Results of Proficiency Test
Cyclohexane
March 2018

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

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1 INTRODUCTION

At the request of several participants, the Institute of Interlaboratory Studies decided to organise an interlaboratory study for Cyclohexane in the 2017/2018 PT program.
In the interlaboratory study 11 laboratories in 9 different countries did register for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2018 proficiency test for Cyclohexane are presented and discussed. This report is also 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 tests (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample of Cyclohexane (1 litre bottle, labelled #18300). The participants were requested to report rounded and unrounded test results. The unrounded test 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/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary bulk material of Cyclohexane, approximately 40 litre was purchased from a local chemical supplier. From this batch, after homogenisation, 24 brown glass bottles of 1 litre were filled and labelled #18300. The homogeneity of the subsamples #18300 was checked by determination of Density at 20°C, according to ISO12185 on 4 stratified randomly selected samples.

Cyclohexane	Density at 20°C in kg/L
sample #18300-1	0.77852
sample #18300-2	0.77852
sample #18300-3	0.77852
sample #18300-4	0.77852

Table 1: homogeneity test results of Cyclohexane subsamples #18300

From the above test results the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference test method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 20°C in kg/L
r (observed)	0.00000
reference test method	ISO12185:96
0.3*R (reference test method)	0.00015

Table 2: evaluation of repeatability of subsamples #18300

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

To each of the participating laboratories one litre bottle of Cyclohexane, labelled #18300, was sent on February 14, 2018. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Cyclohexane, packed in amber glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine on the Cyclohexane sample #18300: Acid Wash Color, Appearance, Color Pt/Co, Density at 20°C, Distillation (IBP, 50% recovered, DP), Freezing Point, Purity, Benzene, n-Hexane, Methylcyclohexane, Methylcyclopentane, Refractive Index at 25°C and Sulphur.

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 calculations.

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

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and the original test results are placed under ‘Remarks’ in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation. First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’.

After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

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

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

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 z-scores

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

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

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

The z-scores were calculated according to:

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

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

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

	$\lvert z \rvert$	< 1	good
1 <	$\lvert z \rvert$	< 2	satisfactory
2 <	$\lvert z \rvert$	< 3	questionable
3 <	$\lvert z \rvert$		unsatisfactory

4 Evaluation

In this interlaboratory study, no problems were encountered with dispatch of the samples. Two participants reported the test results after the final reporting date and one other laboratory did not report any test results. Not all laboratories were able to report all analyses requested.
Finally, in total 120 numerical test results were reported by 10 participants. Observed were 10 outlying results, which is 8.3% of the total of numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D7266) and an added designation for the year that the method was adopted or revised (e.g. D7266:13e1). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D7266:13e1(2018)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D7266:13e1 will be used.

Acid Wash Color: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D848:14.
For the statistical analysis, a result expressed as y- or y+ was changed into a numerical value as follows: y- changed into y-0.25 and y+ into y+0.25.

Appearance: No analytical problems were observed. All labs agreed about the appearance of the sample, which was bright, clear and free of suspended matter (Pass).

Color Pt/Co: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5386:16 and of ASTM D1209:05(2011).

Density at 20°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. In total four statistical outliers were observed and two other test results were excluded. However, all calculated reproducibilities after rejection of the suspect data are in agreement with the requirements of ASTM D850-Automated:16.
From the reported test results of the 50% recovered, it appears that two participants probably did not correct the test results for barometric pressure and thermometer inaccuracy as described in ASTM D850 (paragraph 11). One of these two laboratories also reported a distillation range of more than 2°C, therefore the other non-outlying test results were excluded.

Freezing Point: This determination may not be problematic. Only three participants reported a test result, of which one was excluded as the reported test method was a test method for Jet Fuel. The other two participants agreed on a Freezing Point of 5.90°C. Due to the low number of test results no z-scores were calculated.

Purity: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7266:13e1(2018).

Benzene: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier was in agreement with the requirements of ASTM D7266:13e1(2018).

n-Hexane: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of D7266:13e1(2018).

Methylcyclohexane: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of D7266:13e1(2018).

Metylcyclopentane: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of D7266:13e1 (2018).

Refractive Index: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1218:12(2016).

Sulphur: Six participants reported a test result and agreed on a value for Sulphur smaller than 1 mg/kg.

UV Absorbance: This determination may be problematic. Five participants reported a test result. The test results for 260 and 240 nm were larger than one and were not evaluated. The determination at 280 nm may be problematic. One statistical outlier was observed. Since no method or precision data is available, no z-scores were calculcated. The calculated reproducibility of 0.026 at 0.433 Absorbance is similar to or better than the calculated reproducibility of for example Methanol (iis17C09, Rcalc = 0.068 at 0.514 Absorbance) or Ethanol (iis17C16, Rcalc = 0.077 at 0.490 Absorbance).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 *sd	R (lit.)
Acid Wash Color		7	0.8 (1-)	0.3	2.1
Appearance		10	Pass	n.a.	n.a.
Color Pt/Co		7	3.7	2.8	5.2
Density at 20°C	kg/L	8	0.7786	0.0002	0.0005
Distillation, IBP	°C	6	80.3	0.1	0.4
Distillation, 50% rec.	°C	6	80.7	0.1	0.4
Distillation, DP	°C	7	81.5	0.4	0.4
Freezing Point	°C	2	5.9	n.a.	n.a.
Purity	%M/M	9	99.810	0.083	0.123
Benzene	mg/kg	7	8	2	7
n-Hexane	mg/kg	8	138	17	10
Methylcyclohexane	mg/kg	9	101	30	36
Methylcyclopentane	mg/kg	7	96	24	12
Refractive Index at 25°C		8	1.4264	0.0004	0.0005
Sulphur	mg/kg	6	<1	n.a.	n.a.
UV Absorbance at 280 nm		3	0.433	0.026	n.a.

Table 3: reproducibilities on sample #18300

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

Unfortunately, not all laboratories performed all tests, resulting in a low number of results for some tests. Hopefully in the next PT more test results will be reported.

