**Institute for
Interlaboratory Studies**

Results of Proficiency Test
Cyclohexane
February 2022

CONTENTS

1   INTRODUCTION

Since 2018 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Cyclohexane every year. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of Cyclohexane.

In this interlaboratory study 13 laboratories in 8 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the Cyclohexane proficiency test 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 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 of Cyclohexane in a 1 liter bottle labelled #22022.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the statistical evaluation.

2.1   QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. 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 a 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 20 liters of Cyclohexane was obtained from a local supplier. After homogenization 21 amber glass bottles of 1L were filled and labelled #22022.
The homogeneity of the subsamples was checked by determination of Density at 20˚C in accordance with ASTM D4052 on 4 stratified randomly selected subsamples.

	Density at 20°C in kg/L
sample #22022-1	0.77852
sample #22022-2	0.77853
sample #22022-3	0.77852
sample #22022-4	0.77852

Table 1: homogeneity test results of subsamples #22022

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 20°C in kg/L
r (observed)	0.00001
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #22022

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 one sample Cyclohexane labelled #22022 was sent on February 2, 2022. 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 ANALYZES

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

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 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 result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1   STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the 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 represented 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 of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, 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:

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

The $z_{(target)}$ 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.
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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another two weeks. One participant reported the test results after the extended reporting date. Three other participants did not report any test results at all. Not all participants were able to report all tests requested.
In total 10 participants reported 104 numerical test results. Observed were 4 outlying test results, which is 3.8%. 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 reported test results in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D7266) and an added designation for the year that the test 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 test result tables of appendix 1 only the method number (sub) 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 D848:18.

Appearance: This determination was not problematic. All participants agreed on the appearance which was bright and clear (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 or ASTM D1209:05(2019).

Density at 20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility for Initial Boiling Point is in agreement with the requirements of ASTM D850:21 for automated, but not in agreement with the requirements of ASTM D850:21 for manual mode. The calculated reproducibilities for 50% recovered and Dry Point are in agreement with the respective requirements for automated and manual mode.

Freezing Point: Only four participants reported a test result. Therefore, no z-scores were calculated.

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

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

n-Hexane: 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 strict requirements of ASTM D7266:13e1(2018). The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

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

Methylcyclopentane: 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 D7266:13e1(2018).

Refractive Index: 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 D1218:21.

Sulfur: This determination may not be problematic. All reporting participants agreed on a concentration lower than 1 mg/kg. Therefore, no z-scores were calculated.

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 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acid Wash Color		7	0.6	0.7	2.0
Appearance		9	Pass (B&C)	n.a.	n.a.
Color Pt/Co		8	2.1	2.7	4.9
Density at 20°C	kg/L	9	0.7786	0.0002	0.0005
Distillation, IBP	°C	7	80.4	0.5	0.6
Distillation, 50% recovered	°C	7	80.7	0.1	0.2
Distillation, Dry Point	°C	7	80.8	0.3	0.5
Freezing Point	°C	3	6.4	0.4	n.e.
Purity	%M/M	10	99.959	0.021	0.022
Benzene	mg/kg	10	28	10	25
n-Hexane	mg/kg	7	133	18	6
Methylcyclohexane	mg/kg	9	183	65	62
Methylcyclopentane	mg/kg	6	4	1	4
Refractive Index at 20°C		6	1.4263	0.0002	0.0005
Sulfur	mg/kg	8	<1	n.e.	n.e.

Table 3: reproducibilities of tests on sample #22022

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

4.3   COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2022 WITH PREVIOUS PTS

	February 2022	February 2021	February 2020	February 2019	March 2018
Number of reporting laboratories	10	11	9	10	10
Number of test results	104	111	88	104	120
Number of statistical outliers	4	2	5	2	10
Percentage of statistical outliers	3.8%	1.8%	5.7%	1.9%	8.3%

Table 4: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

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

Parameter	February 2022	February 2021	February 2020	February 2019	March 2018
Acid Wash Color	++	++	n.e.	++	++
Color Pt/Co	+	++	++	+	+
Density at 20°C	++	+	+	++	++
Distillation	+	+	+	++	++
Freezing Point	n.e.	n.e.	n.e.	n.e.	n.e.
Purity	+/-	+	+	++	+
Benzene	++	++	++	++	++
n-Hexane	--	-	n.e.	-	-
Methylcyclohexane	+/-	++	+	++	+
Methylcyclopentane	++	+	++	++	--
Refractive Index at 20°C	++	+	++	-	+
Sulfur	n.e.	n.e.	n.e.	n.e.	n.e.

