Results of Proficiency Test
Methylmethacrylate (MMA)
January 2018

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 2017/2018. In this interlaboratory study 16 laboratories from 13 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2018 Methylmethacrylate proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analysis for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one bottle of 0.5L of Methylmethacrylate (labelled #18002). Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. 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 March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary sample material of approximately 20 litre of Methylmethacrylate was obtained from a local supplier. From this batch, after homogenisation, 34 amber glass bottles of 0.5 L were filled and labelled #18002.
The homogeneity of the subsamples was checked by determination of Density at 20°C according to ISO12185.

	Density at 20°C in kg/L
sample #18002-1	0.94338
sample #18002-2	0.94338
sample #18002-3	0.94338
sample #18002-4	0.94338

table 1: homogeneity test results of subsamples #18002

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 at 20°C in kg/L
r (observed)	0.00000
reference test method	ISO12185:96
0.3 * R (reference test method)	0.00015

table 2: evaluation of the repeatability of subsamples #18002

The calculated repeatability was less than 0.3 times the reproducibility of the corresponding reference test method. Therefore, homogeneity of the subsamples #18002 was assumed.

One bottle of 0.5 L, labelled #18002 was dispatched to each of the participating laboratories on January 24, 2018.

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 #18002: Acidity (as Acrylic Acid), Appearance, Colour Pt/Co, Density at 20°C, Inhibitor as Topanol A, Water, Purity (on dry basis – acidity and/or on dry basis), Acetone, Ethylacrylate, Ethylmethacrylate, Methanol, Methylacrylate, Methylisobutyrate, Methylpropionate, Methyl alpha-Hydroxy Isobutyrate and Other Impurities.

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

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

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.
Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the 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 March 2017 (iis-protocol, version 3.4).

For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

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

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.
Finally, the reproducibilities were calculated from the standard deviations by multiplying 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 was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.
The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.
When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

$z_{(target)}$ = (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.
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. One participant did not report any test results and one participant reported the test results after the final reporting date. Not all laboratories were able to perform all analyses requested. In total 15 participants reported 126 numerical results. Observed were 3 outlying results, which is 2.4% 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 test methods, which are used by the different laboratories, are considered for explaining the observed differences when possible and applicable. These test methods are also listed 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 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(2011)). In the test results tables of appendix 1 only the test method number and year of adoption or revision (e.g. D1209:05) are 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 D1613:17.

Appearance: No analytical problems were observed. All laboratories agreed about the appearance of sample #18002, which was bright and clear and passes the test.

Colour 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(2011).

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

Inhibitor as Topanol A: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good 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 on dry basis – acidity and Purity on dry basis: 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 group performance against ASTM D3362:05. These determinations seem not problematic. No statistical outliers were observed. The calculated reproducibilities are in 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. Only a few test results were reported.

Ethylmethacrylate: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated using the Horwitz equation. The low number of reported test results may cause the higher reproducibility.

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

Methylisobutyrate: 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.

Methylpropionate: 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.

Methyl alpha-Hydroxy Isobutyrate: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated using the Horwitz equation.

Other Impurities: 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.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the calculated reproducibility (2.8 * sd) as declared by the relevant reference test methods and the reproducibility (R (lit)) as found for the group of participating laboratories. The average test results of the evaluated parameters, calculated reproducibilities and reproducibilities, derived from reference test methods (in casu ASTM, EN, ISO and IP reference methods) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acrylic Acid	mg/kg	13	12.7	11.5	14
Appearance		15	pass	n.a.	n.a.
Colour Pt/Co		11	3.1	2.6	7
Density at 20°C	kg/L	15	0.9434	0.0001	0.0005
Inhibitor as Topanol A	mg/kg	12	9.7	2.2	3.2
Water	mg/kg	14	192	37	32
Purity on dry basis - acidity	%M/M	12	99.940	0.048	0.27
Purity on dry basis	%M/M	9	99.953	0.067	0.27
Ethylmethacrylate	mg/kg	4	3.3	1.6	1.2
Methanol	mg/kg	5	4.0	2.4	1.5
Methylacrylate	mg/kg	7	86.2	23.7	19.7
Methylisobutyrate	mg/kg	6	199.5	50.7	40.3
Methylpropionate	mg/kg	7	13.8	4.7	4.2
Methyl alpha-Hydro Isobutyrate	mg/kg	5	63.0	9.7	15.1
Other impurities	mg/kg	3	229.3	139.9	101.4

table 3: reproducibilities of test results of sample #18002

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 JANUARY 2018 WITH PREVIOUS PTS

	January 2018	June 2016	June 2014	May 2011	April 2009
Number of reporting labs	15	12	11	11	11
Number of results reported	126	112	99	85	97
Statistical outliers	3	4	2	3	6
Percentage outliers	2.4%	3.6%	2.0%	3.5%	6.2%

table 4: comparison with previous proficiency tests

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

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

	January 2018	June 2016	June 2014	May 2011	April 2009
Acidity as Acrylic Acid	+	+	++	+/-	-
Colour Pt/Co	++	++	++	++	++
Density at 20°C	++	++	++	++	++
Inhibitor as Topanol A	+	--	++	-	++
Water	-	-	--	++	++
Purity on dry basis - acidity	++	++	n.e.	n.e.	n.e.
Purity on dry basis	++	++	n.e.	n.e.	n.e.
Acetone	n.e.	(--)	n.e.	n.e.	n.e.
Ethylacrylate	n.e.	n.e.	n.e.	n.e.	n.e.
Ethylmethacrylate	n.e.	n.e.	n.e.	n.e.	n.e.
Methanol	-	(--)	-	n.e.	n.e.
Methylacrylate	-	+/-	+	+/-	-
Methylisobutyrate	-	-	+	++	++
Methylpropionate	-	+/-	n.e.	n.e.	n.e.
Methyl-alpha-hydro-isobutyrate	+	++	n.e.	n.e.	n.e.
Other impurities	-	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 method is listed in the above table. The following performance categories were used:

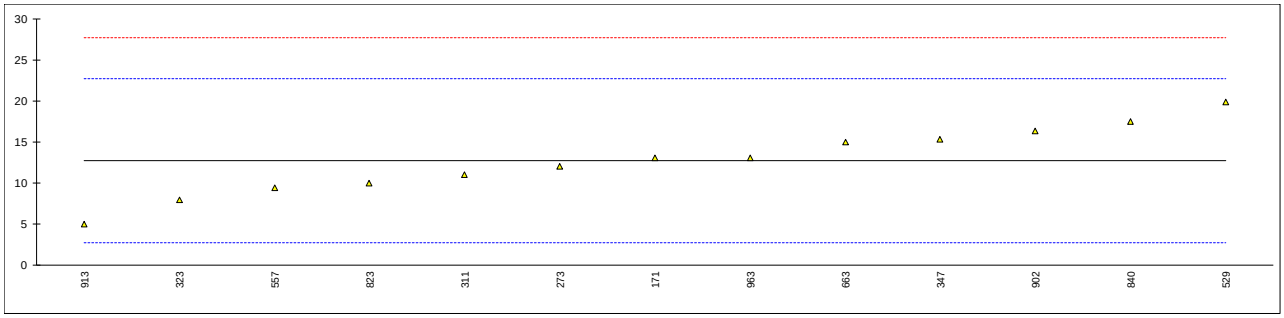
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test method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

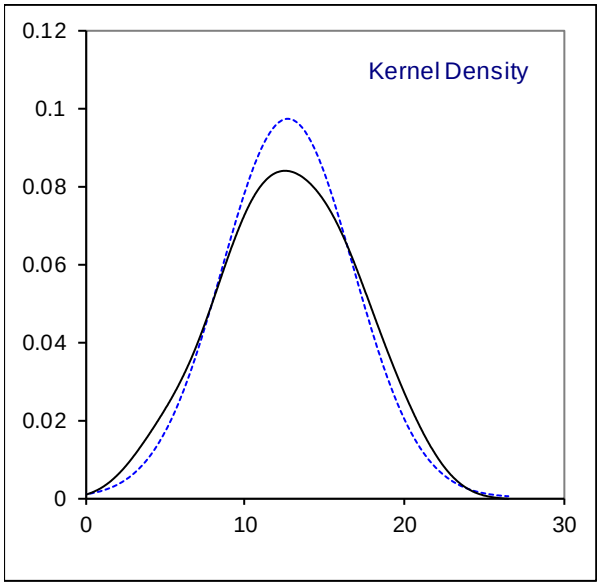
APPENDIX 1

Determination of Acidity as Acrylic Acid on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1613	13		0.05	
273	D1613	12		-0.15	
311	D1613	11		-0.35	
323	D1613	8		-0.95	
347	D1613	15.3		0.51	
522		-----		-----	
529	D1613	19.87		1.43	
557	D1613	9.48443		-0.65	
663	D1613	15.0		0.45	
823	D1613	10		-0.55	
840	D1613	17.5		0.95	
902	D1613	16.3		0.71	
913	D1613	5		-1.55	
962		-----		-----	
963	D1613	13		0.05	
1067		-----		-----	

normality	OK
n	13
outliers	0
mean (n)	12.73
st.dev. (n)	4.103
R(calc.)	11.49
st.dev.(D1613:17)	5
R(D1613:17)	14





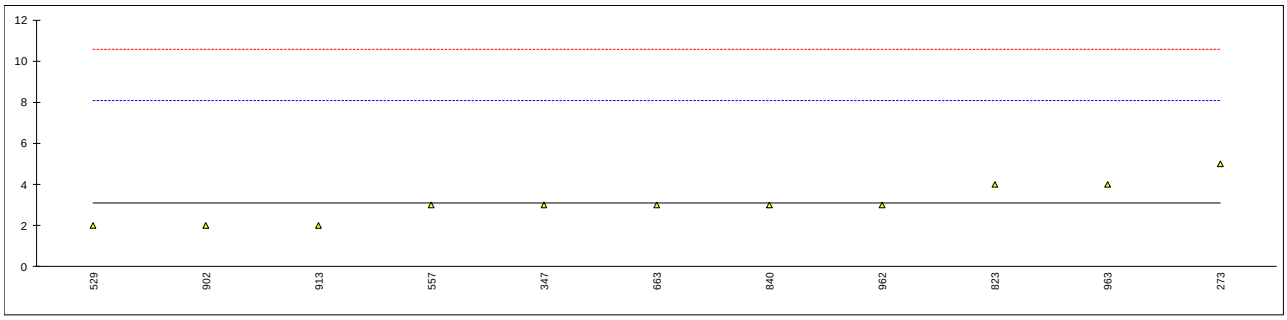
Determination of Appearance on sample #18002;

