

**Results of Proficiency Test  
Acetic Acid  
February 2017**

**Organised by:** Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## **1 INTRODUCTION**

Since 2003, the Institute for Interlaboratory Studies organizes a proficiency test for Acetic Acid. During the annual proficiency test program of 2016/2017, it was decided to continue the proficiency test for the analysis of Acetic Acid in accordance with the latest applicable version of the product specification ASTM D3620. In this interlaboratory study, 25 laboratories in 16 different countries did register 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](http://www.iisnl.com).

## **2 SET UP**

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one bottle of 0.5L Acetic Acid (labelled #17002). The sample was spiked with Iron(III)Chloride.

Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluations.

### **2.1 ACCREDITATION**

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

### **2.2 PROTOCOL**

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

### **2.3 CONFIDENTIALITY STATEMENT**

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the

identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

## 2.4 SAMPLES

The necessary bulk material of Acetic Acid was obtained from a chemical producer. The approximately 25 litres of Acetic Acid was spiked with 250.7 mg Iron(III)Chloride.6H<sub>2</sub>O. After homogenisation, this material was divided over 43 amber glass bottles of 0.5 L and labelled #17002.

The homogeneity of subsamples #17002 was checked by determination of Chloride in accordance with an in-house test method and Density in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	<i>Chloride in mg/kg</i>	<i>Density at 20 °C in kg/l</i>
sample #17002-1	4.3	1.04938
sample #17002-2	4.2	1.04940
sample #17002-3	4.2	1.04942
sample #17002-4	4.2	1.04940
sample #17002-5	4.2	1.04941
sample #17002-6	4.2	1.04940
sample #17002-7	4.2	1.04940
sample #17002-8	4.2	1.04940

Table 1: homogeneity test results of subsamples #17002

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test method and the estimated reproducibility calculated using the Horwitz equation in agreement with the procedure of ISO 13528, Annex B2 in the next table;

	<i>Chloride in mg/kg</i>	<i>Density at 20 °C in kg/l</i>
r (observed)	0.10	0.00003
reference method	Horwitz	ISO12185:96
0.3*R (reference method)	0.46	0.00015

Table 2: evaluation of the repeatabilities of subsamples #17002

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

To each of the participating laboratories 1 \* 0.5 litre, labelled #17002 was sent on January 25, 2017. An SDS was added to the sample package.

## 2.5 STABILITY OF THE SAMPLES

The stability of Acetic Acid, 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 asked to determine Acetaldehyde, Appearance, Anorganic Chloride as Cl, Colour Pt/Co, Density at 20 °C, Formic Acid, Freezing Point, Iron as Fe, Nonvolatile Matter, Purity via Freezing Point, Purity via titration, Sulphate as SO<sub>4</sub> and Water.

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/sqs-iis/](http://www.kpmd.co.uk/sqs-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](http://www.iisnl.com).

## 3 RESULTS

During five weeks after sample dispatch, the results of the individual laboratories were gathered via the data entry portal [www.kpmd.co.uk/sqs-iis/](http://www.kpmd.co.uk/sqs-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 reanalyses). Additional or corrected test results are used for data analysis and original 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

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

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

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

## 3.2 GRAPHICS

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

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

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

### 3.3 Z-SCORES

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

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

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

The z-scores were calculated according to:

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

The  $Z_{(\text{target})}$  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 interlaboratory study, some problems were encountered with dispatch of the samples. Participants in Brazil and Saudi Arabia received the sample late or not at all. One participant reported after the final reporting date and three laboratories did not report any test results at all. Not all laboratories were able to perform all analyses requested. In total 152 numerical test results were reported by 22 participants. Observed were 5 outlying results, which is 3.3% of the total of numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “unknown” 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 test results are discussed per test. The specified test methods and requirements were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the reported data. The abbreviations, used in these tables, are listed in appendix 3.

For comparison of the results of this interlaboratory study, the requirements from the specification ASTM D3620:04 (2009) "Standard Specification for Glacial Acetic Acid" were used. Regrettably, for many determinations this specification is referring to ASTM E302:95 "Standard Test Methods for Monobasic Organic Acids", which was withdrawn already in 2001. As there was no replacement, this specification was used as reference method.

For the determination of the Purity by Titration, the method used for comparison is ASTM E301:94, which was also withdrawn, with no replacement, in 2001. However, no other useful standardised method is published yet. As there was no replacement, this withdrawn specification was used as reference method.

The target reproducibility used for the determination of the Purity by Freezing Point is calculated from the values in table 1 from ASTM E302 and the target reproducibility from ASTM E302.

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. D1209) and an added designation for the year that the method was adopted or revised (e.g. D1209:05). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1209:05 (2011)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

Acetaldehyde: Only four participants reported a numerical result. Therefore no significant conclusions were drawn. Please note that ASTM D2191 is meant for vinyl acetate.

Appearance: No analytical problems were observed. Most labs agreed about the appearance, which is bright, clear and free of suspended matter or pass in accordance with ASTM E2680. One lab reported "fails" (several fibres in the sample).

Anorganic Chloride: This determination may not be problematic. The calculated reproducibility is in agreement with the estimated reproducibility using the Horwitz equation. The average recovery of Anorganic Chloride



(theoretical increment of 4.35 mg/kg) may be good: "less than 95%". The actual blank concentration for Anorganic Chloride is unknown.

