

**Results of Proficiency Test  
Vinyl Acetate Monomer  
February 2017**

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

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## **1 INTRODUCTION**

Since 2007, a proficiency test for Vinyl Acetate Monomer (VAM) is organised every year by the Institute for Interlaboratory Studies (iis). During the planning of the annual proficiency testing program 2016/2017, it was decided to continue the round robin for the analysis of Vinyl Acetate Monomer. The proficiency test on Vinyl Acetate Monomer has been organised in accordance with the latest applicable version of the ASTM D2190 specification and a number of additional tests requested by some participants. In this interlaboratory study, 27 laboratories in 19 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the 2017 proficiency test are presented and discussed. This report is also electronically available through the iis website [www.iisnl.com](http://www.iisnl.com).

## **2 SET UP**

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample of 0.5 L of Vinyl Acetate Monomer, labelled #17001. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

### **2.1 QUALITY SYSTEM**

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

### **2.2 PROTOCOL**

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). The protocol can be downloaded from iis website [www.iisnl.com](http://www.iisnl.com), from the FAQ page.

### **2.3 CONFIDENTIALITY STATEMENT**

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

## 2.4 SAMPLES

The necessary bulk material for sample #17001 was obtained from a local trader. The approximately 25 litre bulk sample was homogenised in a pre-cleaned drum and 48 amber glass bottles of 0.5L were filled and labelled #17001. The homogeneity of these subsamples was checked by determination of Density at 20°C in accordance with ASTM D4052 and Water in accordance with ASTM D1364 on 8 stratified randomly selected samples.

	<i>Density at 20°C in kg/L</i>	<i>Water in mg/kg</i>
sample #17001-1	0.93215	140
sample #17001-2	0.93218	140
sample #17001-3	0.93218	150
sample #17001-4	0.93217	140
sample #17001-5	0.93217	150
sample #17001-6	0.93219	150
sample #17001-7	0.93219	150
sample #17001-8	0.93219	150

Table 1: homogeneity test results of subsamples #17001

From the test results of table 1, 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 the next table:

	<i>Density at 20°C in kg/L</i>	<i>Water in mg/kg</i>
r (observed)	0.00004	18
reference test method	ASTM D4052:16	ASTM D1364:02(2012)
0.3*R (reference test method)	0.00015	22

Table 2: evaluation of the repeatabilities of subsamples #17001

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

To each of the participating laboratories 1 bottle of 0.5 L Vinyl Acetate Monomer, labelled #17001, was sent on January 25, 2017. An SDS was added to the sample package.

## 2.5 STABILITY OF THE SAMPLES

The stability of Vinyl Acetate Monomer packed in 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 one to all of the following parameters; Acidity (without and/or with Nitrogen purge), Apparent Specific Gravity 20/20°C, Colour Pt/Co, Density at 20°C, Distillation (IBP, 50% recovery, Dry Point, Distillation Range), Inhibitor as Hydroquinone, Purity by GC inclusive Acetaldehyde, Acetone, Ethyl Acetate, Methyl Acetate and Water.

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

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal [www.kpmd.co.uk/sgs-iis/](http://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](http://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/](http://www.kpmd.co.uk/sgs-iis/). The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

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

### 3.1 STATISTICS

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

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

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

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

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

### 3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

### 3.3 Z-SCORES

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

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

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

The z-scores were calculated according to:

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

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

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

	$ z  < 1$	good
1 <	$ z  < 2$	satisfactory
2 <	$ z  < 3$	questionable
3 <	$ z $	unsatisfactory

## 4 EVALUATION

In this proficiency test, some problems were encountered with the dispatch of the samples. Participants in Brazil, Mexico, India and Saudi Arabia received the samples late or not at all due to problems at customs. Of the 27 participants, one participant reported the test results after the final reporting date and four other participants did not report any test result at all.

Not all participants were able to report test results for all the requested tests. Finally, 23 participants reported in total 266 numerical test results. Observed were 8 outlying test results, which is 3.0% 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 TEST

In this section, the reported test results are discussed per test.

The test methods, which are used by the different laboratories, are taken into account for explaining the observed differences when possible and applicable. These test methods are also listed in the tables together with the reported test results. The abbreviations, used in these tables, are listed in appendix 3.

Unfortunately, a suitable reference test method providing the precision data is not available for all determinations. For the tests without precision data the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

In the iis PT reports, ASTM test methods are referred to with a number (e.g. D2086) and an added designation for the year that the test method was adopted or revised (e.g. D2086:08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086:08 (2012)). In the test results tables of appendix 1 only the test method number and year of adoption or revision (D2086:08) will be used.

Acidity: This determination (with and without Nitrogen purging) was problematic. The acidity value determined with Nitrogen purging was not significantly different to the acidity without Nitrogen purging. In the determination without Nitrogen purging one statistical outlier was observed and no outliers with Nitrogen purging. The calculated reproducibilities of both determinations after rejection of the statistical outlier are not in agreement with the requirements of ASTM D2086:08(2012). Please note that the reproducibility of ASTM D2086:08(2012) was determined with only two laboratories (see note 5 in §13.2.2 of ASTM D2086:08(2012)). The calculated reproducibility of



the determination without purging was of the same order as observed in the previous PT (iis16C01). The calculated reproducibility for Acidity purged with nitrogen was much larger than the previous PT (iis16C01).

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

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

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

Inhibitor (Hydroquinone): 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 D2193:06(2012).

Purity: Regretfully, no reference test method with precision data exists for this determination; therefore no z-scores were calculated. No statistical outliers were observed. The calculated reproducibility was much larger than observed in previous PT (iis16C01).

Acetaldehyde: The determination of this impurity may be problematic. Two statistical outliers were observed. The reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility using the Horwitz equation.

Acetone: The determination of this impurity may be problematic. No statistical outliers were observed but one test result excluded (see appendix 1). The reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility using the Horwitz equation.

Ethyl Acetate: The determination of this impurity may not be problematic. Two statistical outliers were observed and one other test result excluded (see appendix 1). However, the calculated reproducibility after rejection of the suspect data is in agreement with the estimated reproducibility using the Horwitz equation.

Methyl Acetate: The determination of this impurity 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).

#### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The assigned values, calculated reproducibilities and reproducibilities, derived from reference test methods (in casu ASTM, ISO standards) are compared in the next table.

