Results of Proficiency Test
Methyl methacrylate (MMA)
February 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 2009 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Methyl methacrylate (MMA) once every two years. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of Methyl methacrylate.

In this interlaboratory study 15 laboratories in 13 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the Methyl methacrylate proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one sample of approximately 500 mL Methyl methacrylate labelled #22002.
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/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of approximately 20 liters of Methyl methacrylate was obtained from a local supplier. After homogenization 39 amber glass bottles of 0.5L were filled and labelled #22002.
The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ISO12185 on 4 stratified randomly selected subsamples.

	Density at 20°C in kg/L
sample #22002-1	0.94333
sample #22002-2	0.94332
sample #22002-3	0.94333
sample #22002-4	0.94333

Table 1: homogeneity test results of subsamples #22002

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

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

Table 2: evaluation of the repeatability of subsamples #22002

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

To each of the participating laboratories one sample Methyl methacrylate labelled #22002 was sent on January 12, 2022. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

The participants were asked to determine Acidity as Acrylic acid, Appearance, Color Pt/Co, Density at 20°C, Inhibitor as Topanol A, Water, Purity (two different definitions), Acetone, Ethyl Acrylate, Ethyl Methacrylate, Methanol, Methyl Acrylate, Methyl Isobutyrate, Methyl Propionate, Methyl alpha-hydroxyisobutyrate and Other impurities.
It was also asked what type of column was used in the GC analysis.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendices 1 and 2 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 the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the unrounded (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

$z_{(target)}$ = (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. 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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. Two participants reported test results after the extended final reporting date and one participant did not report any test results. Not all participants were able to report all tests requested.
In total 14 participants reported 95 numerical test results. Observed were 5 outlying test results, which is 5.3%. In proficiency tests 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 in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D1209) and an added designation for the year that the test method was adopted or revised (e.g. D1209:05). If applicable, a designation in parentheses is added to designate the year of re-approval (e.g. D1209:05(2019)). In the test result tables of appendix 1 only the test method number and year of adoption or revision (e.g. D1209:05) are used.

Acidity as Acrylic acid: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D1613:17.

Appearance: This determination was not problematic. All reporting laboratories agreed about the appearance of the sample, which was bright and clear and passes the test.

Color Pt/Co: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1209:05(2019).

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.

Inhibitor as Topanol A: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Water: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM E1064:16.

Regretfully, ASTM D3362 was withdrawn in 2011 with no replacement. As there is no other suitable reference test method with precision data available, it was decided to evaluate the GC Determination with the requirements of ASTM D3362:05.
Nine participants reported which column was used for GC analysis. No clear conclusion could be drawn due to different types of columns mentioned.

Purity (100%-impurities-water-acidity): This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D3362:05.

Purity on dry basis (100%-impurities): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D3362:05.

Ethyl Acrylate: This determination was not problematic. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Methyl Isobutyrate: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Methyl Propionate: This determination was not problematic. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.

The majority of the participants agreed on a content near or below the detection limits of other requested impurities. Therefore, no z-scores are calculated. The reported test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference test methods (in casu ASTM and ISO test methods) or estimated using the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity as Acrylic acid	mg/kg	13	11.7	16.3	14
Appearance		14	pass	n.a.	n.a.
Color Pt/Co		12	3.0	3.1	7
Density at 20°C	kg/L	13	0.9434	0.0001	0.0005
Inhibitor as Topanol A	mg/kg	11	9.6	2.8	3.1
Water	mg/kg	10	78.3	23.2	12.4
Purity [1])	%M/M	11	99.968	0.014	0.27
Purity on dry basis [2])	%M/M	10	99.976	0.012	0.27
Ethyl Acrylate	mg/kg	3	123.9	20.5	26.9
Methyl Isobutyrate	mg/kg	4	6.1	4.3	2.1
Methyl Propionate	mg/kg	3	14.3	4.0	4.3

Table 3: reproducibilities of tests on sample #22002

[1]) Purity = 100% - impurities - water - acidity

[2]) Purity on dry basis = 100% - impurities

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

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

	February 2022	February 2020	January 2018	June 2016	June 2014
Number of reporting laboratories	14	12	15	12	11
Number of test results	95	99	126	112	99
Number of statistical outliers	5	3	3	4	2
Percentage of statistical outliers	5.3%	3.0%	2.4%	3.6%	2.0%

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 reference test methods. The conclusions are given in the following table.

