Results of Proficiency Test Methylmethacrylate (MMA) June 2014

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

On request of several laboratories, the Institute for Interlaboratory Studies decided to organise again a proficiency test for the analysis of Methylmethacrylate (MMA) during the annual proficiency testing program 2013/2014. In this interlaboratory study 11 laboratories from 11 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2014 Methylmethacrylate (MMA) proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to send 1* 0.5L bottle of MMA, labelled #14085. Participants were requested to report rounded and unrounded results. The unrounded 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 (R007). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Also customer's satisfaction is measured on regular basis by the distribution of 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 April 2014 (iis-protocol, version 3.3).

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 10 litre bulk material was obtained from a local supplier. From this 10 litre batch were, after homogenizing, 18 brown glass bottles of 0.5 litre (labelled #14085) filled.

The homogeneity of the subsamples was checked by determination of Density @ 20°C according to ISO12185 and by determination of Methylacrylate according to an in house test method on 4 stratified randomly selected samples.

	Density in kg/L	Methylacrylate in mg/kg
sample #14085-1	0.94342	24.4
sample #14085-2	0.94342	24.7
sample #14085-3	0.94342	25.4
sample #14085-4	0.94342	24.8

table 1: homogeneity test results of subsamples #14085

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density in kg/L	Methylacrylate in mg/kg
r (sample #14085)	0.00000	1.2
reference test method	D4052:02e1	Horwitz
0.3 * R (reference test)	0.00015	2.1

table 2: repeatability of subsample #14085

The calculated repeatabilities were less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples #14085 was assumed.

One bottle 0.5 L, labelled #14085 was dispatched to each of the participating laboratories on May 28, 2014.

2.5 STABILITY OF THE SAMPLES

The stability of the Methylmethacrylate, packed in the brown glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine on sample #14085: Acidity (as Acrylic Acid), Appearance, Colour Pt/Co, Density @ 20°C, Inhibitor as Topanol A, Water, Purity (both on “as-is” and on dry basis), Acetone, Methanol, Methylacrylate, Methylisobutyrate, Ethylmethacrylate and Other Impurities.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website www.iisnl.com. A SDS and a form to confirm receipt of the samples were added to the sample package

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated per determination in the appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after deadline, a reminder fax was sent to those laboratories that had not yet reported. Shortly after the deadline, the available results were screened for suspect data. A 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 (raw data of the) reported results. Additional or corrected results have been used for data analysis and the original results are placed under 'Remarks' in the result tables in Appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ (iis-protocol, April 2014 version 3.3). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.
In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and Rosner outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 3, no.14). Stragglers are marked by D(0.05) for the Dixon 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 the averages and the 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 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 analysis results are plotted. The corresponding laboratory numbers are under 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 “x”. 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 (see appendix 3; nos.12 and 13). Also a normal Gauss curve was projected over the Kernel Density Graph.

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 spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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 in accordance with:

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

The $z_{(target)}$ scores are listed in the 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 maybe as follows:

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

4 EVALUATION

In this proficiency test, all laboratories reported results. Two laboratories reported results after the final reporting data. In total 11 participants reported 99 numerical results. Observed were 2 outlying results, which is 2.0% of the numerical results.

4.1 EVALUATION PER TEST

In this section, the results are discussed per sample and 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.
In the iis PT reports, ASTM methods are referred to with a number (e.g. D2086) and an added designation for the year that the method was adopted or revised (e.g. D2086-08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086-08 (2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

Four data sets proved to have a normal distribution. For the most tests the number of reported test results was too small to determine whether the data set was normally distributed. In these cases the results of the statistical evaluations should be used with care.

Acidity as Acrylic Acid: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1613:06(2012).

Appearance: No analytical problems were observed. All labs agreed about the appearance of the sample #14085, which was bright, clear and free of suspended matter.

Colour Pt/Co: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1209:05e1(2011).

Density @ 20°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D4052:02e1.

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

Water: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all agreement with the requirements of ASTM E1064:12

(See §15.1.3, note 9). The reproducibility mentioned under §15.3.2, table 2 is not suitable because it is for Polyols only.

Purity “as is”: Regretfully, no suitable standardized method exists with precision data. Therefore no significant conclusions were drawn.

Purity on dry basis: Regretfully, no suitable standardized method exists with precision data. Therefore no significant conclusions were drawn.

Acetone: No significant conclusions were drawn. All laboratories agreed on a value of <10 mg/kg.

Methanol: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility, calculated using the Horwitz equation.

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

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

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

Other Impurities: Regretfully, no suitable standardized method exists with precision data. Therefore no significant conclusions were drawn.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and these parameters as found for the group of participating laboratories. The average results and the calculated reproducibilities are compared in the next tables with the reproducibilities, derived from literature standards (in casu the EN, ASTM and ISO standards), see tables in appendix 1.

