Results of Proficiency Test
Cyclohexane
February 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse Netherlands

Author: ing. G.A. Oosterlaken – Buijs
Correctors: ing. A.S. Noordman – de Neef & A. Lewinska, MSc.

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1 INTRODUCTION

Last year the Institute for Interlaboratory Studies (iis) organizes a proficiency test (PT) for Cyclohexane for the first time. During the annual proficiency testing program 2018/2019 it was decided to continue the round robin for the analysis of Cyclohexane.
In the interlaboratory study 10 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 2019 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 test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample of 1L of Cyclohexane, labelled #19022.
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 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

Approximately 25 liter bulk material of Cyclohexane was obtained from a local chemical supplier. After homogenisation 20 amber glass bottles of 1L were filled and labelled #19022. The homogeneity of the subsamples #19022 was checked by determination of Density at 20°C, according to ASTM D4052 on 4 stratified randomly selected samples.

	Density at 20°C in kg/L
sample #19022-1	0.77848
sample #19022-2	0.77848
sample #19022-3	0.77848
sample #19022-4	0.77848

Table 1: homogeneity test results of subsamples #19022

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 #19022

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 1 bottle of 1L Cyclohexane, labelled #19022, was sent on February 6, 2019. 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 sample #19022: Acid Wash Color, Appearance, Color Pt/Co, Density at 20°C, Distillation (IBP, 50% recovered and DP), Freezing Point, Purity, Benzene, n-Hexane, Methylcyclohexane, Methylcyclopentane, Refractive Index at 20°C, Sulfur and UV Absorbance.

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 appropriate 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 reanalyzes). Additional or corrected test results are used for data analysis and 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 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.

According to ISO 5725 the original test results per determination were submitted to Dixon's, 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. 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 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 or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

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

In this interlaboratory study, no problems were encountered with dispatch of the samples. One participant reported the test results after the final reporting date. Not all laboratories were able to report all analyses requested.
Finally, in total 10 participants reported 104 numerical test results. Observed were 2 outlying test results, which is 1.9% 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 D848:18.

Appearance: All participants agreed on the appearance, 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 or ASTM D1209:05(2011).

Density at 20°C: 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 ISO12185:96.

Distillation: This determination was not problematic. No statistical outliers were observed. All calculated reproducibilities are in agreement with the requirements of ASTM D850:18 automated and manual modes.

Freezing Point: Only two participants reported a numeric test results. Therefore, 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. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7266:13e1(2018).

n-Hexane: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM 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).

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 D7266:13e1(2018).

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

Sulfur: All participants reported a test result and agreed on a value for Sulfur less than 1 mg/kg. Therefore, no z-scores were calculated.

UV Absorbance: Three participants reported test results. Therefore, no z-scores were calculated. All three participants agreed on a “Pass”.

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 number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, ISO test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acid Wash Color		7	0.8 (1-)	0.3	2.1
Appearance		10	Pass (B&C)	n.a.	n.a.
Color Pt/Co		9	2.8	4.2	5.0
Density at 20°C	kg/L	8	0.7785	0.0002	0.0005
Distillation, IBP	°C	6	80.5	0.2	0.4
Distillation, 50% rec.	°C	6	80.7	0.2	0.7
Distillation, DP	°C	6	80.8	0.4	0.7
Freezing Point	°C	2	6.4	n.a.	n.a.
Purity	%M/M	9	99.941	0.016	0.034
Benzene	mg/kg	7	2.9	0.7	2.6
n-Hexane	mg/kg	9	215	20	11
Methylcyclohexane	mg/kg	9	51	5	20
Methylcyclopentane	mg/kg	8	135	8	16
Refractive Index at 20°C		8	1.4265	0.0007	0.0005
Sulfur	mg/kg	10	<1	n.a.	n.a.
UV Absorbance at 280 nm		3	0.01	n.a.	n.a.
UV Absorbance at 260 nm		3	<0.02	n.a.	n.a.
UV Absorbance at 240 nm		3	<0.06	n.a.	n.a.
UV Absorbance, evaluation		3	Pass	n.a.	n.a.

Table 3: reproducibilities of tests on sample #19022

Without further statistical calculations, it could 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.