	February 2022	February 2020	January 2018	June 2016	June 2014
Acidity as Acrylic acid	-	+/-	+	+	++
Color Pt/Co	++	++	++	++	++
Density at 20°C	++	++	++	++	++
Inhibitor as Topanol A	+	+	+	--	++
Water	-	--	-	-	--
Purity [1])	++	++	++	++	n.e.
Purity on dry basis [2])	++	++	++	++	n.e.
Acetone	n.e.	+/-	n.e.	(--)	n.e.
Ethyl Acrylate	+	-	n.e.	n.e.	n.e.
Ethyl Methacrylate	n.e.	n.e.	n.e.	n.e.	n.e.
Methanol	n.e.	--	-	(--)	-
Methyl Acrylate	n.e.	n.e.	-	+/-	+
Methyl Isobutyrate	--	-	-	-	+
Methyl Propionate	+/-	n.e.	-	+/-	n.e.
Methyl alpha-hydroxy isobutyrate	n.e.	n.e.	+	++	n.e.
Other impurities	n.e.	(--)	-	n.e.	n.e.

Table 5: comparison determinations against the reference test method

Results between brackets are near or below the detection limits

[1]) Purity = 100% - impurities - water - acidity

[2]) Purity on dry basis = 100% - impurities

The following performance categories were used:

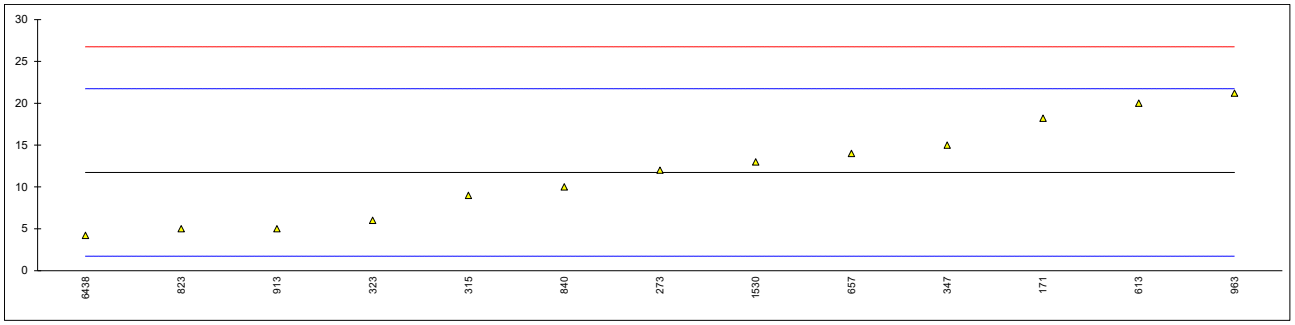
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

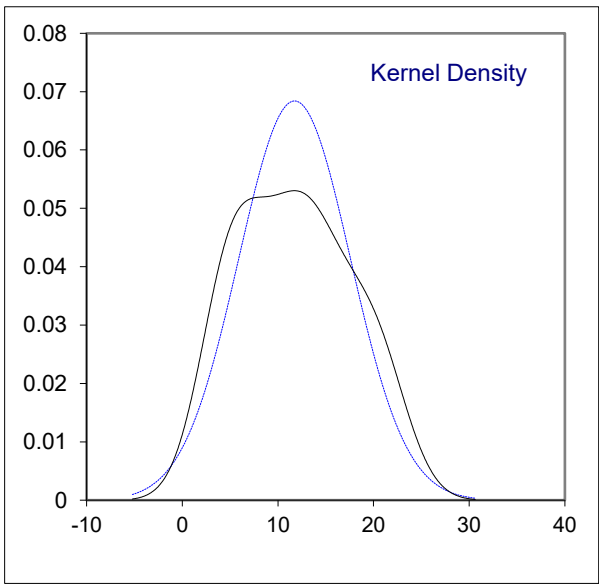
APPENDIX 1

Determination of Acidity as Acrylic acid on sample #22002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1613	18.2		1.29	
273	D1613	12		0.05	
315	D1613	9		-0.55	
323	D1613	6		-1.15	
347	D1613	15		0.65	
557		-----		-----	
613	D1613	20		1.65	
657	D1613	14		0.45	
823	D1613	5		-1.35	
840	D1613	10.0		-0.35	
913	D1613	5		-1.35	
962		-----		-----	
963	D1613	21.2		1.89	
1530	D1613	12.99	C	0.25	first reported 28.26
6438	D1613	4.2		-1.51	

normality	OK
n	13
outliers	0
mean (n)	11.74
st.dev. (n)	5.831
R(calc.)	16.33
st.dev.(D1613:17)	5
R(D1613:17)	14





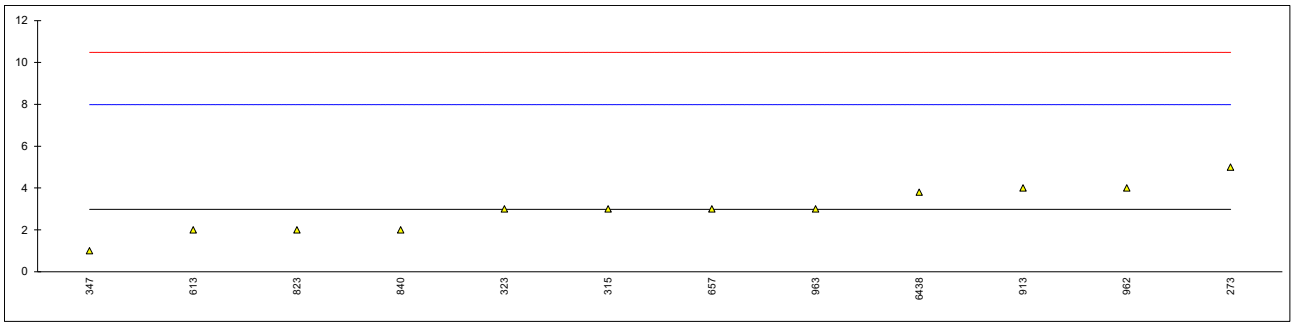
Determination of Appearance on sample #22002;