Parameter	unit	n	average	R (Calc.)	R (lit)
Acidity as Acrylic Acid	mg/kg	11	14.9	7.3	14.0
Appearance		11	Pass	n.e	n.e
Colour Pt/Co		9	3.6	3.2	7.0
Density @ 20°C	kg/L	10	0.9434	0.0001	0.0005
Inhibitor as Topanol A	mg/kg	8	11.1	1.4	3.5
Water	mg/kg	10	100.4	35.0	17.2
Purity “as is” / as received	%M/M	9	99.958	0.016	unknown
Purity (on dry basis)	%M/M	9	99.970	0.010	unknown
Acetone	mg/kg	8	<10	n.e.	n.e.
Methanol	mg/kg	7	23.8	9.4	6.6
Methylacrylate	mg/kg	6	22.4	5.4	6.3
Methylisobutylyrate	mg/kg	6	66.4	13.6	15.8
Ethylmethacrylate	mg/kg	4	5.9	1.8	2.0
Other impurities	mg/kg	6	192.9	142.9	unknown

table 3: reproducibilities of results of sample #14085

Without further statistical calculations, it can be concluded that 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 JUNE 2014 WITH PREVIOUS PTS

	June 2014	May 2011	April 2009	April 1999
Number of reporting labs	11	11	11	10
Number of results reported	99	85	97	106
Statistical outliers	2	3	6	8
Percentage outliers	2.0%	3.5%	6.2%	7.5%

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:

	June 2014	May 2011	April 2009	April 1999
Acidity as Acrylic Acid	++	+/-	-	-
Colour Pt/Co	++	++	++	++
Density @ 20°C	++	++	++	++
Inhibitor as Topanol A	++	-	++	--
Water	--	++	++	++
Purity “as is”	n.e.	n.e.	n.e.	n.e.
Purity on dry basis	n.e.	n.e.	n.e.	n.e.
Acetone	n.e.	n.e.	n.e.	n.e.
Methanol	-	n.e.	n.e.	n.e.
Methylacrylate	+	+/-	-	n.e.
Methylisobutylyrate	+	++	++	n.e.
Ethylmethacrylate	+	+	-	n.e.
Other impurities	n.e.	n.e.	n.e.	n.e.

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:

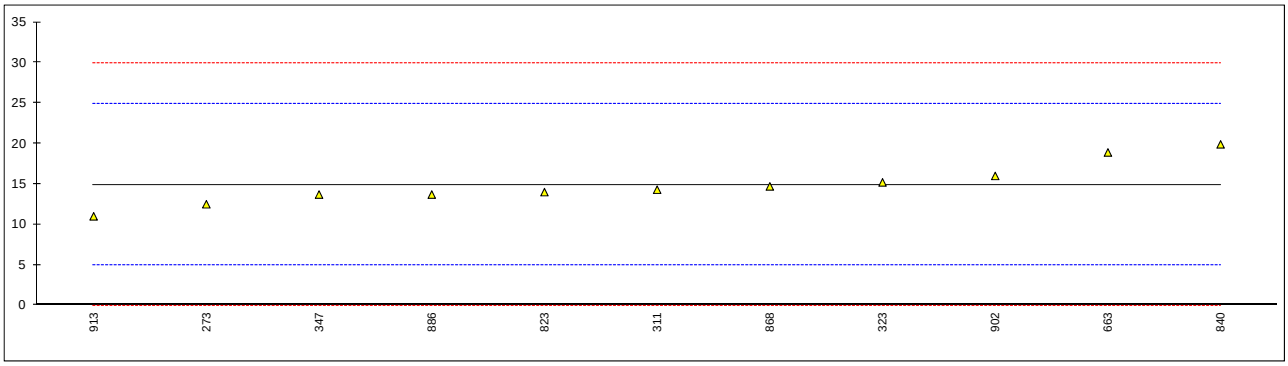
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- - : group performed worse than the standard
- -- : group performed much worse than the standard
- n.e.: not evaluated

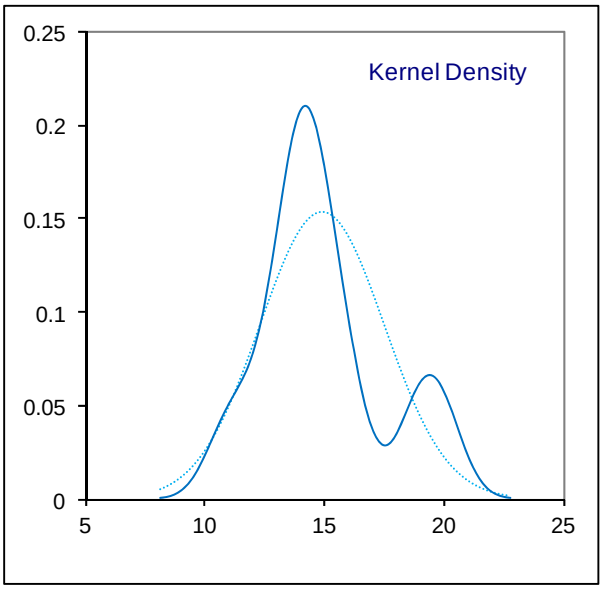
APPENDIX 1

Determination of Acidity as Acrylic Acid on sample #14085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273	D1613	12.5		-0.48	
311	D1613	14.3		-0.12	
323	D1613	15.2	C	0.06	first reported: 20
347	D1613	13.7		-0.24	
663	D1613	18.9		0.80	
823	D1613	14		-0.18	
840	D1613	19.9		1.00	
868	D1613	14.7		-0.04	
886	D1613	13.7		-0.24	
902	D1613	16		0.22	
913	D1613	11.00		-0.78	

normality	OK
n	11
outliers	0
mean (n)	14.90
st.dev. (n)	2.595
R(calc.)	7.27
R(D1613:06)	14.00





Determination of Appearance on sample #14085;

lab	method	value	mark	z(targ)	remarks
273	E2680	Pass		-----	
311	E2680	C&F		-----	
323	E2680	Pass		-----	
347	E2680	Pass		-----	
663	INH-14085	Pass		-----	
823	E2680	Pass		-----	
840	E2680	Pass		-----	
868	E2680	Pass		-----	
886	Visual	CFSM		-----	
902	E2680	B&C		-----	
913	E2680	Pass		-----	
	normality	n.a			
	n	11			
	outliers	0			
	mean (n)	Pass			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(E2680:09)	n.a			

