Results of Proficiency Test
Vinyl Acetate Monomer
February 2018

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

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1 INTRODUCTION

Since 2007, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Vinyl Acetate Monomer (VAM) every year. During the annual proficiency testing program 2017/2018, it was decided to continue the round robin for the analysis of VAM in accordance with the latest applicable version of ASTM D2190 and some additional tests.
In this interlaboratory study 26 laboratories in 18 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2018 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample 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 #18001. 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 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).
The 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

Approximately 25 liter bulk sample was obtained from a local chemical supplier. After homogenization in a pre-cleaned can, 48 amber glass bottles of 0.5L were filled and labelled #18001. The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ISO12185 and Water in accordance with ASTM D1364 on respectively 8 and 7 stratified randomly selected samples.

	Density at 20°C in kg/L	*Water in mg/kg*
sample #18001-1	0.93219	59
sample #18001-2	0.93219	68
sample #18001-3	0.93219	80
sample #18001-4	0.93219	70
sample #18001-5	0.93219	60
sample #18001-6	0.93219	73
sample #18001-7	0.93219	77
sample #18001-8	0.93219	--

Table 1: homogeneity test results of subsamples #18001

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test method or with the corresponding repeatability of the reference test method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 20°C in kg/L	*Water in mg/kg*
r (observed)	0.00000	22
reference test method	ISO12185:96	ASTM D1364:02(2012)
0.3*R (reference test method)	0.00015	--
r (reference test method)	--	25

Table 2: evaluation of the repeatabilities of subsamples #18001

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

To each of the participating laboratories 1 bottle of 0.5 L VAM, labelled #18001, was sent on January 24, 2018. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of VAM packed in an amber glass bottle was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine on sample #18001; 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 and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods 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 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 visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve 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 or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other 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:

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
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 and Mexico received the samples late or not at all due to problems with custom clearance. Of the 26 participants, two participants reported the test results after the final reporting date and one other participant did not report any test result at all.

Not all participants were able to report test results for all the requested tests. Finally, 25 participants reported in total 282 numerical test results. Observed were 10 outlying test results, which is 3.5% 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 TEST

In this section, the reported test results are discussed per test.
The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also listed in the tables together with the original data. 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 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 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 (e.g. D2086:08) will be used.

Acidity: This determination (with and without Nitrogen purging) was problematic. The acidity value determined with Nitrogen purging was significantly different to the acidity “without Nitrogen purging” when using the standard deviation of the test method.
In the determination “without Nitrogen purging” three statistical outliers were observed and one statistical outlier in the determination “with Nitrogen purging”. The calculated reproducibilities of both determinations after rejection of the statistical outliers 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)).

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 ISO12185:96.

Colour 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 good agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibilities after rejection of the statistical outlier are all in good agreement with the requirements of ASTM D1078:11 (Automated method).

Inhibitor (Hydroquinone): This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM 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 smaller than observed in previous PT (iis17C01).

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

Acetone: No evaluation was made as all participants reported a “less than” test result.

Ethyl Acetate: The determination of this impurity was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the estimated reproducibility using the Horwitz equation.

Methyl Acetate: The determination of this impurity was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility using the Horwitz equation.

Water: 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 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_2 purging)	mg/kg	12	15.5	8.3	6
Acidity (with N_2 purging)	mg/kg	6	13.1	11.8	6
Apparent Specific Gravity 20/20°C		24	0.9339	0.0002	0.0005
Colour Pt/Co		18	3.3	3.1	7
Density at 20°C	kg/L	25	0.9322	0.0001	0.0005
Distillation, Initial Boiling Point	°C	19	72.5	0.4	1.1
Distillation, 50% recovery	°C	18	72.7	0.2	0.5
Distillation, Dry Point	°C	19	72.8	0.4	0.8
Distillation, Boiling Range	°C	18	0.35	0.39	0.69
Inhibitor as Hydroquinone	mg/kg	21	3.4	1.3	1.0
Purity	%M/M	20	99.975	(0.013)	(0.029)
Acetaldehyde	mg/kg	19	26.7	12.7	7.3
Ethyl Acetate	mg/kg	18	188	18	38
Methyl Acetate	mg/kg	13	8.3	2.4	2.7
Water	mg/kg	22	66.6	41.4	49.0

Table 3: reproducibilities of tests on sample #18001

The calculated reproducibility between brackets is compared against the reproducibility of the previous PT (iis17C01)

4.3 EVALUATION OF THE PROFICIENCY TEST OF FEBRUARY 2018 WITH PREVIOUS PTS

	February 2018	*February 2017*	*February 2016*	*February 2015*	*February 2014*
Number of reporting labs	25	23	25	20	23
Number of results reported	282	266	275	253	240
Number of statistical outliers	10	8	9	2	9
Percentage outliers	3.5%	3.0%	3.3%	0.8%	3.8%

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:

	February 2018	*February 2017*	*February 2016*	*February 2015*	*February 2014*
Acidity (without N_2 purging)	-	--	--	--	-
Acidity (with N_2 purging)	--	--	--	--	-
Apparent Specific Gravity	++	++	++	++	n.e.
Colour Pt/Co	++	++	++	+	n.e.
Density at 20°C	++	++	++	++	++
Distillation	++	++	++	++	++
Inhibitor as Hydroquinone	-	+/-	-	-	-
Purity	(++)	(--)	(+)	(+)	n.e.
Acetaldehyde	--	-	+/-	-	++
Acetone	n.e.	-	n.e.	-	n.e.
Ethyl Acetate	++	+	+/-	-	++
Methyl Acetate	+	-	+	-	-
Water	+	++	++	+	+/-

Table 5: comparison determinations against the reference test methods

The calculated reproducibility between brackets are compared against the reproducibility of the previous PT

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:

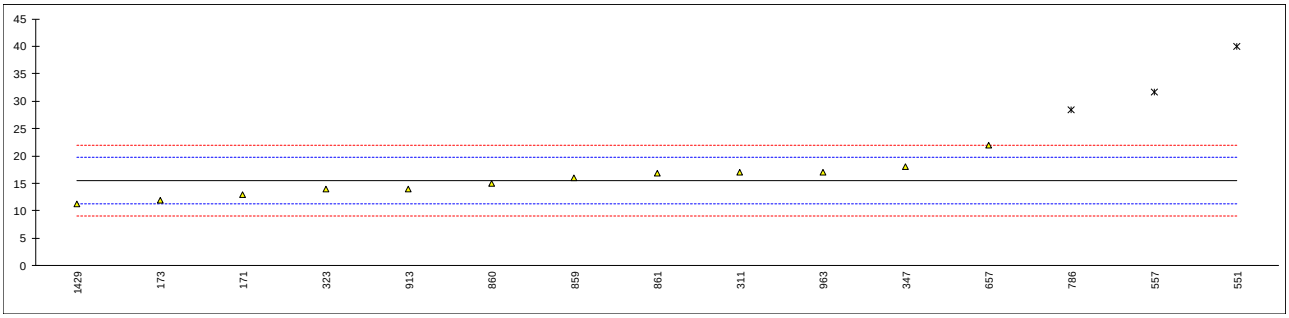
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test method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

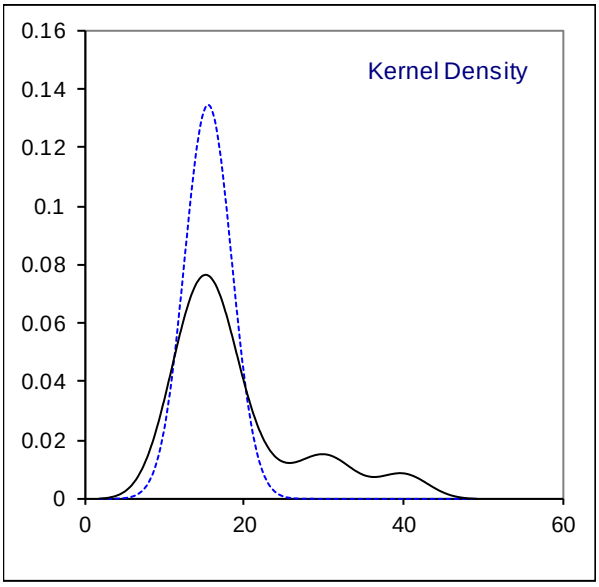
APPENDIX 1

Determination of Acidity (without N_2 purging) on sample #18001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2086	13		-1.17	
173	INH-14	12		-1.64	
174		-----		-----	
273		-----		-----	
311	D2086	17	C	0.70	First reported 0.0017 mg/kg
323	D2086	14		-0.70	
347	D2086	18.0		1.16	
391		-----		-----	
446		-----		-----	
522		-----		-----	
551	D2086	40	G(0.05)	11.43	
557	D2086	31.730	DG(0.01)	7.57	
657	D2086	22		3.03	
786	D2086	28.5	DG(0.01)	6.06	
823		-----		-----	
825		-----		-----	
840		-----		-----	
859	D2086	16		0.23	
860	D2086	15		-0.24	
861	D2086	16.9		0.65	
913	D2086	14		-0.70	
963	D2086	17	C	0.70	First reported 37
974		-----		-----	
1107		-----		-----	
1429	D2086	11.2		-2.01	
7006		-----		-----	

normality	OK
n	12
outliers	3
mean (n)	15.508
st.dev. (n)	2.9688
R(calc.)	8.313
st.dev.(D2086:08)	2.1429
R(D2086:08)	6

