Results of Proficiency Test
Ethyl Acetate & n-Butyl Acetate
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Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Authors: ing. L. Dijkstra
Correctors: dr. R.G. Visser & ing. R.J. Starink

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CONTENTS

1 INTRODUCTION

On request of several participants, the Institute for Interlaboratory Studies decided to organise new proficiency tests for the analysis of Ethyl Acetate and n-Butyl Acetate during the annual proficiency testing program 2014/2015.
In the interlaboratory study for Ethyl Acetate 11 laboratories in 8 different countries have participated. And in the interlaboratory study for n-Butyl Acetate 9 laboratories in 7 different countries have participated. See appendix 3 for the number of participants per country for each proficiency test.
In this report, the results of the 2015 Ethyl Acetate and the n-Butyl Acetate proficiency tests are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of these proficiency tests. The analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to use one sample per product. Depending on the registration one sample of Ethyl Acetate (0.5 litre bottle, labelled #15038) and/or one sample of n-Butyl Acetate (0.5 litre bottle, labelled #15039) was sent to the participants.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010 (R007). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Also customer's satisfaction is measured on regular basis by the distribution of questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website www.iisnl.com.

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

Ethyl Acetate

The necessary amount of bulk material of Ethyl Acetate was obtained from a local supplier. After homogenisation, a part of the 30 litre of Ethyl Acetate was divided over 30 brown glass bottles of 500 mL and labelled #15038.
The homogeneity of the subsamples #15038 was checked by determination of density in accordance with ASTM D4052 and water in accordance with ASTM D1364 on 4 stratified randomly selected samples.

	Density @20°C in kg/L	*Water in mg/kg*
sample #15038-1	0.90046	50
sample #15038-2	0.90046	39
sample #15038-3	0.90045	40
sample #15038-4	0.90046	41

table 1: homogeneity test results of subsamples #15038

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the target methods in agreement with the procedure of ISO 13528, Annex B2 in table 2.

	Density @20°C in kg/L	*Water in mg/kg*
r (sample #15038)	0.00001	14.2
reference test	D4052:02e1	ASTM D1364:02(2012)
0.3*R (reference test)	0.00015	39.1

table 2: repeatabilities of subsamples #15038

The calculated repeatabilities for density and water are in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, homogeneity of the subsamples was assumed.

n-Butyl Acetate

The necessary amount of bulk material of n-Butyl-Acetate was also obtained from a local supplier. After homogenisation, a part of the 30 litre n-Butyl Acetate was divided over 30 brown glass bottles of 500 mL and labelled #15039.
The homogeneity of the subsamples #15039 was checked by determination of density in accordance with ASTM D4052 and water in accordance with ASTM D1364 on 4 stratified randomly selected samples.

	Density @20°C in kg/L	*Water in mg/kg*
sample #15039-1	0.88129	84
sample #15039-2	0.88129	84
sample #15039-3	0.88129	79
sample #15039-4	0.88129	84

table 3: homogeneity test results of subsamples #15039

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the target methods in agreement with the procedure of ISO 13528, Annex B2 in table 2.

	Density @20°C in kg/L	*Water in mg/kg*
r (sample #15039)	0.00000	7.0
reference test	ASTM D4052:02e1	ASTM D1364:02(2012)
0.3*R (reference test)	0.00015	16.4

table 4: repeatabilities of subsamples #15039

The calculated repeatabilities for density and water are in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, homogeneity of the subsamples was assumed.

Depending on their registration to each of the participating laboratories one 0.5 litre bottle of Ethyl Acetate labelled #15038 and/or one 0.5 litre bottle of n-Butyl Acetate labelled #15039 was/were sent on March 25, 2015.

2.5 STABILITY OF THE SAMPLES

The stability of Ethyl Acetate and n-Butyl Acetate, packed in the brown glass bottles was checked. The material has been found stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine on the Ethyl Acetate sample #15038: Acidity as Acetic Acid, Color Pt/Co, Density at 20^{o}C, Specific Gravity 20/20°C, Distillation (IBP, 50% recovery, Dry Point, Distillation Range), Nonvolatile matter, Purity of Ethyl Acetate, Ethanol and Water.
On the n-Butyl Acetate sample #15039 was requested: Acidity as Acetic Acid, Color Pt/Co, Density at 20^{o}C, Specific Gravity 20/20°C, Distillation (IBP, 50% recovery, Dry Point, Distillation Range), Nonvolatile matter, Purity of n-Butyl Acetate, Butanol and Water.

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

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.
Directly after the deadline, a reminder fax was sent to those laboratories that had not yet reported any results at that moment.
Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

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

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<…' or '>…' were not used in the statistical evaluation.

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

According to ISO 5725 the original results per determination were submitted to Dixon’s and/or Grubbs' and/or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test (no. 15). Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are on the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This method is for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.13 and 14). Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8.

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

The z-scores were calculated according to:

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

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

	$\lvert z\rvert < 1$	good
$1 <$	$\lvert z\rvert < 2$	satisfactory
$2 <$	$\lvert z\rvert < 3$	questionable
$3 <$	$\lvert z\rvert$	unsatisfactory

4 EVALUATION

In this interlaboratory study, no problems with sample despatch were encountered.

For both samples #15038 (Ethyl Acetate) and #15039 (n-Butyl Acetate), one laboratory did not report any test results. And one laboratory reported test results after the final reporting date for sample #15039 (n-Butyl Acetate).
Finally, for sample #15038 (Ethyl Acetate) and sample #15039 (n-Butyl Acetate) in total 180 results were submitted. Observed were in total 3 outlying results, which is 1.7%. In proficiency studies, outlier percentages of 3% - 7.5% are normal.

4.1 EVALUATION PER TEST

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

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

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

For Ethyl Acetate sample #15038

Acidity as acetic acid: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility in good agreement with the requirements of ASTM D1613:06(2012).

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

Density at 20°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D4052:02e1.

Specific Gravity 20/20°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D4052:02e1.

Distillation: This determination was not problematic. One statistical outlier was observed for 50% recovered. The calculated reproducibilities are all in good agreement with the requirements of ASTM D1078:11 (automated and manual method).
The 50% recovered test results show that one participant probably did not correct the test result for barometric pressure and/or thermometer inaccuracy as described in ASTM D1078:11 (paragraph 13, Table 3).

Nonvolatile matter: All reported results are near or below the limit of detection.

Purity: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D3545:06(2012).

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

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

For n-Butyl Acetate sample #15039

Acidity as acetic acid: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers in good agreement with the requirements of ASTM D1613:06(2012).

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

Density at 20^{o}C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D4052:02e1.

Specific Gravity 20/20°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D4052:02e1.

Distillation: This determination was not problematic. No statistical outliers were observed. The calculated reproducibilities are all in agreement with the requirements of ASTM D1078:11 (automated and manual method).

Nonvolatile matter: This determination may be problematic. The calculated reproducibility is not in agreement with the requirements of ASTM D1353:13. The low number of reported test results may explain (part of) the large spread.

Purity: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D3545:06(2012).

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

Water: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements 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 standard and the reproducibility as found for the group of participating laboratories. The assigned values, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM, ISO, EN standards) are compared in the next table.

Parameter	unit	n	Mean	2.8 * sd	R (lit)
Acidity as acetic acid	mg/kg	10	14.4	5.3	14.0
Color Pt/Co		8	3	3	7
Density at 20°C	kg/L	10	0.9005	0.0003	0.0005
Specific Gravity 20/20°C		10	0.9021	0.0003	0.0005
Distillation, Initial Boiling Point	ºC	7	76.9	0.4	1.2
Distillation, 50% recovery	ºC	6	77.2	0.0	0.5
Distillation, Dry Point	ºC	7	77.3	0.3	0.8
Distillation, Distillation Range	ºC	7	0.4	0.3	0.7
Nonvolatile matter	mg/100 ml	8	<1	n.a.	n.a.
Purity of Ethyl Acetate	%M/M	8	99.961	0.028	0.090
Ethanol	mg/kg	8	42.3	15.7	10.8
Water, titrimetric	mg/kg	9	76.0	52.1	52.3

table 5: performance evaluation sample #15038

Parameter	unit	n	Mean	2.8 * sd	R (lit)
Acidity as acetic acid	mg/kg	6	10.8	1.6	14.0
Color Pt/Co		8	13	4	7
Density at 20°C	kg/L	8	0.8813	0.0003	0.0005
Specific Gravity 20/20°C		8	0.8829	0.0003	0.0005
Distillation, Initial Boiling Point	ºC	6	125.4	0.5	2.0
Distillation, 50% recovery	ºC	6	126.1	0.1	0.9
Distillation, Dry Point	ºC	6	126.5	0.3	1.4
Distillation, Distillation Range	ºC	6	1.0	0.6	0.8
Nonvolatile matter	mg/100 ml	5	1.8	1.7	0.8
Purity of n-Butyl Acetate	%M/M	8	99.808	0.164	0.200
n-Butanol	mg/kg	8	1066	325	167
Water, titrimetric	mg/kg	7	100.5	28.9	60.2

