

**Results of Proficiency Test
Crude Oil Assay
November 2021**

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Authors: ing. M. Meijer & ing. C.M. Nijssen-Wester
Correctors: ing. A.S. Noordman-de Neef & ing. R.J. Starink
Report: iis21R02

April 2022

CONTENTS

1	INTRODUCTION	3
2	SET UP.....	3
2.1	ACCREDITATION.....	3
2.2	PROTOCOL	3
2.3	CONFIDENTIALITY STATEMENT	3
2.4	SAMPLES	4
2.5	STABILITY OF THE SAMPLES	4
2.6	ANALYZES	5
3	RESULTS.....	5
3.1	STATISTICS	6
3.2	GRAPHICS	6
3.3	Z-SCORES.....	7
4	EVALUATION	7
4.1	EVALUATION PER TEST	8
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	13
4.3	COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2021 WITH PREVIOUS PTS.....	14
5	CONCLUSION	14
Appendices:		
1.	Data, statistical and graphic results.....	15
	A – Analysis of original sample.....	15
	B – Distillation results D2892 and D5236	19
	C – Analysis of distillation fractions	28
	D – Simulated distillation and Effective Cut Point D2892.....	40
2.	True Boiling Point graph, cumulative %M/M vs Temperature AET in °C.....	43
3.	True Boiling Point graph, Sulfur in %M/M vs Temperature AET in °C	44
4.	True Boiling Point graph, Nitrogen in mg/kg vs Temperature AET in °C	45
5.	Details of Distillation and Pressure during ASTM D2892 and ASTM D5236	46
6.	Number of participants per country	47
7.	Abbreviations and literature	48

1 INTRODUCTION

Since 2004 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Crude Oil Assay every two years. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of Crude Oil Assay.

In this interlaboratory study 26 laboratories in 16 different countries registered for participation. See appendix 6 for the number of participants per country. In this report the results of the Crude Oil Assay proficiency test are presented and discussed. This report is electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.

It was decided to send one sample Crude Oil Assay in one or more 5-liter cans (dependent on the need of participants) labelled #21225.

The 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 agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of approximately 400 liters of Crude Oil was obtained from a local refinery. After homogenization 77 metal cans of 5L were filled and labelled #21225.

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

	Density at 15 °C in kg/m ³
sample #21225-1	873.05
sample #21225-2	873.04
sample #21225-3	873.09
sample #21225-4	873.15
sample #21225-5	873.16
sample #21225-6	873.34
sample #21225-7	873.18
sample #21225-8	873.23

Table 1: homogeneity test results of subsamples #21225

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

	Density at 15 °C in kg/m ³
r (observed)	0.28
reference test method	ASTM D5002:19
0.3 x R (reference test method)	1.08

Table 2: evaluation of the repeatability of subsamples #21225

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories the requested amount of Crude Oil labelled #21225 was sent on October 13, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Crude Oil was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine on the original sample: Density at 15°C, Sulfur, Nitrogen and Water. The participants were further requested to determine a Crude Oil Assay consisting of a True Boiling Point Distillation in accordance with ASTM D2892 (collection of 8 fractions) and subsequently a distillation in accordance with ASTM D5236 (collection of 4 fractions). Also was requested to determine Density, Sulfur and Nitrogen on all fractions where possible.

Furthermore, it was requested to determine:

- on the light and heavy naphtha fractions (fractions 2 and 3) a PNA or PiPNA analysis
- on the combined fractions of light and medium gasoil (fractions 5 and 6) Distillation and Flash Point PMcc
- on the separate fractions of light Gasoil (fraction 5) and medium Gasoil (fraction 6) a simulated distillation determination (on both fractions).

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

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

3 RESULTS

During thirteen weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/ and via e-mail for the SimDist data only. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

After appropriate time intervals, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the closure of the Data Entry Portal the available test results were screened for gross errors and/or missing data. The laboratories that produced these gross errors were asked to check the reported test results (no reanalyzes) and laboratories were reminded that data was missing. Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

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

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

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are shown as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

In this proficiency test no major problems were encountered with sample dispatch. To allow for as many participants to report their test results and to obtain these test results in digital format (except for the SimDist test results) the data entry portal was left open until mid January 2022. This corresponds to an extended reporting time of about four weeks. After this period six participants did not report any test results. Not all participants were able to report all tests requested. In total the 20 participants reported 1402 numerical test results. Observed were 64 outlying test results, which is 4.6%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 7.

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

For objective evaluation iis uses reproducibilities taken from reference test methods to calculate target z-scores. Regretfully this is not possible for the Density, Sulfur and Nitrogen test results from the fractions. These test results were determined on a distillation fraction produced by the laboratory itself and therefore also the distillation uncertainty is included in these test results. Therefore, other ways were sought to enable objective evaluation of the test results gathered. Calculated (theoretical) Density, Sulfur and Nitrogen of the original Crude Oil sample were calculated by iis from the masses of the collected fractions and the respective reported Density, Sulfur and Nitrogen test results using below formulae. These calculated (theoretical) values were compared with the measured values as reported by the participants.

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

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

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

Analysis of the original sample (appendix 1A)

Density at 15°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5002:19.

Sulfur: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D4294:16e1.

Nitrogen: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D5762:18a.

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

Distillation results D2892 and D5236 (appendix 1B)

Seventeen laboratories performed the atmospheric distillation (D2892) and thirteen also the vacuum distillation (D5236).

All of the participants reported to have used a starting pressure at atmospheric conditions for the atmospheric distillation (D2892). ASTM D2892 states that it is not allowed to let the Observed Boiling Temperature of the liquid exceed 310°C or the Observed Vapor Temperature exceed 210°C as mentioned in paragraph 10.3.7 of ASTM D2892. According to paragraphs 10.3 till 10.5 of the same method this can be prevented by lowering the pressure in the system. The method also states that after atmospheric pressure a pressure around 100 mmHg should be applied and if necessary or applicable also a lower pressure between 100 and 2 mmHg is permitted in only one step.

Laboratory 1613 still applied a pressure of 100 mmHg for fraction 6, while the Observed Vapor Temperature was above 210°C (see appendix 5).

Laboratory 6201 applied a pressure of 2 mmHg after the atmospheric pressure (fraction 4/5) while skipping the pressure step of 100 mmHg (see appendix 5).

These differences in temperature and/or pressure does not appear to have a significant effect on the reported test results of the fractions of the atmospheric distillation.

The details of the distillation(s) reported by the participants can be found in appendices 1B and 5.

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

- D2892:** This distillation was not problematic. In total four statistical outliers were observed and one other test result was excluded. The calculated reproducibilities after rejection of the suspect data are in agreement with the requirements of ASTM D2892:20.
- D5236:** This distillation may be problematic. Two statistical outliers were observed. Since the reproducibility of ASTM D5236 is not expressed in mass %, but in °C per 10% liquid volume recovered, the reproducibility of this method cannot be used for the test results in this PT. Since ASTM D2892 also gives a reproducibility at lower operating pressure, this has been used as a guideline in the evaluation. Because of this no z-scores are calculated. The calculated reproducibilities after rejection of the statistical outliers are not in agreement with the requirements of ASTM D2892:20 (low pressure), except for the VGO fraction at 470-520°C.
- Mass balance:** The evaluation of the Total Mass balance showed eight recoveries that meet the ASTM D2892 (paragraph 11.2) requirement of 0.4% max loss and the ASTM D5236 requirement of a recovery between 99.6 and 100.1%. Remarkably, three recoveries were equal to 100%. They were excluded in this table for it is unlikely that no loss occurred in both distillations.
- Volume balance:** All laboratories reported a test result for the mass fraction for Gas (LPG), but some did not report the Density of this fraction (see appendix 1C). In order to calculate the individual volume recoveries, the mean value of Density of the group was used when the Density of Gas (LPG) was not reported.
- Calculation (theoretical) values:** The Density, Sulfur and Nitrogen test results do show some deviating results. These deviations may be (partly) explained by the cumulative effect of variations caused by distillation and by subsequent analytical determinations. Still, the calculated averages for Density, Sulfur and Nitrogen are in line with the averages of the reported Density, Sulfur and Nitrogen test values (see table 3).

parameter	unit	average laboratories	average iis	absolute difference	R(lit)
Density	kg/L	0.8737	0.8711	0.0026	0.0036
Sulfur	%M/M	2.616	2.521	0.095	0.1319
Nitrogen	mg/kg	1254	1245	9	331

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

Analysis of distillation fractions (appendix 1C)

- Density:** This determination on the 12 collected distillation fractions show relatively small differences between the reported test results of the participating laboratories, except for the first Gas (LPG) fraction. In total seven statistical outliers were observed. Observed variations are most probably caused by differences in the distillations and not by differences in the performance of the Density determinations. In each case the observed reproducibility will be the sum of the (smaller) variation in the Density determination and the (larger) variation caused by the distillation. Therefore, the observed reproducibilities were not compared with the literature requirements and consequently no z-scores are calculated.
The average Density for the first fraction (gas <30°C) is 0.5752 kg/L, which is in good agreement with the Density of a mixture of C4 hydrocarbons and Isopentane.
- Sulfur:** This determination on the 12 collected distillation fractions show problems for a few laboratories. In total ten statistical outliers were observed. The observed reproducibility per fraction will be the sum of the variation in the Sulfur determination and the variation caused by the distillation. Therefore, the observed reproducibilities were not compared with the literature requirements and consequently no z-scores are calculated.
- Nitrogen:** This determination on the 12 collected distillation fractions show problems for a few laboratories. In total five statistical outliers were observed. The observed reproducibility per fraction will be the sum of the variation in the Nitrogen determination and the variation caused by the distillation. Therefore, the observed reproducibilities were not compared with the requirements and consequently no z-scores are calculated.
- P(iP)NA:** This determination was performed on fraction 2 (Light Naphtha) and on fraction 3 (Heavy Naphtha) and reported in %M/M and %V/V. On the Light Naphtha six statistical outliers were observed. More analytical problems were observed on fraction 3 (Heavy Naphtha) where in total sixteen statistical outliers were observed. Each observed reproducibility will be the sum of the variation in the analytical method and the variation caused by the distillation D2892. Therefore, the observed reproducibilities were not compared with the requirements and consequently no z-scores are calculated. The variety of test methods used may partly explain the variations observed during this PT. The set-up of the correct integration window is most critical in the case of testing high naphthenic distillation fractions.
- Distillation:** This determination was performed on the combined fractions 5 and 6 and may be problematic. Seven statistical outliers were observed and two other test results were excluded. Each observed reproducibility will be the sum of the variation in this distillation of the combined fractions and the variation caused by the D2892 distillation. Therefore, the observed reproducibilities

were not compared with the requirements and consequently no z-scores are calculated.

Flash point: This determination was performed on the combined fractions 5 and 6 and was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D93-A:20. Each observed test result will be the sum of the variation in this distillation of the combined fractions and the variation caused by the D2892 distillation.

Simulated Distillation and Effective Cut Point D2892 (appendix 1D)

SimDist: This determination was performed on both fractions 5 (Light Gasoil) and 6 (Medium Gasoil). This simulated distillation was performed by eleven participants on both fractions. Most reported test results are in line with each other. In total nine outliers were observed. The goal was to enable evaluation of the column efficiency in accordance with appendix X2 of ASTM D2892:20.

ECP/N_{minimum}: From the reported test results, the ECP (effective cut point) and the Standard Efficiency N_{minimum} were calculated. The ECP for one laboratory does not meet the requirements of ASTM D2892:20. The Standard Efficiency N_{minimum} requirements of ASTM D2892:20 is met by eight laboratories. The other three laboratories are not in agreement with the requirements of ASTM D2892:20 and had yielded (too) low total sum of mass fractions on distillation fraction 5.

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 iis19R02. A number of laboratories participated in both rounds and it is good to see that some participants improved (e.g. laboratory 1065) or maintained the quality of the distillation. Regretfully for other participants the performance of the distillation did not improve, based on the calculation of the ECP and the Standard Efficiency.

True Boiling Point graphs (appendices 2, 3 and 4)

Graph Cumulative %M/M (appendix 2): The true boiling point graph (cumulative %M/M vs temperature AET) of the laboratories show a good correlation. The line for laboratory 1108 is deviating from fraction 4 onwards. This is in line with the deviating test results in the distillation found in fraction 4 and higher for this laboratory. The line for laboratory 6432 is deviating at the high end of the graph due to reporting the same mass fractions for the last two fractions of the vacuum distillation.

Graph Sulfur (appendix 3): The true boiling point graph (Sulfur in %M/M vs temperature AET) of most laboratories show a good correlation, except for the line of laboratory 1108 which reported deviating Sulfur test results from fraction 4 and higher.

Graph Nitrogen (appendix 4): The true boiling point graph (Nitrogen in mg/kg vs temperature AET) of most laboratories show a good correlation. Laboratories 1108, 1539 and 1613 are not included in the graph due to missing data for Nitrogen.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility ($2.8 \cdot$ standard deviation) and the target reproducibility derived from reference test methods (in casu ASTM and EN standards) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Density of original sample	kg/L	20	0.8737	0.0011	0.0036
Sulfur of original sample	%M/M	16	2.616	0.109	0.135
Nitrogen of original sample	mg/kg	14	1254	349	334
Water of original sample	%V/V	15	0.031	0.067	0.030
D2892 distillation					
Gas LPG < 30°C	%M/M	17	1.5	1.2	1.3
Light Naphtha 30 - 90°C	%M/M	17	5.2	1.4	1.3
Heavy Naphtha 90 - 180°C	%M/M	17	12.8	1.0	1.3
Kerosene 180 - 215°C	%M/M	16	5.1	0.5	1.5
LGO 215 - 250°C	%M/M	16	5.3	0.7	1.5
MGO 250 - 310°C	%M/M	16	9.5	1.2	2.0
HGO 310 - 370°C	%M/M	16	9.5	0.8	2.0
Residue > 370°C	%M/M	15	50.5	2.9	n.a.
D5236 distillation					
VGO 370 - 420°C	%M/M	13	4.2	4.0	(2)
VGO 420 - 470°C	%M/M	13	8.6	4.5	(2)
VGO 470 - 520°C	%M/M	12	6.9	1.2	(2)
Residue > 520°C	%M/M	12	30.6	3.4	n.a.

Table 4: reproducibilities of test on sample #21225

For results between brackets no z-scores are calculated

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

For the analytical tests performed on the individual distillation fractions it was not possible to evaluate the reproducibilities against literature values as explained in paragraph 4.1.

Therefore, the values are not summarized in a table and can be found in appendix 1C.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2021 WITH PREVIOUS PTS

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

	November 2021	November 2019	November 2017	November 2015	November 2013
Density of original sample	++	++	+	+	++
Sulfur of original sample	+	+/-	++	-	-
Nitrogen of original sample	+/-	-	--	+	--
Water of original sample	--	-	+	--	-
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.a.	n.a.	n.a.	n.a.	n.a.
D5236 distillation					
VGO 370 - 420°C	(--)	(--)	(--)	(--)	(--)
VGO 420 - 470°C	(--)	(-)	(+/-)	(-)	(--)
VGO 470 - 520°C	(+)	(+/-)	(++)	(+/-)	(-)
Residue > 520°C	n.a.	n.a.	n.a.	n.a.	n.a.

Table 5: comparison determinations against the reference test methods

For results between brackets no z-scores are calculated

The following performance categories were used:

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

5 CONCLUSION

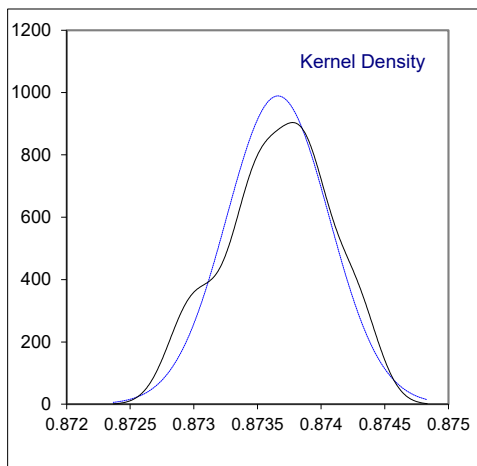
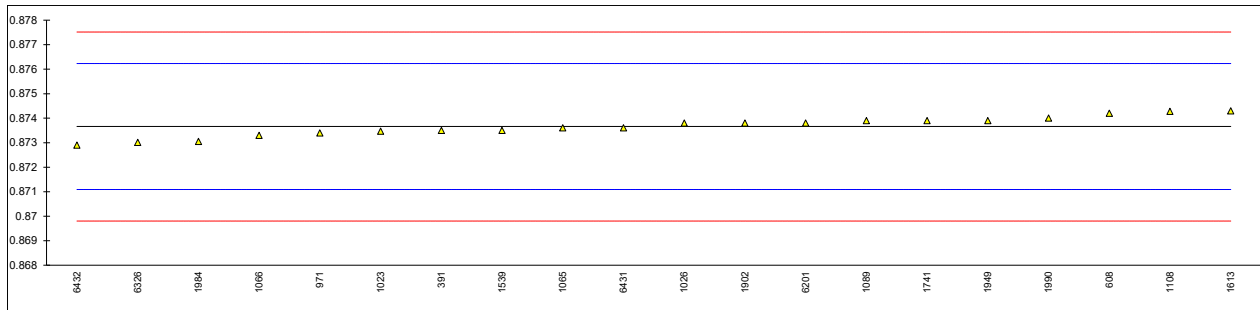
In spite of the practical problems and the differences between the methods used, the distillation graphs of most participating laboratories show a good correlation.