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

Density at 20 °C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96 and also with D4052:16.

Formic Acid: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D3546:05(2011).

Freezing Point: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the withdrawn method ASTM E302:95.

Iron as Fe: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM E394:15. The average recovery of Iron (theoretical increment of 2.28 mg/kg) may be good: "less than 96%". The actual blank concentration for Iron is unknown.

Nonvolatile Matter: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D1353:13.

Purity (from FP): Regretfully, no suitable reference method with precision data exists for this determination. Therefore, a target reproducibility was calculated out of table 1 of the withdrawn ASTM E302:95 and the reproducibility data of the withdrawn ASTM E302:95. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility limits.

Purity (titration): This determination may not be problematic. No statistical outlier was observed and the calculated reproducibility is in agreement with the requirements of ASTM E301:94.

Sulphate as SO<sub>4</sub>: Only two participants reported test results, of which one participant reported a numerical result. Therefore no significant conclusions were drawn.

Water: This determination was not problematic. No statistical outlier was observed and the calculated is in agreement with the requirements of the withdrawn method ASTM E302:95.

#### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R (lit)</i>
Acetaldehyde	mg/kg	8	<15	n.a.	n.a.
Appearance		19	Pass	n.a.	n.a.
Anorganic Chloride	mg/kg	8	4.1	0.3	1.5
Colour Pt/Co		19	11.7	4.9	7.0
Density at 20°C	kg/L	18	1.0494	0.0002	0.0005
Formic Acid	mg/kg	12	59	118	360
Freezing Point	°C	17	16.37	0.11	0.25
Iron as Fe	mg/kg	15	2.18	0.50	1.03
Nonvolatile Matter	mg/100 mL	12	2.0	1.2	0.8
Purity (Freezing Point)	%M/M	16	99.87	0.07	0.13
Purity (Titration)	%M/M	6	99.81	0.27	0.54
Sulphate as SO <sub>4</sub>	mg/kg	2	<1	n.a.	n.a.
Water	%M/M	20	0.105	0.018	0.050

Table 3: reproducibilities of tests on sample #17002

Without further statistical calculations, it can be concluded that for many 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 FEBRUARY 2017 WITH PREVIOUS PTS

	<i>February 2017</i>	<i>February 2015</i>	<i>February 2013</i>	<i>February 2011</i>	<i>March 2010</i>
Number of rep. participants	22	22	23	28	26
Number of results reported	152	159	177	236	193
Statistical outliers	5	6	10	10	8
Percentage outliers	3.3%	3.8%	5.7%	4.2%	4.2%

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

<i>Determination</i>	<i>February 2017</i>	<i>February 2015</i>	<i>February 2013</i>	<i>February 2011</i>	<i>March 2010</i>
Acetaldehyde	n.e.	n.e.	++	++	++
Anorganic Chloride	++	++	++	++	++
Colour	++	+	++	++	++
Density at 20 °C	++	++	++	++	++
Formic Acid	++	++	++	++	++
Freezing Point	++	++	++	++	++
Iron as Fe	++	-	++	++	--
Nonvolatile matter	-	+/-	--	++	++
Purity (Freezing point)	++	++	++	++	++
Purity (Titration)	++	++	+	++	++
Sulphate as SO <sub>4</sub>	n.e.	n.e.	--	--	--
Water	++	++	++	++	++

Table 5: comparison determinations against the standard

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 Acetaldehyde on sample #17002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	INH-245	<1	C	----	first reported: < 0.0001mg/kg
174		----		----	
311		----		----	
319		----		----	
323	D2191	<10		----	
343	D2191	<10		----	
347		----		----	
357		----		----	
395		----		----	
551		----		----	
558		----		----	
609	D2191	6		----	
663		----		----	
786	In house	<10		----	
823	D2191	10		----	
859	D2191	93	D(0.01)	----	possibly false positive
861		----		----	
912		----		----	
913		----		----	
963		----		----	
1107		----		----	
1649		<0,1		----	
7002		----		----	
7015	D2191	4.33		----	
7016		----		----	
	normality	n.a.			
	n	8			
	outliers	1			
	mean (n)	<15			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D2191:06)	n.a.			

