Results of Proficiency Test
n-Butylacrylate
April 2017

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies organizes a proficiency scheme for n-Butylacrylate. During the annual proficiency testing program 2016/2017, it was decided to organize again a round robin for the analysis of n-Butylacrylate.
In this interlaboratory study 20 laboratories in 16 different countries registered, see appendix 2 for the number of participants per country. In this report the results of the 2017 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one bottle of 500 ml filled with n-Butylacrylate, labelled #17062 .
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/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 March 2017 (iis-protocol, version 3.4).
This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary 20 litre bulk material for sample #17062 was obtained from a local supplier. After homogenisation in a precleaned can, 35 subsamples were transferred to brown glass bottles of 0.5L and labelled #17062. The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ASTM D4052 and Water content in accordance with ASTM D1364 on 4 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in mg/kg
sample #17062-1	0.89895	200
sample #17062-2	0.89895	210
sample #17062-3	0.89894	200
sample #17062-4	0.89895	200

table 1: homogeneity test results of subsamples #17062

From the above test results the repeatabilities were calculated and compared to 0.3 times the corresponding reproducibilities of the reference test methods 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 (observed)	0.00001	14.0
reference test method	ISO12185:96	ASTM D1364:02(2012)
0.3 x R(reference test method)	0.00015	25.6

table 2: evaluation of the repeatability of subsamples #17062

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

One 0.5L bottle, labelled #17062 was dispatched to each of the participating laboratories on April 5, 2017.

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 #17062: Acidity (as Acrylic Acid), Appearance, Colour Pt/Co, Density at 20°C, Inhibitor as monomethyl Ether of Hydroquinone (MEHQ), Purity by GC as received, Purity by GC on dry basis , a number of GC-impurities (n-Butanol, n-Butylacetate, n-Butylpropionate, di-n-Butylether, iso-Butylacrylate, iso-Butylpropionate, other impurities and total impurities) and Water.

It was explicitly requested to treat the samples as if they were routine samples. Therefore, each laboratory is advised to perform only those analyses that normally are done in daily routine (but the laboratories are allowed to do all analyses). Furthermore, it was requested to report the test results using the indicated units on the report form and not to round the test results more, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the

rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

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

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

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. 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 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 test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM or IP reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other targets values were used. In some cases a reproducibility based on former iis proficiency tests could be used.

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

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
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. Laboratories in Brazil, Mexico, Saudi Arabia and Indonesia received the samples late or not at all due to several problems (i.e.customs clearance). Not all laboratories were able to report all analyses requested.
Finally, in total 16 participants did report 160 numerical test results. Observed were 6 outlying test results, which is 3.8% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are normal.

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

4.1 EVALUATION PER TEST

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

Unfortunately, a suitable reference test method providing the precision data is not available for all determinations. For the tests that have no available precision data the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

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

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 requirement of ASTM D1613:17.

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #17062, which is pass (= bright, clear and free of suspended matter).

Colour Pt/Co: 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 requirement 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 requirement of ISO12185:96.

MEHQ: This determination may be problematic. No statistical outliers were observed and the calculated reproducibility is not, but almost in agreement with the requirement of ASTM D3125:06(2012).

Purity as received and on dry basis: These determinations were not problematic. No statistical outliers were observed. The reported purities test results from three laboratories were marked with an “E” as the reported test result for purity “as received” was larger than the reported test result for purity “on dry basis”, which is not possible. Presumable the test results were mixed-up? However, it was decided not to exclude these test results as the difference between the two purities is relatively small and the number of test results low.
The calculated reproducibilities are both in good agreement with the requirements of ASTM D3362:05(2011), which test method was withdrawn with no replacement.

n-Butanol: This determination may be problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not in agreement with the reproducibility estimated using the Horwitz equation.

n-Butylacetate: 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 reproducibility estimated using the Horwitz equation.

n-Butylpropionate: 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 reproducibility estimated using the Horwitz equation.

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

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

Isobutylpropionate: No significant conclusions could be drawn as only two laboratories reported a test result.

Other Impurities: The reported test results varied strongly: from 114 – 491 mg/kg. No significant conclusions were drawn.

Total Impurities: 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 (5 components).

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirement of ASTM D1364:02(2012).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of the participating laboratories. The target reproducibilities derived from the reference test methods (in casu ASTM test methods) or the estimated reproducibility calculated using the Horwitz equation and the calculated reproducibilities (2.8 * sd) of the samples (see appendix 1) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acrylic Acid	mg/kg	12	23.3	11.0	14
Appearance		14	Pass	n.a.	n.a.
Colour Pt/Co		11	4.2	3.4	7
Density at 20°C	kg/L	15	0.8990	0.0002	0.0005
Inhibitor as MEHQ	mg/kg	14	15.2	2.8	2.3
Purity as received	%M/M	11	99.770	0.109	0.27
Purity on dry basis	%M/M	11	99.778	0.077	0.27
n-Butanol	mg/kg	10	115	42	25
n-Butylacetate	mg/kg	11	272	33	52
n-Butylpropionate	mg/kg	10	341	39	64
di-n-Butylether	mg/kg	11	719	185	120
iso-Butylacrylate	mg/kg	9	445	39	80
iso-Butylpropionate	mg/kg	2	n.a.	n.a.	n.a.
Total impurities	mg/kg	8	2182	816	687
Water	mg/kg	15	217	93	88

table 3: reproducibilities of test results of sample #17062

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 reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2017 WITH THE PREVIOUS PTS

	April 2017	June 2015	May 2012	April 2010	April 2008
Number of reporting labs	16	13	14	17	17
Number of results reported	160	117	138	202	140
Statistical outliers	6	2	5	19	5
Percentage outliers	3.8%	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 reference test methods. The conclusions are given the following table:

Determination	April 2017	June 2015	May 2012	April 2010	April 2008
Acidity as Acrylic Acid	+	++	++	-	+
Colour Pt/Co	++	++	++	++	+
Density at 20°C	++	++	++	++	++
Inhibitor as MEHQ	-	++	++	+/-	++
Purity as received	++	++	++	++	++
Purity on dry basis	++	++	++	++	++
n-Butanol	-	++	+/-	--	+
n-Butylacetate	+	++	++	+	+
n-Butylpropionate	+	--	+/-	+/-	+/-
di-n-Butylether	-	+	++	--	+
iso-Butylacrylate	++	++	++	++	++
iso-Butylpropionate	n.e.	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 reference test methods is listed in the above table. The following performance categories were used:

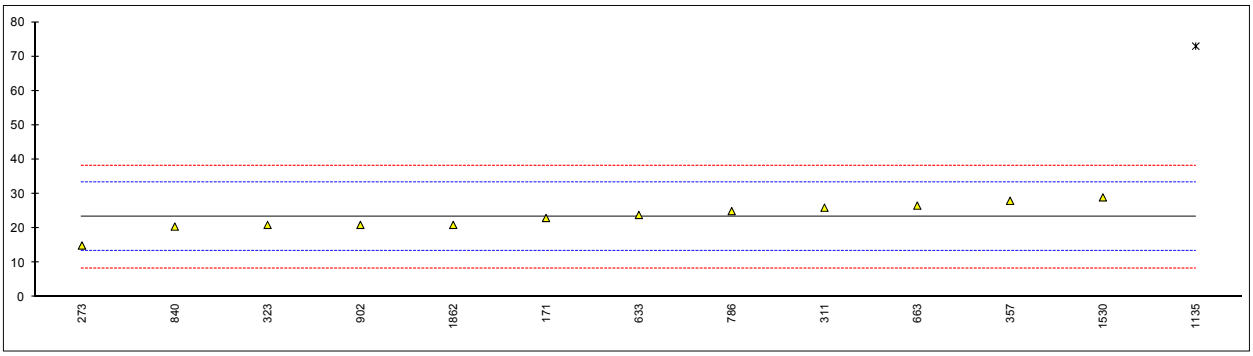
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

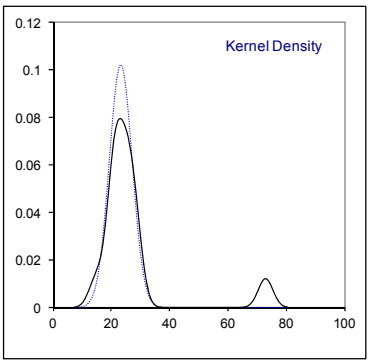
APPENDIX 1

Determination of Acidity, as Acrylic Acid on sample #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171	D1613	23		-0.07	
174		-----		-----	
273	D1613	15	C	-1.67	first reported: 51
311	D1613	26		0.53	
323	D1613	21		-0.47	
357	D1613	28		0.93	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633	D1613	23.885		0.11	
663	D1613	26.6		0.65	
786	D1613	25		0.33	
840	D1613	20.5		-0.57	
886		-----		-----	
902	D1613	21		-0.47	
963		-----		-----	
1135	D1613	73	C,G(0.01)	9.93	first reported: 61
1530	D1613	29		1.13	
1862	D1613	21		-0.47	

normality	OK
n	12
outliers	1
mean (n)	23.332
st.dev. (n)	3.9158
R(calc.)	10.964
R(D1613:17)	14





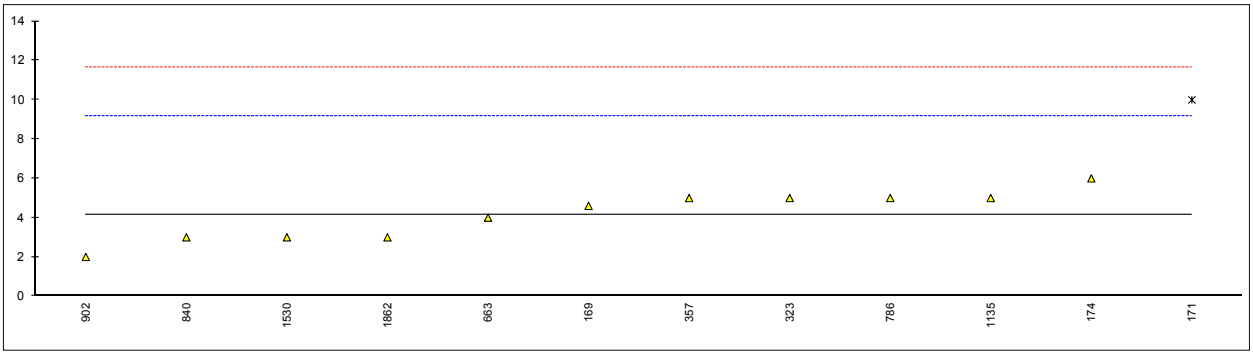
Determination of Appearance on sample #17062;

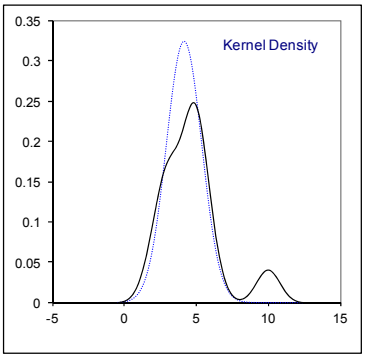
lab	method	value	mark	z(targ)	remarks
169	Visual	BC&FSM		-----	
171	E2680	pass		-----	
174	E2680	PASS		-----	
273	E2680	Pass		-----	
311	E2680	pass		-----	
323	E2680	clear & bright		-----	
357	E2680	Pass		-----	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633	Visual	Clear and Bright		-----	
663	Visual	Pass		-----	
786		-----		-----	
840	E2680	Pass		-----	
886		-----		-----	
902	E2680	PASS		-----	
963		-----		-----	
1135	Visual	Clear		-----	
1530	Visual	c&b		-----	
1862	Visual	Pass		-----	
	normality	n.a.			
	n	14			
	outliers	0			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit.)	n.a.			

