

**Results of Proficiency Test
n-Butyl Acetate & Ethyl Acetate
April 2017**

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis17C06 & iis17C07

July 2017

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1 INTRODUCTION

In 2015 Institute for Interlaboratory Studies organised two new proficiency tests for the analyses of n-Butyl Acetate and Ethyl Acetate on request of several participants. During the annual proficiency testing program 2016/2017 it was decided to continue with the round robins for the analyses of n-Butyl Acetate and Ethyl Acetate.

In the interlaboratory studies for n-Butyl Acetate 12 laboratories in 11 different countries and for Ethyl Acetate 14 laboratories in 11 different countries did register for participation. See appendix 2 for the number of participants per country for each proficiency test.

In this report, the results of the 2017 proficiency tests n-Butyl Acetate and Ethyl Acetate 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 the proficiency tests (PTs). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided, depending on the registration, to send one sample of n-Butyl Acetate (0.5 litre bottle, labelled #17060) and/or one sample of Ethyl Acetate (0.5 litre bottle, labelled #17061). 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 organisation of the proficiency tests was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

n-Butyl Acetate

The necessary bulk material of n-Butyl Acetate was obtained from a local chemical supplier. After homogenisation, 24 amber glass bottles of 0.5 litre were filled and labelled #17060. The homogeneity of the subsamples #17060 was checked by determination of Density at 20°C in accordance with ISO12185 and Water in accordance with ASTM D1364 on 4 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in mg/kg
sample #17060-1	0.88126	160
sample #17060-2	0.88125	160
sample #17060-3	0.88126	160
sample #17060-4	0.88126	160

Table 1: homogeneity test results of subsamples #17060

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

	Density at 20°C in kg/L	Water in mg/kg
r (observed)	0.00001	0.0
Reference test method	ASTM D4052:16	ASTM D1364:02(2012)
0.3*R (reference test method)	0.00015	22.8

Table 2: evaluation of the repeatabilities of subsamples #17060

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

Ethyl Acetate

The necessary bulk material of Ethyl Acetate was obtained from a local chemical supplier. After homogenisation, 24 amber glass bottles of 0.5 litre were filled and labelled #17061. The homogeneity of the subsamples #17061 was checked by determination of Density at 20°C in accordance with ISO12185 and Water in accordance with ASTM D1364 on 4 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in mg/kg
sample #17061-1	0.90048	60
sample #17061-2	0.90047	70
sample #17061-3	0.90048	60
sample #17061-4	0.90048	60

Table 3: homogeneity test results of subsamples #17061

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

	Density at 20°C in kg/L	Water in mg/kg
r (observed)	0.00001	14.0
Reference test method	ASTM D4052:16	ASTM D1364:02(2012)
0.3*R (reference test method)	0.00015	14.2

Table 4: evaluation of the repeatabilities of subsamples #17061

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

Depending on the registration to each of the participating laboratories one 0.5 litre bottle of n-Butyl Acetate labelled #17060 and/or one 0.5 litre bottle of Ethyl Acetate labelled #17061 was sent on April 5, 2017. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of n-Butyl Acetate and Ethyl 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 n-Butyl Acetate sample #17060: Acidity as Acetic Acid, Color Pt/Co, Density at 20°C, Specific Gravity 20/20°C, Distillation (IBP, 50% recovery, Dry Point and Distillation Range), Nonvolatile matter, Purity of n-Butyl Acetate, n-Butanol and Water.

On Ethyl Acetate sample #17061 was requested: Acidity as Acetic Acid, Color Pt/Co, Density at 20°C, Specific Gravity 20/20°C, Distillation (IBP, 50% recovery, Dry Point, and Distillation Range), Nonvolatile matter, Purity of Ethyl Acetate, Ethanol and Water.

It was explicitly requested to treat the samples as if they were routine samples 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 calculations.

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

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

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

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...>' or '>...>' were not used in the statistical evaluation.

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

According to ISO 5725 the original test results per determination were submitted to Dixon's 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. 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 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 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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

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

This 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

The z-scores were calculated according to:

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

The $Z_{(\text{target})}$ scores are listed in the result tables of 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 interlaboratory study, some problems were encountered with dispatch of the samples. All participants timely received sample #17060 (n-Butyl Acetate). However, participants in Brazil and Saudi Arabia received sample #17061 (Ethyl Acetate) late or not at all and did not report any test results.

Finally, for sample #17060 (n-Butyl Acetate) and sample #17061 (Ethyl Acetate) in total 246 numerical test results were reported by 15 participants. Observed were 11 outlying results, which is 4.5% of the total of numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER SAMPLE AND PER TEST

In this section, the reported test results are discussed per sample and 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 methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

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

In the iis PT reports, ASTM 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(2011)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D1209:05 will be used.

For n-Butyl Acetate sample #17060

Acidity as acetic acid: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D1613:17.

Color Pt/Co: The determination was not problematic. No statistical outliers were observed. 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. The calculated reproducibility is in agreement with the requirements of ASTM D4052:16.

Specific Gravity 20/20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D4052:16.

Distillation: The determination of the Initial Boiling Point (IBP) may be problematic depending on the test method used as reference method. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1078:11 - automated method, but is not in agreement with the requirements of ASTM D1078:11 - manual method.

The determination of the Temperature at 50% Recovery (50% Rec) and the Dry Point (DP) was not problematic. No statistical outliers were observed. The calculated reproducibilities are in agreement with the requirements of ASTM D1078:11 (automated and manual method).

The determination of the Distillation range (Distil. Range) 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 D1078:11 (automated and manual method).

Nonvolatile matter: 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 D1353:13.

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

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

Water: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D1364:02(2012).

For Ethyl Acetate sample #17061

Acidity as acetic acid: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D1613:17.

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(2011).

Density at 20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D4052:16.

Specific Gravity 20/20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D4052:16.

Distillation: This determination of Initial Boiling Point (IBP), Temperature at 50% Recovery (50% Rec) and Dry Point (DP) was not problematic. One statistical outlier was observed for IBP, also one for 50% Recovered and two statistical outliers were observed for DP. However, the calculated reproducibilities after rejection of the statistical outliers are all in agreement with the requirements of ASTM D1078:11 (automated and manual method).

From the reported test results of the 50% recovered, it appears 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).

The determination of the Distillation range (Distil. Range) was not problematic. No statistical outliers were observed and one test result was excluded. However, the calculated reproducibility after rejection of the suspect data is good in agreement with the requirements of ASTM D1078:11 (automated and manual method).

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

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

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

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full 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 average results of the evaluated parameters, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM, ISO, EN standards) are compared in the next tables.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as acetic acid	mg/kg	11	19.8	18.9	14
Color Pt/Co		12	22	6	7
Density at 20°C	kg/L	12	0.8813	0.0002	0.0005
Specific Gravity 20/20°C		11	0.8828	0.0001	0.0005
Distillation, Initial Boiling Point	°C	8	125.0	1.6	2.0
Distillation, 50% recovery	°C	8	126.1	0.0	0.9
Distillation, Dry Point	°C	8	126.2	0.3	1.4
Distillation, Distillation Range	°C	7	1.1	0.9	0.8
Nonvolatile matter	mg/100 ml	8	7.4	3.9	3.2
Purity of n-Butyl Acetate	%M/M	10	99.801	0.182	0.2
n-Butanol	mg/kg	9	1106.6	207.3	172.6 *)
Water, titrimetric	mg/kg	11	156.3	43.6	75.0

Table 5: reproducibilities of tests on sample #17060

*) against the Horwitz equation

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as acetic acid	mg/kg	10	11.4	11.2	14
Color Pt/Co		10	3	3	7
Density at 20°C	kg/L	12	0.9005	0.0002	0.0005
Specific Gravity 20/20°C		12	0.9021	0.0001	0.0005
Distillation, Initial Boiling Point	°C	10	76.9	0.3	1.2
Distillation, 50% recovery	°C	10	77.2	0.1	0.5
Distillation, Dry Point	°C	9	77.3	0.2	0.8
Distillation, Distillation Range	°C	10	0.4	0.5	0.7
Nonvolatile matter	mg/100 ml	12	<1	n.a.	n.a.
Purity of Ethyl Acetate	%M/M	9	99.972	0.024	0.09
Ethanol	mg/kg	7	39	8	10 *)
Water, titrimetric	mg/kg	12	70.8	51.5	50.5

Table 6: reproducibilities of tests on sample #17061

*) against the Horwitz equation

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.

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

	April 2017	April 2015
Total Number of reporting labs	15	12
Number of results reported	246	180
Statistical outliers	11	3
Percentage outliers	4.5%	1.7%

Table 7: comparison with previous proficiency test.

