

Results of Proficiency Test
n-Butylacrylate
May 2012

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies organizes on regular basis a proficiency test for the analysis of n-Butylacrylate. As part of the annual proficiency test program of 2011/2012, the Institute decided to continue this proficiency test on n-Butylacrylate. In this interlaboratory study, 16 laboratories from 15 different countries have participated. See appendix 2 for the number of participants per country.

In this report, the results of the n-Butylacrylate 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. It was decided to send one 0.5 litre bottle. The analyses for fit-for-use and homogeneity testing were subcontracted.

Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010 and ILAC-G13:2007. 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

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 25 litre bulk material for sample #12073 was obtained from a local supplier. After homogenisation in a precleaned can, 48 subsamples were transferred to brown glass bottles of 0.5L and labelled #12073. The homogeneity of the subsamples was checked by

determination of Density in accordance with ASTM D4052 and Water in accordance with ASTM D1364 on 8 stratified randomly selected samples.

	Density @20°C in kg/L	Water in mg/kg
sample #12073-1	0.89901	78
sample #12073-2	0.89900	76
sample #12073-3	0.89901	78
sample #12073-4	0.89897	84
sample #12073-5	0.89899	78
sample #12073-6	0.89898	80
sample #12073-7	0.89900	81
sample #12073-8	0.89901	82

table 1: homogeneity test results of subsamples #12073

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 @20°C in kg/L	Water in mg/kg
r (sample #12073)	0.00004	7.3
reference test method	ASTM D4052:11	ASTM D1364:07
0.3 x R(reference test)	0.00015	16.1

table 2: evaluation of homogeneity of subsamples #12073

The calculated repeatabilities are each less than 0.3 times the reproducibility of the corresponding reference method. Therefore, homogeneity of the samples was assumed.

One 0.5L bottle, labelled #12073 was dispatched to each of the participating laboratories on May 9, 2012.

2.5 STABILITY OF THE SAMPLES

In order to be sure that the material, which was used in this proficiency test, was stable for the valid period, the stability of the material packed in the brown glass bottles was checked prior to use.

2.6 ANALYSES

The participants were requested to determine on sample #12073: Acidity (free acid as Acrylic Acid), Appearance, Colour Pt/Co, Density @ 20°C, MEHQ, Water, Purity as received, Purity on dry basis and some GC-impurities (n-Butanol, n-Butylacetate, n-Butylmetacrylate, n-Butylpropionate, Di-n-Butylether, 2-Ethylhexylacrylate, Isobutylacrylate, other impurities and unknown impurities).

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website.

A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original 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 fax was sent to those laboratories that had not reported results at that moment.

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 results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' 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 statistical evaluation of the results should be used with due care.

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

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

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

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a "x". Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nr.12 and 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 target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated according to:

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

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

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 the fit-for-useness of the reported test result.

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
$ z > 3$	unsatisfactory

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples for Brazil and India. Two laboratories received the samples late. Finally, in total 14 participants did report 138 numerical test results. Observed were 5 outlying test results, which is 3.6% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are normal.

Not normal distributions were found with the following determinations: Density and Isobutylacrylate. 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: This determination was not problematic. Two statistical outliers were detected. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D1613:06.
- Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #12073, which is pass (= bright, clear and free of suspended matter).
- Colour Pt/Co: No analytical problems were observed. No statistical outliers were detected and the calculated reproducibility is in full agreement with the requirements of ASTM D1209:11.
- Density @ 20°C: No analytical problems were observed. No statistical outliers were detected and the calculated reproducibility is in good agreement with the requirements of ASTM D4052:11.
- MEHQ: This determination was problematic for one laboratory. One statistical outlier was detected and the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D3125:06.
- Water: This determination was not problematic. One statistical outlier was detected. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D1364:07.
- Purity as received: No analytical problems were observed. No statistical outlier was detected and the calculated reproducibility is in full agreement with the

requirements of ASTM D3362:11, which was withdrawn with no replacement.

Purity on dry basis: No analytical problems were observed. One statistical outlier was detected and the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D3362:11, which was withdrawn with no replacement.

n-Butanol: This determination was problematic for one laboratory that reported a false negative. No statistical outliers were observed and the calculated reproducibility is in full agreement with the calculated reproducibility estimated using the Horwitz equation.

n-Butylacetate: No analytical problems were observed. No statistical outliers were observed and the calculated reproducibility is in full agreement with the calculated reproducibility estimated using the Horwitz equation.

n-Butylmetacrylate: For this impurity all, except for one (false positive result?), reporting participants agreed on a result below 10. Therefore no significant conclusions were drawn.

n-Butylpropionate: No analytical problems were observed. No statistical outliers were observed and the calculated reproducibility is in full agreement with the calculated reproducibility estimated using the Horwitz equation.

Di-n-Butylether: No analytical problems were observed. No statistical outliers were observed and the calculated reproducibility is in full agreement with the calculated reproducibility estimated using the Horwitz equation.

2-Ethylhexylacrylate: For this impurity all reporting participants agreed on a result below 10. Therefore no significant conclusions were drawn.

Isobutylacrylate: No analytical problems were observed. No statistical outliers were observed and the calculated reproducibility is in good agreement with the calculated reproducibility estimated using the Horwitz equation.

Other Impurities: No statistical outliers were detected, but the reported test results vary strongly: from 73 - 580 mg/kg. Because no target reproducibility is known, no significant conclusions were drawn.

Total Impurities: No statistical outliers were detected, but the reported test results vary strongly: from 2076 - 2796 mg/kg. Because no target reproducibility is known, 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 ASTM test methods) and estimated by using the Horwitz equation, see tables in appendix 1.