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

APPENDIX 1A – ANALYSIS OF ORIGINAL SAMPLE

Determination of Density at 15°C on original sample #21225; results in kg/L

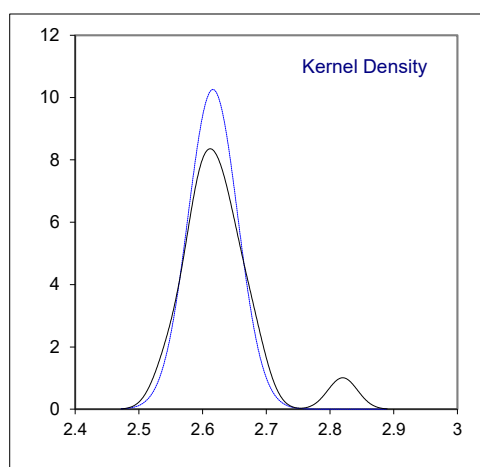
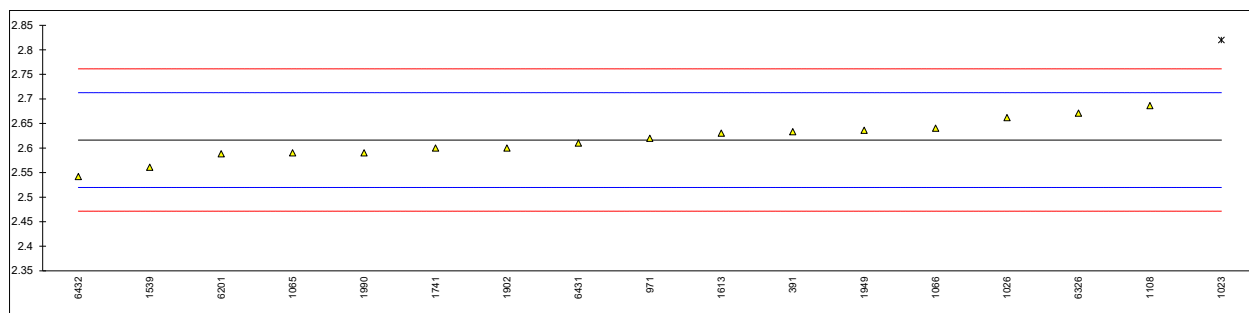
lab	method	value	mark	z(targ)	remarks
171		----		----	
391	D5002	0.8735		-0.13	
442		----		----	
445		----		----	
608	D5002	0.8742		0.42	
971	D5002	0.8734		-0.20	
1023	D5002	0.87347	C	-0.15	reported 873.47 kg/L
1026	D5002	0.87380		0.11	
1065	D4052	0.8736		-0.05	
1066	D5002	0.8733		-0.28	
1089	D5002	0.8739		0.19	
1108	D5002	0.87428		0.48	
1539	D4052	0.8735		-0.13	
1613	D5002	0.8743	C	0.50	reported 874.3 kg/L
1741	D4052	0.8739		0.19	
1842		----		----	
1902	D5002	0.8738		0.11	
1949	D5002	0.8739		0.19	
1984	D5002	0.87305		-0.48	
1990	D4052	0.8740		0.26	
6156		----		----	
6201	D5002	0.8738		0.11	
6326	D4052	0.873013		-0.50	
6379		----		----	
6431	D5002	0.8736		-0.05	
6432	D4052	0.8729		-0.59	
normality		OK			
n		20			
outliers		0			
mean (n)		0.8737			
st.dev. (n)		0.00040			
R(calc.)		0.0011			
st.dev.(D5002:19)		0.00129			
R(D5002:19)		0.0036			



Determination of Sulfur on original sample #21225; results in %M/M

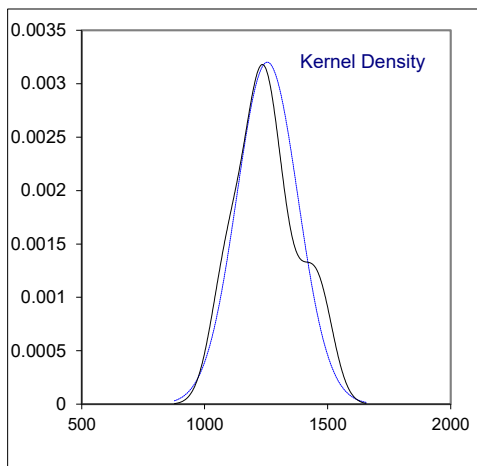
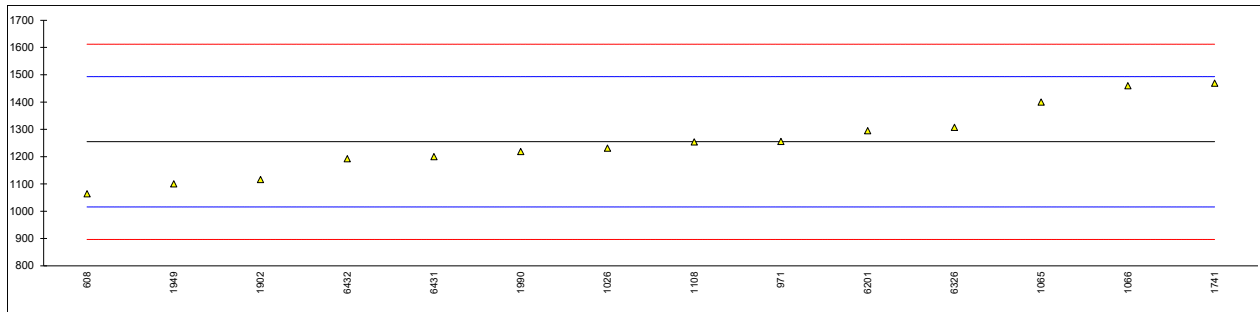
lab	method	value	mark	z(targ)	remarks
171		----		----	
391	D4294	2.633		0.35	
442		----		----	
445		----		----	
608		----		----	
971	D4294	2.62		0.08	
1023	IP336	2.82	G(0.01)	4.23	
1026	D2622	2.662		0.95	
1065	D4294	2.59		-0.54	
1066	D2622	2.64		0.49	
1089		----		----	
1108		2.686		1.45	
1539	D4294	2.561		-1.14	
1613	D4294	2.63		0.29	
1741	D4294	2.60		-0.34	
1842		----		----	
1902	D4294	2.60		-0.34	
1949	D4294	2.636		0.41	
1984		----		----	
1990	D4294	2.590		-0.54	
6156		----		----	
6201	D4294	2.588		-0.58	
6326	D2622	2.6708		1.13	
6379		----		----	
6431	D4294	2.61		-0.13	
6432	D4294	2.542		-1.54	

normality OK
n 16
outliers 1
mean (n) 2.6162
st.dev. (n) 0.03888
R(calc.) 0.1089
st.dev.(D4294:16e1) 0.04824
R(D4294:16e1) 0.1351



Determination of Nitrogen on original sample #21225; results in mg/kg

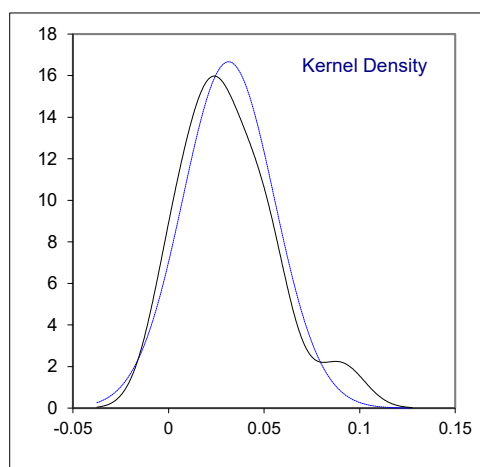
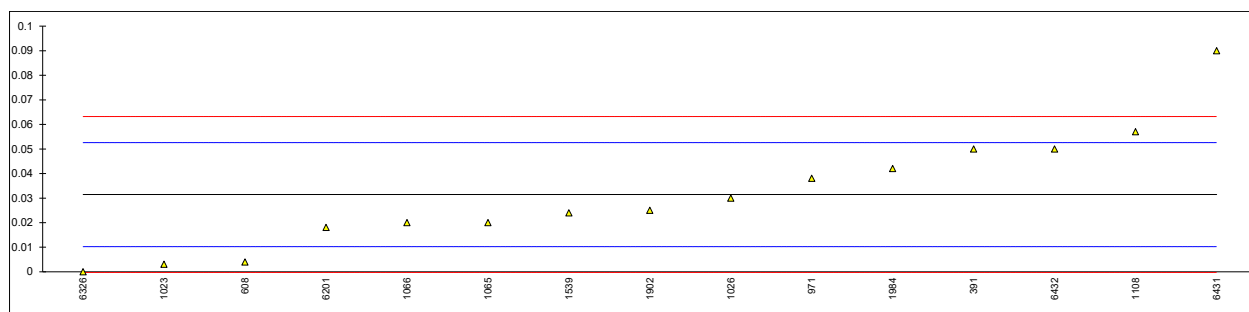
lab	method	value	mark	z(targ)	remarks
171		----		----	
391		----		----	
442		----		----	
445		----		----	
608	D5762	1063.9		-1.60	
971	D5762	1256		0.01	
1023		----		----	
1026	D5762	1231		-0.20	
1065	D4629	1400		1.22	
1066	D5762	1460		1.72	
1089		----		----	
1108		1253.4		-0.01	
1539		----		----	
1613		----		----	
1741	D5762	1469		1.80	
1842		----		----	
1902	D5762	1116		-1.16	
1949	D5762	1100		-1.30	
1984		----		----	
1990	D5762	1218.92		-0.30	
6156		----		----	
6201	D5762	1295		0.34	
6326	D5762	1307.538		0.45	
6379		----		----	
6431	D5762	1200		-0.46	
6432	D5762	1192		-0.52	
normality		OK			
n		14			
outliers		0			
mean (n)		1254.48			
st.dev. (n)		124.636			
R(calc.)		348.98			
st.dev.(D5762:18a)		119.176			
R(D5762:18a)		333.69			



Determination of Water on original sample #21225; results in %V/V

lab	method	value	mark	z(targ)	remarks
171		----		----	
391	D4928	0.050		1.76	
442		----		----	
445		----		----	
608	D4928	0.004		-2.59	
971	D4928	0.038		0.62	
1023	D4928	0.00305		-2.68	
1026	D4006	0.03		-0.13	
1065	D6304	0.02		-1.08	
1066	D4377	0.02		-1.08	
1089		----		----	
1108		0.057		2.42	
1539	D4377	0.024		-0.70	
1613	D4006	<0.05		----	
1741		----		----	
1842		----		----	
1902	D4006	0.025		-0.60	
1949	D4006	Less 0.025		----	
1984	D4377	0.042		1.00	
1990	D4007	<0.05		----	
6156		----		----	
6201	D4377	0.018		-1.27	
6326	D4007	0.00		-2.96	
6379		----		----	
6431	D4928	0.09		5.53	
6432	D4006	0.050		1.76	

normality suspect
n 15
outliers 0
mean (n) 0.0314
st.dev. (n) 0.02393
R(calc.) 0.0670
st.dev.(D4377:00) 0.01059
R(D4377:00) 0.0297



APPENDIX 1B – DISTILLATION RESULTS D2892 AND D5236

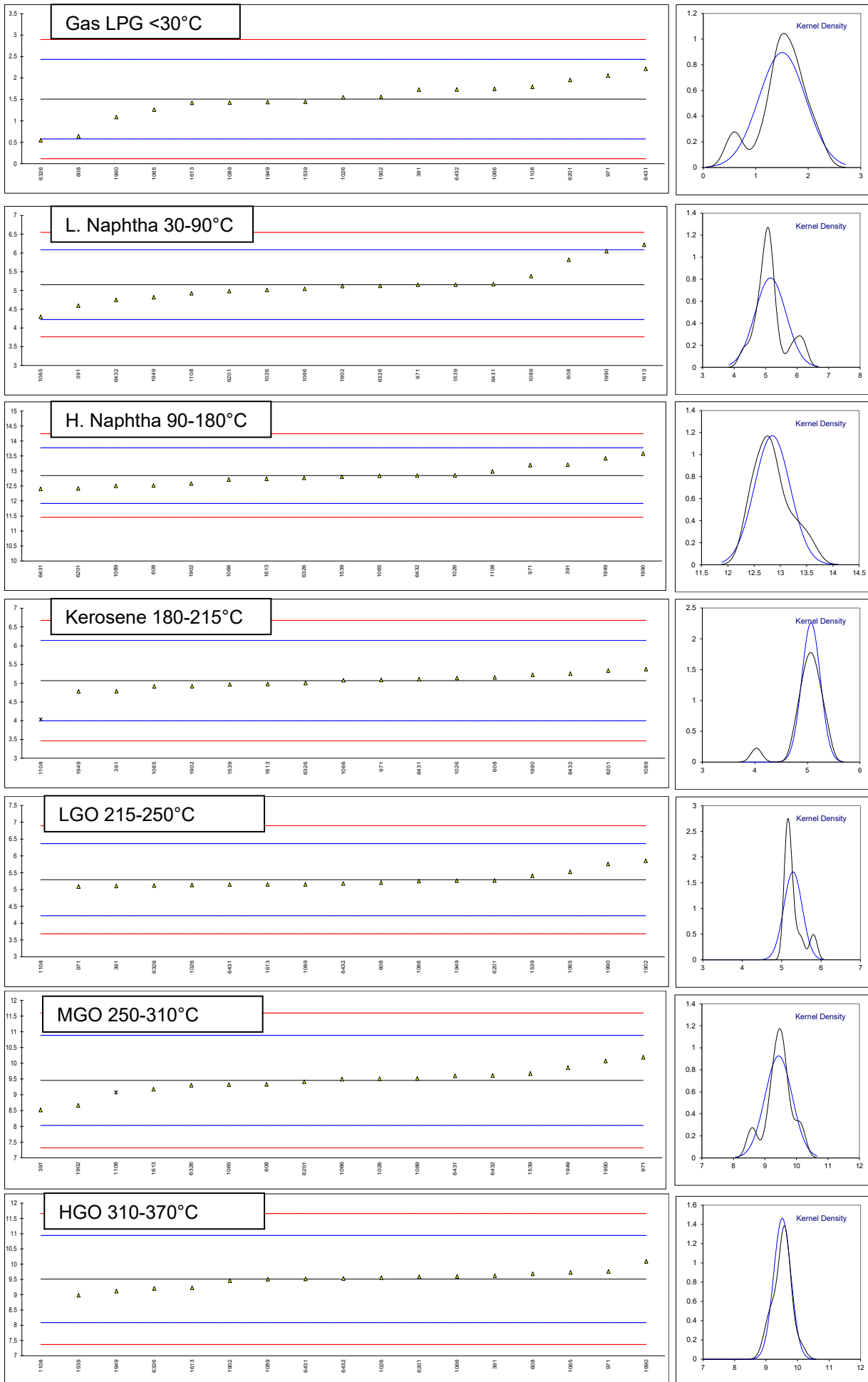
Determination of true boiling point curve of D2892; individual fractions in %M/M

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	----	----	----	----	----	----	----	----
391	1.7282	4.5964	13.2087	4.7874	5.1063	8.5225	9.6225	52.2867
442	----	----	----	----	----	----	----	----
445	----	----	----	----	----	----	----	----
608	0.6394	5.8187	12.5144	5.1572	5.2086	9.3294	9.6871	----
971	2.0544	5.1529	13.1962	5.0889	5.0889	10.1938	9.7614	49.4620
1023	----	----	----	----	----	----	----	----
1026	1.5483	5.0136	12.8536	5.1365	5.1365	9.5112	9.5603	49.1066
1065	1.2604	4.3002	12.8392	4.9130	5.5258	9.3257	9.7297	51.7260
1066	1.7474	5.0431	12.7156	5.0806	5.2528	9.4993	9.5966	50.7068
1089	1.4251	5.3845	12.5059	5.3766	5.1516	9.5176	9.5097	50.8250
1108	1.7910	4.9254	12.9851	4.0299 G(1)	1.6418 G(1)	9.0746 ex	4.6567 G(1)	60.5373 G(1)
1539	1.4439	5.1532	12.8091	4.9670	5.4114	9.6760	8.9803	50.2871
1613	1.4200	6.2201	12.7399	4.9800	5.1501	9.1799	9.2300	50.3900
1741	----	----	----	----	----	----	----	----
1842	----	----	----	----	----	----	----	----
1902	1.5584	5.1193	12.5816	4.9198	5.8541	8.6674	9.4642	51.2438
1949	1.4357	4.8196	13.4212	4.7848	5.2650	9.8656	9.1192	50.9331
1984	----	----	----	----	----	----	----	----
1990	1.0891	6.0507	13.5809	5.2231	5.7605	10.0773	10.0916	48.1268
6156	----	----	----	----	----	----	----	----
6201	1.9520	4.9812	12.4186	5.3381	5.2693	9.4163	9.5865	49.6357
6326	0.5454	5.1229	12.7723	5.0059	5.1219	9.3067	9.2084	50.7203
6379	----	----	----	----	----	----	----	----
6431	2.2161	5.1710	12.4054	5.1079	5.1444	9.6033	9.5186	50.6391
6432	1.7306	4.7486	12.8470	5.2537	5.1827	9.6169	9.5291	51.0200
normality	OK	OK	OK	OK	not OK	OK	OK	OK
n	17	17	17	16	16	16	16	15
outliers	0	0	0	1	1	0+1ex	1	1
mean (n)	1.5050	5.1542	12.8467	5.0700	5.2894	9.4568	9.5122	50.4739
st.dev. (n)	0.44571	0.49169	0.34022	0.17669	0.23330	0.43429	0.27263	1.04773
R(calc.)	1.2480	1.3767	0.9526	0.4947	0.6532	1.2160	0.7634	2.9337
st.dev.(D2892:20)	0.46429	0.46429	0.46429	0.53571	0.53571	0.71429	0.71429	n.a.
R(D2892:20)	1.3	1.3	1.3	1.5	1.5	2.0	2.0	n.a.

lab 1108 MGO test result excluded because of statistical outliers in fractions before and after

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	----	----	----	----	----	----	----	----
391	0.48	-1.20	0.78	-0.53	-0.34	-1.31	0.15	----
442	----	----	----	----	----	----	----	----
445	----	----	----	----	----	----	----	----
608	-1.86	1.43	-0.72	0.16	-0.15	-0.18	0.24	----
971	1.18	0.00	0.75	0.04	-0.37	1.03	0.35	----
1023	----	----	----	----	----	----	----	----
1026	0.09	-0.30	0.01	0.12	-0.29	0.08	0.07	----
1065	-0.53	-1.84	-0.02	-0.29	0.44	-0.18	0.30	----
1066	0.52	-0.24	-0.28	0.02	-0.07	0.06	0.12	----
1089	-0.17	0.50	-0.73	0.57	-0.26	0.09	0.00	----
1108	0.62	-0.49	0.30	-1.94	-6.81	-0.54	-6.80	----
1539	-0.13	0.00	-0.08	-0.19	0.23	0.31	-0.74	----
1613	-0.18	2.30	-0.23	-0.17	-0.26	-0.39	-0.40	----
1741	----	----	----	----	----	----	----	----
1842	----	----	----	----	----	----	----	----
1902	0.11	-0.08	-0.57	-0.28	1.05	-1.11	-0.07	----
1949	-0.15	-0.72	1.24	-0.53	-0.05	0.57	-0.55	----
1984	----	----	----	----	----	----	----	----
1990	-0.90	1.93	1.58	0.29	0.88	0.87	0.81	----
6156	----	----	----	----	----	----	----	----
6201	0.96	-0.37	-0.92	0.50	-0.04	-0.06	0.10	----
6326	-2.07	-0.07	-0.16	-0.12	-0.31	-0.21	-0.43	----
6379	----	----	----	----	----	----	----	----
6431	1.53	0.04	-0.95	0.07	-0.27	0.21	0.01	----
6432	0.49	-0.87	0.00	0.34	-0.20	0.22	0.02	----

