

Results of Proficiency Test
Crude Oil Assay
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Organised by: Institute for Interlaboratory Studies (iis)
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1 INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies organizes a proficiency scheme for Crude Oil Assay. During the annual proficiency test program of 2017/2018, it was decided to continue the proficiency test of the analysis for the Crude Oil Assay.

In this interlaboratory study 23 laboratories in 16 different countries registered for participation. See appendix 6 for the number of participants per country.

In this report, the results of the 2017 proficiency test for Crude Oil Assay are presented and discussed. This report is electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send samples of 5 L, labelled #17218. Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. 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 March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and 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. 300 litre of Crude Oil was homogenised. After homogenisation, the bulk material was divided over 58 metal cans of 5 liter and labelled #17218.

The homogeneity of the subsamples #17218 was checked by determination of Density at 15°C in accordance with ASTM D5002:16 on 8 stratified randomly selected samples.

	Density at 15 °C in kg/m ³
sample #17218-1	798.15
sample #17218-2	798.83
sample #17218-3	798.19
sample #17218-4	798.14
sample #17218-5	797.96
sample #17218-6	798.38
sample #17218-7	798.16
sample #17218-8	798.57

Table 1: homogeneity test results of subsamples #17218

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:

	Density at 15 °C in kg/m ³
r (observed)	0.79
reference test method	ASTM D5002:16
0.3 * R (ref. test method)	0.99

Table 2: evaluation of the repeatability on subsamples #17218

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one or more cans of 5 L (as required) were sent on October 18, 2017. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of crude oil, packed in metal cans with red plastic bag was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine a Crude Oil Assay consisting of a True Boiling Point Distillation in accordance with ASTM D2892 (collection of 8 fractions) and subsequently a distillation in accordance with ASTM D5236 (collection of 4 fractions).

On the original sample Density, Sulphur, Nitrogen and Water was requested to be analysed. 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 of light and medium gasoil a D86-distillation and on the individual fractions 5 (LGO) and 6 (MGO) also a simulated distillation determination was requested.

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

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

3 RESULTS

During twelve weeks after sample dispatch the test results of the individual laboratories were gathered. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

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

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

3.1 STATISTICS

The protocol followed in the 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 March 2017 (iis-protocol, version 3.4).

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's 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. 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 visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected 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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

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

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

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

The z-scores were calculated according to:

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

The $Z_{(\text{target})}$ scores are listed in the result tables of appendix 1.

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

- $|z| < 1$ good
- $1 < |z| < 2$ satisfactory
- $2 < |z| < 3$ questionable
- $3 < |z|$ unsatisfactory

4 EVALUATION

In the proficiency test no major problems were encountered with sample dispatch. After the period of five weeks after sample dispatch only eight participants had reported test results. Eleven laboratories reported test results after the final reporting date, the last test results were submitted by the end of March 2018. Four laboratories did not report any test results at all. Evaluation of test results and preparation of the final report were delayed significantly by these late reported test results.

For objective evaluation iis uses standard reproducibilities to calculate target z-scores. Regrettably this is not possible for most density, sulphur and nitrogen data in this proficiency test, due to the fact that these test 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 test results gathered.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D4377) and an added designation for the year that the method was adopted or revised (e.g. D4377:00). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D4377:00(2011)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D4377:00 will be used.

From the masses of the collected fractions and the respective reported density, sulphur and nitrogen test 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.

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

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

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

4.1 EVALUATION PER TEST

Analysis of the original sample:

Density: The density determination on the original crude sample was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D5002:16.

Sulphur: The sulphur determination on the original crude sample was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D4294:16e1.

Nitrogen: The nitrogen determination on the original crude sample was very problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D5762:17.

Water: The water determination on the original crude sample was not problematic at the low level of 0.04%V/V. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D4377:00(2011).

Distillation test results D2892 and D5236:

The details of the distillation(s), reported by the participants can be found in Appendix 5. Looking at the pressure at the start of the atmospheric distillation (D2892), only one laboratory reported to have used a starting pressure below atmospheric. It concerns lab 1720 with 718 mm Hg, this does not seem to have an effect on the test results of the fractions of the atmospheric distillation. The cuts of the vacuum distillation (D5236) for this laboratory were all either outlier or excluded. As a consequence the majority of the test results of these last four fractions were outliers. The other test results of laboratory 1720 for these four fractions were excluded.

In total, nineteen laboratories reported distillation test results. Two laboratories only performed the atmospheric distillation (D2892) and seventeen also the vacuum distillation (D5236).

The evaluation of the Total Mass balance showed only seven (!) 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%. Remarkably, four recoveries were equal to or higher than 100%. They were excluded in this table for it is highly unlikely that no loss occurred in both distillations. Test results of another two laboratories were excluded because no mass fraction for Gas (LPG) were reported.

Some laboratories did report the mass fraction of the Gas (LPG) but not the density of this fraction. In order to calculate the individual volume recoveries, the consensus value of the group was used when the density of Gas (LPG) was not reported.

After calculation of the Total Volume recovery, one laboratory (lab 1714) had a significantly higher volume recovery than mass recovery. As it turned out, the sample intake that was reported did not match the cumulative mass recoveries of the samples.

D2892: This distillation was problematic depending on the fraction. In total two statistical outliers were detected. However, after rejection of the statistical outliers, the calculated reproducibilities for fraction 4 to 7 are in agreement with the requirements of ASTM D2892:17, while those of fractions one to three are not.

D5236: This distillation may be problematic depending on the fraction. In total two statistical outliers were detected and four test results were excluded from the statistical evaluation. Since the reproducibility of ASTM D5236 is not expressed in mass %, but in °C per 10% liquid volume recovered, the reproducibility of this method cannot be used for the test results in this PT. Since ASTM D2892 also gives a reproducibility at lower operating pressure, this has been used as a guideline in the evaluation. Because of this, no z-scores were calculated. The calculated reproducibilities for fraction 10 and 11 after rejection of the suspect data are in agreement with the requirements of ASTM D2892:17 (low pressure), while that of fraction 9 is not in agreement.

The starts of the true boiling point curves (cumulative %M/M vs temperature AET) of the laboratories show a high resemblance (see Appendix 2). The curve for laboratory 1065 shows a small negative deviation along the whole curve (30°C-520°C). The curve of

laboratory 1613 shows a negative deviation for all parts of the curve (30-520°C). The curve of laboratory 1720 shows a negative deviation at the upper part of the curve (370°C-520°C). This is in line with the deviating test results found for this lab in the vacuum distillation.

The true boiling point curves (Sulphur in %M/M vs temperature AET) of most laboratories show a high resemblance (see Appendix 3). Except for the curve of lab 1720, which reported highly deviating sulfur test results for the fractions MGO and HGO.

The true boiling point curves (Nitrogen in %M/M vs temperature AET) of most laboratories show a high resemblance (see Appendix 4). Again laboratory 1720 has a positive deviation of the curve in the upper part (vacuum distillation). Laboratory 1714 has reported one deviating nitrogen result, which is probably a typing error of due to problems with the nitrogen determination and not with the distillation.

Analysis of distillation fractions:

Density: The density test results on the 12 collected distillation fractions show relatively large differences between the reported test results of the participating laboratories, although in total twelve statistical outliers were observed. The relatively large observed variations 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) variation in the density result and the (large) variation 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) is 0.5837 kg/L, which is in good agreement with the density of a mixture of C4 hydrocarbons and isopentane.

Sulphur: The sulphur test results on the 12 collected distillation fractions show problems for a few participating laboratories. In total six statistical outliers were observed and another four test results were excluded. The observed reproducibility per fraction will be the sum of the variation in the sulphur result and the variation caused by the distillation. Therefore, the observed reproducibilities were not compared with the literature requirements and consequently no z-scores were calculated.

Nitrogen: The nitrogen test results on the 12 collected distillation fractions show problems for a few participating laboratories. In total nine statistical outliers from 3 laboratories were detected. The observed reproducibility per fraction will be the sum of the variation in the nitrogen result and the variation caused by the distillation. Therefore, the observed reproducibilities were not compared with the requirements and consequently no z-scores were calculated.

The theoretical overall Density, Sulphur and Nitrogen results, calculated from the fractions, are discussed in paragraph 4.4 of this report.

P(iP)NA: This determination was performed on fractions 2 (light naphtha) and 3 (heavy naphtha) only. On the light naphtha fraction 2 eight statistical outliers were detected and four test results excluded (all from one laboratory). More analytical problems were observed on fraction 3 (heavy naphtha). In total eleven statistical outliers were observed (from 5 laboratories) and ten test results were excluded (all from 1 laboratory). One laboratory (1720) used ASTM D5134, a method that is applicable up to C9. Therefore, the test results of this laboratory on fraction 3 were excluded. Each observed reproducibility will be the sum of the variation in the analytical method and the variation 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 variations observed during this PT. The set-up of the correct integration window is most critical in the case of testing high naphthenic distillation fractions.

Flash point: This determination was performed on the combined fractions 5+6 only. The determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D93-A:16a.

D86 distillation: This determination was performed on the combined fractions 5+6 only. No analytical problems were encountered. Ten statistical outliers were observed. Six out of seven test results for laboratory 1720 were outliers, therefore the other test result were excluded. Each observed reproducibility will be the sum of the variation in the D86 result and the variation caused by the D2892. Therefore, the observed reproducibilities were not compared with the requirements and consequently no z-scores were calculated.

Simulated Distillation and Effective Cut Point:

Simdist: This determination was performed on both fractions 5 (light gasoil) and 6 (medium gasoil). This simulated distillation was performed by nine participants on both fractions. Most reported test results showed a close resemblance. The goal was to enable evaluation of the column efficiency in accordance with appendix X2 of ASTM D2892:17a. From the reported test results, the ECP (effective cut point) and the standard efficiency N_{minimum} were calculated. A number of test results were not in agreement with the ASTM D2892:17a requirements. The strength of this quality control method becomes clear when the test results of this round are compared with the test results of the previous round iis15R02. A number of laboratories participated in both rounds and it is good to see that some participants improved the distillation (like laboratory 1990 (ECP and Standard Efficiency) and 171 (Standard Efficiency)). Regretfully for other participants the performance of the distillation did not improve, based on the calculation of the ECP and the Standard Efficiency.

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.

Original sample (analysis and distillation):

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sd_R</i>	<i>R (lit)</i>
Density of #17218	kg/m ³	18	799.0	2.3	3.3
Sulphur of #17218	%M/M	15	0.580	0.023	0.051
Nitrogen of #17218	mg/kg	10	261.2	136.4	69.5
Water of #17218	%V/V	12	0.043	0.024	0.033
D2892 distillation	True boiling point curve				
Gas LPG < 30°C	%M/M	16	3.8	2.1	1.3
Light Naphtha 30 - 90°C	%M/M	19	10.0	1.9	1.3
Heavy Naphtha 90 - 180°C	%M/M	19	24.6	1.7	1.3
Kerosene 180 - 215°C	%M/M	19	7.8	1.4	1.5
LGO 215 - 250°C	%M/M	19	7.9	0.9	1.5
MGO 250 - 310°C	%M/M	19	12.2	2.0	2
HGO 310 - 370°C	%M/M	19	10.2	2.0	2
Residue > 370°C	%M/M	18	22.9	4.0	n.a.
D5236 distillation	True boiling point curve				
VGO 370 - 420°C	%M/M	15	4.8	4.6	(2)
VGO 420 - 470°C	%M/M	15	6.3	2.1	(2)
VGO 470 - 520°C	%M/M	16	4.0	1.0	(2)
Residue > 520°C	%M/M	16	7.5	1.4	n.a.

Table 3: reproducibilities of test results on original crude sample #17218

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

Analysis on distillation fractions:

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. For Sulphur and Nitrogen the reproducibilities of the method have been added in brackets for information purposes.

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R (lit)</i>
Density at 15°C of fract. 1: gas <30°C	kg/L	10	0.5837	0.0268	n.r.
Density at 15°C of fract. 2: 30-90°C	kg/L	18	0.6719	0.0080	n.r.
Density at 15°C of fract. 3: 90-180°C	kg/L	18	0.7566	0.0035	n.r.
Density at 15°C of fract. 4: 180-215°C	kg/L	18	0.7982	0.0031	n.r.
Density at 15°C of fract. 5: 215-250°C	kg/L	19	0.8142	0.0032	n.r.
Density at 15°C of fract. 6: 250-310°C	kg/L	18	0.8395	0.0038	n.r.
Density at 15°C of fract. 7: 310-370°C	kg/L	18	0.8652	0.0066	n.r.
Density at 15°C of fract. 8: >370°C	kg/L	17	0.9114	0.0057	n.r.
Density at 15°C of fract.9: 370-420°C	kg/L	15	0.8840	0.0085	n.r.
Density at 15°C of fract.10: 420-470°C	kg/L	15	0.8931	0.0066	n.r.
Density at 15°C of fract. 11: 470-520°C	kg/L	15	0.9079	0.0073	n.r.
Density at 15°C of fract. 12: >520°C	kg/L	15	0.9500	0.0104	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 *sd</i>	<i>R (lit)</i>
Sulphur of fract. 1: gas <30°C	%M/M	4	(0.021)	n.a.	n.a.
Sulphur of fract. 2: 30-90°C	%M/M	12	0.091	0.012	(0.016)
Sulphur of fract. 3: 90-180°C	%M/M	14	0.212	0.039	(0.027)
Sulphur of fract. 4: 180-215°C	%M/M	15	0.212	0.031	(0.027)
Sulphur of fract. 5: 215-250°C	%M/M	15	0.259	0.045	(0.030)
Sulphur of fract. 6: 250-310°C	%M/M	13	0.545	0.062	(0.049)
Sulphur of fract. 7: 310-370°C	%M/M	14	1.097	0.177	(0.077)
Sulphur of fract. 8: >370°C	%M/M	14	1.226	0.077	(0.083)
Sulphur of fract.9: 370-420°C	%M/M	14	1.013	0.131	(0.073)
Sulphur of fract.10: 420-470°C	%M/M	14	1.070	0.110	(0.076)
Sulphur of fract. 11: 470-520°C	%M/M	14	1.182	0.164	(0.081)
Sulphur of fract. 12: >520°C	%M/M	14	1.495	0.165	(0.094)

table 5: reproducibilities of sulphur determinations on distillation fractions

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sd</i>	<i>R (lit)</i>
Nitrogen of fract. 1: gas <30°C	mg/kg	3	n.a.	n.a.	n.a.
Nitrogen of fract. 2: 30-90°C	mg/kg	6	0.445	0.519	(0.534)
Nitrogen of fract. 3: 90-180°C	mg/kg	7	0.483	0.478	(0.556)
Nitrogen of fract. 4: 180-215°C	mg/kg	7	0.654	1.109	(0.651)
Nitrogen of fract. 5: 215-250°C	mg/kg	8	0.911	1.574	(0.772)
Nitrogen of fract. 6: 250-310°C	mg/kg	12	7.027	9.533	(2.209)
Nitrogen of fract. 7: 310-370°C	mg/kg	12	203.1	134.1	(54.0)
Nitrogen of fract. 8: >370°C	mg/kg	11	1034	351	(275)
Nitrogen of fract.9: 370-420°C	mg/kg	12	430.1	157.6	(114.4)
Nitrogen of fract.10: 420-470°C	mg/kg	12	574.0	163.3	(152.7)
Nitrogen of fract. 11: 470-520°C	mg/kg	12	786.9	437.6	(209.3)
Nitrogen of fract. 12: >520°C	mg/kg	11	1931	796	n.a.

Table 6: reproducibilities of nitrogen determinations on distillation fractions

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sd</i>	<i>R (lit)</i>
total paraffins	%V/V	12	84.76	3.48	n.r.
C1-C4	%V/V	10	0.37	1.06	n.r.
n-paraffins	%V/V	11	41.77	3.08	n.r.
i-paraffins	%V/V	11	42.95	4.66	n.r.
naphthenes	%V/V	12	13.78	3.18	n.r.
aromatics	%V/V	12	1.44	0.48	n.r.
total paraffins	%M/M	13	82.41	3.57	n.r.
C1-C4	%M/M	11	0.35	0.90	n.r.
n-paraffins	%M/M	12	40.14	2.58	n.r.
i-paraffins	%M/M	12	42.20	4.20	n.r.
naphthenes	%M/M	13	15.68	3.19	n.r.
aromatics	%M/M	13	1.90	0.60	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 *sd</i>	<i>R (lit)</i>
total paraffins	%V/V	10	54.64	2.12	n.r.
C1-C4	%V/V	9	<0.1	n.a.	n.r.
n-paraffins	%V/V	9	24.72	2.43	n.r.
i-paraffins	%V/V	11	31.12	9.97	n.r.
naphthenes	%V/V	11	32.66	6.73	n.r.
aromatics	%V/V	12	13.24	3.10	n.r.
total paraffins	%M/M	11	51.49	2.13	n.r.
C1-C4	%M/M	11	<0.1	n.a.	n.r.
n-paraffins	%M/M	10	22.95	2.36	n.r.
i-paraffins	%M/M	12	29.54	8.87	n.r.
naphthenes	%M/M	11	33.20	4.41	n.r.
aromatics	%M/M	13	15.20	3.39	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>
Flash point	°C	8	101.3	6.7	7.2
Distillation - IBP	°C	11	224.8	20.1	(12.4)
Distillation - 5% recovered	°C	11	238.0	4.5	(7.1)
Distillation - 10% recovered	°C	11	240.1	4.9	(5.3)
Distillation - 50% recovered	°C	11	253.0	6.2	(3)
Distillation - 90% recovered	°C	11	279.9	8.6	(4.2)
Distillation - 95% recovered	°C	11	286.5	10.0	(6.0)
Distillation - FBP	°C	10	293.5	9.5	(7.1)

Table 9: reproducibilities of tests on combined distillation fractions 5+6

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2017 WITH PREVIOUS PTS

The performance of the determinations of the proficiency tests was compared to the requirements of the respective standards. The conclusions are given in the following table:

	November 2017	November 2015	November 2013	November 2011
Density of original sample	+	+	++	++
Sulphur of original sample	++	-	-	+
Nitrogen of original sample	--	+	--	-
Water of original sample	+	--	-	n.e.
D2892 distillation				
Gas LPG < 30°C	-	+/-	+	++
Light Naphtha 30 - 90°C	-	+	+	+
Heavy Naphtha 90 - 180°C	-	+	+/-	+
Kerosene 180 - 215°C	+/-	+	+	++
LGO 215 - 250°C	+	++	+	+
MGO 250 - 310°C	+/-	++	+/-	+
HGO 310 - 370°C	+/-	+	+	+
Residue > 370°C	n.e.	n.e.	n.e.	n.e.
D5236 distillation				
VGO 370 - 420°C	(--)	(--)	--	n.e.
VGO 420 - 470°C	(+/-)	(-)	--	n.e.
VGO 470 - 520°C	(++)	(+/-)	-	n.e.
sum VGO 520 - 565°C+Residue >565°C	n.e.	n.e.	n.e.	n.e.

Table 5: comparison determinations against the standard

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method
- n.e.: not evaluated

4.4 DISCUSSION

In this PT, most laboratories (seventeen in total) reported test results within 10 weeks of the dispatch date. The normal time schedule is not sufficient for the completion of a round robin on Crude Oil Assay, although 8 to 10 weeks seems to be an acceptable time frame.

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 test results do show some aberrant results. These deviations may be (partly) explained by the cumulative effect of variations caused by distillation and by subsequent analytical determinations.

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, sulphur and nitrogen content (see below table).

