Results of Proficiency Test
Acetone
September 2017

CONTENTS

Appendices:

1   INTRODUCTION

Since 1999, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Acetone. During the annual proficiency testing program 2017/2018, it was decided to continue the round robin for the analysis of Acetone in accordance with the latest applicable version of the specification ASTM D329.
In this interlaboratory study 24 laboratories in 14 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2017 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 organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send a one litre glass bottle filled with Acetone (labelled #17155).
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/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 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 Organization, 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, approximately 47 litre, for sample #17155 was obtained from a local supplier. The original batch of 47 litre Acetone was spiked with 2.03 g Diacetone Alcohol and 1.03 g Mesityloxide. After homogenisation in a precleaned drum, 44 amber glass bottles of 1L were filled and labelled #17155. The homogeneity of the subsamples was checked by determination of Water in accordance with ASTM D1364 and Diacetone alcohol in accordance with an in house method on 4 stratified randomly selected samples.

	Water in mg/kg	Diacetone alcohol in mg/kg
sample #17155-1	2430	62
sample #17155-2	2410	61
sample #17155-3	2380	61
sample #17155-4	2390	61

Table 1: homogeneity test results of subsamples #17155

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the reference test methods and in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Water in mg/kg	Diacetone alcohol in mg/kg
r (observed)	62	1
reference test method	ASTM D1364:02	Horwitz
0.3 x R(reference test method)	81	4

Table 2: evaluation of homogeneity of subsamples #17155

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories 1 * 1 litre glass bottle, labelled #17155 was sent on August 16, 2017. A SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were requested to determine: Acidity, Aldehydes, Appearance, Chloride as Cl, Colour Pt/Co, Density at 20°C, Specific gravity 20/20°C, Distillation (IBP, MBP, DP and distillation range), Water Miscibility, Nonvolatile Matter, Permanganate Time Test at 25°C, Purity on dry basis, Diacetone alcohol, Mesityloxide, Methanol, Refractive Index at 20°C and Water on sample #17155 in accordance with specification ASTM D329:07(2013).

It was explicitly requested to treat the samples as if they were routine samples 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 reference 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. 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. 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 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:

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

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:

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

4   EVALUATION

In this proficiency test, some problems were encountered with the dispatch of the samples to Brazil, India and Vietnam due to custom clearance problems. From the total of 24 participants, three participants did not report any test result. In total 246 numerical results were reported. Observed were 17 outlying test results, which is 6.9%. 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 test methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in Appendix 3.

Unfortunately, a suitable standard test method, providing the precision data, is not available for all determinations. For the tests, that have no available precision data, the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D329) and an added designation for the year that the method was adopted or revised (e.g. D329:07). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D329:07(2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D329:07 will be used.

Acidity: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D1613:17.

Aldehydes: Five participants reported the test as pass, in accordance ASTM D329:07(2013), which describes a pass/fail test.

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #17155, which is bright and clear (or Pass).

Chloride, Inorganic: The Chloride content was near or below the detection limit. Therefore, no significant conclusions were drawn.

Colour: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1209:05(2011).

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

Specific Gravity 20/20°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. In total seven statistical outliers were observed. All three calculated reproducibilities (for IBP, MBP and DP) after rejection of the statistical outliers were in good agreement with the requirements of ASTM D1078:11 (automated and manual mode).

Water Miscibility: This determination was not problematic. All laboratories reported this test pass. The analytical method described in ASTM D1722:09 is a pass/fail test.

NVM: The NVM content was near or below the detection limit. Therefore, no significant conclusions were drawn.

Permanganate Time Test at 25°C: This determination may not problematic. No statistical outliers were observed. All participants agreed on a result far above 30 minutes. When a statistical evaluation is performed on the actually reported results, the calculated reproducibility is not at all in agreement with the (extrapolated) requirements of ASTM D1363:06(2011). However, as it is unknown whether a Permanganate Time Test result of >100 minutes is in the applicability range, it is therefore difficult to draw any conclusions. Therefore, no z-scores were calculated.

Purity on DB: No statistical outliers were observed. The calculated reproducibility is smaller than the calculated reproducibility of the 2015 PT on Acetone iis15C09 (0.016 %MM vs 0.028 %M/M).

Diacetone alcohol: This determination may be very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the estimated requirements, calculated using the Horwitz equation. The small number of reported test results may (partly) explain the large spread. The average recovery of Diacetone alcohol (theoretical increment of 68.6 mg Diacetone alcohol/kg) may be good: "less than 91%" (the actual blank Diacetone alcohol content is unknown).

Mesityloxide: This determination may be very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the estimated requirements, calculated using the Horwitz equation. The small number of reported test results may (partly) explain the large spread. The average recovery of Mesityloxide (theoretical increment of 30.3 mg Mesityloxide/kg) may be good: "less than 88%" (the actual blank Mesityloxide content is unknown).

Methanol: This determination may be very problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the estimated requirements based on the Horwitz equation.

Refractive index: This test was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1218:12.

Water: This determination was problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D1364:02(2012).

4.2   PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as acetic acid	mg/kg	17	10.7	7.1	14
Aldehydes	---	5	Pass	n.a.	n.a.
Appearance	---	20	Pass	n.a.	n.a.
Chloride as Cl	mg/kg	6	<1	n.a.	n.a.
Colour Pt/Co	---	15	3.4	3.8	7
Density at 20ºC	kg/L	19	0.7906	0.0002	0.0005
Specific Gravity 20/20ºC		16	0.7920	0.0002	0.0005
Initial Boiling Point	°C	16	56.0	0.2	0.9
Mid Boiling Point	°C	15	56.1	0.1	0.4
Dry Point	°C	16	56.4	0.3	0.6
Miscibility with water	---	17	Pass	n.a.	n.a.
Nonvolatile Matter	mg/100 mL	15	0.4	0.7	(0.2)
Permanganate Time Test at 25°C	min	10	102	57	(26)
Purity on dry basis	%M/M	12	99.971	0.016	n.a.
Diacetone alcohol	mg/kg	11	62	30	15
Mesityloxide	mg/kg	11	27	15	7
Methanol	mg/kg	14	186	84	38
Refractive Index	---	14	1.3588	0.0004	0.0005
Water	mg/kg	13	2379	335	270

Table 3: Reproducibilities for sample #17155

*) Values between brackets are close or below the limit of detection

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

4.3   COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2017 WITH PREVIOUS PTS

	September 2017	September 2015	September 2013	September 2011
Number of reporting labs	21	21	21	18
Number of results reported	246	250	273	198
Statistical outliers	17	10	7	3
Percentage outliers	6.9%	4.0%	2.6%	1.5%

Table 4: comparison of summary data with previous proficiency tests.

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

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

Determination	September 2017	September 2015	September 2013	September 2011
Acidity as acetic acid	++	++	++	+
Chloride as Cl	n.e.	n.e.	n.e.	n.e.
Colour Pt/Co	++	++	++	++
Density at 20ºC	++	++	++	++
Specific gravity 20/20ºC	++	++	+	n.e.
Distillation	++	++	++	++
Nonvolatile Matter	(--)	(--)	++	++
Permanganate Time Test	(--)	(--)	(--)	n.e.
Diacetone alcohol	--	-	--	--
Mesityloxide	--	-	+/-	n.e.
Methanol	--	--	-	+
Refractive Index	+	+/-	+	+/-
Water	-	-	+/-	--

Table 5: comparison determinations against the standard

*) Consensus values between brackets were close or below the limit of detection

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

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

APPENDIX 1

Determination of Acidity on sample #17155; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	D1613	9.1		-0.31	
171	D1613	8		-0.53	
174	D1613	11.3		0.13	
311	D1613	11		0.07	
323	D1613	8		-0.53	
343	D1613	22.8	DG(0.01)	2.43	
345	D1613	15		0.87	
349	D1613	8.4		-0.45	
444	D1613	21.2	DG(0.01)	2.11	
445	D1613	14		0.67	
551		-----		-----	
557		-----		-----	
657	D1613	8		-0.53	
786	D1613	12		0.27	
840	D1613	12.2		0.31	
886		-----		-----	
902	D1613	12		0.27	
913		-----		-----	
962		-----		-----	
963	D1613	13.2		0.51	
1016	D1613	10.8		0.03	
1429	D1613	5.7		-0.99	
1689	D1613	9.75		-0.18	
1974	D1613	13		0.47	

normality	OK
n	17
outliers	2
mean (n)	10.67
st.dev. (n)	2.533
R(calc.)	7.09
R(D1613:17)	14

