

Results of Proficiency Test
n-Butylacrylate
April 2010

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

On request, the Institute for Interlaboratory Studies decided to organize again a proficiency test for the analysis of n-Butylacrylate during the annual proficiency testing program 2009/2010. In this interlaboratory study 19 laboratories in 15 different countries have participated. See appendix 2 for the number of participants in 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 sample of 500 mL. 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 guide 43, ISO17043:2010 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 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 10 litre bulk material for sample #1047 was obtained from a local supplier. After homogenisation in a precleaned can, 39 subsamples were transferred to brown glass bottles of 500 mL and labelled #1047. The homogeneity of the subsamples was checked by determination of Density in accordance with ASTM D4052:09 and Water in accordance with ASTM D1364:07 on 4 stratified randomly selected samples.

	Density @15°C in kg/L	Water in mg/kg
sample #1047-1	0.90370	85
sample #1047-2	0.90370	81
sample #1047-3	0.90370	88
sample #1047-4	0.90371	85

table 1: homogeneity test of subsamples #1047

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 @15°C in kg/L	Water in mg/kg
r (sample #1047)	0.00001	8.0
reference test method	R(D4052:09)	ASTM D1364:07
0.3 x R(reference test)	0.00015	16.6

table 2: evaluation of homogeneity of subsamples #1047

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 500 mL bottle, labelled #1047 was dispatched to each of the participating laboratories on May 5, 2010.

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 #1047: 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, 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 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.

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.

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 South Africa. Three laboratories received the samples late. Finally, in total 17 participants did report 202 numerical test results. Observed were 19 outlying test results, which is 9.4% of the numerical test results.

Not normal distributions were found with the following determinations: Density and Water. 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 problematic. Three statistical outliers were detected and the calculated reproducibility, after exclusion of the statistical outliers, is not in agreement with the requirements of ASTM D1613:06.
- Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #1047, which is bright, clear and free of suspended matter. Several laboratories may be able to improve the uniformity of reporting as a new standardized method is available for Appearance since 2009, being ASTM E2680. According this method the appearance should be reported as 'pass' (or 'fail').
- Colour Pt/Co: No analytical problems have been observed. No statistical outliers were detected and the calculated reproducibility is in full agreement with the requirements of ASTM D1209:05
- Density @ 20°C: No analytical problems have been observed. Only one statistical outlier was detected and the calculated reproducibility, after exclusion of the statistical outlier, is in full agreement with the requirements of ASTM D4052:09.
- MEHQ: This determination was problematic for several laboratories. Three statistical outliers were detected. However, after exclusion of the statistical outliers, the calculated reproducibility is in full agreement with the requirements of ASTM D3125:06.
- Water: This determination was very problematic. No statistical outliers were detected. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D1364:07.
- Purity as received: No analytical problems have been observed. Only one statistical outlier was detected and the calculated reproducibility, after exclusion of the statistical outlier, is in full agreement with the requirements of ASTM D3362:05.
- Purity on dry basis: No analytical problems have been observed. Two statistical outliers were detected. However, after exclusion of the statistical outliers, the calculated reproducibility is in full agreement with the requirements of ASTM D3362:05.

- n-Butanol: The determination of this impurity may be problematic. Only one statistical outlier was detected. Also, one false negative test result was reported. However, after exclusion of the statistical outlier, the calculated reproducibility is not at all in agreement with the reproducibility estimated from the Horwitz equation.
- n-Butylacetate: No analytical problems have been observed. Only one statistical outlier was detected and the calculated reproducibility is, after exclusion of the statistical outlier, in full agreement with the reproducibility estimated from the Horwitz equation.
- n-Butylmetacrylate: For this impurity all reporting participants agreed on a result below 10. Only three laboratories reported a numerical result.
- n-Butylpropionate: The determination of this impurity may be problematic for two laboratories. Two statistical outliers were detected. However, after exclusion of the statistical outliers, the calculated reproducibility is in full agreement with the reproducibility estimated from the Horwitz equation.
- Di-n-Butylether: The determination of this impurity may be problematic. No statistical outliers were detected. However the calculated reproducibility is not at all in agreement with the reproducibility estimated from the Horwitz equation.
- 2-Ethylhexylacrylate: For this impurity all reporting participants, except one, agreed on a result below 10. Only one laboratory reported a numerical result.
- Isobutylacrylate: No analytical problems have been observed. Only one statistical outlier was detected and the calculated reproducibility is, after exclusion of the statistical outlier, in full agreement with the reproducibility estimated from the Horwitz equation.
- Other Impurities: Only one statistical outlier was detected, but the reported test results vary strongly: from 30 - 587 mg/kg. Because no target reproducibility is known, no significant conclusions were drawn.
- Unknown Impurities: Two statistical outliers were detected and the reported test results vary strongly: from 41 - 2410 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), see tables in appendix 1.

Parameter	unit	n	average	R (Calc.)	R (lit)
Free Acid as Acrylic Acid	%M/M	12	0.0061	0.0017	0.0014
Colour Pt/Co		14	4.1	3.7	7.0
Density @ 20°C	kg/L	15	0.8989	0.0001	0.0005
MEHQ	mg/kg	13	13.6	1.9	2.0
Water	mg/kg	17	103	130	61
Purity as received	%M/M	13	99.77	0.07	0.27
Purity on dry basis	%M/M	12	99.77	0.04	0.27
n-Butanol	mg/kg	12	94	51	21
n-Butylacetate	mg/kg	13	455	70	81
n-Butylmetacrylate	mg/kg	3	4.5	3.8	1.6
n-Butylpropionate	mg/kg	12	294	55	56
Di-n-Butylether	mg/kg	14	971	310	155
2-Ethylhexylacrylate	mg/kg	10	<10	n.a.	n.a.
Isobutylacrylate	mg/kg	14	193	27	39
Other impurities	mg/kg	11	178	318	Unknown
Unknown impurities	mg/kg	8	2259	244	Unknown

table 4: reproducibilities of results of sample #1047

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 APRIL 2010 WITH THE PREVIOUS PTS

	April 2010	April 2008	May 2007	May 2006
Number of reporting labs	17	17	15	12
Number of results reported	202	140	143	104
Statistical outliers	19	5	17	10
Percentage outliers	9.4%	3.6%	11.9%	9.6%

table 5: 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	April 2010	April 2008	May 2007	May 2006	May 2005
Free Acid as Acrylic Acid	-	+	--	++	+/-
Colour Pt/Co	++	+	++	++	++
Density @ 15°C	++	++	++	++	++
MEHQ	+/-	++	++	++	++
Water	--	++	++	++	++
Purity as received	++	++	++	++	++
Purity on dry basis	++	++	++	++	++
n-Butanol	--	+	--	n.e.	n.e.
n-Butylacetate	+	+	--	-	++
n-Butylpropionate	+/-	+/-	--	--	++
Di-n-Butylether	--	+	-	++	++
Isobutylacrylate	++	++	++	++	++

table 6: comparison determinations against the target reproducibility requirements