Table 5: comparison determinations against the reference test methods

In the table above 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

APPENDIX 1
Determination of Acid Wash Color on sample #22022

lab	method	Reported test value	iis conversion *)	mark	z(targ)	remarks
171	D848	1-	0.75		0.20	
315	D848	0+	0.25		-0.50	
323	D848	1-	0.75		0.20	
396		-----	-----		-----	
657	D848	0+	0.25		-0.50	
847		-----	-----		-----	
859		-----	-----		-----	
963	D848	1-	0.75		0.20	
1081		-----	-----		-----	
1135	D848	1-	0.75		0.20	
1264		-----	-----		-----	
1530	D848	<1	0.75		0.20	
6315		-----	-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	0.607
st.dev. (n)	0.2440
R(calc.)	0.683
st.dev.(D848:18)	0.7074
R(D848:18)	1.981

*) 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).
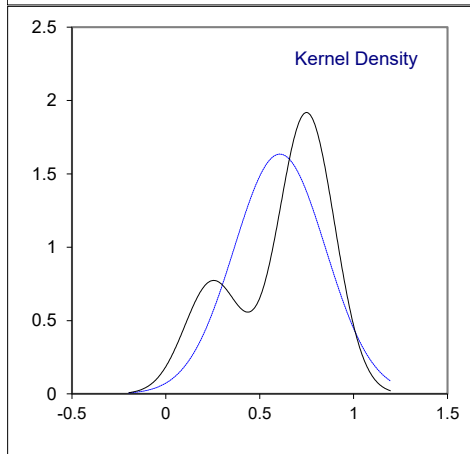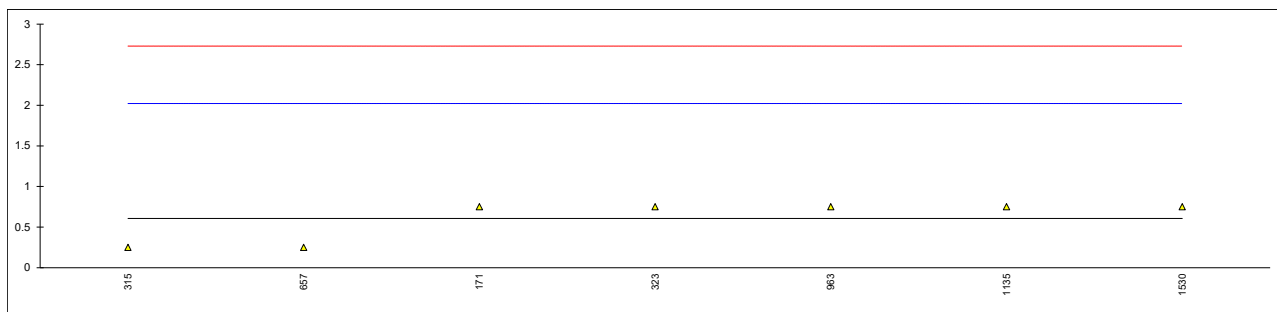
Determination of Appearance on sample #22022

lab	method	value	mark	z(targ)	remarks
171	E2680	Pass		-----	
315	E2680	pass		-----	
323	Visual	C&B		-----	
396	Visual	Clear & Bright		-----	
657	Visual	Clear & free from suspended solid		-----	
847		-----		-----	
859		-----		-----	
963	Visual	Clear		-----	
1081		b/c		-----	
1135	D4176	CFSM		-----	
1264		-----		-----	
1530	Visual	c&b		-----	
6315		-----		-----	

n	9
outliers	0
mean (n)	Pass (bright and Clear)

Determination of Color Pt/Co on sample #22022

lab	method	value	mark	z(targ)	remarks
171	D5386	1.9		-0.11	
315	D5386	3		0.52	
323	D5386	2		-0.05	
396	D1209	<5		-----	
657	D5386	2.83		0.42	
847		-----		-----	
859		-----		-----	
963	D5386	2		-0.05	
1081	D5386	3		0.52	
1135	D5386	0		-1.19	
1264		-----		-----	
1530	D1209	<3		-----	
6315	ISO6271	2		-0.05	

normality	unknown
n	8
outliers	0
mean (n)	2.09
st.dev. (n)	0.973
R(calc.)	2.73
st.dev.(D5386:16)	1.758
R(D5386:16)	4.92
Compare	
R(D1209:05)	7
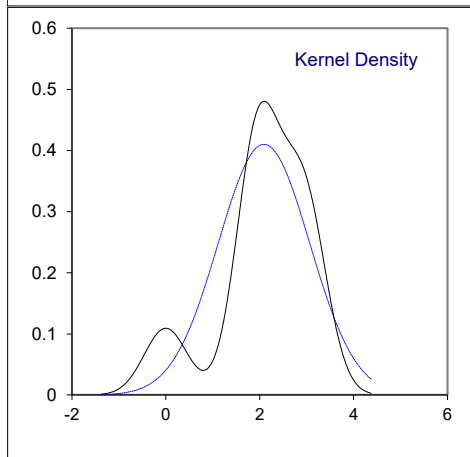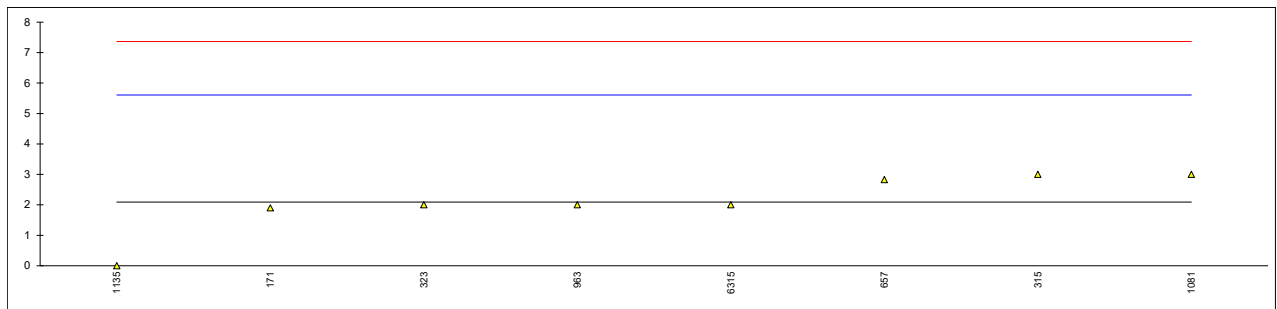
Determination of Density at 20°C on sample #22022; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.77855		-0.19	
315	D4052	0.7785		-0.47	
323	D4052	0.7786		0.09	
396	D4052	0.7787		0.65	
657	D4052	0.7786		0.09	
847		-----		-----	
859		-----		-----	
963	ISO12185	0.7786		0.09	
1081		-----		-----	
1135	ISO12185	0.7786		0.09	
1264		-----		-----	
1530	ISO12185	0.77850		-0.47	
6315	ISO12185	0.7786		0.09	