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2019 WITH THE PREVIOUS PT

	February 2019	March 2018
Number of reporting participants	10	10
Number of test results	104	120
Number of statistical outliers	2	10
Percentage outliers	1.9%	8.3%

Table 4: comparison with the previous proficiency test

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 respective reference test methods. The conclusions are given the following table:

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

Table 5: comparison determinations against the reference test methods

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

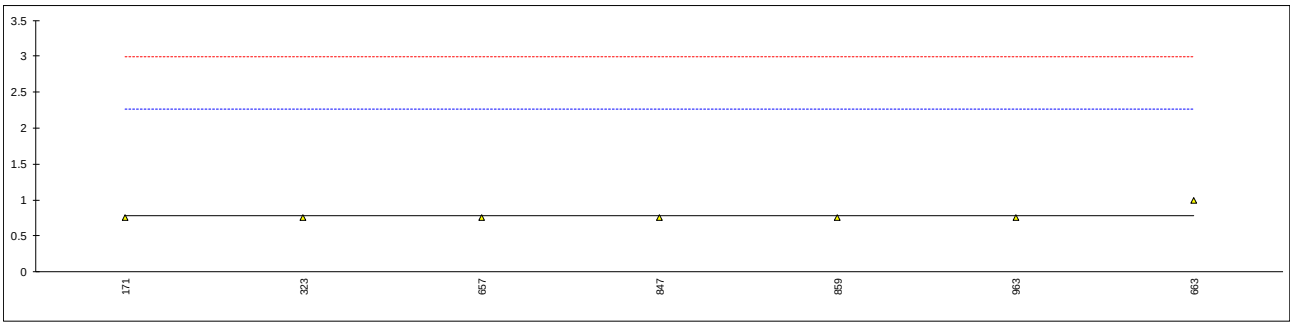
Determination of Acid Wash Color (acid layer) on sample #19022

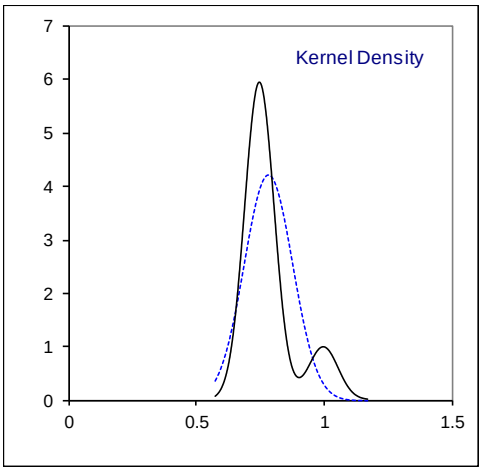
lab	method	value	mark	z(targ)	remarks
171	D848	1-		-0.05	
311		-----		-----	
323	D848	-1		-0.05	
657	D848	1-		-0.05	
663	D848	No. 1		0.29	
847	D848	No.1-		-0.05	
859	D848	1_		-0.05	
963	D848	1-		-0.05	
1081		-----		-----	
1669		-----		-----	

normality		unknown
n		7
outliers		0
mean (n)		0.79 (1-)
st.dev. (n)		0.094
R(calc.)		0.26
st.dev.(D848:18)	*)	0.736
R(D848:18)	*)	2.06

*) precision data of Benzene is used

In the calculation of the mean, standard deviation, reproducibility and for 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).





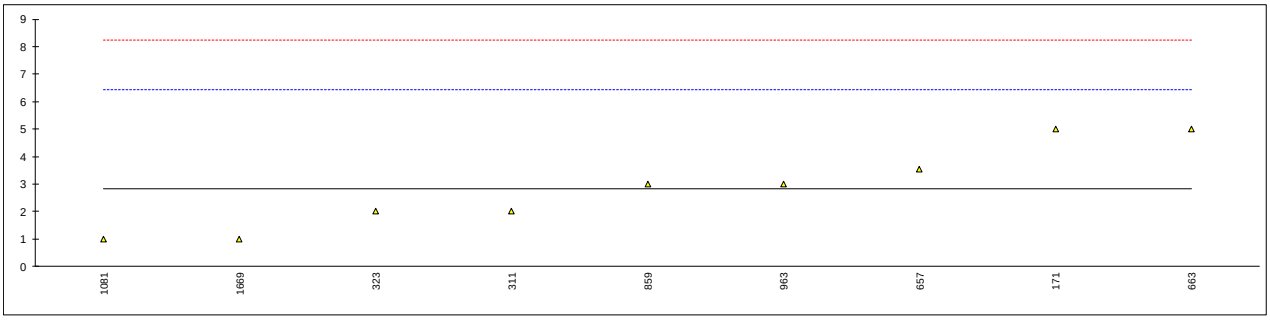
Determination of Appearance on sample #19022

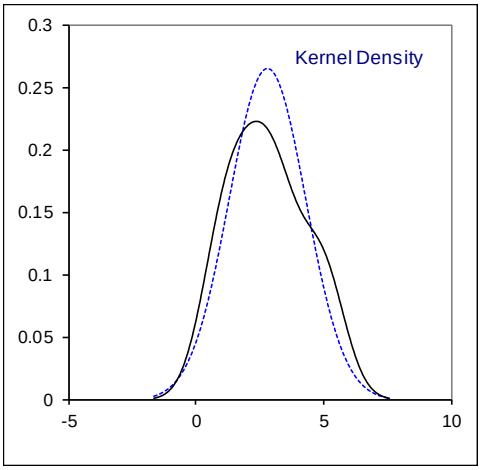
lab	method	value	mark	z(targ)	remarks
171	E2680	Pass		-----	
311	E2680	pass		-----	
323	E2680	clear & bright		-----	
657	E2680	Pass		-----	
663	E2680	Free of Haze, Particulates or suspended matters		-----	
847	Visual	clear and bright		-----	
859	E2680	Clear&Bright		-----	
963	Visual	pass		-----	
1081	In house	B/C		-----	
1669	Visual	Claro y Brillante		-----	
	n	10			
	mean (n)	Pass (B&C)			