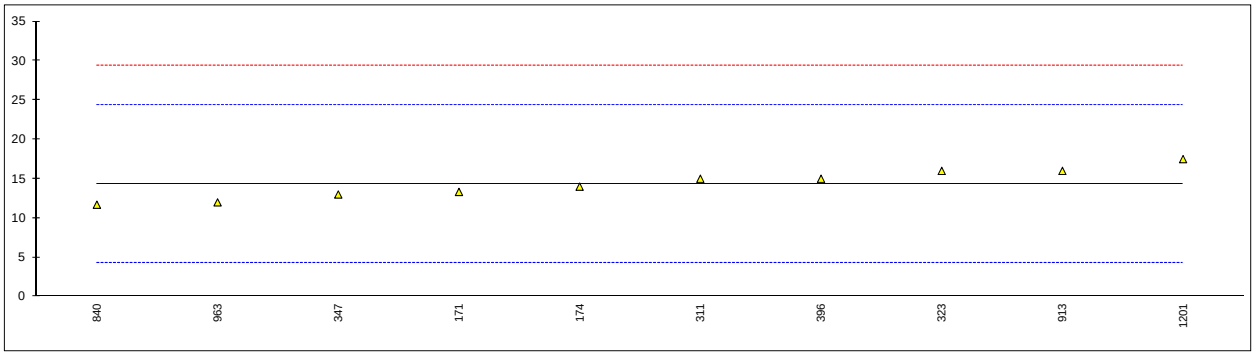
table 6: performance evaluation sample #15039

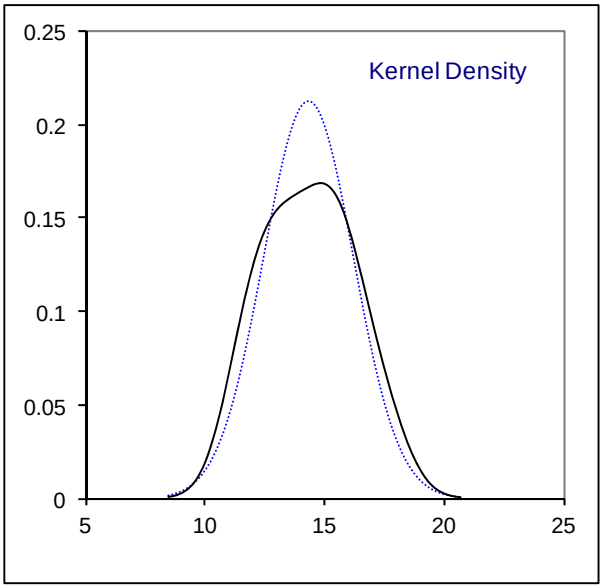
Without further statistical calculations it can be concluded that for a number of tests there is a good compliance of the group of participants with the relevant standards. The tests that are problematic have been discussed in paragraph 4.1.

APPENDIX 1

Determination of Acidity as Acetic Acid on Ethyl Acetate sample #15038; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1613	13.326		-0.21	
174	D1613	14		-0.07	
311	D1613	15		0.13	
323	D1613	16		0.33	
347	D1613	13		-0.27	
396	D1613	15	C	0.13	Probably unit error, reported: 0.0015
840	D1613	11.7		-0.53	
912		-----		-----	
913	D1613	16		0.33	
963	D1613	12		-0.47	
1201	D1613	17.5		0.63	
	normality	OK			
	n	10			
	outliers	0			
	mean (n)	14.35			
	st.dev. (n)	1.878			
	R(calc.)	5.26			
	R(D1613:06)	14.00			

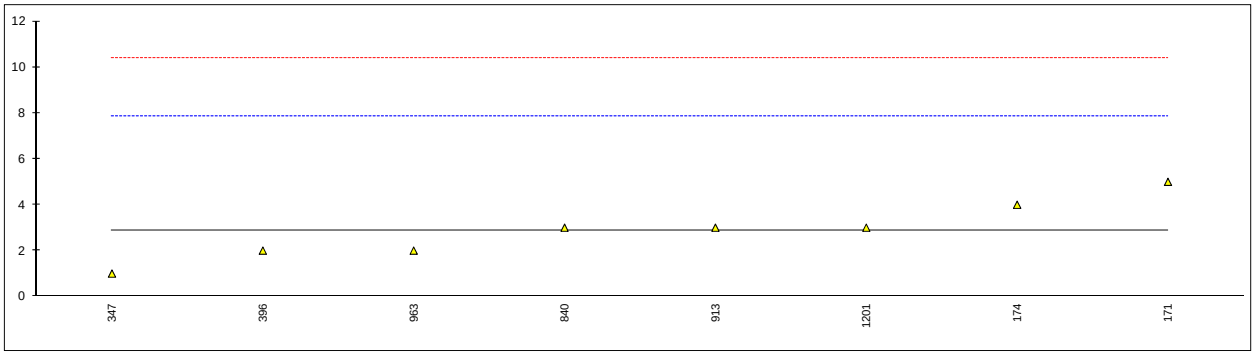


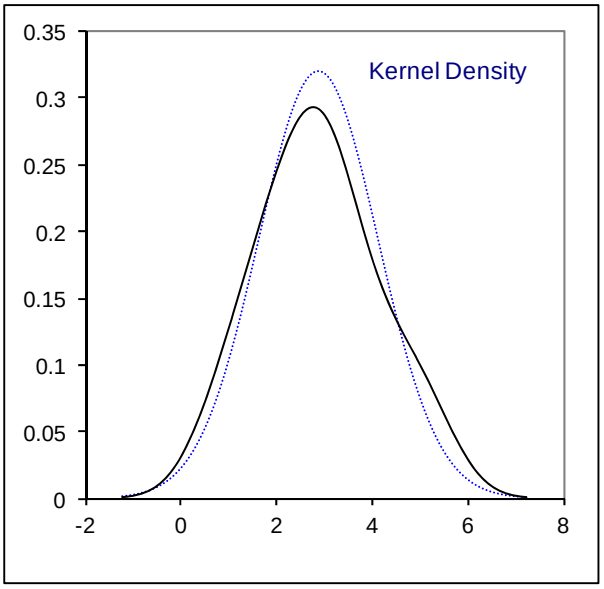


Determination of Color Pt/Co on Ethyl Acetate sample #15038

lab	method	value	mark	z(targ)	remarks
171	D1209	5		0.85	
174	D1209	4		0.45	
311	D1209	<5		-----	
323	D1209	<5		-----	
347	D5386	1		-0.75	
396	D1209	2		-0.35	
840	D1209	3		0.05	
912		-----		-----	
913	D5386	3		0.05	
963	D1209	2		-0.35	
1201	D5386	3		0.05	

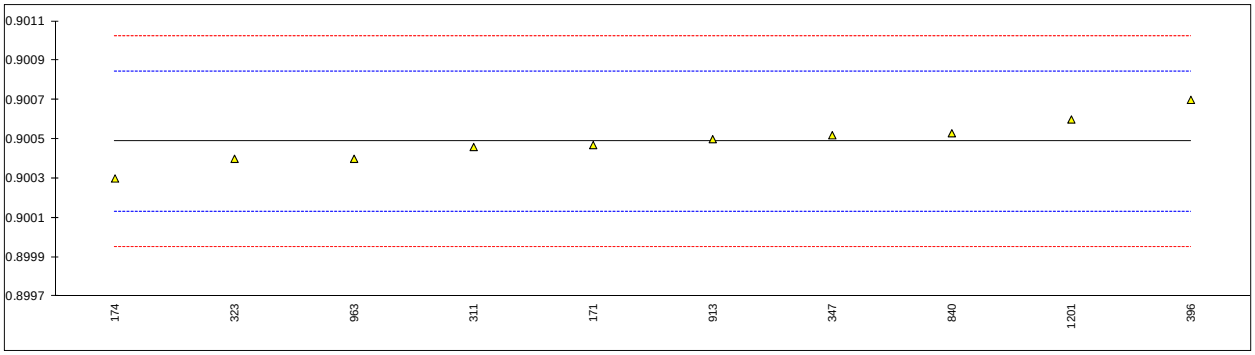
normality	unknown
n	8
outliers	0
mean (n)	2.9
st.dev. (n)	1.25
R(calc.)	3.5
R(D1209:05)	7.0

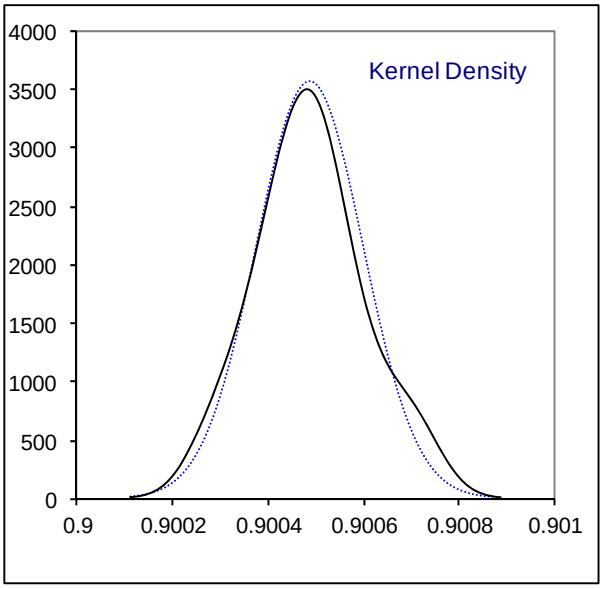




Determination of Density at 20 °C on Ethyl Acetate sample #15038; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.90047		-0.10	
174	D4052	0.9003		-1.05	
311	D4052	0.90046		-0.16	
323	D4052	0.9004		-0.49	
347	D4052	0.90052		0.18	
396	D4052	0.9007		1.19	
840	D4052	0.90053		0.24	
912		-----		-----	
913	D4052	0.9005		0.07	
963	D4052	0.9004		-0.49	
1201	D4052	0.9006		0.63	
	normality	OK			
	n	10			
	outliers	0			
	mean (n)	0.90049			
	st.dev. (n)	0.000112			
	R(calc.)	0.00031			
	R(D4052:02e1)	0.00050			