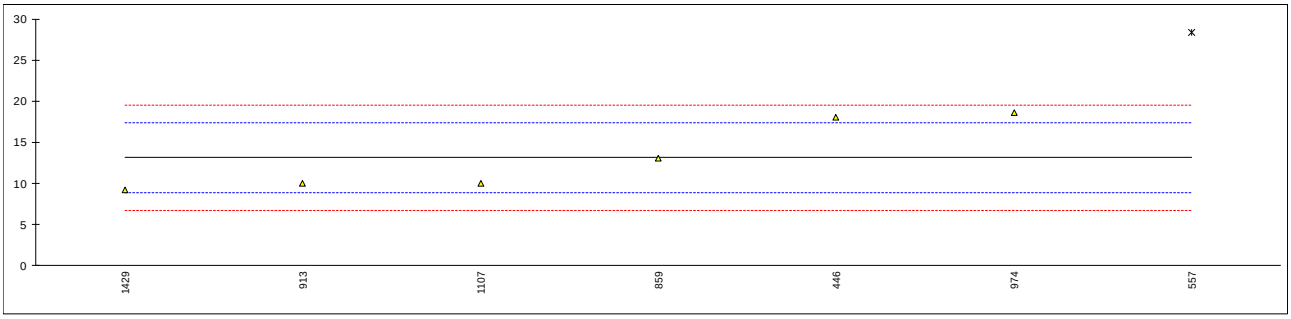


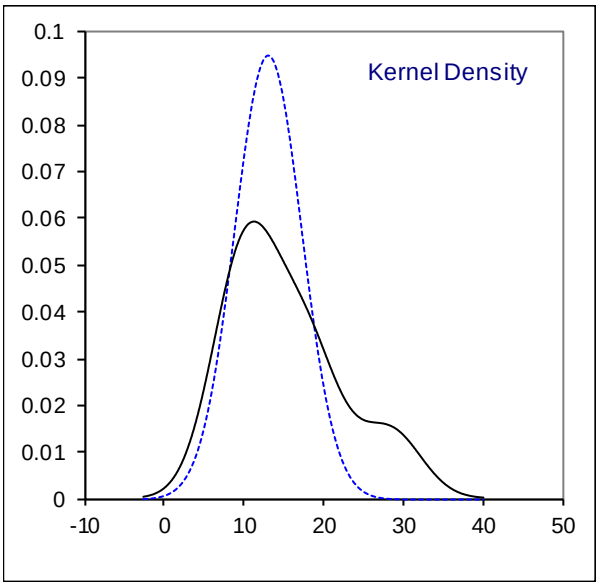


Determination of Acidity (with N_2 purging) on sample #18001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173		-----		-----	
174		-----		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
347		-----		-----	
391		-----		-----	
446	INH-40	18		2.27	
522		-----		-----	
551		-----		-----	
557	D2086	28.401	G(0.05)	7.13	
657		-----		-----	
786		-----		-----	
823		-----		-----	
825		-----		-----	
840		-----		-----	
859	D2086	13		-0.06	
860		-----		-----	
861		-----		-----	
913	D2086	10		-1.46	
963		-----		-----	
974	INH-40	18.6	C	2.55	First reported 37
1107	D2086	10		-1.46	
1429	D2086	9.2		-1.84	
7006		-----		-----	

normality	unknown
n	6
outliers	1
mean (n)	13.133
st.dev. (n)	4.2117
R(calc.)	11.793
st.dev.(D2086:08)	2.1429
R(D2086:08)	6

