Results of Proficiency Test
n-Butylacrylate
March 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. M. Meijer
Corrector: ing. A.S. Noordman-de Neef
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CONTENTS

1 INTRODUCTION

Since 2004 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of n-Butylacrylate every two year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of n-Butylacrylate.

In this interlaboratory study 23 laboratories in 20 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the n-Butylacrylate proficiency test for 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 organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one sample n-Butylacrylate in a 0.5 liter bottle labelled #21027.
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/IEC17043:2010. 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 regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). 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

A batch of approximately 20 liters of n-Butylacrylate was obtained from a chemical supplier. After homogenization 32 amber glass bottles of 0.5L were filled and labelled #21027.
The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ASTM D4052 and Water in accordance with ASTM E1064 on 4 stratified randomly selected subsamples.

	Density at 20°C in kg/L	Water in mg/kg
sample #21027-1	0.89901	180
sample #21027-2	0.89900	180
sample #21027-3	0.89902	180
sample #21027-4	0.89898	180

Table 1: homogeneity test results of subsamples #21027

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 20°C in kg/L	Water in mg/kg
r (observed)	0.00005	0.0
reference test method	ISO12185:96	E1064:16
0.3 x R (reference test method)	0.00015	8.6

Table 2: evaluation of the repeatabilities of subsamples #21027

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

To each of the participating laboratories one sample n-Butylacrylate labelled #21027 was sent on February 24, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of n-Butylacrylate packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine: Acidity as Acrylic acid, Appearance, Color Pt/Co, Density at 20°C, Inhibitor as MEHQ, Purity by GC as received, Purity by GC on dry basis, n-Butanol, n-Butylacetate, n-Butylpropionate, di-n-Butylether, Isobutylacrylate, Isobutylpropionate, Other impurities, Total impurities and Water.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, 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 evaluations.

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 (when applicable) 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the 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 June 2018 (iis-protocol, version 3.5).
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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in 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 target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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. 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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. Three participants reported test results after the final reporting date and two other participants did not report any test results. Not all participants were able to report all tests requested.

In total 21 participants reported 187 numerical test results. Observed were 12 outlying test results, which is 6.4%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all 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 were used by the various laboratories were 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 explained in appendix 3. Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

In the iis PT reports ASTM methods are referred to with a number (e.g. D1209) and an added designation for the year that the 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(2019)). In the test results tables of appendix 1 only the method number and year of adoption or revision (e.g. D1209:05) will be used.

Acidity as Acrylic acid: This determination was problematic for a number of laboratories. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D1613:17.

Appearance: This determination was not problematic. All reporting participants agreed on a result of Pass (Clear and Bright).

Color Pt/Co: This determination was not problematic. No statistical outliers were observed.
The calculated reproducibility is in agreement with the requirements of ASTM D1209:05(2019).

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

Inhibitor as MEHQ: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D3125:06(2012).

Purity as received and on dry basis: These determinations were not problematic. One statistical outlier was observed. The calculated reproducibilities after rejection of the statistical outlier are both in agreement with the requirements of ASTM D3362:05. Please note: this test method was withdrawn in 2011 with no replacement.

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

n-Butylacetate: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.

n-Butylpropionate: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.

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

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

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

Other impurities: No significant conclusions could be drawn as only three laboratories reported a test result. Therefore no z-scores were calculated.

Total impurities: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation (5 components).

Water: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM E1064:16. When only E1064 test results were evaluated the calculated reproducibility is still not in agreement with the requirements of ASTM E1064:16.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method or as declared by the estimated target reproducibility calculated with the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, EN and ISO test methods) or estimated using the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity as Acrylic acid	mg/kg	15	39.5	15.0	14
Appearance		20	Pass (C&B)	n.a.	n.a.
Color Pt/Co		18	3.8	3.6	7
Density at 20°C	kg/L	18	0.8990	0.0002	0.0005
Inhibitor as MEHQ	mg/kg	19	14.1	2.6	2.1
Purity as received	%M/M	15	99.804	0.101	0.27
Purity on dry basis	%M/M	11	99.806	0.035	0.27
n-Butanol	mg/kg	11	167	27	35
n-Butylacetate	mg/kg	11	485	36	86
n-Butylpropionate	mg/kg	10	388	40	71

Parameter	unit	n	average	2.8 * sd	R(lit)
di-n-Butylether	mg/kg	10	467	79	83
Isobutylacrylate	mg/kg	10	235	25	46
Isobutylpropionate	mg/kg	1	n.a.	n.a.	n.a.
Other impurities	mg/kg	3	n.e.	n.e.	n.e.
Total impurities	mg/kg	6	1967	319	629
Water	mg/kg	18	203	46	32

Table 3: reproducibilities of tests on sample #21027

Without further statistical calculations, it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2021 WITH PREVIOUS PTS

	March 2021	March 2019	April 2017	June 2015	May 2012
Number of reporting laboratories	21	24	16	13	14
Number of test results	187	193	160	117	138
Number of statistical outliers	12	8	6	2	5
Percentage of statistical outliers	6.4%	4.1%	3.8%	1.7%	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 to the requirements of the reference test methods. The conclusions are given in the following table.