normality	OK
n	9
outliers	0
mean (n)	0.77858
st.dev. (n)	0.000061
R(calc.)	0.00017
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005
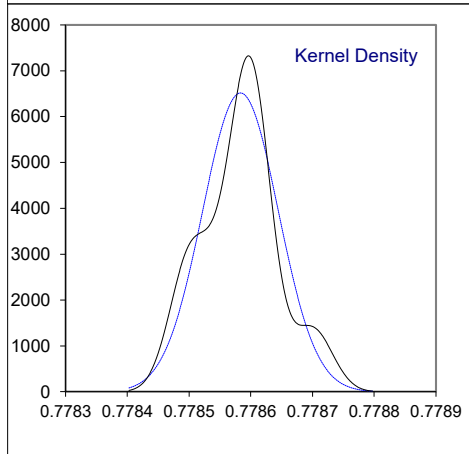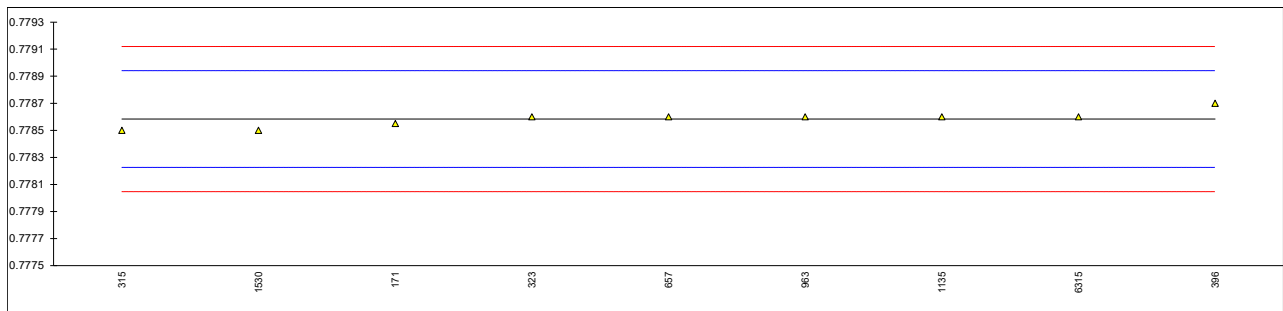
Determination of Distillation on sample #22022; results in °C

