

Results of Proficiency Test
Crude Oil Assay
December 2009

Organised by: Institute for Interlaboratory Studies (iis)
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1 INTRODUCTION

During the planning of the annual proficiency testing program 2004/2005, it was decided to organize a special round robin for a Crude Oil TBP / Assay. After the finishing of this round robin a questionnaire was sent to all participating laboratories and from the returned questionnaires it became clear that a repetition (slightly changed) every two years would be welcomed. Therefore the Crude Oil Assay round robin was repeated in 2007 and 2009. In the international interlaboratory study of 2009, 16 laboratories from 12 different countries have participated. See appendix 6 for a list of participants in alphabetical country order. In this report the results of the 2009 Crude Oil Assay proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (i.i.s.) in Spijkenisse, The Netherlands, was the organizer of this proficiency test. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43 and ILAC-G13:2007. This ensures 100% confidentiality of participant's data. Also customer's satisfaction is measured on a 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 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of November 2008 (iis-protocol, version 3.1).

2.3 SAMPLES

The necessary bulk material was obtained from a local crude oil storage facility. The approx. 190 litre of Crude Oil was homogenised in a metal drum. After homogenisation, 40 subsamples were transferred to 5000 mL metal cans labelled #0984. The homogeneity of the subsamples was checked by determination of Density in accordance with ASTM D5002:05 of 4 stratified random selected samples.

	<i>Density @ 15 °C in kg/L</i>
sample #0984-1	0.85486
sample #0984-2	0.85481
sample #0984-3	0.85482
sample #0984-4	0.85491

table 1: homogeneity test of subsamples #0984

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

	<i>sample #0984</i>
Repeatability density kg/L	0.00013
Reference method	ASTM D5002:05
$0.3 \times R_{(\text{Reference method})}$	0.00106

table 2: repeatabilities of subsamples #0984

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

To each of the participating laboratories one or more cans of 5000 mL (as required) were sent on October 14, 2009.

2.4 ANALYSES

The participants were requested to determine a Crude Oil Assay consisting of a True Boiling Point Distillation in accordance with ASTM D2892 and the collection of 8 fractions. On the original sample and on all fractions Density, Sulphur and Nitrogen content should be determined where possible. Furthermore on the light and heavy naphtha fractions a PIONA or PONA analysis was requested; on the combined fractions 4 +5 (kerosene and light gasoil) a D86-distillation and on the individual fractions 4 and 5 also a simdist determination.

To get comparable results a detailed report form, on which the units and the standard methods were printed, was sent together with each set of samples. Also a letter of instructions and a SDS were added to the package.

3 RESULTS

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

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

Also after the deadline the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the raw data of these tests (no reanalysis). Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of November 2008 (iisprotocol, version 3.1).

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

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

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This method is producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 7; nr.12 and 13).

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

The z-scores were calculated in accordance with:

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

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

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

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

4 EVALUATION

In the proficiency test some problems were encountered with sample despatch to Belarus. After the period of 4 weeks after sample despatch - originally set to report results - only eight participants had reported results. Therefore the deadline was extended to January 4, 2010. Evaluation of results and preparation of the final report were delayed significantly by these problems.

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

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

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

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

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

4.1 EVALUATION PER TEST

- Density:** The density determination on the original crude sample showed that two participating laboratories had analytical problems. Remarkably these were not the two laboratories that used D4052 as test method, a method that is not applicable for crude oil.
- Two statistical outliers were detected. However, the observed reproducibility, after rejection of the statistical outliers, is in good agreement with the requirements of ASTM D5002:05.
- The density results on the 8 collected distillation fractions show large differences between the reported test results of the participating laboratories, although no statistical outliers were detected.
- The 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.
- One reported density for the first fraction (gas <30°C) is significantly high (≥ 0.61 kg/L; compare with the density of 0.6 kg/L for isopentane with bp 28°C and the density of 0.626 for n-pentane with bp 36.1°C).
- When these four high results are excluded, the average density of the ten remaining results is 0.5680 kg/L, which is in good agreement with the density of a mix of C3 and C4 hydrocarbons and isopentane.
- A high density result for the first fraction may possibly be caused by degassing problems.
- Sulphur:** The sulfur determination on the original crude sample showed no analytical problems. No statistical outliers were detected and the observed reproducibility is in good agreement with the requirements of ASTM D4294:03.
- The sulfur results on the 8 collected distillation fractions show problems for several participating laboratories. In total 6 statistical outliers (=7%) were detected (4 from laboratory 442).
- The observed reproducibility per fraction will be the sum of the spread in the sulfur 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.
- However, it is remarkable to see the relatively low spread of the sulfur determinations for all fractions.
- Nitrogen:** The nitrogen determination on the original crude sample showed no analytical problems. One statistical outlier was detected and the observed reproducibility, after rejection of the statistical outlier, is in good agreement with the requirements of ASTM D5762:08. This is remarkable as 4 laboratories used method D4629 that is not applicable

for Crude Oil, but limited to 'liquid hydrocarbons boiling in the range from approximately 50°C to 400°C'.

The nitrogen results on the 8 collected distillation fractions show problems for several participating laboratories. In total 5 statistical outliers (=10%) were detected (3 from laboratory 1066).

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.

PNA:

This determination was performed on fractions 2 (light naphtha) and 3 (heavy naphtha) only. Some analytical problems were observed. In total 15 statistical outliers were detected (=16%! Of which 5 from laboratory 1023). It was remarkable to find that most laboratories did not add the amount C1-C4 to the total paraffine result. This was corrected manually by iis for fraction 2. Several laboratories reported significant amounts of C1-C4 for fraction 3 (heavy naphtha; 90-180), which is very unlikely. Each observed reproducibility will be the sum of the spread in the D5443 (or other PNA method used) 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.

D86 distill:

This determination was performed on the combined fractions 4+5 only. Some individual problems were observed. In total only 5 statistical outliers were detected (=3.7%; Of which 4 from laboratory 494). Remarkably, several laboratories reported the presence of C1-C4 in the combined distillation fraction 4+5 (180-250°C). Each observed reproducibility will be the sum of the spread in the D86 result and the spread caused by the D2892. Therefore the observed reproducibilities were not compared with the requirements and consequently no z-scores were calculated.

Simdist:

This determination was performed on fractions 4 and 5 (kerosene and light gasoil) only. The goal was to enable evaluation of the column efficiency in accordance with appendix X2 of ASTM D2892:05. Almost all reported results were suitable to calculate the ECP (effective cut point) and the standard efficiency N_{minimum} . Most results were in agreement with the ASTM D2892:05 requirements.

D2892:

Only for the heaviest fractions (250-310°C and >310°C) some problems have been observed. In total only 3 statistical outliers were detected (=2.7%). After exclusion of the statistical outliers, all calculated reproducibilities, except for fractions 6 (MGO 250-310°C) and 7 (HGO 310-370°C), are in good agreement with requirements of ASTM D2892:05.

The starts of the true boiling point curves (cum%M/M vs temp AET) of all laboratories show a large resemblance. The curve of two laboratories

(442 and 494) show a positive deviation at the upper part of the curve (250°C-310°C).

The true boiling point curves curves (S in %M/M vs temp AET) of most laboratories show a large resemblance. The curves of two laboratories (1023 and 1065) show a positive deviation at the lower part of the curve (30°C-190°C). The deviations are most probably due to analytical problems with the sulfur determination (on several fractions) and not with the distillation.

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

The evaluation of the Total Mass balance showed 4 very low recoveries that do not meet the ASTM D2892 (paragraph 11.2) requirement of 0.4% max. loss. One laboratory (445) obviously did correct for loss before reporting the D2892 results.

The evaluation of the Total Volume balance showed 3 recoveries (%V/V) that are lower than the respective recoveries in %M/M. The opposite is 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 D2892:05.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sd_R</i>	<i>R (lit)</i>
Density of #0984	kg/L	12	0.8552	0.0007	0.0035
Sulfur of #0984	%M/M	11	1.08	0.10	0.14
Nitrogen of #0984	mg/kg	7	906	199	241
D2892 distillation					
LPG fraction < 30°C	%M/M	14	1.34	1.23	1.2
light naphtha 30 - 90°C	%M/M	14	4.49	1.06	1.2
heavy naphtha 90 - 180°C	%M/M	14	13.6	0.86	1.2
kerosene 180 - 215°C	%M/M	14	5.42	1.06	1.2
LGO 215 - 250°C	%M/M	13	6.38	0.74	1.4
MGO 250 - 310°C	%M/M	13	11.9	1.54	1.4
HGO 310 - 370°C	%M/M	13	11.3	2.26	1.4
residue > 370°C	%M/M	14	44.6	4.17	n.a

table 3: reproducibilities of original crude sample #0984

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

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

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R (lit)</i>
Density @15°C of cut 1: gas <30°C	kg/L	10	0.5680	0.0442	n.r.
Density @15°C of cut 2: 30-90°C	kg/L	14	0.6793	0.0185	n.r.
Density @15°C of cut 3: 90-180°C	kg/L	14	0.7566	0.0042	n.r.
Density @15°C of cut 4: 180-215°C	kg/L	14	0.7963	0.0081	n.r.
Density @15°C of cut 5: 215-250°C	kg/L	14	0.8148	0.0075	n.r.
Density @15°C of cut 6: 250-310°C	kg/L	14	0.8375	0.0060	n.r.
Density @15°C of cut 7: 310-370°C	kg/L	14	0.8628	0.0089	n.r.
Density @15°C of residue: >370°C	kg/L	13	0.9468	0.0119	n.r.

table 4: reproducibilities of density determinations on distillation fractions

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sdR</i>	<i>R (lit)</i>
Sulfur on cut 1: gas <30°C	%M/M	4	0.007	0.011	n.r.
Sulfur on cut 2: 30-90°C	%M/M	11	0.010	0.006	n.r.
Sulfur on cut 3: 90-180°C	%M/M	11	0.032	0.008	n.r.
Sulfur on cut 4: 180-215°C	%M/M	12	0.078	0.012	n.r.
Sulfur on cut 5: 215-250°C	%M/M	12	0.160	0.058	n.r.
Sulfur on cut 6: 250-310°C	%M/M	12	0.478	0.087	n.r.
Sulfur on cut 7: 310-370°C	%M/M	11	0.878	0.109	n.r.
Sulfur on residue: >370°C	%M/M	11	2.032	0.166	n.r.

table 5: reproducibilities of sulfur determinations on distillation fractions

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sdR</i>	<i>R (lit)</i>
Nitrogen on cut 1: gas <30°C	mg/kg	3	n.a.	n.a.	n.r.
Nitrogen on cut 2: 30-90°C	mg/kg	4	1.08	2.52	n.r.
Nitrogen on cut 3: 90-180°C	mg/kg	4	0.52	1.28	n.r.
Nitrogen on cut 4: 180-215°C	mg/kg	5	1.50	1.86	n.r.
Nitrogen on cut 5: 215-250°C	mg/kg	7	5.65	8.50	n.r.
Nitrogen on cut 6: 250-310°C	mg/kg	7	35.2	21.0	n.r.
Nitrogen on cut 7: 310-370°C	mg/kg	9	233	186	n.r.
Nitrogen on residue: >370°C	mg/kg	8	1825	1822	n.r.

table 6: reproducibilities of nitrogen determinations on distillation fractions

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sdR</i>	<i>R (lit)</i>
Total Paraffines	%V/V	9	82.16	9.86	n.r.
C1-C4	%V/V	9	1.90	4.47	n.r.
n-paraffines	%V/V	9	42.97	4.05	n.r.
i-paraffines	%V/V	9	37.30	3.07	n.r.
naphthalenes	%V/V	9	17.67	5.38	n.r.
aromatics	%V/V	9	2.01	0.98	n.r.
Total Paraffines	%M/M	10	77.95	9.61	n.r.
C1-C4	%M/M	10	1.52	3.20	n.r.
n-paraffines	%M/M	9	40.91	4.28	n.r.
i-paraffines	%M/M	9	35.85	4.49	n.r.
naphthalenes	%M/M	10	20.43	6.04	n.r.
aromatics	%M/M	10	2.64	1.15	n.r.

table 7: reproducibilities of PNA determination on distillation fraction 2 (light naphtha)

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 *sdR</i>	<i>R (lit)</i>
Total Paraffines	%V/V	8	57.90	5.26	n.r.
C1-C4	%V/V	6	0.014	0.041	n.r.
n-paraffines	%V/V	6	27.25	0.58	n.r.
i-paraffines	%V/V	8	30.17	5.09	n.r.
naphthalenes	%V/V	6	29.64	2.95	n.r.
aromatics	%V/V	6	12.66	1.29	n.r.
Total Paraffines	%M/M	8	54.68	4.45	n.r.
C1-C4	%M/M	5	0.016	0.039	n.r.
n-paraffines	%M/M	6	25.51	0.57	n.r.
i-paraffines	%M/M	7	28.50	4.31	n.r.
naphthalenes	%M/M	7	30.57	3.12	n.r.
aromatics	%M/M	8	14.74	3.00	n.r.

table 8: reproducibilities of PNA 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	11	189.5	16.0	n.r.
5% evaporated	°C	11	195.8	17.6	n.r.
10% evaporated	°C	11	198.9	12.9	n.r.
50% evaporated	°C	10	211.5	4.7	n.r.
90% evaporated	°C	10	230.1	6.1	n.r.
95% evaporated	°C	10	235.3	7.1	n.r.
fbp	°C	12	246.3	14.3	n.r.
5% recovered	°C	11	196.2	19.6	n.r.
10% recovered	°C	11	199.5	13.7	n.r.
50% recovered	°C	11	212.7	7.5	n.r.
90% recovered	°C	10	231.3	6.0	n.r.
95% recovered	°C	11	237.5	10.8	n.r.

table 9: reproducibilities of D86 determination on combined distillation fraction 3+4

4.3 DISCUSSION

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

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

The density, sulphur and nitrogen results do show more aberrant results. These deviations may be (partly) explained by the cumulative effect of spread caused by distillation and by subsequent analytical determinations.

