Results of Proficiency Test
n-Butylacrylate
June 2015

CONTENTS

Appendices:

1   INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies organizes a proficiency scheme for
n-Butylacrylate. During the annual proficiency testing program 2014/2015, it was decided to
organize again a round robin for the analysis of n-Butylacrylate.
In this interlaboratory study 15 laboratories in 14 different countries have participated. See
appendix 2 for the number of participants per country. In this report, the results of the 2015
proficiency test are presented and discussed. This report is also electronically available
through the iis internet site www.iisnl.com.

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 filled with
n-Butylacrylate. 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/IEC 17043:2010. This ensures strict adherence to protocols for
sample preparation and statistical evaluation and 100% confidentiality of participant's data.
Feedback from the participants on the reported data is encouraged and customer's
satisfaction is measured on a regular basis by sending out questionnaires.

2.2   PROTOCOL

The protocol followed in the 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 April 2014 (iis-protocol, version 3.3).
This protocol is electronically available through the iis internet site www.iisnl.com, from the
FAQ page.

2.3   CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary 20 litre bulk material for sample #15085 was obtained from a local supplier. After homogenisation in a precleaned can, 25 subsamples were transferred to brown glass bottles of 0.5L and labelled #15085. The homogeneity of the subsamples was checked by determination of Density in accordance with ASTM D4052 and Water in accordance with ASTM D1364 on 4 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in mg/kg
sample #15085-1	0.89899	78
sample #15085-2	0.89899	79
sample #15085-3	0.89899	72
sample #15085-4	0.89899	77

table 1: homogeneity test results of subsamples #15085

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 at 20°C in kg/L	Water in mg/kg
r (sample #15085)	0.00000	8.7
reference test method	ISO12185:96	ASTM D1364:07
0.3 x R(reference test)	0.00015	15.7

table 2: evaluation of homogeneity of subsamples #15085

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 #15085 was dispatched to each of the participating laboratories on May 27, 2015.

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 #15085: Acidity (free acid as Acrylic Acid), Appearance, Colour Pt/Co, Density at 20°C, Monomethyl Ether Hydroquinone (MEHQ), Purity as received, Purity on dry basis , a number of GC-impurities (n-Butanol, n-Butylacetate, n-Butylpropionate, Di-n-Butylether, Isobutylacrylate, Isobutylpropionate, other impurities and total impurities) and Water,.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The detailed report form was also made available for download on the iis website www.iisnl.com.

A SDS and a form to confirm receipt of the sample 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

The protocol followed in the organisation of this proficiency test is described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

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, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. 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.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test (ref. 15). Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2   GRAPHICS

In order to visualise 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 on the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected 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 method is for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.13 and 14). Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3   Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM 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.

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

The z-scores were calculated according to:

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

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

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

4   EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples for Brazil, Indonesia, Saudi Arabia and South Africa. Finally, in total 13 participants did report 117 numerical test results. Observed were 2 outlying test results, which is 1.7% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are normal.

4.1   EVALUATION PER TEST

In this section, the results are discussed per sample and test. The methods, which are used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3. When no suitable test method is available, the Horwitz equation was used.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D1613) and an added designation for the year that the method was adopted or revised (e.g. D1613:06). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1613:06(2012)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D1613:06 will be used.

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

Acidity: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D1613:06(2012).

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #15085, which is pass (= bright, clear and free of suspended matter).

Colour Pt/Co: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1209:05(2011).

Density at 20°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ISO12185:96.

MEHQ: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D3125:06(2012).

Purity as received: This determination was not problematic. No statistical outliers were detected. The reported purity results from two laboratories were excluded as the reported result for purity "as received" was larger than the reported result for purity " on dry basis", which is not possible. The calculated reproducibility after rejection of the suspect data is in good agreement with the requirements of ASTM D3362:05(2011), which was withdrawn with no replacement.

Purity on dry basis: This determination was not problematic. No statistical outliers were detected. The reported purity results from two laboratories were excluded as the reported result for purity "as received" was larger than the reported result for purity " on dry basis", which is not possible. The calculated reproducibility after rejection of the suspect data is in good agreement with the requirements of ASTM D3362:05(2011), which was withdrawn with no replacement.

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

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

n-Butylpropionate: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the calculated reproducibility estimated using the Horwitz equation.

Di-n-Butylether: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the calculated reproducibility estimated using the Horwitz equation.

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

Isobutylpropionate: No significant conclusions could be drawn as only one laboratory reported a result.

Other Impurities: The reported test results varied strongly: from 129 – 899.87 mg/kg. No significant conclusions were drawn. One laboratory probably did not identify isobutylacrylate.

Total Impurities: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the calculated reproducibility estimated using the Horwitz equation (5 comp.). After manual summation of all impurities by iis, it appears that only two laboratories reported all impurities (Total impurities reported = Total impurities calculated by iis). The other laboratories did not report all impurities detected.

Water: This determination was not problematic. No statistical outliers were detected. The calculated reproducibility is in full agreement with the requirements of ASTM D1364:07(2012).

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)
Acidity as Acrylic Acid	%M/M	11	0.0011	0.0009	0.0014
Appearance		12	Pass	n.a.	n.a.
Colour Pt/Co		8	3.7	4.4	7.0
Density at 20°C	kg/L	11	0.8990	0.0001	0.0005
MEHQ	mg/kg	12	14.9	1.5	2.2
Purity as received	%M/M	7	99.779	0.104	0.270
Purity on dry basis	%M/M	7	99.782	0.104	0.270
n-Butanol	mg/kg	7	171	17	35
n-Butylacetate	mg/kg	8	354	46	66
n-Butylpropionate	mg/kg	7	391	145	71
Di-n-Butylether	mg/kg	5	131	22	28
Isobutylacrylate	mg/kg	6	897	79	144
Isobutylpropionate	mg/kg	1	n.a.	n.a.	n.a.
Total impurities	mg/kg	7	2289	440	716
Water	mg/kg	11	106	59	62

table 3: reproducibilities of results of sample #15085

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 JUNE 2015 WITH THE PREVIOUS PTS

