

Results of Proficiency Test
Crude Oil Assay
November 2015

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

Authors: ing. C.M. Nijssen-Wester
Correctors: dr. R.G. Visser & L. Sweere
Report: iis15R02 - revised

July 2016

SUMMARY OF CHANGES

This revised report replaces the original report iis15R02 of April 2016.

iis discovered that parts of Appendix 2 and 3 were missing from the report.

Therefore, the graph of True boiling point curve, cum%M/M vs temp AET in °C and the title for Appendix 3 were added to this revised report.

Therefore the following pages in this report have been revised:

- Contents on page 3 (pages were renumbered)
- Appendix 2 (page 44 in the original report)
- Appendix 3 (page 44 in the original report)

CONTENTS

1	INTRODUCTION	5
2	SET UP	5
2.1	ACCREDITATION	5
2.2	PROTOCOL.....	5
2.3	CONFIDENTIALITY STATEMENT	5
2.4	SAMPLES	6
2.5	ANALYSES	6
3	RESULTS.....	7
3.1	STATISTICS	7
3.2	GRAPHICS	8
3.3	Z-SCORES	8
4	EVALUATION	9
4.1	EVALUATION PER TEST	10
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	14
4.3	COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2015 WITH PREVIOUS PTS.....	17
4.4	DISCUSSION.....	18
Appendices:		
1.	Data and statistical results, result tables	19
	A – Analysis of original sample	20
	B - Distillation results D2892 and D5236	24
	C – Analysis of distillation fractions	30
	D – Simulated distillation and Effective Cut Point D2892	44
2.	True Boiling Point curve, cum%M/M vs temp AET in °C.....	46
3.	True Boiling Point curve, Sulphur in %M/M vs temp AET in °C.....	47
4.	True Boiling Point curve, Nitrogen in mg/kg vs temp AET in °C.....	48
5.	Details of Distillation	49
6.	Number of participants per country	50
7.	Abbreviations and literature	51

- empty page

1 INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies organizes a proficiency test for Crude Oil Assay. During the annual proficiency test program of 2015/2016, it was decided to continue the proficiency test for the analysis for Crude Oil Assay.

In this interlaboratory study 25 laboratories in 18 different countries have participated. See appendix 6 for the number of participants per country.

In this report, the results of the 2015 proficiency test for Crude Oil Assay are presented and discussed. This report is electronically available through the iis internet site <http://www.iisnl.com>.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. It was decided to send samples of 5 L, labelled #15216. 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 April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis internet site 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. 200 litre of Crude Oil was homogenised in a metal drum. After homogenisation, the bulk material was divided over 40 metal cans of 5 liter and labelled #15216.

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

	<i>Density at 15 °C in kg/m³</i>
sample #15216-1	874.35
sample #15216-2	874.33
sample #15216-3	874.31
sample #15216-4	874.53
sample #15216-5	874.25
sample #15216-6	874.36
sample #15216-7	874.20
sample #15216-8	874.32

table 1: homogeneity test results of subsamples #15216

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

	<i>Density at 15 °C in kg/m³</i>
r (observed)	0.27
reference test method	ASTM D5002:13
0.3 * R (ref. test method)	1.08

table 2: evaluation of the density repeatability on subsamples #15216

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

To each of the participating laboratories one or more cans of 5 L (as required) were sent on October 21, 2015.

2.5 ANALYSES

The participants were requested to determine a Crude Oil Assay consisting of a True Boiling Point Distillation in accordance with ASTM D2892 (collection of 8 fractions) and subsequently a distillation in accordance with ASTM D5236 (collection of 5 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 kerosene and light gasoil a D86-distillation and on the individual fractions 4 (LGO) and 5 (MGO) also a simulated distillation determination was requested.

To get comparable results a detailed report form, on which the units and the standard methods were printed, was sent by e-mail together with a letter of instructions to each participant. A SDS and a form to confirm receipt of the samples was added to the sample package.

3 RESULTS

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

After the planned deadline, a reminder was sent to those laboratories that had not yet reported any 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 these tests (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.

3.1 STATISTICS

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

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 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 spread 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:

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

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

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

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

4 EVALUATION

In the proficiency test no major problems were encountered with sample dispatch. After the period of four weeks after sample dispatch thirteen participants had reported test results. Seven laboratories reported test results after the final reporting date, the last test results were submitted at the beginning of March 2016. Five 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. Regretfully 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.

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.

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

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

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

Looking at the test results of the laboratories on the fractions of VGO (520-565°C) and Residue >565°C, it was found that the distillation end point was determined differently among the participants. The method D5236 states in Note 4 and Note 5 that the distillation can be stopped when cracking of the oil starts appearing. Therefore these two fractions can be different for each participant. It was decided not to statistically evaluate the VGO (520-565°C) and Residue >565°C fractions. For the evaluation of the distillation the sum of these two fractions were taken.

During the execution of this PT, some ASTM test methods from this PT were updated (to a 2016 version). These updates did not change the precision data, so the 2016 method was used in this report.

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 problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D4294:16ε1.

Nitrogen: The nitrogen determination on the original crude sample was problematic for some laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5762:12.

Water: The water determination on the original crude sample was very problematic at the low level of 0.04%V/V. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D4377:11.

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 distillation (D2892), it is remarkable that two laboratories reported to have used a starting pressure below atmospheric. It concerns lab 574 with 560 mm Hg and lab 1543 with 720 mm Hg. Possibly typing errors were made. Seven out of twelve distillation test results for lab 574 and eight out of sixteen distillation test results were marked to be a statistical outlier according to Dixon, Grubbs or Rosner outlier tests. Therefore the distillation test results, which were not already marked as an outlier, were excluded from the statistical evaluation for labs 574 and 1543. Two laboratories reported to either using a different cutpoint between fraction 3 and 4 at 160°C (lab 442) or to not separating the LGO and MGO fraction (lab 1714). The test results of these laboratories for the respective fractions were excluded.

In total, twenty laboratories reported distillation test results. Three laboratories only performed the atmospheric distillation (D2892) and seventeen also the vacuum distillation (D5236). Ten laboratories were able to distil all fractions up to the residue >565°C. Seven laboratories reported cracking when distilling the last two fractions with end points varying from 520°C to 557.3°C. Not all laboratories reported separate test results for fraction 12 (520-565°C) and/or fraction 13 (>565°C), therefore it is decided to use the sum of both fractions in the evaluation of the distillation. Method D5236 allows a laboratory to stop the distillation when cracking occurs, see D5236 Note 4 and Note 5.

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

For the individual volume recoveries only seven recoveries could be taken into account due to the lack of available densities (mainly for fraction <30°C).

After calculation of the Total Volume recovery, two of the seven recoveries (labs 391 and 1990) were smaller than the Total Mass recovery. This is not to be expected as due to volume expansion a volume gain is to be expected rather than a volume loss, see paragraph 11.6 of ASTM D2892:15.

D2892: This distillation was problematic for a number of laboratories. In total fifteen statistical outliers were detected (=10%) and ten test results were excluded from the statistical evaluation. However, after exclusion of the suspect data, all calculated reproducibilities are in good agreement with the requirements of ASTM D2892:15.

D5236: This distillation may be problematic for a number of laboratories. In total five statistical outliers were detected (=7.7%) and three 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. Since the calculated reproducibility of both VGO (370-420°C) and VGO (120-470 °C) do not meet the requirements of ASTM D2892:15 and the other two fractions had to be summarized, it was decided not to assign z-scores to the test results of this distillation.

The starts of the true boiling point curves (cum%M/M vs temp AET) of the laboratories show a high resemblance (see Appendix 2). The curve of laboratory 574 shows a highly positive deviation most part of the curve (70°C-430°C). This could be caused by the lower pressure at the start of the distillation. The curve of laboratory 1543 shows a positive deviation at the upper part of the curve (370-550°C). The curve for laboratory 1990 shows a small negative deviation along the whole curve (30°C-570°C). Also visible is the fact that laboratory 1714 combined the LGO and MGO fractions.

The true boiling point curves (S in %M/M vs temp AET) of most laboratories show a high resemblance (see Appendix 3). The curves for laboratories 574, 1543 and 1613 show a positive deviation at most temperatures of the curve. The deviations for labs 574 and 1543

could be caused by the lower pressure at the start of the distillation, the deviation for laboratory 1613 is probably due to analytical problems with the sulphur determination (on several fractions) and not with the distillation.

The true boiling point curves (N in %M/M vs temp AET) of most laboratories show a high resemblance (see Appendix 4). Two laboratories, 1065 and 1543 show a positive deviation. The deviation of laboratory 1065 is probably due to problems with the nitrogen determination and not with the distillation.

Analysis of distillation fractions:

Density: The density test results on the 13 collected distillation fractions show relatively large differences between the reported test results of the participating laboratories, although in total only six statistical outliers (=2.7%) were observed and another four test results were excluded (from 5 laboratories). The relatively large observed spreads are most probably caused by differences in the distillations and not by differences in the performance of the density determinations. In each case the observed reproducibility will be the sum of the (small) spread in the density result and the (large) spread caused by the distillation. Therefore the observed reproducibilities were not compared with the literature requirements and consequently no z-scores were calculated.

The average density for the first fraction (gas <30°C) is 0.5687 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 13 collected distillation fractions show problems for several participating laboratories. In total twelve statistical outliers (=7%) were observed and another three test results were excluded (from 6 laboratories). The observed reproducibility per fraction will be the sum of the spread in the sulphur result and the spread caused by the distillation. Therefore the observed reproducibilities were not compared with the literature requirements and consequently no z-scores were calculated. The gradual decrease of the relative standard deviation from the first to the last distillation fraction is in agreement with the expectations.

Nitrogen: The nitrogen test results on the 13 collected distillation fractions show problems for several participating laboratories. In total fourteen statistical outliers (=14%) from 3 laboratories were detected. The observed reproducibility per fraction will be the sum of the spread in the nitrogen result and the spread caused by the distillation. Therefore the observed reproducibilities were not compared with the requirements and consequently no z-scores were calculated. The gradual decrease of the relative standard deviation from the first to the last distillation fraction is in agreement with the expectations.

The theoretical Density, Sulphur and Nitrogen results, calculated from the fractions, will be discussed in paragraph 4.3.

P(iP)NA: This determination was performed on fractions 2 (light naphtha) and 3 (heavy naphtha) only. On the light naphtha fraction 2, 'only' eight statistical outliers were detected (5.7%). Serious analytical problems were observed on fraction 3 (heavy naphtha). In total eighteen statistical outliers were detected (=18%) on fraction 3.

One laboratory (1543) 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 spread in the analytical method and the spread caused by the D2892. Therefore the observed reproducibilities were not compared with the requirements and consequently no z-scores were calculated. The variety of test methods used may partly explain the large spreads observed during this PT. The set-up of the correct integration window is most critical in the case of testing high naphthenic distillation fractions.

D86 distillation: This determination was performed on the combined fractions 4+5 only. No analytical problems were encountered. Ten statistical outliers were observed (=6.3%). Five out of seven test results for laboratory 574 were outliers, therefore the other two test results were excluded. Each observed reproducibility will be the sum of the spread in the D86 result and the spread caused by the D2892. Therefore the observed reproducibilities were not compared with the requirements and consequently no z-scores were calculated.

Simulated Distillation and Effective Cut Point:

Simdist: This determination was performed on both fractions 4 (kerosene) and 5 (light gasoil) only. This simulated distillation was performed by six 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 D2892:15.

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 D2892:15 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 iis13R02. A number of laboratories participated in both rounds and it is good to see that some participants improved the distillation (like lab. 171 (ECP and Standard Efficiency) and 1095 (ECP)). Regrettably 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 #15216	kg/m ³	20	874.3	1.9	3.6
Sulphur of #15216	%M/M	16	2.577	0.223	0.134
Nitrogen of #15216	mg/kg	9	1249	280	332
Water of #15216	%V/V	13	0.0409	0.0725	0.0324
D2892 distillation	True boiling point curve				
Gas LPG < 30°C	%M/M	18	1.4	1.3	1.3
Light Naphtha 30 - 90°C	%M/M	16	5.1	1.0	1.3
Heavy Naphtha 90 - 180°C	%M/M	16	12.8	0.9	1.3
Kerosene 180 - 215°C	%M/M	16	5.1	0.8	1.5
LGO 215 - 250°C	%M/M	16	5.3	0.6	1.5
MGO 250 - 310°C	%M/M	15	9.3	0.9	2.0
HGO 310 - 370°C	%M/M	18	9.7	1.7	2.0
Residue > 370°C	%M/M	17	50.5	2.2	n.a.
D5236 distillation	True boiling point curve				
VGO 370 - 420°C	%M/M	15	4.6	4.3	(2.0)
VGO 420 - 470°C	%M/M	15	8.6	3.1	(2.0)
VGO 470 - 520°C	%M/M	15	7.1	1.9	(2.0)
sum VGO 520 - 565°C+Residue >565°C	%M/M	15	30.0	2.5	n.a.

table 3: reproducibilities of test results on original crude sample #15216

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

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	11	0.5687	0.0725	n.r.
Density at 15°C of fract. 2: 30-90°C	kg/L	19	0.6682	0.0088	n.r.
Density at 15°C of fract. 3: 90-180°C	kg/L	18	0.7488	0.0044	n.r.
Density at 15°C of fract. 4: 180-215°C	kg/L	17	0.7922	0.0028	n.r.
Density at 15°C of fract. 5: 215-250°C	kg/L	18	0.8102	0.0056	n.r.
Density at 15°C of fract. 6: 250-310°C	kg/L	18	0.8428	0.0038	n.r.
Density at 15°C of fract. 7: 310-370°C	kg/L	19	0.8761	0.0082	n.r.
Density at 15°C of residue: >370°C	kg/L	19	0.9839	0.0067	n.r.
Density at 15°C of fract.9: 370-420°C	kg/L	15	0.9038	0.0114	n.r.
Density at 15°C of fract.10: 420-470°C	kg/L	16	0.9193	0.0136	n.r.
Density at 15°C of fract. 11: 470-520°C	kg/L	16	0.9412	0.0125	n.r.
Density at 15°C of fract. 12: 520-565°C	kg/L	15	0.9578	n.a.	n.r.
Density at 15°C of residue: >565°C	kg/L	14	1.0437	n.a.	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	3	0.021	n.a.	n.a.
Sulphur of fract. 2: 30-90°C	%M/M	14	0.012	0.014	n.a.
Sulphur of fract. 3: 90-180°C	%M/M	13	0.043	0.012	(0.010)
Sulphur of fract. 4: 180-215°C	%M/M	15	0.152	0.026	(0.021)
Sulphur of fract. 5: 215-250°C	%M/M	12	0.369	0.084	(0.038)
Sulphur of fract. 6: 250-310°C	%M/M	11	1.270	0.088	(0.085)
Sulphur of fract. 7: 310-370°C	%M/M	13	2.189	0.156	(0.120)
Sulphur of residue: >370°C	%M/M	14	4.297	0.489	(0.186)
Sulphur of fract.9: 370-420°C	%M/M	13	2.530	0.447	(0.132)
Sulphur of fract.10: 420-470°C	%M/M	13	2.809	0.383	(0.141)
Sulphur of fract. 11: 470-520°C	%M/M	13	3.280	0.486	(0.156)
Sulphur of fract. 12: 520-565°C	%M/M	12	3.860	n.a.	n.a.
Sulphur of residue: >565°C	%M/M	13	5.451	n.a.	n.a.

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	2	n.a.	n.a.	n.a.
Nitrogen of fract. 2: 30-90°C	mg/kg	6	0.2	0.2	(0.4)
Nitrogen of fract. 3: 90-180°C	mg/kg	5	0.3	0.3	(0.5)
Nitrogen of fract. 4: 180-215°C	mg/kg	5	0.5	0.7	(0.6)
Nitrogen of fract. 5: 215-250°C	mg/kg	5	1.2	1.3	(0.9)
Nitrogen of fract. 6: 250-310°C	mg/kg	7	13.1	15.2	(3.1)
Nitrogen of fract. 7: 310-370°C	mg/kg	9	235.2	130.5	(62.6)
Nitrogen of residue: >370°C	mg/kg	10	2429	692	(646)
Nitrogen of fract.9: 370-420°C	mg/kg	8	559	304	(149)
Nitrogen of fract.10: 420-470°C	mg/kg	8	745	272	(198)
Nitrogen of fract. 11: 470-520°C	mg/kg	8	1175	380	(313)
Nitrogen of fract. 12: 520-565°C	mg/kg	7	1647	n.a.	n.a.
Nitrogen of residue: >565°C	mg/kg	9	4129	n.a.	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	9	88.78	1.09	n.r.
C1-C4	%V/V	12	1.27	2.43	n.r.
n-paraffins	%V/V	11	48.72	2.35	n.r.
i-paraffins	%V/V	11	40.11	2.52	n.r.
naphthenes	%V/V	12	9.80	2.12	n.r.
aromatics	%V/V	11	1.39	0.39	n.r.
total paraffins	%M/M	9	87.08	1.47	n.r.
C1-C4	%M/M	12	1.19	1.93	n.r.
n-paraffins	%M/M	11	47.65	2.80	n.r.
i-paraffins	%M/M	11	39.47	2.60	n.r.
naphthenes	%M/M	12	11.12	2.33	n.r.
aromatics	%M/M	11	1.80	0.61	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	8	67.03	2.55	n.r.
C1-C4	%V/V	7	0.02	n.a.	n.r.
n-paraffins	%V/V	9	31.75	2.18	n.r.
i-paraffins	%V/V	7	35.08	2.32	n.r.
naphthenes	%V/V	9	19.68	2.92	n.r.
aromatics	%V/V	9	13.01	0.58	n.r.
total paraffins	%M/M	8	64.12	2.10	n.r.
C1-C4	%M/M	7	0.02	n.a.	n.r.
n-paraffins	%M/M	9	29.89	2.19	n.r.
i-paraffins	%M/M	7	34.06	2.09	n.r.
naphthenes	%M/M	9	20.72	3.51	n.r.
aromatics	%M/M	9	14.99	1.03	n.r.

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

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sdR</i>	<i>R (lit)</i>
IBP	°C	14	190.5	9.8	n.r.
5% evaporated	°C	12	198.6	9.6	n.r.
10% evaporated	°C	12	200.5	8.1	n.r.
50% evaporated	°C	12	211.8	8.5	n.r.
90% evaporated	°C	12	230.9	11.9	n.r.
95% evaporated	°C	12	235.7	12.6	n.r.
FBP	°C	13	243.0	12.4	n.r.
5% recovered	°C	13	199.3	9.2	n.r.
10% recovered	°C	13	201.1	8.3	n.r.
50% recovered	°C	13	212.1	7.9	n.r.
90% recovered	°C	11	230.1	6.5	n.r.
95% recovered	°C	11	235.1	6.3	n.r.

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2015 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 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.
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.