Determination of Color (Pt/Co scale) on sample #19022

lab	method	value	mark	z(targ)	remarks
171	D1209	5		1.20	
311	D5386	2		-0.47	
323	D5386	2		-0.47	
657	D5386	3.54		0.39	
663	D5386	5		1.20	
847	D1209	<5		-----	
859	D5386	3		0.09	
963	D5386	3		0.09	
1081	D5386	1		-1.02	
1669	D1209	1		-1.02	

normality	OK
n	9
outliers	0
mean (n)	2.84
st.dev. (n)	1.502
R(calc.)	4.21
st.dev.(D5386:16)	1.800
R(D5386:16)	5.04
compare	
R(D1209:05(2011)	7

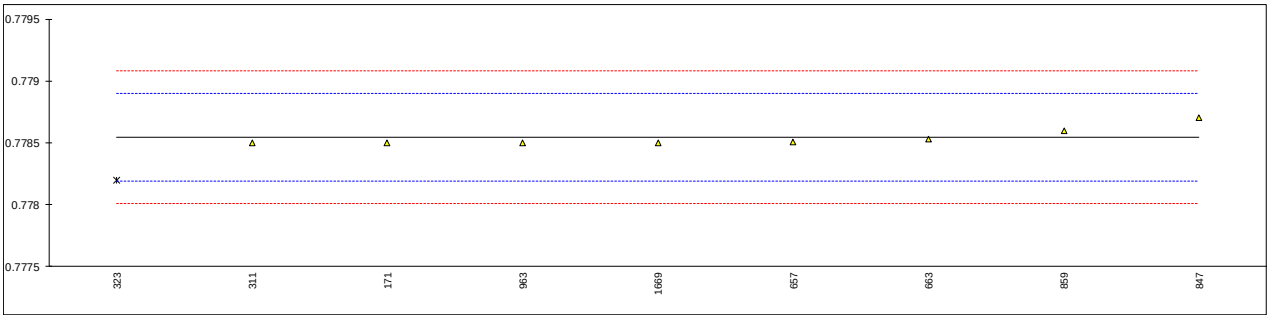


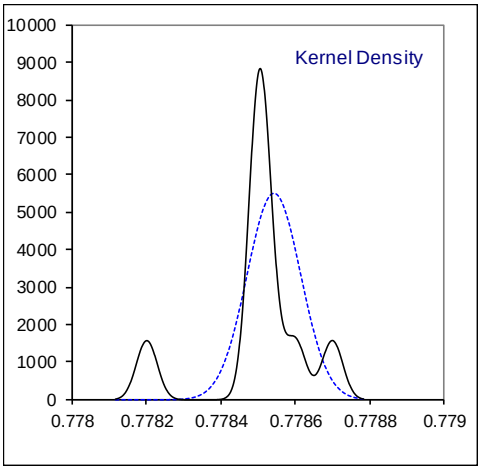


Determination of Density at 20°C on sample #19022; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.7785		-0.24	
311	D4052	0.7785		-0.24	
323	ISO12185	0.7782	D(0.01)	-1.92	
657	D4052	0.77851		-0.18	
663	D4052	0.77853		-0.07	
847	D4052	0.7787		0.88	
859	D4052	0.7786		0.32	
963	ISO12185	0.7785		-0.24	
1081		-----		-----	
1669	D4052	0.7785		-0.24	

normality	not OK
n	8
outliers	1
mean (n)	0.77854
st.dev. (n)	0.000072
R(calc.)	0.00020
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005