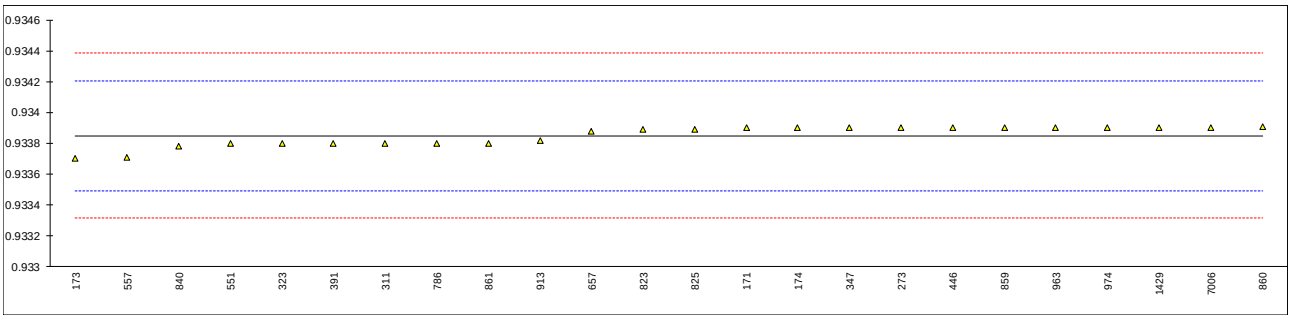


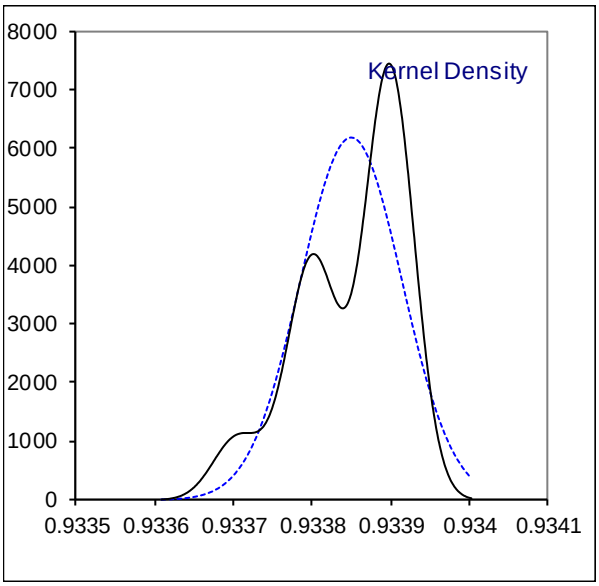


Determination of App. Specific Gravity 20/20°C on sample #18001;

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9339		0.28	
173	D4052	0.9337		-0.84	
174	D4052	0.9339		0.28	
273	D4052	0.9339		0.28	
311	D4052	0.9338		-0.28	
323	D4052	0.9338		-0.28	
347	D4052	0.93390		0.28	
391	ISO12185	0.9338		-0.28	
446	D4052	0.9339		0.28	
522		-----		-----	
551	D4052	0.9338		-0.28	
557	D4052	0.93371		-0.78	
657	D4052	0.93388		0.17	
786	D4052	0.9338		-0.28	
823	D4052	0.93389		0.23	
825	D4052	0.93389		0.23	
840	D4052	0.93378		-0.39	
859	D4052	0.9339		0.28	
860	D4052	0.93391		0.34	
861	D4052	0.9338		-0.28	
913	D4052	0.93382		-0.16	
963	D4052	0.9339		0.28	
974	D4052	0.9339		0.28	
1107		-----		-----	
1429	D4052	0.9339		0.28	
7006	D4052	0.9339		0.28	

normality	OK
n	24
outliers	0
mean (n)	0.93385
st.dev. (n)	0.000064
R(calc.)	0.00018
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

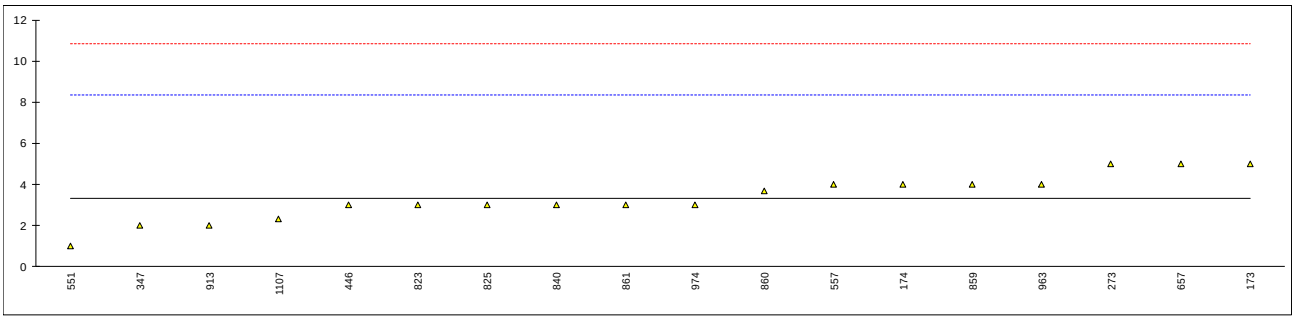


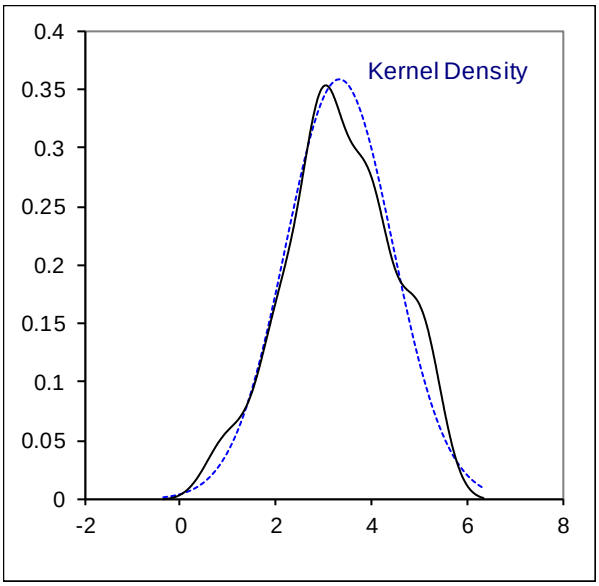


Determination of Colour Pt/Co on sample #18001;