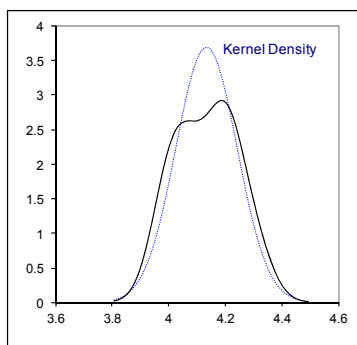
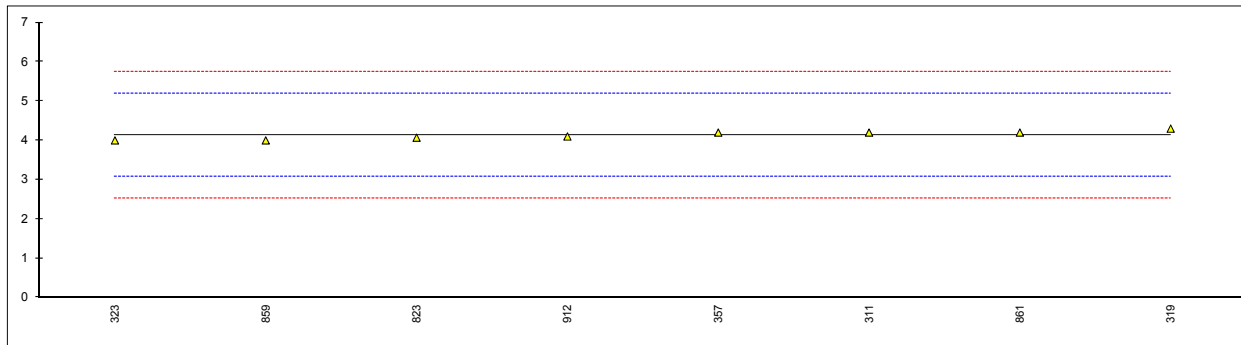
Determination of Appearance on sample #17002;

lab	method	value	mark	z(targ)	remarks
173	E2680	Pass		----	
174	E2680	CFSM		----	
311	E2680	pass		----	
319	Visual	clear colorless liquid		----	
323	E2680	C&B		----	
343	E2680	PASS		----	
347	E2680	PASS		----	
357	E2680	Pass		----	
395	E2680	PASS		----	
551		----		----	
558		----		----	
609	E2680	PASS		----	
663		----		----	
786	E2680	Pass		----	
823	E2680	Pass		----	
859	E2680	PASS		----	
861	Visual	Bright&Clear		----	
912	E2680	Pass		----	
913	D2680	Clear		----	
963		----		----	
1107	E2680	fails		----	reported: the samples contained several fibres
1649		colorless, clear		----	
7002		Pass		----	
7015		----		----	
7016	D4176	Clear without impurities		----	
	normality	n.a.			
	n	19			
	outliers	1			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Lit.)	n.a.			

C&B = Clear and Bright  
 CFSM = Clear and free of suspended matter

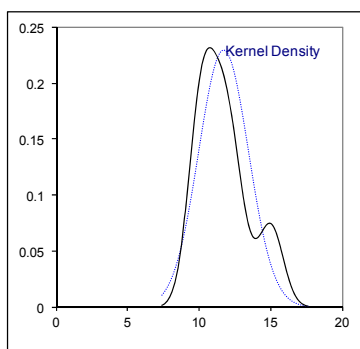
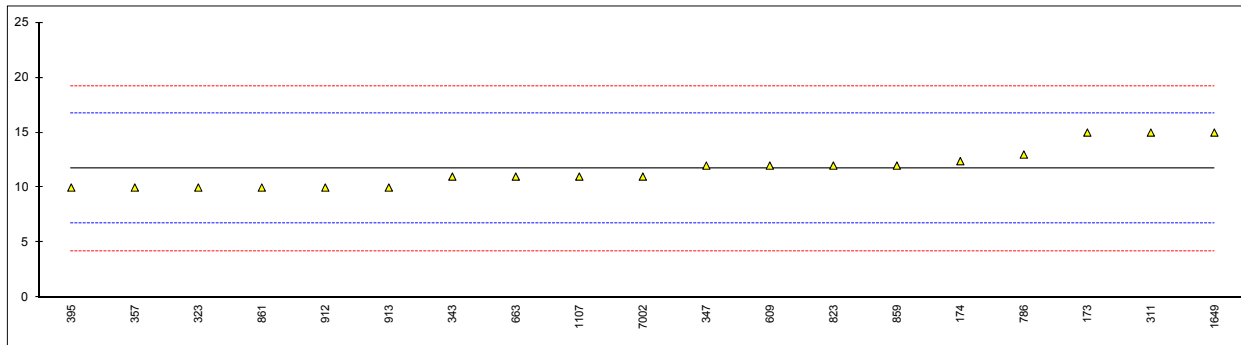
Determination of Chloride, Inorganic as Cl on sample #17002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	INH-0221	>0.5		----	
174		----		----	
311	INH-158	4.2		0.12	
319	ISO753-8	4.3		0.31	
323		4		-0.25	
343		----		----	
347		----		----	
357	INH-709	4.2		0.12	
395		----		----	
551		----		----	
558		----		----	
609	INH-70020	<4		----	
663		----		----	
786		----		----	
823	INH-45	4.07		-0.12	
859	INH-001	4		-0.25	
861	INH-001	4.2		0.12	
912	INH-695	4.1	C	-0.06	first reported: 1.2
913		----		----	
963		----		----	
1107		----		----	
1649		----		----	
7002		----		----	
7015		----		----	
7016		----		----	
normality		unknown			
n		8			
outliers		0		<u>Spike</u>	
mean (n)		4.13		4.35	Recovery < 95%
st.dev. (n)		0.108			
R(calc.)		0.30			
R(Horwitz)		1.50			