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

4.4 DISCUSSION

In this PT, most laboratories (sixteen in total) reported test results within 6 weeks of the dispatch date. The normal time schedule is not sufficient for the completion of a round robin on Crude Oil Assay, although 6 to 8 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 more aberrant results. These deviations may be (partly) explained by the cumulative effect of spread 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	average difference	average recovery
Density	kg/L	0.8743	0.8733	-0.0010	100%
Sulphur	%M/M	2.577	2.516	-0.061	98%
Nitrogen	mg/kg	1249	1255	+6	100%

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

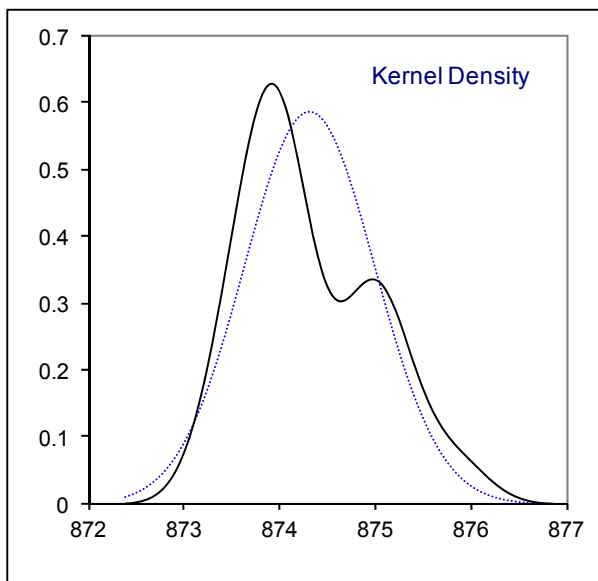
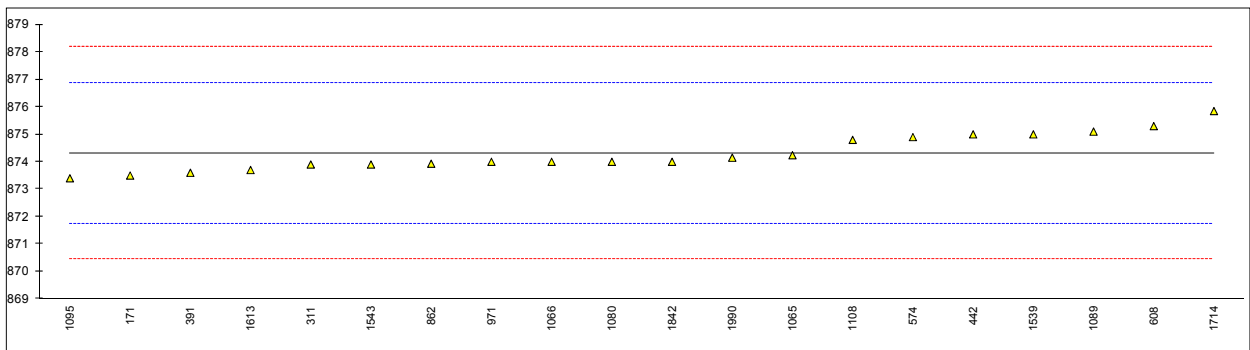
APPENDIX 1

APPENDIX 1A – ANALYSIS OF ORIGINAL SAMPLE

Determination of Density at 15°C on original sample #15216; results in kg/m³

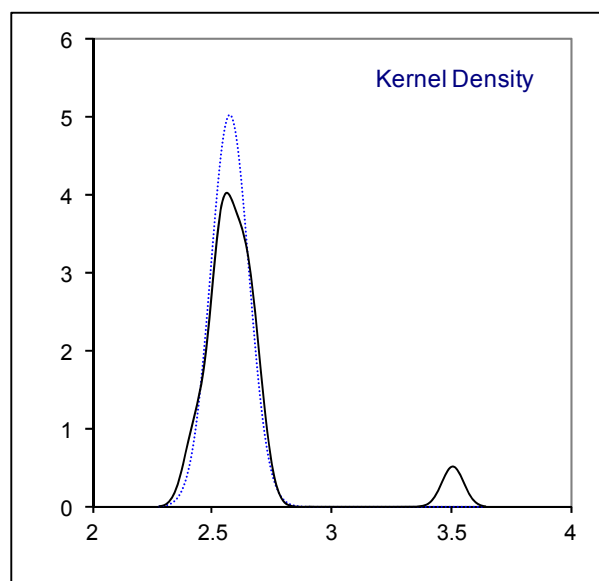
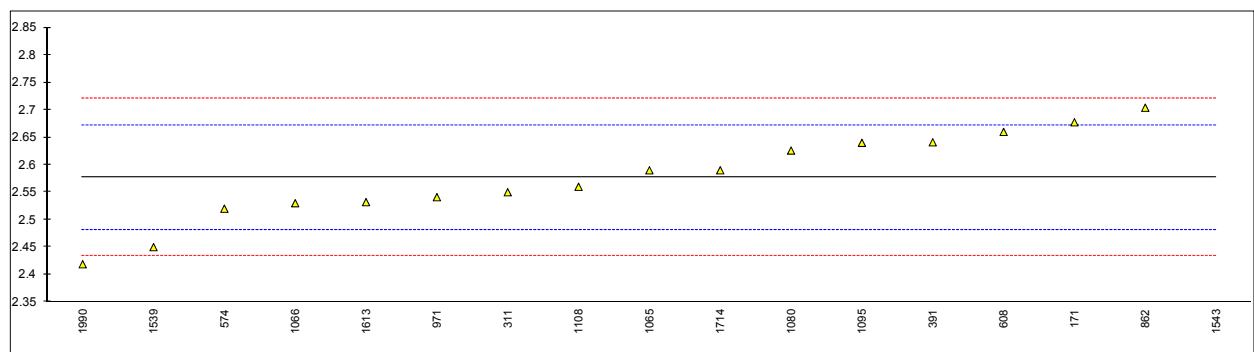
lab	method	value	mark	z(targ)	remarks
171	D5002	873.5		-0.63	
311	D5002	873.9		-0.32	
391	D5002	873.6		-0.55	
442	IP365	875.0		0.53	
445		----		----	
574	D7042	874.9		0.46	
608	D5002	875.3	C	0.77	reported: 0.8753 kg/m ³
862	D5002	873.93		-0.30	
971	D5002	874.0		-0.24	
1065	D1298	874.24	C	-0.06	reported: 0.87424 kg/m ³
1066	D5002	874.0		-0.24	
1080	D5002	874.0		-0.24	
1089	D5002	875.1		0.61	
1095	D5002	873.4	C	-0.71	reported: 0.8734 kg/m ³
1108	D5002	874.8		0.38	
1264		----		----	
1539	D5002	875.0		0.53	
1543	D5002	873.9	C	-0.32	first reported: 0.8733 kg/m ³
1613	D5002	873.7		-0.48	
1616		----		----	
1714	D5002	875.85		1.19	
1720		----		----	
1842	IP365	874.0	C	-0.24	reported: 0.8740 kg/m ³
1990	D4052	874.15		-0.13	
6009		----		----	

normality OK
n 20
outliers 0
mean (n) 874.31
st.dev. (n) 0.679
R(calc.) 1.90
R(D5002:16) 3.60



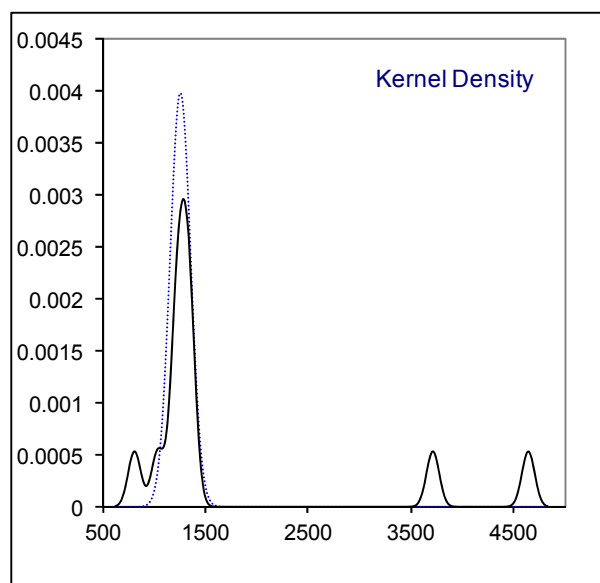
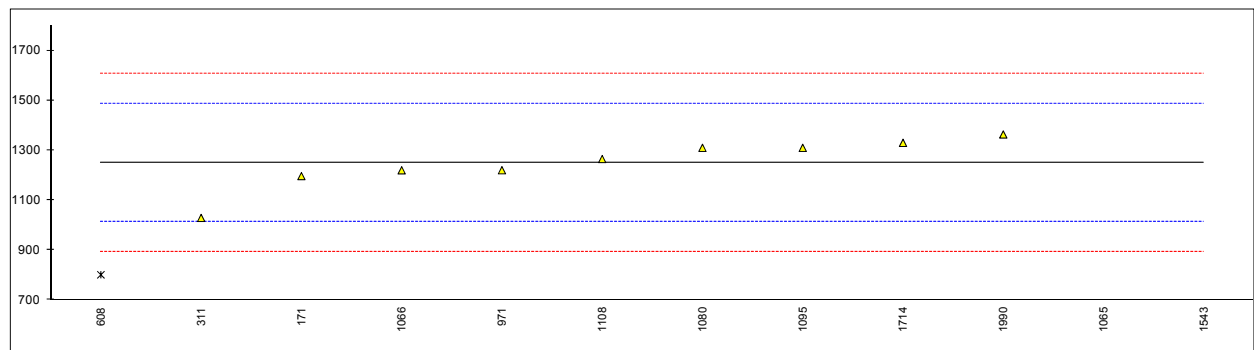
Determination of Sulphur on original sample #15216; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D4294	2.6775		2.11	
311	D4294	2.55		-0.56	
391	D4294	2.641		1.34	
442		----		----	
445		----		----	
574	D4294	2.52		-1.19	
608	D4294	2.6599		1.74	
862	D2622	2.704		2.66	
971	D4294	2.541		-0.75	
1065	ISO8754	2.59		0.27	
1066	D4294	2.53		-0.98	
1080	ISO8754	2.626		1.03	
1089		----		----	
1095	D4294	2.64		1.32	
1108	D4294	2.56		-0.35	
1264		----		----	
1539	ISO8754	2.45		-2.66	
1543	D4294	3.5096	G(0.01)	19.53	
1613	D4294	2.532		-0.94	
1616		----		----	
1714	D2622	2.59		0.27	
1720		----		----	
1842		----		----	
1990	D4294	2.4188		-3.31	
6009		----		----	
normality		OK			
n		16			
outliers		1			
mean (n)		2.5769			
st.dev. (n)		0.07952			
R(calc.)		0.2227			
R(D4294:16ε1)		0.1337			



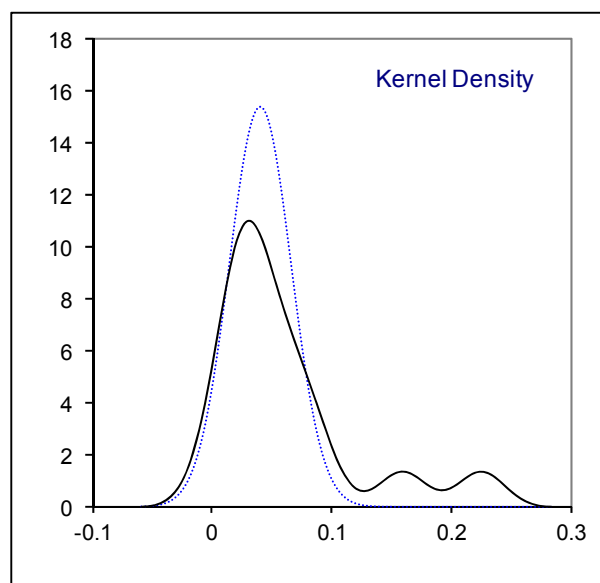
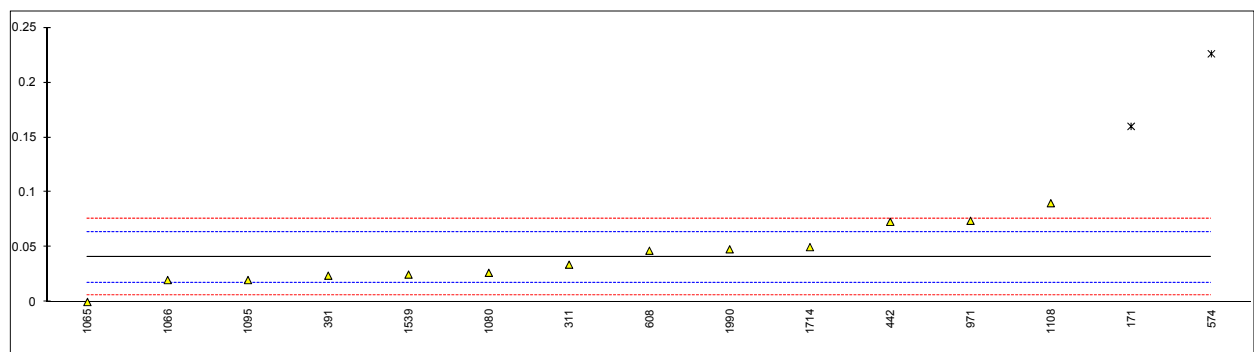
Determination of Nitrogen on original sample #15216; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D5762	1196.9		-0.44	
311	D5762	1029		-1.86	
391		----		----	
442		----		----	
445		----		----	
574		----		----	
608	D5762	801.06	G(0.05)	-3.78	
862		----		----	
971	D5762	1220.21		-0.25	
1065	D5762	3715.693	G(0.01)	20.78	
1066	D5762	1220		-0.25	
1080	ISO8754	1310		0.51	method ISO8754 is probably a typing error
1089		----		----	
1095	D5762	1310		0.51	
1108	D5762	1265		0.13	
1264		----		----	
1539		----		----	
1543	D5291	4648	G(0.05)	28.63	
1613		----		----	
1616		----		----	
1714	D5762	1330		0.68	
1720		----		----	
1842		----		----	
1990	D5762	1363.65		0.96	
6009		----		----	
normality		not OK			
n		9			
outliers		3			
mean (n)		1249.42			
st.dev. (n)		100.116			
R(calc.)		280.33			
R(D5762:12)		332.35			



Determination of Water on original sample #15216; results in %V/V

lab	method	value	mark	z(targ)	remarks
171	D4377	0.16	C,G(0.05)	10.30	first reported: 0.1804
311	D4928	0.034		-0.59	
391	D4377	0.024		-1.46	
442	IP386	0.07305		2.78	
445		----		----	
574	D4377	0.2261	G(0.05)	16.02	
608	D4377	0.0467		0.50	
862	D4006	<0.025		----	
971	D4928	0.074		2.86	
1065	D4006	0		-3.53	
1066	D4377	0.02		-1.80	
1080	D4377	0.0266		-1.23	
1089		----		----	
1095	D6304	0.02		-1.80	
1108	D4377	0.09		4.25	
1264		----		----	
1539	ISO9029	0.025		-1.37	
1543		----		----	
1613	D95	<0.05		----	
1616		----		----	
1714	D4006	0.050		0.79	
1720		----		----	
1842		----		----	
1990	D6304	0.048		0.62	
6009		----		----	
normality		OK			
n		13			
outliers		2			
mean (n)		0.04087			
st.dev. (n)		0.025891			
R(calc.)		0.07249			
R(D4377:11)		0.03238			

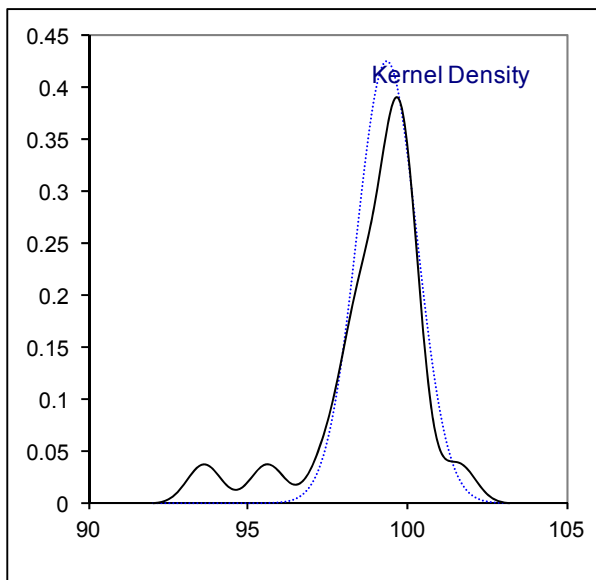
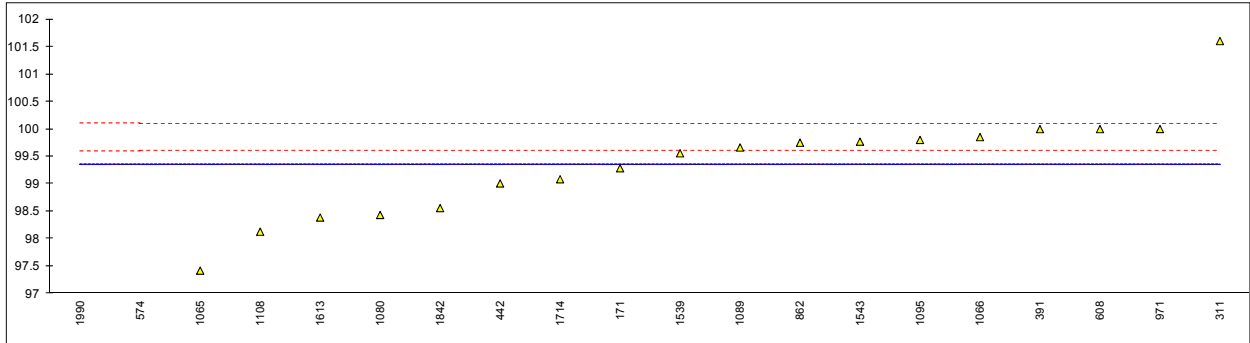