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

Parameter	unit	average measured result	average theoretical result	average difference	average recovery
Density	kg/L	0.8551 5	0.8536 4	-0.0015 1	-----
Sulphur	%M/M	1.079	1.093	+0.013	101%
Nitrogen	mg/L	906.1	722.1	-183.9	80%

The theoretical calculated density is a measure for the volume balance. The lowest density result (lab 862) does correspond with a total volume recovered of 100.25%, while the highest density result (lab 391) corresponds with a total volume recovered of 99.69%. The total mass balance varied from 99.06% up to 100.0%.

ASTM D2892 does not give criteria for recovery, except that is stated that weight loss in excess of 0.4 % is a reason to discard the distillation data (see paragraph 11.2). All but three laboratories did fulfil this latter requirement in this proficiency test (see page 29). For comparison, in ASTM D5236 is stated that the total recovery must be between 99.6 % and 100.1 % of the weight of the charge to be acceptable.

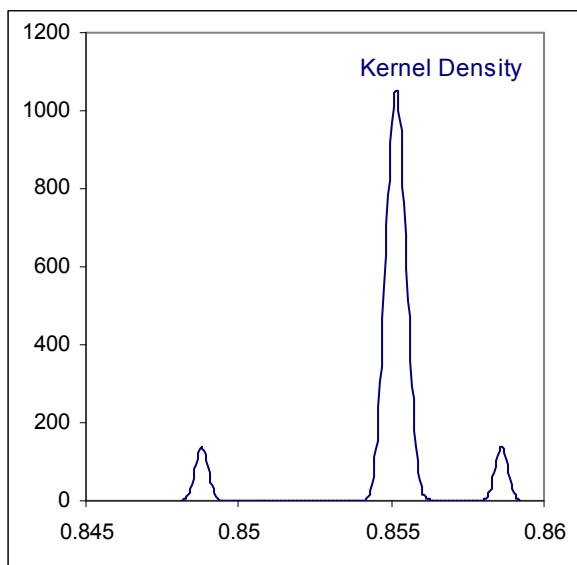
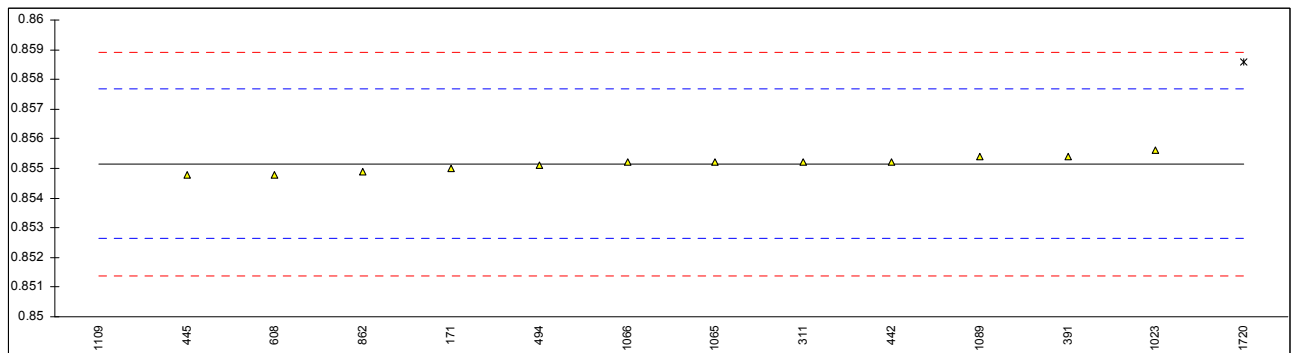
The average theoretical sulphur content shows an excellent resemblance to the average measured sulphur content and also the individual differences are remarkably small (0.006 - 0.054 %M/M) and even the calculated reproducibility of the calculated theoretical sulphur content is in good agreement with the requirement of ASTM D4294:03. The sulphur determination obviously is much less sensitive than the density determination. This will be caused by the fact that the sulfur is present as a series of homologs.

The average theoretical nitrogen content shows a fair resemblance to the average measured nitrogen content (722 vs 906 mg/kg). However, the individual differences vary over a large range (3 – 432 mg/kg). Also, the calculated reproducibility of the calculated theoretical nitrogen content is not at all in agreement with the requirement of ASTM D5762:08. Most probably the problematic nitrogen determination and the relatively high limit of detection of this determination are the main causes for the large spread rather than the distillation.

APPENDIX 1 RESULTS TABLES

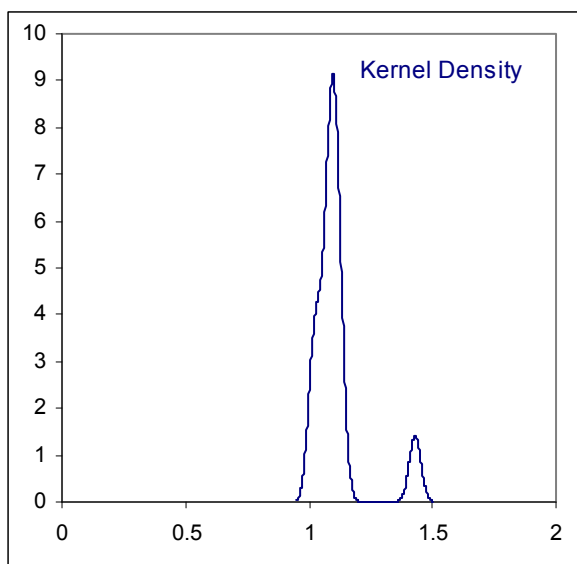
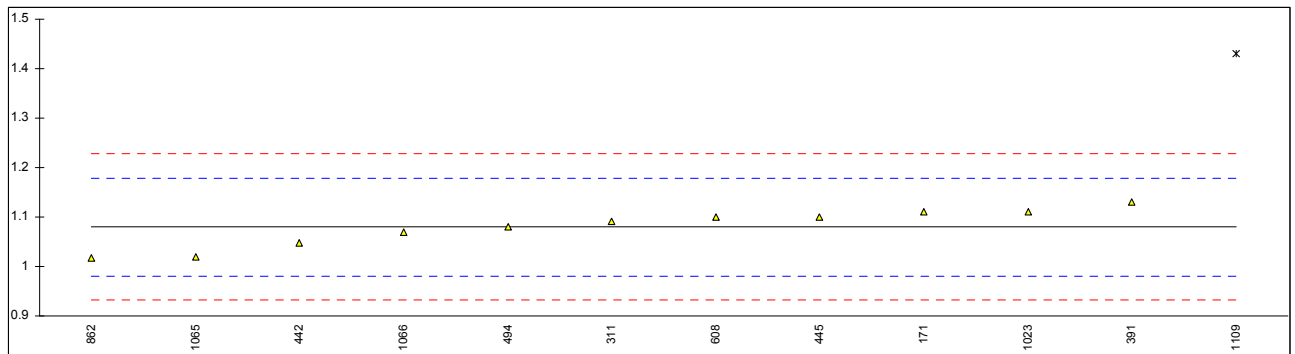
Determination of Density @15°C on original sample #0984; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D1298	0.8550		-0.12	
311	D5002	0.8552		0.04	
391	D1298	0.8554		0.20	
442	IP365	0.8552		0.04	
445	D4052	0.8548		-0.28	
494	D4052	0.8551		-0.04	
608	D5002	0.8548		-0.28	
862	D5002	0.8549		-0.20	
1023	D5002	0.8556		0.36	
1065	D5002	0.8552		0.04	
1066	D5002	0.8552		0.04	
1089	D5002	0.8554		0.20	
1109	D1298	0.8488	G(0.01)	-5.05	
1432		----		----	
1720	D5002	0.8586	G(0.01)	2.74	reported 858.6 kg/m ³
1842		----		----	
normality		OK			
n		12			
outliers		2			
mean (n)		0.85515			
st.dev. (n)		0.000247			
R(calc.)		0.00069			
R(D5002:05)		0.00352			



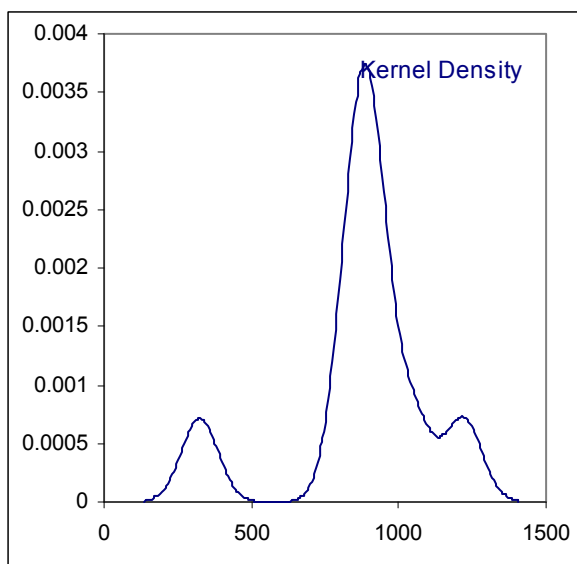
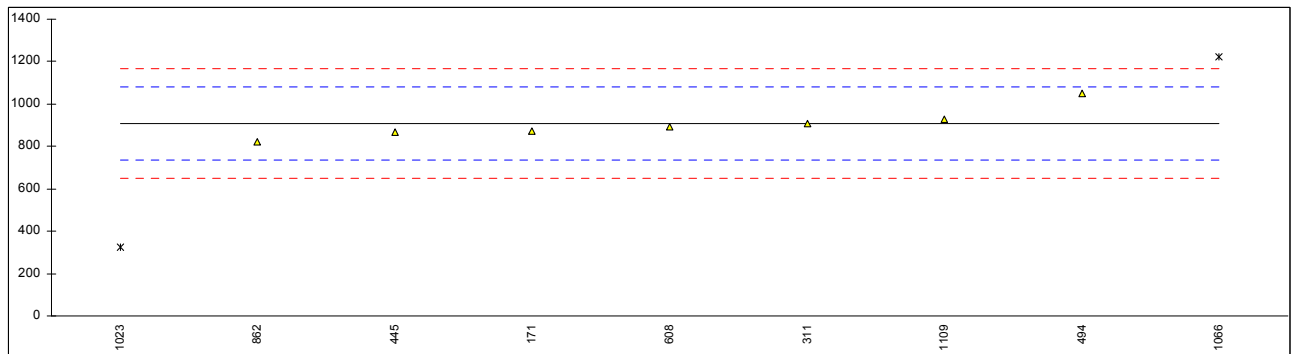
Determination of Sulphur on original sample #0984; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D4294	1.11		0.62	
311	D2622	1.091		0.23	
391	ISO8754	1.13		1.03	
442	IP336	1.0470		-0.66	
445	IP336	1.10		0.42	
494	ISO8754	1.08		0.01	
608	D4294	1.099		0.40	
862	D2622	1.017		-1.27	
1023	IP336	1.11		0.62	
1065	IP336	1.02		-1.21	
1066	D2622	1.07		-0.19	
1089		----		----	
1109	D4294	1.43	G(0.01)	7.12	
1432		----		----	
1720		----		----	
1842		----		----	
normality		OK			
n		11			
outliers		1			
mean (n)		1.079			
st.dev. (n)		0.0373			
R(calc.)		0.105			
R(D4294:03)		0.138			