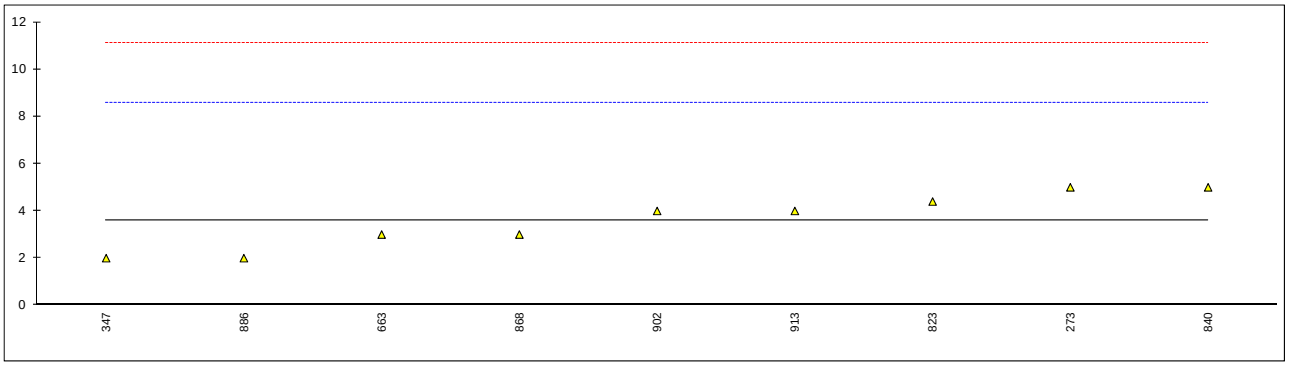
Abbreviations:

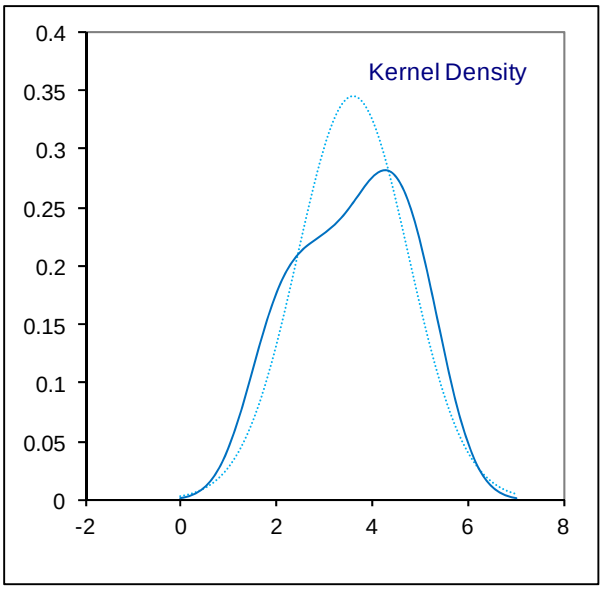
B&C = bright and clear
C&F = clear and free
CFSM = clear free of suspended matter

Determination of Colour Pt/Co on sample #14085;

lab	method	value	mark	z(targ)	remarks
273	D1209	5.0		0.56	
311	D1209	<5		-----	
323	D1209	<5		-----	
347	D5386	2		-0.64	
663	D1209	3		-0.24	
823	D5386	4.4		0.32	
840	D1209	5		0.56	
868	D1209	3		-0.24	
886	D1209	2		-0.64	
902	D1209	4		0.16	
913	D5386	4		0.16	

normality	OK
n	9
outliers	0
mean (n)	3.60
st.dev. (n)	1.158
R(calc.)	3.24
R(D1209:05e1)	7.00