Determination of Colour Pt/Co on sample #17062;

lab	method	value	mark	z(targ)	remarks
169	D5386	4.6		0.18	
171	D1209	10	G(0.05)	2.34	
174	D1209	6		0.74	
273	D1209	<5		-----	
311	D1209	<5		-----	
323	D1209	5		0.34	
357	D1209	5		0.34	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633	D1209	<0		-----	
663	D1209	4		-0.06	
786	D1209	5		0.34	
840	D1209	3		-0.46	
886	D1209	<5		-----	
902	D5386	2		-0.86	
963		-----		-----	
1135	D1209	5		0.34	
1530	D1209	3		-0.46	
1862	D1209	3		-0.46	

normality	OK
n	11
outliers	1
mean (n)	4.15
st.dev. (n)	1.230
R(calc.)	3.44
R(D1209:05)	7

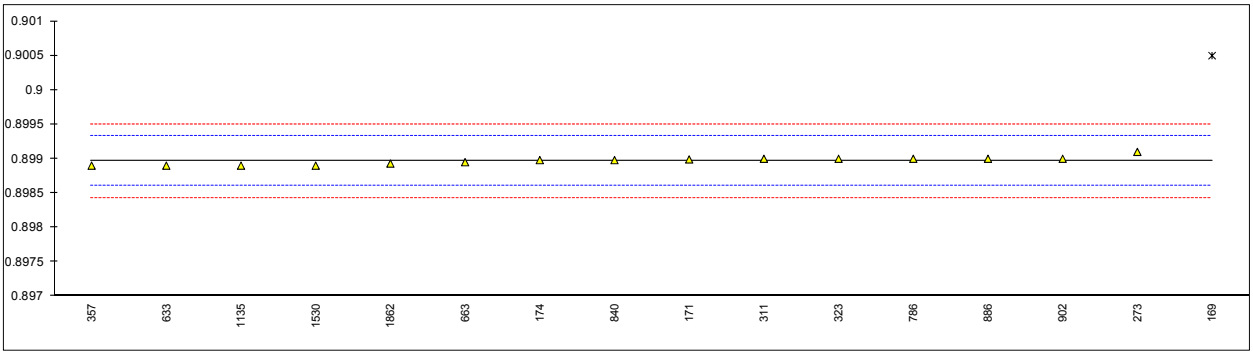


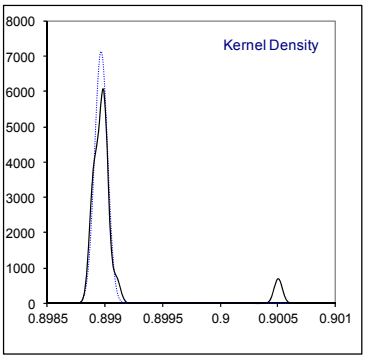


Determination of Density at 20°C on sample #17062; results in kg/L

lab	method	value	mark	z(targ)	remarks
169	D4052	0.9005	G(0.01)	8.57	
171	D4052	0.89899		0.12	
174	D4052	0.89898		0.06	
273	D4052	0.8991		0.73	
311	D4052	0.8990		0.17	
323	D4052	0.8990		0.17	
357	D4052	0.89890		-0.39	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633	D4052	0.8989		-0.39	
663	D4052	0.89895		-0.11	
786	D4052	0.8990		0.17	
840	D4052	0.89898		0.06	
886	D4052	0.8990		0.17	
902	D4052	0.8990		0.17	
963		-----		-----	
1135	ISO12185	0.8989		-0.39	
1530	ISO12185	0.8989	C	-0.39	reported: 898.9 kg/l
1862	ISO12185	0.89893		-0.22	

normality	OK
n	15
outliers	1
mean (n)	0.89897
st.dev. (n)	0.000056
R(calc.)	0.00016
R(ISO12185:96)	0.0005