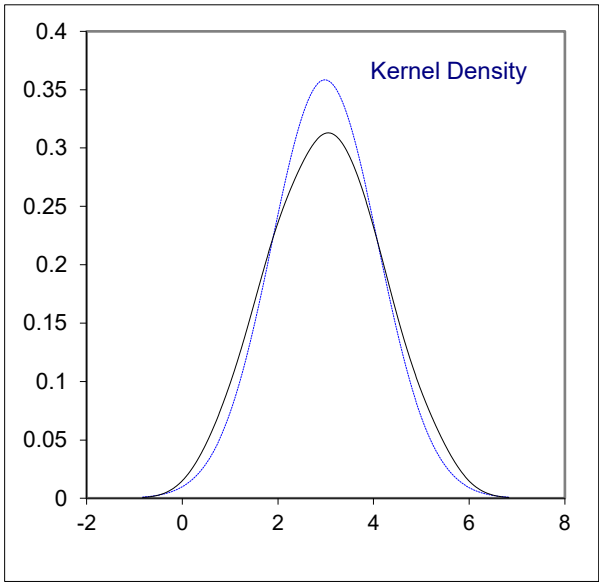
lab	method	value	mark	z(targ)	remarks
171	E2680	Pass		-----	
273	Visual	Bright & Clear		-----	
315	E2680	pass		-----	
323	E2680	CBL		-----	
347	E2680	Pass		-----	
557		-----		-----	
613	DOWM 101967	Pass		-----	
657	Visual	Clear		-----	
823	E2680	Pass		-----	
840	E2680	Pass		-----	
913	E2680	Clear and Bright		-----	
962	Visual	Clear & Bright		-----	
963	Visual	Clear		-----	
1530	E2680	c&b		-----	
6438	Visual	Clear & Bright		-----	
	n	14			
	mean (n)	Pass/ Clear & Bright			

Determination of Color Pt/Co on sample #22002;

lab	method	value	mark	z(targ)	remarks
171	D1209	<5		-----	
273	D1209	5		0.81	
315	D5386	3		0.01	
323	D5386	3		0.01	
347	D5386	1		-0.79	
557		-----		-----	
613	D5386	2		-0.39	
657	D1209	3		0.01	
823	D5386	2		-0.39	
840	D5386	2.0		-0.39	
913	D5386	4		0.41	
962	D1209	4		0.41	
963	D1209	3		0.01	
1530	D1209	<3		-----	
6438	D1209	3.8		0.33	

normality	OK
n	12
outliers	0
mean (n)	2.98
st.dev. (n)	1.113
R(calc.)	3.12
st.dev.(D1209:05)	2.5
R(D1209:05)	7