| Lab	method	IBP	mark	z(targ)	50%	mark	z(targ)	DP	mark	z(targ)	range	mark
171	D850-automated	80.5		0.55	80.6		-1.38	80.7		-0.44	0.2	
315	D1078	80.5		0.55	80.7		0.41	80.8		0.18	0.3	
323	D850-automated	80.5		0.55	80.7		0.41	81.0		1.40	0.5	
396		-----		-----	-----		-----	-----		-----	-----	
657	D850-automated	80.3		-0.41	80.7		0.41	80.8		0.18	0.5	
847		-----		-----	-----		-----	-----		-----	-----	
859		-----		-----	-----		-----	-----		-----	-----	
963	D850-automated	80.2		-0.89	80.64		-0.67	80.7		-0.44	0.5	
1081		-----		-----	-----		-----	-----		-----	-----	
1135	D850-automated	80.1		-1.38	80.7		0.41	80.7		-0.44	0.6	
1264		-----		-----	-----		-----	-----		-----	-----	
1530		-----		-----	-----		-----	-----		-----	-----	
6315	D850-automated	80.6		1.03	80.7		0.41	80.7		-0.44	0.1	
	normality	unknown			unknown			unknown				
	n	7			7			7				
	outliers	0			0			0				
	mean (n)	80.39			80.68			80.77				
	st.dev. (n)	0.186			0.041			0.111				
	R(calc.)	0.52			0.11			0.31				
	st.dev.(D850-A:21)	0.208			0.056			0.163				
	R(D850-A:21)	0.58			0.16			0.46				
Compare												
	R(D850-M:21)	0.41			0.65			0.65				
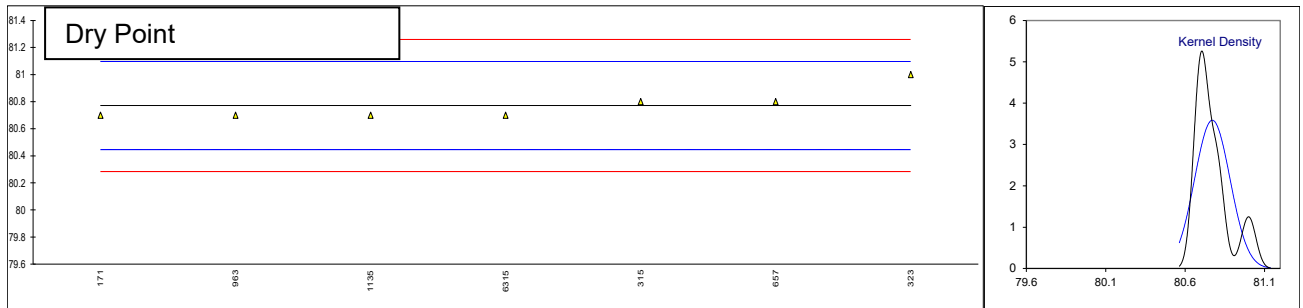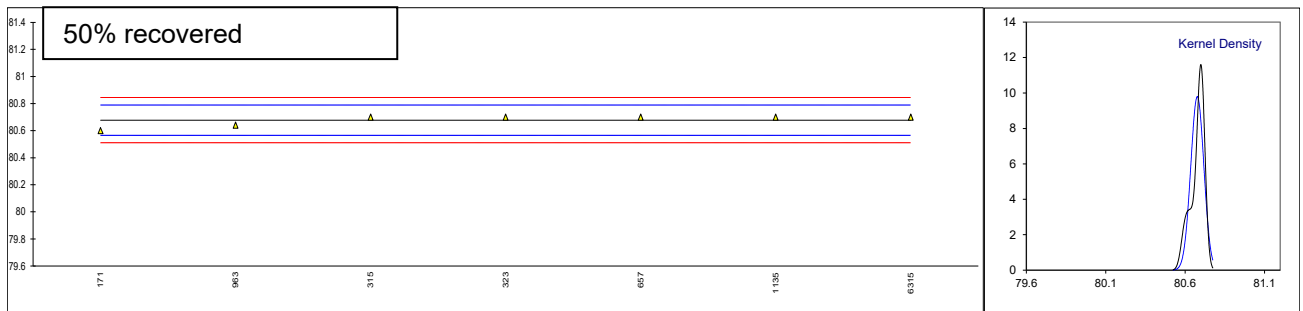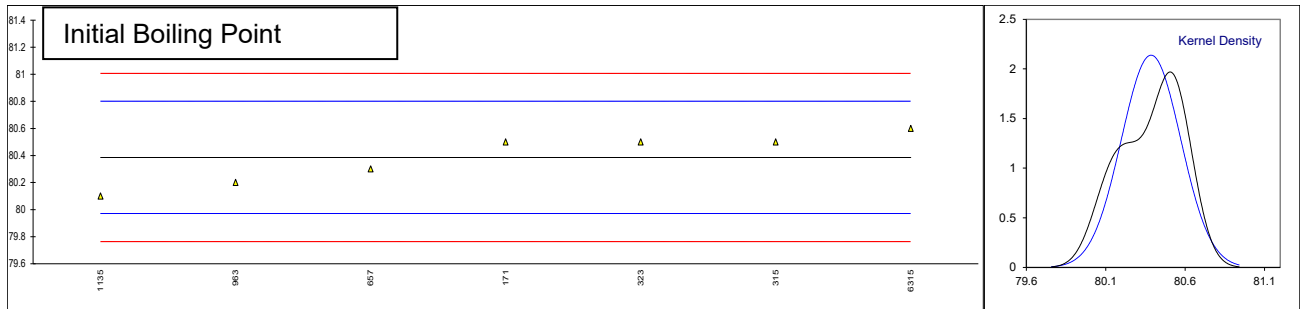
Determination of Freezing Point on sample #22022; results in °C

lab	method	value	mark	z(targ)	remarks
171	D2386	7.3	ex	-----	ex: test method is not intended for High purity Hydrocarbons
315		-----		-----	
323	D1015	6.5		-----	
396	INH-3230	6.2		-----	
657		-----		-----	
847		-----		-----	
859		-----		-----	
963		-----		-----	
1081		-----		-----	
1135	D1015	6.43		-----	
1264		-----		-----	
1530		-----		-----	
6315		-----		-----	

