	----	----	----	----	----	----	----
1065	<u>0.5</u>	1.0	2.5	7.0	21.0	22.0	24.0	14.3	5.8
1066	0	0.8	1.9	10.6	19.9	28.5	25.2	12.1	1.0
1080	----	----	----	----	----	----	----	----	----
1089	----	----	----	----	----	----	----	----	----
1095	0	0	0.2	2.1	<u>5.7</u>	<u>15.0</u>	25.0	23.0	22.5
1108	----	----	----	----	----	----	----	----	----
1264	----	----	----	----	----	----	----	----	----
1340	----	----	----	----	----	----	----	----	----
1613	----	----	----	----	----	----	----	----	----
1616	----	----	----	----	----	----	----	----	----
1714	0	1.9	4.3	8.9	16.6	20.3	22.8	16.0	8.3
1720	----	----	----	----	----	----	----	----	----
1842	----	----	----	----	----	----	----	----	----
1949	----	----	----	----	----	----	----	----	----
normality	n.a.	OK	OK	OK	OK	OK	OK	OK	OK
n	n.a.	8	8	8	6	6	8	8	7
outliers	n.a.	0	0	0	2	2	0	0	0 + 1
mean (n)	n.a.	0.88	2.41	6.87	19.00	23.88	24.44	16.04	10.79
st.dev. (n)	n.a.	0.902	1.676	3.451	2.680	3.539	1.394	5.030	8.910
R(calc.)	n.a.	2.53	4.69	9.66	7.50	9.91	3.90	14.08	24.95

NB. Bold and underlined figures are statistical outliers (Grubbs and/or Dixon)

Determination of Effective Cut Point (ECP) from the simdist data

Lab	Overlap cuts 4 and 5 in °C	ECP in °C	Difference with AET=215 °C	Conclusion*	Remarks
171	175 – 233	210.4	-4.63	OK	
311	195 – 234	216.3	1.28	OK	
391	----	----	----	----	
442	----	----	----	----	
445	191 – 235	213.5	-1.51	OK	
574	----	----	----	----	
608	----	----	----	----	
862	203 – 239	222.8	7.82	Not OK	
971	----	----	----	----	
1023	----	----	----	----	
1065	177 – 266	213.0	-2.04	OK	
1066	193 – 236	212.2	-2.82	OK	
1080	----	----	----	----	
1089	----	----	----	----	
1095	197 – 242	225.1	10.13	Not OK	
1108	----	----	----	----	
1264	----	----	----	----	
1340	----	----	----	----	
1613	----	----	----	----	
1616	----	----	----	----	
1714	183 – 235	213.0	-2.05	OK	
1720	----	----	----	----	
1842	----	----	----	----	
1949	----	----	----	----	

* Acc. To ASTM D2892:13, Appendix X2.6.5.1 the difference between ECP and AET should not exceed 0.7R °C (0.7 x 8 = 5.6°C)

Determination of Standard Efficiency N_{minimum} from the simdist data

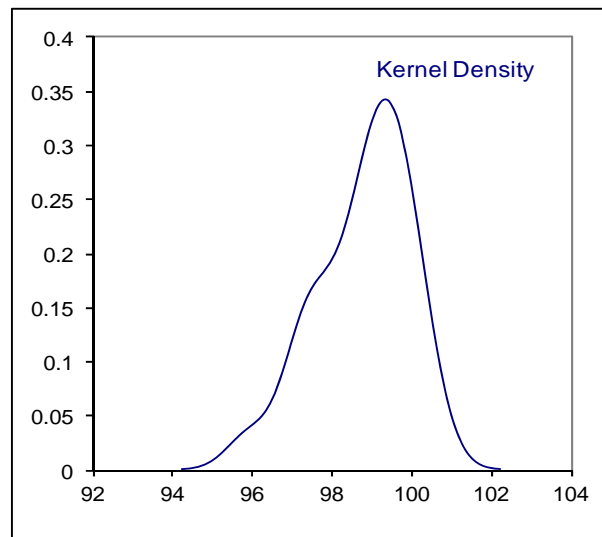
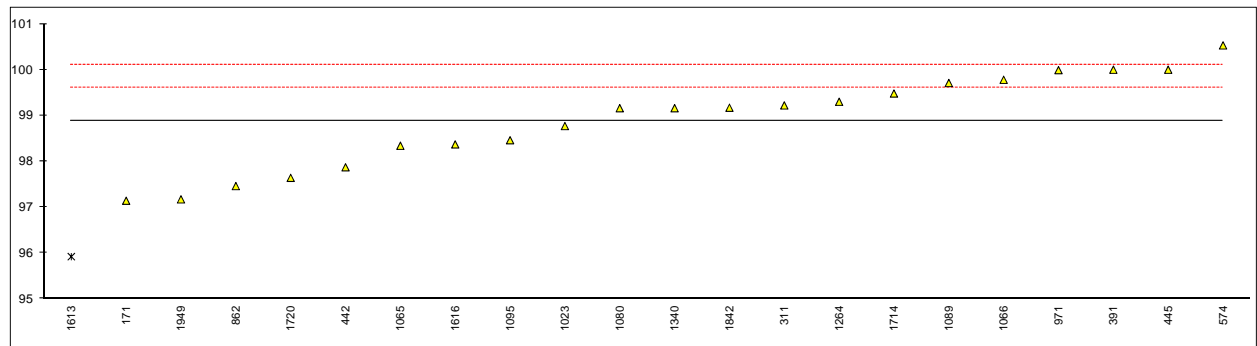
Lab	N_{actual}	N_{minimum}	Requirement $5.4 < N_{\text{minimum}} < 6.8^*$	Remarks
171	6.1	5.2	Not OK	
311	6.9	5.9	OK	
391	----	----	----	
442	----	----	----	
445	7.8	6.7	OK	
574	----	----	----	
608	----	----	----	
862	7.0	6.0	OK	
971	----	----	----	
1023	----	----	----	
1065	8.6	7.3	Not OK	
1066	9.7	8.3	Not OK	
1080	----	----	----	
1089	----	----	----	
1095	7.9	7.5	Not OK	
1108	----	----	----	
1264	----	----	----	
1340	----	----	----	
1613	----	----	----	
1616	----	----	----	
1714	5.7	4.9	Not OK	
1720	----	----	----	
1842	----	----	----	
1949	----	----	----	