Parameter	unit	average measured result	average theoretical result	Absolute difference in averages	average recovery
Density	kg/L	0.7990	0.7962	-0.0028	100%
Sulphur	%M/M	0.5802	0.5485	-0.0317	106%
Nitrogen	mg/kg	261.2	255.4	-5.8	102%

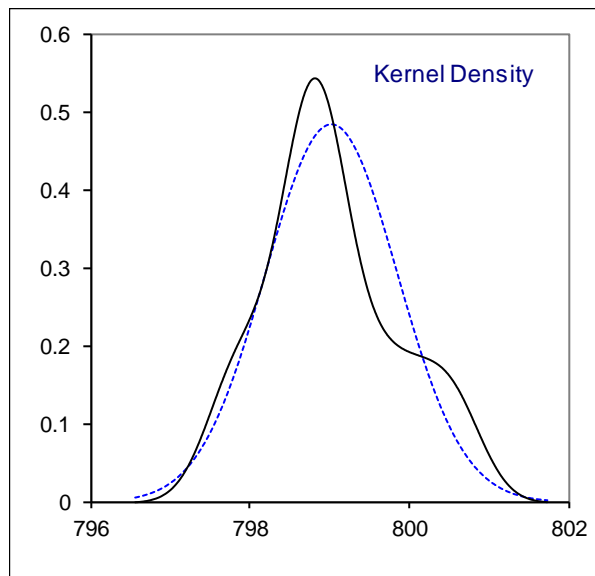
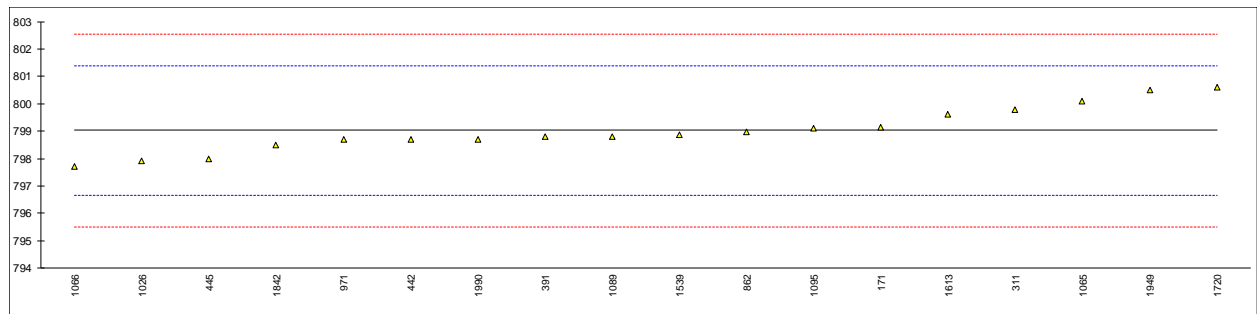
Table 10: comparison of actual measured values and theoretical values calculated from the fractions

APPENDIX 1A – ANALYSIS OF ORIGINAL SAMPLE

Determination of Density at 15°C on original sample #17218; results in kg/m³

lab	method	value	mark	z(targ)	remarks
171	D5002	799.14		0.10	
311	D5002	799.8		0.66	
391	D5002	798.8		-0.19	
442	IP365	798.7		-0.28	
445	D5002	798.0		-0.87	
574		----		----	
608		----		----	
862	D5002	798.96		-0.06	
971	D5002	798.7		-0.28	
1026		797.9		-0.96	
1065	D4052	800.1		0.91	
1066	D5002	797.7		-1.13	
1080		----		----	
1089	D5002	798.8		-0.19	
1095	D5002	799.1		0.06	
1539	ISO12185/D5002	798.87		-0.13	
1613	D5002	799.6		0.49	
1714		----	W	----	first reported: 886.0
1720	D5002	800.6		1.34	
1842	IP365	798.5	C	-0.45	reported: 0.7985 kg/m ³
1949	D5002	800.5		1.25	
1990	D4052	798.7		-0.28	
6156		----		----	

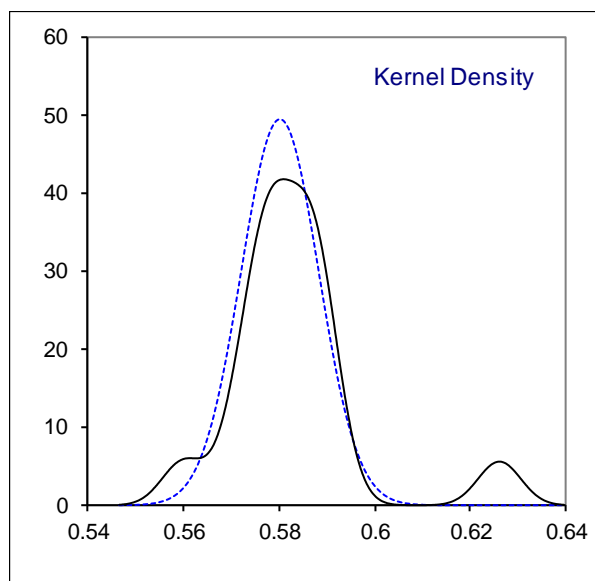
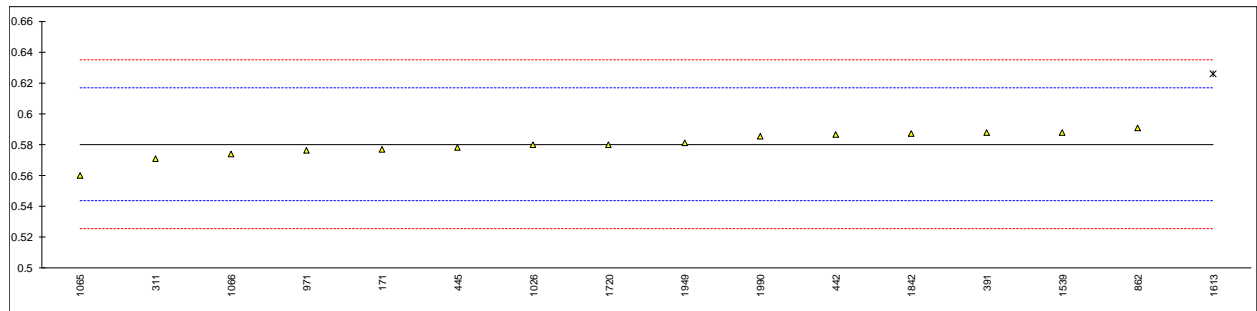
normality OK
n 18
outliers 0
mean (n) 799.03
st.dev. (n) 0.8245
R(calc.) 2.31
st.dev.(D5002:16) 1.176
R(D5002:16) 3.29



Determination of Sulphur on original sample #17218; results in %M/M

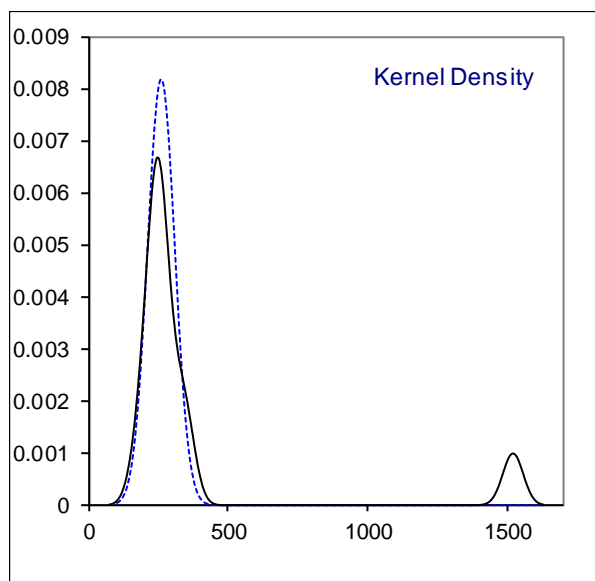
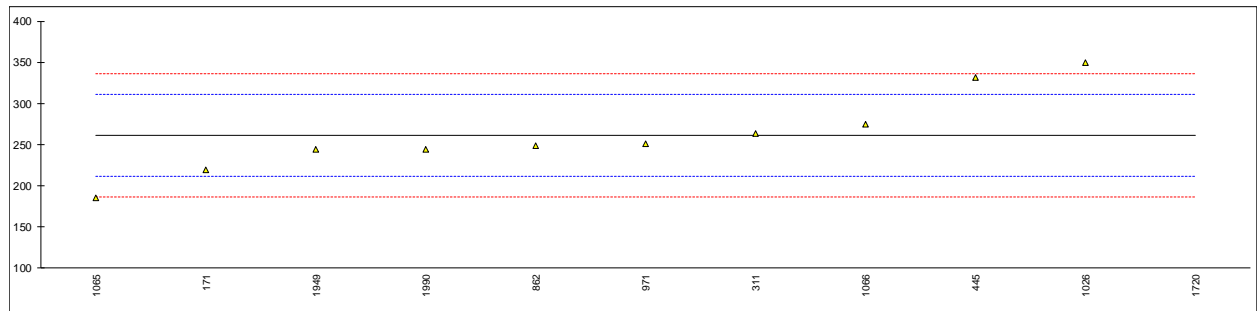
lab	method	value	mark	z(targ)	remarks
171	D4294	0.5767		-0.19	
311	D4294	0.571		-0.50	
391	ISO8754	0.588		0.43	
442	IP336	0.5865		0.35	
445	D4294	0.578		-0.12	
574		----		----	
608		----		----	
862	D2622	0.591		0.59	
971	D4294	0.576		-0.23	
1026		0.58		-0.01	
1065	D4294	0.56		-1.10	
1066	D4294	0.574		-0.34	
1080		----		----	
1089		----		----	
1095		----		----	
1539	ISO8754	0.588		0.43	
1613	D4294	0.626	G(0.01)	2.51	
1714		----	W	----	first reported: 0.636
1720	D4294	0.580		-0.01	
1842	INH-05	0.587		0.37	
1949	D4294	0.5809		0.04	
1990	D4294	0.5852		0.28	
6156		----		----	

normality suspect
n 15
outliers 1
mean (n) 0.5802
st.dev. (n) 0.00809
R(calc.) 0.0226
st.dev.(D4294:16e1) 0.01827
R(D4294:16e1) 0.0512



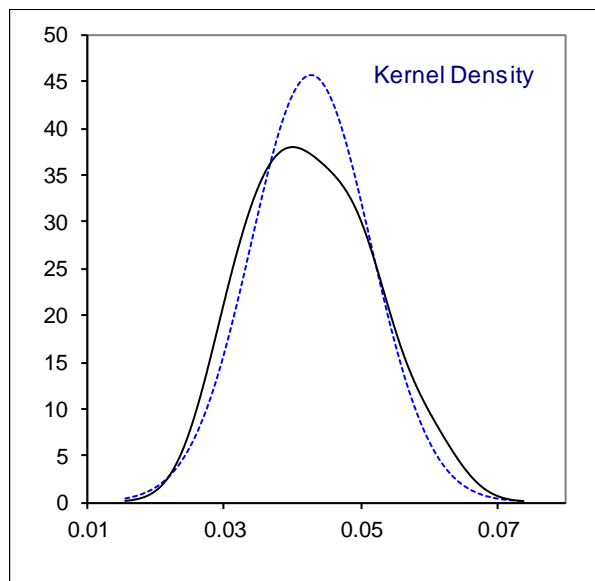
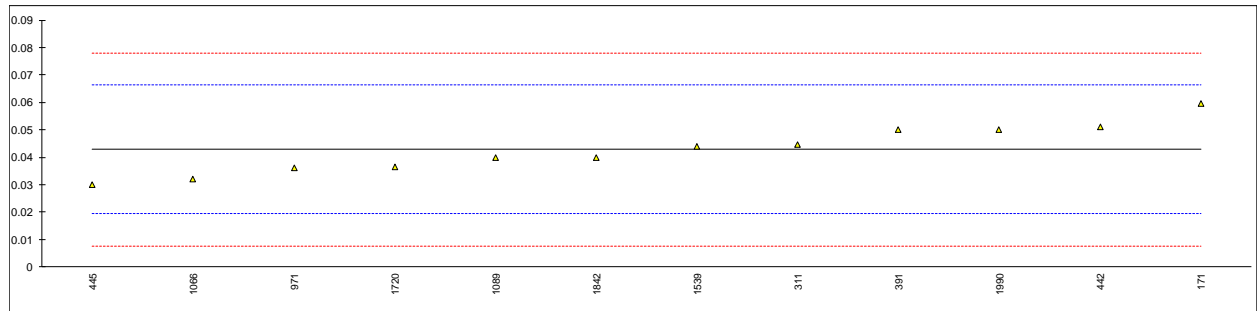
Determination of Nitrogen on original sample #17218; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D5762	219.76		-1.67	
311	D5762	263		0.07	
391		----		----	
442		----		----	
445	D5762	331.4		2.83	
574		----		----	
608		----		----	
862	D5762	249.2		-0.48	
971	D5762	251		-0.41	
1026		350		3.58	
1065	D5762	185		-3.07	
1066	D5762	274.77		0.55	
1080		----		----	
1089		----		----	
1095		----		----	
1539		----		----	
1613		----		----	
1714		----	W	----	first reported: 1400
1720	D5762	1519.42	G(0.01)	50.70	
1842		----		----	
1949	D5762	243.7		-0.71	
1990	D5762	244.22		-0.68	
6156		----		----	
normality		OK			
n		10			
outliers		1			
mean (n)		261.21			
st.dev. (n)		48.725			
R(calc.)		136.43			
st.dev.(D5762:17)		24.814			
R(D5762:17)		69.48			



Determination of Water on original sample #17218; results in %V/V

lab	method	value	mark	z(targ)	remarks
171	D4377	0.0595		1.42	
311	D4377	0.0445		0.15	
391	D4377	0.05		0.61	
442	IP386	0.051		0.70	
445	D4928	0.03		-1.09	
574		----		----	
608		----		----	
862		----		----	
971	D4928	0.036		-0.58	
1026		<0.01		----	
1065	D4006	<0.025		----	
1066	D4377	0.032		-0.92	
1080		----		----	
1089	D4377	0.04		-0.24	
1095	D6304	<0.02		----	
1539	D4377	0.044		0.10	
1613	D4377	<0.02		----	
1714		----		----	
1720	D4377	0.0364		-0.54	
1842	D6304	0.04		-0.24	
1949	D4006	<0.025		----	
1990	D4007	0.05		0.61	
6156		----		----	
normality		OK			
n		12			
outliers		0			
mean (n)		0.0428			
st.dev. (n)		0.00873			
R(calc.)		0.0244			
st.dev.(D4377:00)		0.01174			
R(D4377:00)		0.0329			

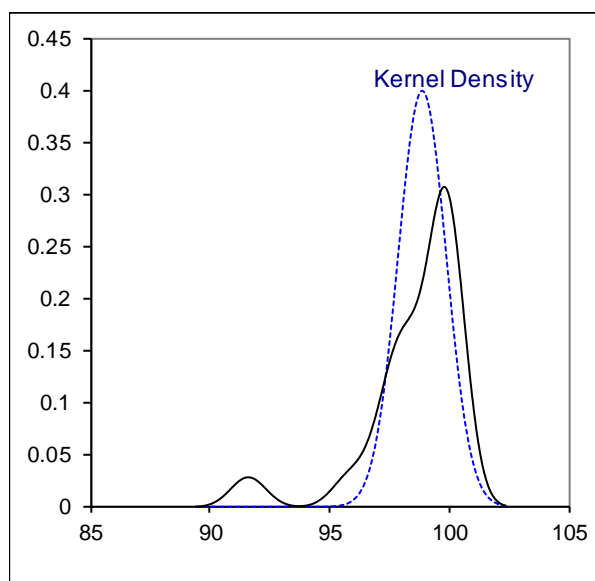
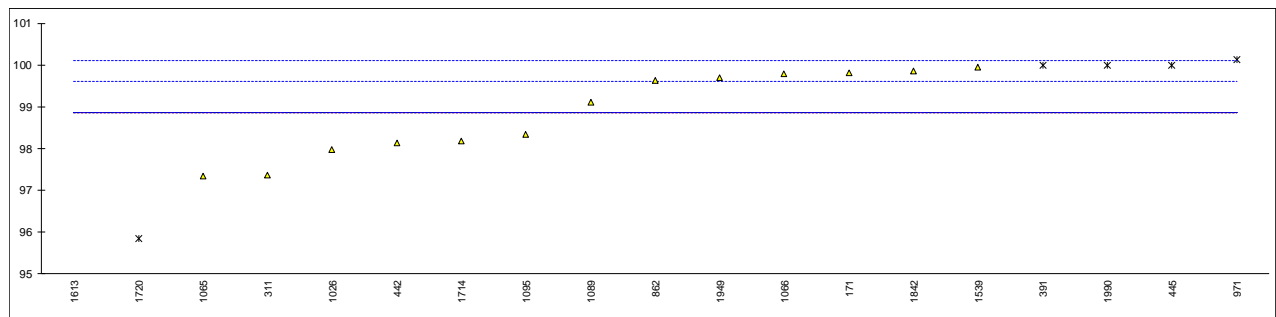


APPENDIX 1B – DISTILLATION RESULTS D2892 AND D5236

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

lab	method	value	mark	remarks
171	calc	99.82		
311	calc	97.36		
391	calc	100.00	ex	excluded, recovery corrected for loss?
442	calc	98.13		
445	calc	100.00	ex	excluded, recovery corrected for loss?
574		-----		
608		-----		
862	calc	99.62		
971	calc	100.12	ex	excluded, recovery corrected for loss?
1026	calc	97.98		
1065	calc	97.34		
1066	calc	99.80		
1080		-----		
1089	calc	99.12		
1095	calc	98.33		
1539	calc	99.95		
1613	calc	91.63	ex	excluded, for no mass fraction was reported for Gas (LPG)
1714	calc	98.18		
1720	calc	95.85	ex	excluded, for no mass fraction was reported for Gas (LPG)
1842	calc	99.86		
1949	calc	99.69		
1990	calc	100.00	ex	excluded, recovery corrected for loss?
6156		-----		
	normality	OK		
	n	13		
	outliers	0 (+6ex)		
	mean (n)	98.86		
	st.dev. (n)	0.998		
	R(calc.)	2.79		

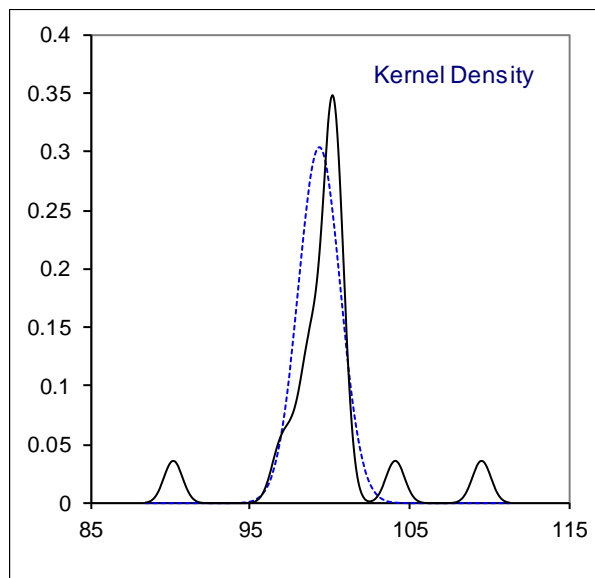
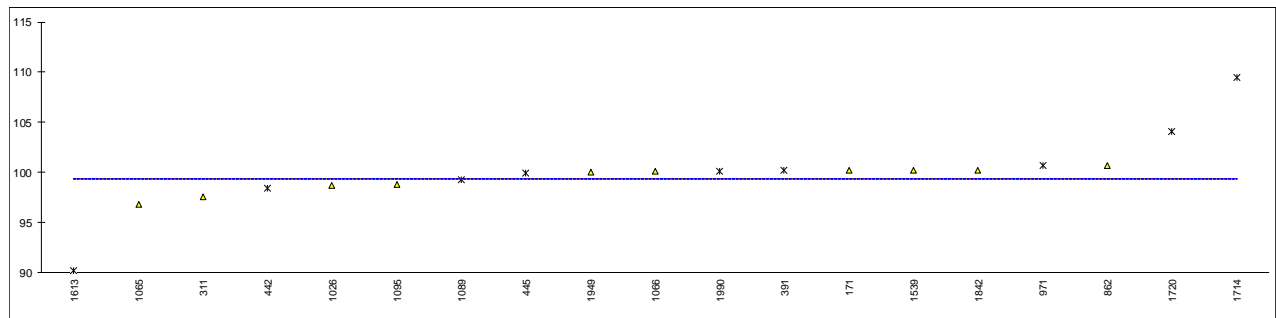
In below graph, the dotted lines represent the ASTM D5236 requirements for recovery: 99.6% < recovery < 100.1%, of which the lower line at 99.6% is equal to the 0.4% max. loss requirement of ASTM D2892:17a, paragraph 11.2.



