

Results of Proficiency Test
Methylmethacrylate (MMA)
June 2016

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 Methylmetacrylate (MMA) during the annual proficiency testing program 2015/2016. In this interlaboratory study 12 laboratories from 11 different countries have registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2016 Methylmetacrylate 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 Spijkensisse, the Netherlands, was the organizer of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. In this proficiency test, the participants received one 0.5 litre sample of Methylmetacrylate (labelled #16110). 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 Spijkensisse, 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% confidentiality of participant's data. Also 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 April 2014 (iis-protocol, version 3.3). This protocol can be downloaded via the FAQ page of the iis website www.iisnl.com.

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 sample material of approximately 20 litre of Methylmetacrylate was obtained from a local supplier. From this batch, after homogenisation, 35 amber glass bottles of 0.5 litre (labelled #16110) were filled.

The homogeneity of the subsamples was checked by determination of Density at 20°C according to ISO12185 and by determination of Water according to ASTM E1064 on 4 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in mg/kg
sample #16110-1	0.94335	120
sample #16110-2	0.94334	118
sample #16110-3	0.94335	118
sample #16110-4	0.94335	115

table 1: homogeneity test results of subsamples #16110

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

	Density at 20°C in kg/L	Water in mg/kg
r (observed)	0.00001	6
reference test method	ISO12185:96	E1064:16
0.3 * R (reference test)	0.00015	6

table 2: evaluation of the repeatabilities of subsamples #16110

The calculated repeatabilities were less or equal to 0.3 times the reproducibilities of the corresponding reference test methods. Therefore, homogeneity of the subsamples #16110 was assumed.

One bottle of 0.5 L, labelled #16110 was dispatched to each of the participating laboratories on June 1, 2016.

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 #16110: Acidity (as Acrylic Acid), Appearance, Colour Pt/Co, Density at 20°C, Inhibitor as Topanol A, Water, Purity (both on "as-is" and on dry basis), Acetone, Ethylacrylate, Ethylmetacrylate, Methanol, Methylacrylate, Methylisobutyrate, Methylpropionate, Methyl-alpha-hydroxy-isobutyrate and Other Impurities.

To get comparable results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results. 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

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 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. 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 the original test results per determination were submitted subsequently to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of 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 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 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. 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. This 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 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 test result tables of appendix 1.

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare. Therefore, the usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test, no problems were encountered with the dispatch of the samples. All laboratories were able to report in time. Not all laboratories were able to perform all analyses requested. In total 12 participants reported 112 numerical results. Observed were 4 outlying results, which is 3.6% of the numerical results. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the reported 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 test results. 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. D1613) and an added designation for the year that the method was adopted or revised (e.g. D1613:06). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1613:06(2012)). In the results tables of appendix 1 only the method number and year of adoption or revision will be used.

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.

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 #16110, which was bright and clear and pass the test.

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 D1209:05(2011).

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

Inhibitor as Topanol A: 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.

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

Purity "as is": Regretfully, ASTM D3362 was withdrawn in 2011 with no replacement. As there is no other suitable standard with precision data available, it was decided to evaluate the group performance against ASTM D3362:05. This determination seems not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D3362:05.

Purity on dry basis: Regretfully, ASTM D3362 was withdrawn in 2011 with no replacement. As there is no other suitable standard with precision data available, it was decided to evaluate the group performance against ASTM D3362:05. This determination seems not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D3362:05.

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

Ethylacrylate: No significant conclusions were drawn. All laboratories agreed on a value of <5 mg/kg.

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

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

Methylacrylate: This determination may not be problematic. No statistical outliers were observed, but one result was excluded for statistical evaluation. However, the calculated reproducibility after rejection of the suspect data is in agreement with the estimated reproducibility, calculated using the Horwitz equation.

Methylisobutyrate: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

Methylpropionate: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the estimated reproducibility calculated using the Horwitz equation.

Methyl-alpha-hydroxy-isobutyrate: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full 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 reference test method and the reproducibility as found for the group of participating laboratories. The assigned values, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM, ISO, EN standards) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acrylic Acid	mg/kg	11	17.0	13.0	14.0
Appearance		12	pass	n.a.	n.a.
Colour Pt/Co		10	3.8	2.6	7.0
Density at 20°C	kg/L	12	0.9434	0.0003	0.0005
Inhibitor as Topanol A	mg/kg	7	5.0	2.9	1.8
Water	mg/kg	11	131	26	22
Purity "as is" / as received	%M/M	9	99.955	0.032	0.270
Purity (on dry basis)	%M/M	7	99.969	0.016	0.270
Acetone	mg/kg	4	5.2	5.5	(1.8)
Ethylacrylate	mg/kg	3	<5	n.a.	n.a.
Ethylmethacrylate	mg/kg	6	<10	n.a.	n.a.
Methanol	mg/kg	3	4.6	3.7	(1.6)
Methylacrylate	mg/kg	4	7.3	2.4	2.4
Methylisobutyrate	mg/kg	5	69	20	16
Methylpropionate	mg/kg	6	106	23	23
Methyl-alpha-hydro-isobutyrate	mg/kg	4	73	10	17
Other impurities	mg/kg	4	76	104	n.a.

table 3: reproducibilities of test results of sample #16110

results between brackets are near or below the lower detection limit

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2016 WITH PREVIOUS PTS

	June 2016	June 2014	May 2011	April 2009	April 1999
Number of reporting labs	12	11	11	11	10
Number of results reported	112	99	85	97	106
Statistical outliers	4	2	3	6	8
Percentage outliers	3.6%	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 reference test methods. The conclusions are given the following table:

	June 2016	June 2014	May 2011	April 2009	April 1999
Acidity as Acrylic Acid	+	++	+/-	-	-
Colour Pt/Co	++	++	++	++	++
Density at 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.
Methylisobutyrate	-	+	++	++	n.e.
Methylpropionate	+/-	n.e.	n.e.	n.e.	n.e.
Methyl-alpha-hydro-isobutyrate	++	n.e.	n.e.	n.e.	n.e.
Other impurities	n.e.	n.e.	n.e.	n.e.	n.e.

table 5: comparison determinations against the reference test method
results between brackets are near or below the lower detection limit

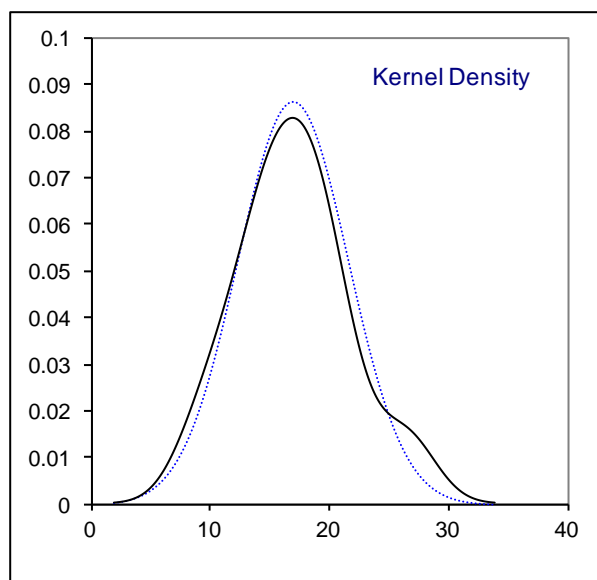
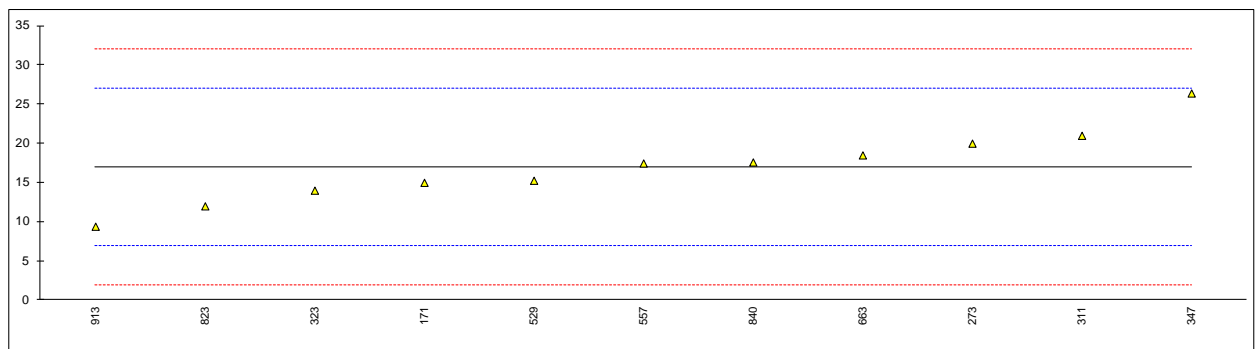
The performance of the determinations against the requirements of the respective reference test methods is listed in the above table. The following performance categories were used:

- ++: 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 #16110; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1613	15		-0.39	
273	D1613	20		0.61	
311	D1613	21		0.81	
323	D1613	14		-0.59	
347	D1613	26.4		1.89	
522		-----		-----	
529	D1613	15.264		-0.34	
557	D1613	17.4673417		0.10	
663	D1613	18.5		0.31	
823	D1613	12		-0.99	
840	D1613	17.6		0.13	
913	D1613	9.4		-1.51	
normality		OK			
n		11			
outliers		0			
mean (n)		16.97			
st.dev. (n)		4.636			
R(calc.)		12.98			
R(D1613:06)		14.00			



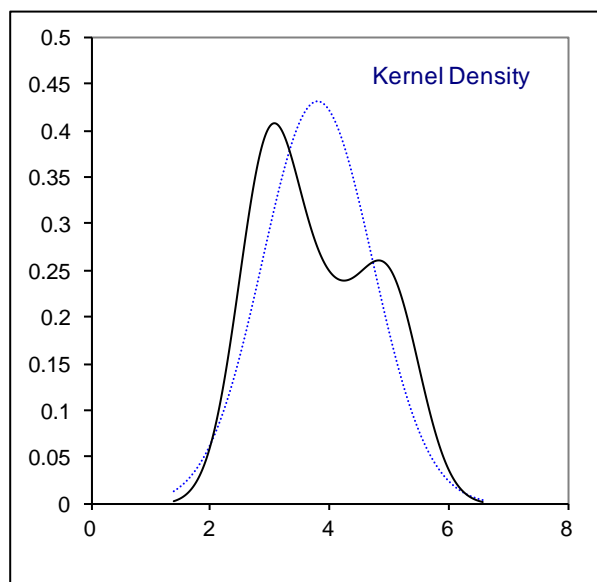
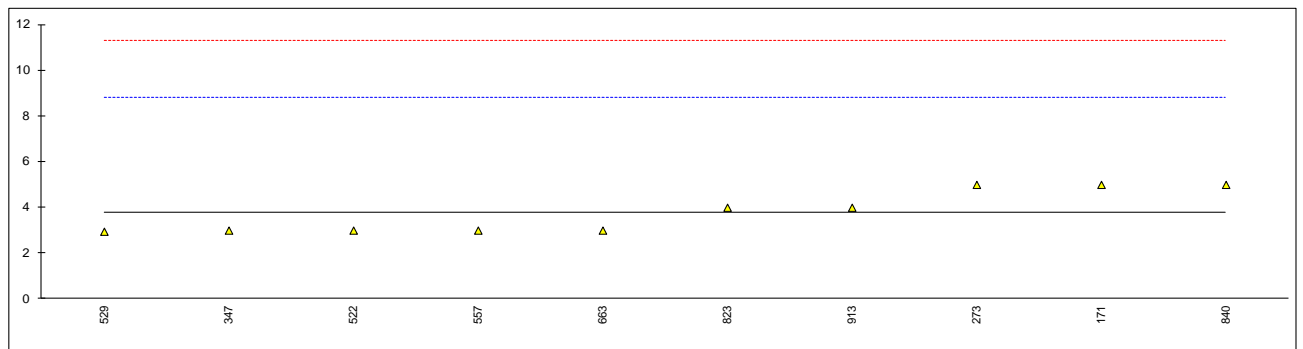
Determination of Appearance on sample #16110;