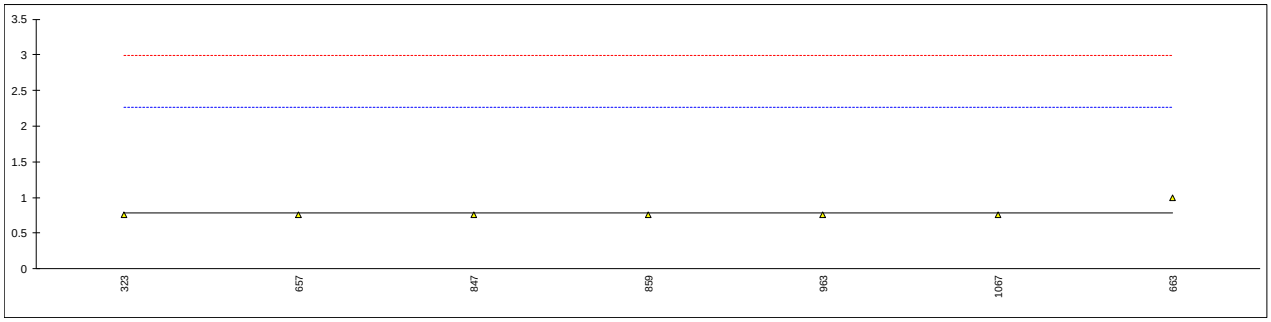
APPENDIX 1

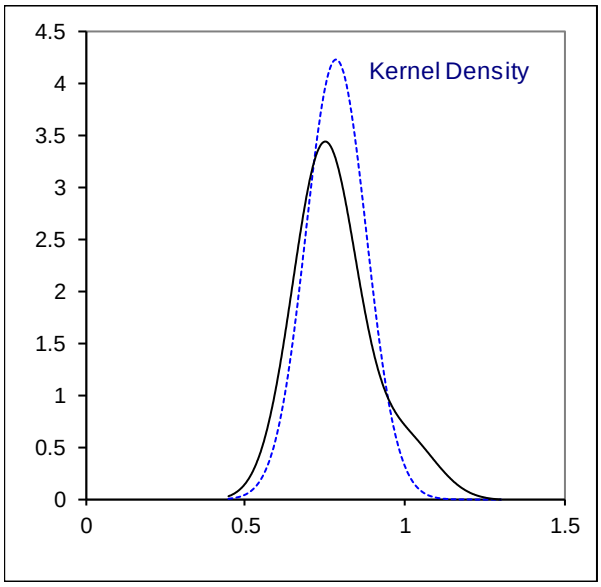
Determination of Acid Wash Color (acid layer) on sample #18300

lab	method	value	mark	z(targ)	remarks
171	D848	pass		-----	
311		n		-----	
323	D848	-1		-0.05	
490		n		-----	
657	D848	1-		-0.05	
663	D848	No.1		0.29	
847	D848	No.1-		-0.05	
859	D848	NO.1_		-0.05	
963	D848	1-		-0.05	
1067	D848	1-		-0.05	
1669		n		-----	

normality	unknown
n	7
outliers	0
mean (n)	0.79 (1-)
st.dev. (n)	0.094
R(calc.)	0.26
st.dev.(D848:14)	0.736
R(D848:14)	2.06

*) In the calculation of the mean, standard deviation, reproducibility and in the graphs, a reported value of 'y-', '-y' or '<y' is changed into y-0.25 (for example 1- into 0.75) and 'y+' is changed into y+0.25 (for example 0+ into 0.25).



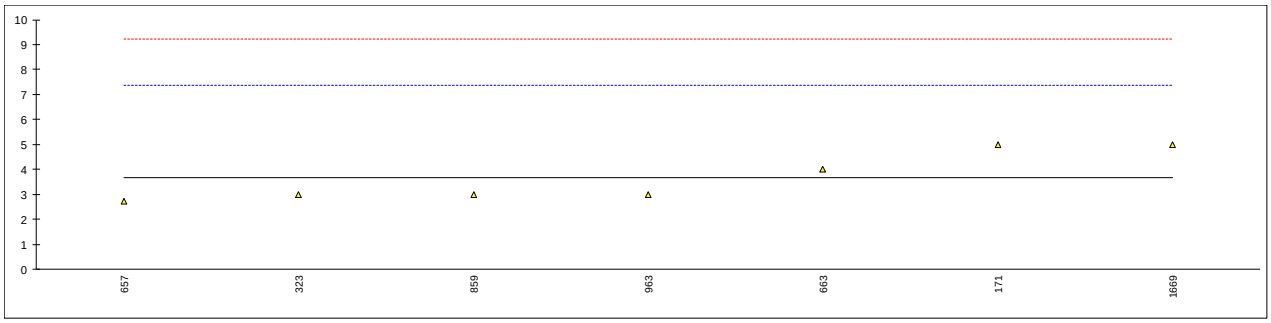


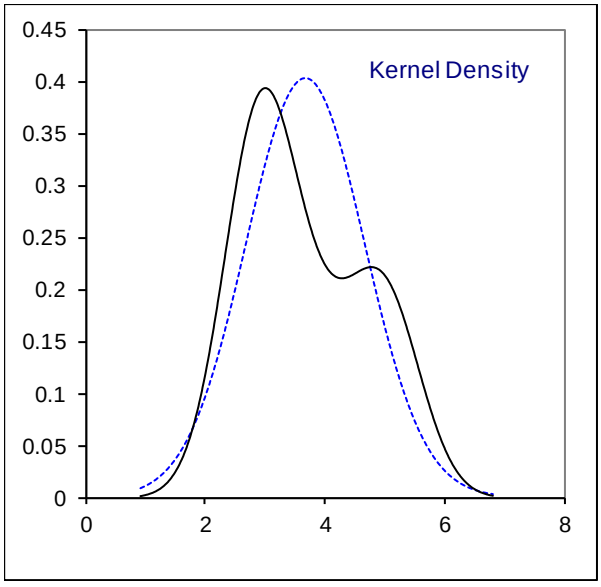
Determination of Appearance on sample #18300

lab	method	value	mark	z(targ)	remarks
171	E2680	Clear and Free		-----	
311	D4176	pass		-----	
323	E2680	pass		-----	
490		-----		-----	
657	E2680	Pass		-----	
663	E2680	Free of haze, particulates or suspended matter		-----	
847	Visual	clear&bright		-----	
859	E2680	Pass		-----	
963	E2680	Pass		-----	
1067	Visual	Clear and Bright		-----	
1669	Visual	Claro y Brillante		-----	
	n	10			
	mean (n)	Pass (B&C)			