* Acc. To ASTM D2892:13, Appendix X2.5.9.2 the acceptable upper and lower limits (14-18 theoretical plates) are 5.4, resp. 6.8

Total Mass balance/Total Mass recovery, results in %M/M

lab	method	value	Mark	remarks
171	calc	97.14		
311	calc	99.22		
391	calc	100.00		
442	calc	97.87		
445	calc	100.00		
574	calc	100.53		
608	calc	-----		
862	calc	97.46		
971	calc	99.99		
1023	calc	98.77		
1065	calc	98.34		
1066	calc	99.78		
1080	calc	99.16		
1089	calc	99.71		
1095	calc	98.46		
1108	calc	-----		
1264	calc	99.30		
1340	calc	99.16		
1613	calc	95.92	G(0.05)	
1616	calc	98.37		
1714	calc	99.48		
1720	calc	97.64		
1842	calc	99.17		
1949	calc	97.17		
normality		OK		
n		21		
outliers		1		
mean (n)		98.91		
st.dev. (n)		0.996		
R(calc.)		2.79		

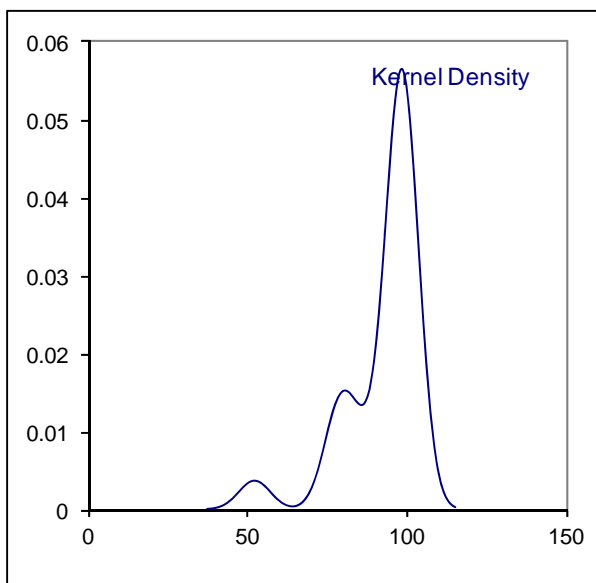
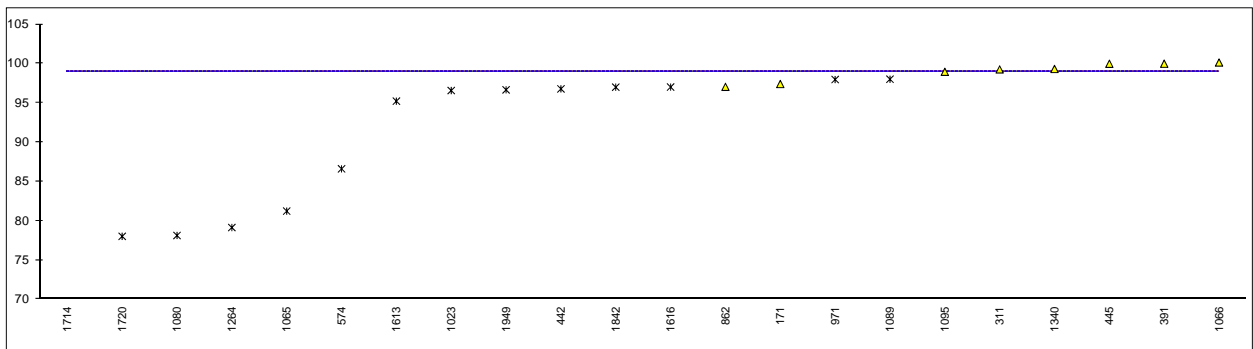
In below graph, the dotted lines represent the ASTM D5236 requirements for recovery: 99.6% < recovery < 100.1%, of which the lower line at 99.6% is equal to the 0.4% max. loss requirement of ASTM D2892, paragraph 11.2.



