Results of Proficiency Test Methylmethacrylate (MMA) May 2011

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 2010/2011. In this interlaboratory study 12 laboratories from 12 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the Methylmethacrylate (MMA) proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organizer of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to send 1* 0.5L bottle of MMA, labelled #11047. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO 17043:2010. This ensures 100% confidentiality of participant's data. Also, 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 Organization, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2), which can be downloaded from www.iisnl.com.

2.3 CONFIDENTIALITY STATEMENT

All data present 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 25 litre bulk material was retained from the previous proficiency test in 2009. After homogenisation in a precleaned can, 35 subsamples were transferred to brown glass bottles of 0.5L and labelled #11047. The homogeneity of the subsamples was checked by determination of Density @ 20°C acc. ASTM D4052:02e1 and Water acc. ASTM E1064:08 on 4 stratified randomly selected samples.

	Density in kg/L	Water in %M/M		
sample #11047-1	0.94343	0.0291		
sample #11047-2	0.94345	0.0294		
sample #11047-3	0.94346	0.0292		
sample #11047-4	0.94352	0.0293		

table 1: homogeneity test results of subsamples #11047

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 in kg/L	Water in %M/M		
r (sample #11047)	0.00014	0.0004		
reference test method	D4052:02e1	E1064:08		
0.3 * R (reference test)	0.00015	0.0015		

table 2: repeatability of subsample #11047

The repeatability of the results for Density and for Water on sample #11047 were in full agreement with the repeatability as required by the respective standards. Therefore, homogeneity of subsample #11047 was assumed.

One bottle 0.5 L, labelled #11047 was dispatched to each of the participating laboratories on April 5, 2011.

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 #11047: Acidity (as Acrylic Acid), Appearance, Colour Pt/Co, Density @ 20°C, Inhibitor as Topanol A, Water, Purity (both on "as-is" and on dry basis), Acetone, Methanol, Methylacrylate, Methylisobutyrate, Ethylmethacrylate and other Impurities.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards, was sent together with each set of samples. Also, a letter of instructions and a SDS were added to the package.

3 RESULTS

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

Directly after deadline, a reminder fax was sent to those laboratories that had not yet reported. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the (raw data of the) reported results. Additional or corrected results have been used for data analysis and the original results are placed under 'Remarks' in the result tables in Appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the conclusions of statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests.

Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility

limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a 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 (see appendix 3, nr.12-13).

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the result tables in appendix 1. Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore the usual interpretation of z-scores is as follows:

 $\begin{aligned} |z| < 1 & \text{good} \\ 1 < |z| < 2 & \text{satisfactory} \\ 2 < |z| < 3 & \text{questionable} \\ |z| > 3 & \text{unsatisfactory} \end{aligned}$

4 EVALUATION

In this proficiency test, only one laboratory did receive the samples late due to problems with custom clearance. One laboratory decided not report any results. In total 11 participants reported 85 numerical results. Observed were 3 outlying results, which is 3.5% of the numerical results.

Not all original data sets proved to have a normal distribution. Not normal Gaussian distributions were found with the following determinations: Colour, Density and Purity "as is". In these cases the statistical evaluation should be used with due care.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data (see appendix 1). The abbreviations, used in these tables, are listed in appendix 3.

- Acidity asThis determination was not problematic. The reported results varyAcrylic Acid:from 8 25 mg/kg. No statistical outliers were observed and the
calculated reproducibility is in agreement with the requirements of ASTM
D1613:06.
- <u>Appearance:</u> This determination was not problematic. All laboratories agree that the sample is clear ("Pass").
- <u>Colour Pt/Co</u>: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1209:11.
- <u>Density @ 20°C</u>: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D4052:02e1.
- Inhibitor asNo analytical problems have been observed. No statistical outliers wereTopanol A:observed and the calculated reproducibility is (almost) in agreement with
the estimated reproducibility, calculated using the Horwitz equation.
- <u>Water:</u> No analytical problems have been observed. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM E1064:08.
- <u>Purity "as is"</u>: Regretfully, no suitable standardized method exists with precision data. Therefore no significant conclusions were drawn. However one statistical outlier was observed.
- <u>Purity on dry basis</u>: Regretfully, no suitable standardized method exists with precision data. Therefore no significant conclusions were drawn. However, one statistical outlier was observed
- <u>Acetone</u>: In this determination, all reporting participants agreed on a result below 10 mg/kg. Therefore, no significant conclusions were drawn.

