Institute for
Interlaboratory Studies

Results of Proficiency Test
n-Butylacrylate
July 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: Mrs. E.R. Montenij-Bos & ing. G.A. Oosterlaken-Buijs
Approved by: ing. A.S. Noordman-de Neef

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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 2022/2023 it was decided to continue the round robin for the analysis of n-Butylacrylate.

In this interlaboratory study 18 laboratories in 15 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 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 analyzes 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 L bottle labelled #23115.
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 22.5 liters of n-Butylacrylate was obtained from a chemical supplier. After homogenization 40 amber glass bottles of 0.5 L were filled and labelled #23115.
The homogeneity of the subsamples was checked by determination of Density at 20 °C in accordance with ASTM D4052 on 4 stratified randomly selected subsamples.

	Density at 20 °C in kg/L
sample #23115-1	0.89902
sample #23115-2	0.89903
sample #23115-3	0.89903
sample #23115-4	0.89903

Table 1: homogeneity test results of subsamples #23115

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 20 °C in kg/L
r (observed)	0.00001
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #23115

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample n-Butylacrylate labelled #23115 was sent on June 21, 2023. 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 (derived from e.g. ISO or ASTM test methods), 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

In this proficiency test some problems were encountered with the dispatch of the samples. Therefore, the reporting time on the data entry portal was extended with another week. Two participants reported test results after the extended reporting date and two other participants did not report any test results. Not all participants did report all tests requested.
In total 16 participants reported 146 numerical test results. Observed were 9 outlying test results, which is 6.2%. 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 in appendix 1. 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). When a method has been reapproved an “R” will be added and the year of approval (e.g. D1209:05R19).

Acidity as Acrylic acid: 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 D1613:17R23.

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. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D1209:05R19.

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. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D3125:06R12. Please note: this test method was withdrawn in 2021 with no replacement.

Purity as received and on dry basis: These determinations were not problematic. No statistical outliers were observed. Both the calculated reproducibilities are in agreement with the respective requirements of ASTM D3362:05. Please note: this test method was withdrawn in 2011 with no replacement.

n-Butanol: 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.

n-Butylacetate: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not 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. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation.

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

Isobutylpropionate: No test results were reported. This component will be removed from the PT scheme in next PT over two years.

Other impurities: No significant conclusions could be drawn as only two laboratories reported a test result. This parameter will be removed from the PT scheme in next PT over two years.

Total impurities: 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 (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 E1064:23.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method 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 reference methods are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity as Acrylic acid	mg/kg	13	42.9	18.5	14
Appearance		14	Pass (C&B)	n.a.	n.a.
Color Pt/Co		15	4.9	3.0	7
Density at 20 °C	kg/L	14	0.8990	0.0002	0.0005
Inhibitor as MEHQ	mg/kg	15	15.7	4.2	2.4
Purity as received	%M/M	11	99.84	0.07	0.27
Purity on dry basis	%M/M	9	99.84	0.02	0.27
n-Butanol	mg/kg	9	283	56	54
n-Butylacetate	mg/kg	9	507	101	89
n-Butylpropionate	mg/kg	7	308	40	58
di-n-Butylether	mg/kg	6	140	15	30
Isobutylacrylate	mg/kg	9	136	41	29
Total impurities	mg/kg	5	1529	255	508
Water	mg/kg	13	115	46	18

Table 3: reproducibilities of tests on sample #23115

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 JULY 2023 WITH PREVIOUS PTS

	July 2023	March 2021	March 2019	April 2017	June 2015
Number of reporting laboratories	16	21	24	16	13
Number of test results	146	187	193	160	117
Number of statistical outliers	9	12	8	6	2
Percentage of statistical outliers	6.2%	6.4%	4.1%	3.8%	1.7%

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	July 2023	March 2021	March 2019	April 2017	June 2015
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	-	+	--	++	++
Total impurities	++	+	+	-	++
Water	--	-	(--)	+/-	+/-

Table 5: comparison determinations to the reference test methods

For results between brackets no z-scores are calculated

The following performance categories were used:

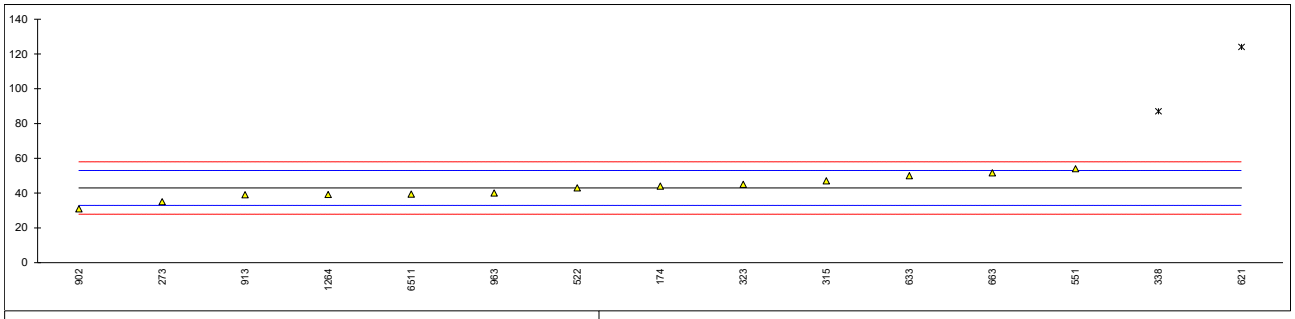
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

