Results of Proficiency Test
Crude Oil Assay
November 2019

SUMMARY OF CHANGES

This revised report replaces the original report iis19R02 of May 2020.

Because of a participant's question iis discovered that the calculation of Effective Cut Point (ECP) and Nminimum in appendix 1D had to be revised. These parameters are calculated from the SimDist data of two overlapping fractions by calculating yields of fixed temperature intervals. The interval chosen by iis was ten degrees Celsius, which is in line with method ASTM D2892. The method describes in paragraph X2.4.2 to take intervals between five and fifteen degrees. ECP and Nminimum are calculated from a line based on data from these intervals. The participant pointed out that when calculating with intervals of 5 degrees this line would have more datapoints and therefore be a better estimate of the slope and intersection of the line to calculate ECP and Nminimum. After recalculation with 5 degrees intervals by iis, this was confirmed. Therefore a revised report was made.

In this revised report, only the conclusions and tables of ECP and Nminimum were revised, respectively in paragraph 4.1D and appendix 1D.

The following pages in this report have been revised:

- Paragraph 4.1D page 13 and 14 (page 12 and 13 in the orginal report)
- Appendix 1D: page 24 (page 23 in the original report)
- Page numbering was changed, due to the addition of this page. This affected the Content on page 3 (page 2 in the original report).

CONTENTS

1   INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies organizes a proficiency scheme for Crude Oil Assay every two years. During the annual proficiency test program of 2019/2020, it was decided to continue the proficiency test for the Assay analyzes of Crude Oil.

In this interlaboratory study 24 laboratories in 16 different countries registered for participation. See appendix 6 for the number of participants per country.
In this report the results of this 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 analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send subsamples in 5 L cans, labelled #19225. 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/IEC17043:2010 (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 organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is 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

A batch of approximately 300 liters of Crude Oil was obtained from a local crude oil storage facility. After homogenization 68 metal cans of 5 L were filled and labelled #19225.
The homogeneity of the subsamples #19225 was checked by determination of Density at 15°C in accordance with ASTM D5002 on 8 stratified randomly selected subsamples.

	Density at 15 °C in kg/m^3
sample #19225-1	872.88
sample #19225-2	872.95
sample #19225-3	872.99
sample #19225-4	872.94
sample #19225-5	873.09
sample #19225-6	872.93
sample #19225-7	873.18
sample #19225-8	873.07

Table 1: homogeneity test results of subsamples #19225

From the above test results of the homogeneity tests, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15 °C in kg/m^3
r (observed)	0.28
reference test method	ASTM D5002:18e1
0.3 * R (ref. test method)	1.08

Table 2: evaluation of the repeatability on subsamples #19225

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 30, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Crude Oil packed in glass bottles with a red plastic bag was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine on the original sample Density, Sulfur, Nitrogen and Water. The participants were further 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). Also was requested to determine Density, Sulfur and Nitrogen on all fractions where possible.
Furthermore, it was requested to determine:
- on the light and heavy naphtha fractions (fractions 2 and 3) a PNA or PIONA analysis
- on the combined fractions of light and medium gasoil (fractions 5 and 6) Flash Point PMCC and a distillation
- on the separate fractions of light Gasoil (fraction 5) and medium Gasoil (fraction 6) a simulated distillation determination (on both fractions).

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

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form (as a word file) 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 sixteen weeks after sample dispatch the test results of the individual laboratories were gathered by e-mail. 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. After the deadline the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the 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 organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

According to ISO5725 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. In this PT, the criterion of ISO13528, paragraph 9.2.1 was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2　GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. 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:

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

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

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

$$|z| \quad < 1 \text{ good}$$
$$1 < \quad |z| \quad < 2 \text{ satisfactory}$$
$$2 < \quad |z| \quad < 3 \text{ questionable}$$
$$3 < \quad |z| \qquad \text{unsatisfactory}$$

4 EVALUATION

In the proficiency test no major problems were encountered with sample dispatch. After the period of five weeks from sample dispatch only eight participants had reported test results. Nine laboratories reported test results after the final reporting date, the last test results were submitted by the end of January 2020. Seven laboratories did not report any test results at all. Evaluation of test results and preparation of the final report were delayed by these late reported test results or waiting for the not reported test results. Not all laboratories were able to report all analyzes requested.

In total 17 participants reported 184 numerical test results for the distillation fractions and 857 numerical test results for the determinations on the fractions. Observed were 4 outliers for the distillation test results and 31 outliers for the determinations, which is respectively 2.2% and 3.6% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the table together with the original data. The abbreviations, used in these tables, are listed in appendix 7.

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.

For objective evaluation iis uses reproducibilities taken from reference test methods to calculate target z-scores. Regretfully this is not possible for the Density, Sulfur and Nitrogen test results from the fractions. 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. Calculated (theoretical) Density, Sulfur and Nitrogen of the original Crude Oil sample were calculated by iis from the masses of the collected fractions and the respective reported Density, Sulfur and Nitrogen test results using below formulae. These calculated (theoretical) values were compared with the measured values as reported by the participants.

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

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

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

4.1A - Analysis of the original sample (appendix 1A)

Density: The Density determination on the original Crude Oil sample was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D5002:19.

Sulfur: The Sulfur determination on the original Crude Oil sample was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D4294:16e1.

Nitrogen: The Nitrogen determination on the original Crude Oil sample was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D5762:18a.

Water: The water determination on the original Crude Oil sample was problematic at the low level of 0.03%V/V. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D4377:00(2011). This method was withdrawn by ASTM in January 2020, because it was not reapproved within eight years.

4.1B - Distillation test results D2892 and D5236 (appendix 1B)

In July 2018 a new version of ASTM D5236 and in November 2019 a new version of ASTM D2892 was published. This PT was dispatched at the end of October 2019. It was decided to use the version of 2019 for ASTM D2892 already because the reproducibilities were not affected compared to the earlier version.

In total, seventeen laboratories reported distillation test results. Five laboratories only performed the atmospheric distillation (D2892) and twelve also the vacuum distillation (D5236). The details of the distillation(s) reported by the participants can be found in appendix 1B and 5.

Looking at the pressure at the start of the atmospheric distillation (D2892), no participant reported to have used a starting pressure below atmospheric. ASTM D2892 states that it is not allowed to let the Observed Boiling Temperature of the liquid exceed 310°C or the Observed Vapor Temperature exceed 210°C (paragraph 10.3.7 of ASTM D2892). According to paragraphs 10.3 till 10.5 of the same method this can be prevented by lowering the pressure in the system. The method also states that after atmospheric pressure a pressure around 100 mmHg should be applied and if necessary or applicable also a lower pressure of between 100 and 2 mmHg is permitted in only one step. Laboratory 445 applied a pressure of 10 mmHg after the atmospheric pressure (fraction 4/5), skipping the pressure step of 100 mmHg. Two laboratories (608 and 1613) still applied a pressure of 100 mmHg for fraction 6, while the Observed Vapor Temperature was above 210°C. And laboratory 1065 applied a pressure for fraction 6 and 7 of 1.00 mmHg, which is not permitted according to ASTM D2892. These differences in temperature and/or pressure does not appear to have a significant effect on the reported test results of the fractions of the atmospheric distillation.

However, it appears to have an effect on the calculation of the Standard Efficiency $N_{minimum}$, calculated from the SimDist data from fraction 5 and 6.

For ASTM D5236, all participants started the distillation at a pressure between 0.1 and 10 mmHg and no participant used a pressure lower than 0.1 mmHg, which is not permitted according to ASTM D5236.

The evaluation of the Total Mass balance showed only five (!) recoveries that do meet the ASTM D2892 (paragraph 11.2) requirement of 0.4% max loss and the ASTM D5236 requirement of a recovery between 99.6 and 100.1%. Remarkably, four recoveries were equal to 100%. They were excluded in this table for it is highly unlikely that no loss occurred in both distillations.

All laboratories reported a test result for the mass fraction for Gas (LPG), but some did not report 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.

D2892:   This distillation was not problematic. In total four statistical outliers were observed. However, after rejection of the statistical outliers the calculated reproducibilities are in agreement with the requirements of ASTM D2892:19.

D5236:   This distillation may be problematic. No statistical outliers were observed. 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 are all, but one, not in agreement with the requirements of ASTM D2892:19 (low pressure).

Graph Cumulative %M/M (appendix 2): The starts of the true boiling point curves (cumulative %M/M *vs* temperature AET) of the laboratories show a high resemblance. The line for laboratory 1990 shows a small negative deviation along a part of 250°C-390°C. This is in line with the deviating test result found for this lab in fraction 5 of the distillation.

4.1C - Analysis of distillation fractions (appendix 1C)

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 only 3 statistical outliers were observed. Two other test results were excluded. 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 determination 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 0.5719 kg/L, which is in good agreement with the Density of a mixture of C4 hydrocarbons and Isopentane.

Sulfur:

The Sulfur test results on the 12 collected distillation fractions show problems for a few participating laboratories. In total seven statistical outliers were observed and another test result was excluded. The observed reproducibility per fraction will be the sum of the variation in the Sulfur determination and the variation caused by the distillation. Therefore, the observed reproducibilies 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 two statistical outliers were observed. The observed reproducibility per fraction will be the sum of the variation in the Nitrogen determination and the variation caused by the distillation. Therefore, the observed reproducibilies were not compared with the requirements and consequently no z-scores were calculated.

Calculation (theoretical) values: The Density, Sulfur and Nitrogen test results do show some deviating 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 Density, Sulfur and Nitrogen are in line with the averages of the reported Density, Sulfur and Nitrogen test values (see below table).

parameter	unit	average reported by labs	average calculated by iis	absolute difference
Density	kg/L	0.8734	0.8720	-0.0014
Sulfur	%M/M	2.620	2.545	-0.075
Nitrogen	mg/kg	1271	1317	46

Table 3: comparison of actual reported values to calculated values by iis from the fractions

Graph Sulfur (appendix 3): The true boiling point curves (Sulfur in %M/M *vs* temperature AET) of most laboratories show a high resemblance. Except for the line of lab 608, which reported deviating Sulfur test results for the fractions 7 and 9-12 (HGO and VGO).

Graph Nitrogen (appendix 4): The true boiling point curves (Nitrogen in %M/M *vs* temperature AET) of most laboratories show a high. Laboratory 445 has a positive deviation and laboratory 1026 a negative deviation in the upper part (vacuum distillation). Both laboratories reported deviating test results for the fractions 9 to 11 (VGO).

P(iP)NA: This determination was performed on fraction 2 (Light Naphtha) and on fraction 3 (Heavy Naphtha) only. On the Light Naphtha three statistical outliers were observed. More analytical problems were observed on fraction 3 (Heavy Naphtha). In total ten statistical outliers were observed. Each observed reproducibility will be the sum of the variation in the analytical method and the variation caused by the distillation 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 and 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:19.

Distillation: This determination was performed on the combined fractions 5 and 6 only. No analytical problems were encountered. Two statistical outliers were observed. Each observed reproducibility will be the sum of the variation in this distillation of the combined fractions and the variation caused by the D2892 distillation. Therefore, the observed reproducibilities were not compared with the requirements and consequently no z-scores were calculated.

4.1D - Simulated Distillation and Effective Cut Point D2892

SimDist: This determination was performed on both fractions 5 (Light Gasoil) and 6 (Medium Gasoil). This simulated distillation was performed by ten participants on both fractions. Most reported test results showed a close resemblance. In total three outliers were observed. The goal was to enable evaluation of the column efficiency in accordance with appendix X2 of ASTM D2892:19.

ECP/$N_{minimum}$: From the reported test results, the ECP (effective cut point) and the Standard Efficiency $N_{minimum}$ were calculated, based on temperature intervals of 5 degrees. The ECP for one laboratory did not meet the requirements of ASTM D2892:19. The Standard Efficiency $N_{minimum}$ requirements of ASTM D2892:19 were met by four laboratories. A fifth laboratory was very close to the lower limit. One laboratory did not submit any Observed Vapor Temperatures, so the calculation could not be done. The other five laboratories were not in agreement with the requirements of ASTM D2892 and had either problems with pressure and/or temperature used in the distillation itself or with the determination of the Simulated Distillation test (see also the discussion on page 10). One laboratory submitted SimDist result in Fahrenheit, which had to be calculated to Celsius first.

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 iis17R02. A number of laboratories participated in both rounds and it is good to see that some participants improved or maintained the quality of the distillation (like laboratory 445, 1026 and 1066). 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 number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, EN standards) are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Density of original sample	kg/m^3	16	873.4	0.8	3.6
Sulfur of original sample	%M/M	16	2.620	0.134	0.135
Nitrogen of original sample	mg/kg	9	1271	537	338
Water of original sample	%V/V	13	0.033	0.044	0.030
D2892 distillation	True boiling point curve				
Gas LPG < 30°C	%M/M	16	1.6	1.0	1.3
Light Naphtha 30 - 90°C	%M/M	17	5.2	0.9	1.3
Heavy Naphtha 90 - 180°C	%M/M	17	12.9	0.7	1.3
Kerosene 180 - 215°C	%M/M	17	5.2	1.0	1.5
LGO 215 - 250°C	%M/M	15	5.2	0.4	1.5
MGO 250 - 310°C	%M/M	17	9.3	1.4	2.0
HGO 310 - 370°C	%M/M	16	9.6	1.5	2.0
Residue > 370°C	%M/M	17	50.5	2.9	n.a.
D5236 distillation	True boiling point curve				
VGO 370 - 420°C	%M/M	12	5.0	5.2	(2)
VGO 420 - 470°C	%M/M	12	8.0	3.6	(2)
VGO 470 - 520°C	%M/M	12	6.8	2.0	(2)
Residue > 520°C	%M/M	12	30.7	3.6	n.a.

Table 4: reproducibilities of test results on original Crude Oil sample #19225

Results mentioned between brackets are for indication only

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.

The analytical tests performed on the individual distillation fractions it was not possible to evaluate the reproducibilities against literature values as explained in paragraph 4.1. Therefore, the values are not given in a table and can be found in appendix 1C.

4.3   COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2019 WITH PREVIOUS PTS

The performance of the determinations of the proficiency tests was compared to the requirements of the respective reference test methods. The conclusions are given in the following table.

	November 2019	November 2017	November 2015	November 2013	November 2011
Density of original sample	++	+	+	++	++
Sulfur 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.	n.e.
D5236 distillation					
VGO 370 - 420°C	(--)	(--)	(--)	(--)	n.e.
VGO 420 - 470°C	(-)	(+/-)	(-)	(--)	n.e.
VGO 470 - 520°C	(+/-)	(++)	(+/-)	(-)	n.e.
Residue > 520°C	n.e.	n.e.	n.e	n.e.	n.e.

Table 5: comparison determinations against the reference test method

Results mentioned between brackets are for indication only

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

5   CONCLUSION

In this PT most laboratories (sixteen 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.

Although it can be concluded that most of the participants have no problem with the determination in Crude Oil Assay analyzes in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

APPENDIX 1A – ANALYSIS OF ORIGINAL SAMPLE

Determination of Density at 15°C on original sample #19225; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
171	D5002	873.33		-0.04	
339		-----		-----	
391	D5002	873.5		0.09	
442	IP365	873.3		-0.07	
445	D5002	872.9		-0.38	
608	D5002	873.0		-0.30	
862		-----		-----	
971	D5002	873.4		0.01	
1011		-----		-----	
1026	D5002	873.61		0.18	
1065	D4052	873.0		-0.30	
1066	D5002	873.3		-0.07	
1067		-----		-----	
1089	D5002	873.9	C	0.40	reported: 0.8739 kg/m^3
1108	D5002	873.71		0.25	
1412	D5002	873.5		0.09	
1455		-----		-----	
1613	D5002	873.4		0.01	
1714	D5002	873.3		-0.07	
1720		-----		-----	
1842		873.7	C	0.25	reported: 0.8737 kg/m^3
1949	D5002	873.3		-0.07	
1990	D4052	875.3	G(0.01)	1.49	
6156		-----		-----	

normality	OK	
n	16	
outliers	1	
mean (n)	873.38	
st.dev. (n)	0.272	
R(calc.)	0.76	
st.dev.(D5002:19)	1.285	
R(D5002:19)	3.60	
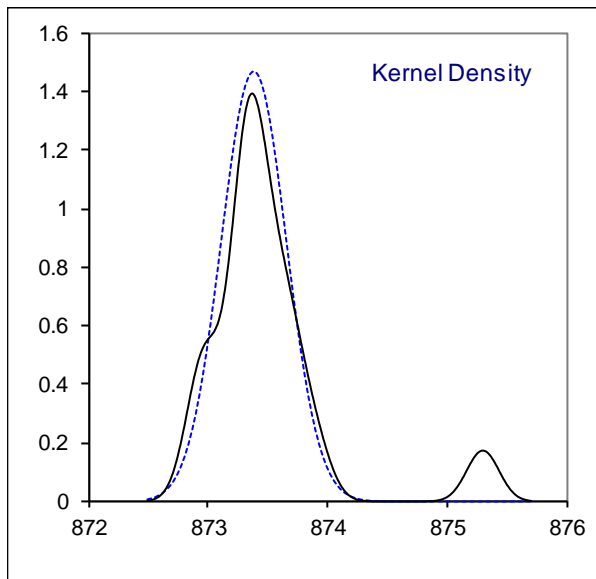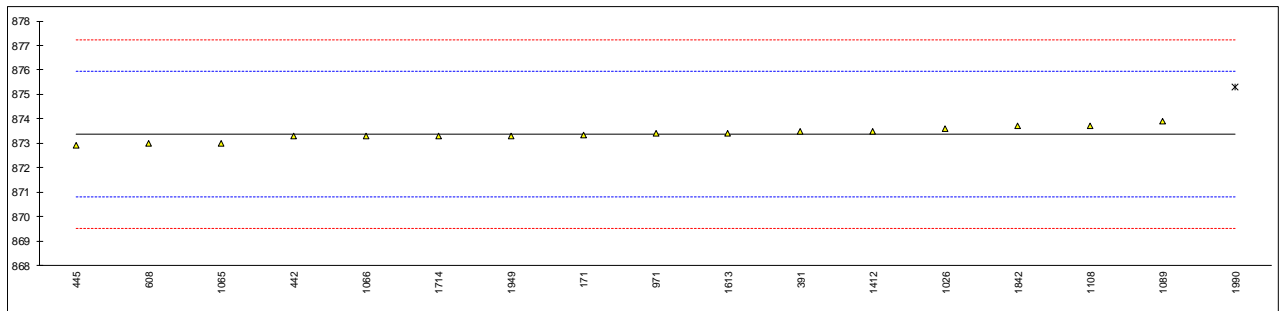
Determination of Sulfur on original sample #19225; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D4294	2.70741		1.82	
339		-----		-----	
391	D4294	2.609		-0.22	
442	IP336	2.644		0.50	
445	D4294	2.689		1.44	
608	D4294	2.5610		-1.21	
862		-----		-----	
971	D4294	2.614		-0.12	
1011		-----		-----	
1026	D2622	2.675		1.15	
1065	D4294	2.60		-0.41	
1066	D2622	2.556		-1.32	
1067		-----		-----	
1089		-----		-----	
1108	D4294	2.578		-0.86	
1412	D4294	2.67		1.04	
1455		-----		-----	
1613	D4294	2.636		0.34	
1714	D2622	2.59		-0.61	
1720		-----		-----	
1842	D4294	2.58		-0.82	
1949	D4294	2.567		-1.09	
1990	D4294	2.6377		0.37	
6156		-----		-----	