	June 2015	May 2012	April 2010	April 2008
Number of reporting labs	13	14	17	17
Number of results reported	117	138	202	140
Statistical outliers	2	5	19	5
Percentage outliers	1.7%	3.6%	9.4%	3.6%

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	June 2015	May 2012	April 2010	April 2008
Free Acid as Acrylic Acid	++	++	-	+
Colour Pt/Co	++	++	++	+
Density at 20°C	++	++	++	++
MEHQ	++	++	+/-	++
Purity as received	++	++	++	++
Purity on dry basis	++	++	++	++
n-Butanol	++	+/-	--	+
n-Butylacetate	++	++	+	+
n-Butylpropionate	--	+/-	+/-	+/-
Di-n-Butylether	+	++	--	+
Isobutylacrylate	++	++	++	++
Isobutylpropionate	n.e.	n.e.	n.e.	n.e.
Total impurities	++	n.e.	n.e.	n.e.
Water	+/-	++	--	++

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 #15085; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	D1613	0.001169		0.14	
174	D1613	0.00122		0.25	
273	D1613	0.0014		0.61	
311	D1613	0.0013		0.41	
323	D1613	0.0009		-0.39	
357	D1613	0.0010		-0.19	
522		-----		-----	
551		-----		-----	
621	D1613	0.000419		-1.36	
633	D1613	0.00173		1.27	
663	D1613	0.0024	G(0.05)	2.61	
840	D1613	0.00106		-0.07	
886	D1613	0.0010		-0.19	
902	D1613	0.00087		-0.45	
1685		-----		-----	

normality	suspect
n	11
outliers	1
mean (n)	0.00110
st.dev. (n)	0.000335
R(calc.)	0.00094
R(D1613:06)	0.00140

Kernel Density
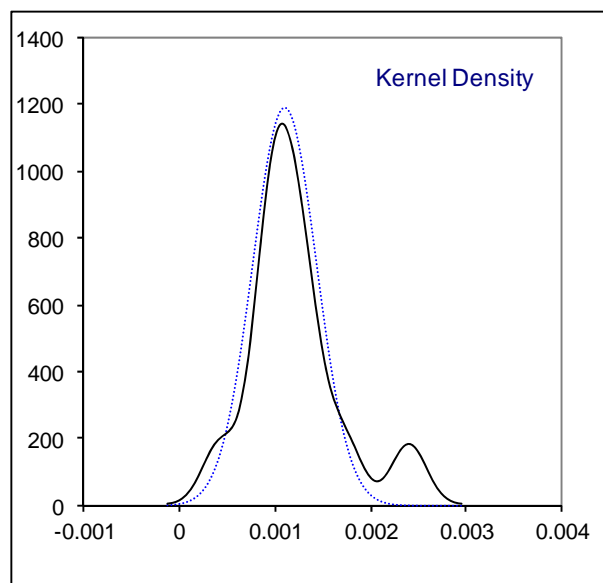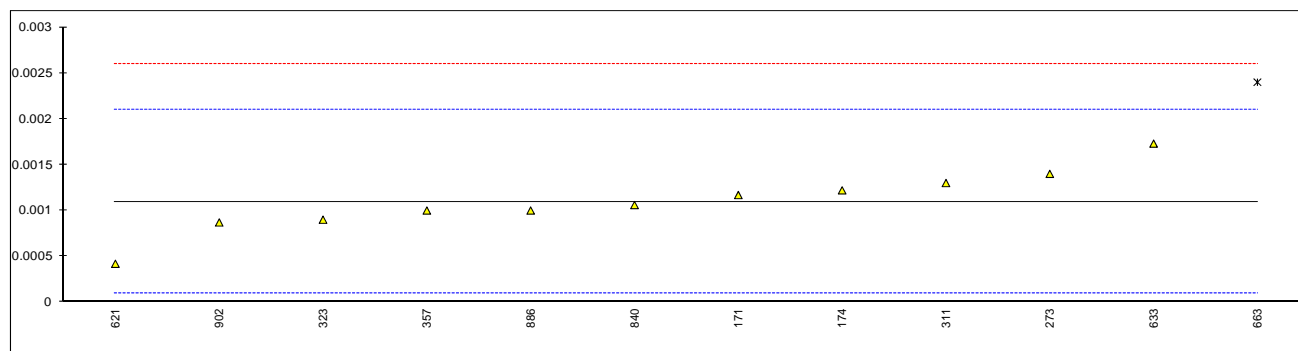
Determination of Appearance on sample #15085;