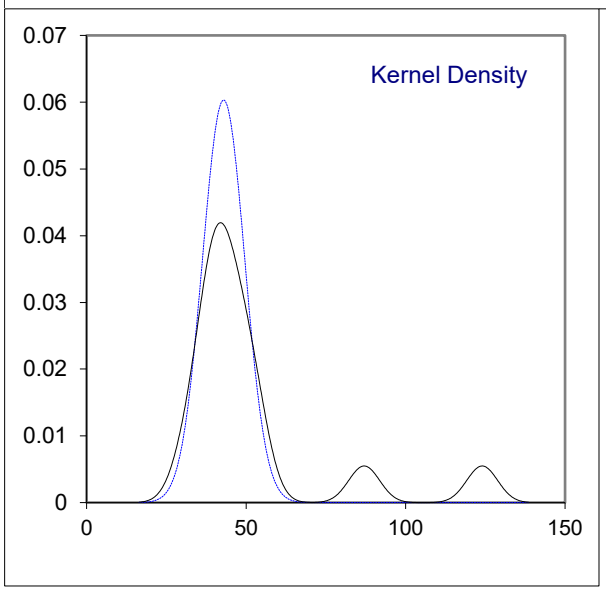
APPENDIX 1

Determination of Acidity as Acrylic acid on sample #23115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D1613	44		0.21	
273	D1613	35		-1.59	
315	D1613	47		0.81	
323	D1613	45		0.41	
338	D1613	87	C,G(0.01)	8.81	First reported 20
522	D1613	43		0.01	
551	D1613	54		2.21	
613		-----		-----	
621	D1613	124	G(0.01)	16.21	
633	D1613	50		1.41	
663	D1613	51.6		1.73	
886		-----		-----	
902	D1613	31	C	-2.39	First reported 0.0015 %M/M
913	D1613	39		-0.79	
963	D1613	40.0		-0.59	
1264	D1613	39.20		-0.75	
6511	D1613	39.40		-0.71	

normality	OK
n	13
outliers	2
mean (n)	42.938
st.dev. (n)	6.6163
R(calc.)	18.526
st.dev.(D1613:17R23)	5
R(D1613:17R23)	14





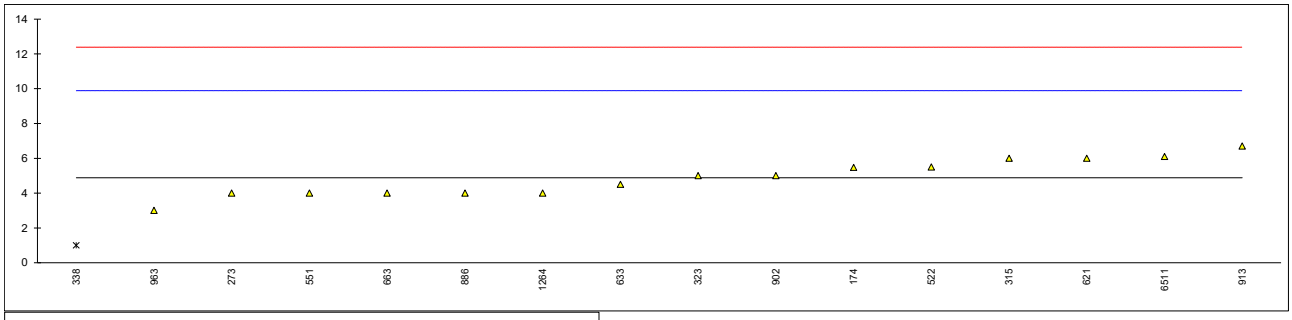
Determination of Appearance on sample #23115;

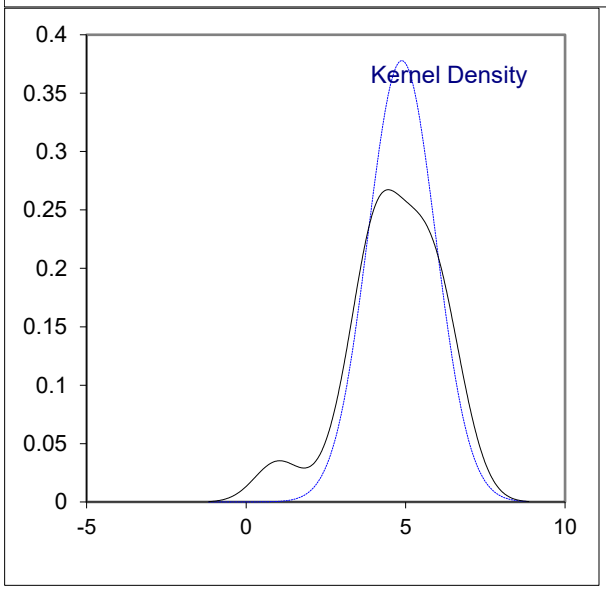
lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	Visual	Clear & Free		-----	
273	Visual	Pass		-----	
315	E2680	pass		-----	
323	Visual	PASS		-----	
338	Visual	C&B FFSM		-----	
522	Visual	Pass		-----	
551	E2680	Pass		-----	
613		-----		-----	
621	E2680	PASS		-----	
633	Visual	Clear & Bright		-----	
663	Visual	PASS		-----	
886		-----		-----	
902	E2680	PASS		-----	
913	E2680	Clear & Bright		-----	
963	E2680	Pass		-----	
1264	Visual	Pass		-----	
6511		-----		-----	
	n	14			
	mean (n)	Pass (C&B)			

Determination of Color Pt/Co on sample #23115;

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D5386	5.47		0.23	
273	D1209	4		-0.35	
315	D5386	6		0.45	
323	D1209	5		0.05	
338	D1209	1	G(0.05)	-1.55	
522	D1209	5.5		0.25	
551	D1209	4		-0.35	
613		-----		-----	
621	D1209	6		0.45	
633	D1209	4.5		-0.15	
663	D1209	4		-0.35	
886	D1209	4		-0.35	
902	D1209	5		0.05	
913	D5386	6.7		0.73	
963	D1209	3		-0.75	
1264	D1209	4		-0.35	
6511	D5386	6.1		0.49	

normality	OK
n	15
outliers	1
mean (n)	4.88
st.dev. (n)	1.056
R(calc.)	2.96
st.dev.(D1209:05R19)	2.5
R(D1209:05R19)	7