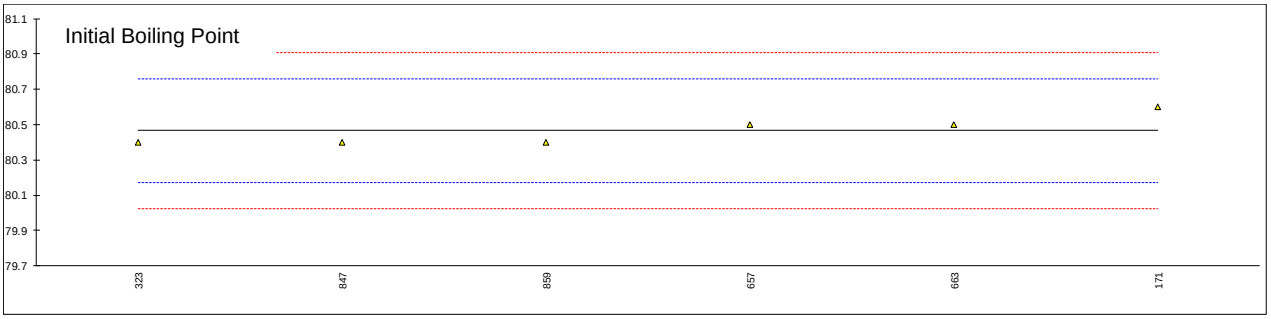


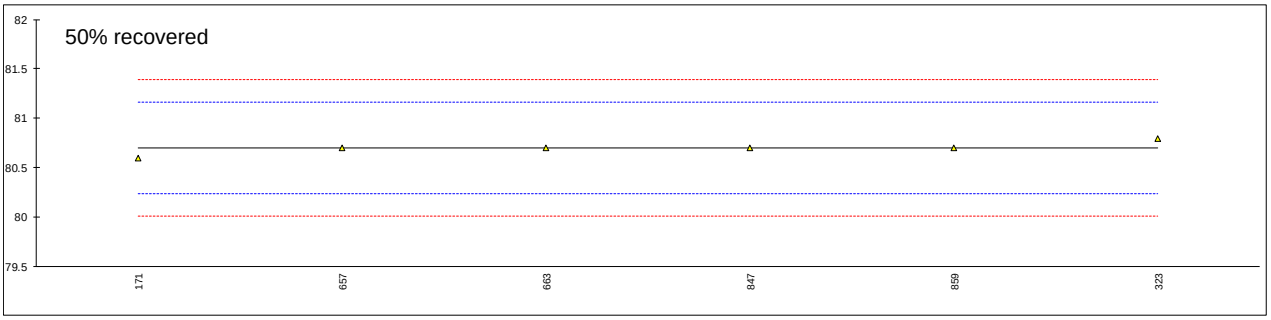
Determination of Distillation on sample #19022; results in °C

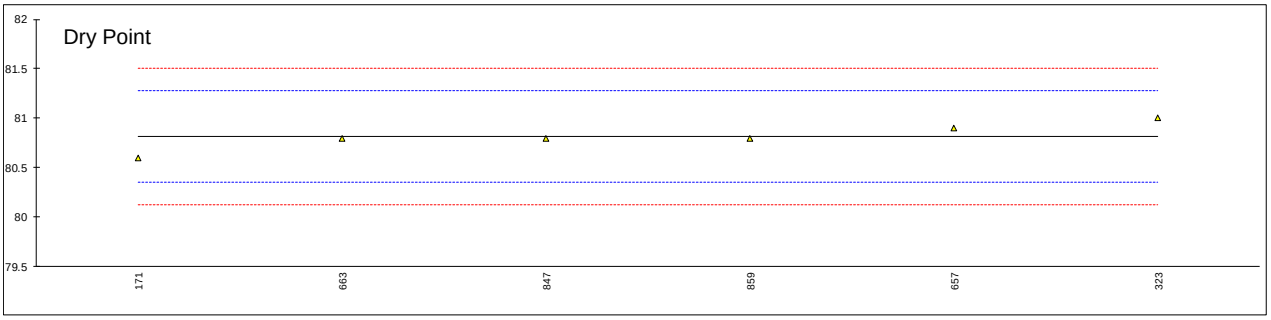
Lab	method	IBP	mark	z(targ)	50%	mark	z(targ)	DP	mark	z(targ)	range	mark
171	D850-automated	80.6		0.91	80.6		-0.43	80.6		-0.94	-----	
311		-----		-----	-----		-----	-----		-----	-----	
323	D850-manual	80.4		-0.45	80.8		0.43	81.0	C	0.79	0.6	C
657	D850-manual	80.5		0.23	80.7		0.00	80.9		0.36	0.4	
663	D850-automated	80.5		0.23	80.7		0.00	80.8		-0.07	0.3	
847	D850-manual	80.4		-0.45	80.7		0.00	80.8		-0.07	0.4	
859	D850-manual	80.4		-0.45	80.7		0.00	80.8		-0.07	0.4	
963		-----		-----	-----		-----	-----		-----	-----	
1081		-----		-----	-----		-----	-----		-----	-----	
1669		-----		-----	-----		-----	-----		-----	-----	
	normality	unknown			unknown			unknown				
	n	6			6			6				
	outliers	0			0			0				
	mean (n)	80.47			80.70			80.82				
	st.dev. (n)	0.082			0.063			0.133				
	R(calc.)	0.23			0.18			0.37				
	st.dev.(D850-M:18)	0.147			0.231			0.231				
	R(D850-M:18) *)	0.41			0.65			0.65				
	compare											
	R(D850-A:18) *)	0.58			0.16			0.46				