Total Volume balance/Total Volume recovery, results in %V/V

lab	method	value	mark	remarks
171	calc	97.41		
311	calc	99.27		
391	calc	100.00		
442	calc	96.79	ex	Density of the gas fraction was not provided
445	calc	99.99		
574	calc	86.61	ex	Densities of the gas fraction and final residue were not provided
608	calc	-----		
862	calc	97.05		
971	calc	97.98	ex	Density of the gas fraction was not provided
1023	calc	96.58	ex	Density of the gas fraction was not provided
1065	calc	81.32	ex	Densities of the gas fraction and final residue were not provided
1066	calc	100.15		
1080	calc	78.13	ex	Density of the gas fraction was not provided
1089	calc	98.02	ex	Density of the gas fraction was not provided
1095	calc	98.97		
1108	calc	-----		
1264	calc	79.14	ex	Density of the final residue was not provided
1340	calc	99.33		
1613	calc	95.23	ex	Density of the gas fraction was not provided
1616	calc	97.02	ex	Density of the gas fraction was not provided
1714	calc	52.15	ex	Densities of the gas fraction and final residue were not provided
1720	calc	78.02	ex	Density of the final residue was not provided
1842	calc	97.01	ex	Density of the gas fraction was not provided
1949	calc	96.67	ex	Density of the gas fraction was not provided

normality OK
n 8 + 14 excl
outliers 0
mean (n) 99.02
st.dev. (n) 1.184
R(calc.) 3.32



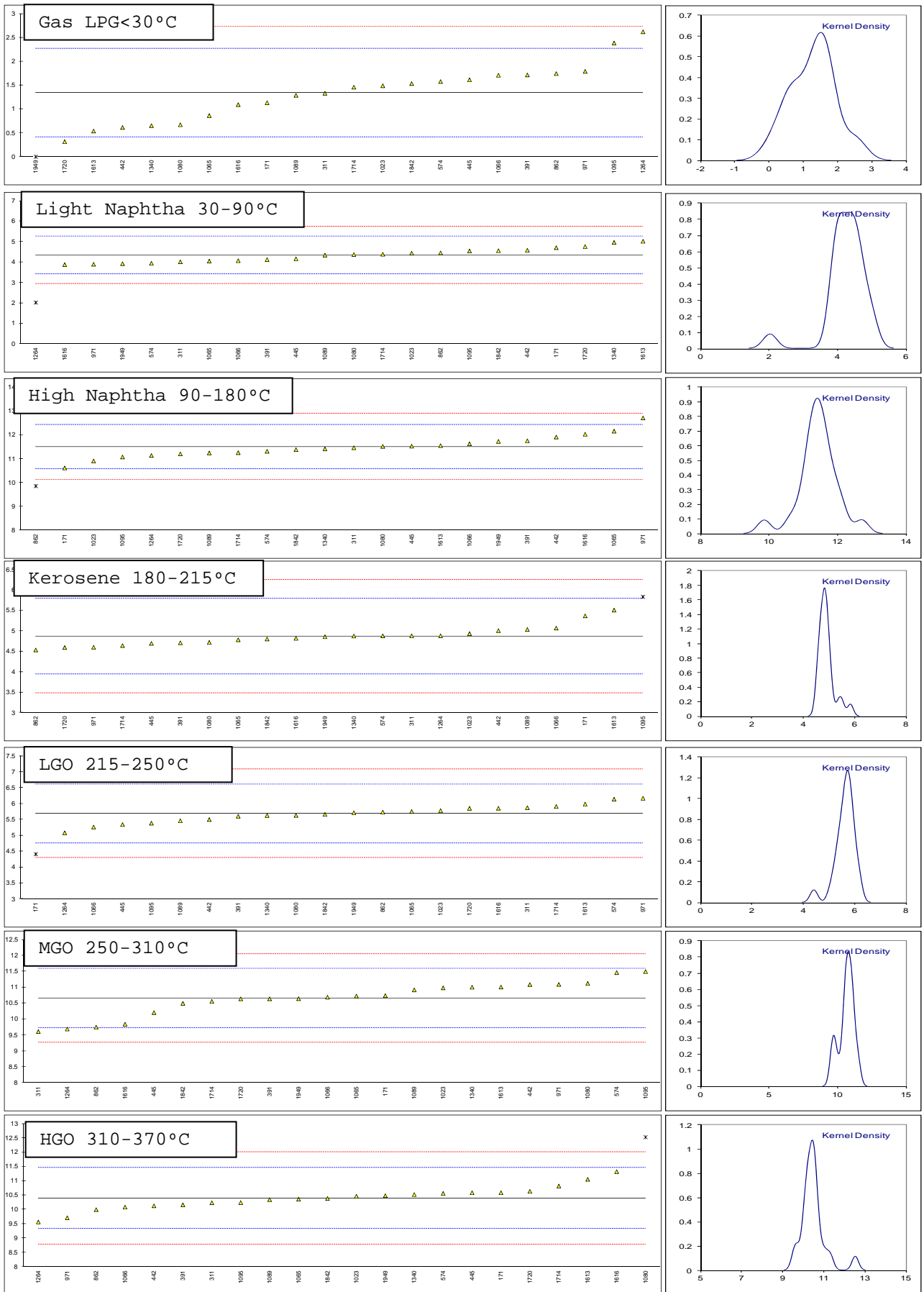
Determination of true boiling point curve of D2892; individual fractions in %M/M