Kernel Density
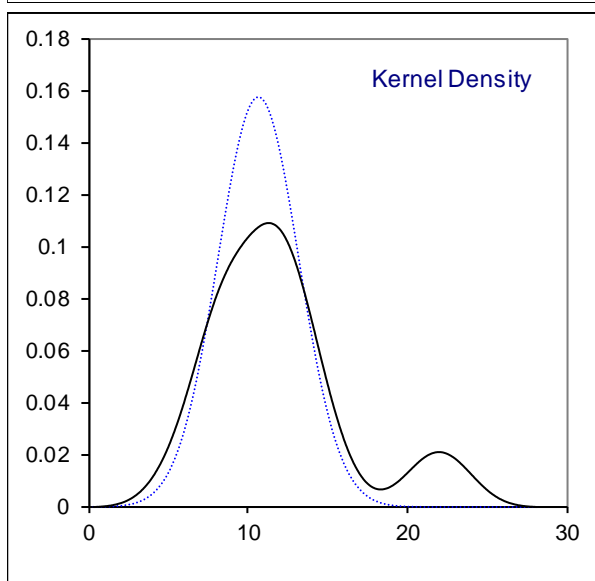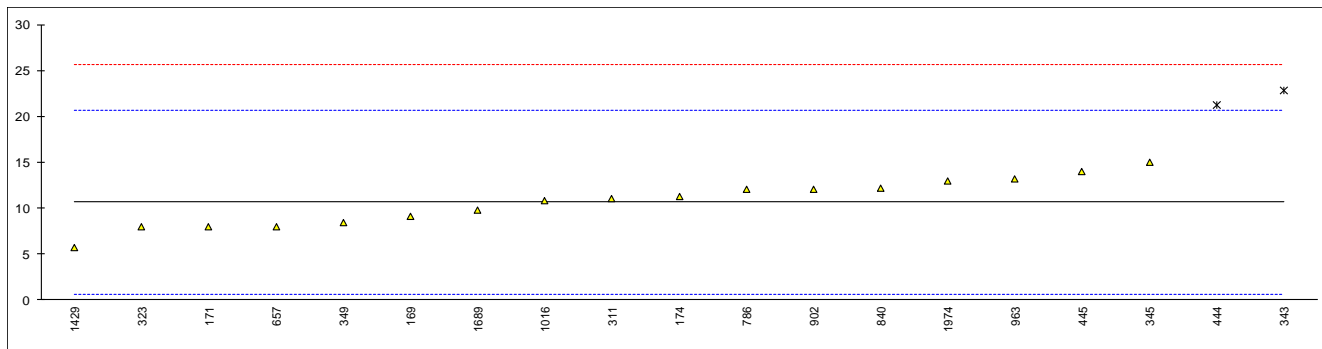
Determination of Aldehydes on sample #17155;

lab	method	value	mark	z(targ)	remarks
169	D329	Pass		-----	
171	D329	Pass		-----	
174		-----		-----	
311		-----		-----	
323	D329	PASS		-----	
343	D329	Pass		-----	
345		-----		-----	
349		-----		-----	
444		-----		-----	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657		-----		-----	
786		-----		-----	
840		-----		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1016		-----		-----	
1429		-----		-----	
1689		-----		-----	
1974	D329	pass		-----	

normality	unknown
n	5
outliers	n.a.
mean (n)	Pass
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D329:07)	n.a.

Determination of Appearance on sample #17155;

lab	method	value	mark	z(targ)	remarks
169	Visual	BC&FSM		-----	
171	E2680	Pass		-----	
174	Visual	Clear&Bright		-----	
311	E2680	pass		-----	
323	D4176	C&B		-----	
343	Visual	Clear&Bright		-----	
345	Visual	pass		-----	
349	E2680	pass		-----	
444	E2680	Pass		-----	
445	E2680	Pass		-----	
551		-----		-----	
557		-----		-----	
657	E2680	Clear&Bright		-----	
786	E2680	Pass		-----	
840	E2680	Pass		-----	
886		-----		-----	
902	E2680	Pass		-----	
913		-----		-----	
962	D4176	Pass		-----	
963	E2680	Pass		-----	
1016	In house	Pass		-----	
1429	E2680	Clear & Bright		-----	
1689	E2680	Pass		-----	
1974	D4176	Clear,bright&free		-----	

normality	unknown
n	20
outliers	n.a.
mean (n)	Pass
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit)	n.a.

Determination of Chloride Inorganic as Cl on sample #17155; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171	IMPCA002	<0.25		-----	
174		-----		-----	
311	INH-158	<0.20		-----	
323	E2469	<1		-----	
343		-----		-----	
345		-----		-----	
349		-----		-----	
444		-----		-----	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657		-----		-----	
786	IMPCA002	<0.25		-----	
840	IMPCA002	<0.1		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1016		-----		-----	
1429	ISO6227	<0.5		-----	
1689		-----		-----	
1974		-----		-----	

normality	unknown
n	6
outliers	n.a.
mean (n)	<1
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit)	n.a.

Determination of Colour as Pt/Co on sample #17155;

lab	method	value	mark	z(targ)	remarks
169	D1209	<5		-----	
171	D1209	5		0.66	
174	D1209	5		0.66	
311	D1209	<5		-----	
323	D1209	<5		-----	
343	D1209	4		0.26	
345	D1209	2		-0.54	
349	D5386	2		-0.54	
444	D5386	3.4		0.02	
445	D6045	2		-0.54	
551		-----		-----	
557		-----		-----	
657	D1209	5		0.66	
786	D1209	5		0.66	
840	D1209	3		-0.14	
886	D1209	< 5		-----	
902	D5386	3		-0.14	
913		-----		-----	
962	D1209	2		-0.54	
963	D1209	2		-0.54	
1016	D1209	2		-0.54	
1429	D1209	<5		-----	
1689		-----		-----	
1974	D1209	5		0.66	

normality	OK
n	15
outliers	0
mean (n)	3.4
st.dev. (n)	1.34
R(calc.)	3.8
R(D1209:05)	7

Kernel Density
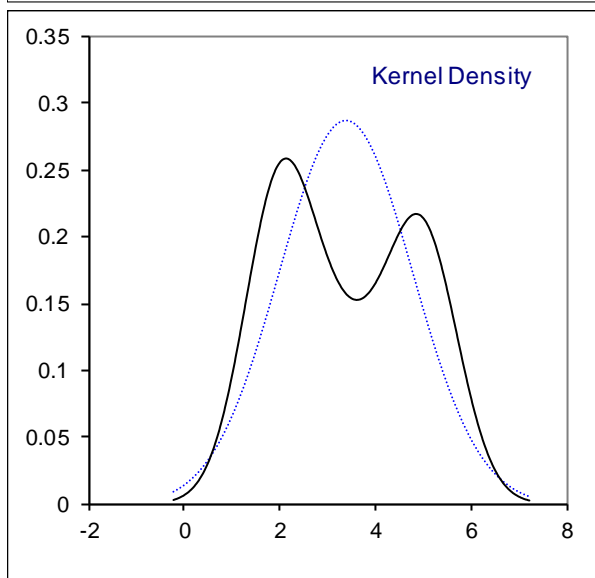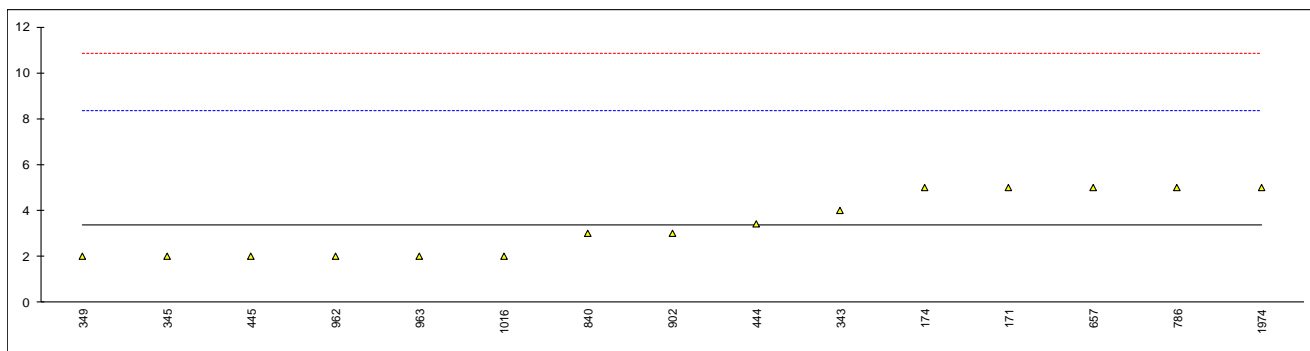
Determination of Density at 20°C on sample #17155; results in kg/L

