Results of Proficiency Test
Methyl Methacrylate (MMA)
February 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: ing. A.S. Noordman-de Neef & ing. M. Meijer
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1 INTRODUCTION

Since 2009, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Methyl Methacrylate (MMA) once every two year. During the annual proficiency testing (PT) program 2019/2020 it was decided to continue the round robin for the analysis of Methyl Methacrylate.

In this interlaboratory study 13 laboratories from 11 different countries registered for participation for the PT on Methyl Methacrylate. See appendix 3 for the number of participants per country.
In this report the results of the 2020 proficiency test on Methyl Methacrylate are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample of 0.5 L Methyl Methacrylate labelled #20002.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of approximately 20 liters of Methyl Methacrylate obtained from a European supplier was spiked with Methanol and Acetone. After homogenization 38 amber 0.5 L glass bottles were filled and labelled #20002. The homogeneity of the subsamples #20002 was checked by determination of Density in accordance with ISO12185 and Methanol in accordance with an in-house test method on 4 stratified randomly selected samples.

	Density at 20°C in kg/L	Methanol in mg/kg
sample #20002-1	0.94333	40.0
sample #20002-2	0.94334	40.0
sample #20002-3	0.94335	40.0
sample #20002-4	0.94333	40.0

Table 1: homogeneity test results of subsamples #20002

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

	Density at 20°C in kg/L	Methanol in mg/kg
r (observed)	0.00003	0.0
reference method	ISO12185:96	Horwitz
0.3 * R (reference method)	0.00015	3.1

Table 2: evaluation of the repeatabilities of subsamples #20002

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

To each of the participating laboratories one bottle of 0.5L labelled #20002 was sent on January 15, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

The participants were asked to determine on sample #20002: Acidity as Acrylic Acid, Appearance, Color Pt/Co, Density at 20°C, Inhibitor as Topanol A, Water, Purity (two different definitions), Acetone, Ethyl Acrylate, Ethyl Methacrylate, Methanol, Methyl Acrylate, Methyl Isobutyrate, Methyl Propionate, Methyl alpha-hydroxyisobutyrate and Other Impurities.
It was also requested to report what type of column was used for the GC analysis.

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

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate 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 appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under ‘Remarks’ in the 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 June 2018 (iis-protocol, version 3.5).
For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

According to ISO5725 the original test results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis, the reported 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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

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

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

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Only one participant did not report any test results. All other participants reported before the deadline. Not all laboratories were able to perform all analyzes requested.
Twelve participants reported 99 numerical test results. Observed were 3 statistical outlying test results, which is 3.0%. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported test results. The abbreviations, used in these tables, are explained in appendix 4.

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

In the iis PT reports, ASTM test methods are referred to with a number (e.g. D1209) and an added designation for the year that the test method was adopted or revised (e.g. D1209:05). If applicable, a designation in parentheses is added to designate the year of re-approval (e.g. D1209:05(2011)). In the test result tables of appendix 1 only the test method number and year of adoption or revision (e.g. D1209:05) are used.

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

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

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

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

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

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

Acetone: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility calculated using the Horwitz equation.

Ethyl Acrylate: 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.

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.

Methyl Isobutyrate: 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.

Other Impurities: In total five test results were reported. The calculated reproducibility is large. Therefore, no z-scores were calculated.

The majority of the participants agreed on a content close to or below the quantification limits of Ethyl Methacrylate, Methyl Acrylate, Methyl Propionate and Methyl alpha-hydroxyisobutyrate. Therefore, no z-scores were calculated. The test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acrylic Acid	mg/kg	10	11.3	13.1	14
Appearance		12	pass	n.a.	n.a.
Color Pt/Co		10	2.8	3.8	7
Density at 20°C	kg/L	12	0.9434	0.0002	0.0005
Inhibitor as Topanol A	mg/kg	10	9.7	2.7	3.1
Water	mg/kg	10	98.0	36.7	16.8
Purity *)	%M/M	9	99.959	0.015	0.27
Purity on dry basis *)	%M/M	8	99.968	0.012	0.27
Acetone	mg/kg	5	31.9	8.8	8.5
Ethyl Acrylate	mg/kg	4	90.8	34.5	20.6
Methanol	mg/kg	7	36.9	21.5	9.6
Methyl Isobutyrate	mg/kg	6	39.9	16.4	10.3
Other impurities	mg/kg	5	191.6	281.9	(87.0)

Table 3: reproducibilities of test results on sample #20002

*) see definition in paragraph 4.1 and appendix 1

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2020 WITH PREVIOUS PTS

	February 2020	January 2018	June 2016	June 2014	May 2011
Number of reporting labs	12	15	12	11	11
Number of results reported	99	126	112	99	85
Number of statistical outliers	3	3	4	2	3
Percentage outliers	3.0%	2.4%	3.6%	2.0%	3.5%

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.

	February 2020	January 2018	June 2016	June 2014	May 2011
Acidity as Acrylic Acid	+/-	+	+	++	+/-
Color Pt/Co	++	++	++	++	++
Density at 20°C	++	++	++	++	++
Inhibitor as Topanol A	+	+	--	++	-
Water	--	-	-	--	++
Purity *)	++	++	++	n.e.	n.e.
Purity on dry basis 8)	++	++	++	n.e.	n.e.
Acetone	+/-	n.e.	(--)	n.e.	n.e.
Ethyl Acrylate	-	n.e.	n.e.	n.e.	n.e.
Ethyl Methacrylate	n.e.	n.e.	n.e.	n.e.	n.e.
Methanol	--	-	(--)	-	n.e.
Methyl Acrylate	n.e.	-	+/-	+	+/-
Methyl Isobutyrate	-	-	-	+	++
Methyl Propionate	n.e.	-	+/-	n.e.	n.e.
Methyl alpha-hydroisobutyrate	n.e.	+	++	n.e.	n.e.
Other impurities	(--)	-	n.e.	n.e.	n.e.