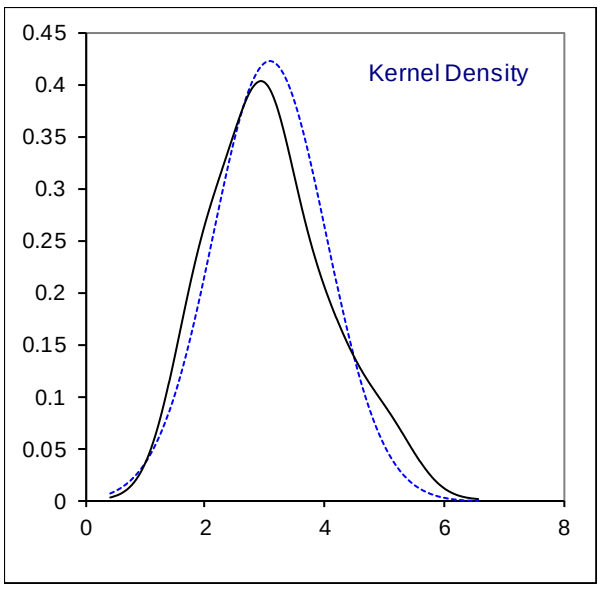
lab	method	value	mark	z(targ)	remarks
171	E2680	Pass		-----	
273	Visual	Pass		-----	
311	E2680	pass		-----	
323	D4176	clear & bright		-----	
347	E2680	Pass		-----	
522		-----		-----	
529	Visual	pass		-----	
557	Visual	Pass		-----	
663	Visual	Pass		-----	
823	E2680	Pass		-----	
840	E2680	Pass		-----	
902	E2680	PASS		-----	
913	E2680	CFSM		-----	
962	D4176	BCSFM		-----	
963	E2680	Pass		-----	
1067	Visual	Bright & Clear		-----	
	normality	n.a.			
	n	15			
	outliers	n.a.			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	st.dev.(E2680:16)	n.a.			
	R(E2680:16)	n.a.			

Determination of Colour Pt/Co on sample #18002;

lab	method	value	mark	z(targ)	remarks
171	D1209	<5		-----	
273	D1209	5		0.76	
311	D1209	<5		-----	
323	D1209	<5		-----	
347	D5386	3		-0.04	
522		-----		-----	
529	D1209	2		-0.44	
557	D1209	3		-0.04	
663	D1209	3		-0.04	
823	D5386	4		0.36	
840	D1209	3		-0.04	
902	D5386	2		-0.44	
913	D5386	2		-0.44	
962	D1209	3		-0.04	
963	D1209	4		0.36	
1067	D1209	< 5		-----	

normality	OK
n	11
outliers	0
mean (n)	3.09
st.dev. (n)	0.944
R(calc.)	2.64
st.dev.(D1209:05)	2.500
R(D1209:05)	7

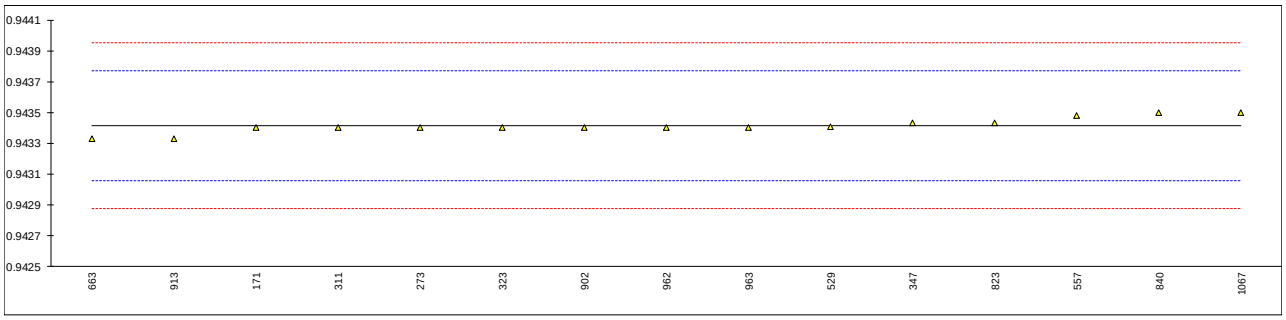


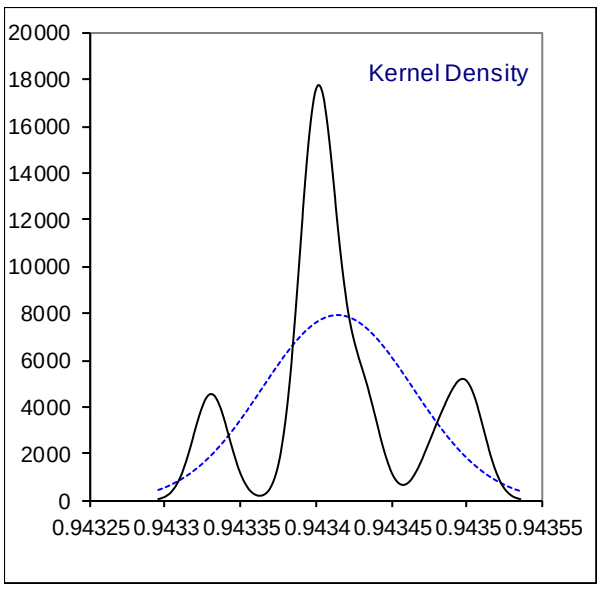


Determination of Density at 20°C on sample #18002; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9434		-0.08	
273	D4052	0.9434		-0.08	
311	D4052	0.9434		-0.08	
323	D4052	0.9434		-0.08	
347	D4052	0.94343		0.09	
522		-----		-----	
529	D4052	0.94341		-0.02	
557	D4052	0.94348		0.37	
663	D4052	0.94333		-0.47	
823	D4052	0.94343		0.09	
840	D4052	0.94350		0.48	
902	D4052	0.9434		-0.08	
913	D4052	0.94333		-0.47	
962	ISO12185	0.9434		-0.08	
963	D4052	0.9434		-0.08	
1067	ISO12185	0.9435		0.48	