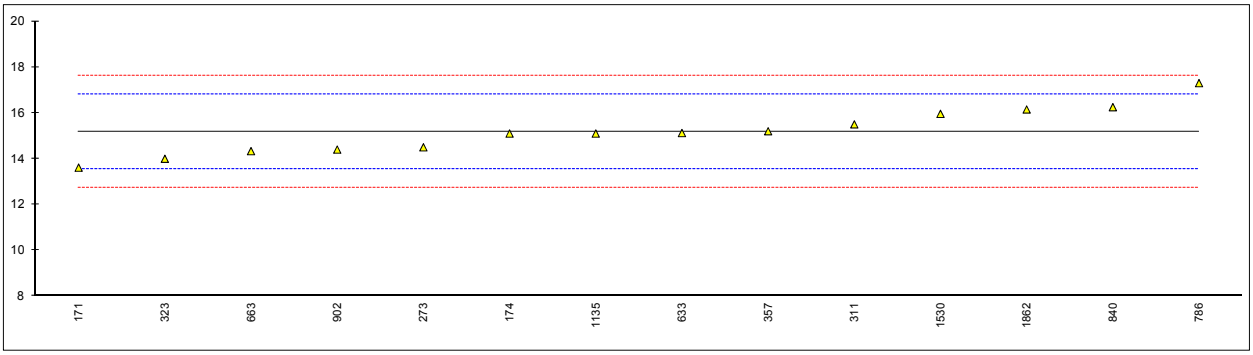


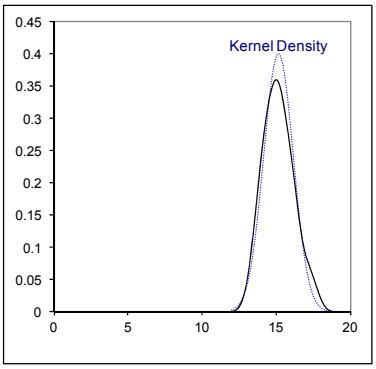


Determination of Inhibitor as MEHQ on sample #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171	D3125	13.611		-1.93	
174	D3125	15.1		-0.10	
273	D3125	14.5		-0.84	
311	D3125	15.5		0.39	
323	D3125	14.0		-1.45	
357	D3125	15.2		0.02	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633	D3125	15.1207		-0.07	
663	D3125	14.33		-1.05	
786	INH-2435	17.3		2.61	
840	D3125	16.25		1.32	
886		-----		-----	
902	D3125	14.4		-0.96	
963		-----		-----	
1135	D3125	15.1		-0.10	
1530	D3125	15.96		0.96	
1862	D3125	16.15		1.19	

normality	OK
n	14
outliers	0
mean (n)	15.18
st.dev. (n)	0.998
R(calc.)	2.80
R(D3125:06)	2.28

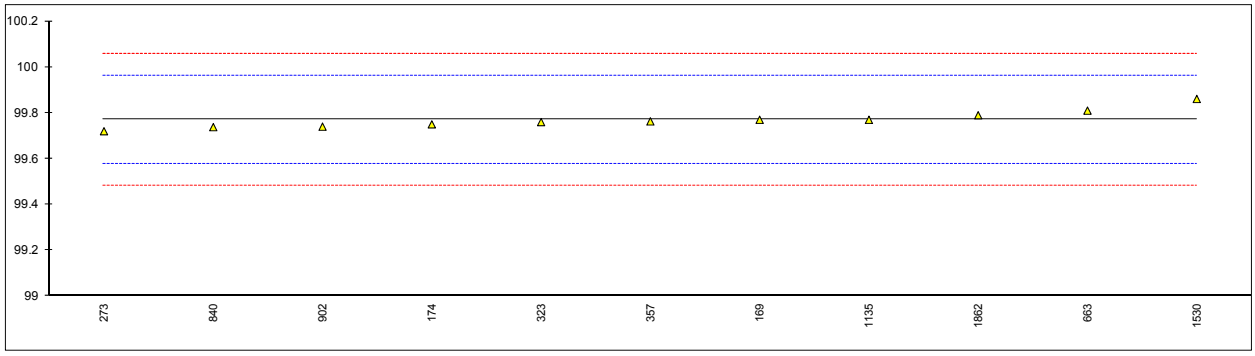


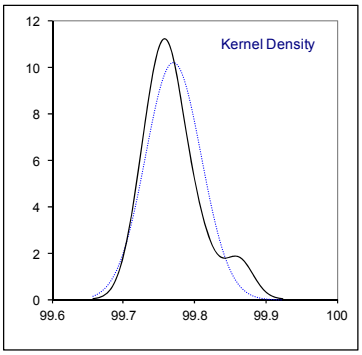


Determination of Purity by GC as received on sample #17062; results in %M/M

lab	method	value	mark	z(targ)	remarks
169	D3362	99.77	E	0.00	E: test result “on dry basis” < test result “as received”
171		-----		-----	
174	D3362	99.750		-0.21	
273	INH-1.4	99.72		-0.52	
311		-----		-----	
323	D3362	99.76		-0.11	
357	D3362	99.763		-0.07	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663	INH-1.4	99.81		0.41	
786		-----		-----	
840	INH-2017	99.738		-0.33	
886		-----		-----	
902	INH-226	99.74		-0.31	
963		-----		-----	
1135	D3362	99.77		0.00	
1530		99.861	E	0.94	E: test result “on dry basis” < test result “as received”
1862		99.79	E	0.21	E: test result “on dry basis” < test result “as received”

normality	not OK
n	11
outliers	0
mean (n)	99.7702
st.dev. (n)	0.03906
R(calc.)	0.1094
R(D3362:05)	0.27