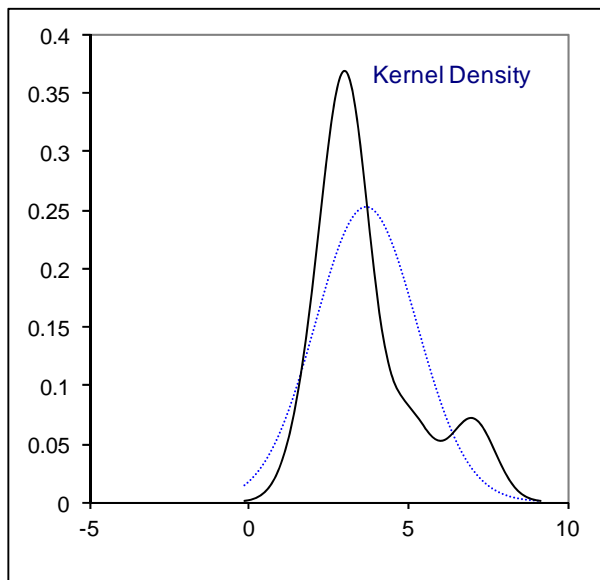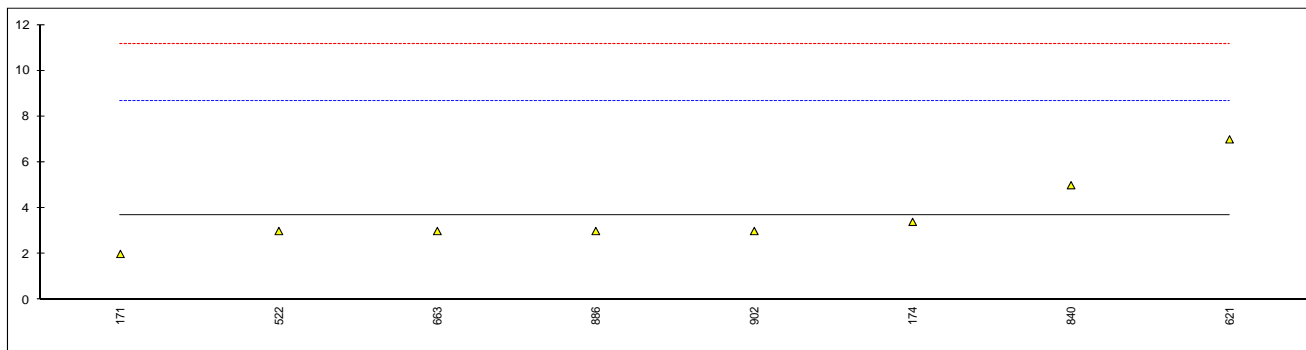| lab	method	value	mark	z(targ)	remarks
171	E2680	Pass		-----	
174	E2680	Pass		-----	
273	E2680	Pass		-----	
311	E2680	Pass		-----	
323	E2680	CFFSM		-----	
357	E2680	Pass		-----	
522	Visual	Pass		-----	
551		-----		-----	
621	Visual	C&B		-----	
633	Visual	C&B		-----	
663	Visual	Pass		-----	
840	E2680	Pass		-----	
886		-----		-----	
902	E2680	Pass		-----	
1685		-----		-----	

CFFSM　　= Clear and free from suspended matter
C&B　　　= Bright and clear

Determination of Colour Pt/Co on sample #15085;

lab	method	value	mark	z(targ)	remarks
171	D1209	2		-0.67	
174	D1209	3.4		-0.11	
273	D1209	<5		-----	
311	D1209	<5		-----	
323	D1209	<5		-----	
357	D1209	<5		-----	
522	D1209	3		-0.27	
551		-----		-----	
621	D1209	7		1.33	
633		-----		-----	
663	D1209	3		-0.27	
840	D1209	5		0.53	
886	D1209	3		-0.27	
902	D5386	3		-0.27	
1685		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	3.67
st.dev. (n)	1.582
R(calc.)	4.43
R(D1209:05)	7.00

Determination of Density at 20°C on sample #15085; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.89907		0.33	
174	D4052	0.8990		-0.06	
273	D4052	0.8990		-0.06	
311	D4052	0.8990		-0.06	
323	D4052	0.8990		-0.06	
357	ISO12185	0.89899		-0.12	
522	D4052	0.8991		0.50	
551		-----		-----	
621	D4052	0.8990	C	-0.06	Reported 899.0 kg/m3
633	D4052	0.8993	G(0.01)	1.62	
663	D4052	0.8990		-0.06	
840	D4052	0.89896		-0.29	
886		-----		-----	
902	D4052	0.8990		-0.06	
1685		-----		-----	

normality	not OK
n	11
outliers	1
mean (n)	0.89901
st.dev. (n)	0.000039
R(calc.)	0.00011
R(ISO12185:96)	0.00050

Kernel Density
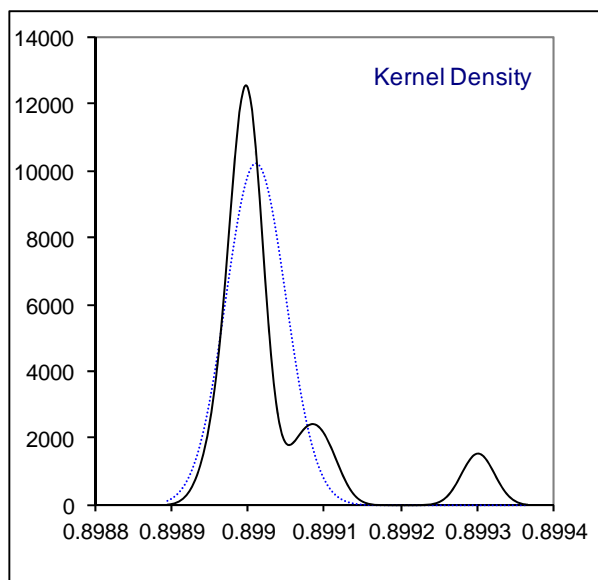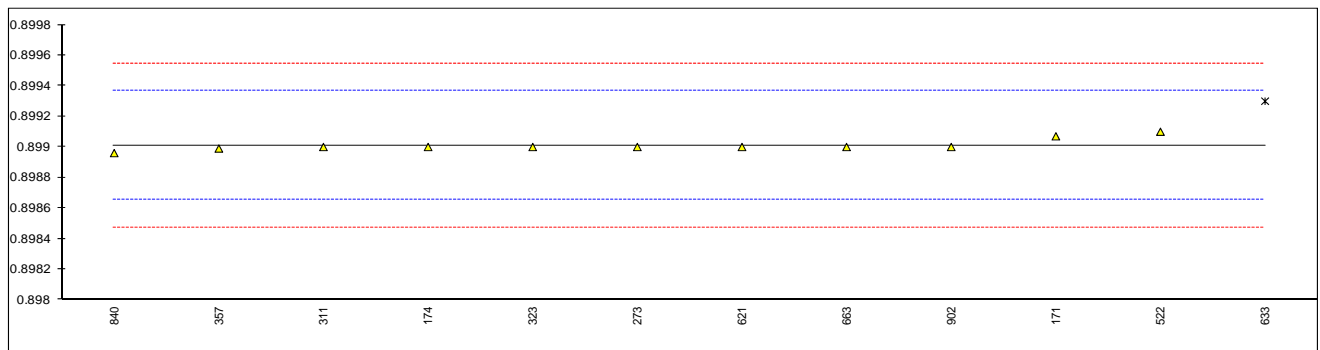
Determination of MEHQ on sample #15085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D3125	14.2		-0.85	
174	D3125	15.9		1.28	
273	D3125	14.7		-0.22	
311	D3125	15.2		0.41	
323	D3125	14.1		-0.98	
357	D3125	15.0		0.15	
522	D3125	15.2		0.41	
551		-----		-----	
621		-----		-----	
633	D3125	14.22		-0.82	
663	D3125	14.865		-0.02	
840	D3125	14.74		-0.17	
886	D3125	15.6		0.91	
902	D3125	14.8		-0.10	
1685		-----		-----	