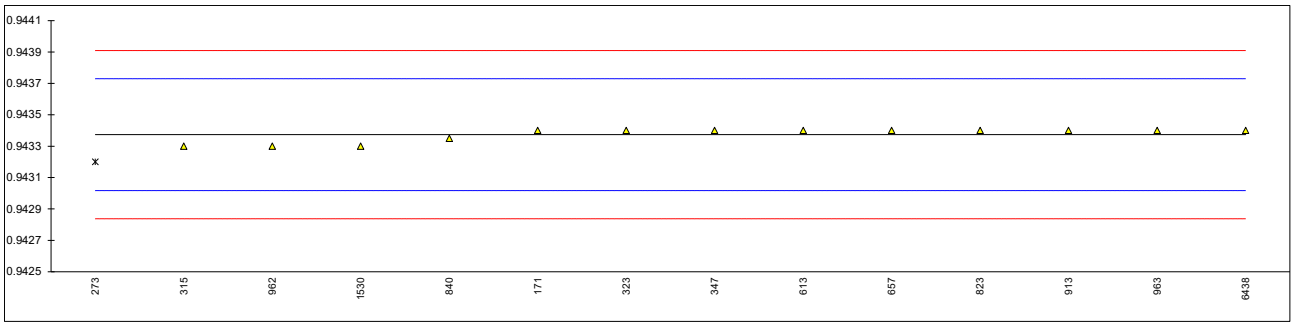


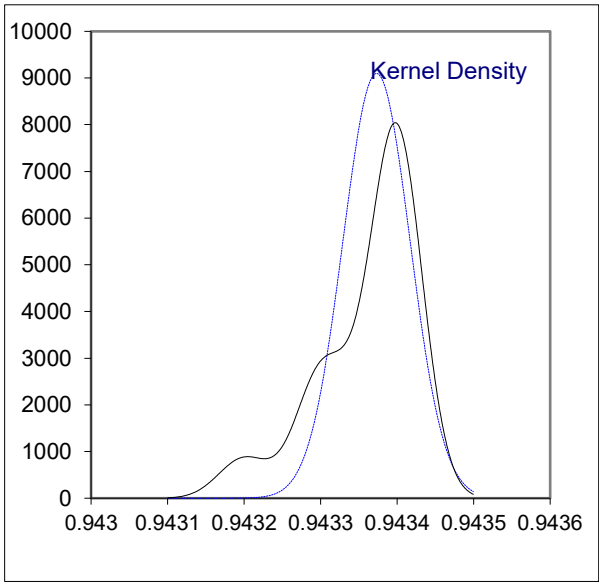


Determination of Density at 20°C on sample #22002; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9434		0.15	
273	D4052	0.9432	G(0.05)	-0.97	
315	D4052	0.9433		-0.41	
323	D4052	0.9434		0.15	
347	D4052	0.9434		0.15	
557		-----		-----	
613	D4052	0.9434		0.15	
657	D4052	0.9434		0.15	
823	D4052	0.9434		0.15	
840	D4052	0.94335		-0.13	
913	D4052	0.9434		0.15	
962	D4052	0.9433		-0.41	
963	D4052	0.9434		0.15	
1530	ISO12185	0.9433	C	-0.41	first reported 0.9237
6438	D4052	0.9434		0.15	

normality	OK
n	13
outliers	1
mean (n)	0.94337
st.dev. (n)	0.000044
R(calc.)	0.00012
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

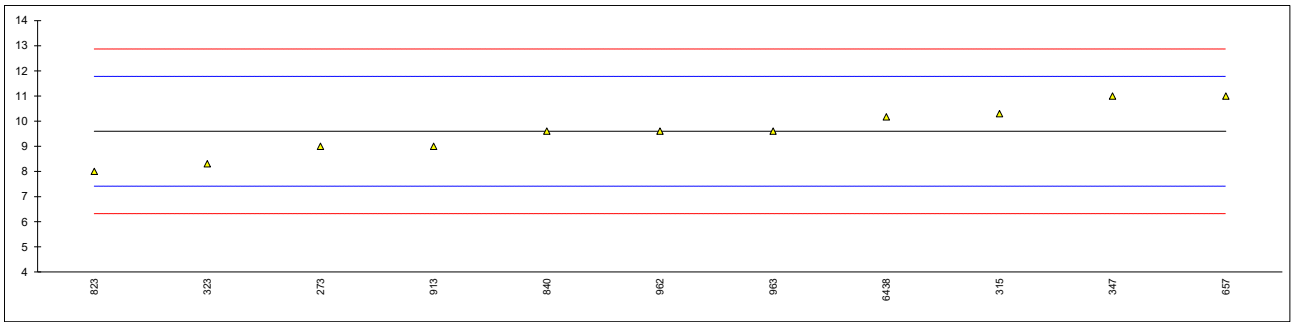


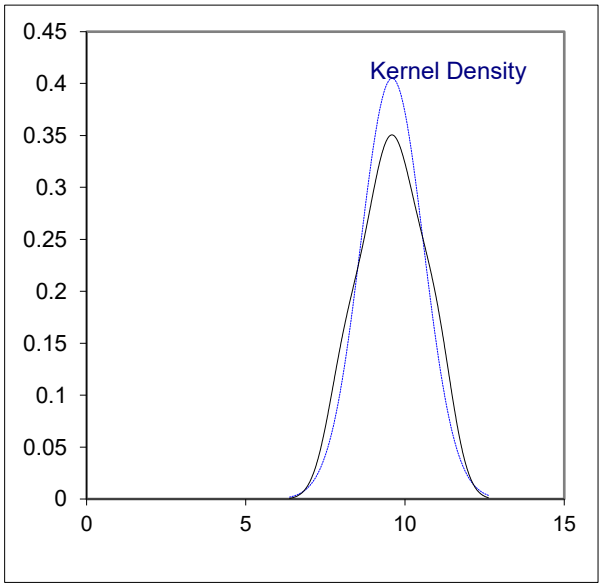


Determination of Inhibitor as Topanol A on sample #22002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273	INH-E20B	9.0		-0.55	
315	INH-510	10.3		0.64	
323	INH-0002	8.3		-1.19	
347	INH-2	11		1.28	
557		-----		-----	
613		-----		-----	
657	INH-2	11		1.28	
823	INH-2	8		-1.46	
840	INH-0002	9.6		0.00	
913	INH-0002	9		-0.55	
962	In house	9.6		0.00	
963	INH-30	9.6		0.00	
1530		-----		-----	
6438	In house	10.17		0.52	

normality	OK
n	11
outliers	0
mean (n)	9.60
st.dev. (n)	0.985
R(calc.)	2.76
st.dev.(Horwitz)	1.093
R(Horwitz)	3.06