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following tables:

	April 2017	April 2015
Acidity as acetic acid	-	++
Color Pt/Co	+/-	+
Density at 20°C	++	+
Specific Gravity 20/20°C	++	+
Distillation, IBP	+	++
Distillation, 50%	++	++
Distillation, DP	++	++
Distillation Range	+/-	+
Nonvolatile matter	-	--
Purity	+/-	+
n-Butanol	-	-
Water	+	++

Table 8: comparison determinations on n-Butyl Acetate against the standards

	April 2017	April 2015
Acidity as acetic acid	+	++
Color Pt/Co	++	++
Density at 20°C	++	+
Specific Gravity 20/20°C	++	+
Distillation, IBP	++	++
Distillation, 50%	++	++
Distillation, DP	++	++
Distillation Range	+	++
Nonvolatile matter	n.e.	n.e.
Purity	++	++
Ethanol	+	-
Water	+/-	+/-

Table 9: comparison determinations on Ethyl Acetate against the standards

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

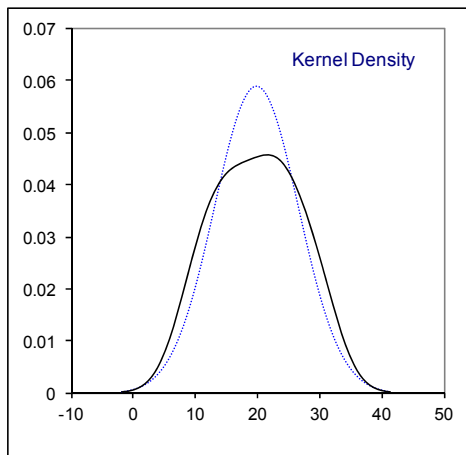
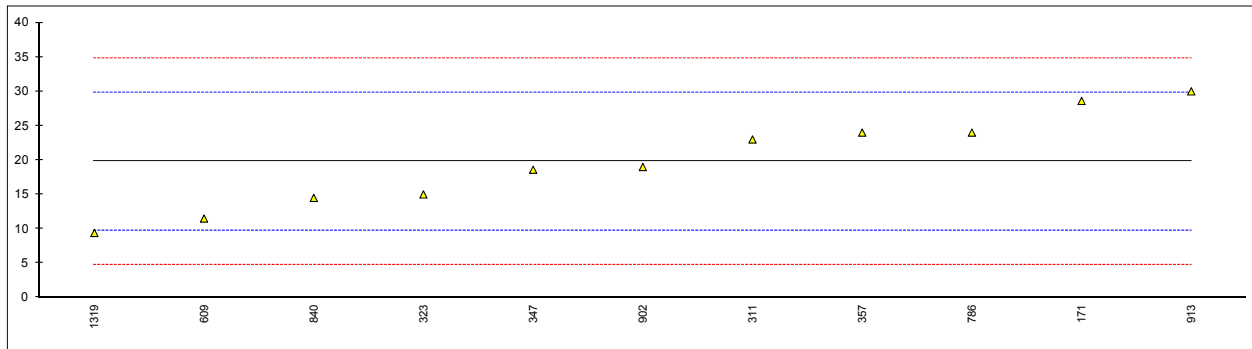
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

APPENDIX 1

Determination of Acidity as Acetic Acid on n-Butyl Acetate sample #17060; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1613	28.6	C	1.76	First reported 0.00286
174		----		----	
311	D1613	23		0.64	
323	D1613	15		-0.96	
347	D1613	18.6		-0.24	
357	D1613	24		0.84	
609	D1613	11.5		-1.66	
786	D1613	24		0.84	
840	D1613	14.5		-1.06	
902	D1613	19		-0.16	
913	D1613	30		2.04	
1319	D1613	9.4		-2.08	

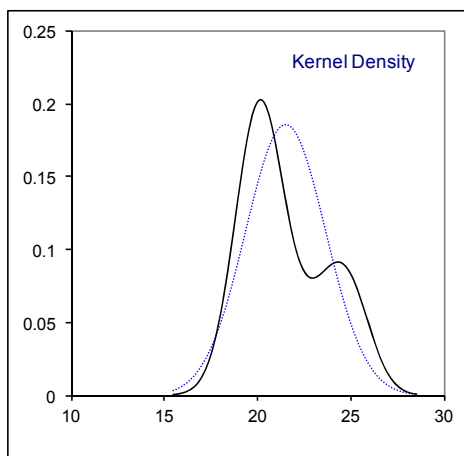
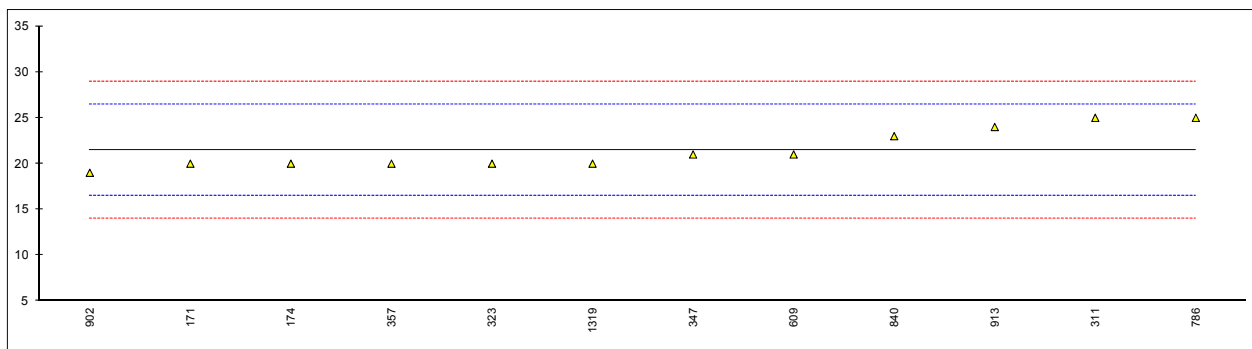
normality OK
n 11
outliers 0
mean (n) 19.78
st.dev. (n) 6.762
R(calc.) 18.93
R(D1613:17) 14



Determination of Color Pt/Co on n-Butyl Acetate sample #17060

lab	method	value	mark	z(targ)	remarks
171	D1209	20		-0.60	
174	D1209	20		-0.60	
311	D1209	25		1.40	
323	D1209	20		-0.60	
347	D5386	21		-0.20	
357	D1209	20		-0.60	
609	D1209	21		-0.20	
786	D1209	25		1.40	
840	D1209	23		0.60	
902	D5386	19		-1.00	
913	D5386	24		1.00	
1319	D1209	20		-0.60	

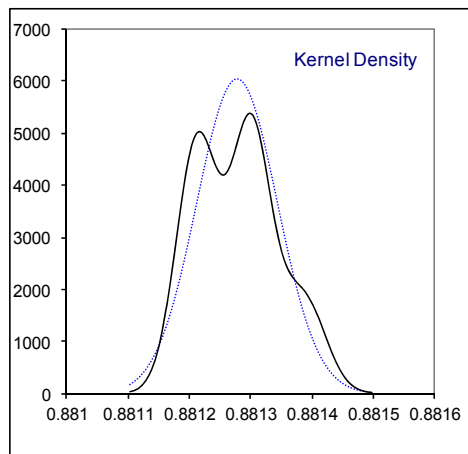
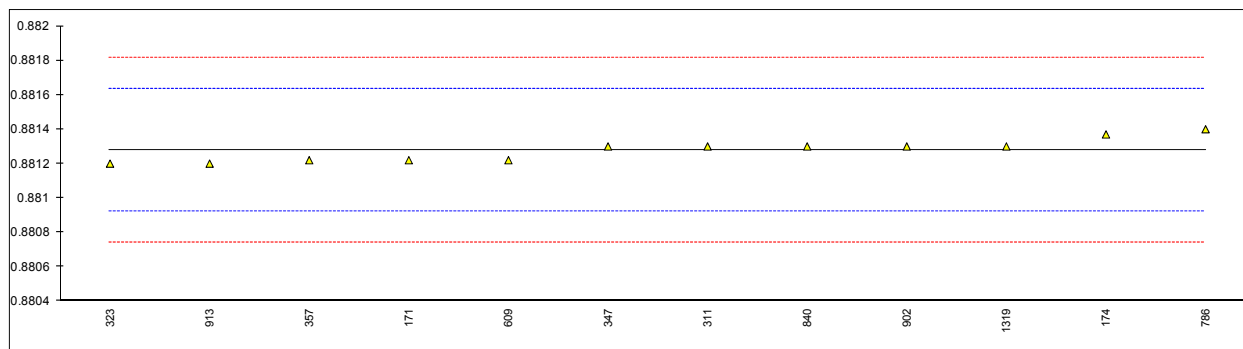
normality OK
n 12
outliers 0
mean (n) 21.5
st.dev. (n) 2.15
R(calc.) 6.0
R(D1209:05) 7



Determination of Density at 20°C on n-Butyl Acetate sample #17060; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.88122		-0.32	
174	D4052	0.88137		0.52	
311	D4052	0.8813		0.13	
323	D4052	0.8812		-0.43	
347	D4052	0.8813		0.13	
357	D4052	0.88122		-0.32	
609	D4052	0.88122		-0.32	
786	D4052	0.8814		0.69	
840	D4052	0.88130		0.13	
902	D4052	0.8813		0.13	
913	D4052	0.8812		-0.43	
1319	D4052	0.88130		0.13	