Parameter	unit	n	Mean	2.8 * sd	R (lit)
Acidity (without N <sub>2</sub> purging)	mg/kg	14	36.1	14.2	6.0
Acidity (with N <sub>2</sub> purging)	mg/kg	7	32.9	20.5	6.0
Apparent Specific Gravity 20/20°C		21	0.9339	0.0001	0.0005
Colour Pt/Co		16	3.7	3.7	7.0
Density at 20°C	kg/L	22	0.9322	0.0002	0.0005
Distillation, Initial Boiling Point	°C	17	72.4	0.2	1.1
Distillation, 50% recovery	°C	18	72.7	0.1	0.5
Distillation, Dry Point	°C	18	72.9	0.3	0.8
Distillation, Boiling Range	°C	17	0.4	0.3	0.7
Inhibitor as Hydroquinone	mg/kg	16	1.8	1.1	1.0
Purity	%M/M	19	99.971	(0.029)	(0.011)
Acetaldehyde	mg/kg	15	36.4	12.8	9.5
Acetone	mg/kg	6	4.2	2.2	1.5
Ethyl Acetate	mg/kg	14	136.6	25.3	29.2
Methyl Acetate	mg/kg	16	27.0	9.8	7.4
Water	mg/kg	20	159.3	35.7	75.7

Table 3: reproducibilities of tests on sample #17001

The calculated reproducibility between brackets is compared against the reproducibility of the previous PT (iis16C01)

### 4.3 EVALUATION OF THE PROFICIENCY TEST OF FEBRUARY 2017 WITH PREVIOUS PTS

	<i>February 2017</i>	<i>February 2016</i>	<i>February 2015</i>	<i>February 2014</i>	<i>February 2013</i>
Number of reporting labs	23	25	20	23	24
Number of results reported	266	275	253	240	243
Number of statistical outliers	8	9	2	9	9
Percentage outliers	3.0%	3.3%	0.8%	3.8%	3.7%

Table 4: comparison to previous proficiency tests.

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

The performance of the determinations of the proficiency test was compared against the requirements of the respective reference test methods. The conclusions are given the following table:

	<i>February 2017</i>	<i>February 2016</i>	<i>February 2015</i>	<i>February 2014</i>	<i>February 2013</i>
Acidity (without N <sub>2</sub> purging)	--	--	--	-	--
Acidity (with N <sub>2</sub> purging)	--	--	--	-	--
Apparent Specific Gravity	++	++	++	n.e.	n.e.
Colour Pt/Co	++	++	+	n.e.	n.e.
Density at 20°C	++	++	++	++	++
Distillation	++	++	++	++	++
Inhibitor as Hydroquinone	+/-	-	-	-	-
Purity	(--)	(+)	(+)	n.e.	n.e.
Acetaldehyde	-	+/-	-	++	++
Acetone	-	n.e.	-	n.e.	n.e.
Ethyl Acetate	+	+/-	-	++	+/-
Methyl Acetate	-	+	-	-	-
Water	++	++	+	+/-	+/-

Table 5: comparison determinations against the reference standards

The calculated reproducibility between brackets is compared against the reproducibility of the previous PT (iis16C01)

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

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

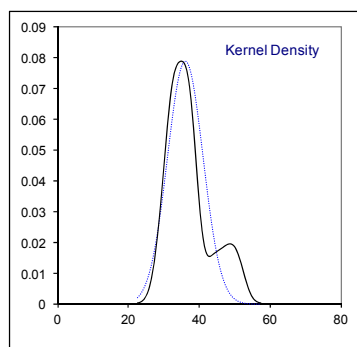
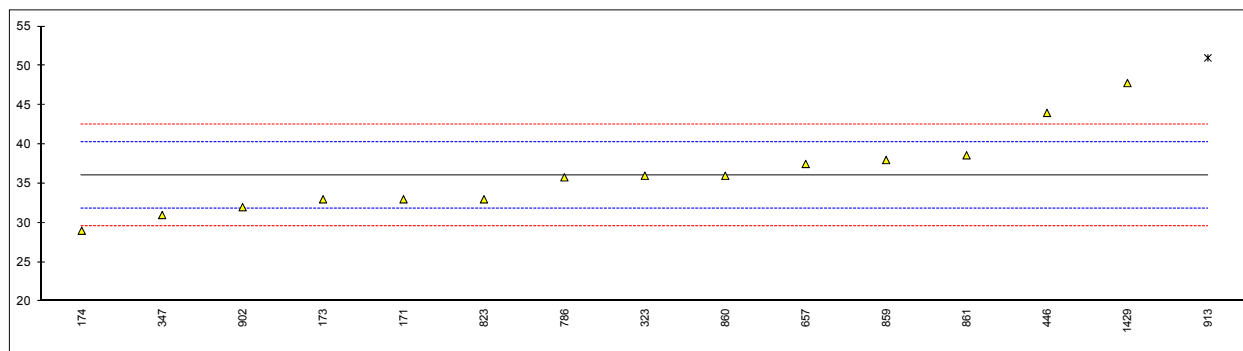
**APPENDIX 1**

Determination of Acidity (without N<sub>2</sub> purging) on sample #17001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2086	33		-1.42	
173	INH-44	33		-1.42	
174	D2086	29		-3.29	
273		----		----	
311		----		----	
323	D2086	36	C	-0.02	first reported: 63
337		----		----	
347	D2086	31		-2.36	
391		----		----	
395		----		----	
446	D2086	44		3.71	
522		----		----	
551		----		----	
557		----		----	
657	D2086	37.5		0.68	
786	D2086	35.8		-0.12	
823	D2086	33		-1.42	
840		----		----	
859	D2086	38		0.91	
860	D2086	36		-0.02	
861	D2086	38.6		1.19	
902	D2086	32		-1.89	
913	D2086	51.0	G(0.05)	6.98	
963		----		----	
974		----		----	
1107		----		----	
1429		47.8		5.48	

normality not OK  
n 14  
outliers 1  
mean (n) 36.05  
st.dev. (n) 5.063  
R(calc.) 14.18  
R(D2086:08) 6.00

Compare R(iis16C01)=13.74

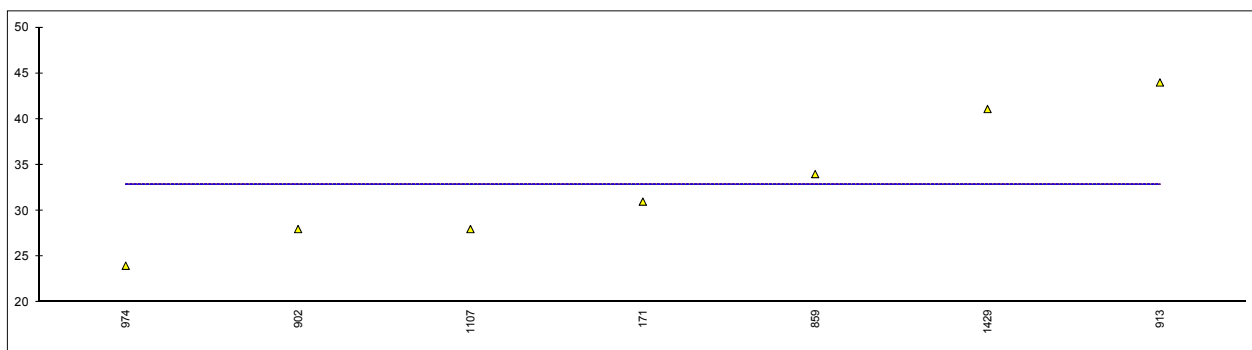


Determination of Acidity (with N<sub>2</sub> purging) on sample #17001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2086	31		----	
173		----		----	
174		----		----	
273		----		----	
311		----		----	
323		----		----	
337		----		----	
347		----		----	
391		----		----	
395		----		----	
446		----		----	
522		----		----	
551		----		----	
557		----		----	
657		----		----	
786		----		----	
823		----		----	
840		----		----	
859	D2086	34		----	
860		----		----	
861		----		----	
902	D2086	28		----	
913	D2086	44.0		----	
963		----		----	
974	D2086	24		----	
1107	D2086	28		----	
1429		41.1		----	

normality unknown  
n 7  
outliers 0  
mean (n) 32.87  
st.dev. (n) 7.331  
R(calc.) 20.53  
R(D2086:08) 6.00