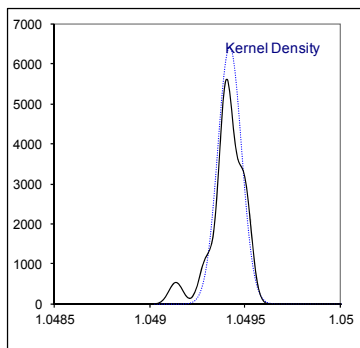
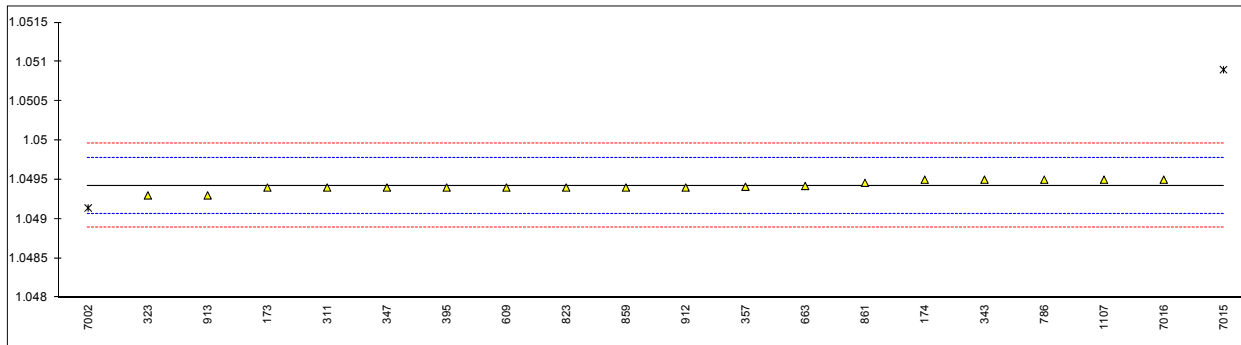
Determination of Colour Pt/Co on sample #17002

lab	method	value	mark	z(targ)	remarks
173	D1209	15		1.32	
174	D5386	12.4		0.28	
311	E302	15		1.32	
319		----		----	
323	D1209	10		-0.68	
343	D5386	11		-0.28	
347	D5386	12		0.12	
357	D1209	10		-0.68	
395	D1209	10		-0.68	
551		----		----	
558		----		----	
609	D1209	12		0.12	
663	D1209	11		-0.28	
786	D1209	13		0.52	
823	D1209	12		0.12	
859	D1209	12		0.12	
861	D1209	10	C	-0.68	reported: 5-10 (off hue)
912	D1209	10		-0.68	
913	D5386	10		-0.68	
963		----		----	
1107	D5386	11		-0.28	
1649		15	C	1.32	first reported: 80
7002		11		-0.28	
7015		----		----	
7016		----		----	
normality		OK			
n		19			
outliers		0			
mean (n)		11.71			
st.dev. (n)		1.740			
R(calc.)		4.87			
R(D1209:05)		7.00			



Determination of Density at 20°C on sample #17002; results in kg/L

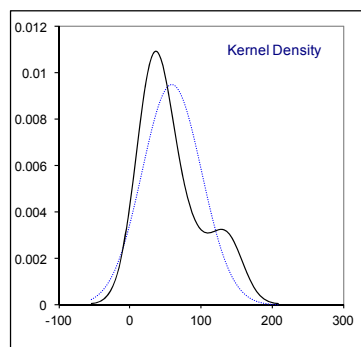
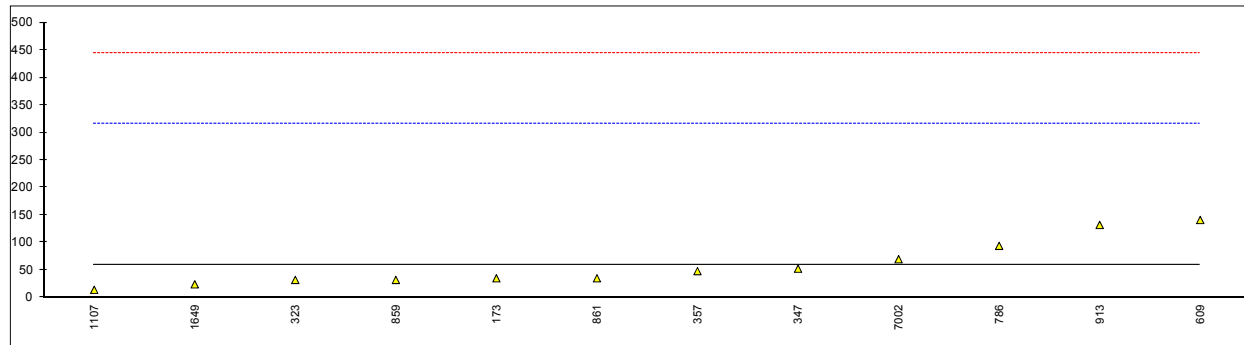
lab	method	value	mark	z(targ)	remarks
173	D4052	1.0494		-0.12	
174	D4052	1.0495		0.44	
311	D4052	1.0494		-0.12	
319		----		----	
323	D4052	1.0493		-0.68	
343	D4052	1.0495		0.44	
347	D4052	1.0494		-0.12	
357	D4052	1.04941		-0.07	
395	D4052	1.0494		-0.12	
551		----		----	
558		----		----	
609	D4052	1.0494		-0.12	
663	D4052	1.04942		-0.01	
786	D4052	1.0495		0.44	
823	D4052	1.0494		-0.12	
859	D4052	1.0494		-0.12	
861	D4052	1.04946		0.21	
912	D4052	1.0494		-0.12	
913	D4052	1.0493		-0.68	
963		----		----	
1107	D4052	1.0495		0.44	
1649		----		----	
7002	ISO12185	1.04914	G(0.01)	-1.58	
7015	ISO12185	1.0509	G(0.01)	8.28	
7016	ISO12185	1.0495		0.44	
normality		OK			
n		18			
outliers		2			
mean (n)		1.04942			
st.dev. (n)		0.000062			
R(calc.)		0.00017			
R(ISO12185:96)		0.00050			
				Compare R(D4052:16)=0.00050	