Methanol:In this determination, all reporting participants, except one, agreed on a
result below 10 mg/kg. Therefore no significant conclusions were drawn.
Only one laboratory reported a numerical result.Methylacrylate:No analytical problems have been observed. No statistical outliers were
observed and the calculated reproducibility is in agreement with the
estimated reproducibility, calculated using the Horwitz equation.Methylisobutyrate:No analytical problems have been observed. No statistical outliers were
observed and the calculated reproducibility is in agreement with the
estimated reproducibility, calculated using the Horwitz equation.

- the estimated reproducibility, calculated using the Horwitz equation.Ethylmethacrylate:No analytical problems have been observed. No statistical outliers were
- observed and the calculated reproducibility is in agreement with the estimated reproducibility, calculated using the Horwitz equation.
- <u>Other Impurities:</u> Regretfully, no suitable standardized method exists with precision data. The results vary from 209 – 252 mg/kg. Therefore, no significant conclusions were drawn.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	R (Calc.)	R (lit)
Acidity as Acrylic Acid	mg/kg	11	15.5	14.3	14.0
Colour Pt/Co		8	3.1	1.0	7.0
Density @ 20°C	kg/L	11	0.9434	0.0001	0.0005
Inhibitor as Topanol A	mg/kg	10	13.6	4.4	4.1
Water	mg/kg	10	303.2	49.7	71.0
Purity "as is" / as received	%M/M	8	99.937	0.012	unknown
Purity (on dry basis)	%M/M	7	99.968	0.005	unknown
Acetone	mg/kg	0	<10	n.a.	n.a.
Methanol	mg/kg	0	<10	n.a.	n.a.
Methylacrylate	mg/kg	3	11.2	3.7	3.5
Methylisobutylyrate	mg/kg	5	60.9	7.2	14.7
Ethylmethacrylate	mg/kg	3	10.0	2.7	3.2
Other impurities	mg/kg	5	224.8	50.5	unknown

table 3: reproducibilities of results of sample #11047

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2011 WITH PREVIOUS PTS

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

table 4: comparison with previous proficiency tests

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

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

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

table 5: comparison determinations against the standard

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

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

Sample #11047 was already used before, in PT iis09C06. When the data of both PTs are compared it is remarkable to notice that the consensus values for both rounds differ not significantly (except for water), while the spread has improved for many tests, see table 6.

		#0939 (in	iis09C06)	#11047 (in iis11C05)		
Parameter	unit	average	R (Calc.)	average	R (Calc.)	
Acidity as Acrylic Acid	mg/kg	15.0	16.4	15.5	14.3	
Colour Pt/Co		3.8	2.1	3.1	1.0	
Density @ 20°C	kg/L	0.9434	0.0002	0.9434	0.0001	
Inhibitor as Topanol A	mg/kg	15.0	1.7	13.6	4.4	
Water	mg/kg	199.9	62.1	303.2	49.7	
Purity "as is" / as received	%M/M	99.96	0.03	99.94	0.01	
Purity (on dry basis)	%M/M	99.97	0.03	99.97	0.01	
Methylacrylate	mg/kg	9.9	4.4	11.2	3.7	
Methylisobutylyrate	mg/kg	59.8	6.2	60.9	7.2	
Ethylmethacrylate mg/kg		8.0	3.3	10.0	2.7	
Other impurities	mg/kg	218.3	103.3	224.8	50.5	

table 6: Comparison of samples #0939 (in iis09C06) and #11047 (in iis11C05)