*) precision data of Toluene is used

Lab 323 first reported 81.4 and 1.0 respectively







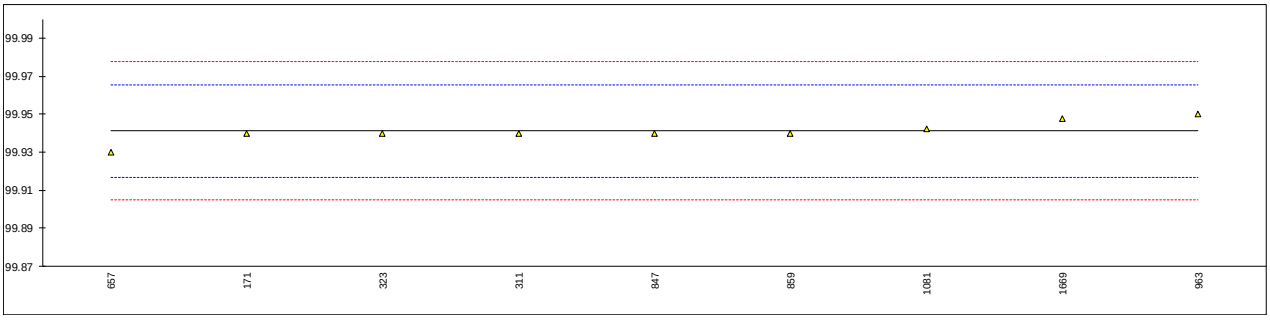
Determination of Freezing Point on sample #19022; results in °C

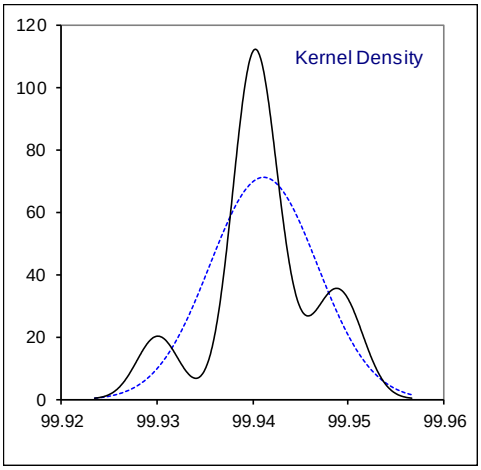
lab	method	value	mark	z(targ)	remarks
171		-----		-----	
311	D1493	6.35		-----	
323	D1016	6.4		-----	
657		-----		-----	
663		-----		-----	
847		-----		-----	
859		-----		-----	
963		-----		-----	
1081		-----		-----	
1669		-----		-----	
	n	2			
	mean (n)	6.4			

Determination of Purity of Cyclohexane on sample #19022; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D7266	99.94		-0.09	
311	D3054	99.94		-0.09	
323	D7266	99.94		-0.09	
657	D7266	99.9301		-0.91	
663		-----		-----	
847	D7266	99.94		-0.09	
859	D7266	99.94		-0.09	
963	D7266	99.95		0.73	
1081	D3054	99.9424695		0.11	
1669	D7266	99.9477	C	0.54	first reported 99.9743

normality	suspect
n	9
outliers	0
mean (n)	99.9411
st.dev. (n)	0.00561
R(calc.)	0.0157
st.dev.(D7266:13e1)	0.01214
R(D7266:13e1)	0.0340