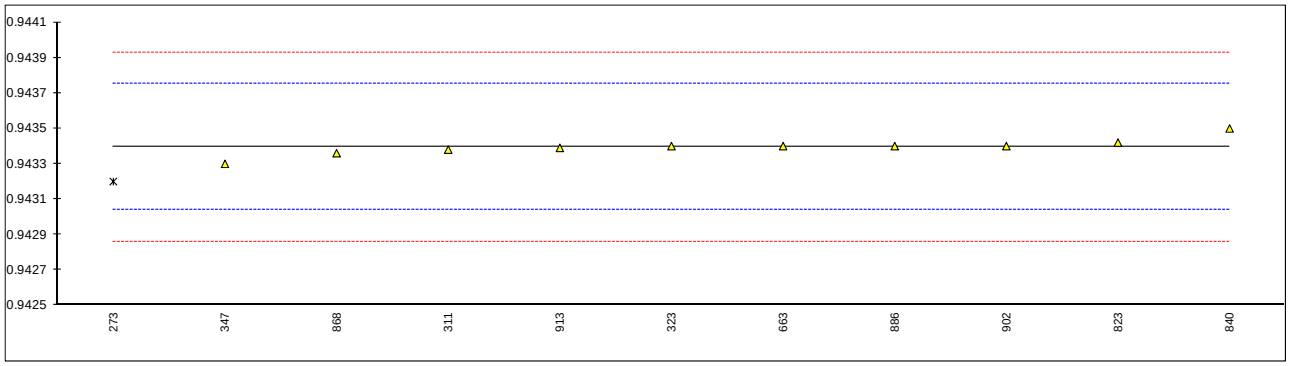


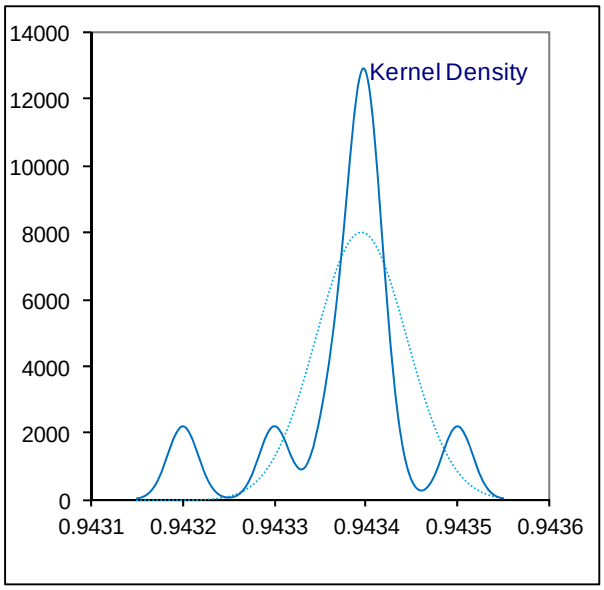


Determination of Density @ 20°C on sample #14085; results in kg/L

lab	method	value	mark	z(targ)	remarks
273	D4052	0.9432	G(0.05)	-1.09	
311	D4052	0.94338		-0.08	
323	D4052	0.9434		0.03	
347	D4052	0.9433		-0.53	
663	D4052	0.9434		0.03	
823	D4052	0.94342		0.14	
840	D4052	0.94350		0.59	
868	D4052	0.94336		-0.20	
886	D4052	0.9434		0.03	
902	D4052	0.9434		0.03	
913	D4052	0.94339		-0.03	

normality	not OK
n	10
outliers	1
mean (n)	0.94339
st.dev. (n)	0.000050
R(calc.)	0.00014
R(D4052:02e1)	0.00050

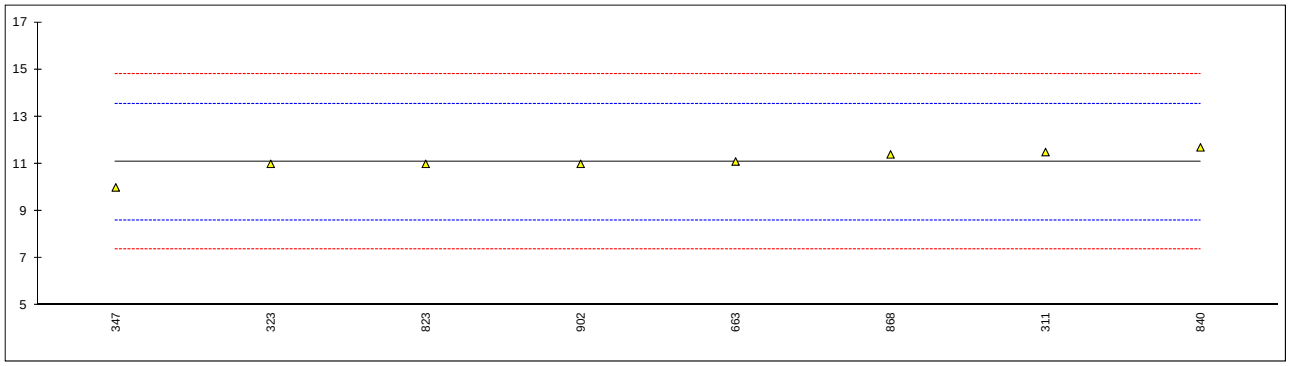


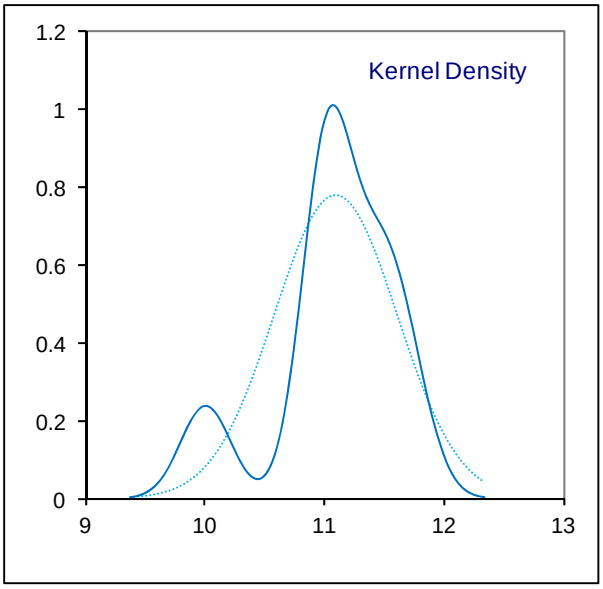


Determination of Inhibitor as Topanol A on sample #14085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
311	INH-510	11.5		0.33	
323	INH-0002	11		-0.07	
347	INH-0002	10		-0.88	
663	INH-8001	11.1	C	0.01	first reported: 21.1
823	INH-0002	11		-0.07	
840	INH-0002	11.7		0.50	
868	INH-044	11.4		0.25	
886		-----		-----	
902	INH-94	11		-0.07	
913		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	11.09
st.dev. (n)	0.514
R(calc.)	1.44
R(Horwitz)	3.46