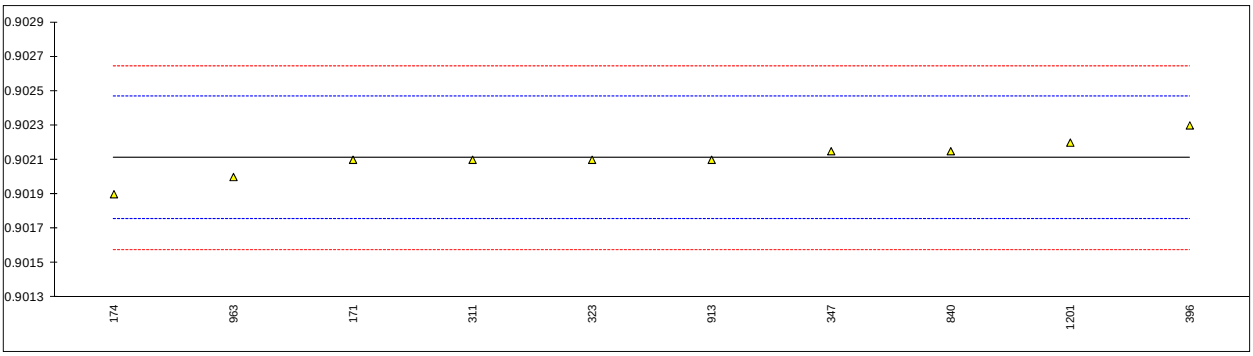


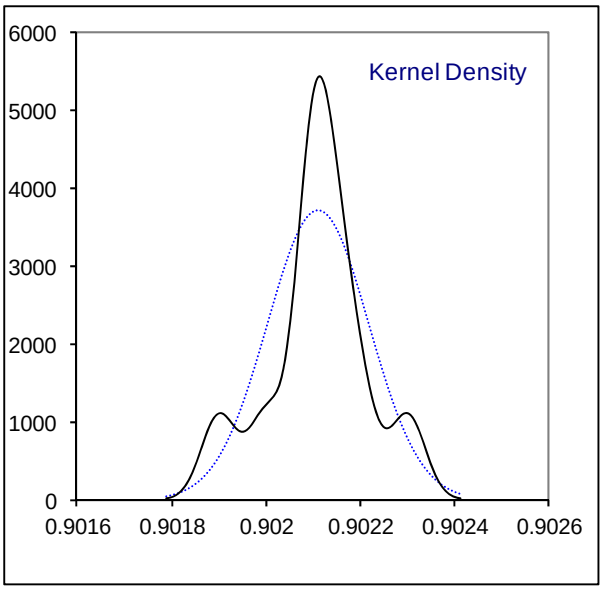


Determination of Specific Gravity 20/20 °C on sample #15038

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9021		-0.06	
174	D4052	0.9019		-1.18	
311	D4052	0.9021		-0.06	
323	D4052	0.9021		-0.06	
347	D4052	0.90215		0.22	
396	D4052	0.9023		1.06	
840	D4052	0.90215		0.22	
912		-----		-----	
913	D4052	0.9021		-0.06	
963	D4052	0.9020		-0.62	
1201	D4052	0.9022		0.50	

normality	suspect
n	10
outliers	0
mean (n)	0.90211
st.dev. (n)	0.000107
R(calc.)	0.00030
R(D4052:02e1)	0.00050





Determination of Distillation on Ethyl Acetate sample #15038; results in ºC

lab	method	mode	IBP	mark	50% rec	mark	DP	mark	Distil. Range	mark
171			-----		-----		-----		-----	
174	D1078	Automated	77.0		77.2		77.4		0.4	
311	D1078	Automated	76.9		77.2		77.3		0.4	
323	D1078	Manual	76.7		76.8	G(0.01)	77.1		0.4	
347			-----		-----		-----		-----	
396			-----		-----		-----		-----	
840	D1078	Automated	77.06		77.20		77.30		0.24	
912			-----		-----		-----		-----	
913	D1078	Manual	76.8		77.2		77.4		0.6	
963	D1078	Manual	77.0		77.2		77.3		0.3	
1201	D1078	Automated	77.0		77.2		77.3		0.3	
	normality		unknown		unknown		unknown		unknown	
	n		7		6		7		7	
	outliers		0		1		0		0	
	mean (n)		76.92		77.20		77.30		0.38	
	st.dev. (n)		0.130		0.000		0.100		0.117	
	R(calc.)		0.36		0.00		0.28		0.33	
	R(D1078:11)	Auto	1.20		0.53		0.83		0.70	
	R(D1078:11)	Manual	0.82		0.50		1.01		0.63	

Theoretical mid boiling point = 77.2ºC

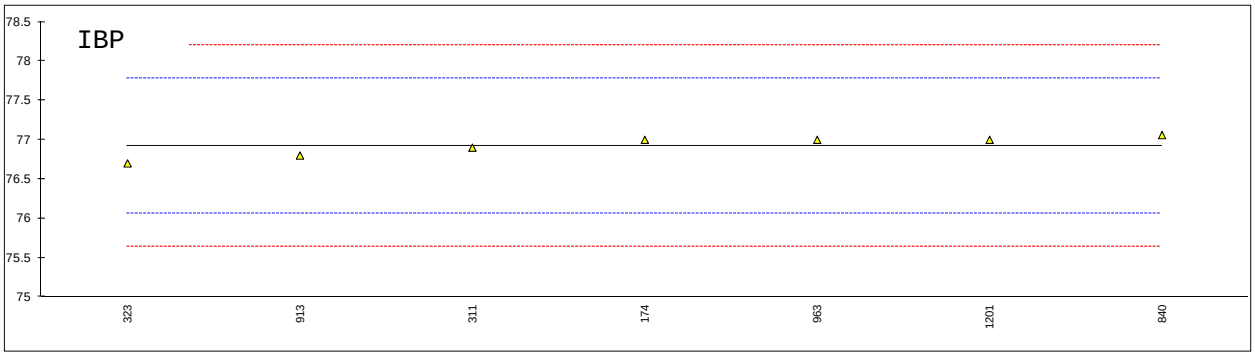
After manual correction:

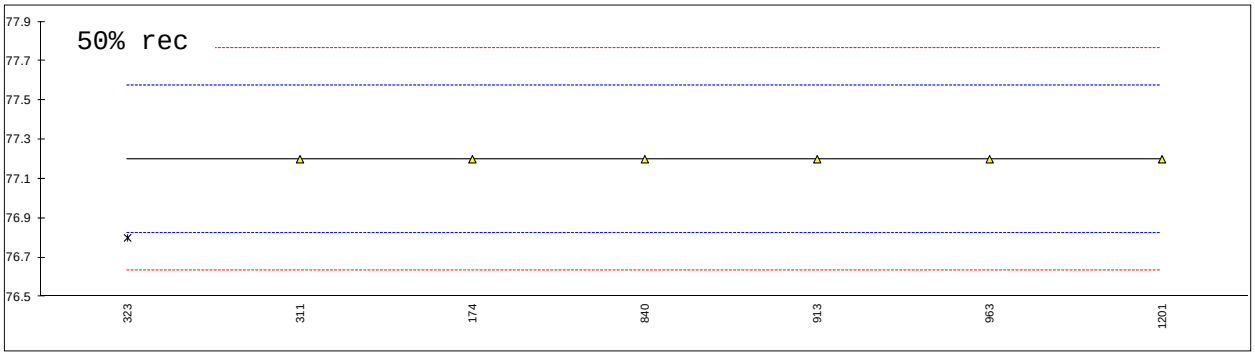
323	D1078	Manual	77.1		77.2		77.5			
	normality		unknown		unknown		unknown			
	n		7		7		7			
	outliers		0		0		0			
	mean (n)		76.98		77.21		77.36			
	st.dev. (n)		0.101		0.000		0.079			
	R(calc.)		0.28		0.00		0.22			
	R(D1078:11)	Auto	1.20		0.53		0.83			
	R(D1078:11)	Manual	0.82		0.50		1.01			

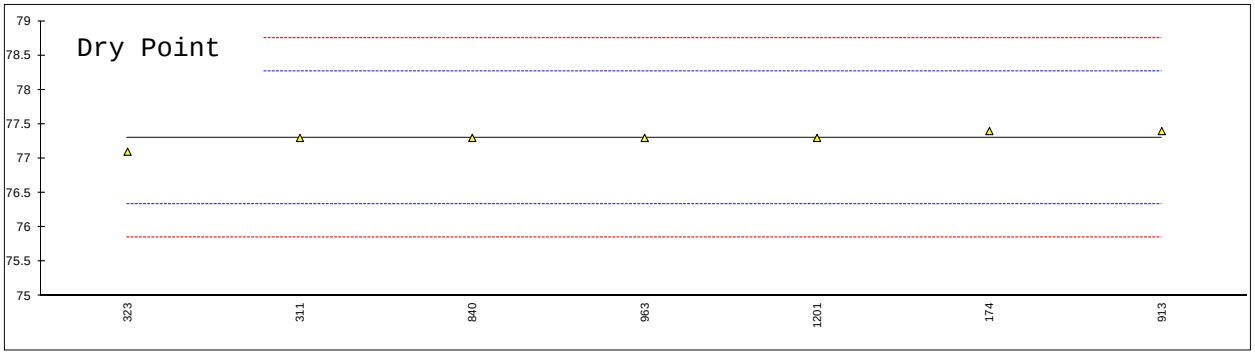
z-scores of Distillation on sample #15038