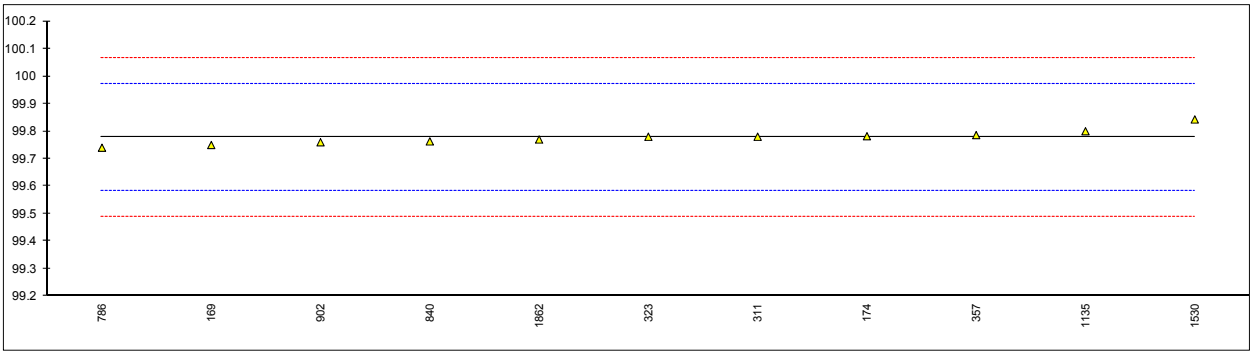


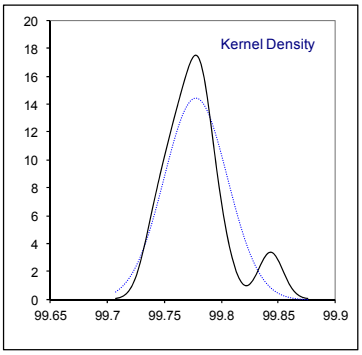


Determination of Purity by GC on dry basis on sample #17062; results in %M/M

lab	method	value	mark	z(targ)	remarks
169	D3362	99.75	E	-0.29	E: test result “on dry basis” < test result “as received”
171		-----		-----	
174	D3362	99.782		0.05	
273		-----		-----	
311	INH-796	99.78		0.02	
323	D3362	99.78		0.02	
357	D3362	99.786		0.09	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
786	INH-2435	99.74		-0.39	
840	INH-2017	99.763		-0.15	
886		-----		-----	
902	INH-226	99.76	C	-0.18	first reported: 9.76
963		-----		-----	
1135	D3362	99.80		0.23	
1530		99.843	E	0.68	E: test result “on dry basis” < test result “as received”
1862		99.77	E	-0.08	E: test result “on dry basis” < test result “as received”

normality	not OK
n	11
outliers	0
mean (n)	99.7776
st.dev. (n)	0.02764
R(calc.)	0.0774
R(D3362:05)	0.27

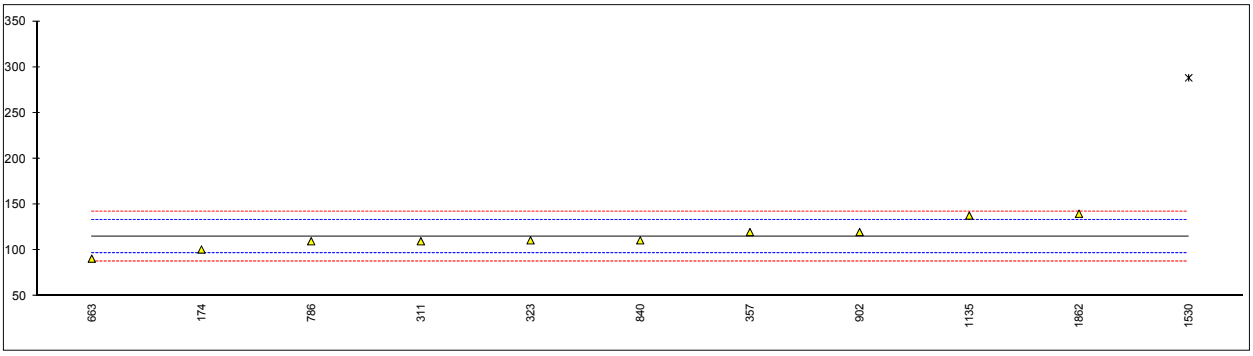


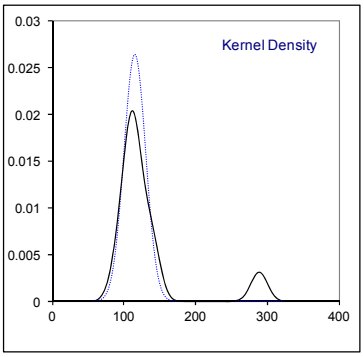


Determination of n-Butanol on sample #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	D3362	<50	C	<-7.23	first reported: 590; probably a false negative test result?
171		-----		-----	
174	D3362	101		-1.57	
273		-----		-----	
311	INH-796	110		-0.58	
323	D3362	111		-0.47	
357	D3362	120		0.53	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663	INH-1.4	91		-2.68	
786	INH-2435	110		-0.58	
840	INH-2017	111		-0.47	
886		-----		-----	
902	INH-226	120		0.53	
963		-----		-----	
1135	D3362	138		2.53	
1530		288.3	G(0.01)	19.19	probably mixed-up with n-Butylpropionate?
1862		140		2.75	

normality	OK
n	10
outliers	1
mean (n)	115.20
st.dev. (n)	15.120
R(calc.)	42.34
R(Horwitz)	25.26

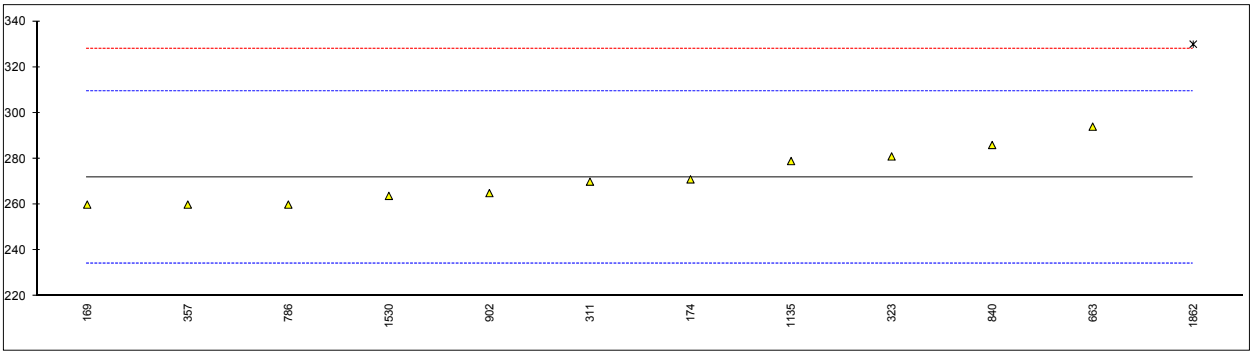


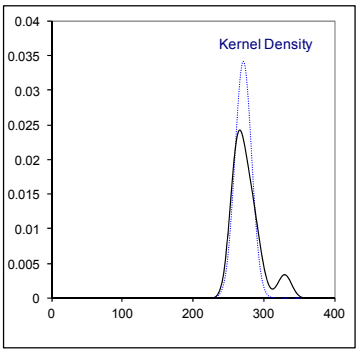


Determination of n-Butylacetate on sample #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	D3362	260	C	-0.63	first reported: 30
171		-----		-----	
174	D3362	271		-0.04	
273		-----		-----	
311	INH-796	270		-0.10	
323	D3362	281		0.49	
357	D3362	260		-0.63	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663	INH-1.4	294		1.19	
786	INH-2435	260		-0.63	
840	INH-2017	286		0.76	
886		-----		-----	
902	INH-226	265		-0.36	
963		-----		-----	
1135	D3362	279		0.38	
1530		263.8		-0.43	
1862		330	G(0.01)	3.11	

normality	OK
n	11
outliers	1
mean (n)	271.80
st.dev. (n)	11.674
R(calc.)	32.69
R(Horwitz)	52.38