Table 5: comparison determinations against the reference test method
results between brackets are near or below the lower detection limit
*) see definition in paragraph 4.1 and appendix 1

The following performance categories were used:

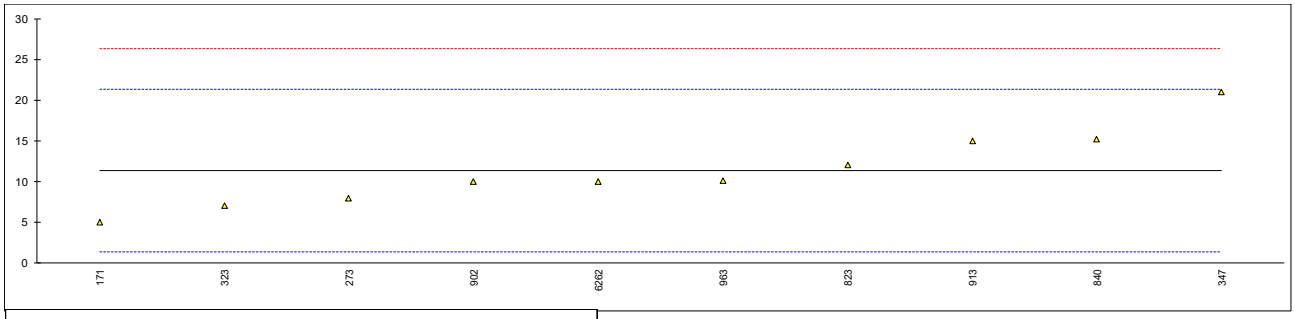
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.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

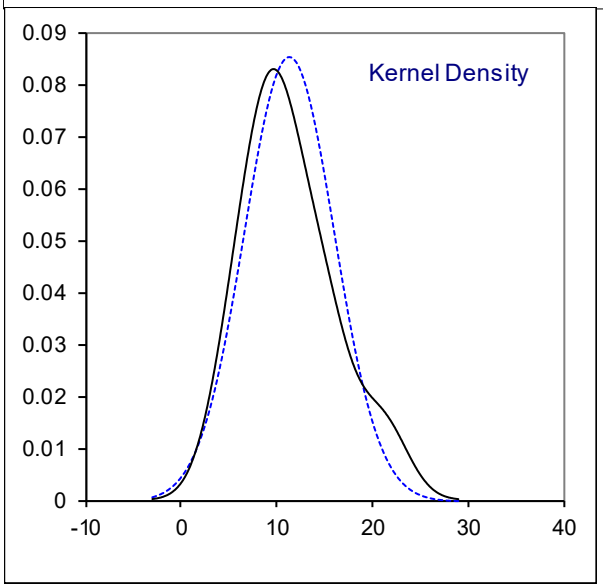
APPENDIX 1

Determination of Acidity as Acrylic Acid on sample #20002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1613	5	C	-1.27	First reported as 0.005 %M/M
273	D1613	8		-0.67	
311		-----		-----	
323	D1613	7		-0.87	
347	D1613	21		1.93	
557		-----		-----	
823	D1613	12		0.13	
840	D1613	15.2		0.77	
902	D1613	10		-0.27	
913	D1613	15		0.73	
962		-----		-----	
963	D1613	10.1		-0.25	
6262	D1613	10		-0.27	

normality	OK
n	10
outliers	0
mean (n)	11.33
st.dev. (n)	4.683
R(calc.)	13.11
st.dev.(D1613:17)	5
R(D1613:17)	14





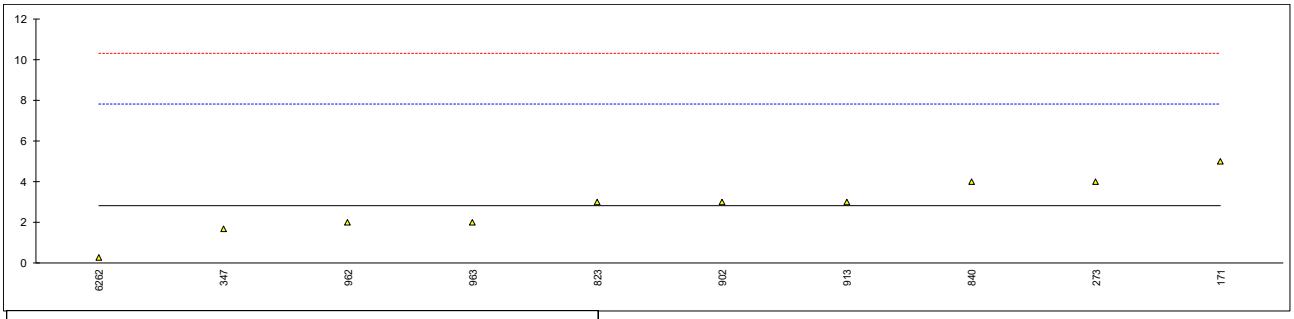
Determination of Appearance on sample #20002;