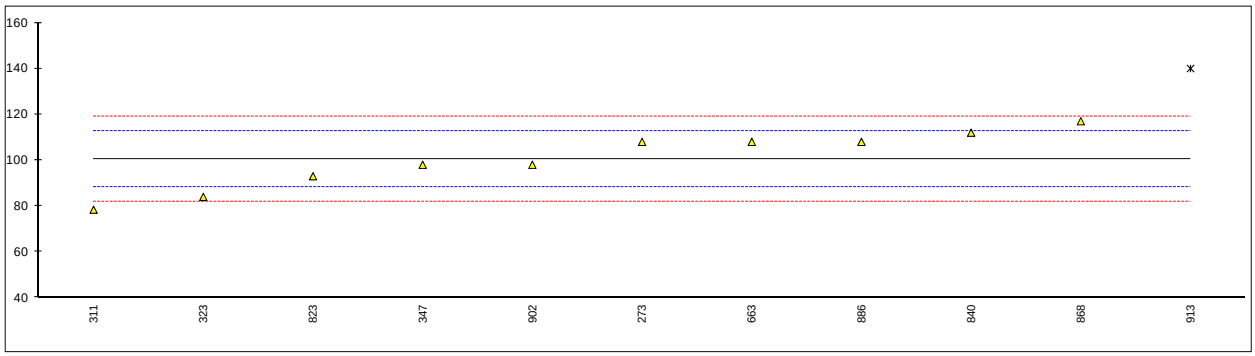


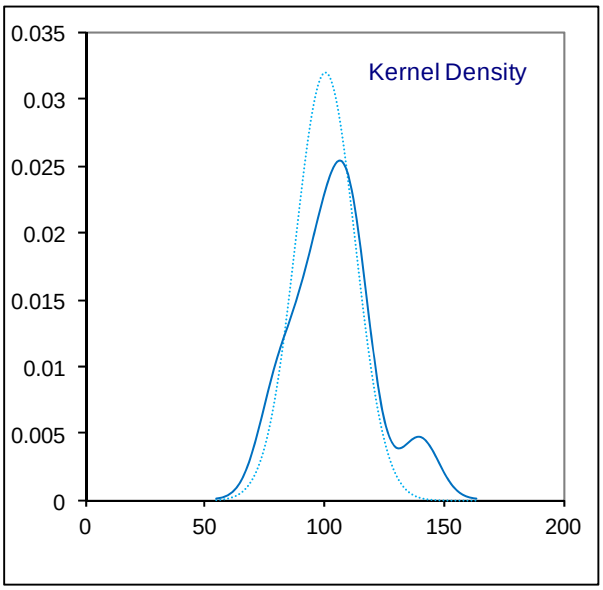


Determination of Water on sample #14085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273	E1064	108.0		1.23	
311	E1064	78.4		-3.59	
323	E1064	84		-2.68	
347	E1064	98		-0.40	
663	E203	108		1.23	
823	E1064	93		-1.21	
840	E1064	112	C	1.88	first reported: 136
868	E1064	117		2.70	
886	E1064	108		1.23	
902	E1064	98		-0.40	
913	E1064	140	G(0.05)	6.45	

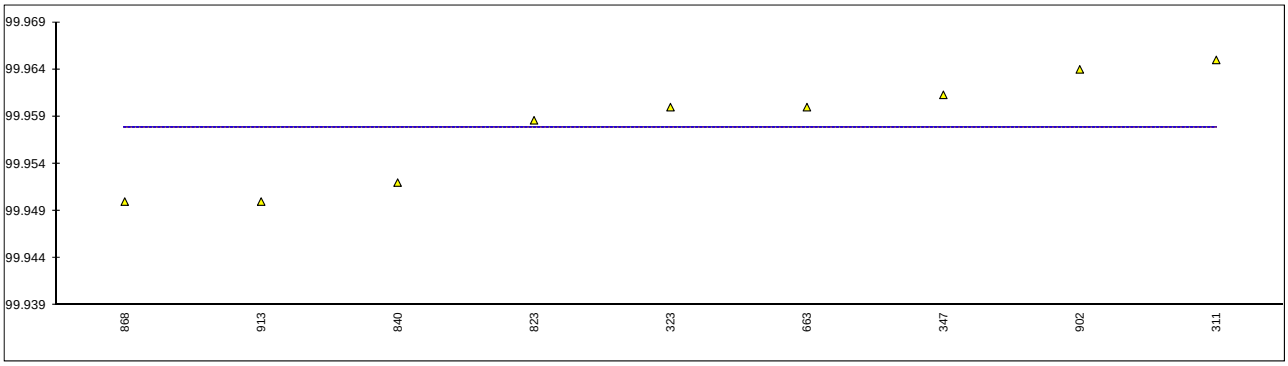
normality	suspect
n	10
outliers	1
mean (n)	100.44
st.dev. (n)	12.484
R(calc.)	34.95
R(E1064:12)	17.18

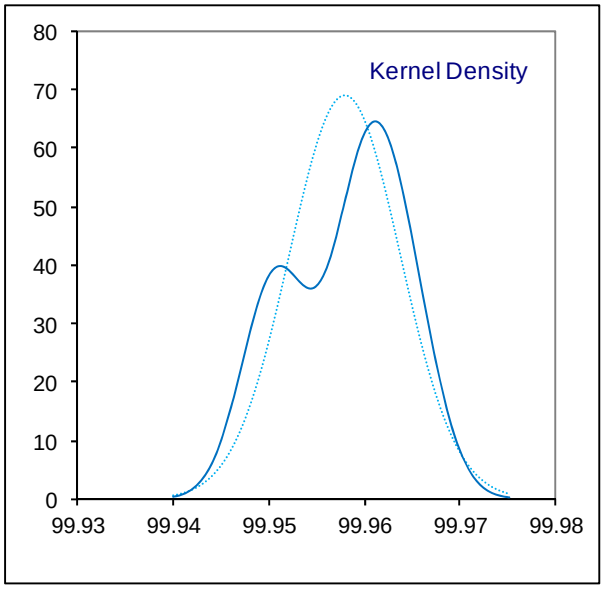




Determination of Purity (“as is” / as received) on sample #14085; results in %M/M