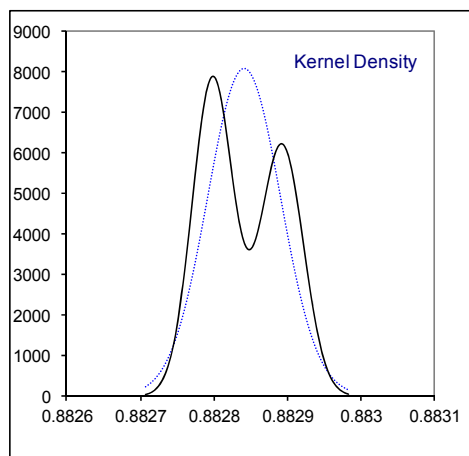
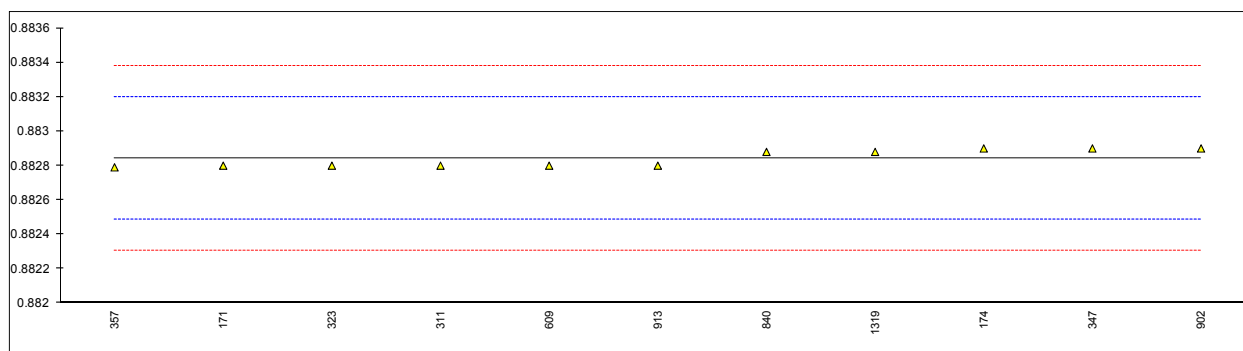
normality OK
 n 12
 outliers 0
 mean (n) 0.88128
 st.dev. (n) 0.000066
 R(calc.) 0.00019
 R(D4052:16) 0.0005



Determination of Specific Gravity 20/20°C on n-Butyl Acetate sample #17060

lab	method	value	mark	z(targ)	remarks
171	D4052	0.8828		-0.23	
174	D4052	0.8829		0.33	
311	D4052	0.8828		-0.23	
323	D4052	0.8828		-0.23	
347	D4052	0.8829		0.33	
357	D4052	0.88279		-0.29	
609	D4052	0.8828		-0.23	
786		----		----	
840	D4052	0.88288		0.22	
902	D4052	0.8829		0.33	
913	D4052	0.8828		-0.23	
1319	D4052	0.88288		0.22	

normality OK
n 11
outliers 0
mean (n) 0.88284
st.dev. (n) 0.000049
R(calc.) 0.00014
R(D4052:16) 0.0005



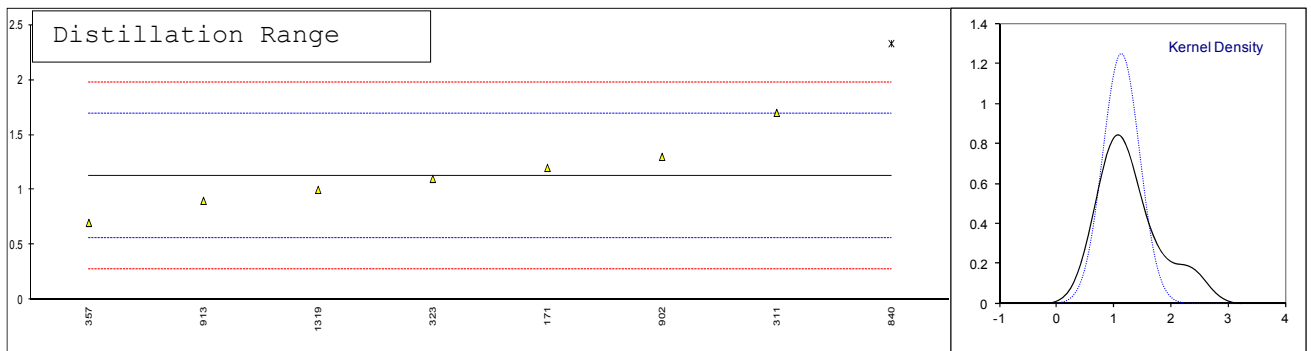
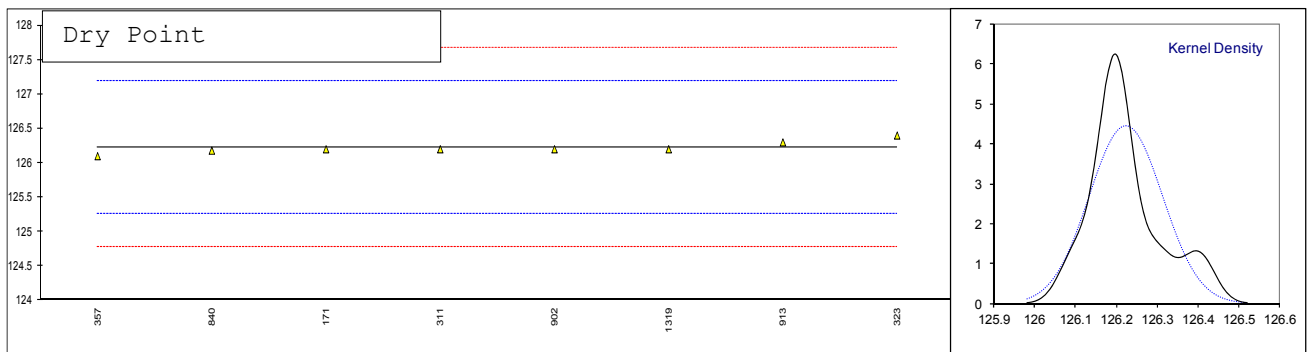
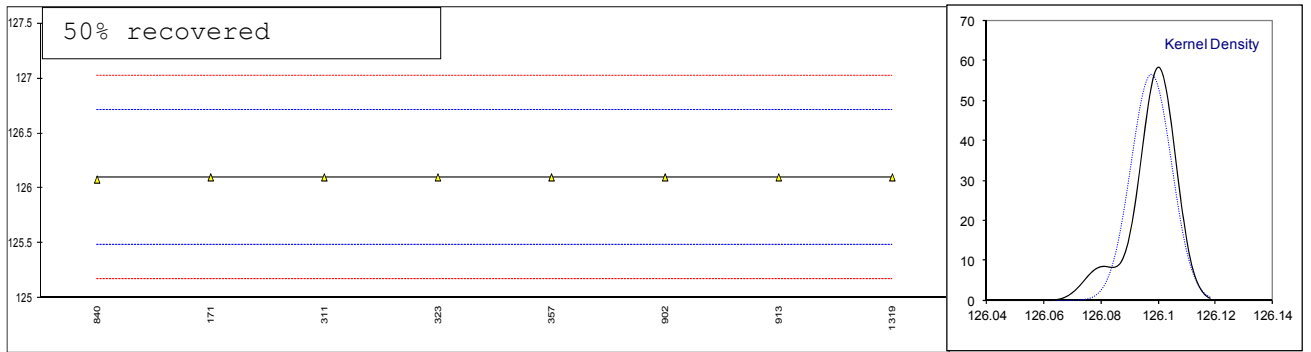
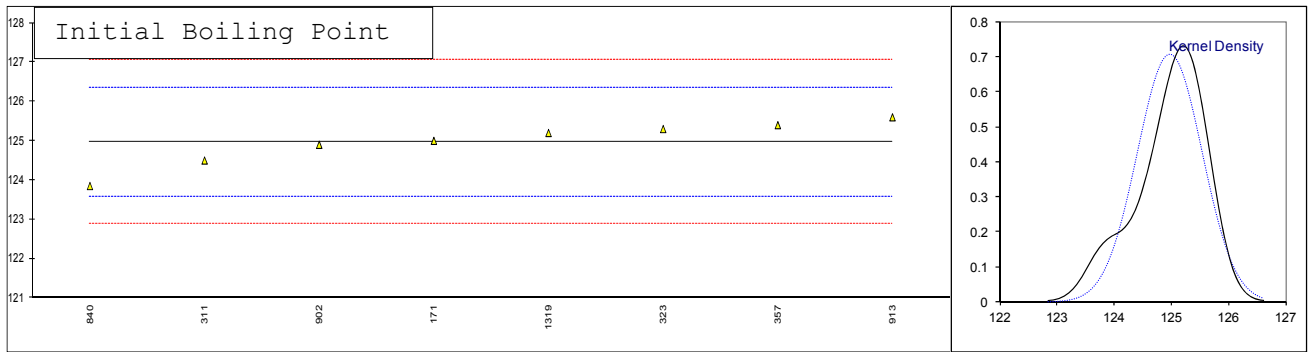
Determination of Distillation on n-Butyl Acetate sample #17060; results in °C

lab	method	mode	IBP	mark	50% rec	mark	DP	mark	Distil. Range	mark
171	D1078	Automated	125.0		126.1		126.2		1.2	
174			----		----		----		----	
311	D1078	Automated	124.5		126.1		126.2		1.7	
323	D1078	Manual	125.3		126.1		126.4		1.1	
347			----		----		----		----	
357	D1078	Automated	125.4		126.1		126.1		0.7	
609			----		----		----		----	
786			----		----		----		----	
840	D1078	Automated	123.85		126.08		126.18		2.33	G(0.05)
902	D1078	Automated	124.9		126.1		126.2		1.3	
913	D1078	Manual	125.6		126.1		126.3		0.9	
1319	D1078	Manual	125.2		126.1		126.2		1.0	
	normality		unknown		unknown		unknown		unknown	
	n		8		8		8		7	
	outliers		0		0		0		1	
	mean (n)		124.97		126.10		126.22		1.13	
	st.dev. (n)		0.564		0.007		0.090		0.320	
	R(calc.)		1.58		0.02		0.25		0.90	
	R(D1078:11)	Automated	1.95		0.86		1.35		0.79	
Compare	R(D1078:11)	Manual	1.34		0.82		1.64		0.71	