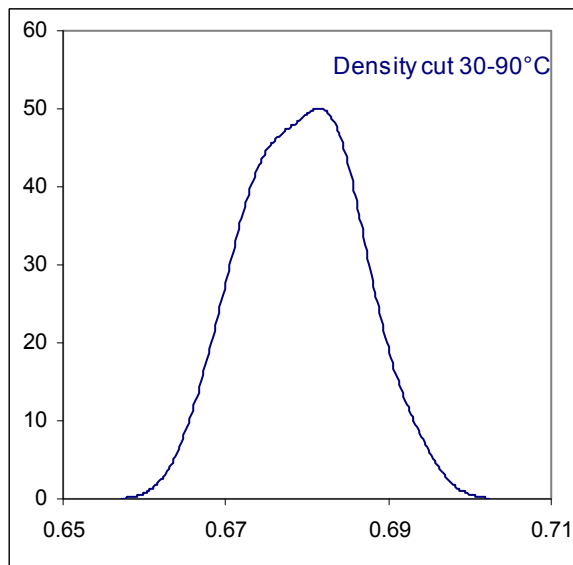
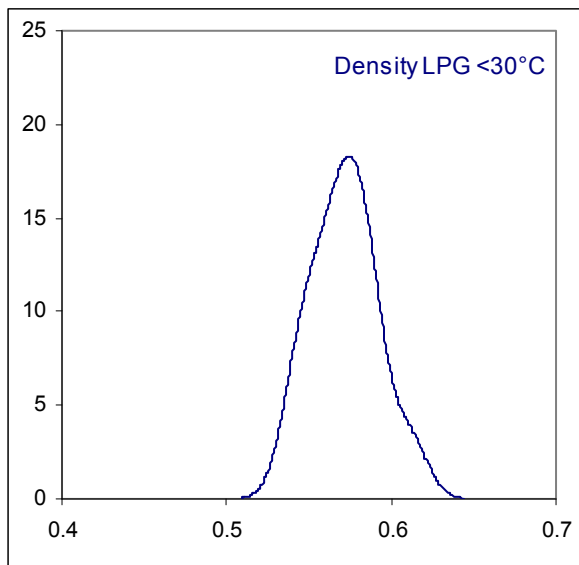
Determination of Nitrogen on original sample #0984; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D4629	873		-0.38	
311	D5762	910		0.05	
391		----		----	
442		----		----	
445	D5762	865		-0.48	
494	D5762	1048		1.65	
608	D4629	894		-0.14	
862	D4629	823.4		-0.96	
1023	UOP269	----		----	reported 325 mg/kg base N, not total N
1065		----		----	
1066	D5762	1220	G(0.05)	3.65	
1089		----		----	
1109	D4629	929		0.27	
1432		----		----	
1720		----		----	
1842		----		----	
normality		OK			
n		7			
outliers		1			
mean (n)		906.1			
st.dev. (n)		71.23			
R(calc.)		199.5			
R(D5762:08)		241.0			



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

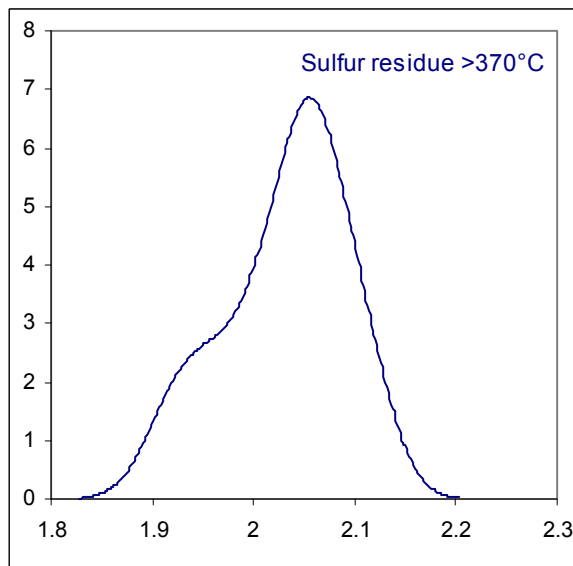
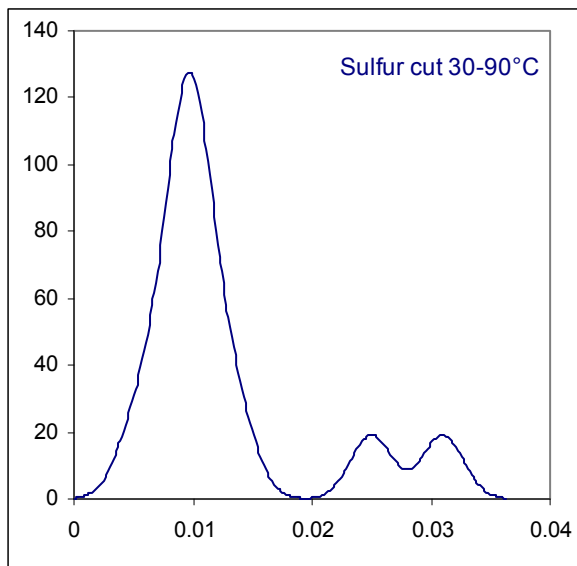
lab	Gas LPG <30°C	L.Naphtha 30-90°C	H.Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C	remarks
171	0.5857	0.6742	0.7571	0.8004	0.8123	0.8353	0.8587	0.9485	
311	0.5526	0.6824	0.7558	0.7970	0.8143	0.8388	0.8622	0.9451	
391	0.5757	0.6863	0.7597	0.7962	0.8141	0.8395	0.8654	0.9505	
442	0.5674	0.6918	0.7548	0.7942	0.8172	0.8359	0.8676	0.9575	
445	0.5691	0.6678	0.7552	0.7980	0.8134	0.8350	0.8592	0.9444	
494	0.5850	0.6856	0.7583	0.7988	0.8174	0.8426	0.8692	0.9505	
608	0.5495	0.6751	0.7563	0.7962	0.8142	0.8382	0.8587	0.9431	
862	0.5419	0.6733	0.7565	0.7971	0.8147	0.8392	0.8630	0.9457	
1023	0.6107 ex	0.6822	0.7583	0.8003	0.8161	0.8371	0.8653	0.9467	see page 7
1065	----	0.6841	0.7563	0.7960	0.8122	0.8385	0.8618	0.9434	
1066	0.58578	0.6774	0.7568	0.7973	0.8132	0.8365	0.8628	0.9463	
1089	----	0.6712	0.7539	0.7941	0.8120	0.8369	0.8612	0.9449	
1109	0.5672	0.6808	0.7565	0.7896	0.8220	0.8364	0.8609	0.9411	
1432	----	----	----	----	----	----	----	----	
1720	----	0.6784	0.7564	0.7933	0.8144	0.8349	0.8634	----	
1842	----	----	----	----	----	----	----	----	
normality	OK	OK	OK	OK	not OK	OK	OK	OK	
n	10	14	14	14	14	14	14	13	
outliers	0	0	0	0	0	0	0	0	
mean (n)	0.56799	0.67933	0.75656	0.79632	0.81482	0.83749	0.86281	0.94675	
st.dev. (n)	0.015778	0.006623	0.001494	0.002873	0.002670	0.002134	0.003181	0.004245	
R(calc.)	0.04418	0.01854	0.00418	0.00805	0.00748	0.00598	0.00891	0.01188	



Determination of Sulphur on distillation fractions; results 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	remarks
171	0.0010	0.0073	0.0320	0.085	0.144	0.437	0.832	2.06	
311	----	0.0090	0.0296	0.0768	0.154	0.491	0.885	2.064	
391	0.008	<i>0.025</i>	0.038	0.082	0.17	0.51	0.92	2.11	
442	<i>1.0630</i>	<i>0.0310</i>	<i>0.0185</i>	<i>0.0535</i>	0.1500	0.4470	0.8910	2.0570	fr 0.0891
445	0.01	0.01	0.03	0.07	0.15	0.45	0.84	1.99	
494	----	0.012	0.033	0.083	0.20	0.55	0.93	2.05	
608	----	0.010	0.0322	C 0.074	0.161	0.485	0.854	2.040	fr 0.322
862	----	0.010	0.034	0.077	0.156	0.476	0.845	2.010	
1023	----	0.01	0.032	C 0.08	0.18	0.49	0.94	2.10	fr 0.32
1065	----	0.01	C 0.03	0.08	0.12	0.47	0.84	1.92	fr 0.12
1066	0.0070	0.0137	0.0358	0.0777	0.157	0.460	0.879	1.95	
1089	----	----	----	----	----	----	----	----	
1109	----	0.009	0.028	0.073	0.181	0.464	----	----	
1432	----	----	----	----	----	----	----	----	
1720	----	0.0053	<i>0.0191</i>	0.0791	----	----	----	----	
1842	----	----	----	----	----	----	----	----	
normality	n.a.	not OK	OK	OK	OK	OK	OK	OK	
n	4	11	11	12	12	12	11	11	
outliers	1	2	2	1	0	0	0	0	
mean (n)	0.0065	0.0097	0.0322	0.0781	0.1602	0.4775	0.8778	2.0319	
st.dev. (n)	0.00387	0.00219	0.00291	0.00433	0.02058	0.03102	0.03899	0.05920	
R(calc.)	0.0108	0.0061	0.0082	0.0121	0.0576	0.0868	0.1092	0.1658	

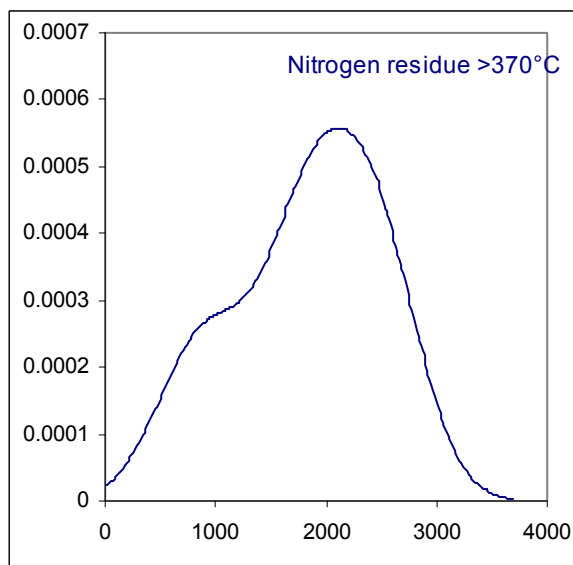
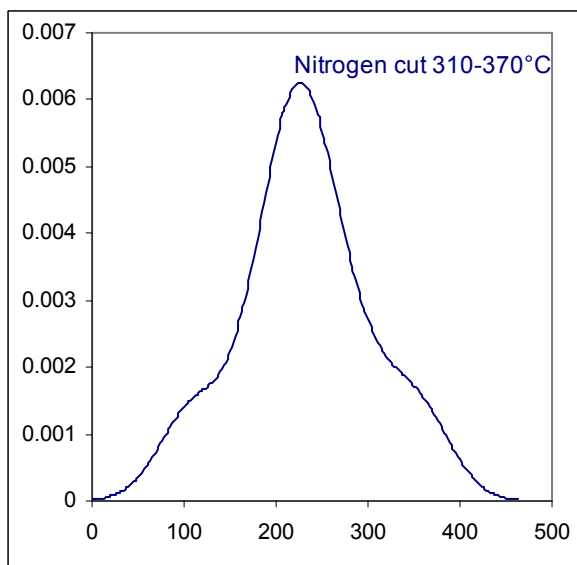
NB. Bold, italic and underlined figures are statistical outliers (Grubbs and/or Dixon)