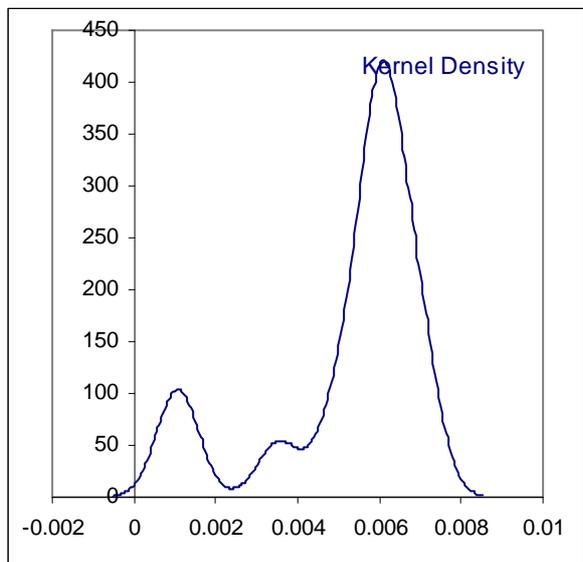
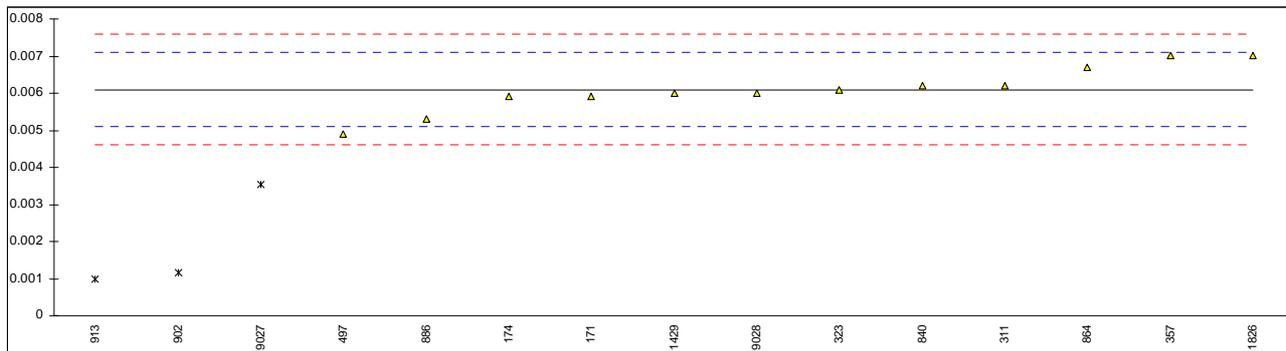
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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 #1047; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D1613	0.0059	C	-0.40	first reported 59.2
174	D1613	0.0059		-0.40	
273		-----		-----	
311	D1613	0.0062		0.20	
323	D1613	0.0061		0.00	
347		-----		-----	
357	D1613	0.0070		1.80	
497	D1613	0.0049		-2.40	
613		-----		-----	
840	D1613	0.00619		0.18	
864	D1613	0.0067		1.20	
886	D1613	0.0053	C	-1.60	first reported 0.053
902	D1613	0.00115	C,DG(0.01)	-9.90	first reported 0.0115
913	D1613	0.00099	DG(0.01)	-10.22	
974		-----		-----	
1429	D1613	0.0060		-0.20	
1826	D1613	0.0070		1.80	
9027	INH-16005	0.00353	G(0.05)	-5.14	
9028	INH-9015	0.006		-0.20	
normality		OK			
n		12			
outliers		3			
mean (n)		0.00610			
st.dev. (n)		0.000616			
R(calc.)		0.00173			
R(D1613:06)		0.00140			



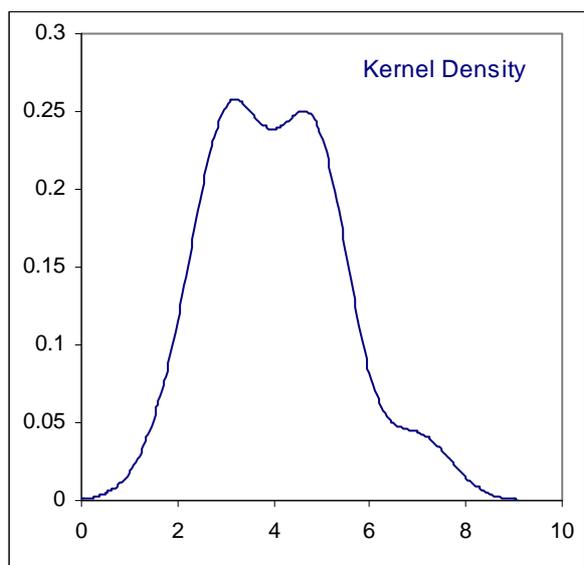
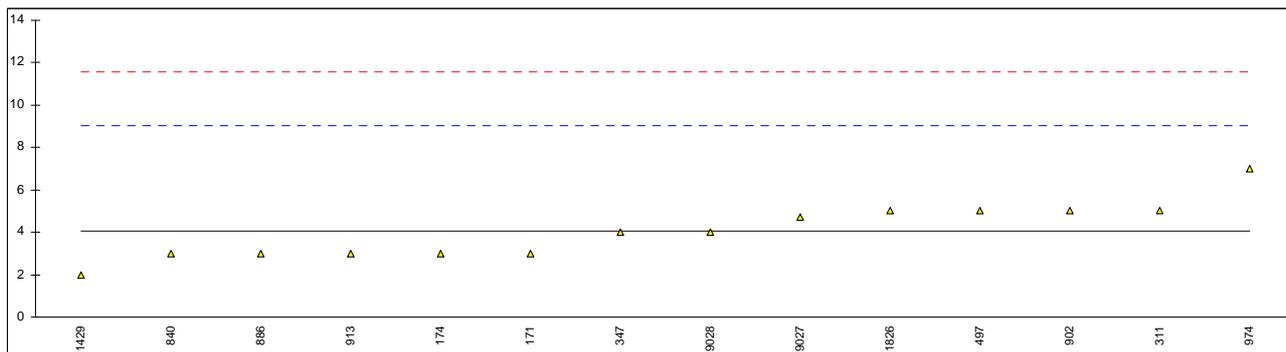
Determination of Appearance on sample #1047;

lab	method	value	mark	z(targ)	remarks
171	E2680	C&F		----	
174	E2680	C&F		----	
273		----		----	
311	E2680	pass		----	
323	E2680	pass		----	
347	E2680	pass		----	
357	E2680	pass		----	
497	E2680	B&C		----	
613		----		----	
840	D2680	pass		----	
864	E2680	pass		----	
886	E2680	pass		----	
902	E2680	pass		----	
913	E2680	CFFSM		----	
974	VISUAL	pass		----	
1429	E2680	B&C		----	
1826	E2680	C&F		----	
9027	VISUAL	pass		----	
9028	INH-9002	pass		----	