Parameter	March 2021	March 2019	April 2017	June 2015	May 2012
Acidity as Acrylic acid	+/-	--	+	++	++
Color 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	+/-	-	-	+	++
Isobutylacrylate	+	--	++	++	++
Isobutylpropionate	n.e.	n.e.	n.e.	n.e.	n.e.
Total impurities	+	+	-	++	n.e.
Water	-	(--)	+/-	+/-	++

Table 5: comparison determinations against the reference test methods

The following performance categories were used:

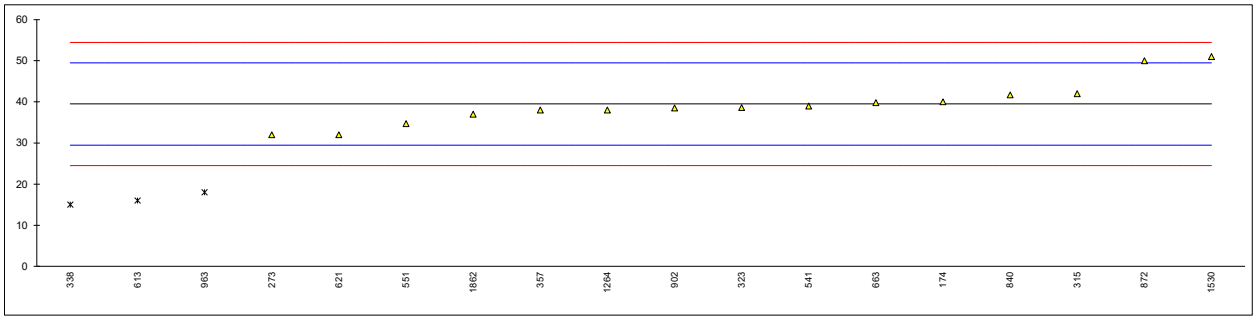
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

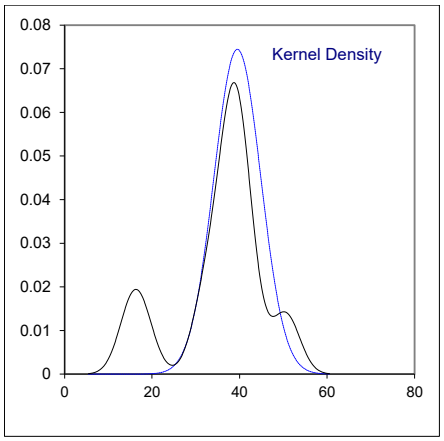
APPENDIX 1

Determination of Acidity as Acrylic acid on sample #21027; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D1613	40		0.10	
273	D1613	32.0		-1.50	
315	D1613	42		0.50	
323	D1613	38.6		-0.18	
338	D1613	15	G(0.05)	-4.90	
357	D1613	38		-0.30	
522		-----		-----	
541	D1613	39.0		-0.10	
551	D1613	34.7		-0.96	
613	D1613	16	C,G(0.05)	-4.70	first reported 0.0008 %M/M
621	D1613	32		-1.50	
633		-----		-----	
663	D1613	39.8		0.06	
840	D1613	41.7		0.44	
872	D1613	50		2.10	
886		-----		-----	
902	D1613	38.5		-0.20	
913		-----		-----	
963	D1613	18	C,G(0.05)	-4.30	first reported 0.0010 %M/M
1264	D1613	38		-0.30	
1530	D1613	51		2.30	
1862	D1613	37		-0.50	

normality	suspect
n	15
outliers	3
mean (n)	39.487
st.dev. (n)	5.3604
R(calc.)	15.009
st.dev.(D1613:17)	5
R(D1613:17)	14





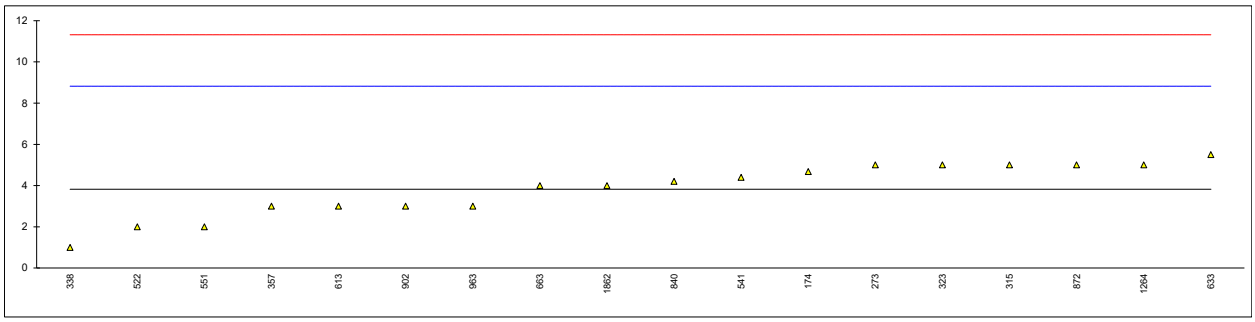
Determination of Appearance on sample #21027;

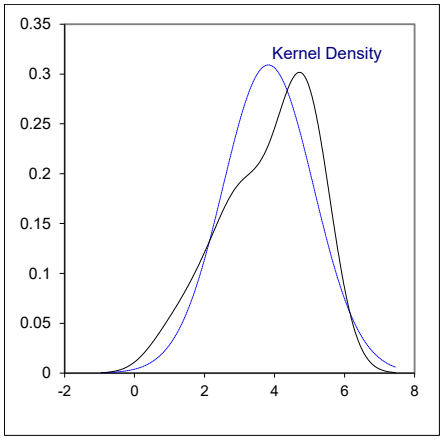
lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	Visual	Clear & Free		-----	
273	Visual	Pass		-----	
315	E2680	pass		-----	
323	INH-001	C&B		-----	
338	Visual	Clear and Bright		-----	
357	E2680	Pass		-----	
522	Visual	PASS		-----	
541	E2680	Pass		-----	
551	E2680	Pass		-----	
613	INH-101967	Pass		-----	
621	D4176	Pass		-----	
633	Visual	Clear & Bright		-----	
663	Visual	Pass		-----	
840	E2680	Pass		-----	
872	E2680	Pass		-----	
886		-----		-----	
902	E2680	PASS		-----	
913		-----		-----	
963	E2680	Pass		-----	
1264	Visual	Pass		-----	
1530	Visual	C&B		-----	
1862	Visual	Pass		-----	
	n	20			
	mean (n)	Pass (C&B)			