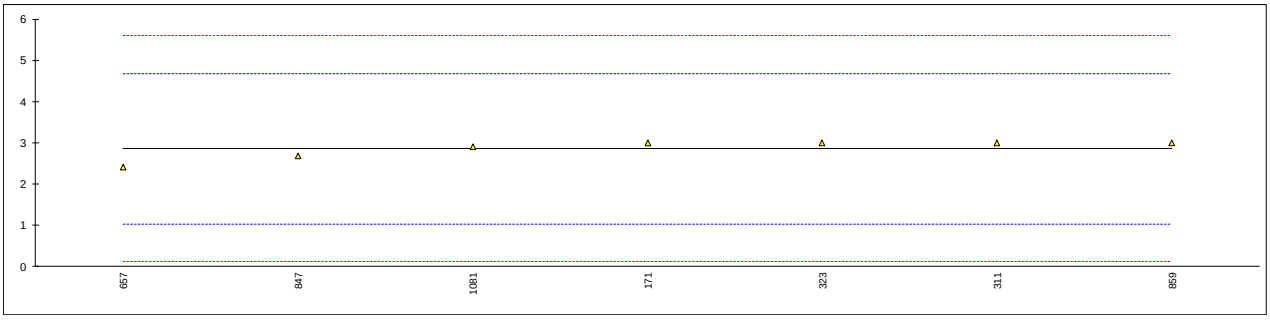


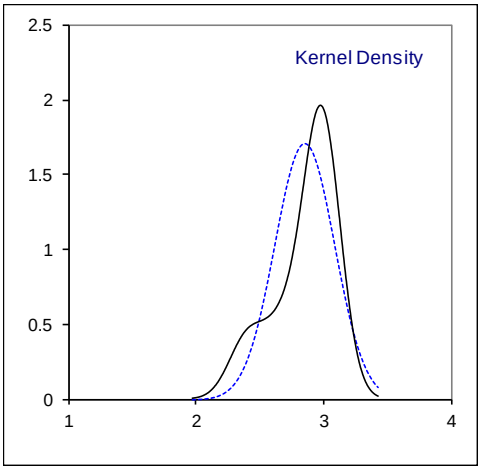


Determination of Benzene on sample #19022 in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7266	3		0.16	
311	D3054	3		0.16	
323	D7266	3		0.16	
657	D7266	2.4		-0.50	
663		-----		-----	
847	D7266	2.67		-0.20	
859	D7266	3		0.16	
963		-----		-----	
1081	D3054	2.90247		0.05	
1669	D7266	<5		-----	

normality	unknown
n	7
outliers	0
mean (n)	2.9
st.dev. (n)	0.23
R(calc.)	0.7
st.dev.(D7266:13e1)	0.91
R(D7266:13e1)	2.6

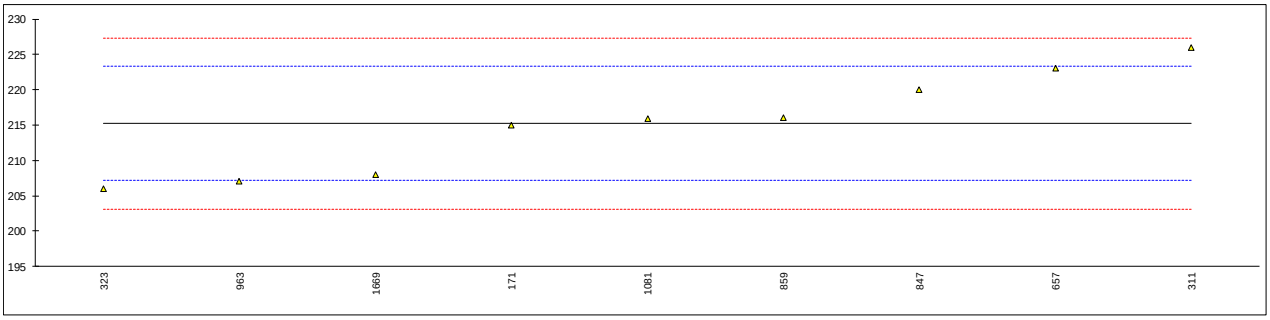


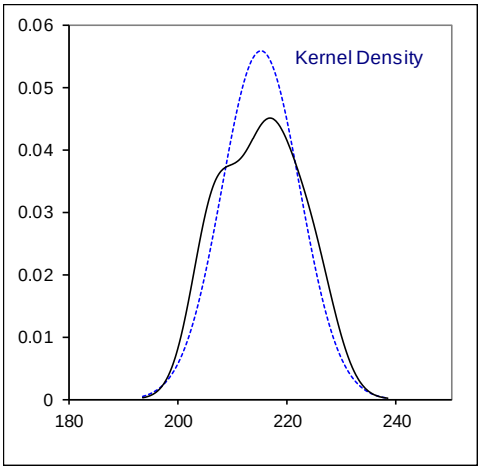


Determination of n-Hexane on sample #19022; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7266	215		-0.05	
311	D3054	226		2.68	
323	D7266	206		-2.29	
657	D7266	223.0		1.93	
663		-----		-----	
847	D7266	220		1.19	
859	D7266	216		0.20	
963	D7266	207		-2.04	
1081	D3054	215.97189		0.19	
1669	D7266	207.9425	C	-1.80	first reported 103.5

normality	OK
n	9
outliers	0
mean (n)	215.2
st.dev. (n)	7.13
R(calc.)	20.0
st.dev.(D7266:13e1)	4.03
R(D7266:13e1)	11.3