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

lab	method	value	mark	remarks
171	calc	100.20		
311	calc	97.56		
391	calc	100.19	ex	excluded, recovery corrected for loss?
442	calc	98.45	ex	not enough data
445	calc	99.96	ex	excluded, recovery corrected for loss?
574		-----		
608		-----		
862	calc	100.73		
971	calc	100.65	ex	excluded, recovery corrected for loss?
1026	calc	98.70		
1065	calc	96.81		
1066	calc	100.09		
1080		-----		
1089	calc	99.27	ex	not enough data
1095	calc	98.83		
1539	calc	100.21		
1613	calc	90.19	ex	excluded, for no mass fraction was reported for Gas (LPG)
1714	calc	109.51	G(0.05)	
1720	calc	104.10	ex	excluded, for no mass fraction was reported for Gas (LPG)
1842	calc	100.27		
1949	calc	100.04		
1990	calc	100.13	ex	excluded, recovery corrected for loss?
6156		-----		

normality OK
n 10
outliers 1 (+8ex)
mean (n) 99.34
st.dev. (n) 1.315
R(calc.) 3.68



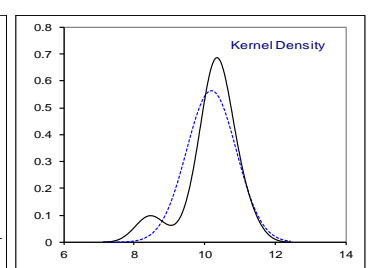
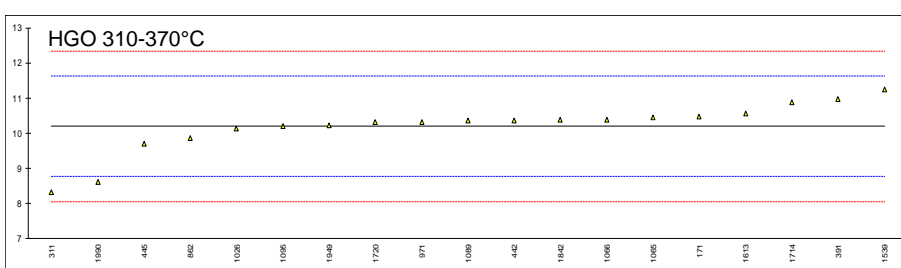
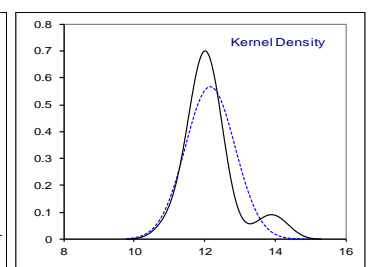
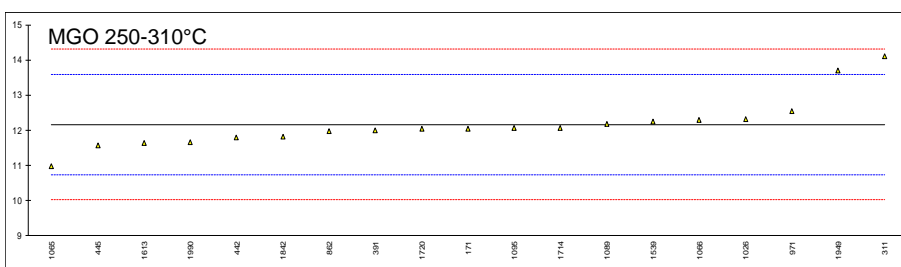
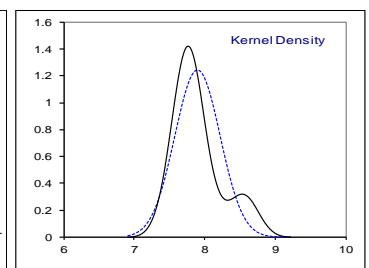
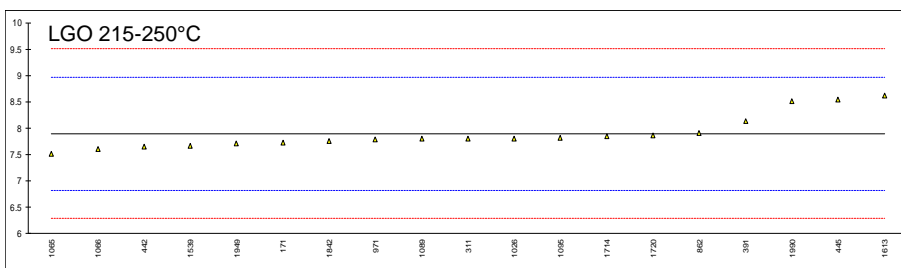
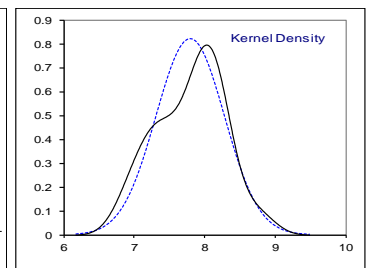
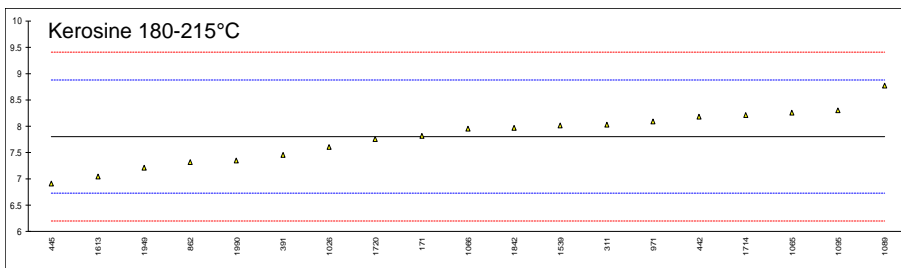
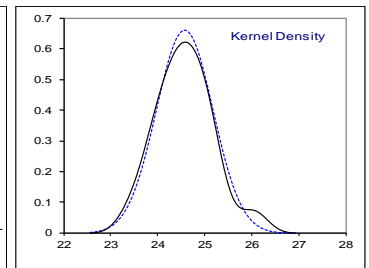
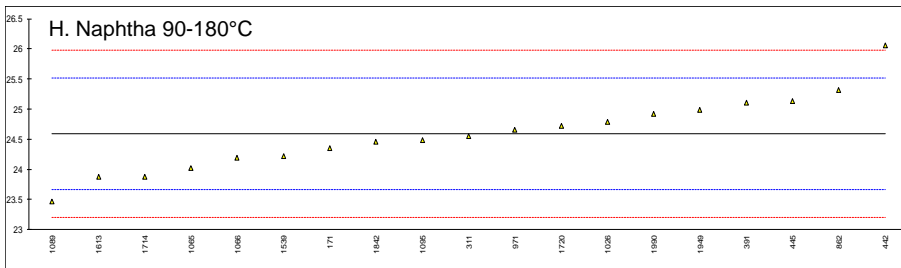
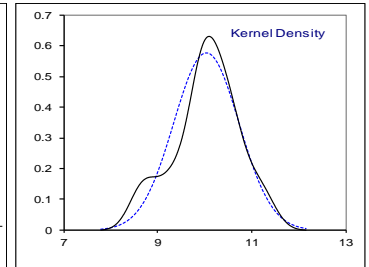
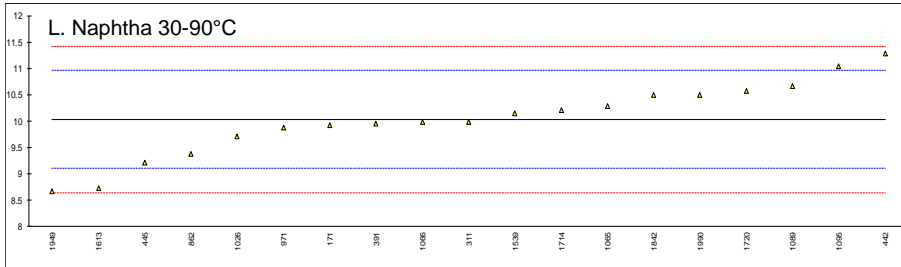
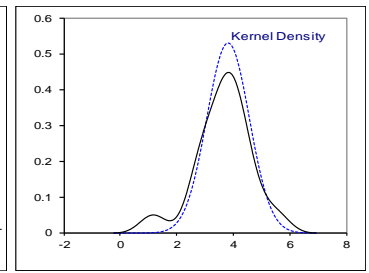
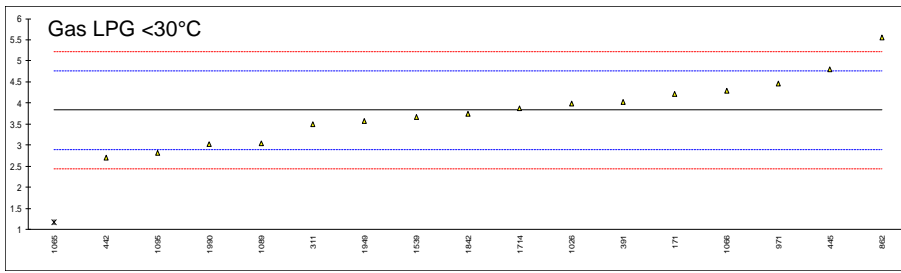
Determination of true boiling point curve of D2892; individual fractions in %M/M

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	4.2127	9.9292	24.3527	7.8221	7.7226	12.0457	10.4766	23.2564
311	3.4884	9.9894	24.5507	8.0338	7.7960	14.1121	8.3245	21.1681
391	4.0332	9.9484	25.1017	7.4532	8.1352	12.0045	10.9643	22.3596
442	2.7051	11.2773	26.0552	8.1856	7.6535	11.7822	10.3686	20.0980
445	4.7947	9.2090	25.1327	6.9061	8.5457	11.5632	9.7084	24.1402
574	----	----	----	----	----	----	----	----
608	----	----	----	----	----	----	----	----
862	5.5555	9.3755	25.3192	7.3213	7.9012	11.9733	9.8675	21.5113
971	4.4538	9.8809	24.6615	8.0908	7.7849	12.5460	10.3115	22.4408
1026	3.9814	9.7170	24.7932	7.6030	7.8070	12.3132	10.1250	25.5906
1065	1.1742	10.2775	24.0224	8.2583	7.5111	10.9874	10.4501	24.6575
1066	4.2841	9.9834	24.1869	7.9546	7.6040	12.2916	10.3870	23.1606
1080	----	----	----	----	----	----	----	----
1089	3.0514	10.6685	23.4637	8.7690	7.7943	12.1749	10.3564	22.8395
1095	2.8203	11.0404	24.4825	8.2934	7.8135	12.0549	10.2049	21.7452
1539	3.6652	10.1463	24.2198	8.0108	7.6615	12.2367	11.2461	22.7763
1613	----	8.7305	23.8743	7.0419	8.6148	11.6385	10.5629	22.0013
1714	3.8752	10.2085	23.8774	8.2044	7.8426	12.0735	10.8803	22.6856
1720	----	10.5774	24.7257	7.7563	7.8634	12.0415	10.3060	22.5831
1842	3.7368	10.4924	24.4560	7.9614	7.7555	11.8256	10.3867	33.2873
1949	3.5781	8.6606	24.9816	7.2135	7.7177	13.6885	10.2303	23.6245
1990	3.0206	10.4974	24.9176	7.3435	8.5046	11.6507	8.6066	25.4590
6156	----	----	----	----	----	----	----	----
normality	OK	OK	OK	OK	not OK	not OK	not OK	OK
n	16	19	19	19	19	19	19	18
outliers	1	0	0	0	0	0	0	1
mean (n)	3.8285	10.0321	24.5882	7.8012	7.8963	12.1581	10.1981	22.8943
st.dev. (n)	0.75181	0.69242	0.60361	0.48620	0.32029	0.70571	0.70606	1.42916
R(calc.)	2.1051	1.9388	1.6901	1.3614	0.8968	1.9760	1.9770	4.0017
st.dev.(D2892:17)	0.46429	0.46429	0.46429	0.53571	0.53571	0.71429	0.71429	n.a.
R(D2892:17)	1.3	1.3	1.3	1.5	1.5	2	2	n.a.

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

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	0.83	-0.22	-0.51	0.04	-0.32	-0.16	0.39	----
311	-0.73	-0.09	-0.08	0.43	-0.19	2.74	-2.62	----
391	0.44	-0.18	1.11	-0.65	0.45	-0.22	1.07	----
442	-2.42	2.68	3.16	0.72	-0.45	-0.53	0.24	----
445	2.08	-1.77	1.17	-1.67	1.21	-0.83	-0.69	----
574	----	----	----	----	----	----	----	----
608	----	----	----	----	----	----	----	----
862	3.72	-1.41	1.57	-0.90	0.01	-0.26	-0.46	----
971	1.35	-0.33	0.16	0.54	-0.21	0.54	0.16	----
1026	0.33	-0.68	0.44	-0.37	-0.17	0.22	-0.10	----
1065	-5.72	0.53	-1.22	0.85	-0.72	-1.64	0.35	----
1066	0.98	-0.10	-0.86	0.29	-0.55	0.19	0.26	----
1080	----	----	----	----	----	----	----	----
1089	-1.67	1.37	-2.42	1.81	-0.19	0.02	0.22	----
1095	-2.17	2.17	-0.23	0.92	-0.15	-0.14	0.01	----
1539	-0.35	0.25	-0.79	0.39	-0.44	0.11	1.47	----
1613	----	-2.80	-1.54	-1.42	1.34	-0.73	0.51	----
1714	0.10	0.38	-1.53	0.75	-0.10	-0.12	0.96	----
1720	----	1.17	0.30	-0.08	-0.06	-0.16	0.15	----
1842	-0.20	0.99	-0.28	0.30	-0.26	-0.47	0.26	----
1949	-0.54	-2.95	0.85	-1.10	-0.33	2.14	0.05	----
1990	-1.74	1.00	0.71	-0.85	1.14	-0.71	-2.23	----
6156	----	----	----	----	----	----	----	----



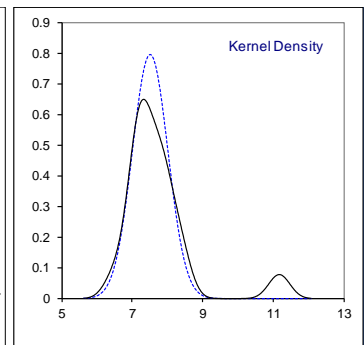
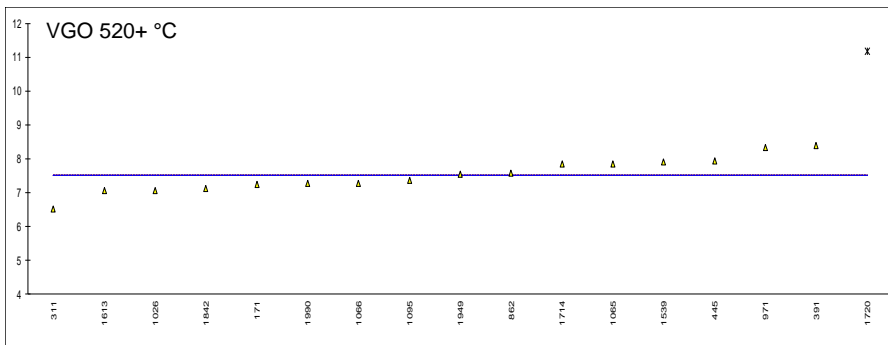
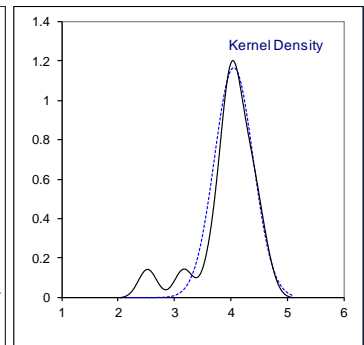
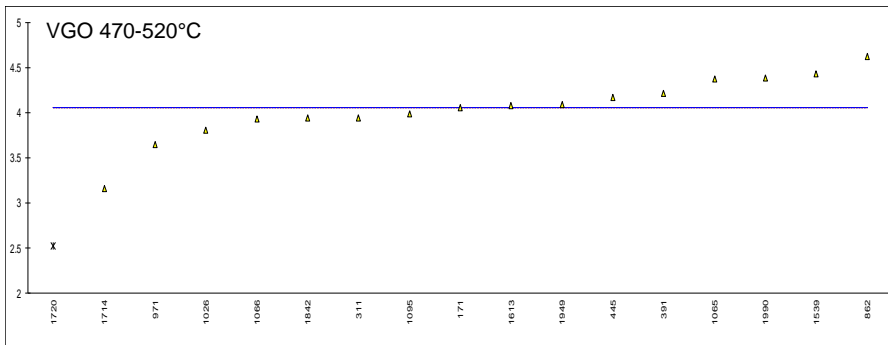
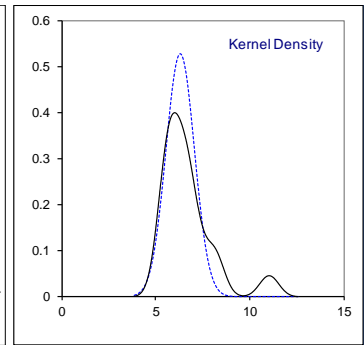
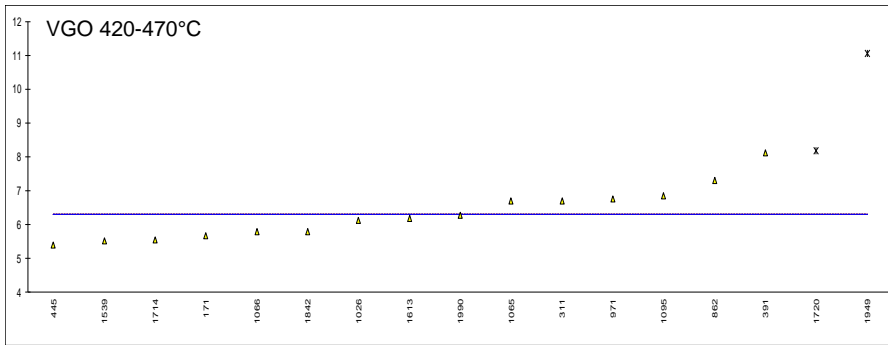
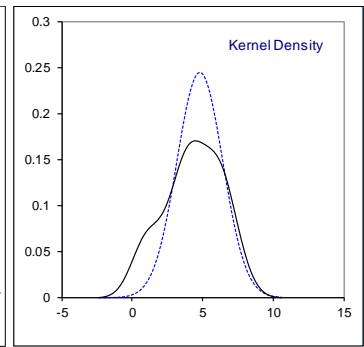
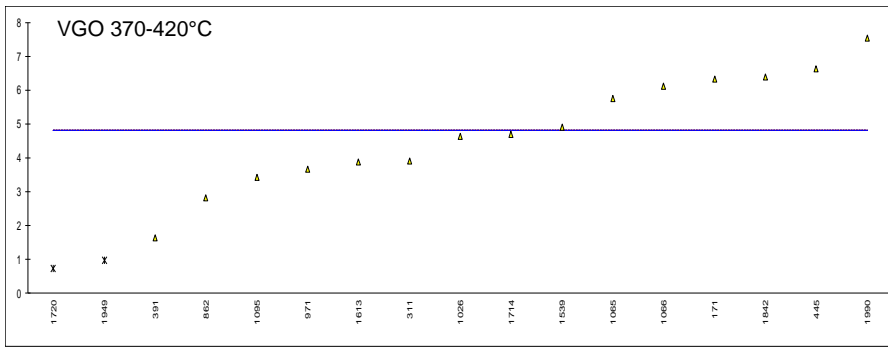
Determination of true boiling point curve D5236 (continued); individual fractions in %M/M