lab	method	value	mark	z(targ)	remarks
169	D4052	0.7907		0.46	
171	D4052	0.7906		-0.10	
174	D4052	0.79074		0.69	
311	D4052	0.79057		-0.27	
323	D4052	0.7905		-0.66	
343	D4052	0.7906		-0.10	
345	D4052	0.7907	C	0.46	First reported 790.7
349		-----		-----	
444	D4052	0.7906		-0.10	
445	D4052	0.7906		-0.10	
551		-----		-----	
557		-----		-----	
657	D4052	0.79067		0.29	
786	D4052	0.7907		0.46	
840	D4052	0.79065		0.18	
886	D4052	0.7906		-0.10	
902	D4052	0.7906		-0.10	
913		-----		-----	
962	D4052	0.7906		-0.10	
963	D4052	0.7906		-0.10	
1016	ISO12185	0.7906		-0.10	
1429	D4052	0.7905		-0.66	
1689	ISO3675	0.7906		-0.10	
1974		-----		-----	

normality	OK
n	19
outliers	0
mean (n)	0.79062
st.dev. (n)	0.000063
R(calc.)	0.00018
R(ISO12185:96)	0.0005
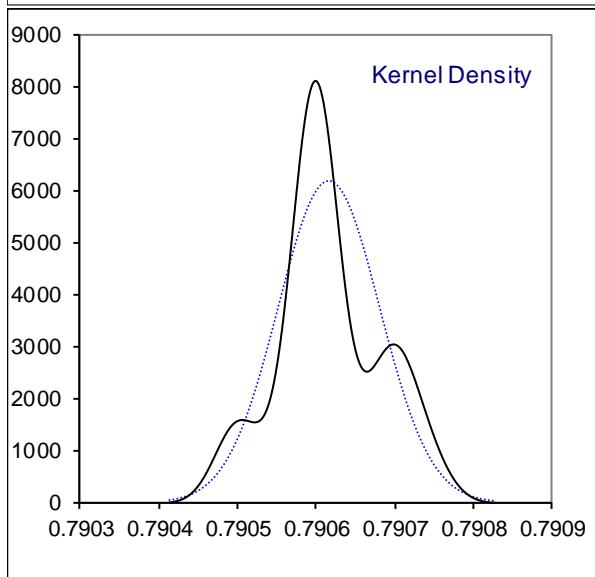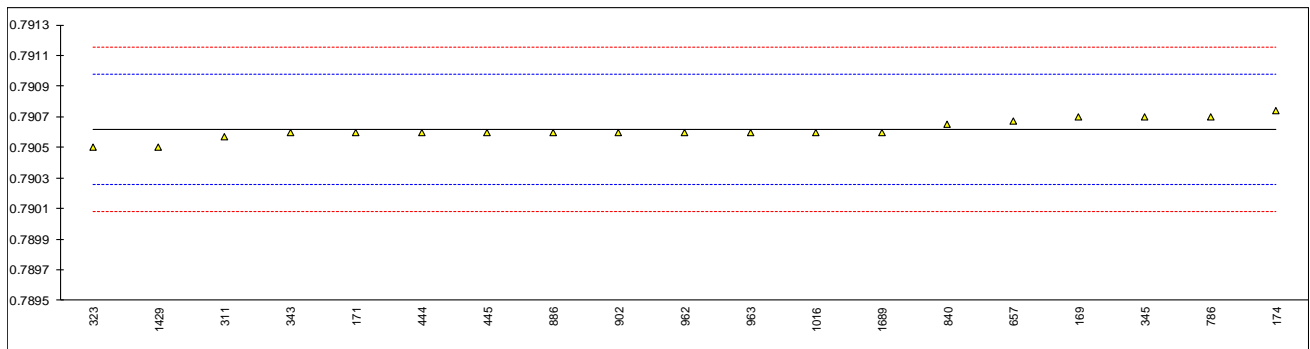
Determination of Specific Gravity 20/20 °C on sample #17155;

lab	method	value	mark	z(targ)	remarks
169	D4052	0.7921		0.43	
171	D4052	0.7920		-0.13	
174	D4052	0.7921		0.43	
311	D4052	0.7920		-0.13	
323	D4052	0.7919		-0.69	
343	D4052	0.7921		0.43	
345	D4052	0.7921		0.43	
349		-----		-----	
444	D4052	0.7920		-0.13	
445	D4052	0.7920		-0.13	
551		-----		-----	
557		-----		-----	
657	D4052	0.79209		0.38	
786		-----		-----	
840	D4052	0.79207		0.27	
886	D4052	0.7920		-0.13	
902	D4052	0.7920		-0.13	
913		-----		-----	
962	D4052	0.7920		-0.13	
963	ISO12185	0.7920		-0.13	
1016		-----		-----	
1429	D4052	0.7919		-0.69	
1689		-----		-----	
1974		-----		-----	

normality	OK	
n	16	
outliers	0	
mean (n)	0.79202	
st.dev. (n)	0.000066	
R(calc.)	0.00018	
R(ISO12185:96)	0.0005	
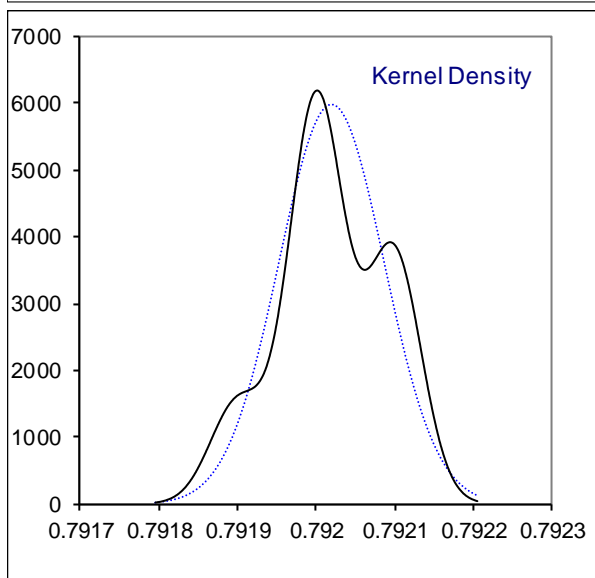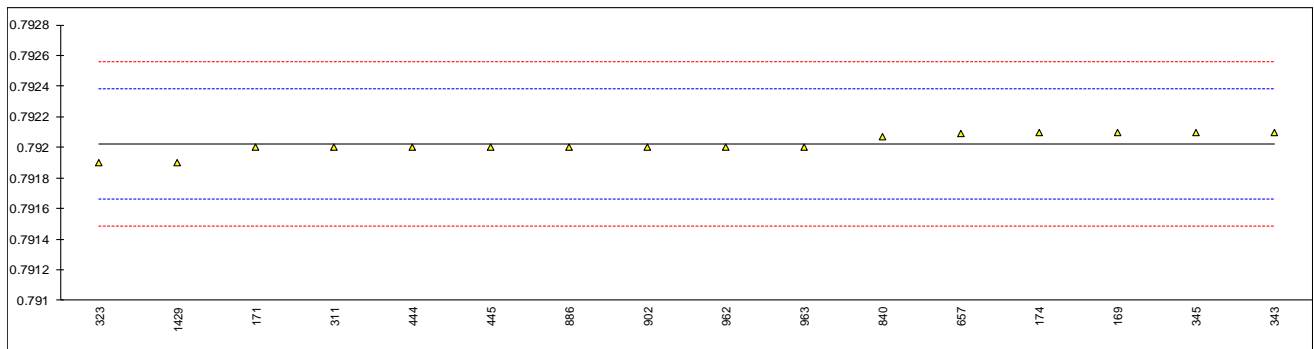
Determination of IBP, 50% recovered and DP at 760 mmHg on sample #17155; results in °C

lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	DP	mark	z(targ)	range
169	D1078-automated	55.9		-0.17	56.1		-0.10	56.4		0.13	0.5
171	D1078-automated	55.9		-0.17	56.1		-0.10	56.3		-0.33	0.4
174		-----		-----	-----		-----	-----		-----	-----
311	D1078-automated	55.9		-0.17	56.1		-0.10	56.4		0.13	0.5
323	D1078-manual	55.9		-0.17	56.1		-0.10	56.5		0.59	0.6
343	D1078-automated	55.9		-0.17	56.1		-0.10	56.2		-0.80	0.3
345	D1078-automated	55.8		-0.49	55.9	DG(1)	-1.56	56.2		-0.80	-----
349		-----		-----	-----		-----	-----		-----	-----
444	D1078-manual	56.1		0.47	56.1		-0.10	56.5		0.59	0.4
445	D1078-manual	56.0		0.15	56.1		-0.10	56.4		0.13	0.4
551		-----		-----	-----		-----	-----		-----	-----
557		-----		-----	-----		-----	-----		-----	-----
657	D1078-manual	56.0		0.15	56.1		-0.10	56.6		1.06	0.6
786	D1078-manual	56.0		0.15	56.1		-0.10	56.2		-0.80	0.2
840	D1078-automated	56.06		0.34	56.10		-0.10	56.45		0.36	0.39
886		55.6	DG(1)	-1.13	55.9	DG(1)	-1.56	56.0	DG(5)	-1.73	0.4
902	D1078-automated	55.9		-0.17	56.1		-0.10	56.3		-0.33	0.4
913		-----		-----	-----		-----	-----		-----	-----
962	D1078-automated	55.5	DG(1)	-1.46	55.7	G(1)	-3.02	56.0	DG(5)	-1.73	0.4
963	D1078-automated	55.9		-0.17	56.1		-0.10	56.4		0.13	0.5
1016		55.9		-0.17	56.1		-0.10	56.4		0.13	-----
1429	D1078-automated	56.0		0.15	56.2		0.63	56.3		-0.33	0.3
1689		-----		-----	-----		-----	-----		-----	-----
1974	D1078-automated	56.1		0.47	56.2		0.63	56.4		0.13	-----
	normality	OK			not OK			OK			
	n	16			15			16			
	outliers	2			3			2			
	mean (n)	55.95			56.11			56.37			
	st.dev. (n)	0.085			0.035			0.115			
	R(calc.)	0.24			0.10			0.32			
	R(D1078:11-A)	0.87			0.38			0.60			
comp	R(D1078:11-B)	0.60			0.36			0.73			
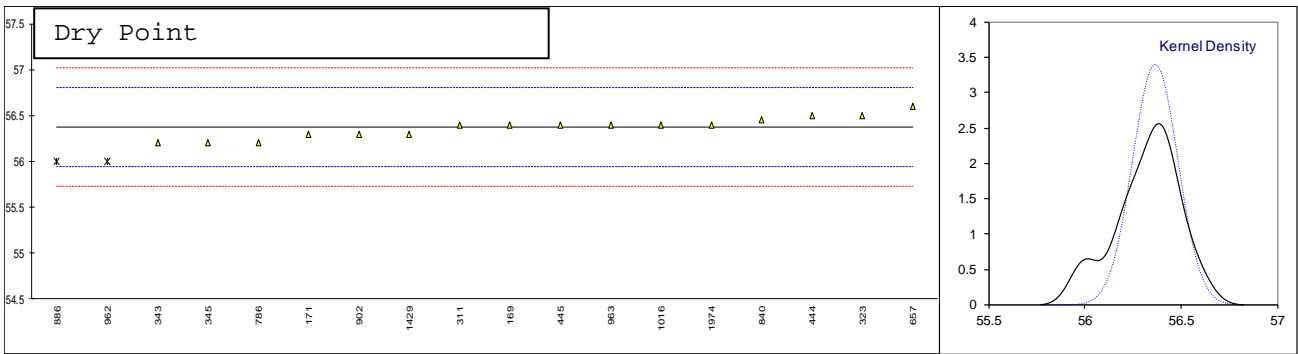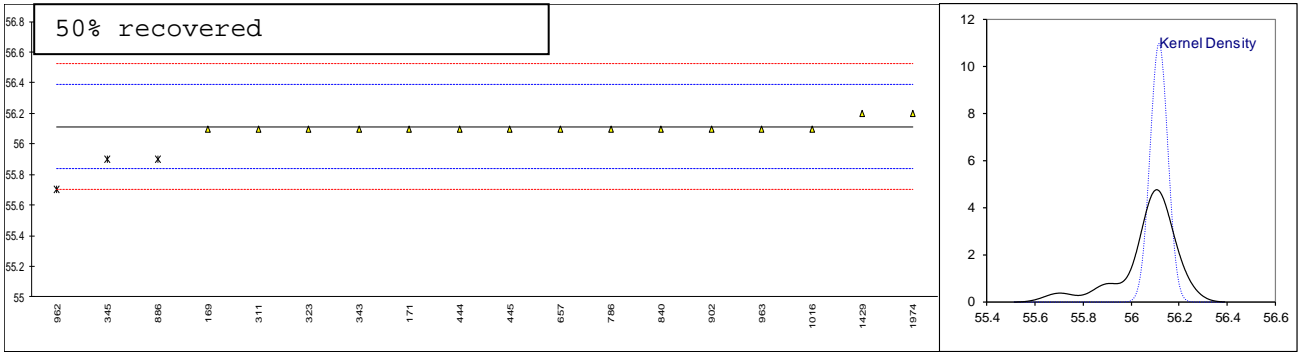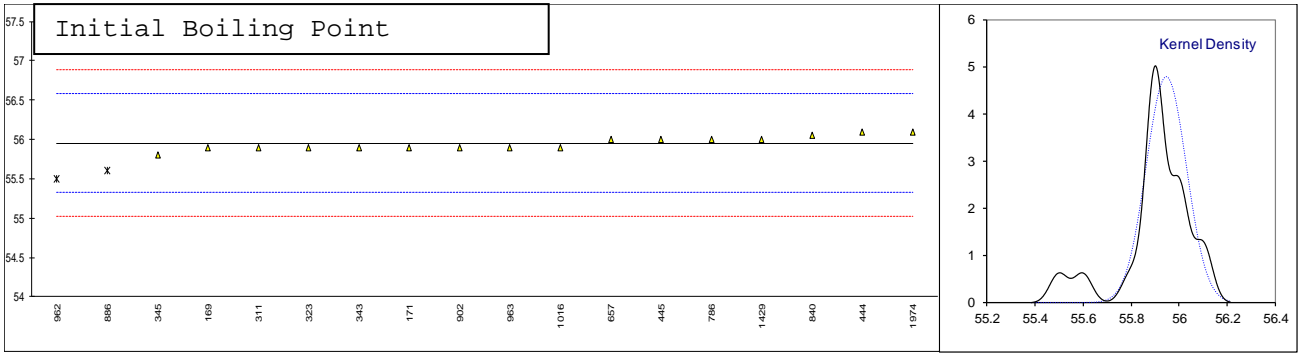
Determination of Miscibility with Water on sample #17155;