lab	method	value	mark	z(targ)	remarks
171	E2680	pass		----	
273	E2680	Pass		----	
311	E2680	pass		----	
323	Visual	clear&bright		----	
347	E2680	PASS		----	
522	Visual	pass		----	
529	E2680	Pass		----	
557	Visual	pass		----	
663	D4176	Bright&clear		----	
823	E2680	Pass		----	
840	E2680	Pass		----	
913	E2680	Clear		----	
	normality	n.a.			
	n	12			
	outliers	n.a.			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(E2680:09e1)	n.a.			

Determination of Colour Pt/Co on sample #16110;

lab	method	value	mark	z(targ)	remarks
171	D1209	5		0.48	
273	D1209	5		0.48	
311	D1209	<5		-----	
323	D1209	<5		-----	
347	D5386	3		-0.32	
522	D1219	3		-0.32	
529	D1209	2.95		-0.34	
557	D1209	3		-0.32	
663	D1209	3		-0.32	
823	D1209	4		0.08	
840	D1209	5		0.48	
913	D5386	4		0.08	

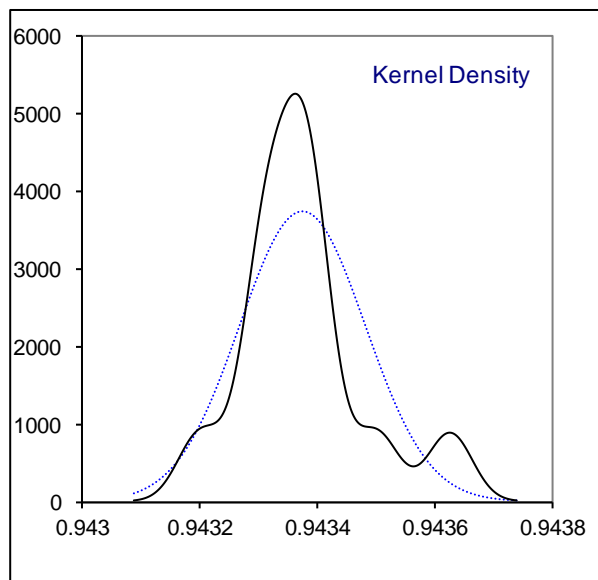
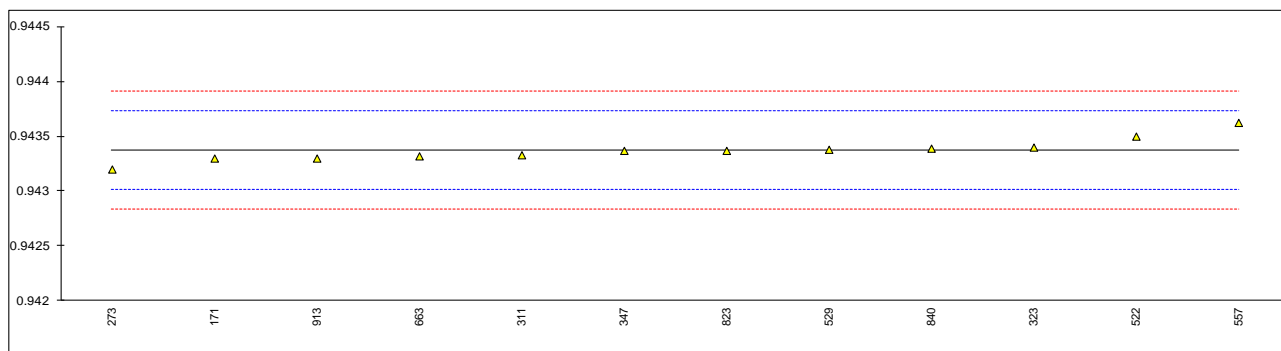
normality OK
n 10
outliers 0
mean (n) 3.80
st.dev. (n) 0.924
R(calc.) 2.59
R(D1209:05) 7.00