lab	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue 520+ °C	remarks
171	6.3122	5.6565	4.0512	7.2365	
311	3.9112	6.6860	3.9376	6.5275	
391	1.6406	8.1137	4.2120	8.3932	
442	----	----	----	----	
445	6.6395	5.4005	4.1677	7.9349	
574	----	----	----	----	
608	----	----	----	----	
862	2.8270	7.2855	4.6159	7.5831	
971	3.6508	6.7548	3.6508	8.3373	
1026	4.6369	6.1331	3.7994	7.0731	
1065	5.7444	6.6858	4.3679	7.8594	
1066	6.1116	5.7923	3.9250	7.2771	
1080	----	----	----	----	
1089	----	----	----	----	
1095	3.4346	6.8532	3.9846	7.3509	
1539	4.9033	5.5305	4.4231	7.9096	
1613	3.8694	6.1696	4.0708	7.0565	
1714	4.6808	5.5455	3.1625	7.8321	
1720	<i>0.7209</i>	<i>8.1730</i>	<i>2.5191</i>	<i>11.1701</i>	
1842	6.3955	5.8041	3.9370	7.1065	
1949	<i>0.9718</i>	<i>11.0404</i>	4.0853	7.5271	
1990	7.5420	6.2796	4.3778	7.2595	
6156	----	----	----	----	
normality	OK	OK	suspect	OK	
n	15	15	16	16	
outliers	0 (+2ex)	1 (+1ex)	0 (+1ex)	1	
mean (n)	4.8200	6.3127	4.0480	7.5165	
st.dev. (n)	1.62805	0.75605	0.34300	0.50181	
R(calc.)	4.5585	2.1169	0.9604	1.4051	
st.dev.(D2892:17)	(0.71429)	(0.71429)	(0.71429)	n.a.	
R(D2892:17)	(2)	(2)	(2)	n.a.	

NB. Bold and underlined figures are statistical outliers (Grubbs)
 Bold and italic figures are excluded test results, for possible different cut points, although total mass is correct.

Corresponding z-scores for above mass fractions:

lab	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue 520+ °C
171	----	----	----	----
311	----	----	----	----
391	----	----	----	----
442	----	----	----	----
445	----	----	----	----
574	----	----	----	----
608	----	----	----	----
862	----	----	----	----
971	----	----	----	----
1026	----	----	----	----
1065	----	----	----	----
1066	----	----	----	----
1080	----	----	----	----
1089	----	----	----	----
1095	----	----	----	----
1539	----	----	----	----
1613	----	----	----	----
1714	----	----	----	----
1720	----	----	----	----
1842	----	----	----	----
1949	----	----	----	----
1990	----	----	----	----
6156	----	----	----	----



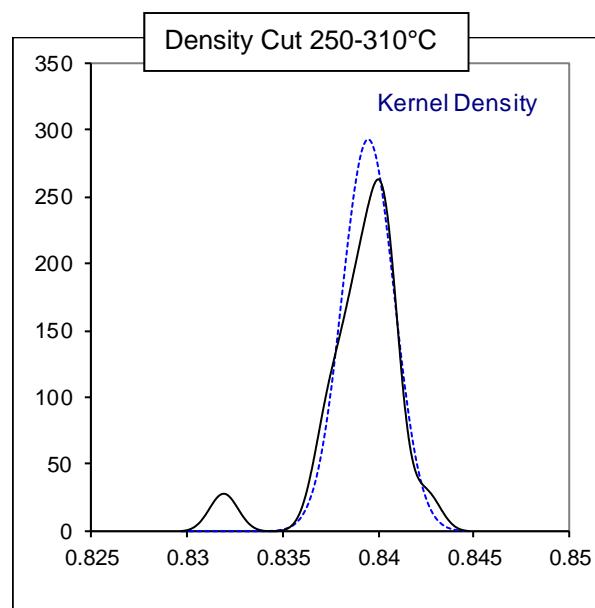
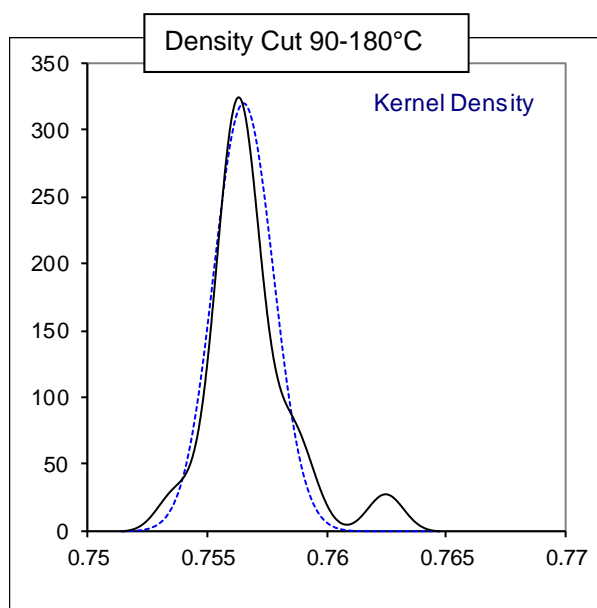
APPENDIX 1C – ANALYSIS OF DISTILLATION FRACTIONS

Determination of Density at 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	----	0.6729	0.7564	0.7980	0.8134	0.8370	0.8662	0.9099
311	D4052	0.5933	0.6783	0.7584	0.7989	0.8160	0.8425	0.8693	0.9127
391	D4052	0.5853	0.6729	0.7571	0.7990	0.8141	0.8404	0.8655	0.9186
442	IP365	----	0.6697	0.7625	0.7996	0.8155	0.8402	0.8658	0.9137
445	D4052	0.6052	0.6783	0.7570	0.7986	0.8160	0.8383	0.8656	0.9137
574		----	----	----	----	----	----	----	----
608		----	----	----	----	----	----	----	----
862	D4052	0.5759	0.6696	0.7565	0.8001	0.8145	0.8376	0.8686	0.9124
971	D4052	0.5776	0.6733	0.7569	0.7983	0.8153	0.8407	0.8639	0.9159
1026	D4052	0.5856	0.6708	0.7561	0.7985	0.8130	0.8391	0.8640	0.9121
1065	D4052	----	0.6721	0.7564	0.7981	0.8139	0.8402	0.8612	0.9093
1066	D4052	0.5821 C	0.6738	0.7560	0.7981	0.8130	0.8389	0.8646	0.9105
1080		----	----	----	----	----	----	----	----
1089	D4052	0.5758	0.6725	0.7567	0.7979	0.8152	0.8401	0.8661	0.9100
1095	D4052	0.5736	0.6689	0.7584	0.7984	0.8147	0.8405	0.8539	0.9128
1539	ISO12185	----	0.6711	0.7564	0.7982	0.8140	0.8400	0.8656	----
1613	D4052	----	0.6872	0.7592	0.7989	0.8152	0.8392	0.8672	0.9121
1714	D4052	----	0.6722	0.7558	0.7976	0.8144	0.8375	0.8678	0.9101
1720	D4052	----	0.6688	0.7558	0.7959	0.8125	0.8403	0.8635	0.9084
1842	IP365	----	0.6696	0.7561	0.7978	0.8136	0.8385	0.8636	0.9114
1949	D4052	----	0.6703	0.7537	0.7955	0.8119	0.8319	0.8649	0.9107
1990	D4052	0.5828	0.6688	0.7552	0.8053	0.8141	0.8392	0.8600	0.9083
6156		----	----	----	----	----	----	----	----
	normality	not OK	not OK	suspect	suspect	OK	OK	OK	OK
	n	10	18	18	18	19	18	18	17
	outliers	0	1	1	1	0	1	1	1
	mean (n)	0.5837	0.6719	0.7566	0.7982	0.8142	0.8395	0.8652	0.9114
	st.dev. (n)	0.00958	0.00285	0.00125	0.00110	0.00115	0.00136	0.00235	0.00204
	R(calc.)	0.0268	0.0080	0.0035	0.0031	0.0032	0.0038	0.0066	0.0057
	RSD	1.6%	0.4%	0.2%	0.1%	0.1%	0.2%	0.3%	0.2%

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

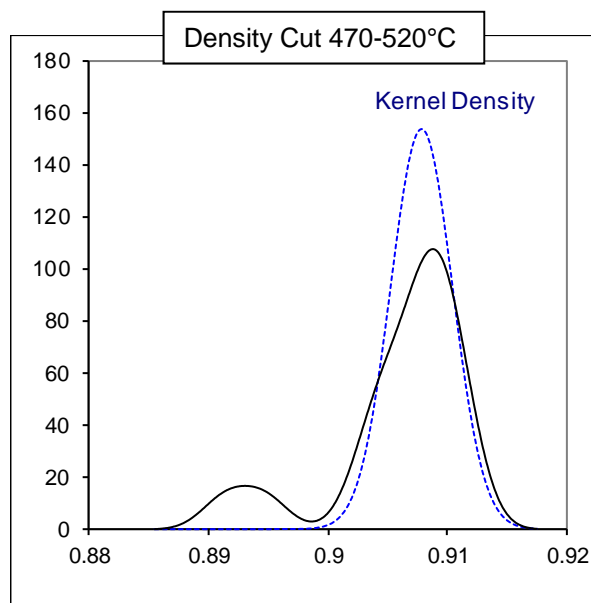
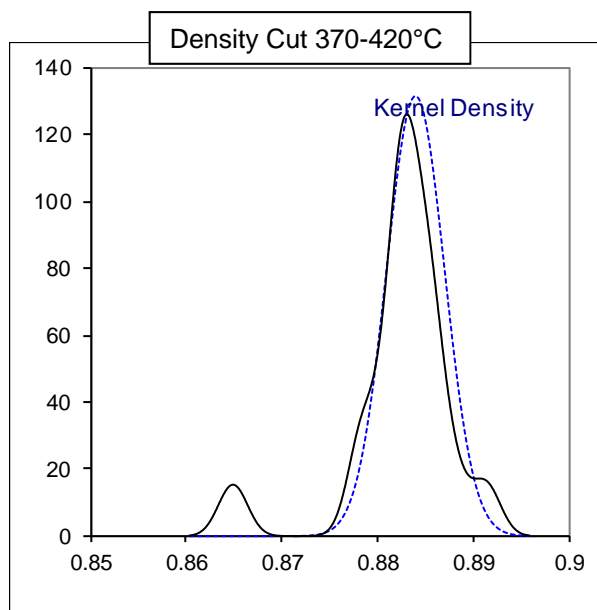
Lab 1066 first reported for Gas LPG: 0.5582 (fraction <-15°) and 0.6122 (fraction 15-30°C), iis calculated 0.5821



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

lab	Method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue >520°C	remarks on method used
171	D4052	0.8832	0.8938	0.9074	0.957	
311	D4052	0.8856	0.8937	0.9100	0.9542	
391	D4052	0.8914	0.8983	0.9116	0.9482	LPG: ISO8973
442		----	----	----	----	
445	D4052	0.8880	0.8965	0.9091	0.9519	LPG: GC + D4052 + calc.
574		----	----	----	----	
608		----	----	----	----	
862	ISO12185	0.8855	0.8904	0.9076	0.9495	LPG: D2598
971	D4052	0.8825	0.8911	0.9046	0.9526	
1026		<u>0.8649</u>	<u>0.8764</u>	<u>0.8914</u>	<u>0.9313</u>	LPG: EN-ISO8973
1065	D4052	0.8786	0.8904	0.9074	0.9487	
1066	D4052	0.8820	0.8940	0.9097	0.9514	LPG: D2598
1080		----	----	----	----	
1089		----	----	----	----	LPG: manually
1095	D4052	0.8828	0.8931	0.9092	0.9482	LPG: UOP539
1539	ISO12185	0.8853	0.8921	0.9030	0.9436	
1613	D4052	0.8855	0.8922	0.9050	0.9473	
1714	D4052	0.8826	0.8924	0.9045	0.9438	
1720	D4052	<i>0.8784</i>	<i>0.8859</i>	<u>0.8947</u>	<u>0.9333</u>	
1842	IP365	0.8834	0.8954	0.9112	0.9537	
1949	D4052	0.8810	0.8898	0.9079	0.9514	
1990	D4052	0.8830	0.8930	0.9098	0.9486	LPG: D2598
6156		----	----	----	----	
	normality	suspect	OK	OK	OK	
	n	15	15	15	15	
	outliers	1 (+1ex)	1 (+1ex)	2	2	
	mean (n)	0.8840	0.8931	0.9079	0.9500	
	st.dev. (n)	0.00304	0.00235	0.00260	0.00370	
	R(calc.)	0.0085	0.0066	0.0073	0.0104	
	RSD	0.3%	0.3%	0.3%	0.4%	

NB. Bold and underlined figures are statistical outliers (Grubbs)
 Bold and italic figures are excluded test results of lab 1720, since they were outlying/suspect results in the distillation D5236.



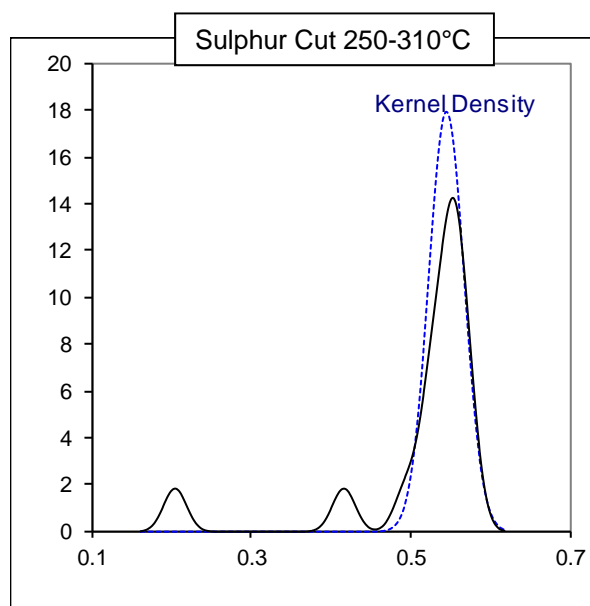
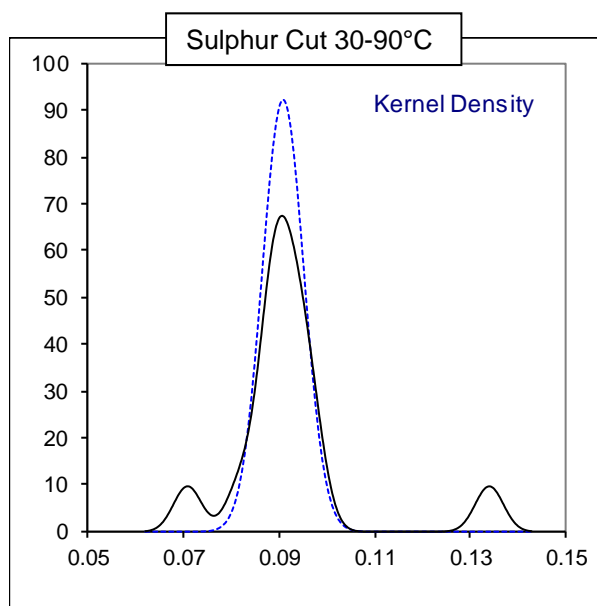
Determination of Sulphur on distillation fractions; results in %M/M

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	D4292	----	0.0881	0.209	0.204	0.256	0.525	1.11	1.23
311	D2622	0.0345	0.0977	0.219	0.199	0.241	0.575 C	1.18	1.28
391	ISO8754	----	0.087	0.22	0.23	0.26	0.56	1.04	1.35
442	IP336	----	----	----	----	----	----	----	1.227
445	IP336	0.015	0.071	0.21	0.20	0.27	0.56	1.16	1.24
574		----	----	----	----	----	----	----	----
608		----	----	----	----	----	----	----	----
862	D4294	----	0.134	0.182	0.196	0.252	0.495	1.085	1.169
971	D4294	----	0.095	0.190	0.208	0.269	0.557	0.970	1.210
1026	D2622	0.00005	0.09	0.20	0.21	0.24	0.53	1.12	1.25
1065	D4294	----	0.089	0.22	0.22	0.25	0.55	1.05	1.25
1066	D2622	0.0325	0.0933	0.228	0.208	0.247	0.417	1.104	1.229
1080		----	----	----	----	----	----	----	----
1089		----	----	----	----	----	----	----	----
1095		----	----	----	----	----	----	----	----
1539	ISO8754	----	0.092	0.23	0.23	0.26	0.55	1.12	----
1613	D4294	----	0.096	0.200	0.223	0.287	0.572	1.190	1.210
1714	D2622	----	0.082	0.218	0.222	0.259	0.523	1.11	1.19
1720	D4294	----	0.0919	0.2125	0.2098	0.2294	0.2056	0.0727	1.21
1842		----	----	----	----	----	----	----	----
1949	D4294	----	0.0893	0.2220	0.2152	0.2772	0.5425	1.113	1.239
1990	D4294	----	----	----	0.2051	0.2803	0.5513	1.0021	1.2239
6156		----	----	----	----	----	----	----	----
	normality	n.a.	OK	OK	OK	OK	OK	OK	OK
	n	4	12	14	15	15	13	14	14
	outliers	n.a.	2	0	0	0	2	1	1
	mean (n)	(0.0205)	0.0909	0.2115	0.2120	0.2585	0.5454	1.0967	1.2256
	st.dev. (n)	n.a.	0.00433	0.01407	0.01090	0.01611	0.02222	0.06321	0.02741
	R(calc.)	n.a.	0.0121	0.0394	0.0305	0.0451	0.0622	0.1770	0.0768
	st.dev.(D4294:16e1)	n.a.	(0.00553)	(0.00953)	(0.00955)	(0.01085)	(0.01756)	(0.02754)	(0.02958)
	R(D4294:16e1)	n.a.	(0.0155)	(0.0267)	(0.0267)	(0.0304)	(0.0492)	(0.0771)	(0.0828)
	RSD	n.a.	4.8%	6.7%	5.1%	6.2%	4.1%	5.8%	2.2%