Parameter	unit	n	average	R (calc.)	R (lit)
Free Acid as Acrylic Acid	%M/M	10	0.0010	0.0007	0.0014
Colour Pt/Co		9	3.4	5.1	7.0
Density @ 20°C	kg/L	13	0.8990	0.0002	0.0005
MEHQ	mg/kg	13	15.7	1.2	2.4
Water	mg/kg	12	95.5	48.4	58.6
Purity as received	%M/M	11	99.75	0.12	0.27
Purity on dry basis	%M/M	8	99.75	0.05	0.27
n-Butanol	mg/kg	7	118	25	26
n-Butylacetate	mg/kg	9	170	20	35
n-Butylmetacrylate	mg/kg	7	<10	n.a.	n.a.
n-Butylpropionate	mg/kg	8	379	65	68
Di-n-Butylether	mg/kg	8	123	18	27
2-Ethylhexylacrylate	mg/kg	6	<10	n.a.	n.a.
Isobutylacrylate	mg/kg	7	1231	77	189
Other impurities	mg/kg	5	371	n.a.	n.a.
Total impurities	mg/kg	7	2447	729	n.a.

table 3: reproducibilities of results of sample #12073

Without further statistical calculations it can be concluded that for many tests there is a very 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 2012 WITH THE PREVIOUS PTS

	May 2012	April 2010	April 2008	May 2007
Number of reporting labs	14	17	17	15
Number of results reported	138	202	140	143
Statistical outliers	5	19	5	17
Percentage outliers	3.6%	9.4%	3.6%	11.9%

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:

Determination	May 2012	April 2010	April 2008	May 2007
Free Acid as Acrylic Acid	++	-	+	--
Colour Pt/Co	++	++	+	++
Density @ 20°C	++	++	++	++
Water	++	--	++	++
MEHQ	++	+/-	++	++
Purity as received	++	++	++	++
Purity on dry basis	++	++	++	++
n-Butanol	+/-	--	+	--
n-Butylacetate	++	+	+	--
n-Butylpropionate	+/-	+/-	+/-	--
Di-n-Butylether	++	--	+	-
Isobutylacrylate	++	++	++	++

table 5: comparison determinations against the target reproducibility requirements

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

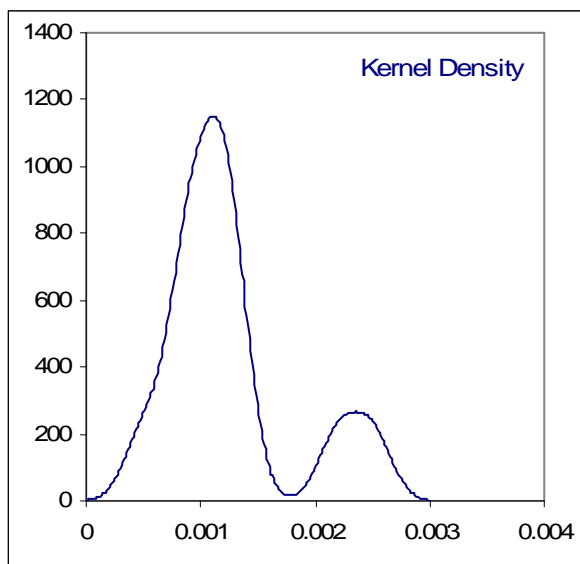
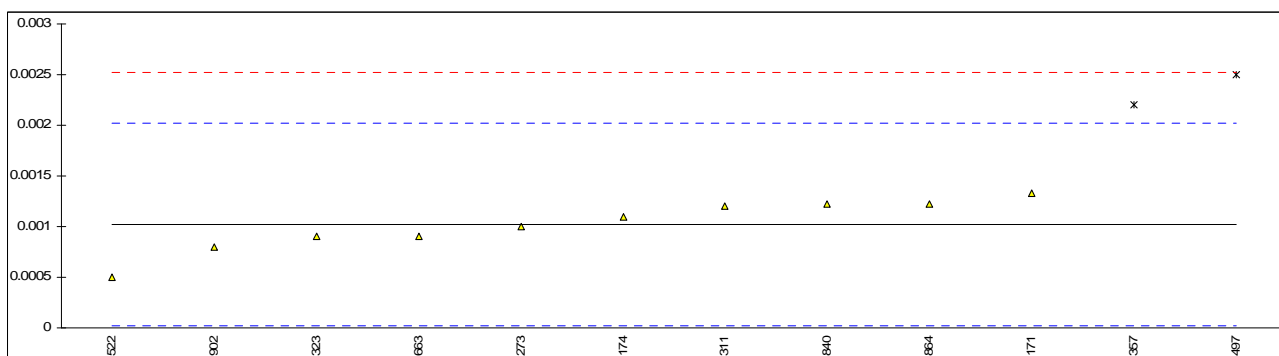
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.d.: not determined
- n.e.: not evaluated

APPENDIX 1

Determination of Acidity (Free Acid as Acrylic Acid) on sample #12073; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D1613	0.001335		0.64	
174	D1613	0.0011		0.17	
273	D1613	0.0010		-0.03	
311	D1613	0.0012		0.37	
323	D1613	0.0009		-0.23	
357	D1613	0.0022	C,G(0.01)	2.37	First reported 0.00204
497	D1613	0.0025	G(0.01)	2.97	
522	D1613	0.0005		-1.03	
551		----		----	
613		----		----	
663	D1613	0.0009		-0.23	
840	D1613	0.00122		0.41	
864	D1613	0.00122		0.41	
886		----		----	
902	D1613	0.0008		-0.43	
913		----		----	

normality OK
n 10
outliers 2
mean (n) 0.00102
st.dev. (n) 0.000251
R(calc.) 0.00070
R(D1613:06) 0.00140



Determination of Appearance on sample #12073;