Determination of Density at 20°C on sample #16110; results in kg/L

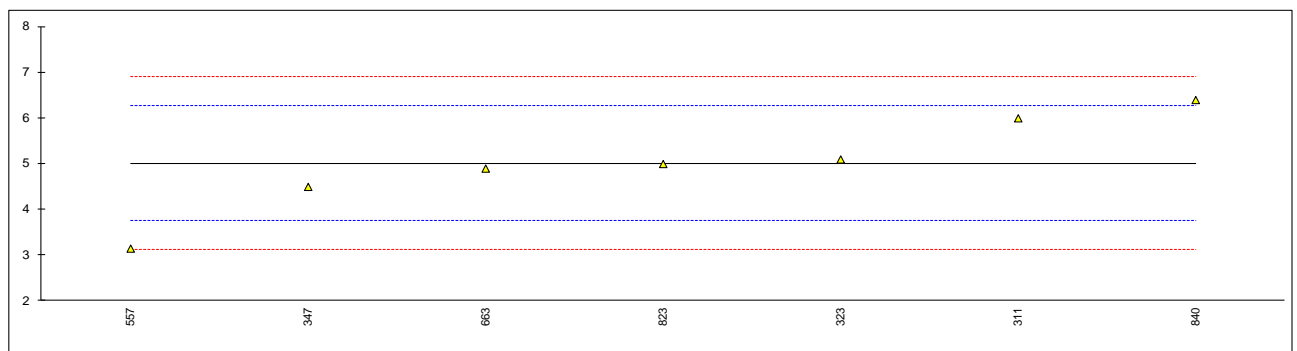
lab	method	value	mark	z(targ)	remarks
171	D4052	0.9433		-0.41	
273	D4052	0.9432		-0.97	
311	D4052	0.94333		-0.24	
323	D4052	0.9434		0.15	
347	D4052	0.94337		-0.02	
522	D4052	0.9435		0.71	
529	D4052	0.94338		0.04	
557	D4052	0.943625	C	1.41	First reported 0.944625
663	D4052	0.94332		-0.30	
823	ISO12185	0.94337		-0.02	
840	D4052	0.94339		0.09	
913	D4052	0.9433		-0.41	

normality not OK
 n 12
 outliers 0
 mean (n) 0.943374
 st.dev. (n) 0.0001070
 R(calc.) 0.000300
 R(ISO12185:96) 0.000500



Determination of Inhibitor as Topanol A on sample #16110; results in mg/kg

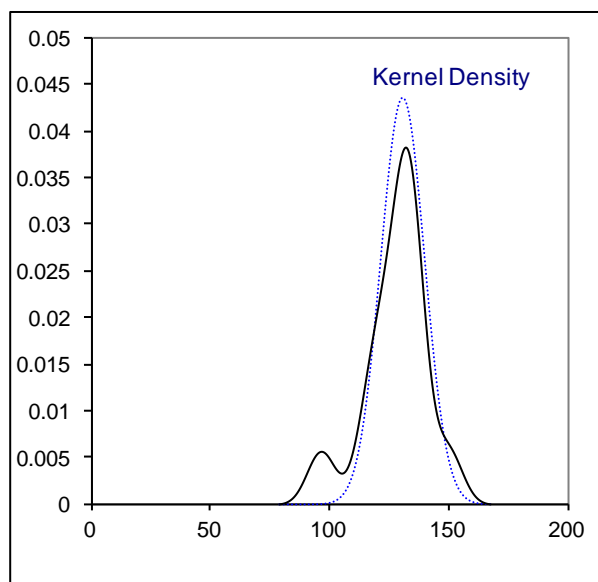
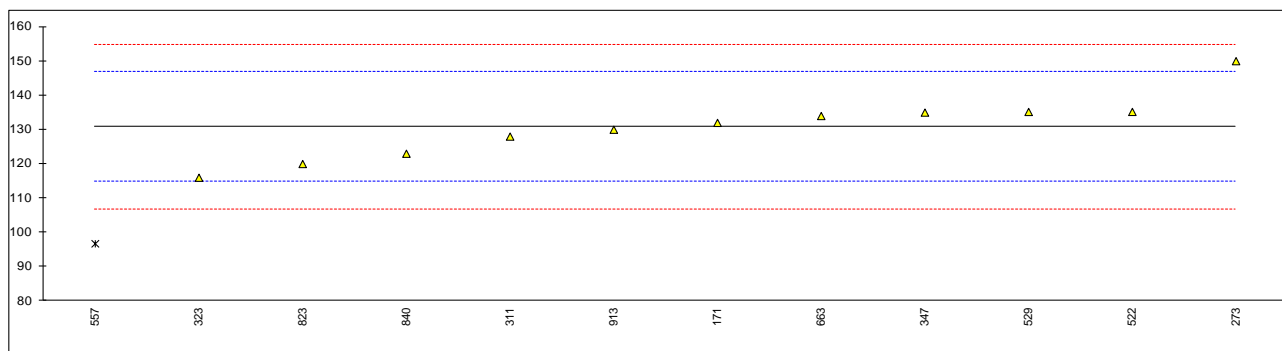
lab	method	value	mark	z(targ)	remarks
171		----		----	
273		----		----	
311	INH-510	6.0		1.58	
323	INH-0002	5.1		0.15	
347	INH-0002	4.5		-0.81	
522		----		----	
529		----		----	
557	INH-048	3.1498824		-2.95	
663	INH-8001	4.9		-0.17	
823	INH-2	5		-0.01	
840	INH-2	6.4		2.22	
913	INH-0002	<10		----	
	normality	unknown			
	n	7			
	outliers	0			
	mean (n)	5.01			
	st.dev. (n)	1.053			
	R(calc.)	2.95			
	R(Horwitz)	1.76			