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

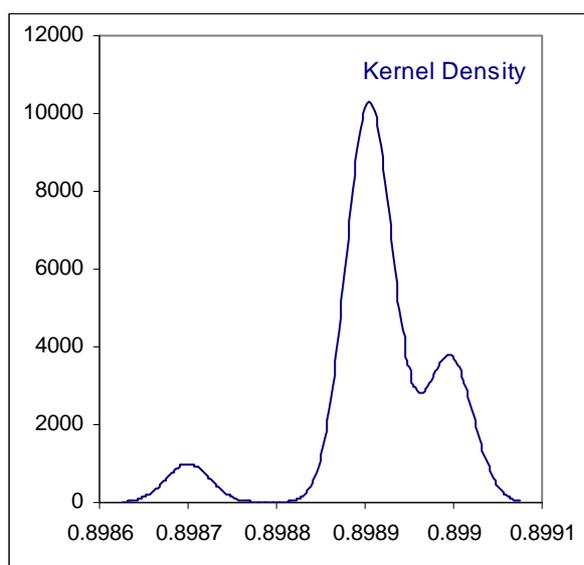
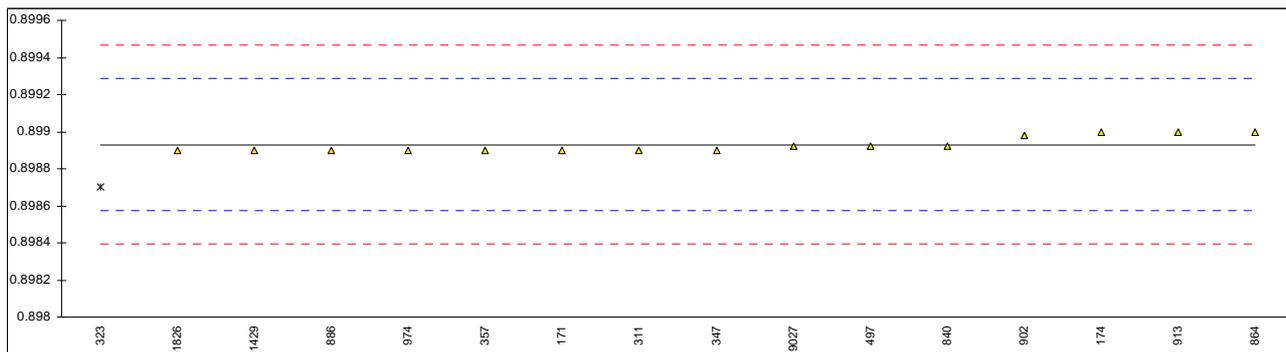
Determination of Colour Pt/Co on sample #1047;

lab	method	value	mark	z(targ)	remarks
171	D1209	3		-0.42	
174	D1209	3		-0.42	
273		-----			
311	D1209	5		0.38	
323	D1209	<5		-----	
347	D1209	4		-0.02	
357	D1209	<5		-----	
497	D1209	5		0.38	
613		-----			
840	D1209	3		-0.42	
864	D1209	<5		-----	
886	D1209	3		-0.42	
902	D5386	5		0.38	
913	D5386	3.0		-0.42	
974	D1209	7		1.18	
1429	D5386	2		-0.82	
1826	D1209	5		0.38	
9027	INH-3001	4.7		0.26	
9028	INH-9001	4		-0.02	
normality		OK			
n		14			
outliers		0			
mean (n)		4.05			
st.dev. (n)		1.314			
R(calc.)		3.68			
R(D1209:05)		7			



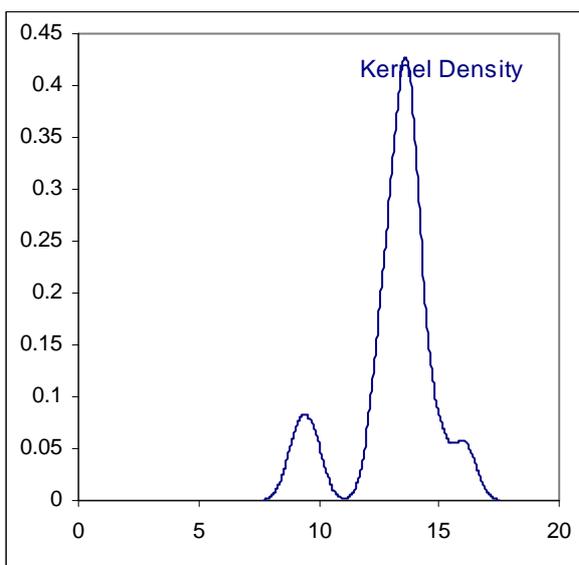
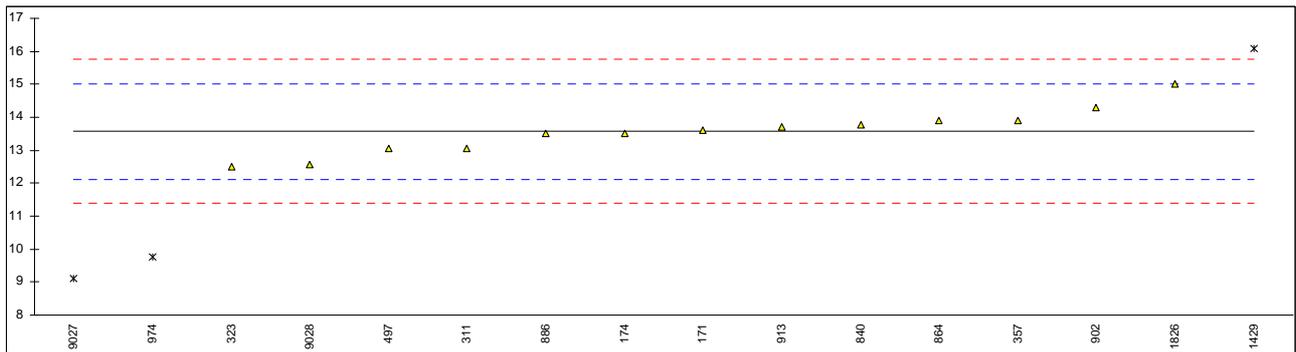
Determination of Density @20°C on sample #1047; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.8989		-0.16	
174	D4052	0.8990		0.40	
273		-----		-----	
311	D4052	0.8989		-0.16	
323	D4052	0.8987	G(0.01)	-1.28	
347	D4052	0.8989		-0.16	
357	D4052	0.8989		-0.16	
497	D4052	0.89892		-0.05	
613		-----		-----	
840	D4052	0.89892		-0.05	
864	D4052	0.8990		0.40	
886	D4052	0.8989		-0.16	
902	D4052	0.89898		0.28	
913	D4052	0.8990		0.40	
974	D4052	0.8989		-0.16	
1429	D4052	0.8989	C	-0.16	first reported 0.9005
1826	D4052	0.8989		-0.16	
9027	INH-1001	0.89892		-0.05	
9028		-----		-----	
normality		not OK			
n		15			
outliers		1			
mean (n)		0.89893			
st.dev. (n)		0.000042			
R(calc.)		0.00012			
R(D4052:09)		0.0005			