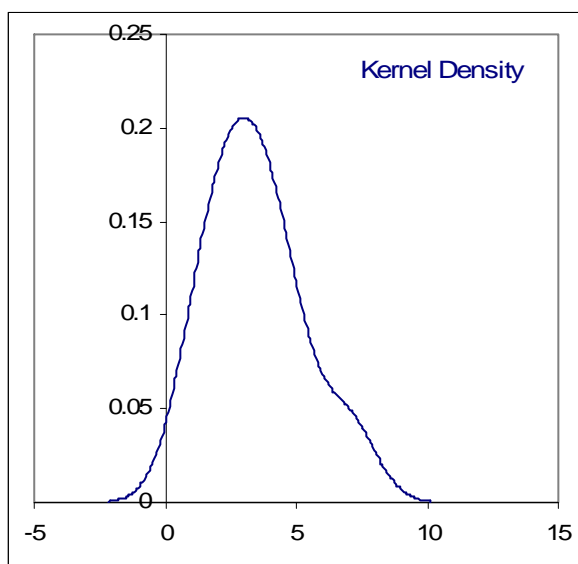
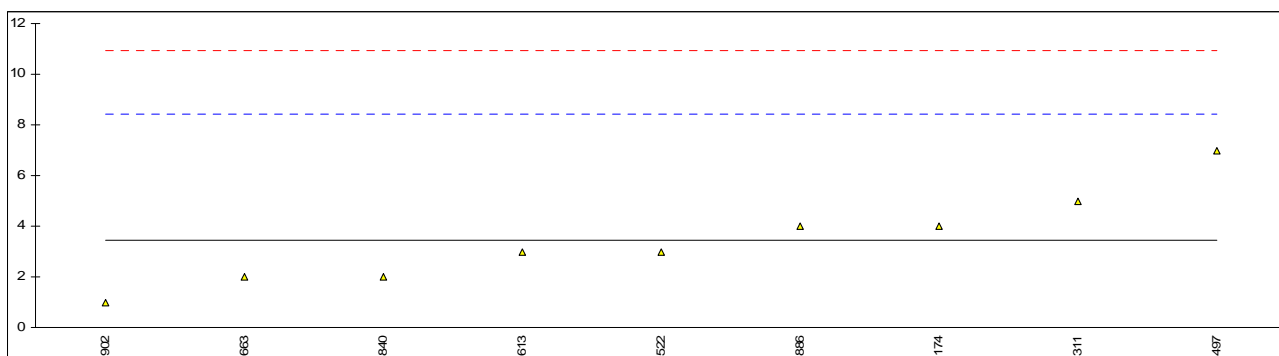
lab	method	value	mark	z(targ)	remarks
171	E2680	pass		----	
174	E2680	pass		----	
273	E2680	pass		----	
311	E2680	C&F		----	
323	E2680	pass		----	
357	E2680	pass		----	
497	E2680	pass		----	
522	E2680	CLMS		----	
551		----		----	
613		----		----	
663	E2680	pass		----	
840	E2680	pass		----	
864	E2680	pass		----	
886	E2680	pass		----	
902	E2680	pass		----	
913		----		----	
	normality	n.a.			
	n	13			
	outliers	n.a.			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(E2680:09e1)	n.a.			

C&F = Clear and free
 CFMS = Clear and free from suspended matter
 B&C = Bright and clear
 CCL = Clear colourless liquid

Determination of Colour Pt/Co on sample #12073;

lab	method	value	mark	z(targ)	remarks
171	D1209	<5		----	
174	D1209	4		0.22	
273	D1209	<10		----	
311	D1209	5		0.62	
323	D1209	<5		----	
357	D1209	<5		----	
497	D1209	7		1.42	
522	D1209	3		-0.18	
551		----		----	
613	D5386	3		-0.18	
663	D1209	2		-0.58	
840	D1209	2		-0.58	
864	D1209	<5		----	
886	D1209	4		0.22	
902	D5386	1		-0.98	
913		----		----	

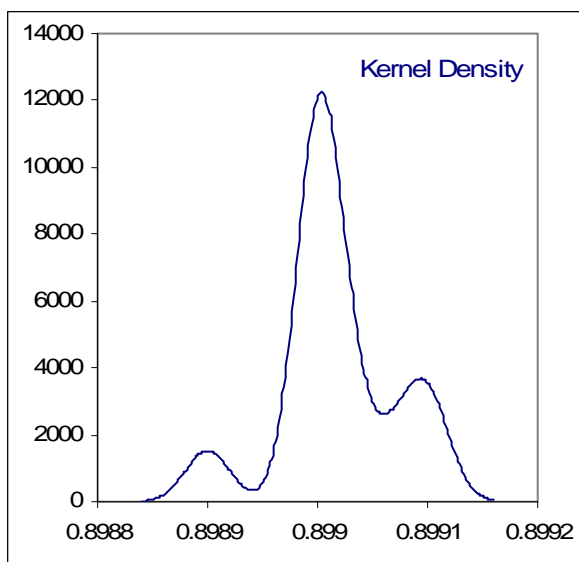
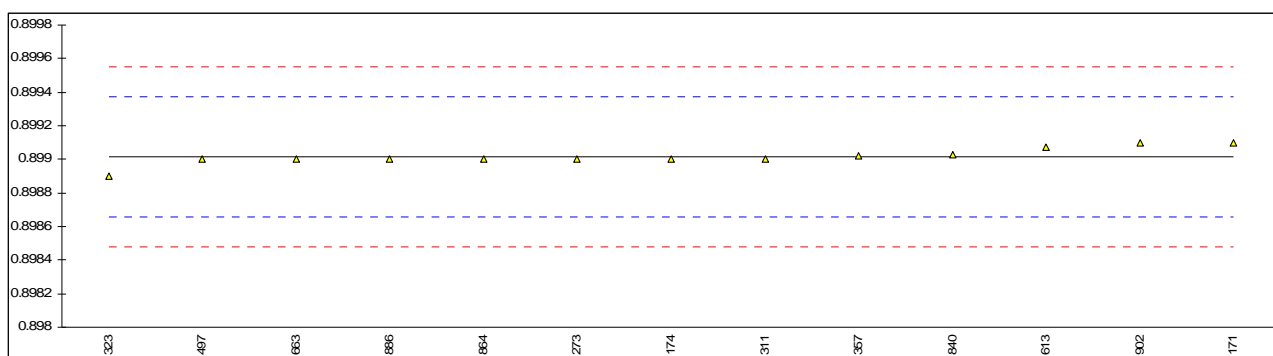
normality OK
n 9
outliers 0
mean (n) 3.4
st.dev. (n) 1.81
R(calc.) 5.1
R(D1209:11) 7.0



Determination of Density @20°C on sample #12073; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.8991		0.46	
174	D4052	0.8990		-0.10	
273	D4052	0.8990		-0.10	
311	D4052	0.8990		-0.10	
323	D4052	0.8989		-0.66	
357	D4052	0.89902		0.02	
497	D4052	0.8990		-0.10	
522		-----		-----	
551		-----		-----	
613	D4052	0.89907		0.30	
663	D4052	0.8990		-0.10	
840	D4052	0.89903		0.07	
864	D4052	0.8990		-0.10	
886	D4052	0.8990		-0.10	
902	D4052	0.89910		0.46	
913		-----		-----	