Determination of Water on sample #16110; results in mg/kg

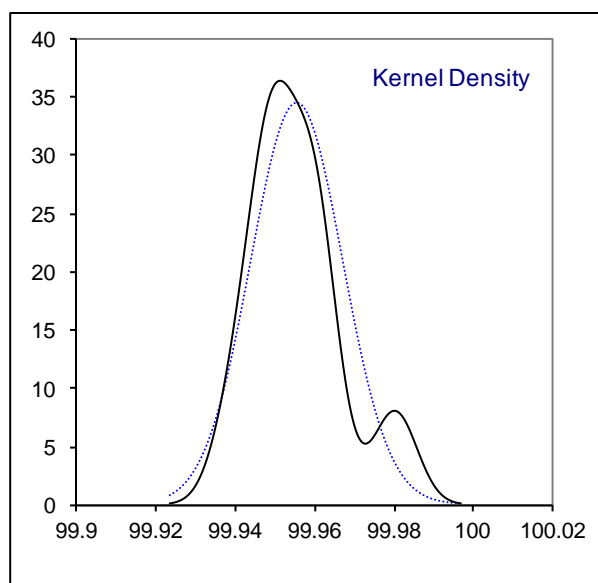
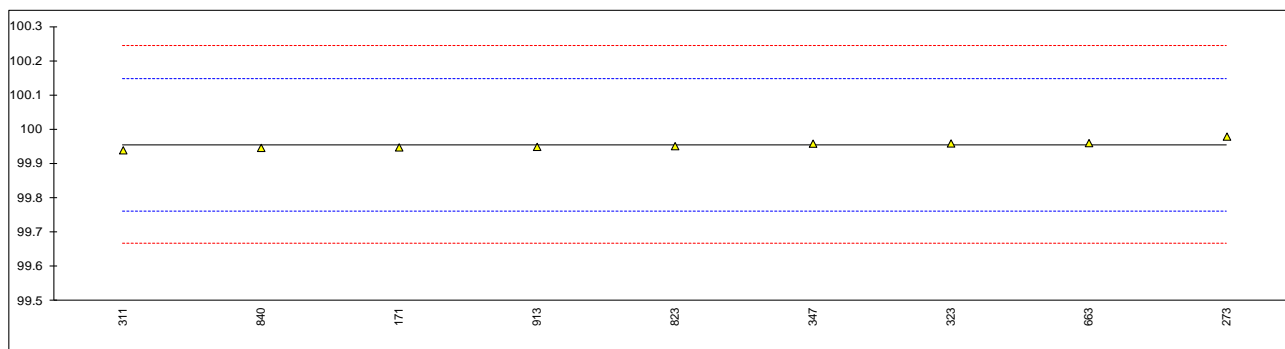
lab	method	value	mark	z(targ)	remarks
171	E1064	132		0.15	
273	E203	150		2.41	
311	E1064	128	C	-0.35	First reported 170
323	E1064	116		-1.85	
347	E1064	135		0.53	
522	E203	135.2		0.56	
529	E1064	135.19		0.55	
557	D1364	96.7348985	D(0.05)	-4.26	
663	E1064	134		0.41	
823	E1064	120		-1.35	
840	E1064	123		-0.97	
913	E1064	130		-0.10	

normality suspect
n 11
outliers 1
mean (n) 130.76
st.dev. (n) 9.174
R(calc.) 25.69
R(E1064:16) 22.36



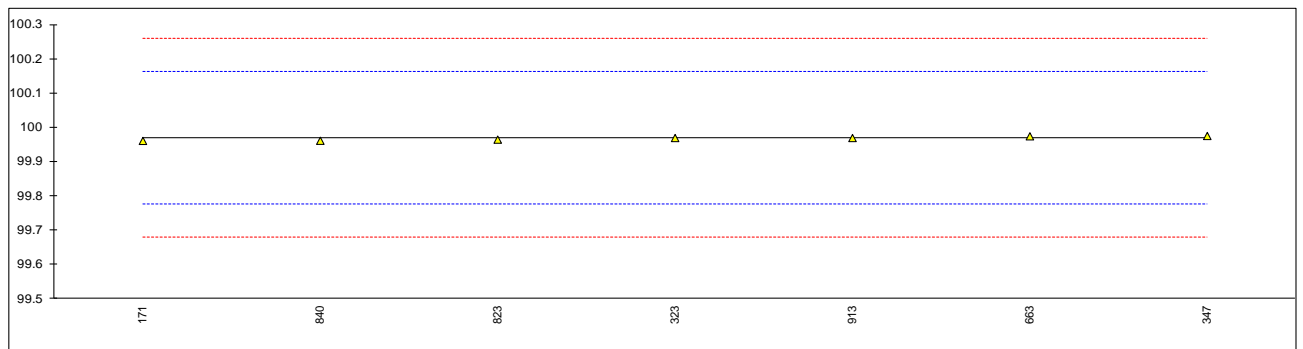
Determination of Purity (“as is” / as received) on sample #16110; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	INH-01	99.94845		-0.07	
273	INH-408	99.98		0.26	
311	INH-114	99.94		-0.16	
323	D3362Mod.	99.96		0.05	
347	INH-0002	99.9594		0.04	
522		----		----	
529		----		----	
557		----		----	
663	INH-8001	99.961		0.06	
823	INH-2	99.9521		-0.03	
840	INH-2	99.947		-0.09	
913	INH-0002	99.95		-0.06	
	normality	not OK			
	n	9			
	outliers	0			
	mean (n)	99.9553			
	st.dev. (n)	0.01156			
	R(calc.)	0.0324			
	R(D3362:05)	0.2700			Compare R(iis14C04) = 0.0162