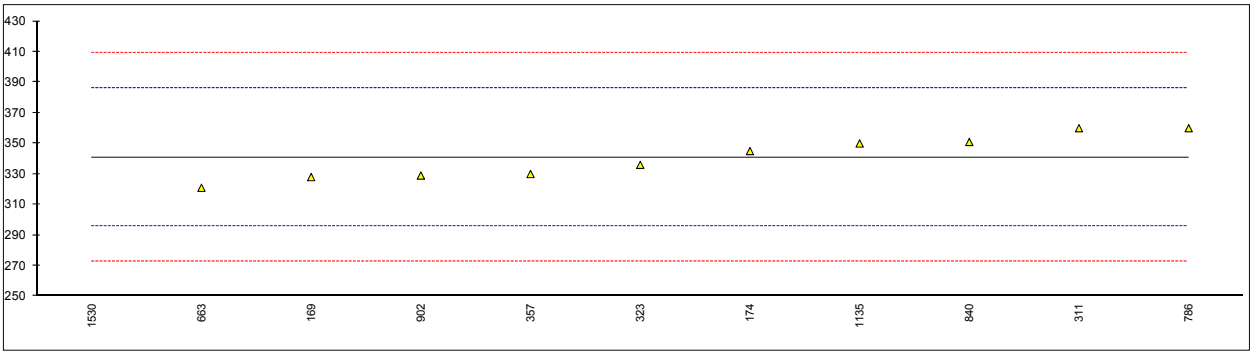


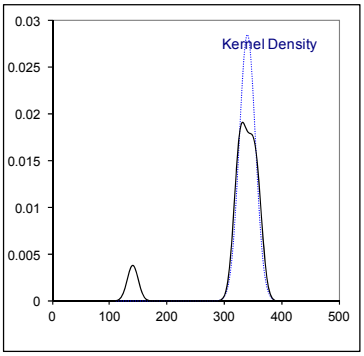


Determination of n-Butylpropionate on sample #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	D3362	328		-0.57	
171		-----		-----	
174	D3362	345		0.18	
273		-----		-----	
311	INH-796	360		0.84	
323	D3362	336		-0.22	
357	D3362	330		-0.48	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663	INH-1.4	321		-0.88	
786	INH-2435	360		0.84	
840	INH-2017	351		0.44	
886		-----		-----	
902	INH-226	329		-0.53	
963		-----		-----	
1135	D3362	350		0.40	
1530		141.0	G(0.01)	-8.82	probably mixed-up with n-Butanol?
1862		-----		-----	

normality	OK
n	10
outliers	1
mean (n)	341.00
st.dev. (n)	14.055
R(calc.)	39.36
R(Horwitz)	63.51

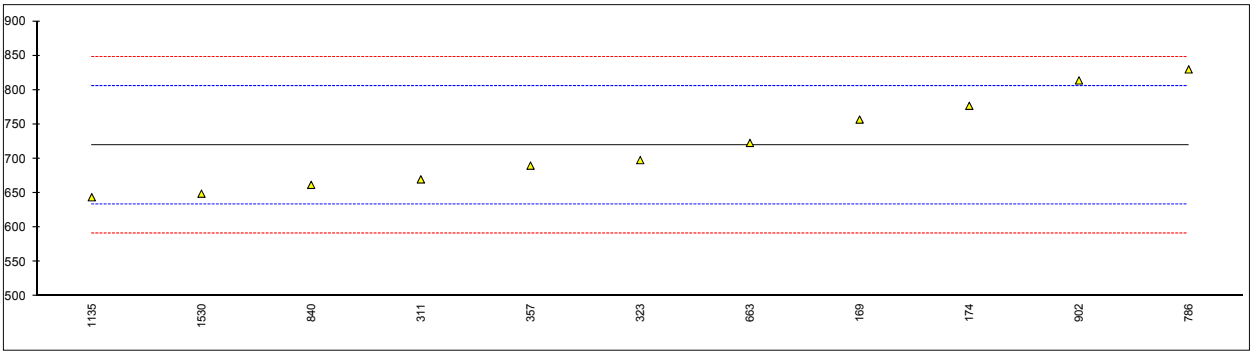


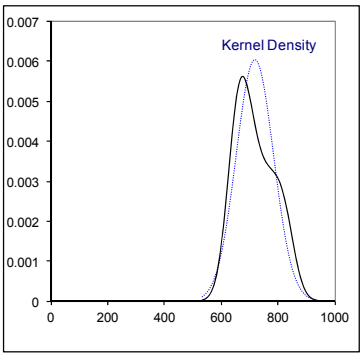


Determination of di-n-Butylether on sample #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	D3362	757		0.88	
171		-----		-----	
174	D3362	777		1.35	
273		-----		-----	
311	INH-796	670		-1.16	
323	D3362	698		-0.50	
357	D3362	690		-0.69	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663	INH-1.4	723		0.08	
786	INH-2435	830		2.58	
840	INH-2017	662		-1.34	
886		-----		-----	
902	INH-226	814		2.21	
963		-----		-----	
1135	D3362	644		-1.76	
1530		649.2		-1.64	
1862		-----		-----	

normality	OK
n	11
outliers	0
mean (n)	719.47
st.dev. (n)	66.028
R(calc.)	184.88
R(Horwitz)	119.75