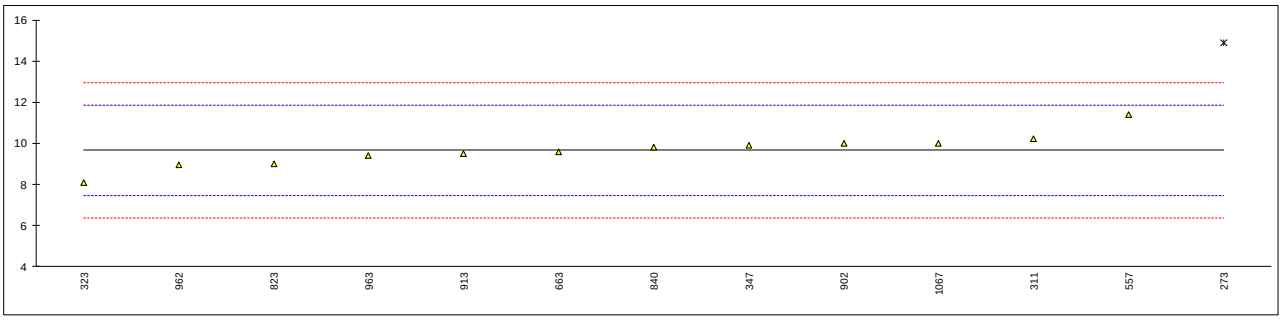
normality	OK
n	15
outliers	0
mean (n)	0.943414
st.dev. (n)	0.0000501
R(calc.)	0.000140
st.dev.(ISO12185:96)	0.0001786
R(ISO12185:96)	0.0005

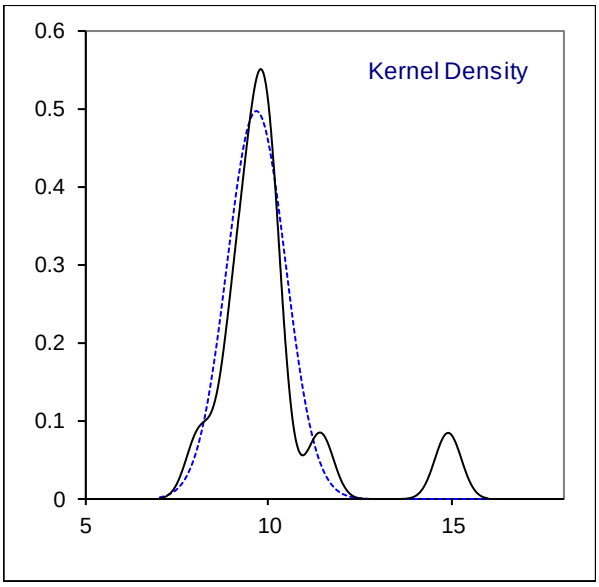




Determination of Inhibitor as Topanol A on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273	INH-2	14.9	C,D(0.01)	4.78	first reported 6
311	INH-510	10.2		0.50	
323	INH-09	8.1		-1.42	
347	INH-2	9.9		0.22	
522		-----		-----	
529		-----		-----	
557	INH-048	11.4		1.59	
663	INH-8001	9.6		-0.05	
823	INH-2	9		-0.60	
840		9.8		0.13	
902	INH-2	10		0.31	
913		9.5		-0.14	
962		8.97		-0.62	
963	In house	9.4		-0.23	
1067	In house	10		0.31	
	normality	suspect			
	n	12			
	outliers	1			
	mean (n)	9.66			
	st.dev. (n)	0.802			
	R(calc.)	2.24			
	st.dev.(Horwitz)	1.137			
	R(Horwitz)	3.18			