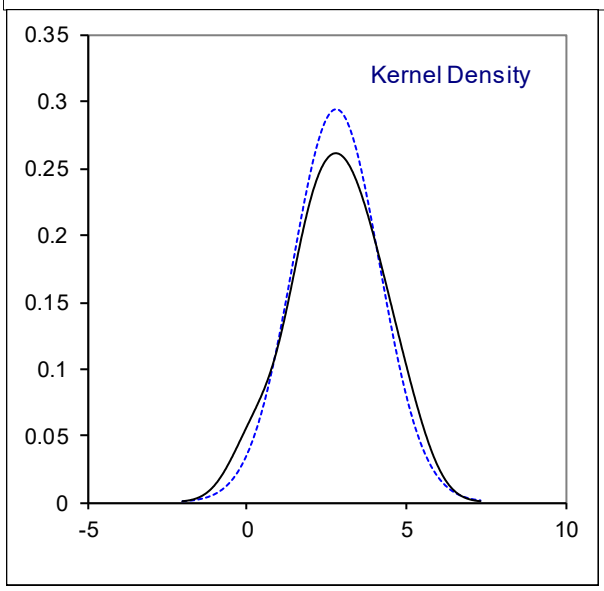
lab	method	value	mark	z(targ)	remarks
171	E2680	Pass		-----	
273	Visual	Bright & Clear		-----	
311	E2680	pass		-----	
323	D4176	clear & bright		-----	
347	E2680	Pass		-----	
557		-----		-----	
823	E2680	Pass		-----	
840	E2680	Pass		-----	
902	E2680	Pass		-----	
913	E2680	Pass		-----	
962	D4176	Clear and bright		-----	
963	Visual	Clear		-----	
6262	Visual	Clear & bright, free from sediment		-----	
	n	12			
	mean (n)	Pass			

Determination of Color Pt/Co on sample #20002;

lab	method	value	mark	z(targ)	remarks
171	D1209	5		0.88	
273	D1209	4		0.48	
311	D1209	<5		-----	
323	D1209	<5		-----	
347	D5386	1.7		-0.44	
557		-----		-----	
823	D5386	3		0.08	
840	D1209	4		0.48	
902	D5386	3		0.08	
913	D5386	3		0.08	
962	D1209	2		-0.32	
963	D1209	2		-0.32	
6262	D1209	0.3		-1.00	

normality	OK
n	10
outliers	0
mean (n)	2.80
st.dev. (n)	1.357
R(calc.)	3.80
st.dev.(D1209:05)	2.5
R(D1209:05)	7

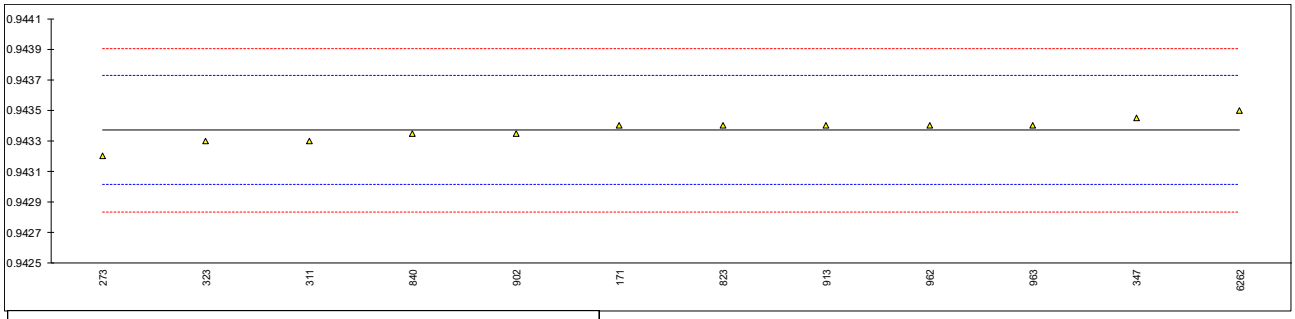


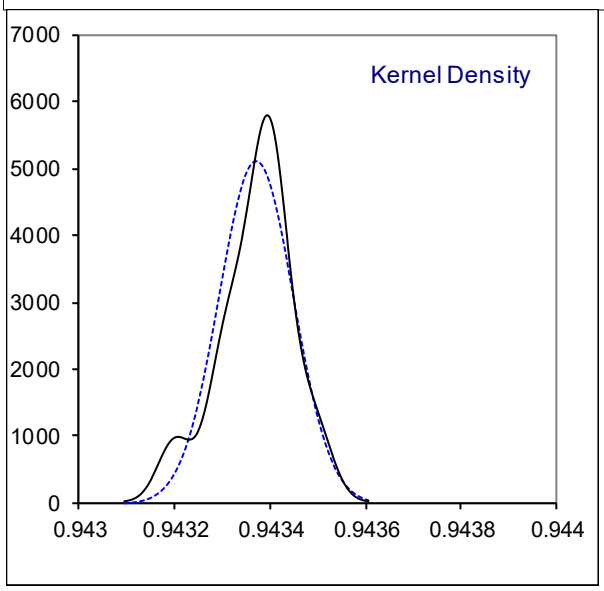


Determination of Density at 20°C on sample #20002; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9434		0.16	
273	D4052	0.9432		-0.96	
311	D4052	0.9433		-0.40	
323	D4052	0.9433		-0.40	
347	D4052	0.94345		0.44	
557		-----		-----	
823	D4052	0.94340		0.16	
840	D4052	0.94335		-0.12	
902	ISO12185	0.94335		-0.12	
913	D4052	0.9434		0.16	
962	D4052	0.9434		0.16	
963	D4052	0.9434		0.16	
6262	D4052	0.9435		0.72	

normality	suspect
n	12
outliers	0
mean (n)	0.94337
st.dev. (n)	0.000078
R(calc.)	0.00022
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