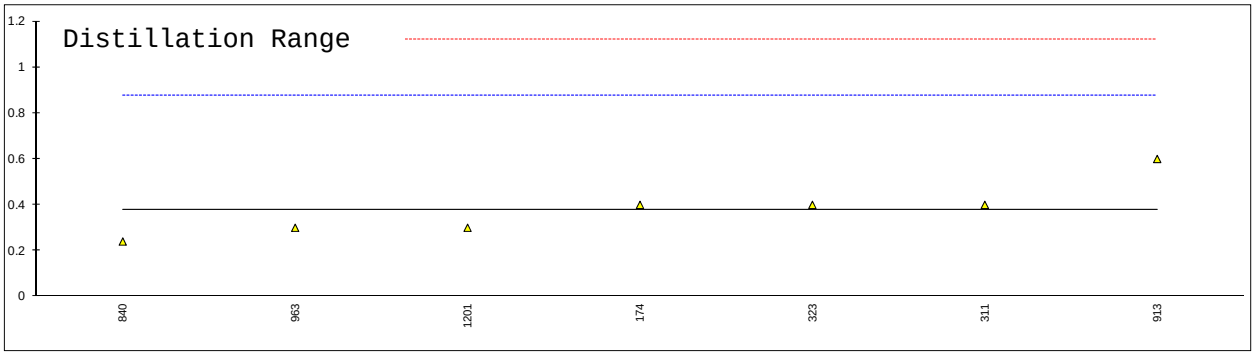
lab	IBP	50% rec	DP	Distil. Range
171	-----	-----	-----	-----
174	0.18	0.00	0.34	0.09
311	-0.05	0.00	0.00	0.09
323	-0.52	**-2.12**	-0.68	0.09
347	-----	-----	-----	-----
396	-----	-----	-----	-----
840	0.32	0.00	0.00	-0.55
912	-----	-----	-----	-----
913	-0.29	0.00	0.34	0.90
963	0.18	0.00	0.00	-0.31
1201	0.18	0.00	0.00	-0.31

Z-scores underlined and bold belong to the statistical outliers acc. to Grubbs/Dixon/Rosner outlier test.


IBP
78.5
78
77.5
77
76.5
76
75.5
75
323
913
311
174
963
1201
840

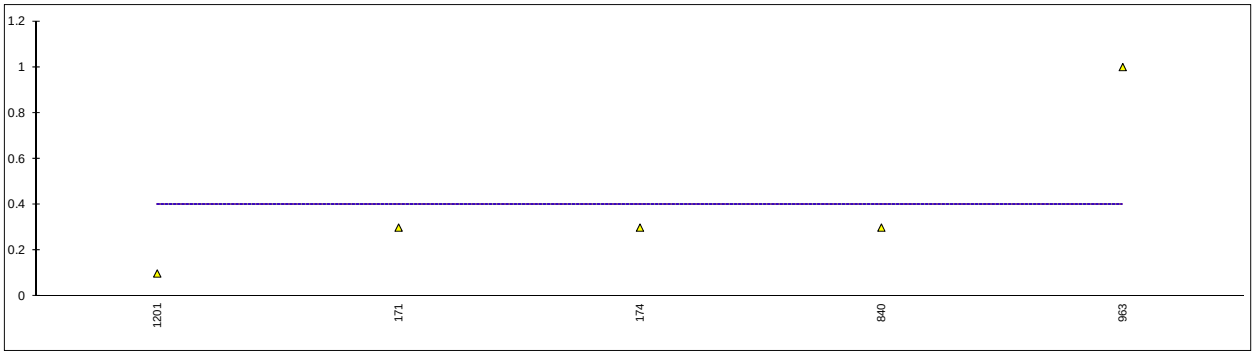

50% rec
77.9
77.7
77.5
77.3
77.1
76.9
76.7
76.5
323
311
174
840
913
963
1201


Dry Point
79
78.5
78
77.5
77
76.5
76
75.5
75
323
311
840
963
1201
174
913


Distillation Range
1.2
1
0.8
0.6
0.4
0.2
0
840
963
1201
174
323
311
913

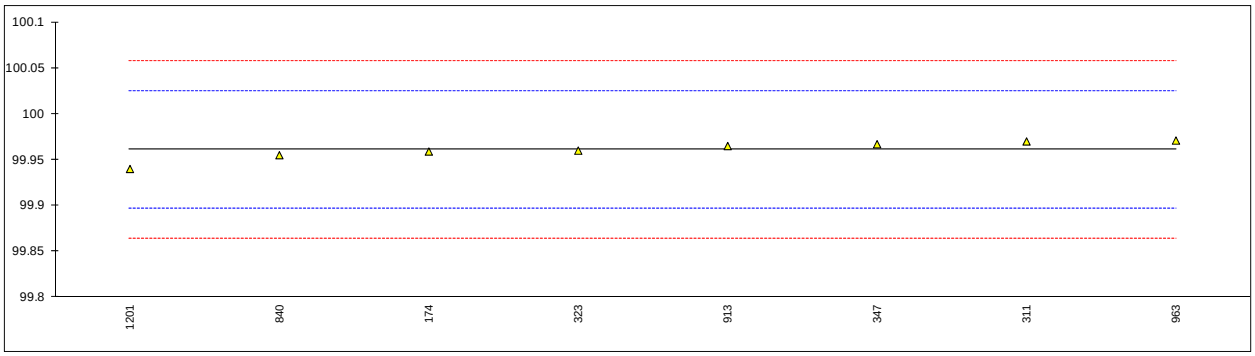
Determination of Nonvolatile matter on Ethyl Acetate sample #15038; results in mg/100ml

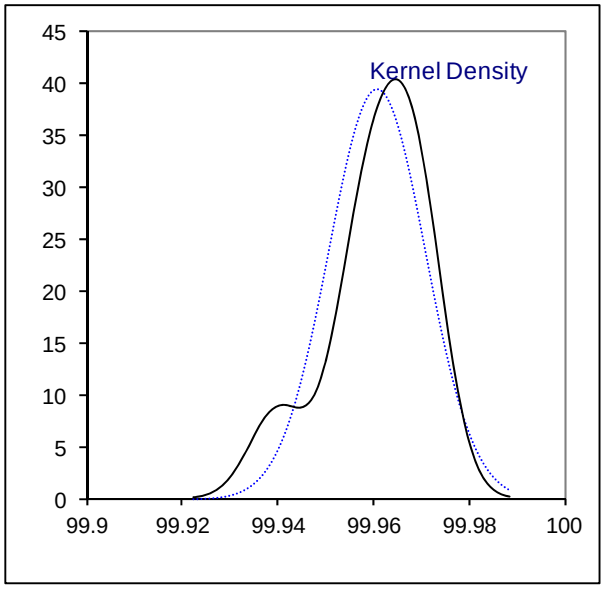
lab	method	value	mark	z(targ)	remarks
171	D1353	0.300		-----	
174	D1353	0.3		-----	
311	D1353	<1		-----	
323	D1353	<1		-----	
347	D1353	<1		-----	
396		-----		-----	
840	D1353	0.3		-----	
912		-----		-----	
913	D1353	<1.0		-----	
963	D1353	1.0		-----	
1201	D1353	0.1		-----	
	normality	n.a			
	n	8			
	outliers	n.a.			
	mean (n)	<1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a			
	R(D1353:13)	n.a			



Determination of Purity of Ethyl Acetate sample #15038; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3545	99.959		-0.06	
311	INH-650	99.97		0.28	
323	INH-110	99.96		-0.03	
347	D3545	99.967		0.19	
396		-----		-----	
840	D3545	99.955		-0.18	
912		-----		-----	
913	D3545	99.965		0.13	
963	D3545	99.971		0.32	
1201	D3545	99.94		-0.65	
	normality	unknown			
	n	8			
	outliers	0			
	mean (n)	99.9609			
	st.dev. (n)	0.01011			
	R(calc.)	0.0283			
	R(D3545:06)	0.0900			