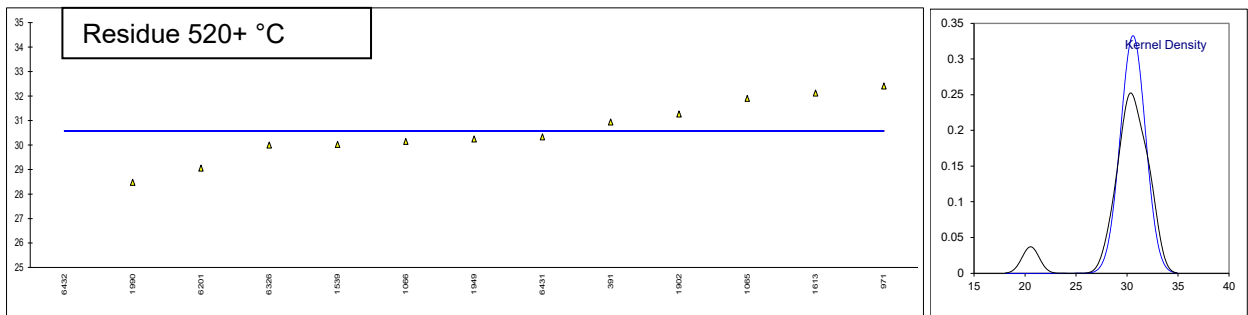
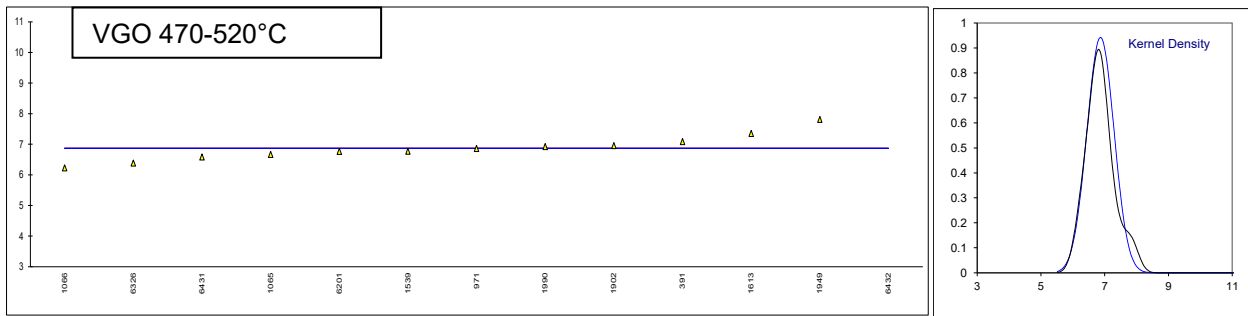
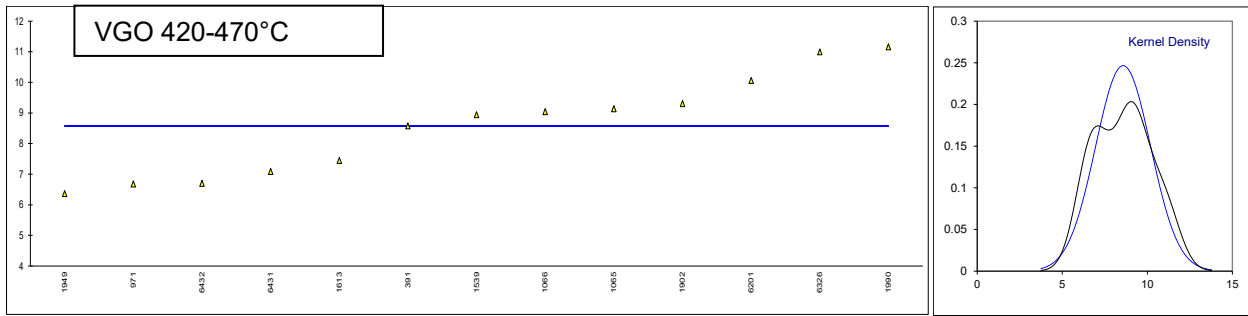
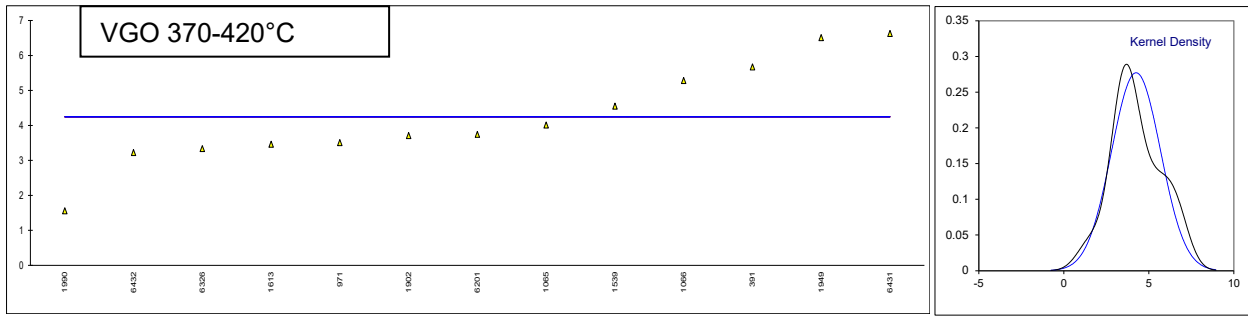


Determination of true boiling point curve of D5236; individual fractions in %M/M

lab	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue 520+ °C
171	----	----	----	----
391	5.6755	8.5798	7.0872	30.9442
442	----	----	----	----
445	----	----	----	----
608	----	----	----	----
971	3.5084	6.6805	6.8599	32.4131
1023	----	----	----	----
1026	----	----	----	----
1065	4.0118	9.1406	6.6656	31.9080
1066	5.2839	9.0448	6.2300	30.1481
1089	----	----	----	----
1108	----	----	----	----
1539	4.5528	8.9406	6.7702	30.0235
1613	3.4664	7.4457	7.3549	32.1230
1741	----	----	----	----
1842	----	----	----	----
1902	3.7133	9.3027	6.9559	31.2719
1949	6.5088	6.3643	7.8128	30.2472
1984	----	----	----	----
1990	1.5587	11.1621	6.9245	28.4814
6156	----	----	----	----
6201	3.7452	10.0592	6.7680	29.0633
6326	3.3367	10.9958	6.3852	30.0027
6379	----	----	----	----
6431	6.6275	7.0913	6.5822	30.3381
6432	3.2237	6.7022	20.5470 G(1)	20.5470 G(1)
normality	OK	OK	suspect	OK
n	13	13	12	12
outliers	0	0	1	1
mean (n)	4.2471	8.5777	6.8664	30.5804
st.dev. (n)	1.43878	1.61814	0.42331	1.19922
R(calc.)	4.0286	4.5308	1.1853	3.3578
st.dev.(D2892:20)	(0.71429)	(0.71429)	(0.71429)	n.a.
R(D2892:20)	(2.0)	(2.0)	(2.0)	n.a.

Corresponding z-scores for above mass fractions:

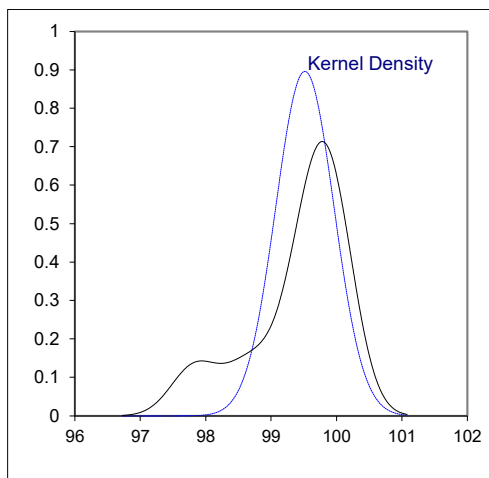
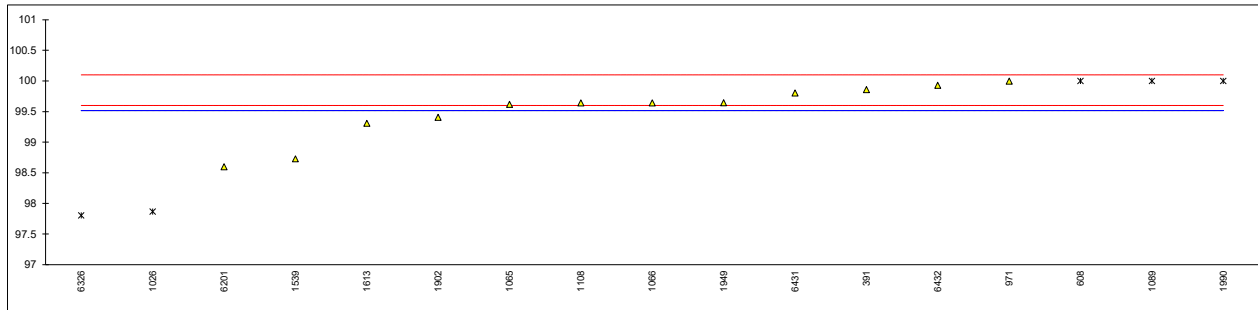
lab	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue 520+ °C
171	----	----	----	----
391	----	----	----	----
442	----	----	----	----
445	----	----	----	----
608	----	----	----	----
971	----	----	----	----
1023	----	----	----	----
1026	----	----	----	----
1065	----	----	----	----
1066	----	----	----	----
1089	----	----	----	----
1108	----	----	----	----
1539	----	----	----	----
1613	----	----	----	----
1741	----	----	----	----
1842	----	----	----	----
1902	----	----	----	----
1949	----	----	----	----
1984	----	----	----	----
1990	----	----	----	----
6156	----	----	----	----
6201	----	----	----	----
6326	----	----	----	----
6379	----	----	----	----
6431	----	----	----	----
6432	----	----	----	----



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

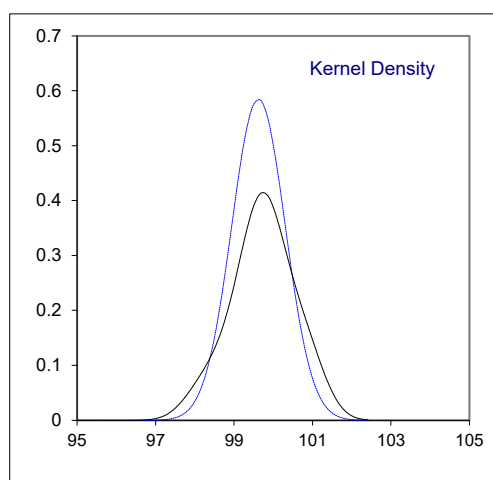
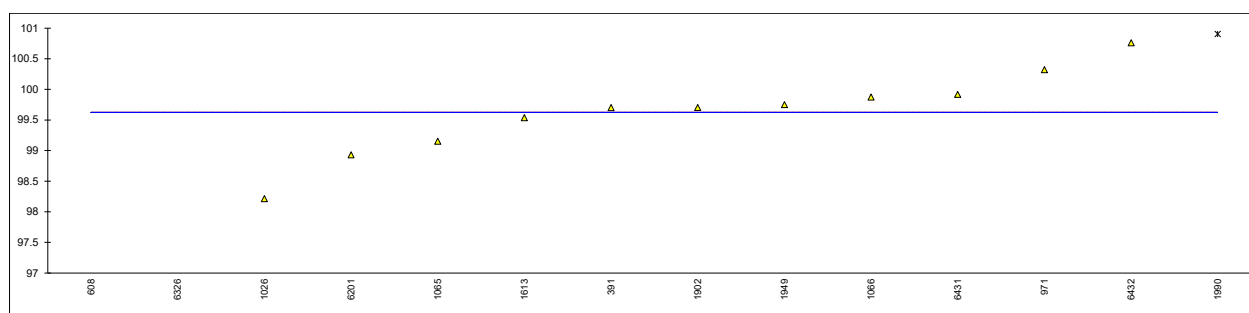
lab	method	value	mark	remarks
171		----		
391	calc	99.8587		
442		----		
445		----		
608	calc	100.0000	ex	test result excluded, iis corrected recovery for loss
971	calc	99.9984		
1023		----		
1026	calc	97.8667	DG(0.05)	
1065	calc	99.6200		
1066	calc	99.6421		
1089	calc	100.0000	ex	test result excluded, iis corrected recovery for loss
1108	calc	99.6418		
1539	calc	98.7281		
1613	calc	99.3100		
1741		----		
1842		----		
1902	calc	99.4085		
1949	calc	99.6442		
1984		----		
1990	calc	100.0000	ex	test result excluded, possibly laboratory corrected recovery for loss?
6156		----		
6201	calc	98.5977		
6326	calc	97.8037	DG(0.05)	
6379		----		
6431	calc	99.8058		
6432	calc	99.9286		
normality		OK		
n		12		
outliers		2+3ex		
mean (n)		99.52		
st.dev. (n)		0.445		
R(calc.)		1.25		

In below graph, the red lines represent the ASTM D5236:2018a 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 D2892:2020, paragraph 11.2.



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

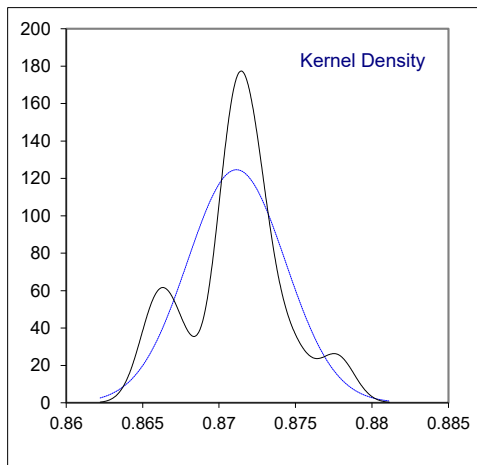
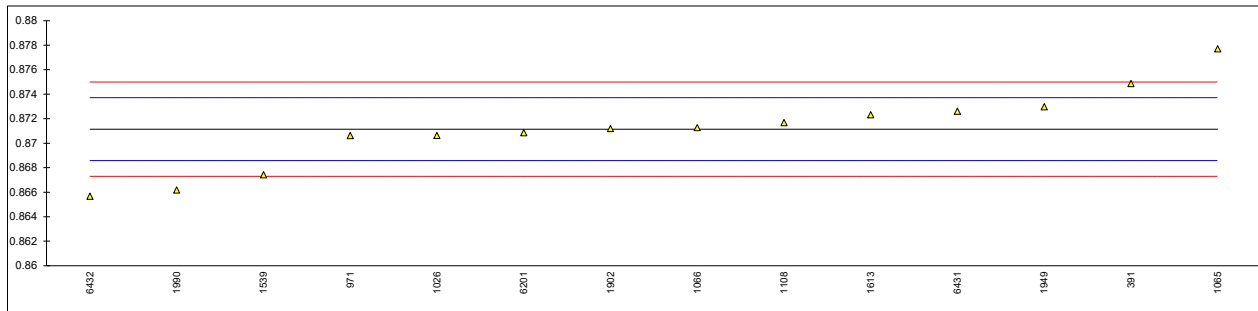
lab	method	value	mark	remarks
171		-----		
391	calc	99.7023		
442		-----		
445		-----		
608	calc	54.0526	ex	test result excluded, only the first seven fractions were reported
971	calc	100.3221		
1023		-----		
1026	calc	98.2147		
1065	calc	99.1545		
1066	calc	99.8745		
1089	calc	-----		
1108		-----		
1539		-----		
1613	calc	99.5363		
1741		-----		
1842		-----		
1902	calc	99.7040		
1949	calc	99.7504		
1984		-----		
1990	calc	100.9050	ex	test result excluded, recovery corrected for loss?
6156		-----		
6201	calc	98.9312		
6326	calc	72.1796	ex	test result excluded, Density fraction 12 was not reported
6379		-----		
6431	calc	99.9190		
6432	calc	100.7625		
normality		OK		
n		11		
outliers		0+3ex		
mean (n)		99.62		
st.dev. (n)		0.682		
R(calc.)		1.91		



Determination of calculated (theoretical) Density at 15°C of sample #21225; results in kg/L

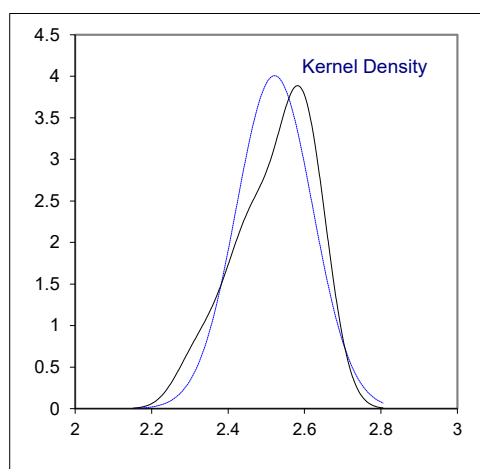
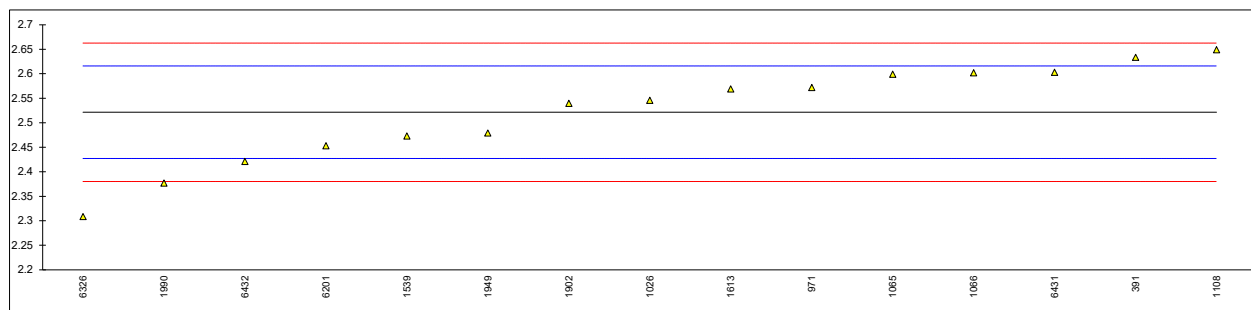
lab	method	calculated	mark	z(targ)	reported	remarks
171		----		----	----	
391	calculated by iis	0.8749		2.91	0.8735	
442		----		----	----	
445		----		----	----	
608		----		----	0.8742	not enough data to calculate
971	calculated by iis	0.8706		-0.41	0.8734	
1023		----		----	0.87347	not enough data to calculate
1026	calculated by iis	0.87062		-0.40	0.87380	
1065	calculated by iis	0.8777	*)	5.12	0.8736	
1066	calculated by iis	0.8713		0.10	0.8733	
1089		----		----	0.8739	not enough data to calculate
1108	calculated by iis	0.87168		0.42	0.87428	
1539	calculated by iis	0.8674	*)	-2.90	0.8735	
1613	calculated by iis	0.8723	*)	0.91	0.8743	
1741		----		----	0.8739	not enough data to calculate
1842		----		----	----	
1902	calculated by iis	0.8712		0.04	0.8738	
1949	calculated by iis	0.8730		1.43	0.8739	
1984		----		----	0.87305	not enough data to calculate
1990	calculated by iis	0.8662		-3.87	0.8740	
6156		----		----	----	
6201	calculated by iis	0.8709	*)	-0.22	0.8738	
6326		----		----	0.873013	not enough data to calculate
6379		----		----	----	
6431	calculated by iis	0.8726		1.15	0.8736	
6432	calculated by iis	0.8657	*)	-4.27	0.8729	
normality		OK				
n		14			n 20	
outliers		0			outliers 0	
mean (n)		0.87114			mean (n) 0.8737	
st.dev. (n)		0.003201				
R(calc.)		0.00896				
st.dev.(D5002:19)		0.001282				
R(D5002:19)		0.00359				

*) no Density reported for fraction 1 (LPG), for calculation purposes the consensus value of the group (0.5752 kg/L) was used.