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

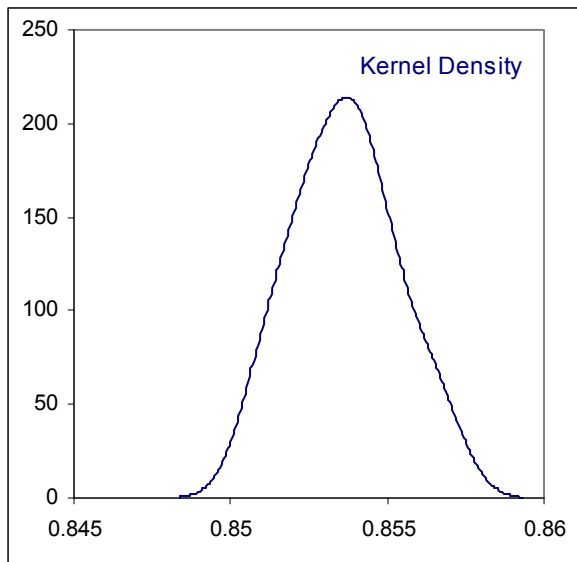
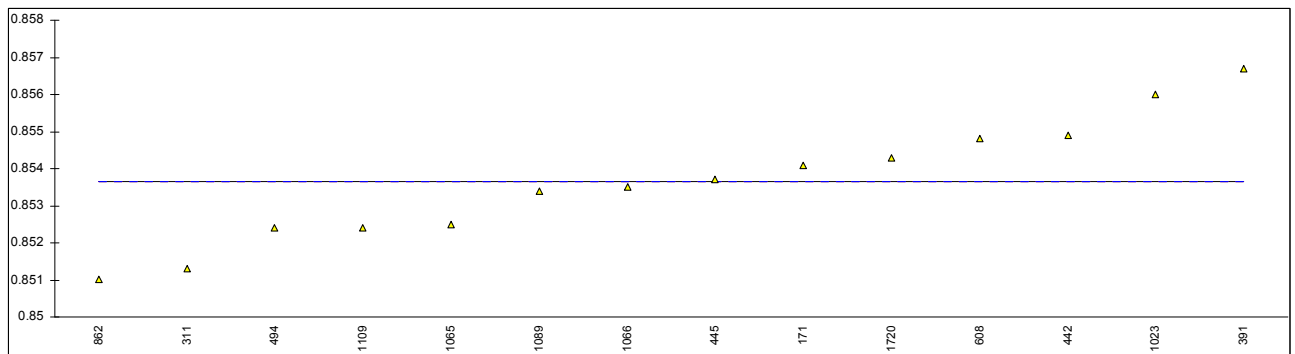
lab	Gas LPG <30°C	L.Naphtha 30-90°C	H.Naphtha 90-180°C	Kerosene 180-215°C	LGO 215-250°C	MGO 250-310°C	HGO 310-370°C	Residue >370°C	remarks
171	<1	<1	<1	<1	3	<u>69</u>	232	1949	
311	----	<0.3	0.3	1.3	3.5	38	C 240	C 2300	fr 23.8; 90.2
391	----	----	----	----	----	----	----	----	
442	----	----	----	----	----	----	----	----	
445	<40	<40	<40	<40	<40	<40	234	1031	
494	----	<0.3	<0.3	1.6	9.6	<u>81</u>	353	2500	
608	----	<1	<1	<1	<1	34	205	1766	
862	----	2.2	1.2	2.6	6.1	37.1	239.5	1763.8	
1023	----	----	<1	<1	10	25	110	760	
1065	----	----	----	----	----	----	----	----	
1066	<1	1.4	<u>9</u>	<u>11</u>	<u>31</u>	40.5	289	2530	
1089	----	----	----	----	----	----	----	----	
1109	----	0.50	0.24	0.93	4.36	45.66	----	----	
1432	----	----	----	----	----	----	----	----	
1720	----	0.22	0.33	1.08	2.96	26	190	----	
1842	----	----	----	----	----	----	----	----	
normality	n.a.	n.a.	n.a.	OK	OK	OK	OK	OK	
n	3	4	4	5	7	7	9	8	
outliers	n.a.	0	1	1	1	2	0	0	
mean (n)	n.a.	1.08	0.518	1.502	5.646	35.180	232.50	1824.98	
st.dev. (n)	n.a.	0.901	0.4565	0.6636	3.0351	7.5174	66.510	650.883	
R(calc.)	n.a.	2.52	1.278	1.858	8.498	21.049	186.23	1822.47	

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



Determination of calculated theoretical Density @15°C of sample #0984; results in kg/L

lab	method	value	mark	z(targ)	remarks	Meas. density	difference
171	calc.	0.8541		----		0.8550	-0.0009
311	calc.	0.8513		----		0.8552	-0.0039
391	calc.	0.8567		----		0.8554	0.0013
442	calc.	0.8549		----		0.8552	-0.0003
445	calc.	0.8537		----		0.8548	-0.0011
494	calc.	0.8524		----		0.8551	-0.0027
608	calc.	0.8548		----		0.8548	0.0000
862	calc.	0.8510		----		0.8549	-0.0039
1023	calc.	0.8560		----		0.8556	0.0004
1065	calc.	0.8525		----		0.8552	-0.0027
1066	calc.	0.8535		----		0.8552	-0.0017
1089	calc.	0.8534		----		0.8554	-0.0020
1109	calc.	0.8524		----		0.8488	0.0036
1432		----		----	no data available	----	----
1720	calc.	0.8543		----		0.8586	-0.0043
1842		----		----	no data available	----	----
normality		OK					
n		14					
outliers		0					
mean (n)		0.85364	Average:		0.85515	-0.00151	
st.dev. (n)		0.001651					
R(calc.)		0.00462					
R(D5002:05)		0.00352					

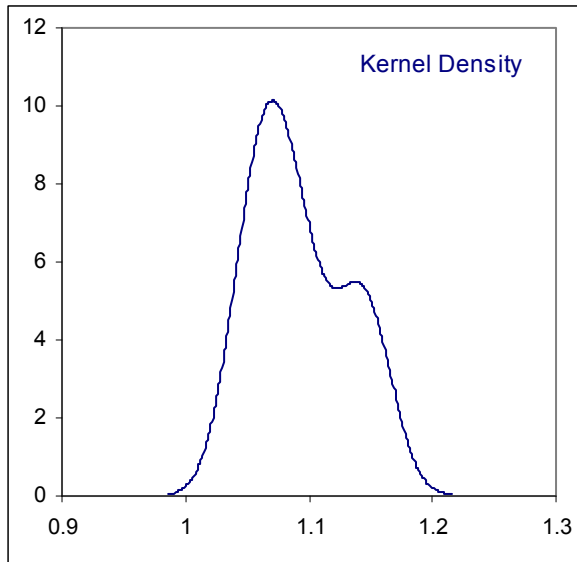
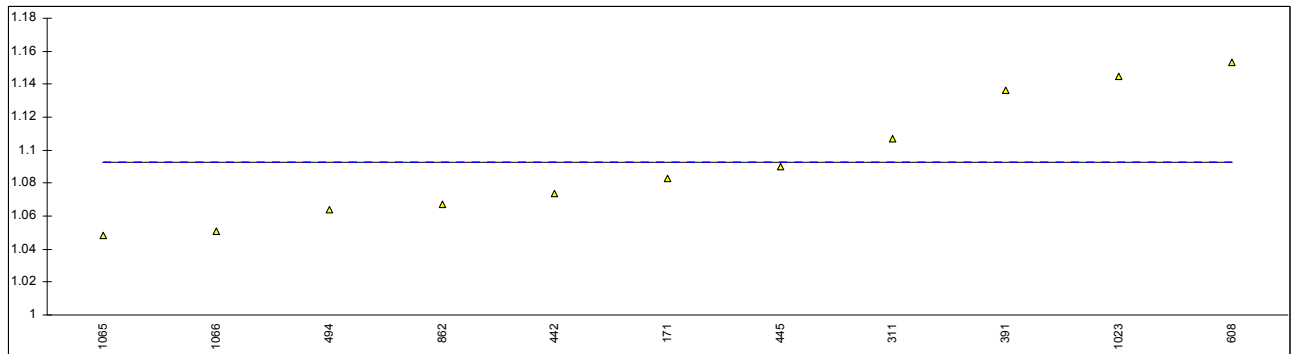


Determination of calculated theoretical sulphur content on sample #0984; results in %M/M

lab	method	value	mark	z(targ)	remarks	Meas. sulphur	difference
171	calc.	1.083		----		1.11	-0.027
311	calc.	1.107		----		1.091	0.016
391	calc.	1.136		----		1.13	0.006
442	calc.	1.074		----		1.047	0.027
445	calc.	1.090		----		1.10	-0.010
494	calc.	1.064		----		1.08	-0.016
608	calc.	1.153		----		1.099	0.054
862	calc.	1.067		----		1.017	0.050
1023	calc.	1.145		----		1.11	0.035
1065	calc.	1.048		----		1.02	0.028
1066	calc.	1.051		----		1.07	-0.019
1089		----		----	no data available	----	----
1109		----		----	not enough data available	1.43	----
1432		----		----	no data available	----	----
1720		----		----	not enough data available	----	----
1842		----		----	no data available	----	----

normality OK
n 11
outliers 0
mean (n) 1.0925
st.dev. (n) 0.03758
R(calc.) 0.1052
R(D4294:03) 0.1395

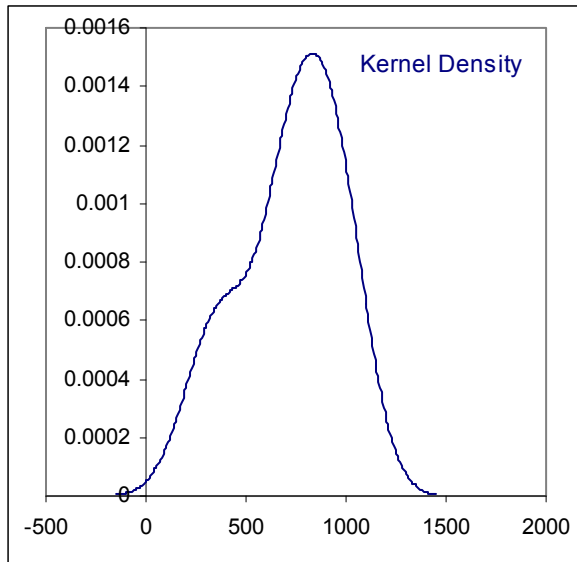
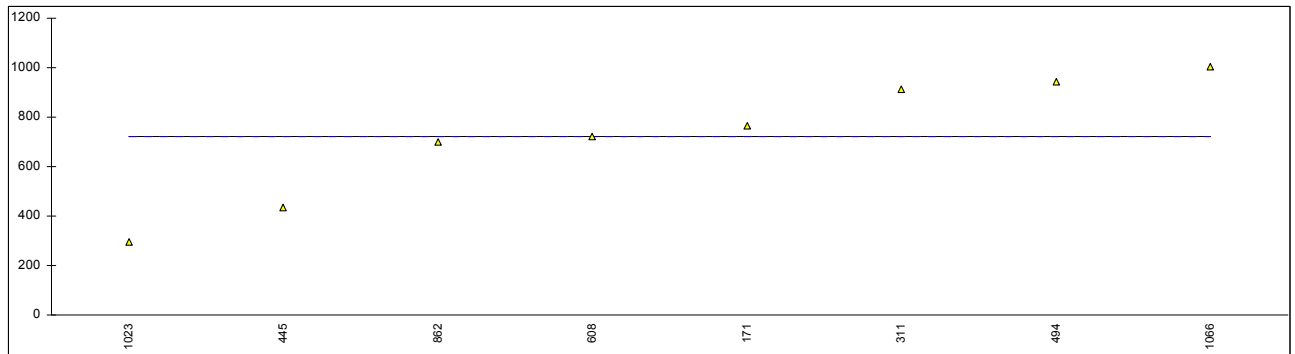
Average: 1.0795 0.0130



Determination of calculated theoretical nitrogen content on sample #0984; results in mg/kg

lab	method	value	mark	z(targ)	remarks	Meas. nitrogen	difference
171	calc.	766		----		873	-107
311	calc.	913		----		910	3
391		----		----	no data available	----	----
442		----		----	no data available	----	----
445	calc.	433		----		865	-432
494	calc.	944		----		1048	-104
608	calc.	722		----		894	-172
862	calc.	699		----		823.4	-124.4
1023	calc.	297		----		325	-28
1065	calc.	----		----	no data available	----	----
1066	calc.	1003		----		1220	-217
1089		----		----	no data available	----	----
1109		----		----	not enough data available	929	----
1432		----		----	no data available	----	----
1720		----		----	not enough data available	----	----
1842		----		----	no data available	----	----