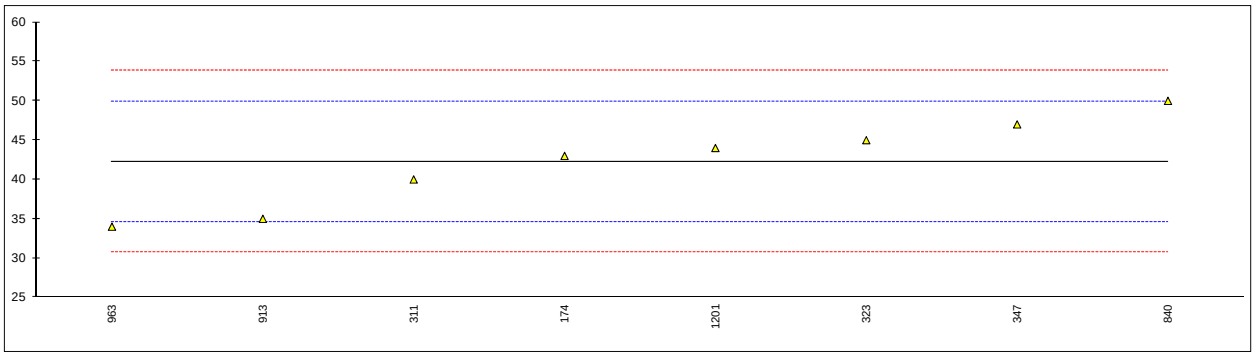


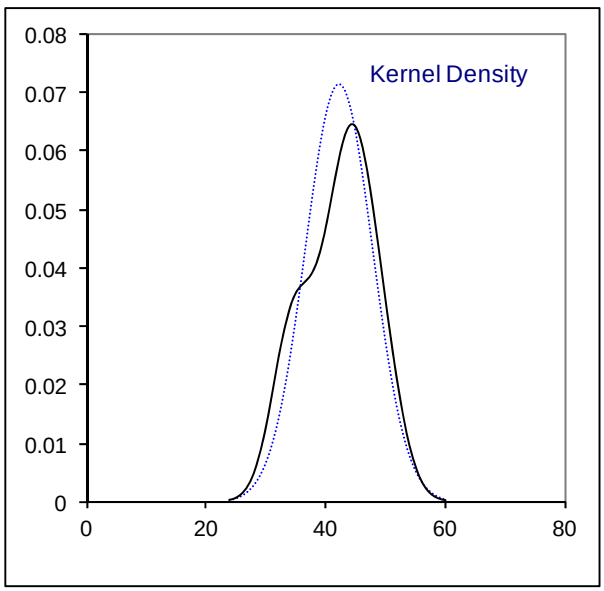


Determination of Ethanol on Ethyl Acetate sample #15038; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3545	43		0.19	
311	INH-650	40		-0.58	
323	INH-110	45		0.71	
347	D3545	47		1.23	
396		-----		-----	
840	D3545	50		2.01	
912		-----		-----	
913	D3545	35		-1.88	
963	D3545	34		-2.14	
1201	D3545	44		0.45	

normality	unknown
n	8
outliers	0
mean (n)	42.3
st.dev. (n)	5.60
R(calc.)	15.7
R(Horwitz)	10.8

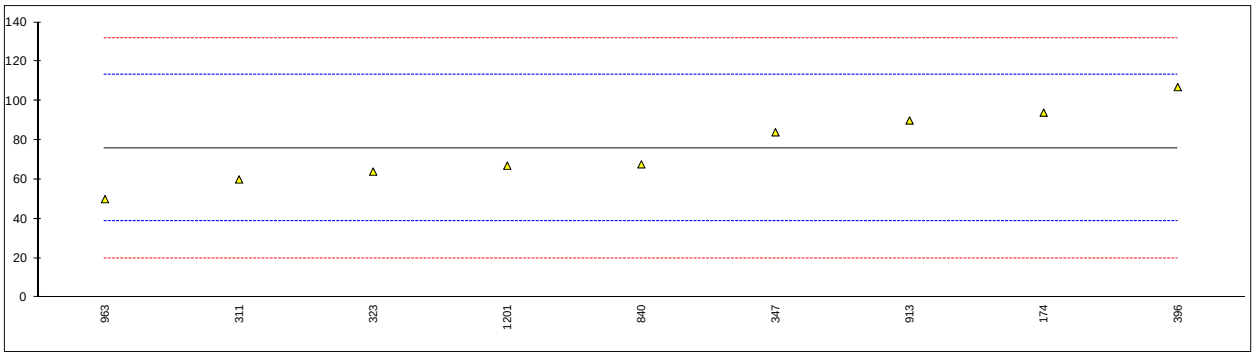


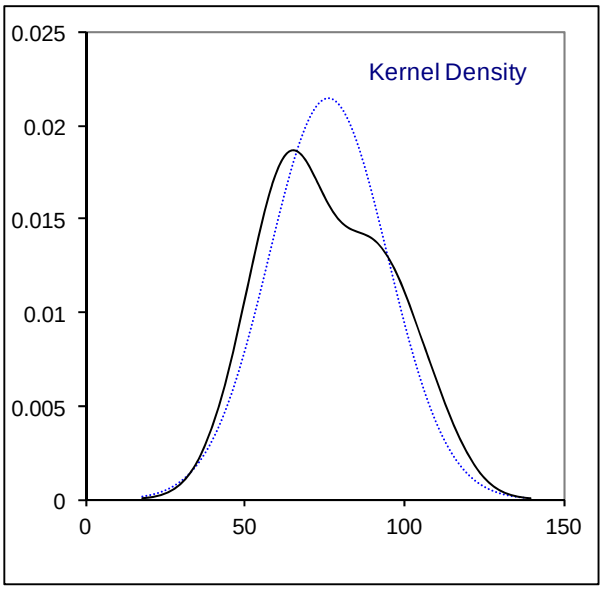


Determination of Water titrimetric on Ethyl Acetate, sample #15038; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D1364	94		0.97	
311	D1364	60		-0.85	
323	D1364	64		-0.64	
347	E1064	84		0.43	
396	D1364	107		1.66	
840	E1064	67.7		-0.44	
912		-----		-----	
913	D1364	90		0.75	
963	D1364	50		-1.39	
1201	E1064	67		-0.48	

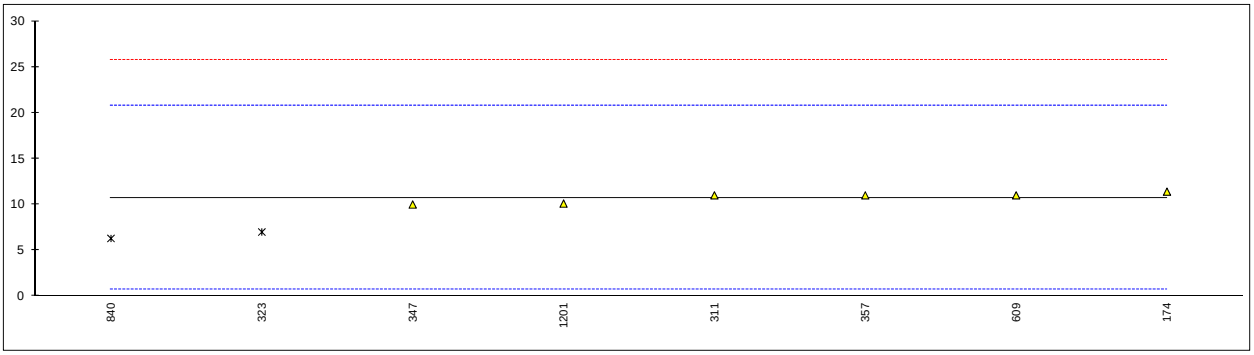
normality	OK
n	9
outliers	0
mean (n)	75.97
st.dev. (n)	18.611
R(calc.)	52.11
R(D1364:02)	52.30

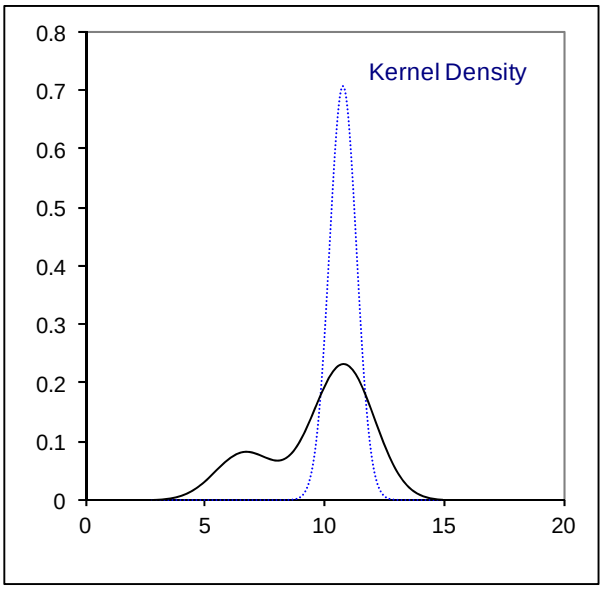




Determination of Acidity as Acetic Acid on n-Butyl Acetate sample #15039; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D1613	11.4		0.13	
311	D1613	11		0.05	
323	D1613	7	DG(0.05)	-0.75	
347	D1613	10		-0.15	
357	D1613	11	C	0.05	first reported: 0.0011
609	D1613	11	C	0.05	first reported: 45
840	D1613	6.3	DG(0.05)	-0.89	
1201	D1613	10.1		-0.13	
	normality	unknown			
	n	6			
	outliers	2			
	mean (n)	10.75			
	st.dev. (n)	0.565			
	R(calc.)	1.58			
	R(D1613:06)	14.00			