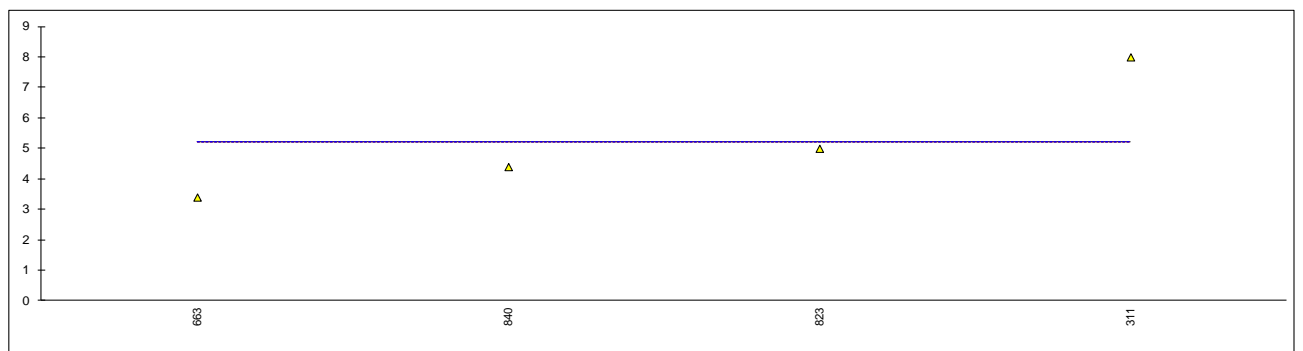
Determination of Purity (on dry basis) on sample #16110; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	INH-01	99.961650		-0.07	
273		----		----	
311		----		----	
323	D3362Mod.	99.97		0.01	
347	INH-0002	99.9760		0.08	
522		----		----	
529		----		----	
557		----		----	
663	INH-8001	99.975		0.07	
823	INH-2	99.9653		-0.03	
840	INH-2	99.962		-0.07	
913	INH-0002	99.97		0.01	
	normality	unknown			
	n	7			
	outliers	0			
	mean (n)	99.9686			
	st.dev. (n)	0.00581			
	R(calc.)	0.0163			
	R(D3362:05)	0.2700			



Determination of Acetone on sample #16110; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		----		----	
273		----		----	
311	INH-114	8		----	
323		----		----	
347	INH-0002	<5		----	
522		----		----	
529		----		----	
557		----		----	
663	INH-8001	3.4		----	
823	INH-2	5		----	
840	INH-2	4.4		----	
913		----		----	
	normality	Unknown			
	n	4			
	outliers	n.a.			
	mean (n)	5.20			
	st.dev. (n)	1.980			
	R(calc.)	5.54			
	R(Horwitz)	(1.82)			



Determination of Ethylacrylate on sample #16110; results in mg/kg

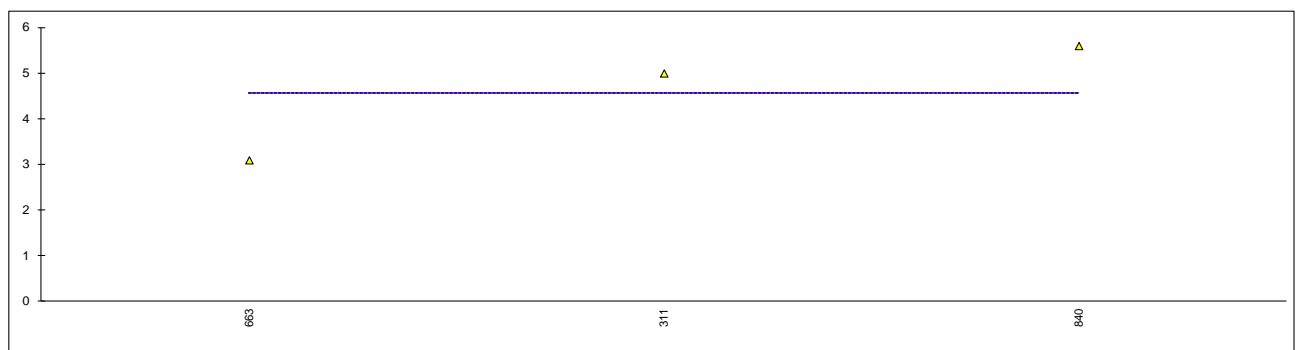
lab	method	value	mark	z(targ)	remarks
171		----		----	
273		----		----	
311		----		----	
323		----		----	
347	INH-0002	<5		----	
522		----		----	
529		----		----	
557		----		----	
663		----		----	
823	INH-2	<5		----	
840	INH-2	<2		----	
913		----		----	
	normality	unknown			
	n	3			
	outliers	n.a.			
	mean (n)	<5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	n.a.			

Determination of Ethylmethacrylate on sample #16110; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		----		----	
273		----		----	
311	INH-114	0		----	
323	D3362Mod.	<10		----	
347	INH-0002	<5		----	
522		----		----	
529		----		----	
557		----		----	
663	INH-8001	1.4		----	
823	INH-2	<5		----	
840	INH-2	3.2		----	
913		----		----	
	normality	unknown			
	n	6			
	outliers	n.a.			
	mean (n)	<10			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of Methanol on sample #16110; results in mg/kg

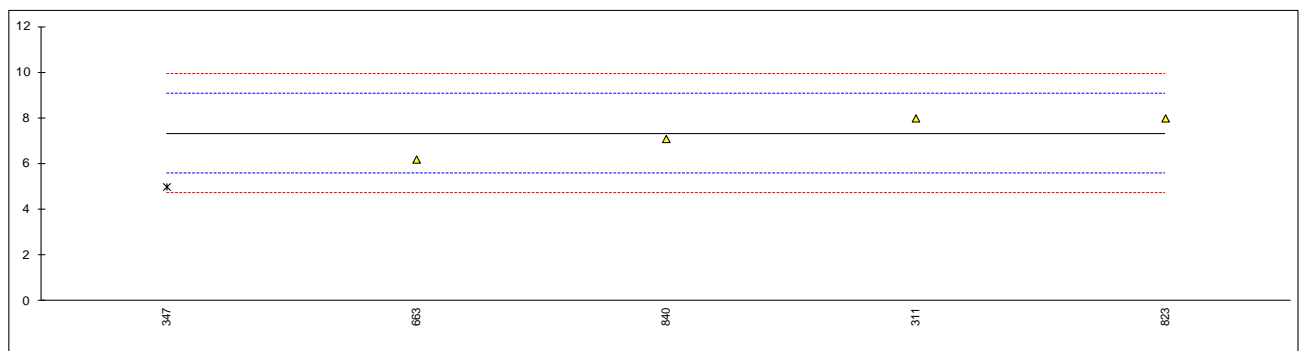
lab	method	value	mark	z(targ)	remarks
171	INH-01	<100		-----	
273				-----	
311	INH-114	5		-----	
323				-----	
347	INH-0002	<5		-----	
522				-----	
529				-----	
557				-----	
663	INH-8001	3.1		-----	
823	INH-2	<5		-----	
840	INH-2	5.6		-----	
913				-----	
	normality	Unknown			
	n	3			
	outliers	n.a.			
	mean (n)	4.57			
	st.dev. (n)	1.305			
	R(calc.)	3.65			
	R(Horwitz)	(1.63)			