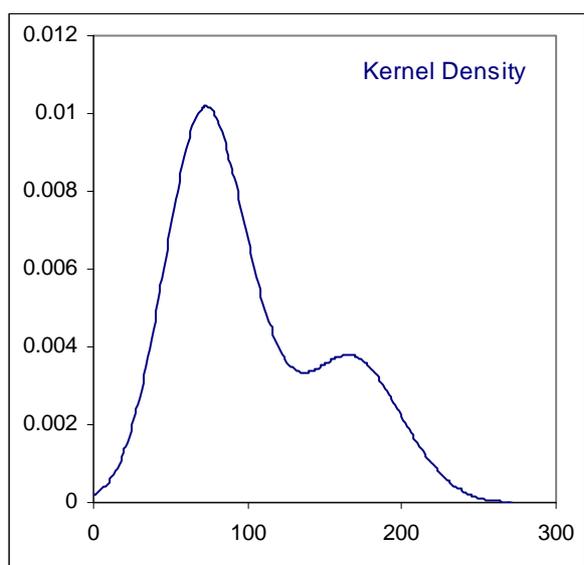
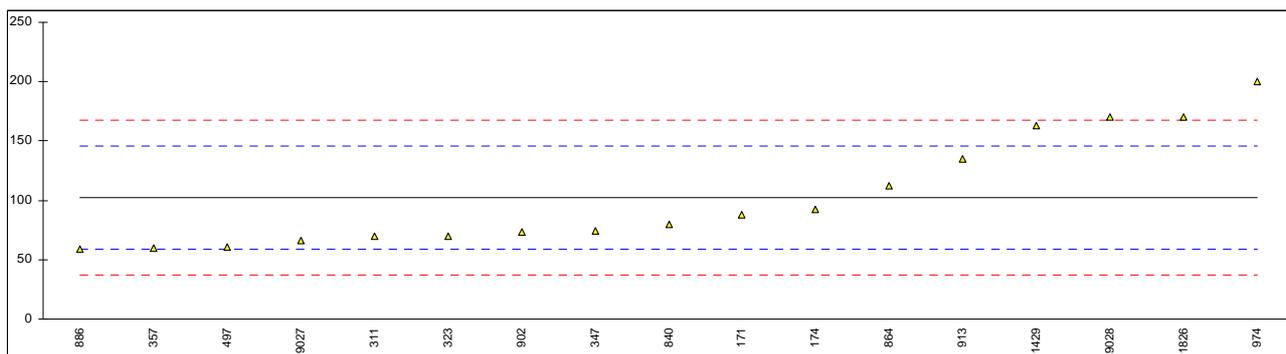
Determination of MEHQ on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3125	13.6		0.05	
174	D3125	13.5		-0.09	
273				-----	
311	D3125	13.06		-0.69	
323	D3125	12.5		-1.47	
347				-----	
357	D3125	13.9		0.46	
497	D3125	13.06		-0.69	
613				-----	
840	D3125	13.76		0.27	
864	D3125	13.9		0.46	
886	D3125	13.5		-0.09	
902	D3125	14.3		1.01	
913	D3125	13.7		0.19	
974	D3125	9.7601	DG(0.01)	-5.24	
1429	D3125	16.1	D(0.05)	3.49	
1826	D3125	15.0		1.98	
9027	INH-16005	9.1	DG(0.01)	-6.14	
9028	INH-1900	12.560		-1.38	
normality		OK			
n		13			
outliers		3			
mean (n)		13.56			
st.dev. (n)		0.680			
R(calc.)		1.90			
R(D3125:06)		2.03			



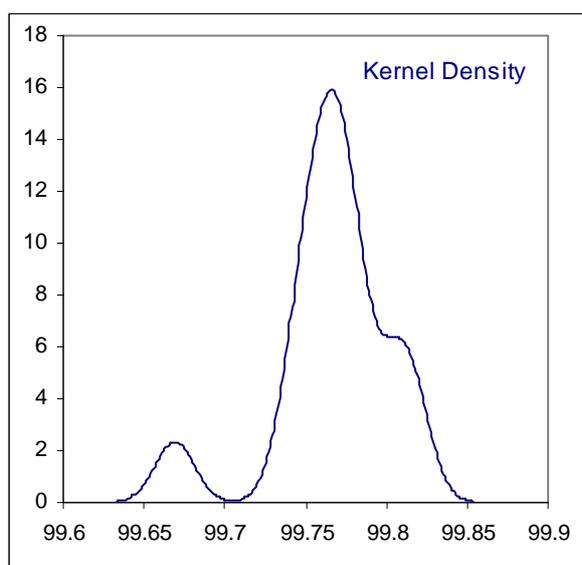
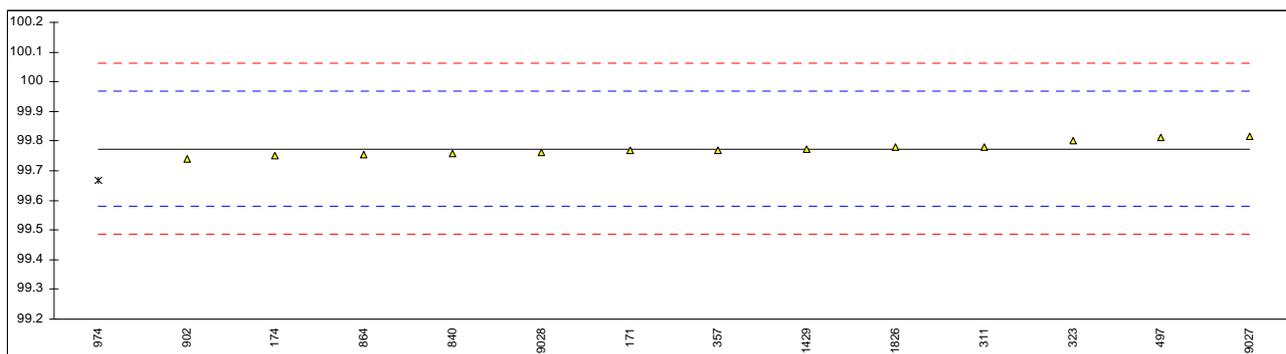
Determination of Water on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	87.8		-0.68	
174	D1364	92		-0.49	
273		-----		-----	
311	D1364	70		-1.50	
323	D1364	70		-1.50	
347	E1064	74		-1.31	
357	E1064	60		-1.96	
497	D1364	61		-1.91	
613		-----		-----	
840	E1064	80		-1.04	
864	E1064	112		0.44	
886	D1364	59.0		-2.01	
902	D1364	73		-1.36	
913	D1364	135		1.50	
974	E203	200		4.49	
1429	D1364	163		2.79	
1826	D1364	170		3.11	
9027	INH-007	66.1		-1.68	
9028	INH-9000	170	C	3.11	first reported 0.01
normality		not OK			
n		17			
outliers		0			
mean (n)		102.5			
st.dev. (n)		46.64			
R(calc.)		130.6			
R(D1364:07)		60.8			