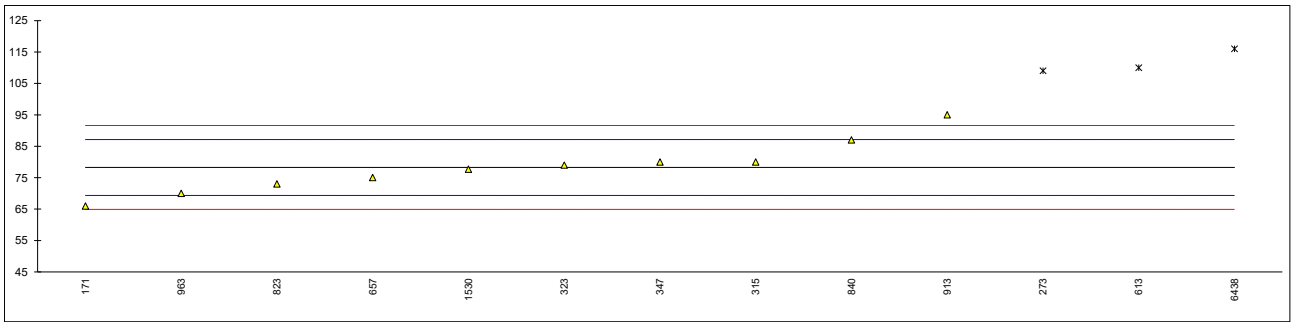


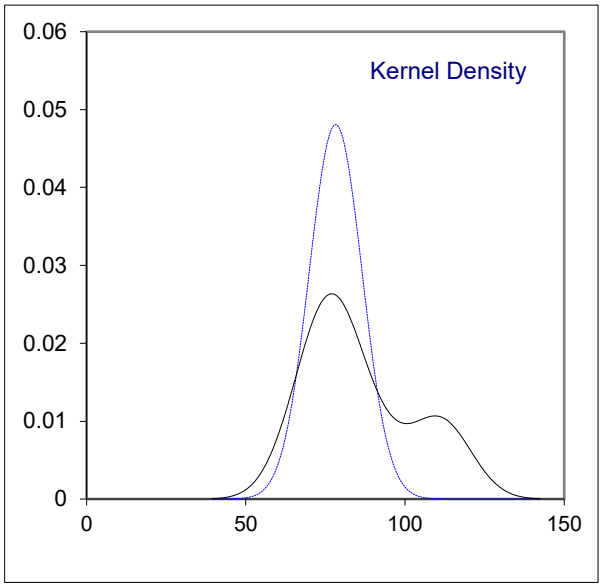


Determination of Water on sample #22002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	E1064	66		-2.76	
273	E1064	109	G(0.05)	6.91	
315	E1064	80		0.39	
323	E1064	79		0.16	
347	E1064	80		0.39	
557		-----		-----	
613	E203	110	G(0.05)	7.14	
657	E1064	75		-0.74	
823	E1064	73		-1.19	
840	E1064	87		1.96	
913	E1064	95		3.76	
962		-----		-----	
963	E1064	70		-1.86	
1530	E1064	77.7		-0.13	
6438	E1064	116	C,G(0.05)	8.49	first reported 105.8

normality	OK
n	10
outliers	3
mean (n)	78.27
st.dev. (n)	8.302
R(calc.)	23.25
st.dev.(E1064:16)	4.445
R(E1064:16)	12.44