Determination of Color (Pt/Co scale) on sample #18300

lab	method	value	mark	z(targ)	remarks
171	D1209	5		0.72	
311	D1209	<5		-----	
323	D5386	3		-0.37	
490		-----		-----	
657	D5386	2.73		-0.51	
663	D5386	4		0.18	
847	D1209	<5		-----	
859	D5386	3		-0.37	
963	D1209	3		-0.37	
1067	D1209	< 5		-----	
1669	D1209	5		0.72	
	normality	unknown			
	n	7			
	outliers	0			
	mean (n)	3.68			
	st.dev. (n)	0.990			
	R(calc.)	2.77			
	st.dev.(D5386:16)	1.846			
	R(D5386:16)	5.17			compare R(D1209:05(2011)) = 7

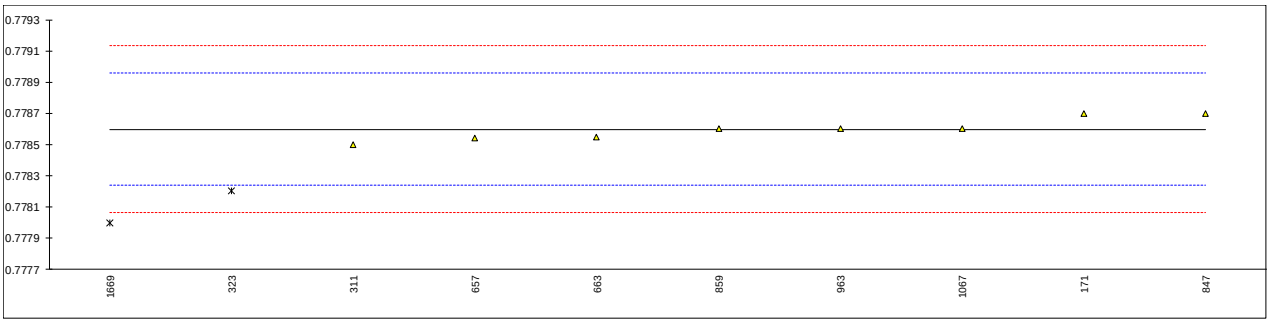


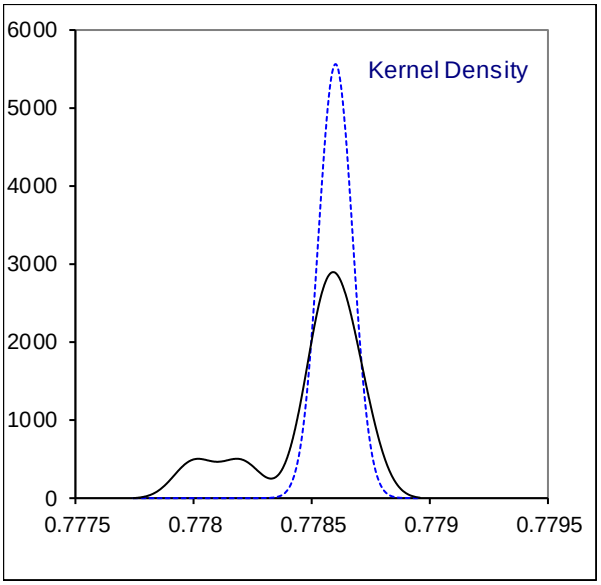


Determination of Density at 20°C on sample #18300; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7787		0.57	
311	D4052	0.7785		-0.55	
323	D4052	0.7782	DG(0.01)	-2.23	
490		-----		-----	
657	D4052	0.77854		-0.33	
663	D4052	0.77855		-0.27	
847	D4052	0.7787		0.57	
859	D4052	0.7786		0.01	
963	D4052	0.7786		0.01	
1067	D4052	0.7786		0.01	
1669	D4052	0.778	C,DG(0.01)	-3.35	first reported: 0.7870 (not at 20°C)

normality	OK
n	8
outliers	2
mean (n)	0.77860
st.dev. (n)	0.000072
R(calc.)	0.00020
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