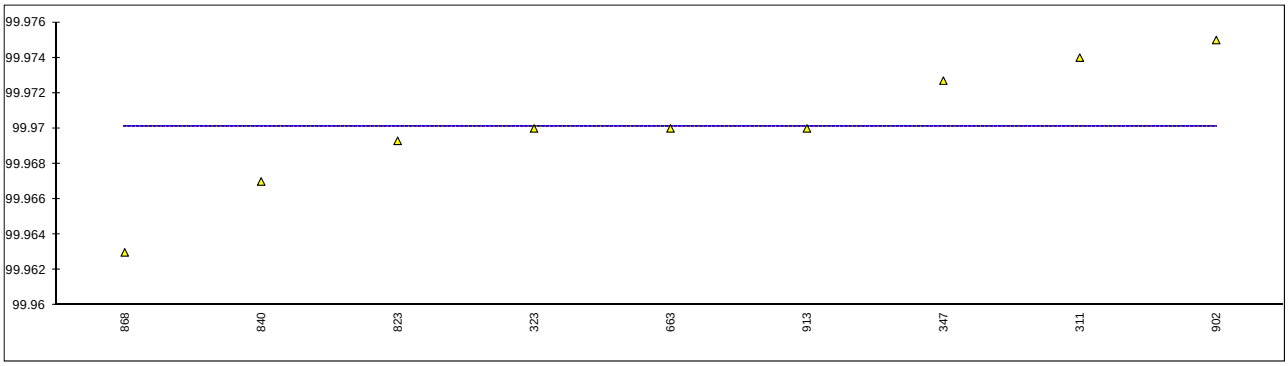
lab	method	value	mark	z(targ)	remarks
273		-----		-----	
311	INH-114	99.965		-----	
323	INH-CM	99.96		-----	
347	INH-0002	99.9613		-----	
663	INH-8001	99.96		-----	
823	INH-0002	99.9586		-----	
840	INH-0002	99.952		-----	
868	INH-044	99.950		-----	
886		-----		-----	
902	INH-80	99.964		-----	
913	INH-0002	99.95		-----	
	normality	OK			
	n	9			
	outliers	0			
	mean (n)	99.9579			
	st.dev. (n)	0.00579			
	R(calc.)	0.0162			
	R(lit)	unknown			Compare R(iis11C05) = 0.0124

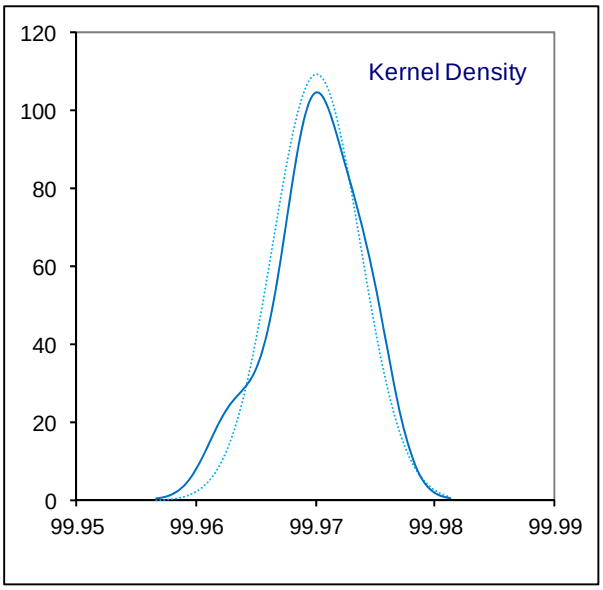




Determination of Purity (on dry basis) on sample #14085; results in %M/M

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
311	INH-114	99.974		-----	
323	INH-CM	99.97		-----	
347	INH-0002	99.9727		-----	
663	INH-8001	99.97		-----	
823	INH-0002	99.9693		-----	
840	INH-0002	99.967		-----	
868	INH-044	99.963		-----	
886		-----		-----	
902	INH-80	99.975		-----	
913	INH-0002	99.97		-----	
	normality	OK			
	n	9			
	outliers	0			
	mean (n)	99.9701			
	st.dev. (n)	0.00365			
	R(calc.)	0.0102			
	R(lit)	unknown			Compare R(iis11C05) = 0.0045