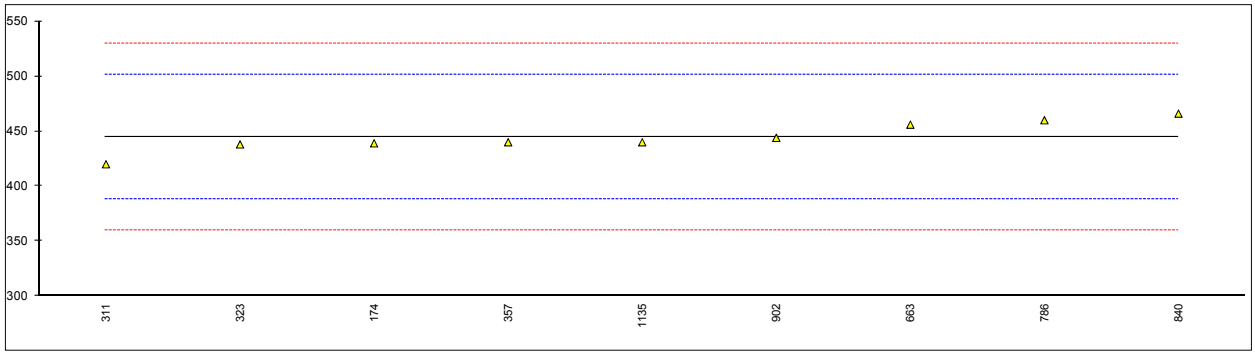


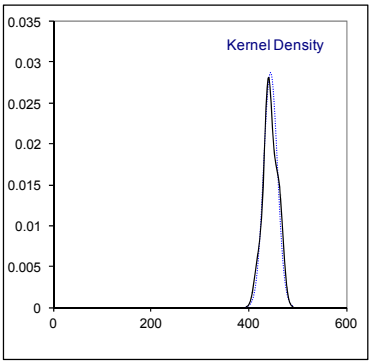


Determination of iso-Butylacrylate on sample #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171		-----		-----	
174	D3362	439		-0.20	
273		-----		-----	
311	INH-796	420		-0.87	
323	D3362	438		-0.24	
357	D3362	440		-0.17	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663	INH-1.4	456		0.39	
786	INH-2435	460		0.54	
840	INH-2017	466		0.75	
886		-----		-----	
902	INH-226	444		-0.03	
963		-----		-----	
1135	D3362	440		-0.17	
1530		-----		-----	
1862		-----		-----	

normality	OK
n	9
outliers	0
mean (n)	444.78
st.dev. (n)	13.908
R(calc.)	38.94
R(Horwitz)	79.59



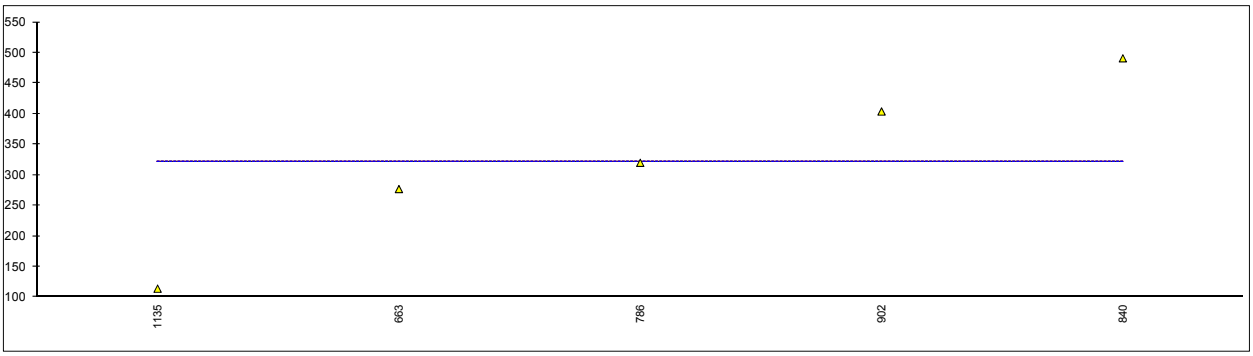


Determination of iso-Butylpropionate on sample #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171		-----		-----	
174		-----		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
357		-----		-----	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
786		-----		-----	
840	INH-2017	<10		-----	
886		-----		-----	
902		-----		-----	
963		-----		-----	
1135	D3362	55		-----	
1530		-----		-----	
1862		-----		-----	
	normality	n.a.			
	n	2			
	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 #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171		-----		-----	
174		-----		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
357		-----		-----	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663	INH-1.4	277		-----	including Benzaldehyde (40 mg/kg) and sec-butyl Acrylate (40 mg/kg)
786	INH-2435	320		-----	
840	INH-2017	491		-----	
886		-----		-----	
902	INH-226	404		-----	
963		-----		-----	
1135	D3362	114		-----	
1530		-----		-----	
1862		-----		-----	
	normality	n.a.			
	n	5			
	outliers	n.a.			
	mean (n)	321.20			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit.)	n.a.			