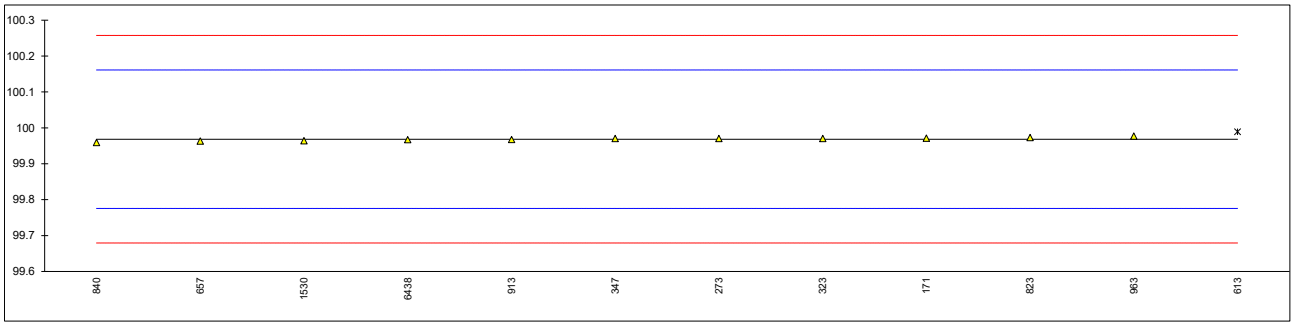


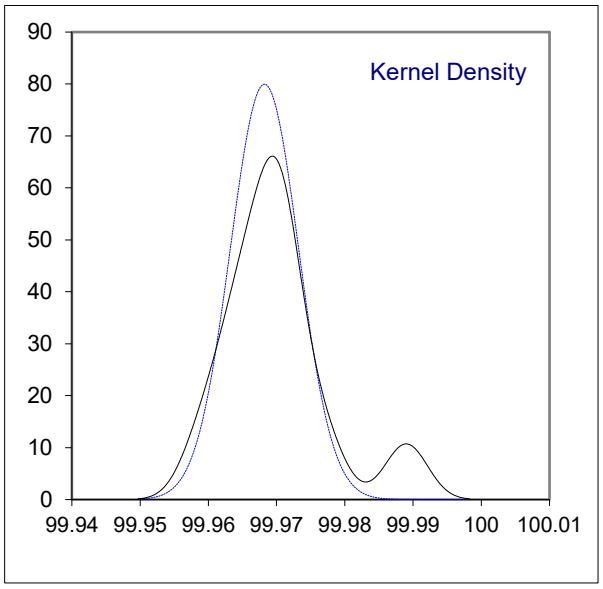
Determination of Purity*) on sample #22002; results in %M/M

lab	method	value	mark	z(targ)	type of column	remarks
171	In house	99.9712		0.03	Petrocol DH 150 GC DP MMacryl-01.1	
273	INH-E20B	99.97		0.02	DB-1 - 30m x 0.450mm x 2.55u	
315		-----		-----	60 m x 0,32 mm (ID), CP WAX 52 CB, Df: 1,2 µm	
323	D3362Mod.	99.97		0.02	DB1 - 30m x 0.32 mm x 1.0 µm	
347	INH-2	99.970		0.02	CP SIL 5CB 25mx0,32mmx1,2um	
557		-----		-----		
613	QAL-1504	99.989	G(0.05)	0.22		
657	INH-8001	99.963		-0.05	DB-1 (30m x 0.32mm x 1.00 µm)	
823	INH-2	99.9726		0.05	CP-Sil 5CB, 25 m * 0.320 mm * 1.20 µm	
840	INH-0002	99.959		-0.10		
913	INH-0002	99.967		-0.01		
962		-----		-----		
963	INH-30	99.977		0.09	DB 1701 (60mX0.25mX1.0µm)	
1530		99.964		-0.04		
6438	In house	99.9666		-0.02	Agilent HP-5 column, 30mX0.320mm,0.25µm	

normality	OK
n	11
outliers	1
mean (n)	99.9682
st.dev. (n)	0.00499
R(calc.)	0.0140
st.dev.(D3362:05)	0.09643
R(D3362:05)	0.27

*) Purity= 100% - impurities – water – acidity



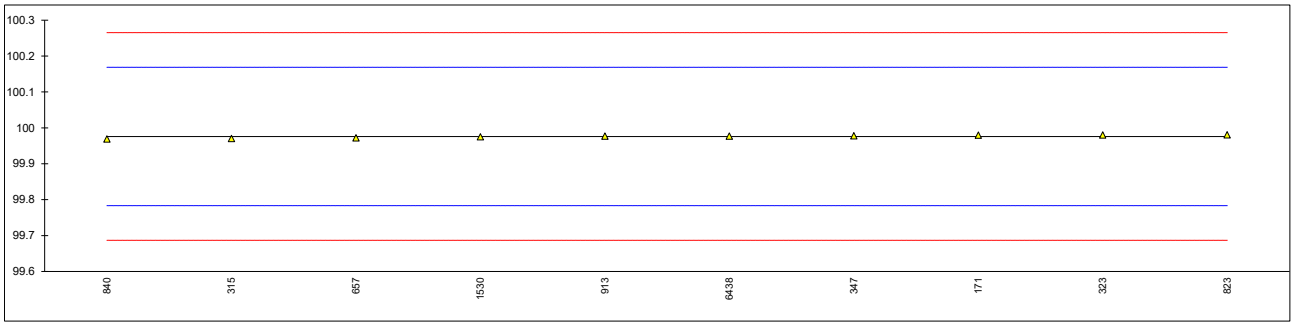


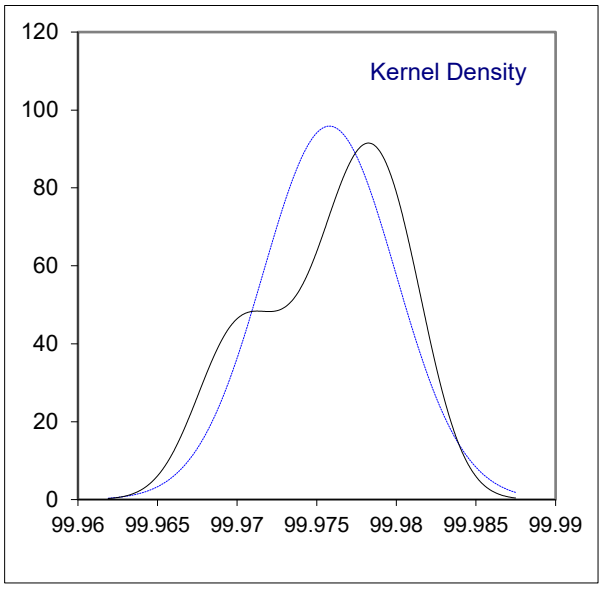
Determination of Purity on dry basis*) on sample #22002; results in %M/M