normality	OK
n	12
outliers	0
mean (n)	14.88
st.dev. (n)	0.551
R(calc.)	1.54
R(D3125:06)	2.23

Kernel Density
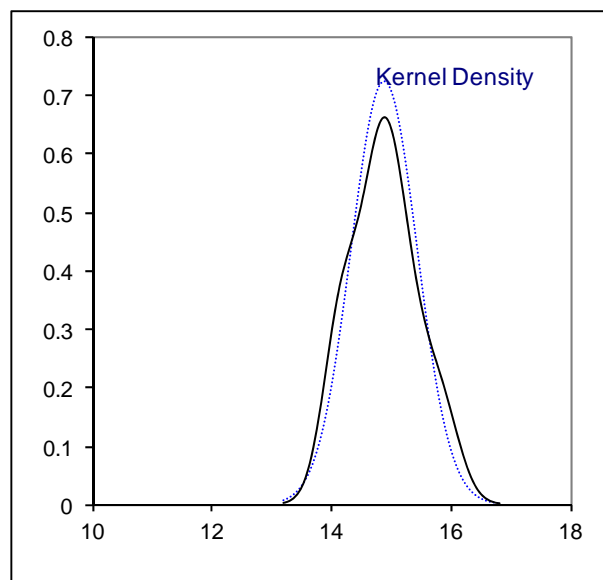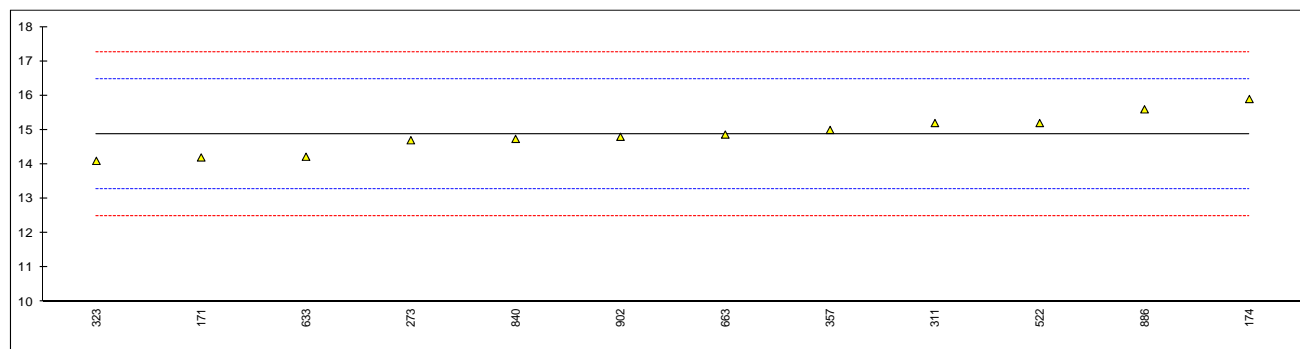
Determination of Purity as received on sample #15085; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3362	99.787		0.08	
273		-----		-----	
311		-----		-----	
323	D3362	99.76		-0.20	
357	D3362	99.751		-0.29	
522	D3362	99.80		0.22	
551		-----		-----	
621	D3362	99.62	ex	-1.65	Result excluded as result "on dry basis" < result "as received".
633	INH-007	99.8045	ex	0.27	Result excluded as result "on dry basis" < result "as received".
663		99.85		0.74	
840	INH-004	99.744		-0.36	
886		-----		-----	
902	D3362	99.76		-0.20	
1685		-----		-----	