|	
normality	unknown
n	3
outliers	0 (+1ex)
mean (n)	6.38
st.dev. (n)	0.157
R(calc.)	0.44
st.dev.(D1015:05)	(0.0054)
R(D1015:05)	(0.015)
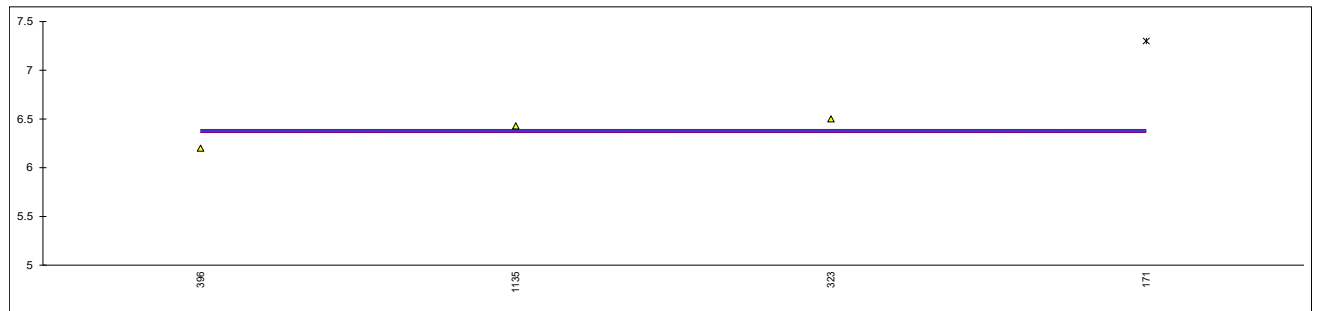
Determination of Purity of Cyclohexane on sample #22022; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D7266	99.96		0.11	
315	D3054	99.95		-1.18	
323	D7266	99.96		0.11	
396	D7266	99.95		-1.18	
657	D7266	99.9540		-0.66	
847		-----		-----	
859		-----		-----	
963	D7266	99.965		0.75	
1081		99.9570		-0.28	
1135	D7266	99.97	C	1.40	First reported 99.91
1264		-----		-----	
1530	D7266	99.9702		1.42	
6315	D7871	99.9553		-0.50	

normality	OK
n	10
outliers	0
mean (n)	99.95915
st.dev. (n)	0.007371
R(calc.)	0.02064
st.dev.(D7266:13e1)	0.007767
R(D7266:13e1)	0.02175
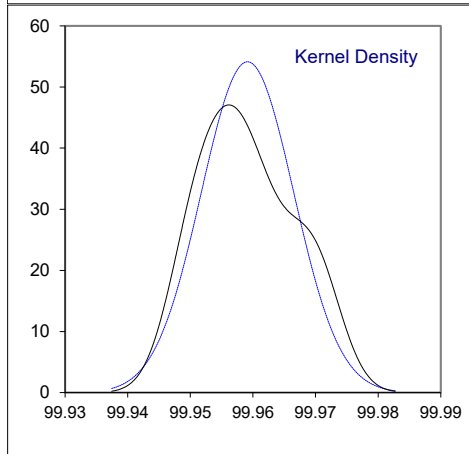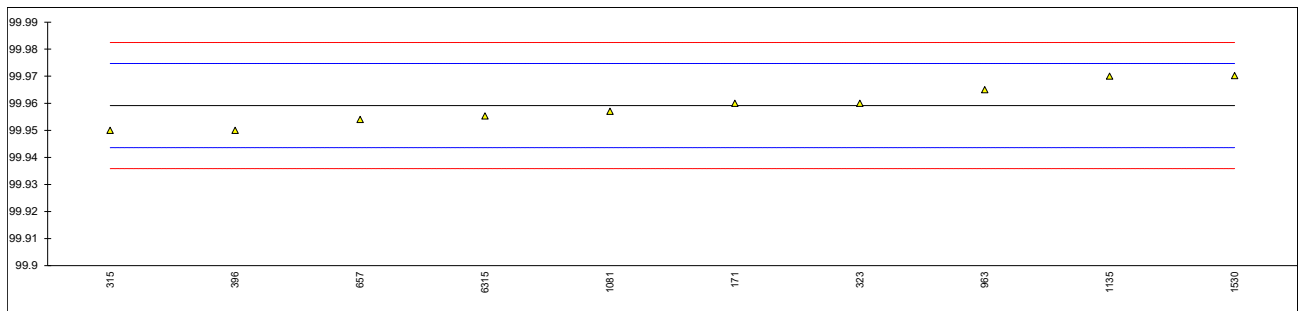
Determination of Benzene on sample #22022 in mg/kg
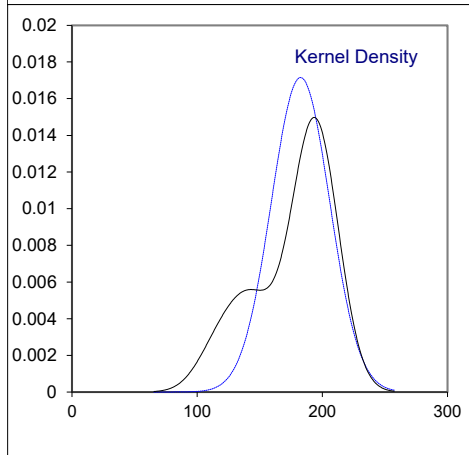
lab	method	value	mark	z(targ)	remarks
171	D7266	24.98		-0.34	
315	D3054	32		0.44	
323	D7266	33		0.55	
396	D7266	25		-0.34	
657	D7266	29.2039		0.13	
847		-----		-----	
859		-----		-----	
963	D7266	30		0.22	
1081		30.63675		0.29	
1135	D7266	21		-0.79	
1264		-----		-----	
1530	D7266	26		-0.23	
6315	D7871	28.7		0.07	