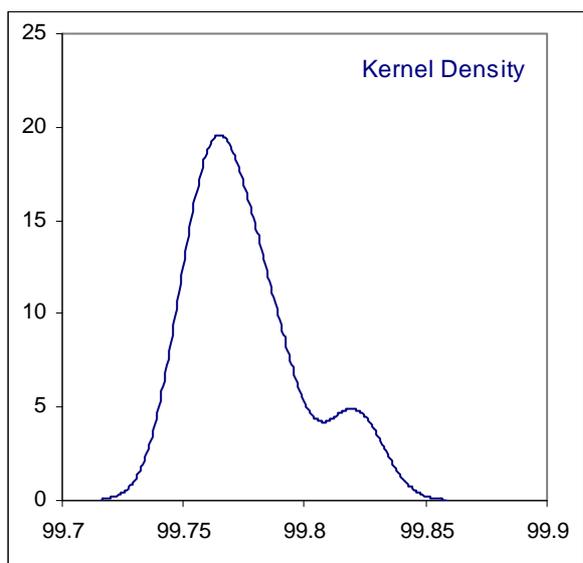
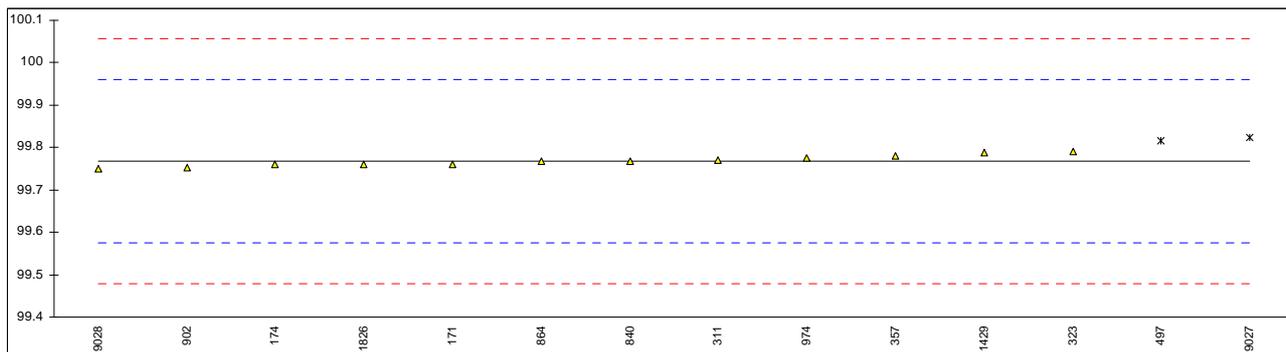
Determination of Purity as received by GLC on sample #1047; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D3362mod	99.769		-0.05	
174	D3362	99.749		-0.26	
273		-----		-----	
311	inh-117	99.78		0.06	
323	inh-307	99.80		0.27	
347		-----		-----	
357	inh-052	99.77		-0.04	
497	in house	99.811		0.38	
613		-----		-----	
840	inh-0004	99.759		-0.16	
864	D3362	99.756		-0.19	
886		-----		-----	
902	D3362	99.741		-0.34	
913		-----		-----	
974	D3362	99.669	G(0.05)	-1.09	
1429	D3362	99.772		-0.02	
1826	in house	99.78		0.06	
9027	calc	99.817		0.44	
9028	D3362	99.76		-0.15	
normality		OK			
n		13			
outliers		1			
mean (n)		99.774			
st.dev. (n)		0.0232			
R(calc.)		0.065			
R(D3362:05)		0.270			



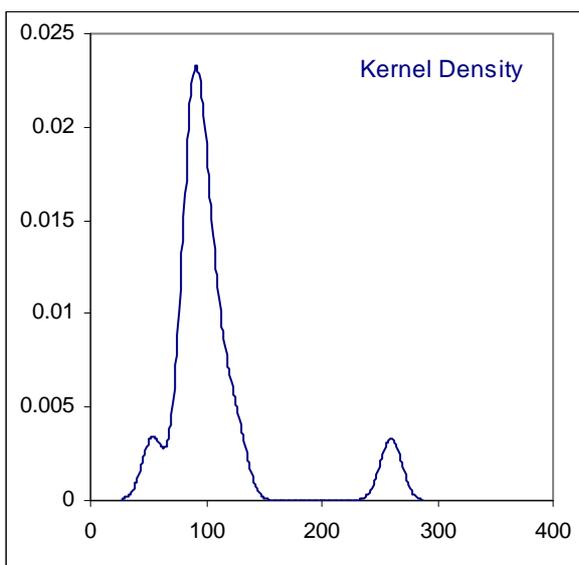
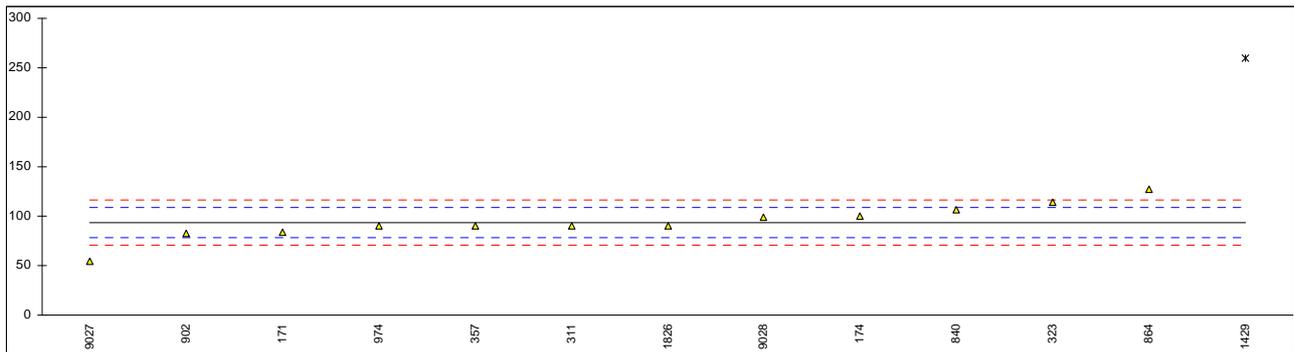
Determination of Purity on dry basis by GLC on sample #1047; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D3362mod	99.760		-0.09	
174	D3362	99.759		-0.10	
273		-----		-----	
311	inh-117	99.77		0.02	
323	inh-307	99.79		0.22	
347		-----		-----	
357	inh-052	99.78		0.12	
497	in house	99.817	DG(0.05)	0.50	
613		-----		-----	
840	inh-0004	99.767		-0.01	
864	D3362	99.767		-0.01	
886		-----		-----	
902	D3362	99.753		-0.16	
913		-----		-----	
974	D3362	99.776		0.08	
1429	D3362	99.788		0.20	
1826	in house	99.76		-0.09	
9027	calc	99.824	DG(0.05)	0.58	
9028	D3362	99.75		-0.19	
normality		OK			
n		12			
outliers		2			
mean (n)		99.768			
st.dev. (n)		0.0130			
R(calc.)		0.036			
R(D3362:05)		0.270			