normality	not OK
n	7
outliers	0 (+2 excl)
mean (n)	99.7789
st.dev. (n)	0.03714
R(calc.)	0.1040
R(D3362:05)	0.2700
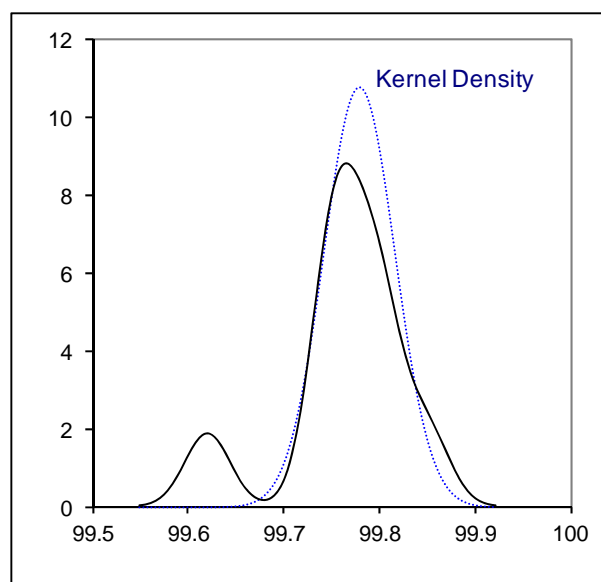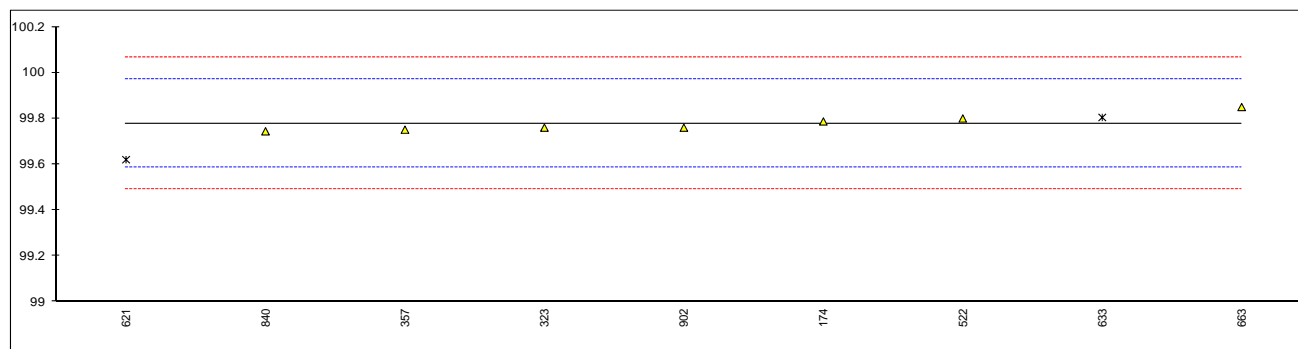
Determination of Purity on dry basis by GC on sample #15085; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3362	99.80		0.18	
273		-----		-----	
311	INH-117	99.76		-0.23	
323	INH-307	99.77		-0.13	
357	D3362	99.759		-0.24	
522		-----		-----	
551		-----		-----	
621	D3362	99.61	ex	-1.79	Result excluded as result "on dry basis" < result "as received".
633	INH-007	99.7894	ex	0.07	Result excluded as result "on dry basis" < result "as received".
663		99.86		0.81	
840	INH-004	99.757		-0.26	
886		-----		-----	
902	D3362	99.77		-0.13	
1685		-----		-----	

normality	not OK
n	7
outliers	0 (+ 2 excl)
mean (n)	99.7823
st.dev. (n)	0.03727
R(calc.)	0.1044
R(D3362:05)	0.2700
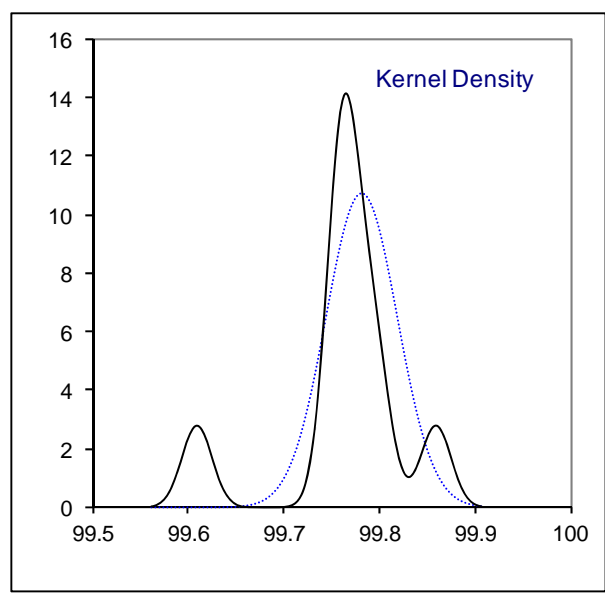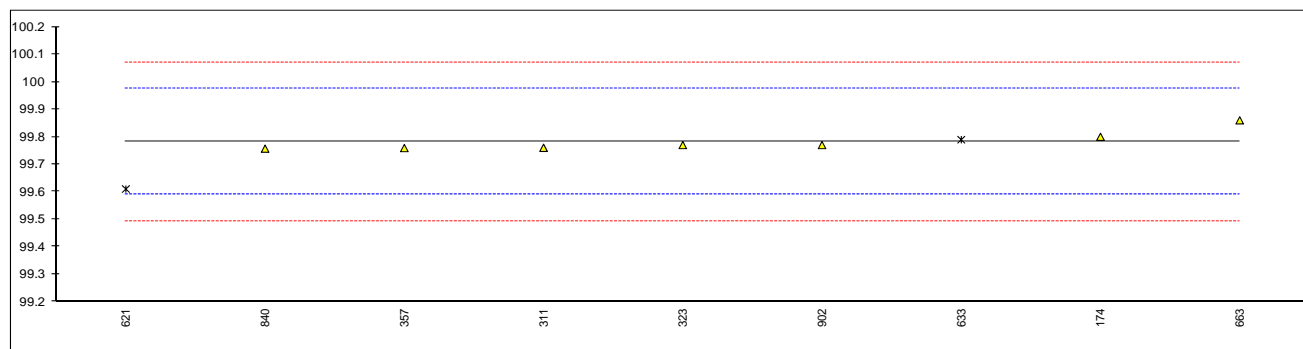
Determination of n-Butanol on sample #15085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3362	170	C	-0.11	First reported 17
273		-----		-----	
311	INH-117	170		-0.11	
323	INH-307	179		0.60	
357	D3362	180		0.68	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633	INH-007	169.976		-0.11	
663		-----		-----	
840	INH-004	165.5		-0.46	
886		-----		-----	
902	D3362	165		-0.50	
1685		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	171.4
st.dev. (n)	5.96
R(calc.)	16.7
R(Horwitz)	35.4
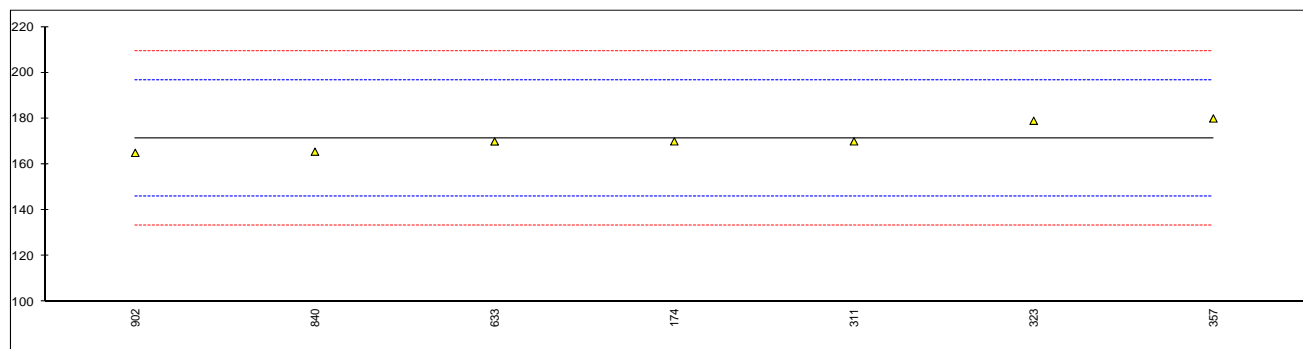
Determination of n-Butylacetate on sample #15085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3362	369		0.65	
273		-----		-----	
311	INH-117	370		0.69	
323	INH-307	344		-0.42	
357	D3362	360		0.26	
522		-----		-----	
551		-----		-----	
621	D3362	359	C	0.22	Reported 0.0359 %M/M
633	INH-007	319.955		-1.45	
663		-----		-----	
840	INH-004	360.2		0.27	
886		-----		-----	
902	D3362	349		-0.21	
1685		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	353.9
st.dev. (n)	16.32
R(calc.)	45.7
R(Horwitz)	65.5