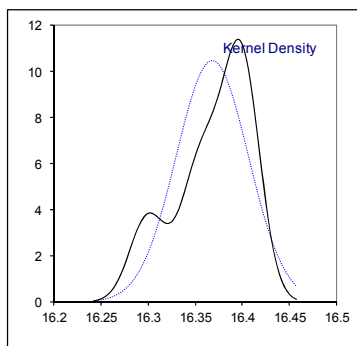
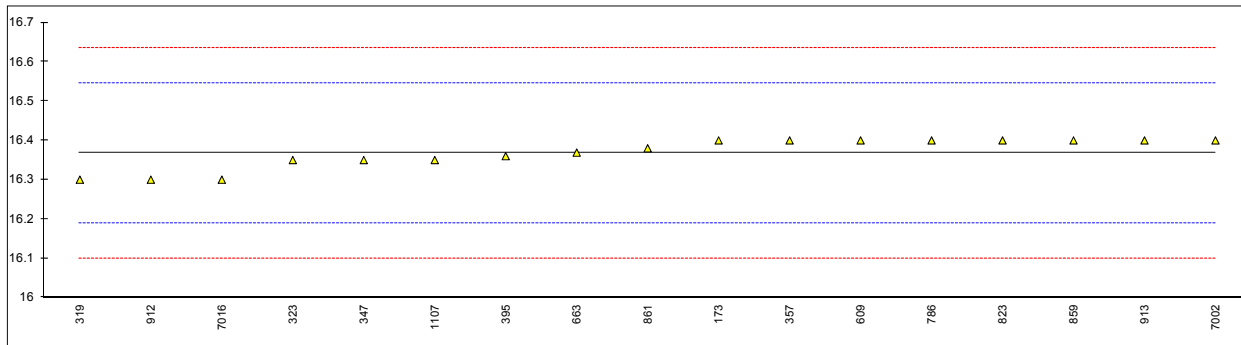
Determination of Formic Acid on sample #17002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	D3546	35	C	-0.19	first reported: 0.0035 mg/kg
174		----		----	
311		----		----	
319		----		----	
323	D3546	32		-0.21	
343		----		----	
347	D3546	52.5		-0.05	
357	D3546	48		-0.09	
395		----		----	
551		----		----	
558		----		----	
609	D3546	141		0.64	
663		----		----	
786	GOST19814	94		0.27	
823		----		----	
859	D3546	32		-0.21	
861	D3546	35		-0.19	
912		----		----	
913	D3546	132	C	0.57	reported: 0.0132 mg/kg
963		----		----	
1107	D3546	14		-0.35	
1649		24		-0.27	
7002	D3546	69.7		0.08	
7015		----		----	
7016		----		----	
normality		suspect			
n		12			
outliers		0			
mean (n)		59.1			
st.dev. (n)		42.01			
R(calc.)		117.6			
R(D3546:05)		360.0			



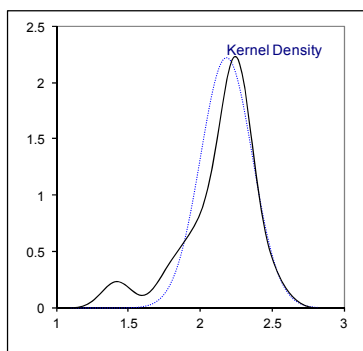
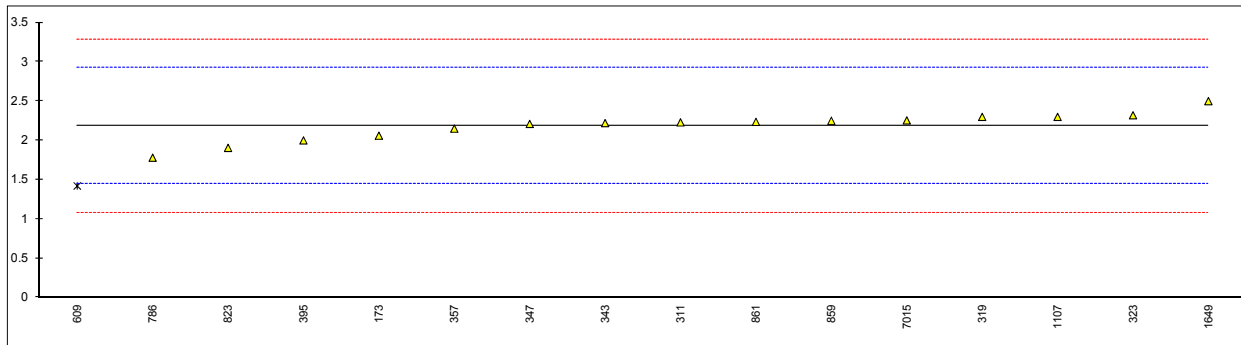
Determination of Freezing Point on sample #17002; results in °C