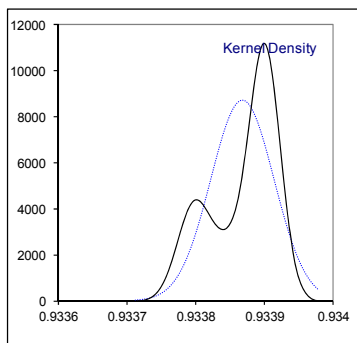
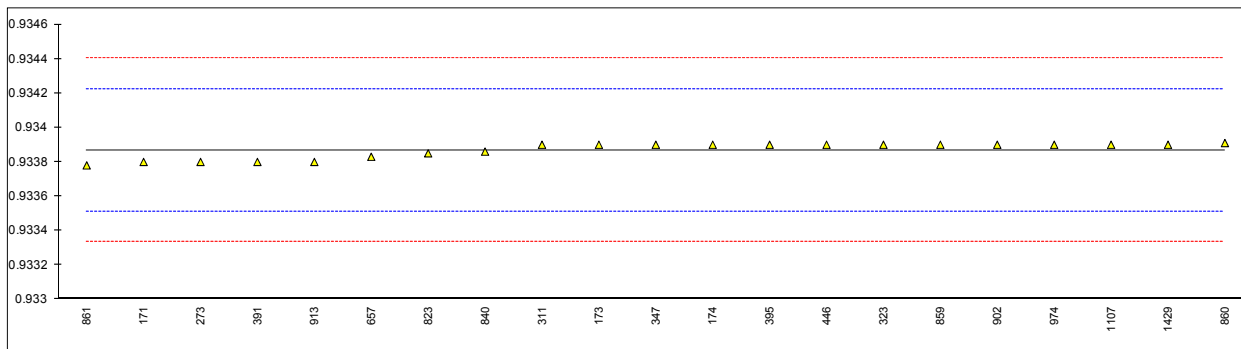
Compare R(iis16C01)=10.01



Determination of App. Specific Gravity 20/20°C on sample #17001;

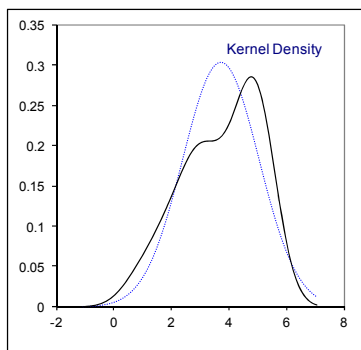
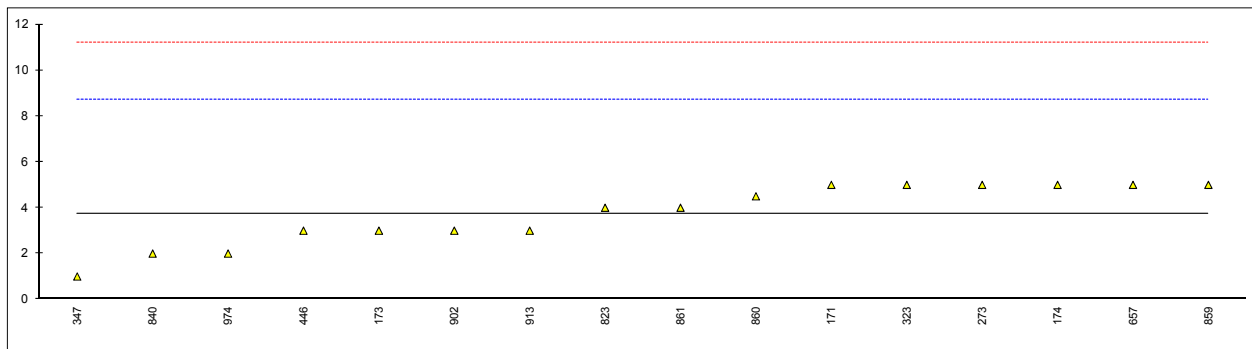
lab	method	value	mark	z(targ)	remarks
171	D4052	0.9338		-0.38	
173	D4052	0.9339		0.18	
174	D4052	0.9339		0.18	
273	D4052	0.9338		-0.38	
311	D4052	0.9339		0.18	
323	D4052	0.9339		0.18	
337		----		----	
347	D4052	0.9339		0.18	
391	D4052	0.9338		-0.38	
395	D4052	0.9339		0.18	
446	D4052	0.9339	C	0.18	first reported: 0.9039
522		----		----	
551		----		----	
557		----		----	
657	D4052	0.93383		-0.21	
786		----		----	
823	D4052	0.93385		-0.10	
840	D4052	0.93386		-0.05	
859	D4052	0.9339		0.18	
860	D4052	0.93391		0.23	
861	D4052	0.93378		-0.49	
902	D4052	0.9339		0.18	
913	D4052	0.9338		-0.38	
963		----		----	
974	D4052	0.9339		0.18	
1107	D4052	0.9339		0.18	
1429		0.9339		0.18	

normality OK  
n 21  
outliers 0  
mean (n) 0.93387  
st.dev. (n) 0.000046  
R(calc.) 0.00013  
R(D4052:16) 0.00050