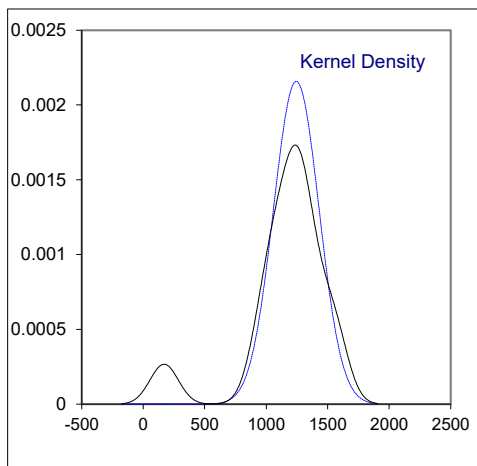
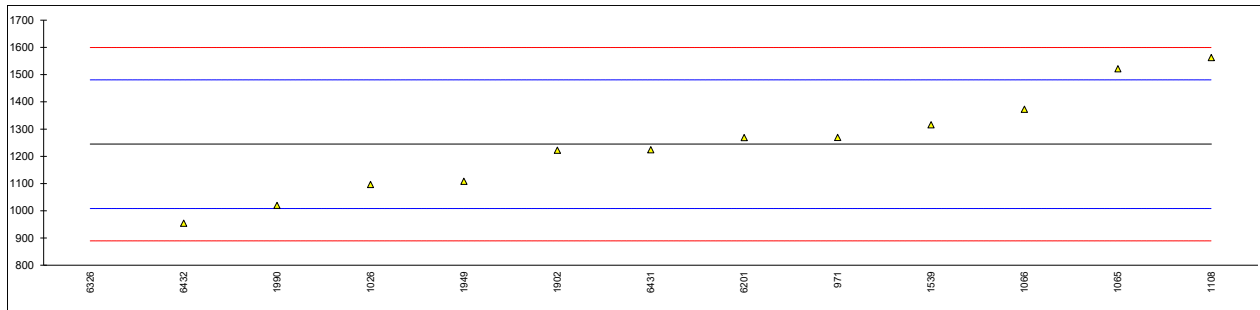
Determination of calculated (theoretical) Sulfur on sample #21225; results in %M/M

lab	method	calculated	mark	z(targ)	reported	remarks
171		----		----	----	
391	calculated by iis	2.633		2.37	2.633	
442		----		----	----	
445		----		----	----	
608		----		----	----	
971	calculated by iis	2.57		1.07	2.62	
1023		----		----	2.82	G(0.01) not enough data to calculate
1026	calculated by iis	2.546		0.51	2.662	
1065	calculated by iis	2.60		1.64	2.59	
1066	calculated by iis	2.60		1.71	2.64	
1089		----		----	----	
1108	calculated by iis	2.649		2.70	2.686	
1539	calculated by iis	2.473		-1.03	2.561	
1613	calculated by iis	2.57		1.01	2.63	
1741		----		----	2.60	not enough data to calculate
1842		----		----	----	
1902	calculated by iis	2.54		0.38	2.60	
1949	calculated by iis	2.479		-0.90	2.636	
1984		----		----	----	
1990	calculated by iis	2.377		-3.07	2.590	
6156		----		----	----	
6201	calculated by iis	2.453		-1.45	2.588	
6326	calculated by iis	2.3084		-4.52	2.6708	
6379		----		----	----	
6431	calculated by iis	2.60		1.73	2.61	
6432	calculated by iis	2.421		-2.13	2.542	
normality		OK				
n		15			n 16	
outliers		0			outliers 1	
mean (n)		2.5215			mean (n) 2.6162	
st.dev. (n)		0.09956				
R(calc.)		0.2788				
st.dev.(D4294:16e1)		0.04710				
R(D4294:16e1)		0.1319				



Determination of calculated (theoretical) Nitrogen on sample #21225; results in mg/kg

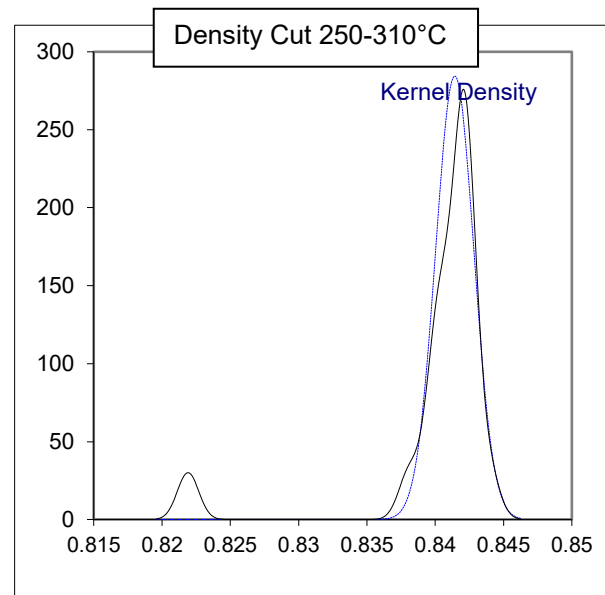
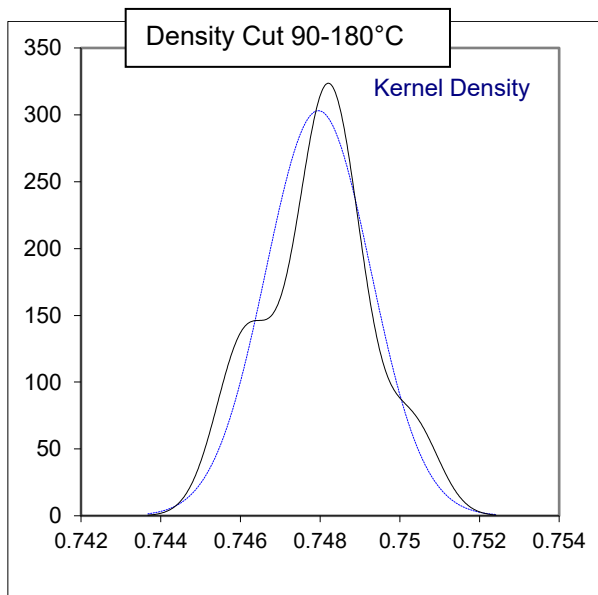
lab	method	calculated	mark	z(targ)	reported	remarks
171		----		----	----	
391		----		----	----	
442		----		----	----	
445		----		----	----	
608		----		----	----	
971	calculated by iis	1269		0.21	1256	
1023		----		----	----	
1026	calculated by iis	1097		-1.25	1231	
1065	calculated by iis	1521		2.34	1400	
1066	calculated by iis	1373		1.09	1460	
1089		----		----	----	
1108	calculated by iis	1562.8		2.69	1253.4	
1539	calculated by iis	1315		0.60	----	
1613		----		----	----	
1741		----		----	1469	not enough data to calculate
1842		----		----	----	
1902	calculated by iis	1222		-0.19	1116	
1949	calculated by iis	1109		-1.15	1100	
1984		----		----	----	
1990	calculated by iis	1019.86		-1.90	1218.92	
6156		----		----	----	
6201	calculated by iis	1269		0.20	1295	
6326	calculated by iis	169.996	G(0.01)	-9.09	1307.538	
6379		----		----	----	
6431	calculated by iis	1224		-0.18	1200	
6432	calculated by iis	955		-2.45	1192	
normality		OK				
n		12			n 14	
outliers		1			outliers 0	
mean (n)		1244.66			mean (n) 1254.48	
st.dev. (n)		184.968				
R(calc.)		517.91				
st.dev.(D5762:18a)		118.243				
R(D5762:18a)		331.08				



APPENDIX 1C – ANALYSIS OF DISTILLATION FRACTIONS

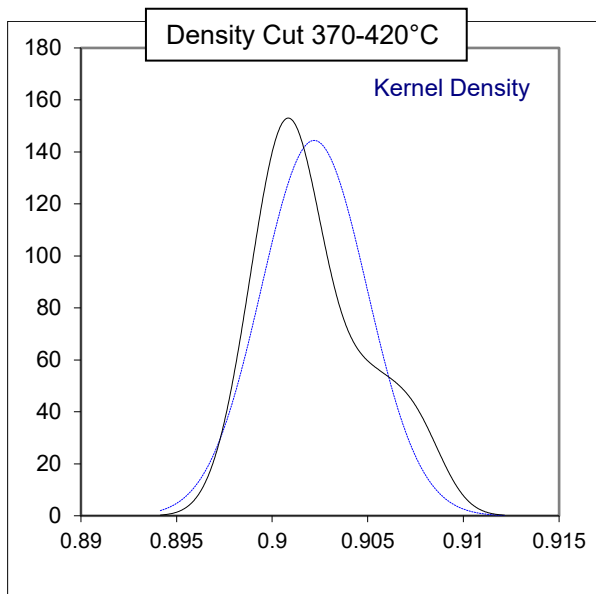
Determination of Density at 15°C on distillation fractions; results in kg/L

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		----	----	----	----	----	----	----	----
391	D4052	0.5830	0.6695	0.7489	0.7919	0.8095	0.8381	0.8683	0.9852
442		----	----	----	----	----	----	----	----
445		----	----	----	----	----	----	----	----
608	D4052	0.5468	0.6610	0.7480	0.7915	0.8073	0.8424	0.8738	----
971	D4052	0.5855	0.6699	0.7487	0.7925	0.8096	0.8409	0.8779	0.9853
1023		----	----	----	----	----	----	----	----
1026	D4052	0.55083	0.66785	0.74805	0.79097	0.80752	0.84249	0.87501	0.9853
1065	D4052	----	0.6717	0.7474	0.7911	0.8073	0.8420	0.8736	0.9841
1066	D4052	0.5837	0.6683	0.7483	0.7909	0.8078	0.8422	0.8746	0.9832
1089		----	----	----	----	----	----	----	----
1108	D4052	0.5527	0.6706	0.7484	0.7912	0.8006 D(1)	0.8219 D(1)	0.8464 D(1)	0.9657 D(1)
1539	D4052	----	0.6686	0.7478	0.7901	0.8077	0.8421	0.8739	----
1613	D4052	----	0.6669	0.7502	0.7916	0.8097	0.8401	0.8753	0.9837
1741	D4052	----	0.671	0.7479	0.7913	0.8089	0.8439	0.8756	0.9818
1842		----	----	----	----	----	----	----	----
1902	D4052	0.5647	0.6658	0.7462	0.7874 D(5)	0.8087	0.8416	0.8723	0.9826
1949	D4052	0.6196	0.6657	0.7463	0.7898	0.8072	0.8423	0.8719	0.9818
1984		----	----	----	----	----	----	----	----
1990	D4052	0.5699	0.6656	0.7487	0.7915	0.8080	0.8420	0.8756	0.9829
6156		----	----	----	----	----	----	----	----
6201	D4052	----	0.6714	0.7482	0.7927	0.8100	0.8403	0.8747	0.9837
6326	D4052	----	0.6586 D(5)	0.7457	0.7899	0.8057	0.8397	0.8693	0.9824
6379		----	----	----	----	----	----	----	----
6431	D4052	0.5956	0.6771	0.7504	0.7918	0.8092	0.8422	0.8756	0.9851
6432	D4052	NIL	0.6663	0.7462	0.7907	0.8069	0.8405	0.8734	0.9833
normality	OK	suspect	OK	OK	OK	OK	suspect	OK	OK
n	10	16	17	16	16	16	16	14	14
outliers	0	1	0	1	1	1	1	1	1
mean (n)	0.5752	0.6686	0.7480	0.7912	0.8082	0.8414	0.8738	0.9836	0.9836
st.dev. (n)	0.02278	0.00359	0.00132	0.00083	0.00122	0.00140	0.00243	0.00126	0.00126
R(calc.)	0.0638	0.0101	0.0037	0.0023	0.0034	0.0039	0.0068	0.0035	0.0035
RSD	4.0%	0.5%	0.2%	0.1%	0.2%	0.2%	0.3%	0.1%	0.1%



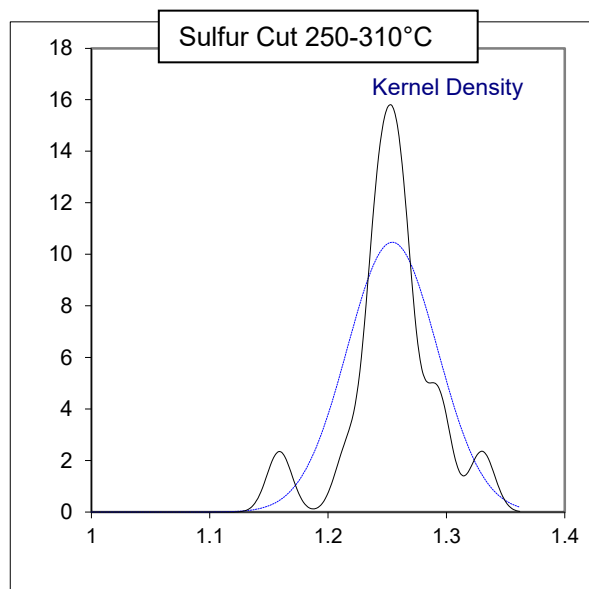
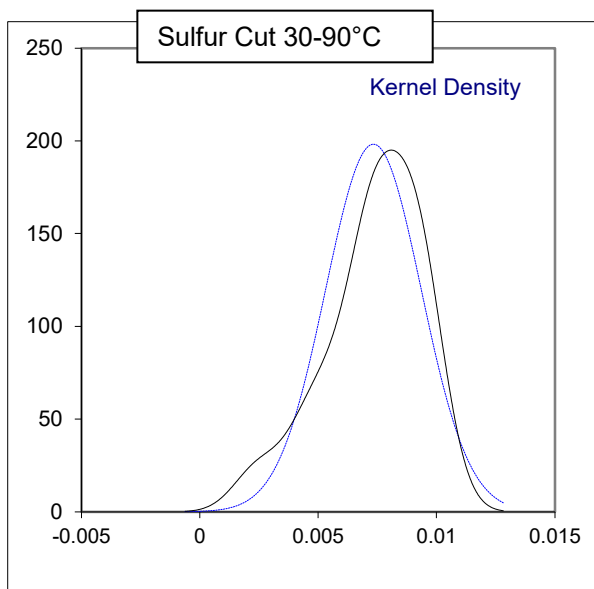
Determination of Density at 15°C on distillation fractions (continued); results in kg/L

lab	Method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue >520°C	remarks
171		----	----	----	----	
391	D4052	0.9003	0.9264	0.9461	1.0310	LPG: ISO8973 / Residue>520°C: D70
442		----	----	----	----	
445		----	----	----	----	
608		----	----	----	----	LPG: GPA2261
971	D4052	0.9009	0.9117	0.9327	1.0287	
1023		----	----	----	----	
1026	D4052	0.8992	0.9156	0.9422	1.0326	LPG: ISO8973
1065	D4052	0.9017	0.9175	0.9399	1.0307	
1066	D4052	0.8985	0.9214	0.9446	1.0268	
1089		----	----	----	----	
1108		----	----	----	----	LPG: D2598
1539	D4052	0.9078	0.9200	0.9387	1.0084	D(1)
1613	D4052	0.9025	0.9193	0.9415	1.0298	
1741	D4052	0.9001	0.9134	0.933	----	
1842		----	----	----	----	
1902	D4052	0.9004	0.9160	0.9383	1.0264	LPG: D2598 / Residue>520°C: D70
1949	D4052	0.9008	0.9177	0.9399	1.0296	Residue>520°C: D5002
1984		----	----	----	----	
1990	D4052	0.9027	0.9152	0.9408	1.0244	LPG: D2598 / Residue>520°C: D70
6156		----	----	----	----	
6201	D4052	0.9014	0.9190	0.9442	1.0330	
6326	D4052	0.9048	0.9181	0.9405	----	LPG: D2598
6379		----	----	----	----	
6431	D4052	0.9069	0.9276	0.9449	1.0289	Residue>520°C: D70
6432	D4052	0.9050	0.9212	0.9450	1.0231	Residue>520°C: D70
	normality	OK	OK	OK	OK	
	n	15	15	15	12	
	outliers	0	0	0	1	
	mean (n)	0.9022	0.9187	0.9408	1.0288	
	st.dev. (n)	0.00276	0.00434	0.00405	0.00307	
	R(calc.)	0.0077	0.0121	0.0113	0.0086	
	RSD	0.3%	0.5%	0.4%	0.3%	