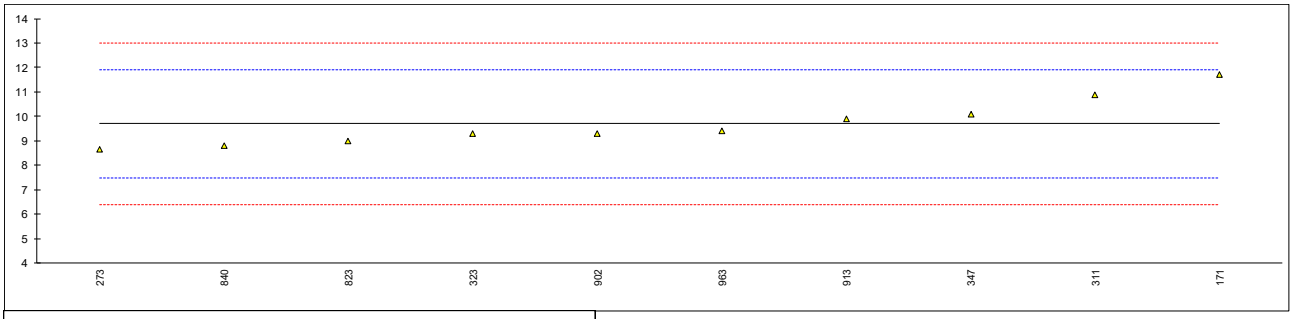


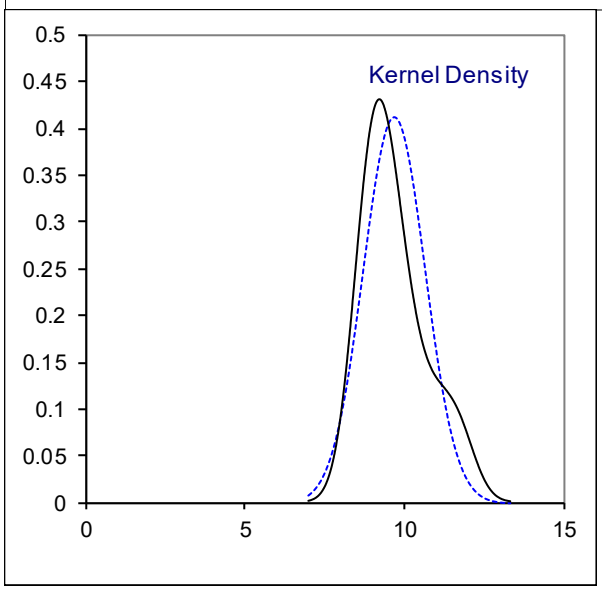


Determination of Inhibitor as Topanol A on sample #20002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01.1	11.7		1.81	
273	INH-2	8.66		-0.95	
311	INH-510	10.9		1.08	
323	INH-0002	9.3		-0.37	
347	INH-002	10.1		0.36	
557		-----		-----	
823	INH-2	9		-0.64	
840	INH-0002	8.8		-0.82	
902	INH-core 2	9.3		-0.37	
913	INH-core 2	9.9		0.18	
962		-----		-----	
963	INH-006C	9.4		-0.28	
6262		-----		-----	

normality	suspect
n	10
outliers	0
mean (n)	9.71
st.dev. (n)	0.968
R(calc.)	2.71
st.dev.(Horwitz)	1.103
R(Horwitz)	3.09