Kernel Density
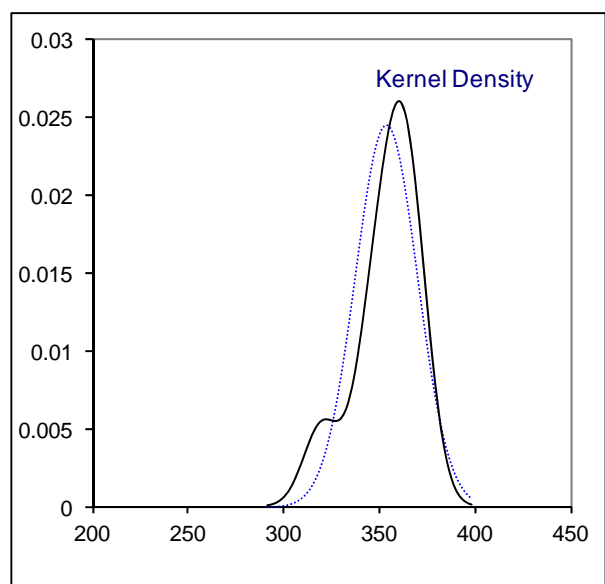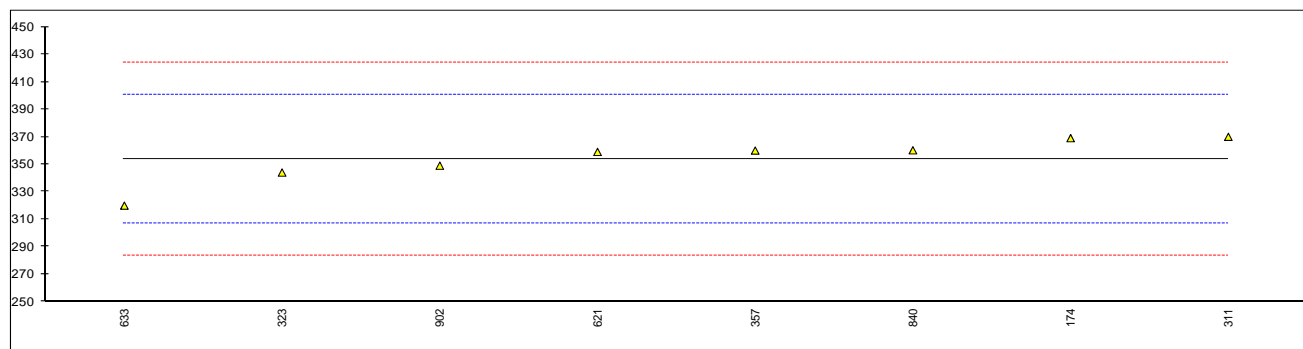
Determination of n-Butylpropionate on sample #15085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3362	291	C	-3.92	First reported 253
273		-----		-----	
311	INH-117	460		2.71	
323	INH-307	386		-0.19	
357	D3362	410		0.75	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633	INH-007	419.941		1.14	
663		-----		-----	
840	INH-004	391.1		0.01	
886		-----		-----	
902	D3362	378		-0.51	
1685		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	390.9
st.dev. (n)	51.89
R(calc.)	145.3
R(Horwitz)	71.3
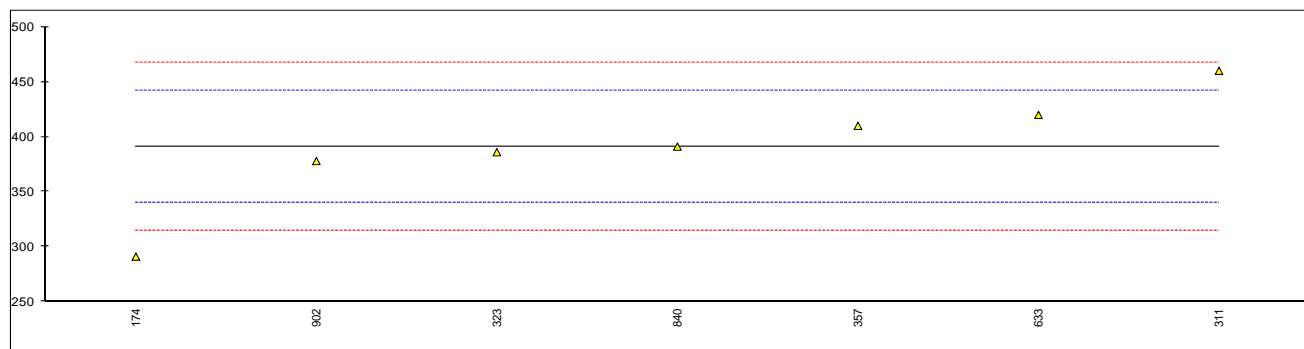
Determination of Di-n-Butylether on sample #15085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3362	143		1.24	
273		-----		-----	
311	INH-118	130		-0.05	
323	INH-307	129		-0.15	
357	D3362	130		-0.05	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
840	INH-004	120.6		-0.99	
886		-----		-----	
902		-----		-----	
1685		-----		-----	