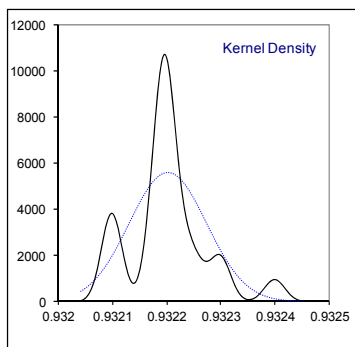
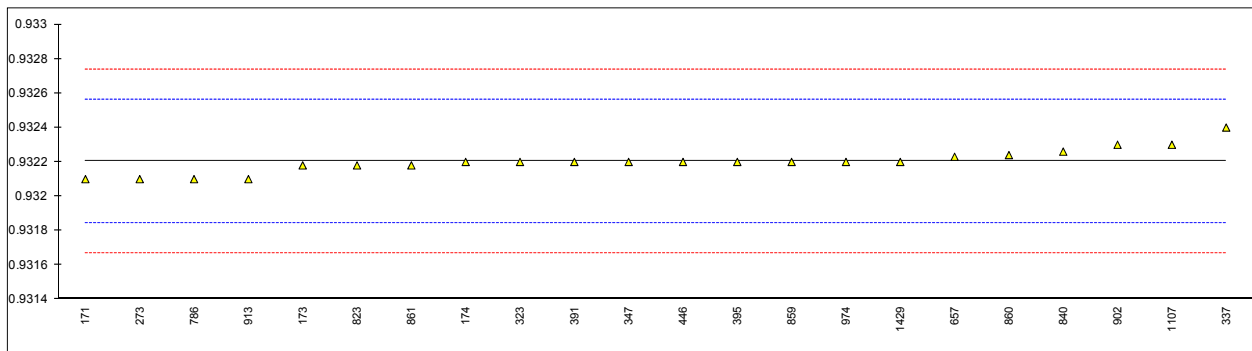
Determination of Colour Pt/Co on sample #17001;

lab	method	value	mark	z(targ)	remarks
171	D1209	5		0.51	
173	D1209	3		-0.29	
174	D1209	5		0.51	
273	D1209	5		0.51	
311		----		----	
323	D1209	5		0.51	
337		----		----	
347	D5386	1		-1.09	
391	D1209	<5		----	
395	D1209	<5		----	
446	D5386	3		-0.29	
522		----		----	
551		----		----	
557		----		----	
657	D1209	5		0.51	
786	D1209	<5		----	
823	D1209	4		0.11	
840	D1209	2		-0.69	
859	D1209	5		0.51	
860	D5386	4.5		0.31	
861	D1209	4		0.11	
902	D5386	3		-0.29	
913	D5386	3		-0.29	
963		----		----	
974	D5386	2		-0.69	
1107		----		----	
1429		<5		----	
normality		OK			
n		16			
outliers		0			
mean (n)		3.72			
st.dev. (n)		1.316			
R(calc.)		3.69			
R(D1209:05)		7.00			



Determination of Density at 20°C on sample #17001; results in kg/L

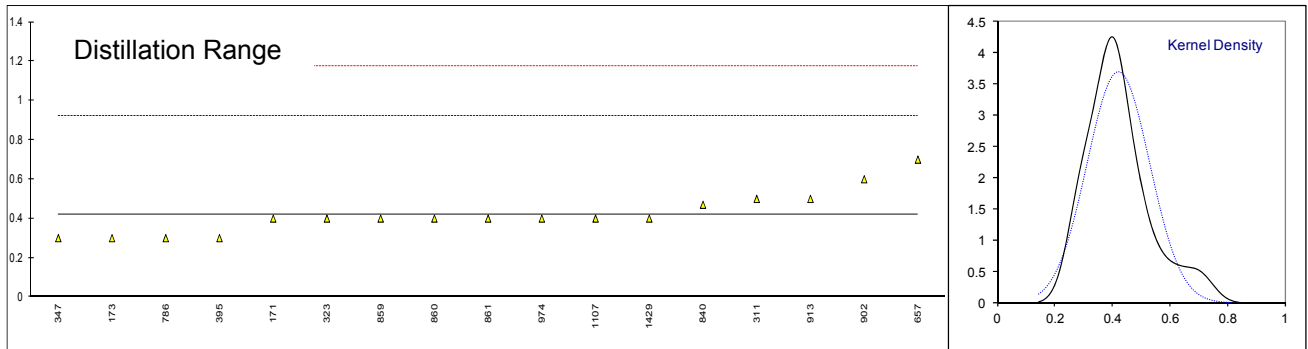
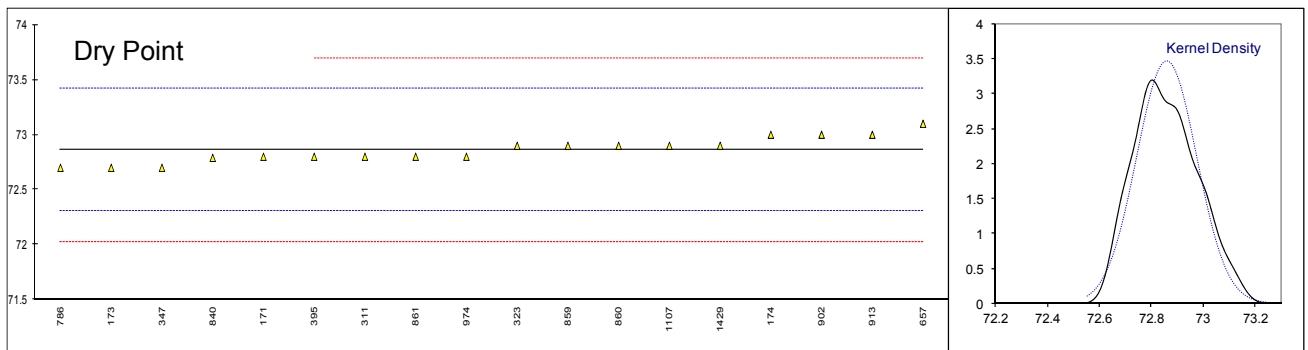
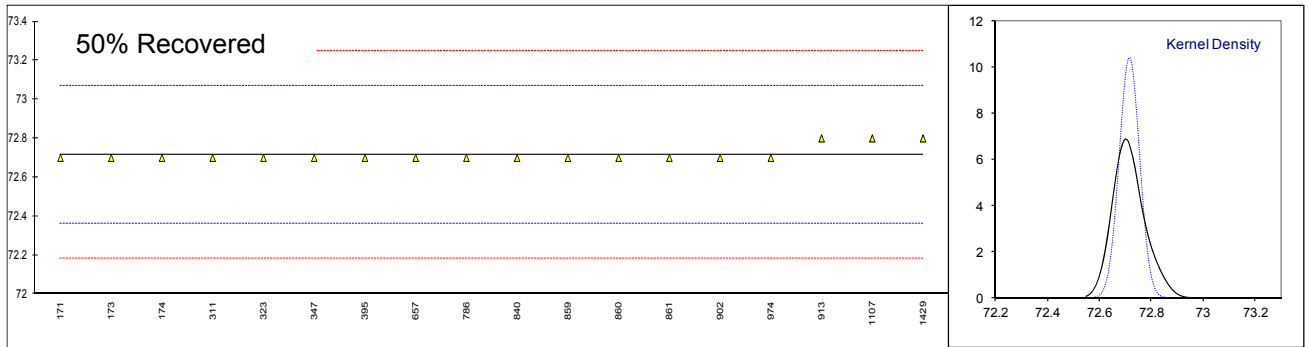
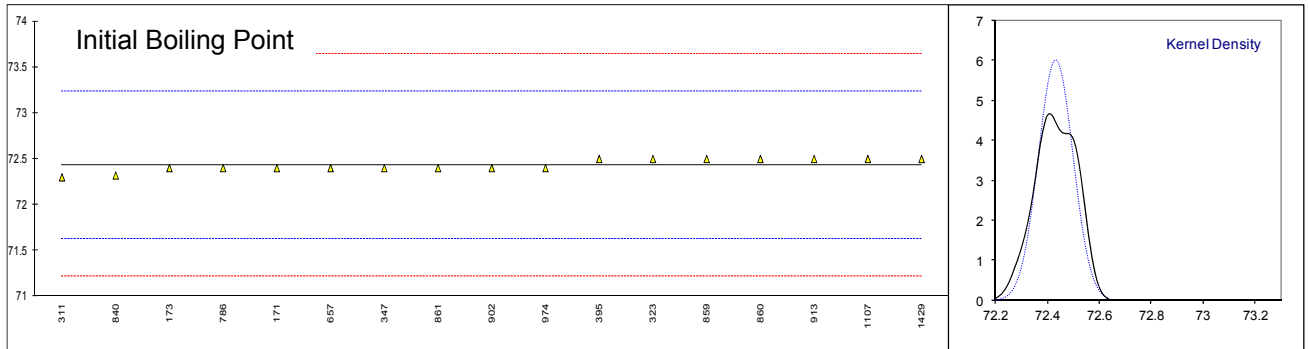
lab	method	value	mark	z(targ)	remarks
171	D4052	0.9321		-0.58	
173	D4052	0.93218		-0.13	
174	D4052	0.9322		-0.02	
273	D4052	0.9321		-0.58	
311		----		----	
323	D4052	0.9322		-0.02	
337	ISO12185	0.9324		1.10	
347	D4052	0.9322		-0.02	
391	D4052	0.9322		-0.02	
395	D4052	0.9322		-0.02	
446	D4052	0.9322		-0.02	
522		----		----	
551		----		----	
557		----		----	
657	D4052	0.93223		0.15	
786	D4052	0.9321		-0.58	
823	D4052	0.93218		-0.13	
840	D4052	0.93226		0.32	
859	D4052	0.9322		-0.02	
860	D4052	0.93224		0.21	
861	D4052	0.93218		-0.13	
902	D4052	0.9323		0.54	
913	D4052	0.9321		-0.58	
963		----		----	
974	D4052	0.9322		-0.02	
1107	D4052	0.9323		0.54	
1429		0.9322		-0.02	
normality		suspect			
n		22			
outliers		0			
mean (n)		0.93220			
st.dev. (n)		0.000071			
R(calc.)		0.00020			
R(D4052:16)		0.00050			