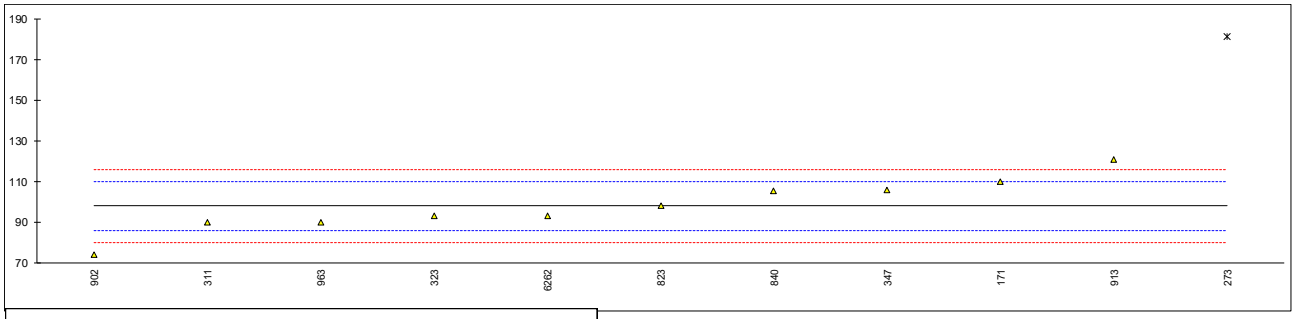


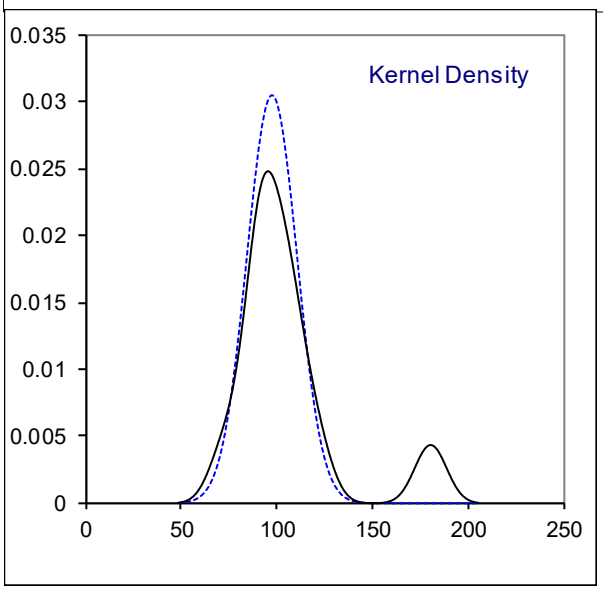


Determination of Water on sample #20002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	E1064	110		2.00	
273	E203	181	C,G(0.01)	13.86	First reported 225
311	E1064	90		-1.34	
323	E1064	93		-0.84	
347	E1064	106		1.33	
557		-----		-----	
823	E1064	98		-0.01	
840	E1064	105.3		1.21	
902	E1064	74		-4.01	
913	E1064	121		3.84	
962		-----		-----	
963	E1064	90		-1.34	
6262	E1064	93		-0.84	

normality	OK
n	10
outliers	1
mean (n)	98.03
st.dev. (n)	13.099
R(calc.)	36.68
st.dev.(E1064:16)	5.987
R(E1064:16)	16.76



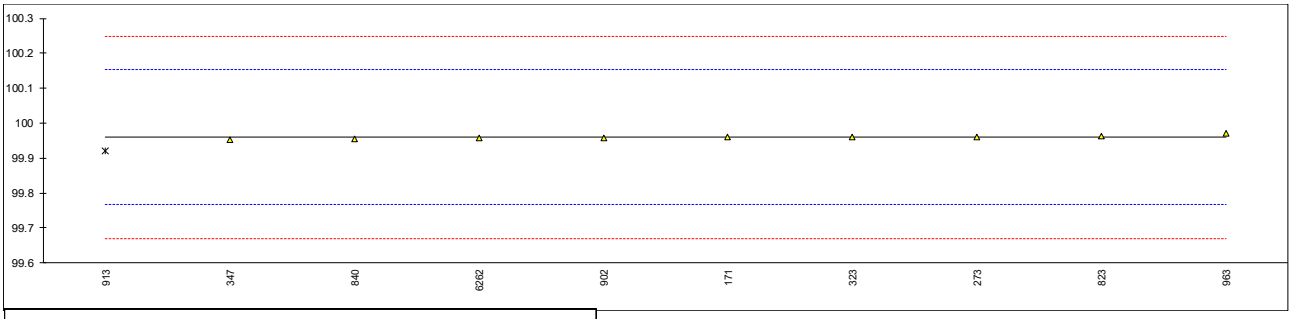


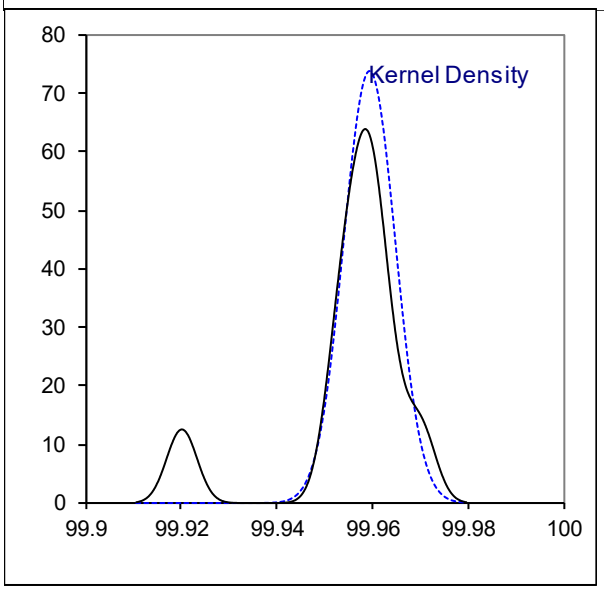
Determination of Purity*) on sample #20002; results in %M/M

lab	method	value	mark	z(targ)	Tyoe of column	remarks
171	INH-01.1	99.96		0.01	DB1701 30m x 0.32u x 1u	
273	INH-1A	99.96		0.01	DB-1 - 30m x 0.450mm x 2.55u	
311		-----		-----	CPWax 52 CB 60 m x 0,32 mm x 1,2 µm	
323	D3362Mod.	99.96		0.01		
347	INH-002	99.952		-0.08		
557		-----		-----		
823	INH-2	99.9640		0.05	CP Sil 5CB 25m * 0.32mm * 1,2 µm	
840	INH-0002	99.954		-0.05		
902	INH-core 2	99.957		-0.02		
913	INH-core 2	99.92	G(0.01)	-0.41	CP Sil 5 CB	
962		-----		-----		
963	INH-006C	99.97		0.11		
6262		99.9563		-0.03	CP-Wax 52CB 60m*320µm*0,5 µm	

normality	OK
n	9
outliers	1
mean (n)	99.9593
st.dev. (n)	0.00540
R(calc.)	0.0151
st.dev.(D3362:05)	0.09643
R(D3362:05)	0.27