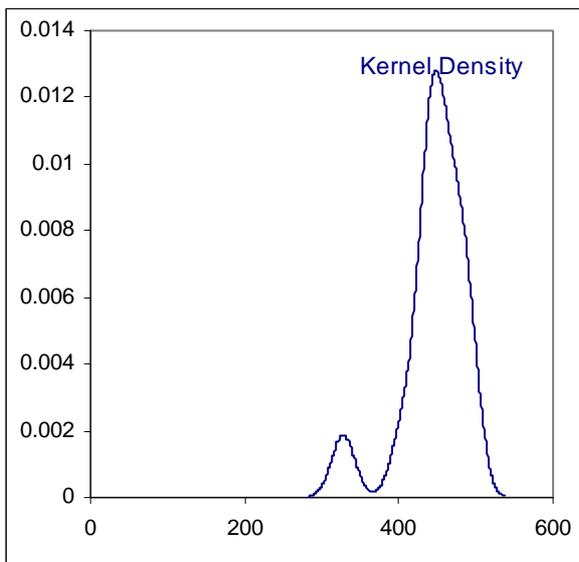
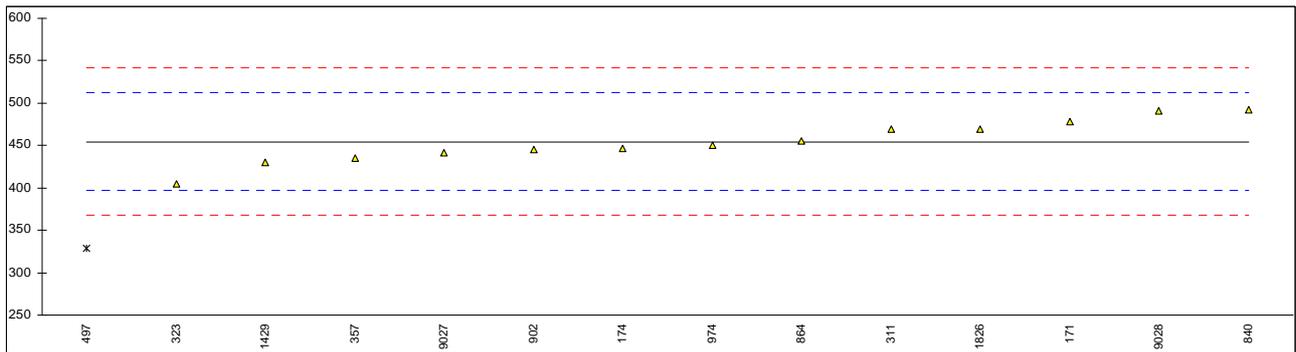
Determination of n-Butanol on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362mod	84.1		-1.29	
174	D3362	100		0.80	
273					
311	inh-117	90		-0.52	
323	inh-307	114		2.65	
347					
357	inh-052	90		-0.52	
497	in house	<10		<-11.06	
613					
840	inh-0004	106		1.59	
864	D3362	127		4.36	
886					
902	D3362	82.65		-1.49	
913					
974	D3362	90		-0.52	
1429	D3362	260	G(0.01)	21.90	co-elutes with n-butylpropionate
1826	in house	90		-0.52	
9027	INH-6001	53.96		-5.27	
9028	INH-1900	99.332		0.71	
	normality	OK			
	n	12			
	outliers	1			
	mean (n)	93.92			
	st.dev. (n)	18.069			
	R(calc.)	50.59			
	R(Horwitz)	21.24			



Determination of n-Butylacetate on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362mod	478.8		0.83	
174	D3362	447		-0.27	
273		-----		-----	
311	inh-117	470		0.53	
323	inh-307	405		-1.72	
347		-----		-----	
357	inh-052	435		-0.68	
497	in house	328	G(0.01)	-4.38	
613		-----		-----	
840	inh-0004	492		1.29	
864	D3362	456		0.04	
886		-----		-----	
902	D3362	445.7		-0.31	
913		-----		-----	
974	D3362	450		-0.16	
1429	D3362	430		-0.85	
1826	in house	470		0.53	
9027	INH-6001	441.3		-0.46	
9028	INH-1900	491	C	1.25	first reported 535.497
normality		OK			
n		13			
outliers		1			
mean (n)		454.75			
st.dev. (n)		25.116			
R(calc.)		70.33			
R(Horwitz)		81.10			

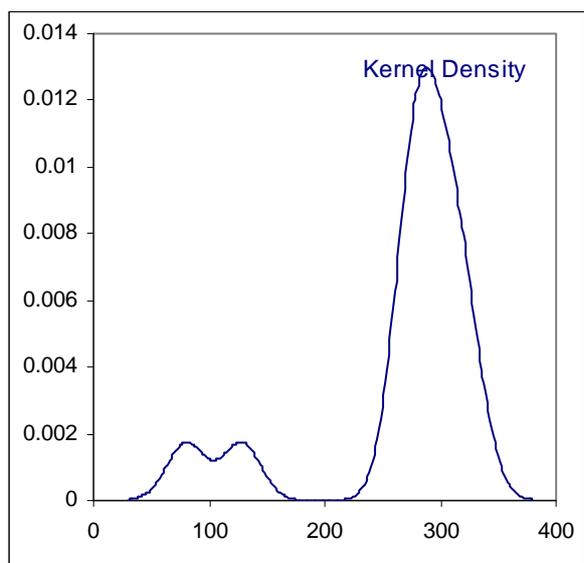
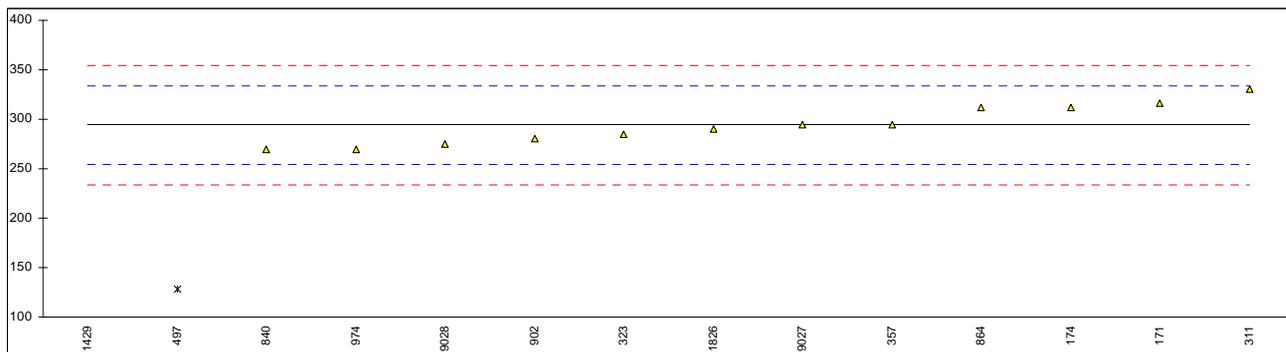


Determination of n-Butylmetacrylate on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362mod	3.4		----	
174	D3362	<10		----	
273		----		----	
311	inh-117	<10		----	
323	inh-307	<20		----	
347		----		----	
357	inh-052	<10		----	
497	in house	<10		----	
613		----		----	
840	inh-0004	6		----	
864	D3362	<10		----	
886		----		----	
902	D3362	<10		----	
913		----		----	
974		----		----	
1429		----		----	
1826	in house	<10		----	
9027		----		----	
9028	INH-1900	4.106		----	
	normality	not OK			
	n	3			
	outliers	0			
	mean (n)	4.502			
	st.dev. (n)	1.3445			
	R(calc.)	3.765			
	R(Horwitz)	(1.608)			