normality	OK
n	16
outliers	0
mean (n)	2.6196
st.dev. (n)	0.04780
R(calc.)	0.1338
st.dev.(D4294:16e1)	0.04828
R(D4294:16e1)	0.1352
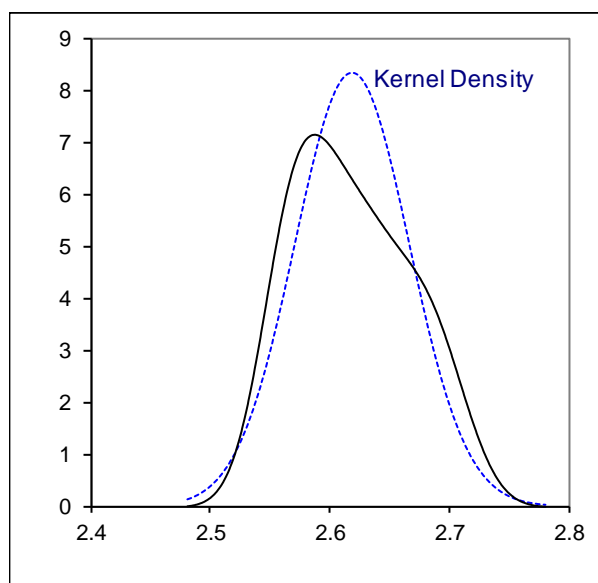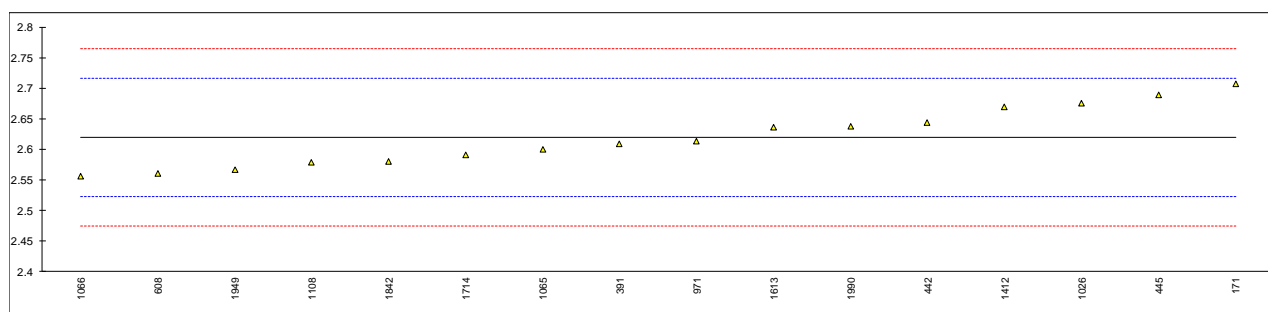
Determination of Nitrogen on original sample #19225; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
339		-----		-----	
391		-----		-----	
442		-----		-----	
445	D5762	1580		2.56	
608		-----		-----	
862		-----		-----	
971	D5762	1266		-0.04	
1011		-----		-----	
1026	D5762	907.22		-3.01	
1065	D4629	1200		-0.58	
1066	D5762	1220		-0.42	
1067		-----		-----	
1089		-----		-----	
1108	D5762	1311		0.33	
1412		-----		-----	
1455		-----		-----	
1613		-----		-----	
1714	D4629	1210		-0.50	
1720		-----		-----	
1842	D5762	1500		1.90	
1949	D5762	1241		-0.25	
1990		-----		-----	
6156		-----		-----	

normality	suspect
n	9
outliers	0
mean (n)	1270.58
st.dev. (n)	191.750
R(calc.)	536.90
st.dev.(D5762:18a)	120.705
R(D5762:18a)	337.97

Kernel Density
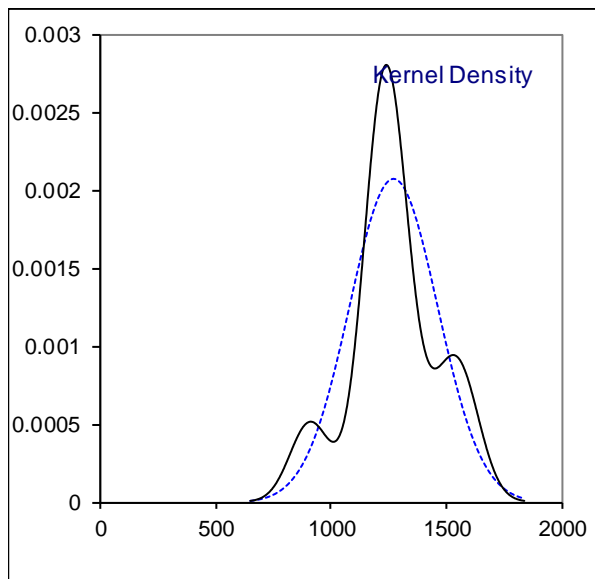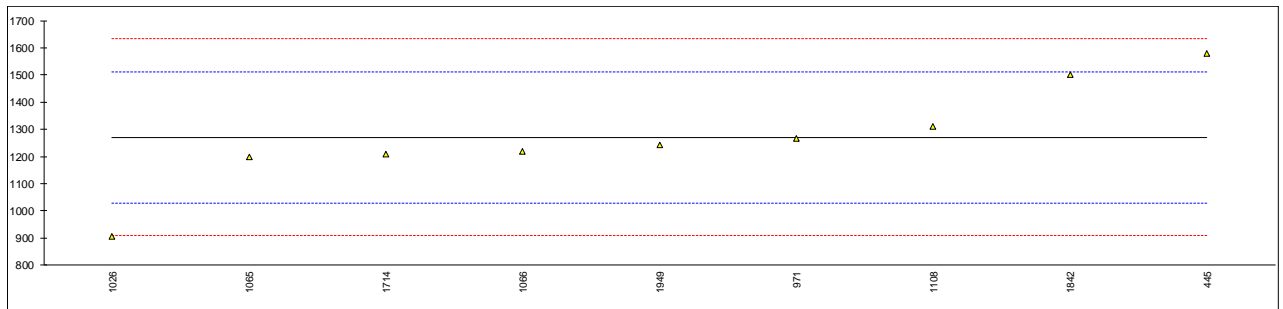
Determination of Water on original sample #19225; results in %V/V

lab	method	value	mark	z(targ)	remarks
171	D4377	0.047565		1.34	
339		-----		-----	
391	D4377	0.025		-0.75	
442	IP386	0.0341		0.09	
445	D4928	0.015		-1.68	
608	D4377	0.026		-0.66	
862		-----		-----	
971	D4928	0.025		-0.75	
1011		-----		-----	
1026	D4006	<0.1		-----	
1065	D4298	0.0145		-1.72	
1066	D4377	0.019		-1.31	
1067		-----		-----	
1089	D4377	0.03		-0.29	
1108	D4377	0.07		3.42	
1412	D4928	0.035		0.18	
1455		-----		-----	
1613	D95	<0.05		-----	
1714	D6304	0.039		0.55	
1720		-----		-----	
1842		-----		-----	
1949	D4006	<0.025		-----	
1990	D4007	0.05		1.57	
6156		-----		-----	

normality	not OK
n	13
outliers	0
mean (n)	0.0331
st.dev. (n)	0.01570
R(calc.)	0.0440
st.dev.(D4377:00)	0.01078
R(D4377:00)	0.0302

Kernel Density
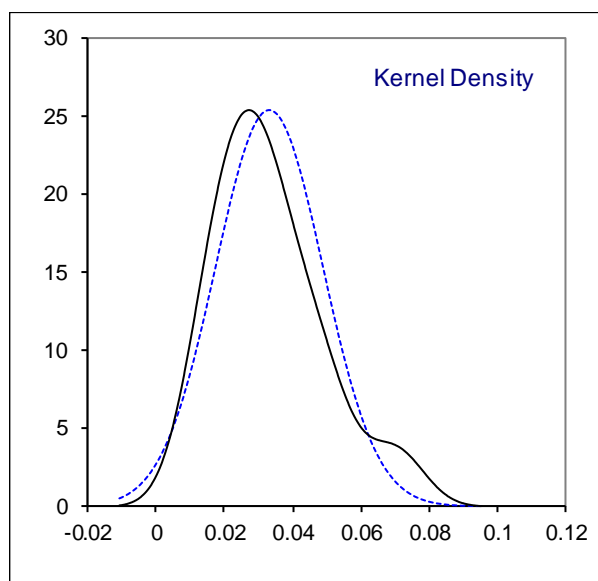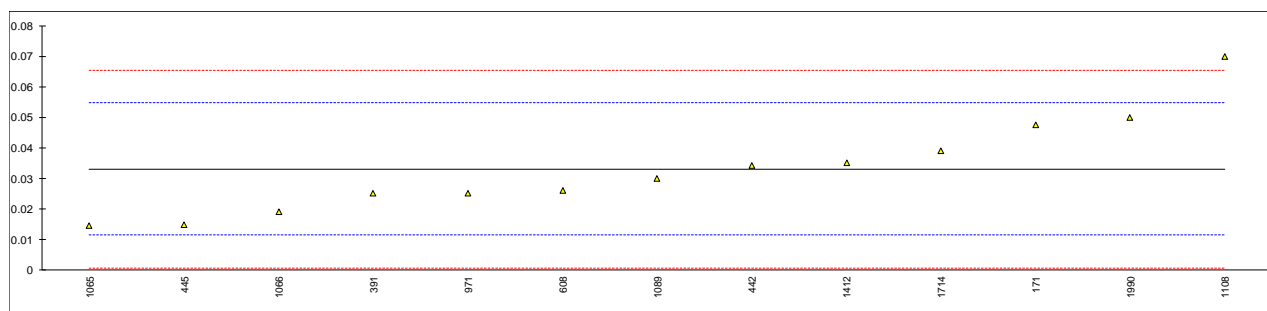
APPENDIX 1B – DISTILLATION RESULTS D2892 AND D5236

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

lab	method	value	mark	remarks
171	calc	99.49		
339	calc	-----		
391	calc	97.19		
442	calc	99.90		
445	calc	99.90		
608	calc	100.00	ex	excluded, recovery corrected for loss?
862	calc	-----		
971	calc	100.00	ex	excluded, recovery corrected for loss?
1011	calc	-----		
1026	calc	98.50		
1065	calc	100.00	ex	excluded, recovery corrected for loss?
1066	calc	99.83		
1067	calc	-----		
1089	calc	99.64		
1108	calc	98.30		
1412	calc	100.00	ex	excluded, recovery corrected for loss?
1455	calc	-----		
1613	calc	99.25		
1714	calc	98.96		
1720	calc	-----		
1842	calc	98.47		
1949	calc	99.71		
1990	calc	97.89		
6156	calc	-----		
	normality	OK		
	n	13		
	outliers	0+4ex		
	mean (n)	99.00		
	st.dev. (n)	0.870		
	R(calc.)	2.43		

In below graph, the dotted lines represent the ASTM D5236:2018a 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 D2892:2019, paragraph 11.2.
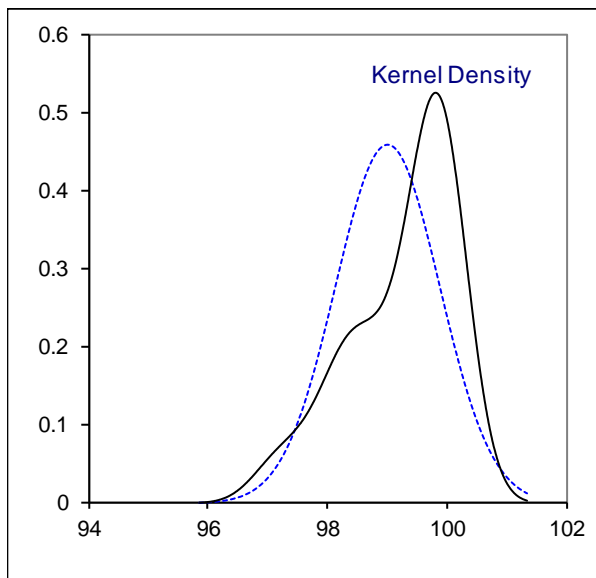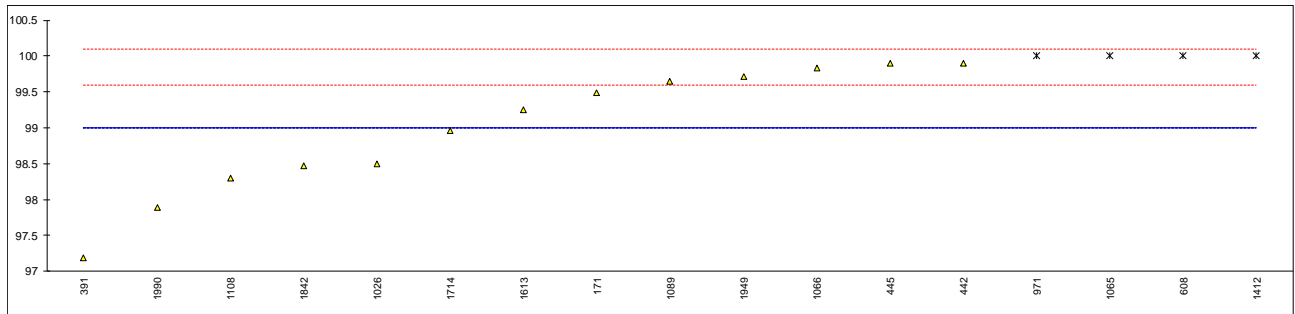
Total Volume balance/Total Volume recovery, results in %V/V

lab	method	value	mark	remarks
171	calc	99.88		
339	calc	-----		
391	calc	97.27		
442	calc	100.22		
445	calc	100.28		
608	calc	100.21	ex	excluded, recovery corrected for loss?
862	calc	-----		
971	calc	100.41	ex	excluded, recovery corrected for loss?
1011	calc	-----		
1026	calc	98.65		
1065	calc	100.13	ex	excluded, recovery corrected for loss?
1066	calc	100.13		
1067	calc	-----		
1089	calc	99.81		
1108	calc	98.28		
1412	calc	100.18	ex	excluded, recovery corrected for loss?
1455	calc	-----		
1613	calc	99.12		
1714	calc	98.91		
1720	calc	-----		
1842	calc	97.94		
1949	calc	99.90		
1990	calc	97.87		
6156	calc	-----		
	normality	OK		
	n	13		
	outliers	0+4ex		
	mean (n)	99.10		
	st.dev. (n)	1.023		
	R(calc.)	2.87		
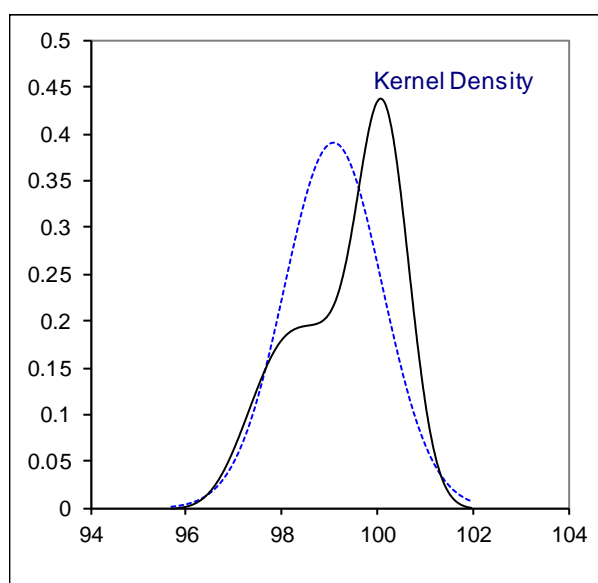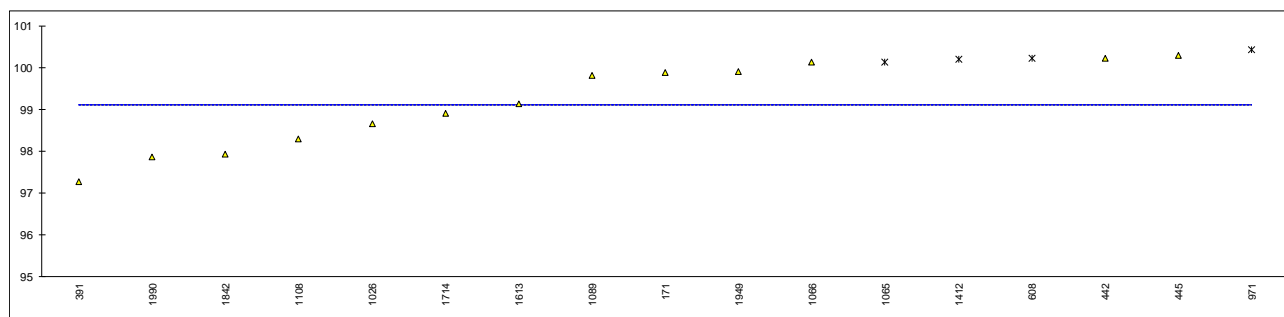
Determination of true boiling point curve of D2892; individual fractions in %M/M

| lab	Gas LPG <30°C	L. Naphtha 30-90°C	H. Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C
171	1.8592	5.1378	12.6657	5.1877	5.0293	9.6510	9.5367	50.4194
339	-----	-----	-----	-----	-----	-----	-----	-----
391	1.5566	4.5805	13.1490	5.0094	5.2473	9.3203	10.2397	48.0856
442	1.4454	5.2955	12.9316	5.7855	5.0147	9.5691	9.5614	50.2989
445	1.9616	5.1068	12.7692	4.6601	5.0889	9.3912	9.3282	51.5919
608	1.0088	5.8024	13.0633	5.0537	5.2798	9.3199	8.7073	51.7648
862	-----	-----	-----	-----	-----	-----	-----	-----
971	2.4031	5.1497	12.9006	5.1497	5.1864	9.6993	9.8105	49.6993
1011	-----	-----	-----	-----	-----	-----	-----	-----
1026	1.4904	4.9768	12.7229	5.1373	5.1106	9.4720	9.4853	50.1077
1065	1.3867	5.6972	13.1758	4.9753	5.2843	8.0628	10.2623	51.1555
1066	2.0984	4.9906	12.8319	5.1207	5.2239	9.2129	9.3644	50.9842
1067	-----	-----	-----	-----	-----	-----	-----	-----
1089	1.4433	5.1443	12.6941	5.3906	5.1860	9.6977	9.5841	50.5038
1108	1.4417	5.1769	12.6802	5.5046	**3.5714**	10.3539	8.4862	51.0813
1412	1.6736	5.1891	13.4456	5.5908	5.6315	8.6509	9.2760	50.5424
1455	-----	-----	-----	-----	-----	-----	-----	-----
1613	1.3480	5.7127	12.8964	4.7109	5.4007	8.7750	**11.4396**	48.9697
1714	1.2147	5.1375	12.3842	5.7579	5.2426	9.6067	10.1826	49.4369
1720	-----	-----	-----	-----	-----	-----	-----	-----
1842	**0.3192**	5.0932	12.8370	5.2186	5.1873	9.2886	9.4938	51.0318
1949	1.5842	4.7797	12.4578	4.4554	5.3638	9.4966	10.4746	51.1011
1990	1.0256	5.3147	12.9681	4.9029	**3.2401**	9.0210	9.2541	52.1601
6156	-----	-----	-----	-----	-----	-----	-----	-----
normality	OK	OK	OK	OK	not OK	suspect	OK	OK
n	16	17	17	17	15	17	16	17
outliers	1	0	0	0	2	0	1	0
mean (n)	1.5588	5.1933	12.8573	5.1536	5.2318	9.3288	9.5654	50.5255
st.dev. (n)	0.37365	0.31451	0.26236	0.37032	0.15596	0.50585	0.54154	1.04067
R(calc.)	1.0462	0.8806	0.7346	1.0369	0.4367	1.4164	1.5163	2.9139
st.dev.(D2892:19)	0.46429	0.46429	0.46429	0.53571	0.53571	0.71429	0.71429	n.a.
R(D2892:19)	1.3	1.3	1.3	1.5	1.5	2.0	2.0	n.a.