Determination of Color Pt/Co on sample #21027;

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D5386	4.68		0.34	
273	D1209	5		0.47	
315	D1209	5		0.47	
323	D1209	5		0.47	
338	D1209	1		-1.13	
357	D5386	3		-0.33	
522	D1209	2		-0.73	
541	D5386	4.4		0.23	
551	D1209	2		-0.73	
613	D5386	3		-0.33	
621	D1209	< 5		-----	
633	D1209	5.5		0.67	
663	D1209	4		0.07	
840	D5386	4.2		0.15	
872	D1209	5		0.47	
886	D1209	<5		-----	
902	D5386	3		-0.33	
913		-----		-----	
963	D1209	3		-0.33	
1264	D1209	5		0.47	
1530	D1209	<3		-----	
1862	D1209	4		0.07	

normality	OK
n	18
outliers	0
mean (n)	3.82
st.dev. (n)	1.291
R(calc.)	3.61
st.dev.(D1209:05)	2.5
R(D1209:05)	7

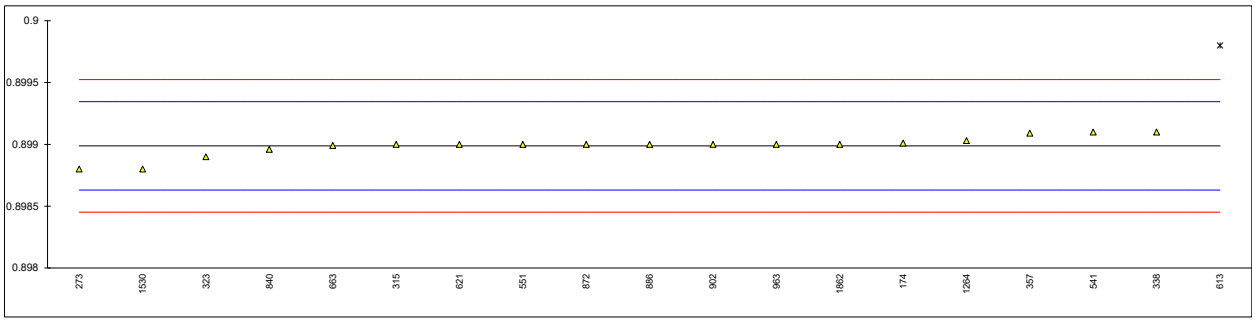


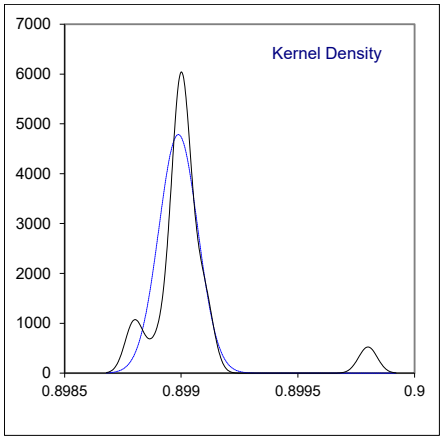


Determination of Density at 20°C on sample #21027; results in kg/L

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D4052	0.89901		0.12	
273	D4052	0.8988		-1.05	
315	D4052	0.8990		0.07	
323	D4052	0.8989		-0.49	
338	ISO12185	0.8991		0.63	
357	D4052	0.89909		0.57	
522		-----		-----	
541	D4052	0.89910		0.63	
551	D4052	0.8990		0.07	
613	D4052	0.8998	C,G(0.01)	4.55	first reported 0.8997
621	D4052	0.8990		0.07	
633		-----		-----	
663	D4052	0.89899		0.01	
840	D4052	0.89896		-0.16	
872	D4052	0.8990		0.07	
886	D4052	0.8990	C	0.07	first reported 899.0 kg/L
902	D4052	0.8990		0.07	
913		-----		-----	
963	ISO12185	0.8990		0.07	
1264	D4052	0.89903		0.24	
1530	ISO12185	0.89880		-1.05	
1862	ISO12185	0.8990		0.07	

normality	suspect
n	18
outliers	1
mean (n)	0.89899
st.dev. (n)	0.000083
R(calc.)	0.00023
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