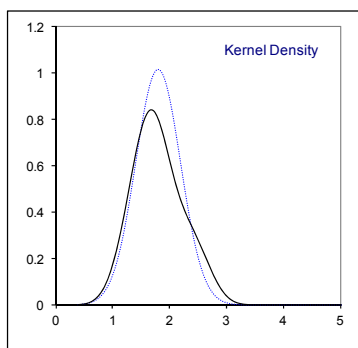
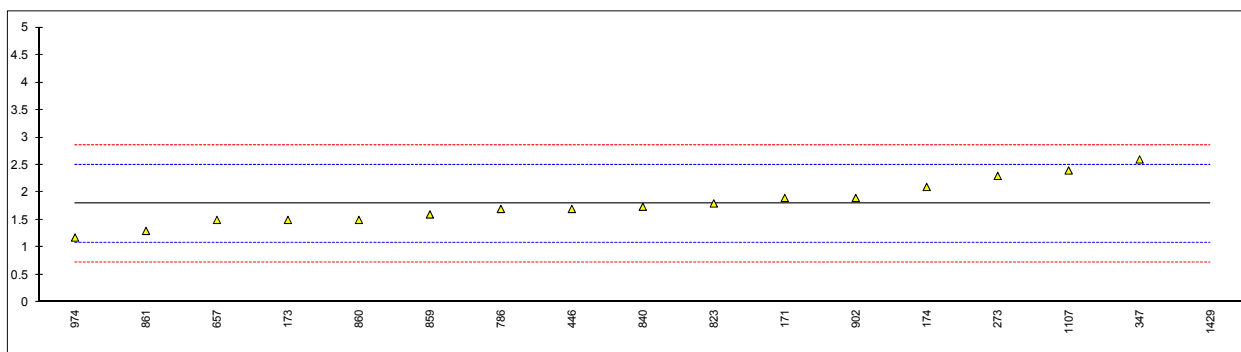
Determination of Distillation on sample #17001; results in °C

lab	method	mode	IBP	mark	z(targ)	50%rec. mark	z(targ)	DP	mark	z(targ)	range	mark	z(targ)
171	D1078	Auto.	72.4		-0.08	72.7	-0.09	72.8		-0.22	0.4		-0.09
173	D1078	Auto.	72.4		-0.08	72.7	-0.09	72.7		-0.58	0.3		-0.49
174	D1078	Auto.	----		----	72.7	-0.09	73.0		0.50	----		----
273			----		----	----	----	----		----	----		----
311		Auto.	72.3		-0.32	72.7	-0.09	72.8		-0.22	0.5		0.31
323	D1078	Man.	72.5		0.17	72.7	-0.09	72.9		0.14	0.4		-0.09
337			----		----	----	----	----		----	----		----
347	D1078	Auto.	72.4		-0.08	72.7	-0.09	72.7		-0.58	0.3		-0.49
391			----		----	----	----	----		----	----		----
395	D1078	Man.	72.5		0.17	72.7	-0.09	72.8		-0.22	0.3		-0.49
446			----		----	----	----	----		----	----		----
522			----		----	----	----	----		----	----		----
551			----		----	----	----	----		----	----		----
557			----		----	----	----	----		----	----		----
657	D1078	Man.	72.4		-0.08	72.7	-0.09	73.1		0.86	0.7		1.11
786	D1078	Man.	72.4		-0.08	72.7	-0.09	72.7		-0.58	0.3		-0.49
823	D1078	Auto.	----		----	----	----	----		----	----		----
840	D1078	Auto.	72.32		-0.27	72.7	-0.09	72.79		-0.25	0.47		0.19
859	D1078	Man.	72.5		0.17	72.7	-0.09	72.9		0.14	0.4		-0.09
860	D1078	Man.	72.5		0.17	72.7	-0.09	72.9		0.14	0.4		-0.09
861	D1078	Man.	72.4		-0.08	72.7	-0.09	72.8		-0.22	0.4		-0.09
902	D1078	Auto.	72.4		-0.08	72.7	-0.09	73.0		0.50	0.6		0.71
913	D1078	Man.	72.5		0.17	72.8	0.47	73.0		0.50	0.5		0.31
963			----		----	----	----	----		----	----		----
974	D1078	Auto.	72.4		-0.08	72.7	-0.09	72.8		-0.22	0.4		-0.09
1107	D1078	Auto.	72.5		0.17	72.8	0.47	72.9		0.14	0.4		-0.09
1429	D1078	Auto.	72.5		0.17	72.8	0.47	72.9		0.14	0.4		-0.09
normality			OK			not OK		OK					not OK
n			17			18		18					17
outliers			0			0		0					0
mean (n)			72.43			72.72		72.86			0.42		
st.dev. (n)			0.066			0.038		0.115			0.108		
R(calc.)			0.19			0.11		0.32			0.30		
R(D1078-A:11)			1.13			0.50		0.78			0.70		