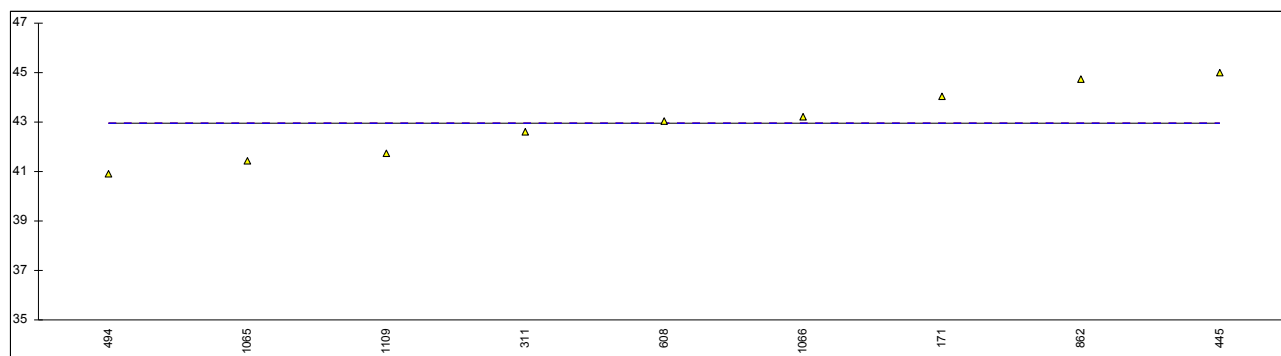
normality OK
 n 8
 outliers 0
 mean (n) 722.1 Average: 906.1
 st.dev. (n) 248.23
 R(calc.) 695.1
 R(D5762) 192.1 difference -184.0



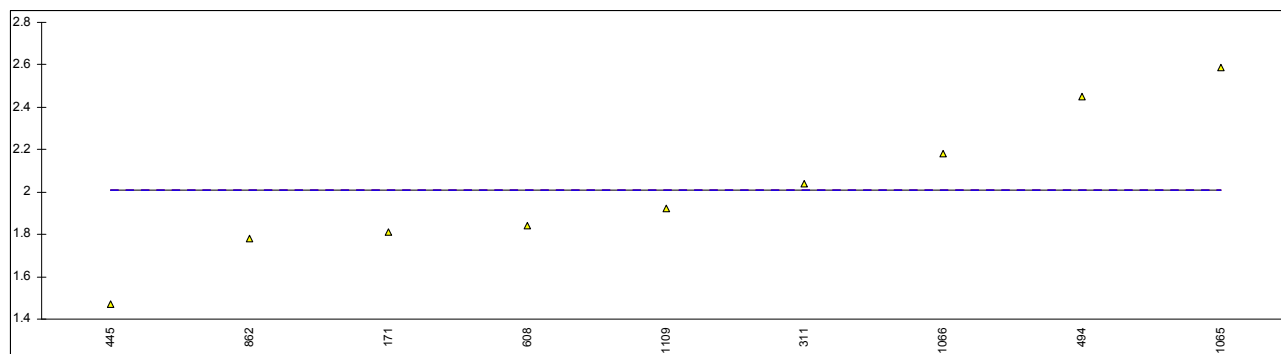
Determination of PNA on distillation fraction 2 (light naphtha); results in %V/V

lab	method	total par*	C1-C4	n-par	i-par	naphth.	arom.	remarks	
171	D5443	85.990	C	4.799	44.028	37.163	16.996	1.813	added C1-C4 to reported total
311	D5443	80.36	C	1.58	42.61	36.17	19.14	2.04	added C1-C4 to reported total
391		----		----	----	----	----		
442		----		----	----	----	----		
445	D5443	86.95		3.33	45.01	38.61	14.91	1.47	
494	D4629	78.39	C	0.51	40.90	36.98	19.67	2.45	added C1-C4 to reported total
608	D6730mod	83.5112	C	1.3232	43.0331	39.1549	15.9699	1.8421	added C1-C4 to reported total
862	D6730	85.748	C	3.419	44.738	37.591	15.547	1.782	added C1-C4 to reported total
1023		----		----	----	----	----		
1065	in house	77.419	C	0.309	41.431	35.679	20.301	2.589	added C1-C4 to reported total
1066	D5443	81.48	C	1.52	43.21	36.75	17.85	2.18	added C1-C4 to reported total
1089		----		----	----	----	----		
1109	D6839	79.62	C	0.29	41.76	37.57	18.64	1.92	added C1-C4 to reported total
1432		----		----	----	----	----		
1720		----		----	----	----	----		
1842		----		----	----	----	----		
	normality	OK	OK	OK	OK	OK	OK		
	n	9	9	9	9	9	9		
	outliers	0	0	0	0	0	0		
	mean (n)	82.163	1.898	42.969	37.296	17.669	2.010		
	st.dev. (n)	3.5206	1.5965	1.4455	1.0979	1.9211	0.3495		
	R(calc.)	9.858	4.470	4.047	3.074	5.379	0.979		

* C1-C4 was included in the total paraffines content



n-paraffines

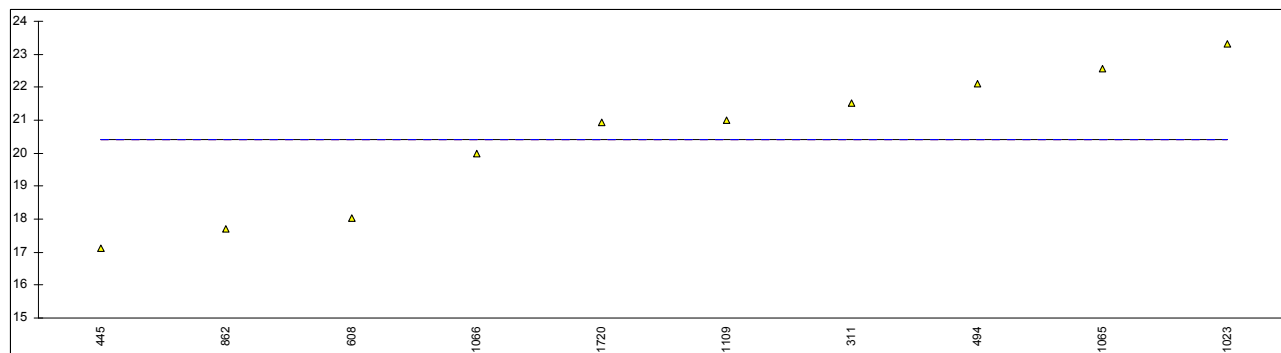


aromatics

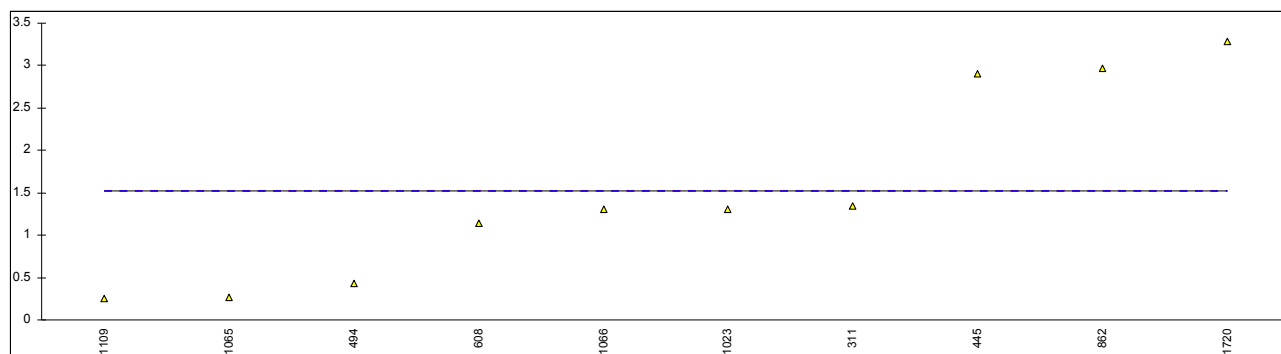
Determination of PNA on distillation fraction 2 (light naphtha); results in %M/M

lab	method	total par*	C1-C4	n-par	i-par	naphth.	arom.	remarks
171		----	----	----	----	----	----	
311	D5443	77.12	1.34	40.70	35.08	21.53	2.63	added C1-C4 to reported total
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445	D5443	83.84	2.90	43.41	37.53	17.12	1.95	
494	D4629	75.16	0.43	39.04	35.69	22.11	3.16	added C1-C4 to reported total
608	D6730mod	80.7076	1.1449	41.4478	38.1149	18.0256	2.4117	added C1-C4 to reported total
862	D6730	82.618	2.969	42.975	36.674	17.695	2.341	added C1-C4 to reported total
1023	D5134	73.832	1.310	39.705	32.817	23.310	2.843	
1065	in house	74.372	0.262	39.584	34.526	22.576	3.314	added C1-C4 to reported total
1066	D5443	78.49	1.31	41.37	35.81	19.99	2.84	added C1-C4 to reported total
1089		----	----	----	----	----	----	
1109	D6839	76.63	0.25	39.95	36.43	21.01	2.50	added C1-C4 to reported total
1432		----	----	----	----	----	----	
1720	D5443	76.70	3.29	----	----	20.92	2.38	
1842		----	----	----	----	----	----	
normality	OK	not OK	OK	OK	OK	OK	OK	
n	10	10	9	9	10	10	10	
outliers	0	0	0	0	0	0	0	
mean (n)	77.947	1.521	40.909	35.852	20.429	2.637		
st.dev. (n)	3.4305	1.1442	1.5284	1.6023	2.1583	0.4097		
R(calc.)	9.605	3.204	4.280	4.487	6.043	1.147		

* C1-C4 was included in the total paraffines content



naphthenes



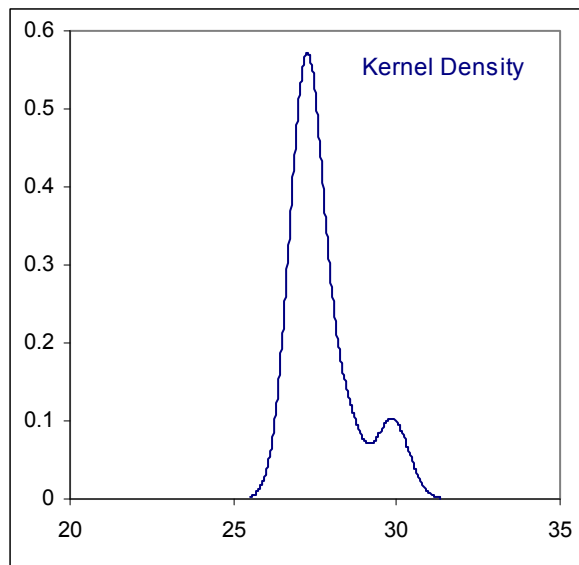
C1-C4

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

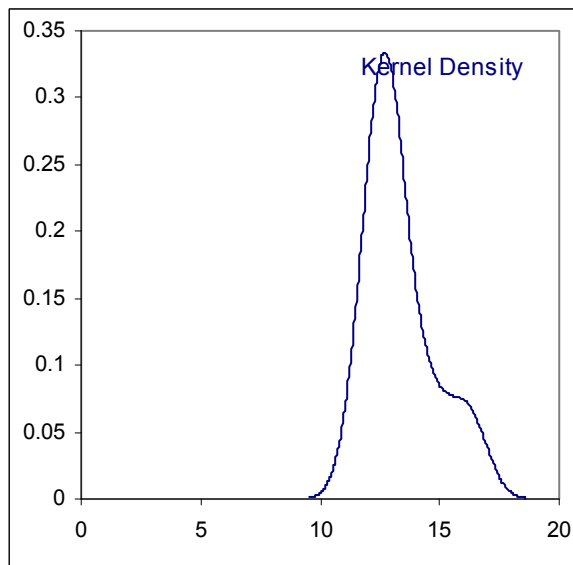
lab	method	total par*	C1-C4	n-par	i-par	naphth.	arom.	remarks
171	D5443	61.329	0.237 ex	<u>28.445</u>	32.884	<u>22.471</u>	<u>16.200</u>	
311	D5443	56.18	0.25 ex	27.33	28.85	31.27	12.56	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445	D5443	57.49	0.17 ex	26.99	30.50	28.85	11.93	
494	D6839	57.11	<0.01 nb	27.49	29.62	29.97	12.92	nb: used 0.005 in stat. calc.
608	D6730mod	58.1659	0.0000	<u>29.8800</u>	28.2859	28.2582	12.4052	
862	D6730	60.033	0.025	27.177	32.856	<u>24.778</u>	13.198	fr. 84.811
1023		----	----	----	----	----	----	
1065	in house	55.986	0.033	27.462	28.524	29.415	14.600	
1066	D5443	56.94	<0.01 nb	27.07	29.87	30.09	<u>12.96</u>	nb: used 0.005 in stat. calc.
1089		----	----	----	----	----	----	
1109		----	----	----	----	----	----	
1432		----	----	----	----	----	----	
1720		----	----	----	----	----	----	
1842		----	----	----	----	----	----	
normality	OK	OK	OK	OK	OK	OK	OK	
n	8	5	6	8	6	6	6	
outliers	0	0	2	0	2	2	2	
mean (n)	57.904	0.014	27.253	30.174	29.642	12.662		
st.dev. (n)	1.8790	0.0145	0.2070	1.8159	1.0538	0.4593		
R(calc.)	5.261	0.041	0.580	5.085	2.951	1.286		