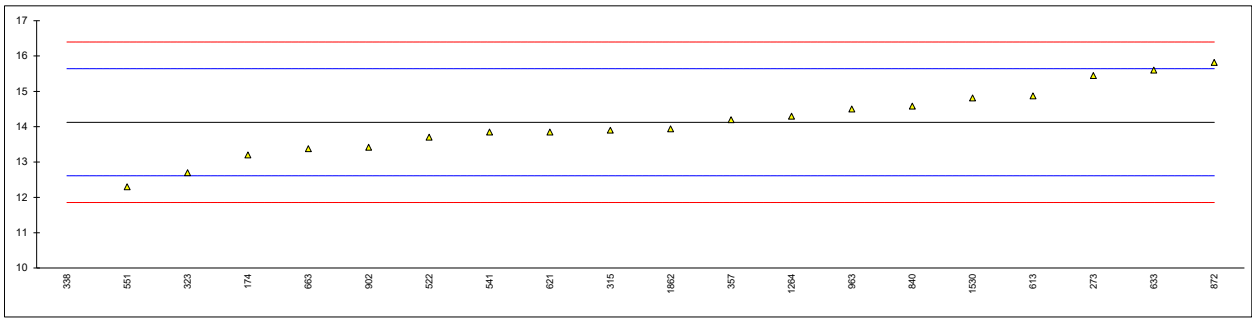


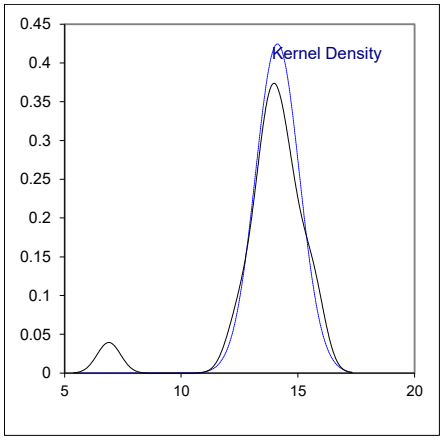


Determination of Inhibitor as MEHQ on sample #21027; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3125	13.2		-1.22	
273	D3125	15.45		1.75	
315	D3125	13.9		-0.30	
323	D3125	12.7		-1.88	
338	D3125	6.9	R(0.01)	-9.55	waiting for a maintenance on the analyzer
357	D3125	14.2		0.10	
522	D3125	13.7		-0.56	
541	D3125	13.85		-0.36	
551	D3125	12.3		-2.41	
613		14.87	C	0.99	first reported 17.33
621	D3125	13.85		-0.36	
633	D3125	15.6		1.95	
663	D3125	13.375		-0.99	
840	D3125	14.58		0.60	
872	INH-175	15.82		2.24	
886		-----		-----	
902	D3125	13.42		-0.93	
913		-----		-----	
963	D3125	14.5	C	0.50	first reported 10.93
1264	D3125	14.3		0.23	
1530	D3125	14.81		0.91	
1862	D3125	13.94		-0.24	

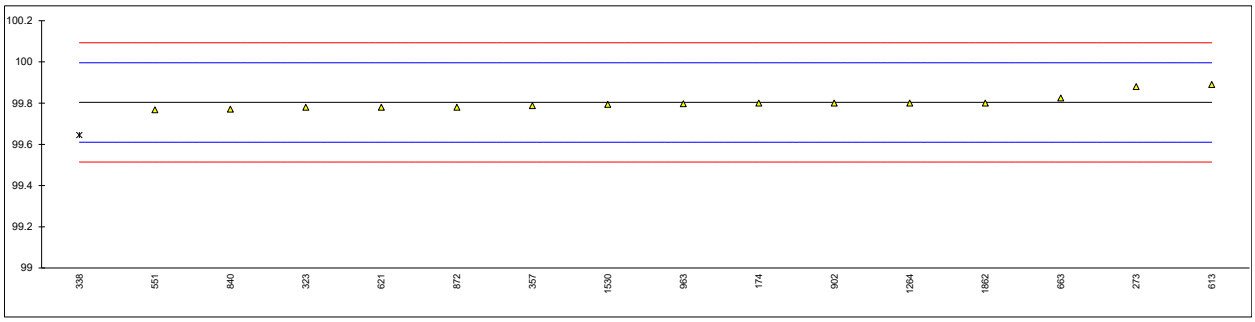
normality	OK
n	19
outliers	1
mean (n)	14.12
st.dev. (n)	0.940
R(calc.)	2.63
st.dev.(D3125:06)	0.757
R(D3125:06)	2.12

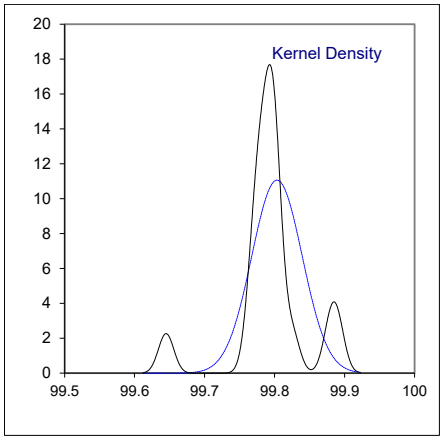




Determination of Purity by GC as received on sample #21027; results in %M/M

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	99.80		-0.04	
273	D3362	99.88		0.79	
315		-----		-----	
323	D3362-mod	99.78		-0.24	
338		99.645	G(0.05)	-1.64	
357	D3362	99.788		-0.16	
522		-----		-----	
541		-----		-----	
551	INH-3088	99.7674		-0.37	
613		99.89		0.90	
621	D3362	99.78		-0.24	
633		-----		-----	
663	INH-1.4	99.825		0.22	
840	INH-004	99.771		-0.34	
872	INH-175	99.78		-0.24	
886		-----		-----	
902	INH-226	99.80		-0.04	
913		-----		-----	
963	INH-102538	99.797		-0.07	
1264	In house	99.800		-0.04	
1530	D3362	99.7944		-0.09	
1862	MSTM4293	99.80		-0.04	
	normality	not OK			
	n	15			
	outliers	1			
	mean (n)	99.8035			
	st.dev. (n)	0.03608			
	R(calc.)	0.1010			
	st.dev.(D3362:05)	0.09643			
	R(D3362:05)	0.27			