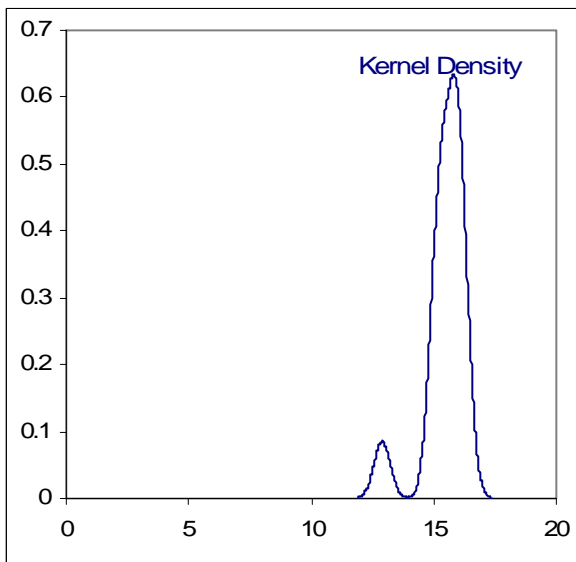
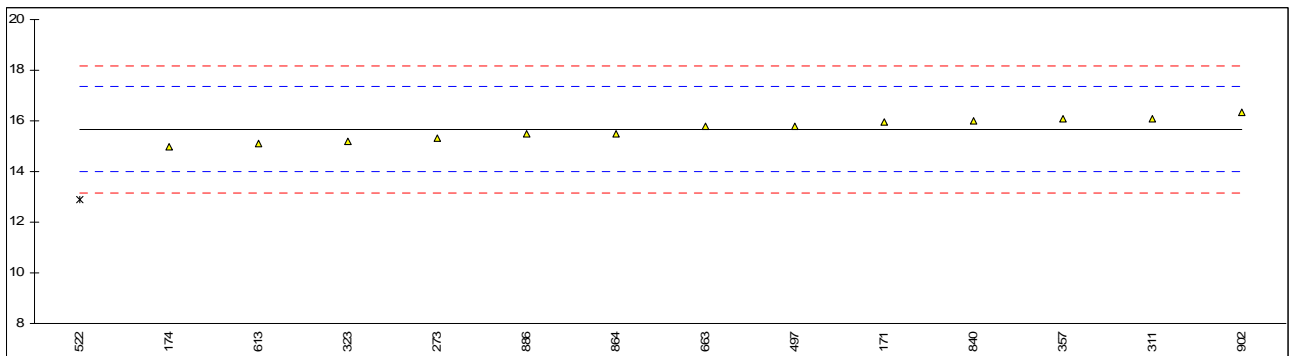
normality not OK
n 13
outliers 0
mean (n) 0.89902
st.dev. (n) 0.000052
R(calc.) 0.00015
R(D4052:11) 0.00050



Determination of MEHQ on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3125	15.96		0.35	
174	D3125	15		-0.80	
273	D3125	15.3		-0.44	
311	D3125	16.1		0.51	
323	D3125	15.2		-0.56	
357	D3125	16.07		0.48	
497	D3125	15.8		0.16	
522	D3125	12.9	G(0.01)	-3.30	
551		-----		-----	
613	D3125	15.11		-0.67	
663	D3125	15.8		0.16	
840	D3125	16.00		0.39	
864	D3125	15.5		-0.20	
886	D3125	15.5		-0.20	
902	D3125	16.35		0.81	
913		-----		-----	

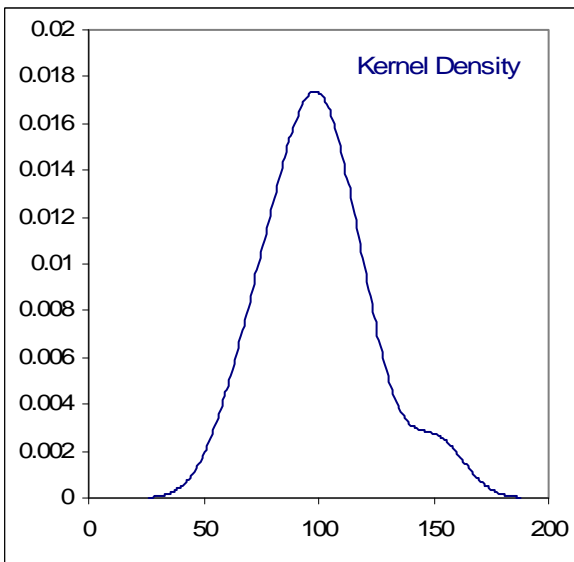
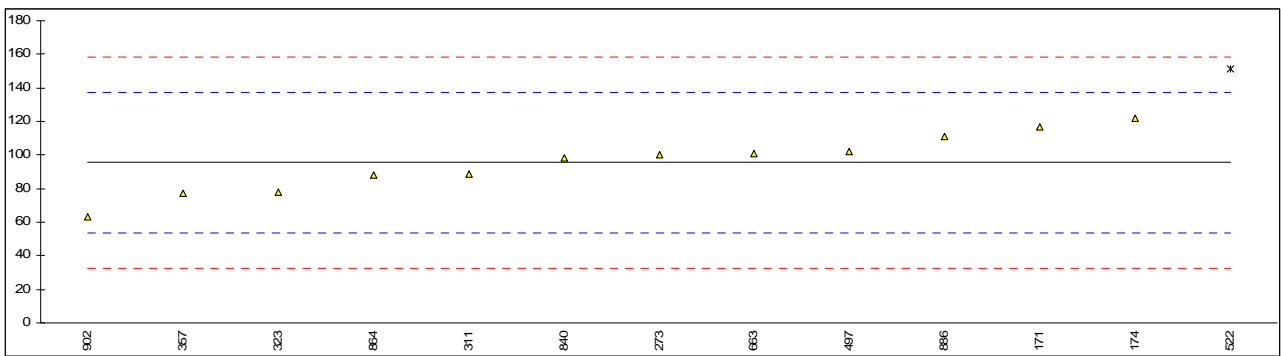
normality OK
n 13
outliers 1
mean (n) 15.668
st.dev. (n) 0.4294
R(calc.) 1.202
R(D3125:06) 2.350



Determination of Water on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	116.5		1.00	
174	D1364	122		1.27	
273	D1364	100		0.22	
311	D1364	89		-0.31	
323	D1364	78		-0.83	
357	E1064	77		-0.88	
497	D1364	102		0.31	
522	E203	151	G(0.05)	2.65	
551		-----		-----	
613		-----		-----	
663	E1064	101		0.26	
840	E1064	98		0.12	
864	E1064	88		-0.36	
886	D1364	111.1		0.75	
902	D1364	63		-1.55	
913		-----		-----	