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

* C1-C4 was NOT included in the total paraffines content



n-paraffines



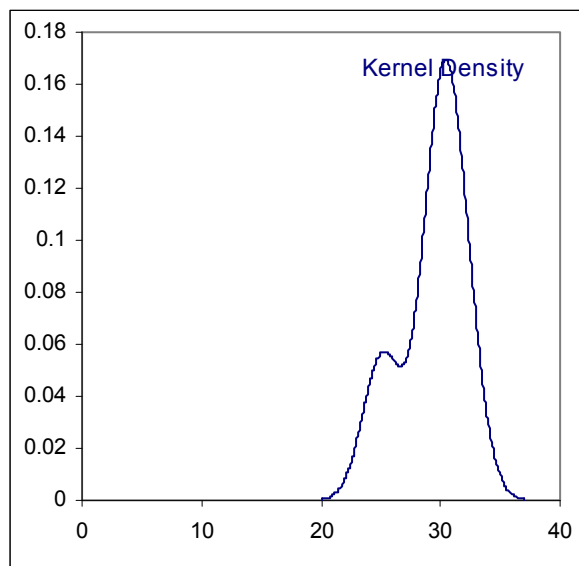
aromatics

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

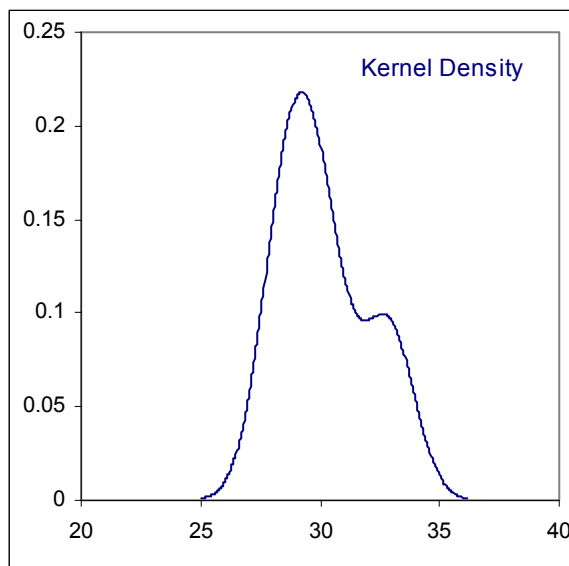
lab	method	total par*	C1-C4	n-par	i-par	naphth.	arom.	remarks
171		----	----	----	----	----	----	
311	D5443	53.04	<u>0.19</u>	25.49	27.55	32.49	14.46	
391		----	----	----	----	----	----	
442		----	----	----	----	----	----	
445	D5443	54.43	<u>0.11</u>	25.17	29.26	29.80	13.75	
494	D6839	54.27	<0.01 nb	25.67	28.60	30.97	14.76	nb: used 0.005 in stat. calc.
608	D6730mod	55.4426	0.0000	<u>28.2586</u>	27.1840	29.0447	14.4159	
862	D6730	57.082	0.019	25.630	31.452	<u>25.673</u>	15.324	fr. 82.755
1023	D5134	<u>42.335</u>	0.014	<u>27.599</u>	<u>14.722</u>	<u>24.606</u>	<u>10.004</u>	
1065	in house	52.706	0.026	25.707	26.999	30.427	16.867	
1066	D5443	53.83	<0.01 nb	25.41	28.42	31.19	14.98	nb: used 0.005 in stat. calc.
1089		----	----	----	----	----	----	
1109		----	----	----	----	----	----	
1432		----	----	----	----	----	----	
1720	D5443	56.62	0.04	----	----	30.03	13.35	
1842		----	----	----	----	----	----	
normality	OK	OK	OK	OK	OK	OK	OK	
n	8	7	6	7	7	8		
outliers	1	2	2	1	2	1		
mean (n)	54.678	0.016	25.513	28.495	30.565	14.738		
st.dev. (n)	1.5881	0.0141	0.2024	1.5382	1.1152	1.0703		
R(calc.)	4.447	0.039	0.567	4.307	3.123	2.997		

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

* C1-C4 was NOT included in the total paraffines content



Naphthenes



i-paraffines

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

Lab method	IBP	5% evap	10% evap	50% evap	90% evap	95% evap	FBP	remarks
171 D86	184.5	194.6	197	211	227.7	232	242.7	
311 D86	196.9	202.2	204.1	215.0	233.4	238.0	254.9	
391 D86	191.0	197.4	202.2	211.8	229.4	233.2	242.0	
442	----	----	----	----	----	----	----	
445 D86	185.9	192.9	195.9	209.4	227.2	232.6	241.6	
494 D86	194.1	203.6	205.3	218.5	239.9	245.4	253.3	
608 D86	180.50	190.00	194.25	209.80	231.25	236.25	241.50	
862 D86	191.7	200.6	201.9	213.4	233.2	237.9	251.6	
1023 D86	194	----	----	----	----	----	248	
1065 D86	189.9	198.9	200.0	211.1	228.7	232.9	243.2	
1066 D86	195.3	199.3	200.7	210.1	231.4	238.4	C 249.7	fr 249.7
1089	----	----	----	----	----	----	----	
1109 D86	146.8	181.9	189.8	211.8	230.2	234.5	240.5	
1432	----	----	----	----	----	----	----	
1720 D86	181.0	192.5	197.0	211.5	228.5	237.5	246.0	
1842	----	----	----	----	----	----	----	
normality	OK	OK	OK	OK	OK	OK	OK	
n	11	11	11	10	10	10	12	
outliers	1	0	0	1	1	1	0	
mean (n)	189.53	195.81	198.92	211.49	230.10	235.33	246.25	
st.dev. (n)	5.730	6.300	4.615	1.690	2.178	2.547	5.094	
R(calc.)	16.04	17.64	12.92	4.73	6.10	7.13	14.26	

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

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

Lab method	% rec.	5% rec	10% rec	50% rec	90% rec	95% rec	% res.	remarks
171	----	----	----	----	----	----	----	
311 D86	99.1	202.2	204.1	215.0	233.4	238.0	0.9	
391 D86	98	199.1	203.2	212.0	230.2	234.1	1	
442	----	----	----	----	----	----	----	
445 D86	99.0	185.9	192.9	209.4	227.2	232.6	1.0	
494 D86	99.1	203.7	205.3	218.6	240.0	245.5	0.8	
608 D86	98.5	190.50	194.50	210.00	231.50	236.50	1.1	
862 D86	99.3	200.6	201.9	213.4	233.2	237.9	0.7	
1023 D86	99	201	203	215	234	240	0.5	
1065 D86	98.8	199.1	199.9	211.2	229.0	233.1	1.0	
1066 D86	97.5	199.9	200.9	210.5	232.6	241.3	1.3	
1089	----	----	----	----	----	----	----	
1109 D86	98.3	182.9	190.6	212.0	230.6	235.0	1.2	
1432	----	----	----	----	----	----	----	
1720 D86	98.5	193.0	198.0	212.5	231.5	238.0	1.2	
1842	----	----	----	----	----	----	----	
normality		not OK	OK	OK	OK	OK		
n		11	11	11	10	11		
outliers		0	0	0	1	0		
mean (n)		196.17	199.48	212.69	231.32	237.45		
st.dev. (n)		7.006	4.885	2.686	2.131	3.841		
R(calc.)		19.62	13.68	7.52	5.97	10.75		

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

Results of Simdist on distillation fraction 4 (Kerosene); yields of fractions in %M/M.

Lab	190-198°C	198-206°C	206-214°C	214-222°C	222-230°C	230-238°C	238-246°C	remarks
171	18.5	28.0	14.0	8.0	2.0	0.0	0.0	
311	24.0	17.0	15.0	13.0	2.5	0.5	0.0	
391	26.0	17.0	18.0	12.5	1.5	0.1	0.0	
442	----	----	----	----	----	----	----	
445	11.0	20.3	15.8	14.3	<u>9.7</u>	1.5	0.0	
494	----	----	----	----	----	----	----	
608	20.0	10.0	12.0	18.0	4.5	3.0	<u>0.5</u>	
862	8.0	18.0	20.0	16.0	<u>7.0</u>	2.5	<u>0.5</u>	
1023	----	----	----	----	----	----	----	
1065	15.0	28.0	9.5	8.5	2.5	0.5	0.1	
1066	17.7	28.0	13.0	9.0	3.0	1.5	0.2	
1089	----	----	----	----	----	----	----	
1109	----	----	----	----	----	----	----	
1432	----	----	----	----	----	----	----	
1720	----	----	----	----	----	----	----	
1842	----	----	----	----	----	----	----	
normality	OK	OK	OK	OK	OK	OK	not OK	
n	8	8	8	8	6	8	6	
outliers	0	0	0	0	2	0	2	
mean (n)	17.53	20.79	14.66	12.41	2.67	1.20	0.05	
st.dev. (n)	6.101	6.645	3.338	3.673	1.033	1.117	0.084	
R(calc.)	17.08	18.61	9.35	10.28	2.89	3.13	0.23	

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

Results of Simdist on distillation fraction 5 (light gasoil); yields of fractions in %M/M.

Lab	190-198°C	198-206°C	206-214°C	214-222°C	222-230°C	230-238°C	238-246°C	remarks
171	1.5	3.0	7.5	20.0	15.0	22.0	18.0	
311	0.9	2.0	5.1	13.5	15.5	27.0	13.0	
391	0.1	1.5	4.0	15.0	15.0	29.0	15.0	
442	----	----	----	----	----	----	----	
445	1.4	2.8	5.0	11.6	14.1	18.2	20.0	
494	----	----	----	----	----	----	----	
608	<u>3.5</u>	2.0	4.0	11.0	<u>9.0</u>	24.0	11.0	
862	1.0	3.5	4.5	10.5	14.0	20.0	20.5	
1023	----	----	----	----	----	----	----	
1065	1.5	4.0	7.0	20.0	17.0	18.0	18.0	
1066	1.8	4.5	8.0	17.4	13.2	15.4	16.0	
1089	----	----	----	----	----	----	----	
1109	----	----	----	----	----	----	----	
1432	----	----	----	----	----	----	----	
1720	----	----	----	----	----	----	----	
1842	----	----	----	----	----	----	----	
normality	OK	OK	OK	OK	OK	OK	OK	
n	7	8	8	8	7	8	8	
outliers	1	0	0	0	1	0	0	
mean (n)	1.17	2.91	5.64	14.88	14.83	21.70	16.44	
st.dev. (n)	0.565	1.051	1.615	3.886	1.231	4.710	3.332	
R(calc.)	1.58	2.94	4.52	10.88	3.45	13.19	9.330	

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

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

Lab	Overlap cuts 4 and 5 in °C	ECP in °C	Difference with AET=215 °C	Conclusion*	remarks
171	196 - 224	211.3	-3.67	OK	
311	199 - 227	214.1	-0.85	OK	
391	205 - 224	215.1	+0.05	OK	
442	----	----	----	----	
445	191 – 232	216.8	+1.82	OK	
494	----	----	----	----	
608	184 – 237	216.6	+1.58	OK	
862	190 – 240	217.3	+2.33	OK	
1023	----	----	----	----	
1065	194 - 224	211.1	-3.94	OK	
1066	196 - 232	211.6	-3.39	OK	
1089	----	----	----	----	
1109	----	----	----	----	
1432	----	----	----	----	
1720	----	----	----	----	
1842	----	----	----	----	