Theoretical mid boiling point = 126.1 °C

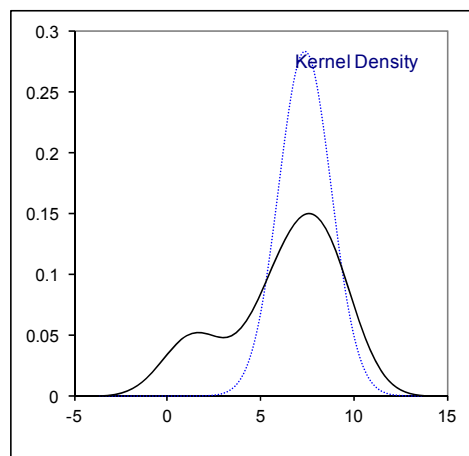
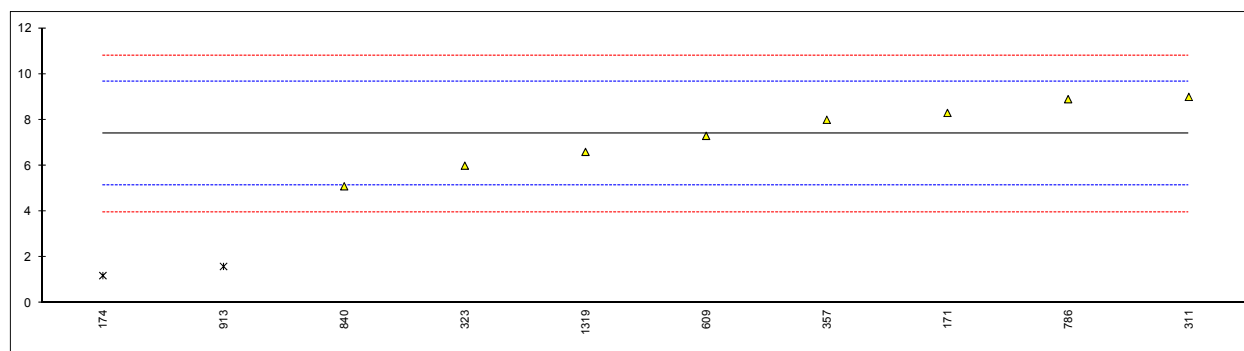
z-scores of Distillation on n-Butyl Acetate sample #17060

lab	method	IBP z(targ)	50% rec z(targ)	DP z(targ)	Distil. Range z(targ)
171	D1078	0.04	0.01	-0.05	0.25
174		----	----	----	----
311	D1078	-0.67	0.01	-0.05	2.02
323	D1078	0.48	0.01	0.37	-0.10
347		----	----	----	----
357	D1078	0.62	0.01	-0.25	-1.51
609		----	----	----	----
786		----	----	----	----
840	D1078	-1.61	-0.06	-0.09	4.24
902	D1078	-0.10	0.01	-0.05	0.61
913	D1078	0.91	0.01	0.16	-0.81
1319	D1078	0.33	0.01	-0.05	-0.45



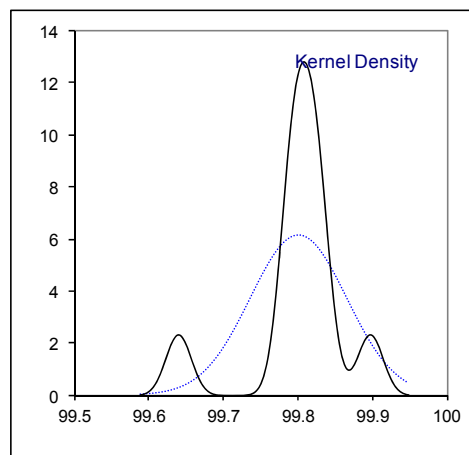
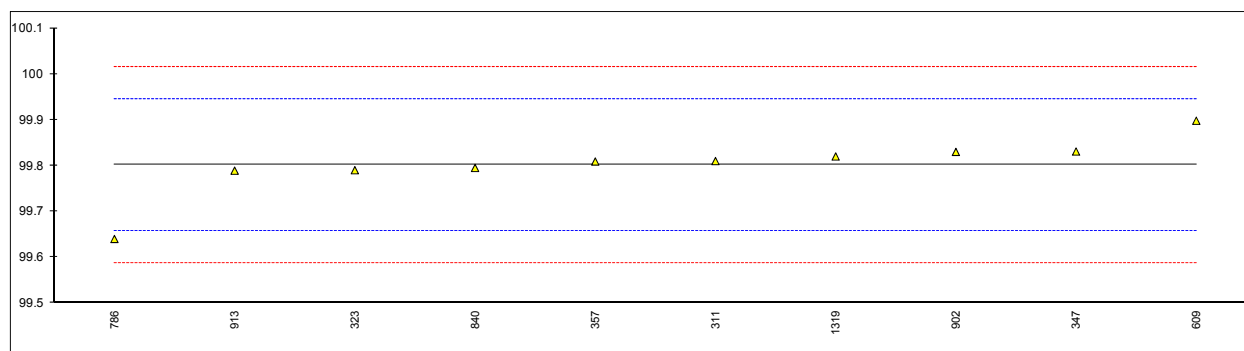
Determination of Nonvolatile matter on n-Butyl Acetate sample #17060; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
171	D1353	8.3	C	0.79	First reported 0.0083
174	D1353	1.2	G(0.05)	-5.44	
311	D1353	9		1.40	
323	D1353	6		-1.23	
347		----		----	
357	D1353	8		0.53	
609	D1353	7.3		-0.09	
786	D1353	8.9		1.32	
840	D1353	5.10		-2.02	
902		----		----	
913	D1353	1.6	C,G(0.05)	-5.09	First reported 0.6
1319	D1353	6.6		-0.70	
normality		OK			
n		8			
outliers		2			
mean (n)		7.40			
st.dev. (n)		1.408			
R(calc.)		3.94			
R(D1353:13)		3.19			



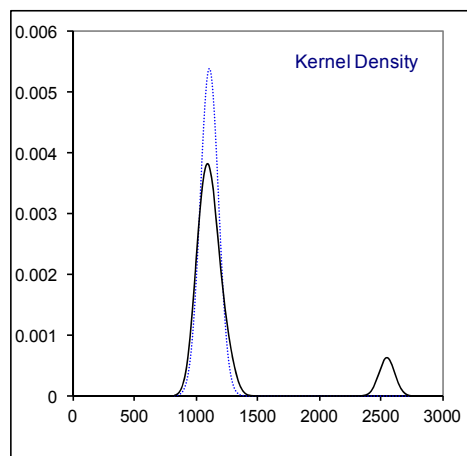
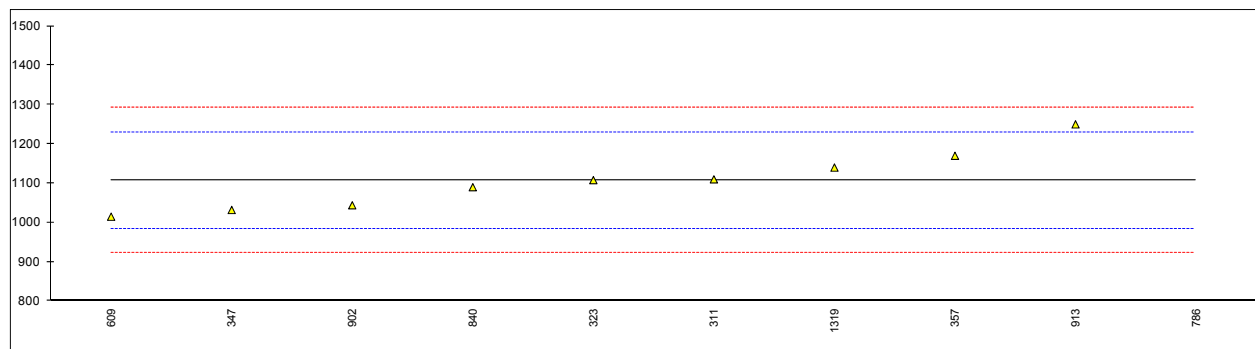
Determination of Purity of n-Butyl Acetate sample #17060; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		----		----	
174		----		----	
311	INH-172	99.81		0.12	
323	D3545	99.79		-0.16	
347	D3545	99.8308		0.41	
357	D3545	99.809		0.11	
609	In house	99.898		1.36	
786	GOST8981	99.64		-2.26	
840	D3545	99.795		-0.09	
902	INH-126	99.83		0.40	
913	D3545	99.789		-0.17	
1319	JISK0114	99.82		0.26	
	normality	not OK			
	n	10			
	outliers	0			
	mean (n)	99.8012			
	st.dev. (n)	0.06486			
	R(calc.)	0.1816			
	R(D3545:06)	0.2			