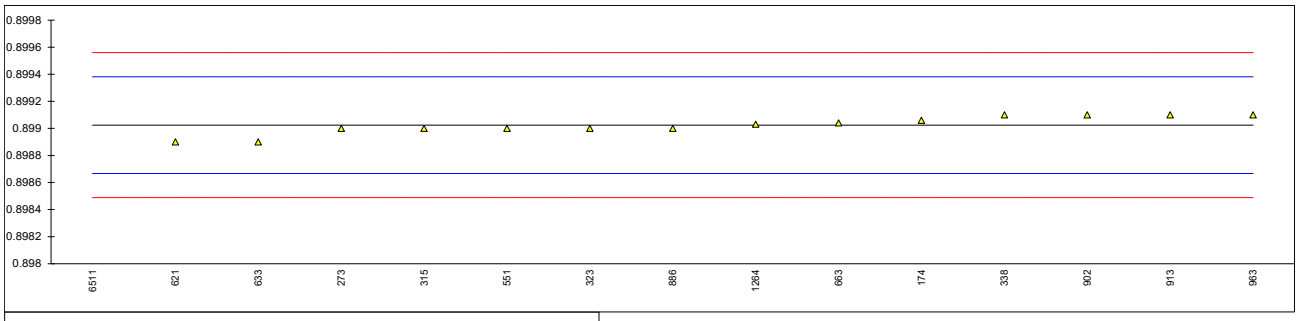


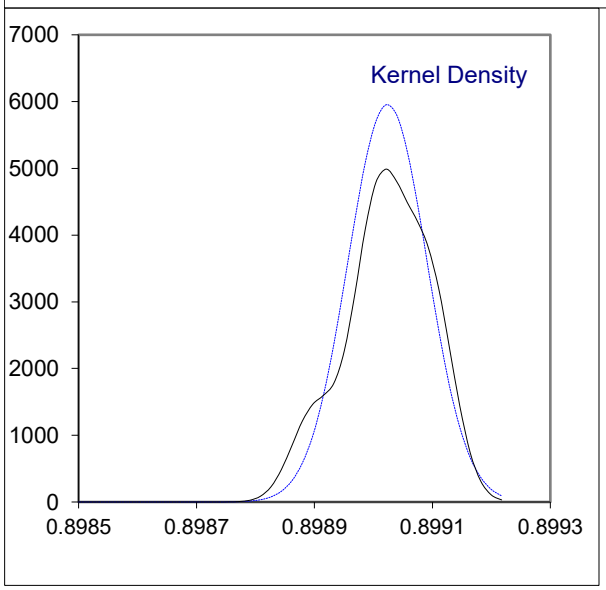


Determination of Density at 20 °C on sample #23115; results in kg/L

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D4052	0.89906		0.20	
273	D4052	0.8990	C	-0.13	First reported 0.8988
315	D4052	0.8990		-0.13	
323	D4052	0.8990		-0.13	
338	ISO12185	0.8991		0.43	
522		-----		-----	
551	D4052	0.8990		-0.13	
613		-----		-----	
621	D4052	0.8989		-0.69	
633	D4052	0.8989	C	-0.69	First reported 0.8992
663	D4052	0.89904		0.09	
886	D4052	0.8990		-0.13	
902	D4052	0.8991		0.43	
913	D4052	0.8991		0.43	
963	ISO12185	0.8991		0.43	
1264	ISO12185	0.89903		0.04	
6511	D4052	0.8948	G(0.01)	-23.65	

normality	OK
n	14
outliers	1
mean (n)	0.89902
st.dev. (n)	0.000007
R(calc.)	0.00019
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

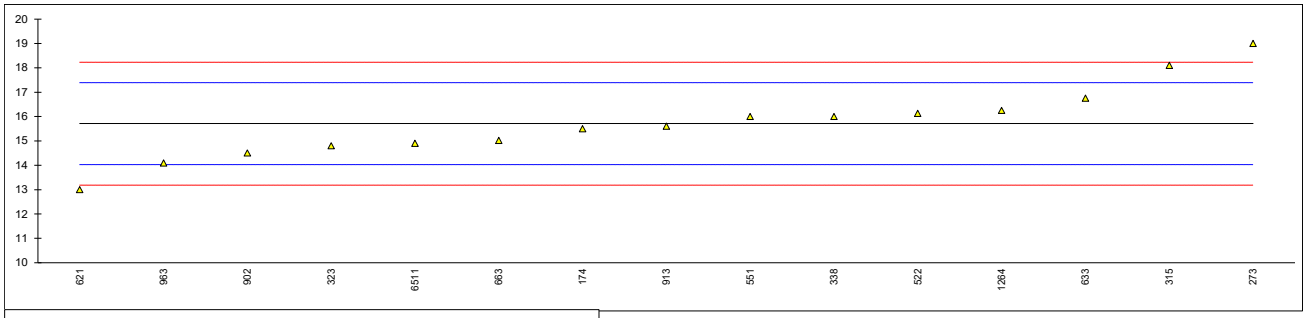


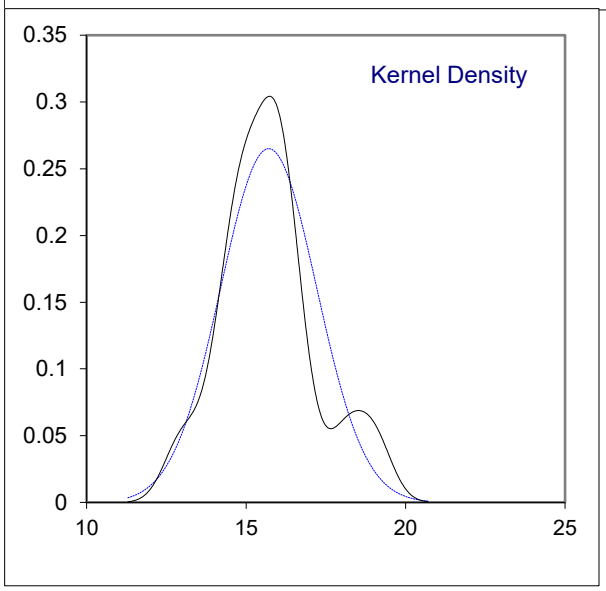


Determination of Inhibitor as MEHQ on sample #23115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3125	15.5		-0.25	
273	D3125	19		3.91	
315	D3125	18.1		2.84	
323	D3125	14.8		-1.08	
338	D3125	16		0.35	
522	D3125	16.13		0.50	
551	D3125	16.0		0.35	
613		-----		-----	
621	D3125	13		-3.22	
633	D3125	16.75		1.24	
663	D3125	15.019		-0.82	
886		-----		-----	
902	D3125	14.5		-1.44	
913	D3125	15.6		-0.13	
963	D3125	14.09		-1.92	
1264	D3125	16.25		0.64	
6511	D3125	14.9		-0.96	

normality	OK
n	15
outliers	0
mean (n)	15.71
st.dev. (n)	1.505
R(calc.)	4.21
st.dev.(D3125:06R12)	0.842
R(D3125:06R12)	2.36