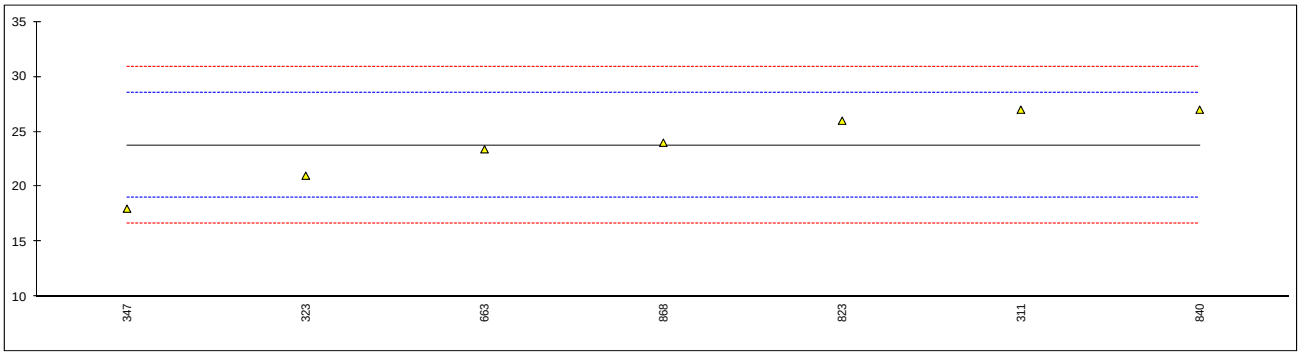
Determination of Acetone on sample #14085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
311	INH-114	<10		-----	
323	INH-CM	<10		-----	
347	INH-0002	<5		-----	
663	INH-8001	<1		-----	
823	INH-0002	1		-----	
840	INH-0002	0.8		-----	
868	INH-044	<10		-----	
886		-----		-----	
902		-----		-----	
913	INH-0002	<10		-----	
	normality	n.a			
	n	8			
	outliers	n.a			
	mean (n)	<10			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(Horwitz)	n.a			

Determination of Methanol on sample #14085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
311	INH-114	27	C	1.37	First reported 18
323	INH-CM	21		-1.17	
347	INH-0002	18		-2.44	
663	INH-8001	23.4	C	-0.16	first reported: 34.5
823	INH-0002	26		0.94	
840	INH-0002	27		1.37	
868	INH-044	24		0.10	
886		-----		-----	
902		-----		-----	
913		-----		-----	

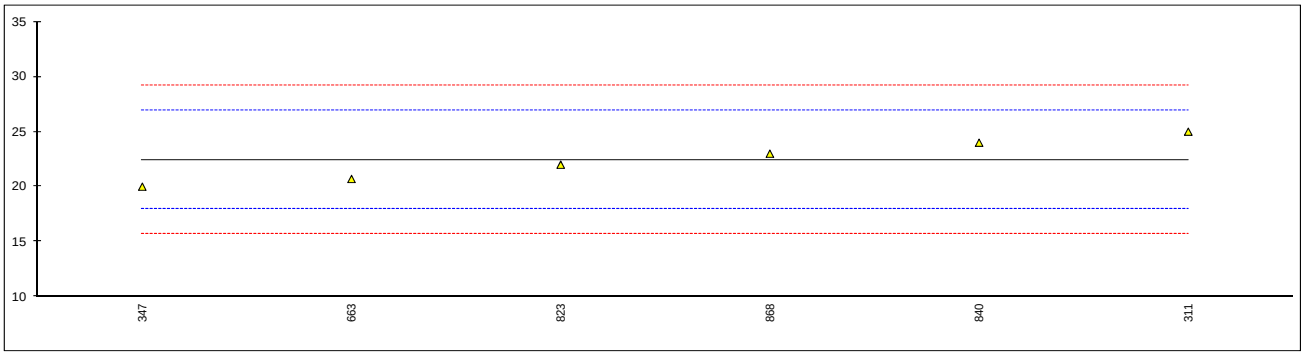
normality	unknown
n	7
outliers	0
mean (n)	23.77
st.dev. (n)	3.342
R(calc.)	9.36
R(Horwitz)	6.61



Determination of Methylacrylate on sample #14085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
311	INH-114	25	C	1.13	First reported 15
323		-----		-----	
347	INH-0002	20		-1.09	
663	INH-8001	20.7		-0.78	
823	INH-0002	22		-0.20	
840	INH-0002	24		0.69	
868	INH-044	23		0.24	
886		-----		-----	
902		-----		-----	
913		-----		-----	

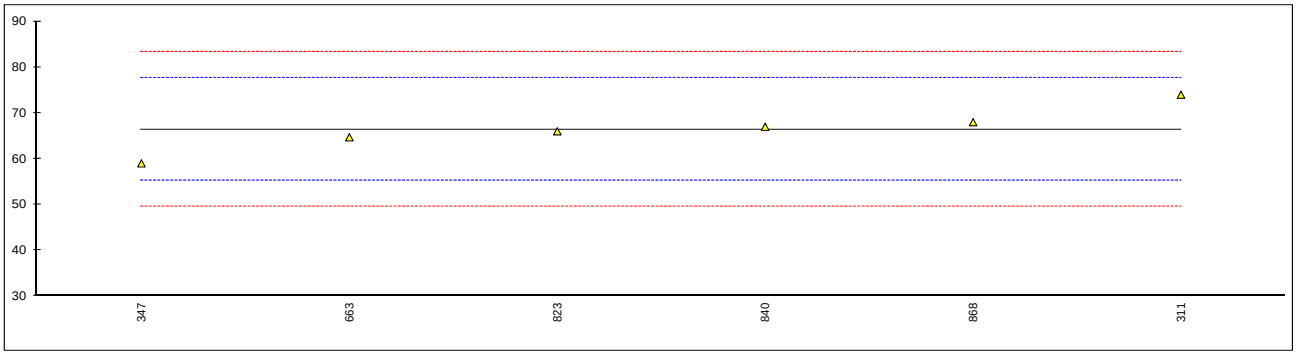
normality	unknown
n	6
outliers	0
mean (n)	22.45
st.dev. (n)	1.922
R(calc.)	5.38
R(Horwitz)	6.30