APPENDIX 1B – DISTILLATION RESULTS D2892 AND D5236

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

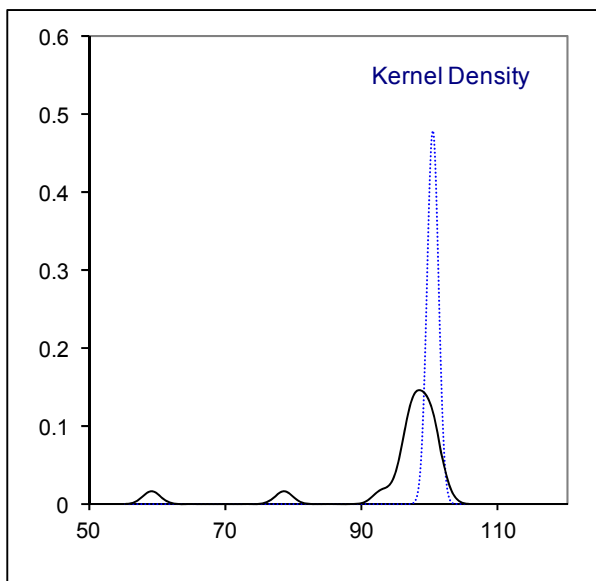
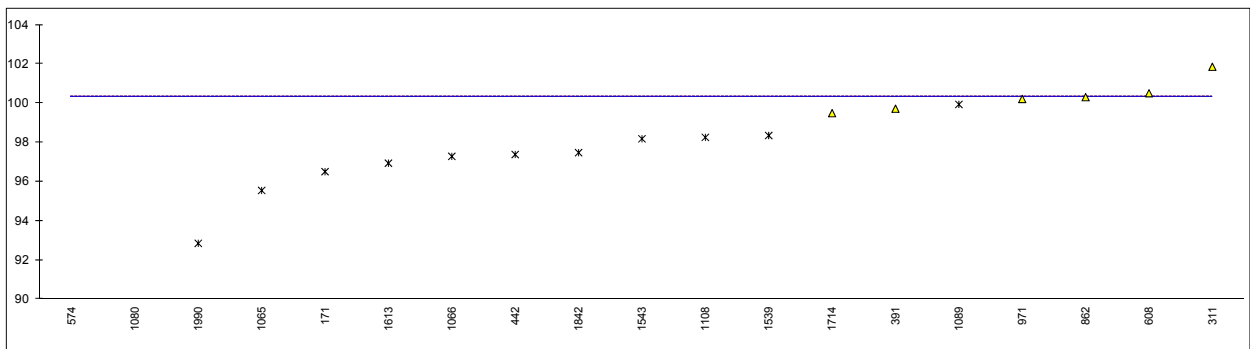
lab	method	value	Mark	remarks
171	calc	99.29		
311	calc	101.60		
391	calc	100.00		
442	calc	99.01		
445	calc	-----		
574	calc	95.61	G(0.05)	
608	calc	100.00		
862	calc	99.75		
971	calc	100.00		
1065	calc	97.42		
1066	calc	99.85		
1080	calc	98.44		
1089	calc	99.66		
1095	calc	99.80		
1108	calc	98.13		
1264	calc	-----		
1539	calc	99.56		
1543	calc	99.77		
1613	calc	98.39		
1616	calc	-----		
1714	calc	99.09		
1720	calc	-----		
1842	calc	98.56		
1990	calc	93.62	G(0.01)	
6009	calc	-----		
normality		suspect		
n		18		
outliers		2		
mean (n)		99.35		
st.dev. (n)		0.942		
R(calc.)		2.64		

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



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

lab	method	value	mark	remarks
171	calc	96.51	ex	density cut 1 not reported
311	calc	101.86		
391	calc	99.72		
442	calc	97.39	ex	not enough data
445	calc	-----		
574	calc	59.32	ex	not enough data
608	calc	100.51		
862	calc	100.31		
971	calc	100.22		
1065	calc	95.55	ex	density cut 1 not reported
1066	calc	97.29	ex	density cut 1 not reported
1080	calc	78.63	ex	dens. 1 and res 565+ n rep.
1089	calc	99.94	ex	not enough data
1095	calc	-----		
1108	calc	98.26	ex	not enough data
1264	calc	-----		
1539	calc	98.36	ex	density cut 1 not reported
1543	calc	98.19	ex	density res. 565+ not rep.
1613	calc	96.94	ex	density cut 1 not reported
1616	calc	-----		
1714	calc	99.49		
1720	calc	-----		
1842	calc	97.48	ex	density cut 1 not reported
1990	calc	92.86	D(0.01)	
6009	calc	-----		
normality		not OK		
n		6		
outliers		1 (+12ex)		
mean (n)		100.35		
st.dev. (n)		0.832		
R(calc.)		2.33		



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

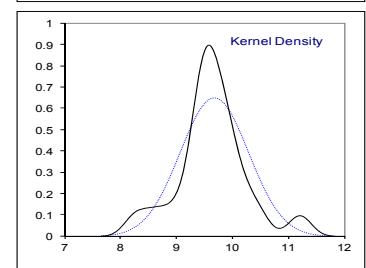
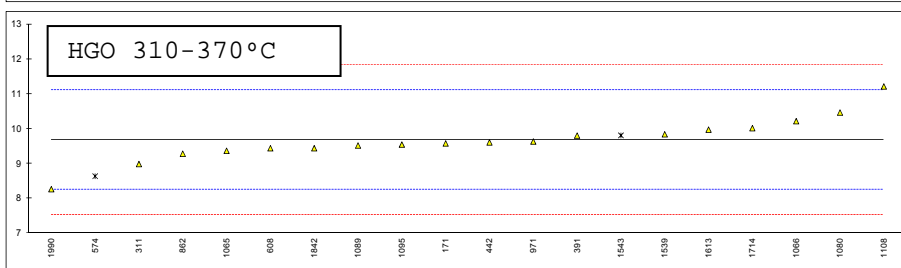
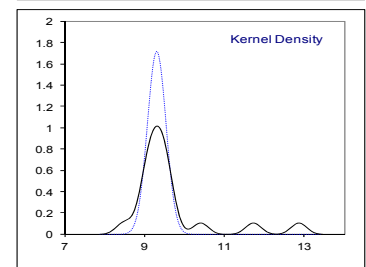
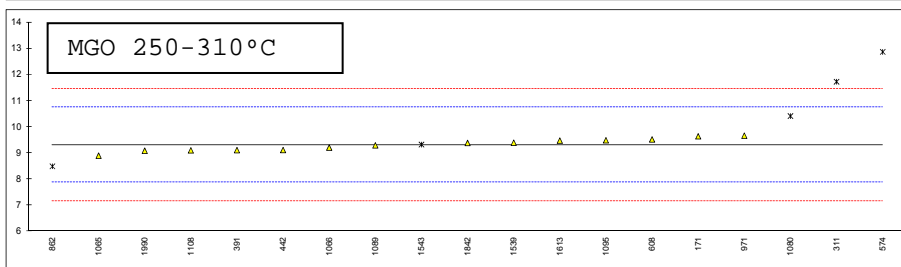
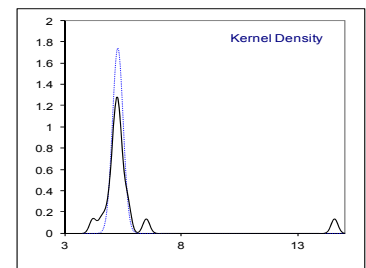
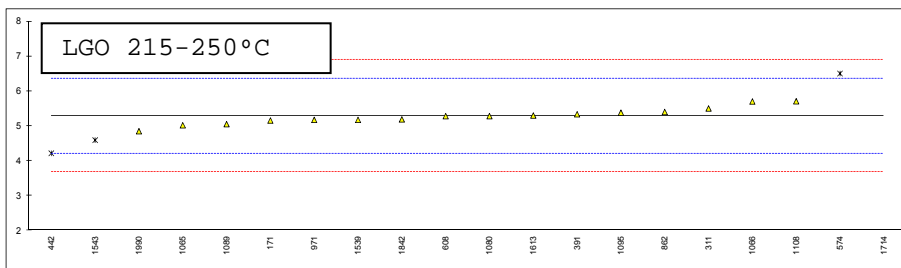
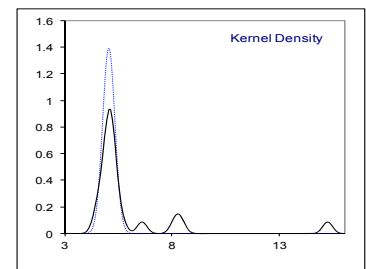
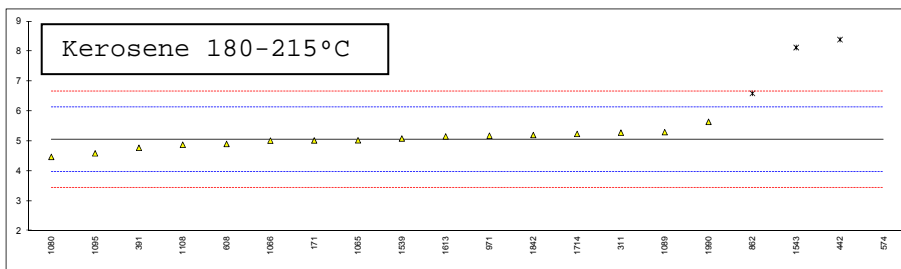
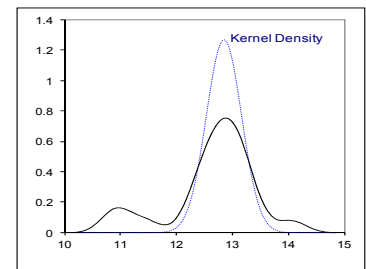
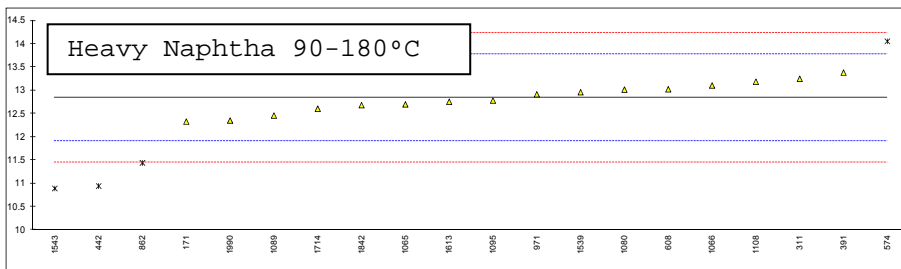
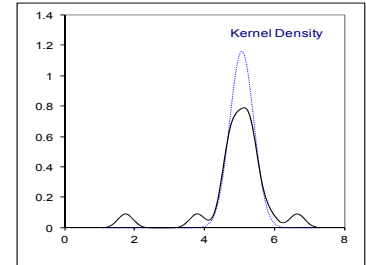
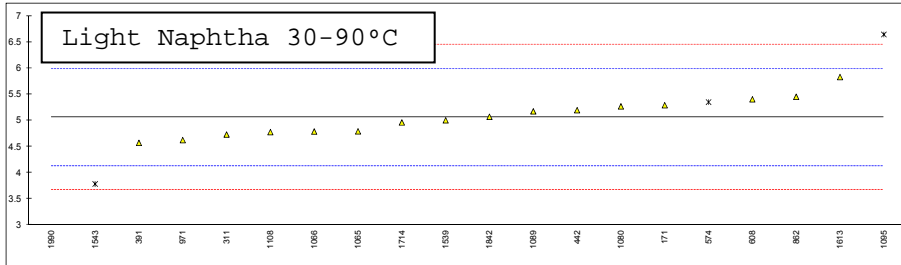
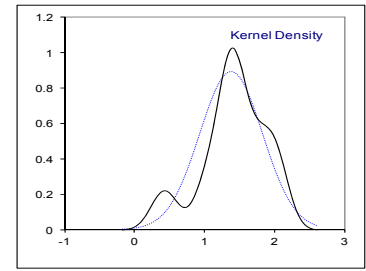
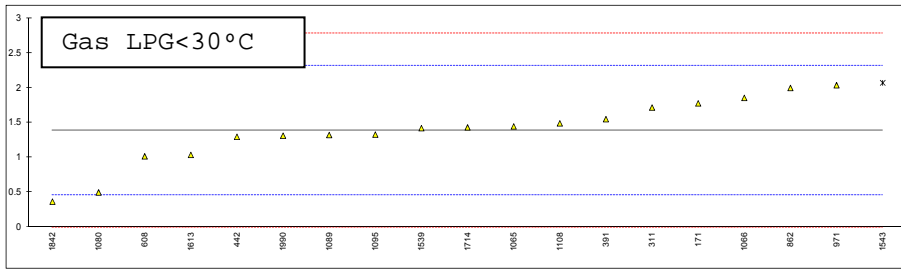
lab	Gas LPG <30°C	L. Naphtha 30-90°C	H. Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C
171	1.7743	5.2911	12.3305	5.0228	5.1584	9.6417	9.5811	50.4876
311	1.7151	4.7303	13.2503	5.2835	5.5048	11.7289	8.9903	50.8437
391	1.5488	4.5705	13.3838	4.7775	5.3383	9.1147	9.8066	51.4597
442	1.2963	5.1955	10.9440 *	8.3969 *	4.2185	9.1174	9.6111	50.2312
445	----	----	----	----	----	----	----	----
574	----	5.3505 *	14.0563	15.2239	6.5058	12.8682	8.6403 *	----
608	1.0156	5.4046	13.0255	4.9063	5.2848	9.5241	9.4424	51.3968
862	1.9960	5.4523	11.4403	6.5978	5.4034	8.4942	9.2868	51.0812
971	2.0354	4.6239	12.9162	5.1825	5.1770	9.6681	9.6350	50.7218
1065	1.4442	4.7907	12.7000	5.0274	5.0230	8.9020	9.3688	50.1644
1066	1.8531	4.7872	13.1055	5.0189	5.7049	9.2121	10.2189	49.9540
1080	0.4980	5.2682	13.0176	4.4732	5.2857	10.4141	10.4665	49.5457
1089	1.3210	5.1738	12.4605	5.2994	5.0558	9.2968	9.5215	51.5361
1095	1.3277	6.6419	12.7820	4.5919	5.3849	9.4898	9.5494	50.0354
1108	1.4884	4.7767	13.1880	4.8806	5.7113	9.1035	11.2150	47.7674
1264	----	----	----	----	----	----	----	----
1539	1.4190	5.0023	12.9607	5.0889	5.1770	9.3980	9.8435	49.4227
1543	2.0686 *	3.7847	10.8915	8.1311	4.5964 *	9.3247 *	9.8108 * C	51.1632 *
1613	1.0355	5.8295	12.7574	5.1573	5.3032	9.4794	9.9771	48.8501
1616	----	----	----	----	----	----	----	----
1714	1.4299	4.9614	12.6072	5.2420	14.5740 *	----	10.0200	50.8256
1720	----	----	----	----	----	----	----	----
1842	0.3669	5.0698	12.6829	5.2033	5.1907	9.3892	9.4434	51.2153
1990	1.3121	1.7362	12.3526	5.6461	4.8509	9.0921	8.2704	50.7621
6009	----	----	----	----	----	----	----	----
normality	OK	OK	OK	OK	OK	OK	not OK	OK
n	18	16	16	16	16	15	18	17
outliers	0 (+1ex)	3 (+1ex)	3 (+1ex)	3 (+1ex)	2 (+2ex)	3 (+1ex)	0 (+2ex)	1 (+1ex)
mean (n)	1.3821	5.0580	12.8450	5.0501	5.2846	9.2616	9.6804	50.5020
st.dev. (n)	0.44869	0.34367	0.31533	0.28653	0.22970	0.30833	0.61343	0.76986
R(calc.)	1.2563	0.9623	0.8829	0.8023	0.6432	0.8633	1.7176	2.1556
R(D2892:15)	1.3000	1.3000	1.3000	1.5000	1.5000	2.0000	2.0000	n.a.

NB. Bold and underlined figures are statistical outliers (Dixon, Grubbs and/or Rosner) and excluded test results (*)

*) lab 442 test results for H. Naphtha and Kerosene were excluded because cutpoint was 160°C instead of 180°C
lab 574 and 1543 test results were excluded for distillation was started with lower pressure than atmospheric (760 mm Hg)
lab 1714 test result for LGO was excluded, because LGO and MGO fraction was combined in one fraction.

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	0.84	0.50	-1.11	-0.05	-0.24	0.53	-0.14	----
311	0.72	-0.71	0.87	0.44	0.41	3.45	-0.97	----
391	0.36	-1.05	1.16	-0.51	0.10	-0.21	0.18	----
442	-0.18	0.30	-4.09	6.25	-1.99	-0.20	-0.10	----
445	----	----	----	----	----	----	----	----
574	----	0.63	2.61	18.99	2.28	5.05	-1.46	----
608	-0.79	0.75	0.39	-0.27	0.00	0.37	-0.33	----
862	1.32	0.85	-3.03	2.89	0.22	-1.07	-0.55	----
971	1.41	-0.93	0.15	0.25	-0.20	0.57	-0.06	----
1065	0.13	-0.58	-0.31	-0.04	-0.49	-0.50	-0.44	----
1066	1.01	-0.58	0.56	-0.06	0.78	-0.07	0.75	----
1080	-1.90	0.45	0.37	-1.08	0.00	1.61	1.10	----
1089	-0.13	0.25	-0.83	0.47	-0.43	0.05	-0.22	----
1095	-0.12	3.41	-0.14	-0.86	0.19	0.32	-0.18	----
1108	0.23	-0.61	0.74	-0.32	0.80	-0.22	2.15	----
1264	----	----	----	----	----	----	----	----
1539	0.08	-0.12	0.25	0.07	-0.20	0.19	0.23	----
1543	1.48	-2.74	-4.21	5.75	-1.28	0.09	0.18	----
1613	-0.75	1.66	-0.19	0.20	0.03	0.30	0.42	----
1616	----	----	----	----	----	----	----	----
1714	0.10	-0.21	-0.51	0.36	17.34	----	0.48	----
1720	----	----	----	----	----	----	----	----
1842	-2.19	0.03	-0.35	0.29	-0.18	0.18	-0.33	----
1990	-0.15	-7.15	-1.06	1.11	-0.81	-0.24	-1.97	----
6009	----	----	----	----	----	----	----	----