Determination of n-Butanol on n-Butyl Acetate sample #17060; results in mg/kg

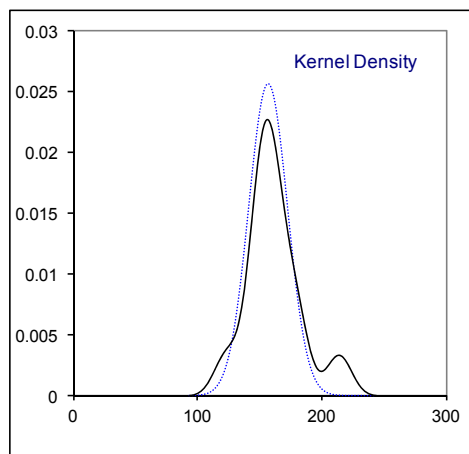
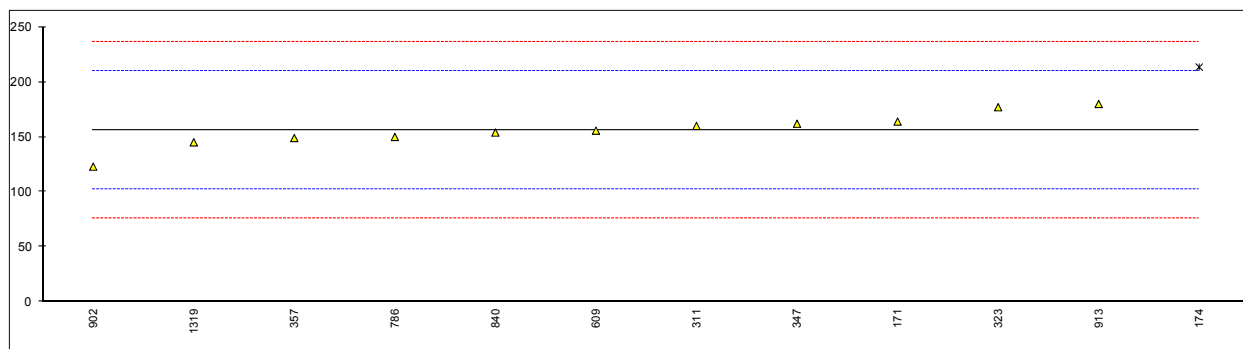
lab	method	value	mark	z(targ)	remarks
171		----		----	
174		----		----	
311	INH-172	1110		0.06	
323	D3545	1108		0.02	
347	D3545	1032		-1.21	
357	D3545	1170	C	1.03	First reported 0.117
609	In house	1015		-1.49	
786	GOST8981	2545	C,G(0.01)	23.33	First reported 2073
840	D3545	1090		-0.27	
902	INH-126	1044	C	-1.01	First reported 44
913	D3545	1250	C	2.33	First reported 0.125
1319	JISK0114	1140		0.54	
normality		OK			
n		9			
outliers		1			
mean (n)		1106.6			
st.dev. (n)		74.05			
R(calc.)		207.3			
R(Horwitz)		172.6			



Determination of Water titrimetric on n-Butyl Acetate sample #17060; results in mg/kg

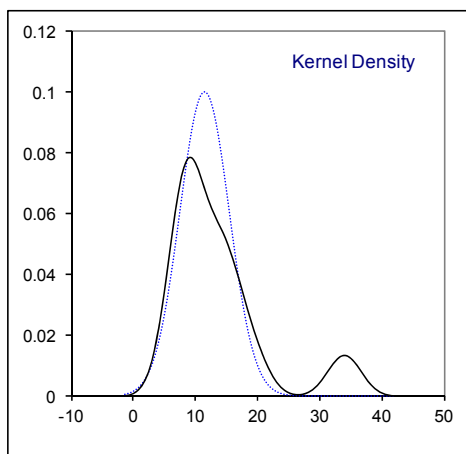
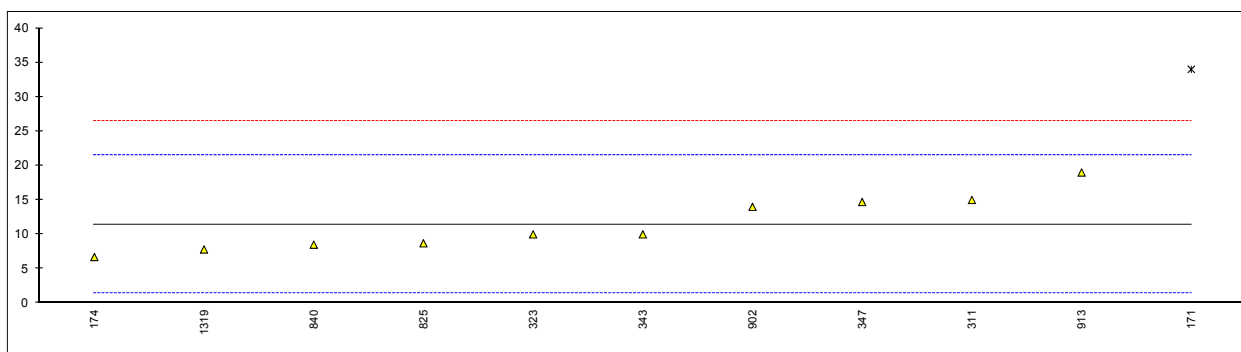
lab	method	value	mark	z(targ)	remarks
171	D1364	164		0.29	
174	E203	213.5	D(0.05)	2.13	
311	D1364	160		0.14	
323	D1364	177		0.77	
347	E1064	162		0.21	
357	E1064	149		-0.27	
609	E1064	155.72		-0.02	
786	D1364	150		-0.24	
840	E1064	154		-0.09	
902	D1364	123		-1.24	
913	D1364	180		0.88	
1319	D1364	145.1		-0.42	

normality suspect
n 11
outliers 1
mean (n) 156.35
st.dev. (n) 15.584
R(calc.) 43.63
R(D1364:02) 75.02



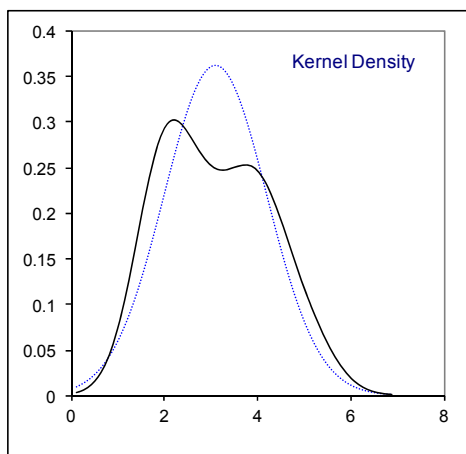
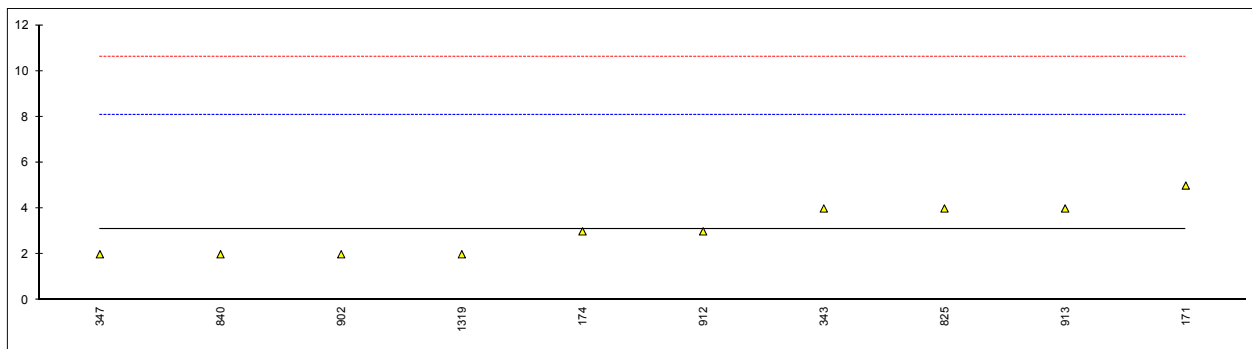
Determination of Acidity as Acetic Acid on Ethyl Acetate sample #17061; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1613	34	C,G(0.01)	4.51	First reported 0.0034
174	D1613	6.7		-0.95	
311	D1613	15		0.71	
323	D1613	10		-0.29	
343	D1613	10.0		-0.29	
347	D1613	14.7		0.65	
551		----		----	
825	D1613	8.7		-0.55	
840	D1613	8.5		-0.59	
902	D1613	14		0.51	
912		----		----	
913	D1613	19		1.51	
963		----		----	
1319	D1613	7.8		-0.73	
normality	OK				
n	10				
outliers	1				
mean (n)	11.44				
st.dev. (n)	3.986				
R(calc.)	11.16				
R(D1613:17)	14				