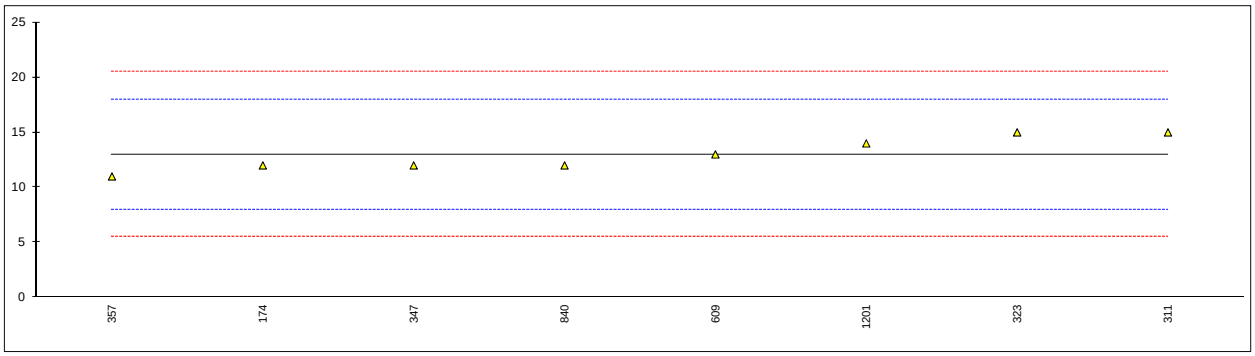


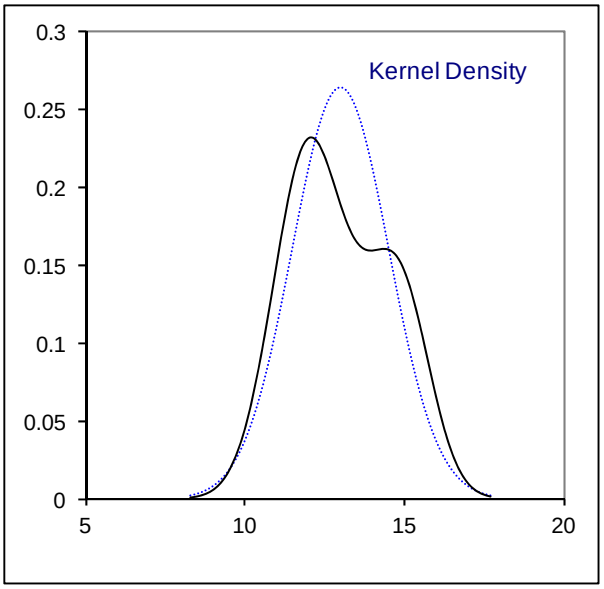


Determination of Color Pt/Co on n-Butyl Acetate sample #15039;

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D1209	12		-0.40	
311	D1209	15		0.80	
323	D1209	15		0.80	
347	D5386	12		-0.40	
357	D5386	11		-0.80	
609	INH-03	13		0.00	
840	D1209	12		-0.40	
1201	D5386	14		0.40	

normality	unknown
n	8
outliers	0
mean (n)	13.0
st.dev. (n)	1.51
R(calc.)	4.2
R(D1209:05)	7.0

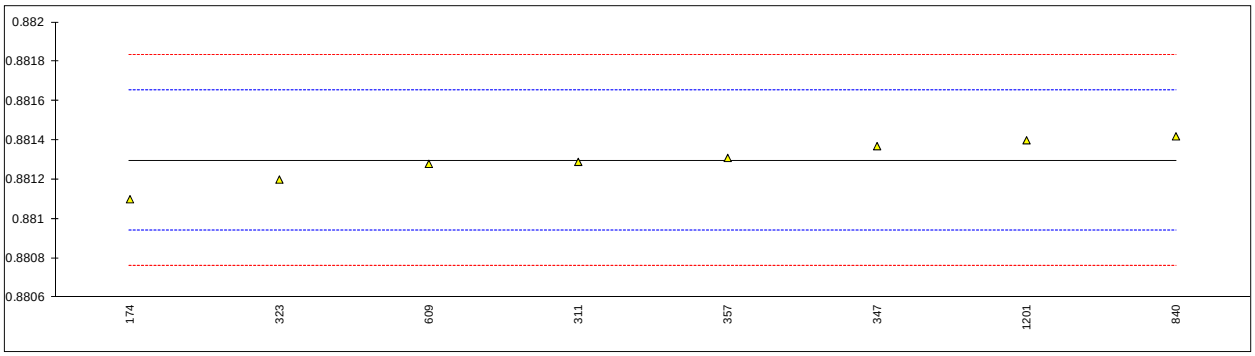


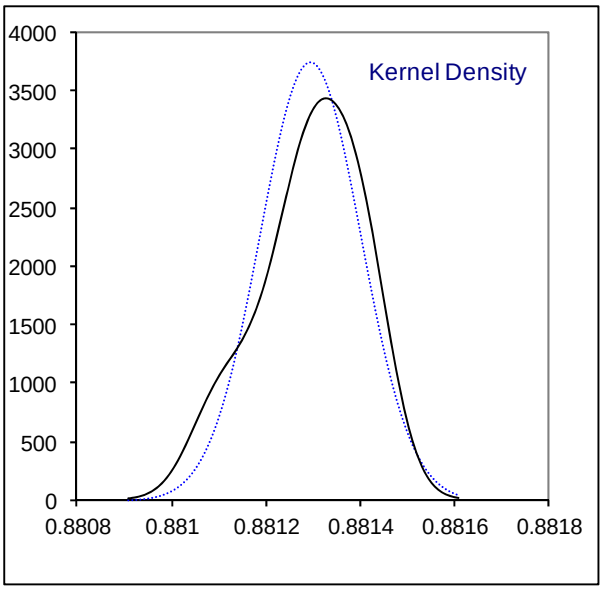


Determination of Density at 20 °C on n-Butyl Acetate sample #15039; results in kg/L

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D4052	0.8811		-1.10	
311	D4052	0.88129		-0.04	
323	D4052	0.8812		-0.54	
347	D4052	0.88137		0.41	
357	D4052	0.88131		0.08	
609	D4052	0.88128		-0.09	
840	D4052	0.88142		0.69	
1201	D4052	0.8814		0.58	

normality	unknown
n	8
outliers	0
mean (n)	0.88130
st.dev. (n)	0.000107
R(calc.)	0.00030
R(D4052:02e1)	0.00050

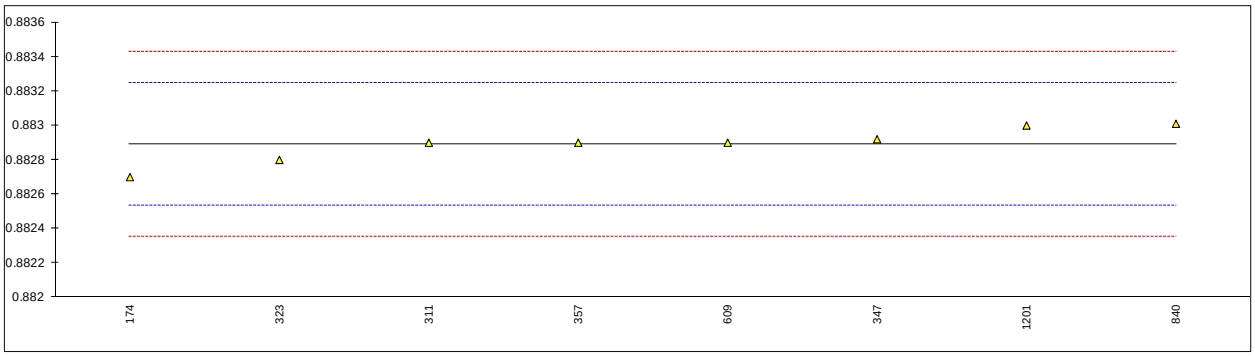


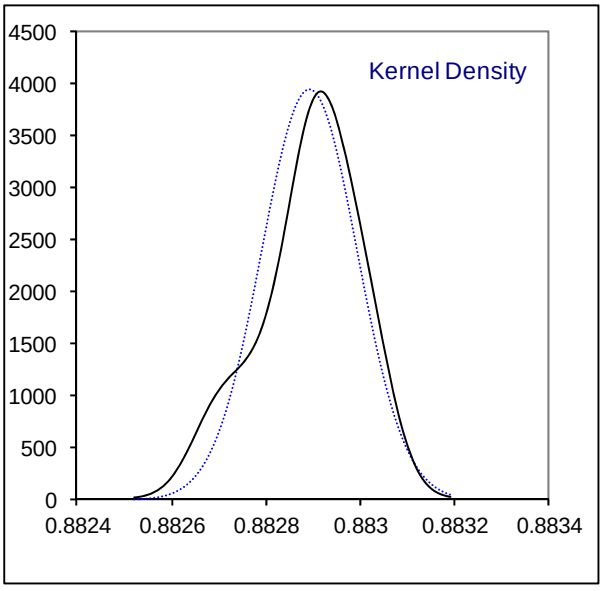


Determination of Specific Gravity 20/20 °C on n-Butyl Acetate, sample #15039

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D4052	0.8827		-1.07	
311	D4052	0.8829		0.05	
323	D4052	0.8828		-0.51	
347	D4052	0.88292		0.16	
357	D4052	0.88290		0.05	
609	D4052	0.8829		0.05	
840	D4052	0.88301		0.67	
1201	D4052	0.8830		0.61	

normality	unknown
n	8
outliers	0
mean (n)	0.88289
st.dev. (n)	0.000101
R(calc.)	0.00028
R(D4052:02e1)	0.00050