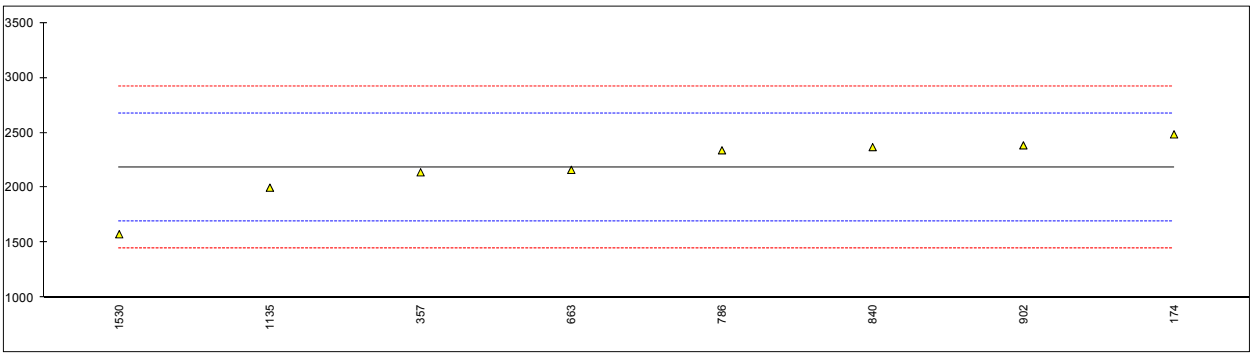


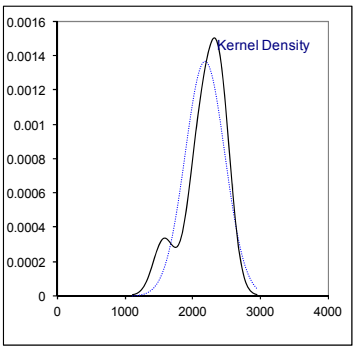
Determination of Total Impurities on sample #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171		-----		-----	
174	D3362	2485	E	1.23	gap of 552 mg/kg *)
273		-----		-----	
311		-----		-----	
323		-----		-----	
357	D3362	2140	E	-0.17	gap of 300 mg/kg *)
522		-----		-----	
551		-----		-----	
621		-----		-----	
633		-----		-----	
663	INH-1.4	2162		-0.08	
786	INH-2435	2340		0.64	
840	INH-2017	2369		0.76	
886		-----		-----	
902	INH-226	2385		0.83	
963		-----		-----	
1135	D3362	2000		-0.74	
1530		1576.8	E	-2.47	gap of 235 mg/kg *)
1862		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	2182.23
st.dev. (n)	291.426
R(calc.)	815.99
R(Horwitz 5 comp)	687.24

*) iis could not reproduce the total of impurities. Unfortunately, the laboratory did not report a test result under “Other Impurities”

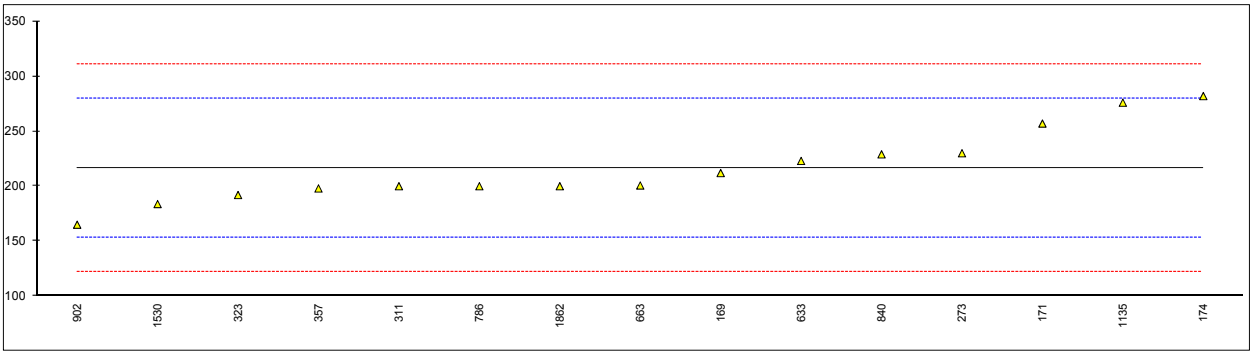


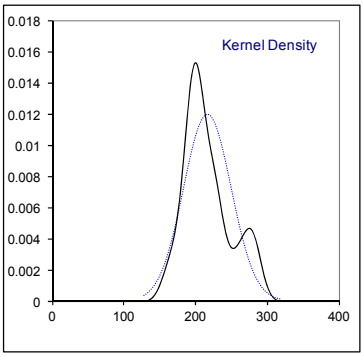


Determination of Water, titrimetric on sample #17062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	E1064	212		-0.14	
171	D1364	257		1.28	
174	E203	282		2.07	
273	E203	230	C	0.43	first reported: 0.023 mg/kg
311	D1364	200		-0.53	
323	D1364	192		-0.78	
357	E1064	198		-0.59	
522		-----		-----	
551		-----		-----	
621		-----		-----	
633	E1064	223	C	0.20	first reported: 0.0223 mg/kg
663	E1064	200.7		-0.50	
786	D1364	200		-0.53	
840	E1064	229		0.39	
886		-----		-----	
902	D1364	165		-1.64	
963		-----		-----	
1135	D1364	276		1.88	
1530	D1364	183.8		-1.04	
1862	E1064	200		-0.53	

normality	OK
n	15
outliers	0
mean (n)	216.57
st.dev. (n)	33.383
R(calc.)	93.47
R(D1364:02)	88.30





APPENDIX 2

Number of participants per country

2 labs in BELGIUM
1 lab in BRAZIL
1 lab in FINLAND
1 lab in GERMANY
1 lab in INDONESIA
1 lab in MEXICO
1 lab in NETHERLANDS
1 lab in PHILIPPINES
2 labs in RUSSIAN FEDERATION
1 lab in SAUDI ARABIA
1 lab in SOUTH AFRICA
1 lab in TAIWAN
1 lab in THAILAND
1 lab in TURKEY
3 labs in UNITED STATES OF AMERICA
1 lab in VIETNAM

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
2 ASTM E178:89
3 ASTM E1301:89
4 ISO 5725:86
5 ISO 5725, parts 1-6, 1994
6 ISO13528:05
7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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)
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