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

Lab 1066 first reported for Gas LPG only fraction 15-30°C

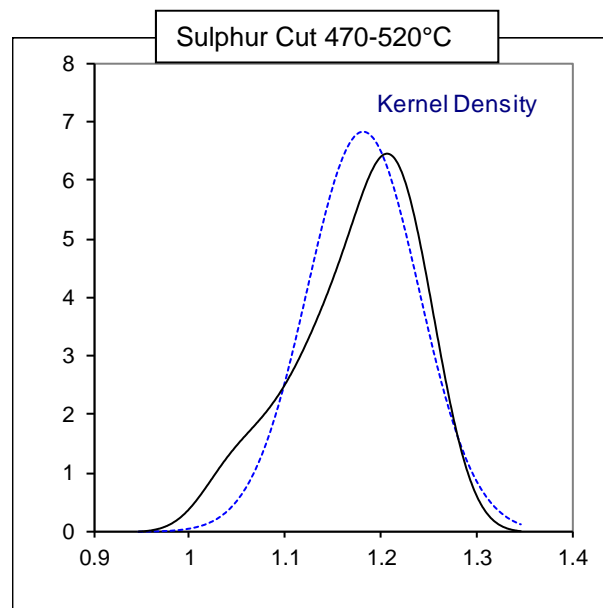
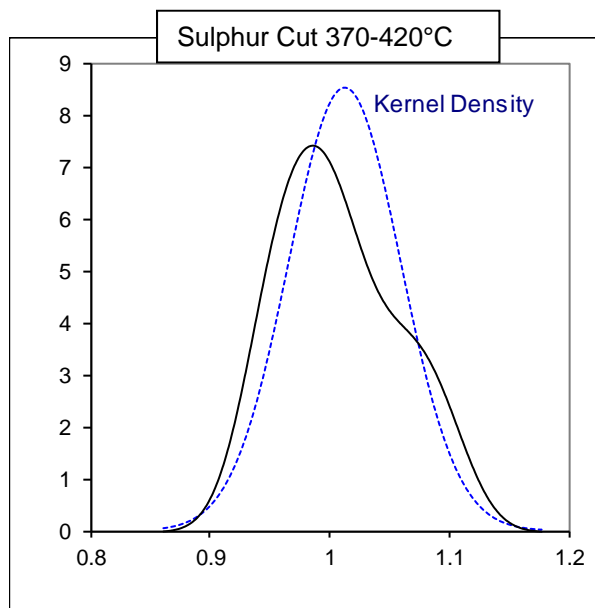
Lab 311 first reported for MGO: 0.966



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	Residue >520°C	remarks on method
171	D4294	1.01	1.09	1.19	1.48	LPG: D2622
311	D4294	1.05	1.09	1.25	1.58	LPG: D6667, Keros.: D5453, MGO/HGO/Res.: D4294
391		1.10	1.13	1.18	1.59	
442		----	----	----	----	
445	IP336	1.03	1.09	1.20	1.51	LPG/Naphta L: IP490
574		----	----	----	----	
608		----	----	----	----	
862	D4294	0.984	0.996	1.141	1.422	
971	D4294	1.003	1.021	1.140	1.482	
1026	D2622	0.97	1.07	1.22	1.57	LPG: D4045
1065	D4294	0.99	1.09	1.22	1.52	
1066	D2622	0.957	1.043	1.225	1.520	
1080		----	----	----	----	
1089		----	----	----	----	
1095		----	----	----	----	
1539	ISO8754	1.01	1.03	1.11	1.42	
1613	D4294	1.081	1.115	1.207	1.472	
1714	D2622	0.95	1.11	1.04	1.45	Naphtha L/Naphtha M: D5453
1720	D4294	0.938	0.9943	1.0807	1.3817	
1842		----	----	----	----	
1949	D4294	0.9746	1.055	1.253	1.511	
1990	D4294	1.0656	1.0442	1.1665	1.4087	
6156		----	----	----	----	
	normality	OK	OK	suspect	OK	
	n	14	14	14	14	
	outliers	0 (+1ex)	0 (+1ex)	0 (+1ex)	0 (+1ex)	
	mean (n)	1.0125	1.0696	1.1816	1.4954	
	st.dev. (n)	0.04684	0.03914	0.05847	0.05880	
	R(calc.)	0.1312	0.1096	0.1637	0.1647	
	st.dev.(D4294:16e1)	(0.02615)	(0.02710)	(0.02890)	(0.03363)	
	R(D4294:16e1)	(0.0732)	(0.0759)	(0.0809)	(0.0942)	
	RSD	4.6%	3.7%	4.9%	3.9%	

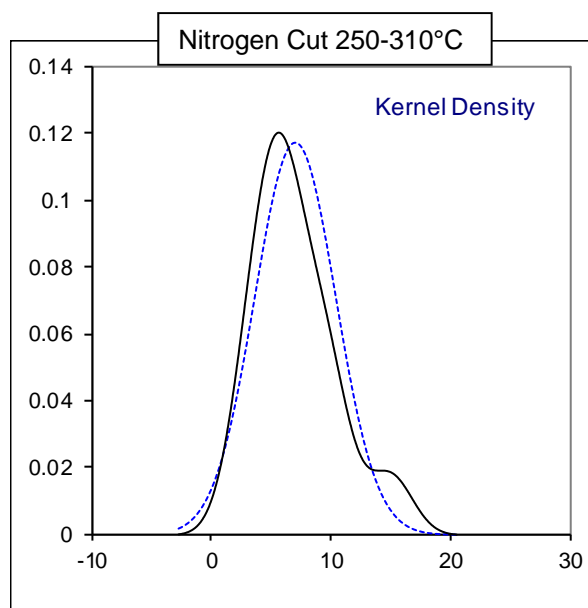
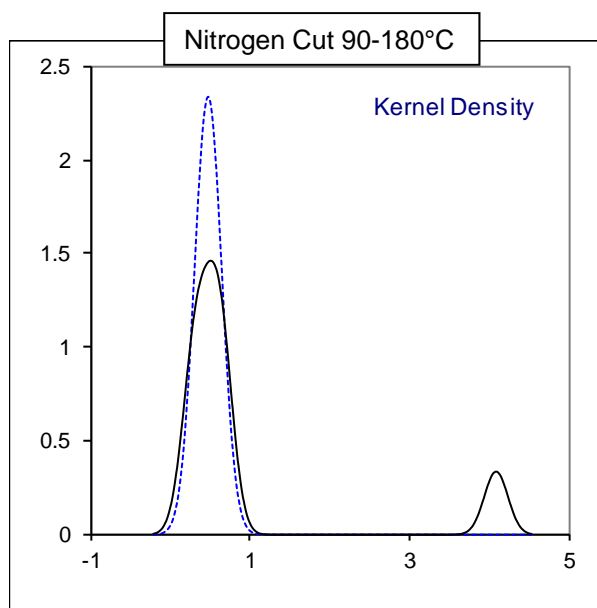
NB. Bold and italic figures are excluded test results of lab 1720, since they were outlying/suspect results in the distillation D5236.



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	----	0.5	0.7	0.7	0.7	4.9	180	920
311	D4629	3.2	<0.3	0.6	<u>3.4</u>	2.1	15	240	1100
391		----	----	----	----	----	----	----	----
442		----	----	----	----	----	----	----	----
445	D4629	<1	<1	<1	<1	<1	6	250	1300
574		----	----	----	----	----	----	----	----
608		----	----	----	----	----	----	----	----
862	D4629	----	0.7	0.5	1.5	1.4	7.0	188	1050
971	D4629	----	0.32	0.37	0.55	0.73	10.1	256	1023
1026	D4629	----	<0.3	<u>4.1</u>	<u>4.0</u>	<u>3.7</u>	9.9	169	990
1065		----	----	----	----	----	----	----	----
1066	D4629	<0.5	<0.5	<0.5	<0.5	<0.5	5.0	215	1120
1080		----	----	----	----	----	----	----	----
1089		----	----	----	----	----	----	----	----
1095		----	----	----	----	----	----	----	----
1539		----	----	----	----	----	----	----	----
1613		----	----	----	----	----	----	----	----
1714	D4629	----	<0.3	<0.3	0.43	0.70	5.2	204	830
1720	D4629	----	0.40	0.34	0.40	0.49	2.80	121.0	<u>2109.7</u>
1842	INH-12	----	<0.2	<0.2	<0.2	<0.2	3.4	173	1118
1949	D4629	----	0.57	0.63	0.66	0.74	8.44	286.1	943.9
1990	D4629	----	0.18	0.24	0.34	0.43	6.58	155.55	983.73
6156		----	----	----	----	----	----	----	----
	normality	n.a.	unknown	unknown	not OK	not OK	not OK	OK	suspect
	n	3	6	7	7	8	12	12	11
	outliers	n.a.	0	1	2	1	0	0	1
	mean (n)	n.a.	0.4450	0.4829	0.6543	0.9113	7.0267	203.14	1034.4
	st.dev. (n)	n.a.	0.18523	0.17075	0.39623	0.56220	3.40470	47.885	125.35
	R(calc.)	n.a.	0.5186	0.4781	1.1094	1.5742	9.5331	134.08	351.0
	st.dev.(D4629:17)	n.a.	(0.19052)	(0.19870)	(0.23235)	(0.27556)	(0.78885)	----	----
	R(D4629:17)	n.a.	(0.5335)	(0.5564)	(0.6506)	(0.7716)	(2.2088)	----	----
	st.dev.(D5762:17)	----	----	----	----	----	----	(19.298)	(98.27)
	R(D5762:17)	----	----	----	----	----	----	(54.03)	(275.2)
	RSD	n.a.	42%	35%	61%	62%	49%	24%	12%

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

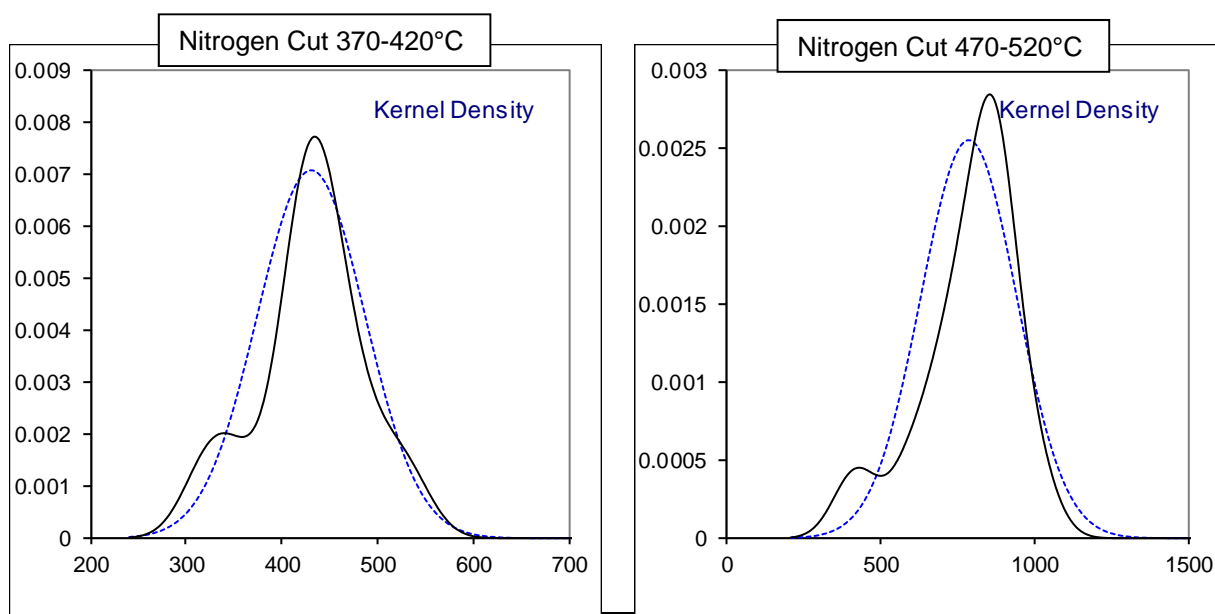


Determination of Nitrogen on distillation fractions (Continued); results in mg/kg

lab	method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue >520°C	remarks on method used
171	D5762	350	480	700	1600	HGO/Residue >370°C: D5762
311	D5762	430	540	840 C	2100 C	Residue >370°C: D5762
391		----	----	----	----	
442		----	----	----	----	
445	D5762	530	700	1000	2400	HGO/Residue >370°C: D5762
574		----	----	----	----	
608		----	----	----	----	
862	D5762	430	520	889	2020	Residue >370°C: D5762
971	D5762	411	542	781	1769	HGO/Residue >370°C: D5762
1026	D5762	410	530	600	1770	Residue >370°C: D5762
1065	D5762	440	638	733	----	
1066	D5762	455	585	870	2240	HGO/Residue >370°C: D5762
1080		----	----	----	----	
1089		----	----	----	----	
1095		----	----	----	----	
1539		----	----	----	----	
1613		----	----	----	----	
1714	D5762	320	610	420	1640	HGO/Residue >370°C: D5762
1720	D5762	<u>1417.66</u>	<u>1438.7</u>	<u>2241.1</u>	<u>2570.1</u>	Residue >370°C: D5762
1842	INH-12	431	580	832	2247	
1949	D5762	461.5	582.6	897.7	1610.0	HGO/Residue >370°C: D5762
1990	D5762	492.28	580.83	880.47	1851.97	HGO/Residue >370°C: D5762
6156		----	----	----	----	
	normality	OK	OK	suspect	OK	
	n	12	12	12	11	
	outliers	1	1	1	1	
	mean (n)	430.06	574.04	786.93	1931.6	
	st.dev. (n)	56.293	58.320	156.282	284.16	
	R(calc.)	157.62	163.30	437.59	795.6	
	st.dev.(D5762:17)	(40.856)	(54.533)	(74.758)	n.a.	
	R(D5762:17)	(114.40)	(152.69)	(209.32)	n.a.	
	RSD	13%	10%	20%	15%	

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

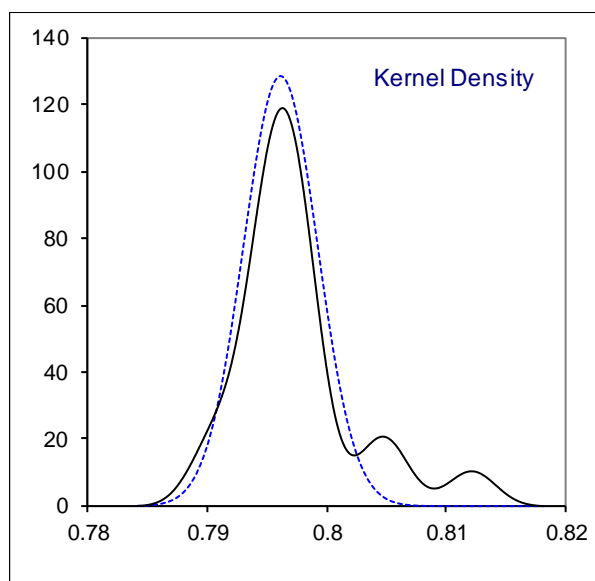
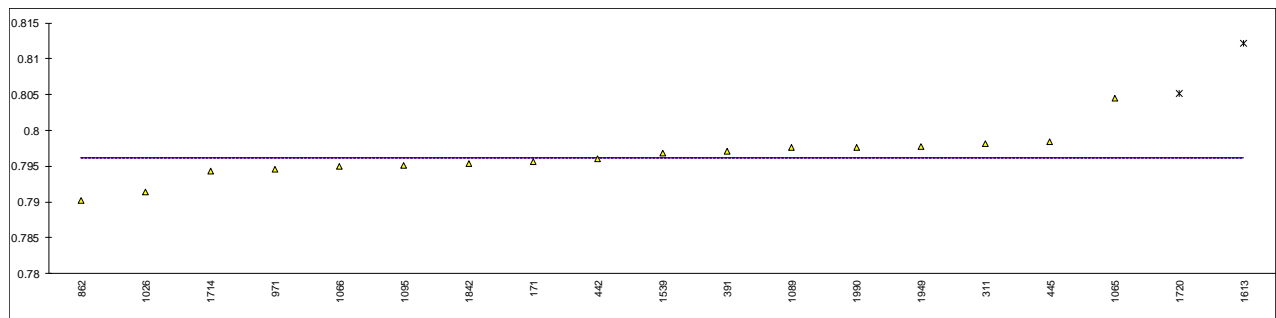
Lab 311 first reported for VGO: 2100 and for Residue >520°C: 840



Determination of calculated theoretical Density at 15°C of sample #17218; results in kg/L

lab	method	value	mark	z(targ)	Remarks	Meas. density	difference
171	calc. by iis	0.7957		----	*)	0.79914	-0.0035
311	calc. by iis	0.7981		----		0.7998	-0.0017
391	calc. by iis	0.7971		----		0.7988	-0.0017
442	calc. by iis	0.7961		----	*)	0.7987	-0.0026
445	calc. by iis	0.7984		----		0.7980	0.0004
574		----		----		----	----
608		----		----		----	----
862	calc. by iis	0.7902		----		0.79896	-0.0087
971	calc. by iis	0.7945		----		0.7987	-0.0042
1026	calc. by iis	0.7914		----		0.7979	-0.0065
1065	calc. by iis	0.8045		----	*)	0.8001	0.0044
1066	calc. by iis	0.7950		----		0.7977	-0.0027
1080		----		----		----	----
1089	calc. by iis	0.7976		----		0.7988	-0.0012
1095	calc. by iis	0.7951		----		0.7991	-0.0040
1539	calc. by iis	0.7968		----	*)	0.7989	-0.0021
1613	calc. by iis	0.8122	ex	----	no mass for cut 1 reported	0.7996	----
1714	calc. by iis	0.7944		----	*)	----	----
1720	calc. by iis	0.8052	ex	----	no mass for cut 1 reported	0.8006	----
1842	calc. by iis	0.7953		----	*)	0.7985	-0.0032
1949	calc. by iis	0.7977		----	*)	0.8005	-0.0028
1990	calc. by iis	0.7977		----		0.7987	-0.0010
6156		----		----		----	----
normality		not OK					
n		17					
outliers		0 (+2ex)					
mean (n)		0.79622		Average:		0.79903	-0.0026
st.dev. (n)		0.003097					
R(calc.)		0.00867					
st.dev. (D5002:16)		(0.001172)					
R(D5002:16)		(0.00328)					

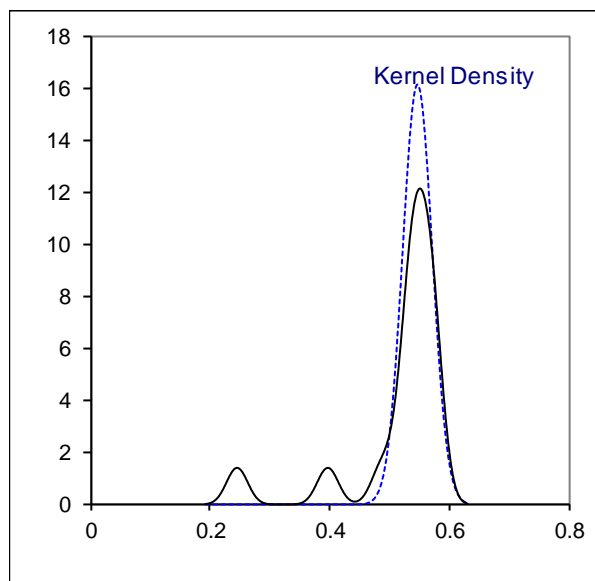
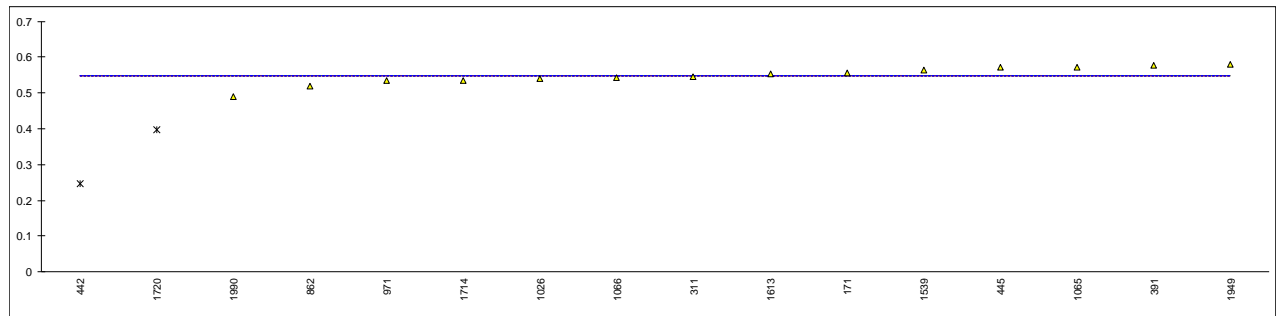
*) no density reported for fraction 1 (LPG), for calculation purposes the consensus value of the group (0.5837 kg/L) was used.