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



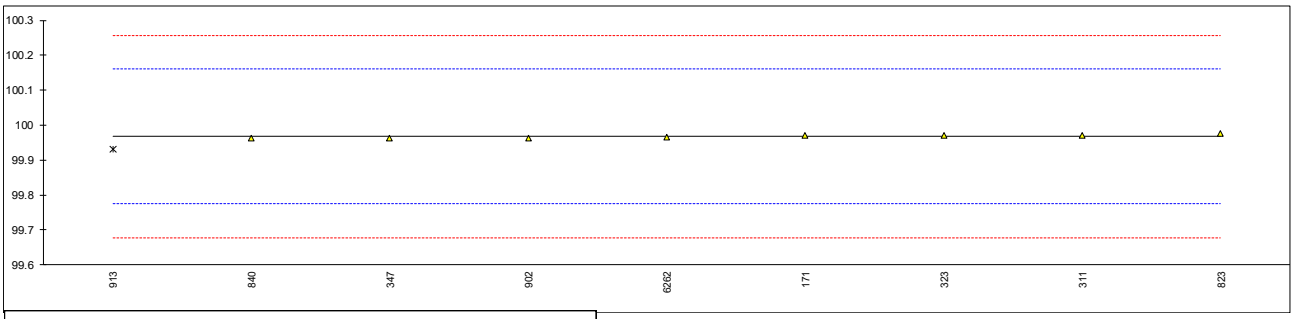


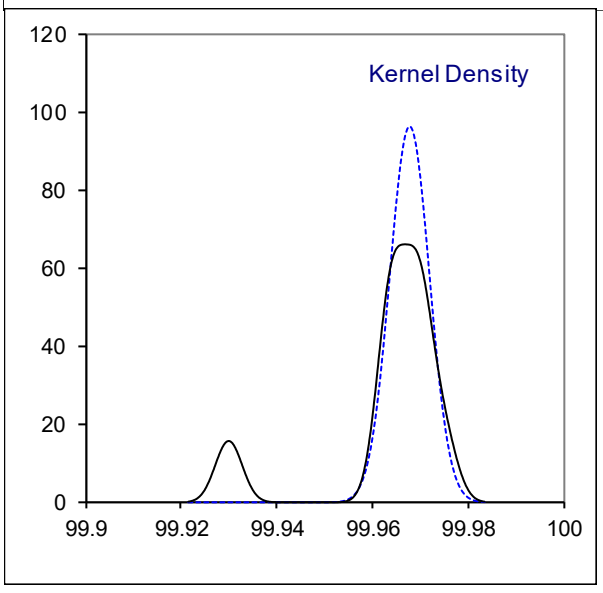
Determination of Purity on dry basis*) on sample #20002; results in %M/M

lab	method	value	mark	z(targ)	Type of column used	remarks
171	INH-01.1	99.97		0.02	DB1701 30m x 0.32u x 1u	
273		-----		-----	DB-1 - 30m x 0.450mm x 2.55u	
311	INH-114	99.97		0.02	CPWax 52 CB 60 m x 0,32 mm x 1,2 µm	
323	D3362Mod.	99.97		0.02		
347	INH-002	99.964		-0.04		
557		-----		-----		
823	INH-2	99.9750		0.07	CP Sil 5CB 25m * 0.32mm * 1,2 µm	
840	INH-0002	99.963		-0.05		
902	INH-core 2	99.964		-0.04		
913	INH-core 2	99.93	G(0.01)	-0.39	CP Sil 5 CB	
962		-----		-----		
963		-----		-----		
6262		99.9666		-0.01	CP-Wax 52CB 60m*320µm*0,5 µm	

normality	OK
n	8
outliers	1
mean (n)	99.9678
st.dev. (n)	0.00414
R(calc.)	0.0116
st.dev.(D3362:05)	0.09643
R(D3362:05)	0.27

*) Purity on dry basis = 100% - impurities

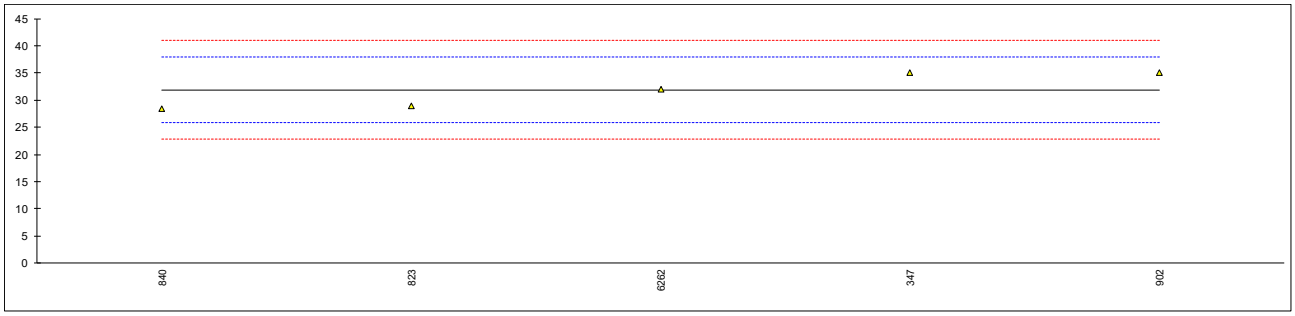




Determination of Acetone on sample #20002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
311	INH-114	<10		<-7.23	False negative test result?
323		-----		-----	
347	INH-002	35		1.02	
557		-----		-----	
823	INH-2	29		-0.96	
840	INH-0002	28.5		-1.12	
902	INH-core 2	35		1.02	
913	INH-core 2	<10		<-7.23	False negative test result?
962		-----		-----	
963		-----		-----	
6262		32		0.03	