lab	method	value	mark	z(targ)	remarks
173	INH-124	16.4		0.36	
174		----		----	
311		----		----	
319	E302	16.30		-0.76	
323	E302	16.35		-0.20	
343		----		----	
347	E302	16.35		-0.20	
357	E302	16.40		0.36	
395	INH-124	16.36		-0.09	
551		----		----	
558		----		----	
609	INH-70013	16.4		0.36	
663	D6875	16.369		0.01	
786	E302	16.4		0.36	
823	E302	16.40		0.36	
859	E302	16.40		0.36	
861	E302	16.38		0.13	
912	E302	16.3		-0.76	
913	E302	16.4		0.36	
963		----		----	
1107	E302	16.35		-0.20	
1649		----		----	
7002	E302	16.4		0.36	
7015		----		----	
7016	E302	16.3		-0.76	
normality		OK			
n		17			
outliers		0			
mean (n)		16.368			
st.dev. (n)		0.0381			
R(calc.)		0.107			
R(E302:95)		0.250			



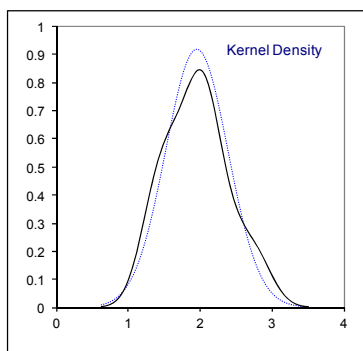
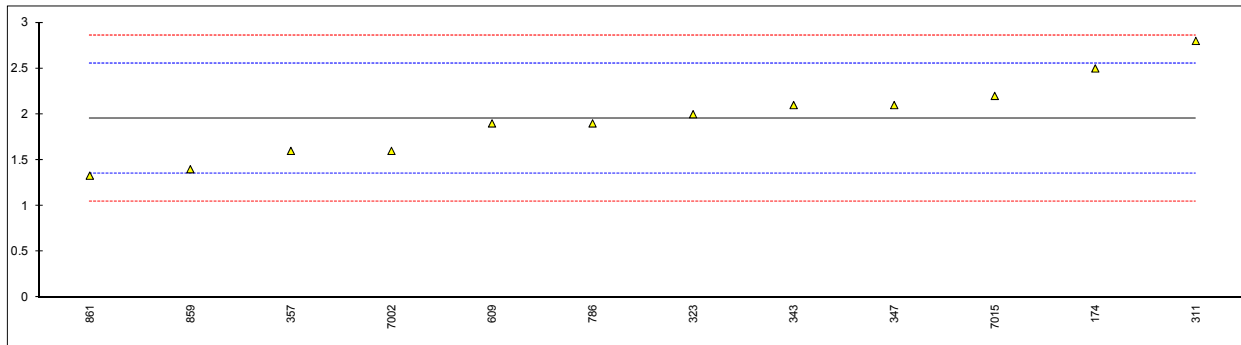
Determination of Iron as Fe on sample #17002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	E394	2.06		-0.33	
174		----		----	
311	E394	2.23		0.13	
319	E394	2.3		0.32	
323	E394	2.32		0.38	
343	E394	2.22		0.11	
347	E394	2.21		0.08	
357	E394	2.15		-0.08	
395	E394	2.00		-0.49	
551		----		----	
558		----		----	
609	E394	1.42	G(0.05)	-2.06	
663		----		----	
786	E394	1.78		-1.09	
823	E394	1.905		-0.75	
859	E394	2.25		0.19	
861	E394	2.236		0.15	
912		----		----	
913		----		----	
963		----		----	
1107	E394	2.3		0.32	
1649		2.5	C	0.86	first reported: 25
7002		----		----	
7015	E394	2.2552		0.20	
7016		----		----	
normality		OK			
n		15			
outliers		1		<u>Spike</u>	
mean (n)		2.181		2.28	Recovery < 96%
st.dev. (n)		0.1798			
R(calc.)		0.504			
R(E394:15)		1.034			