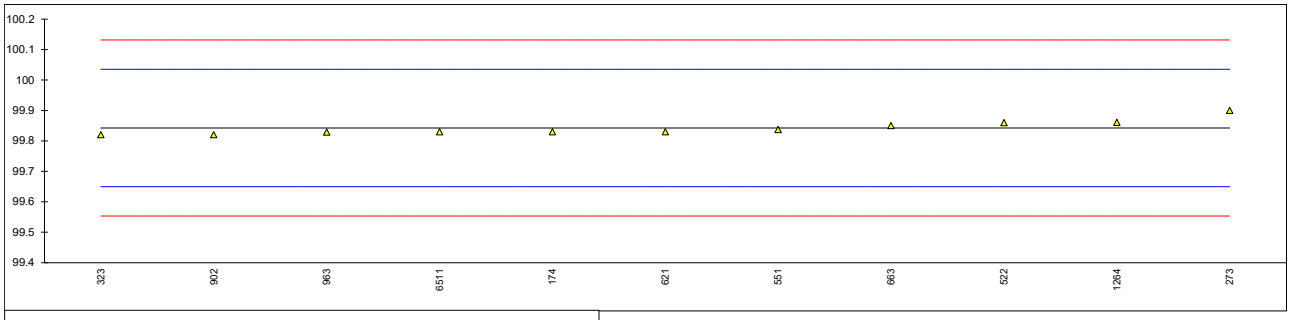


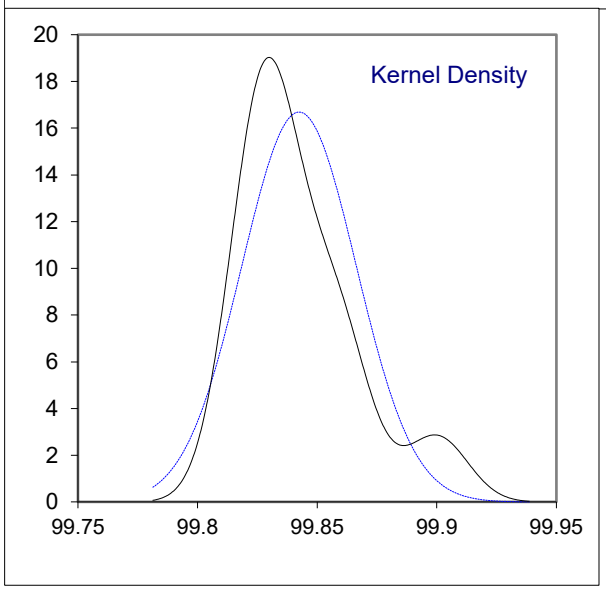


Determination of Purity by GC as received on sample #23115; results in %M/M

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	99.83		-0.13	
273	D3362	99.9		0.60	
315	INH-796	-----		-----	
323	D3362	99.82		-0.23	
338		-----		-----	
522	D3362	99.86		0.18	
551	D3362	99.837	C	-0.06	First reported 99.751
613		-----		-----	
621	D3362	99.83		-0.13	
633		-----		-----	
663	INH-1.4	99.85		0.08	
886		-----		-----	
902	D3362	99.82		-0.23	
913		-----		-----	
963	DOWM102538	99.829		-0.14	
1264	D3362	99.861		0.19	
6511	In house	99.8297		-0.13	

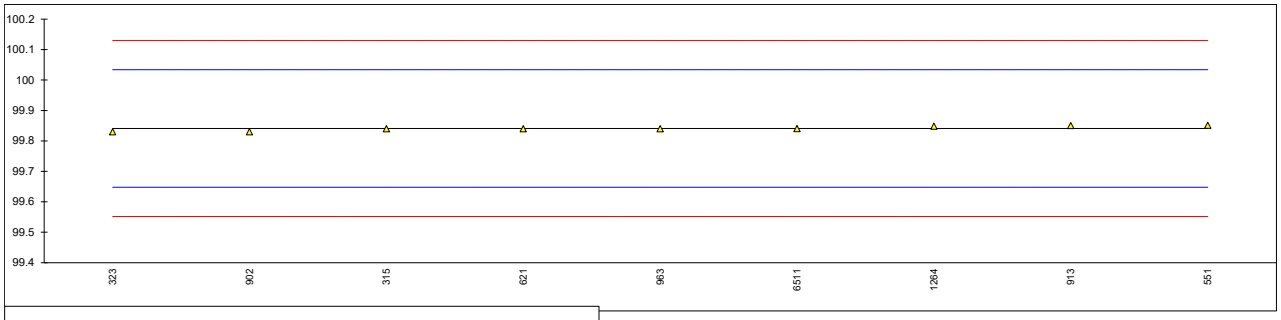
normality	not OK
n	11
outliers	0
mean (n)	99.8424
st.dev. (n)	0.02390
R(calc.)	0.0669
st.dev.(D3362:05)	0.09643
R(D3362:05)	0.27