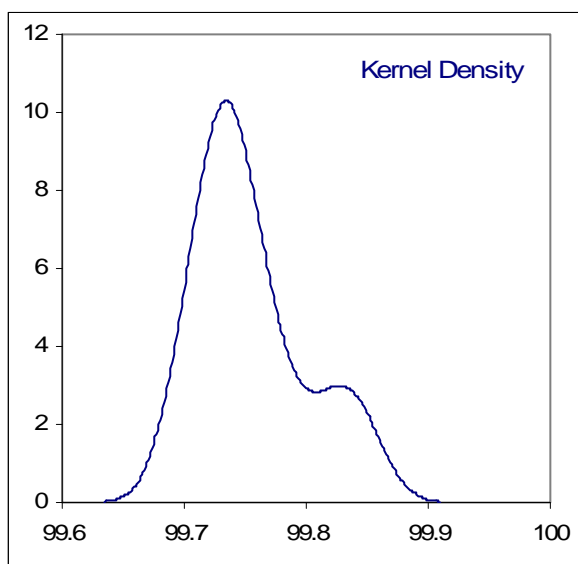
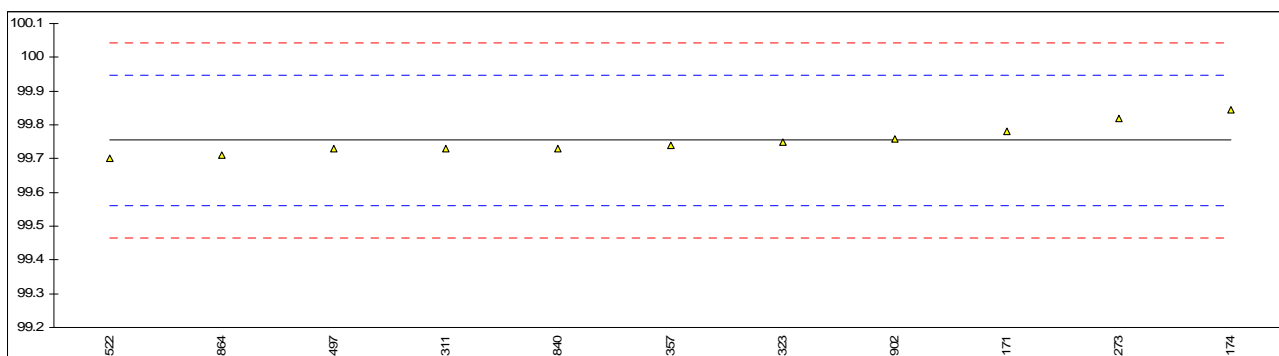
normality OK
n 12
outliers 1
mean (n) 95.47
st.dev. (n) 17.291
R(calc.) 48.42
R(D1364:07) 58.62



Determination of Purity as received by GLC on sample #12073; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D3362Mod.	99.782	C	0.29	Results probably switched. Result as received > Result on dry basis
174	D3362	99.845		0.94	
273		99.82		0.68	
311	INH-117	99.73	C	-0.25	Results probably switched. Result as received > Result on dry basis
323	INH-307	99.75		-0.05	
357	INH-052	99.74		-0.15	
497	INH-1460	99.73	C	-0.25	Results probably switched. Result as received > Result on dry basis
522	D3362	99.7		-0.56	
551		----		----	
613		----		----	
663		----		----	
840	INH-004	99.731		-0.24	
864	D3362	99.711		-0.45	
886		----		----	
902		99.759	C	0.05	Results probably switched. Result as received > Result on dry basis
913		----		----	

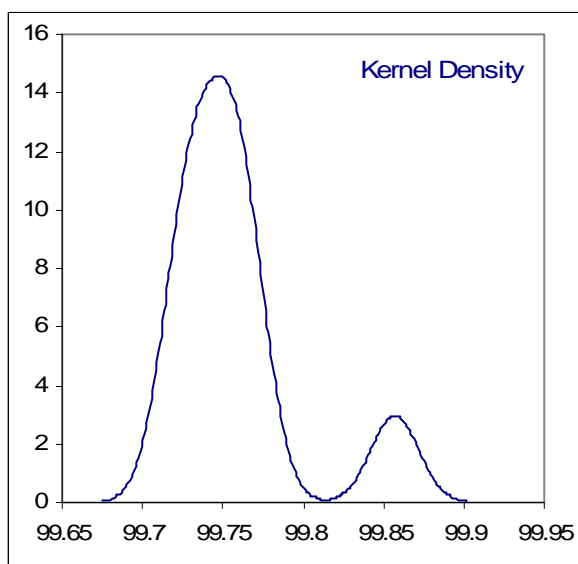
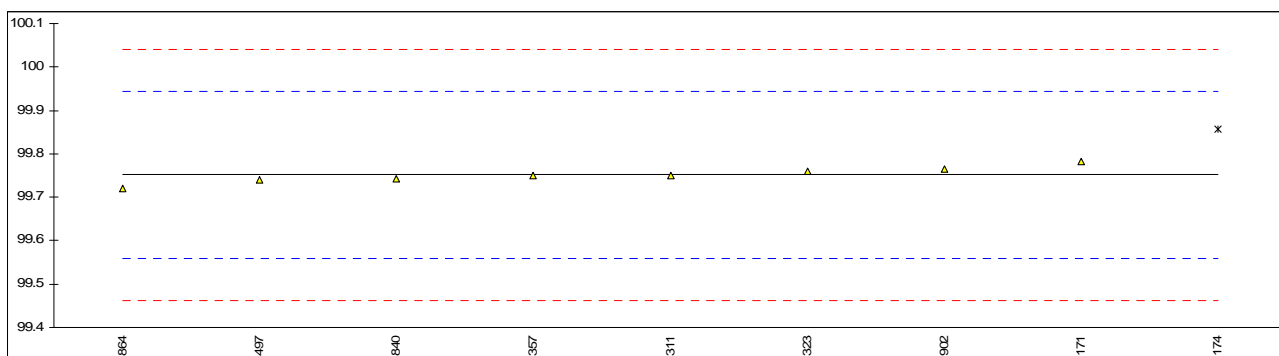
normality OK
n 11
outliers 0
mean (n) 99.7544
st.dev. (n) 0.04484
R(calc.) 0.1255
R(D3362:11) 0.2700