normality	unknown
n	5
outliers	0
mean (n)	130.5
st.dev. (n)	8.02
R(calc.)	22.4
R(Horwitz)	28.1
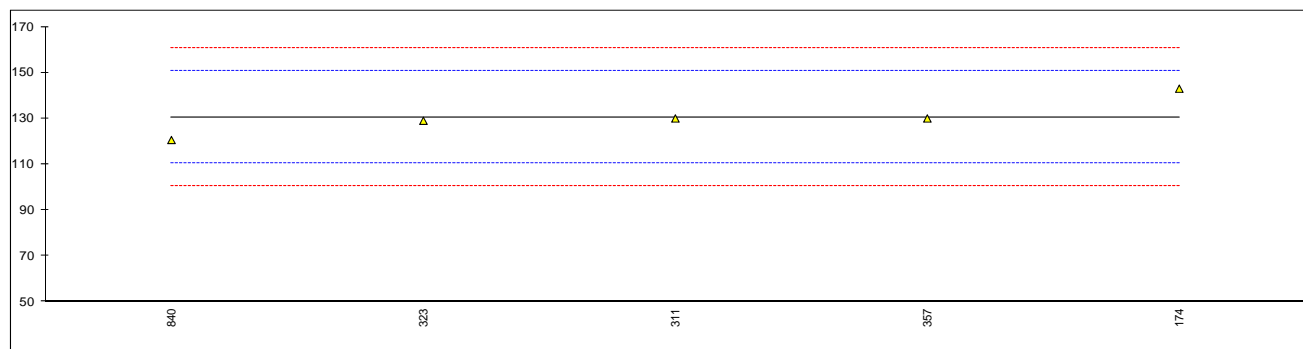
Determination of Isobutylacrylate on sample #15085; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
171		-----		-----	
174	D3362	946		0.95	
273		-----		-----	
311	INH-117	870		-0.52	
323	INH-307	890		-0.13	
357	D3362	880		-0.33	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	See other impurities
840	INH-004	912.9		0.31	
886		-----		-----	
902	D3362	882		-0.29	
1685		-----		-----	

normality	unknown
n	6
outliers	0
mean (n)	896.8
st.dev. (n)	28.09
R(calc.)	78.7
R(Horwitz)	144.4
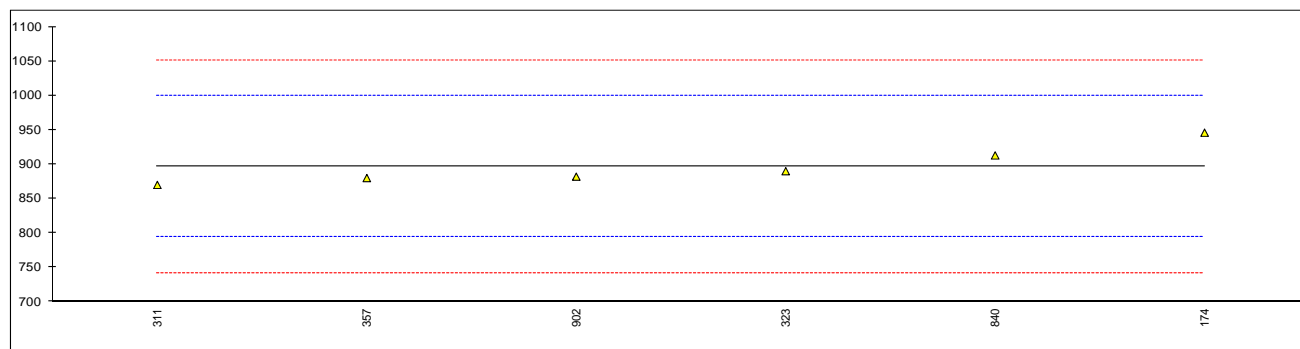
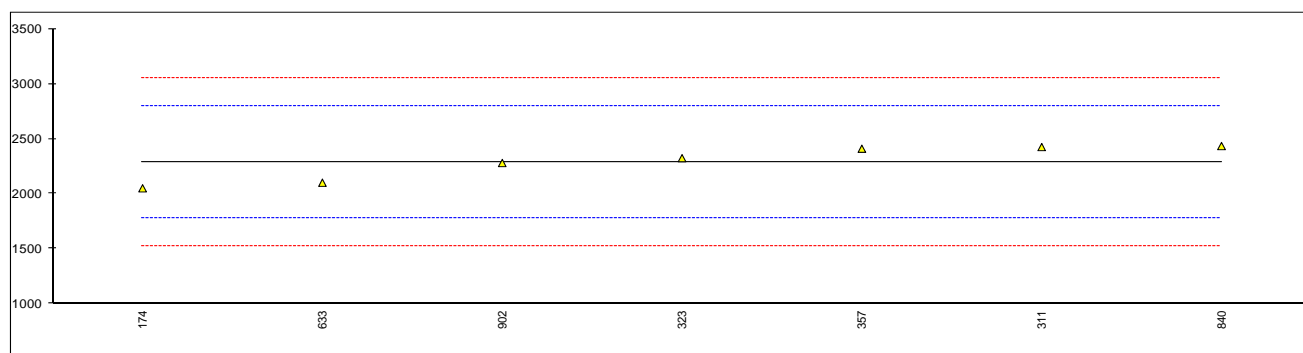Determination of Isobutylpropionate on sample #15085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174		-----		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
357		-----		-----	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
840		-----		-----	
886		-----		-----	
902	D3362	37		-----	
1685		-----		-----	

normality	n.a.	
n	1	
outliers	n.a.	
mean (n)	n.a.	
st.dev. (n)	n.a.	
R(calc.)	n.a.	
R(lit)	n.a.	