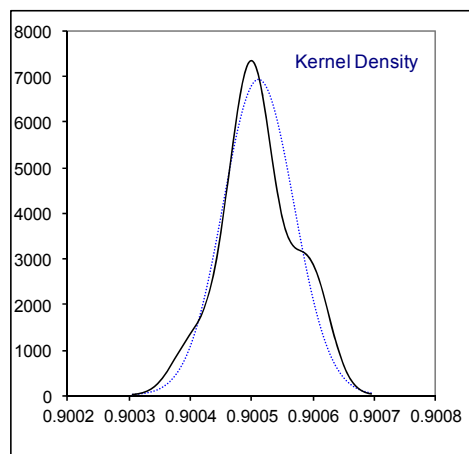
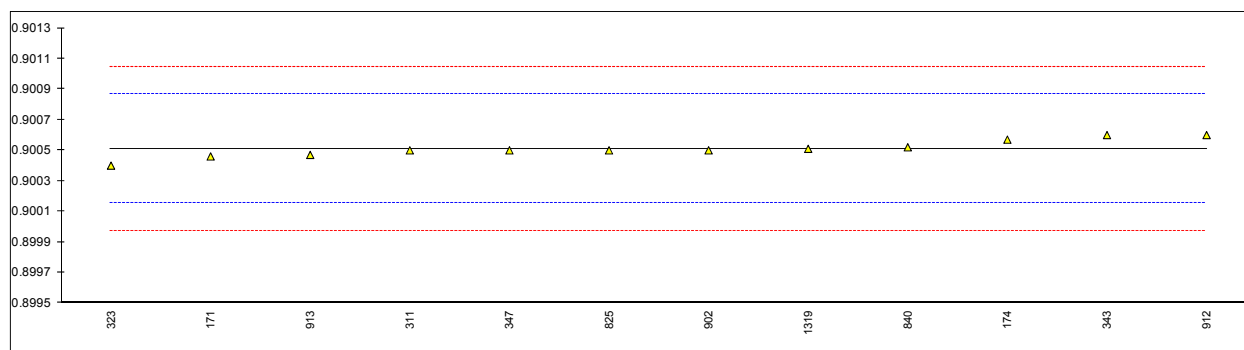
Determination of Color Pt/Co on Ethyl Acetate sample #17061

lab	method	value	mark	z(targ)	remarks
171	D1209	5		0.76	
174	D1209	3		-0.04	
311	D1209	<5		----	
323	D1209	<5		----	
343	D5386	4		0.36	
347	D5386	2		-0.44	
551		----		----	
825	D1209	4		0.36	
840	D1209	2		-0.44	
902	D5386	2		-0.44	
912	D5386	3		-0.04	
913	D5386	4		0.36	
963		----		----	
1319	D1209	2		-0.44	
normality		OK			
n		10			
outliers		0			
mean (n)		3.1			
st.dev. (n)		1.10			
R(calc.)		3.1			
R(D1209:05)		7			



Determination of Density at 20°C on Ethyl Acetate sample #17061; results in kg/L

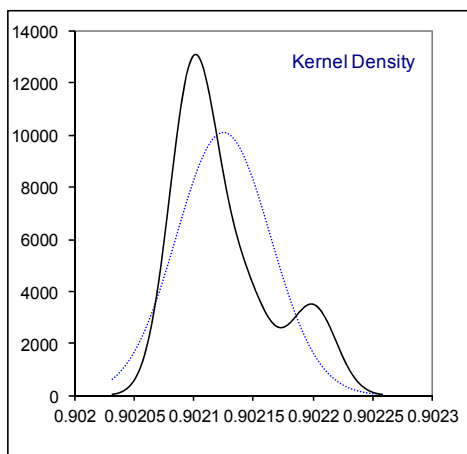
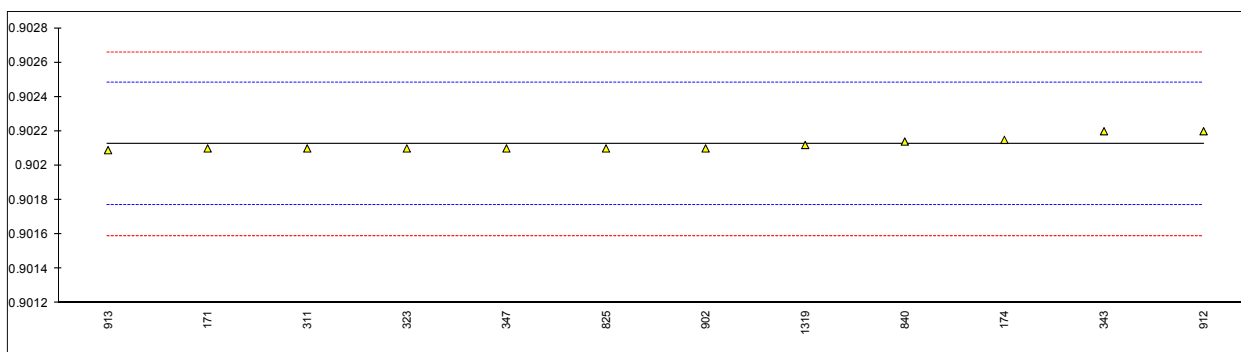
lab	method	value	mark	z(targ)	remarks
171	D4052	0.90046		-0.28	
174	D4052	0.90057		0.33	
311	D4052	0.9005		-0.06	
323	D4052	0.9004		-0.62	
343	D4052	0.9006		0.50	
347	D4052	0.9005		-0.06	
551		----		----	
825	D4052	0.9005		-0.06	
840	D4052	0.90052		0.05	
902	D4052	0.9005		-0.06	
912	D4052	0.9006		0.50	
913	D4052	0.90047		-0.23	
963		----		----	
1319	D4052	0.90051		0.00	
normality		OK			
n		12			
outliers		0			
mean (n)		0.90051			
st.dev. (n)		0.000058			
R(calc.)		0.00016			
R(D4052:16)		0.0005			



Determination of Specific Gravity 20/20°C on Ethyl Acetate sample #17061

lab	method	value	mark	z(target)	remarks
171	D4052	0.9021		-0.14	
174	D4052	0.90215		0.14	
311	D4052	0.9021		-0.14	
323	D4052	0.9021		-0.14	
343	D4052	0.9022		0.42	
347	D4052	0.9021		-0.14	
551		----		----	
825	D4052	0.9021		-0.14	
840	D4052	0.90214		0.08	
902	D4052	0.9021		-0.14	
912	D4052	0.9022		0.42	
913	D4052	0.90209		-0.20	
963		----		----	
1319	D4052	0.90212		-0.03	

normality suspect
n 12
outliers 0
mean (n) 0.90212
st.dev. (n) 0.000039
R(calc.) 0.00011
R(D4052:16) 0.0005



Determination of Distillation on Ethyl Acetate sample #17061; results in °C

lab	method	mode	IBP	mark	50% rec	mark	DP	mark	Distil. Range	mark
171	D1078	Automated	77.0		77.2		77.3		0.3	
174			----		----		----		----	
311	D1078	Automated	76.9		77.2		77.3		0.4	
323	D1078	Manual	77.0		77.2		77.3		0.3	
343	D1078	Automated	76.9		77.2		77.3		0.4	
347	D1078	Automated	76.9		77.2		77.4		0.5	
551			----		----		----		----	
825	D1078	Automated	76.9		77.1		77.1	DG(0.01)	0.2	
840	D1078	Automated	76.80		77.20		77.29		0.49	
902	D1078	Automated	76.6		77.2		77.4		0.8	
912	D1078	Manual	77.0		77.2		77.4		0.4	
913	D1078	Manual	76.9		77.2		77.4		0.5	
963			----		----		----		----	
1319	D1078	Manual	76.3	G(0.05)	76.8	G(0.01)	77.0	DG(0.01)	0.7	ex
	normality		not OK		not OK		OK		not OK	
	n		10		10		9		10	
	outliers		1		1		2		0 (+1 ex)	
	mean (n)		76.89		77.19		77.34		0.43	
	st.dev. (n)		0.120		0.032		0.054		0.163	
	R(calc.)		0.34		0.089		0.15		0.46	
	R(D1078:11)	Automated	1.20		0.53		0.83		0.70	
Compare	R(D1078:11)	Manual	0.82		0.50		1.01		0.63	