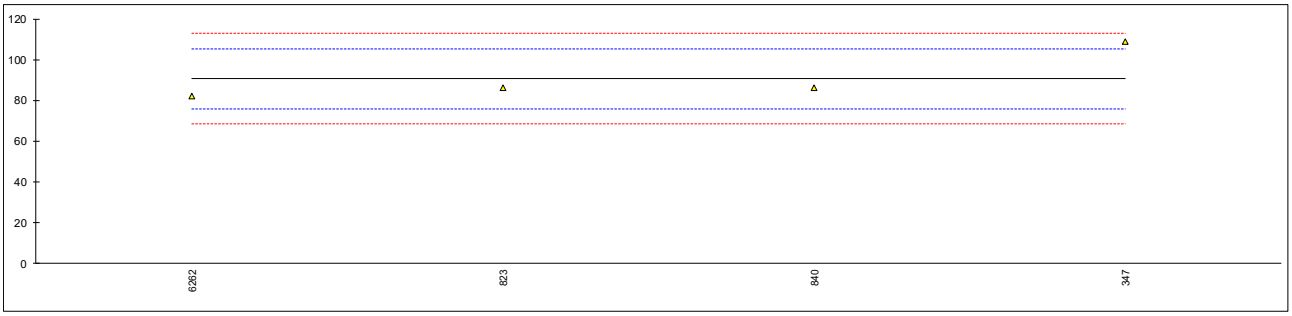
normality	unknown
n	5
outliers	0
mean (n)	31.90
st.dev. (n)	3.130
R(calc.)	8.77
st.dev.(Horwitz)	3.031
R(Horwitz)	8.49



Determination of Ethyl Acrylate on sample #20002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
347	INH-002	109		2.48	
557		-----		-----	
823	INH-2	86		-0.64	
840	INH-0002	86.0		-0.64	
902	INH-core 2	<5		<-11.64	False negative test result?
913		-----		-----	
962		-----		-----	
963		-----		-----	
6262		82		-1.19	

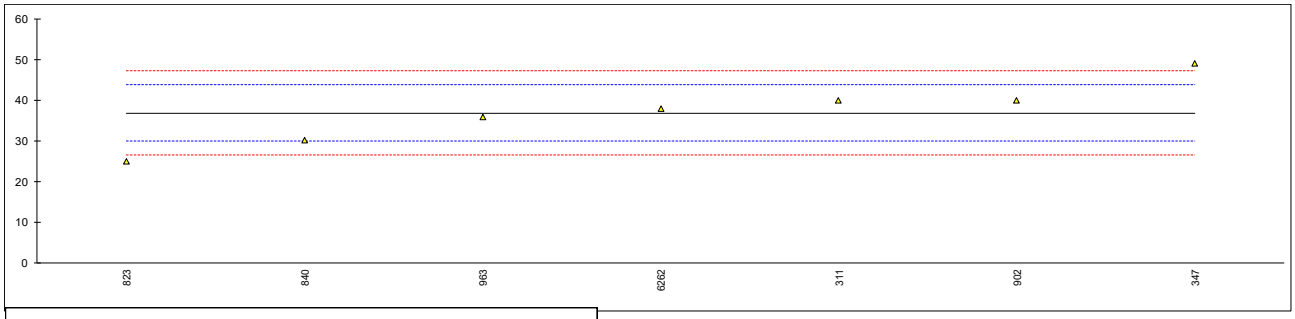
normality	unknown
n	4
outliers	0
mean (n)	90.75
st.dev. (n)	12.312
R(calc.)	34.47
st.dev.(Horwitz)	7.367
R(Horwitz)	20.63

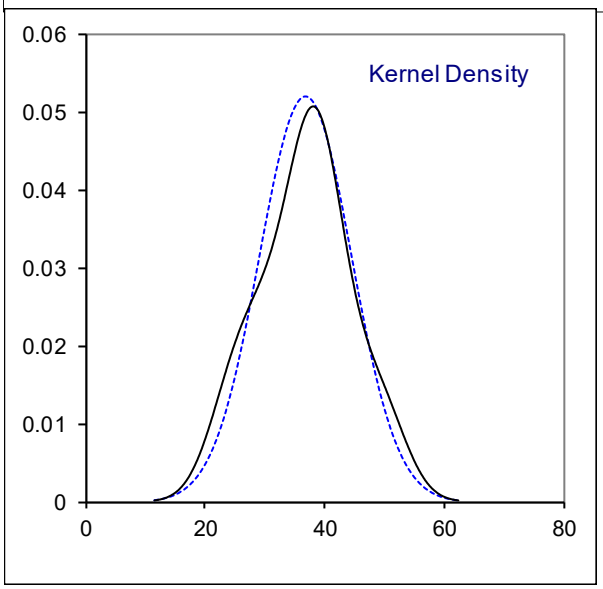


Determination of Methanol on sample #20002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
311	INH-114	40		0.92	
323		-----		-----	
347	INH-002	49		3.54	
557		-----		-----	
823	INH-2	25		-3.46	
840	INH-0002	30.2		-1.94	
902	INH-core 2	40		0.92	
913	INH-core 2	<10		<-7.84	False negative test result?
962		-----		-----	
963	INH-006C	35.8		-0.31	
6262		38		0.33	

normality	unknown
n	7
outliers	0
mean (n)	36.86
st.dev. (n)	7.689
R(calc.)	21.52
st.dev.(Horwitz)	3.427
R(Horwitz)	9.59