NB. Bold and underlined figures are statistical outliers

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	0.65	-0.12	-0.41	0.06	-0.38	0.45	-0.04	-----
339	-----	-----	-----	-----	-----	-----	-----	-----
391	0.00	-1.32	0.63	-0.27	0.03	-0.01	0.94	-----
442	-0.24	0.22	0.16	1.18	-0.41	0.34	-0.01	-----
445	0.87	-0.19	-0.19	-0.92	-0.27	0.09	-0.33	-----
608	-1.18	1.31	0.44	-0.19	0.09	-0.01	-1.20	-----
862	-----	-----	-----	-----	-----	-----	-----	-----
971	1.82	-0.09	0.09	-0.01	-0.08	0.52	0.34	-----
1011	-----	-----	-----	-----	-----	-----	-----	-----
1026	-0.15	-0.47	-0.29	-0.03	-0.23	0.20	-0.11	-----
1065	-0.37	1.09	0.69	-0.33	0.10	-1.77	0.98	-----
1066	1.16	-0.44	-0.05	-0.06	-0.01	-0.16	-0.28	-----
1067	-----	-----	-----	-----	-----	-----	-----	-----
1089	-0.25	-0.11	-0.35	0.44	-0.09	0.52	0.03	-----
1108	-0.25	-0.04	-0.38	0.66	-3.10	1.44	-1.51	-----
1412	0.25	-0.01	1.27	0.82	0.75	-0.95	-0.41	-----
1455	-----	-----	-----	-----	-----	-----	-----	-----
1613	-0.45	1.12	0.08	-0.83	0.32	-0.78	2.62	-----
1714	-0.74	-0.12	-1.02	1.13	0.02	0.39	0.86	-----
1720	-----	-----	-----	-----	-----	-----	-----	-----
1842	-2.67	-0.22	-0.04	0.12	-0.08	-0.06	-0.10	-----
1949	0.05	-0.89	-0.86	-1.30	0.25	0.23	1.27	-----
1990	-1.15	0.26	0.24	-0.47	-3.72	-0.43	-0.44	-----
6156	-----	-----	-----	-----	-----	-----	-----	-----
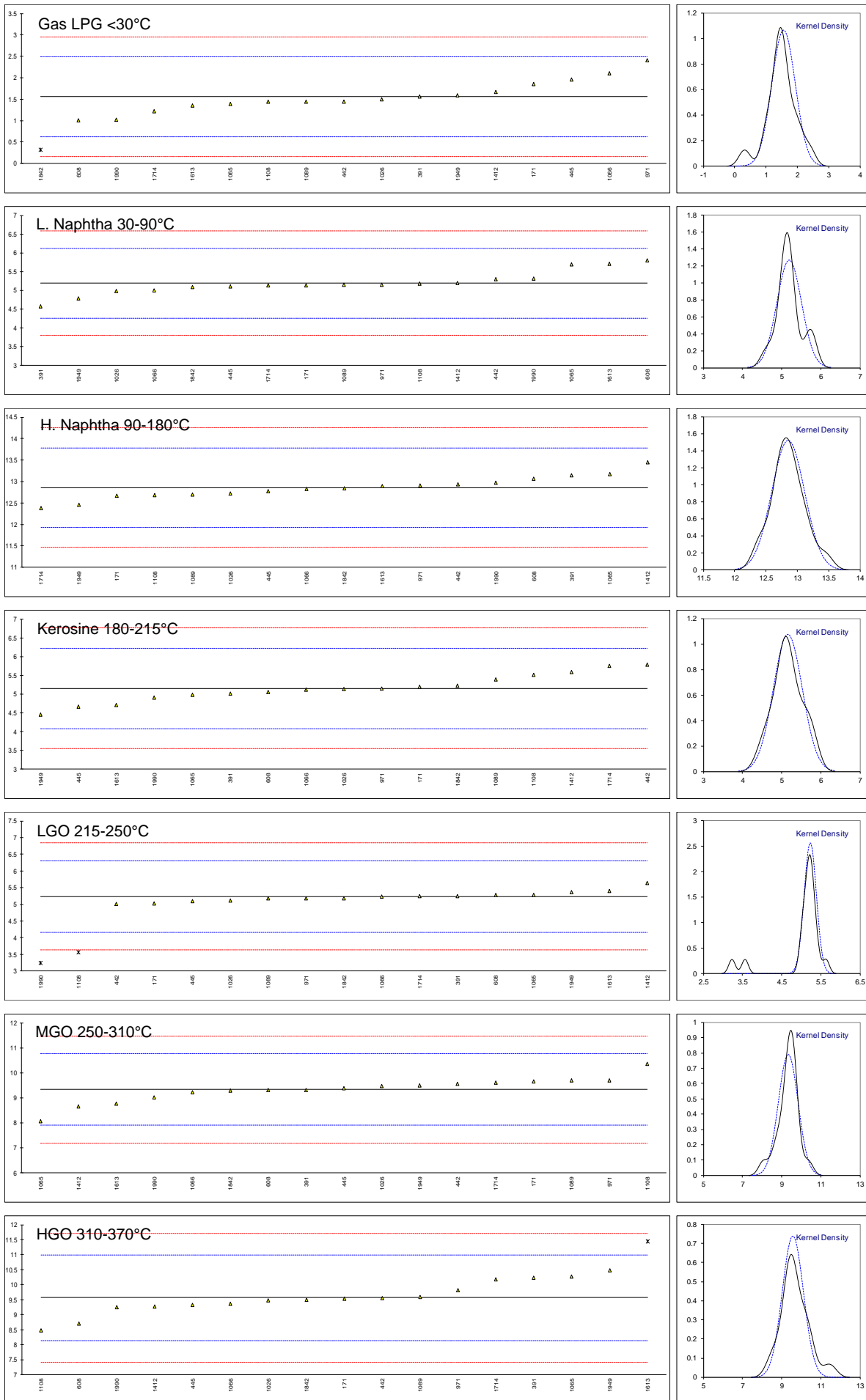
Determination of true boiling point curve D5236 (continued); individual fractions in %M/M

lab	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue 520+ °C	remarks
171	-----	-----	-----	-----	
339	-----	-----	-----	-----	
391	3.7434	7.4386	6.0408	30.8628	
442	-----	-----	-----	-----	
445	6.9919	6.6000	6.9273	31.0727	
608	7.6328	8.4958	7.5259	28.1103	
862	-----	-----	-----	-----	
971	3.6695	6.6239	6.7266	32.6806	
1011	-----	-----	-----	-----	
1026	2.0536	10.2993	5.5964	32.1583	
1065	4.5794	9.2791	6.1954	31.1015	
1066	8.1134	7.0383	6.4980	29.3345	
1067	-----	-----	-----	-----	
1089	-----	-----	-----	-----	
1108	-----	-----	-----	-----	
1412	-----	-----	-----	-----	
1455	-----	-----	-----	-----	
1613	2.8914	6.1263	7.9529	31.9991	
1714	5.0006	7.8862	6.8464	29.7036	
1720	-----	-----	-----	-----	
1842	5.0150	8.7666	6.8869	30.3632	
1949	5.4932	7.9916	7.1604	30.4559	
1990	4.6946	9.3885	7.8244	30.2525	
6156	-----	-----	-----	-----	
normality	OK	OK	OK	OK	
n	12	12	12	12	
outliers	0	0	0	0	
mean (n)	4.9899	7.9945	6.8485	30.6746	
st.dev. (n)	1.84838	1.28988	0.70839	1.27897	
R(calc.)	5.1755	3.6117	1.9835	3.5811	
st.dev.(D2892:19)	(0.71429)	(0.71429)	(0.71429)	n.a.	
R(D2892:19)	(2)	(2)	(2)	n.a.	

Corresponding z-scores for above mass fractions:

lab	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue 520+ °C
171	-----	-----	-----	-----
339	-----	-----	-----	-----
391	-----	-----	-----	-----
442	-----	-----	-----	-----
445	-----	-----	-----	-----
608	-----	-----	-----	-----
862	-----	-----	-----	-----
971	-----	-----	-----	-----
1011	-----	-----	-----	-----
1026	-----	-----	-----	-----
1065	-----	-----	-----	-----
1066	-----	-----	-----	-----
1067	-----	-----	-----	-----
1089	-----	-----	-----	-----
1108	-----	-----	-----	-----
1412	-----	-----	-----	-----
1455	-----	-----	-----	-----
1613	-----	-----	-----	-----
1714	-----	-----	-----	-----
1720	-----	-----	-----	-----
1842	-----	-----	-----	-----
1949	-----	-----	-----	-----
1990	-----	-----	-----	-----
6156	-----	-----	-----	-----

VGO 370-420°C

VGO 420-470°C

VGO 470-520°C

Residue 520+ °C
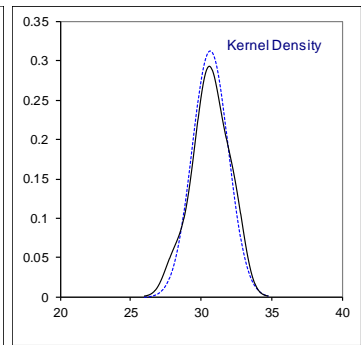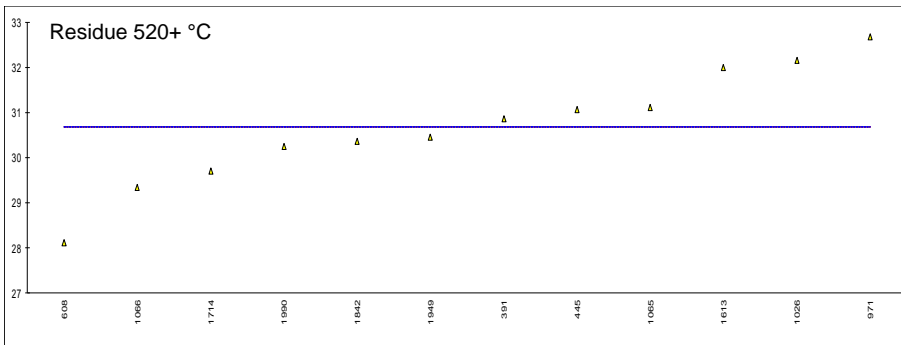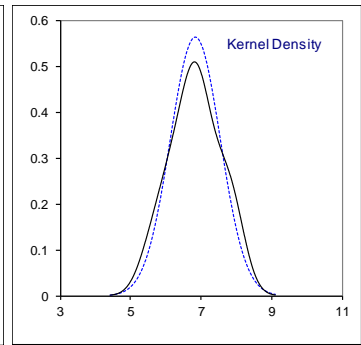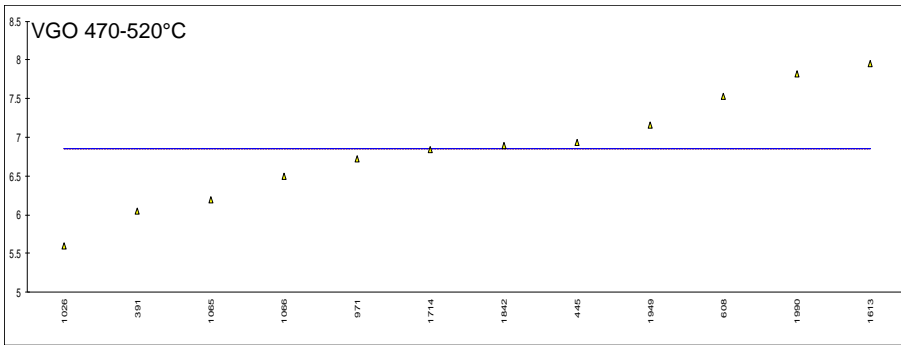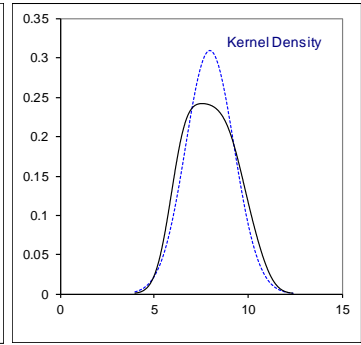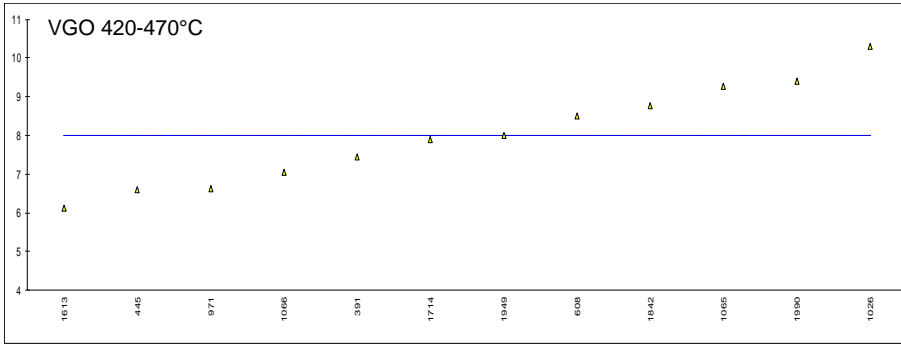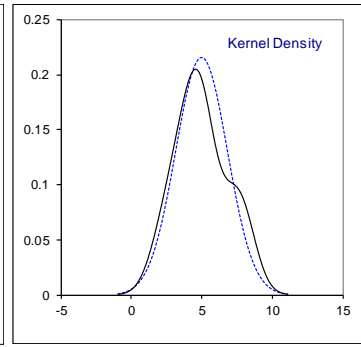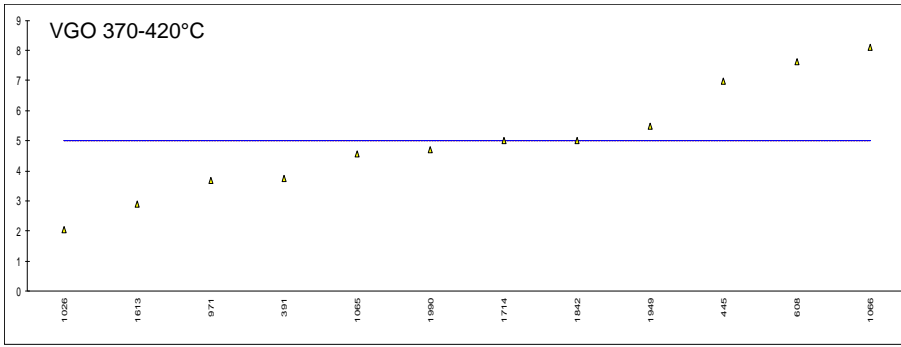
APPENDIX 1C – ANALYSIS OF DISTILLATION FRACTIONS