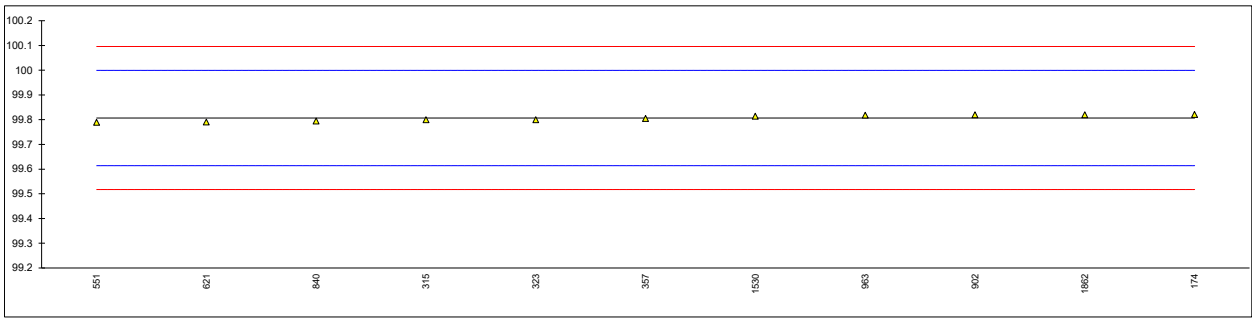


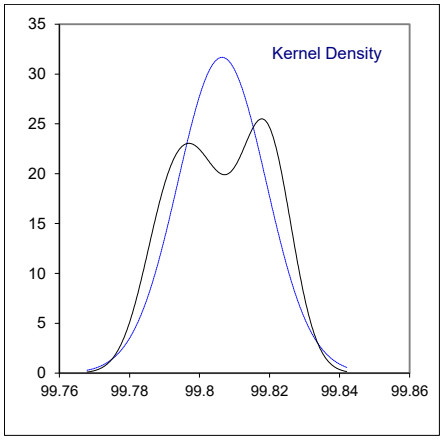


Determination of Purity by GC on dry basis on sample #21027; results in %M/M

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	99.821		0.15	
273		-----		-----	
315	INH-796	99.80		-0.07	
323	D3362-mod	99.80		-0.07	
338		-----		-----	
357	D3362	99.805		-0.01	
522		-----		-----	
541		-----		-----	
551	INH-3088	99.7889		-0.18	
613		-----		-----	
621	D3362	99.79		-0.17	
633		-----		-----	
663		-----		-----	
840	INH-004	99.794		-0.13	
872		-----		-----	
886		-----		-----	
902	INH-226	99.82		0.14	
913		-----		-----	
963	INH-102538	99.818		0.12	
1264		-----		-----	
1530	D3362	99.8139		0.08	
1862	MSTM4293	99.82		0.14	

normality	OK
n	11
outliers	0
mean (n)	99.8064
st.dev. (n)	0.01260
R(calc.)	0.0353
st.dev.(D3362:05)	0.09643
R(D3362:05)	0.27

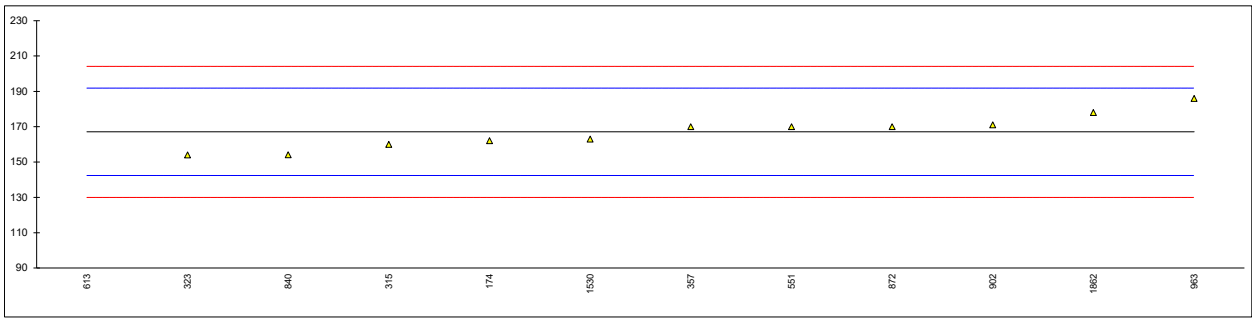


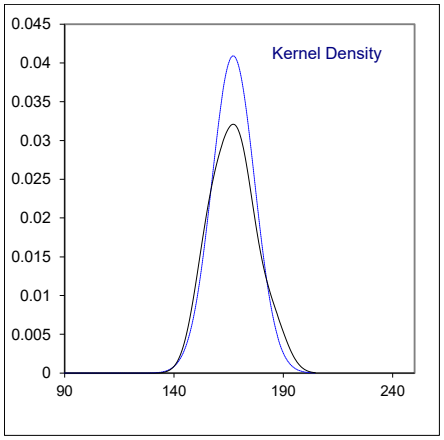


Determination of n-Butanol on sample #21027; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	162		-0.41	
273		-----		-----	
315	INH-796	160		-0.57	
323	D3362-mod	154		-1.06	
338		-----		-----	
357	D3362	170		0.23	
522		-----		-----	
541		-----		-----	
551	INH-3088	170		0.23	
613		0	CG(0.01)	-13.50	first reported <0.1
621		-----		-----	
633		-----		-----	
663		-----		-----	
840	INH-004	154.1		-1.05	
872	INH-175	170		0.23	
886		-----		-----	
902	INH-226	171		0.32	
913		-----		-----	
963	INH-102538	186	C	1.53	first reported 227
1264		-----		-----	
1530	D3362	163		-0.33	
1862	MSTM4293	178		0.88	

normality	OK
n	11
outliers	1
mean (n)	167.10
st.dev. (n)	9.748
R(calc.)	27.30
st.dev.(Horwitz)	12.374
R(Horwitz)	34.65