lab	Gas LPG <30°C	L. Naphtha 30-90°C	H. Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C
171	1.1343	4.7132	10.6127	5.3701	4.4116	10.7387	10.5865	50.9871
311	1.3316	4.0209	11.4621	4.8825	5.8747	9.6084	10.2350	52.5849
391	1.7158	4.1323	11.7529	4.7120	5.6059	10.6399	10.1659	51.2752
442	0.6150	4.5864	11.9106	5.0095	5.5008	11.0894	10.1284	49.0282
445	1.6161	4.1693	11.5253	4.6979	5.3437	10.2061	10.5863	51.8553
574	1.5776	3.9502	11.3120	4.8808	6.1419	11.4632	10.5622	48.7954
608	----	----	----	----	----	----	----	----
862	1.7439	4.4584	9.8544	4.5367	5.7347	9.7483	9.9960	53.9276
971	1.7912	3.9008	12.7106	4.6040	6.1696	11.0919	9.7121	49.8607
1023	1.4897	4.4418	10.9094	4.9372	5.7822	10.9841	10.4649	49.7591
1065	0.8642	4.0589	12.1573	4.7856	5.7550	10.7267	10.3625	50.0728
1066	1.7098	4.0742	11.6285	5.0718	5.2650	10.6914	10.0830	51.3465
1080	0.6717	4.3762	11.5183	4.7219	5.6307	11.1232	12.5259	48.7010
1089	1.2889	4.3458	11.2429	5.0378	5.4637	10.9197	10.3456	51.0665
1095	2.3865	4.5548	11.0732	5.8366	5.3866	11.4960	10.2414	48.0158
1108	----	----	----	----	----	----	----	----
1264	2.6215	2.0261	11.1359	4.8830	5.0842	9.6909	9.5641	54.5063
1340	0.6528	4.9680	11.4142	4.8763	5.6288	11.0046	10.5213	50.0972
1613	0.5398	5.0256	11.5511	5.5114	5.9886	11.0085	11.0540	47.7414
1616	1.0935	3.8849	12.0288	4.8201	5.8561	9.8417	11.3237	49.9137
1714	1.4602	4.3906	11.2542	4.6440	5.9125	10.5587	10.8206	50.6577
1720	0.3164	4.7672	11.2038	4.5962	5.8537	10.6394	10.6344	49.8821
1842	1.5351	4.5647	11.3822	4.8050	5.6630	10.4961	10.3869	50.5335
1949	0.00 ex	3.9300	11.7279	4.8594	5.7138	10.6431	10.4826	49.8939
normality	OK	OK	OK	OK	OK	OK	OK	OK
n	21	21	21	21	21	22	21	22
outliers	0 + 1 ex	1	1	1	1	0	1	0
mean (n)	1.3407	4.3483	11.5007	4.8687	5.6836	10.6550	10.3932	50.4774
st.dev. (n)	0.59390	0.34743	0.45853	0.24012	0.27726	0.54087	0.40339	1.69534
R(calc.)	1.6629	0.9728	1.2839	0.6723	0.7763	1.5144	1.1295	4.7469
R(D2892:13)	1.3	1.3	1.3	1.3	1.5	1.5	1.5	n.a.

NB. Bold and underlined figures are statistical outliers (Grubbs and/or Dixon)

Corresponding z-scores for above mass fractions:

lab	Gas LPG <30°C	L. Naphtha 30-90°C	H. Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C
171	-0.44	0.79	-1.91	1.08	-2.37	0.16	0.36	----
311	-0.02	-0.71	-0.08	0.03	0.36	-1.95	-0.30	----
391	0.81	-0.47	0.54	-0.34	-0.15	-0.03	-0.42	----
442	-1.56	0.51	0.88	0.30	-0.34	0.81	-0.49	----
445	0.59	-0.39	0.05	-0.37	-0.63	-0.84	0.36	----
574	0.51	-0.86	-0.41	0.03	0.86	1.51	0.32	----
608	----	----	----	----	----	----	----	----
862	0.87	0.24	-3.55	-0.72	0.10	-1.69	-0.74	----
971	0.97	-0.96	2.61	-0.57	0.91	0.82	-1.27	----
1023	0.32	0.20	-1.27	0.15	0.18	0.61	0.13	----
1065	-1.03	-0.62	1.41	-0.18	0.13	0.13	-0.06	----
1066	0.79	-0.59	0.28	0.44	-0.78	0.07	-0.58	----
1080	-1.44	0.06	0.04	-0.32	-0.10	0.87	3.98	----
1089	-0.11	-0.01	-0.56	0.36	-0.41	0.49	-0.09	----
1095	2.25	0.44	-0.92	2.08	-0.55	1.57	-0.28	----
1108	----	----	----	----	----	----	----	----
1264	2.76	-5.00	-0.79	0.03	-1.12	-1.80	-1.55	----
1340	-1.48	1.33	-0.19	0.02	-0.10	0.65	0.24	----
1613	-1.73	1.46	0.11	1.38	0.57	0.66	1.23	----
1616	-0.53	-1.00	1.14	-0.10	0.32	-1.52	1.74	----
1714	0.26	0.09	-0.53	-0.48	0.43	-0.18	0.80	----
1720	-2.21	0.90	-0.64	-0.59	0.32	-0.03	0.45	----
1842	0.42	0.47	-0.26	-0.14	-0.04	-0.30	-0.01	----
1949	-2.89	-0.90	0.49	-0.02	0.06	-0.02	0.17	----