* Acc. To ASTM D2892:05, 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

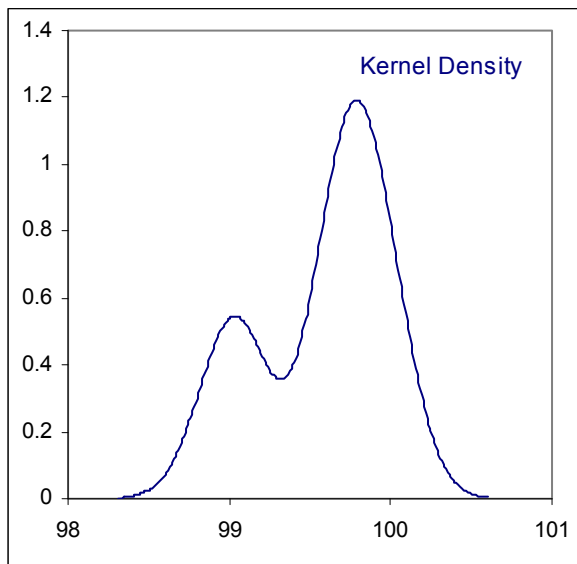
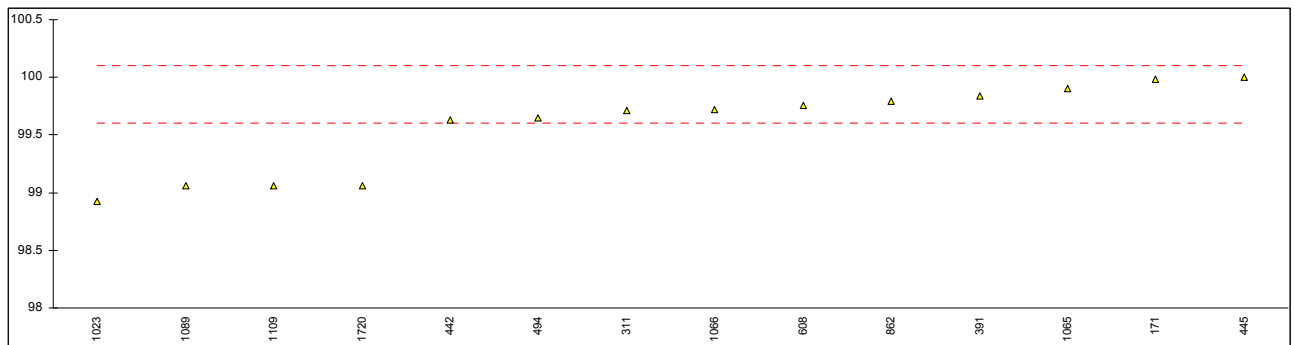
Lab	N_{actual}	N_{minimum}	Requirement $5.4 < N_{\text{minimum}} < 6.8^*$	remarks
171	6.9	6.9	not OK	
311	6.8	6.8	OK	
391	8.1	8.1	not OK	
442	----	----	----	
445	6.7	6.7	OK	
494	----	----	----	
608	5.4	5.4	OK	
862	5.5	5.5	OK	
1023	----	----	----	
1065	6.2	6.1	OK	
1066	5.4	5.3	not OK	
1089	----	----	----	
1109	----	----	----	
1432	----	----	----	
1720	----	----	----	
1842	----	----	----	

* Acc. To ASTM D2892:05, Appendix X2.5.9.2 the acceptable upper and lower limits (14-18 theoretical plates) are 5.4, resp. 6.8

Total Mass balance/Total Mass recovery of D2892, results in %M/M

lab	method	value	mark	remarks
171	calc.	99.98		
311	calc.	99.71		
391	calc.	99.84		
442	calc.	99.63		
445	calc.	100.00		
494	calc.	99.65		
608	calc.	99.76		
862	calc.	99.79		
1023	calc.	98.92		
1065	calc.	99.90		
1066	calc.	99.72		
1089	calc.	99.06		
1109	calc.	99.06		
1432		----		
1720	calc.	99.06		
1842		----		
normality		not OK		
n		14		
outliers		0		
mean (n)		99.578		
st.dev. (n)		0.3795		
R(calc.)		1.063		

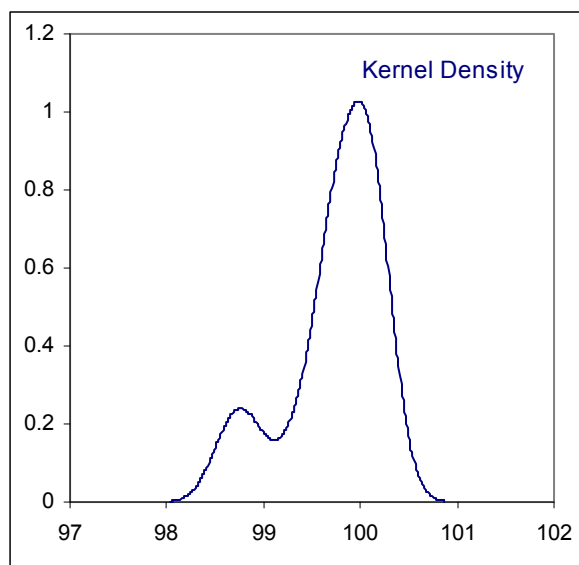
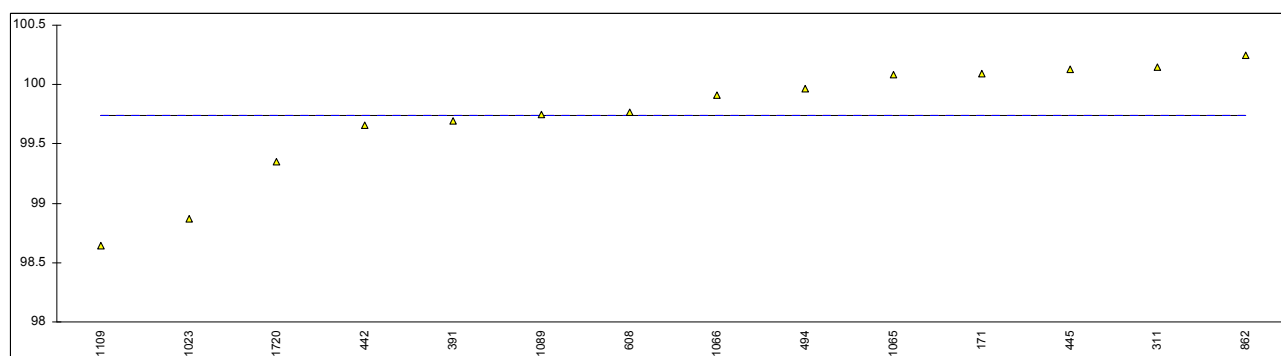
In below graph, the dotted lines represent the ASTM D5236 requirements for recovery: $99.6\% < \text{recovery} < 100.1\%$, of which the lower line at 99.6% is equal to the 0.4% max. loss requirement of ASTM D2892, paragraph 11.2.



Total Volume balance/Total Volume recovery of D2892, results in %V/V

lab	method	value	mark	remarks
171	calc.	100.09		
311	calc.	100.15		
391	calc.	99.69		< 99.84, see paragraph 4.1
442	calc.	99.66		
445	calc.	100.13		
494	calc.	99.97		
608	calc.	99.77		
862	calc.	100.25		
1023	calc.	98.87		< 98.92, see paragraph 4.1
1065	calc.	100.08		
1066	calc.	99.91		
1089	calc.	99.75		
1109	calc.	98.64		< 99.06, see paragraph 4.1
1432		----		
1720	calc.	99.35		
1842		----		

normality OK
n 14
outliers 0
mean (n) 99.736
st.dev. (n) 0.4827
R(calc.) 1.352



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

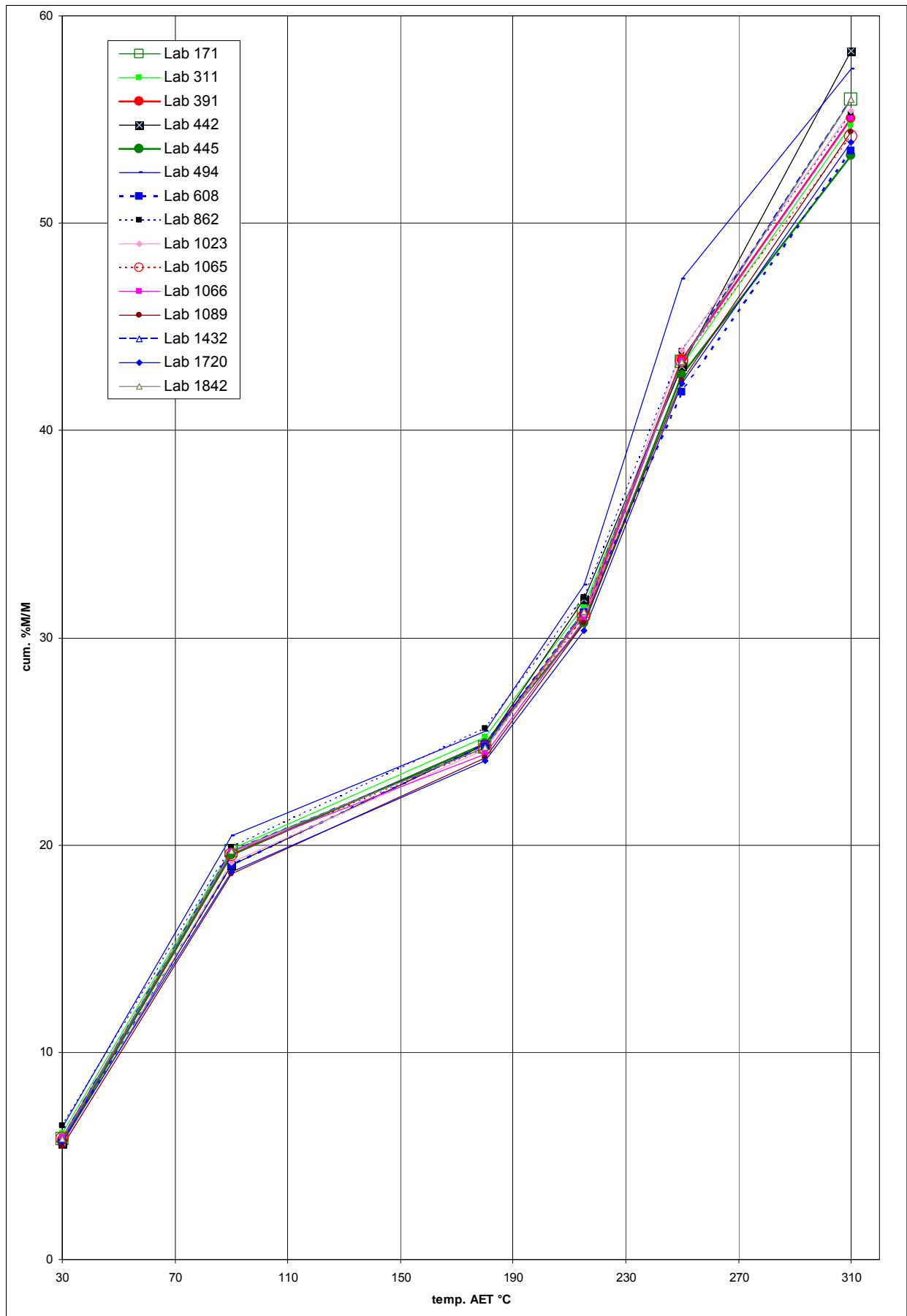
	Gas LPG	L. Naphtha	H. Naphtha	Kerosene	LGO	MGO	HGO	Residue
lab	<30°C	30-90°C	90-180°C	180-215°C	215-250°C	250-310°C	310-370°C	>370°C
171	0.8994	4.9151	13.8997	4.9822	6.5746	12.0608	12.6234	44.0287
311	1.8895	4.2084	13.7131	5.3822	6.3556	11.5660	11.5946	45.0043
391	1.5106	4.2486	13.7488	5.3887	6.2438	12.2470	11.6752	44.7805
442	1.9414	3.6542	13.4329	5.8223	6.9865	11.2667	<u>15.1869</u>	41.3343
445	1.0555	4.6417	13.8504	5.3501	5.8060	11.9649	10.6102	46.7212
494	2.0527	4.3310	14.0627	5.0364	<u>7.0603</u>	<u>14.7681</u>	10.1307	42.2112
608	0.6638	4.9668	13.3943	5.8023	6.3666	10.6437	11.6565	46.2701
862	1.3088	5.1378	13.4640	5.6955	6.3270	11.8989	11.4694	44.4850
1023	0.9347	4.6025	13.6339	5.4622	6.5073	12.7228	11.5397	43.5124
1065	1.5892	4.2379	13.6876	5.1606	6.3226	12.3718	10.7997	45.7280
1066	1.5255	4.4036	13.7222	4.7178	6.4771	12.4969	11.6993	44.6766
1089	0.9063	4.5517	13.1434	5.6119	6.4981	11.6953	12.0130	44.6440
1109	1.4290	4.2823	13.2098	6.1395	6.2000	12.2091	9.6397	45.9551
1432	----	----	----	----	----	----	----	----
1720	1.0045	4.6803	12.9959	5.3911	6.2785	11.9259	11.6082	45.1783
1842	----	----	----	----	----	----	----	----
normality	OK	OK	OK	OK	OK	OK	not OK	OK
n	14	14	14	14	13	13	13	14
outliers	0	0	0	0	1	1	1	0
mean (n)	1.33649	4.49013	13.56847	5.42448	6.38028	11.92846	11.31227	44.60926
st.dev. (n)	0.438808	0.380185	0.307070	0.376942	0.265047	0.549610	0.806377	1.489151
R(calc.)	1.22866	1.06452	0.85980	1.05544	0.74213	1.53891	2.25786	4.16962
R(D2892:05)	1.2	1.2	1.2	1.2	1.4	1.4	1.4	n.a.