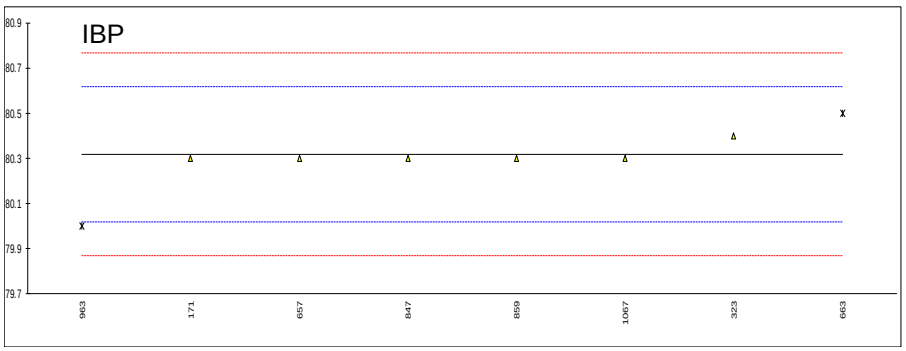


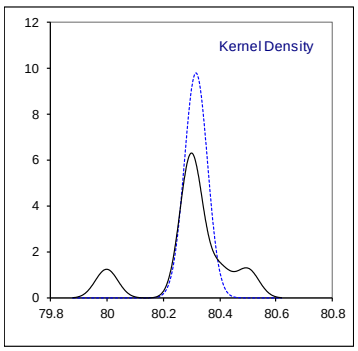


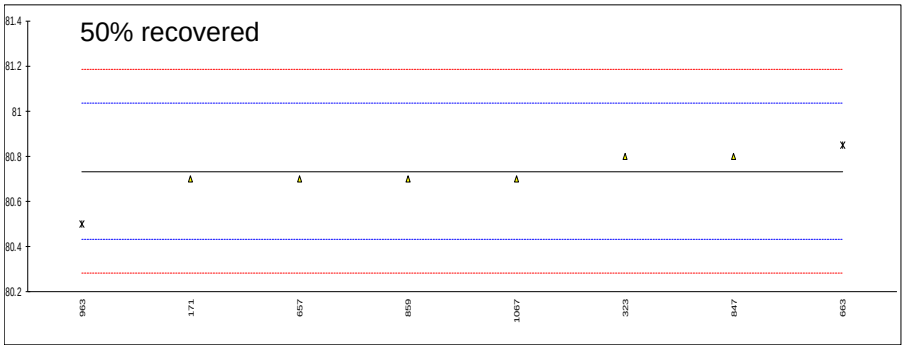
Determination of Distillation on sample #18300; results in °C

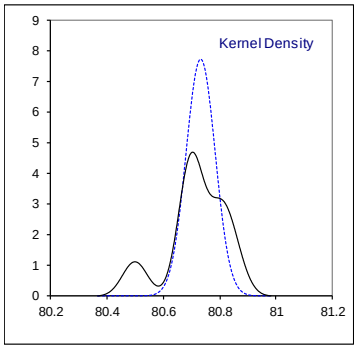
Lab	method	IBP	mark	z(targ)	50%	mark	z(targ)	DP	mark	z(targ)	range	mark	z(targ)
171	D850-automated	80.3		-0.11	80.7		-0.22	81.2		-1.81	-----		-----
311		-----		-----	-----		-----	-----		-----	-----		-----
323	D850-manual	80.4		0.56	80.8		0.44	81.5		0.19	1.1		-----
490		-----		-----	-----		-----	-----		-----	-----		-----
657	D850-manual	80.3		-0.11	80.7		-0.22	81.5		0.19	1.2		-----
663	D850-automated	80.50	ex	1.22	80.85	ex	0.78	82.80	D(0.01)	8.86	2.30	D(0.01)	-----
847	D850-manual	80.3		-0.11	80.8		0.44	81.6		0.86	1.3		-----
859	D850-manual	80.3		-0.11	80.7		-0.22	81.5		0.19	1.2		-----
963	D850-automated	80.0	D(0.01)	-2.11	80.5	D(0.05)	-1.56	81.4		-0.48	1.4		-----
1067	D850-manual	80.3		-0.11	80.7		-0.22	81.6		0.86	1.3		-----
1669		-----		-----	-----		-----	-----		-----	-----		-----
	normality	unknown			unknown			unknown			unknown		
	n	6			6			7			6		
	outliers	1 (+1ex)			1 (+1ex)			1			1		
	mean (n)	80.32			80.73			81.47			1.25		
	st.dev. (n)	0.041			0.052			0.138			0.105		
	R(calc.)	0.11			0.14			0.39			0.29		
	st.dev.(D850-auto:16)	0.15			0.15			0.15			n.a.		
	R(D850-auto:16)	0.42			0.42			0.42			n.a.		

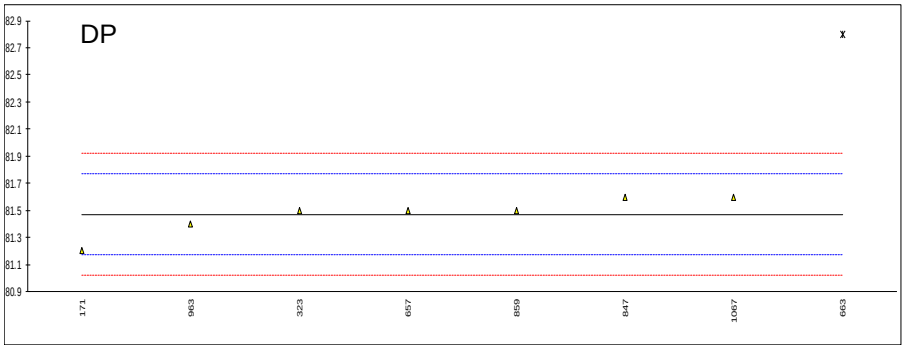
Lab 663: the distillation range was higher than 2°C and not corrected for barometric and temperature deviations, therefore the other non-outlying results were excluded.