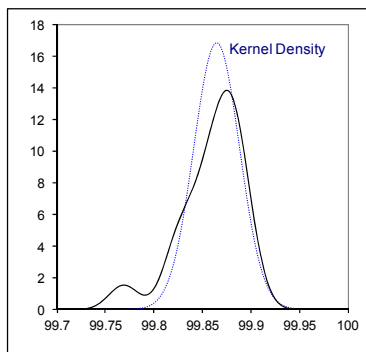
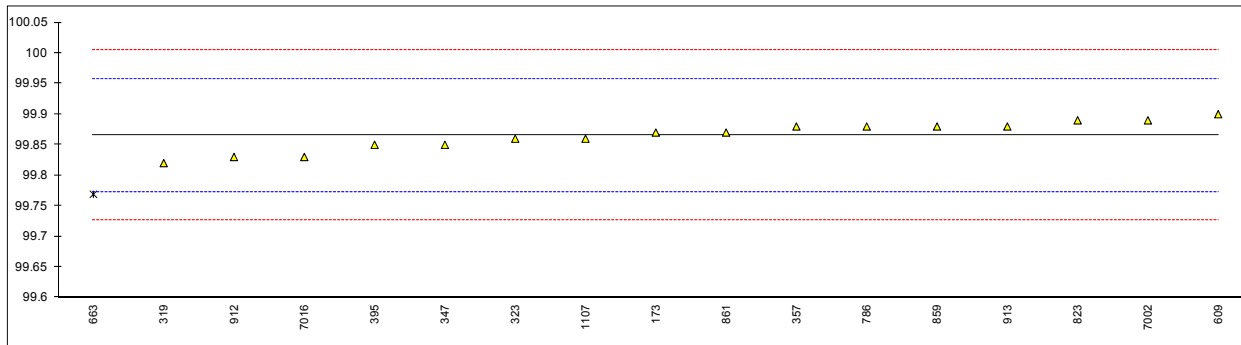
Determination of Nonvolatile Matter on sample #17002; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
173		----		----	
174	D1353	2.5		1.82	
311	D1353	2.8		2.82	
319		----		----	
323	D1353	2		0.16	
343	D1353	2.1		0.49	
347	D1353	2.1		0.49	
357	D1353	1.6		-1.17	
395		----		----	
551		----		----	
558		----		----	
609	D1353	1.9		-0.17	
663		----		----	
786	D1353	1.9		-0.17	
823		----		----	
859	D1353	1.4		-1.84	
861	D1353	1.33		-2.07	
912		----		----	
913		----		----	
963		----		----	
1107		----		----	
1649		----		----	
7002	D1353	1.6		-1.17	
7015	D1353	2.2		0.82	
7016		----		----	
normality		OK			
n		12			
outliers		0			
mean (n)		1.95			
st.dev. (n)		0.434			
R(calc.)		1.22			
R(D1353:13)		0.84			



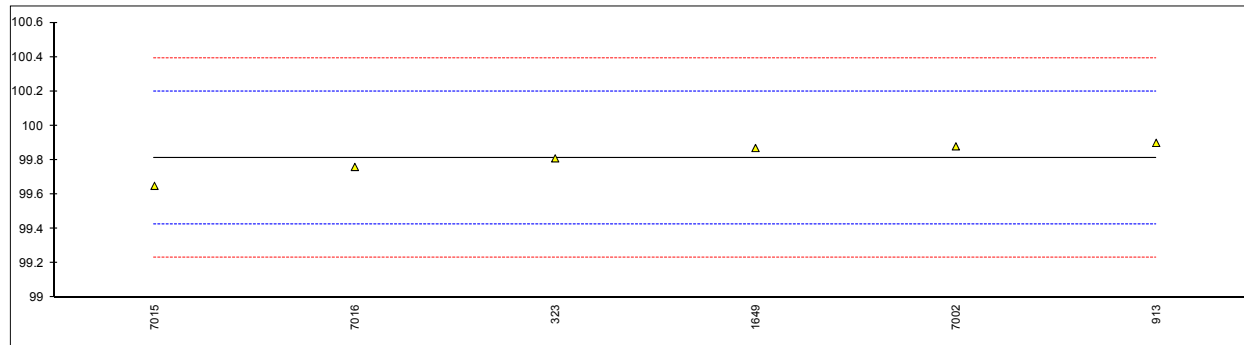
Determination of Purity via Freezing Point on sample #17002; results in %M/M

lab	method	value	mark	z(targ)	remarks
173	INH-124	99.87		0.11	
174		----		----	
311		----		----	
319	E302	99.82		-0.97	
323	E302	99.86		-0.11	
343		----		----	
347	E302	99.85		-0.32	
357	E302	99.88		0.32	
395	INH-124	99.85		-0.32	
551		----		----	
558		----		----	
609	INH-70014	99.90		0.75	
663	BS576Part2	99.769	G(0.05)	-2.07	
786	E302	99.88		0.32	
823	E302	99.89		0.54	
859	E302	99.88		0.32	
861	E302	99.870		0.11	
912	E302	99.83		-0.75	
913	E302	99.88		0.32	
963		----		----	
1107		99.86		-0.11	
1649		----		----	
7002	E302	99.89		0.54	
7015		----		----	
7016	E302	99.83		-0.75	
normality		OK			
n		16			
outliers		1			
mean (n)		99.865			
st.dev. (n)		0.0237			
R(calc.)		0.066			
R(E302:95)		0.130			



Determination of Purity via titration on sample #17002; results in %M/M

lab	method	value	mark	z(targ)	remarks
173		----		----	
174		----		----	
311		----		----	
319		----		----	
323	E301	99.81		-0.01	
343		----		----	
347		----		----	
357		----		----	
395		----		----	
551		----		----	
558		----		----	
609		----		----	
663		----		----	
786		----		----	
823		----		----	
859		----		----	
861		----		----	
912		----		----	
913	E301	99.90		0.46	
963		----		----	
1107		----		----	
1649		99.87		0.30	
7002	E301	99.88		0.35	
7015	E301	99.65		-0.84	
7016	E301	99.76		-0.27	
normality		unknown			
n		6			
outliers		0			
mean (n)		99.812			
st.dev. (n)		0.0945			
R(calc.)		0.265			
R(E301:94)		0.540			