Determination of Inhibitor as Hydroquinone on sample #17001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2193	1.9	C	0.29	first reported: 4.1
173	D2193	1.5		-0.83	
174	D2193	2.1		0.85	
273	D2193	2.3		1.41	
311		----		----	
323	D2193	<2		----	
337		----		----	
347	D2193	2.6		2.25	
391		----		----	
395		----		----	
446	D2193	1.7		-0.27	
522		----		----	
551		----		----	
557		----		----	
657	D2193	1.5	C	-0.83	first reported: 3.41
786	D2193	1.7		-0.27	
823	D2193	1.8		0.01	
840	D2193	1.74		-0.15	
859	D2193	1.6		-0.55	
860	D2193	1.5		-0.83	
861	D2193	1.30		-1.39	
902	D2193	1.9		0.29	
913		----		----	
963		----		----	
974	D2193	1.18		-1.72	
1107	In house	2.4		1.69	
1429		10.6	D(0.01)	24.65	
normality		OK			
n		16			
outliers		1			
mean (n)		1.79			
st.dev. (n)		0.394			
R(calc.)		1.10			
R(D2193:06)		1.00			

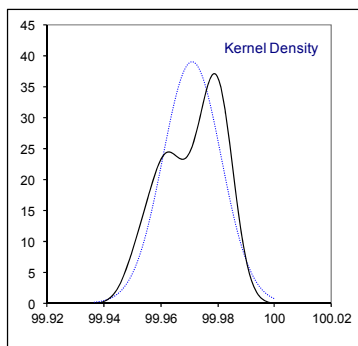
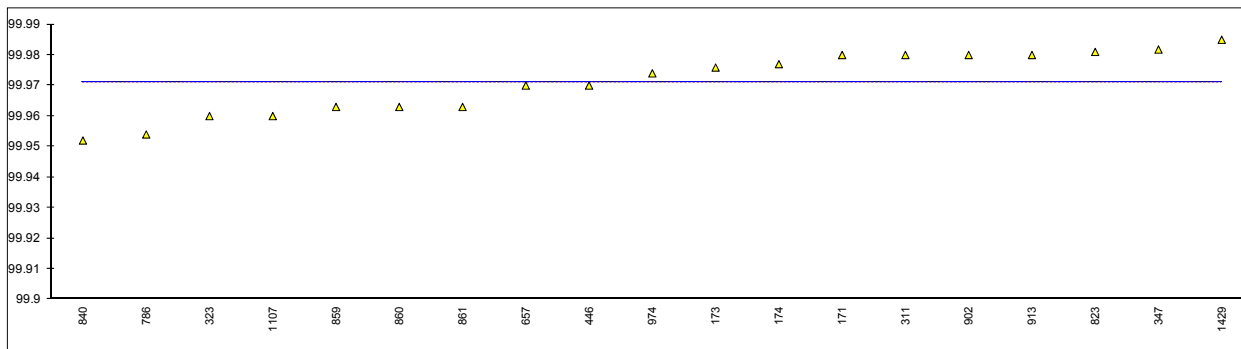


Determination of Purity on sample #17001; results in % M/M

lab	method	value	mark	z(targ)	remarks
171	INH-1-12	99.98		----	
173	INH-257	99.9759		----	
174	In house	99.977		----	
273		----		----	
311	INH-122	99.98		----	
323		99.96		----	
337		----		----	
347		99.9818		----	
391		----		----	
395		----		----	
446	INH-102582	99.97		----	
522		----		----	
551		----		----	
557		----		----	
657	INH-0047	99.97		----	
786	INH-9955	99.954		----	
823	INH-021	99.981		----	
840	SH/T1628.2	99.952		----	
859	SH/T1628.2	99.963		----	
860	SH/T1628.2	99.963		----	
861	SH/T1628.2	99.963		----	
902	INH/22	99.98		----	
913	INH-12345	99.98		----	
963		----		----	
974		99.974		----	
1107	In house	99.96		----	
1429		99.985		----	

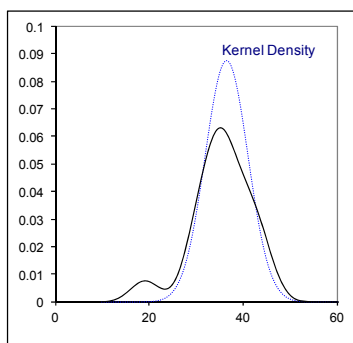
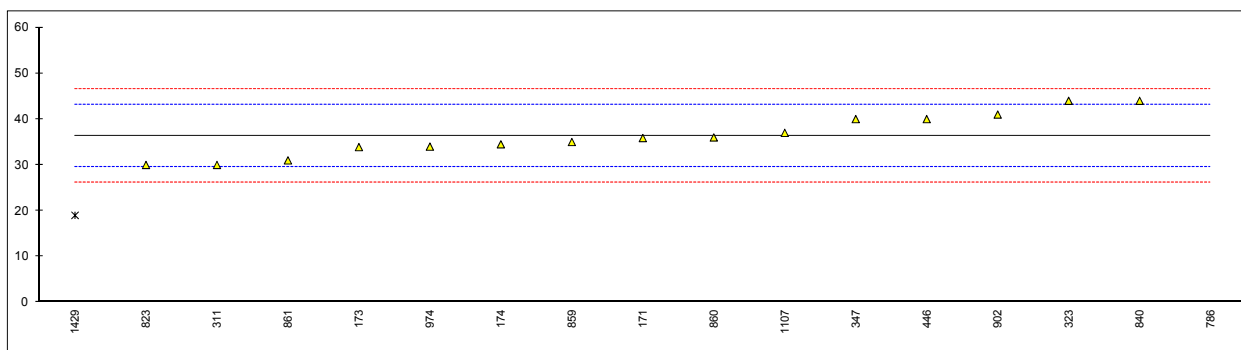
normality OK  
n 19  
outliers 0  
mean (n) 99.9710  
st.dev. (n) 0.01025  
R(calc.) 0.0287  
R(lit.) unknown