lab	method	value	mark	z(targ)	remarks
169	D1722	Pass		-----	
171	D1722	Passes		-----	
174	D1722	Pass		-----	
311	D1722	pass		-----	
323	D1722	PASS		-----	
343		-----		-----	
345		-----		-----	
349		-----		-----	
444	D1722	Pass		-----	
445	D1722	Pass		-----	
551		-----		-----	
557		-----		-----	
657	D1722	Pass		-----	
786	D1722	Pass		-----	
840	D1722	passes test		-----	
886		-----		-----	
902	D1722	Pass		-----	
913		-----		-----	
962	D1722	Pass		-----	
963	D1722	Pass		-----	
1016	D1722	Pass		-----	
1429	D1722	pass		-----	
1689	D1722	passes test		-----	
1974	D1722	Pass		-----	

|		
normality	unknown
n	17
outliers	n.a.
mean (n)	Pass
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit)	n.a.

Determination of Nonvolatile Matter on sample #17155; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
169	D1353	0.9		-----	
171	D1353	0.1		-----	
174	D1353	0.6		-----	
311	D1353	<1		-----	
323	D1353	<1		-----	
343	D1353	0.4		-----	
345	D1353	0.3	C	-----	First reported 5
349		-----		-----	
444	D1353	0		-----	
445	D1353	0.45		-----	
551		-----		-----	
557		-----		-----	
657	D1353	0.7		-----	
786	D1353	0.25		-----	
840	D1353	0.3		-----	
886		-----		-----	
902	D1353	0.2		-----	
913		-----		-----	
962		-----		-----	
963	D1353	0.3		-----	
1016	D1353	0.5		-----	
1429	D1353	0.7		-----	
1689	D1353	0.51		-----	
1974	D1353	<1		-----	

normality	OK
n	15
outliers	0
mean (n)	0.41
st.dev. (n)	0.244
R(calc.)	0.68
R(D1353:13)	(0.18)

Kernel Density
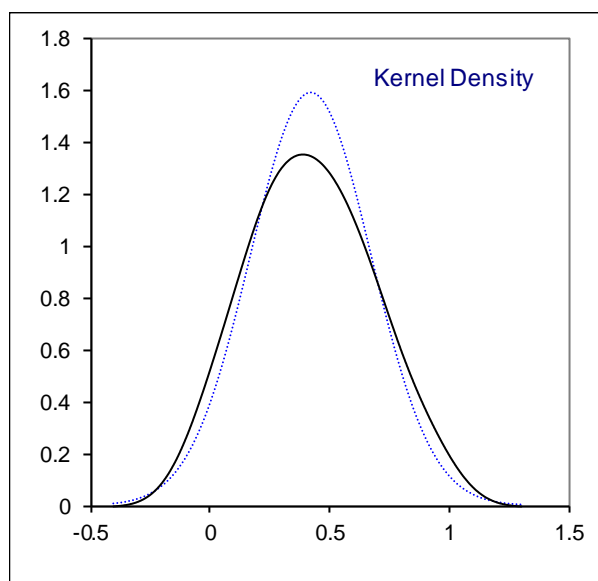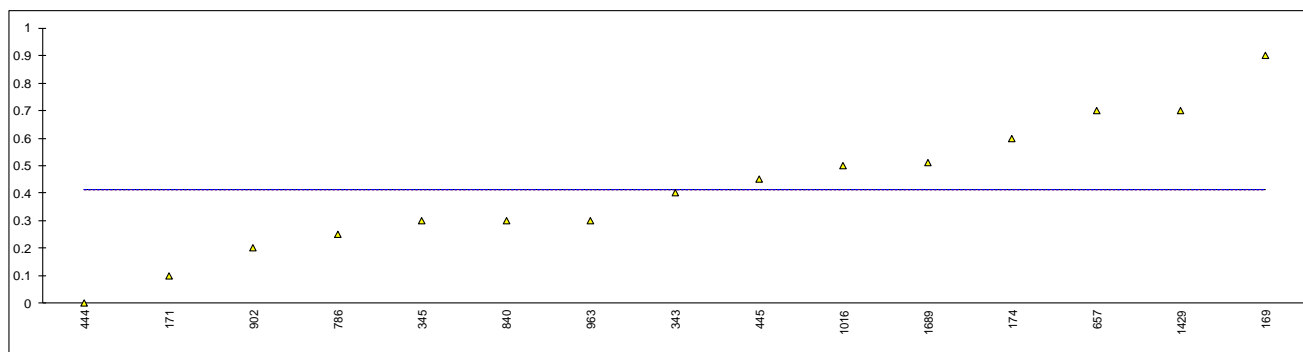
Determination of Permanganate Time Test at 25°C on sample #17155; results in minutes

lab	method	value	mark	z(targ)	remarks
169	D1363	>120		-----	
171	D1363	>60		-----	
174	D1363	130		-----	
311	D1363	120		-----	
323	D1363	>25		-----	
343	D1363	120		-----	
345	D1363	78		-----	
349		-----		-----	
444	D1363	80		-----	
445	D1363	77		-----	
551		-----		-----	
557		-----		-----	
657	D1363	> 30		-----	
786	D1363	110		-----	
840	D1363	95		-----	
886		-----		-----	
902	D1363	>30		-----	
913		-----		-----	
962		-----		-----	
963	D1363	120		-----	
1016	D1363	90		-----	
1429		-----		-----	
1689		-----		-----	
1974	D1363	>120		-----	

normality	OK	
n	10	
outliers	0	
mean (n)	102.0	
st.dev. (n)	20.27	
R(calc.)	56.8	
R(D1363:06)	(25.9)	

Kernel Density
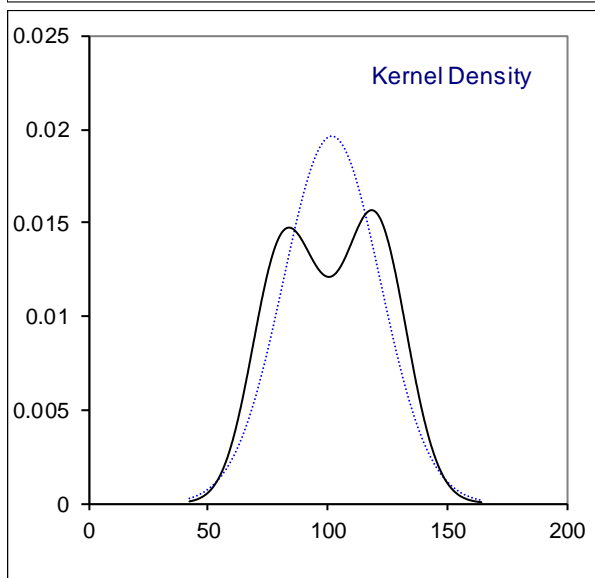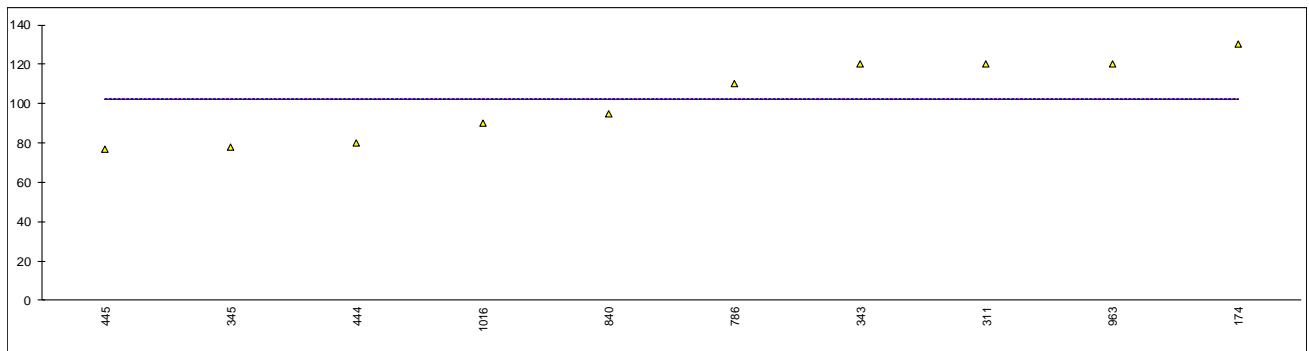
Determination of Purity by GC on Dry Basis on sample #17155, results in %M/M