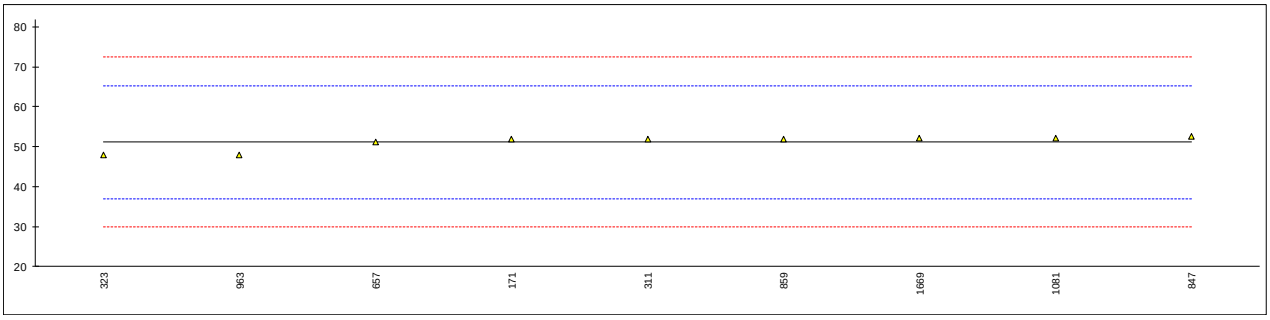


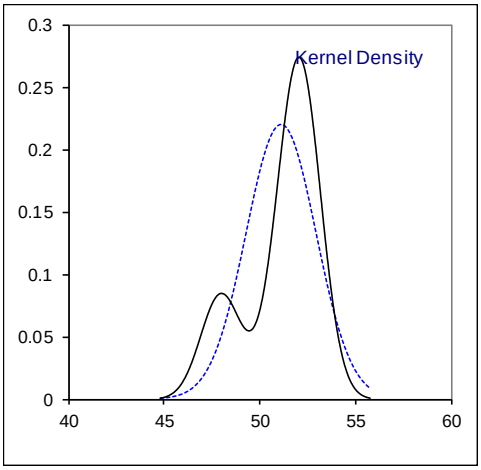


Determination of Methylcyclohexane on sample #19022; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7266	52		0.12	
311	D3054	52		0.12	
323	D7266	48		-0.44	
657	D7266	51.1		0.00	
663		-----		-----	
847	D7266	52.6		0.21	
859	D7266	52		0.12	
963	D7266	48		-0.44	
1081	D3054	52.21965		0.15	
1669	D7266	52.2132	C	0.15	first reported 22.8

normality	OK
n	9
outliers	0
mean (n)	51.1
st.dev. (n)	1.82
R(calc.)	5.1
st.dev.(D7266:13e1)	7.11
R(D7266:13e1)	19.9

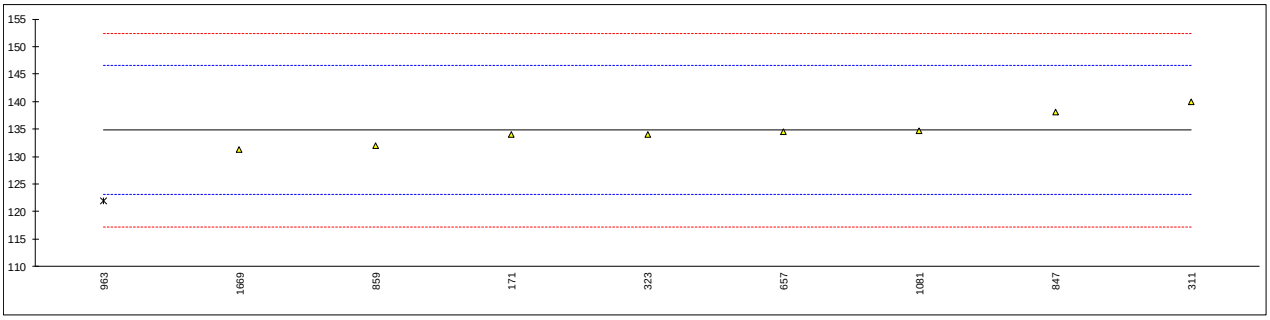


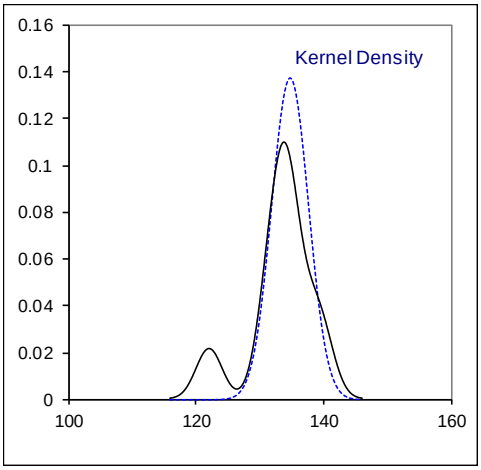


Determination of Methylcyclopentane on sample #19022; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7266	134		-0.14	
311	D3054	140		0.89	
323	D7266	134		-0.14	
657	D7266	134.5		-0.05	
663		-----		-----	
847	D7266	138		0.54	
859	D7266	132		-0.48	
963	D7266	122	D(0.05)	-2.19	
1081	D3054	134.75957		-0.01	
1669	D7266	131.2387	C	-0.61	first reported 65.3

normality	OK
n	8
outliers	1
mean (n)	134.8
st.dev. (n)	2.91
R(calc.)	8.1
st.dev.(D7266:13e1)	5.86
R(D7266:13e1)	16.4