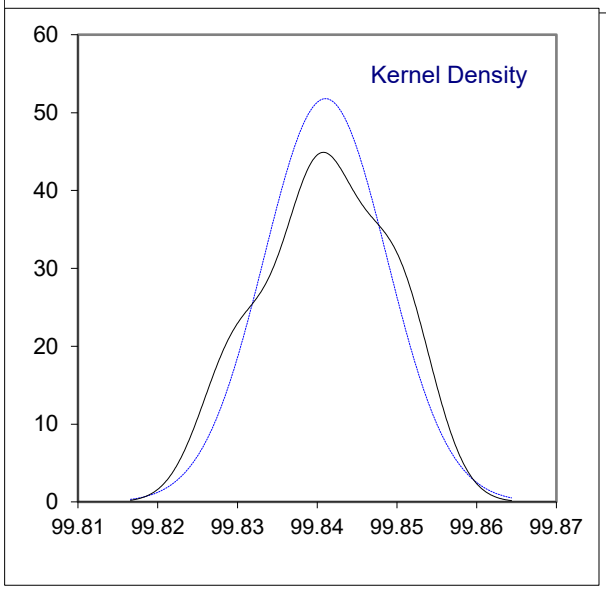




Determination of Purity by GC on dry basis on sample #23115; results in %M/M

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174		-----		-----	
273		-----		-----	
315	INH-796	99.84		-0.01	
323	D3362	99.83		-0.11	
338		-----		-----	
522		-----		-----	
551	D3362	99.851	C	0.10	First reported 99.765
613		-----		-----	
621	D3362	99.84		-0.01	
633		-----		-----	
663		-----		-----	
886		-----		-----	
902	D3362	99.83		-0.11	
913	D3362	99.85		0.09	
963	DOWM102538	99.84		-0.01	
1264	D3362	99.848		0.07	
6511	In house	99.8405		-0.01	
	normality	OK			
	n	9			
	outliers	0			
	mean (n)	99.8411			
	st.dev. (n)	0.00771			
	R(calc.)	0.0216			
	st.dev.(D3362:05)	0.09643			
	R(D3362:05)	0.27			

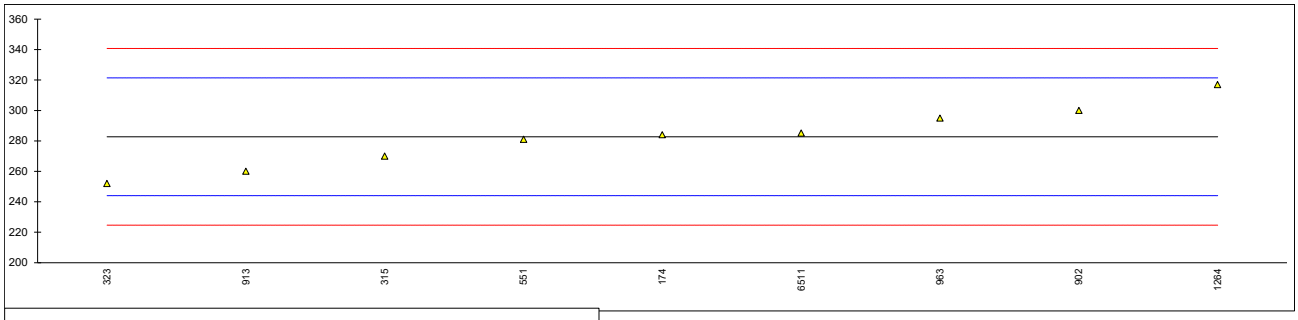


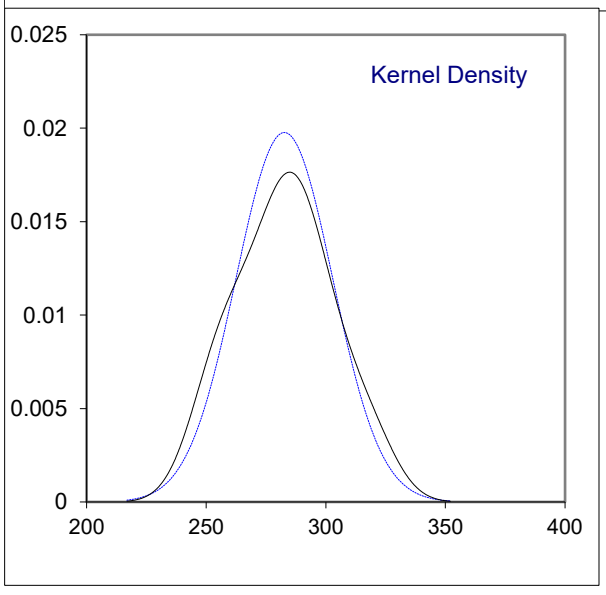


Determination of n-Butanol on sample #23115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	284		0.07	
273		-----		-----	
315	INH-796	270		-0.66	
323	D3362	252		-1.59	
338		-----		-----	
522		-----		-----	
551	D3362	281		-0.09	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
886		-----		-----	
902	D3362	300		0.90	
913	D3362	260		-1.17	
963	INH-102538	295		0.64	
1264	D3362	317	C	1.77	First reported 166.24
6511	In house	285.06		0.12	

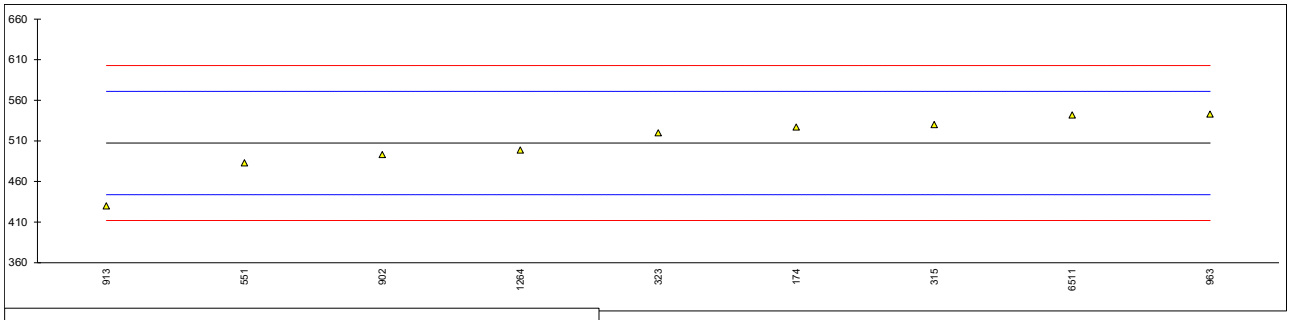
normality	OK
n	9
outliers	0
mean (n)	282.67
st.dev. (n)	20.175
R(calc.)	56.49
st.dev.(Horwitz)	19.340
R(Horwitz)	54.15