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

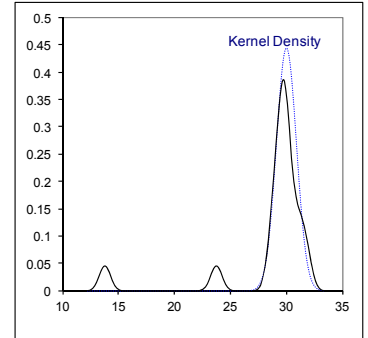
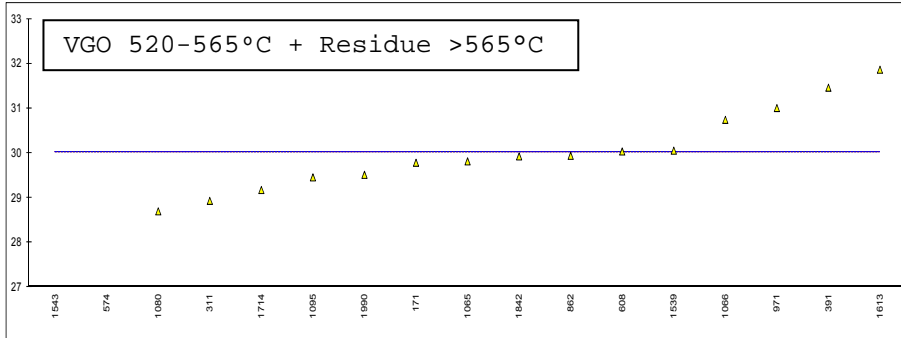
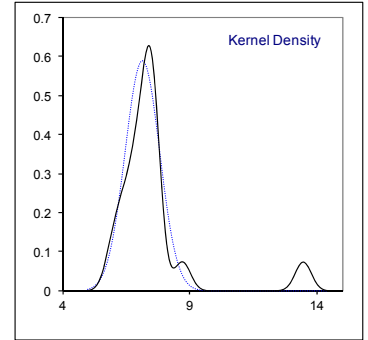
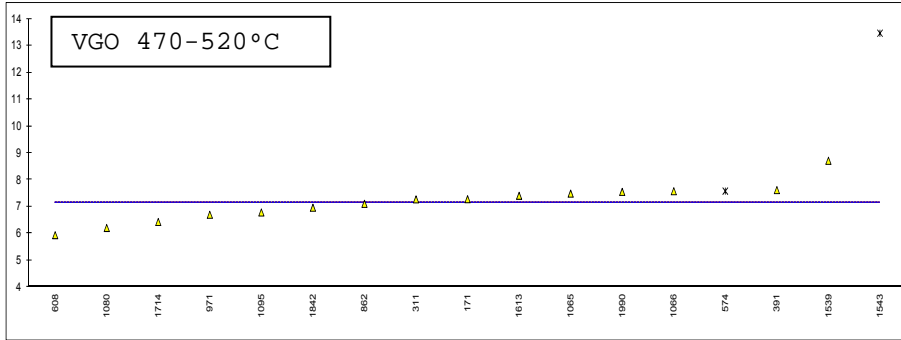
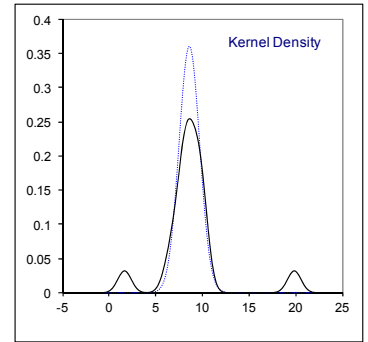
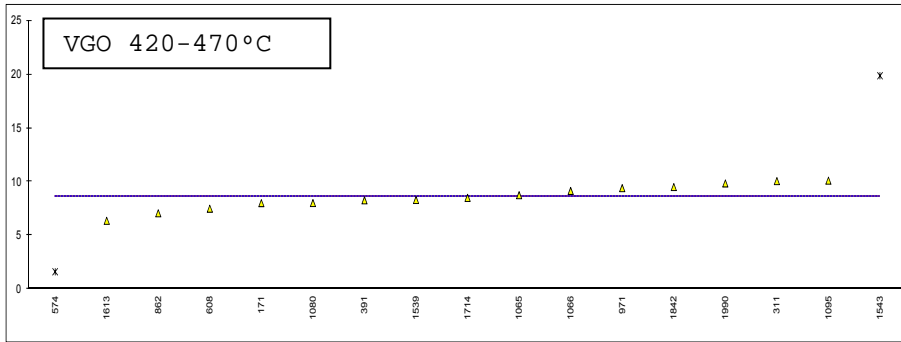
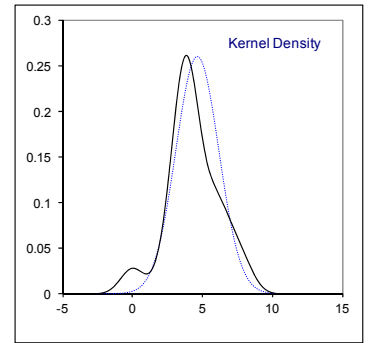
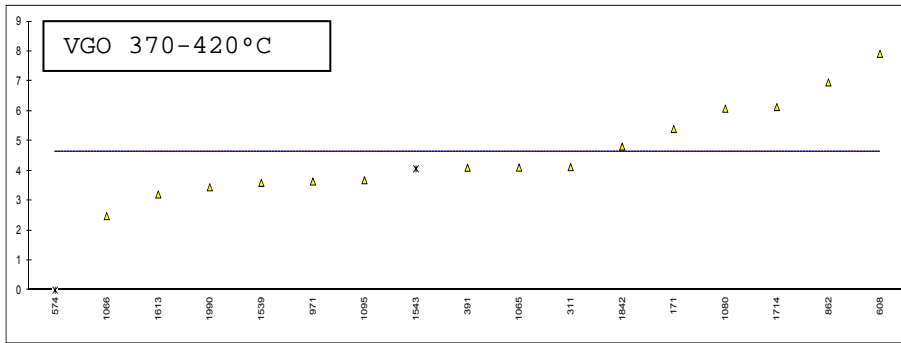
lab	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	sum of VGO 520-565°C + Residue >565°C	remarks
171	5.4017	8.0185	7.2811	29.7863	
311	4.1217	10.0692	7.2752	28.9350	
391	4.1013	8.2747	7.6212	31.4625	
442	----	----	----	----	
445	----	----	----	----	
574	0.0000 *	1.6264	7.5833 *	23.7535	
608	7.9177	7.4984	5.9437	30.0370	
862	6.9597	7.0701	7.1099	29.9416	
971	3.6408	9.4123	6.7022	31.0080	
1065	4.1061	8.7528	7.4889	29.8166	
1066	2.4818	9.1484	7.5796	30.7441	
1080	6.0807	8.0203	6.2118	28.7000	
1089	----	----	----	----	
1095	3.6814	10.1063	6.7884	29.4593	
1108	----	----	----	----	
1264	----	----	----	----	
1539	3.5935	8.3079	8.7124	30.0562	
1543	4.0668 *	19.8698	13.4612	13.7653	fr. for HGO 113.2 g, corrected this to 1130.2 g
1613	3.2062	6.3709	7.4089	31.8642	
1616	----	----	----	----	
1714	6.1351	8.5063	6.4343	29.1766	
1720	----	----	----	----	
1842	4.8113	9.5115	6.9649	29.9276	
1990	3.4460	9.8476	7.5547	29.5162	
6009	----	----	----	----	
normality	OK	OK	suspect	OK	
n	15	15	15	15	
outliers	0 (+2ex)	2	1 (+1ex)	2	
mean (n)	4.6457	8.5943	7.1385	30.0287	
st.dev. (n)	1.53623	1.10826	0.67818	0.89812	
R(calc.)	4.3014	3.1031	1.8990	2.5147	
R(D2892:15)	(2.0000)	(2.0000)	(2.0000)	n.a.	

NB. Bold and underlined figures are statistical outliers (Dixon, Grubbs and/or Rosner) and excluded test results (*)

*) lab 574 and 1543 test results were excluded for D2892 distillation was started with lower pressure than atmospheric (760 mm Hg)

Corresponding z-scores for above mass fractions:

lab	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	sum of VGO 520-565°C + Residue >565°C
171	----	----	----	----
311	----	----	----	----
391	----	----	----	----
442	----	----	----	----
445	----	----	----	----
574	----	----	----	----
608	----	----	----	----
862	----	----	----	----
971	----	----	----	----
1065	----	----	----	----
1066	----	----	----	----
1080	----	----	----	----
1089	----	----	----	----
1095	----	----	----	----
1108	----	----	----	----
1264	----	----	----	----
1539	----	----	----	----
1543	----	----	----	----
1613	----	----	----	----
1616	----	----	----	----
1714	----	----	----	----
1720	----	----	----	----
1842	----	----	----	----
1990	----	----	----	----
6009	----	----	----	----



APPENDIX 1C – ANALYSIS OF DISTILLATION FRACTIONS

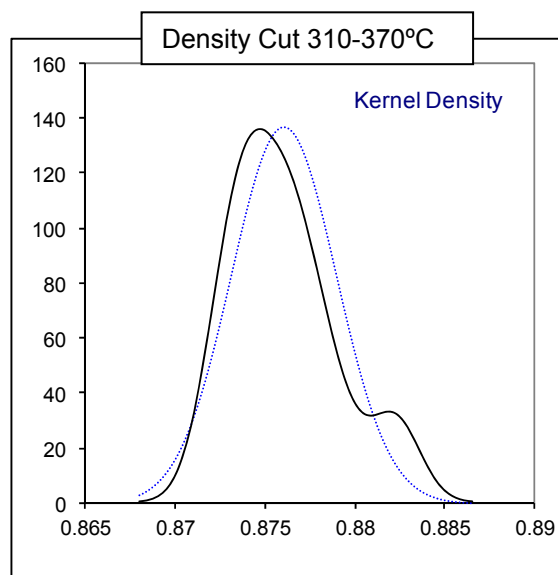
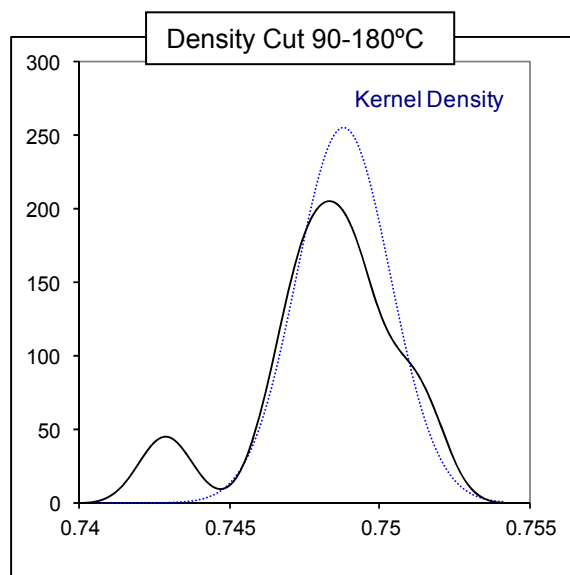
Determination of Density at 15°C on distillation fractions; results in kg/L

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	----	0.66947	0.74876	0.79134	0.80812	0.84177	0.87512	0.98118
311	D4052	0.5859	0.6748	0.7495	0.7916	0.8105	0.8464	0.8824	0.9862
391	D4052	0.5932	0.6672	0.7480	0.7926	0.8088	0.8427	0.8741	0.9876
442	IP365	----	0.6641	0.7425**	0.7895**	0.8116	0.8420	0.8734	0.9836
445		----	----	----	----	----	----	----	----
574	D7042	----	0.6719 *	0.7432	0.8108	0.8476	0.8801	----	----
608	D4052	0.5347	0.6609	0.7483	0.7926	0.8087	0.8438	0.8777	0.9872
862	D4052	0.5547	0.6662	0.7514	0.7941	0.8115	0.8441	0.8743	0.9835
971	D4052	0.5845	0.6706	0.7495	0.7930	0.8100	0.8433	0.8766	0.9828
1065	D4052	----	0.6678	0.7474	0.7918	0.8083	0.8425	0.8722	0.9824
1066	D4052	----	0.6684	0.7491	0.7930	0.8098	0.8440	0.8783	0.9850
1080	D4052	----	0.6704	0.7473	0.7915	0.8078	0.8423	0.8758	0.9854
1089	D4052	0.5715	0.6639	0.7476	0.7910	0.8079	0.8412	0.8731	0.9813
1095	D4052	0.5436	0.6701	0.7505	0.7912	0.8124	0.8403	0.8740	0.9830
1108	D4052	0.5714	0.6702	0.7507	0.7924	0.8142	0.8433	0.8794	0.9889
1264		----	----	----	----	----	----	----	----
1539	ISO12185	----	0.6686	0.7486	0.7923	0.8092	0.8432	0.8769	0.9836
1543	D4052	0.5480*	0.6705*	0.7463*	0.7830*	0.8135*	0.8433*	0.8758*	0.9823*
1613	D4052	----	0.6672	0.7517	0.7938	0.8123	0.8426	0.8821	0.9846
1616		----	----	----	----	----	----	----	----
1714	D4052	0.6205*	0.6665* C	0.7467*	0.7905*	0.8277*/**	----	0.8769*	0.9798*
1720		----	----	----	----	----	----	----	----
1842	IP365	----	0.6673	0.7476	0.7912	0.8080	0.8411	0.8727	0.9847
1990	D4052	0.5476	0.6717	0.7490	0.7929	0.8101	0.8427	0.8748	0.9813
6009		----	----	----	----	----	----	----	----
normality		OK	OK	OK	OK	OK	suspect	OK	OK
n		11	19	18	17	18	18	19	19
outliers		0	0 (+1ex)	1 (+1ex)	2 (+1ex)	1 (+1ex)	1	0	0
mean (n)		0.5687	0.6682	0.7488	0.7922	0.8102	0.8428	0.8761	0.9839
st.dev. (n)		0.02588	0.00316	0.00156	0.00101	0.00202	0.00137	0.00292	0.00241
R(calc.)		0.0725	0.0088	0.0044	0.0028	0.0056	0.0038	0.0082	0.0067
RSD		4.6%	0.5%	0.2%	0.1%	0.2%	0.2%	0.3%	0.2%

NB: Bold and underlined figures are statistical outliers (Grubbs, Dixon and /or Rosner) or excluded test results.

*) Lab 1543 and 1714 corrected all test results for a possible unit error (result was in kg/m³ but reported as kg/L).
 **) Test result lab 442 excluded, cut of L. Naphtha ended at 160°C and cut of H. Naphtha started at 160°C instead of 180°C.
 Test result lab 1714 excluded, fraction LGO and MGO were combined (fraction LGO from 215-310°C).
 Test result for L. Naphtha for lab 574 was excluded, see §4.1.

Lab 1714 first reported for L. Naphtha 66.5 kg/L



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

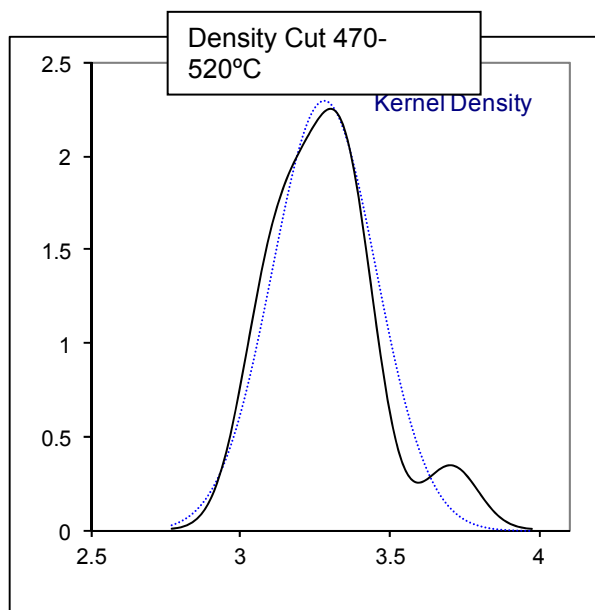
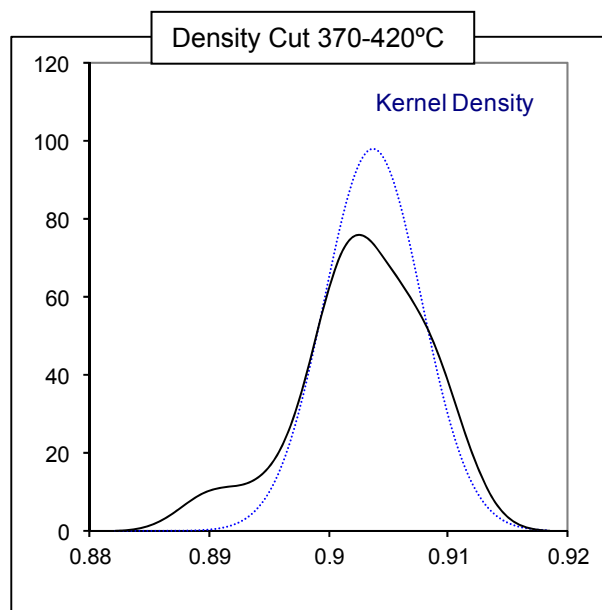
lab	Method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	VGO 520-565°C	Residue >565°C	remarks on method used
171	D4052	0.90500	0.92008	0.94045	0.95504	1.056	Res.>565°C: D70
311	D4052	0.9066	0.9239	0.9473	0.9638	1.0348	LPG: calc.
391	D4052	0.9077	0.9195	0.9432	0.9619	1.0510	LPG: ISO8973, Res.>565°C:D70
442		----	----	----	----	----	
445		----	----	----	----	----	
574		----	----	----	----	----	
608	D4052	0.9092	0.9316	0.9495	----	1.0273	LPG: D2163, Res.>565°C:D70
862	D4052	0.9079	0.9158	0.9415	0.9523	1.0484	LPG: D2598, >565°C:GB/T11137
971	D4052	0.9035	0.9167	0.9383	0.9534	1.0365	
1065	D1298	0.8902**	0.9161	0.9348	0.9624	1.0651	
1066	D4052	0.8956	0.9141	0.9404	0.9592	1.0416	
1080	D4052	0.9038	0.9262	0.9465	0.9637	----	
1089		----	----	----	----	----	LPG: measured manually
1095	D4052	0.9013	0.9176	0.9427	0.9619	1.0635	LPG: UOP539, Res.>565°C:D1298
1108		----	----	----	----	----	LPG: D2598
1264		----	----	----	----	----	
1539	ISO12185	0.9105	0.9176	0.9344	0.9542	1.0215	
1543	D4052	0.9006*	0.9142*	0.9387*	0.9556*	----	LPG: calc.
1613	D4052	0.9027	0.9172	0.9346	0.9468	1.0341	Res. >565°C: DIN51757
1616		----	----	----	----	----	
1714	D4052	0.9004*	0.9247*	0.9412*	0.9590*	1.0425*	
1720		----	----	----	----	----	
1842	IP365	0.9016	0.9182	0.9443	0.9607	1.0466	
1990	D4052	0.8999	0.9158	0.9407	0.9576	1.0434	Res. >565°C: D70
6009		----	----	----	----	----	
normality		OK	not OK	OK	n.a.	n.a.	
n		15	16	16	15	14	
outliers		1	0	0	n.a.	n.a.	
mean (n)		0.9038	0.9193	0.9412	0.9578	1.0437	
st.dev. (n)		0.00407	0.00487	0.00445	n.a.	n.a.	
R(calc.)		0.0114	0.0136	0.0125	n.a.	n.a.	
RSD		0.5%	0.5%	0.5%	n.a.	n.a.	

NB: Bold and underlined figures are statistical outliers (Grubbs, Dixon and /or Rosner) or excluded test results.

*) Lab 1543 and 1714 corrected all test results for a possible unit error (result was in kg/m³ but reported as kg/L).

**) Lab 1065 reported VGO 370-420°C as 'approx. density'

Lab 1714 first reported all test results of VGO as density at 70°C.