Determination of Purity on dry basis by GLC on sample #12073; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D3362	99.782	C	0.32	Results probably switched. Result as received > Result on dry basis
174	D3362	99.857	G(0.01)	1.10	
273		-----		-----	
311	INH-117	99.75	C	-0.01	Results probably switched. Result as received > Result on dry basis
323	INH-307	99.76		0.09	
357	INH-052	99.75		-0.01	
497	INH-1460	99.74	C	-0.12	Results probably switched. Result as received > Result on dry basis
522		-----		-----	
551		-----		-----	
613		-----		-----	
663		-----		-----	
840	INH-004	99.742		-0.10	
864	D3362	99.720		-0.32	
886		-----		-----	
902	D3362	99.766	C	0.15	Results probably switched. Result as received > Result on dry basis
913		-----		-----	

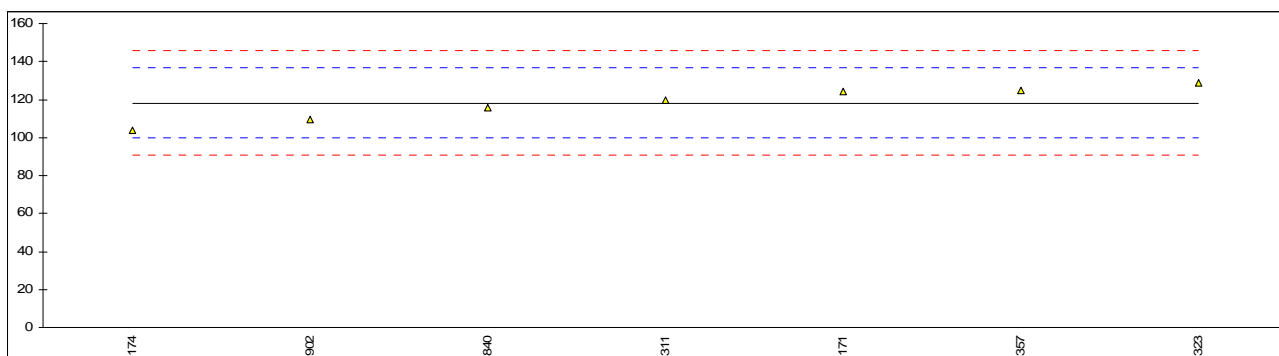
normality OK
n 8
outliers 1
mean (n) 99.7512
st.dev. (n) 0.01864
R(calc.) 0.0522
R(D3362:11) 0.2700



Determination of n-Butanol on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362	124		0.63	
174	D3362	104		-1.54	
273		-----		-----	
311	INH-117	120	C	0.19	First reported 0.0120
323	INH-307	129		1.17	
357	INH-052	125		0.74	
497	INH-1460	<10	ex	-----	False negative?
522		-----		-----	
551		-----		-----	
613		-----		-----	
663		-----		-----	
840	INH-004	116		-0.24	
864		-----		-----	
886		-----		-----	
902	D3362	109.5		-0.94	
913		-----		-----	

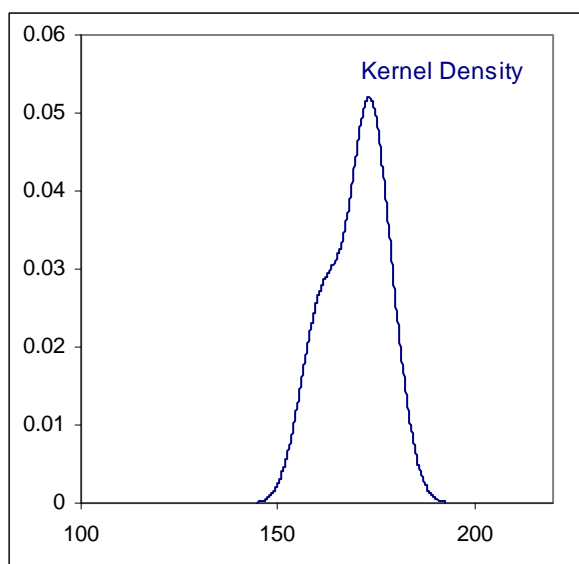
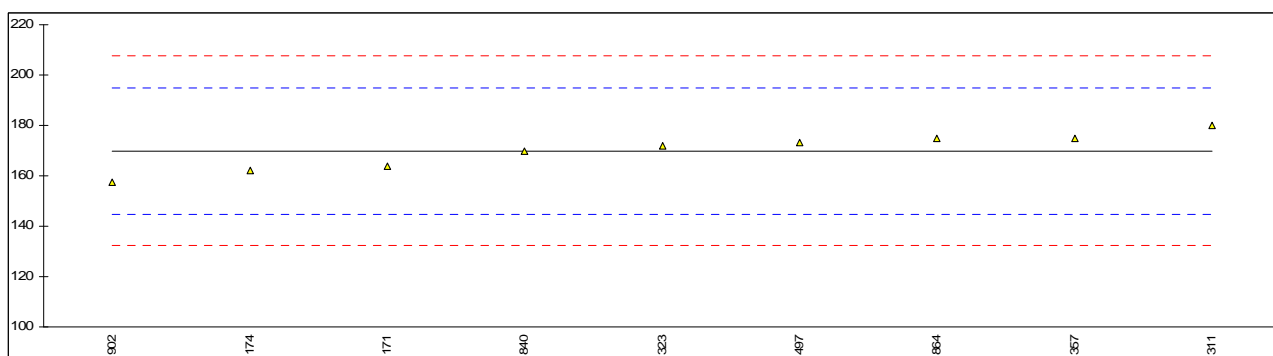
normality OK
n 7
outliers 0
mean (n) 118.2
st.dev. (n) 8.96
R(calc.) 25.1
R(Horwitz) 25.8