Determination of Methylacrylate on sample #16110; results in mg/kg

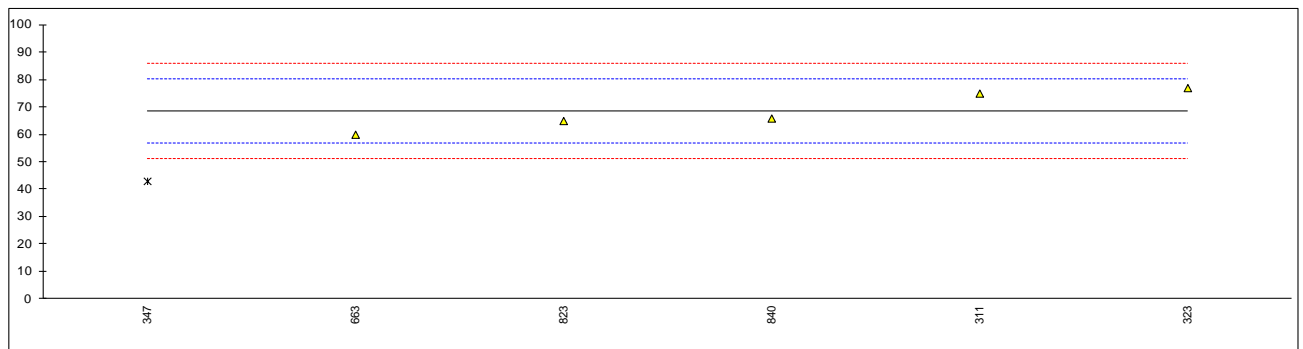
lab	method	value	mark	z(targ)	remarks
171	INH-01	<100		----	
273		----		----	
311	INH-114	8		0.78	
323		----		----	
347	INH-0002	5	ex	-2.68	Test result excluded,
522		----		----	
529		----		----	
557		----		----	
663	INH-8001	6.2		-1.30	
823	INH-2	8		0.78	
840	INH-2	7.1		-0.26	
913		----		----	
	normality	unknown			
	n	4			
	outliers	0 (+1 excl)			
	mean (n)	7.32			
	st.dev. (n)	0.862			
	R(calc.)	2.41			
	R(Horwitz)	2.43			

Lab 347: result excluded as other reported impurities were statistical outliers. Determination of impurities is not independent of each other.



Determination of Methylisobutyrate on sample #16110; results in mg/kg

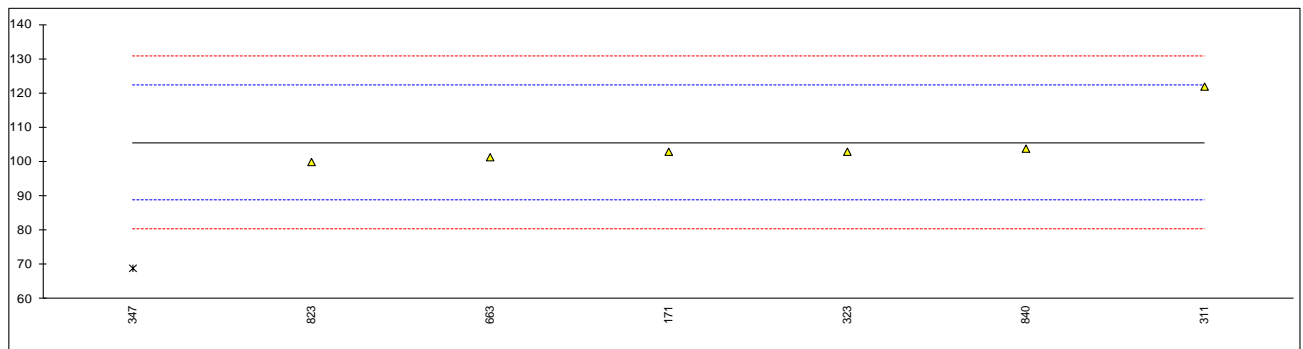
lab	method	value	mark	z(targ)	remarks
171		----		----	
273		----		----	
311	INH-114	75		1.11	
323	D3362Mod.	77		1.45	
347	INH-0002	43	G(0.05)	-4.41	
522		----		----	
529		----		----	
557		----		----	
663	INH-8001	60.0		-1.48	
823	INH-2	65		-0.62	
840	INH-2	65.9		-0.46	
913		----		----	
normality		unknown			
n		5			
outliers		1			
mean (n)		68.58			
st.dev. (n)		7.172			
R(calc.)		20.08			
R(Horwitz)		16.26			