lab	method	value	mark	z(targ)	type of column	remarks
171	In house	99.97961		0.04	Petrocol DH 150 GC DP MMacryl-01.1	
273		-----		-----	DB-1 - 30m x 0.450mm x 2.55u	
315	INH-2	99.97		-0.06	60 m x 0,32 mm (ID), CP WAX 52 CB, Df: 1,2 µm	
323	D3362Mod.	99.98		0.04	DB1 - 30m x 0.32 mm x 1.0 µm	
347	INH-2	99.978		0.02	CP SIL 5CB 25mx0,32mmx1,2um	
557		-----		-----		
613		-----		-----		
657	INH-8001	99.972		-0.04	DB-1 (30m x 0.32mm x 1.00 µm)	
823	INH-2	99.9804		0.05	CP-Sil 5CB, 25 m * 0.320 mm * 1.20 µm	
840	INH-0002	99.969		-0.07		
913	INH-0002	99.977		0.01		
962		-----		-----		
963		-----		-----	DB 1701 (60mX0.25mX1.0µm)	
1530		99.975		-0.01		
6438	In house	99.977		0.01	Agilent HP-5 column, 30mX0.320mm,0.25µm	

normality	OK
n	10
outliers	0
mean (n)	99.9758
st.dev. (n)	0.00416
R(calc.)	0.0117
st.dev.(D3362:05)	0.09643
R(D3362:05)	0.27

*) Purity on dry basis = 100% - impurities

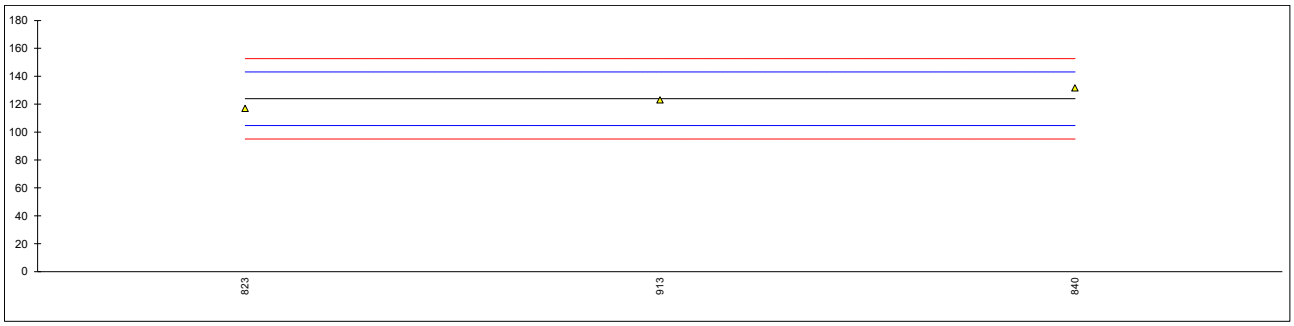




Determination of Ethyl Acrylate on sample #22002 results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
315		-----		-----	
323		-----		-----	
347		-----		-----	
557		-----		-----	
613		-----		-----	
657		-----		-----	
823	INH-2	117		-0.72	
840	INH-0002	131.6		0.81	
913	INH-0002	123		-0.09	
962		-----		-----	
963		-----		-----	
1530		-----		-----	
6438		-----		-----	

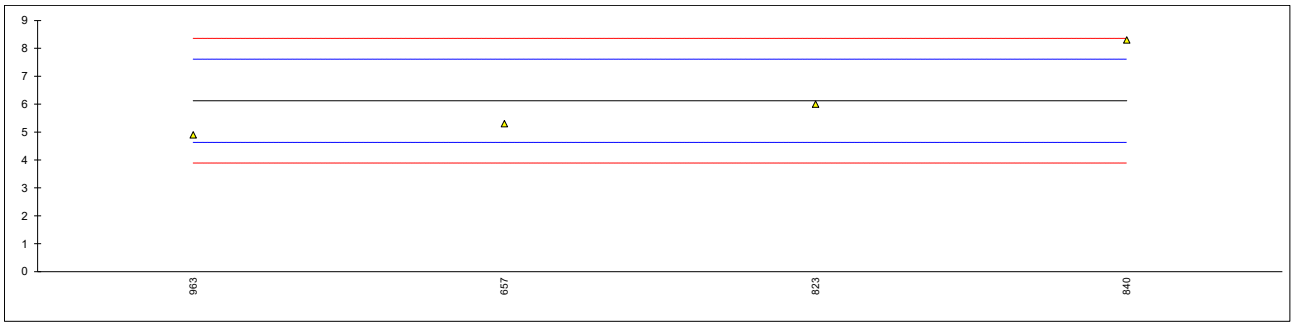
normality	unknown
n	3
outliers	0
mean (n)	123.87
st.dev. (n)	7.338
R(calc.)	20.55
st.dev.(Horwitz)	9.595
R(Horwitz)	26.87