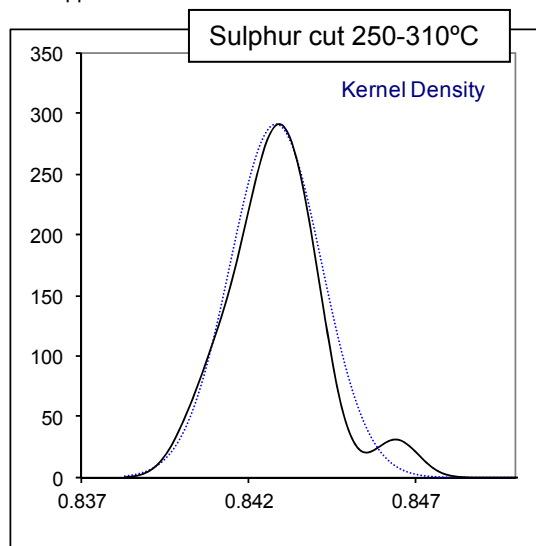
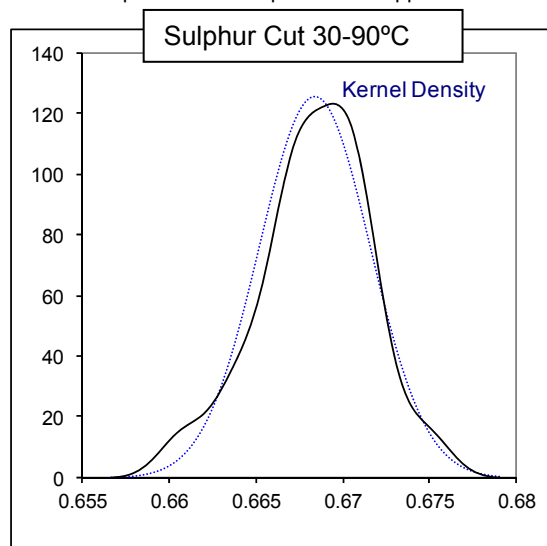
Determination of Sulphur on distillation fractions; results in %M/M

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	D4294	----	0.01253	0.04465	0.1433	0.3280	1.2317 C	2.2042	4.4257
311	D4294	0.0039	0.018	0.045	0.158	0.374	1.354	2.311	4.384
391	D4294	----	0.011	0.044	0.155	0.364	1.30	2.16	4.45
442		----	----	----	----	----	----	----	----
445		----	----	----	----	----	----	----	----
574	D4294	----	0.0062 *	0.0355 *	0.493	1.40	2.25	2.74	----
608	D4294	----	0.00755 C	0.0370	0.1395	0.3296	1.2274	2.0914	4.5372
862	D2622	0.0527	0.0140	0.0429	0.150	0.397	1.32	2.20	4.23
971	D4294	----	0.0120	0.0390	0.147	0.332	1.260	2.200	4.350
1065	ISO8754	----	0.02	0.05	0.17	0.36	1.27	2.13	3.97
1066	D2622	----	0.0030	0.0353	0.135	0.371	1.27	2.21	4.19
1080		----	----	----	----	----	----	----	----
1089		----	----	----	----	----	----	----	----
1095	D4294	0.0052 C	0.0146	0.0474	0.147	0.420	1.24	2.20	4.44
1108	D4294	----	0.0165	0.0479	0.1595	0.501	1.42	2.26	4.38
1264		----	----	----	----	----	----	----	----
1539	ISO8754	----	0.0132	0.043	0.154	0.362	1.28	2.15	4.22
1543	D4294	----	----	----	0.1488	0.4642	1.6225	2.9185	----
1613	D4294	----	0.0135	0.0434	0.163	0.411	1.315	2.674	4.403
1616		----	----	----	----	----	----	----	----
1714	D2622	----	0.0034 C	0.0130 C	0.147	0.863*	----	2.17	4.22
1720		----	----	----	----	----	----	----	----
1842		----	----	----	----	----	----	----	----
1990	D4294	----	0.0101	0.0391	0.1572	0.3751	1.2578	2.1697	3.9578
6009		----	----	----	----	----	----	----	----
normality		n.a.	OK	OK	OK	OK	OK	suspect	OK
n		3	14	13	15	12	11	13	14
outliers		n.a.	0 (+1ex)	1 (+1ex)	1	3 (+1ex)	4	3	0
mean (n)		0.02060	0.01210	0.04297	0.1516	0.3686	1.2702	2.1889	4.2970
st.dev. (n)		n.a.	0.00492	0.004347	0.00920	0.03011	0.03155	0.05557	0.17462
R(calc.)		n.a.	0.01377	0.01217	0.0258	0.0843	0.0883	0.1556	0.4889
R(D4294:16)		n.a.	n.a.	(0.0096)	(0.0215)	(0.0382)	(0.0848)	(0.1204)	(0.1860)
RSD in %		n.a.	41%	10%	6.1%	8.2%	2.5%	2.5%	4.1%

NB: Bold and underlined figures are statistical outliers (Grubbs, Dixon and /or Rosner) or excluded test results.

*) Test result lab 1714 excluded, fraction LGO and MGO were combined (fraction LGO from 215-310°C).
 Test result for L. Naphtha and H. Naphtha for lab 574 was excluded, see §4.1.

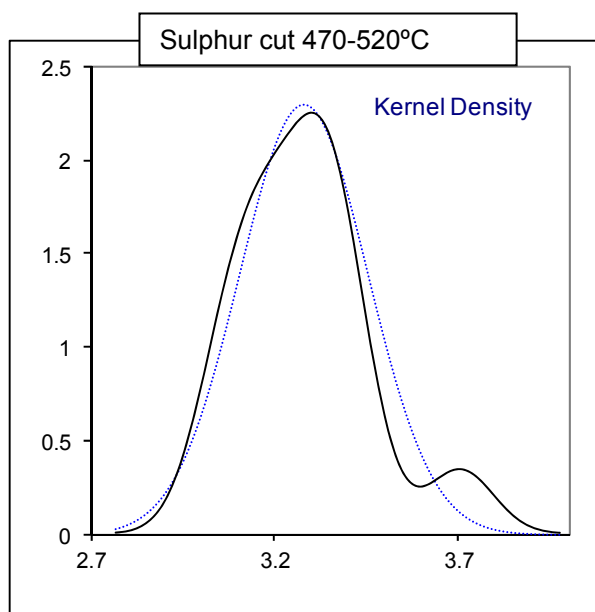
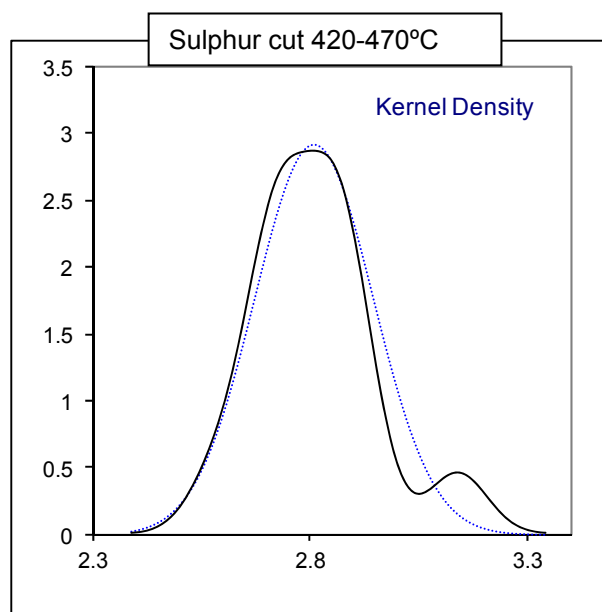
Lab 171 first reported for MGO: 0.84177
 Lab 608 first reported for L. Naphtha: 0.0755
 Lab 1095 reported for Gas LPG: 52 %M/M (probably a unit error?)
 Lab 1714 reported for L. Naphtha: 34.00 ppm and for H. Naphtha: 134.00 ppm



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

lab	Method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	VGO 520-565°C	Residue >565°C	remarks on method
171	D4294	2.6013	2.8522	3.3457	3.8170	5.6168	L. and H. Naphtha: D2622
311	D4294	2.52	2.90	3.37	3.92	5.88	LPG: D6667
391	D4294	2.62	2.75	3.42	4.09	4.48	
442		----	----	----	----	----	
445		----	----	----	----	----	
574		----	----	----	----	----	
608	D4294	2.4273	2.8136	3.3537	----	5.7173	L. Naphtha: D5453
862	D2622	2.77	2.91	3.33	3.73	5.14	LPG: D5453
971	D4294	2.494	2.728	3.157	3.580	5.390	
1065	ISO8754	2.28	2.69	3.12	3.86	5.41	L. and H. Naphtha: D7212
1066	D2622	2.29	2.59	3.09	3.61	5.32	
1080		----	----	----	----	----	
1089		----	----	----	----	----	
1095	D4294	2.51	2.84	3.24	4.07	5.64	LPG: D6667
1108		----	----	----	----	----	
1264		----	----	----	----	----	
1539	ISO8754	2.71	2.73	3.04	3.73	5.27	L. Naphtha: ISO20846
1543		----	----	----	----	----	
1613	D4294	2.770	3.141	3.704	4.361	5.485	
1616		----	----	----	----	----	
1714	D2622	2.45	2.87	3.27	3.79	5.86	L. and H. Naphtha: D5453
1720		----	----	----	----	----	
1842		----	----	----	----	----	
1990	D4294	2.4526	2.7069	3.2012	3.7567	5.6584	L. and H. Naphtha: D5453
6009		----	----	----	----	----	
normality		OK	not OK	suspect	n.a	n.a.	
n		13	13	13	12	13	
outliers		0	0	0	n.a.	n.a.	
mean (n)		2.5304	2.8094	3.2801	3.8596	5.4513	
st.dev. (n)		0.15959	0.13666	0.17370	n.a.	n.a.	
R(calc.)		0.4468	0.3826	0.4864	n.a.	n.a.	
R(D4294:16)		(0.1322)	(0.1414)	(0.1563)	n.a.	n.a.	
RSD in %		6.3%	4.9%	5.3%	n.a.	n.a.	

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



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

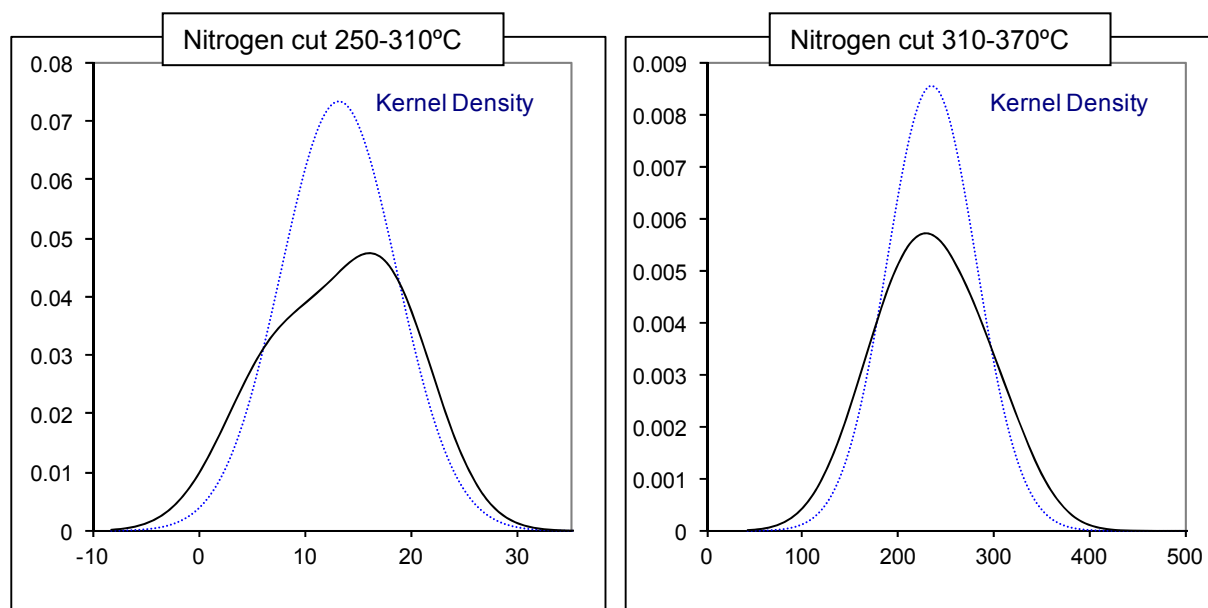
lab	method	Gas LPG <30°C	L.Naphtha 30-90°C	H. Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C
171	D4629	----	<0.3	<0.3	<0.3	<0.3	9.88	183.87**	2405.8**
311	D4629	0.9	0.25	0.4	0.846 C	1.715 C	<0.3 C	300**	2400**
391		----	----	----	----	----	----	----	----
442		----	----	----	----	----	----	----	----
445		----	----	----	----	----	----	----	----
574		----	----	----	----	----	----	----	----
608	D5762	----	n.d.	n.d.	n.d.	n.d.	6.73	234	2105
862		----	----	----	----	----	----	----	----
971	D4629	----	0.17	0.24	0.38	1.08	18.49	239.8**	2416.9**
1065	D5762	----	2153	2346	2275	2270	2295	2464	2560
1066	D4629	<2	<2	<2	<2	<2	5.9	259**	2400**
1080		----	----	----	----	----	----	----	----
1089		----	----	----	----	----	----	----	----
1095	D4629	----	0.20	0.3	0.6	1.4	17	222	2950**
1108	D5762	----	0.19	0.25	0.49	1.4	17.3	302	2180
1264		----	----	----	----	----	----	----	----
1539		----	----	----	----	----	----	----	----
1543		----	----	----	----	----	----	----	7106
1613		----	----	----	----	----	----	----	----
1616		----	----	----	----	----	----	----	----
1714	D4629	----	0.31	0.93	<3**	6.6*/**	----	167**	2650**
1720		----	----	----	----	----	----	----	----
1842		----	----	----	----	----	----	----	----
1990	D4629	----	0.29	0.13	0.20	0.47	16.69	209.18**	2223.91**
6009		----	----	----	----	----	----	----	----
normality		n.a.	unknown	unknown	unknown	unknown	unknown	OK	suspect
n		2	6	5	5	5	7	9	10
outliers		n.a.	1	2	1	2	1	1	1
mean (n)		n.a.	0.24	0.26	0.50	1.21	13.14	235.21	2429.2
st.dev. (n)		n.a.	0.057	0.098	0.242	0.472	5.440	46.621	246.95
R(calc.)		n.a.	0.16	0.27	0.68	1.32	15.23	130.54	691.5
R(D4629:12)		n.a.	(0.38)	(0.49)	(0.57)	(0.89)	(3.05)	(62.56)**	(646.2)**
RSD in %		n.a.	24%	37%	48%	39%	41%	20%	10%

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

*) Test result lab 1714 excluded, fraction LGO and MGO were combined (fraction LGO from 215-310°C).

***) Test results of these laboratories were done with D5762, Reproducibility based on D5762:12

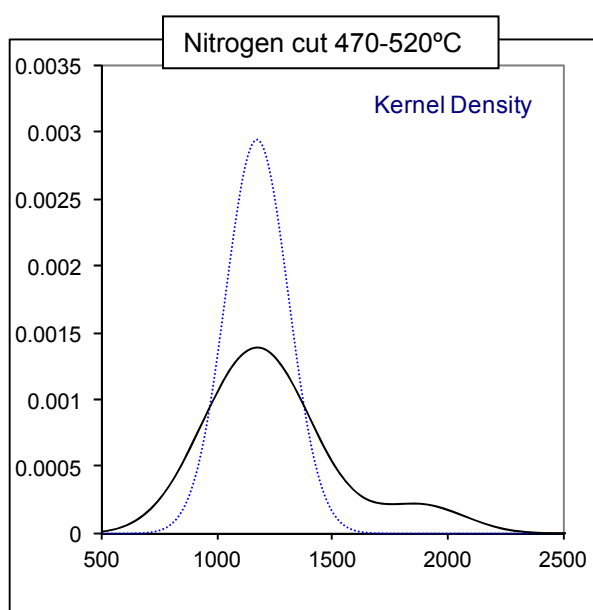
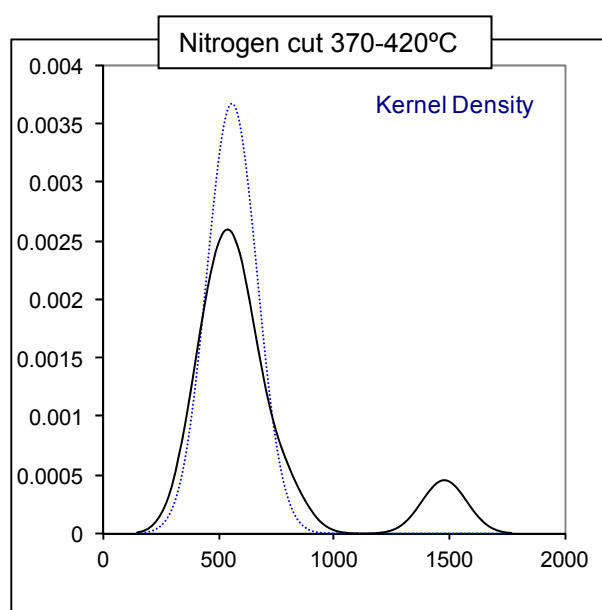
Lab 311 first reported for Kerosene, LGO and MGO: <40



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

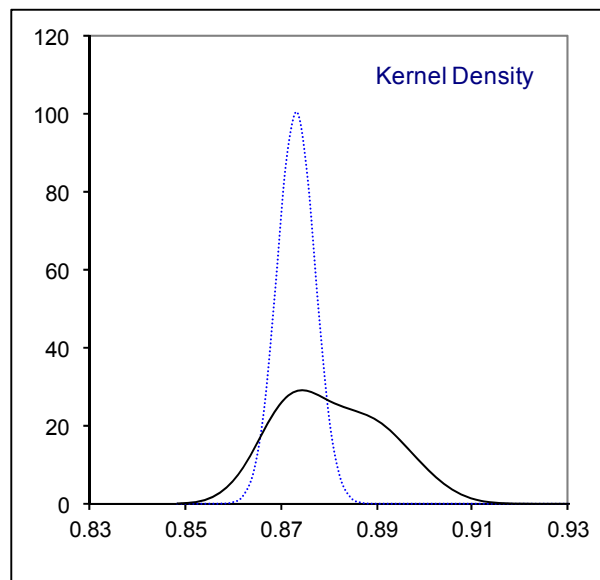
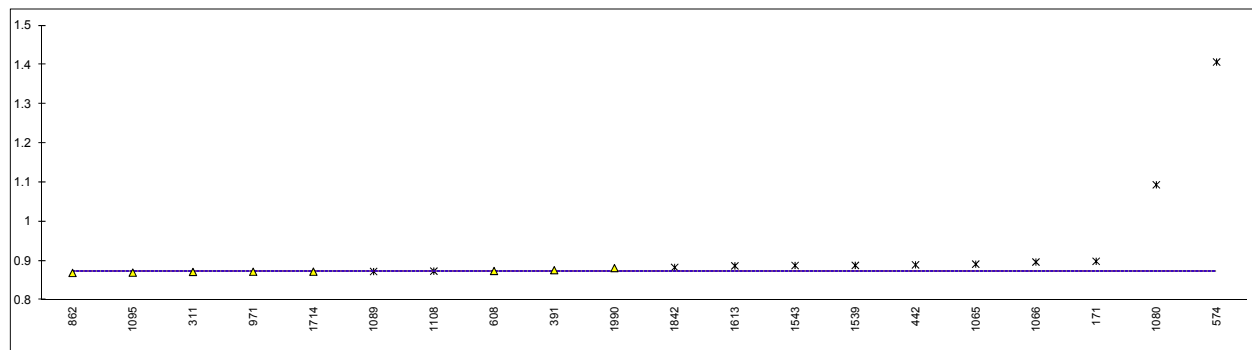
lab	method	VGO	VGO	VGO	VGO	Residue	remarks on method used
		370-420°C	420-470°C	470-520°C	520-565°C		
171	D5762	442.3	662.2	1012.5	1368.5	3815.8	HGO and Res.>370°C: D5762
311	D5762	600	800	1200	1700	3700	HGO and Res.>370°C: D5762
391		----	----	----	----	----	
442		----	----	----	----	----	
445		----	----	----	----	----	
574		----	----	----	----	----	
608	D5762	571	846	1228	----	3293	
862		----	----	----	----	----	
971	D5762	588.6	753.4	1397.7	1929.4	3604.6	HGO and Res.>370°C: D5762
1065	D5762	1479	1630	1887	2538	6125	HGO and Res.>370°C: D5762
1066	D5762	440	590	990	1450	3600	
1080		----	----	----	----	----	
1089		----	----	----	----	----	
1095	D5762	774	871	1260	1780	5310	Res.>370°C: D5762
1108		----	----	----	----	----	
1264		----	----	----	----	----	
1539		----	----	----	----	----	
1543	D5291	----	2711	5339	3678	----	
1613		----	----	----	----	----	
1616		----	----	----	----	----	
1714	D5762	484	671	1096	1635	4620	fraction LGO to Res.>370°C: D5762
1720		----	----	----	----	----	
1842		----	----	----	----	----	
1990	D5762	573.26	767.41	1218.85	1666.88	3096.70	HGO and Res.>370°C: D5762
6009		----	----	----	----	----	
normality		suspect	OK	OK	n.a.	n.a.	
n		8	8	8	7	9	
outliers		1	2	2	n.a.	n.a.	
mean (n)		559.15	745.13	1175.38	1647.11	4129.46	
st.dev. (n)		108.585	97.124	135.777	n.a.	n.a.	
R(calc.)		304.04	271.95	380.18	n.a.	n.a.	
R(D5762:12)		(148.73)	(198.20)	(312.65)	n.a.	n.a.	
RSD in %		19%	13%	12%	n.a.	n.a.	