APPENDIX 1

Determination of Acidity as Acrylic Acid on sample #11047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273	D1613	16		0.10	
311	D1613	16		0.10	
323	D1613	20		0.90	
338					
347	D1613	19.7		0.84	
357	D1613	25		1.90	
823	D1613	8		-1.50	
840	D1613	19.0		0.70	
868	D1613	12.8		-0.54	
886	D1613	11		-0.90	
902	D1613	13		-0.50	
913	D1613	10.2		-1.06	
		<u></u>			
	normality	OK			
	n	11			
	outliers	0			
	mean (h)	15.5			
	st.aev. (h)	5.09			
	R(Calc.) P(D1613:06)	14.3			





Determination of Appearance on sample #11047;

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lab	method	value	mark	z(targ)	remarks
273	E2680	PASS			
311	E2680	PASS			
323	E2680	CFFSM			
338					
347	E2680	PASS			
357	E2680	PASS			
823	E2680	PASS			
840	E2680	PASS			
868	E2680	PASS			
886	VISUAL	CFSM			
902	VISUAL	CFFSM			
913	E2680	CFFSM			
	normality	n.a.			
	n	11			
	outliers	n.a.			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(E2680)	n.a.			

Abbreviations:

CFFSM

= clear free from suspended matter

Determination of Colour Pt/Co on sample #11047;

lab	method	value	mark	z(targ)	remarks				
273	D1209	4		0.35					
311 323	D1209 D1209	<5 <5							
338	2.200								
347 257	D1209	3		-0.05					
823	D1209	3		-0.05					
840	D1209	3		-0.05					
868 886	D1209 D1209	3		-0.05 -0.05					
902	D5386	3		-0.05					
913	D5386	3.0		-0.05					
	normality	not OK							
	n autliara	8							
	mean (n)	0 3.1							
	st.dev. (n)	0.35							
	R(calc.) R(D1209·11)	1.0 7.0							
	R(D1203.11)	7.0							
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10 -									
8 -									
6 -									
4 -									Δ
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1.6	-			ensity					
1.4	-		Λ						
1.2									
1	-								
0.8	-								
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0.2	-			\mathbf{i}					
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	0 1	2	3 4	1	5				

Determination of Density @ 20°C on sample #11047; results in g/L

lab	method	value	mark	z(targ)	remarks						
273	D4052	0.9434		0.08							
311	D4052	0.94342		0.19							
323	D4052	0.9434		0.08							
338											
347	D4052	0.94332		-0.37							
357	D4052	0.9434		0.08							
823	D4052	0.9434		0.08							
840	D4052	0.94336		-0.14							
868	D4052	0.94343		0.25							
886	D4052	0.9433		-0.48							
902	D4052	0.94345		0.36							
913	D4052	0.94336		-0.14							
	normality	not OK									
	n	11									
	outliers	0									
	mean (n)	0 04330									
	st dev (n)	0.04000									
	R(calc.)	0.000040									
	R(D4052:02e1)	0.00050									
	H(B 1002.0201)	0.00000									
0.0445											
0.9445 T											
0.944 -											
0.9435 -											۵
	Δ Δ	۵	Δ		<u>م</u>		Δ		4		
0943											
0.010											
0.9425 -											
0.942 ⊥	98 2	9	<i>с</i>		3 E	1	<u>ព</u>	6	~		Ŋ
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100	<u>ກ</u>										
900	- 00		Kernel De	nsity							
800	- 00	\wedge									
700	- 00										
600	- 00										
500	- 00										
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300	00 -										
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			\sim								
	0 +										
	0.9432 0.9433	3 0.9434	0.9435	0.943	36						

Determination of Inhibitor as Topanol A on sample #11047; results in mg/kg

lab	method	value	mark	z(targ)	remarks	_
273 311 323 338 347 357 823 840 868 886 902 913	INH-510 INH-0002 INH-136 INH-0002 INH-0002 INH-044 INH-070 INH-94 INH-0002 normality n outliers mean (n) st.dev. (n) R(calc.) R(Horwitz)	12 11 13.2 13.1 14 15.9 13.7 12.7 15.8 14.8 OK 10 0 13.62 1.573 4.41 4.12	С	 -1.10 -1.78 -0.29 -0.35 0.26 1.55 0.05 -0.63 1.48 0.80	First reported 26.0	
19 - 17 - 15 - 13 - 11 - 9 - 7 -		Δ	Δ	Δ	Δ Δ Δ Δ Δ	
5		~	ω	~		_
	Ŕ	ર્સ	8	8	<u> </u>	
0.25 0.2 0.15 0.1 0.05		10	Kernel	Density	20	