Determination of n-Butylacetate on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362	164		-0.46	
174	D3362	162	C	-0.62	First reported 104
273		-----		-----	
311	INH-117	180	C	0.81	First reported 0.0180
323	INH-307	172		0.17	
357	INH-052	175		0.41	
497	INH-1460	173		0.25	
522		-----		-----	
551		-----		-----	
613		-----		-----	
663		-----		-----	
840	INH-004	170		0.01	
864	D3362	175		0.41	
886		-----		-----	
902	D3362	157.5		-0.98	
913		-----		-----	

normality OK
n 9
outliers 0
mean (n) 169.8
st.dev. (n) 7.24
R(calc.) 20.3
R(Horwitz) 35.1



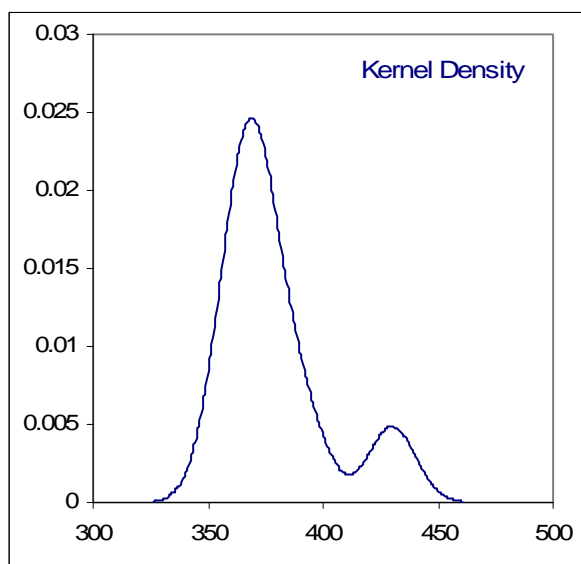
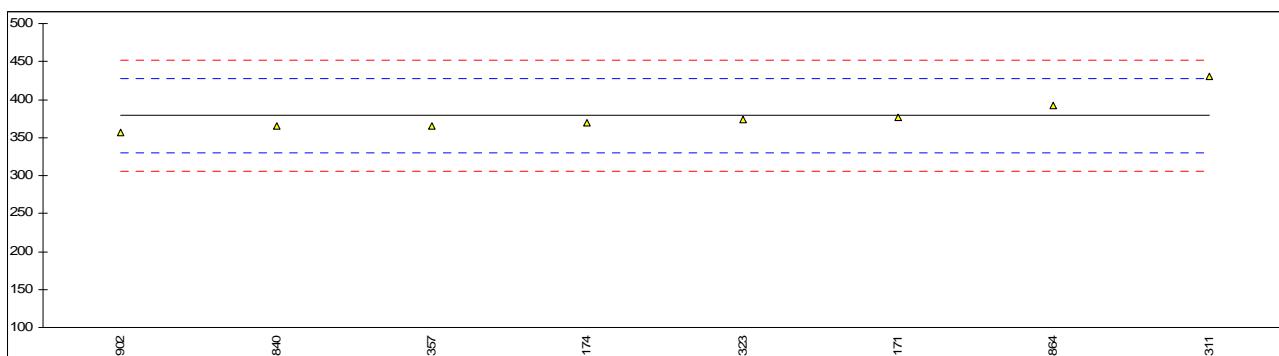
Determination of n-Butylmetacrylate on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362	4		----	
174	D3362	<10	C	----	First reported 71
273		----		----	
311	INH-117	<10	C	----	First reported <0.001
323	INH-307	<20		----	
357	INH-052	<10		----	
497	INH-1460	53		----	False positive result?
522		----		----	
551		----		----	
613		----		----	
663		----		----	
840	INH-004	4		----	
864	D3362	3.4		----	
886		----		----	
902	D3362	<10		----	
913		----		----	
	normality	n.a.			
	n	7			
	outliers	0			
	mean (n)	<10			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of n-Butylpropionate on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362	377		-0.07	
174	D3362	370	C	-0.35	First reported 204
273				-----	
311	INH-117	430	C	2.07	First reported 0.043
323	INH-307	374		-0.19	
357	INH-052	365		-0.56	
497				-----	
522				-----	
551				-----	
613				-----	
663				-----	
840	INH-004	365		-0.56	
864	D3362	392		0.53	
886				-----	
902	D3362	357.3		-0.87	
913				-----	

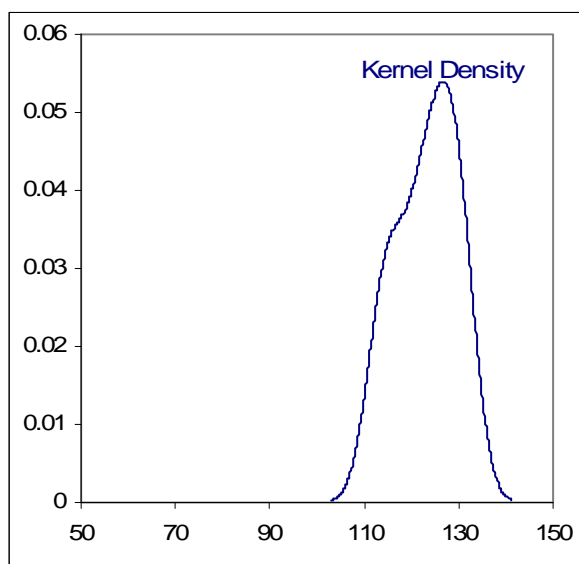
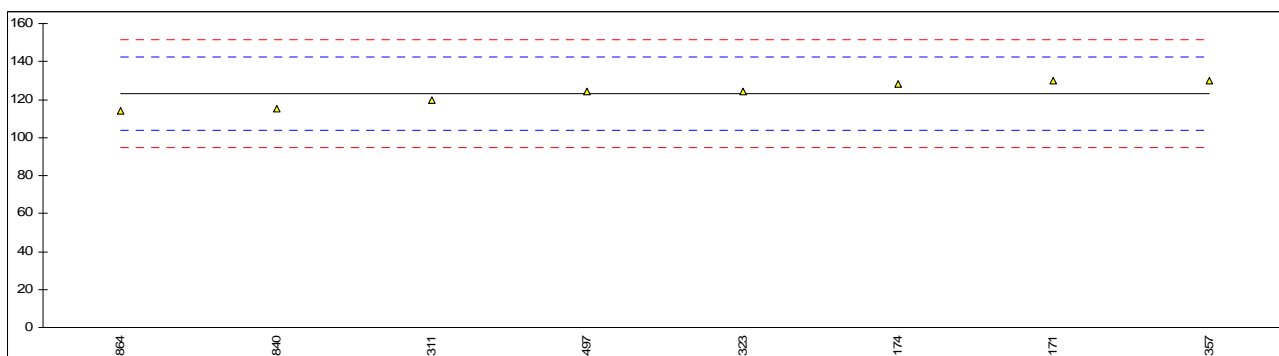
normality OK
n 8
outliers 0
mean (n) 378.8
st.dev. (n) 23.12
R(calc.) 64.74
R(Horwitz) 69.44