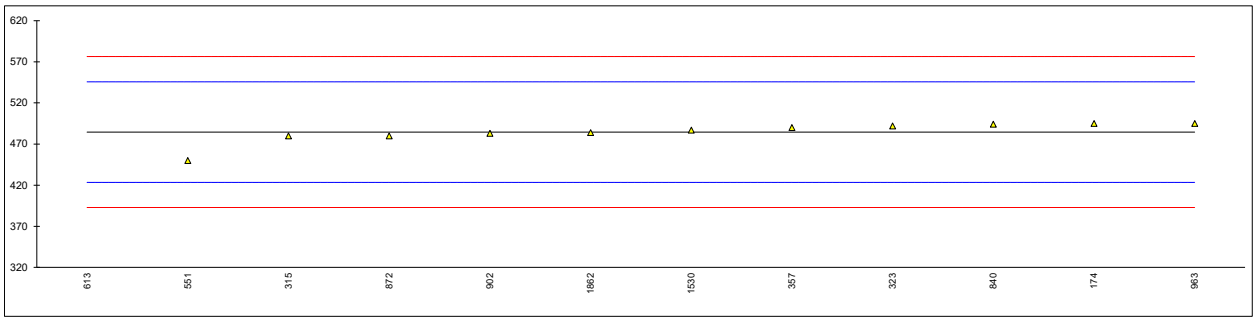


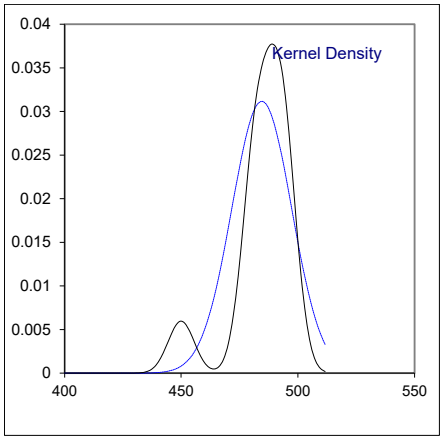


Determination of n-Butylacetate on sample #21027; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	495		0.34	
273		-----		-----	
315	INH-796	480		-0.15	
323	D3362-mod	492		0.24	
338		-----		-----	
357	D3362	490		0.18	
522		-----		-----	
541		-----		-----	
551	INH-3088	450		-1.13	
613		0	C,G(0.01)	-15.85	first reported <0.1
621		-----		-----	
633		-----		-----	
663		-----		-----	
840	INH-004	494.2		0.32	
872	INH-175	480		-0.15	
886		-----		-----	
902	INH-226	483		-0.05	
913		-----		-----	
963	INH-102538	495		0.34	
1264		-----		-----	
1530	D3362	487		0.08	
1862	MSTM4293	484		-0.02	

normality	not OK
n	11
outliers	1
mean (n)	484.56
st.dev. (n)	12.808
R(calc.)	35.86
st.dev.(Horwitz)	30.569
R(Horwitz)	85.59

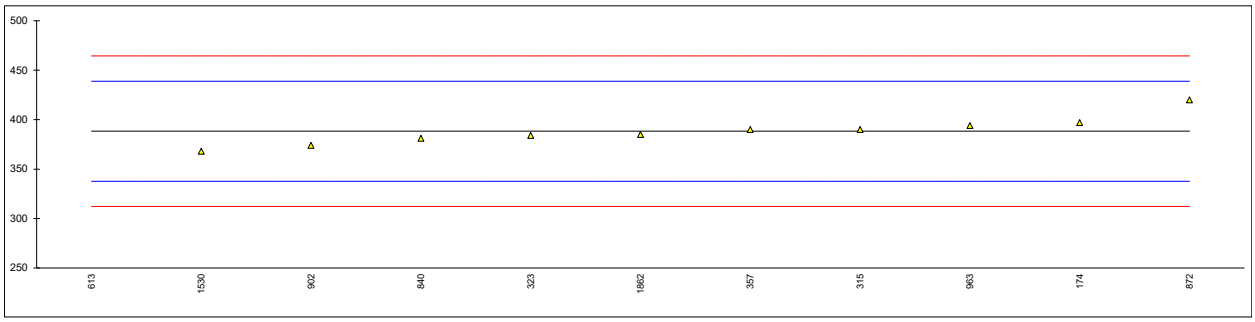


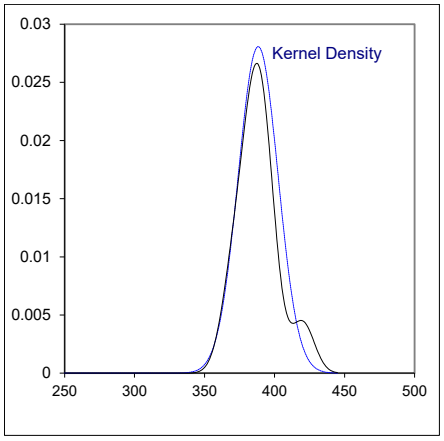


Determination of n-Butylpropionate on sample #21027; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	397		0.34	
273		-----		-----	
315	INH-796	390		0.07	
323	D3362-mod	384		-0.17	
338		-----		-----	
357	D3362	390		0.07	
522		-----		-----	
541		-----		-----	
551		-----		-----	
613		0	C,G(0.01)	-15.33	first reported <0.1
621		-----		-----	
633		-----		-----	
663		-----		-----	
840	INH-004	381.0		-0.29	
872	INH-175	420		1.25	
886		-----		-----	
902	INH-226	374		-0.56	
913		-----		-----	
963	INH-102538	394	C	0.23	first reported 247
1264		-----		-----	
1530	D3362	368		-0.80	
1862	MSTM4293	385		-0.13	

normality	not OK
n	10
outliers	1
mean (n)	388.30
st.dev. (n)	14.213
R(calc.)	39.80
st.dev.(Horwitz)	25.327
R(Horwitz)	70.91