Determination of Water on sample #11047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273 311 323 338	E1064 E1064 E1064	303 310 290		-0.01 0.27 -0.52	
347 357 823 840 868 886 902 913	E1064 E1064 E1064 E1064 E1064 E1064 E1064 E203	297 330 290 335 363 285 306.7 285	C G(0.05)	-0.24 1.06 -0.52 1.26 2.36 -0.72 0.14 -0.72	First reported 530
	normality n outliers mean (n) st.dev. (n) R(calc.) R(E1064:08)	OK 10 1 303.17 17.756 49.72 71.00			
⁴⁰⁰ T					
350 -					×
300 -		۵	Δ	Δ	ΔΔ
250 -					
200 -					
150				~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	N M N 7 N 0 0
	8 5	8	8	8	± 10 − 10 − 10 − 10 − 10 − 10 − 10 − 10
0.0	2		Korpol D	oncity	
0.01	8 -		Λ Λ	Chisity	
0.01	6 -		1		
0.01	4 -		11		
0.01	2-				
0.0			[]		
0.00					
0.00					
0.00	0				
0.00	0			<u>_</u>	
	0 100	200	300 4	00 50	
]

Determination of Purity ("as is" / as received) on sample #11047; results in %M/M

lab	method	value	mark	z(targ)	remarks				
273 311 323 338 347 357 823 840 868 886	INH114 D3362mod INH-31 INH-031 INH-0002 INH-0002 INH-042	99.94 99.94 99.94 99.94 99.94 99.94 99.94 99.931 99.929	С	 	First reported 99.	92			
902 913	INH-80 INH-0002	99.938 99.97	C G(0.01)		First reported 99.8	388			
	normality n outliers mean (n) st.dev. (n) R(calc.) R(lit)	not OK 8 1 99.937 0.0044 0.012 unknown			Compare R(iis090	C06) = 0.03			
99.98 - 99.97 -									*
99.96 - 99.95 -									
99.94 -		<u> </u>	A	A	Δ	۵	۵	۵	
99.93 - 99.92 - 99.91 -	۵	•							
99.9	8	64	47	8	ñ		ŝ	22	
	α	ø	ň	o		<u>м</u>	M		5
30	-	\wedge	Kernel D	ensity					
25	_								
15									
10	-								
0 · 0	0.85 99.9	99.95	100	100.0	75				

Determination of Purity (on dry basis) on sample #11047; results in %M/M

lab	method	value	mark	z(targ)	remarks
273					
311					
323	D3362mod	99.97			
338					
347	INH-31	99.900			
823	INH-0002	99.97			
840	INH-0002	99.966			
868	INH-042	99.967			
886	INH-084	99.968			
902	INH-80	99.932	C,G(0.01)		First reported 99.884
913					
	normality	ок			
	n	7			
	outliers	1			
	mean (n)	99.968			
	St.dev. (n)	0.0016			
	R(lit)	Unknown			Compare R(iis09C06) = 0.03
	()	•			
99.98 _T					
99.97					
00.07		Δ	Δ		<u> </u>
99.96 -					
99.95 -					
99.94 -					
99.93 -	×				
99.92 -					
00.04					
99.91 +					
99.9 ⊥	80	840	ŝ		8 8 8
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			(\		
120	1				
100	-				
			/ /		
80	-				
60	1				
40					
40	1				
20	$ \land $				
20] /\				
0	\downarrow			\mathbf{X}	
9	9.92 99	94	99.96	99,98	8
				,	

Determination of Acetone on sample #11047; results in mg/kg

lab	mothod	value	mark	z(torc)	romarka
	method	value	IIIdi K	z(tary)	Terridiks
273					
311	INH114	<10			
323					
338					
347					
357	INH-031	<10			
823	INH-0002	<5			
840	INH-0002	<2	n.d.		
868	INH-042	<10			
886					
902					
013					
515					
	normality	n 0			
	normality	n.a.			
	n 	0			
	outliers	0			
	mean (n)	<10			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of Methanol on sample #11047; results in mg/kg

lab	method	value	mark	z(targ)	remarks	
273						
311	INH114	<10				
323	D3362mod	<10				
338						
347						
357	INH-031	<10				
823	INH-0002	<5				
840	INH-0002	21.0			False positive?	
868	INH-042	<10				
886						
902						
913						
	normality	na				
	normality	11.a.				
	outliere	0				
	mean (n)	-10				
	st dev (n)	<u></u>				
	P(calc)	n.a.				
	R(lit)	n.a				
	ix(iii)	n.a.				