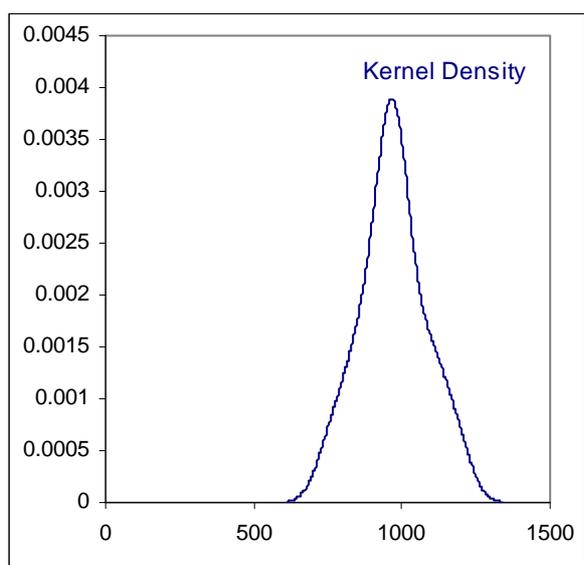
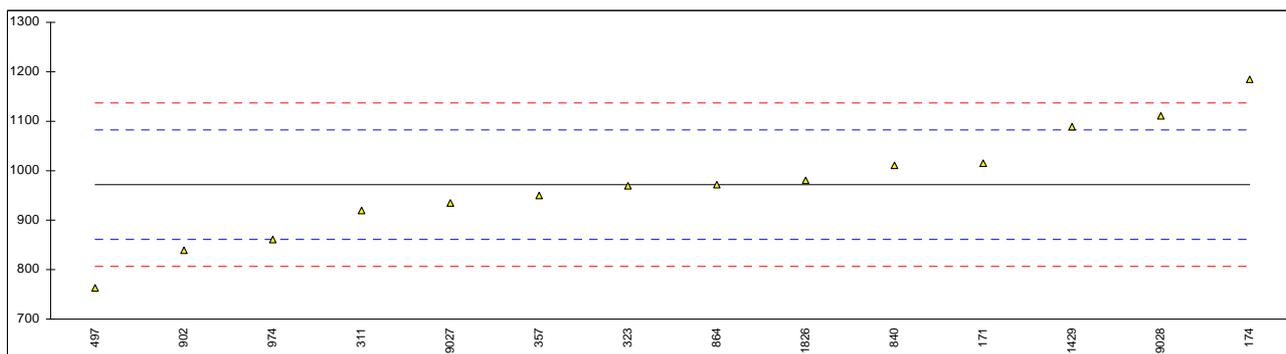
Determination of n-Butylpropionate on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362mod	316.1		1.10	
174	D3362	312		0.89	
273		-----		-----	
311	inh-117	330		1.79	
323	inh-307	285		-0.46	
347		-----		-----	
357	inh-052	295		0.04	
497	in house	128	G(0.01)	-8.31	
613		-----		-----	
840	inh-0004	270	C	-1.21	first reported 225
864	D3362	312		0.89	
886		-----		-----	
902	D3362	280.8		-0.67	
913		-----		-----	
974	D3362	270		-1.21	
1429	D3362	80	G(0.05)	-10.71	co-elutes with n-butanol
1826	in house	290		-0.21	
9027	INH-6001	294.1		0.00	
9028	INH-1900	275.178		-0.95	
	normality	OK			
	n	12			
	outliers	2			
	mean (n)	294.18			
	st.dev. (n)	19.565			
	R(calc.)	54.78			
	R(Horwitz)	56.02			



Determination of Di-n-Butylether on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362mod	1014.5		0.78	
174	D3362	1184		3.85	
273		-----		-----	
311	inh-118	920		-0.93	
323	inh-307	969		-0.04	
347		-----		-----	
357	inh-052	950		-0.39	
497	in house	763		-3.78	
613		-----		-----	
840	inh-0004	1011		0.72	
864	D3362	972		0.01	
886		-----		-----	
902	D3362	840	C	-2.38	first reported 813.6
913		-----		-----	
974	D3362	860		-2.02	
1429	D3362	1090		2.15	
1826	in house	980		0.16	
9027	INH-6001	935.8		-0.65	
9028	INH-1900	1110.889		2.53	
	normality	OK			
	n	14			
	outliers	0			
	mean (n)	971.44			
	st.dev. (n)	110.837			
	R(calc.)	310.34			
	R(Horwitz)	154.54			

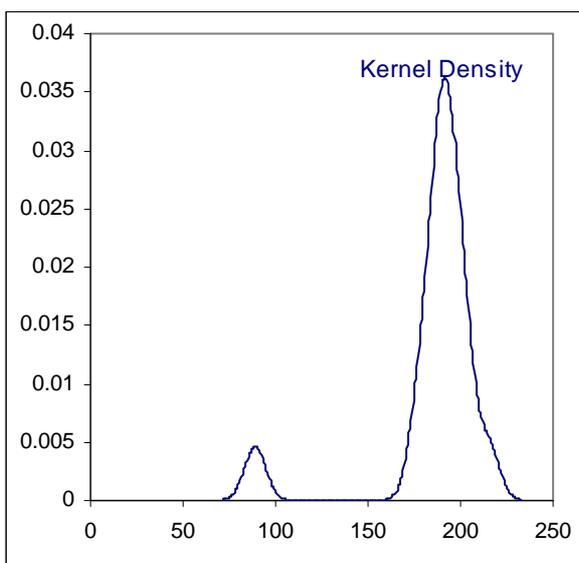
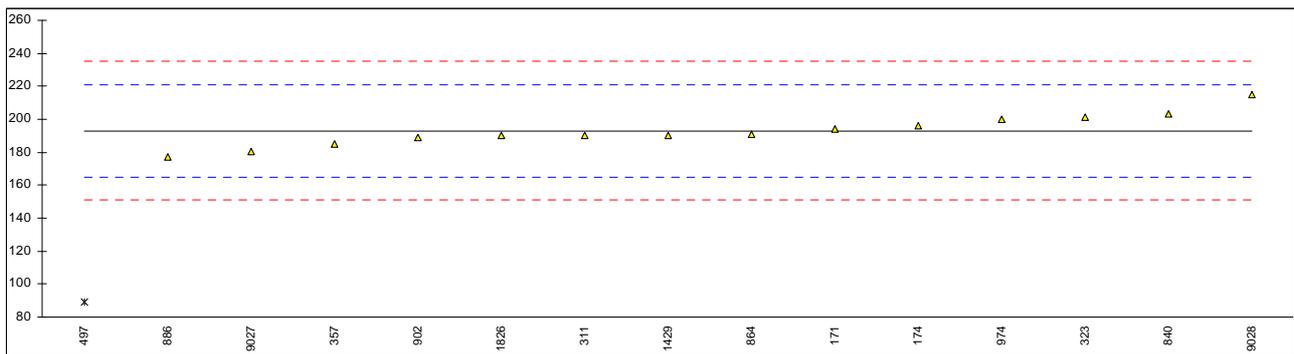


Determination of 2-Ethylhexylacrylate on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362mod	<1		----	
174	D3362	<10		----	
273		----		----	
311	inh-117	<10		----	
323	inh-307	<20		----	
347		----		----	
357	inh-052	<10		----	
497	in house	<10		----	
613		----		----	
840	inh-0004	<5		----	
864	D3362	<10		----	
886		----		----	
902	D3362	<10		----	
913		----		----	
974		----		----	
1429	D3362	30	C	----	first reported 40
1826	in house	<10		----	
9027		----		----	
9028		----		----	
	normality	n.a.			
	n	n.a.			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	n.a.			