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



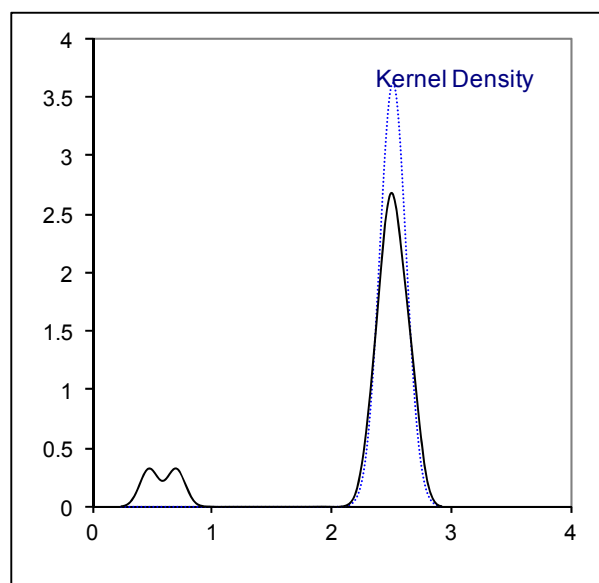
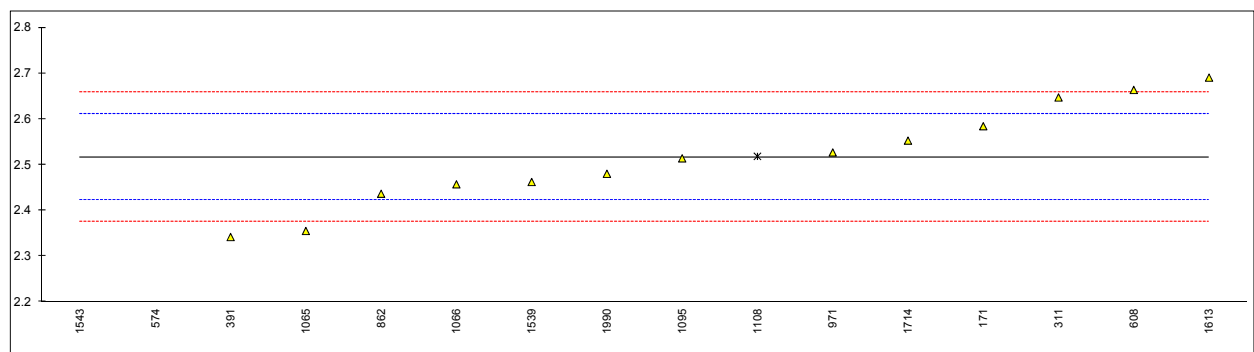
Determination of calculated theoretical Density at 15°C of sample #15216; results in kg/L

lab	method	value	mark	z(targ)	remarks	Meas. density	difference
171	calc.	0.8987	ex	----	density cut 1 not reported	0.8735	----
311	calc.	0.8716		----		0.8739	-0.0023
391	calc.	0.8760		----		0.8736	0.0024
442	calc.	0.8896	ex	----	not enough data	0.8750	----
445	calc.	----		----		----	----
574	calc.	1.4070	ex	----	not enough data	0.8749	----
608	calc.	0.8741		----		0.8753	-0.0012
862	calc.	0.8690		----		0.87393	-0.0049
971	calc.	0.8721		----		0.8740	-0.0019
1065	calc.	0.8914	ex	----	density cut 1 not reported	0.87424	----
1066	calc.	0.8969	ex	----	density cut 1 not reported	0.8740	----
1080	calc.	1.0938	ex	----	dens. 1 and res 565+ n rep.	0.8740	----
1089	calc.	0.8727	ex	----	not enough data	0.8751	----
1095	calc.	0.8698		----		0.8734	-0.0036
1108	calc.	0.8736	ex	----	not enough data	0.8748	----
1264	calc.	----		----		----	----
1539	calc.	0.8883	ex	----	density cut 1 not reported	0.8750	----
1543	calc.	0.8880	ex	----	density res. 565+ not rep.	0.8739	----
1613	calc.	0.8870	ex	----	density cut 1 not reported	0.8737	----
1616	calc.	----		----		----	----
1714	calc.	0.8723		----		0.87585	-0.0036
1720	calc.	----		----		----	----
1842	calc.	0.8831	ex	----	density cut 1 not reported	0.8740	----
1990	calc.	0.8814		----		0.87415	0.0073
6009	calc.	----		----		----	----
normality		not OK					
n		8					
outliers		0 (+12 ex)					
mean (n)		0.87331		Average:		0.87431	-0.0010
st.dev. (n)		0.003963					
R(calc.)		0.01110					
R(D5002:16)		0.00360					



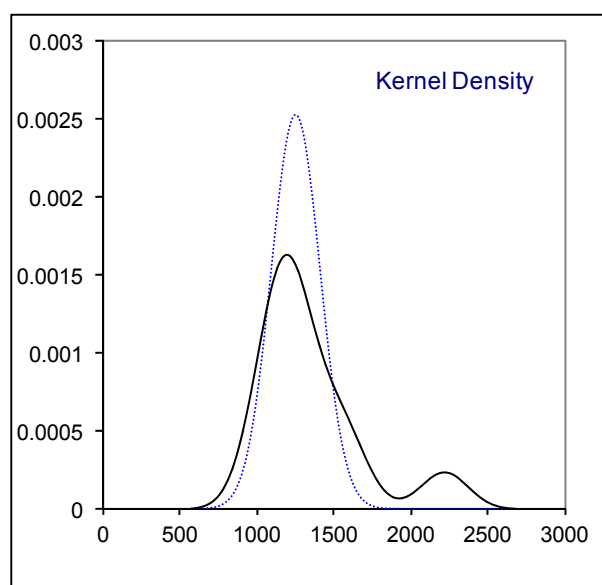
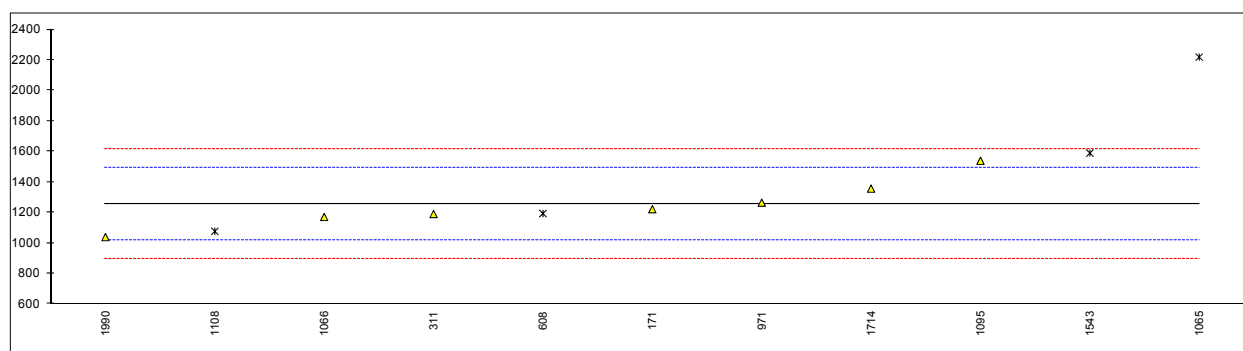
Determination of calculated theoretical sulphur content on sample #15216; results in %M/M

lab	method	Value	mark	z(targ)	remarks	Meas. sulphur	Δ absolute	rel%
171	calc.	2.5844		----		2.6775	-0.0931	-3.5
311	calc.	2.6472		----		2.55	0.0972	3.8
391	calc.	2.3420		----		2.641	-0.2990	-11.3
442	calc.	----		----		----	----	
445	calc.	----		----		----	----	
574	calc.	0.6977	ex	----	not enough data available	2.52	----	
608	calc.	2.6637		----		2.6599	0.0038	0.1
862	calc.	2.4367		----		2.704	-0.2673	-9.9
971	calc.	2.5269		----		2.541	-0.0141	-0.6
1065	calc.	2.3553		----		2.59	-0.2347	-9.1
1066	calc.	2.4573		----		2.53	-0.0727	-2.9
1080	calc.	----		----		2.626	----	
1089	calc.	----		----		----	----	
1095	calc.	2.5141		----		2.64	-0.1259	-4.8
1108	calc.	2.5184	ex	----	not enough data available	2.56	----	
1264	calc.	----		----		----	----	
1539	calc.	2.4623		----		2.45	0.0123	0.5
1543	calc.	0.4711	ex	----	not enough data available	3.5096 outlier	----	
1613	calc.	2.6906		----		2.532	0.1586	6.3
1616	calc.	----		----		----	----	
1714	calc.	2.5528		----		2.59	-0.0372	-1.4
1720	calc.	----		----		----	----	
1842	calc.	----		----		----	----	
1990	calc.	2.4803		----		2.4188	0.0615	2.5
6009	calc.	----		----		----	----	
normality		OK						
n		13						
outliers		0 (+3ex)						
mean (n)		2.5164				Average: 2.5769		
st.dev. (n)		0.11017						
R(calc.)		0.3085						
R(D4294:16)		0.1317						



Determination of calculated theoretical nitrogen content on sample #15216; results in mg/kg

lab	method	value	mark	z(targ)	remarks	Meas. nitrogen	Δ absolute	rel%
171	calc.	1221.93		----		1196.9	25.0	2.1
311	calc.	1190.60		----		1029	161.6	15.7
391	calc.	----		----		----	----	
442	calc.	----		----		----	----	
445	calc.	----		----		----	----	
574	calc.	----		----		----	----	
608	calc.	1193.27	ex	----	not enough data available	801.06	outlier	----
862	calc.	----		----		----	----	
971	calc.	1264.81		----		1220.21	44.6	3.7
1065	calc.	2219.57	G(0.05)	----		3715.693	outlier	----
1066	calc.	1170.81		----		1220	-49.2	-4.0
1080	calc.	----		----		1310	----	
1089	calc.	----		----		----	----	
1095	calc.	1539.60		----		1310	229.6	17.5
1108	calc.	1076.77	ex	----	not enough data available	1265	----	
1264	calc.	----		----		----	----	
1539	calc.	----		----		----	----	
1543	calc.	1589.77	ex	----	not enough data available	4648	outlier	----
1613	calc.	----		----		----	----	
1616	calc.	----		----		----	----	
1714	calc.	1356.88		----		1330	26.9	2.0
1720	calc.	----		----		----	----	
1842	calc.	----		----		----	----	
1990	calc.	1038.58		----		1363.65	-325.1	-23.8
6009	calc.	----		----		----	----	
normality		suspect						
n		7						
outliers		1 (+3ex)						
mean (n)		1254.74	Average		1249.42			
st.dev. (n)		158.450						
R(calc.)		443.66						
R(D5762:10)		333.76						

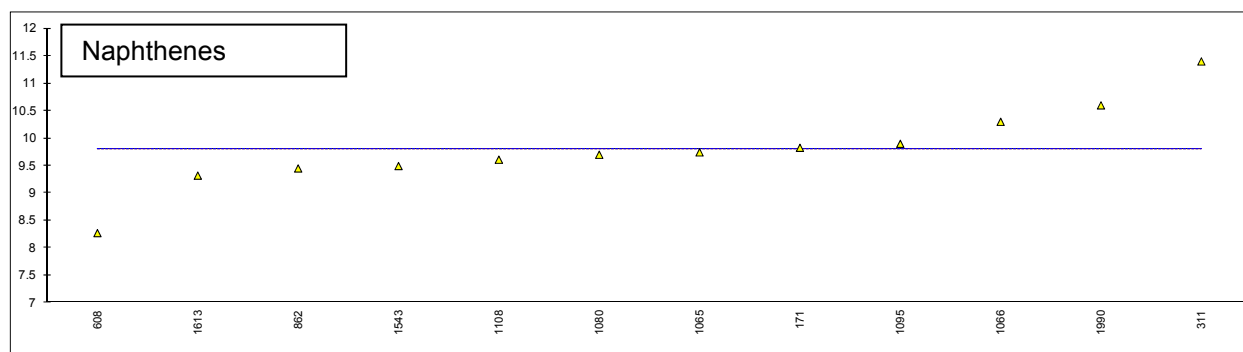
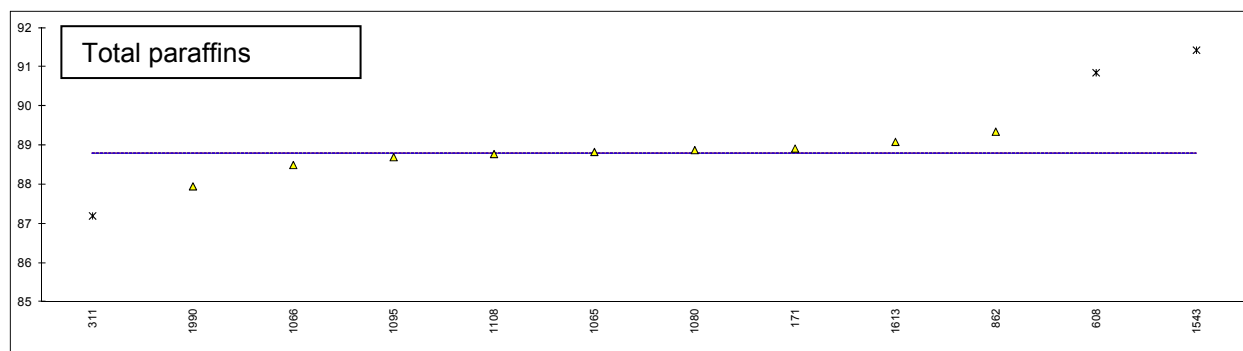


Determination of P(iP)NA on distillation fraction 2 (light naphtha); results in %V/V

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D5134M	88.920	0.483	49.938	38.982	9.827	1.237	n- and i-paraffins: D5443Mod
311	D5443	87.2	1.3	47.9	39.3	11.4	1.4	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445		----	----	----	----	----	----	
574		----	----	----	----	----	----	
608	D6730	90.8542 C	2.0461	49.4220	41.4322	8.2724	0.8490	
862	D6839	89.35	1.10	48.34	41.01	9.45	1.17	
971		----	----	----	----	----	----	
1065	in house	88.834	2.40	49.478	39.356	9.743	1.423	
1066	in house	88.5	2.0	49.0	39.5	10.3	1.2	
1080	in house	88.88	0.29	48.99	39.89	9.70	1.41	
1089		----	----	----	----	----	----	
1095	in house	88.7	0.1	48.1	40.6	9.9	1.4	
1108	D5443	88.78	0.96	49.53	39.25	9.61	1.60	
1264		----	----	----	----	----	----	
1539		----	----	----	----	----	----	
1543	D5134	91.43	2.265	47.83	41.33	9.494	1.397	
1613	D6839	89.09	2.08	----	----	9.32	1.58	
1616		----	----	----	----	----	----	
1714		----	----	----	----	----	----	
1720		----	----	----	----	----	----	
1842		----	----	----	----	----	----	
1990	D6839	87.955	0.200	47.430	40.525	10.600	1.445	
6009		----	----	----	----	----	----	
	normality	not OK	OK	OK	OK	not OK	OK	
	n	9	12	11	11	12	11	
	outliers	3	0	0	0	0	1	
	mean (n)	88.779	1.269	48.723	40.107	9.801	1.387	
	st.dev. (n)	0.3903	0.8675	0.8376	0.9009	0.7562	0.1389	
	R(calc.)	1.093	2.429	2.345	2.522	2.117	0.389	

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

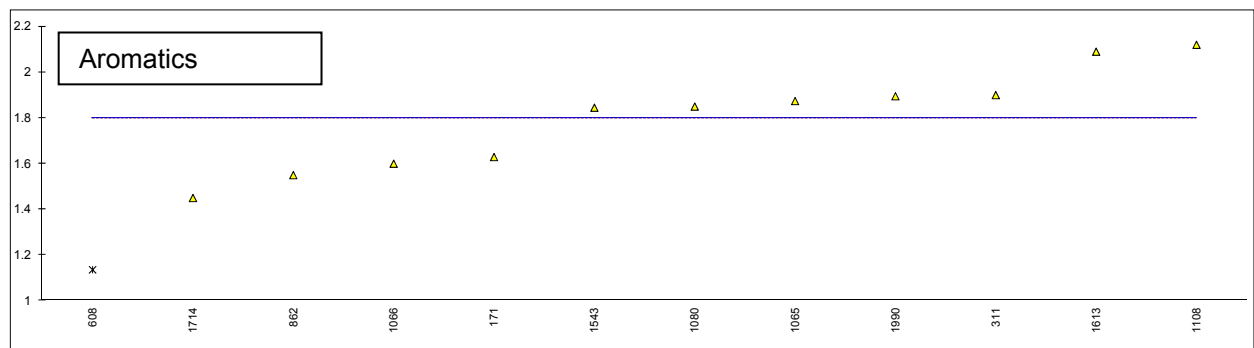
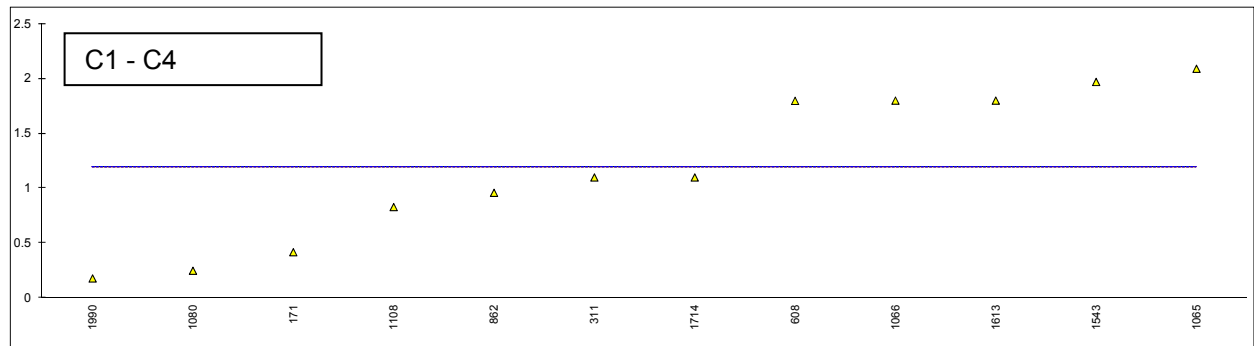
Lab 608 first reported for Total paraffins: 49.4220