Determination of calculated theoretical sulphur content on sample #17218; results in %M/M

lab	method	value	mark	z(targ)	remarks	meas. sulphur	Δ absolute	Δ rel%
171	calc. by iis	0.5556		----		0.5767	-0.0211	-3.7
311	calc. by iis	0.5452		----		0.571	-0.0258	-4.5
391	calc. by iis	0.5763		----		0.588	-0.0117	-2.0
442	calc. by iis	0.2466	ex	----	not enough data	0.5865	----	
445	calc. by iis	0.5714		----		0.578	-0.0066	-1.1
574		----		----		----	----	
608		----		----		----	----	
862	calc. by iis	0.5201		----		0.591	-0.0709	-12.0
971	calc. by iis	0.5347		----		0.576	-0.0413	-7.2
1026	calc. by iis	0.5397		----		0.58	-0.0403	-6.9
1065	calc. by iis	0.5716		----		0.56	0.0116	2.1
1066	calc. by iis	0.5439		----		0.574	-0.0301	-5.2
1080		----		----		----	----	
1089		----		----		----	----	
1095		----		----		----	----	
1539	calc. by iis	0.5645		----		0.588	-0.0235	-4.0
1613	calc. by iis	0.5525		----		0.626 G(0.01)	-0.0735	-11.7
1714	calc. by iis	0.5353		----		----	----	
1720	calc. by iis	0.3984	G(0.01)	----		0.58	-0.1816	-31.3
1842		----		----		0.587	----	
1949	calc. by iis	0.5791		----		0.5809	-0.0018	-0.3
1990	calc. by iis	0.4886		----		0.5852	-0.0966	-16.5
6156		----		----		----	----	

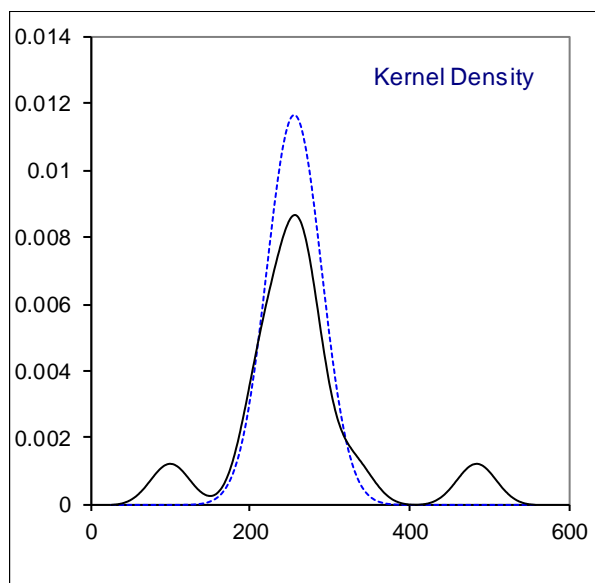
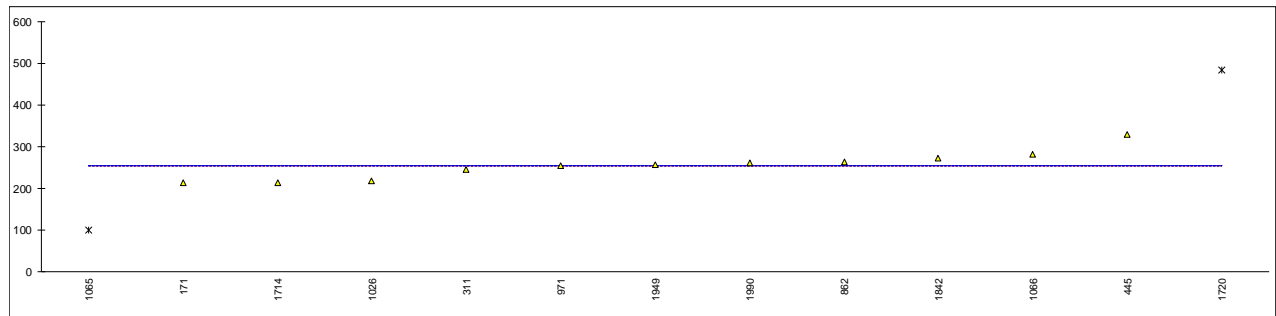
normality	suspect	
n	14	
outliers	1 (+1ex)	
mean (n)	0.5485	Average: 0.5802
st.dev. (n)	0.02473	
R(calc.)	0.0692	
st.dev.(D4294:16e1)	(0.01762)	
R(D4294:16e1)	(0.0493)	



Determination of calculated theoretical nitrogen content on sample #17218; results in mg/kg

lab	method	value	mark	z(targ)	remarks	meas. sulphur	Δ absolute	Δ rel%
171	calc. by iis	212.83		----		219.76	-6.9	-3.2
311	calc. by iis	245.17		----		263	-17.8	-6.8
391		----		----		----	----	----
442		----		----		----	----	----
445	calc. by iis	330.07		----		331.4	-1.3	-0.4
574		----		----		----	----	----
608		----		----		----	----	----
862	calc. by iis	263.64		----		249.2	14.4	5.8
971	calc. by iis	255.28		----		251	4.3	1.7
1026	calc. by iis	217.84		----		350	-132.2	-37.8
1065	calc. by iis	99.95	ex	----	not enough data	185	----	----
1066	calc. by iis	281.79		----		274.77	7.0	2.6
1080		----		----		----	----	----
1089		----		----		----	----	----
1095		----		----		----	----	----
1539		----		----		----	----	----
1613		----		----		----	----	----
1714	calc. by iis	213.36		----		----	----	----
1720	calc. by iis	484.15	G(0.01)	----		1519.42	-1035.3	-68.1
1842	calc. by iis	272.04		----		----	----	----
1949	calc. by iis	257.09		----		243.7	13.4	5.5
1990	calc. by iis	260.75		----		244.22	16.5	6.8
6156		----		----		----	----	----

normality suspect
 n 11
 outliers 1 (+1ex)
 mean (n) 255.44 Average: 261.21
 st.dev. (n) 34.269
 R(calc.) 95.95
 st.dev.(D4294:16e1) (24.267)
 R(D4294:16e1) (67.95)

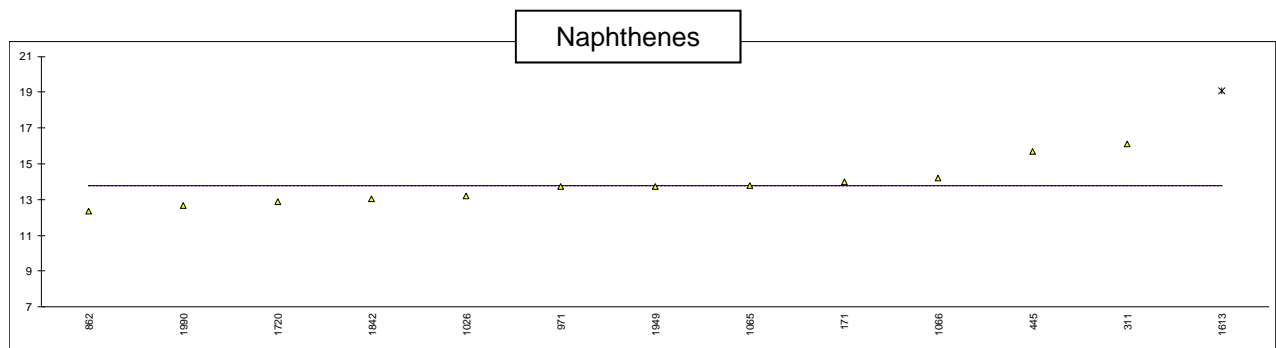
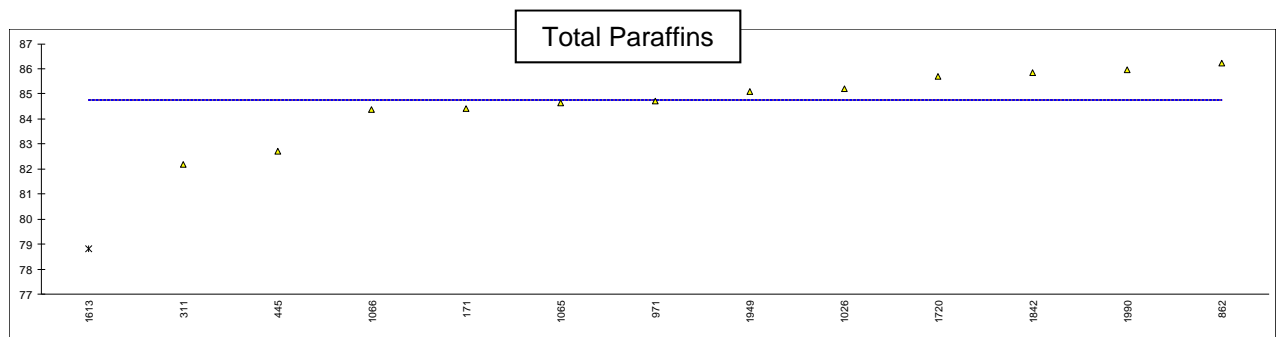


Determination of P(iP)NA on distillation fraction 2 (light naphtha, 30-90°C); results in %V/V

lab	method	total par.	C1-C4	n-par.	i-par.	naphth.	arom.	remarks
171	D6730M	84.426	0.261	41.931	42.495	14.002	1.481	
311	D5443	82.2	0.4 C	40.0	42.1	16.1 C	1.7	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D5443mod	82.7	0.3	42.0	40.8	15.7	1.6	
574		-----	-----	-----	-----	-----	-----	
608		-----	-----	-----	-----	-----	-----	
862	D6839	86.23	0.19	41.04	45.19	12.35	1.40	
971	D6730	84.71	0.15	42.89	41.82	13.71	1.55	
1026	EN-ISO22854	85.2	0.07	43.4	41.8	13.2	1.5	
1065	D5443	84.649	0.90	41.784	42.865	13.769	1.576	
1066	in house	84.37	0.27	43.36	41.01	14.19	1.44	
1080		-----	-----	-----	-----	-----	-----	
1089		-----	-----	-----	-----	-----	-----	
1095		-----	-----	-----	-----	-----	-----	
1539		-----	-----	-----	-----	-----	-----	
1613	D6839	<u>78.83</u>	-----	<u>35.86</u>	<u>42.97</u>	<u>19.08</u>	<u>2.01</u>	
1714		-----	-----	-----	-----	-----	-----	
1720	D5134	85.69	<u>3.84</u>	41.45	44.24	12.89	1.42	
1842	ISO22854	85.83	-----	40.91	44.92	13.04	1.13	
1949	D5443	85.10	1.19	-----	-----	13.75	1.14	
1990	D6839	85.95	0	40.72	45.23	12.69	1.35	
6156		-----	-----	-----	-----	-----	-----	
	normality	OK	not OK	OK	OK	suspect	OK	
	n	12	10	11	11	12	12	
	outliers	1	1	1	0 (+1ex)	1	0 (+1ex)	
	mean (n)	84.755	0.373	41.771	42.952	13.783	1.441	
	st.dev. (n)	1.2421	0.3784	1.1013	1.6638	1.1362	0.1720	
	R(calc.)	3.478	1.059	3.084	4.659	3.181	0.481	

NB. Bold and underlined figures are statistical outliers (Grubbs)
 Bold and italic figures are excluded test results of lab 1613, for three of five test results are outliers.

Lab 311 first reported for C1-C4: 16.1 and for Naphthenes: 0.3

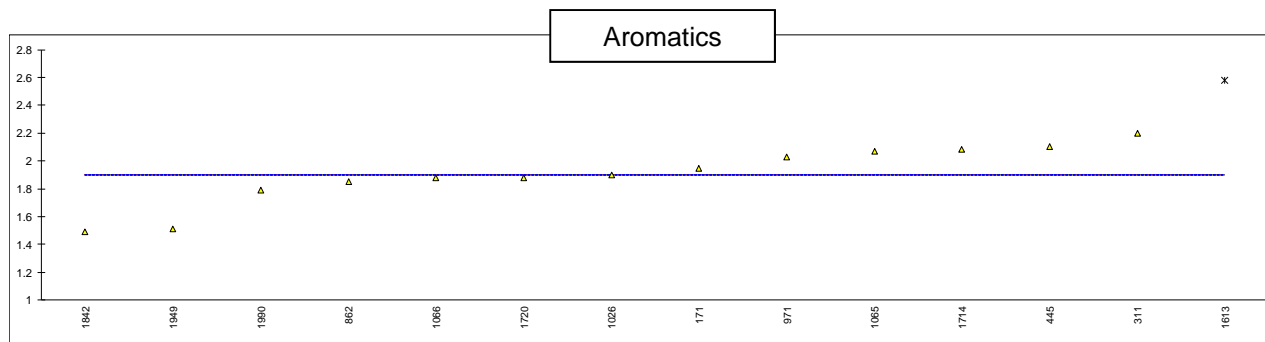
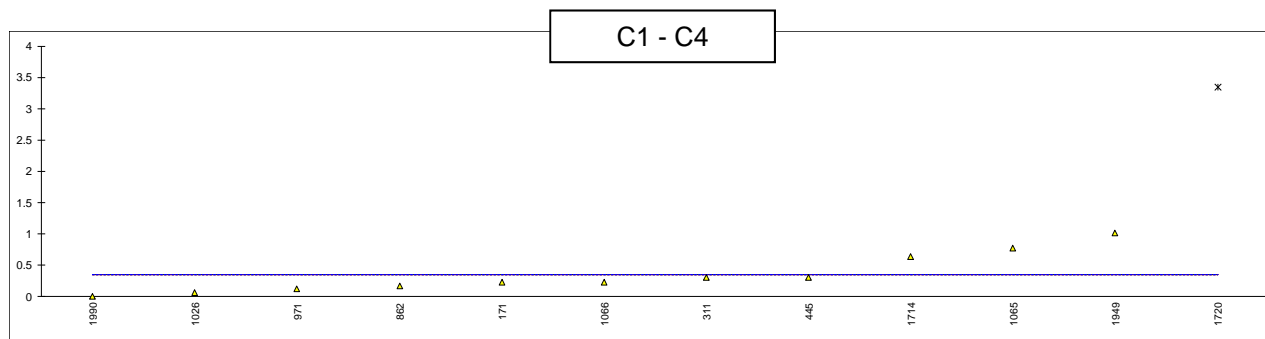


Determination of P(iP)NA on distillation fraction 2 (light naphtha, 30-90°C); results in %M/M

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D6730M	82.067	0.225	40.471	41.596	15.882	1.944	
311	D5443	79.7	0.3	38.2	41.1	18.1	2.2	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D5443mod	80.3	0.3	40.4	39.9	17.6	2.1	
574		-----	-----	-----	-----	-----	-----	
608		-----	-----	-----	-----	-----	-----	
862	D6839	84.07	0.17	39.76	44.31	14.07	1.85	
971	D6730	82.35	0.13	41.37	40.98	15.59	2.03	
1026	EN-ISO22854	83.06	0.06	40.4	42.6	15.0	1.9	
1065	D5443	82.295	0.78	40.371	41.924	15.632	2.067	
1066	in house	82.08	0.23	41.79	40.29	16.03	1.88	
1080		-----	-----	-----	-----	-----	-----	
1089		-----	-----	-----	-----	-----	-----	
1095		-----	-----	-----	-----	-----	-----	
1539		-----	-----	-----	-----	-----	-----	
1613	D6839	<u>76.19</u>	-----	<u>34.42</u>	<u>41.77</u>	<u>21.14</u>	<u>2.58</u>	
1714	D5443	81.91	0.64	39.70	42.21	16.01	2.08	
1720	D5134	83.44	<u>3.35</u>	40.06	43.38	14.68	1.88	
1842	ISO22854	83.54	-----	39.68	43.86	14.97	1.49	
1949	D5443	82.85	1.02	-----	-----	15.64	1.51	
1990	D6839	83.65	0	39.46	44.19	14.57	1.79	
6156		-----	-----	-----	-----	-----	-----	
	normality	OK	suspect	suspect	OK	OK	OK	
	n	13	11	12	12	13	13	
	outliers	1	1	1	0 (+1ex)	1	0 (+1ex)	
	mean (n)	82.409	0.350	40.139	42.195	15.675	1.902	
	st.dev. (n)	1.2760	0.3225	0.9203	1.5014	1.1408	0.2131	
	R(calc.)	3.573	0.903	2.577	4.204	3.194	0.597	

NB. Bold and underlined figures are statistical outliers (Grubbs)
 Bold and italic figures are excluded test results of lab 1613, for three out of five test results are outliers.