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	-----	0.6681	0.7477	0.7912	0.8073	0.8409	0.8736	0.9828
339		-----	-----	-----	-----	-----	-----	-----	-----
391	D4052	0.5759	0.6718	0.7486	0.7920	0.8101	0.8411	0.8746	0.9876
442	IP365	-----	0.6642	0.7481	0.7916	0.8100	0.8412	0.8760	0.9828
445	D4052	0.5659	0.6694	0.7483	0.7925	0.8067	0.8382	0.8715	0.9809
608	D4052	0.5440	0.6604	0.7491	0.7928	0.8093	0.8404	0.8776	0.9813
862		-----	-----	-----	-----	-----	-----	-----	-----
971	D4052	0.5912	0.6729	0.7497	0.7909	0.8093	0.8404	0.8756	0.9831
1011		-----	-----	-----	-----	-----	-----	-----	-----
1026	D4052	0.5669	0.6655	0.7473	0.7907	0.8070	0.8416	0.8738	0.9836
1065	D4052	-----	0.6661	0.7481	0.7918	0.8082	0.8421	0.8711	0.9874
1066	D4052	0.5834	0.6717	0.7494	0.7918	0.8091	0.8426	0.8737	0.9832
1067		-----	-----	-----	-----	-----	-----	-----	-----
1089	D4052	0.5694	0.6632	0.7465	0.7908	0.8083	0.8420	0.8762	0.9838
1108	D4052	0.5806	0.6751	0.7486	0.7926	0.8103	0.8379	0.8730	0.9830
1412	D4052	-----	0.6678	0.7520	0.7932	0.8125	0.8439	0.8759	0.9853
1455		-----	-----	-----	-----	-----	-----	-----	-----
1613	D4052	-----	0.6723	0.7524	0.7920	0.8088	0.839	0.8774	0.9853
1714	D4052	-----	0.6724 C	0.7484 C	0.7913 C	0.8108 C	0.8431 C	0.8782 C	-----
1720		-----	-----	-----	-----	-----	-----	-----	-----
1842	D4052	N/A	0.6657	0.7470	0.7913	0.8073	0.8412	0.8730	0.9833
1949	D4052	-----	0.6655	0.7447	**_0.7866_**	0.8036	0.8375	0.8715	0.9815
1990	D4052	0.5702	0.6627	0.7476	0.7918	0.8080	0.8415	0.8732	**_1.0287_**
6156		-----	-----	-----	-----	-----	-----	-----	-----
	normality	suspect	OK	suspect	OK	suspect	OK	OK	OK
	n	9	17	17	16	17	17	17	15
	outliers	0	0	0	1	0	0	0	1
	mean (n)	0.5719	0.6679	0.7484	0.7918	0.8086	0.8409	0.8745	0.9837
	st.dev. (n)	0.01344	0.00424	0.00184	0.00073	0.00199	0.00181	0.00220	0.00199
	R(calc.)	0.0376	0.0119	0.0051	0.0021	0.0056	0.0051	0.0062	0.0056
	RSD	2.3%	0.6%	0.3%	0.1%	0.2%	0.2%	0.3%	0.2%

NB. Bold and underlined figures are statistical outliers
Lab 1714 first reported: 672.4, 748.4, 791.3, 810.8, 843.1 and 878.2 kg/L respectively
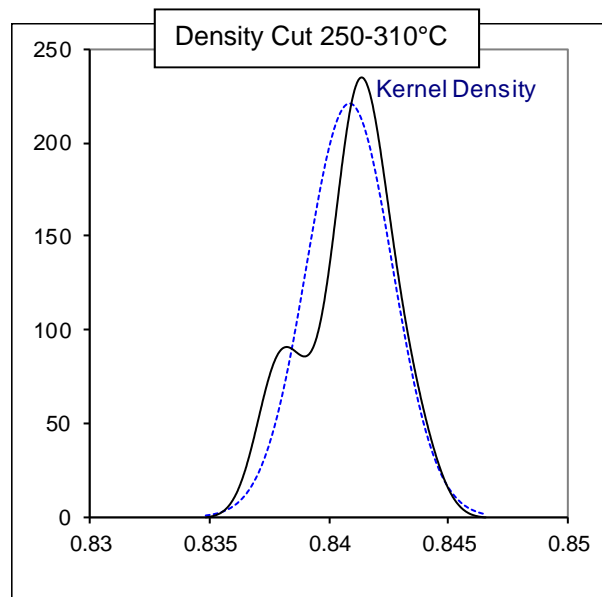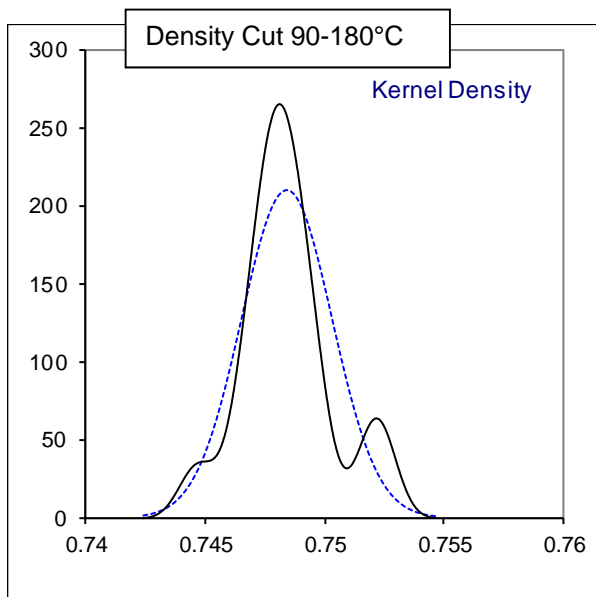
Determination of Density at 15°C on distillation fractions (continued); results in kg/L

lab	Method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue >520°C	remarks
171		-----	-----	-----	-----	Residue >370°C: D5002
339		-----	-----	-----	-----	
391	D4052	0.8979	0.9169	0.9397	1.0297	LPG: ISO8973/D4052
442		-----	-----	-----	-----	
445	D4052	**_0.9113_**	0.9408 ex	0.9236 ex	1.0208	LPG: GC, Residue >370°C and >520°C: D70
608	D4052	0.9011	0.9252	0.9502	1.0323	LPG: D2163, Residue >520°C: D70
862		-----	-----	-----	-----	
971	D4052	0.9006	0.9115	0.9331	1.0267	
1011		-----	-----	-----	-----	
1026	D4052	0.8990	0.9142	0.9382	1.0261	LPG: ISO8973
1065	D4052	0.9017	0.9196	0.9438	1.0301	
1066	IP189/190	0.9052	0.9260	0.9457	1.0293	LPG: calc., VGO 370-520°C IP189/190, Residue >520°C D70
1067		-----	-----	-----	-----	
1089		-----	-----	-----	-----	LPG: manual method
1108		-----	-----	-----	-----	LPG: D2598
1412		-----	-----	-----	-----	
1455		-----	-----	-----	-----	
1613	D4052	0.9045	0.9198	0.9401	1.0307	
1714	D4052	0.9043 C	0.9228 C	0.9462 C	1.0276 C	
1720		-----	-----	-----	-----	
1842	D4052	0.9025	0.9199	0.9438	1.0312	
1949	D4052	0.9020	0.9165	0.9387	1.0297	
1990	D4052	0.9029	0.9171	0.9428	1.0294	LPG: D2598, Residue >520°C: D70
6156		-----	-----	-----	-----	
	normality	OK	OK	OK	not OK	
	n	11	11	11	12	
	outliers	1	0+1ex	0+1ex	0	
	mean (n)	0.9020	0.9190	0.9420	1.0286	
	st.dev. (n)	0.00227	0.00444	0.00468	0.00305	
	R(calc.)	0.0064	0.0124	0.0131	0.0085	
	RSD	0.3%	0.5%	0.5%	0.3%	

NB. Bold and underlined figures are statistical outliers
Lab 445 two test values are excluded as Density of the earlier fraction is higher than the next one which is not possible.
Lab 1714 first reported: 904.3, 922.8, 946.2 and 1027.6 respectively

Density Cut 370-420°C
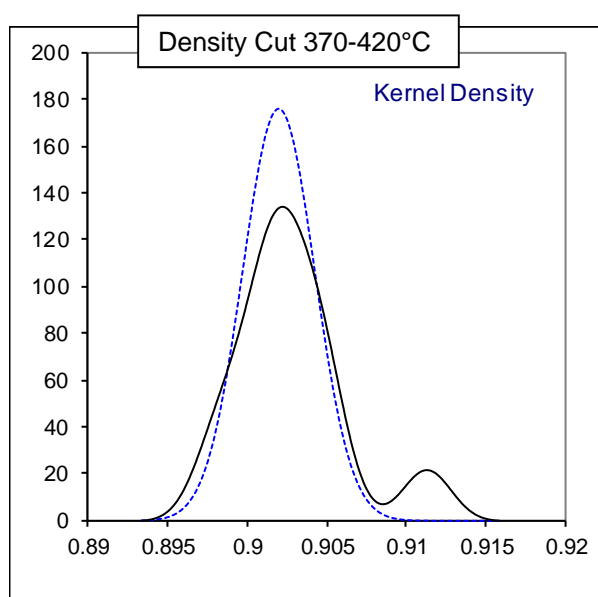
Determination of Sulfur on distillation fractions; results in %M/M

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	D4294	-----	0.0052	0.0400	0.142	0.330	1.23	2.19	4.53
339		-----	-----	-----	-----	-----	-----	-----	-----
391	D4294	-----	0.011	0.035	0.151	0.364	1.30	2.23	4.39
442		-----	-----	-----	-----	-----	-----	-----	-----
445	D4294	0.0040	0.0081	0.038	0.145	0.328	1.19	2.11	4.40
608	D4294	-----	0.0097	0.0340	0.1353	0.3795	1.1835	**1.9309**	4.4301
862		-----	-----	-----	-----	-----	-----	-----	-----
971	D4294	-----	0.0084	0.0470	0.143	0.368	1.299	2.213	4.435
1011		-----	-----	-----	-----	-----	-----	-----	-----
1026	D2622	0.0000834	0.00928	0.043	0.146	0.334	1.266	2.202	4.427
1065	D4294	-----	0.0087	0.043	0.15	0.37	1.25	2.09	4.33
1066	D2622	0.0001	0.0068	0.0384	0.148	0.371	1.26	2.13	4.29
1067		-----	-----	-----	-----	-----	-----	-----	-----
1089		-----	-----	-----	-----	-----	-----	-----	-----
1108	D4294	-----	**0.0168**	0.0452	**0.162**	0.395	1.16	2.12	**4.03**
1412		-----	-----	-----	-----	-----	-----	-----	-----
1455		-----	-----	-----	-----	-----	-----	-----	-----
1613	D4294	-----	0.0096	0.0442	0.1469	0.3610	1.230	2.200	4.335
1714	D2622	0.000110	0.00610	0.0382	0.148	0.404	1.28	2.26	-----
1720		-----	-----	-----	-----	-----	-----	-----	-----
1842	INH-05	-----	0.009	0.049	0.143	0.334	1.22	2.10	4.30
1949	D4294	-----	0.00732	0.03719	0.1339	0.3222	1.244	2.080	4.313
1990	D2622	-----	0.0069	0.0341	0.1447	0.3494	1.2553	2.1512	4.3415
6156		-----	-----	-----	-----	-----	-----	-----	-----
	normality	unknown	OK	OK	OK	OK	OK	OK	OK
	n	4	13	14	13	14	14	13	12
	outliers	0	1	0	1	0	0	1	1
	mean (n)	(0.0011)	0.0082	0.0404	0.1444	0.3579	1.2406	2.1597	4.3768
	st.dev. (n)	n.a.	0.00163	0.00483	0.00511	0.02572	0.04187	0.05913	0.07111
	R(calc.)	n.a.	0.0046	0.0135	0.0143	0.0720	0.1172	0.1656	0.1991
	st.dev.(D4294:16e1)	n.a.	(0.00117)	(0.00377)	(0.00745)	(0.01318)	(0.02892)	(0.04263)	(0.06721)
	R(D4294:16e1)	n.a.	(0.0033)	(0.0105)	(0.0209)	(0.0369)	(0.0810)	(0.1194)	(0.1882)
	RSD	n.a.	20.0%	11.9%	3.5%	7.2%	3.4%	2.7%	1.6%

NB. Bold and underlined figures are statistical outliers
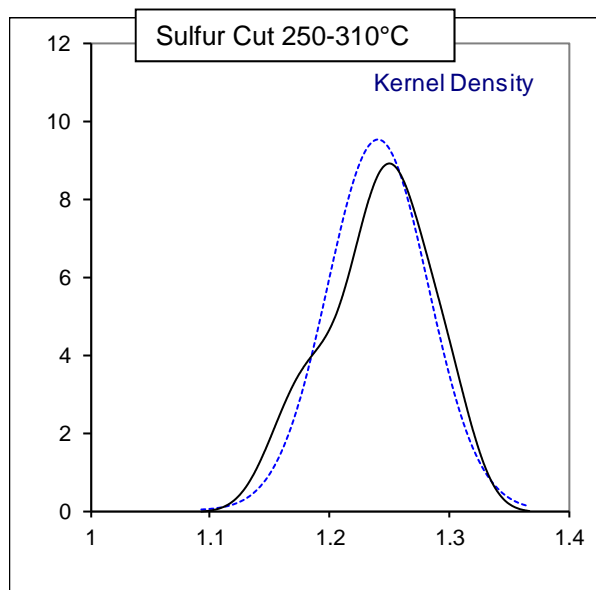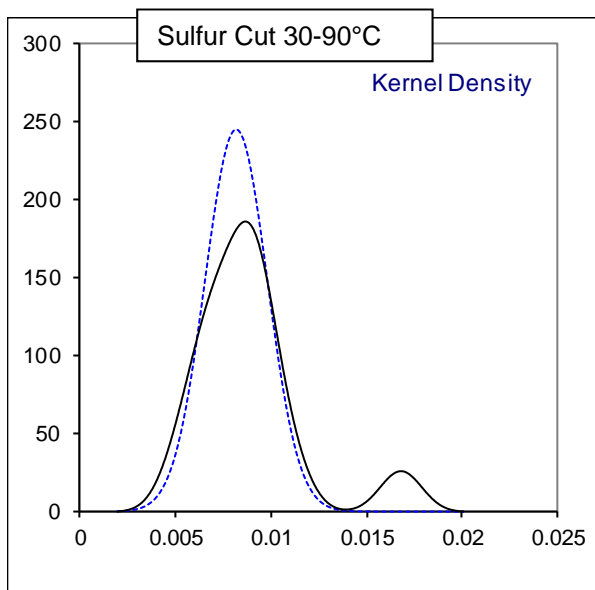
Determination of Sulfur on distillation fractions (continued); results in %M/M

lab	Method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue >520°C	remarks
171		-----	-----	-----	-----	
339		-----	-----	-----	-----	
391	D4294	2.41	2.71	3.17	5.32	
442		-----	-----	-----	-----	
445	D4294	2.72	2.88	3.34	5.47	LPG: D6667
608	D4294	**2.0915**	**2.3069**	**2.6060**	5.5404 ex	
862		-----	-----	-----	-----	
971	D4294	2.509	2.694	3.098	5.252	
1011		-----	-----	-----	-----	
1026	D2622	2.470	2.754	3.201	5.232	LPG: D4045, L.Naphtha/H.Naphtha: ISO20846
1065	D4294	2.44	2.77	3.27	5.25	
1066	D2622	2.53	2.84	3.34	5.33	LPG/L.Naphtha/H.Naphtha: D5453
1067		-----	-----	-----	-----	
1089		-----	-----	-----	-----	
1108		-----	-----	-----	-----	
1412		-----	-----	-----	-----	
1455		-----	-----	-----	-----	
1613	D4294	2.593	2.795	3.255	5.0562	
1714	D2622	2.53	2.81	3.32	5.440	LPG/L.Naphtha/H.Naphtha: D5453
1720		-----	-----	-----	-----	
1842	INH-05	2.52	2.74	3.24	5.35	
1949	D4294	2.567	2.778	3.233	5.128	
1990	D4294	2.6031	2.8079	3.3423	5.4913	L.Naphtha/H.Naphtha: D5453
6156		-----	-----	-----	-----	
	normality	suspect	OK	OK	OK	
	n	11	11	11	11	
	outliers	1	1	1	0+1ex	
	mean (n)	2.5356	2.7799	3.2554	5.3018	
	st.dev. (n)	0.08548	0.05494	0.07875	0.13686	
	R(calc.)	0.2394	0.1538	0.2205	0.3832	
	st.dev.(D4294:16e1)	(0.04727)	(0.04803)	(0.05553)	(0.07605)	
	R(D4294:16e1)	(0.1324)	(0.1345)	(0.1555)	(0.2129)	
	RSD	3.4%	2.0%	2.4%	2.6%	

NB. Bold and underlined figures are statistical outliers
Lab 608 one test value excluded because related test values are statistical outliers.
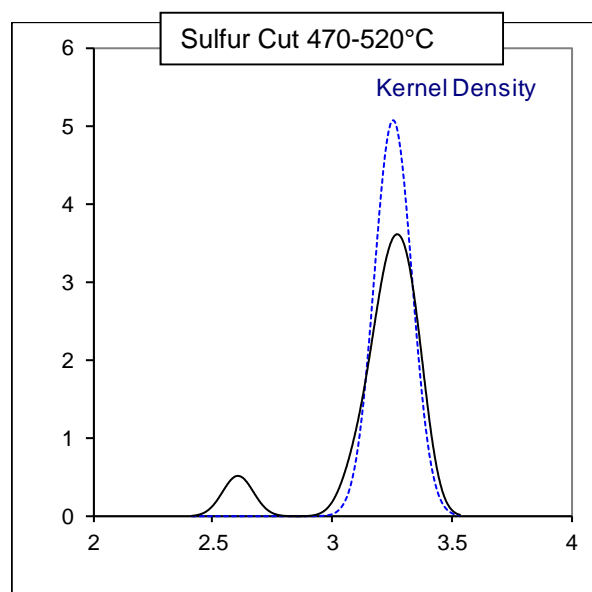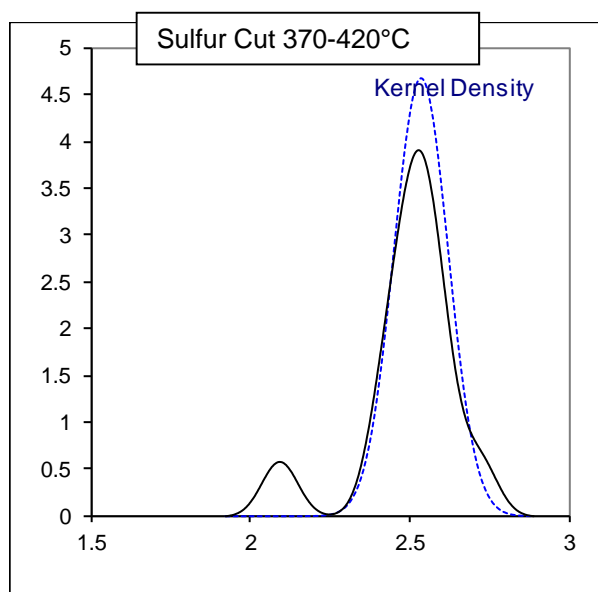
Determination of Nitrogen on distillation fractions; results in mg/kg