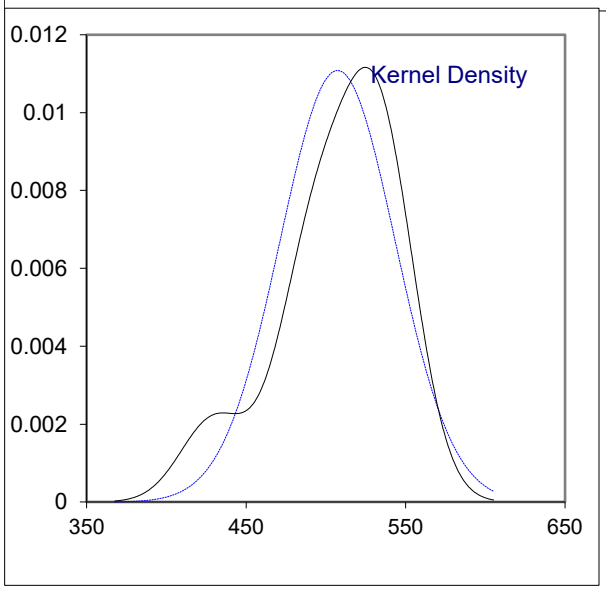




Determination of n-Butylacetate on sample #23115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	527		0.62	
273		-----		-----	
315	INH-796	530		0.71	
323	D3362	520		0.40	
338		-----		-----	
522		-----		-----	
551	D3362	483		-0.77	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
886		-----		-----	
902	D3362	493		-0.45	
913	D3362	430		-2.43	
963	INH-102538	543		1.12	
1264	D3362	498.72		-0.27	
6511	In house	541.895		1.09	
	normality	suspect			
	n	9			
	outliers	0			
	mean (n)	507.40			
	st.dev. (n)	36.020			
	R(calc.)	100.86			
	st.dev.(Horwitz)	31.789			
	R(Horwitz)	89.01			

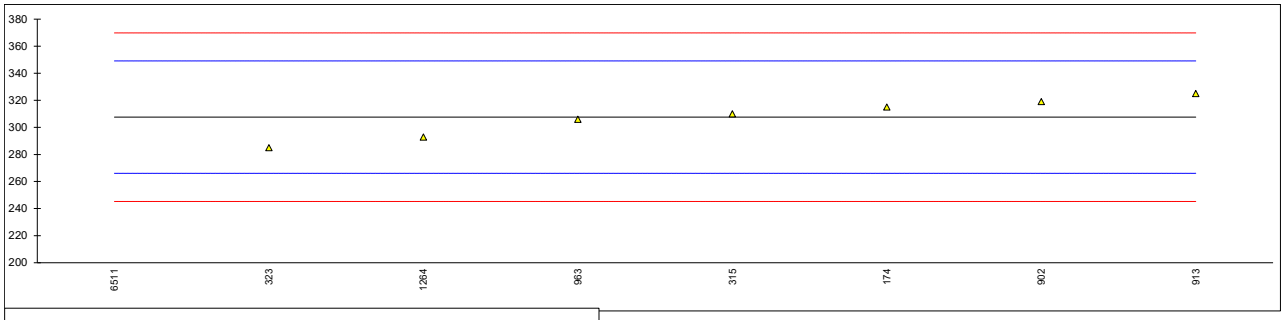


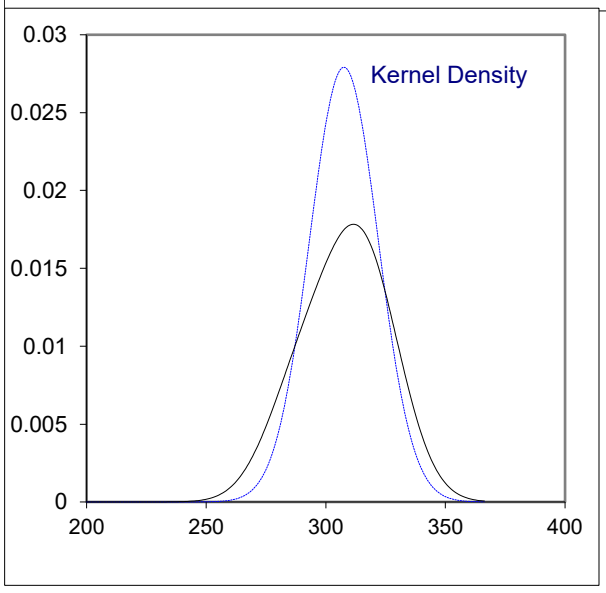


Determination of n-Butylpropionate on sample #23115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	315		0.36	
273		-----		-----	
315	INH-796	310		0.12	
323	D3362	285		-1.08	
338		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
886		-----		-----	
902	D3362	319		0.55	
913	D3362	325		0.84	
963	INH-102538	306		-0.07	
1264	D3362	292.79		-0.71	
6511	In house	30.40	G(0.01)	-13.34	

normality	unknown
n	7
outliers	1
mean (n)	307.54
st.dev. (n)	14.294
R(calc.)	40.02
st.dev.(Horwitz)	20.776
R(Horwitz)	58.17