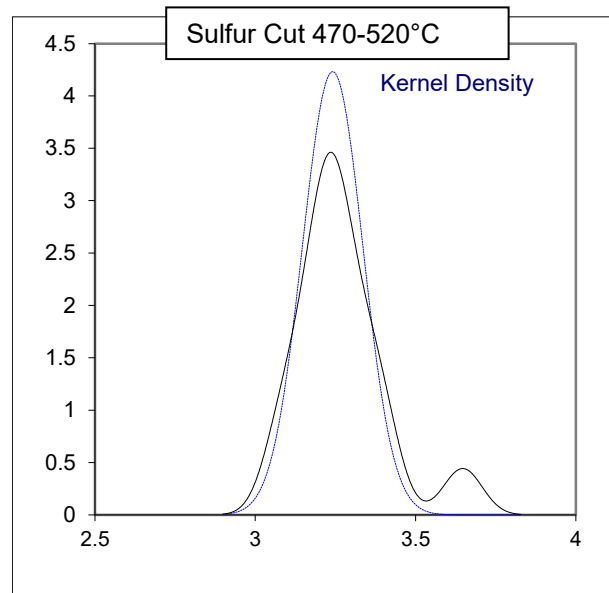
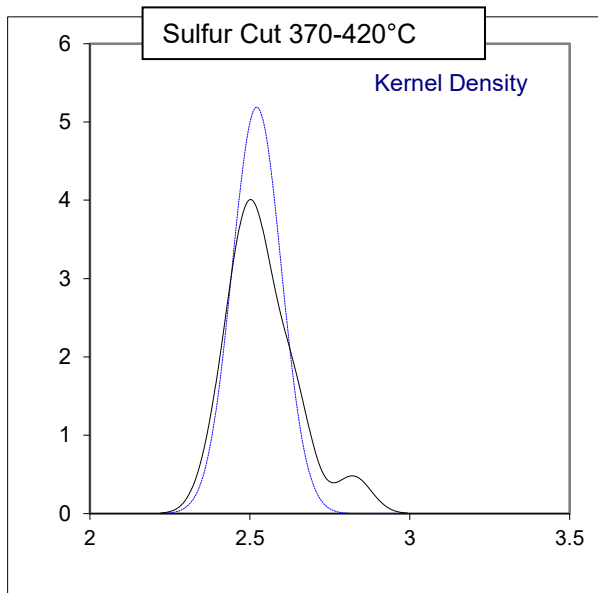
Determination of Sulfur on distillation fractions; results in %M/M

lab	Method	Gas LPG <30°C	L. Naphtha 30-90°C	H. Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C
171		----	----	----	----	----	----	----	----
391	D4294	----	0.0097	0.0446	0.168	0.390	1.216	2.008	4.476
442		----	----	----	----	----	----	----	----
445		----	----	----	----	----	----	----	----
608		----	----	----	----	----	----	----	----
971	D4294	----	0.0078	0.0405	0.148	0.370	1.29	2.21	4.43
1023		----	----	----	----	----	----	----	----
1026	D2622	----	0.008645	0.039270	0.152	0.349	1.295	2.211	4.439
1065	D4294	----	0.009	0.042	0.150	0.348	1.27	2.17	4.34
1066	D2622	0.00022	0.0049	0.03563	0.145	0.345	1.24	2.18	4.45
1089		----	----	----	----	----	----	----	----
1108	D4294	----	0.0094	0.0410	0.1438	0.2096 G(1)	0.7181 G(1)	1.4362 G(1)	4.1324 DG(5)
1539	ISO20846	----	0.007	0.042	0.144	0.343	1.259	2.103	----
1613	D4294	----	0.0095	0.0453	0.01462 G(1)	0.3592	1.250	2.202	4.352
1741	ISO8754	----	0.0072	0.0392	0.16	0.37	1.33	2.24	4.42
1842		----	----	----	----	----	----	----	----
1902	D4294	----	0.0025	0.0409	0.128	0.338	1.24	2.10	4.40
1949	D4294	----	0.00699	0.00964 G(1)	0.1462	0.3439	1.259	2.089	4.245
1984		----	----	----	----	----	----	----	----
1990	D4294	----	0.00895	0.0399	0.145	0.349	1.26	2.19	4.31
6156		----	----	----	----	----	----	----	----
6201	D4294	<0.0001	0.007675	0.039647	0.1653	0.415	1.245	2.17	4.31
6326	D2622	----	0.0046	0.0365	0.1408	0.3067	1.1588	2.059	4.007 G(5)
6379		----	----	----	----	----	----	----	----
6431	D4294	----	0.0060	0.0406	0.165	0.370	1.26	2.23	4.37
6432	D4294	NIL	0.0075	0.0366	0.142	0.334	1.24	2.16	4.39
normality		n.a.	OK	OK	OK	suspect	not OK	OK	OK
n		3	16	15	15	15	15	15	13
outliers		n.a.	0	1	1	1	1	1	2
mean (n)		<0.0003	0.0073	0.0402	0.1495	0.3554	1.2542	2.1548	4.3794
st.dev. (n)		n.a.	0.00201	0.00271	0.01089	0.02545	0.03810	0.06785	0.06627
R(calc.)		n.a.	0.0056	0.0076	0.0305	0.0713	0.1067	0.1900	0.1856
st.dev. (D4294:16e1)		n.a.	(0.00109)	(0.00327)	(0.00762)	(0.01332)	(0.03003)	(0.04256)	(0.06723)
R(D4294:16e1)		n.a.	(0.0031)	(0.0092)	(0.0213)	(0.0373)	(0.0841)	(0.1192)	(0.1883)
RSD		n.a.	27.5%	6.7%	7.3%	7.2%	3.0%	3.1%	1.5%



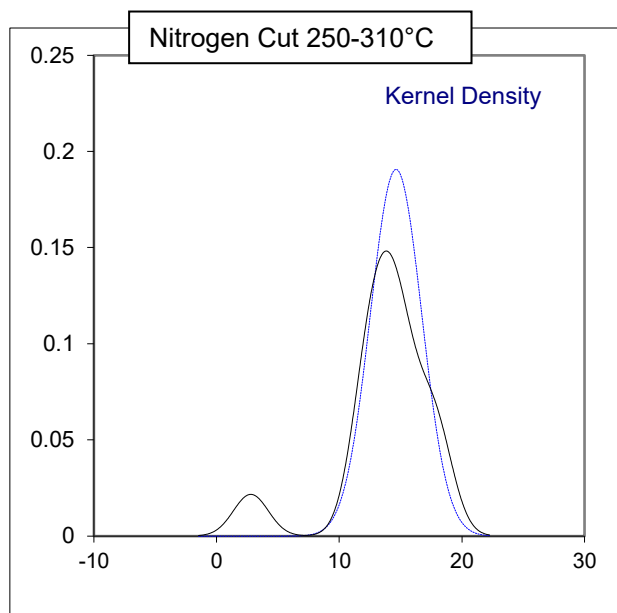
Determination of Sulfur on distillation fractions (continued); results in %M/M

lab	Method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue >520°C	remarks
171		----	----	----	----	
391	D4294	2.497	2.931	3.379	5.395	L.Naphtha: ISO20846
442		----	----	----	----	
445		----	----	----	----	
608		----	----	----	----	
971	D4294	2.51	2.70	3.10	5.28	
1023		----	----	----	----	
1026	D2622	2.439	2.757	3.344	5.449	L.&H.Naphtha: ISO20846
1065	D4294	2.39	2.72	3.21	5.26	
1066	D2622	2.47	2.83	3.26	5.49	
1089		----	----	----	----	
1108		----	----	----	----	
1539	ISO20846	2.823 G(5)	2.909	3.647 G(5)	4.980	
1613	D4294	2.527	2.762	3.249	5.271	
1741	ISO8754	2.55	2.73	3.08	----	L.&H.Naphtha: D5453
1842		----	----	----	----	
1902	D4294	2.50	2.77	3.19	5.21	
1949	D4294	2.498	2.709	3.161	5.143	L.&H.Naphtha: D5453
1984		----	----	----	----	
1990	D4294	2.56	2.71	3.22	5.02	
6156		----	----	----	----	
6201	D4294	2.446	2.743	3.275	5.170	LPG, L.&H.Naphtha, Ker.: D5453 / LGO&MGO: D2622
6326	D2622	2.6315	2.789	3.222	4.610 G(5)	
6379		----	----	----	----	
6431	D4294	2.63	2.93	3.29	5.40	L.Naphtha: D5453
6432	D4294	2.65	2.85	3.40	5.31	L.&H.Naphtha: D5453
	normality	OK	OK	OK	OK	
	n	14	15	14	13	
	outliers	1	0	1	1	
	mean (n)	2.5213	2.7893	3.2414	5.2598	
	st.dev. (n)	0.07691	0.08145	0.09425	0.15539	
	R(calc.)	0.2154	0.2281	0.2639	0.4351	
	st.dev.(D4294:16e1)	(0.04710)	(0.05027)	(0.05538)	(0.07566)	
	R(D4294:16e1)	(0.1319)	(0.1408)	(0.1551)	(0.2119)	
	RSD	3.1%	2.9%	2.9%	3.0%	



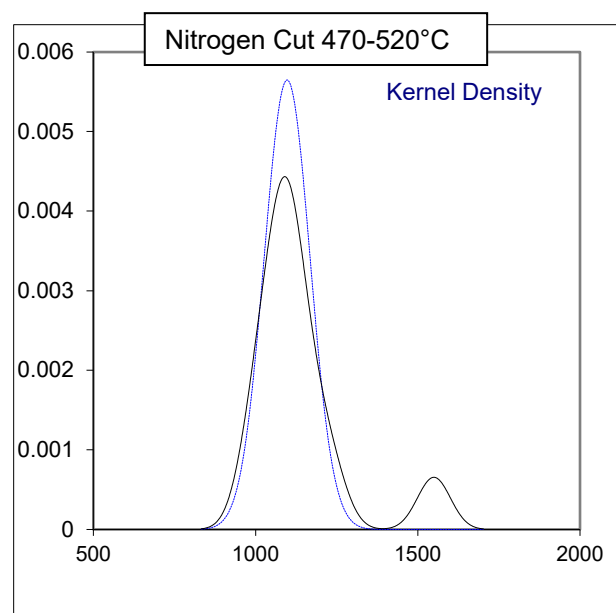
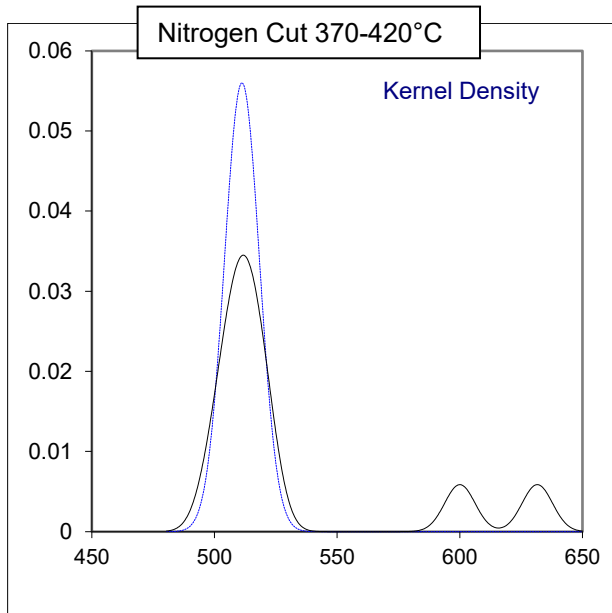
Determination of Nitrogen on distillation fractions; results in mg/kg

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		----	----	----	----	----	----	----	----
391		----	----	----	----	----	----	----	----
442		----	----	----	----	----	----	----	----
445		----	----	----	----	----	----	----	----
608	D4629	----	<1	<1	<1	<1	14.72	188.11	----
971	D5762	----	<0.30	0.33	0.45	1.50	18	248	2476
1023		----	----	----	----	----	----	----	----
1026	D4629	----	0.57	0.57	1.11	1.23	15.10	181.22	2195
1065	D4629	----	<10	<10	<10	<10	14	200	2600
1066	D5762	<5	<5	<5	<5	<5	12	229	2720
1089		----	----	----	----	----	----	----	----
1108	D5762	----	0.1	0.1	0.2	0.2	2.8 G(0.01)	11.2 G(0.01)	2580.3
1539		----	----	----	----	----	----	----	----
1613	D4629	----	<0.1	0.3	0.5	1.0	----	----	----
1741		----	----	----	----	----	----	----	----
1842		----	----	----	----	----	----	----	----
1902	D4629	----	<1.0	<1.0	<1.0	<1.0	15	179	2326
1949	D4629	----	0.28	0.56	0.85	1.36	16.9	215	2120
1984		----	----	----	----	----	----	----	----
1990	D5762	----	----	----	0.83	1.24	12.16	245.89	2257.26
6156		----	----	----	----	----	----	----	----
6201	D5762	<1	<1	<1	<1	<1	18	205	2427
6326	D4629	----	1.1995	0.1877	0.14	0.534	12.78	163.04	2528.26
6379		----	----	----	----	----	----	----	----
6431	D4629	----	<1	<1	<1	<1	14	230	2400
6432	D5762	NIL	0.9	1.0	0.9	1.0	13	188	2292
	normality	n.a.	n.e.	n.e.	n.e.	n.e.	OK	OK	OK
	n	3	13	13	14	14	12	12	12
	outliers	n.a.	n.e.	n.e.	n.e.	n.e.	1	1	0
	mean (n)	<10	<10	<10	<10	<10	14.6383	206.0217	2410.2
	st.dev. (n)	n.a.	n.e.	n.e.	n.e.	n.e.	2.09160	27.65184	179.54
	R(calc.)	n.a.	n.e.	n.e.	n.e.	n.e.	5.8565	77.4252	502.7
	st.dev.(D4629:17)	n.a.	n.e.	n.e.	n.e.	n.e.	(1.15111)	---	---
	R(D4629:17)	n.a.	n.e.	n.e.	n.e.	n.e.	(3.2231)	---	---
	st.dev.(D5762:18a)	---	---	---	---	---	---	(19.57206)	(228.96)
	R(D5762:18a)	---	---	---	---	---	---	(54.8018)	(641.1)
	RSD	---	---	---	---	---	14.3%	13.4%	7.4%



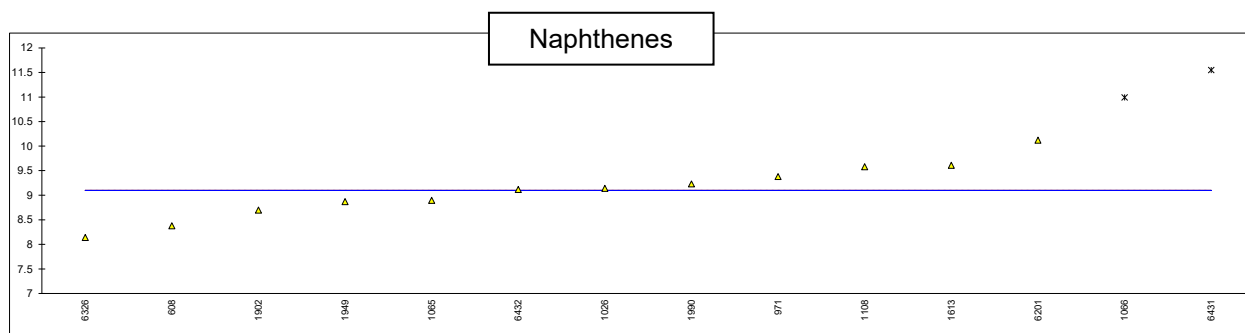
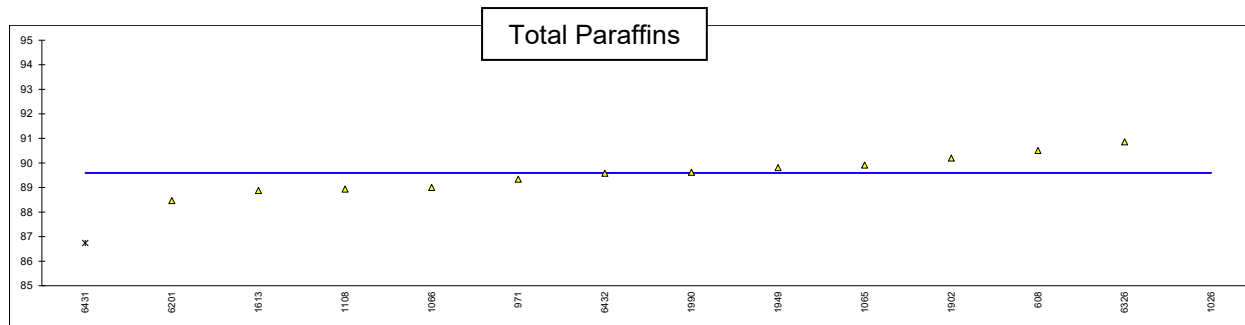
Determination of Nitrogen on distillation fractions (continued); results in mg/kg

lab	method	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C	Residue >520°C	remarks
171		----	----	----	----	
391		----	----	----	----	
442		----	----	----	----	
445		----	----	----	----	
608		----	----	----	----	HGO: D5762
971	D5762	505	722	1100	3400	fractions 2 to 6: D4629
1023		----	----	----	----	
1026	D4629	499	690	1094	2911	fractions 8 to 12: D5762
1065	D4629	510	690	1150	4200	
1066	D5762	515	700	1100	3950	
1089		----	----	----	----	
1108		----	----	----	----	
1539		----	----	1550 G(0.01)	4032	
1613		----	----	----	----	
1741		----	----	----	----	
1842		----	----	----	----	
1902	D4629	513	690	1025	3355	fractions 8 to 12: D5762
1949	D4629	521	704	985	3080	fractions 7 to 12: D5762
1984		----	----	----	----	
1990	D5762	631.54 DG(0.01)	776.70	1234.35	2850.30	fractions 4 to 6: D4629
6156		----	----	----	----	
6201	D5762	520	726	1175	3700	fractions 1 to 5: D4629
6326	D4629	506.14	644.32	1034.53	----	fractions 8 to 12: D5762
6379		----	----	----	----	
6431	D4629	600 DG(0.01)	800	1100	3400	fractions 8 to 12: D5762
6432	D5762	512	698	1075	3170	LPG: D4629
	normality	OK	OK	OK	OK	
	n	9	11	11	11	
	outliers	2	0	1	0	
	mean (n)	511.2	712.8	1097.5	3458.9	
	st.dev. (n)	7.12	43.19	70.64	457.42	
	R(calc.)	19.9	120.9	197.8	1280.8	
	st.dev.(D5762:18a)	(48.57)	(67.72)	(104.27)	(328.60)	
	R(D5762:18a)	(136.0)	(189.6)	(291.9)	(920.1)	
	RSD	1.4%	6.1%	6.4%	13.2%	