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	-----	0.4	0.4	0.4	0.6	12	-----	-----
339		-----	-----	-----	-----	-----	-----	-----	-----
391		-----	-----	-----	-----	-----	-----	-----	-----
442		-----	-----	-----	-----	-----	-----	-----	-----
445	D4629	-----	0.3	0.5	0.6	1.3	18	240	3000
608		-----	-----	-----	-----	-----	-----	-----	-----
862		-----	-----	-----	-----	-----	-----	-----	-----
971	D4629	-----	0.26	0.39	0.45	1.02	19	270	2600
1011		-----	-----	-----	-----	-----	-----	-----	-----
1026	D4629	-----	<1	<1	<1	<1	13.9	201.3	1967
1065	D4629	-----	<10	<10	<10	<10	12	150	2700
1066	D4629	<0.3	<0.3	<0.3	<0.3	<0.3	15.7	192	2400
1067		-----	-----	-----	-----	-----	-----	-----	-----
1089		-----	-----	-----	-----	-----	-----	-----	-----
1108	D5762	-----	0.1	0.3	0.4	1.0	9.6	164	2657
1412		-----	-----	-----	-----	-----	-----	-----	-----
1455		-----	-----	-----	-----	-----	-----	-----	-----
1613		-----	-----	-----	-----	-----	-----	-----	-----
1714	D4629	<0.1	<0.1	<0.1	0.1	1.2	17	220	-----
1720		-----	-----	-----	-----	-----	-----	-----	-----
1842	INH-12	-----	<0.2	<0.2	<0.2	<0.2	6.9	-----	2600
1949	D4629	-----	0.18	0.24	0.40	2.1	16.2	188	2340
1990	D4629	-----	0.092	0.140	0.167	0.549	13.51	-----	-----
6156		-----	-----	-----	-----	-----	-----	-----	-----
	normality	n.a.	unknown	unknown	unknown	unknown	OK	unknown	unknown
	n	2	8	6	7	7	11	8	8
	outliers	0	0	0	0	0	0	0	0
	mean (n)	<0.3	<0.37	0.3283	0.3596	1.1099	13.983	203.16	2533.0
	st.dev. (n)	n.a.	n.a.	0.12844	0.17095	0.51951	3.6844	39.326	303.70
	R(calc.)	n.a.	n.a.	0.3596	0.4787	1.4546	10.316	110.11	850.4
	st.dev.(D4629:17)	n.a.	n.a.	(0.16291)	(0.17072)	(0.30501)	(1.1243)	---	---
	R(D4629:17)	n.a.	n.a.	(0.4562)	(0.4780)	(0.8540)	(3.148)	---	---
	st.dev.(D5762:18a)	---	---	---	---	---	---	(19.300)	240.64
	R(D5762:18a)	---	---	---	---	---	---	(54.04)	673.8
	RSD	n.a.	n.a.	39.1%	47.5%	46.8%	26.3%	19.4%	12.0%
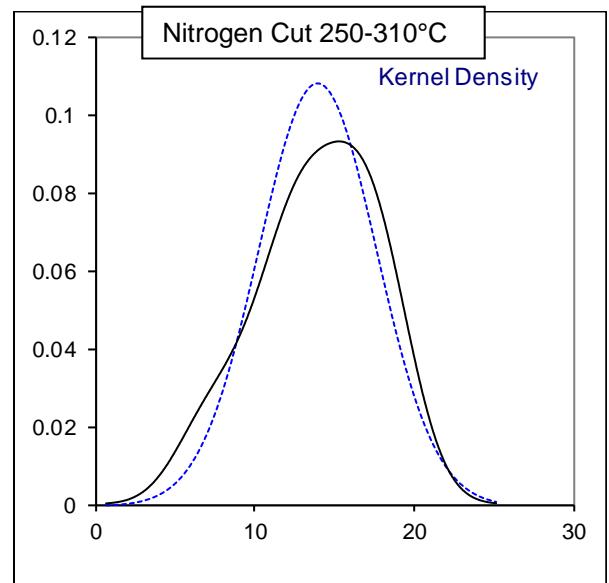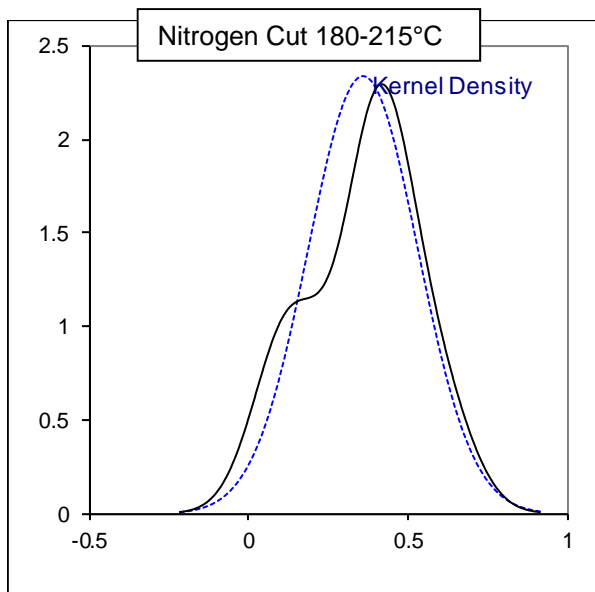
Determination of Nitrogen on distillation fractions (Continued); results in mg/kg

lab	method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue >520°C	remarks
171		-----	-----	-----	-----	
339		-----	-----	-----	-----	
391		-----	-----	-----	-----	
442		-----	-----	-----	-----	
445	D5762	**740**	**980**	1400	4300	HGO/Residue >370°C: D5762
608		-----	-----	-----	-----	
862		-----	-----	-----	-----	
971	D5762	560	720	1100	3500	Residue >370°C: D5762
1011		-----	-----	-----	-----	
1026	D5762	352	525	906	2523	Residue >370°C: D5762
1065	D4629	550	720	1140	4340	
1066	D5762	550	790	1100	3600	HGO/Residue >370°C: D5762
1067		-----	-----	-----	-----	
1089		-----	-----	-----	-----	
1108		-----	-----	-----	-----	
1412		-----	-----	-----	-----	
1455		-----	-----	-----	-----	
1613		-----	-----	-----	-----	
1714	D4629	470	670	1000	3400	
1720		-----	-----	-----	-----	
1842	INH-12	510	730	1200	4200	
1949	D5762	520	780	1120	3450	HGO/Residue >370°C: D5762
1990		-----	-----	-----	-----	
6156		-----	-----	-----	-----	
	normality	unknown	unknown	unknown	unknown	
	n	7	7	8	8	
	outliers	1	1	0	0	
	mean (n)	501.71	705.00	1120.75	3664.13	
	st.dev. (n)	72.897	89.022	144.515	609.113	
	R(calc.)	204.11	249.26	404.64	1705.52	
	st.dev.(D5762:18a)	(47.663)	(66.975)	(106.471)	n.a.	
	R(D5762:18a)	(133.46)	(187.53)	(298.12)	n.a.	
	RSD	14.5%	12.6%	12.9%	16.6%	

NB. Bold and underlined figures are statistical outliers
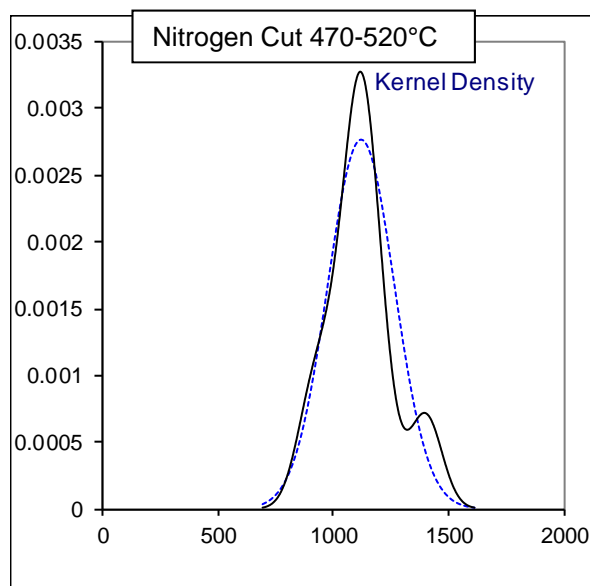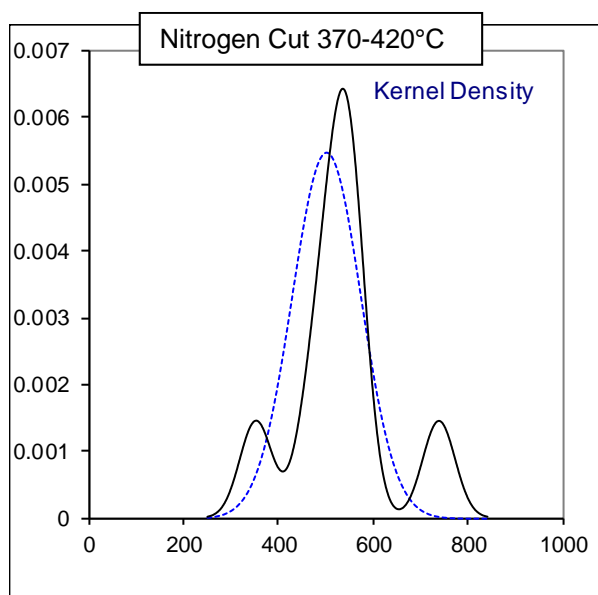
Determination of calculated (theoretical) Density at 15°C of sample #19225; results in kg/L

lab	method	calculated	mark	z(targ)	remarks	reported		difference
171	calc.by iis	0.8699		-1.59	*)	0.87333		-0.0034
339		-----		-----		-----		-----
391	calc.by iis	0.8727		0.61		0.8735		-0.0008
442	calc.by iis	0.8706		-1.09	*)	0.8733		-0.0027
445	calc.by iis	0.8696		-1.87		0.8729		-0.0033
608	calc.by iis	0.8711		-0.65		0.8730		-0.0019
862		-----		-----		-----		-----
971	calc.by iis	0.8698		-1.70		0.8734		-0.0036
1011		-----		-----		-----		-----
1026	calc.by iis	0.8723		0.23		0.87361		-0.0013
1065	calc.by iis	0.8719		-0.06	*)	0.8730		-0.0011
1066	calc.by iis	0.8706		-1.03		0.8733		-0.0027
1067		-----		-----		-----		-----
1089	calc.by iis	0.8716		-0.27		0.8739		-0.0023
1108	calc.by iis	0.8739		1.48		0.87371		0.0002
1412	calc.by iis	0.8719		-0.06	*)	0.8735		-0.0016
1455		-----		-----		-----		-----
1613	calc.by iis	0.8746		2.08	*)	0.8734		0.0012
1714	calc.by iis	0.8738		1.40	*)	0.8733		0.0005
1720		-----		-----		-----		-----
1842	calc.by iis	0.8779	ex	4.66	**)	0.8737		0.0042
1949	calc.by iis	0.8717		-0.23	*)	0.8733		-0.0016
1990	calc.by iis	0.8755		2.74		0.8753	G(0.01)	0.0002
6156		-----		-----		-----		-----

normality	OK						
n	16			n	16		
outliers	0 + 1ex			outliers	1		
mean (n)	0.87196			mean (n)	0.87338		-0.00142
st.dev. (n)	0.001757						
R(calc.)	0.00492						
st.dev.(D5002:19)	0.001283						
R(D5002:19)	0.00359						

*) no Density reported for fraction 1 (LPG), for calculation purposes the consensus value of the group (0.5837 kg/L) was used.
**) this laboratory was outlier in D2892 %M/M for fraction 1. Since the influence of this light fraction on the Density is significant the calculated Density of this laboratory was excluded.
Difference = calculated – reported Density
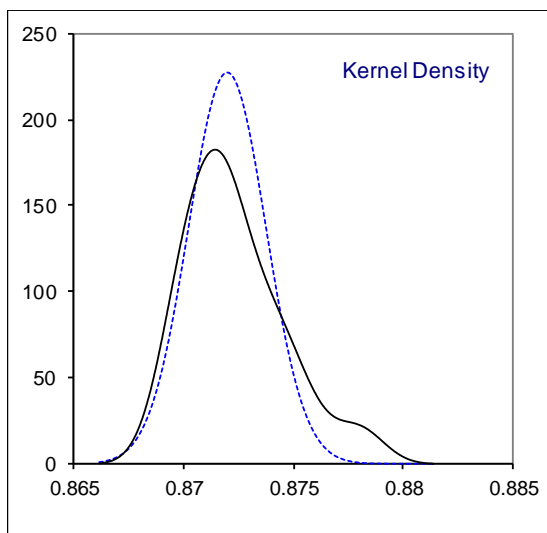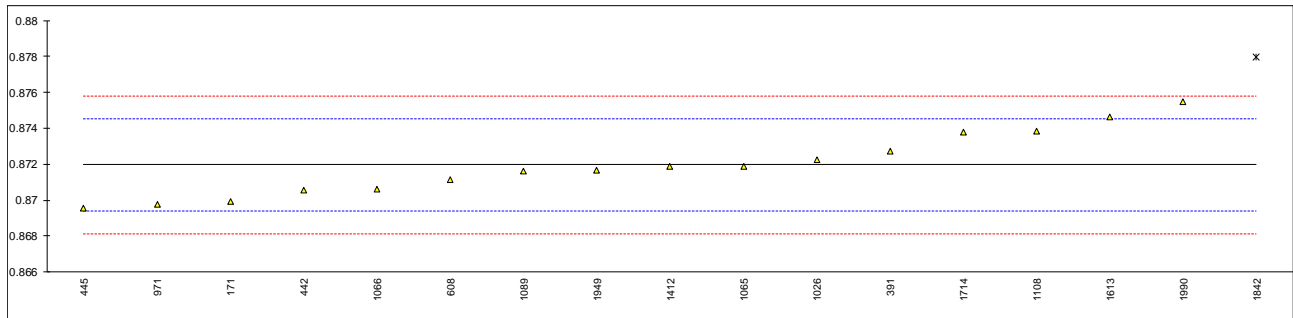
Determination of calculated (theoretical) Sulfur on sample #19225; results in %M/M

lab	method	calculated	mark	z(targ)	remarks	reported	difference
171	calc.by iis	2.6409		2.03		2.70741	-0.0666
339		-----		-----		-----	-----
391	calc.by iis	2.5065		-0.81		2.609	-0.1025
442		-----		-----		2.644	-----
445	calc.by iis	2.6487		2.19		2.689	-0.0403
608	calc.by iis	2.4195		-2.65		2.561	-0.1415
862		-----		-----		-----	-----
971	calc.by iis	2.5713		0.56		2.614	-0.0427
1011		-----		-----		-----	-----
1026	calc.by iis	2.5553		0.22		2.675	-0.1197
1065	calc.by iis	2.5526		0.16		2.6	-0.0474
1066	calc.by iis	2.5335		-0.24		2.556	-0.0225
1067		-----		-----		-----	-----
1089		-----		-----		-----	-----
1108	calc.by iis	2.3882		-3.31		2.578	-0.1898
1412		-----		-----		2.67	-----
1455		-----		-----		-----	-----
1613	calc.by iis	2.5153		-0.62		2.636	-0.1207
1714	calc.by iis	2.5791		0.72		2.59	-0.0109
1720		-----		-----		-----	-----
1842	calc.by iis	2.5527		0.17		2.58	-0.0273
1949	calc.by iis	2.5205		-0.51		2.567	-0.0465
1990	calc.by iis	2.6441		2.09		2.6377	0.0064
6156		-----		-----		-----	-----

normality	OK				
n	14		n	16	
outliers	0		outliers	0	
mean (n)	2.5449		mean (n)	2.6196	-0.0747
st.dev. (n)	0.07610				
R(calc.)	0.2131				
st.dev.(D4294:16e1)	0.04738				
R(D4294:16e1)	0.1327				

Difference = calculated – reported Sulfur

Kernel Density
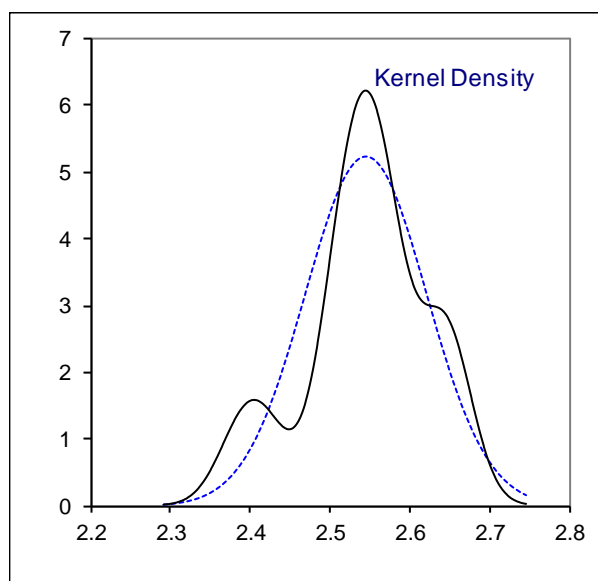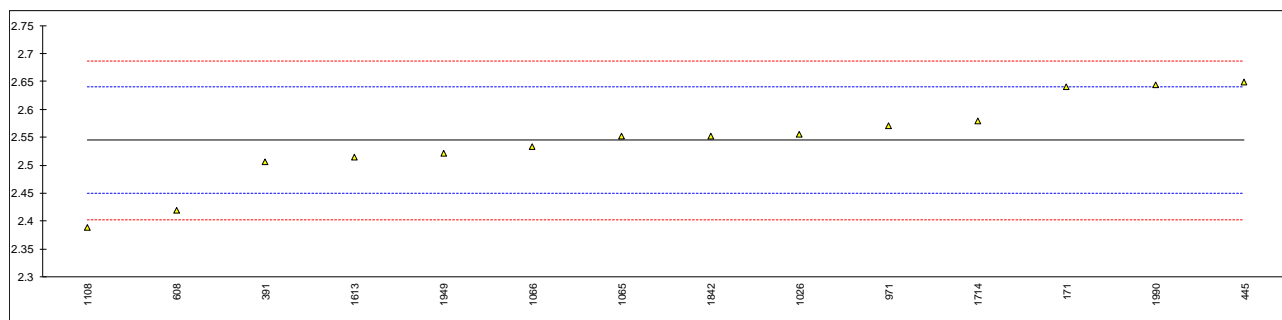
Determination of calculated (theoretical) Nitrogen on sample #19225; results in mg/kg

lab	method	calculated	mark	z(targ)	remarks	reported	difference
171	calc.by iis	1.16	ex	-10.52	not enough data	-----	-----
339		-----		-----		-----	-----
391		-----		-----		-----	-----
442		-----		-----		-----	-----
445	calc.by iis	1573.61		2.05		1580	-6.4
608		-----		-----		-----	-----
862		-----		-----		-----	-----
971	calc.by iis	1314.39		-0.02		1266	48.4
1011		-----		-----		-----	-----
1026	calc.by iis	943.94		-2.98		907.22	36.7
1065	calc.by iis	1528.79		1.70		1200	328.8
1066	calc.by iis	1247.17		-0.56		1220	27.2
1067		-----		-----		-----	-----
1089		-----		-----		-----	-----
1108	calc.by iis	1372.14		0.44		1311	61.1
1412		-----		-----		-----	-----
1455		-----		-----		-----	-----
1613		-----		-----		-----	-----
1714	calc.by iis	1178.76		-1.10		1210	-31.2
1720		-----		-----		-----	-----
1842	calc.by iis	1448.11		1.05		1500	-51.9
1949	calc.by iis	1243.06		-0.59		1241	2.1
1990	calc.by iis	1.22	ex	-10.52	not enough data	-----	-----
6156		-----		-----		-----	-----

normality	OK				
n	9		n	9	
outliers	0 + 2ex		outliers	0	
mean (n)	1316.66		mean (n)	1270.58	46.08
st.dev. (n)	193.510				
R(calc.)	541.83				
st.dev.(D5762:18a)	125.083				
R(D5762:18a)	350.23				