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

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D5134M	87.161	0.419	48.609	38.552	11.194	1.629	
311	D5443	85.2	1.1	46.4	38.8	12.9	1.9	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445		----	----	----	----	----	----	
574		----	----	----	----	----	----	
608	D6730	89.3420	1.7976	48.3318	41.0102	9.4958	1.1361	
862	D6839	87.65	0.96	47.15	40.50	10.78	1.55	
971		----	----	----	----	----	----	
1065	in house	87.012	2.09	48.184	38.828	11.114	1.874	
1066	in house	86.7	1.8	47.6	39.1	11.7	1.6	
1080	in house	87.01	0.25	47.79	39.22	11.12	1.85	
1089		----	----	----	----	----	----	
1095		----	----	----	----	----	----	
1108	D5443	86.87	0.83	48.22	38.65	11.07	2.12	
1264		----	----	----	----	----	----	
1539		----	----	----	----	----	----	
1543	D5134	89.28	1.970	46.52	40.79	10.79	1.845	
1613	D6839	87.27	1.80	----	----	10.64	2.09	
1616		----	----	----	----	----	----	
1714	in house	87.94	1.10	49.25	38.69	10.61	1.45	
1720		----	----	----	----	----	----	
1842		----	----	----	----	----	----	
1990	D6839	86.125	0.180	46.130	39.995	11.985	1.895	
6009		----	----	----	----	----	----	
	normality	OK	OK	OK	OK	suspect	OK	
	n	9	12	11	11	12	11	
	outliers	3	0	0	0	0	1	
	mean (n)	87.082	1.191	47.653	39.467	11.117	1.800	
	st.dev. (n)	0.5264	0.6894	0.9988	0.9292	0.8320	0.2167	
	R(calc.)	1.474	1.930	2.797	2.602	2.330	0.607	

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

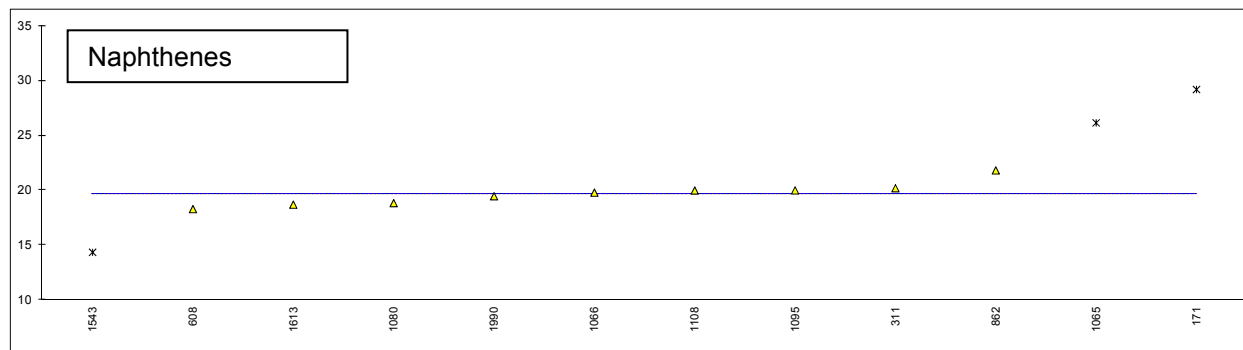
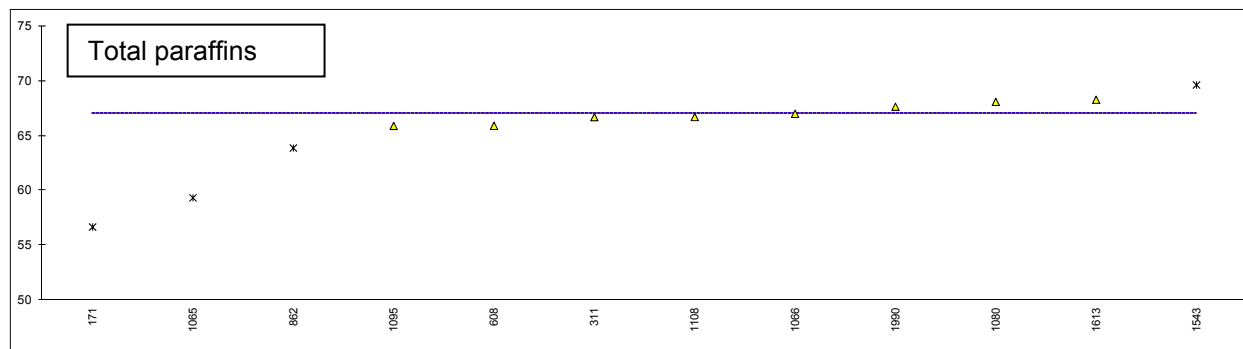


Determination of P(iP)NA on distillation fraction 3 (heavy naphtha); results in %V/V

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D5443	56.68	0.01	----	----	29.19	12.83	
311	D5443	66.7	<0.2	30.6	35.6	20.2	13.1	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445		----	----	----	----	----	----	
574		----	----	----	----	----	----	
608	D6730	65.9090	0.0618	31.5773	34.3317	18.2913	12.6652	
862	D6839	63.88	0	33.20	30.68	21.82	14.29	
971		----	----	----	----	----	----	
1065	in house	59.343	0.40	31.270	28.073	26.151	14.505	
1066	in house	67.0	<0.1	31.4	35	19.8	13.2	
1080	in house	68.09	0.03	31.43	36.64	18.84	12.92	
1089		----	----	----	----	----	----	
1095	in house	65.9	0	31.7	34.2	20.0	13.2	
1108	D5443	66.71	0.04	31.89	34.82	19.99	13.30	
1264		----	----	----	----	----	----	
1539		----	----	----	----	----	----	
1543	D5134	69.63 *	0.0390 *	35.28 *	34.31 *	14.36 *	13.20 *	Method not applicable, see §4.1
1613	D6839	68.28	----	----	----	18.70	13.02	
1616		----	----	----	----	----	----	
1714		----	----	----	----	----	----	
1720		----	----	----	----	----	----	
1842		----	----	----	----	----	----	
1990	D6839	67.645	0	32.700	34.945	19.470	12.885	
6009		----	----	----	----	----	----	
	normality	OK	n.a.	OK	not OK	suspect	OK	
	n	8	7	9	7	9	9	
	outliers	3 (+1ex)	n.a.	0 (+1ex)	2 (+1ex)	2 (+1ex)	2 (+1ex)	
	mean (n)	67.029	0.020	31.752	35.077	19.679	13.013	
	st.dev. (n)	0.9110	n.a.	0.7774	0.8297	1.0429	0.2060	
	R(calc.)	2.551	n.a.	2.177	2.323	2.920	0.577	

NB: Bold and underlined figures are statistical outliers (Dixon, Grubbs and / or Rosner) or test result is excluded

*) Test results of lab 1543 excluded for method not applicable for heavy naphtha (carbon atoms higher than C7)

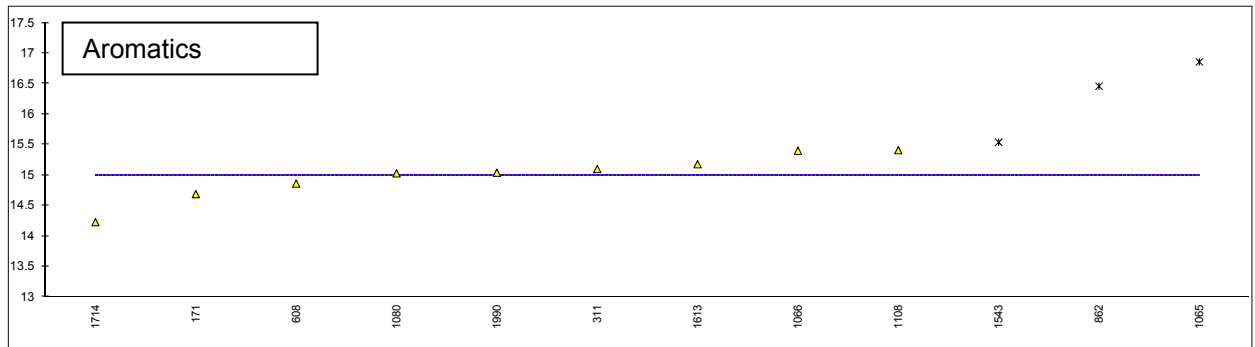
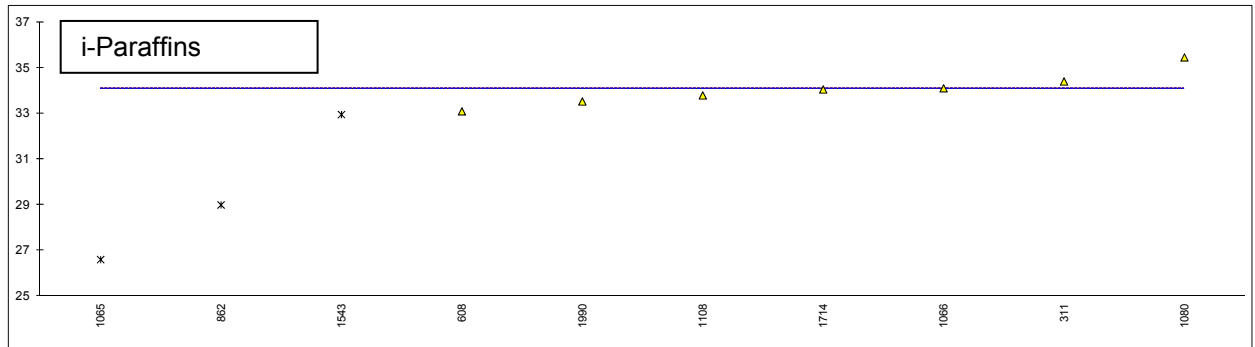


Determination of P(iP)NA on distillation fraction 3 (heavy naphtha); results in %M/M

lab	method	total par	C1-C4	n-par	i-par	naphth.	arom.	Remarks
171	D5443	53.28	0.01	----	----	30.55	14.69	
311	D5443	63.8	<0.2	28.7	34.4	21.1	15.1	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445		----	----	----	----	----	----	
574		----	----	----	----	----	----	
608	D6730	63.0805	0.0481	29.9883	33.0922	19.0978	14.8622	
862		60.16	0	31.16	29.00	23.38	16.46	
971		----	----	----	----	----	----	
1065	in house	55.826	0.31	29.216	26.610	27.316	16.858	
1066	in house	63.8	<0.1	29.7	34.1	20.9	15.4	
1080	in house	65.08	0.02	29.63	35.45	19.76	15.03	
1089		----	----	----	----	----	----	
1095		----	----	----	----	----	----	
1108	D5443	63.73	0.03	29.94	33.79	20.86	15.41	
1264		----	----	----	----	----	----	
1539		----	----	----	----	----	----	
1543	D5134	66.65 *	0.0300 *	33.67 *	32.95 *	15.02 *	15.54 *	Method not applicable, see §4.1
1613	D6839	65.23	----	----	----	19.59	15.18	
1616		----	----	----	----	----	----	
1714	in house	63.71	0.01	29.66	34.05	21.36	14.23	
1720		----	----	----	----	----	----	
1842		----	----	----	----	----	----	
1990	D6839	64.530	0	31.005	33.525	20.430	15.040	
6009		----	----	----	----	----	----	
	normality	OK	n a.	OK	suspect	not OK	suspect	
	n	8	7	9	7	9	9	
	outliers	3 (+1ex)	n.a.	0 (+1ex)	2 (+1ex)	2 (+1ex)	2 (+1ex)	
	mean (n)	64.120	0.017	29.889	34.058	20.720	14.994	
	st.dev. (n)	0.7490	n a.	0.7822	0.7467	1.2548	0.3675	
	R(calc.)	2.097	n a.	2.190	2.091	3.513	1.029	

NB: Bold and underlined figures are statistical outliers (Dixon, Grubbs and / or Rosner) or test result is excluded

*) Test results of lab 1543 excluded for method not applicable for heavy naphtha (carbon atoms higher than C7)



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

Lab method	IBP	5% evap.	10% evap.	50% evap.	90% evap.	95% evap.	FBP	remarks
171 D86	189.4	195.0	197.0	207.5	226.5	231.3	236.4	
311 D86	189.7	200.3	202.0	212.4	232.4	237.5	243.1	
391	189	----	----	----	----	----	240	
442	----	----	----	----	----	----	----	
445	----	----	----	----	----	----	----	
574 D86	<u>196.3</u> *	----	----	----	----	----	<u>311.8</u>	
608 D86	186.8	196.8	199.3	208.4	224.8	229.2	242.5	
862 D86	195.0	204.0	205.1	214.7	232.3	236.4	242.2	
971 D86	189.8	199.2	200.5	212.3	230.8	235.4	245.5	
1065 D86	186.3	192.6	195.5	208.1	227.4	232.0	238.3	
1066	196.3	202.7	203.7	213.2	231.8	235.9	241.1	
1080	----	----	----	----	----	----	----	
1089	----	----	----	----	----	----	----	
1095 D7345	193.1	198.6	199.6	210.0	228.7	234.7	240.6	
1108 D86	188.5	197.1	199.7	216.3	240.5	246.4	<u>264.5</u>	
1264	----	----	----	----	----	----	----	
1539 D86	191.1	199.6	200.7	211.5	229.8	234.2	240.6	
1543	----	----	----	----	----	----	----	
1613 D86	196.7	202.2	204.3	216.3	235.8	240.5	246.4	
1616	----	----	----	----	----	----	----	
1714	----	----	----	----	----	----	----	
1720	----	----	----	----	----	----	----	
1842 D86	186.1	195.2	198.4	210.3	229.5	234.4	251.5	
1990 D86	189.55	----	----	----	----	----	250.25	
6009	----	----	----	----	----	----	----	
normality	OK	OK	OK	OK	suspect	not OK	OK	
n	14	12	12	12	12	12	13	
outliers	0 (+1ex)	0	0	0	0	0	2	
mean (n)	190.53	198.61	200.48	211.75	230.86	235.66	242.96	
st.dev. (n)	3.504	3.427	2.903	3.027	4.243	4.488	4.409	
R(calc.)	9.81	9.60	8.13	8.47	11.88	12.57	12.35	

NB: Bold and underlined figures are statistical outliers (Dixon, Grubbs and / or Rosner) or excluded test results (*)

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

Lab method	5% rec.	10% rec.	50% rec.	90% rec.	95% rec.	%recovery	%residue	remarks
171 D86	195.8	197.3	208.0	227.4	232.9	97.6	1.1	
311 D86	200.4	202.0	212.5	232.6	237.7	98.6	1.2	
391	203	205	213	231	236	99	0.5	
442	----	----	----	----	----	----	----	
445	----	----	----	----	----	----	----	
574 D86	<u>211.4</u> *	<u>218.4</u>	<u>261.8</u>	<u>295.8</u>	<u>301.7</u>	99	0.5	
608 D86	197.5	199.5	208.5	225.0	229.5	98.5	1.2	
862 D86	204.1	205.2	214.7	232.4	236.6	99.0	0.4	
971 D86	199.5	200.9	212.5	231.3	236.1	98.5	1.0	
1065 D86	194.5	196.1	208.9	229.2	235.4	<u>96.5</u>	1.3	
1066	202.9	204.0	213.3	232.1	236.4	<u>98.5</u>	1.0	
1080	----	----	----	----	----	----	----	
1089	----	----	----	----	----	----	----	
1095 D7345	----	----	----	----	----	98.1	1.3	
1108 D86	197.2	200.0	216.4	<u>240.8</u>	<u>246.8</u>	99.2	0.6	
1264	----	----	----	----	----	----	----	
1539 D86	199.7	200.9	211.6	230.2	234.8	98.4	1.1	
1543	----	----	----	----	----	----	----	
1613 D86	203.1	204.6	216.8	<u>237.0</u>	<u>242.6</u>	98.8	1.2	
1616	----	----	----	----	----	----	----	
1714	----	----	----	----	----	----	----	
1720	----	----	----	----	----	----	----	
1842 D86	194.9	198.1	210.0	229.2	234.1	98.7	1.0	
1990 D86	197.80	201.20	211.50	230.95	236.05	98.05	0.9	
6009	----	----	----	----	----	----	----	
normality	OK	OK	OK	suspect	not OK			
n	13	13	13	11	11			
outliers	0 (+1ex)	1	1	3	3			
mean (n)	199.26	201.14	212.13	230.12	235.05			
st.dev. (n)	3.292	2.969	2.813	2.312	2.253			
R(calc.)	9.22	8.31	7.88	6.47	6.31			

NB: Bold and underlined figures are statistical outliers (Dixon, Grubbs and / or Rosner) or excluded test results (*)

APPENDIX 1D – SIMULATED DISTILLATION AND EFFECTIVE CUT POINT D2892

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

Lab	185-192°C	192-199°C	199-206°C	206-213°C	213-220°C	220-227°C	227-234°C	234-241°C	241-248°C
171	13.9	22.8	14.2	11.4	10.0	1.9	0.9	0.0	0.0
311	10.5	12.0	21.0	15.5	13.0	8.3	2.4	0.8	0.0
391	----	----	----	----	----	----	----	----	----
442	----	----	----	----	----	----	----	----	----
445	----	----	----	----	----	----	----	----	----
574	----	----	----	----	----	----	----	----	----
608	14.0	25.2	12.5	15.5	13.2	3.1	1.0	0.0	0.0
862	----	----	----	----	----	----	----	----	----
971	----	----	----	----	----	----	----	----	----
1065	----	----	----	----	----	----	----	----	----
1066	14.1	17.5	23.0	15.7	12.1	3.2	0.0	0.0	0.0
1080	----	----	----	----	----	----	----	----	----
1089	----	----	----	----	----	----	----	----	----
1095	6.3	8.7	6.3	5.4	7.6	4.2	4.3	4.2	1.6
1108	----	----	----	----	----	----	----	----	----
1264	----	----	----	----	----	----	----	----	----
1539	----	----	----	----	----	----	----	----	----
1543	----	----	----	----	----	----	----	----	----
1613	----	----	----	----	----	----	----	----	----
1616	----	----	----	----	----	----	----	----	----
1714	----	----	----	----	----	----	----	----	----
1720	----	----	----	----	----	----	----	----	----
1842	----	----	----	----	----	----	----	----	----
1990	10.0	10.4	21.0	13.9	12.9	12.8	3.9	1.0	0.0
6009	----	----	----	----	----	----	----	----	----
normality	unknown	unknown	unknown	unknown	unknown	unknown	unknown	unknown	n.a
n	6	6	6	6	6	6	6	5	5
outliers	0	0	0	0	0	0	0	1	1
mean (n)	11.48	16.10	16.33	12.90	11.47	5.57	2.10	0.37	0.0
st.dev. (n)	3.1289	6.847	6.446	4.009	2.236	4.167	1.736	0.517	n.a.
R(calc.)	8.76	19.17	18.05	11.23	6.26	11.67	4.86	1.45	n.a.