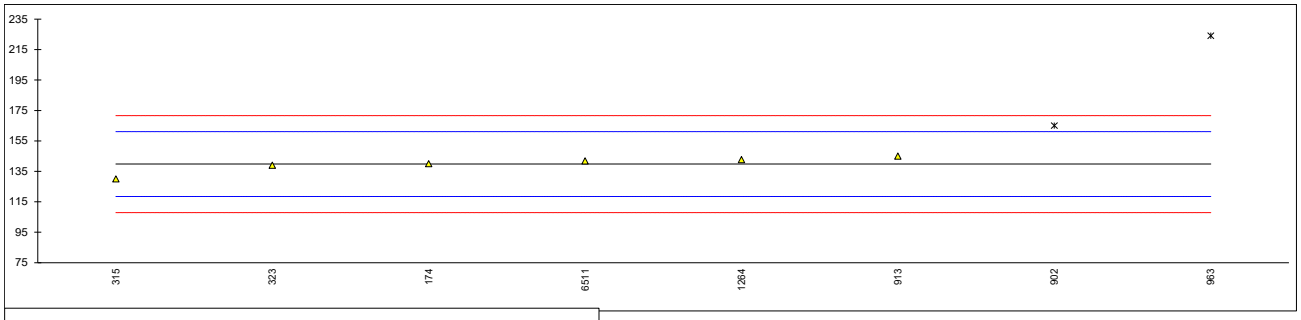


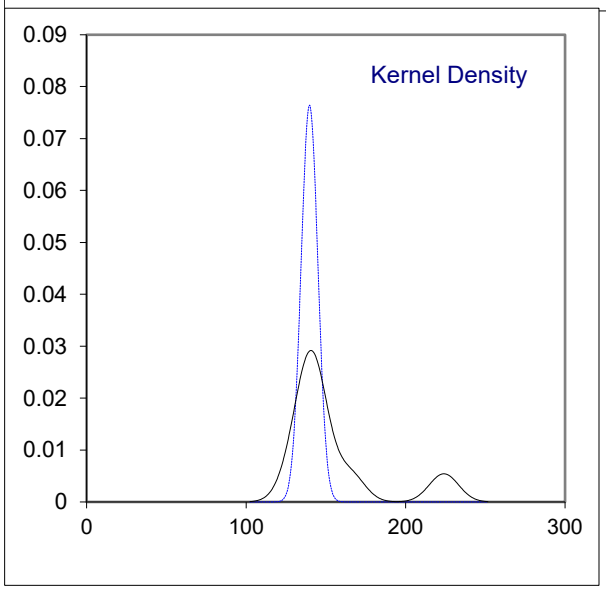


Determination of di-n-Butylether on sample #23115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	140		0.02	
273		-----		-----	
315	INH-796	130		-0.92	
323	D3362	139		-0.07	
338		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
886		-----		-----	
902	D3362	165	G(0.05)	2.37	
913	D3362	145		0.49	
963	INH-102538	224	G(0.01)	7.92	
1264	D3362	142.68		0.28	
6511	In house	141.835		0.20	

normality	unknown
n	6
outliers	2
mean (n)	139.75
st.dev. (n)	5.219
R(calc.)	14.61
st.dev.(Horwitz)	10.631
R(Horwitz)	29.77

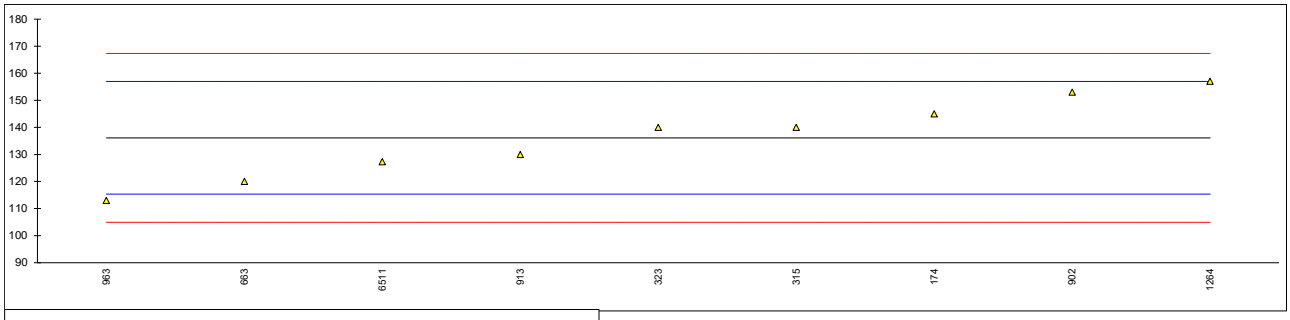


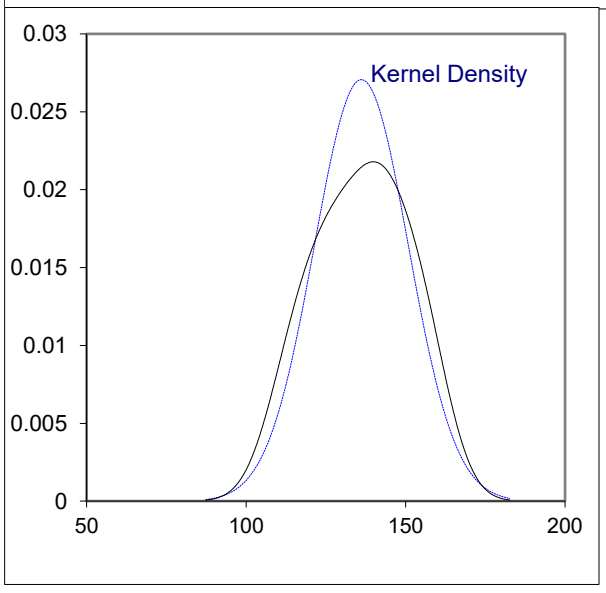


Determination of Isobutylacrylate on sample #23115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	D3362	145		0.85	
273		-----		-----	
315	INH-796	140		0.37	
323	D3362	140		0.37	
338		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663	INH-1.4	120	C	-1.55	First reported 0.01
886		-----		-----	
902	D3362	153		1.62	
913	D3362	130		-0.59	
963	INH-102538	113		-2.23	
1264	D3362	157	C	2.01	First reported 297.18
6511	In house	127.314		-0.85	

normality	OK
n	9
outliers	0
mean (n)	136.15
st.dev. (n)	14.745
R(calc.)	41.29
st.dev.(Horwitz)	10.397
R(Horwitz)	29.11