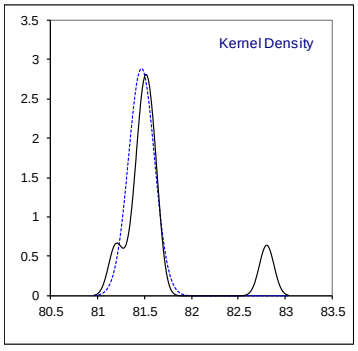






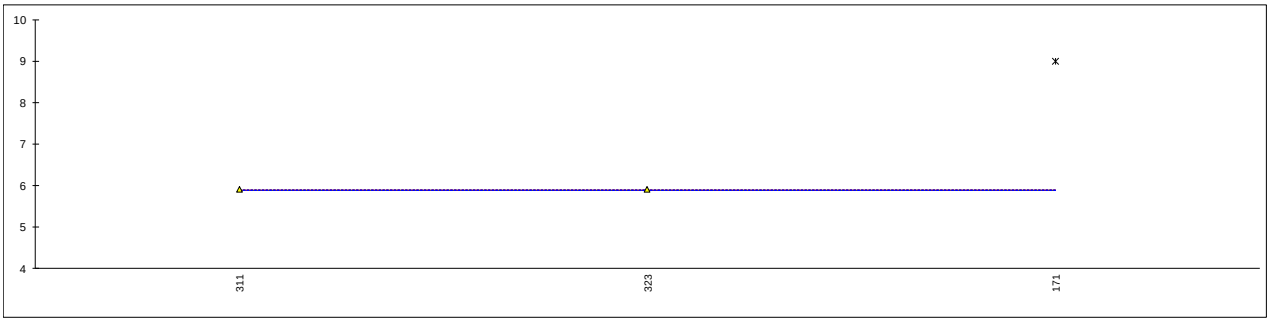






Determination of Freezing Point on sample #18300; results in °C

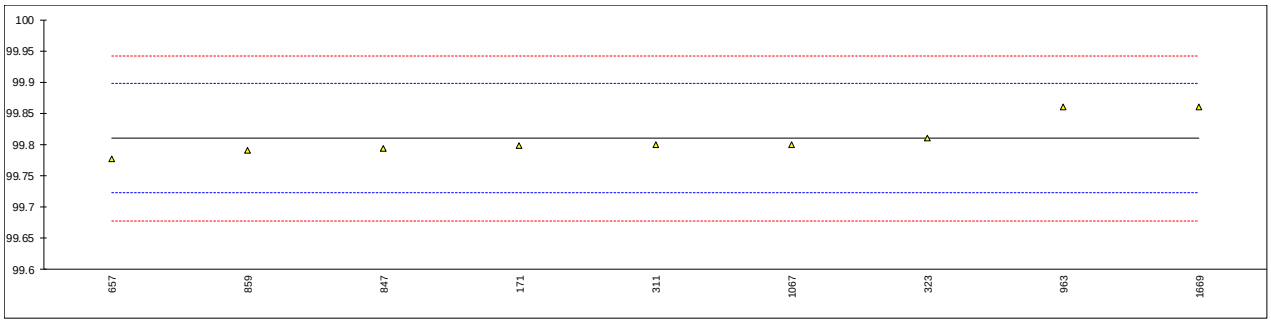
lab	method	value	mark	z(targ)	remarks
171	D5972	9.0	ex	-----	excluded for method reported is for Jet Fuel, see §4
311	D1493	5.90		-----	
323	D1016	5.9		-----	
490		-----		-----	
657		-----		-----	
663		-----		-----	
847		-----		-----	
859		-----		-----	
963		-----		-----	
1067		-----		-----	
1669		-----		-----	
	n	2 (+1ex)			
	mean (n)	5.90			

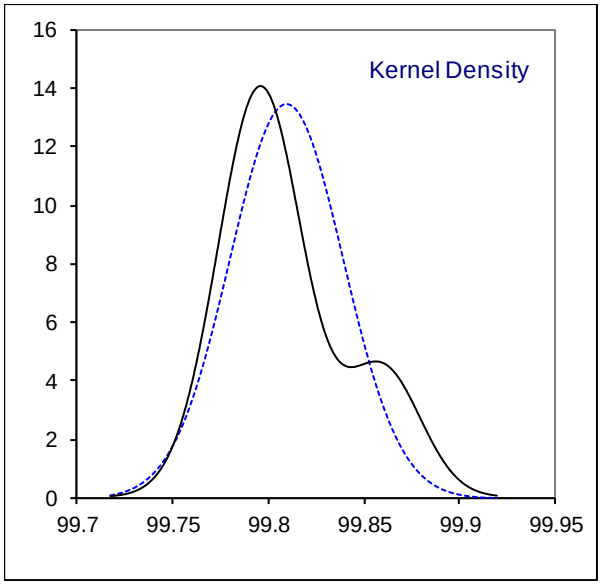


Determination of Purity on sample #18300; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D7266	99.798		-0.27	
311	D3054	99.80		-0.23	
323	D7266	99.81		0.00	
490		-----		-----	
657	D7266	99.7778		-0.73	
663		-----		-----	
847	D7266	99.794		-0.36	
859	D7266	99.79		-0.45	
963	D7266	99.86	C	1.14	first reported: 99.9607
1067		99.8		-0.23	
1669	D7266	99.86	C	1.14	first reported: 99.88

normality	suspect
n	9
outliers	0
mean (n)	99.8100
st.dev. (n)	0.02966
R(calc.)	0.0830
st.dev.(D7266:13e1)	0.04396
R(D7266:13e1)	0.1231