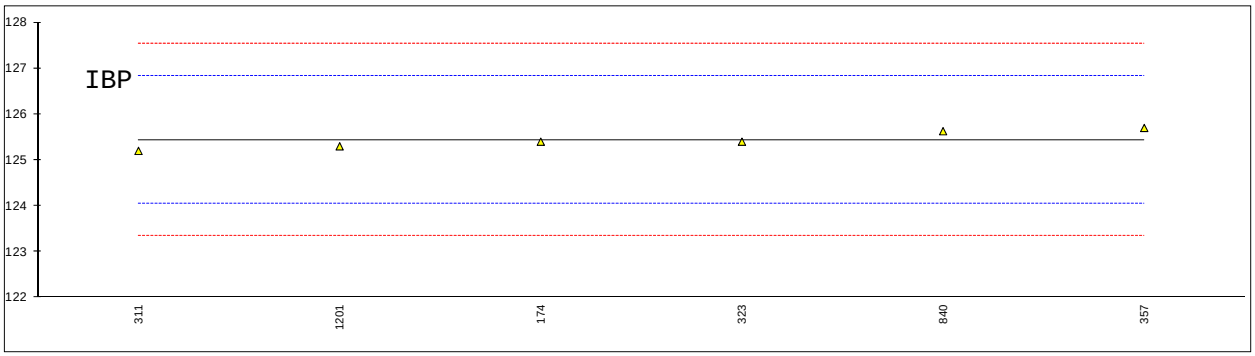
Determination of Distillation on n-Butyl Acetate sample #15039; results in ºC

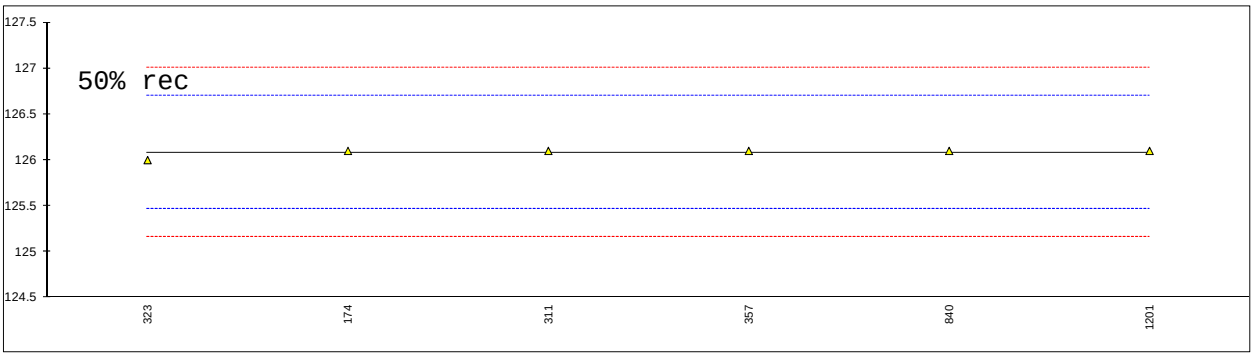
lab		mode	IBP	mark	50%	mark	DP	mark	Distil. Range	mark
171			-----		-----		-----		-----	
174	D1078	Automated	125.4		126.1		126.5		1.1	
311	D1078	Automated	125.2		126.1		126.4		1.2	
323	D1078	Manual	125.4		126.0		126.4		1.0	
347			-----		-----		-----		-----	
357	D1078	Automated	125.7		126.1		126.5		0.8	
609			-----		-----		-----		-----	
840	D1078	Automated	125.63		126.10		126.33		0.70	
1201	D1078	Automated	125.3		126.1		126.6		1.2	
	normality	.	unknown		unknown		unknown		unknown	
	n		6		6		6		6	
	outliers		0		0		0		0	
	mean (n)		125.44		126.08		126.45		1.00	
	st.dev. (n)		0.192		0.041		0.097		0.210	
	R(calc.)		0.54		0.11		0.27		0.59	
	R(D1078:11)	Auto	1.96		0.86		1.35		0.78	
	R(D1078:11)	Manual	1.34		0.82		1.65		0.70	

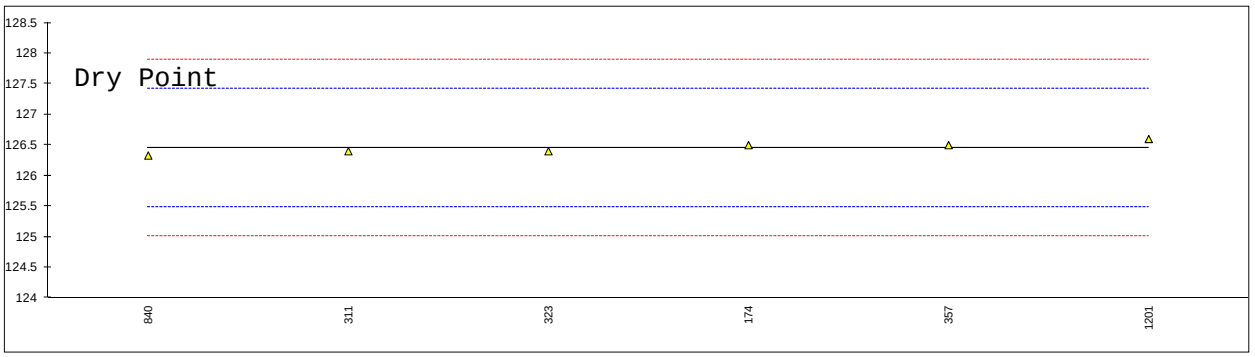
Theoretical mid boiling point = 126.1 ºC

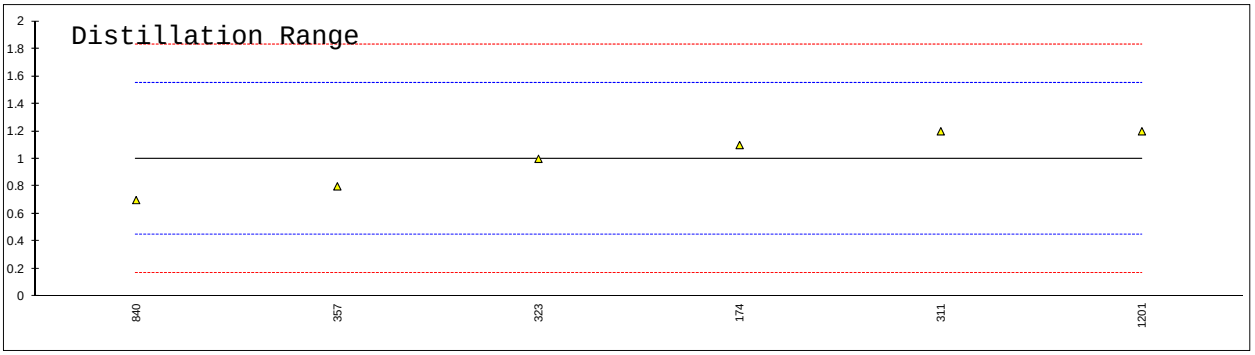
z-scores of Distillation on sample #15039

lab	IBP	50% rec	DP	Distil. Range
171	-----	-----	-----	-----
174	-0.05	0.05	0.09	0.36
311	-0.34	0.05	-0.11	0.72
323	-0.05	-0.27	-0.11	0.00
347	-----	-----	-----	-----
357	0.37	0.05	0.09	-0.72
609	-----	-----	-----	-----
840	0.27	0.05	-0.26	-1.08
1201	-0.20	0.05	0.30	0.72


IBP
128
127
126
125
124
123
122
311
1201
174
323
840
357


50% rec
127.5
127
126.5
126
125.5
125
124.5
323
174
311
357
840
1201

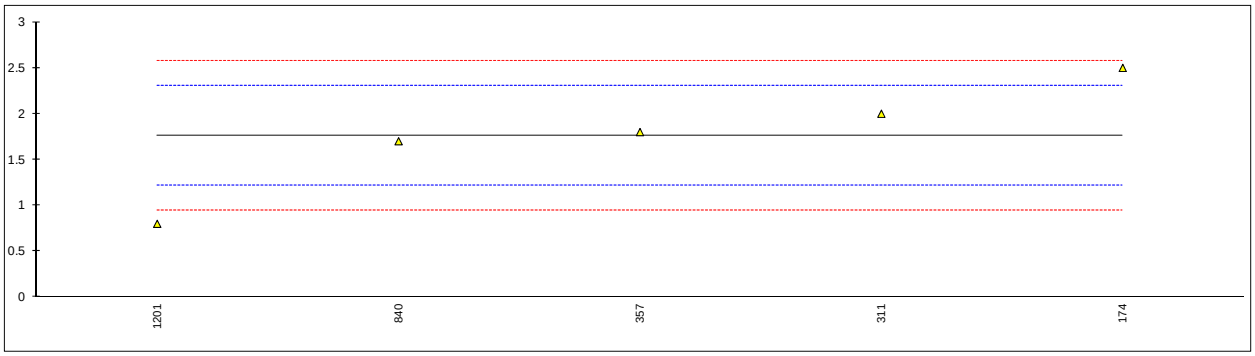

Dry Point
128.5
128
127.5
127
126.5
126
125.5
125
124.5
124
840
311
323
174
357
1201


Distillation Range
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
840
357
323
174
311
1201

Determination of Nonvolatile matter on n-Butyl Acetate sample #15039; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D1353	2.5		2.73	
311	D1353	2		0.88	
323		-----		-----	
347		-----		-----	
357	D1353	1.8		0.15	
609		-----		-----	
840	D1353	1.7		-0.22	
1201	D1353	0.8		-3.54	