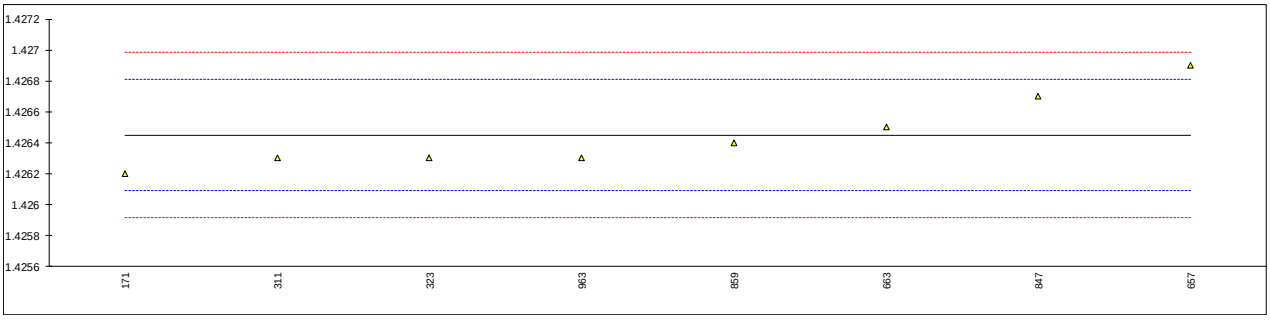


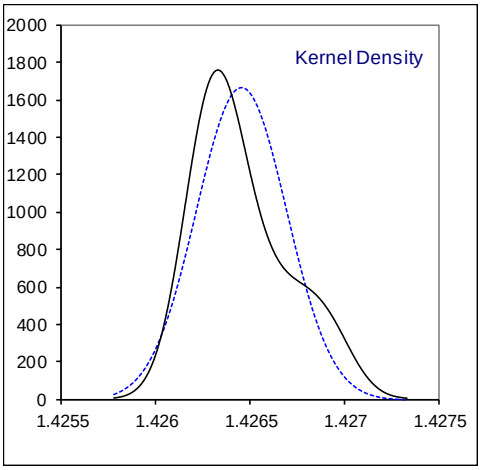


Determination of Refractive Index at 20°C on sample #19022;

lab	method	value	mark	z(targ)	remarks
171	D1218	1.4262		-1.40	
311	D1218	1.4263		-0.84	
323	D1218	1.4263		-0.84	
657	D1218	1.4269		2.52	
663	D1218	1.4265		0.28	
847	D1218	1.4267		1.40	
859	D1218	1.4264		-0.28	
963	D1218	1.4263		-0.84	
1081		-----		-----	
1669		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	1.42645
st.dev. (n)	0.000239
R(calc.)	0.00067
st.dev.(D1218:12)	0.000179
R(D1218:12)	0.0005





Determination of Sulfur on sample #19022; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7183	0.58		-----	
311	D7183	<0.5		-----	
323	D7183	<1		-----	
657	D7183	0.05		-----	
663	D5453	0		-----	
847	D5453	<1		-----	
859	D5453	<1		-----	
963	D5453	<1		-----	
1081	D7183	0.00		-----	
1669	D5453	<0.2		-----	
	n	10			
	mean (n)	<1			

Determination of UV Absorbance (10 mm cuvette) on sample #19022

Lab	cuvet size	280nm	mark	z(targ)	260nm	mark	z(targ)	240nm	mark	z(targ)	Pass/Fail
171		-----		-----	-----		-----	-----		-----	-----
311		-----		-----	-----		-----	-----		-----	-----
323	10 mm	0.01		-----	<0.01		-----	0.03		-----	Pass
657		-----		-----	-----		-----	-----		-----	-----
663		-----		-----	-----		-----	-----		-----	-----
847	10 mm	0.010		-----	0.017		-----	0.056		-----	Pass
859	10 mm	0.011		-----	0.019		-----	0.056		-----	Pass
963		-----		-----	-----		-----	-----		-----	-----
1081		-----		-----	-----		-----	-----		-----	-----
1669		-----		-----	-----		-----	-----		-----	-----
	n	3			3			3			3
	mean (n)	0.01			<0.02			<0.06			Pass

APPENDIX 2

Number of participants per country

1 lab in BELGIUM
2 labs in CHINA, People's Republic
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 = possibly an error in calculations
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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6 ISO 5725, parts 1-6, 1994
7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9 IP 367:84
10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee Technical brief, No 4, January 2001
14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, 127, 1359-1364 (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)