Determination of Methylpropionate on sample #16110; results in mg/kg

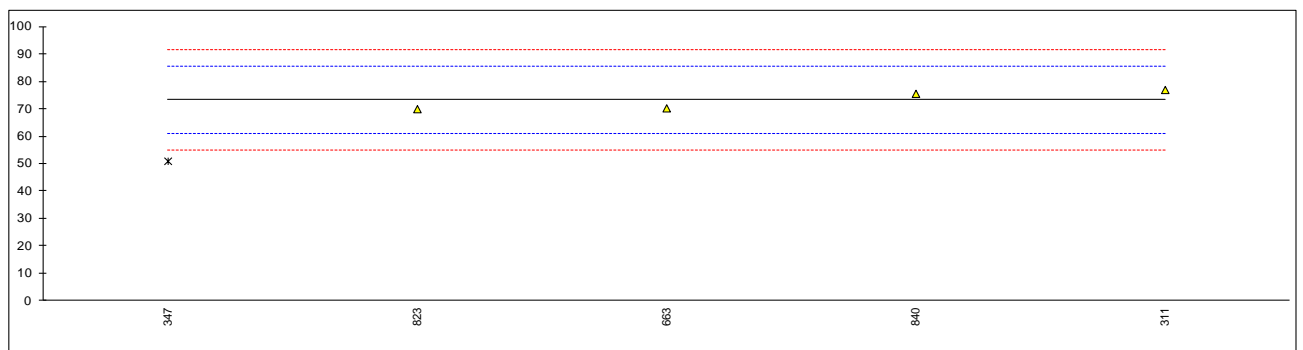
lab	method	value	mark	z(targ)	remarks
171	INH-01	103		-0.30	
273		----		----	
311	INH-114	122		1.96	
323	D3362Mod.	103		-0.30	
347	INH-0002	69	D(0.05)	-4.36	
522		----		----	
529		----		----	
557		----		----	
663	INH-8001	101.4		-0.50	
823	INH-2	100		-0.66	
840	INH-2	103.9		-0.20	
913		----		----	

normality unknown
n 6
outliers 1
mean (n) 105.55
st.dev. (n) 8.177
R(calc.) 22.90
R(Horwitz) 23.45



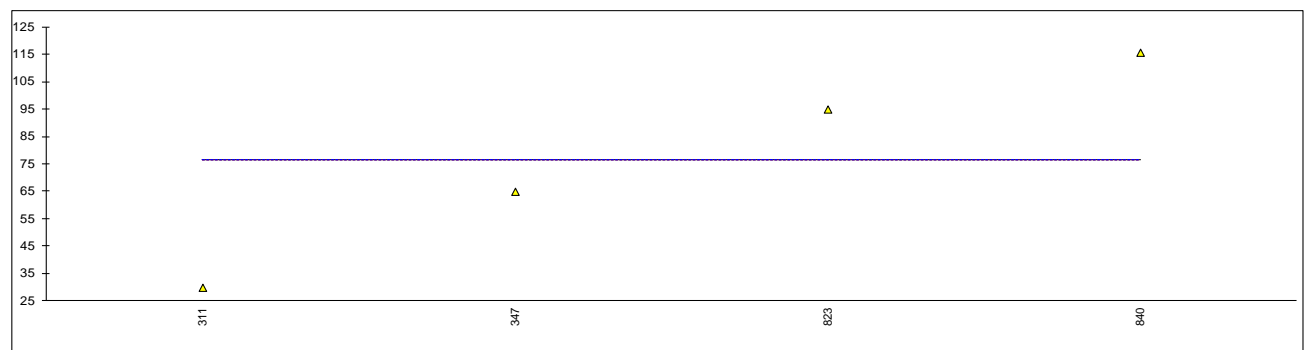
Determination of Methyl-alpha-hydroxy-isobutyrate sample #16110; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		----		----	
273		----		----	
311	INH-114	77		0.61	
323		----		----	
347	INH-0002	51	D(0.05)	-3.62	
522		----		----	
529		----		----	
557		----		----	
663	INH-8001	70.3		-0.48	
823	INH-2	70		-0.53	
840	INH-2	75.6		0.39	
913		----		----	
	normality	unknown			
	n	4			
	outliers	1			
	mean (n)	73.22			
	st.dev. (n)	3.598			
	R(calc.)	10.08			
	R(Horwitz)	17.19			



Determination of Other Impurities on sample #16110; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		----		----	
273		----		----	
311	INH-114	30		----	
323		----		----	
347	INH-0002	65		----	
522		----		----	
529		----		----	
557		----		----	
663		----		----	
823	INH-2	95	C	----	First reported 350
840	INH-2	115.7		----	
913		----		----	
normality		unknown			
n		4			
outliers		0			
mean (n)		76.43			
st.dev. (n)		37.298			
R(calc.)		104.43			
R(lit)		unknown			



APPENDIX 2

Number of participants per country

1 lab in BELGIUM
1 lab in BRAZIL
1 lab in INDIA
2 labs in MEXICO
1 lab in NETHERLANDS
1 lab in SOUTH AFRICA
1 lab in SOUTH KOREA
1 lab in SPAIN
1 lab in THAILAND
1 lab in UNITED STATES OF AMERICA
1 lab in VIETNAM

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= 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
ex	= test result excluded from calculations
S	= scope of reported method is not applicable
n.a.	= not applicable
n.e.	= not evaluated
U	= test result probably reported in a different unit
W	= test 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 E178:08
- 3 ASTM E1301:03
- 4 ISO 5725:86
- 5 ISO 5725, parts 1-6, 1994
- 6 Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)
- 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 The Royal Society of Chemistry 2002, Analyst 2002, 127, 1359-1364, P.J. Lowthian and M. Thompson.
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)