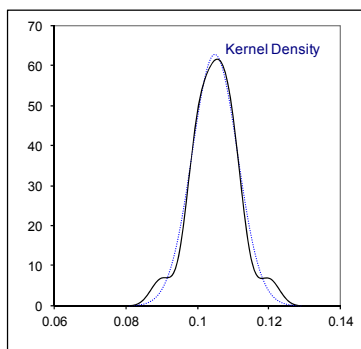
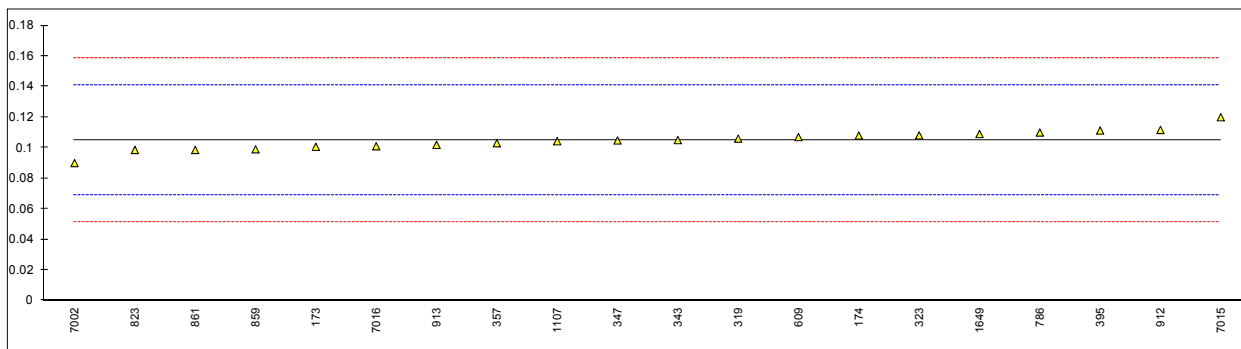


Determination of Sulphate as SO<sub>4</sub> on sample #17002, results in mg/kg

lab	method	value	mark	z(targ)	remarks
173		----		----	
174		----		----	
311		----		----	
319		----		----	
323		----		----	
343	INH-CM	<1		----	
347		----		----	
357	EN15492	0.5		----	
395		----		----	
551		----		----	
558		----		----	
609		----		----	
663		----		----	
786		----		----	
823		----		----	
859		----		----	
861		----		----	
912		----		----	
913		----		----	
963		----		----	
1107		----		----	
1649		----		----	
7002		----		----	
7015		----		----	
7016		----		----	
	normality	n.a.			
	n	2			
	outliers	n.a.			
	mean (n)	<1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	n.a.			

Determination of Water on sample #17002, results in %M/M

lab	method	value	mark	z(targ)	remarks
173	E203	0.1006		-0.24	
174	E203	0.108		0.17	
311		----		----	
319	E1064	0.106		0.06	
323	E302	0.1081		0.18	
343	E1064	0.105		0.01	
347	E1064	0.1048		-0.01	
357	E1064	0.103		-0.11	
395	E1064	0.1113		0.36	
551		----		----	
558		----		----	
609	D1364	0.107		0.12	
663		----		----	
786	E1064	0.110		0.29	
823	D1364	0.0986		-0.35	
859	E1064	0.0990		-0.33	
861	E302	0.0986		-0.35	
912	E203	0.1117	C	0.38	first reported: 0.1117mg/kg
913	E203	0.1019		-0.17	
963		----		----	
1107	E203	0.1043		-0.03	
1649		0.109		0.23	
7002	E302	0.09		-0.83	
7015	E302	0.12		0.85	
7016	E302	0.101		-0.22	
normality		suspect			
n		20			
outliers		0			
mean (n)		0.1049			
st.dev. (n)		0.00635			
R(calc.)		0.0178			
R(E302:95)		0.0500			





## **APPENDIX 2**

### **Number of participants per country**

1 lab in AUSTRIA  
2 labs in BELGIUM  
2 labs in BRAZIL  
2 labs in CHINA, People's Republic  
1 lab in FINLAND  
2 labs in INDIA  
3 labs in IRAN, Islamic Republic of  
1 lab in ITALY  
1 lab in MALAYSIA  
2 labs in NETHERLANDS  
1 lab in RUSSIAN FEDERATION  
1 lab in SAUDI ARABIA  
1 lab in SOUTH KOREA  
2 labs in SPAIN  
1 lab in THAILAND  
2 labs in UNITED STATES OF AMERICA

## APPENDIX 3

### Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
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 E178:89
- 3 ASTM E1301:89
- 4 ISO 5725:86
- 5 ISO 5725, parts 1-6, 1994
- 6 ISO13528:05
- 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
- 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, Analyst, 127, 1359-1364, (2002)
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)
- 16 W. Horwitz and R. Albert, J. AOAC Int., Vol. 79, 3, p. 589, (1996)