lab	method	value	mark	z(targ)	remarks
169	INH-9	99.981		-----	
171		99.98		-----	
174		-----		-----	
311	INH-394	99.965		-----	
323		99.97		-----	
343	INH-CM	99.9760		-----	
345		-----		-----	
349	INH-034	99.9690		-----	
444	INH-001	99.968		-----	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657	INH-009	99.9622		-----	
786		-----		-----	
840	DIN55687	99.969		-----	
886		-----		-----	
902	INH-125	99.969		-----	
913		-----		-----	
962		-----		-----	
963	DIN55687	99.971		-----	
1016	DIN55687	99.966		-----	
1429		-----		-----	
1689		-----		-----	
1974		-----		-----	

normality	OK	
n	12	
outliers	0	
mean (n)	99.97051	
st.dev. (n)	0.005751	
R(calc.)	0.01610	
R(lit)	unknown	Compare R(iis15C09) = 0.0275

Kernel Density
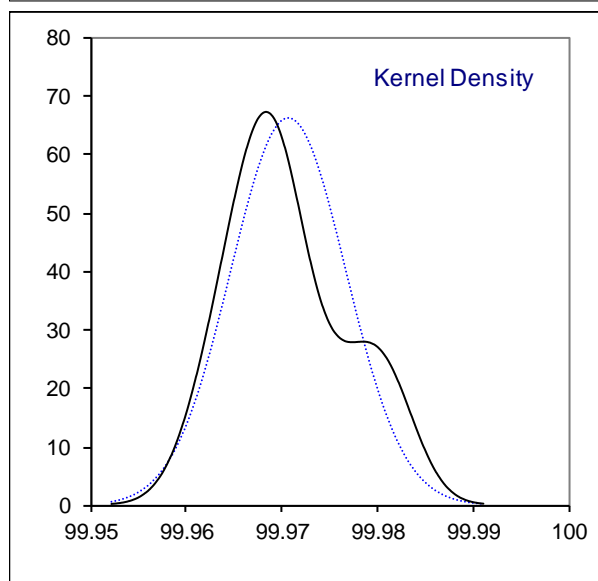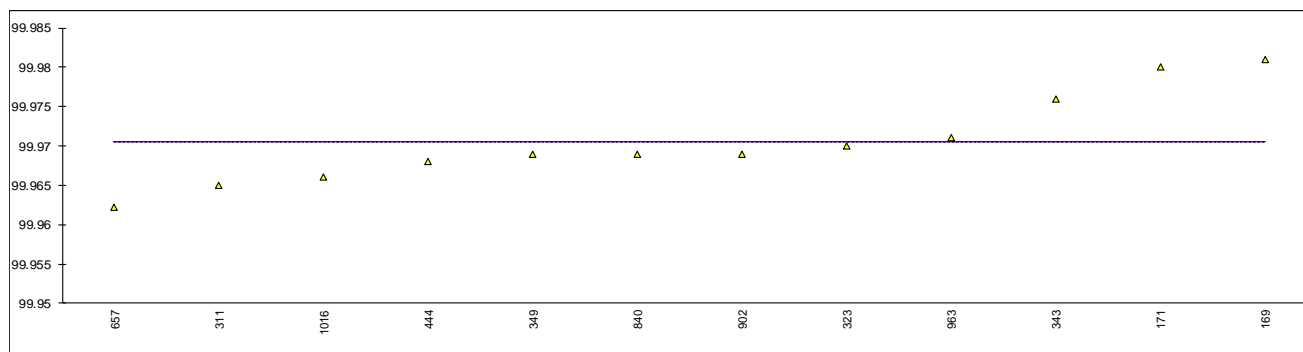
Determination of Diacetone alcohol on sample #17155, results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	INH-9	38.4		-4.47	
171		9	DG(0.05)	-9.96	
174		-----		-----	
311	INH-394	77		2.74	
323		70		1.44	
343	INH-CM	52		-1.93	
345		66		0.69	
349	INH-034	71	C	1.62	First reported 211
444	INH-001	67.2		0.91	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657	OINH-009	64		0.32	
786		-----		-----	
840	DIN55687	62.7		0.07	
886		-----		-----	
902		-----		-----	
913		-----		-----	
962		-----		-----	
963	DIN55687	52.59		-1.82	
1016	DIN55687	64.498		0.41	
1429		-----		-----	
1689		-----		-----	
1974	INH-2017	24	DG(0.05)	-7.16	

normality	suspect			
n	11			
outliers	2	Spike		
mean (n)	62.31	68.6		Recovery <91%
st.dev. (n)	10.808			
R(calc.)	30.26			
R(Horwitz)	14.99			

Kernel Density
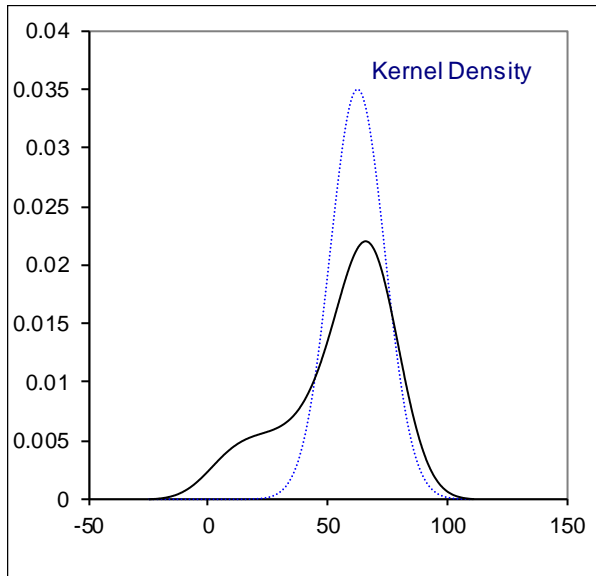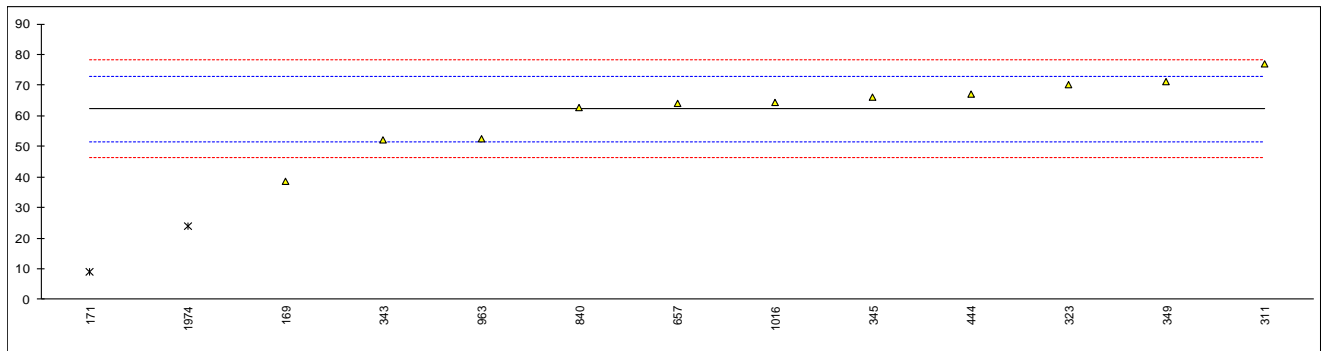
Determination of Mesityloxide on sample #17155, results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171		22	C	-1.81	First reported <1
174		-----		-----	
311	INH-394	30		1.25	
323		31		1.64	
343	INH-CM	21		-2.20	
345		30		1.25	
349	INH-034	28		0.49	
444	INH-001	23.6		-1.20	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657	INH-009	38		4.32	
786		-----		-----	
840	DIN55687	23.8		-1.12	
886		-----		-----	
902		-----		-----	
913		-----		-----	
962		-----		-----	
963	DIN55687	20.24		-2.49	
1016	DIN55687	26.391		-0.13	
1429		-----		-----	
1689		-----		-----	
1974	INH-2017	6	C,D(0.05)	-7.95	First reported 5