normality	OK
n	10
outliers	0
mean (n)	28.052
st.dev. (n)	3.7266
R(calc.)	10.434
st.dev.(D7266:13e1)	8.9527
R(D7266:13e1)	25.068

Kernel Density
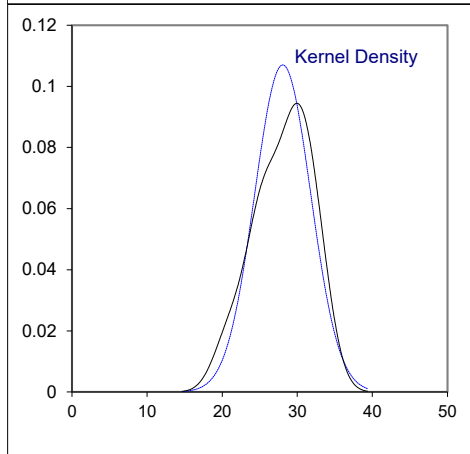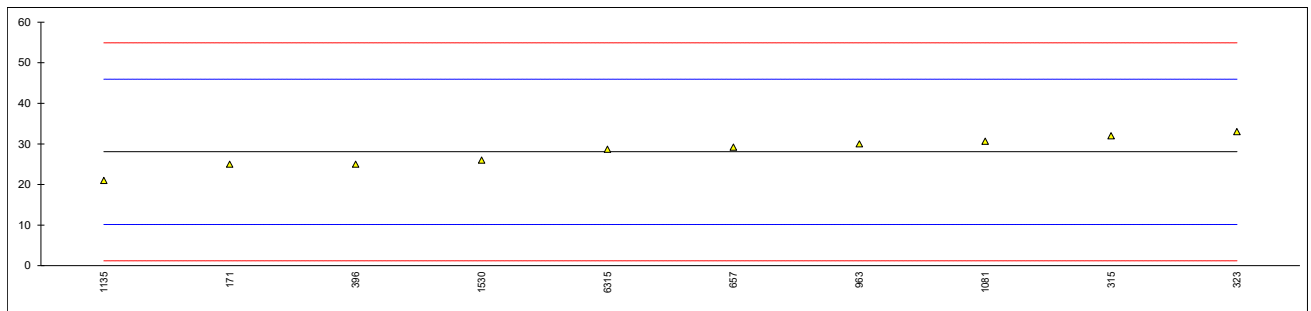
Determination of n-Hexane on sample #22022; results in mg/kg
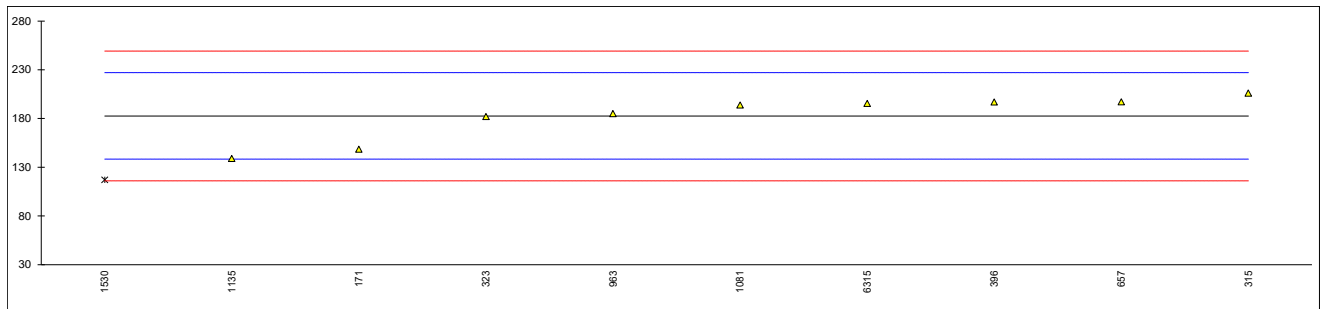
lab	method	value	mark	z(targ)	remarks
171	D7266	109.31	G(0.05)	-10.07	
315	D3054	136		1.47	
323	D7266	142		4.07	
396		-----		-----	
657	D7266	136.7332		1.79	
847		-----		-----	
859		-----		-----	
963	D7266	130		-1.12	
1081		127.90700		-2.03	
1135	D7266	123	C	-4.15	First reported 262
1264		-----		-----	
1530		-----		-----	
6315	D7871	132.5		-0.04	