Difference = calculated – reported Nitrogen
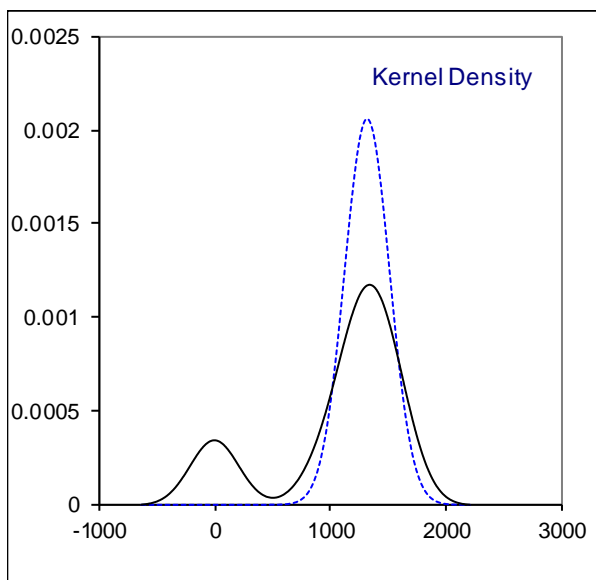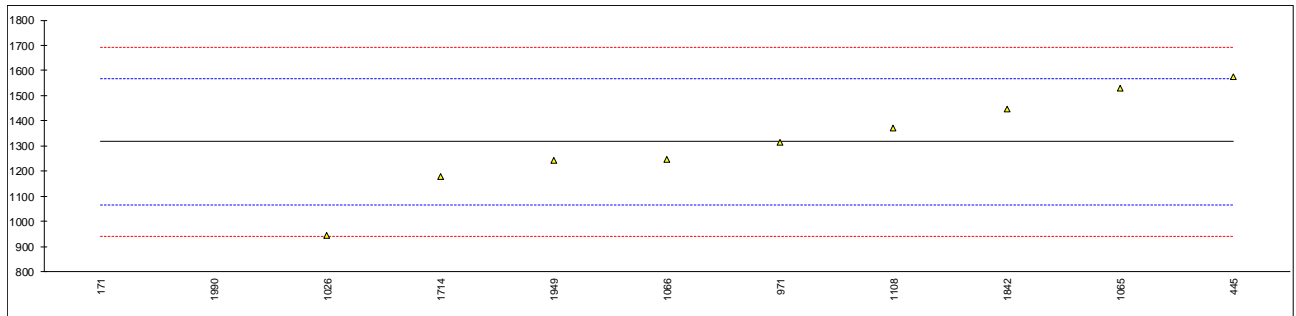
Determination of P(iP)NA on distillation fraction 2 (light Naphtha, 30-90°C); results in %V/V

lab	method	total par.	C1-C4	n-par.	i-par.	naphth.	arom.	remarks
171	D6730Mod.	89.664	0.462	51.035	38.629	9.208	1.103	
339		-----	-----	-----	-----	-----	-----	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D6839	88.92	0.44	49.17	39.75	9.85	1.22	
608		-----	-----	-----	-----	-----	-----	
862		-----	-----	-----	-----	-----	-----	
971	D6730	88.02	0.20	48.62	39.40	10.40	1.46	
1011		-----	-----	-----	-----	-----	-----	
1026	ISO22854	90.13	0.22	49.53	40.60	8.72	1.15	
1065	D5443	89.557	2.73	50.283	39.274	9.228	1.215	
1066	D6839	88.32	1.40	49.14	39.18	10.31	1.37	
1067		-----	-----	-----	-----	-----	-----	
1089		-----	-----	-----	-----	-----	-----	
1108	D5443	86.46	0.68	47.14	39.32	9.26	**4.28**	
1412		-----	-----	-----	-----	-----	-----	
1455		-----	-----	-----	-----	-----	-----	
1613	D6839	88.66	1.37	-----	-----	9.89	1.39	
1714		-----	-----	-----	-----	-----	-----	
1720		-----	-----	-----	-----	-----	-----	
1842	D5443	90.09	0.32	50.28	39.81	8.87	1.07	
1949	D5443	90.21	2.59	-----	-----	8.75	1.04	
1990	D6839	90.39	1.54	49.56	40.83	8.45	1.15	
6156		-----	-----	-----	-----	-----	-----	
	normality	OK	OK	suspect	OK	OK	OK	
	n	11	11	9	9	11	10	
	outliers	0	0	0	0	0	1	
	mean (n)	89.129	1.087	49.418	39.644	9.358	1.217	
	st.dev. (n)	1.2010	0.9189	1.1238	0.6988	0.6637	0.1443	
	R(calc.)	3.363	2.573	3.147	1.957	1.858	0.404	

NB. Bold and underlined figures are statistical outliers
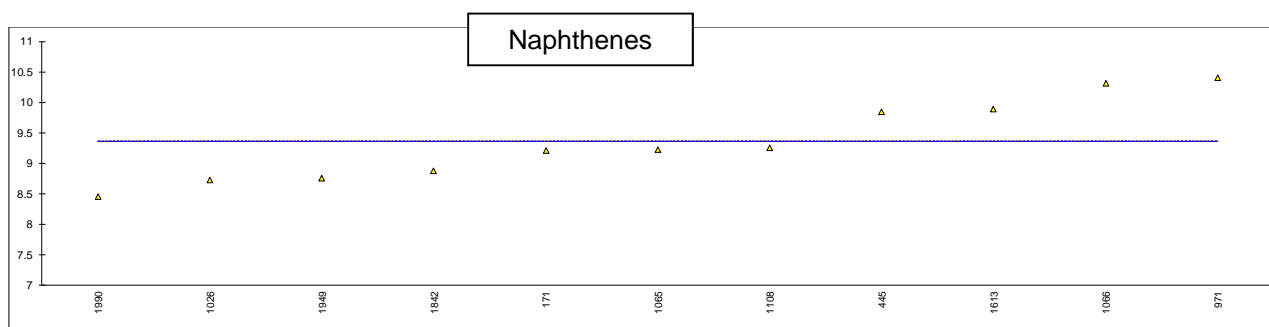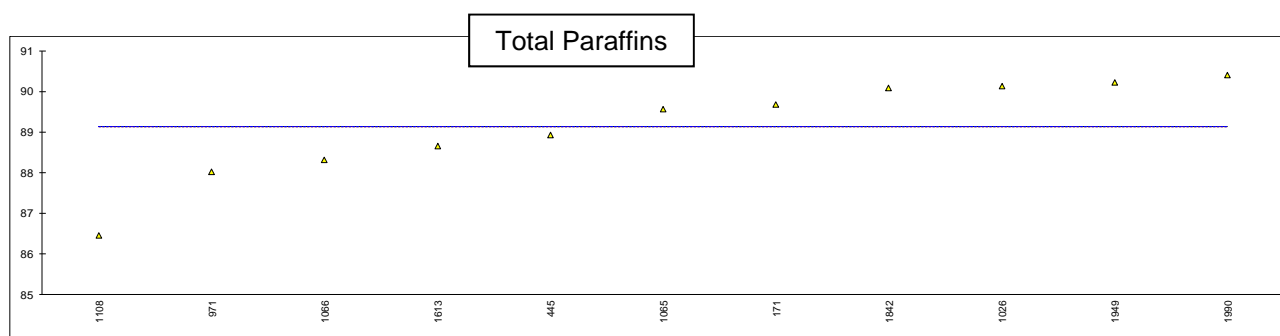
Determination of P(iP)NA on distillation fraction 2 (light Naphtha, 30-90°C); results in %M/M

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D6730Mod.	88.001	0.395	49.707	38.294	10.509	1.459	
339		-----	-----	-----	-----	-----	-----	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D6839	87.21	0.38	47.90	39.31	11.18	1.61	
608		-----	-----	-----	-----	-----	-----	
862		-----	-----	-----	-----	-----	-----	
971	D6730	86.14	0.18	47.71	38.43	11.86	1.89	
1011		-----	-----	-----	-----	-----	-----	
1026	ISO22854	88.52	0.20	48.38	40.14	9.96	1.52	
1065	D5443	87.843	2.37	48.961	38.882	10.548	1.608	
1066	D6839	86.53	1.22	47.78	38.75	11.67	1.78	
1067		-----	-----	-----	-----	-----	-----	
1089		-----	-----	-----	-----	-----	-----	
1108	D5443	83.93	0.59	45.6	38.33	10.52	**5.55**	
1412		-----	-----	-----	-----	-----	-----	
1455		-----	-----	-----	-----	-----	-----	
1613	D6839	86.48	1.18	-----	-----	11.25	1.82	
1714	D5443	86.25	2.88	47.35	38.90	11.83	1.91	
1720		-----	-----	-----	-----	-----	-----	
1842	D5443	**80.09**	0.28	49.09	39.31	10.18	1.42	
1949	D5443	88.47	2.22	-----	-----	10.15	1.39	
1990	D6839	88.79	1.35	48.41	40.38	9.68	1.53	
6156		-----	-----	-----	-----	-----	-----	
	normality	suspect	OK	suspect	OK	OK	OK	
	n	11	12	10	10	12	11	
	outliers	1	0	0	0	0	1	
	mean (n)	87.106	1.104	48.089	39.073	10.778	1.631	
	st.dev. (n)	1.4327	0.9402	1.1345	0.7228	0.7553	0.1893	
	R(calc.)	4.012	2.632	3.177	2.024	2.115	0.530	

NB. Bold and underlined figures are statistical outliers
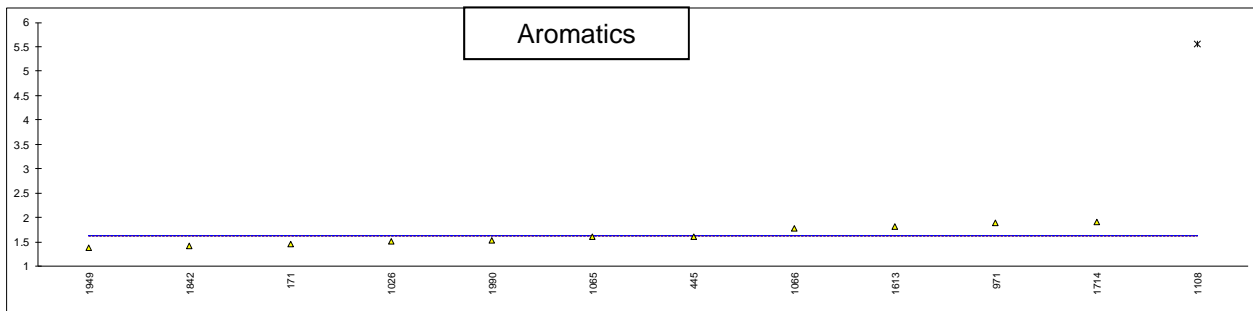
Determination of P(iP)NA on distillation fraction 3 (heavy Naphtha, 90-180°C); results in %V/V

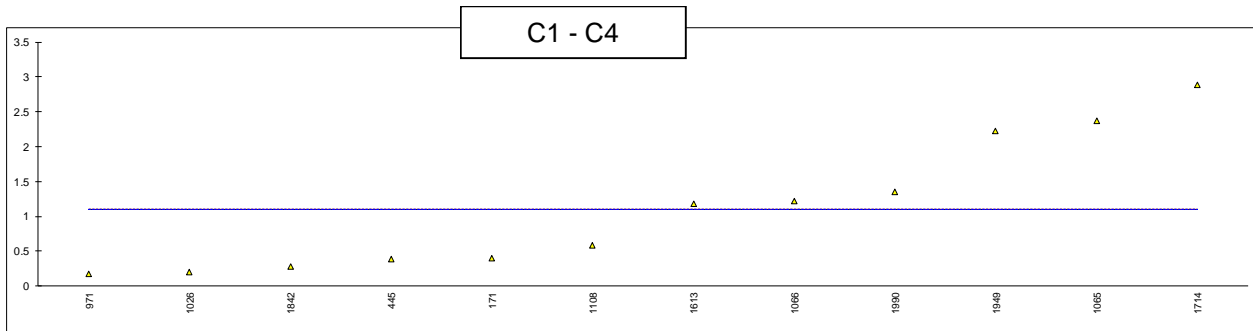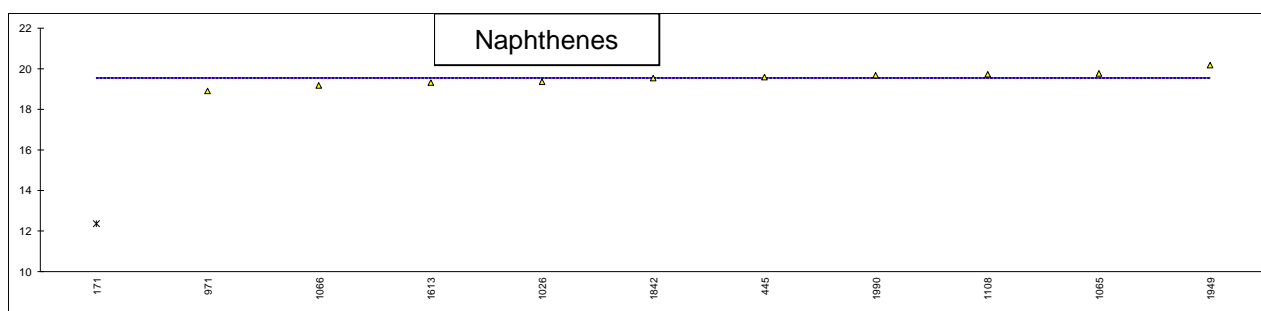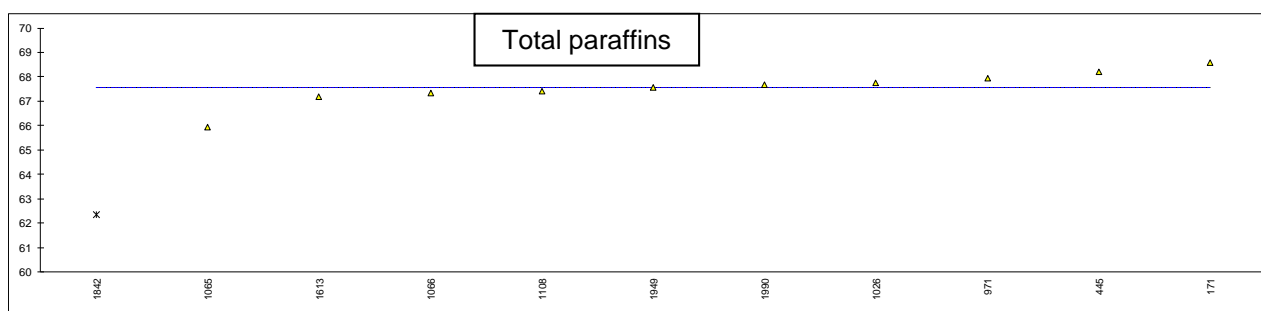| lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D6730Mod.	68.587	0.019	32.327	36.260	**12.377**	14.002	
339		-----	-----	-----	-----	-----	-----	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D6839	68.21	<0.10	32.09	36.12	19.58	12.21	
608		-----	-----	-----	-----	-----	-----	
862		-----	-----	-----	-----	-----	-----	
971	D6730	67.95	0.03	32.05	35.90	18.90	13.00	
1011		-----	-----	-----	-----	-----	-----	
1026	ISO22854	67.76	0.00	31.31	36.45	19.33	12.40	
1065	D5443	65.955	**0.24**	32.166	**33.789**	19.748	14.226	
1066	D6839	67.35	<0.01	31.25	36.10	19.17	12.76	
1067		-----	-----	-----	-----	-----	-----	
1089		-----	-----	-----	-----	-----	-----	
1108	D5443	67.41	0.06	31.33	36.08	19.72	12.94	
1412		-----	-----	-----	-----	-----	-----	
1455		-----	-----	-----	-----	-----	-----	
1613	D6839	67.19	0.0	-----	-----	19.29	13.52	
1714		-----	-----	-----	-----	-----	-----	
1720		-----	-----	-----	-----	-----	-----	
1842	D5443	**62.36**	<0.01	31.12	36.24	19.54	12.63	
1949	D5443	67.57	**0.19**	-----	-----	20.16	12.28	
1990	D6839	67.67	0.07	31.34	36.33	19.68	12.65	
6156		-----	-----	-----	-----	-----	-----	
	normality	not OK	unknown	OK	OK	OK	OK	
	n	10	6	9	8	10	11	
	outliers	1	2	0	1	1	0	
	mean (n)	67.565	0.030	31.665	36.185	19.512	12.965	
	st.dev. (n)	0.7040	0.0297	0.4784	0.1705	0.3538	0.6773	
	R(calc.)	1.971	0.083	1.340	0.478	0.991	1.896	

NB. Bold and underlined figures are statistical outliers

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	D6730Mod.	65.448	0.013	30.580	34.868	**12.888**	16.425	
339		-----	-----	-----	-----	-----	-----	
391		-----	-----	-----	-----	-----	-----	
442		-----	-----	-----	-----	-----	-----	
445	D6839	65.11	<0.10	30.41	34.70	20.60	14.29	
608		-----	-----	-----	-----	-----	-----	
862		-----	-----	-----	-----	-----	-----	
971	D6730	64.59	0.03	30.42	34.17	20.01	15.22	
1011		-----	-----	-----	-----	-----	-----	
1026	ISO22854	64.63	0.00	29.62	35.01	20.35	14.5	
1065	D5443	**62.612**	0.18	30.357	**32.255**	20.697	16.691	
1066	D6839	64.16	<0.01	29.56	34.60	20.09	14.88	
1067		-----	-----	-----	-----	-----	-----	
1089		-----	-----	-----	-----	-----	-----	
1108	D5443	64.41	0.05	29.50	34.91	20.67	15.00	
1412		-----	-----	-----	-----	-----	-----	
1455		-----	-----	-----	-----	-----	-----	
1613	D6839	63.92	0.0	-----	-----	20.16	15.70	
1714	D5443	**67.49**	0.09	30.84	36.65	**15.93**	16.39	
1720		-----	-----	-----	-----	-----	-----	
1842	D5443	64.32	<0.01	29.29	35.03	20.44	14.69	
1949	D5443	64.59	0.14	-----	-----	21.13	14.28	
1990	D6839	64.48	0.06	29.63	34.85	20.73	14.78	
6156		-----	-----	-----	-----	-----	-----	
	normality	OK	OK	OK	not OK	OK	OK	
	n	10	9	10	9	10	12	
	outliers	2	0	0	1	2	0	
	mean (n)	64.566	0.063	30.021	34.976	20.488	15.237	
	st.dev. (n)	0.4414	0.0633	0.5516	0.6806	0.3458	0.8590	
	R(calc.)	1.236	0.177	1.545	1.906	0.968	2.405	