normality	OK			
n	11			
outliers	1	Spike		
mean (n)	26.73	30.3		Recovery <88%
st.dev. (n)	5.330			
R(calc.)	14.92			
R(Horwitz)	7.30			
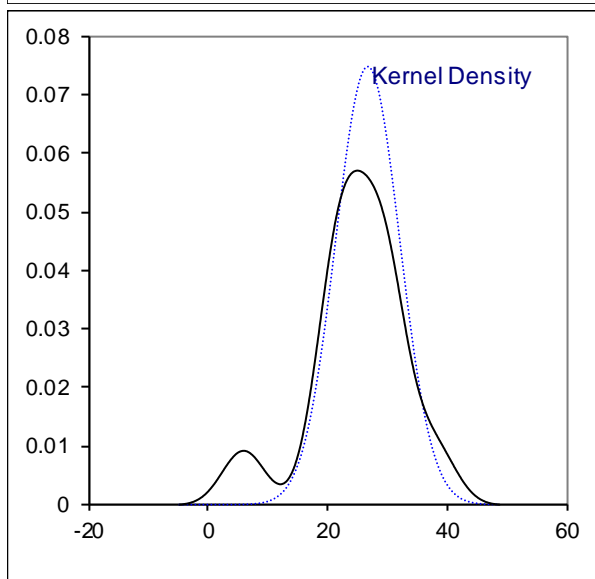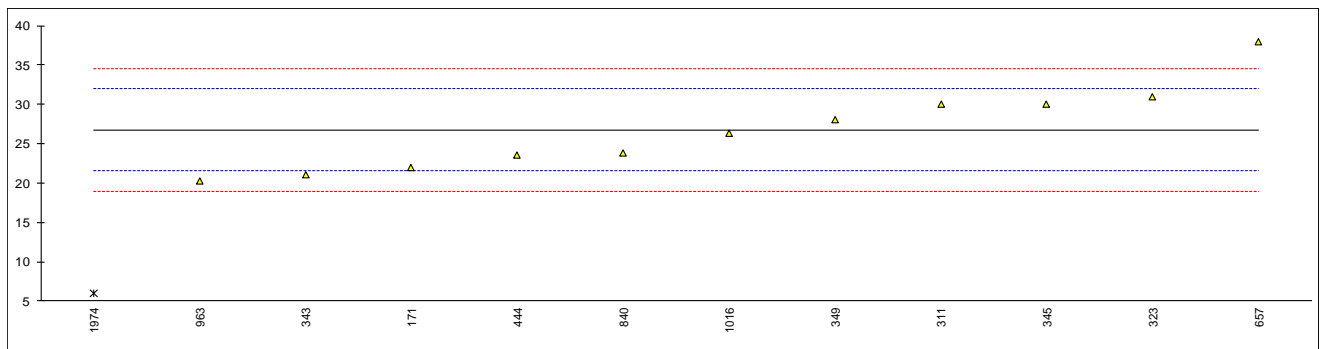
Determination of Methanol on sample #17155, results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	INH-9	144.2		-3.08	
171		131	C	-4.05	First reported <28
174		-----		-----	
311	INH-394	176		-0.73	
323		217		2.29	
343	INH-CM	164		-1.62	
345		197		0.82	
349	INH-034	211	C	1.85	First reported 71
444	INH-001	171.2		-1.09	
445		-----		-----	
551		-----		-----	
557		-----		-----	
657	INH-009	216		2.22	
786	GOST2768	220		2.51	
840	DIN55687	187.0		0.08	
886		-----		-----	
902	INH-125	200		1.04	
913		-----		-----	
962		-----		-----	
963	DIN55687	151.4		-2.55	
1016	DIN55687	217.371		2.32	
1429		-----		-----	
1689		-----		-----	
1974	INH-2017	114	C,G(0.05)	-5.31	First reported 95

normality	OK	
n	14	
outliers	1	
mean (n)	185.94	
st.dev. (n)	30.021	
R(calc.)	84.06	
R(Horwitz)	37.94	
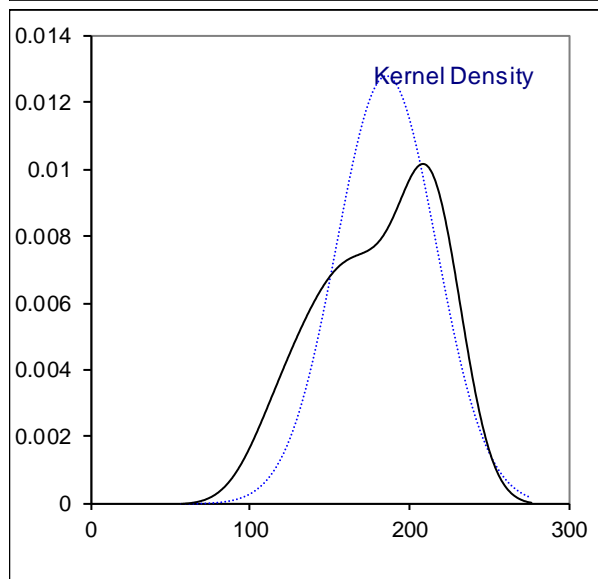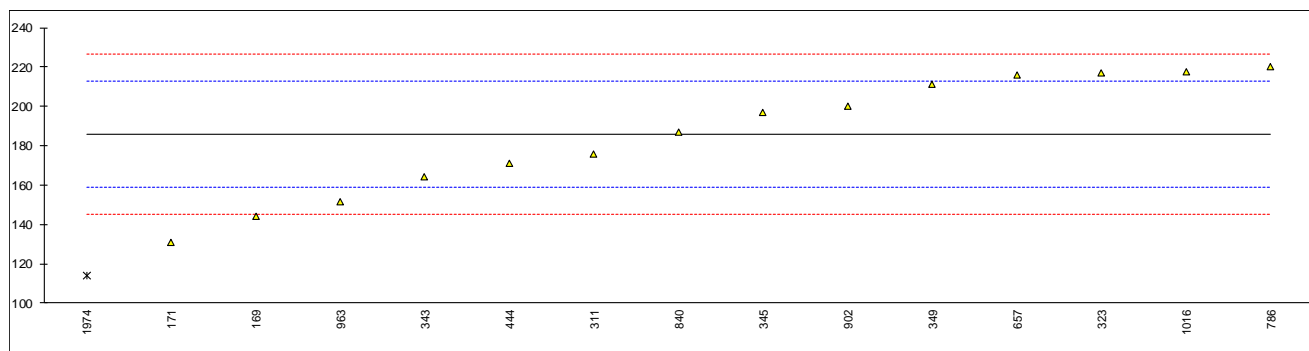
Determination of Refractive Index at 20°C on sample #17155;

lab	method	value	mark	z(targ)	remarks
169	D1218	1.3588		0.13	
171		1.3588		0.13	
174	D1218	1.35889		0.63	
311	D1218	1.3588		0.13	
323	D1218	1.3586		-0.99	
343		-----		-----	
345		-----		-----	
349		-----		-----	
444	D1218	1.3587		-0.43	
445	D1218	1.3590		1.25	
551		-----		-----	
557		-----		-----	
657	D1218	1.35880		0.13	
786	D1218	1.3587		-0.43	
840	D1218	1.3590		1.25	
886		-----		-----	
902		-----		-----	
913		-----		-----	
962		-----		-----	
963	D1218	1.35864		-0.77	
1016	D1218	1.3587		-0.43	
1429	D1218	1.35865		-0.71	
1689		-----		-----	
1974	D1218	1.3588		0.13	

normality	OK
n	14
outliers	0
mean (n)	1.35878
st.dev. (n)	0.000124
R(calc.)	0.00035
R(D1218:12)	0.0005