normality	unknown
n	7
outliers	1
mean (n)	132.591
st.dev. (n)	6.2976
R(calc.)	17.633
st.dev.(D7266:13e1)	2.3126
R(D7266:13e1)	6.475
Compare	
R(Horwitz)	28.466
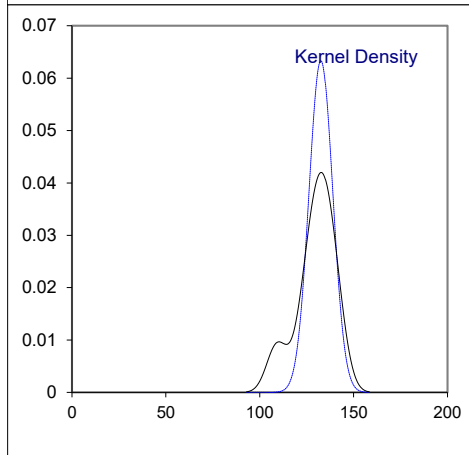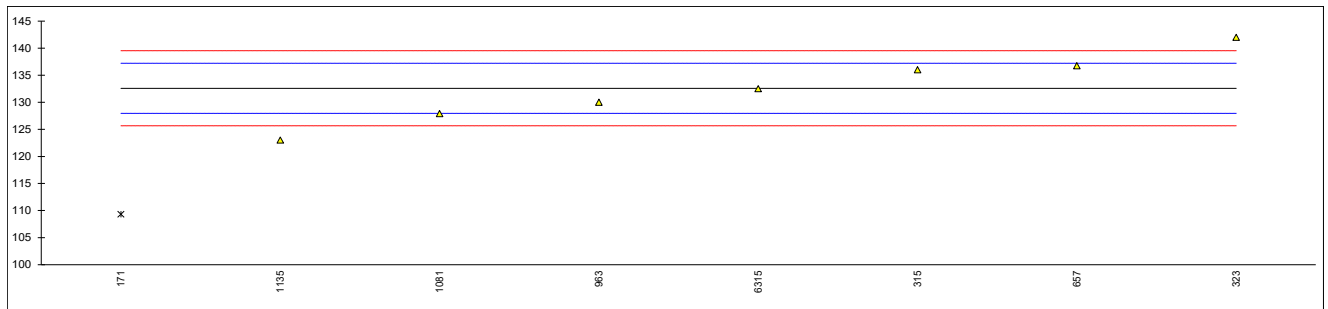
Determination of Methylcyclohexane on sample #22022; results in mg/kg

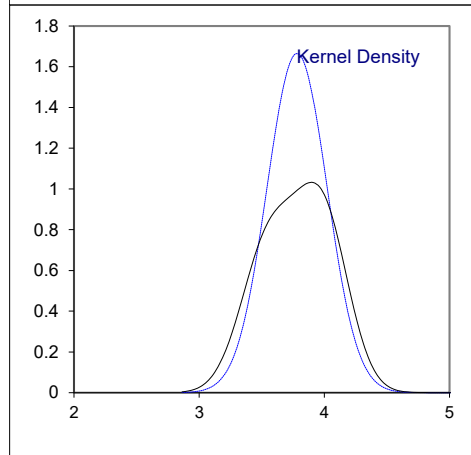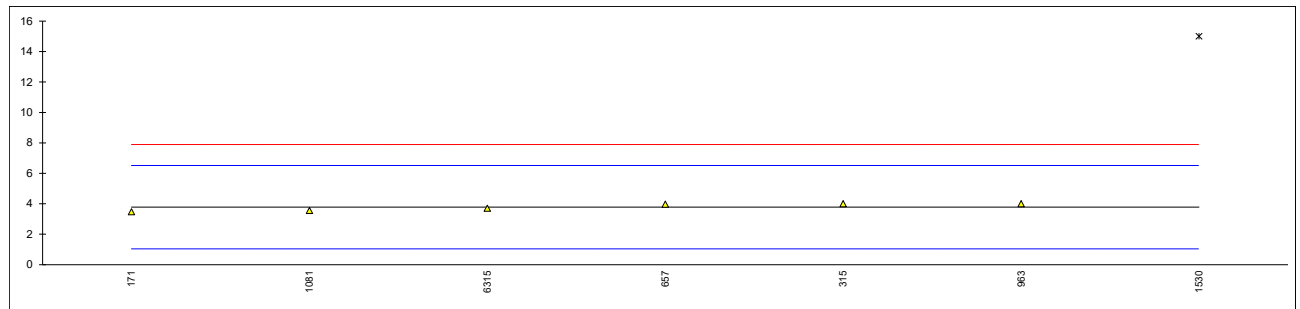| lab	method	value	mark	z(targ)	remarks
171	D7266	148.48		-1.54	
315	D3054	206		1.05	
323	D7266	182		-0.03	
396	D7266	197	C	0.65	First reported 297
657	D7266	197.1706		0.65	
847		-----		-----	
859		-----		-----	
963	D7266	185		0.11	
1081		193.84104		0.50	
1135	D7266	139	C	-1.97	First reported 316
1264		-----		-----	
1530	D7266	117	G(0.05)	-2.96	
6315	D7871	195.4		0.57	

normality	OK
n	9
outliers	1
mean (n)	182.655
st.dev. (n)	23.2612
R(calc.)	65.131
st.dev.(D7266:13e1)	22.1931
R(D7266:13e1)	62.141
Compare	
R(Horwirtz)	37.368