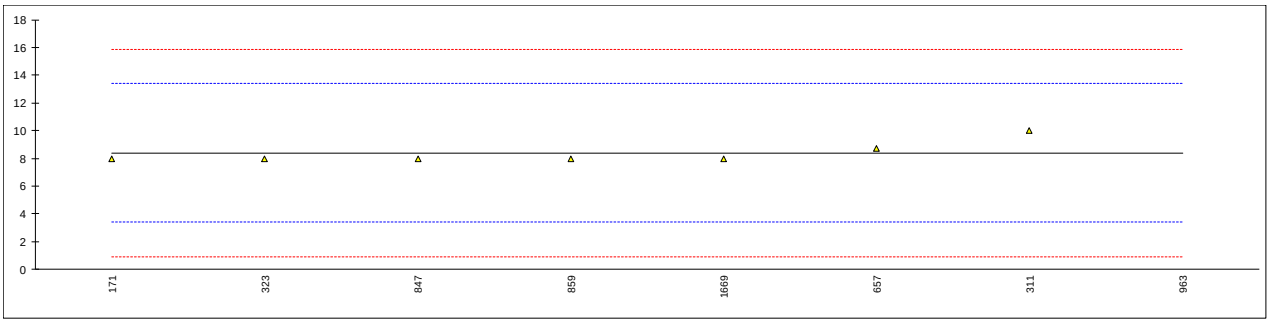


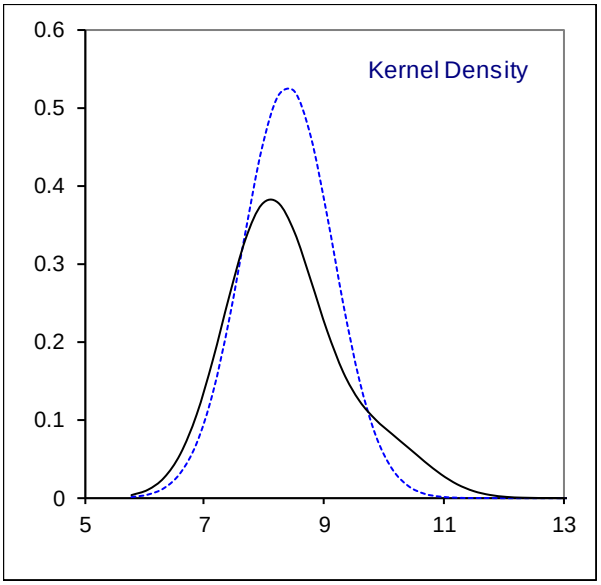


Determination of Benzene on sample #18300 in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7266	8.0		-0.15	
311	D3054	10		0.65	
323	D7266	8		-0.15	
490		-----		-----	
657	D7266	8.7		0.13	
663		-----		-----	
847	D7266	8		-0.15	
859	D7266	8		-0.15	
963	D7266	73	D(0.01)	25.87	
1067		< 10		-----	
1669	D7266	8		-0.15	

normality	unknown
n	7
outliers	1
mean (n)	8.4
st.dev. (n)	0.76
R(calc.)	2.1
st.dev.(D7266:13e1)	2.50
R(D7266:13e1)	7.0

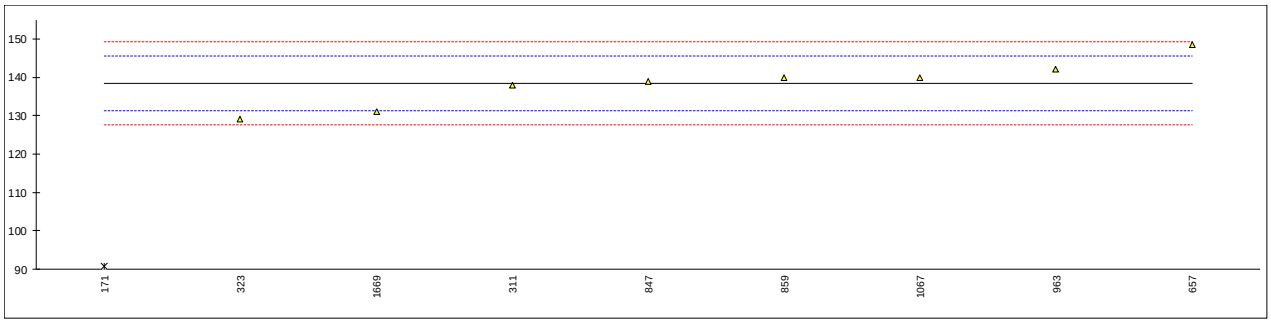


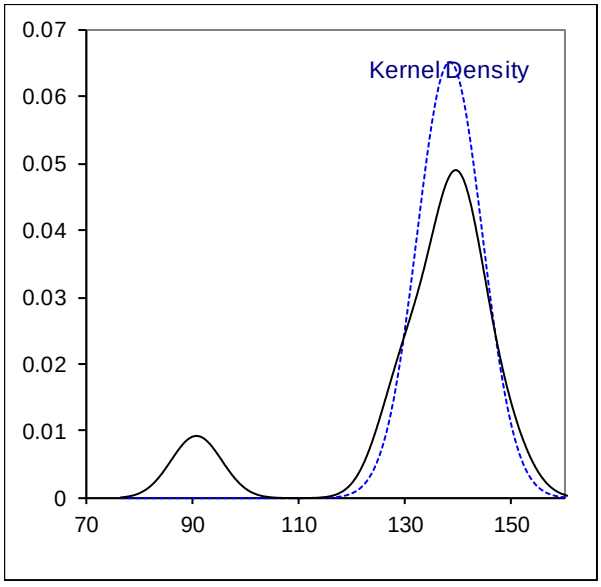


Determination of n-Hexane on sample #18300; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7266	90.87	C,D(0.01)	-13.32	first reported: 8.76
311	D3054	138		-0.12	
323	D7266	129		-2.64	
490		-----		-----	
657	D7266	148.4		2.79	
663		-----		-----	
847	D7266	139		0.16	
859	D7266	140		0.44	
963	D7266	142		1.00	
1067		140		0.44	
1669	D7266	131		-2.08	

normality	OK
n	8
outliers	1
mean (n)	138.4
st.dev. (n)	6.11
R(calc.)	17.1
st.dev.(D7266:13e1)	3.57
R(D7266:13e1)	10