lab	method	value	mark	z(targ)	remarks
171	D1209	<5		-----	
173	D1209	5		0.67	
174	D5386	4		0.27	
273	D1209	5		0.67	
311	D1209	<5		-----	
323	D1209	<5		-----	
347	D5386	2		-0.53	
391	D1209	<5		-----	
446	D5386	3		-0.13	
522		-----		-----	
551	D1209	1		-0.93	
557	D1209	4		0.27	
657	D1209	5		0.67	
786	D1209	<5		-----	
823	D5386	3		-0.13	
825	D1209	3		-0.13	
840	D1209	3		-0.13	
859	D1209	4		0.27	
860	D5386	3.7		0.15	
861	D1209	3		-0.13	
913	D5386	2		-0.53	
963	D1209	4		0.27	
974	D5386	3		-0.13	
1107	D5386	2.3		-0.41	
1429	D1209	< 5		-----	
7006		-----		-----	

normality	OK
n	18
outliers	0
mean (n)	3.33
st.dev. (n)	1.111
R(calc.)	3.11
st.dev.(D1209:05)	2.5
R(D1209:05)	7

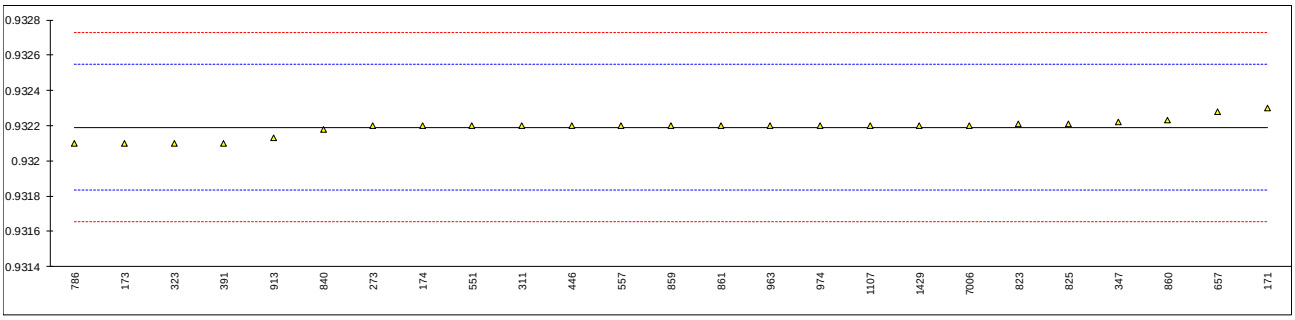


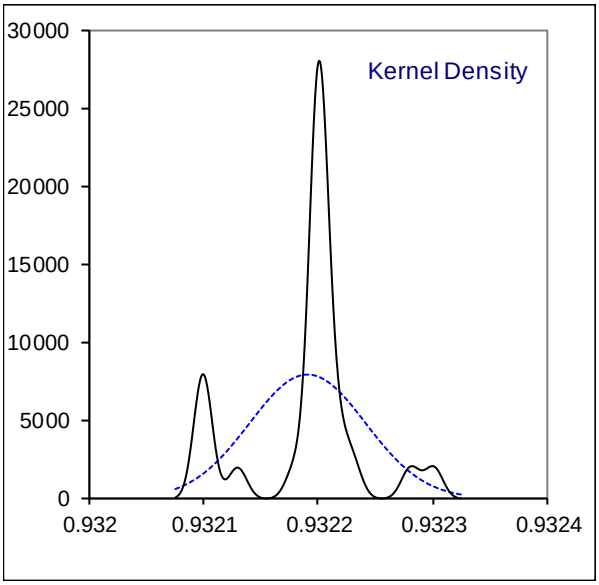


Determination of Density at 20°C on sample #18001; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9323		0.61	
173	D4052	0.9321		-0.51	
174	D4052	0.9322		0.05	
273	D4052	0.9322		0.05	
311	D4052	0.9322		0.05	
323	D4052	0.9321		-0.51	
347	D4052	0.93222		0.16	
391	ISO12185	0.9321		-0.51	
446	D4052	0.9322		0.05	
522		-----		-----	
551	D4052	0.9322	C	0.05	First reported 0.9312
557	D4052	0.93220		0.05	
657	D4052	0.93228		0.50	
786	D4052	0.9321		-0.51	
823	D4052	0.93221		0.11	
825	D4052	0.93221		0.11	
840	D4052	0.93218		-0.06	
859	D4052	0.9322		0.05	
860	D4052	0.93223		0.22	
861	D4052	0.9322		0.05	
913	D4052	0.93213		-0.34	
963	D4052	0.9322		0.05	
974	D4052	0.9322		0.05	
1107	D4052	0.9322		0.05	
1429	D4052	0.9322		0.05	
7006	D4052	0.9322		0.05	

normality	OK
n	25
outliers	0
mean (n)	0.93219
st.dev. (n)	0.000050
R(calc.)	0.00014
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