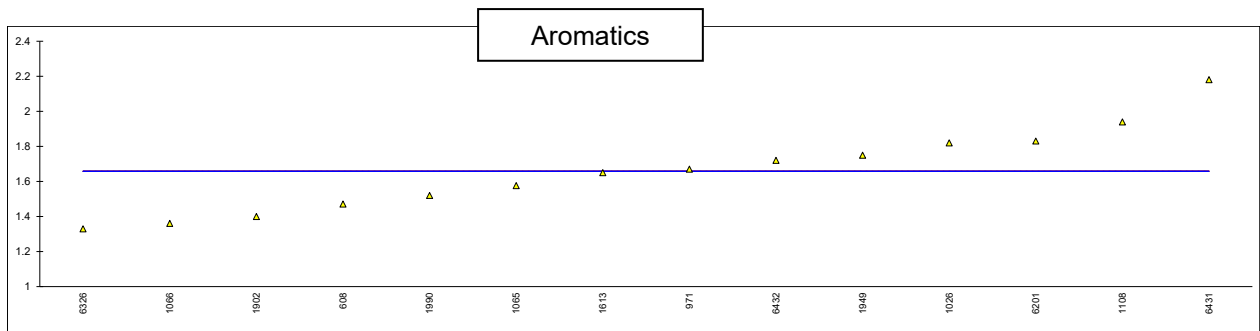
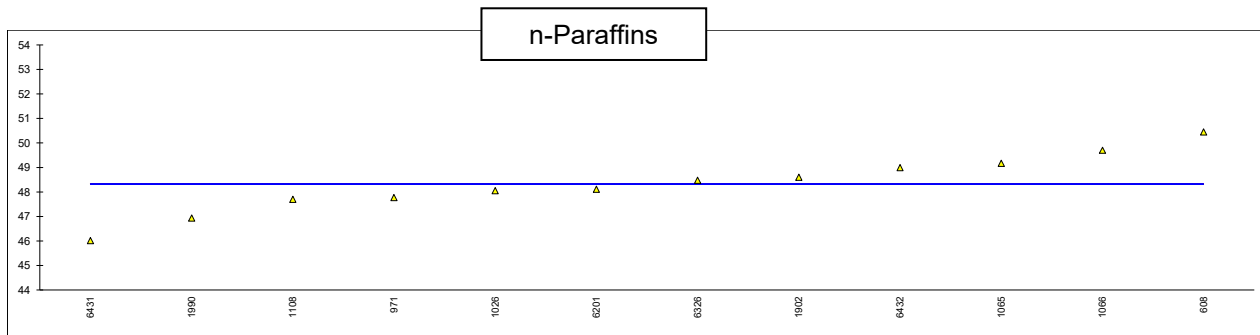
Determination of P(iP)NA on distillation fraction 2 (light Naphtha, 30-90°C); results in %V/V

lab	method	total par.	C1-C4	n-par.	i-par.	naphth.	arom.	remarks
171		----	----	----	----	----	----	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445		----	----	----	----	----	----	
608	D6730	90.5068	----	51.8610	38.6458	8.3788	1.0993	
971	D6839	89.34	0.46	48.96	40.38	9.38	1.27	
1023		----	----	----	----	----	----	
1026	ISO22854	98.62 G(1)	0.02	49.26	40.22	9.14	1.38	
1065	In house	89.916	2.85	50.472	39.444	8.895	1.189	
1066	ISO22854	89.0	3.51	48.3	38.9	10.99 DG(5)	1.79	
1089		----	----	----	----	----	----	
1108	D5443	88.94	0.43	48.91	40.03	9.58	1.48	
1539		----	----	----	----	----	----	
1613	D6839	88.88	1.31	----	----	9.61	1.26	
1741		----	----	----	----	----	----	
1842		----	----	----	----	----	----	
1902	D6839	90.2	0.3	49.7	40.5	8.7	1.1	
1949	D5443	89.81	4.05	----	----	8.87	1.32	
1984		----	----	----	----	----	----	
1990	D6839	89.62	1.17	48.10	41.52	9.23	1.15	
6156		----	----	----	----	----	----	
6201	D6839	88.47	0.84	49.45	39.02	10.12	1.39	
6326	D6839	90.86	0.00	49.5	41.36	8.14	1.0	
6379		----	----	----	----	----	----	
6431	D6733	86.74 G(5)	1.96	47.48	39.26	11.55 DG(5)	1.67	
6432	D6839	89.58	0.58	50.26	39.32	9.12	1.30	
	normality	OK	suspect	OK	OK	OK	OK	
	n	12	13	12	12	12	14	
	outliers	2	0	0	0	2	0	
	mean (n)	89.594	1.345	49.354	39.883	9.097	1.314	
	st.dev. (n)	0.7118	1.3477	1.1702	0.9421	0.5498	0.2200	
	R(calc.)	1.993	3.774	3.277	2.638	1.539	0.616	



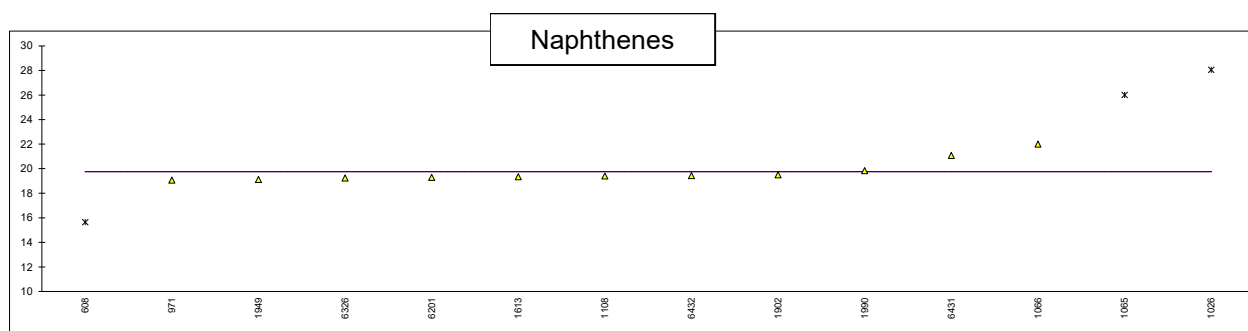
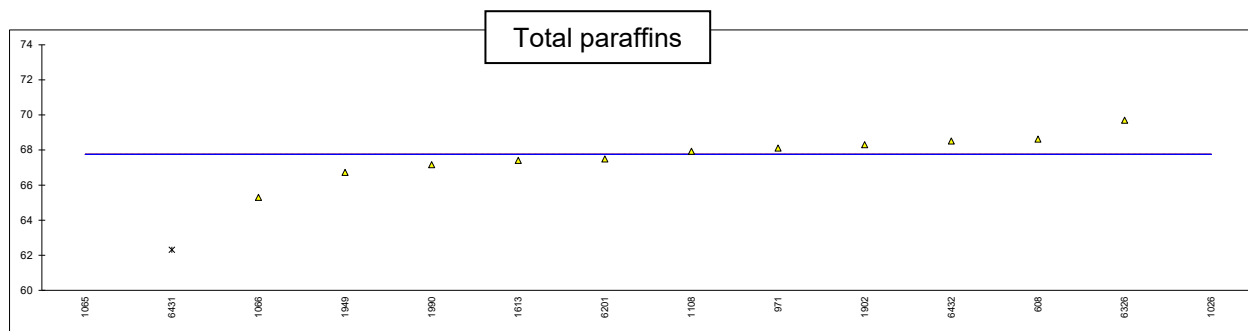
Determination of P(iP)NA on distillation fraction 2 (light Naphtha, 30-90°C); results in %M/M

lab	method	total par.	C1-C4	n-par.	i-par.	naphth.	arom.	remarks
171		----	----	----	----	----	----	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445		----	----	----	----	----	----	
608	D6730	88.8258	----	50.4469	38.3789	9.6867	1.4712	
971	D6839	87.68	0.40	47.77	39.91	10.65	1.67	
1023		----	----	----	----	----	----	
1026	ISO22854	98.18 G(1)	0.02	48.05	39.74	10.40	1.82	
1065	In house	88.243	2.48	49.169	39.074	10.181	1.576	
1066	ISO22854	87.2	3.07	49.7	39.3	9.65	1.36	
1089		----	----	----	----	----	----	
1108	D5443	87.09	0.37	47.70	39.39	10.97	1.94	
1539		----	----	----	----	----	----	
1613	D6839	86.70	1.13	----	----	10.93	1.65	
1741		----	----	----	----	----	----	
1842		----	----	----	----	----	----	
1902	D6839	88.6	0.2	48.6	40.1	9.9	1.4	
1949	D5443	87.97	3.48	----	----	10.28	1.75	
1984		----	----	----	----	----	----	
1990	D6839	87.95	1.03	46.93	41.02	10.53	1.52	
6156		----	----	----	----	----	----	
6201	D6839	86.69	0.73	48.11	38.58	11.46	1.83	
6326	D6839	89.34	0.0	48.47	40.87	9.33	1.33	
6379		----	----	----	----	----	----	
6431	D6733	84.75	1.67	46.01	38.74	13.03 G(5)	2.18	
6432	D6839	87.88	0.51	48.99	38.89	10.41	1.72	
	normality	suspect	suspect	OK	OK	OK	OK	
	n	13	13	12	12	13	14	
	outliers	1	0	0	0	1	0	
	mean (n)	87.609	1.161	48.329	39.499	10.337	1.658	
	st.dev. (n)	1.1714	1.1700	1.1973	0.8560	0.5976	0.2392	
	R(calc.)	3.280	3.276	3.353	2.397	1.673	0.670	



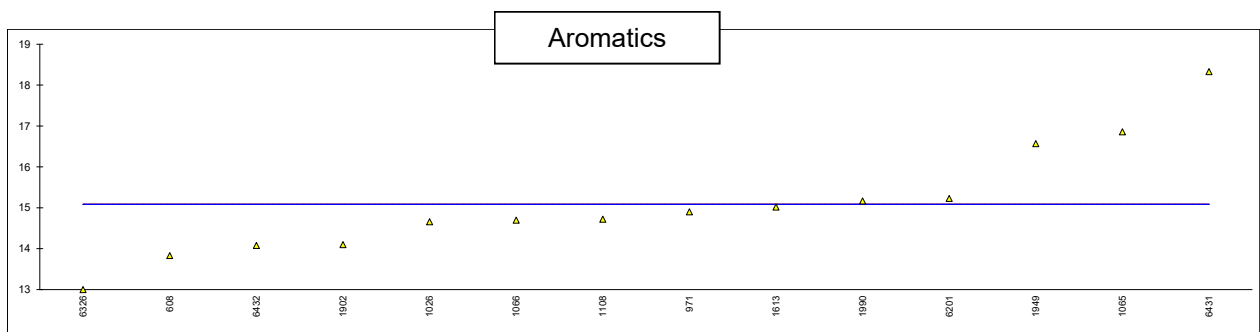
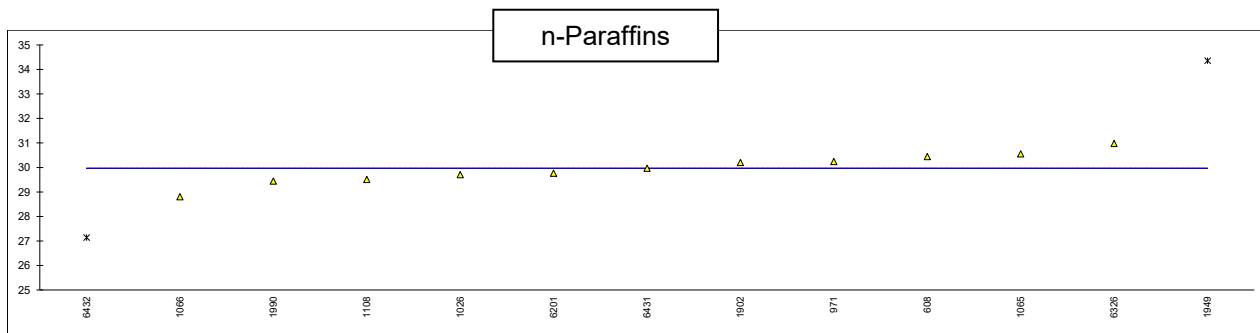
Determination of P(iP)NA on distillation fraction 3 (heavy Naphtha, 90-180°C); results in %V/V

lab	method	total par.	C1-C4	n-par.	i-par.	naphth.	arom.	remarks
171		----	----	----	----	----	----	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445		----	----	----	----	----	----	
608	D6730	68.6215	0.0761	31.9038	36.7177	15.6479 G(5)	11.7528	
971	D6839	68.11	<0.1	31.81	36.30	19.07	12.81	
1023		----	----	----	----	----	----	
1026	ISO22854	87.14 G(1)	0	31.71	27.37	28.06 DG(1)	12.66	
1065	In house	59.463 G(5)	0.11	32.621	26.842	26.013 DG(1)	14.524	
1066	ISO22854	65.3	0.05	30.6	34.7	22.0	12.6	
1089		----	----	----	----	----	----	
1108	D5443	67.93	0.06	31.35	36.58	19.40	12.67	
1539		----	----	----	----	----	----	
1613	D6839	67.41	----	----	----	19.35	12.93	
1741		----	----	----	----	----	----	
1842		----	----	----	----	----	----	
1902	D6839	68.3	<0.1	31.9	36.4	19.5	12.0	
1949	D6729mod	66.726	< 0.010	36.178 G(5)	30.548	19.128	14.146	
1984		----	----	----	----	----	----	
1990	D6839	67.16	0	31.13	36.03	19.84	13	
6156		----	----	----	----	----	----	
6201	D6839	67.49	0.03	31.47	36.02	19.30	13.04	
6326	D6839	69.7	0	32.6	37.1	19.24	11.07	
6379		----	----	----	----	----	----	
6431	D6733	62.31 G(5)	0.11	31.84	30.47	21.07	16.24 G(5)	
6432	D6839	68.52	0	28.74 G(1)	39.78	19.44	12.01	
	normality	suspect	OK	OK	OK	not OK	OK	
	n	11	13	11	13	11	13	
	outliers	3	n.e.	2	0	3	1	
	mean (n)	67.752	<0.15	31.721	34.220	19.758	12.709	
	st.dev. (n)	1.1490	n.e.	0.5896	4.0388	0.9255	0.9229	
	R(calc.)	3.217	n.e.	1.651	11.309	2.591	2.584	



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

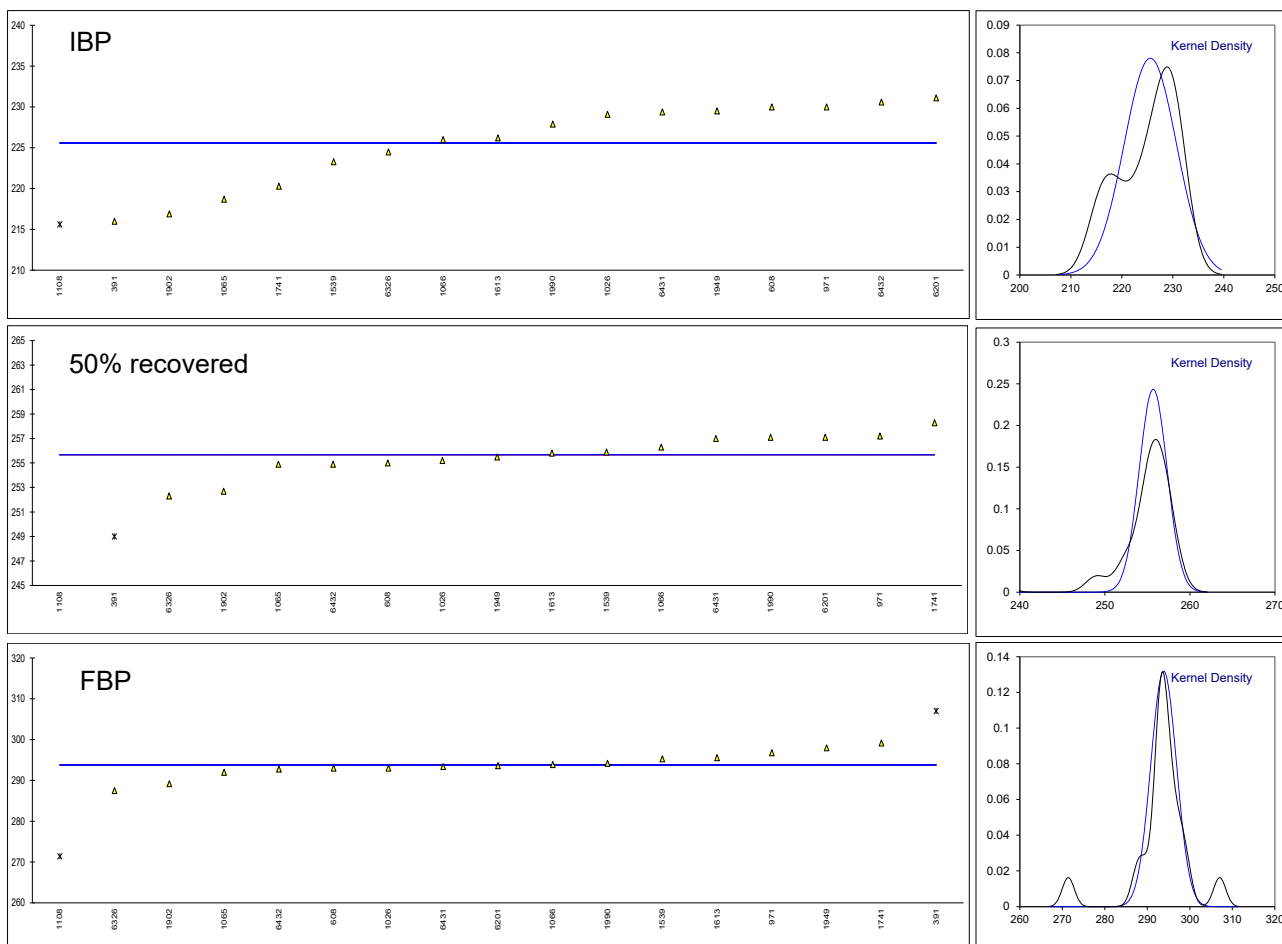
lab	method	total par.	C1-C4	n-par.	i-par.	naphth.	arom.	remarks
171		----	----	----	----	----	----	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445		----	----	----	----	----	----	
608	D6730	65.9968	0.0585	30.4399	35.5569	16.3960	G(5) 13.8327	
971	D6839	65.13	<0.1	30.24	34.89	19.96	14.90	
1023		----	----	----	----	----	----	
1026		85.13	G(1) 0	29.70	25.81	29.62	DG(1) 14.66	
1065	In house	51.025	G(1) 0.09	30.552	25.473	27.117	DG(1) 16.858	
1066	ISO22854	62.1	0.04	28.8	33.2	23.2	14.7	
1089		----	----	----	----	----	----	
1108	D5443	64.96	0.04	29.51	35.45	20.31	14.72	
1539		----	----	----	----	----	----	
1613	D6839	64.13	----	----	----	20.22	15.02	
1741		----	----	----	----	----	----	
1842		----	----	----	----	----	----	
1902	D6839	65.2	<0.1	30.2	35.0	20.5	14.1	
1949	D6729mod	63.627	<0.010	34.349	G(5) 29.278	19.805	16.568	
1984		----	----	----	----	----	----	
1990	D6839	64.00	0	29.44	34.56	20.82	15.17	
6156		----	----	----	----	----	----	
6201	D6839	64.32	0.02	29.76	34.56	20.28	15.23	
6326	D6839	66.72	0.0	30.98	35.74	20.28	13.00	
6379		----	----	----	----	----	----	
6431	D6733	58.99	0.08	29.96	29.03	21.86	18.33	
6432	D6839	65.47	0	27.14	G(5) 38.26	20.48	14.08	
	normality	not OK	OK	OK	OK	not OK	not OK	
	n	12	13	11	13	11	14	
	outliers	2	n.e.	2	0	3	0	
	mean (n)	64.220	<0.1	29.962	32.831	20.701	15.083	
	st.dev. (n)	2.0354	n.e.	0.6069	4.0599	0.9891	1.3671	
	R(calc.)	5.699	n.e.	1.699	11.368	2.769	3.828	