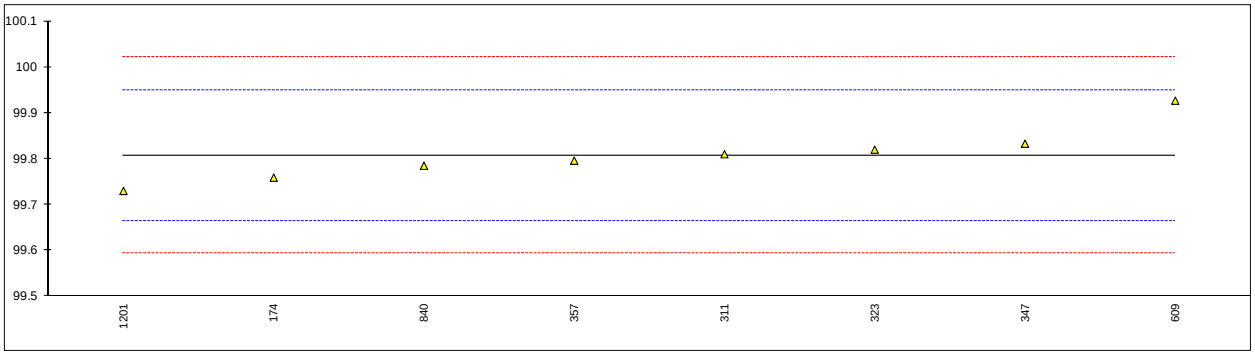
normality	unknown
n	5
outliers	n.a.
mean (n)	1.76
st.dev. (n)	0.619
R(calc.)	1.73
R(D1353:13)	0.76

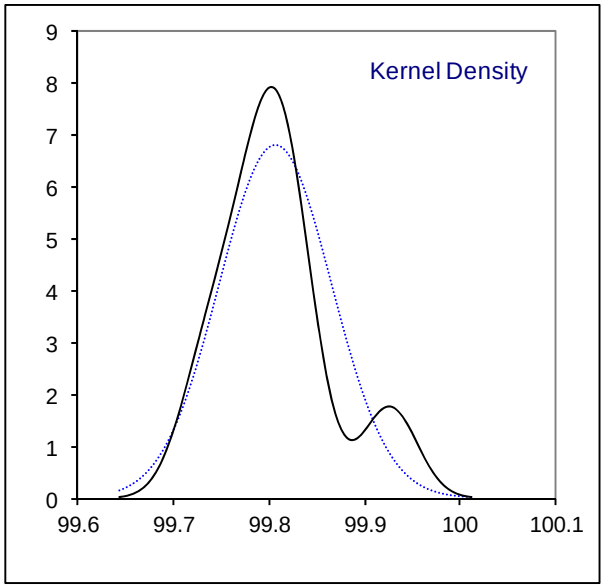


Determination of Purity of n-Butyl Acetate on sample #15039; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3545	99.759		-0.68	
311	INH-172	99.81		0.04	
323	D3545	99.82		0.18	
347	D3545	99.833		0.36	
357	D3545	99.796		-0.16	
609	INH-01	99.9269		1.67	
840	D3545	99.785		-0.31	
1201	D3545	99.73		-1.08	

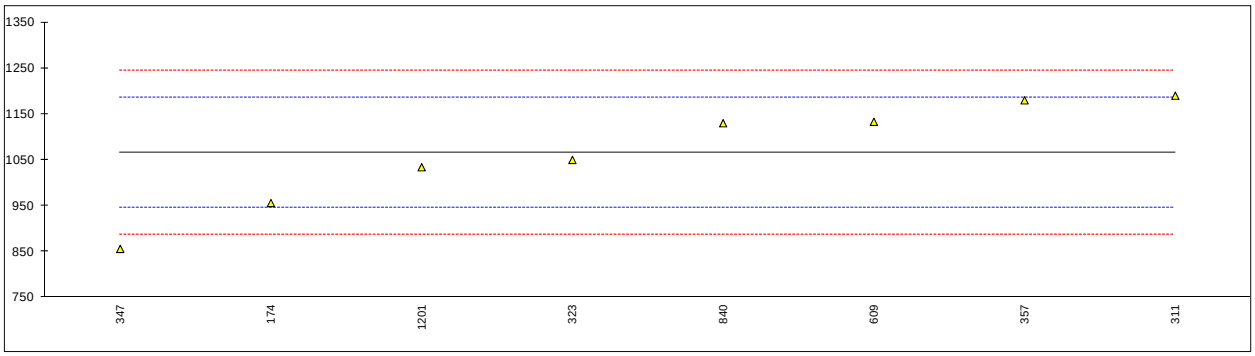
normality	unknown
n	8
outliers	0
mean (n)	99.8075
st.dev. (n)	0.05863
R(calc.)	0.1642
R(D3545:06)	0.2000

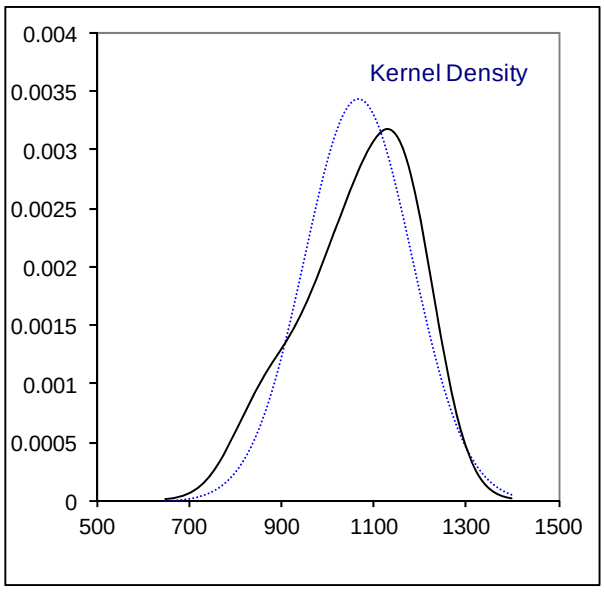




Determination of n-Butanol on n-Butyl Acetate sample # 15039; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3545	956		-1.84	
311	INH-172	1190		2.07	
323	D3545	1050		-0.27	
347	D3545	856		-3.52	
357	D3545	1180		1.91	
609	INH-01	1133	C	1.12	first reported: 634
840	D3545	1130		1.07	
1201	D3545	1034		-0.54	
	normality	unknown			
	n	8			
	outliers	0			
	mean (n)	1066.1			
	st.dev. (n)	116.15			
	R(calc.)	325.2			
	R(Horwitz)	167.3			

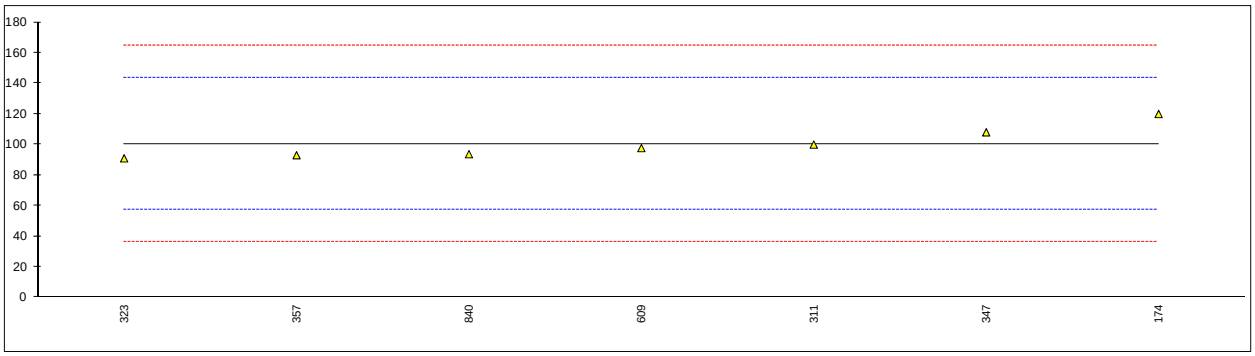




Determination of Water titrimetric on n-Butyl Acetate, sample #15039; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D1364	120		0.91	
311	D1364	100		-0.02	
323	D1364	91		-0.44	
347	E1064	108		0.35	
357	E203	93		-0.35	
609	INH-02	97.695		-0.13	
840	E1064	93.7		-0.32	
1201		-----	W	-----	result withdrawn, first reported: 187

normality	unknown
n	7
outliers	0
mean (n)	100.49
st.dev. (n)	10.307
R(calc.)	28.86
R(D1364:02)	60.15



APPENDIX 2

Number of participants in the Ethyl Acetate PT

1 lab in BELGIUM

2 labs in INDIA

1 lab in ITALY

2 labs in NETHERLANDS

1 lab in SAUDI ARABIA

1 lab in SPAIN

2 labs in UNITED STATES OF AMERICA

1 lab in VIETNAM

Number of participants in the n-Butyl Acetate PT

1 lab in BELGIUM

1 lab in SPAIN

1 lab in FINLAND

1 lab in MALAYSIA

1 lab in VIETNAM

2 labs in UNITED STATES OF AMERICA

2 labs in NETHERLANDS

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
E	= error in calculations
ex	= excluded from calculations
n.e.	= not evaluated
wd	= withdrawn method

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