Determination of Methylacrylate on sample #11047; results in mg/kg

lab	method	value	mark	z(targ)	remarks	
lab 273 311 323 338 347 357 823 840 868 886 902	method INH114 INH-031 INH-002 INH-002 INH-042	value <10 <10 11 12.6 10 	mark	z(targ) -0.16 1.12 -0.96 	remarks	
913	normality n outliers mean (n) st.dev. (n) R(calc.) R(Horwitz)	n.a. 3 0 11.20 1.311 3.67 3.49				
16 - 14 - 12 - 10 - 8 - 6 -		<u> </u>			Δ	
4 - 2 - 0		88			<u>ଥ</u> ି ଛି	

Determination of Methylisobutyrate on sample #11047; results in mg/kg

lab	method	value	mark	z(targ)	remarks	
273 311 323 338 347 357 823 840 868 886 902 913	INH114 INH-031 INH-0002 INH-0002 INH-042	60 57 61 63.3 63 		-0.16 -0.74 0.03 0.47 0.41 		
	normality n outliers mean (n) st.dev. (n) R(calc.) R(Horwitz)	OK 5 0 60.86 2.559 7.16 14.69				
80 - 75 - 70 - 65 - 60 -					Δ Δ Δ	
55 - 50 - 45 - 40 - 35 -						
30	367		311		8 8 8	

Determination of Ethylmethacrylate on sample #11047; results in mg/kg

lab	method	value	mark	z(targ)	remarks	
273 311 323 338 347 357 823 840 868 886 902 913	INH114 INH-031 INH-0002 INH-0002 INH-042 INH-80	<10 <10 <10 11 9.1 10 <10 <10 	С	2(targ) 0.85 -0.82 -0.03 		
	normality n outliers mean (n) st.dev. (n) R(calc.) R(Horwitz)	n.a. 3 0 10.03 0.950 2.66 3.18				
16 - 14 -						-
12 -					· · · · · · · · · · · · · · · · · · ·	-
8 -		۵ 			-	-
6 - 4 -						
2 -						
0		840			8	8

Determination of Other Impurities on sample #11047; results in mg/kg

lab	method	value	mark	z(targ)	remarks		
273							
311	INH114	210					
323							
338							
347		220					
823	INH-031	220					
840	INH-0002	233.2					
868	INH-042	252					
886							
902							
913							
	normality	OK 5					
	n	5					
	mean (n)	224 84					
	st.dev. (n)	18.042					
	R(calc.)	50.52					
	R(lit)	unknown			Compare R(iis09C06) = 103.3		
270 _T							
250 -							۵
230 -						Δ	
					۵		
210 -	۵		Δ				
100							
190 -							
170 -							
150	~				~		~
	ğ		3		Ŕ	840	ä

APPENDIX 2

Number of participants per country

1 laboratory in BELGIUM

- 1 laboratory in FINLAND
- 1 laboratory in FRANCE
- 1 laboratory in INDIA
- 1 laboratory in KOREA
- 1 laboratory in P.R. of CHINA
- 1 laboratory in SOUTH AFRICA
- 1 laboratory in SPAIN
- 1 laboratory in TAIWAN
- 1 laboratory in THE NETHERLANDS
- 1 laboratory in TURKEY
- 1 laboratory in VIETNAM

APPENDIX 3

Abbreviations:

С	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
ex	= excluded from calculations
S	= scope of the reported method is not applicable
n.a.	= not applicable
U	= reported in different unit
W	= result withdrawn on request of the participant
SDS	= Safety Data Sheet

Literature:

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