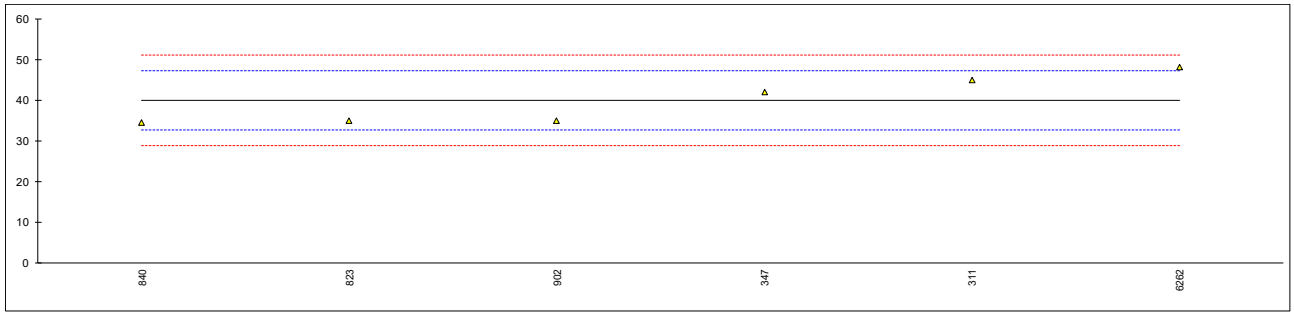




Determination of Methyl Isobutyrate on sample #20002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
273		-----		-----	
311	INH-114	45		1.38	
323		-----		-----	
347	INH-002	42		0.56	
557		-----		-----	
823	INH-2	35		-1.34	
840	INH-0002	34.6		-1.45	
902	INH-core 2	35		-1.34	
913		-----		-----	
962		-----		-----	
963		-----		-----	
6262		48		2.20	

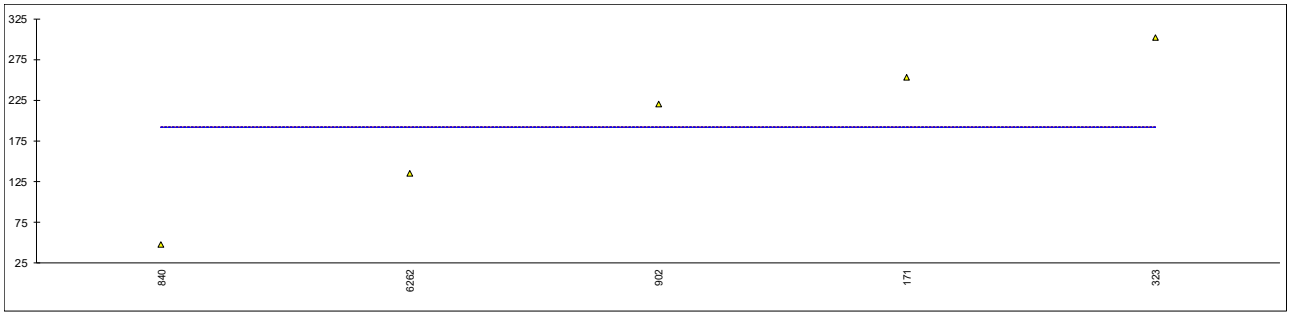
normality	unknown
n	6
outliers	0
mean (n)	39.93
st.dev. (n)	5.867
R(calc.)	16.43
st.dev.(Horwitz)	3.668
R(Horwitz)	10.27



Determination of Other Impurities on sample #20002; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01.1	253		-----	
273		-----		-----	
311		-----		-----	
323	INH-0002	302		-----	
347		-----		-----	
557		-----		-----	
823		-----		-----	
840	INH-0002	48.2		-----	
902	INH-core 2	220		-----	
913		-----		-----	
962		-----		-----	
963		-----		-----	
6262		135		-----	

normality	unknown
n	5
outliers	0
mean (n)	191.64
st.dev. (n)	100.663
R(calc.)	281.86
st.dev.(Horwitz 5 comp)	(31.084)
R(Horwitz 5 comp)	(87.04)



APPENDIX 2

Other reported test results

Determination of individual Components on sample #20002; in mg/kg

lab	Ethyl Methacrylate	Methyl Acrylate	Methyl Propionate	Methyl alpha-hydroxy isobutyrate
171	-----	-----	<100	-----
273	-----	-----	-----	-----
311	35	<10	-----	-----
323	<100	-----	<100	<100
347	-----	-----	14	<5
557	-----	-----	-----	-----
823	<5	<5	<5	<5
840	48.4	<5	11.8	50.5
902	<5	<5	13	<5
913	-----	-----	-----	-----
962	-----	-----	-----	-----
963	-----	-----	-----	-----
6262	0	9	0	0

APPENDIX 3

Number of participants per country

2 labs in	BELGIUM
1 lab in	BRAZIL
1 lab in	INDIA
1 lab in	NETHERLANDS
2 labs in	SAUDI ARABIA
1 lab in	SOUTH AFRICA
1 lab in	SOUTH KOREA
1 lab in	SPAIN
1 lab in	TURKEY
1 lab in	UNITED STATES OF AMERICA
1 lab in	VIETNAM

APPENDIX 4

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
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
SDS	= Safety Data Sheet

Literature

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3 ASTM E1301:03
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12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee, Technical brief, No 4, January 2001.
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