Determination of Di-n-Butylether on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362	130		0.72	
174	D3362	128	C	0.51	First reported 75
273		-----		-----	
311	INH-118	120	C	-0.33	First reported 0.012
323	INH-307	124		0.09	
357	INH-052	130		0.72	
497	INH-1460	124		0.09	
522		-----		-----	
551		-----		-----	
613		-----		-----	
663		-----		-----	
840	INH-004	115		-0.85	
864	D3362	114		-0.96	
886		-----		-----	
902		-----		-----	
913		-----		-----	

normality OK
 n 8
 outliers 0
 mean (n) 123.1
 st.dev. (n) 6.31
 R(calc.) 17.7
 R(Horwitz) 26.7



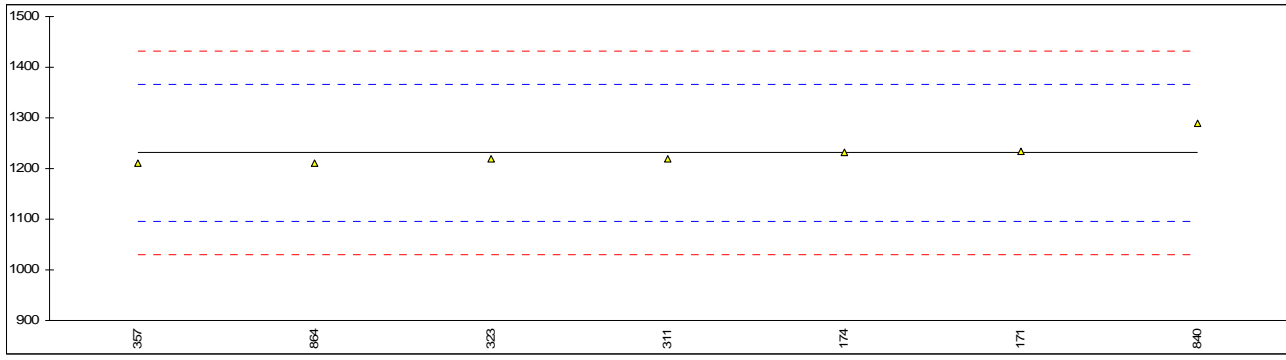
Determination of 2-Ethylhexylacrylate on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362	2		----	
174	D3362	<10		----	
273		----		----	
311	INH-117	10	C	----	First reported 0.001
323	INH-307	<20		----	
357	INH-052	<10		----	
497		----		----	
522		----		----	
551		----		----	
613		----		----	
663		----		----	
840	INH-004	<3		----	
864	D3362	<10		----	
886		----		----	
902	D3362	<10		----	
913		----		----	
	normality	n.a.			
	n	6			
	outliers	n.a.			
	mean (n)	<10			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	n.a.			

Determination of Isobutylacrylate on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362	1234		0.05	
174	D3362	1231	C	0.00	First reported 800
273		-----		-----	
311	INH-117	1220	C	-0.16	First reported 0.122
323	INH-307	1220		-0.16	
357	INH-052	1210		-0.31	
497		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
663		-----		-----	
840	INH-004	1290		0.88	
864	D3362	1211		-0.29	
886		-----		-----	
902		-----		-----	
913		-----		-----	

normality not OK
 n 7
 outliers 0
 mean (n) 1230.9
 st.dev. (n) 27.61
 R(calc.) 77.3
 R(Horwitz) 189.0



Determination of Other Impurities on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362	187		----	
174	D3362	73		----	
273		----		----	
311	INH-117	580	C	----	First reported 0.058
323		----		----	
357	INH-052	495		----	
497		----		----	
522		----		----	
551		----		----	
613		----		----	
663		----		----	
840	INH-004	518		----	
864		----		----	
886		----		----	
902		----		----	
913		----		----	
	normality	n.a.			
	n	5			
	outliers	0			
	mean (n)	370.6			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of Total Impurities on sample #12073; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362	2183		----	
174	D3362	2076	C	----	First reported 1431
273		----		----	
311	INH117/118	2660	C	----	First reported 0.266
323		----		----	
357	INH-052	2500		----	
497		----		----	
522		----		----	
551		----		----	
613		----		----	
663		----		----	
840	INH-004	2578		----	
864	D3362	2796		----	
886		----		----	
902	D3362	2337		----	
913		----		----	
	normality	OK			
	n	7			
	outliers	0			
	mean (n)	2447.1			
	st.dev. (n)	260.31			
	R(calc.)	728.9			
	R(lit)	unknown			

APPENDIX 2

Number of participants per country

- 1 lab in AUSTRALIA
- 1 lab in BELGIUM
- 1 lab in BRAZIL
- 1 lab in FINLAND
- 1 lab in GERMANY
- 1 lab in INDIA
- 1 lab in MEXICO
- 1 lab in P.R. of CHINA
- 1 lab in SOUTH AFRICA
- 1 lab in TAIWAN R.O.C.
- 1 lab in THAILAND
- 1 lab in THE NETHERLANDS
- 1 lab in TURKEY
- 2 labs in U.S.A.
- 1 lab in VIETNAM

APPENDIX 3

Abbreviations:

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

Literature:

- 1 i.i.s. Interlaboratory Studies. Protocol for the Organisation, Statistics and Evaluation, January 2010
- 2 ASTM E178-89
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- 6 M. Thompson and R. Wood. J. AOAC Int. 76. 926. (1993)
- 7 W.J. Youden and E.H. Steiner. Statistical Manual of the AOAC. (1975)
- 8 IP 367/84
- 9 DIN 38402 T41/42
- 10 P.L. Davies. First reported Z. Anal. Chem. 331. 513. (1988)
- 11 J.N. Miller. Analyst. 118. 455. (1993)
- 12 Analytical Methods Committee Technical brief, No4 January 2001.
- 13 The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>).