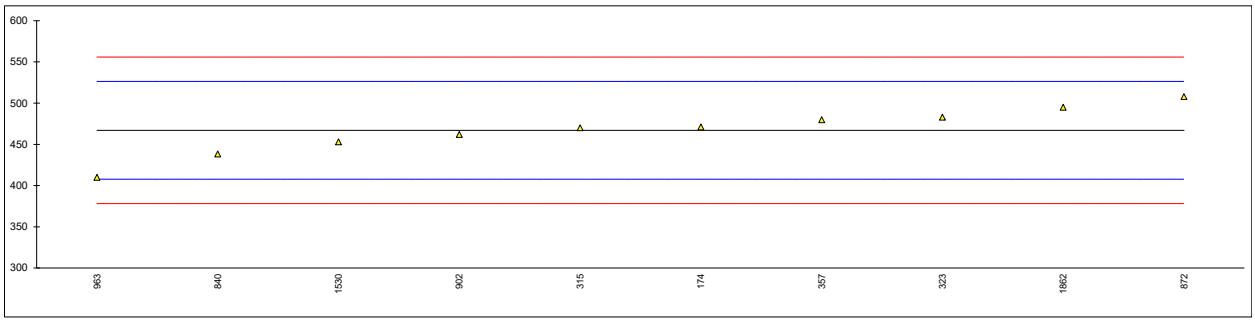


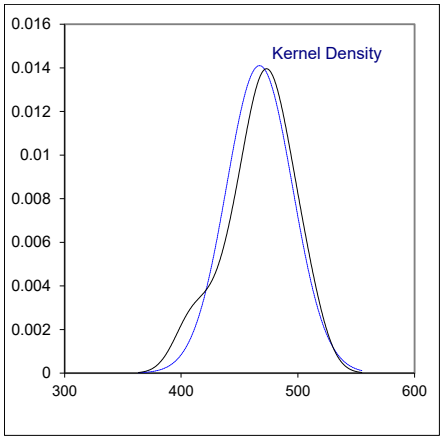


Determination of di-n-Butylether on sample #21027; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	471		0.13	
273		-----		-----	
315	INH-796	470		0.10	
323	D3362-mod	483		0.54	
338		-----		-----	
357	D3362	480		0.44	
522		-----		-----	
541		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
840	INH-004	438.4		-0.97	
872	INH-175	508	C	1.38	first reported 580
886		-----		-----	
902	INH-226	462	C	-0.17	first reported 560
913		-----		-----	
963	INH-102538	410		-1.93	
1264		-----		-----	
1530	D3362	453		-0.47	
1862	MSTM4293	495		0.94	

normality	OK
n	10
outliers	0
mean (n)	467.04
st.dev. (n)	28.290
R(calc.)	79.21
st.dev.(Horwitz)	29.628
R(Horwitz)	82.96

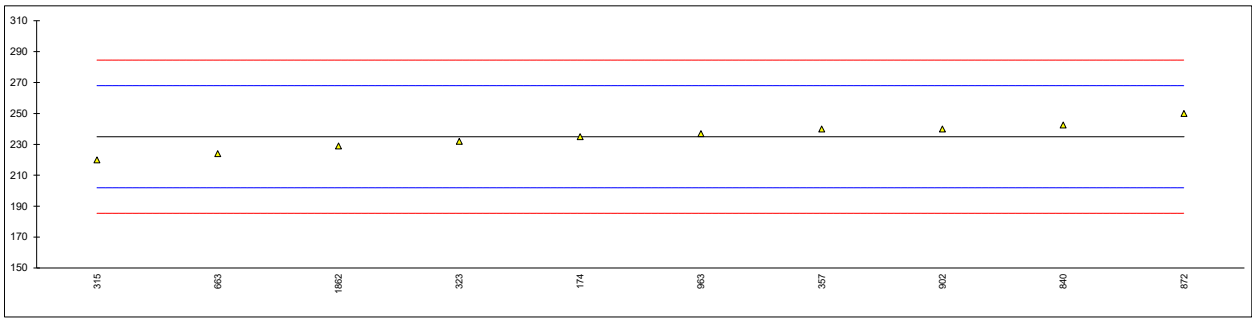


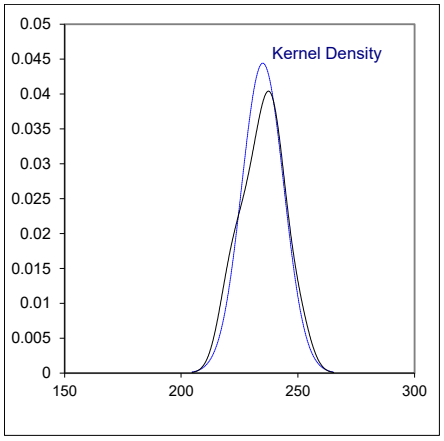


Determination of Isobutylacrylate on sample #21027; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	235		0.00	
273		-----		-----	
315	INH-796	220		-0.90	
323	D3362-mod	232		-0.18	
338		-----		-----	
357	D3362	240		0.31	
522		-----		-----	
541		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663	INH-1.4	224		-0.66	
840	INH-004	242.5		0.46	
872	INH-175	250		0.91	
886		-----		-----	
902	INH-226	240		0.31	
913		-----		-----	
963	INH-102538	237		0.12	
1264		-----		-----	
1530		-----		-----	
1862	MSTM4293	229		-0.36	

normality	OK
n	10
outliers	0
mean (n)	234.95
st.dev. (n)	8.983
R(calc.)	25.15
st.dev.(Horwitz)	16.528
R(Horwitz)	46.28





Determination of Isobutylpropionate on sample #21027; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174		-----		-----	
273		-----		-----	
315		-----		-----	
323		-----		-----	
338		-----		-----	
357		-----		-----	
522		-----		-----	
541		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
840	INH-004	<10		-----	
872		-----		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
963		-----		-----	
1264		-----		-----	
1530		-----		-----	
1862		-----		-----	
	n	1			
	mean (n)	<10			

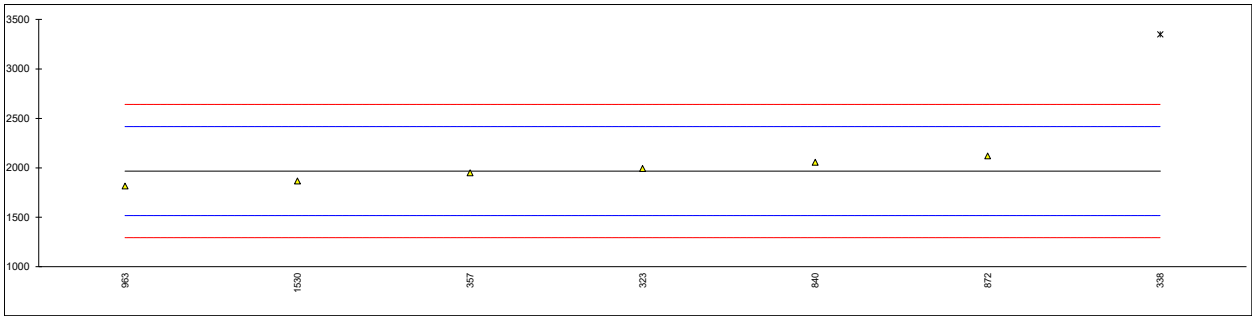
Determination of Other impurities on sample #21027; results in mg/kg