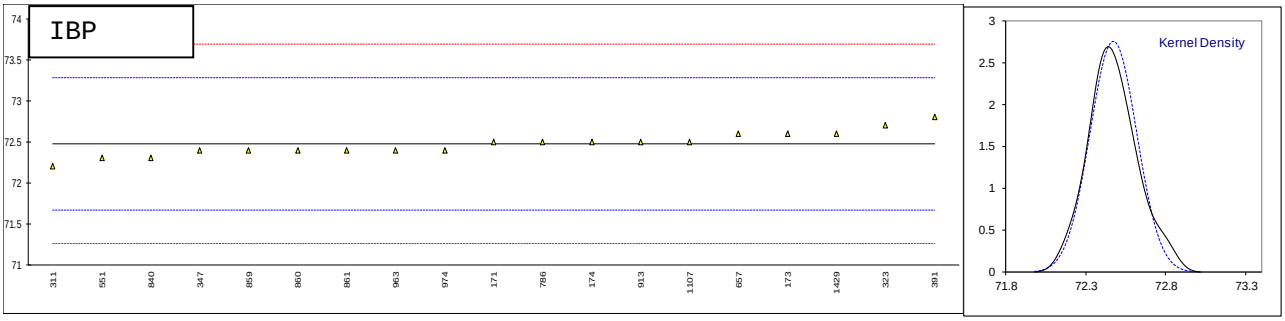


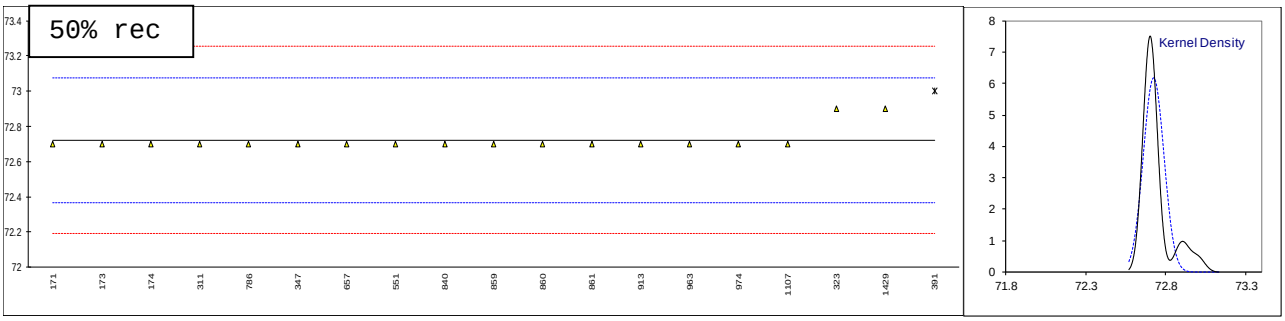


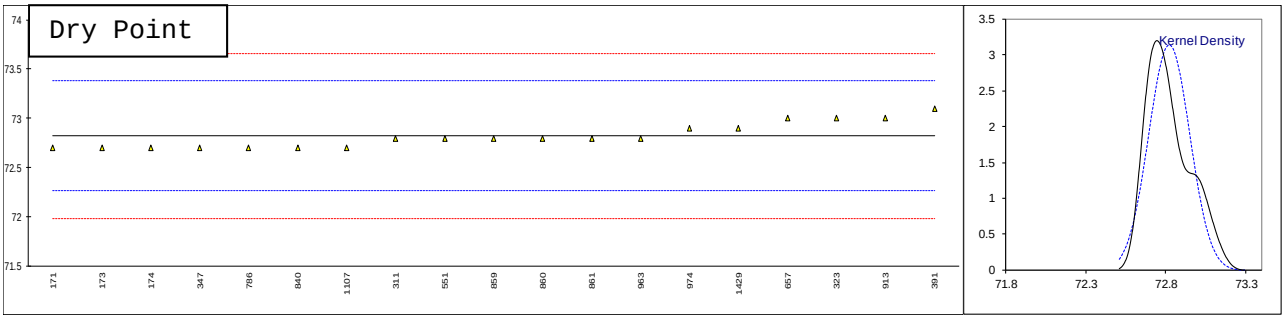
Determination of Distillation on sample #18001; results in °C