Kernel Density

## Determination of Methylcyclopentane on sample #22022; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7266	3.47		-0.23	
315	D3054	4		0.16	
323	D7266	<5		-----	
396		-----		-----	
657	D7266	3.9737		0.14	
847		-----		-----	
859		-----		-----	
963	D7266	4		0.16	
1081		3.55643		-0.17	
1135	D7266	<3		-----	
1264		-----		-----	
1530	D7266	15	G(0.01)	8.19	
6315	D7871	3.7		-0.06	

	normality	unknown
	n	6
	outliers	1
	mean (n)	3.783
	st.dev. (n)	0.2395
	R(calc.)	0.671
	st.dev.(D7266:13e1)	1.3689
	R(D7266:13e1)	3.833
Compare		
	R(Horwitz)	1.387

Kernel Density

Determination of Refractive Index at 20°C on sample #22022;

lab	method	value	mark	z(targ)	remarks
171	D1218	1.42625		-0.33	
315	D1218	1.4263		-0.05	
323	D1218	1.4263		-0.05	
396		-----		-----	
657	D1218	1.42620		-0.61	
847		-----		-----	
859		-----		-----	
963	D1218	1.4264		0.51	
1081		-----		-----	
1135	D1218	1.4264		0.51	
1264		-----		-----	
1530	D1218	1.42680	G(0.05)	2.75	
6315		-----		-----	

normality	unknown
n	6
outliers	1
mean (n)	1.42631
st.dev. (n)	0.000080
R(calc.)	0.00022
st.dev.(D1218:21)	0.000179
R(D1218:21)	0.0005
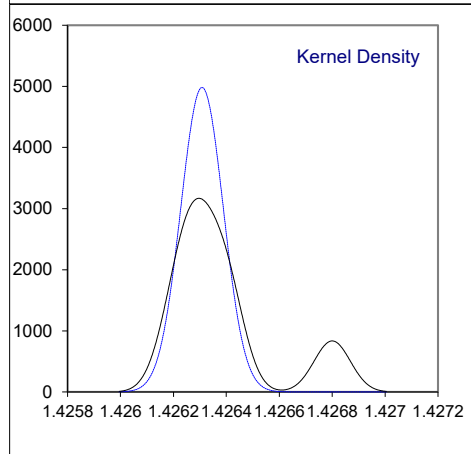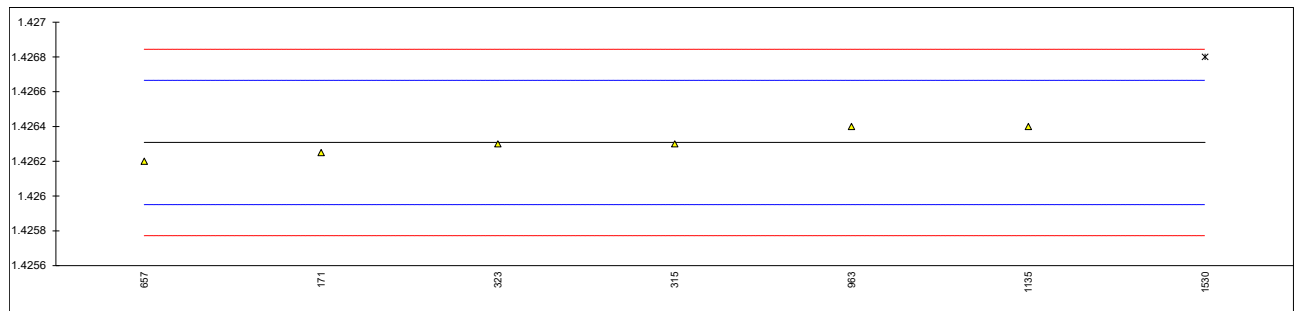
Determination of Sulfur on sample #22022; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D5453	0.5		-----	
315	D7183	<0.1		-----	
323	D5453	<1		-----	
396	IP373	<1		-----	
657	D5453	0.198		-----	
847		-----		-----	
859		-----		-----	
963	D7183	<0.5		-----	
1081	D7183	0.00		-----	
1135	D7183	<0.1		-----	
1264		-----		-----	
1530		-----		-----	
6315		-----		-----	

n	8	
mean (n)	<1	

APPENDIX 2

Number of participants per country

2 labs in BELGIUM
2 labs in CHINA, People's Republic
2 labs in GERMANY
1 lab in ITALY
2 labs in NETHERLANDS
2 labs in SAUDI ARABIA
1 lab in SINGAPORE
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) or D1	= outlier in Dixon's outlier test
D(0.05) or D5	= straggler in Dixon's outlier test
G(0.01) or G1	= outlier in Grubbs' outlier test
G(0.05) or G5	= straggler in Grubbs' outlier test
DG(0.01) or DG1	= outlier in Double Grubbs' outlier test
DG(0.05) or DG5	= 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
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)