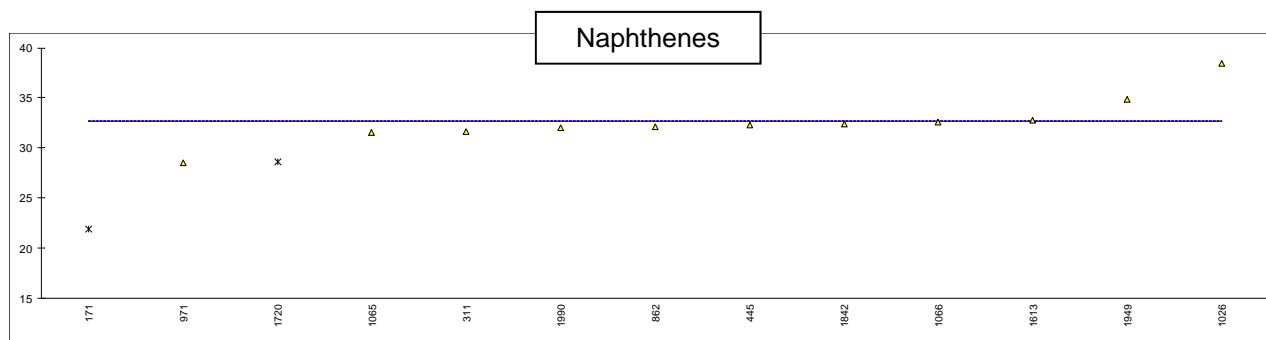
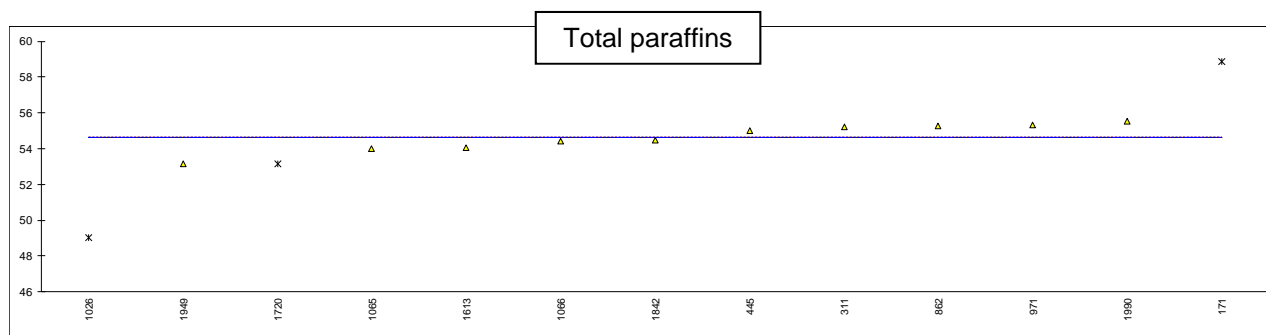
Lab 311 first reported for C1-C4: 0.4



Determination of P(iP)NA on distillation fraction 3 (heavy naphtha, 90-180°C); results in %V/V

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D6730M	58.872	0.003	25.379	33.493	21.904	14.488	Method not applicable
311	D5443	55.2	<0.1	25.1	29.8	31.6	13.2	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D5443mod	55.0	<0.1	26.4	28.7	32.3	12.7	
574		-----	-----	-----	-----	-----	-----	
608		-----	-----	-----	-----	-----	-----	
862	D6839	55.24	0.00	23.94	31.30	32.14	12.61	
971	D6730	55.33	<0.01	19.00	36.33	28.53	15.81	
1026	EN-ISO22854	49.0	-----	24.9	24.1	38.4	12.4	
1065	D5443	53.99	0.08	24.257	29.733	31.588	14.422	
1066	in house	54.41	<0.1	23.97	30.44	32.62	12.97	
1080		-----	-----	-----	-----	-----	-----	
1089		-----	-----	-----	-----	-----	-----	
1095		-----	-----	-----	-----	-----	-----	
1539		-----	-----	-----	-----	-----	-----	
1613	D6839	54.07	-----	17.10	36.97	32.82	13.11	
1714		-----	-----	-----	-----	-----	-----	
1720	D5134	53.17	0.0	25.72	30.45	28.58	15.25	
1842	ISO22854	54.45	-----	23.65	30.77	32.38	12.67	
1949	D5443	53.14	0.16	-----	-----	34.82	12.04	
1990	D6839	55.53	0	24.92	30.61	32.06	12.41	
6156		-----	-----	-----	-----	-----	-----	
	normality	OK	n.a.	OK	OK	not OK	not OK	
	n	10	9	9	11	11	12	
	outliers	2 (+1ex)	n.a.	2 (+1ex)	0 (+1ex)	1 (+1ex)	0 (+1ex)	
	mean (n)	54.636	<0.1	24.724	31.113	32.660	13.236	
	st.dev. (n)	0.7570	n.a.	0.8663	3.5592	2.4043	1.1078	
	R(calc.)	2.120	n.a.	2.426	9.966	6.732	3.102	

NB. Bold and underlined figures are statistical outliers (Grubbs)
 Bold and italic figures are excluded test results of lab 1720 for method D5134 is not applicable to for heavy naphtha (carbon atoms higher than C7).

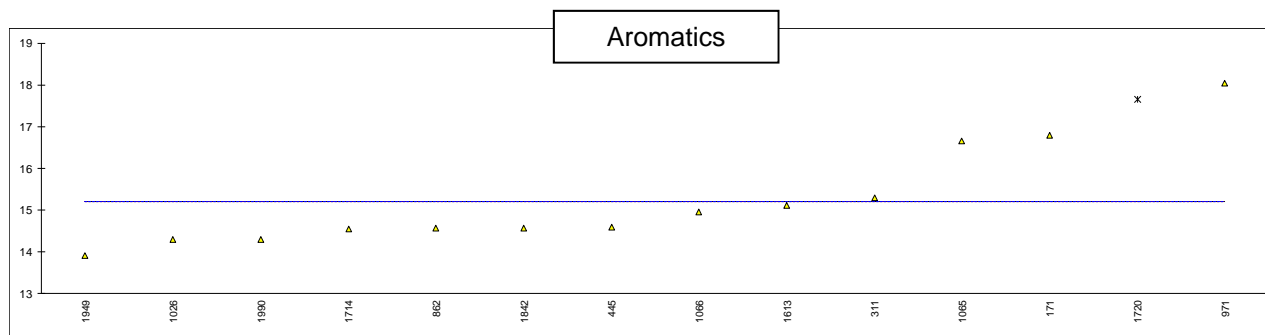
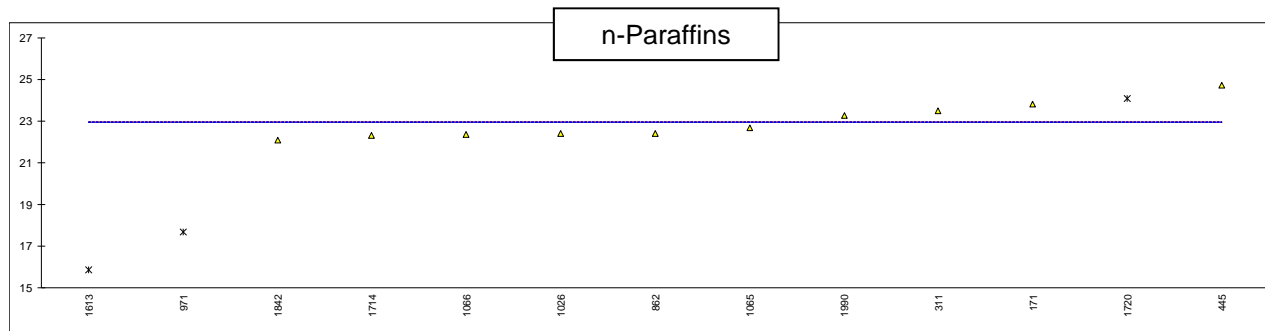


Determination of P(iP)NA on distillation fraction 3 (heavy naphtha, 90-180°C); results in %M/M

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	Remarks
171	D6730M	55.689	0.003	23.799	31.890	22.512	16.788	
311	D5443	52.0	<0.1	23.5	28.2	32.7	15.3	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445	D5443mod	51.8	<0.1	24.7	27.1	33.6	14.6	
574		----	----	----	----	----	----	
608		----	----	----	----	----	----	
862	D6839	52.09	0.00	22.41	29.68	33.35	14.56	
971	D6730	52.19	<0.01	17.69	34.50	29.47	18.03	
1026	EN-ISO22854	45.7	----	22.4	23.3	39.8	14.3	
1065	D5443	50.737	0.06	22.685	28.052	32.608	16.656	
1066	in house	51.25	<0.1	22.37	28.88	33.79	14.96	
1080		----	----	----	----	----	----	
1089		----	----	----	----	----	----	
1095		----	----	----	----	----	----	
1539		----	----	----	----	----	----	
1613	D6839	50.94	----	15.86	35.08	33.94	15.11	
1714	D5443	51.57	0.02	22.31	29.26	32.94	14.55	
1720	D5134	52.84	0.0	24.09	28.76	29.52	17.64	Method not applicable
1842	ISO22854	51.42	----	22.06	29.36	33.44	14.57	
1949	D5443	49.87	0.13	----	----	36.22	13.91	
1990	D6839	52.51	0	23.28	29.23	33.19	14.30	
6156		----	----	----	----	----	----	
	normality	OK	n.a.	suspect	OK	not OK	not OK	
	n	11	11	10	12	11	13	
	outliers	2 (+1ex)	n.a.	2 (+1ex)	0 (+1ex)	2 (+1ex)	0 (+1ex)	
	mean (n)	51.489	<0.1	22.951	29.544	33.204	15.203	
	st.dev. (n)	0.7622	n.a.	0.8431	3.1677	1.5760	1.2110	
	R(calc.)	2.134	n.a.	2.361	8.870	4.413	3.391	

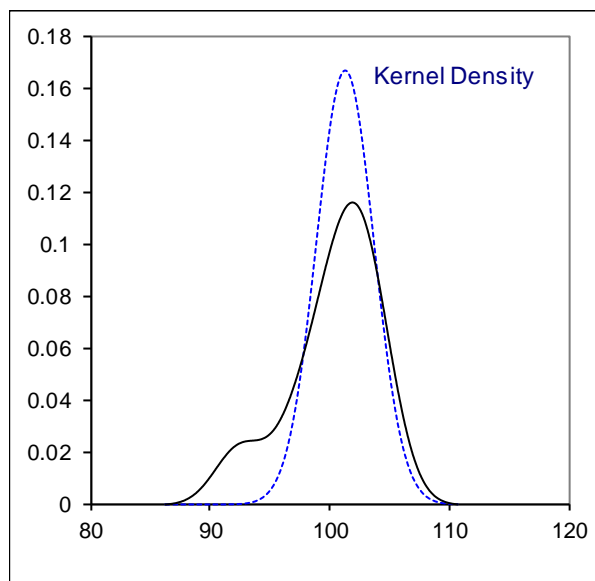
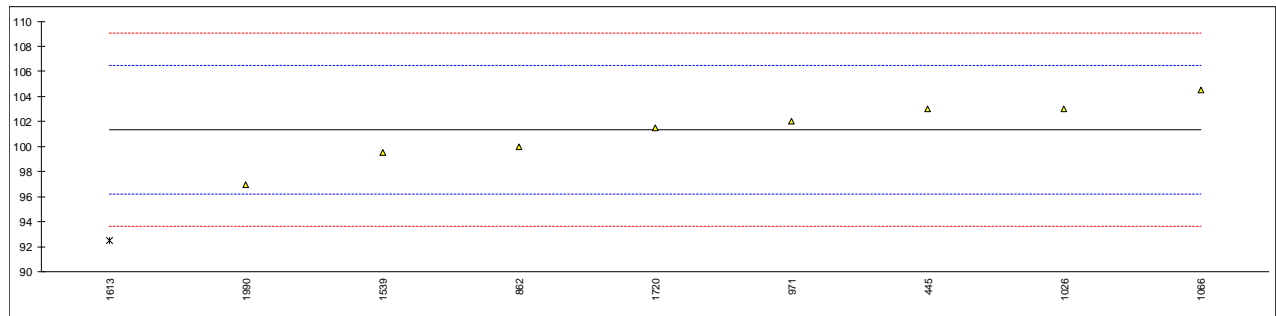
NB. Bold and underlined figures are statistical outliers (Grubbs)

Bold and italic figures are excluded test results of lab 1720 for method D5134 is not applicable to for heavy naphtha (carbon atoms higher than C7).



Determination of Flash point on combined distillation fractions 5 + 6, results in °C

lab	method	value	mark	z(targ)	remarks
171		----		----	
311		----		----	
391		----		----	
442		----		----	
445	D93-A	103.0		0.66	
574		----		----	
608		----		----	
862	D93-A	100.0		-0.51	
971	D93-A	102.0		0.27	
1026		103.0		0.66	
1065		----		----	
1066	D93-A	104.5		1.24	
1080		----		----	
1089		----		----	
1095		----		----	
1539	ISO2719	99.5		-0.71	
1613	D93-A	92.5	G(0.05)	-3.43	
1714		----		----	
1720	D93	101.5		0.07	
1842		----		----	
1949		----		----	
1990	D93-A	97		-1.68	
6156		----		----	
normality		OK			
n		8			
outliers		1			
mean (n)		101.31			
st.dev. (n)		2.390			
R(calc.)		6.69			
st.dev.(D93:16A-A)		2.569			
R(D93:16A-A)		7.19			



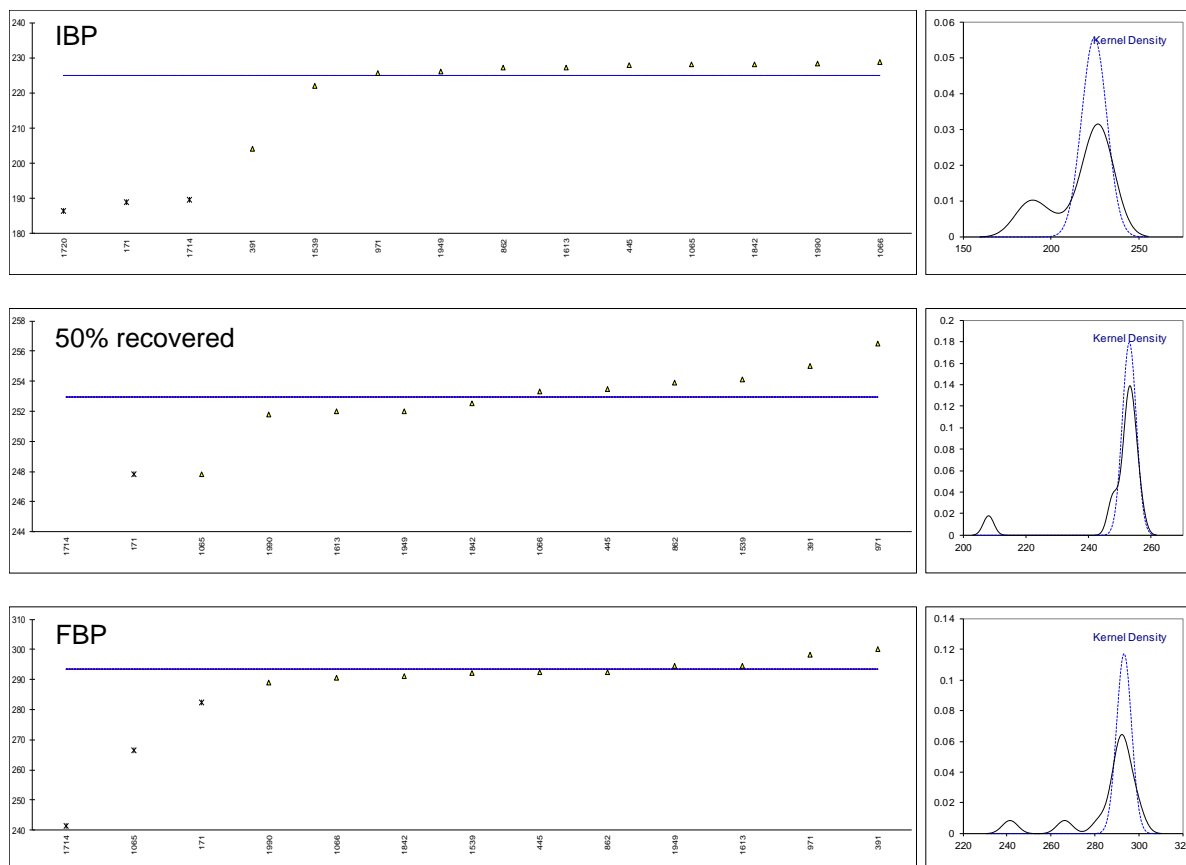
Determination of distillation (D86) on combined distillation fractions 5 + 6, results in °C

lab	method	IBP	5% rec.	10% rec.	50% rec.	90% rec.	95% rec.	FBP	%recov	%resid	%loss*
171	D86	188.9	232.2	233.3	247.8	274.4	281.1	282.3	98	2	0
311		----	----	----	----	----	----	----	----	----	----
391	D86	204	236	239	255	283	293	300	99	0.5	0.5
442		----	----	----	----	----	----	----	----	----	----
445	D86-Auto	227.9	237.5	239.2	253.5	281.6	288.2	292.3	98.2	1.3	0.5
574		----	----	----	----	----	----	----	----	----	----
608		----	----	----	----	----	----	----	----	----	----
862	D86	227.1	238.5	240.5	253.9	281.1	287.3	292.4	98.5	1.3	0.2
971	D86	225.6	241.2	243.4	256.5	284.5	290.4	298.1	99.2	0.5	0.3
1026		----	----	----	----	----	----	----	----	----	----
1065	D86	228.2	236.6	236.9	247.8	273.6	280.2	266.4	99.6	0.4	0
1066	D86	228.8	239.9	241.1	253.3	279.2	285.0	290.6	99.1	0.9	0
1080		----	----	----	----	----	----	----	----	----	----
1089		----	----	----	----	----	----	----	----	----	----
1095		----	----	----	----	----	----	----	----	----	----
1539	ISO3405	221.9	239.3	240.5	254.1	281.1	287.4	292.1	98.1	----	----
1613	D86	227.2	237.1	238.7	252.0	281.4	287.9	294.6	99.0	1.0	0
1714	D86	189.6	193.9	195.7	208.2	229.0	234.8	241.5	97.6	1.5	0.9
1720	D86	186.5	----	----	----	----	----	----	97.0	1.3	1.7
1842	D86	228.2	236.7	240.0	252.5	278.3	284.4	291.2	98.5	1.1	0.4
1949		226.0	237.0	242.0	252.0	278.5	285.5	294.5	98.5	1.3	0.2
1990	D86	228.4	238.1	239.6	251.8	276.5	282.6	289.0	99.4	1.3	-0.7
6156		----	----	----	----	----	----	----	----	----	----
	normality	not OK	OK	OK	not OK	OK	OK	OK			
	n	11	11	11	11	11	11	10			
	outliers	2 (+1ex)	1 (+1ex)	2	1 (+1ex)	1 (+1ex)	1 (+1ex)	2 (+1ex)			
	mean (n)	224.845	237.99	240.08	252.95	279.89	286.54	293.48			
	st.dev. (n)	7.182	1.597	1.741	2.221	3.083	3.554	3.402			
	R(calc.)	20.11	4.47	4.87	6.22	8.63	9.95	9.53			
	st.dev.(D86:17)	(4.417)	(2.550)	(1.886)	(1.071)	(1.499)	(2.148)	(2.536)			
	R(D86:17)	(12.37)	(7.14)	(5.28)	(3)	(4.20)	(6.02)	(7.1)			

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

Bold and italic figures are excluded test results of lab 171 and 1714 for two or more out of seven test results are outliers

*) loss calculated by iis (100% - recovered in % - residue in %), figures in bold indicated that there is no loss.



APPENDIX 1D – SIMULATED DISTILLATION AND EFFECTIVE CUT POINT D2892

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

lab	method	210-220°C	220-230°C	230-240°C	240-250°C	250-260°C	260-270°C	270-280°C
171	D2887	12.4	19.8	27.3	22.1	12.3	1.3	0.0
311	D2887	9.7	15.0	26.5	22.5	19.5	4.5	0.0
391		----	----	----	----	----	----	----
442		----	----	----	----	----	----	----
445	D2887	11.8	14.5	26.0	17.7	19.3	5.0	0.0
574		----	----	----	----	----	----	----
608		----	----	----	----	----	----	----
862		----	----	----	----	----	----	----
971		----	----	----	----	----	----	----
1026	D2887	19.7	20.3	29.1	15.5	9.3	0.8	0.0
1065		12.9	20.0	22.5	23.3	13.3	2.2	0.8
1066	D2887	18.7	21.5	33.5	14.0	8.0	0.0	0.0
1080		----	----	----	----	----	----	----
1089		----	----	----	----	----	----	----
1095		----	----	----	----	----	----	----
1539		----	----	----	----	----	----	----
1613		----	----	----	----	----	----	----
1714		12.4	14.8	28.8	16.4	16.1	3.6	0.3
1720		----	----	----	----	----	----	----
1842		18.0	19.5	30.5	15.0	10.9	1.1	0.0
1949		----	----	----	----	----	----	----
1990		8.7	17.9	21.1	26.2	17.8	4.8	0.0
6156		----	----	----	----	----	----	----
	normality	OK	OK	OK	OK	OK	OK	n.a.
	n	9	9	9	9	9	9	9
	outliers	0	0	0	0	0	0	0
	mean (n)	13.80	18.15	27.26	19.17	14.05	2.59	<1
	st.dev. (n)	4.005	2.697	3.837	4.378	4.323	1.899	n.a.
	R(calc.)	11.22	7.55	10.74	12.26	12.10	5.32	n.a.