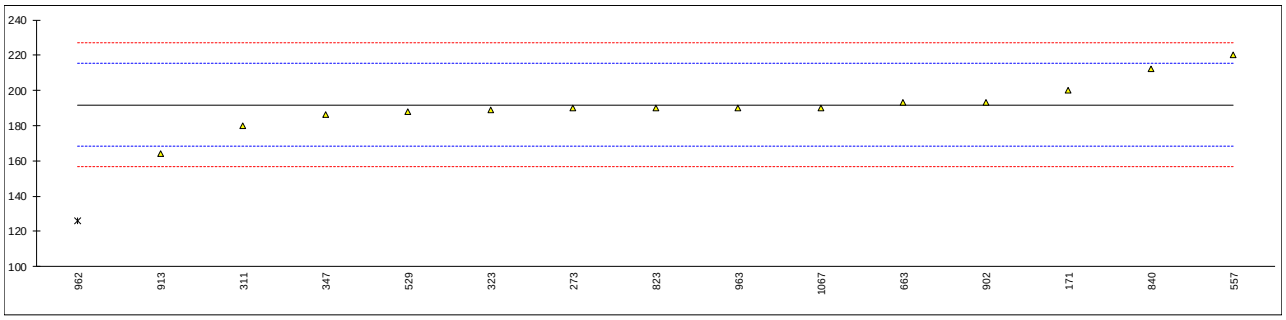


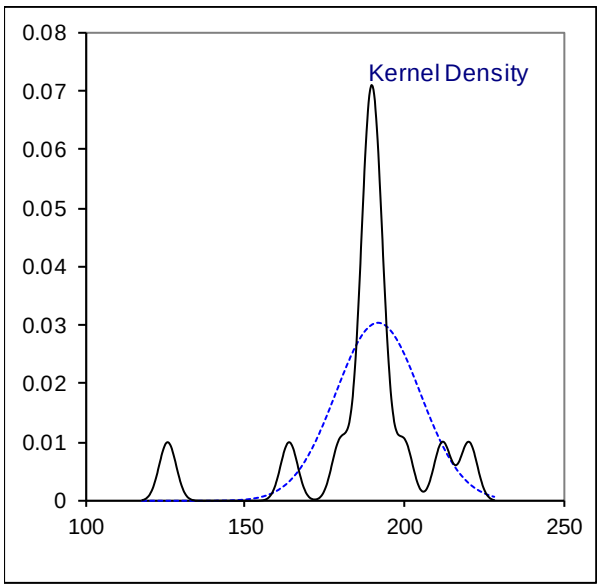


Determination of Water, Coulometric KF titration on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	E1064	200		0.70	
273	203	190	C	-0.15	first reported 250
311	E1064	180		-1.01	
323	E1064	189		-0.24	
347	E1064	186.2		-0.48	
522		-----		-----	
529	E1064	187.96		-0.33	
557	E1064	220.2		2.42	
663	E1064	193		0.10	
823	E1064	190		-0.15	
840	E1064	212		1.72	
902	E1064	193		0.10	
913	E1064	164		-2.37	
962	E1064	126	D(0.01)	-5.62	
963	E1064	190		-0.15	
1067	E1064	190		-0.15	

normality	suspect
n	14
outliers	1
mean (n)	191.81
st.dev. (n)	13.196
R(calc.)	36.95
st.dev.(E1064:16)	11.446
R(E1064:16)	32.05

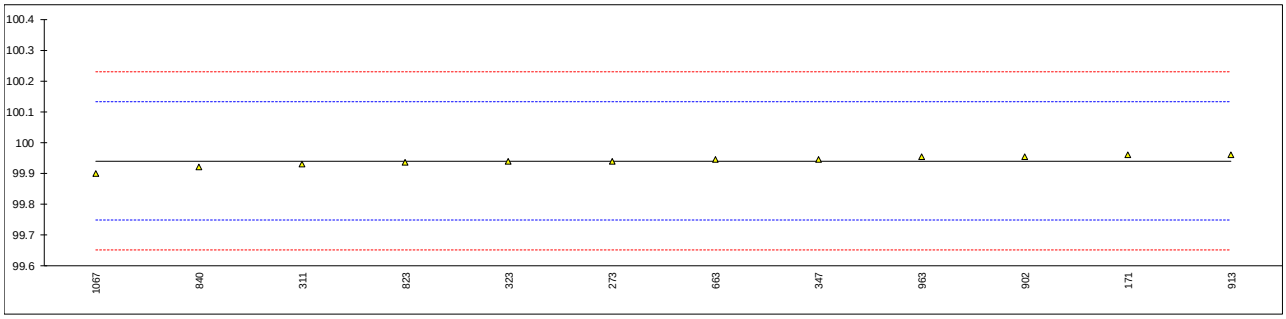


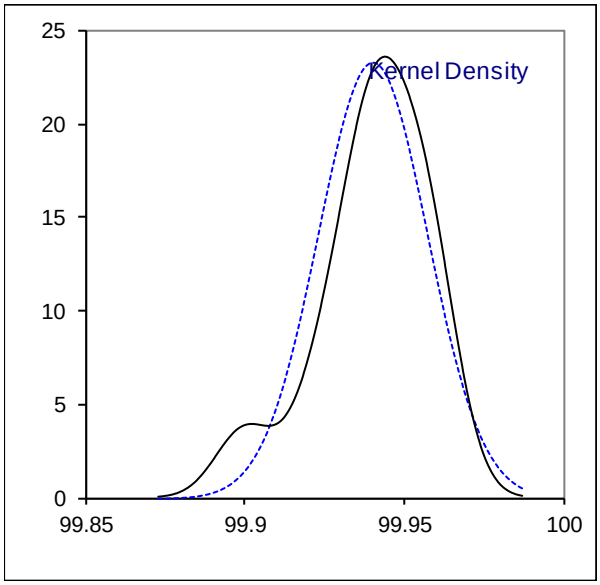


Determination of Purity*) on sample #18002; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	INH-01	99.96		0.20	
273	INH-408	99.94		0.00	
311	INH-2	99.93		-0.11	
323	D3362Mod.	99.94		0.00	
347	INH-2	99.9445		0.04	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663	INH-8001	99.944		0.04	
823	INH-2	99.9362		-0.04	
840		99.922		-0.19	
902	INH-2	99.954		0.14	
913		99.96		0.20	
962		-----		-----	
963	In house	99.953		0.13	
1067	In house	99.9		-0.42	
	normality	suspect			
	n	12			
	outliers	0			
	mean (n)	99.9403			
	st.dev. (n)	0.01716			
	R(calc.)	0.0480			
	st.dev.(D3362:05)	0.09643			
	R(D3362:05)	0.27			

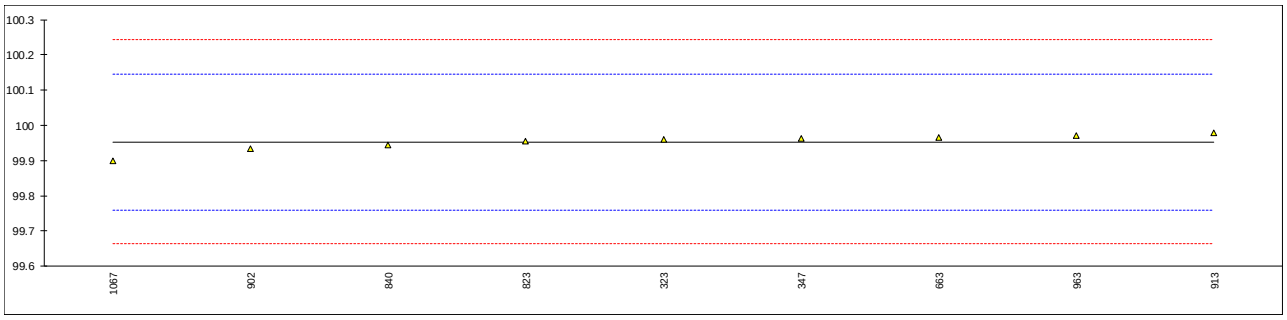
*) Purity= 100% - impurities – water – acidity