Determination of Methylisobutyrate on sample #14085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
311	INH-114	74	C	1.34	First reported 60
323		-----		-----	
347	INH-0002	59		-1.32	
663	INH-8001	64.7		-0.31	
823	INH-0002	66		-0.08	
840	INH-0002	67		0.10	
868	INH-044	68		0.27	
886		-----		-----	
902		-----		-----	
913		-----		-----	

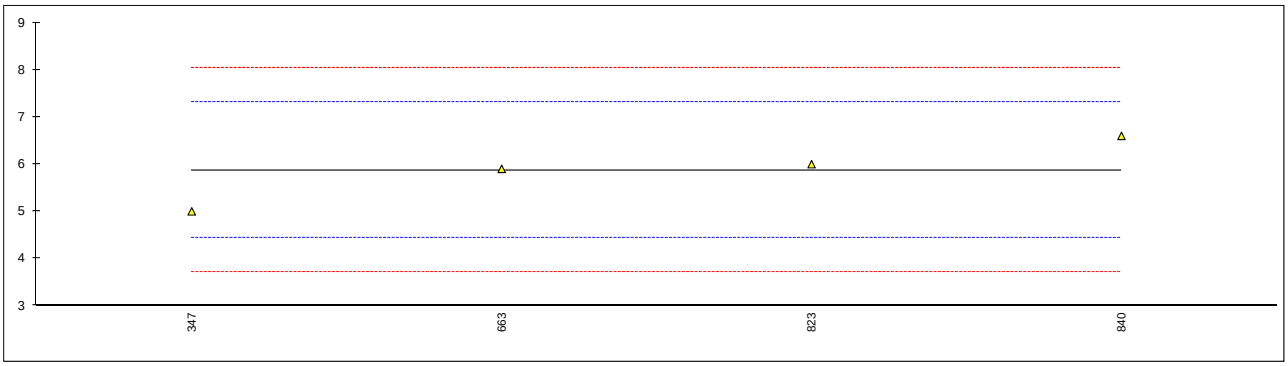
normality	unknown
n	6
outliers	0
mean (n)	66.45
st.dev. (n)	4.868
R(calc.)	13.63
R(Horwitz)	15.83



Determination of Ethylmethacrylate on sample #14085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
311	INH-114	<10		-----	
323	INH-CM	<10		-----	
347	INH-0002	5		-1.22	
663	INH-8001	5.9		0.03	
823	INH-0002	6		0.17	
840	INH-0002	6.6		1.01	
868	INH-044	<10		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	

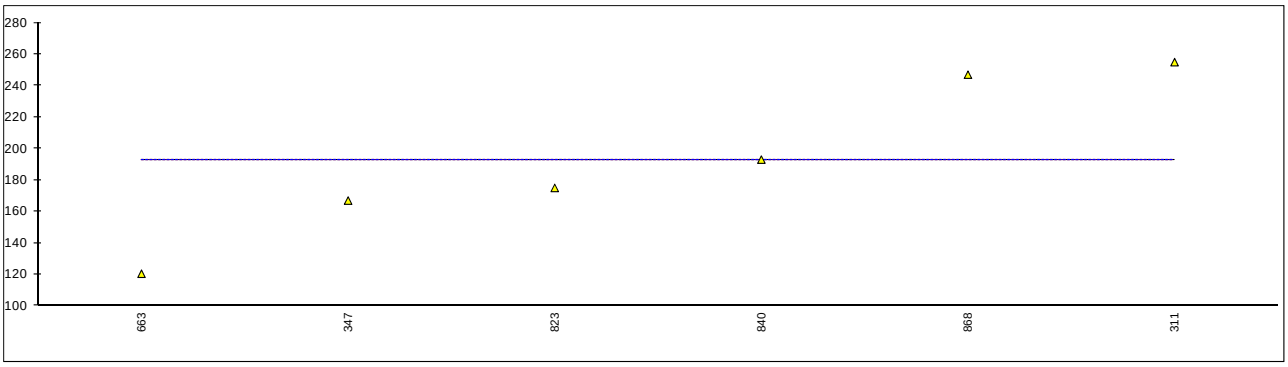
normality	unknown
n	4
outliers	0
mean (n)	5.88
st.dev. (n)	0.660
R(calc.)	1.85
R(Horwitz)	2.02



Determination of Other Impurities on sample #14085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
311	INH-114	255		-----	
323		-----		-----	
347	INH-0002	167		-----	
663	INH-8001	120.5		-----	
823	INH-0002	175		-----	
840	INH-0002	193		-----	
868	INH-044	247		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	

normality	unknown
n	6
outliers	0
mean (n)	192.92
st.dev. (n)	51.022
R(calc.)	142.86
R(lit)	unknown



APPENDIX 2

Number of participants per country

1 lab in BELGIUM
1 lab in CHINA, People's Republic
1 lab in INDIA
1 lab in NETHERLANDS
1 lab in SOUTH AFRICA
1 lab in SOUTH KOREA
1 lab in SPAIN
1 lab in TAIWAN
1 lab in THAILAND
1 lab in TURKEY
1 lab in VIETNAM

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect 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 outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from calculations
S	= scope of the reported method is not applicable
n.a.	= not applicable
U	= reported in different unit
W	= result withdrawn on request of the participant
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014

2 ASTM E178-02

3 ASTM E1301-03

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9 DIN 38402 T41/42

10 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)

11 J.N. Miller, Analyst, 118, 455, (1993)

12 Analytical Methods Committee Technical Brief, No4 January 2001

13 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson.

14 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)