Determination of Methyl Isobutyrate on sample #22002 results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
315	INH-2	<10	C	-----	first reported 16
323		-----		-----	
347		-----		-----	
557		-----		-----	
613		-----		-----	
657	INH-8001	5.3		-1.11	
823	INH-2	6		-0.17	
840	INH-0002	8.3		2.92	
913		-----		-----	
962		-----		-----	
963	INH-30	4.9		-1.64	
1530		-----		-----	
6438		-----		-----	

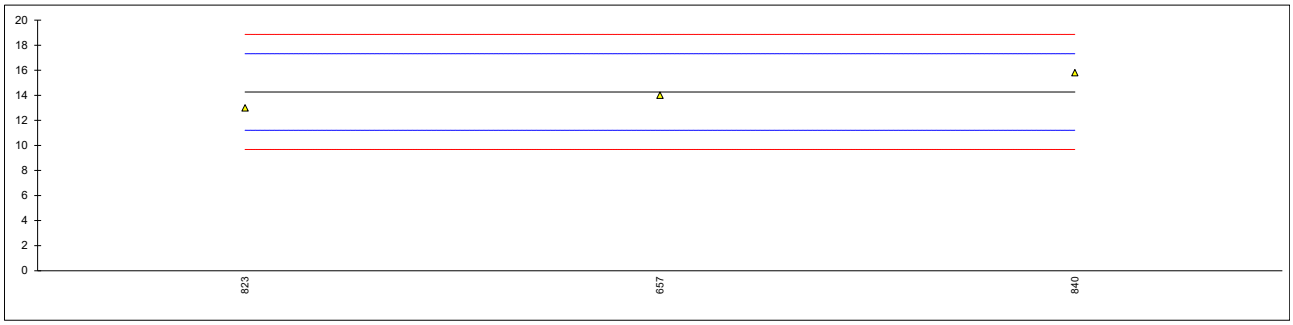
normality	unknown
n	4
outliers	0
mean (n)	6.13
st.dev. (n)	1.520
R(calc.)	4.25
st.dev.(Horwitz)	0.746
R(Horwitz)	2.09



Determination of Methyl Propionate on sample #22002 results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
315		-----		-----	
323	D3362Mod.	< 50		-----	
347		-----		-----	
557		-----		-----	
613		-----		-----	
657	INH-8001	14		-0.17	
823	INH-2	13		-0.83	
840	INH-0002	15.8		1.00	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1530		-----		-----	
6438		-----		-----	

normality	unknown
n	3
outliers	0
mean (n)	14.27
st.dev. (n)	1.419
R(calc.)	3.97
st.dev.(Horwitz)	1.530
R(Horwitz)	4.28



APPENDIX 2 Other reported test results

Determination of individual Components on sample #22002; in mg/kg

lab	Acetone	Ethyl Methacrylate	Methanol	Methyl Acrylate	Methyl alpha-hydroxy isobutyrate	Other impurities
171	-----	-----	-----	-----	-----	-----
273	-----	-----	-----	-----	-----	-----
315	<10	-----	<10	-----	-----	-----
323	-----	< 50	-----	-----	< 100	-----
347	-----	-----	-----	-----	-----	-----
557	-----	-----	-----	-----	-----	-----
613	-----	-----	-----	-----	-----	-----
657	<10	0.8	<10	<10	0.6	-----
823	7	<5	<5	<5	<5	-----
840	7.5	12.9	<5	<5	71.1	50.8
913	<10	-----	<10	-----	-----	-----
962	-----	-----	-----	-----	-----	-----
963	-----	-----	10.6	-----	-----	-----
1530	-----	-----	-----	-----	-----	-----
6438	5.1	-----	0.5	-----	-----	-----

APPENDIX 3

Number of participants per country

1 lab in	UNITED STATES OF AMERICA
1 lab in	AUSTRALIA
1 lab in	BELGIUM
1 lab in	BRAZIL
1 lab in	GERMANY
1 lab in	INDIA
1 lab in	KOREA, Republic of
1 lab in	NETHERLANDS
3 labs in	SAUDI ARABIA
1 lab in	SINGAPORE
1 lab in	SOUTH AFRICA
1 lab in	SPAIN
1 lab in	VIETNAM

APPENDIX 4

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	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

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