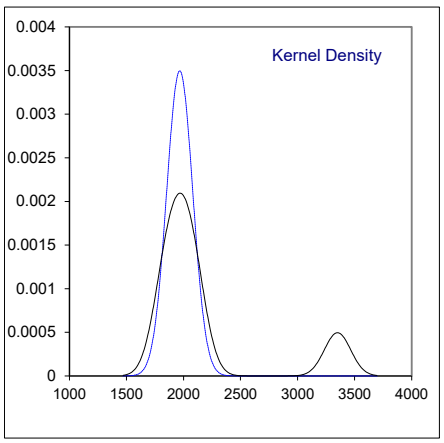
lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174		-----		-----	
273		-----		-----	
315		-----		-----	
323	D3362-mod	250		-----	
338		-----		-----	
357		-----		-----	
522		-----		-----	
541		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
840	INH-004	345.4		-----	
872	INH-175	220		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
963		-----		-----	
1264		-----		-----	
1530		-----		-----	
1862		-----		-----	

Determination of Total impurities on sample #21027; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174		-----		-----	
273		-----		-----	
315		-----		-----	
323	D3362-mod	1994		0.12	
338		3350	C,G(0.01)	6.15	first reported 0.335 mg/kg
357	D3362	1950		-0.08	
522		-----		-----	
541		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
840	INH-004	2055.5		0.39	
872	INH-175	2120		0.68	
886		-----		-----	
902		-----		-----	
913		-----		-----	
963	INH-102538	1816	C	-0.67	first reported 0.1816 mg/kg
1264		-----		-----	
1530	D3362	1867		-0.45	
1862		-----		-----	

normality	unknown
n	6
outliers	1
mean (n)	1967.08
st.dev. (n)	114.070
R(calc.)	319.40
st.dev.(Horwitz 5 comp)	224.728
R(Horwitz 5 comp)	629.24

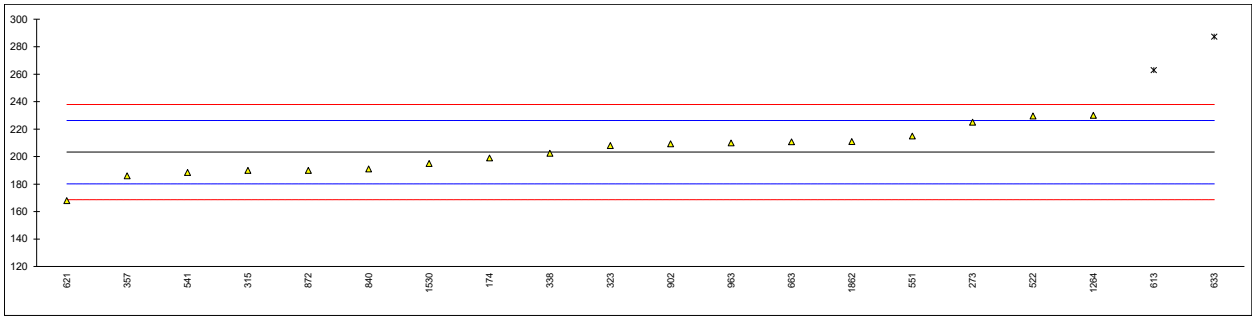


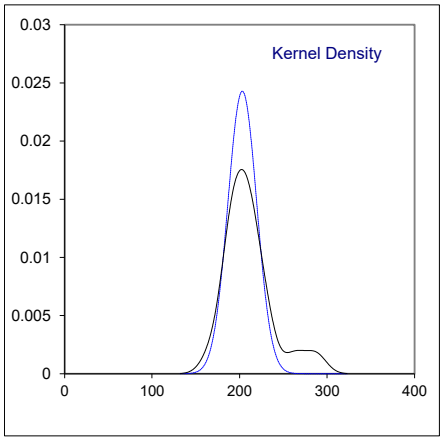


Determination of Water on sample #21027; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	E203	199		-0.37	
273	E203	225.0		1.88	
315	E1064	190		-1.15	
323	E1064	208		0.41	
338	E1064	202.5		-0.07	
357	E1064	186		-1.50	
522	E203	229.6		2.28	
541	E1064	188.5		-1.28	
551	E1064	215		1.02	
613	E203	263	C,R(0.05)	5.18	first reported 310
621	E1064	168		-3.05	
633	E1064	287.3	R(0.05)	7.28	
663	E1064	210.75		0.65	
840	E1064	191		-1.06	
872	D1364	190		-1.15	
886		-----		-----	
902	E1064	209.3		0.52	
913		-----		-----	
963	E1064	210		0.58	
1264	E203	230		2.32	
1530	E1064	195		-0.72	
1862	D1364	211		0.67	

			Only E1064
	normality	OK	OK
	n	18	12
	outliers	2	1
	mean (n)	203.26	197.84
	st.dev. (n)	16.427	13.815
	R(calc.)	46.00	38.68
	st.dev.(E1064:16)	11.542	11.234
	R(E1064:16)	32.32	31.46





APPENDIX 2

Number of participants per country

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

APPENDIX 3

Abbreviations

C	= final 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	= calculation difference between reported test result and result calculated by iis
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, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
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