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

Lab	185-192°C	192-199°C	199-206°C	206-213°C	213-220°C	220-227°C	227-234°C	234-241°C	241-248°C
171	0.0	1.9	2.9	6.0	14.9	9.8	16.1	20.9	13.5
311	0.2	1.3	1.5	4.0	9.0	9.0	14.0	18.5	13.5
391	----	----	----	----	----	----	----	----	----
442	----	----	----	----	----	----	----	----	----
445	----	----	----	----	----	----	----	----	----
574	----	----	----	----	----	----	----	----	----
608	0.0	0.5	1.0	3.5	14.0	9.6	15.3	26.5	11.7
862	----	----	----	----	----	----	----	----	----
971	----	----	----	----	----	----	----	----	----
1065	----	----	----	----	----	----	----	----	----
1066	0.0	0.1	1.2	2.7	10.7	11.3	16.5	20.8	12.7
1080	----	----	----	----	----	----	----	----	----
1089	----	----	----	----	----	----	----	----	----
1095	0.0	1.6	2.0	5.1	11.3	10.5	13.5	21.0	11.5
1108	----	----	----	----	----	----	----	----	----
1264	----	----	----	----	----	----	----	----	----
1539	----	----	----	----	----	----	----	----	----
1543	----	----	----	----	----	----	----	----	----
1613	----	----	----	----	----	----	----	----	----
1616	----	----	----	----	----	----	----	----	----
1714	----	----	----	----	----	----	----	----	----
1720	----	----	----	----	----	----	----	----	----
1842	----	----	----	----	----	----	----	----	----
1990	0.0	1.0	1.3	2.6	7.6	7.8	11.8	17.8	20.4
6009	----	----	----	----	----	----	----	----	----
normality	n.a.	unknown	unknown	unknown	unknown	unknown	unknown	unknown	unknown
n	6	6	6	6	6	6	6	5	5
outliers	n.a.	0	0	0	0	0	0	1	1
mean (n)	0.0	1.08	1.64	3.98	11.25	9.67	14.53	19.80	12.58
st.dev. (n)	n.a.	0.665	0.693	1.353	2.813	1.209	1.774	1.528	0.955
R(calc.)	n.a.	1.86	1.94	3.79	7.89	3.39	4.97	4.28	2.67

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

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

Lab	Overlap cuts 4 and 5 in °C	ECP in °C	Difference with AET=215 °C	Conclusion*	Remarks
171	192-230°C	213.0	-1.95	OK	
311	191-237°C	219.6	+4.58	OK	
391	----	----	----	----	
442	----	----	----	----	
445	----	----	----	----	
574	----	----	----	----	
608	199-230°C	216.2	1.22	OK	
862	----	----	----	----	
971	----	----	----	----	
1065	----	----	----	----	
1066	198-226°C	216.4	1.40	OK	
1080	----	----	----	----	
1089	----	----	----	----	
1095	194-268.5°C	212.04	-2.96	OK	large overlap cuts
1108	----	----	----	----	
1264	----	----	----	----	
1539	----	----	----	----	
1543	----	----	----	----	
1613	----	----	----	----	
1616	----	----	----	----	
1714	----	----	----	----	
1720	----	----	----	----	
1842	----	----	----	----	
1990	189-236°C	223.9	8.92	not OK	
6009	----	----	----	----	

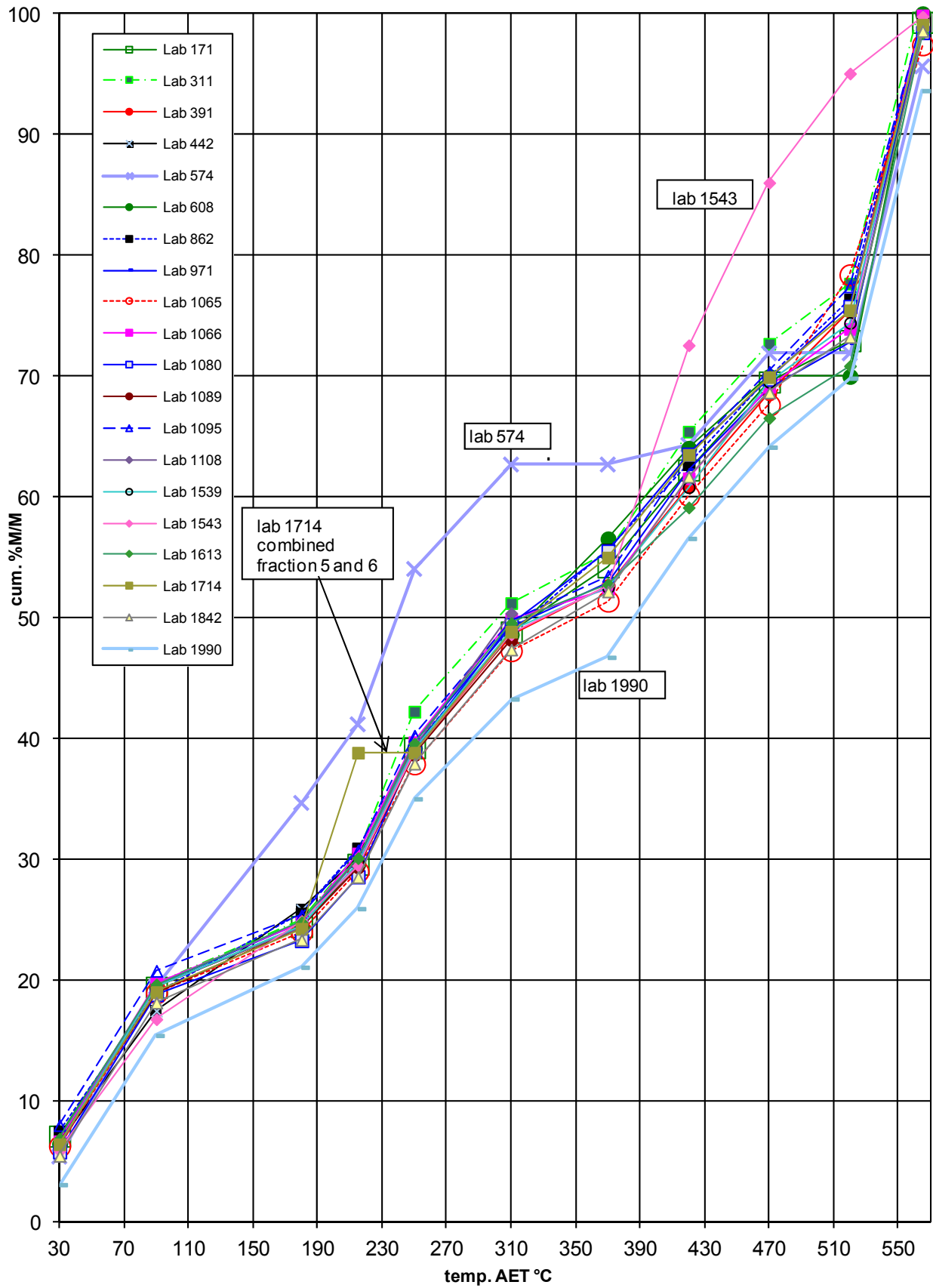
* Acc. To ASTM D2892:13, Appendix X2.6.5.1 the difference between ECP and AET should not exceed $0.7R$ °C ($0.7 \times 8 = 5.6$ °C)

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

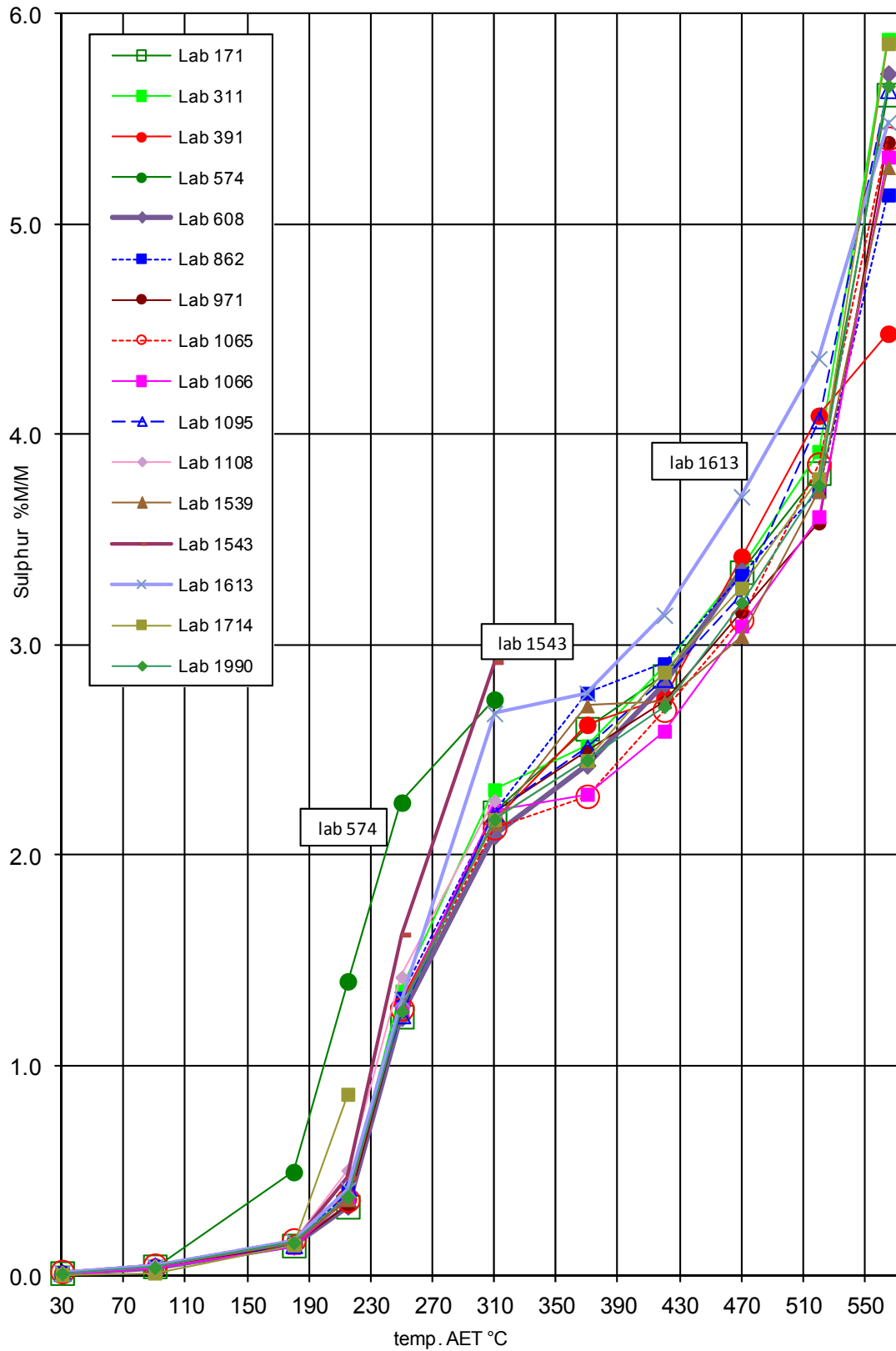
Lab	N_{actual}	N_{minimum}	Requirement $5.4 < N_{\text{minimum}} < 6.8^*$	Remarks
171	7.4	6.3	OK	
311	6.9	5.9	OK	
391	----	----	----	
442	----	----	----	
445	----	----	----	
574	----	----	----	
608	8.6	7.4	not OK	
862	----	----	----	
971	----	----	----	
1065	----	----	----	
1066	10.0	8.6	not OK	
1080	----	----	----	
1089	----	----	----	
1095	3.8	3.1	not OK	
1108	----	----	----	
1264	----	----	----	
1539	----	----	----	
1543	----	----	----	
1613	----	----	----	
1616	----	----	----	
1714	----	----	----	
1720	----	----	----	
1842	----	----	----	
1990	5.4	4.6	not OK	
6009	----	----	----	

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

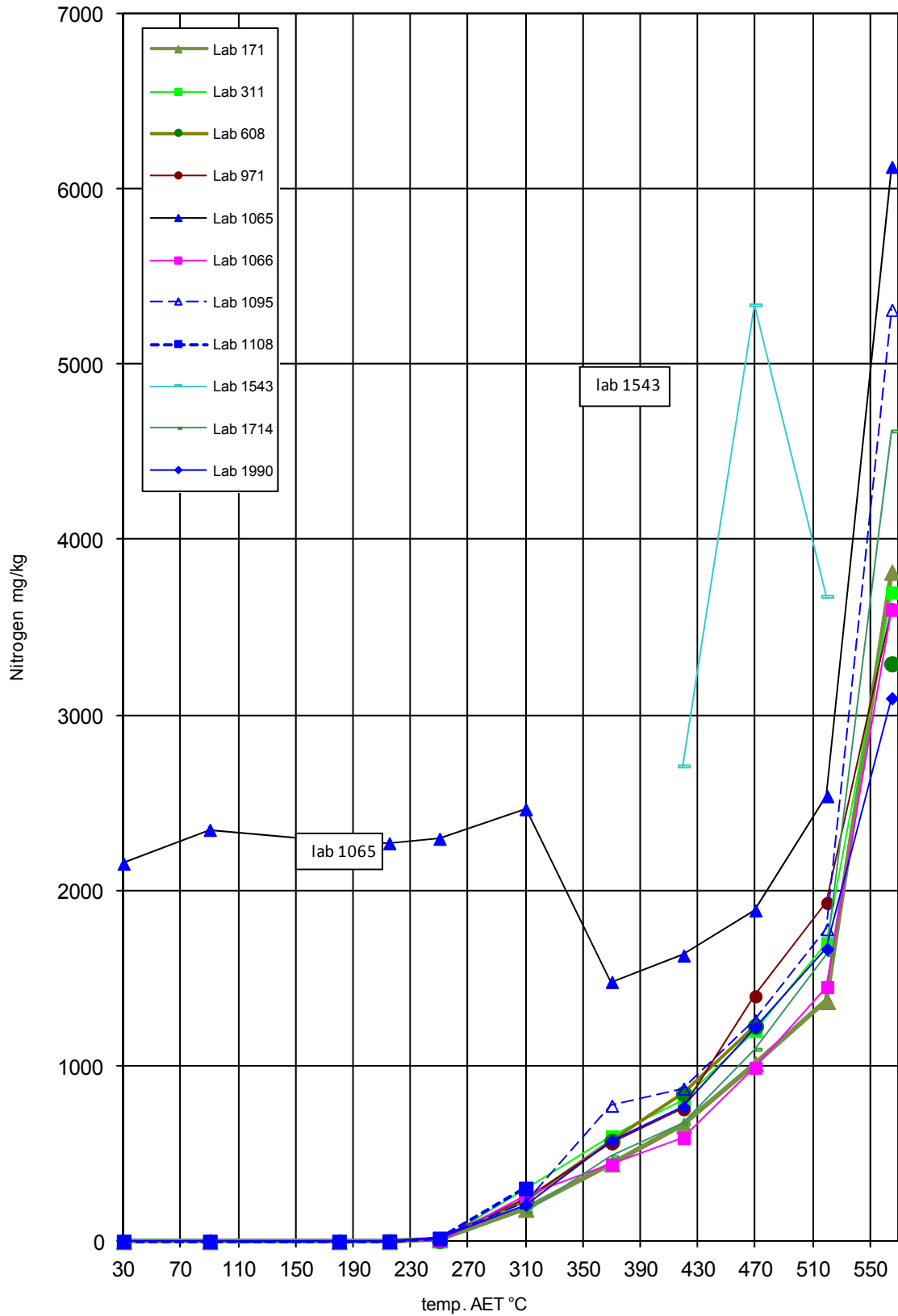
APPENDIX 2 True boiling point curve, cum%M/M vs temp AET in °C



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



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



APPENDIX 5**Details of Distillation**

<u>Lab</u>	<u>Sample received</u>	<u>Distillation started</u>	<u>Results reported</u>	<u>Intake in mL</u>	<u>Pressure at start of dist. in mm Hg</u>	<u>End Point of distillation(s) in °C</u>
171	09-Nov-15	30-Nov-15	16-Dec-15	3968.0	757	551+
311	15-Oct-15	27-Oct-15	27-Nov-15	4137	758	565+
391	03-Nov-15	17-Nov-15	20-Nov-15	4368.0	758.2	565+
442	30-Oct-15	11-Nov-15	20-Nov-15	6550.7	760	370+
445	02-Nov-15	----	----	----	----	----
574	02-Nov-15	11-Nov-15	20-Nov-15	2796.2	560	520+
608	24-Nov-15	19-Jan-16	09-Mar-16	4180.3	760	520+
862	09-Nov-15	----	11-Dec-15	6323.50	760	565+
971	03-Nov-15	08-Nov-15	19-Nov-15	8274.6	760	565+
1065	11-Nov-15	17-Nov-15	26-Nov-15	5219	740	554+
1066	02-Nov-16	16-Nov-15	04-Dec-15	3853.2	761	565+
1080	28-Oct-15	11-Nov-15	20-Nov-15	1310	761.0	565+
1089	03-Nov-15	12-Nov-15	20-Nov-15	3001.6	750.8	370+
1095	----	16-Nov-15	17-Dec-15	8330.1	768	565+
1108	23-Oct-15	02-Nov-15	20-Nov-15	3302.5	755	370+
1264	6-11-205	----	----	----	----	----
1539	23-Oct-15	18-Nov-15	20-Nov-15	3000	750.64	565+
1543	----	----	20-Jan-16	13182.286	720.41	557.3+
1613	12-Nov-15	17-Nov-15	21-Nov-15	4000	760	550+
1616	11-Nov-15	----	----	----	----	----
1714	27-Oct-15	03-Nov-15	26-Nov-15	4232.0	762	565+
1720	18-Nov-15	----	----	----	----	----
1842	30-Oct-15	09-Nov-15	20-Nov-15	2746.1	765.5	565+
1990	30-Oct-15	07-Nov-15	19-Nov-15	8630.75	757.0	557+
6009	----	----	----	----	----	----

Lab Remarks

171	
311	
391	
442	Cut between H. Naphtha and Kerosene was 160° instead of 180°C
445	
574	The sample only distilled up 520°C, after that you could see the cracking .
608	
862	
971	
1065	Fraction 9 approx density, a max temp of 554 was reached
1066	Loss potstill 0.15%
1080	
1089	
1095	Mass of the residue charged to potstill = 2562.4g (vol dist flask D5236 = 6L)
1108	
1264	
1539	
1543	
1613	The blending ratio of comb. fr. 4+5 (180-250°C) is 1:1 (dist acc to ASTM D86)
1616	
1714	Forgot to separate LGO and MGO (no SIMDIST/D86 results)
1720	
1842	
1990	Vacuum Distillation cracked at 557°C
6009	

APPENDIX 6

Number of participants per country

- 1 lab in ALGERIA
- 1 lab in CHINA, People's Republic
- 1 lab in COLOMBIA
- 1 lab in GREECE
- 1 lab in HUNGARY
- 1 lab in ISRAEL
- 1 lab in ITALY
- 1 lab in JORDAN
- 2 labs in MALAYSIA
- 3 labs in NETHERLANDS
- 1 lab in NORWAY
- 1 lab in POLAND
- 1 lab in PORTUGAL
- 1 lab in QATAR
- 2 labs in SUDAN
- 2 labs 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)