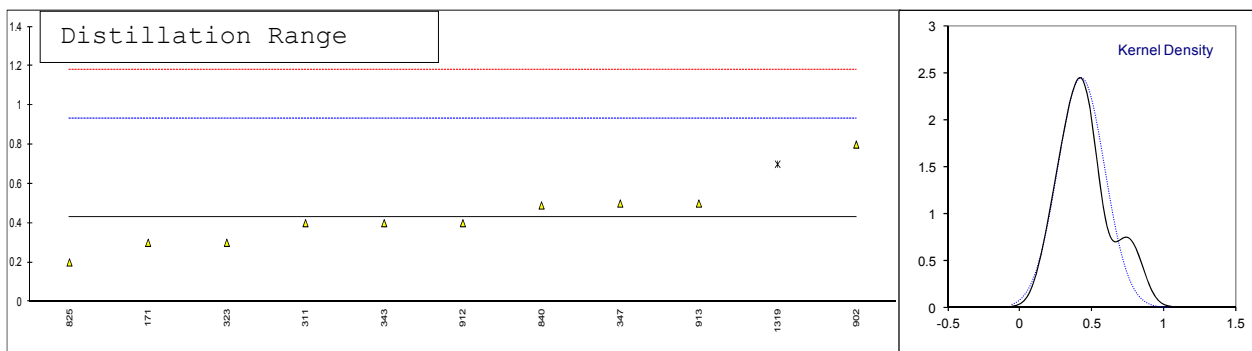
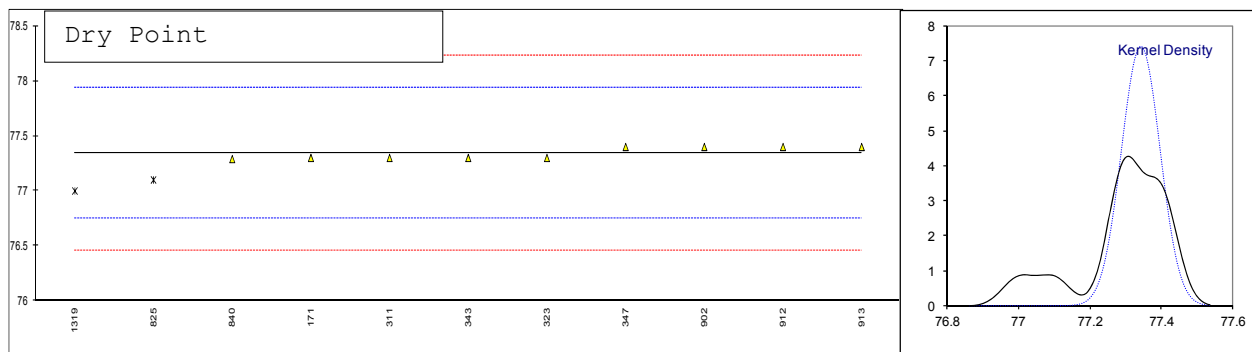
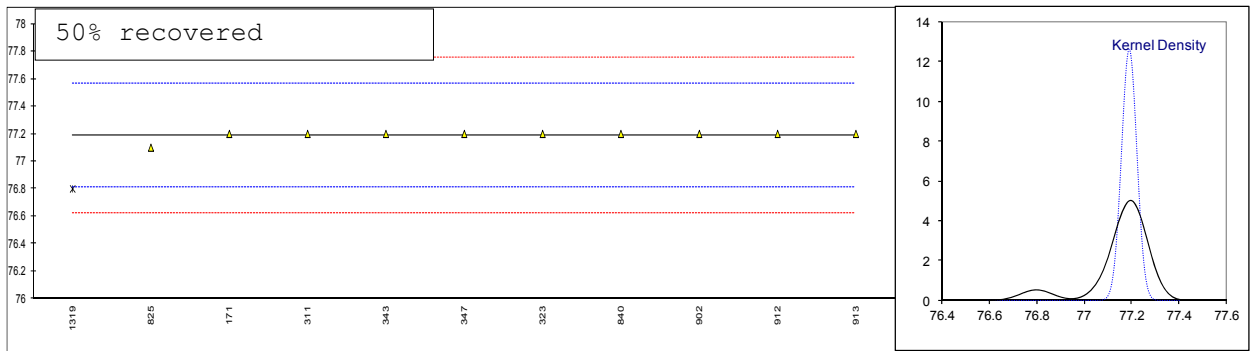
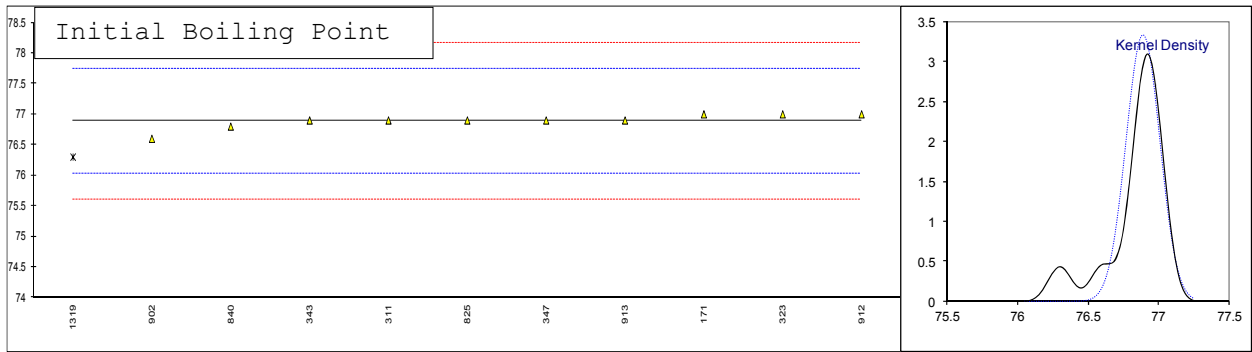
Theoretical mid boiling point = 77.2 °C

After manual correction:

1319	D1078	Manual	76.7	DG(0.05)	77.2		77.4	
	normality		OK		OK		OK	
	n		9		10		10	
	outliers		2		1		1	
	mean (n)		76.92		77.20		77.34	
	st.dev. (n)		0.067		0.000		0.053	
	R(calc.)		0.19		0.00		0.15	
	R(D1078:11)	Automated	1.20		0.53		0.83	
Compare	R(D1078:11)	Manual	0.82		0.50		1.01	

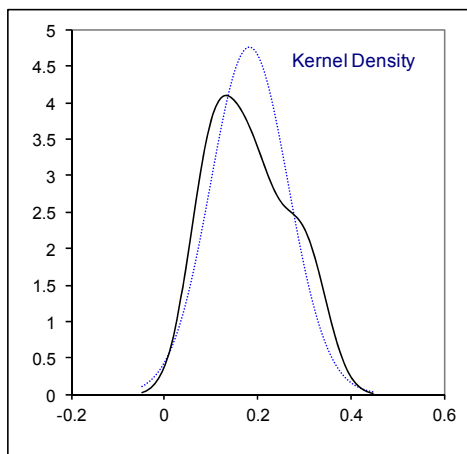
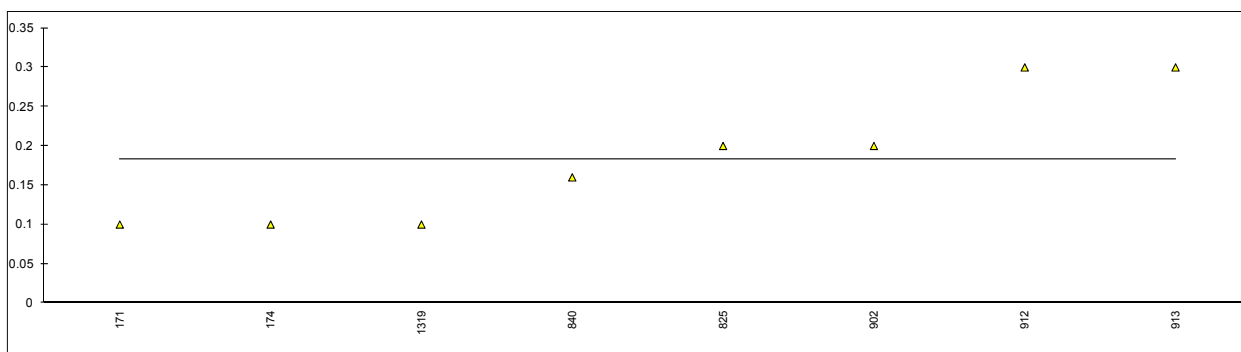
z-scores of Distillation on Ethyl Acetate sample #17061; no manual correction mid boiling point

lab	method	IBP z(targ)	50% rec z(targ)	DP z(targ)	Distil. Range z(targ)
171	D1078	0.26	0.05	-0.15	-0.51
174		----	----	----	----
311	D1078	0.02	0.05	-0.15	-0.12
323	D1078	0.26	0.05	-0.15	-0.51
343	D1078	0.02	0.05	-0.15	-0.12
347	D1078	0.02	0.05	0.19	0.28
551		----	----	----	----
825	D1078	0.02	-0.48	-0.82	-0.91
840	D1078	-0.21	0.05	-0.18	0.24
902	D1078	-0.68	0.05	0.19	1.48
912	D1078	0.26	0.05	0.19	-0.12
913	D1078	0.02	0.05	0.19	0.28
963		----	----	----	----
1319	D1078	-1.38	-2.07	-1.16	1.08