Determination of Distillation on combined fractions 5 and 6, results in °C

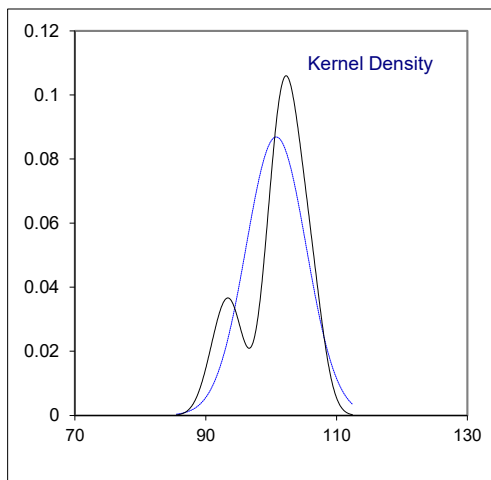
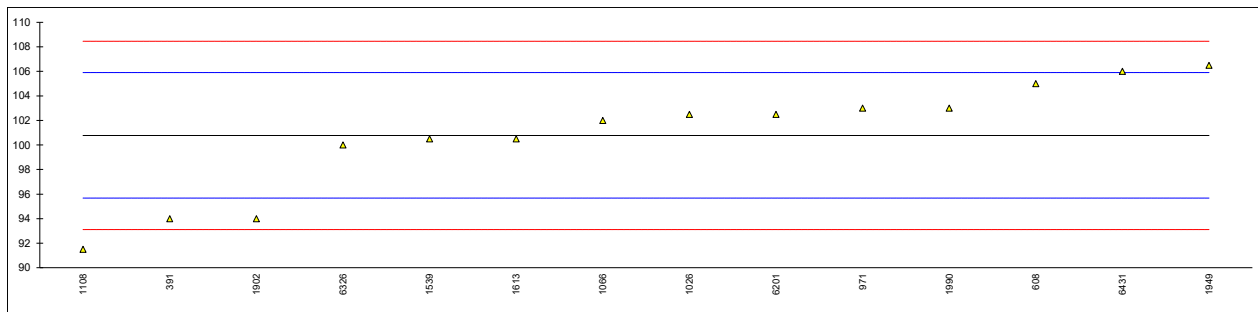
lab	method	IBP	5% rec.	10% rec.	50% rec.	90% rec.	95% rec.	FBP	%residue	%loss
171		----	----	----	----	----	----	----	----	----
391	D86-M	216	232	234	249 G(5)	280	292	307 G(5)	1.8	0.2
442		----	----	----	----	----	----	----	----	----
445		----	----	----	----	----	----	----	----	----
608	D86-M	230.0	238.0	240.0	255.0	280.0	286.0	293.0	1.6	0.4
971	D86-A	230.0	240.1	242.0	257.2	283.2	290.0	296.8	0.4	0.4
1023		----	----	----	----	----	----	----	----	----
1026	D86-A	229.1	----	240.8	255.2	281.6	286.8	293.0	1.5	0.0
1065	D86-A	218.7	229.9	235.4	254.9	280.0	285.9	292.0	1.5	0.4
1066	D86-A	226.0	239.3	241.3	256.3	282.7	288.2	293.9	1	0.5
1089		----	----	----	----	----	----	----	----	----
1108	D86-A	215.6 ex	224.2 ex	226.9 G(5)	237.3 G(1)	256.6 G(1)	262.6 G(1)	271.4 G(1)	0.4	0.1
1539	ISO3405-A	223.3	237.5	239.5	255.9	284.7	291.3	295.3	97.2	----
1613	D86-A	226.2	236.1	239.3	255.8	281.5	288.2	295.6	1.0	0.5
1741	D86-A	220.3	241.0	243.0	258.3	286.5	292.5	299.2	1.0	1.0
1842		----	----	----	----	----	----	----	----	----
1902	D86-A	216.9	229.1	232.6	252.7	278.9	284.3	289.2	1.4	0.3
1949	D86-A	229.5	235.5	238.5	255.5	278.5	291.0	298.0	0.8	0.7
1984		----	----	----	----	----	----	----	----	----
1990	D86-A	227.9	240.7	242.2	257.1	282.7	287.9	294.2	0.6	0.1
6156		----	----	----	----	----	----	----	----	----
6201	D86-A	231.1	240.1	242.0	257.1	282.6	288.0	293.6	1.0	0.1
6326	D86-A	224.5	236	237.8	252.3	277.2	282.7	287.5	1.4	0.7
6379		----	----	----	----	----	----	----	----	----
6431	D86-A	229.4	241.1	243.0	257.0	282.8	289.0	293.4	1.3	0.0
6432	D86-A	230.6	239.0	240.7	254.9	280.7	285.9	292.8	0.4	0.2
	normality	OK	OK	OK	OK	OK	OK	OK		
	n	16	15	16	15	16	16	15		
	outliers	0+1ex	0+1ex	1	2	1	1	2		
	mean (n)	225.59	237.03	239.51	255.68	281.48	288.11	293.83		
	st.dev. (n)	5.110	3.947	3.157	1.640	2.388	2.794	3.024		
	R(calc.)	14.31	11.05	8.84	4.59	6.69	7.82	8.47		
	st.dev.(D86:20b)	(4.431)	(2.540)	(1.882)	(1.071)	(1.508)	(2.171)	(2.536)		
	R(D86:20b)	(12.41)	(7.11)	(5.27)	(3.00)	(4.22)	(6.08)	(7.10)		

Lab 1108 test results at IBP and 5% rec. excluded because of statistical outliers in related distillation parameters



Determination of Flash Point on combined distillation fractions 5 and 6, results in °C

lab	method	value	mark	z(targ)	remarks
171		----		----	
391	D93-A	94		-2.66	
442		----		----	
445		----		----	
608	D93-A	105.0		1.65	
971	D93-A	103.0		0.87	
1023		----		----	
1026	D93-A	102.5		0.67	
1065		----		----	
1066	D93-A	102.0		0.48	
1089		----		----	
1108	D93-A	91.5		-3.63	
1539	D93-A	100.5		-0.11	
1613	D93-A	100.5		-0.11	
1741		----		----	
1842		----		----	
1902	D93-A	94.0		-2.66	
1949	D93-A	106.5		2.24	
1984		----		----	
1990	D93-A	103		0.87	
6156		----		----	
6201	D93-A	102.5		0.67	
6326	D93-A	100		-0.31	
6379		----		----	
6431	D93-A	106		2.04	
6432	D93-A	>100		----	
normality		OK			
n		14			
outliers		0			
mean (n)		100.79			
st.dev. (n)		4.590			
R(calc.)		12.85			
st.dev.(D93-A:20)		2.556			
R(D93-A:20)		7.16			



APPENDIX 1D – SIMULATED DISTILLATION AND EFFECTIVE CUT POINT D2892

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

lab	method	210-215°C	215-220°C	220-225°C	225-230°C	230-235°C	235-240°C	240-245°C
171		----	----	----	----	----	----	----
391		----	----	----	----	----	----	----
442		----	----	----	----	----	----	----
445		----	----	----	----	----	----	----
608		3.3	12.0	8.0	9.0	13.0	19.0	9.0
971		----	----	----	----	----	----	----
1023		----	----	----	----	----	----	----
1026	D2887	4.3	13.0	7.5	11.5	11.5	15.5	8.0
1065		4.4	8.3	13.0	G(1) 9.7	11.8	11.0	15.2 DG(1)
1066	D2887	4.3	11.3	7.8	9.0	11.8	16.7	8.3
1089		----	----	----	----	----	----	----
1108		----	----	----	----	----	----	----
1539		----	----	----	----	----	----	----
1613		----	----	----	----	----	----	----
1741		----	----	----	----	----	----	----
1842		----	----	----	----	----	----	----
1902	D7169	5.1	12.0	8.5	9.1	8.3	15.4	6.3
1949	D7169	6.0	13.0	9.0	12.0	9.0	16.0	7.0
1984		----	----	----	----	----	----	----
1990		3.3	10.0	8.0	8.0	13.0	13.0	14.0 DG(1)
6156		----	----	----	----	----	----	----
6201	D2887	4.1	11.7	7.1	11.3	12.0	18.7	8.0
6326	D2887	4.5	13.6	9.7	8.9	13.1	17.2	9.6
6379		----	----	----	----	----	----	----
6431	D2887	3.5	11.0	7.0	10.0	12.0	19.0	9.0
6432	D7169	10.1 G(1)	12.0	9.4	11.6	10.0	14.2	7.1
	normality	OK	suspect	OK	OK	OK	OK	OK
	n	10	11	10	11	11	11	9
	outliers	1	0	1	0	0	0	2
	mean (n)	4.30	11.63	8.19	10.01	11.41	15.96	8.04
	st.dev. (n)	0.829	1.493	0.924	1.356	1.634	2.547	1.083
	R(calc.)	2.32	4.18	2.59	3.80	4.57	7.13	3.03

lab	method	245-250°C	250-255°C	255-260°C	260-265°C	265-270°C	total sum	remarks
171		----	----	----	----	----	----	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445		----	----	----	----	----	----	
608		8.5	6.5	5.7	0.0	0.0	94.0	
971		----	----	----	----	----	----	
1023		----	----	----	----	----	----	
1026	D2887	8.5	8.5	3.0	1.3	0.0	92.6	
1065		7.3	6.5	5.0	0.9	0.0	93.1	
1066	D2887	7.7	7.0	6.0	1.5	0.9	92.4	
1089		----	----	----	----	----	----	
1108		----	----	----	----	----	----	
1539		----	----	----	----	----	----	
1613		----	----	----	----	----	----	
1741		----	----	----	----	----	----	
1842		----	----	----	----	----	----	
1902	D7169	6.9	5.4	0.0	0.0	0.0	77.1	low yield of fractions?
1949	D7169	7.5	5.5	2.9	1.3	0.6	89.8	low yield of fractions?
1984		----	----	----	----	----	----	
1990		8.0	8.5	7.6	0.0	0.0	93.5	
6156		----	----	----	----	----	----	
6201	D2887	9.0	8.8	2.9	0.0	0.0	93.6	
6326	D2887	6.7	4.8	4.2	0.0	0.0	92.2	
6379		----	----	----	----	----	----	
6431	D2887	9.0	8.0	5.7	0.0	0.0	94.2	
6432	D7169	4.8	4.4	0.0	0.0	0.0	83.6	low yield of fractions?
	normality	suspect	OK	OK	n.a.	n.a.		
	n	11	11	11	11	11		
	outliers	0	0	0	0	0		
	mean (n)	7.63	6.73	3.90	<2	<1		
	st.dev. (n)	1.211	1.572	2.431	n.a.	n.a.		
	R(calc.)	3.39	4.40	6.81	n.a.	n.a.		

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

lab	method	225-230°C	230-235°C	235-240°C	240-245°C	245-250°C	250-255°C	255-260°C	260-265°C	265-270°C
171		----	----	----	----	----	----	----	----	----
391		----	----	----	----	----	----	----	----	----
442		----	----	----	----	----	----	----	----	----
445		----	----	----	----	----	----	----	----	----
608		0.0	0.7	0.7	0.9	2.3	5.3	6.5	5.5	7.0
971		----	----	----	----	----	----	----	----	----
1023		----	----	----	----	----	----	----	----	----
1026	D2887	0.6	0.4	0.8	1.2	2.7	6.3	5.5	6.0	6.5
1065		0.0	0.0	2.0	1.6	3.4	4.0	8.0	5.8	8.5
1066	D2887	0.8	0.8	1.0	1.3	3.1	5.0	6.3	5.2	7.2
1089		----	----	----	----	----	----	----	----	----
1108		----	----	----	----	----	----	----	----	----
1539		----	----	----	----	----	----	----	----	----
1613		----	----	----	----	----	----	----	----	----
1741		----	----	----	----	----	----	----	----	----
1842		----	----	----	----	----	----	----	----	----
1902	D7169	0.0	1.3	1.4	2.8	4.3	7.0	6.3	8.0	7.6
1949	D7169	1.7	1.5	1.5	2.3	4.0	4.5	6.5	5.0	7.0
1984		----	----	----	----	----	----	----	----	----
1990		0.0	0.0	1.5	0.9	1.9	3.7	7.0	4.5	7.0
6156		----	----	----	----	----	----	----	----	----
6201	D2887	0.0	0.0	1.3	0.9	2.6	6.8	4.7	6.3	7.3
6326	D2887	0.0	1.4	1.2	1.4	3.0	4.7	9.9	5.2	7.6
6379		----	----	----	----	----	----	----	----	----
6431	D2887	0.0	1.6	0.8	1.3	2.8	5.5	4.5	5.5	7.0
6432	D7169	0.9	2.0	2.8	4.5	4.4	8.0	6.2	8.4	8.9
	normality	n.a.	n.a.	n.a.	n.a.	n.a.	OK	not OK	OK	OK
	n	11	11	11	11	11	11	11	9	9
	outliers	n.a.	n.a.	n.a.	n.a.	n.a.	0	0	2	2
	mean (n)	<3	<3	<3	<5	<5	5.52	6.48	5.44	7.13
	st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.	1.352	1.503	0.552	0.345
	R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.	3.78	4.21	1.55	0.96

lab	method	270-275°C	275-280°C	280-285°C	285-290°C	290-295°C	295-300°C	300-305°C	305-310°C	total sum
171		----	----	----	----	----	----	----	----	----
391		----	----	----	----	----	----	----	----	----
442		----	----	----	----	----	----	----	----	----
445		----	----	----	----	----	----	----	----	----
608		11.0	6.0	8.0	10.0	7.0	8.0	9.0	5.5	93.5
971		----	----	----	----	----	----	----	----	----
1023		----	----	----	----	----	----	----	----	----
1026	D2887	11.0	6.5	7.5	10.5	6.5	7.0	9.5	5.0	93.5
1065		8.8	9.0	7.7	7.0	9.8	7.5	6.5	5.7	95.2
1066	D2887	11.3	6.0	7.5	9.8	6.2	8.0	8.5	5.0	93.0
1089		----	----	----	----	----	----	----	----	----
1108		----	----	----	----	----	----	----	----	----
1539		----	----	----	----	----	----	----	----	----
1613		----	----	----	----	----	----	----	----	----
1741		----	----	----	----	----	----	----	----	----
1842		----	----	----	----	----	----	----	----	----
1902	D7169	11.2	6.6	7.1	10.7	6.5	6.7	8.0	2.8	98.1
1949	D7169	8.0	7.5	6.5	7.0	8.5	7.0	5.5	7.3	91.3
1984		----	----	----	----	----	----	----	----	----
1990		9.5	7.5	7.5	7.0	10.0	7.0	7.0	8.7	90.7
6156		----	----	----	----	----	----	----	----	----
6201	D2887	11.0	6.7	7.1	10.5	6.4	7.6	9.5	5.1	93.8
6326	D2887	11.4	6.7	8.1	8.2	8.2	7.5	7.4	4.8	96.5
6379		----	----	----	----	----	----	----	----	----
6431	D2887	11.5	6.5	7.3	10.8	5.5	8.0	9.5	5.0	93.0
6432	D7169	7.3	7.0	6.1	9.5	7.1	5.6	5.9	2.4	97.8
	normality	OK	OK	OK	OK	OK	suspect	OK	OK	
	n	11	10	11	11	11	11	11	11	
	outliers	0	1	0	0	0	0	0	0	
	mean (n)	10.18	6.69	7.30	9.17	7.44	7.26	7.84	5.19	
	st.dev. (n)	1.513	0.521	0.597	1.571	1.497	0.717	1.488	1.768	
	R(calc.)	4.24	1.46	1.67	4.40	4.19	2.01	4.17	4.95	

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

lab	Overlap cuts 5 and 6 in °C	ECP in °C	Difference with AET=250 °C	Conclusion	Remarks
171	----	----	----	----	
391	----	----	----	----	
442	----	----	----	----	
445	----	----	----	----	
608	233.5-264.0= 30.5°C	252.7	2.7	OK	
971	----	----	----	----	
1023	----	----	----	----	
1026	228.5-266.5= 38°C	250.1	0.1	OK	
1065	229.8-298.8= 69°C	250.7	0.7	OK	large overlap of cuts
1066	226.5-270.5= 44°C	251.1	1.1	OK	
1089	----	----	----	----	
1108	----	----	----	----	
1539	----	----	----	----	
1613	----	----	----	----	
1741	----	----	----	----	
1842	----	----	----	----	
1902	228.3-263.1= 35°C	246.3	-3.7	OK	
1949	220.0-301.0= 81°C	247.7	-2.3	OK	large overlap of cuts
1984	----	----	----	----	
1990	231.0-265.5= 35°C	253.2	3.2	OK	
6156	----	----	----	----	
6201	233.4-264.6= 31°C	251.3	1.3	OK	
6326	228.2-264.0= 35.8°C	249.2	-0.76	OK	
6379	----	----	----	----	
6431	223-267= 34°C	253.0	3.0	OK	
6432	219.7-260.7= 41°C	242.6	-7.4	not OK *	

* Acc. to ASTM D2892:20, appendix X2.6.5.1 the difference between ECP and AET should not exceed $0.7R$ °C ($0.7 \times 8 = 6$ °C)