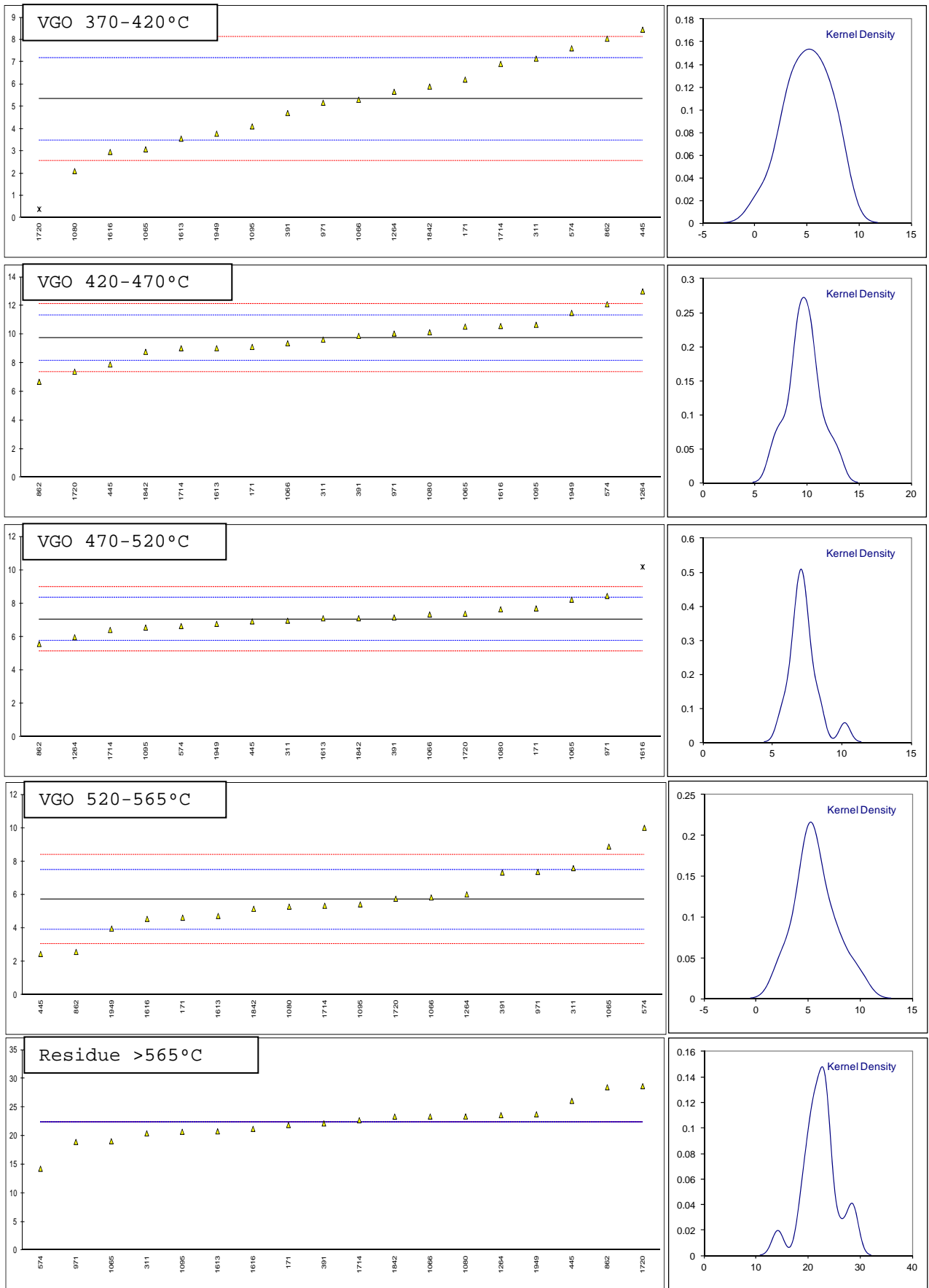
Determination of true boiling point curve D5236 (continued); individual fractions in %M/M

lab	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	VGO 520-565°C	Residue >565°C	remarks
171	6.2121	9.1233	7.7055	4.6314	21.8970	
311	7.1540	9.6345	6.9713	7.5979	20.4439	
391	4.7109	9.8961	7.1657	7.3324	22.1701	
442	----	----	----	----	----	
445	8.4558	7.8998	6.9296	2.4586	26.1116	
574	7.6174	12.1033	6.6489	10.0140	14.2634	
608	----	----	----	----	----	
862	8.0498	6.6901	5.5755	2.5805	28.4967	
971	5.1745	10.0571	8.4516	7.3769	18.9465	
1023	----	----	----	----	----	
1065	3.0801	10.5421	8.2269	8.8865	19.0634	
1066	5.3098	9.3767	7.3410	5.8413	23.3831	
1080	2.1041	10.1482	7.6499	5.2949	23.3923	
1089	----	----	----	----	----	
1095	4.1109	10.6702	6.5592	5.4264	20.7192	
1108	----	----	----	----	----	
1264	5.6681	13.0023	5.9788	6.0239	23.6225	
1340	----	----	----	----	----	
1613	3.5625	9.0341	7.1136	4.7358	20.7926	
1616	2.9640	10.5842	10.1899	4.5612	21.2230	
1714	6.9163	9.0299	6.4151	5.3454	22.7357	
1720	0.3929	7.3942	7.3906	5.7675	28.6806	
1842	5.9001	8.7797	7.1184	5.1651	23.3689	
1949	3.7844	11.4997	6.7816	3.9778	23.7702	
normality	OK	OK	OK	OK	OK	
n	17	18	17	18	18	
outliers	1	0	1	0	0	
mean (n)	5.3397	9.7481	7.0602	5.7232	22.3934	
st.dev. (n)	1.90284	1.58068	0.73226	1.95817	3.40376	
R(calc.)	5.3280	4.4259	2.0503	5.4829	9.5305	
R(D5236:13)	2.6	2.2	1.8	2.5	n.a.	