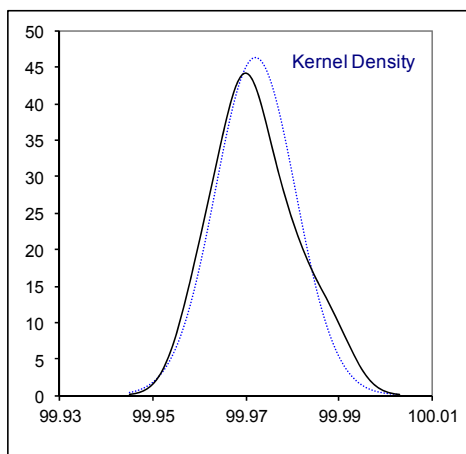
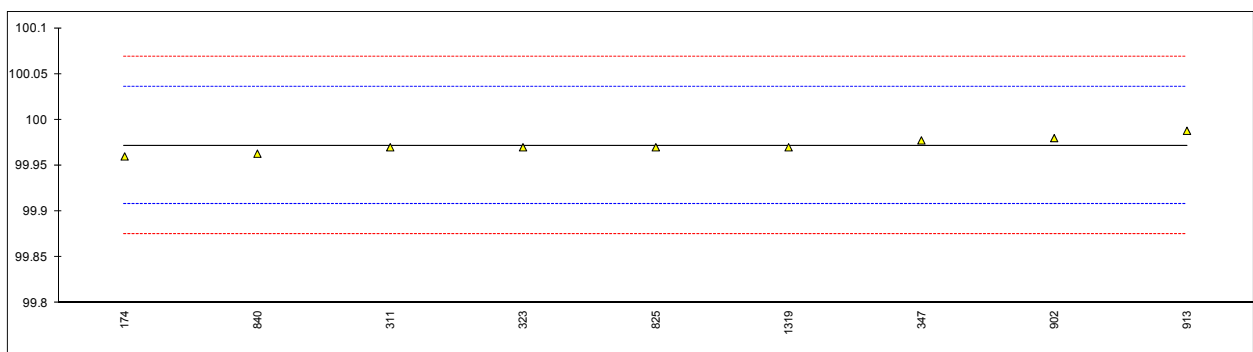
Determination of Nonvolatile matter on Ethyl Acetate sample #17061; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
171	D1353	0.1		----	
174	D1353	0.1		----	
311	D1353	<1.0		----	
323	D1353	<1		----	
343	D1353	<1		----	
347	D1353	<1		----	
551		----		----	
825	D1353	0.2		----	
840	D1353	0.16		----	
902	D1353	0.2		----	
912	D1353	0.3		----	
913	D1353	0.3	C	----	First reported 0.5
963		----		----	
1319	D1353	0.1		----	
	normality	n.a.			
	n	12			
	outliers	n.a.			
	mean (n)	<1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D1353:13)	n.a.			



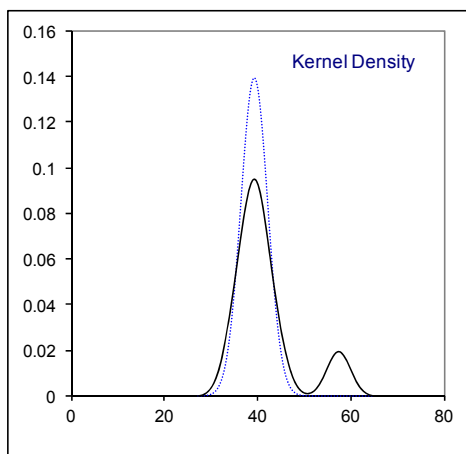
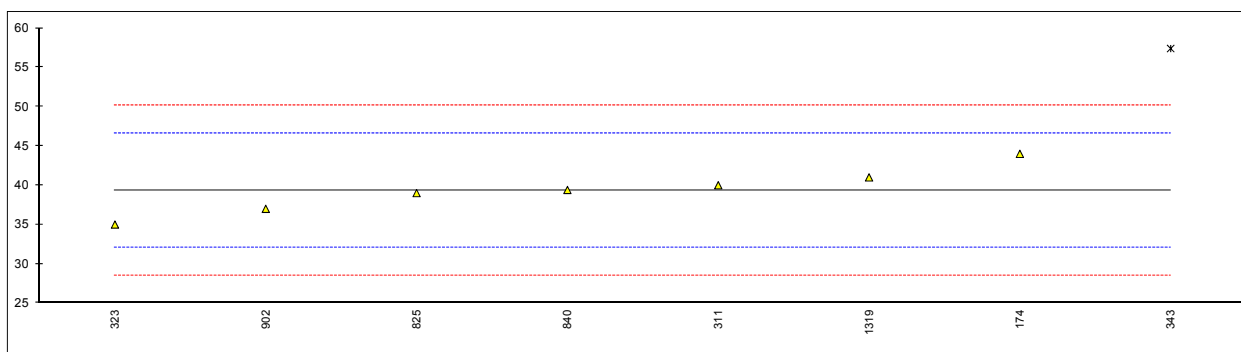
Determination of Purity of Ethyl Acetate sample #17061; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
174	D3545	99.96		-0.37	
311	INH-074	99.97		-0.06	
323	D3545	99.97		-0.06	
343	INH-1243	>99.9		-----	
347	D3545	99.9775		0.17	
551		-----		-----	
825	D3545	99.97		-0.06	
840	D3545	99.963		-0.28	
902	INH-128	99.98		0.25	
912		-----		-----	
913	D3545	99.988		0.50	
963		-----		-----	
1319	JISK0114	99.97		-0.06	
normality		OK			
n		9			
outliers		0			
mean (n)		99.9720			
st.dev. (n)		0.00860			
R(calc.)		0.0241			
R(D3545:06)		0.09			



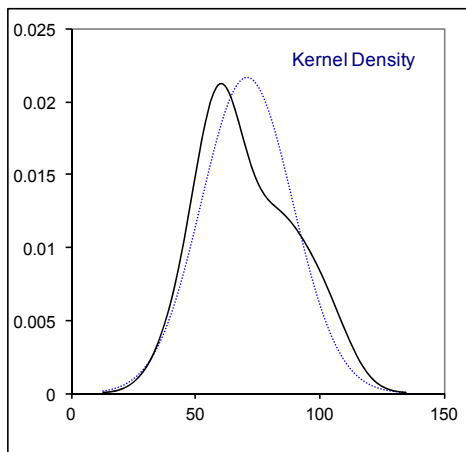
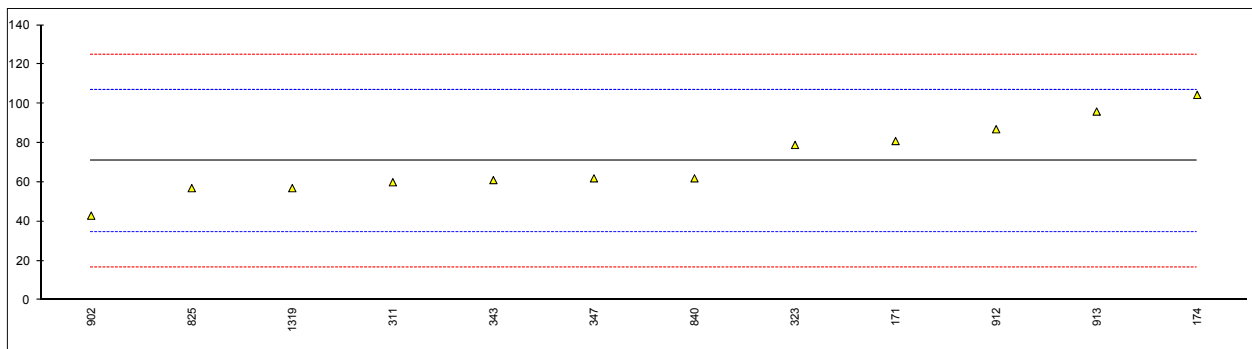
Determination of Ethanol on Ethyl Acetate sample #17061; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		----		----	
174	D3545	44		1.29	
311	INH-074	40		0.18	
323	D3545	35		-1.20	
343	INH-1243	57.4	G(0.01)	4.99	
347	D3545	<100		----	
551		----		----	
825	D3545	39		-0.09	
840	D3545	39.4		0.02	
902	INH-128	37		-0.65	
912		----		----	
913	D3545	<5		<-9.48	False negative test result?
963		----		----	
1319	JISK0114	41		0.46	
normality	unknown				
n	7				
outliers	1				
mean (n)	39.34				
st.dev. (n)	2.867				
R(calc.)	8.0				
R(Horwitz)	10.1				



Determination of Water titrimetric on Ethyl Acetate sample #17061; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	81		0.57	
174	E203	104.5		1.87	
311	D1364	60		-0.60	
323	D1364	79		0.45	
343	E1064	61.1		-0.54	
347	E1064	62		-0.49	
551		----		----	
825	E1064	57		-0.77	
840	E1064	62		-0.49	
902	D1364	43		-1.54	
912	E203	87		0.90	
913	D1364	96		1.40	
963		----		----	
1319	D1364	57		-0.77	
normality		OK			
n		12			
outliers		0			
mean (n)		70.80			
st.dev. (n)		18.399			
R(calc.)		51.52			
R(D1364:02)		50.49			



APPENDIX 2**Number of participants in the n-Butyl Acetate PT iis17C06**

1 lab in BELGIUM
1 lab in FINLAND
1 lab in INDIA
1 lab in JAPAN
1 lab in MALAYSIA
1 lab in NETHERLANDS
1 lab in RUSSIAN FEDERATION
1 lab in SPAIN
1 lab in TURKEY
2 labs in UNITED STATES OF AMERICA
1 lab in VIETNAM

Number of participants in the Ethyl Acetate PT iis17C07

1 lab in BELGIUM
1 lab in BRAZIL
2 labs in INDIA
1 lab in JAPAN
1 lab in NETHERLANDS
1 lab in SAUDI ARABIA
1 lab in SOUTH KOREA
2 labs in SPAIN
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	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from the statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

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