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

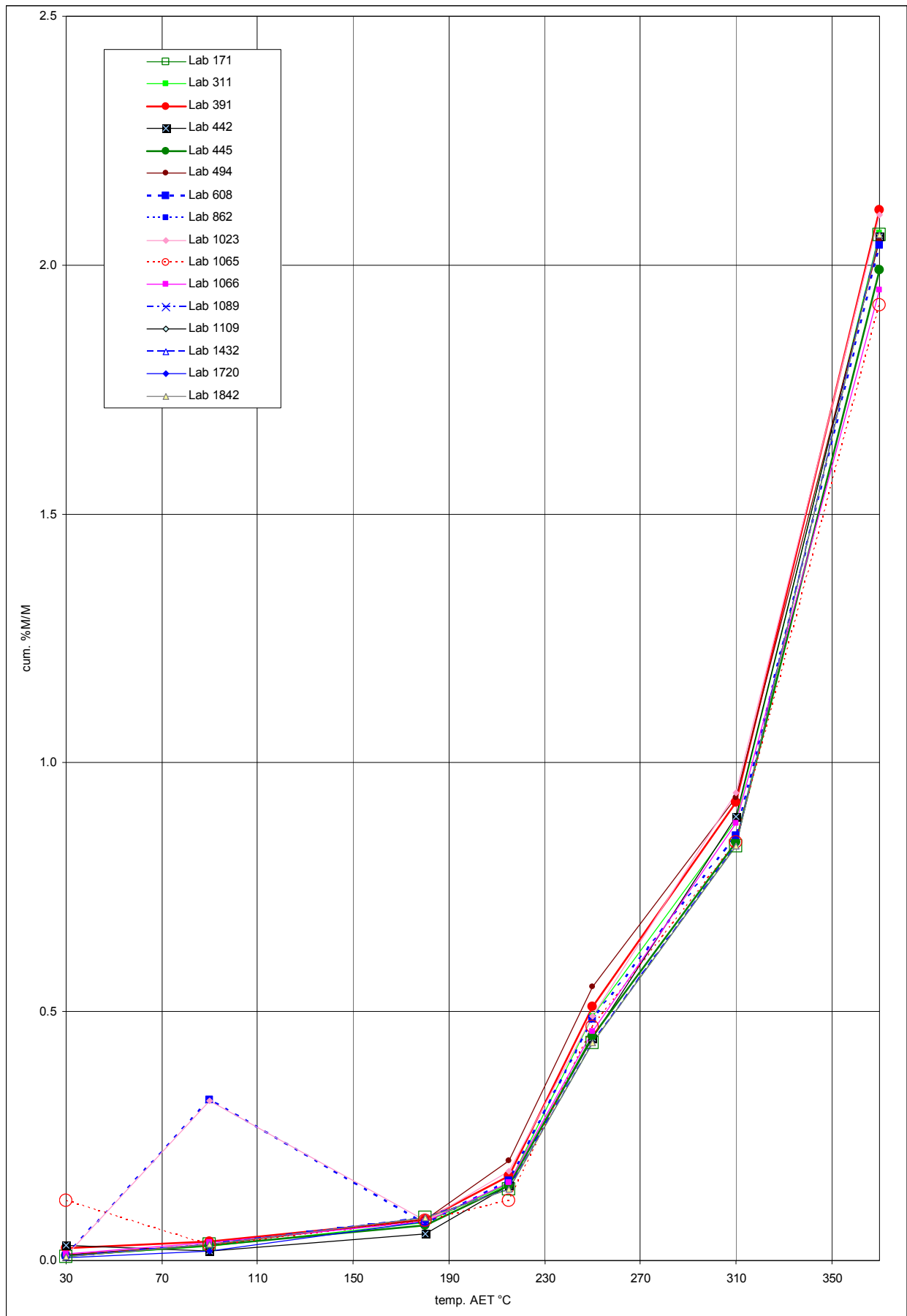
Corresponding z-scores for above results

	Gas LPG	L. Naphtha	H. Naphtha	Kerosene	LGO	MGO	HGO	Residue
lab	<30°C	30-90°C	90-180°C	180-215°C	215-250°C	250-310°C	310-370°C	>370°C
171	-1.02	0.99	0.77	-1.03	0.39	0.26	2.62	----
311	1.29	-0.66	0.34	-0.10	-0.05	-0.72	0.56	----
391	0.41	-0.56	0.42	-0.08	-0.27	0.64	0.73	----
442	1.41	-1.95	-0.32	0.93	1.21	-1.32	7.75	----
445	-0.66	0.35	0.66	-0.17	-1.15	0.07	-1.40	----
494	1.67	-0.37	1.15	-0.91	1.36	5.68	-2.36	----
608	-1.57	1.11	-0.41	0.88	-0.03	-2.57	0.69	----
862	-0.06	1.51	-0.24	0.63	-0.11	-0.06	0.31	----
1023	-0.94	0.26	0.15	0.09	0.25	1.59	0.45	----
1065	0.59	-0.59	0.28	-0.62	-0.12	0.89	-1.03	----
1066	0.44	-0.20	0.36	-1.65	0.19	1.14	0.77	----
1089	-1.00	0.14	-0.99	0.44	0.24	-0.47	1.40	----
1109	0.22	-0.49	-0.84	1.67	-0.36	0.56	-3.35	----
1432	----	----	----	----	----	----	----	----
1720	-0.77	0.44	-1.34	-0.08	-0.20	-0.01	0.59	----
1842	----	----	----	----	----	----	----	----

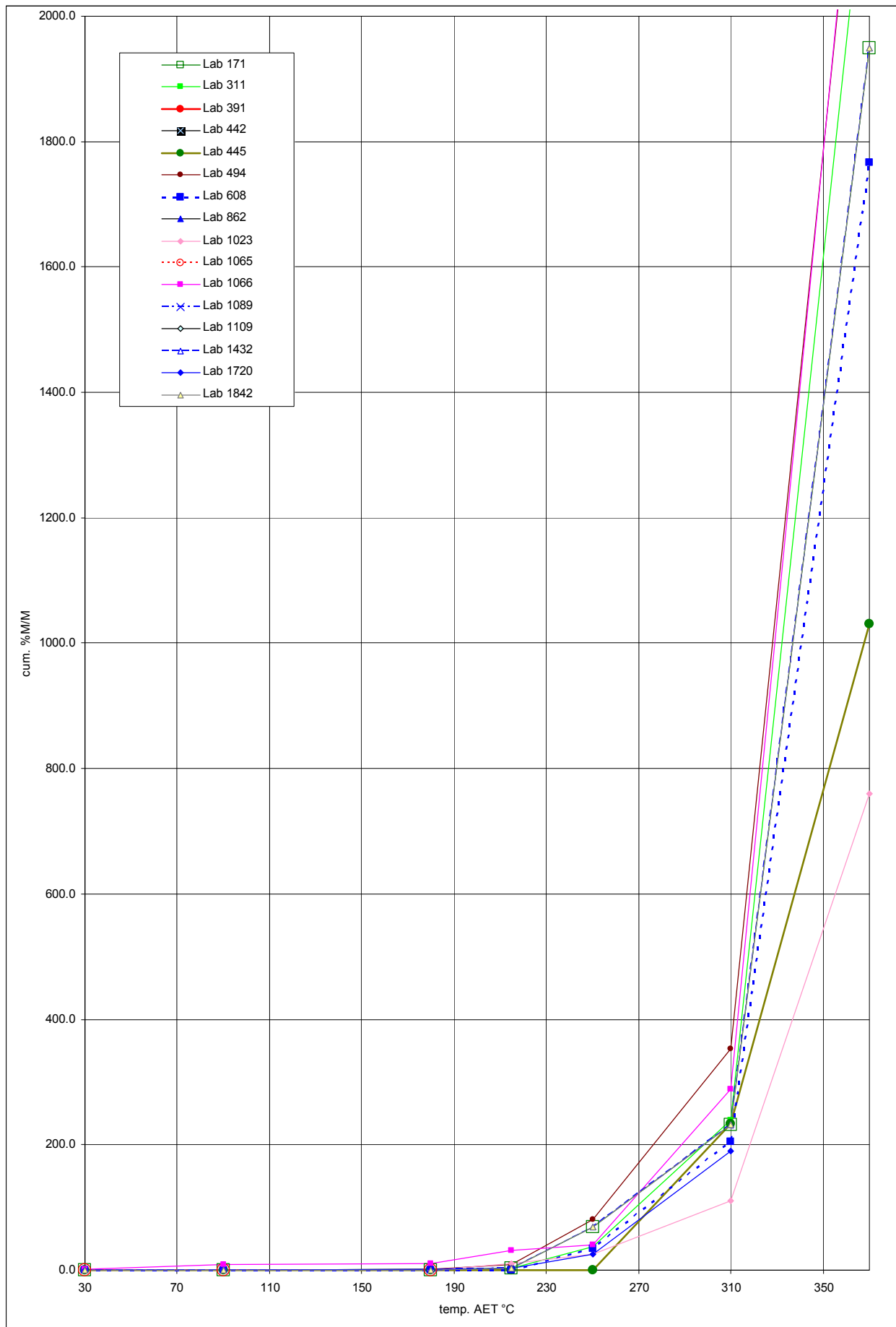
APPENDIX 2 True boiling point curve D2892, cum%M/M vs temp AET in °C



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



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



APPENDIX 5**Details of Distillation**

	Sample received	Distillation started	Results reported	Intake in liters	Remarks
171	28-Oct-2009	9-Nov-2009	12-Nov-2009	4.4	no water detected
311	16-Oct-2009	23-Dec-2009	4-Jan-2010	4.1	
391	22-Oct-2009	2-Nov-2009	13-Nov-2009	6.6	
442	23-Oct-2009	11-Nov-2009	13-Nov-2009	2.4	
445	22-Oct-2009	29-Oct-2009	27-Nov-2009	10	
494	20-Oct-2009	7-Dec-2009	21-Dec-2009	2	
608	26-Oct-2009	5-Nov-2009	10-Nov-2009	8.6	
862	4-Nov-2009	17-Nov-2009	24-Nov-2009	6	
1023	29-Oct-2009	10-Nov-2009	18-Nov-2009	3	
1065	21-Oct-2009	26-Oct-2009	12-Nov-2009	6.9	
1066	22-Oct-2009	13-Nov-2009	18-Dec-2009	6.7	
1089	23-Oct-2009	16-Nov-2009	27-Nov-2009	3.5	hold up (20 g) not incl.; normally added to residue.
1109	30-Oct-2009	5-Nov-2009	20-Nov-2009	2.5	did not reach 370°C, due to pot temp of 320°C
1432					
1720	5-Nov-2009	11-Nov-2009	15-Nov-2009	9.6	
1842					

APPENDIX 6

List of participants

- 1 laboratory in AUSTRALIA
- 1 laboratory in BELARUS REPUBLIC
- 1 laboratory in GERMANY
- 1 laboratory in ISRAEL
- 1 laboratory in ITALY
- 1 laboratory in MALAYSIA
- 2 laboratories in NORWAY
- 1 laboratory in P.R. of CHINA
- 1 laboratory in SUDAN
- 2 laboratories in THE NETHERLANDS
- 1 laboratory in U.S.A.
- 3 laboratories in UNITED KINGDOM

APPENDIX 7

Abbreviations:

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

Literature:

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