Determination of Other Impurities on sample #15085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3362	129		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
357		-----		-----	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633	INH-007	899.873		-----	Probably isobutylacrylate?
663		-----		-----	
840		-----		-----	
886		-----		-----	
902	D3362	469		-----	
1685		-----		-----	

normality	n.a.
n	3
outliers	n.a.
mean (n)	499.3
st.dev. (n)	n.a.
R(calc.)	n.a.
R(Horwitz)	n.a.

Determination of Total Impurities on sample #15085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3362	2050	C	-0.94	First reported 1857
273		-----		-----	
311	INH-117	2425		0.53	
323	INH-307	2324		0.14	
357	D3362	2410		0.47	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633	INH-007	2100.524		-0.74	
663		-----		-----	
840	INH-004	2434.1		0.57	
886		-----		-----	
902	D3362	2280		-0.04	
1685		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	2289.1
st.dev. (n)	157.13
R(calc.)	440.0
R(Horwitz -5 comp)	715.7

Determination of Water, titrimetric on sample #15085; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D1364	115		0.39	
273	E203	140	C	1.53	First reported 0.014 mg/kg
311	D1364	80		-1.19	
323	D1364	84		-1.01	
357	E1064	76		-1.37	
522	E203	101		-0.24	
551		-----		-----	
621	D6304	124.46		0.82	
633	E1064	120		0.62	
663	E1064	90		-0.74	
840	E1064	114.6		0.38	
886		-----		-----	
902	D1364	124.0		0.80	
1685		-----		-----	

normality	OK	
n	11	
outliers	0	
mean (n)	106.28	
st.dev. (n)	21.241	
R(calc.)	59.48	
R(D1364:07)	61.85	

Kernel Density
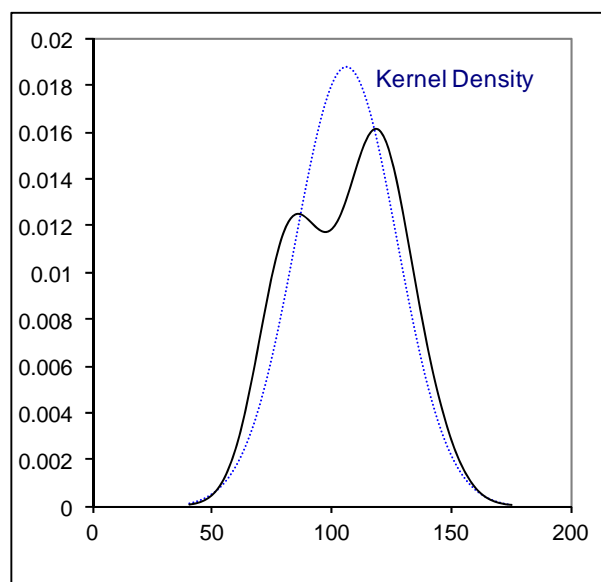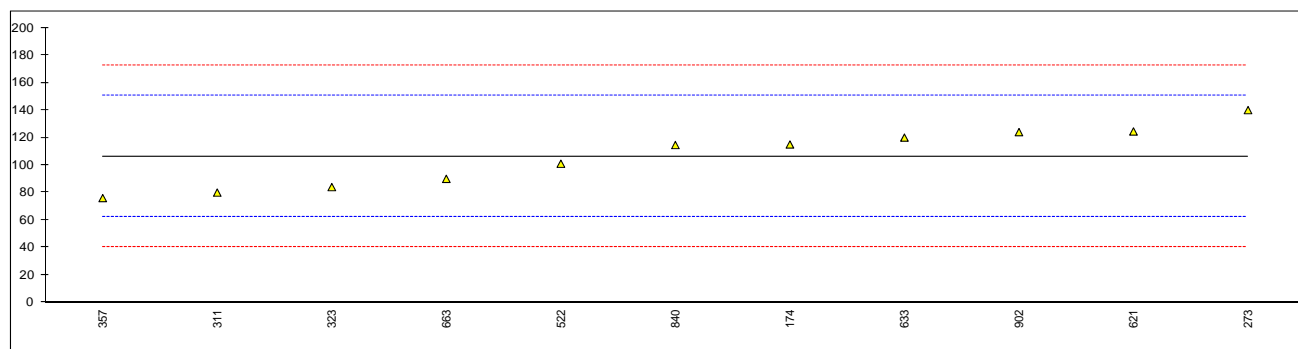
APPENDIX 2

Number of participants per country

1 lab in BELGIUM

1 lab in BRAZIL

1 lab in FINLAND

1 lab in INDONESIA

1 lab in MEXICO

1 lab in NETHERLANDS

1 lab in PHILIPPINES

1 lab in SAUDI ARABIA

1 lab in SOUTH AFRICA

1 lab in TAIWAN R.O.C.

1 lab in THAILAND

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
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
E	= error in calculations
ex	= excluded from calculations
n.e.	= not evaluated
wd	= withdrawn method

Literature:

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2   ASTM E178:89
3   ASTM E1301:89
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5   ISO 5725, parts 1-6, 1994
6   ISO13528-05
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8   W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9   IP 367:84
10   DIN 38402 T41/42
11   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12   J.N. Miller, Analyst, 118, 455, (1993)
13   Analytical Methods Committee Technical brief, No4 January 2001
14   The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson
15   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), pp. 165-172, (1983)