Compare R(iis16C01)=0.0112



Determination of Acetaldehyde on sample #17001; results in mg/kg

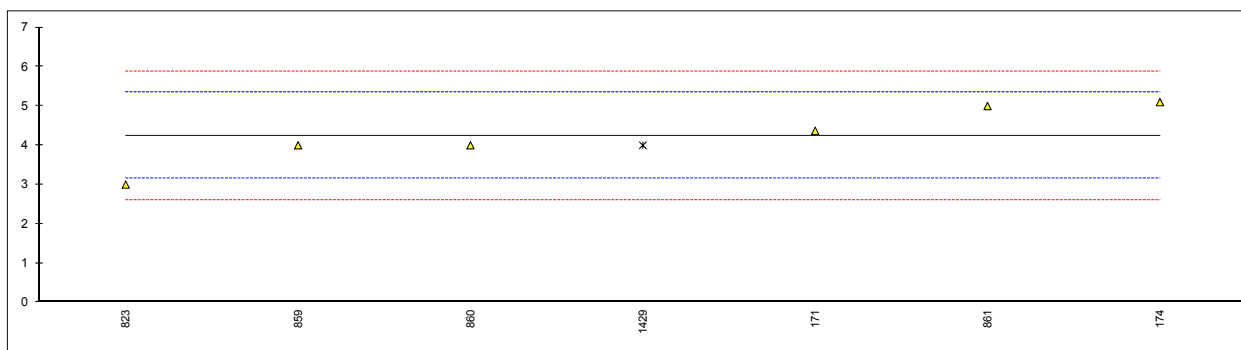
lab	method	value	mark	z(targ)	remarks
171	INH-1-12	35.87		-0.16	
173	INH-47	33.9		-0.74	
174	In house	34.5		-0.57	
273		----		----	
311	INH-122	30		-1.89	
323		44		2.24	
337		----		----	
347		40		1.06	
391		----		----	
395		----		----	
446	INH-102582	40		1.06	
522		----		----	
551		----		----	
557		----		----	
657		----		----	
786	INH-9955	126	C,G(0.01)	26.41	first reported: 86
823	INH-021	30		-1.89	
840	SH/T1628.2	44		2.24	
859	SH/T1628.2	35		-0.42	
860	SH/T1628.2	36		-0.12	
861	SH/T1628.2	31		-1.60	
902	INH/22	41		1.35	
913		----		----	
963		----		----	
974		34		-0.71	
1107	In house	37		0.17	
1429		19	G(0.05)	-5.14	
normality		OK			
n		15			
outliers		2			
mean (n)		36.42			
st.dev. (n)		4.567			
R(calc.)		12.79			
R(Horwitz)		9.50			



Determination of Acetone on sample #17001, results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-1-12	4.37		0.23	
173	INH-47	<10		----	
174	In house	5.1		1.56	
273		----		----	
311	INH-122	<10		----	
323		<10		----	
337		----		----	
347		<10		----	
391		----		----	
395		----		----	
446		----		----	
522		----		----	
551		----		----	
557		----		----	
657	INH-0047	<10		----	
786	INH-9955	<10		----	
823	INH-021	3		-2.28	
840	SH/T1628.2	<10		----	
859	SH/T1628.2	4		-0.45	
860	SH/T1628.2	4		-0.45	
861	SH/T1628.2	5		1.38	
902	INH/22	<10		----	
913	INH-12345	<5		----	
963		----		----	
974		<10		----	
1107		----		----	
1429		4	ex	-0.45	two out of four test results in GC analysis are outliers

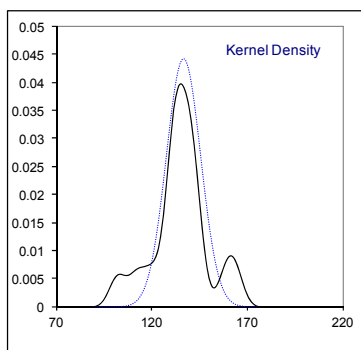
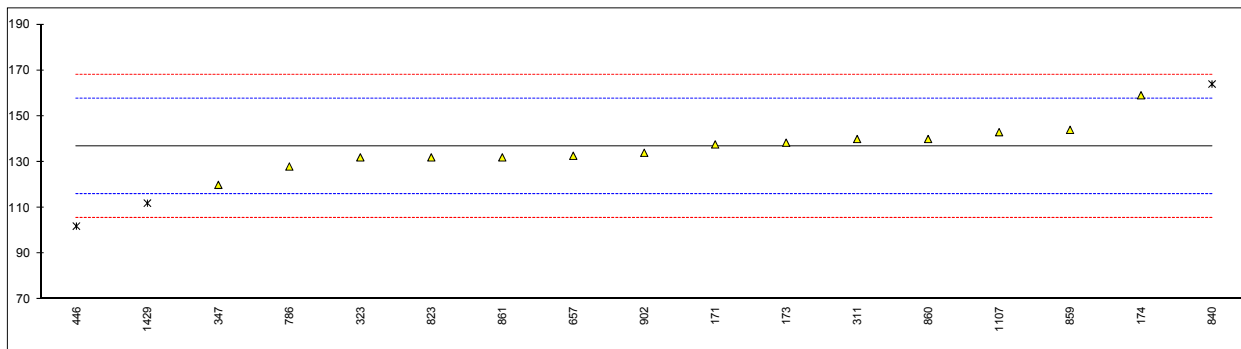
normality unknown  
n 6  
outliers 0+1ex  
mean (n) 4.24  
st.dev. (n) 0.773  
R(calc.) 2.16  
R(Horwitz) 1.53



Determination of Ethyl Acetate on sample #17001, results in mg/kg

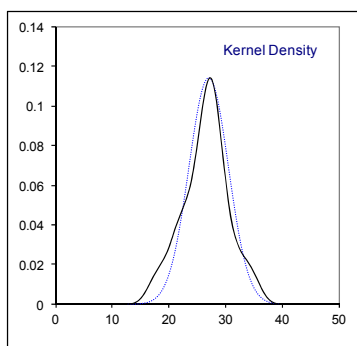
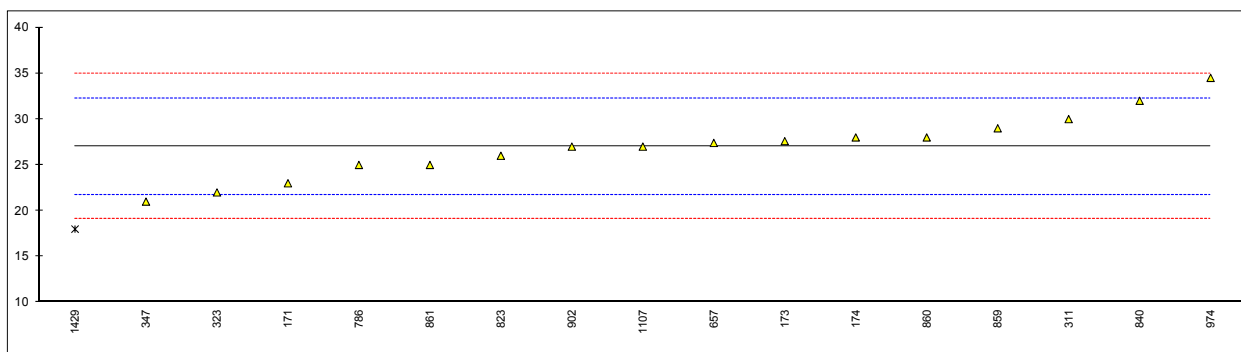
lab	method	value	mark	z(targ)	remarks
171	INH-1-12	137.64		0.10	
173	INH-47	138.4		0.17	
174	In house	159.1		2.15	
273		----		----	
311	INH-122	140		0.32	
323		132		-0.44	
337		----		----	
347		120		-1.59	
391		----		----	
395		----		----	
446	INH-102582	102	G(0.05)	-3.32	
522		----		----	
551		----		----	
557		----		----	
657	INH-0047	132.7		-0.38	
786	INH-9955	128		-0.83	
823	INH-021	132		-0.44	
840	SH/T1628.2	164	G(0.05)	2.62	
859	SH/T1628.2	144		0.71	
860	SH/T1628.2	140		0.32	
861	SH/T1628.2	132		-0.44	
902	INH/22	134		-0.25	
913		----		----	
963		----		----	
974		----		----	
1107	In house	143		0.61	
1429		112	ex	-2.36	two out of four test results in GC analysis are outliers