NB. Bold and underlined figures are statistical outliers
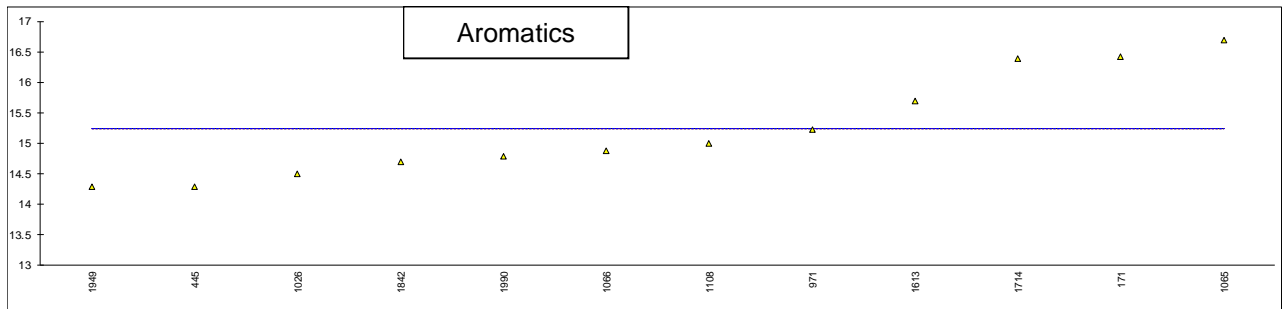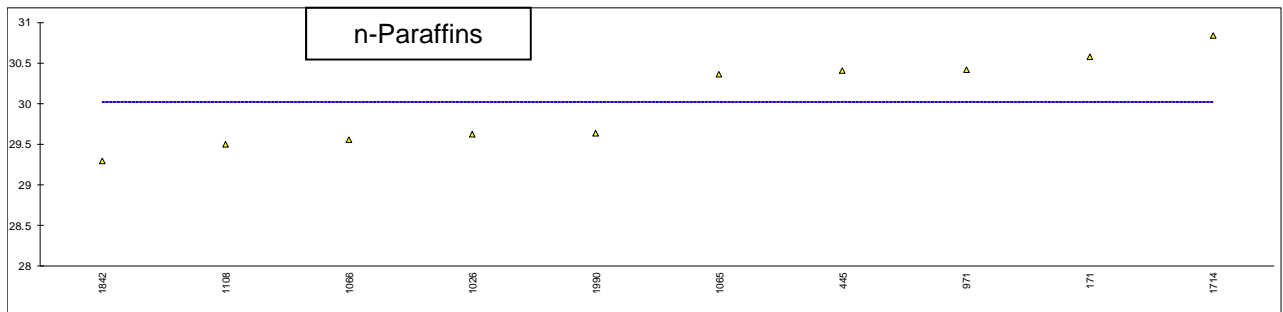
Determination of Flash Point on combined distillation fractions 5 and 6, results in °C

lab	method	value	mark	z(targ)	remarks
171	D93	100.5		-0.37	
339		-----		-----	
391		-----		-----	
442		-----		-----	
445	D93	99.0		-0.95	
608	D93	102.0		0.21	
862		-----		-----	
971	D93-A	105.0		1.38	
1011		-----		-----	
1026	D93-A	98.0		-1.34	
1065		-----		-----	
1066	D93	104.5		1.19	
1067		-----		-----	
1089		-----		-----	
1108	D93	100.0		-0.56	
1412		-----		-----	
1455		-----		-----	
1613	D93	103.5		0.80	
1714	D93	102.5		0.41	
1720		-----		-----	
1842		-----		-----	
1949	D93-A	86.5	D(0.01)	-5.81	
1990	D93-A	99.5		-0.76	
6156		-----		-----	

normality	OK	
n	10	
outliers	1	
mean (n)	101.45	
st.dev. (n)	2.409	
R(calc.)	6.74	
st.dev.(D93-A:19)	2.572	
R(D93-A:19)	7.20	
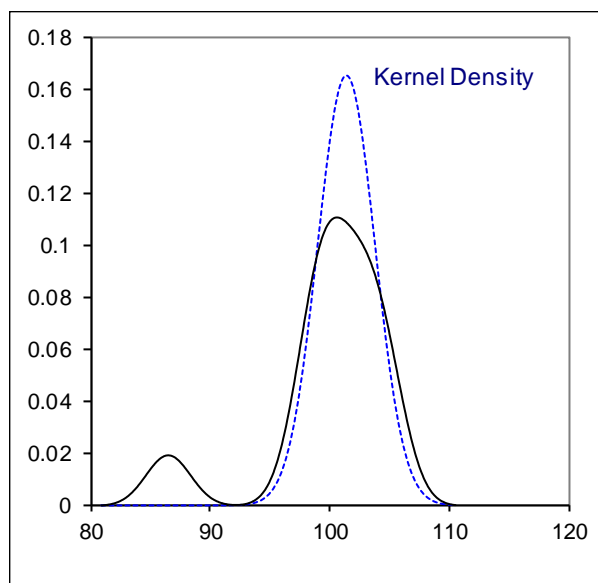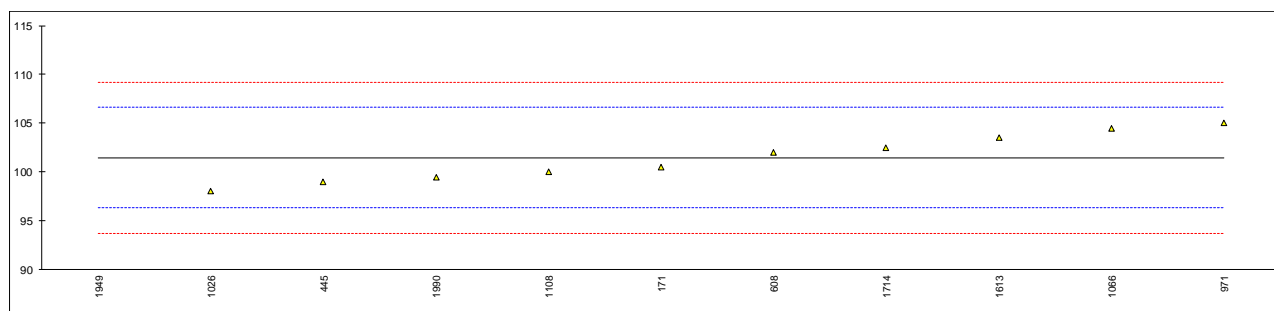
Determination of Distillation on combined fractions 5 and 6, results in °C

lab	method	IBP	5% rec.	10% rec.	50% rec.	90% rec.	95% rec.	FBP	%recov	%resid	%loss*
171	D86-Manual	230	238	240	254	280	286	293	98	1.4	0.6
339		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
391		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
442		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
445	D86	232.4	240.5	241.0	256.0	279.6	289.6	297.6	99.0	0.5	0.5
608	D86	230.1	241.6	244.0	257.7	282.7	287.9	292.3	98.3	1.3	0.4
862		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
971		228.4	241.1	243.1	257.1	282.7	289.6	297.2	99.6	0.2	0.2
1011		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1026	ISO3405	225.0	232.0	233.5	246.0	275.6	282.2	288.6	98.6	1.4	0
1065	D7345	**205.3**	**222.5**	230.2	252.4	275.5	281.5	288.2	98.1	1.4	0.5
1066	D86	229.9	240.7	242.6	257.5	282.7	288.3	293.6	98.5	1.1	0.4
1067		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1089		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1108	D86	222.9	238.5	241.6	259.4	285.9	291.7	297.8	98.5	1.3	0.2
1412		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1455		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1613	D86	229.6	238.5	240.6	255.0	280.7	287.6	296.4	98.5	1.3	0.2
1714	D86	234.3	241.9	243.8	259.6	285.8	291.3	297.6	98.4	0.9	0.7
1720		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1842	D86	229.1	238.5	241.7	255.0	280.0	285.8	293.2	98.7	1.1	0.2
1949		221.0	229.0	235.0	249.0	276.5	283.0	296.0	99.0	0.5	0.5
1990	D86	226.3	236.3	238.0	251.2	279.1	285.0	290.4	-----	-----	-----
6156		-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
	normality	OK	suspect	OK	OK	OK	OK	OK			
	n	12	12	13	13	13	13	13			
	outliers	1	1	0	0	0	0	0			
	mean (n)	228.25	238.05	239.62	254.61	280.52	286.88	293.99			
	st.dev. (n)	3.828	3.953	4.273	4.048	3.417	3.323	3.426			
	R(calc.)	10.72	11.07	11.96	11.34	9.57	9.30	9.59			
	st.dev.(D86:19)	(4.483)	(2.551)	(1.883)	(1.071)	(1.503)	(2.153)	(2.536)			
	R(D86:19)	(12.55)	(7.14)	(5.27)	(3.00)	(4.21)	(6.03)	(7.10)			

NB. Bold and underlined figures are statistical outliers
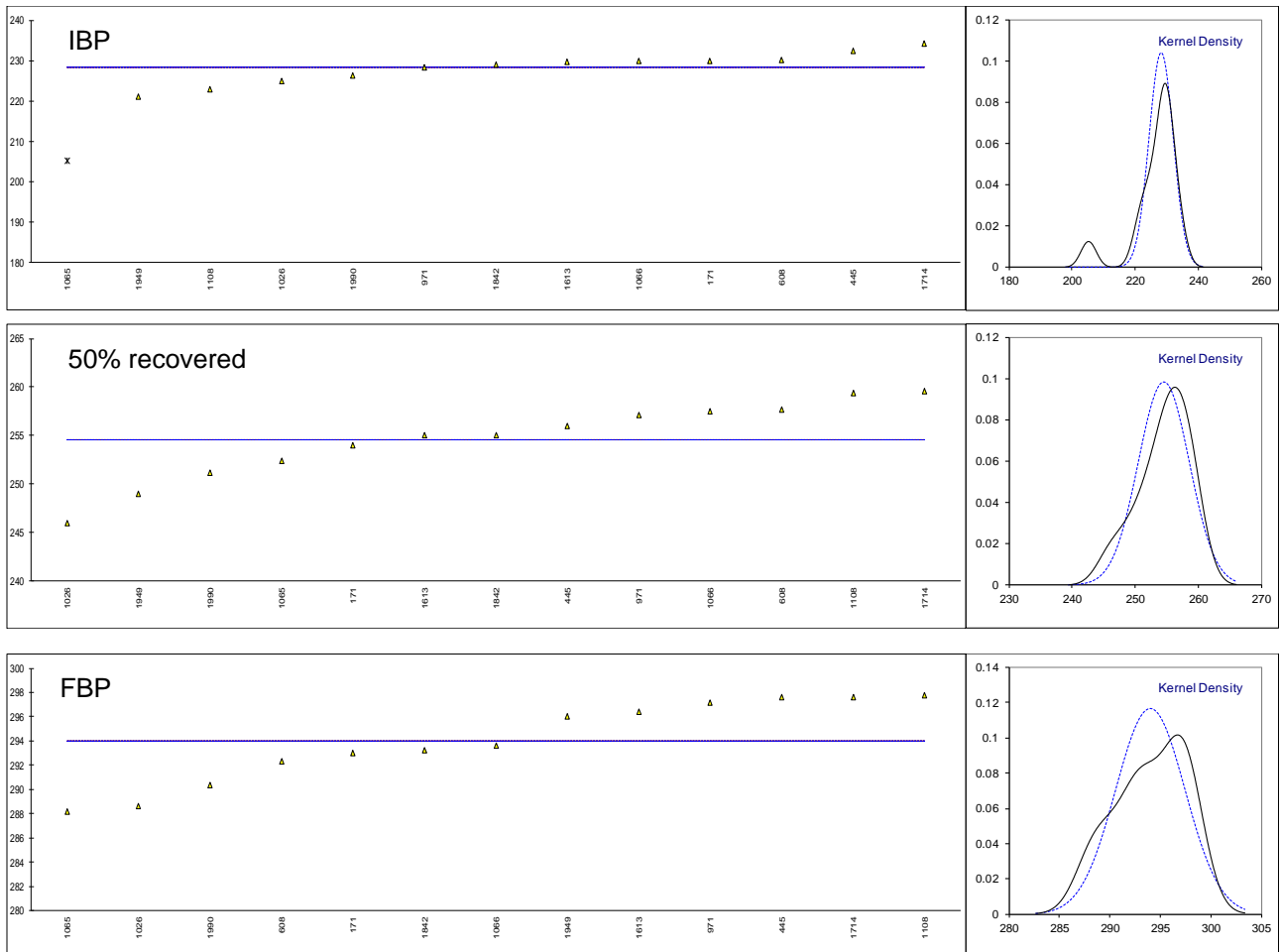
APPENDIX 1D – SIMULATED DISTILLATION AND EFFECTIVE CUT POINT D2892

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	total sum
171	D2887	19.4	23.7	27.2	14.4	6.5	0.0	0.0	91.2
339		-----	-----	-----	-----	-----	-----	-----	-----
391		-----	-----	-----	-----	-----	-----	-----	-----
442		-----	-----	-----	-----	-----	-----	-----	-----
445	D2887	17.0	18.5	28.0	19.5	9.7	0.0	0.0	92.7
608		13.6	16.5	33.3	18.3	13.8	0.0	0.0	95.5
862		-----	-----	-----	-----	-----	-----	-----	-----
971		15.3	20.7	27.2	17.2	11.1	1.9	0.0	93.5
1011		-----	-----	-----	-----	-----	-----	-----	-----
1026	D2887	18.6	20.4	26.5	15.5	10.0	0.0	0.0	90.9
1065	D6352	14.8	20.0	24.0	22.0	13.5	0.0	0.0	94.3
1066	D2887	13.5	16.5	29.5	16.5	15.4	3.3	0.0	94.7
1067		-----	-----	-----	-----	-----	-----	-----	-----
1089		-----	-----	-----	-----	-----	-----	-----	-----
1108		-----	-----	-----	-----	-----	-----	-----	-----
1412		-----	-----	-----	-----	-----	-----	-----	-----
1455		-----	-----	-----	-----	-----	-----	-----	-----
1613		-----	-----	-----	-----	-----	-----	-----	-----
1714	D2887	10.8	13.6	27.2	16.9	18.3	4.8	0.0	91.7
1720		-----	-----	-----	-----	-----	-----	-----	-----
1842		-----	-----	-----	-----	-----	-----	-----	-----
1949	D7169	19.0	17.0	**18.7 G(0.05)**	10.8	8.7	3.8	0.0	**78.0**
1990		14.7	16.5	30.0	17.0	14.3	0.0	0.0	92.4
6156		-----	-----	-----	-----	-----	-----	-----	-----
	normality	OK	OK	suspect	suspect	OK	n.a.	n.a.	
	n	10	10	9	10	10	10	10	
	outliers	0	0	1	0	0	n.a.	n.a.	
	mean (n)	15.67	18.33	28.10	16.82	12.11	<5	0	
	st.dev. (n)	2.784	2.897	2.594	2.983	3.548	n.a.	n.a.	
	R(calc.)	7.80	8.11	7.26	8.35	9.93	n.a.	n.a.	