Determination of Isobutylpropionate on sample #23115; results in mg/kg

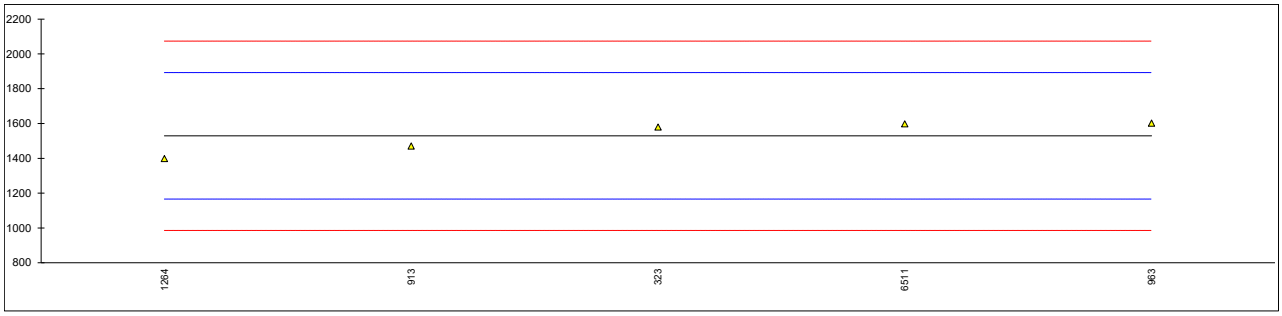
lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174		-----		-----	
273		-----		-----	
315		-----		-----	
323		-----		-----	
338		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
963		-----		-----	
1264	D3362	ND		-----	
6511		-----		-----	

Determination of Other impurities on sample #23115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174		-----		-----	
273		-----		-----	
315		-----		-----	
323	D3362	152		-----	
338		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
963		-----		-----	
1264	D3362	ND		-----	
6511	In house	456.56		-----	

Determination of Total impurities on sample #23115; results in mg/kg

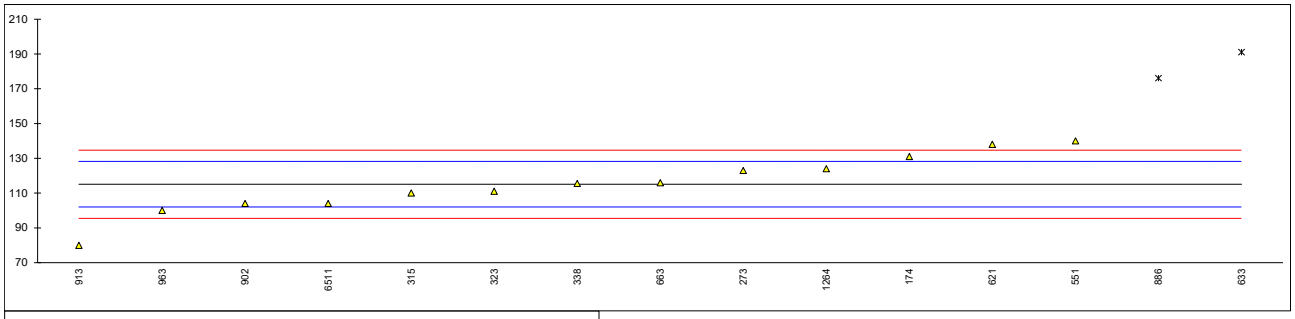
lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174		-----		-----	
273		-----		-----	
315		-----		-----	
323	D3362	1579		0.27	
338		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
621		-----		-----	
633		-----		-----	
663		-----		-----	
886		-----		-----	
902		-----		-----	
913	D3362	1470		-0.33	
963	INH-102538	1602		0.40	
1264	D3362	1398		-0.72	
6511	In house	1598.061		0.38	
	normality	unknown			
	n	5			
	outliers	0			
	mean (n)	1529.4			
	st.dev. (n)	91.16			
	R(calc.)	255.2			
	st.dev.(Horwitz 5 comp)	181.47			
	R(Horwitz 5 comp)	508.1			

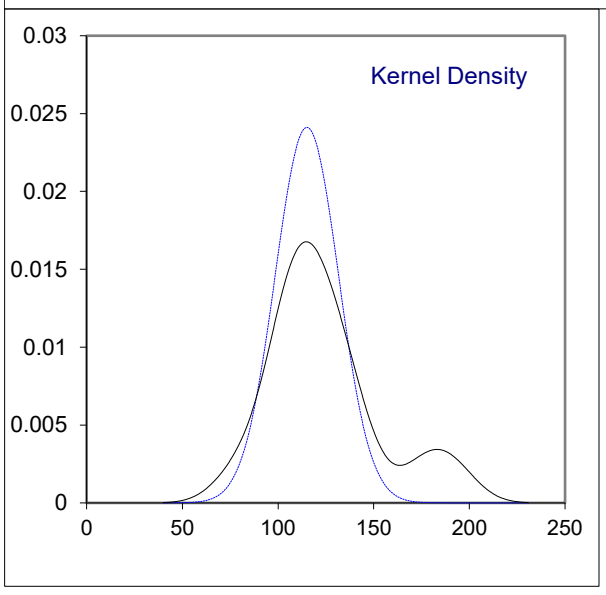


Determination of Water on sample #23115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
174	E203	131		2.43	
273	E1064	123		1.21	
315	E1064	110		-0.78	
323	E1064	111		-0.63	
338	ISO12937	115.5		0.06	
522		-----		-----	
551	E1064	140		3.81	
613		-----		-----	
621	E1064	138		3.50	
633	E1064	191	C,DG(0.05)	11.61	First reported 220.0
663	E1064	115.95		0.13	
886	E1064	176	DG(0.05)	9.31	
902	E1064	104		-1.70	
913	E1064	80		-5.37	
963	E1064	100		-2.31	
1264	E1064	124		1.36	
6511	E1064	104.0		-1.70	

normality	OK
n	13
outliers	2
mean (n)	115.11
st.dev. (n)	16.555
R(calc.)	46.36
st.dev.(E1064:23)	6.537
R(E1064:23)	18.30





APPENDIX 2

Number of participants per country

1 lab in	AUSTRALIA
1 lab in	BELGIUM
1 lab in	BRAZIL
1 lab in	FRANCE
1 lab in	INDIA
1 lab in	INDONESIA
1 lab in	MEXICO
1 lab in	NETHERLANDS
1 lab in	PHILIPPINES
3 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

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

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