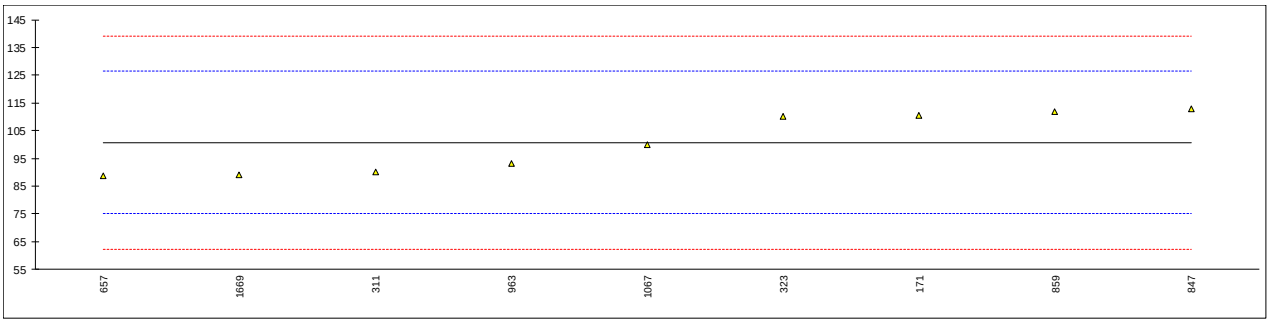


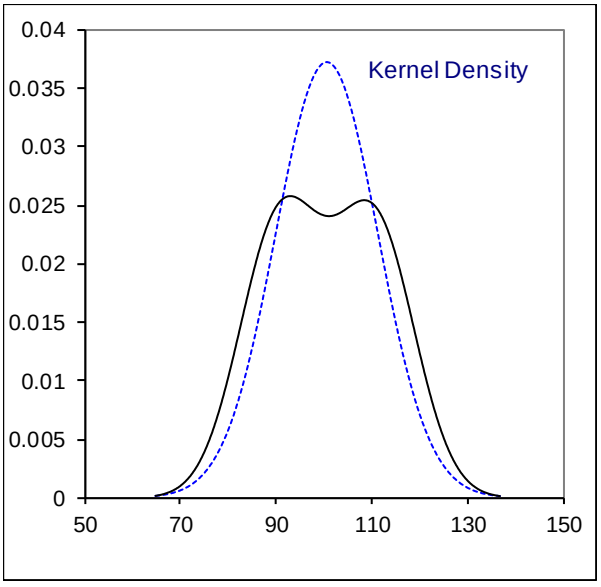


Determination of Methylcyclohexane on sample #18300; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7266	110.6		0.77	
311	D3054	90		-0.84	
323	D7266	110		0.73	
490		-----		-----	
657	D7266	88.8		-0.93	
663		-----		-----	
847	D7266	113		0.96	
859	D7266	112		0.88	
963	D7266	93		-0.60	
1067		100		-0.06	
1669	D7266	89		-0.91	

normality	OK
n	9
outliers	0
mean (n)	100.7
st.dev. (n)	10.70
R(calc.)	30.0
st.dev.(D7266:13e1)	12.80
R(D7266:13e1)	35.9

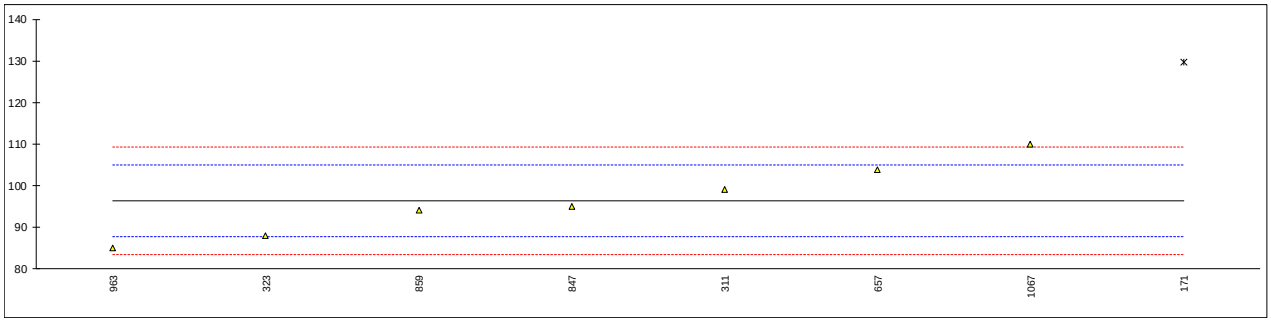


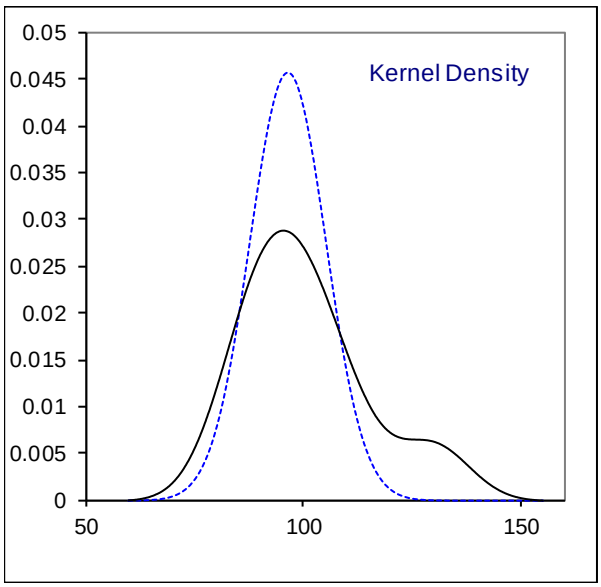


Determination of Methylcyclopentane on sample #18300; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7266	129.76	C,D(0.05)	7.78	first reported: 108
311	D3054	99		0.60	
323	D7266	88		-1.96	
490		-----		-----	
657	D7266	103.9		1.75	
663		-----		-----	
847	D7266	95		-0.33	
859	D7266	94		-0.56	
963	D7266	85		-2.66	
1067		110		3.17	
1669	D7266	<5	C	<-21.33	first reported: 25.6, possible false negative test result?

	unknow
normality	n
n	7
outliers	1
mean (n)	96.4
st.dev. (n)	8.72
R(calc.)	24.4
st.dev.(D7266:13e1)	4.29
R(D7266:13e1)	12