lab	method	IBP	z(targ)	50%rec		z(targ)	DP		z(targ)	range	z(targ)
171	D1078-automated	72.5	0.07	72.7		-0.13	72.7		-0.44	0.2	-0.61
173	D1078-automated	72.6	0.31	72.7		-0.13	72.7		-0.44	0.1	-1.01
174	D1078-automated	72.5	0.07	72.7		-0.13	72.7		-0.44	0.2	-0.61
273	-----	-----	-----	-----		-----	-----		-----	-----	-----
311	D1078-automated	72.2	-0.68	72.7		-0.13	72.8	C	-0.08	0.6	1.01
323	D1078-manual	72.7	0.56	72.9		1.00	73.0		0.64	0.3	-0.20
347	D1078-automated	72.4	-0.18	72.7		-0.13	72.7		-0.44	-----	-----
391	D1078-automated	72.8	0.81	73.0	G(0.05)	1.57	73.1		1.00	0.3	-0.20
446	-----	-----	-----	-----		-----	-----		-----	-----	-----
522	-----	-----	-----	-----		-----	-----		-----	-----	-----
551	D1078-automated	72.3	-0.43	72.7		-0.13	72.8		-0.08	0.5	0.61
557	-----	-----	-----	-----		-----	-----		-----	-----	-----
657	D1078-manual	72.6	0.31	72.7		-0.13	73.0		0.64	0.4	0.20
786	-----	72.5	0.07	72.7		-0.13	72.7		-0.44	0.2	-0.61
823	-----	-----	-----	-----		-----	-----		-----	-----	-----
825	-----	-----	-----	-----		-----	-----		-----	-----	-----
840	D1078-automated	72.3	-0.43	72.7		-0.13	72.7		-0.44	0.4	0.20
859	D1078-manual	72.4	-0.18	72.7		-0.13	72.8		-0.08	0.4	0.20
860	D1078-manual	72.4	-0.18	72.7		-0.13	72.8		-0.08	0.4	0.20
861	D1078-manual	72.4	-0.18	72.7		-0.13	72.8		-0.08	0.4	0.20
913	D1078-manual	72.5	0.07	72.7		-0.13	73.0		0.64	0.5	0.61
963	D1078-automated	72.4	-0.18	72.7		-0.13	72.8		-0.08	0.4	0.20
974	D1078-automated	72.4	-0.18	72.7		-0.13	72.9		0.28	0.5	0.61
1107	-----	72.5	0.07	72.7		-0.13	72.7		-0.44	0.2	-0.61
1429	-----	72.6	0.31	72.9		1.00	72.9		0.28	0.3	-0.20
7006	-----	-----	-----	-----		-----	-----		-----	-----	-----
	normality	OK		not OK			OK			OK	
	n	19		18			19			18	
	outliers	0		1			0			0	
	mean (n)	72.47		72.72			72.82			0.35	
	st.dev. (n)	0.145		0.065			0.127			0.134	
	R(calc.)	0.41		0.18			0.36			0.38	
	st.dev.(D1078-A:11)	0.404		0.177			0.278			0.247	
	R(D1078-A:11)	1.13		0.50			0.78			0.69	