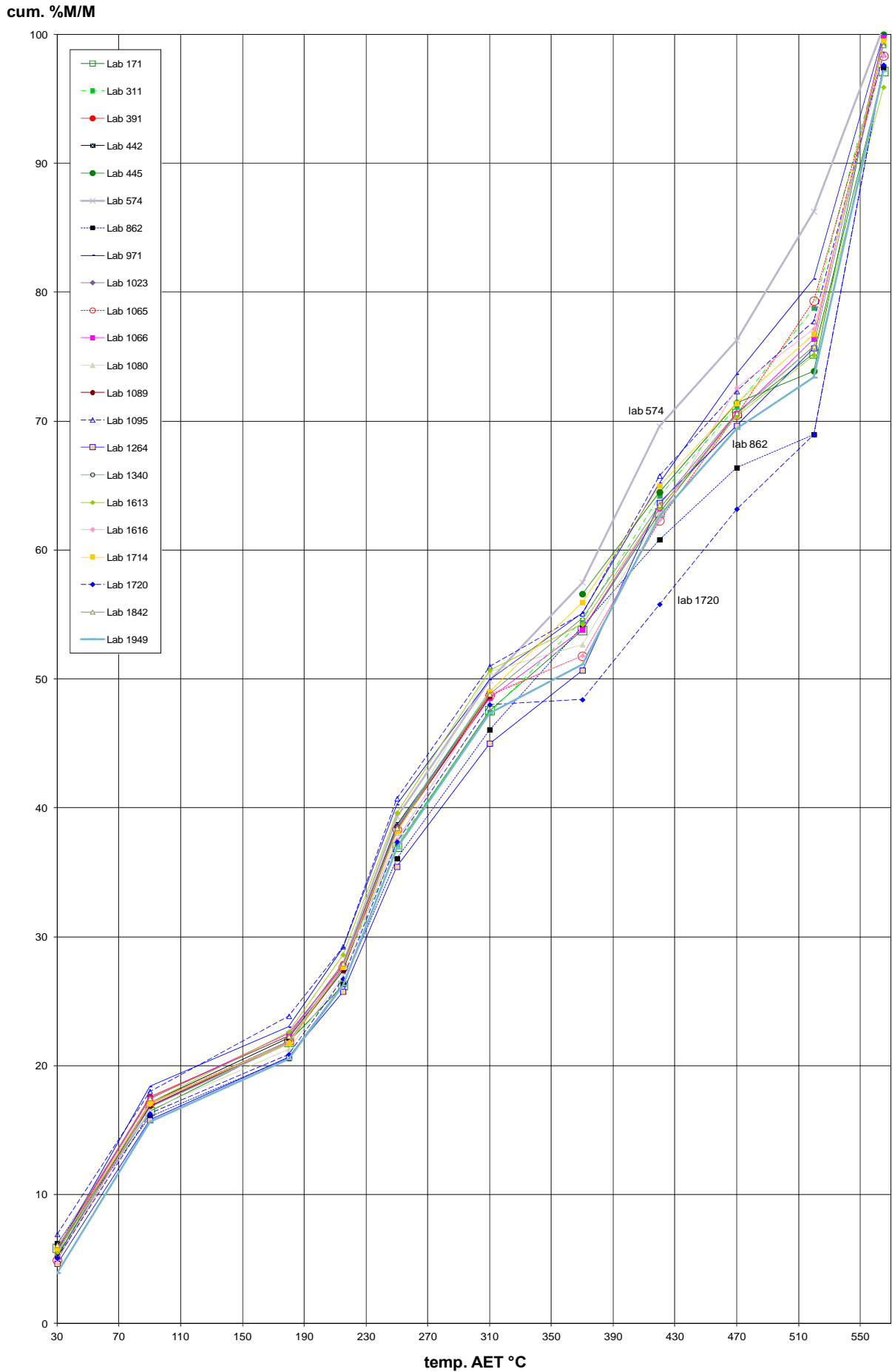
NB. Bold and underlined figures are statistical outliers (Grubbs and/or Dixon)

Corresponding z-scores for above mass fractions:

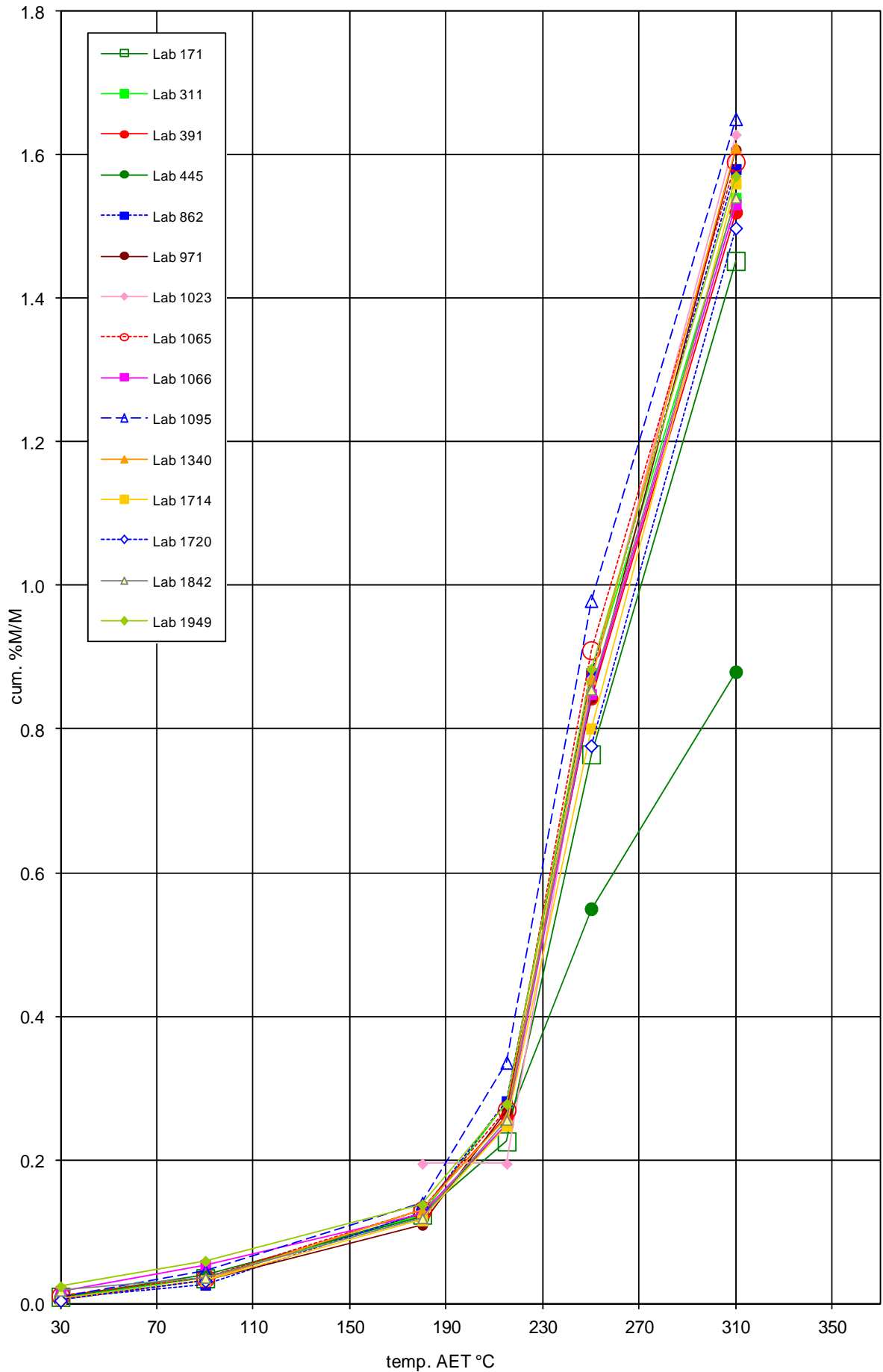
lab	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	VGO 520-565°C	Residue >565°C
171	0.94	-0.80	1.00	-1.22	----
311	1.95	-0.14	-0.14	2.10	----
391	-0.68	0.19	0.16	1.80	----
442	----	----	----	----	----
445	3.36	-2.35	-0.20	-3.66	----
574	2.45	3.00	-0.64	4.81	----
608	----	----	----	----	----
862	2.92	-3.89	-2.31	-3.52	----
971	-0.18	0.39	2.16	1.85	----
1023	----	----	----	----	----
1065	-2.43	1.01	1.81	3.54	----
1066	-0.03	-0.47	0.44	0.13	----
1080	-3.48	0.51	0.92	-0.48	----
1089	----	----	----	----	----
1095	-1.32	1.17	-0.78	-0.33	----
1108	----	----	----	----	----
1264	0.35	4.14	-1.68	0.34	----
1340	----	----	----	----	----
1613	-1.91	-0.91	0.08	-1.11	----
1616	-2.56	1.06	4.87	-1.30	----
1714	1.70	-0.91	-1.00	-0.42	----
1720	-5.33	-3.00	0.51	0.05	----
1842	0.60	-1.23	0.09	-0.63	----
1949	-1.67	2.23	-0.43	-1.95	----



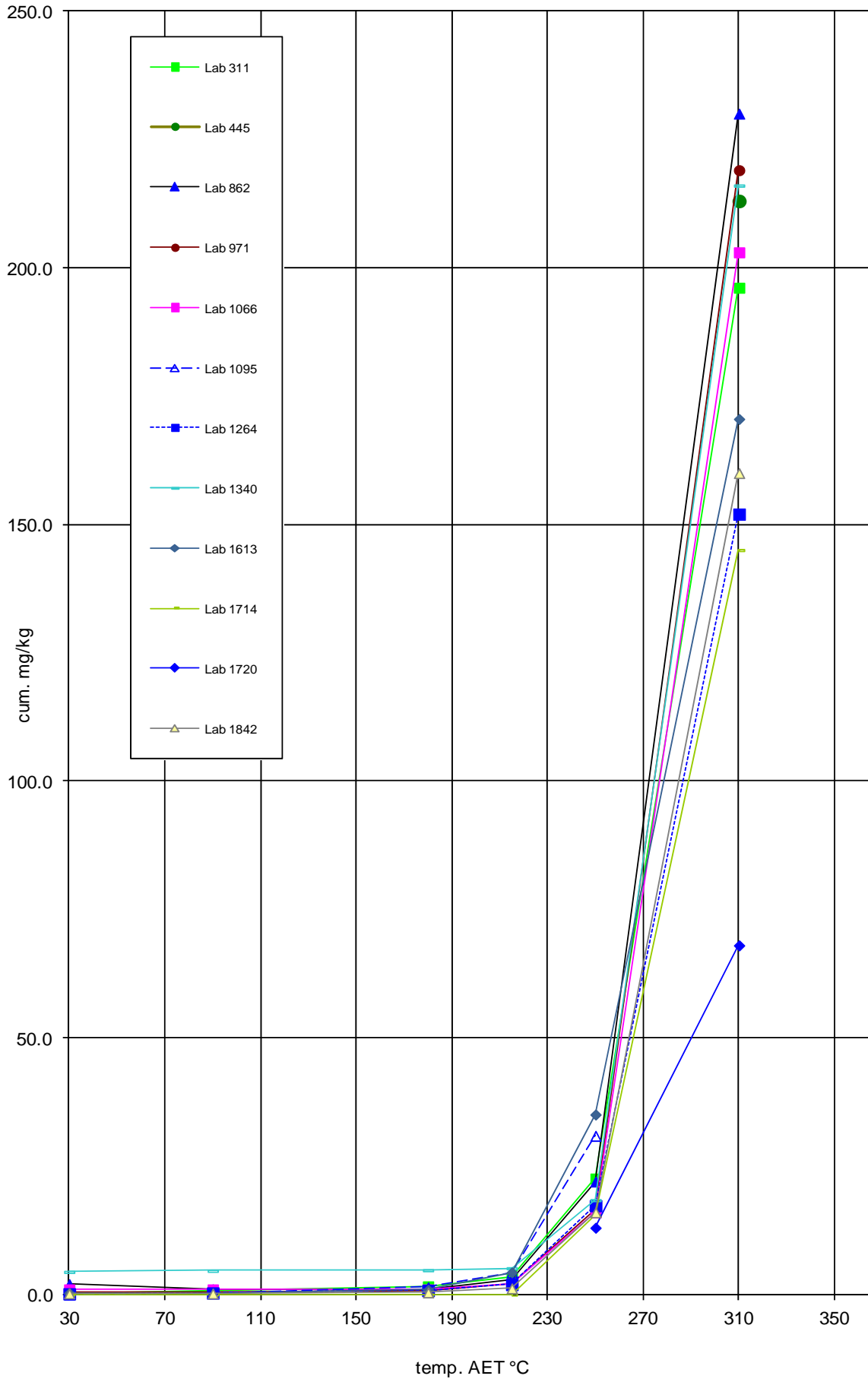
APPENDIX 2 True boiling point curve, cum%M/M vs temp AET in °C