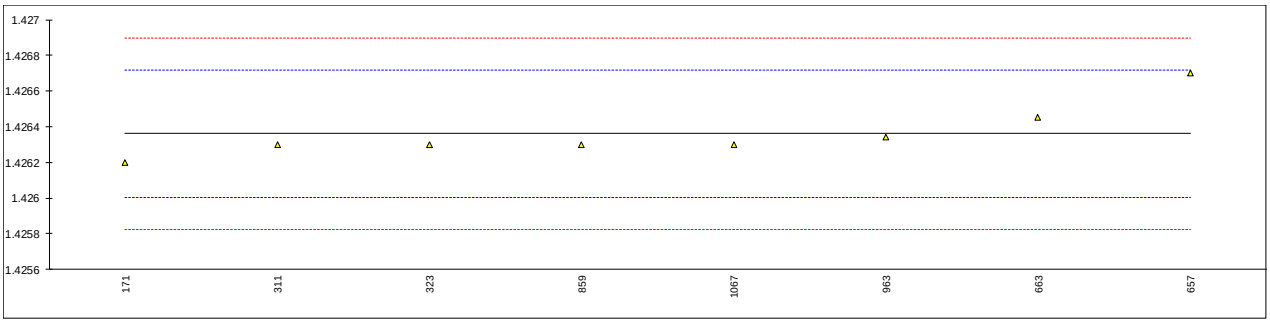


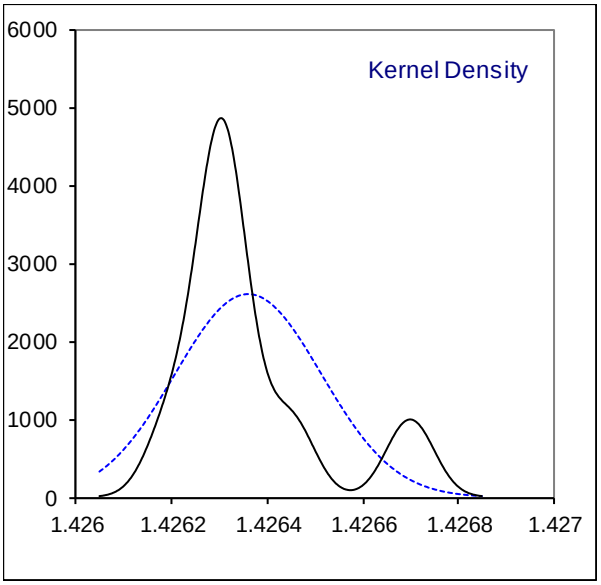


Determination of Refractive Index at 20°C on sample #18300;

lab	method	value	mark	z(targ)	remarks
171	D1218	1.4262	C	-0.90	first reported: 1.4235
311	D1218	1.4263		-0.34	
323	D1218	1.4263		-0.34	
490		-----		-----	
657	D1218	1.4267		1.90	
663	D1218	1.42645		0.50	
847		-----		-----	
859	D1218	1.4263		-0.34	
963	D1218	1.42634		-0.12	
1067	D1218	1.4263		-0.34	
1669		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	1.42636
st.dev. (n)	0.000153
R(calc.)	0.00043
st.dev.(D1218:12)	0.000179
R(D1218:12)	0.0005



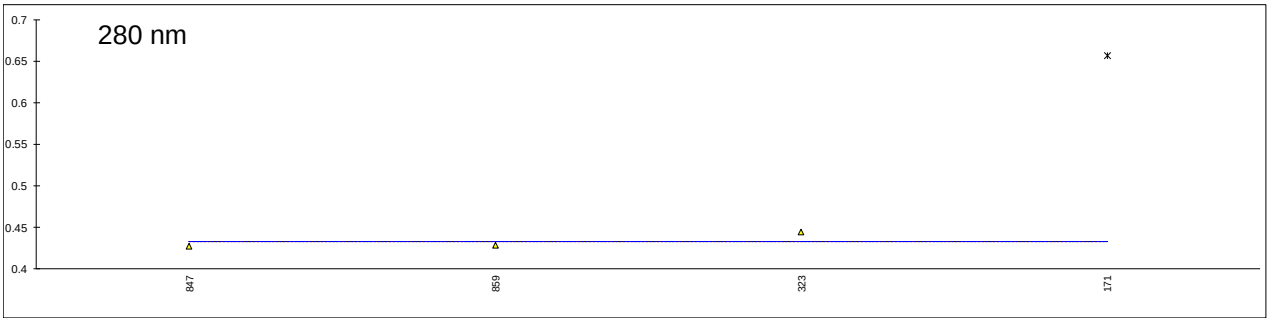


Determination of Sulphur on sample #18300; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311	D5453	<1.0		-----	
323	D6069	< 1		-----	
490		-----		-----	
657	D5453	0.2		-----	
663		-----		-----	
847		-----		-----	
859	D5453	<1		-----	
963		-----		-----	
1067	D5453	< 1.0		-----	
1669	D5453	<0.2		-----	
	n	6			
	mean (n)	<1			

Determination of UV Absorbance (10 mm cuvette) on sample #18300

Lab	method/cuvet size	280nm	mark	z(targ)	260nm	mark	z(targ)	240nm	mark	z(targ)	Pass/Fail
171	10 mm	0.6562	D(0.01)	-----	1.8206		-----	1.2734		-----	fail
311		-----		-----	-----		-----	-----		-----	-----
323	UOP495	0.444		-----	> 1		-----	> 1		-----	fails
490		-----		-----	-----		-----	-----		-----	-----
657		-----		-----	-----		-----	-----		-----	-----
663		-----		-----	-----		-----	-----		-----	-----
847	10 mm	0.427		-----	2.220		-----	1.739		-----	Fail
859	10 mm	0.429		-----	2.195		-----	1.750		-----	Fail
963		-----		-----	-----		-----	-----		-----	-----
1067	10 mm	> 1.0		-----	> 1.0		-----	> 1.0		-----	Fail
1669		-----		-----	-----		-----	-----		-----	-----
	normality	unknown			n.a.			n.a.			n.a.
	n	3			5			5			5
	outliers	1			n.a.			n.a.			n.a.
	mean (n)	0.4333			>1			>1			Fail
	st.dev. (n)	0.00929			n.a.			n.a.			n.a.
	R(calc.)	0.0260			n.a.			n.a.			n.a.



APPENDIX 2

Number of participants

1 lab in BELGIUM
2 labs in CHINA, People's Republic
1 lab in GERMANY
2 labs in NETHERLANDS
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SPAIN
1 lab in THAILAND
1 lab in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
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
SDS	= Safety Data Sheet

Literature:

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