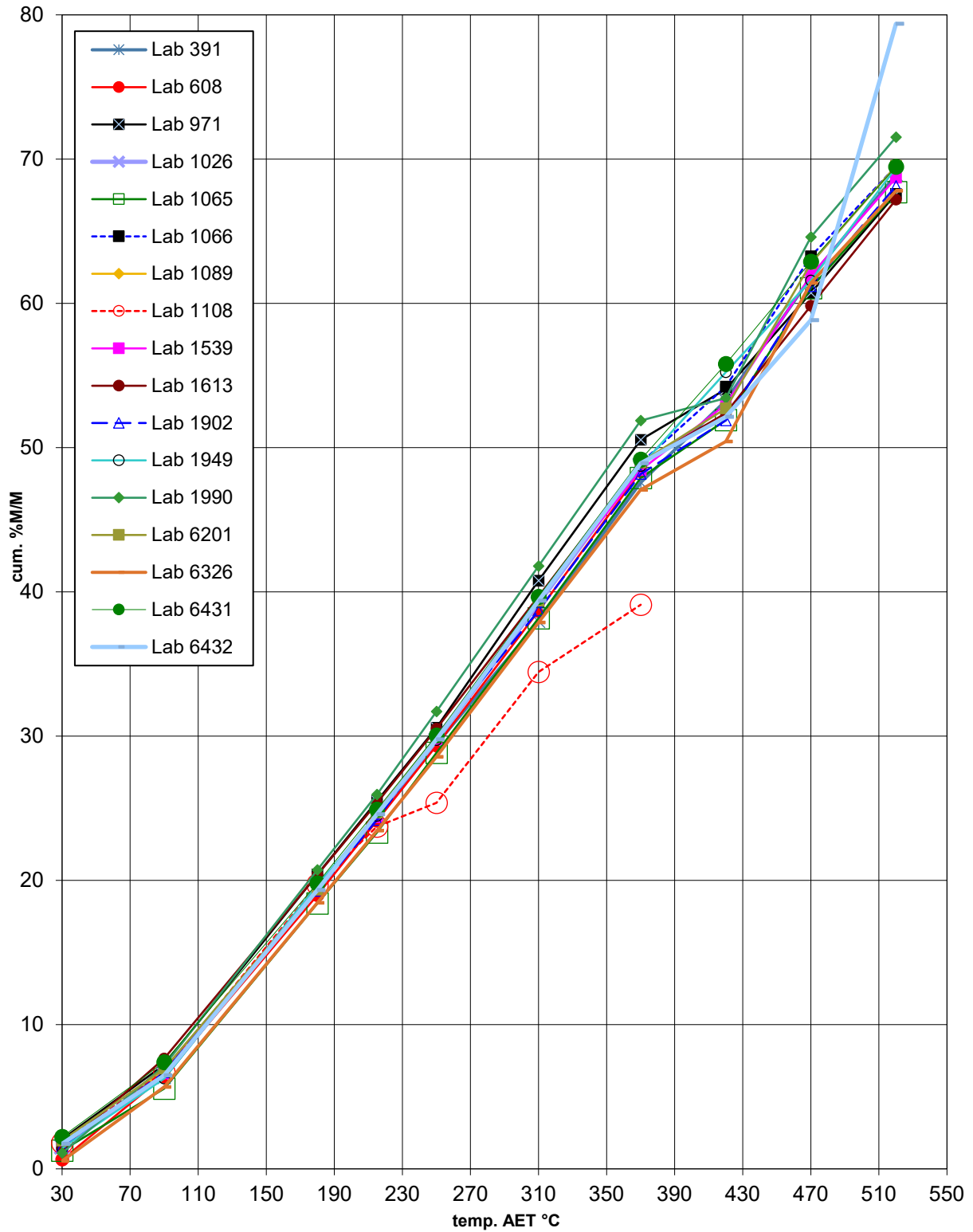
Determination of Standard Efficiency N_{minimum} from the SimDist data

lab	N_{actual}	N_{minimum}	Requirement N_{minimum} *	Remarks
171	----	----	----	
391	----	----	----	
442	----	----	----	
445	----	----	----	
608	8.5	6.7	OK	
971	----	----	----	
1023	----	----	----	
1026	8.4	6.5	OK	
1065	9.5	7.4	OK	
1066	7.8	6.3	OK	
1089	----	----	----	
1108	----	----	----	
1539	----	----	----	
1613	----	----	----	
1741	----	----	----	
1842	----	----	----	
1902	11.0	8.6	not OK	total sum SimDist fraction 5 (too) low?
1949	7.4	5.7	not OK	total sum SimDist fraction 5 (too) low?
1984	----	----	----	
1990	7.4	6.2	OK	
6156	----	----	----	
6201	8.0	6.7	OK	
6326	8.8	7.2	OK	
6379	----	----	----	
6431	7.3	6.0	OK	
6432	6.5	5.5	not OK	total sum SimDist fraction 5 (too) low?

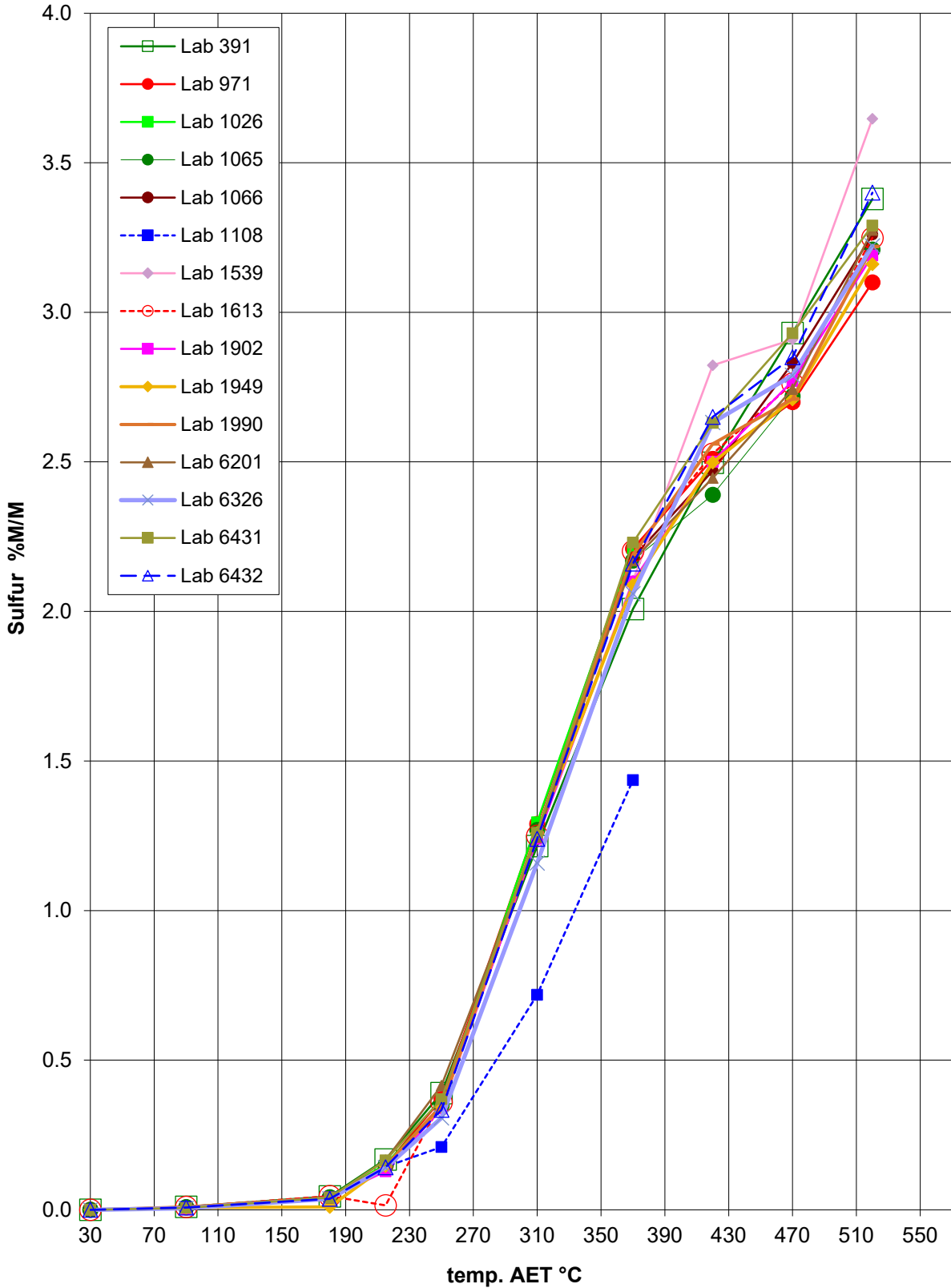
* Acc. to ASTM D2892:20, appendix X2.5.9.2 the acceptable upper and lower limits (14-18 theoretical plates) are 5.8, resp. 7.4

** ASTM D2892 describes three steps: first atmospheric distillation with a maximum Observed Vapor Temperature of 210°C (paragraph 10.3). When further cuts have to be distilled the temperature is reduced by reducing the pressure to 100 mmHg (paragraph 10.4) and if necessary to further stay below 210°C also at lower pressure, but not lower than 2 mmHg (paragraph 10.5).

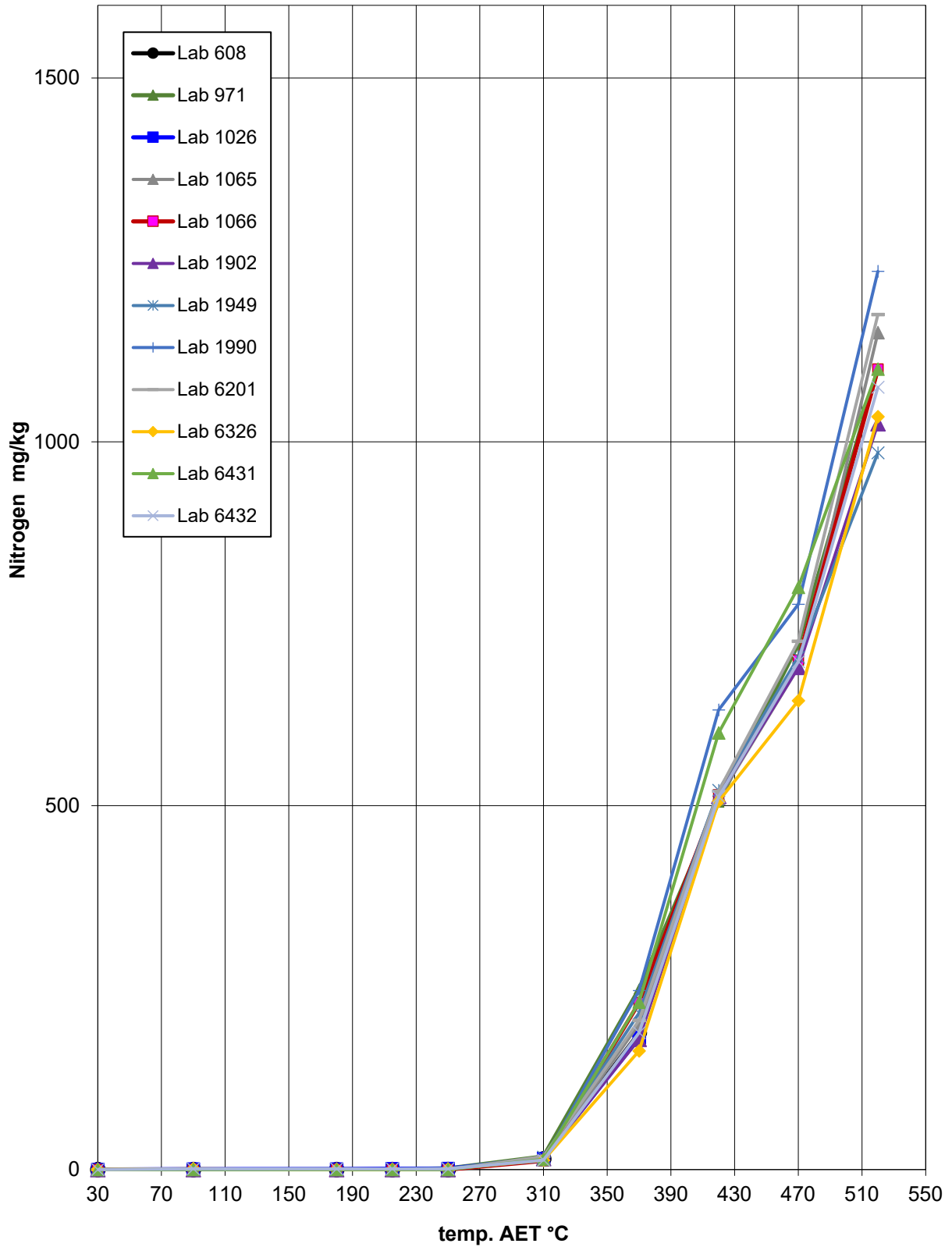
APPENDIX 2 True boiling point graph, cumulative %M/M vs temperature AET in °C



APPENDIX 3 True boiling point graph, Sulfur in %M/M vs temperature AET in °C



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



labs 1108, 1539 and 1613 not included in graph because of insufficient data

APPENDIX 5

Details of Distillation

Lab	Sample received	Distillation started	Distillation finished	Intake in L	Mass charged in grams	Volume charged in mL	End Point distillation(s) in °C
171	3/11/2021	----	----	----	----	----	----
391	21/10/2021	01/12/21	03/12/21	20	10473.4	11990.2	520+
442	----	----	----	----	----	----	----
445	25/10/2021	----	----	----	----	----	----
608	2/12/2021	15/12/2021	21/12/2021	8.0	4081.7	4669.1	370+
971	24/10/2021	10/11/2021	14/11/21	22	6245	7150	520+
1023	22/10/2021	----	----	----	----	----	----
1026	19/10/2021	22/11/21	23/11/21	10	4068.9	4657	370+
1065	2/11/2021	07/11/21	09/11/2021	10	3737	4277.7	520+
1066	23/11/2021	23/11/21	24/11/21	20	6678.4	7647.3	520+
1089	26/10/2021	16/11/21	16/11/21	6	2533.2	2898.7	370+
1108	18/10/2021	16/11/21	18/11/21	6	3350	----	370+
1539	25/10/2021	17/11/2021	18/11/2021	6	3012.7	----	520+
1613	4/11/2021	01/12/2021	2/12/2021	5.0	3497.2	4000	520+
1741	25/10/2021	----	----	----	----	----	----
1842	----	----	----	----	----	----	----
1902	20/10/2021	29/10/21	01/11/21	22	12060	13802	520+
1949	1/12/2021	02/12/21	05/12/21	10.0	4019.0	4598.92	520+
1984	18/10/2021	----	----	----	----	----	----
1990	29/10/2021	15/11/21	18/11/21	50	27914.2	31937.7	520+
6156	----	----	----	----	----	----	----
6201	13/10/2021	08/12/2021	13/12/2021	20	6573.7	7523.1	520+
6326	8/12/2021	12/12/21	14/12/21	20	7774	8904.8	520+
6379	25/10/2021	----	----	----	----	----	----
6431	11/12/2021	11/16/21	11/23/21	12	6024.0	6895.6	520+
6432	19/11/2021	22/11/21	4/12/21	22	7815	8953	520+

Details of Pressure during distillation ASTM D2892 and ASTM D5236

Lab	Gas LPG <30°C	L.Naphtha 30-90°C	H.Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	VGO 370-420°C	VGO 420-470°C	VGO 470-520°C
171	n	n	n	n	n	n	n	n	n	n
391	762	762	762	100.0	100.0	2.0	2.0	0.2	0.2	0.2
442	n	n	n	n	n	n	n	n	n	n
445	n	n	n	n	n	n	n	n	n	n
608	760	760	760	100	100	3	3	n	n	n
971	760	760	760	100	100	5	5	0.1	0.1	0.1
1023	n	n	n	n	n	n	n	n	n	n
1026	767.3	767.8	769.3	99.6	99.6	2.045	2.045	n	n	n
1065	738.3	736.1	734.0	99.3	100.4	2.00	2.00	0.1973	0.1993	0.1972
1066	772.6	772.6	772.6	100	100	5	5	1	1	1
1089	755.6	755.0	753.4	100.0	100.0	10.0	5.0	n	n	n
1108	755	755	755	100.2	10.01	10.01	2.50	n	n	n
1539	761.22906	761.22906	761.59783	100.95826	99.787651	1.993058	1.993058	0.209707	0.226950	0.241730
1613	714	714	714	100	100	100	10	1.27	1.27	1.27
1741	n	n	n	n	n	n	n	n	n	n
1842	n	n	n	n	n	n	n	n	n	n
1902	760	760	760	760	100	2	2	1	0.3	0.3
1949	749.0	749.0	749.0	100.0	100.0	2.0	2.0	1.0	1.0	1.0
1984	n	n	n	n	n	n	n	n	n	n
1990	760	748	748	100	100	10	2	1	0.89	0.77
6156	n	n	n	n	n	n	n	n	n	n
6201	743.5	743.5	743.5	2.0	2.0	2.0	2.0	2.0	1.0	1.0
6326	760	760	760	100.038	99.977	4.944	5.127	1.000	0.63	0.65
6379	n	n	n	n	n	n	n	n	n	n
6431	760	760	760	93.4	93.8	6.4	6.3	1.03	0.21	0.21
6432	753.7	753.5	752.5	100.0	100.0	10.0	10.0	1.00	1.00	0.30

lab 1108 dropped pressure to 10 mmHg already at fraction 5 (LGO)

lab 1613: observed Vapor Temperature in this step >210°C (ASTM D2892, paragraph 10.3)

lab 1902 observed Vapor Temperature in this step >210°C (ASTM D2892, paragraph 10.3)

lab 6201 dropped pressure to 2 mmHg already at fraction 4 (Kerosene)

APPENDIX 6

Number of participants per country

1 lab in FINLAND
1 lab in FRANCE
1 lab in GREECE
1 lab in ISRAEL
1 lab in ITALY
1 lab in JORDAN
4 labs in MALAYSIA
3 labs in NETHERLANDS
2 labs in NORWAY
1 lab in POLAND
1 lab in RUSSIAN FEDERATION
1 lab in SERBIA
2 labs in SINGAPORE
1 lab in UNITED ARAB EMIRATES
3 labs in UNITED KINGDOM
2 labs in UNITED STATES OF AMERICA

APPENDIX 7

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01) or G(1)	= outlier in Grubbs' outlier test
G(0.05) or G(5)	= straggler in Grubbs' outlier test
DG(0.01) or DG(1)	= outlier in Double Grubbs' outlier test
DG(0.05) or DG(5)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
- 5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 8 J.N. Miller, Analyst, 118, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
- 10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
- 11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)