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

lab	220-230°C	230-240°C	240-250°C	250-260°C	260-270°C	270-280°C	280-290°C	290-300°C	300-310°C	total sum
171	0.7	2.3	7.3	13.4	17.8	15.4	16.5	13.75	10.69	97.7
339	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
391	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
442	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
445	0.0	2.3	4.0	14.2	13.5	18.5	16	15.5	12	96.0
608	0.0	**0.0 G(0.05)**	1.9	9.1	12.69	18.14	19	14.28	17.11	92.2
862	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
971	0.7	1.6	5.4	10.5	13.82	17.29	17.06	13.87	12.9	93.1
1011	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1026	0.3	2.1	4.7	13.0	16	15.83	17.5	14.27	12.9	96.5
1065	0.0	1.9	6.5	13.1	15.5	19.7	16.3	16.4	7.93	97.3
1066	0.0	1.9	3.4	10.7	12.5	17.5	18	15	14.33	93.3
1067	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1089	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1108	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1412	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1455	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1613	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1714	0.6	1.9	3.2	8.3	10.89	16.68	17.54	13.07	14.57	**86.8**
1720	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1842	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1949	2.6	**6.7 G(0.05)**	8.7	13.0	13.5	16.5	16.5	10.83	8.67	97.0
1990	0.0	1.6	3.4	11.0	12.5	17.5	16.5	16.5	14.5	93.5
6156	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
norm.	n.a.	OK	OK	OK	OK	OK	OK	suspect	OK	
n	10	8	10	10	10	10	10	10	10	
outliers	n.a.	2	0	0	0	0	0	0	0	
mean	<1	1.93	4.85	11.62	13.87	17.30	17.10	14.35	12.56	
st.dev.	n.a.	0.267	2.107	1.979	2.026	1.275	0.921	1.673	2.833	
R(calc)	n.a.	0.75	5.90	5.54	5.67	3.57	2.58	4.69	7.93	

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	227-263 = 36°C	245.5	-4.5	OK	
339	-----	-----	-----	-----	
391	-----	-----	-----	-----	
442	-----	-----	-----	-----	
445	231-262 = 31°C	249.7	-0.3	OK	
608	245-266 = 21°C	254.3	4.3	OK	narrow overlap of cuts
862	-----	-----		-----	
971	227-270 = 43°C	249.1	-0.9	OK	large overlap of cuts
1011	-----	-----		-----	
1026	229-265 = 36°C	248.2	-1.8	OK	
1065	235-263 = 28°C	252.4	2.4	OK	
1066	230-271 = 41°C	252.4	2.4	OK	
1067	-----				
1089	-----				
1108	-----				
1412	-----				
1455	-----				
1613	-----				
1714	227-272 = 45°C	254.7	4.7	OK	large overlap of cuts
1720	-----				
1842	-----				
1949	226-271 = 45°C	241.4	-8.6	Not OK	total sum SimDist fr.5 low
1990	231-267 = 36°C	250.0	-0.0	OK	
6156	-----				

* Acc. To ASTM D2892:19, appendix X2.6.5.1 the difference between ECP and AET should not exceed 0.7R °C (0.7 x 8 = 6°C)

Determination of Standard Efficiency $N_{minimum}$ from the SimDist data

| Lab	N_{actual}	$N_{minimum}$	Requirement $N_{minimum}$*	Remarks
171	8.9	7.7	Not OK	
339	-----	-----	-----	
391	-----	-----	-----	
442	-----	-----	-----	
445	8.8	6.6	OK	
608	9.1	7.8	Not OK	Fraction 6 distilled at (too) high Obs. Vapor Temperature of 230.6°C?**
862	-----	-----	-----	
971	7.8	6.7	OK	
1011	-----	-----	-----	
1026	8.2	7.1	OK	
1065	5.4	4.7	Not OK	Fraction 6 distilled at (too) low pressure of 1 mmHg?**
1066	7.4	6.4	OK	
1067	-----	-----	-----	
1089	-----	-----	-----	
1108	-----	-----	-----	
1412	-----	-----	-----	
1455	-----	-----	-----	
1613	-----	-----	-----	
1714	6.6	5.7	Not OK	Total sum SimDist fraction 6 (too) low?
1720	-----	-----	-----	
1842	-----	-----	-----	
1949	3.9	3.4	Not OK	Total sum SimDist fraction 5 (too) low?
1990	8.4	not determined	-----	Did not report Observed Vapor Temperatures
6156	-----	-----	-----	

* Acc. To ASTM D2892:19, appendix X2.5.9.2 the acceptable upper and lower limits (14-18 theoretical plates) are 5.8, resp. 7.4
** ASTM D2892 describes three steps: first atmospheric distillation with a maximum Observed Vapor Temperature of 210°C (paragraph 10.3). When further cuts have to be distilled the temperature is reduced by reducing the pressure to 100 mmHg (paragraph 10.4) and if necessary to further stay below 210°C also at lower pressure, but not lower than 2 mmHg (paragraph 10.5).
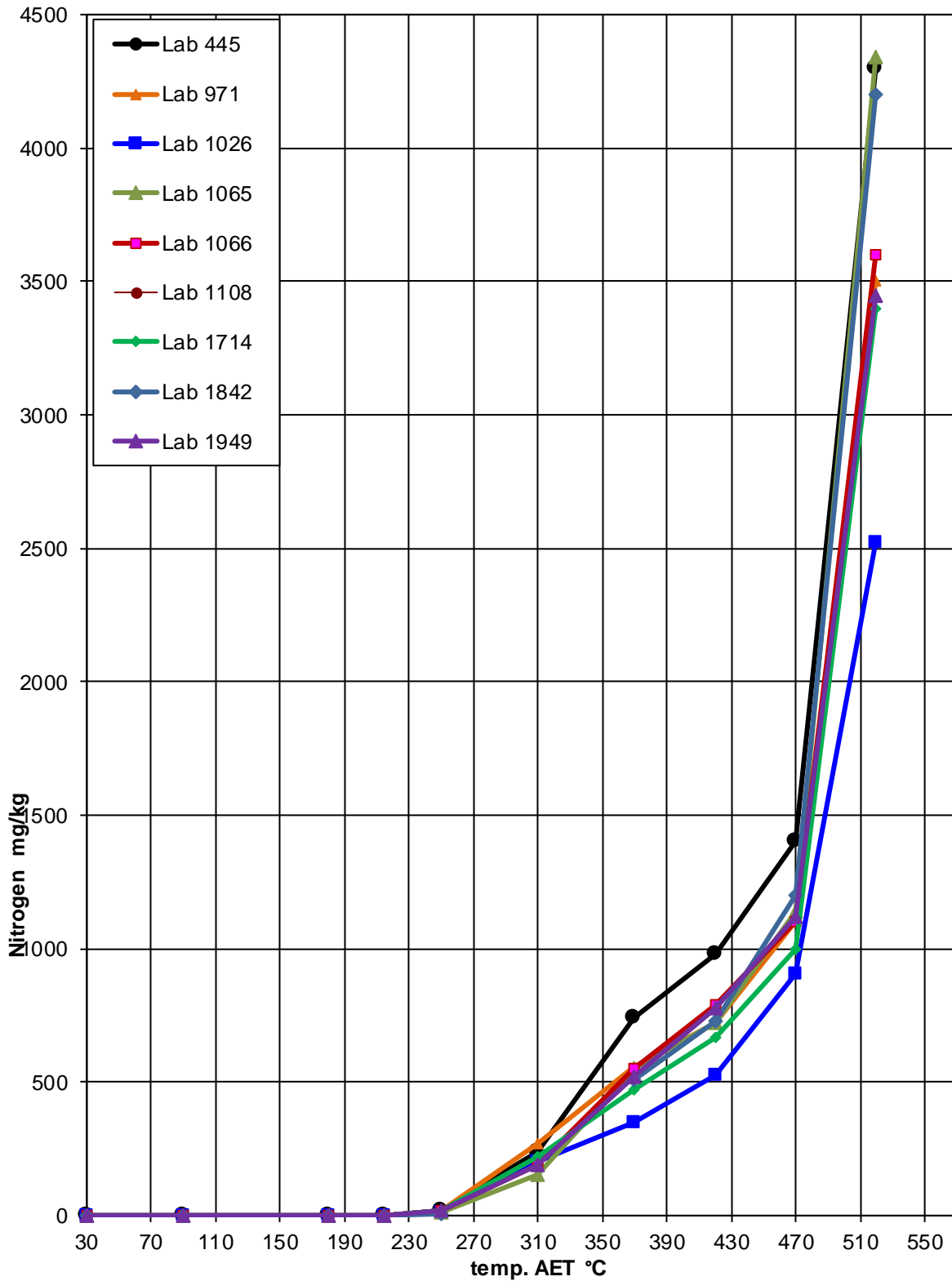
APPENDIX 2   True boiling point curve, cumulative %M/M *vs* temperature AET in °C
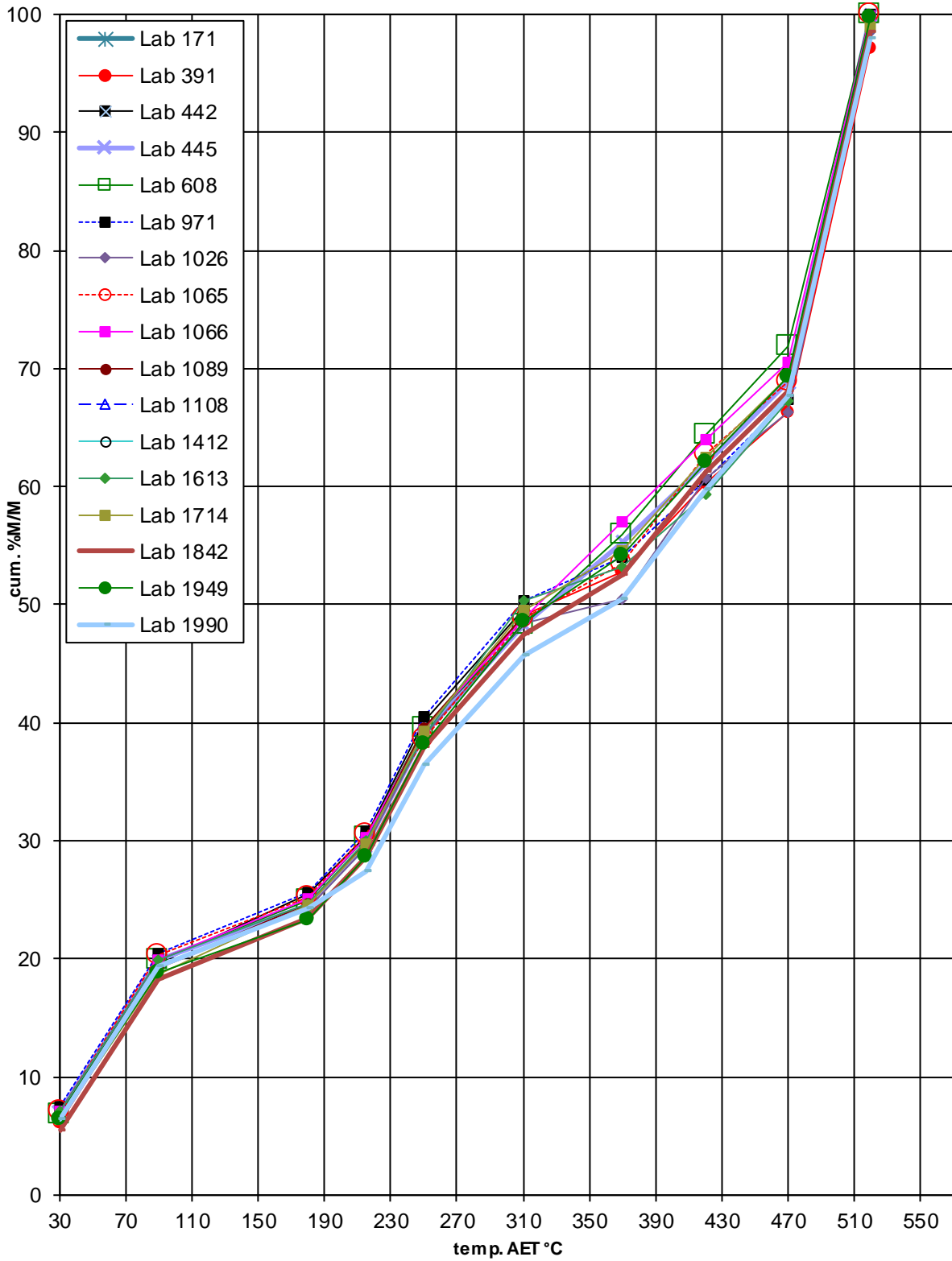
APPENDIX 3 True boiling point curve D2892, Sulfur in %M/M *vs* temperature AET in °C
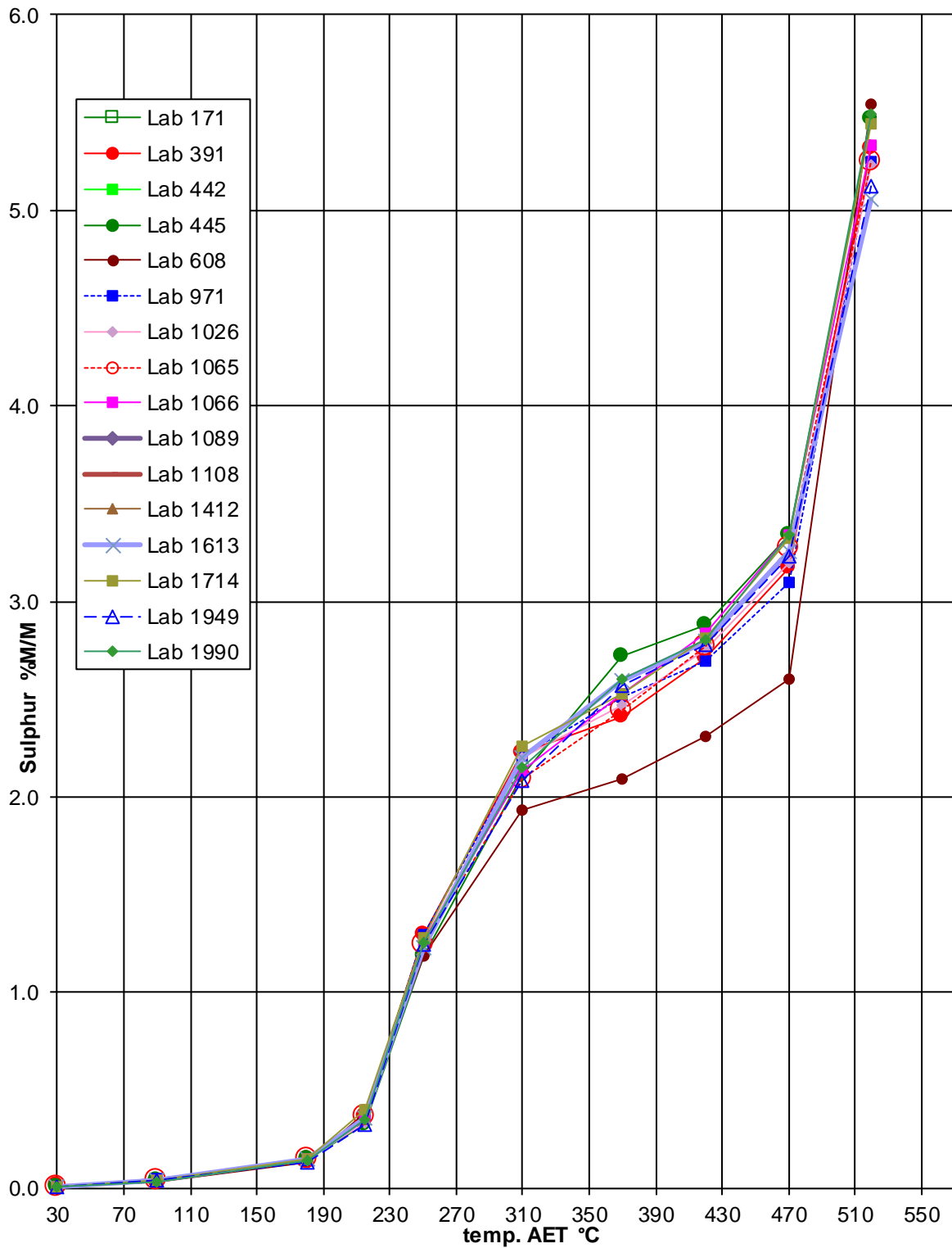
APPENDIX 4 True boiling point curve D2892, Nitrogen in mg/kg *vs* temperature AET in °C

APPENDIX 5

Details of Distillation

Lab	Sample received	Distillation started	Results reported	Intake in L	Mass charged in grams	Volume charged in mL	End Point of distillation(s) in °C
171	26-11-19	09-12-19	24-01-20	6	3410.0	3904.6	370+
339	-----	-----	-----	-----	-----	-----	-----
391	15-11-19	28-11-19	06-12-19	10	4710.3	5391.3	520+
442	08-11-19	18-11-19	06-12-19	22	5296.0	6064.4	370+
445	08-11-19	02-12-19	14-01-20	40	8720	9990	520+
608	27-11-19	29-11-19	23-12-19	8	4113.8	4712.3	520+
862	-----	-----	-----	-----	-----	-----	-----
971	11-11-19	19-11-19	08-12-19	22	8183.0	9367.0	520+
1011	-----	-----	-----	-----	-----	-----	-----
1026	08-11-19	13-11-19	13-12-19	20	7474.7	8556.2	520+
1065	17-1119	20-11-19	05-12-19	10	3851	4411	520+
1066	11-11-19	15-11-19	06-12-19	20	7458	8540	520+
1067	-----	-----	-----	-----	-----	-----	-----
1089	14-11-19	03-12-19	06-12-19	6	2642.3	3023.6	370+
1108	06-11-19	13-11-19	06-12-19	6	3052	3493	370+
1412	08-11-19	28-11-19	06-12-19	12	6898	7893.8	370+
1455	-----	-----	-----	-----	-----	-----	-----
1613	21-11-19	02-12-19	08-12-19	5.0	3494	4000	520+
1714	04-11-19	12-11-19	05-12-19	6	4261.8	4880.1	520+
1720	-----	-----	-----	-----	-----	-----	-----
1842	06-11-19	13-11-19	09-12-19	6	3508.6	4018.1	520+
1949	25-11-19	27-11-19	18-12-19	10	5889.5	6744.0	520+
1990	20-11-19	27-11-19	10-01-20	20	12870	14703.5	520+
6156	-----	-----	-----	-----	-----	-----	-----

Details of Pressure during distillation ASTM D2892 and ASTM D5236

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	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C
171	757	756	756	99.9	100.0	10.31	10.23	n	n	n
339	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
391	763.0	763	763	100.0	100.0	2.0	2.0	0.2	0.2	0.2
442	759.3	759.1	758.6	100	100.0	9.53	9.54	n	n	n
445	776.9	776.6	776.4	**10****	10	10	10	0.400	0.400	0.35
608	760	760	760	100	100.0	**100****	3	1	0.8	0.3
862	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
971	760	760	760	100	100.0	5	5	0.1	0.1	0.1
1011	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1026	765	748	748	100	100.0	2	2	1.00	0.91	0.28
1065	744.0	743.7	742.0	98.7	99.1	**1.00****	**1.00****	1.00	1.00	1.00
1066	751.67	751.59	750.90	100.00	100.03	5.01	5.00	1	1	1
1067	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1089	754.4	754.0	753.6	100.0	100.0	10.0	5.0	n	n	n
1108	-----	755	755	100	10	10	3	n	n	n
1412	760	760	760	100	100	10	10	n	n	n
1455	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1613	760	760	760	100	100	**100****	10	1.0	1.0	1.0
1714	**757***	**757***	**757***	100	100	10	10	1.0	1.0	0.28
1720	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
1842	749.6	749.5	748.2	100	100	10	10	1.0	1.0	0.2
1949	749	749	749	100	100	2	2	1	1	1
1990	760	760	760	100	100.0	10	10	1	0.7	0.2
6156	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

*) lab 1714: reported 1009 (in hPa?) = 757 mmHg
**) lab 445: dropped pressure from 776 to 10 mmHg, without 100 mmHg step (ASTM D2892, paragraph 10.4)
***) lab 608 and lab 1613: Observed Vapor Temperature in this step >210°C (ASTM D2892, paragraph 10.3)
****) lab 1065: pressure below 2 mmHg (not allowed according to ASTM D2892, paragraph 10.5)

APPENDIX 6

Number of participants per country

    1 lab in AZERBAIJAN
    1 lab in CHINA, People's Republic
    1 lab in FRANCE
    1 lab in GREECE
    1 lab in ISRAEL
    1 lab in ITALY
    1 lab in JORDAN
   3 labs in MALAYSIA
   5 labs in NETHERLANDS
    1 lab in NORWAY
    1 lab in PORTUGAL
    1 lab in RUSSION 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 test result after checking of first reported suspect test result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
E = possibly an error in calculations
W = test result withdrawn on request of participant
ex = test result excluded from the statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported
SDS = safety data sheet

Literature

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