Kernel Density
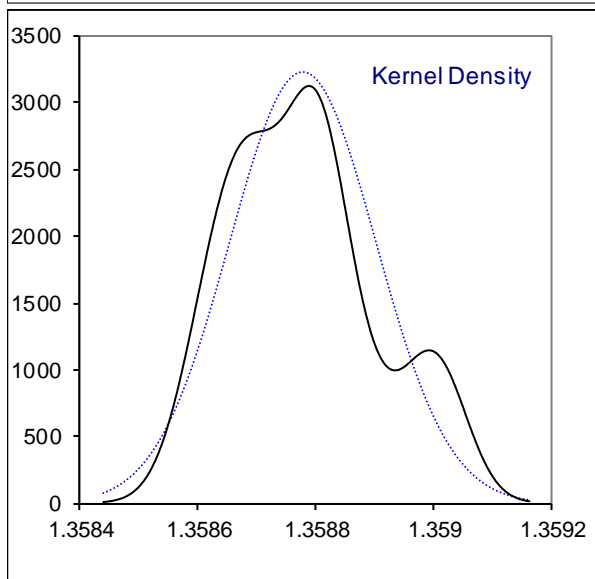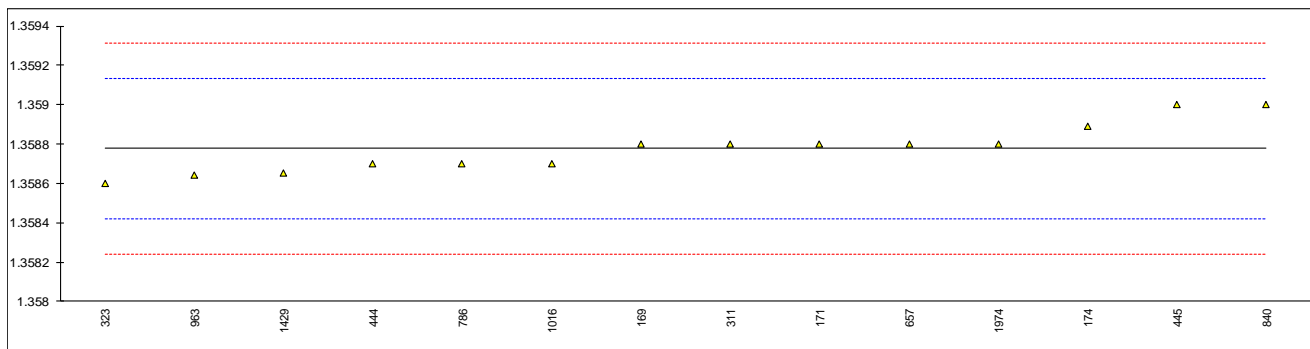
Determination of Water, titrimetric on sample #17155; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	D1364	2429		0.52	
171		1833	DG(0.05)	-5.66	
174	E203	2384		0.05	
311	D1364	2350		-0.30	
323	D1364	2403		0.25	
343	D1364	2440	C	0.63	First reported 244
345	D1364	2234		-1.50	
349	D1364	1950	G(0.05)	-4.45	
444	E203	2333	C	-0.48	First reported 0.2333
445	E203	1895	DG(0.05)	-5.02	
551		-----		-----	
557		-----		-----	
657	E1064	2330		-0.51	
786	D1364	2481		1.06	
840	D1364	2090		-3.00	
886		-----		-----	
902	D1364	2514		1.40	
913		-----		-----	
962		-----		-----	
963	D1364	3155	G(0.05)	8.05	
1016	D1364	2400		0.22	
1429	D1364	2540		1.67	
1689		-----		-----	
1974		-----		-----	

normality	suspect	
n	13	
outliers	4	
mean (n)	2379.1	
st.dev. (n)	119.62	
R(calc.)	334.9	
R(D1364:02)	270	
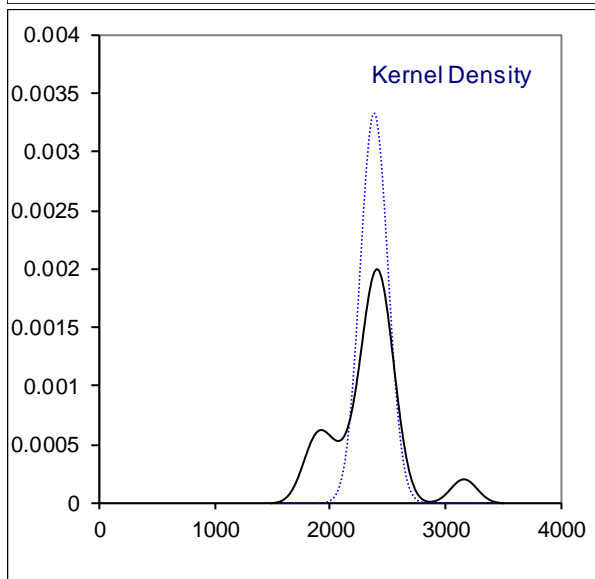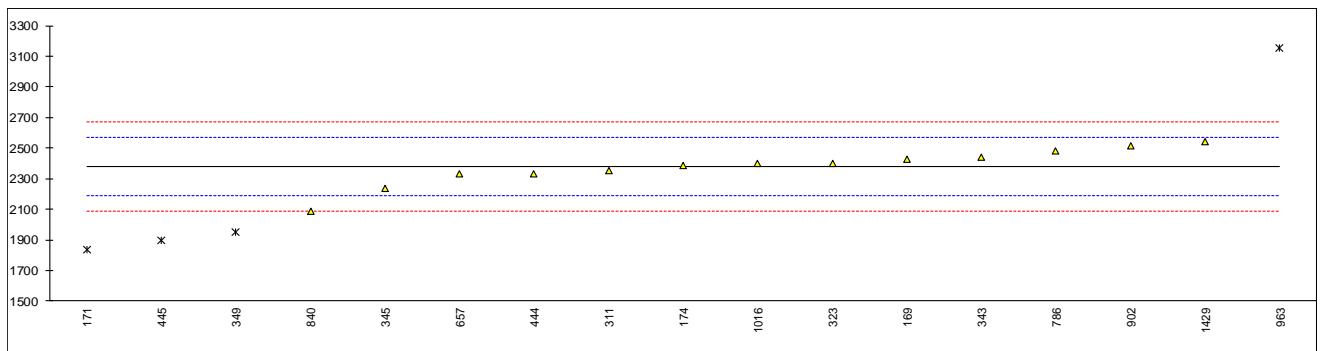
APPENDIX 2

Number of participants per country

1 lab in BELGIUM

2 labs in BRAZIL

1 lab in CHINA P.R. of

1 lab in INDIA

2 labs in NETHERLANDS

1 lab in RUSSIAN FEDERATION

2 labs in SAUDI ARABIA

1 lab in SINGAPORE

3 labs in SPAIN

1 lab in TAIWAN R.O.C.

1 lab in TURKEY

3 labs in UNITED KINGDOM

4 labs in UNITED STATES OF AMERICA.

1 lab in VIETNAM

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)/G(1)	= outlier in Grubbs' outlier test
G(0.05)/G(5)	= straggler in Grubbs' outlier test
DG(0.01)/DG(1)	= outlier in Double Grubbs' outlier test
DG(0.05)/DG(1)	= 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:

1   iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
2   ASTM E178:02
3   ASTM E1301:95(2003)
4   ISO 5725:86
5   ISO 5725, parts 1-6, 1994
6   ISO 13528:05
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
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