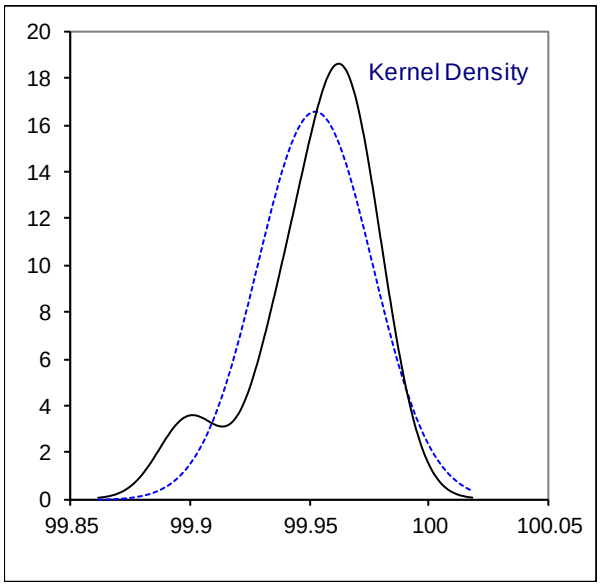




Determination of Purity on dry basis on sample #18002; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
311		-----		-----	
323	D3362Mod.	99.96		0.08	
347	INH-2	99.9631		0.11	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663	INH-8001	99.965		0.13	
823	INH-2	99.9552		0.03	
840		99.943		-0.10	
902	INH-2	99.935		-0.18	
913		99.98		0.28	
962		-----		-----	
963	In house	99.972		0.20	
1067	In house	99.9		-0.55	
	normality	not OK			
	n	9			
	outliers	0			
	mean (n)	99.9526			
	st.dev. (n)	0.02403			
	R(calc.)	0.0673			
	st.dev.(D3362:05)	0.09643			
	R(D3362:05)	0.27			





Determination of Acetone on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
311	INH-2	<10		-----	
323		-----		-----	
347	INH-23	<10		-----	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663	INH-8001	<1.47		-----	
823	INH-2	0		-----	
840		1.7		-----	
902	INH-2	<5		-----	
913		ND		-----	
962		-----		-----	
963		-----		-----	
1067		-----		-----	

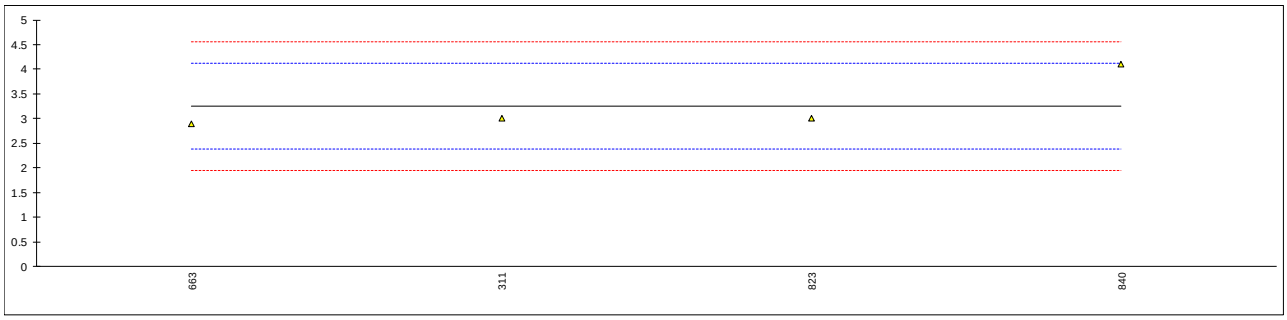
Determination of Ethylacrylate on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01	<100		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
347	INH-2	<10		-----	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663		-----		-----	
823	INH-2	0		-----	
840		<1		-----	
902	INH-2	<5		-----	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1067		-----		-----	

Determination of Ethylmethacrylate on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
311	INH-2	3	C	-0.57	first reported 23
323	D3362Mod.	<10		-----	
347	INH-2	<10		-----	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663	INH-8001	2.9		-0.80	
823	INH-2	3		-0.57	
840		4.1		1.95	
902	INH-2	<5		-----	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1067		-----		-----	

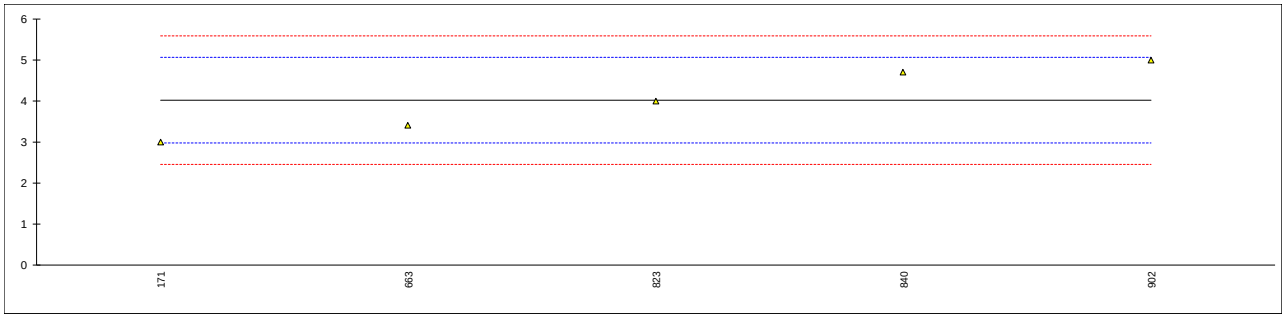
normality	unknown
n	4
outliers	0
mean (n)	3.25
st.dev. (n)	0.569
R(calc.)	1.59
st.dev.(Horwitz)	0.435
R(Horwitz)	1.22