APPENDIX 3 True boiling point curve D2892, Sulphur in %M/M vs temp AET in °C



APPENDIX 4 True boiling point curve D2892, Nitrogen in mg/kg vs temp AET in °C



APPENDIX 5**Details of Distillation**

	Sample received	Distillation started	Results reported	Intake in mL	Remarks
171	25-Oct-13	4-Dec-13	4-Dec-13	3690	
311	12-Oct-13	25-Nov-13	2-Dec-13	4350	
391	23-Oct-13	6-Nov-13	7-Nov-13	4400.4	
442	30-Oct-13	6-Nov-13	13-Nov-13	6335.0	
445	24-Oct-13	25-Oct-13	27-Oct-13	12014	sample starts to crack @ 540°C AET, dist stopped at this point
574	12-Nov-13	13-Nov-13	19-Nov-13	3420.4	
608					
862	4-Nov-13			4495.60	
971	27-Oct-13	5-Nov-13	8-Nov-13	8559	
1023	6-Nov-13	12-Nov-13	15-Nov-13	2596.3	
1065	28-Oct-13	30-Oct-13	6-Nov-13	7000	
1066	28-Oct-13	5-Nov-13	12-Nov-13	3938.9	volume gain reported for ASTM D2892
1080	30-Oct-13	8-Nov-13	11-Nov-13	1151.0	
1089	11-Nov-13	14-Nov-13	15-Nov-13		
1095	30-Oct-13	18-Nov-13	22-Nov-13	8330.1	mass of the residue charged to potstill = 2628g (vol dist flask D5236 = 6L)
1108					
1264	14-Nov-13	22-Dec-13	30-Dec-13		ASTM D2892 dist got interrupted during every cut change, due to malfunctioning of N2 pressure sensor. (see report form)
1340	14-Nov-13	26-Nov-13	26-Nov-13	4000.00	
1613	28-Nov-13	7-Dec-13	8-Dec-13	4000	the blending ratio of combined fractions 4+5 (180-250°C) is 1:1 (dist acc to ASTM D86)
1616	9-Dec-13	11-Dec-13	17-Dec-13	3946	
1714	28-Oct-13	31-Oct-13	5-Nov-13	4135	
1720	14-Nov-13	27-Nov-13	27-Nov-13	8443.61	
1842	24-Oct-13	28-Oct-13	30-Oct-13	3641.6	Charge weight for ASTM D5236 was 501.9g
1949					

APPENDIX 6

Number of participants per country

1 lab in BOSNIA and HERZEGOVINA
1 lab in COLUMBIA
1 lab in GREECE
1 lab in HUNGARY
1 lab in ISRAEL
1 lab in ITALY
1 lab in JORDAN
1 lab in MALAYSIA
2 labs in NORWAY
1 lab in P.R. of CHINA
1 lab in PORTUGAL
1 lab in QATAR
1 lab in RUSSIA
1 lab in SAUDI ARABIA
1 lab in SUDAN
3 labs in THE NETHERLANDS
1 lab in U.A.E.
1 lab in U.S.A.
3 labs in UNITED KINGDOM

APPENDIX 7

Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
E	= error in calculations
ex	= excluded from calculations
n.a.	= not available
n.r.	= not relevant
fr.	= first reported result

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
- 2 ASTM E178-89
- 3 ASTM E1301-89
- 4 ISO 5725-86
- 5 ISO 5725, parts 1-6, 1994
- 6 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 7 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 8 IP 367/84
- 9 DIN 38402 T41/42
- 10 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 11 J.N. Miller, Analyst, 118, 455, (1993)
- 12 Analytical Methods Committee Technical brief, No4 January 2001.
- 13 The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>).