normality not OK  
n 14  
outliers 2+1ex  
mean (n) 136.63  
st.dev. (n) 9.048  
R(calc.) 25.34  
R(Horwitz) 29.20



Determination of Methyl Acetate on sample #17001; results in mg/kg

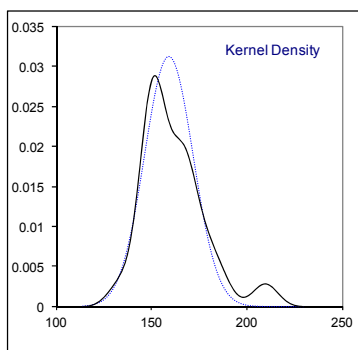
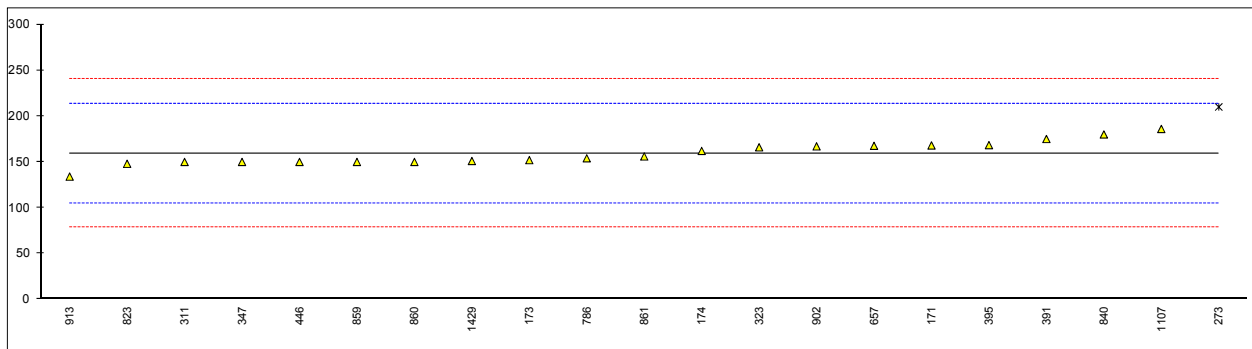
lab	method	value	mark	z(targ)	remarks
171	INH-1-12	23		-1.53	
173	INH-47	27.6		0.22	
174	In house	28		0.37	
273		----		----	
311	INH-122	30		1.13	
323		22		-1.91	
337		----		----	
347		21		-2.29	
391		----		----	
395		----		----	
446		----		----	
522		----		----	
551		----		----	
557		----		----	
657	INH-0047	27.4		0.14	
786	INH-9955	25		-0.77	
823	INH-021	26		-0.39	
840	SH/T1628.2	32		1.89	
859	SH/T1628.2	29		0.75	
860	SH/T1628.2	28		0.37	
861	SH/T1628.2	25		-0.77	
902	INH-122	27		-0.01	
913	INH-12345	<5		<-8.37	possibly a false negative test result?
963		----		----	
974		34.5	C	2.84	first reported: 4.5
1107	In house	27		-0.01	
1429		18	G(0.05)	-3.43	
	normality	OK			
	n	16			
	outliers	1			
	mean (n)	27.03			
	st.dev. (n)	3.491			
	R(calc.)	9.77			
	R(Horwitz)	7.37			





Determination of Water, titrimetric on sample #17001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	168		0.32	
173	E203	152		-0.27	
174	E203	162		0.10	
273	E203	210	G(0.05)	1.88	
311	D1364	150	C	-0.34	first reported: 50
323	D1364	166		0.25	
337		----		----	
347	D1364	150		-0.34	
391	E203	175		0.58	
395	D1364	168.34		0.34	
446	D1364	150		-0.34	
522		----		----	
551		----		----	
557		----		----	
657	E1064	167.6		0.31	
786	D1364	154		-0.19	
823	D1364	148		-0.42	
840	D1364	180		0.77	
859	D1364	150		-0.34	
860	D1364	150		-0.34	
861	D1364	156		-0.12	
902	D1364	167		0.29	
913	D1364	134		-0.93	
963		----		----	
974		----		----	
1107	D1364	186		0.99	
1429		151		-0.30	
	normality	OK			
	n	20			
	outliers	1			
	mean (n)	159.25			
	st.dev. (n)	12.746			
	R(calc.)	35.69			
	R(D1364:02)	75.72			



## **APPENDIX 2**

### **Number of participants per country**

2 labs in BELGIUM  
2 labs in BRAZIL  
3 labs in CHINA, People's Republic  
1 lab in FRANCE  
1 lab in INDIA  
2 labs in ITALY  
1 lab in MEXICO  
1 lab in NETHERLANDS  
1 lab in RUSSIAN FEDERATION  
1 lab in SAUDI ARABIA  
1 lab in SINGAPORE  
1 lab in SOUTH AFRICA  
1 lab in SOUTH KOREA  
1 lab in SPAIN  
1 lab in TURKEY  
1 lab in UNITED ARAB EMIRATES  
2 labs in UNITED KINGDOM  
3 labs in UNITED STATES OF AMERICA  
1 lab in VIETNAM

## APPENDIX 3

### Abbreviations:

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

### Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
- 2 ASTM E178:89
- 3 ASTM E1301:89
- 4 ISO 5725:86
- 5 ISO 5725, parts 1-6, 1994
- 6 ISO13528:05
- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367:84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical brief, No 4 January 2001
- 14 P.J. Lowthian and M.Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2),165-172, (1983)