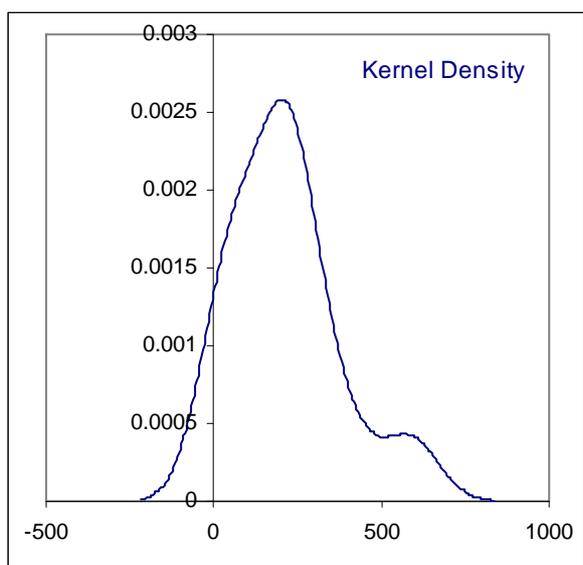
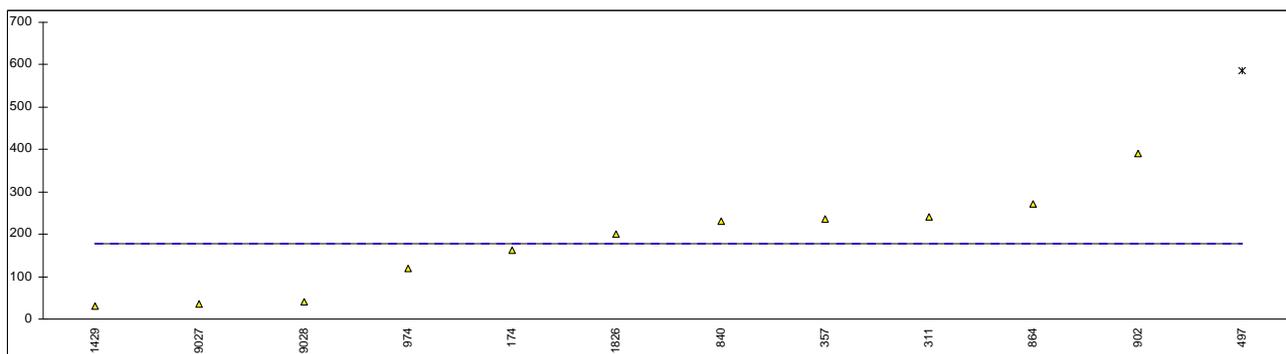
Determination of Isobutylacrylate on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3362mod	194.4		0.10	
174	D3362	196		0.21	
273		-----		-----	
311	inh-117	190		-0.22	
323	inh-307	201		0.57	
347		-----		-----	
357	inh-052	185		-0.57	
497	in house	89	G(0.01)	-7.44	
613		-----		-----	
840	inh-0004	203		0.71	
864	D3362	191		-0.14	
886	GC	177		-1.15	
902	D3362	189		-0.29	
913		-----		-----	
974	D3362	200		0.50	
1429	D3362	190		-0.22	
1826	in house	190		-0.22	
9027	INH-6001	180.6		-0.89	
9028	INH-1900	215.298		1.59	
normality		OK			
n		14			
outliers		1			
mean (n)		193.02			
st.dev. (n)		9.774			
R(calc.)		27.37			
R(Horwitz)		39.16			



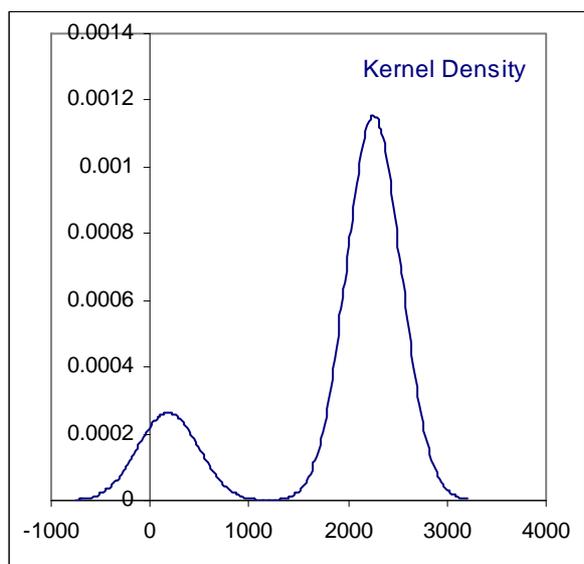
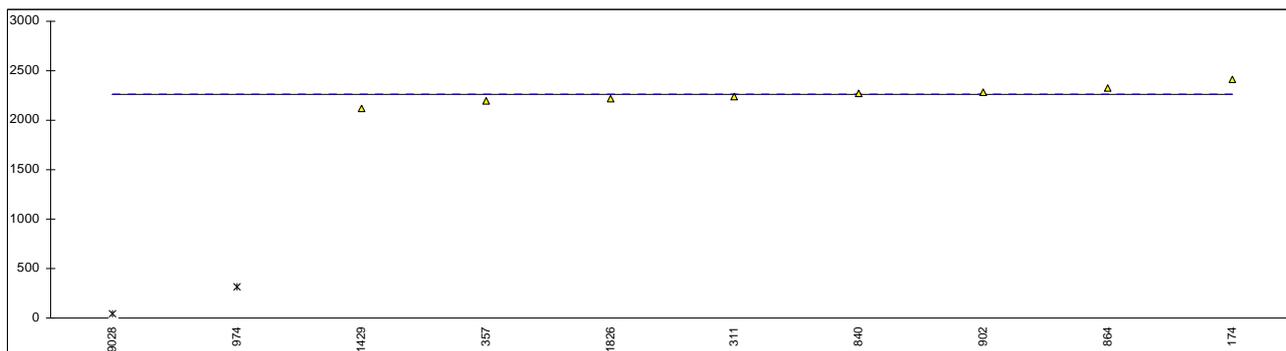
Determination of Other Impurities on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		----		----	
174	D3362	162		----	
273		----		----	
311	inh-117	240		----	
323		----		----	
347		----		----	
357	inh-052	235		----	
497	in house	587	G(0.05)	----	
613		----		----	
840	inh-0004	230		----	
864	D3362	272		----	
886		----		----	
902	D3362	390	C	----	first reported 460
913		----		----	
974		120		----	
1429	D3362	30		----	
1826	in house	200		----	
9027	INH-6001	34.9		----	
9028	INH-1900	40.908		----	
normality		OK			
n		11			
outliers		1			
mean (n)		177.71			
st.dev. (n)		113.515			
R(calc.)		317.84			
R(lit)		unknown			



Determination of Unknown Impurities on sample #1047; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		----		----	
174	D3362	2410		----	
273		----		----	
311	inh-117	2240		----	
323		----		----	
347		----		----	
357	inh-052	2200		----	
497		----		----	
613		----		----	
840	inh-0004	2273		----	
864	D3362	2330		----	
886		----		----	
902		2282		----	
913		----		----	
974		320	DG(0.01)	----	
1429		2120		----	
1826	in house	2220		----	
9027		----		----	
9028	INH-1900	40.908	DG(0.01)	----	
normality		OK			
n		8			
outliers		2			
mean (n)		2259.38			
st.dev. (n)		87.219			
R(calc.)		244.21			
R(lit)		unknown			



APPENDIX 2

Number of participants per country

1 lab in AUSTRALIA
1 lab in BELGIUM
1 lab in FINLAND
2 labs in GERMANY
1 lab in INDIA
1 lab in P.R. of CHINA
1 lab in SOUTH AFRICA
1 lab in SPAIN
1 lab in TAIWAN R.O.C.
2 labs in THE NETHERLANDS
1 lab in TURKEY
1 lab in U.A.E.
3 labs in U.S.A.
1 lab in UNITED KINGDOM
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
- 3 ASTM E1301-89
- 4 ISO 5725-86
- 5 ISO 5725. parts 1-6. 1994
- 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/>).