Determination of Methanol on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01	3		-1.96	
273		-----		-----	
311	INH-2	<10		-----	
323		-----		-----	
347	INH-2	<10		-----	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663	INH-8001	3.4		-1.19	
823	INH-2	4		-0.04	
840		4.7		1.30	
902	INH-2	5		1.88	
913		ND		-----	
962		-----		-----	
963		-----		-----	
1067		-----		-----	

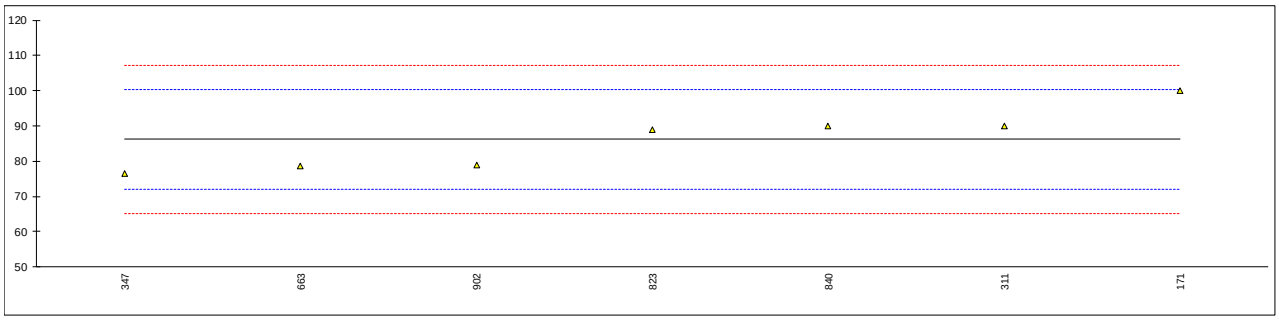
normality	unknown
n	5
outliers	0
mean (n)	4.02
st.dev. (n)	0.844
R(calc.)	2.36
st.dev.(Horwitz)	0.522
R(Horwitz)	1.46



Determination of Methylacrylate on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01	100		1.96	
273		-----		-----	
311	INH-2	90		0.55	
323		-----		-----	
347	INH-2	76.5		-1.37	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663	INH-8001	78.7		-1.06	
823	INH-2	89		0.40	
840		89.9		0.53	
902	INH-2	79		-1.02	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1067		-----		-----	

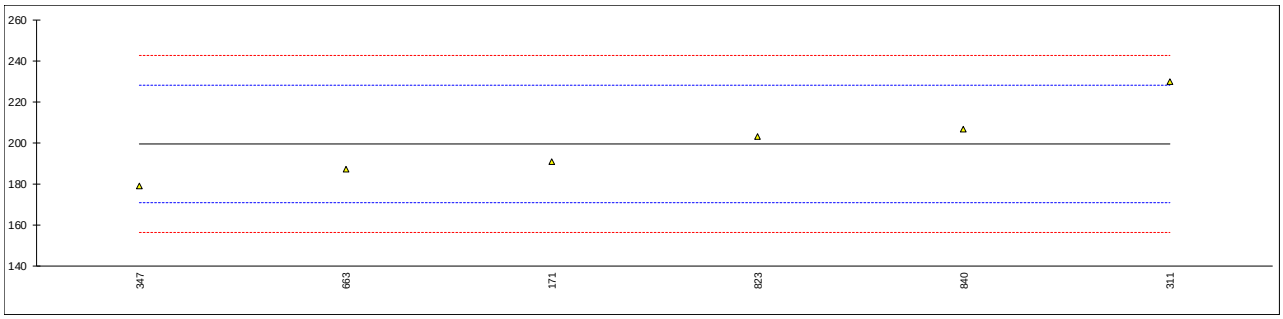
normality	unknown
n	7
outliers	0
mean (n)	86.16
st.dev. (n)	8.452
R(calc.)	23.66
st.dev.(Horwitz)	7.049
R(Horwitz)	19.74



Determination of Methylisobutyrate on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01	191		-0.59	
273		-----		-----	
311	INH-2	230		2.12	
323		-----		-----	
347	INH-2	179.0		-1.42	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663	INH-8001	187.1		-0.86	
823	INH-2	203		0.24	
840		206.8		0.51	
902		-----		-----	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1067		-----		-----	

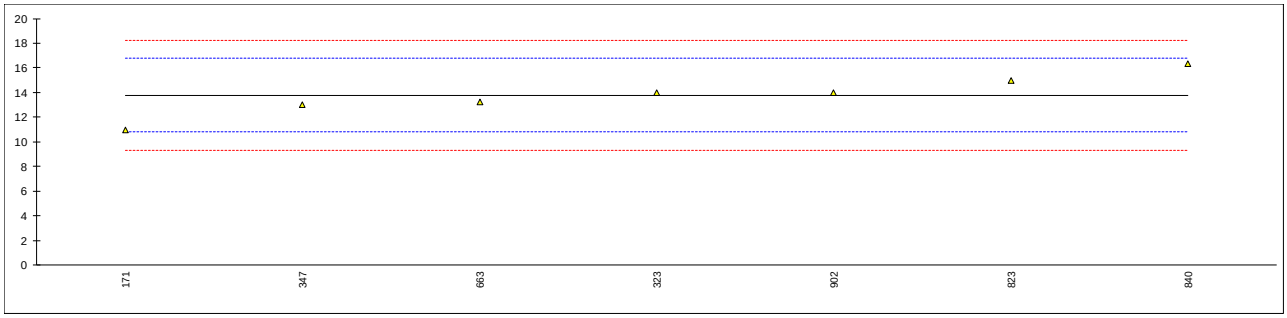
normality	unknown
n	6
outliers	0
mean (n)	199.48
st.dev. (n)	18.122
R(calc.)	50.74
st.dev.(Horwitz)	14.383
R(Horwitz)	40.27