Results of Simdist on distillation fraction 6 (MGO, 250-310°C); yields of fractions in %M/M

lab	230-240°C	240-250°C	250-260°C	260-270°C	270-280°C	280-290°C	290-300°C	300-310°C
171	2.0	4.4	13.6	13.4	18.7	15.8	15.1	12.6
311	0.7	2.3	8.7	10.3	15.0	14.0	12.5	14.5
391	----	----	----	----	----	----	----	----
442	----	----	----	----	----	----	----	----
445	1.9	2.5	9.2	11.6	18.5	15.5	14.0	17.3
574	----	----	----	----	----	----	----	----
608	----	----	----	----	----	----	----	----
862	----	----	----	----	----	----	----	----
971	----	----	----	----	----	----	----	----
1026	1.3	4.4	13.9	14.4	17.9	16.8	12.6	13.2
1065	2.1	6.0	13.5	14.8	19.5	14.8	16.9	9.5
1066	0.9	5.1	15.5	15.5	17.5	16.5	13.0	12.7
1080	----	----	----	----	----	----	----	----
1089	----	----	----	----	----	----	----	----
1095	----	----	----	----	----	----	----	----
1539	----	----	----	----	----	----	----	----
1613	----	----	----	----	----	----	----	----
1714	1.9	2.3	5.4	9.6	11.2	16.3	13.7	15.4
1720	----	----	----	----	----	----	----	----
1842	1.7	4.3	14.0	15.0	19.0	17.5	12.5	13.0
1949	----	----	----	----	----	----	----	----
1990	0.7	2.3	10.6	12.5	22.0	15.2	15.8	12.6
6156	----	----	----	----	----	----	----	----
norm.	OK	OK	OK	OK	not OK	OK	OK	suspect
n	9	9	9	9	9	9	9	9
outliers	0	0	0	0	0	0	0	0
mean	1.46	3.74	11.59	13.02	17.70	15.81	14.01	13.41
st.dev.	0.567	1.415	3.303	2.128	3.068	1.072	1.595	2.167
R(calc)	1.59	3.96	9.25	5.96	8.59	3.00	4.47	6.07

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

Lab	Overlap cuts 5 and 6 in °C	ECP in °C	Difference with AET=250 °C	Conclusion*	Remarks
171	229-264°C = 35°C	250.0	0.0	OK	
311	236-271°C = 35°C	256.9	6.9	Not OK	
391	-----	-----	-----	-----	
442	-----	-----	-----	-----	
445	229-269°C = 40°C	255.6	5.6	OK	
574	-----	-----	-----	-----	
608	-----	-----	-----	-----	
862	-----	-----	-----	-----	
971	-----	-----	-----	-----	
1026	230-262°C = 32°C	248.1	-1.9	OK	
1065	225-275°C = 50°C	249.1	-0.9	OK	
1066	237-260°C = 23°C	249.0	-1.0	OK	narrow overlap of cuts
1080	-----	-----	-----	-----	
1089	-----	-----	-----	-----	
1095	-----	-----	-----	-----	
1539	-----	-----	-----	-----	
1613	-----	-----	-----	-----	
1714	228-271°C = 43°C	253.3	3.3	OK	
1720	-----	-----	-----	-----	
1842	233-264°C = 31°C	249.3	-0.7	OK	
1949	-----	-----	-----	-----	
1990	236-266°C = 30°C	255.9	5.9	OK	
6156	-----	-----	-----	-----	

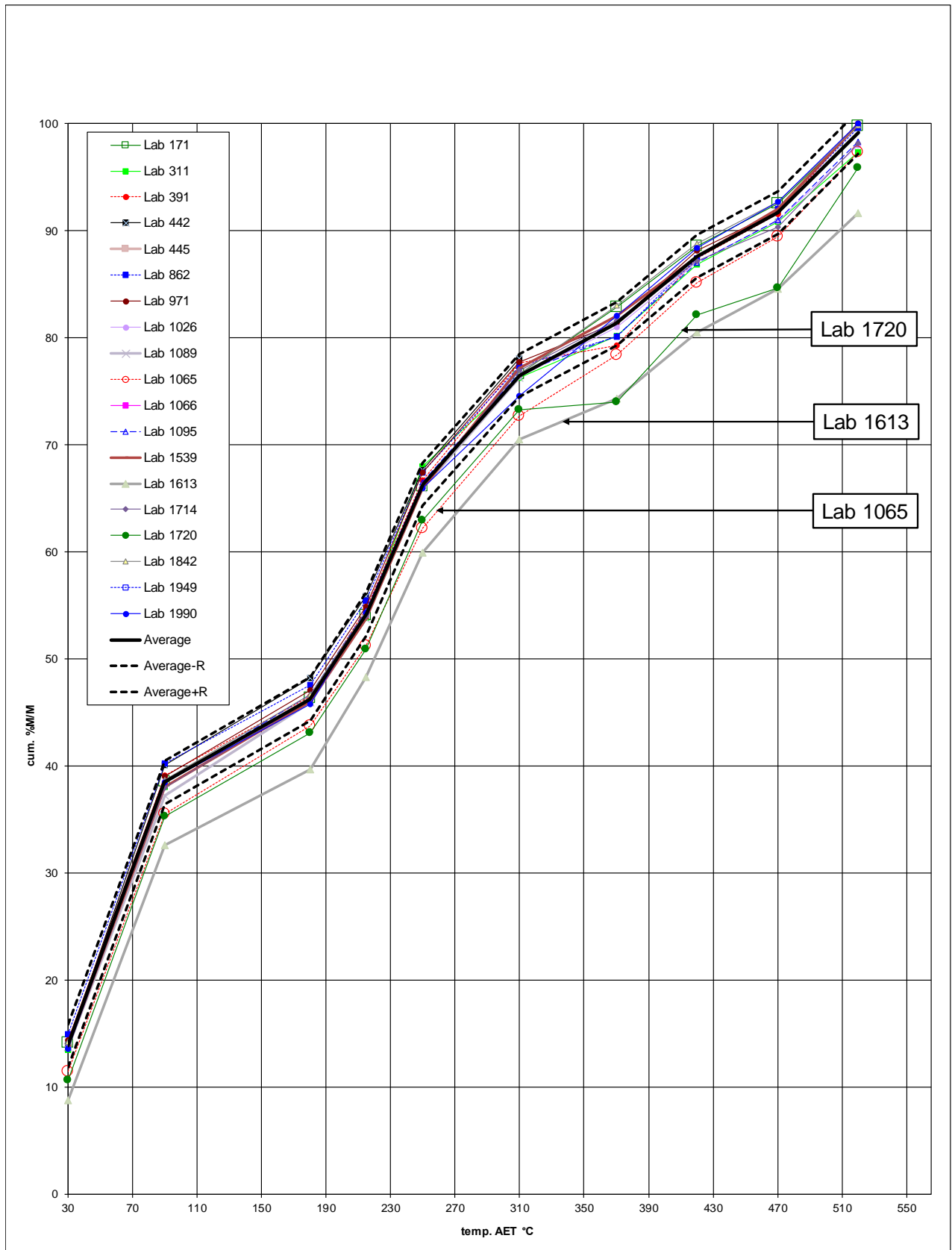
* Acc. To ASTM D2892:17a, Appendix X2.6.5.1 the difference between ECP and AET should not exceed $0.7R$ °C ($0.7 \times 8 = 6$ °C)

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

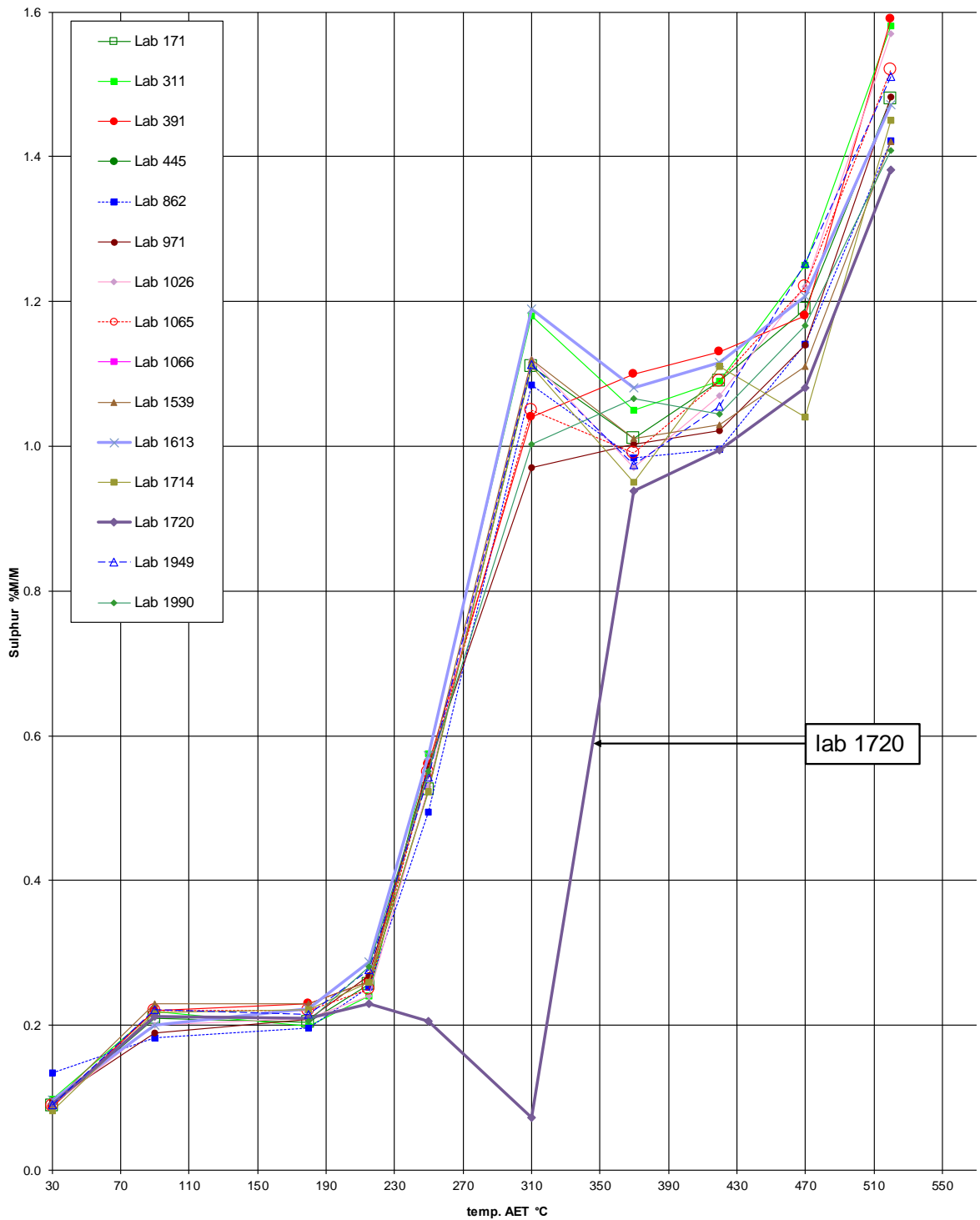
Lab	N_{actual}	N_{minimum}	Requirement 5.9 < N_{minimum} < 7.3*	Remarks
171	8.6	7.4	Not OK	
311	7.3	-----	-----	Observed Vapor Temperature not reported
391	-----	-----	-----	
442	-----	-----	-----	
445	5.9	4.4	Not OK	
574	-----	-----	-----	
608	-----	-----	-----	
862	-----	-----	-----	
971	-----	-----	-----	
1026	8.5	7.3	OK	
1065	6.7	5.8	Not OK	
1066	10.9	9.3	Not OK	
1080	-----	-----	-----	
1089	-----	-----	-----	
1095	-----	-----	-----	
1539	-----	-----	-----	
1613	-----	-----	-----	
1714	7.8	6.7	OK	
1720	-----	-----	-----	
1842	8.9	7.7	Not OK	
1949	-----	-----	-----	
1990	7.5	6.4	OK	
6156	-----	-----	-----	

* Acc. To ASTM D2892:17a, Appendix X2.5.9.2 the acceptable upper and lower limits (14-18 theoretical plates) are 5.9, resp. 7.3

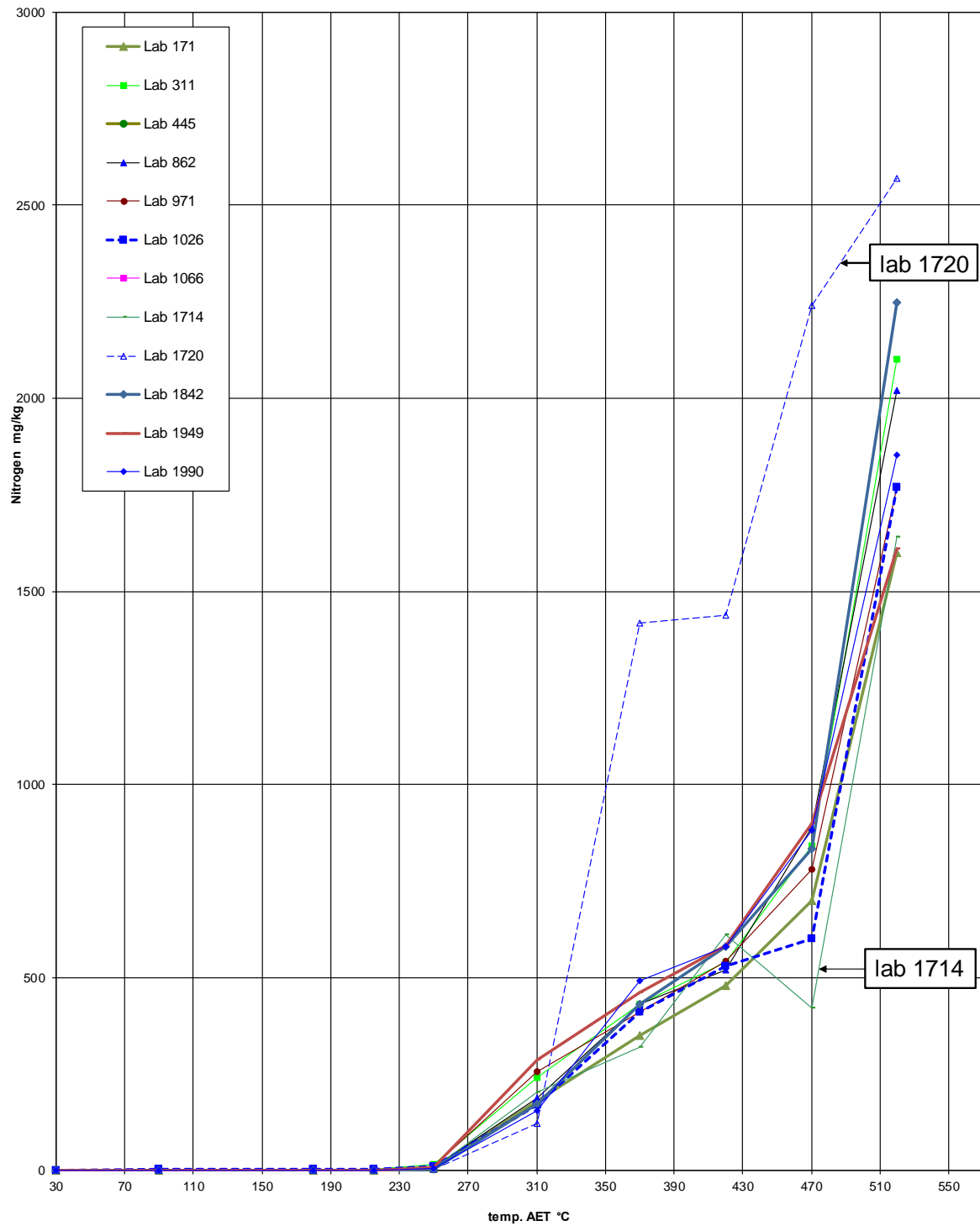
APPENDIX 2 True boiling point curve, cumulative %M/M vs temperature AET in °C



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



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



APPENDIX 5**Details of Distillation**

<u>Lab</u>	<u>Sample received</u>	<u>Distillation started</u>	<u>Results reported</u>	<u>Intake in L</u>	<u>Pressure at start of dist. in mm Hg</u>	<u>End Point of distillation(s) in °C</u>
171	27-Oct-17	01-Nov-17	28-Nov-17	12.4	757	520+
311	27-Oct-17	06-Dec-17	21-Feb-18	10	772	520+
391	27-Oct-17	06-Nov-17	24-Nov-17	10	758.3	520+
442	15-Jan-18	16-Jan-18	29-Jan-18	22	760	370+
445	01-Nov-17	21-Nov-17	15-Dec-17	40	753	520+
574	-----	-----	-----	-----	-----	-----
608	-----	-----	-----	-----	-----	-----
862	-----	-----	28-12-17	8	760	520+
971	28-10-17	12-11-17	24-11-17	22	760	520+
1026	21-12-17	28-02-18	29-03-18	20	760	520+
1065	05-11-17	07-11-17	23-11-17	10	743	520+
1066	24-10-17	06-11-17	06-12-17	10	763	520+
1080	-----	-----	-----	-----	-----	-----
1089	13-11-17	16-11-17	23-11-17	6	749.6	370+
1095	09-11-17	22-11-17	19-12-17	10	758	520+
1539	26-10-17	20-11-17	24-11-17	6	750	520+
1613	29-11-17	19-10-17	06-12-17	6.0	760	520+
1714	26-10-17	31-10-17	23-11-17	5	760	520+
1720	16-11-17	19-11-17	24-11-17	20	718	520+
1842	26-10-17	09-11-17	24-11-17	10	767	520+
1949	13-11-17	14-11-17	07-12-17	10	736	520+
1990	01-11-17	03-11-17	27-11-17	20	760	520+
6156	-----	-----	-----	-----	-----	-----

APPENDIX 6

Number of participants per country

1 lab in CHINA, People's Republic
1 lab in COLOMBIA
1 lab in HUNGARY
1 lab in ISRAEL
1 lab in ITALY
1 lab in JORDAN
3 labs in MALAYSIA
4 labs in NETHERLANDS
1 lab in NORWAY
1 lab in POLAND
1 lab in PORTUGAL
1 lab in RUSSIAN FEDERATION
1 lab in SUDAN
1 lab in UNITED ARAB EMIRATES
3 labs in UNITED KINGDOM
1 lab in UNITED STATES OF AMERICA

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
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
E	= probably error in calculations
W	= result withdrawn on request of participant
ex	= test result excluded from calculations
n.a.	= not applicable
n.d.	= not detected
fr.	= first reported
SDS	= safety data sheet
n.r.	= not relevant

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
- 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/>).
- 14 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), pp. 165-172, (1983)