Determination of Methylpropionate on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01	11		-1.87	
273		-----		-----	
311		-----		-----	
323	D3362Mod.	14		0.14	
347	INH-2	13.0		-0.53	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663	INH-8001	13.2		-0.39	
823	INH-2	15		0.82	
840		16.3		1.69	
902	INH-2	14		0.14	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1067		-----		-----	

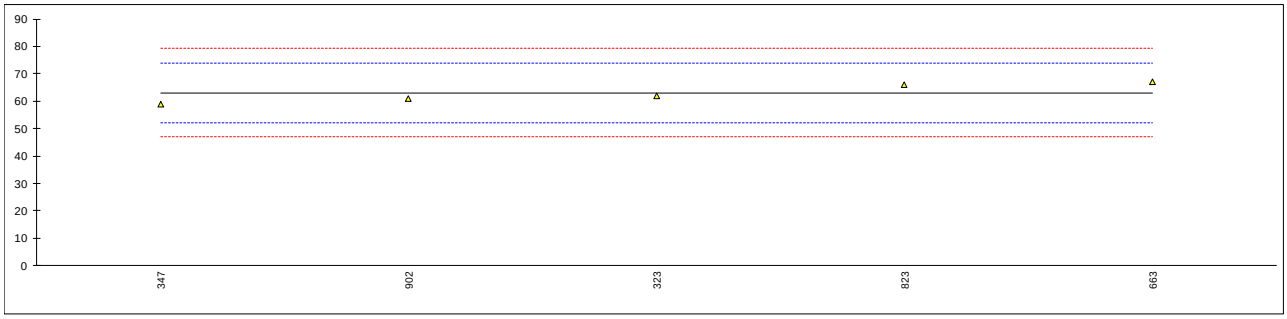
normality	unknown
n	7
outliers	0
mean (n)	13.79
st.dev. (n)	1.664
R(calc.)	4.66
st.dev.(Horwitz)	1.486
R(Horwitz)	4.16



Determination of Methyl alpha-Hydroxy Isobutyrate sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
311		-----		-----	
323	D3362Mod.	62		-0.19	
347	INH-2	59.0		-0.75	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663	INH-8001	67.2		0.77	
823	INH-2	66		0.55	
840		<5	C	<-10.74	first reported 18.9
902	INH-2	61		-0.38	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1067		-----		-----	

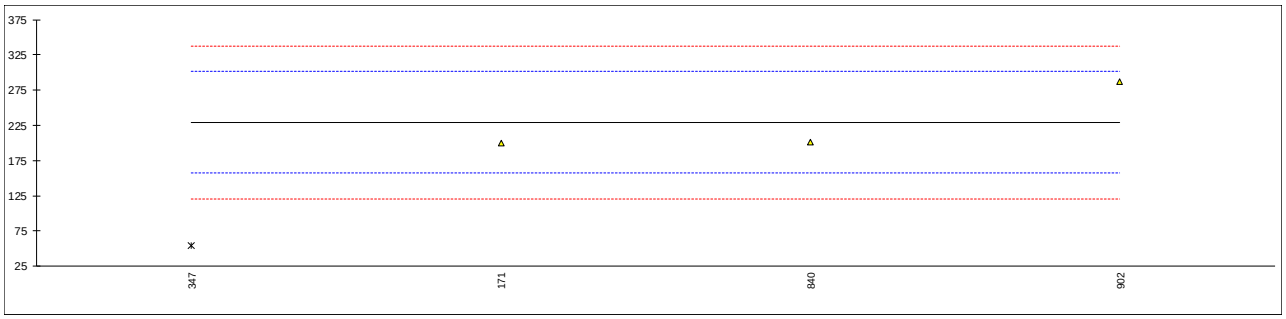
normality	unknown
n	5
outliers	0
mean (n)	63.04
st.dev. (n)	3.451
R(calc.)	9.66
st.dev.(Horwitz)	5.406
R(Horwitz)	15.14



Determination of Other Impurities on sample #18002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01	200		-0.81	
273		-----		-----	
311		-----		-----	
323		-----		-----	
347	INH-2	54.5	D(0.01)	-4.83	
522		-----		-----	
529		-----		-----	
557		-----		-----	
663		-----		-----	
823		-----		-----	
840		200.9		-0.78	
902	INH-2	287		1.59	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1067		-----		-----	

normality	unknown
n	3
outliers	1
mean (n)	229.30
st.dev. (n)	49.972
R(calc.)	139.92
st.dev.(Horwitz 5 comp)	36.202
R(Horwitz 5 comp)	101.37



APPENDIX 2

Number of participants per country

1 lab in BELGIUM
1 lab in BRAZIL
1 lab in INDIA
2 labs in MEXICO
2 labs in NETHERLANDS
2 labs in SAUDI ARABIA
1 lab in SOUTH AFRICA
1 lab in SOUTH KOREA
1 lab in SPAIN
1 lab in THAILAND
1 lab in TURKEY
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
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

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12 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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14 Analytical Methods Committee Technical brief, No 4 January 2001
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