Lab 311: first reported 73.4



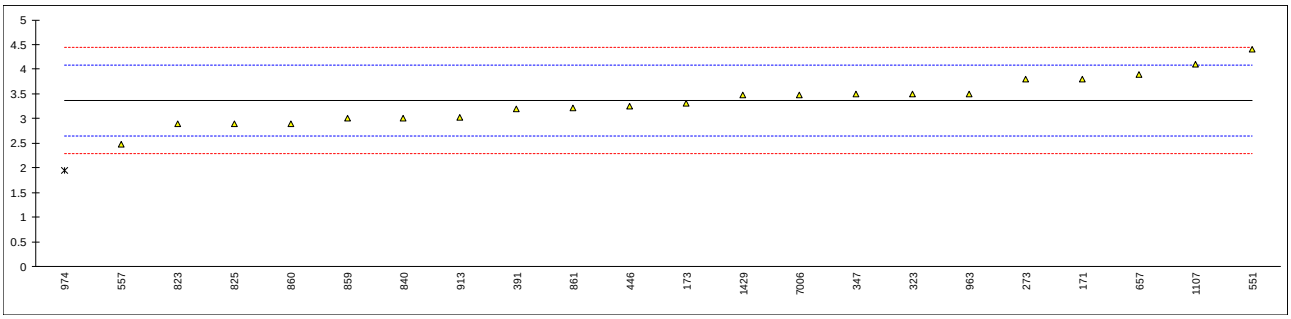


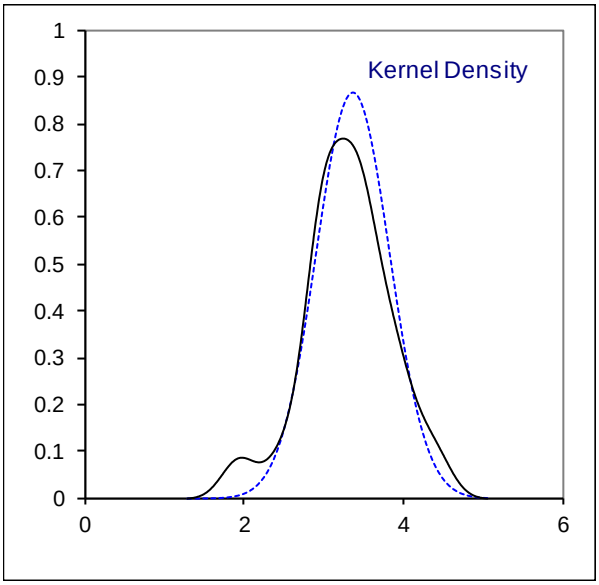


Determination of Inhibitor as Hydroquinone on sample #18001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2193	3.8		1.22	
173	D2193	3.3		-0.18	
174		-----		-----	
273	D2193	3.8		1.22	
311		-----		-----	
323	D2193	3.5		0.38	
347	D2193	3.50		0.38	
391	D2193	3.2		-0.46	
446	INH-40	3.25		-0.32	
522		-----		-----	
551	D2193	4.4		2.90	
557	D2193	2.483		-2.47	
657	D2193	3.9		1.50	
786		-----		-----	
823	D2193	2.9		-1.30	
825	D2193	2.9		-1.30	
840	D2193	3.01		-0.99	
859	D2193	3.0		-1.02	
860	D2193	2.9		-1.30	
861	D2193	3.22		-0.41	
913	D2193	3.03		-0.94	
963	D2193	3.5		0.38	
974	D2193	1.94	D(0.05)	-3.99	
1107	In house	4.1		2.06	
1429	D2193	3.48		0.32	
7006	D2193	3.486		0.34	

normality	OK
n	21
outliers	1
mean (n)	3.36
st.dev. (n)	0.459
R(calc.)	1.29
st.dev.(D2193:06)	0.357
R(D2193:06)	1.0

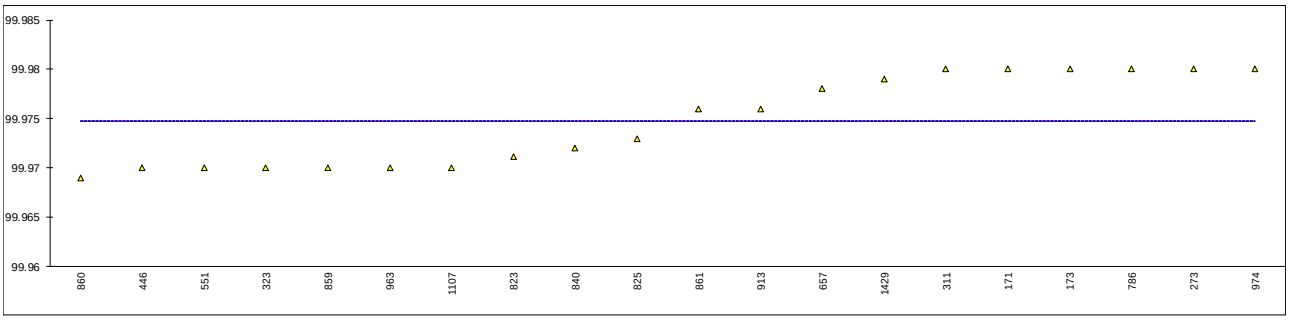


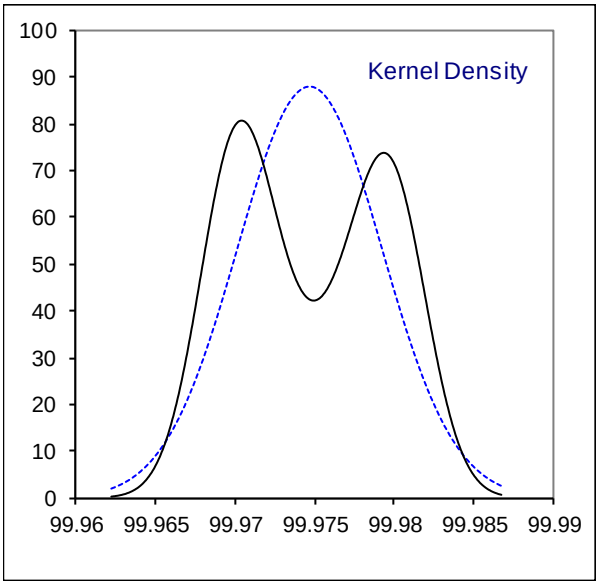


Determination of Purity on sample #18001; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	INH-0001	99.98		-----	
173	INH-257	99.98		-----	
174		-----		-----	
273	INH-102582	99.98		-----	
311	INH-122	99.98		-----	
323	INH-067	99.97		-----	
347		-----		-----	
391		-----		-----	
446	INH-102582	99.97		-----	
522		-----		-----	
551	INH-1355	99.97		-----	
557		-----		-----	
657	INH-0047	99.9781		-----	
786	INH-88	99.98		-----	
823	INH-021	99.9712		-----	
825	INH-021	99.973		-----	
840	INH-001	99.972		-----	
859	SH/T1628.2	99.97		-----	
860	SH/T1628.2	99.969		-----	
861	SH/T1628.2	99.976		-----	
913		99.976		-----	
963	In house	99.970		-----	
974	INH-102582	99.98		-----	
1107	In house	99.97		-----	
1429		99.979		-----	
7006		-----		-----	

	normality	OK
	n	20
	outliers	0
	mean (n)	99.9747
	st.dev. (n)	0.00453
	R(calc.)	0.0127
	st.dev.(lit.)	unknown
	R(lit.)	unknown
Compare		
	R(iis17C01)	0.0287

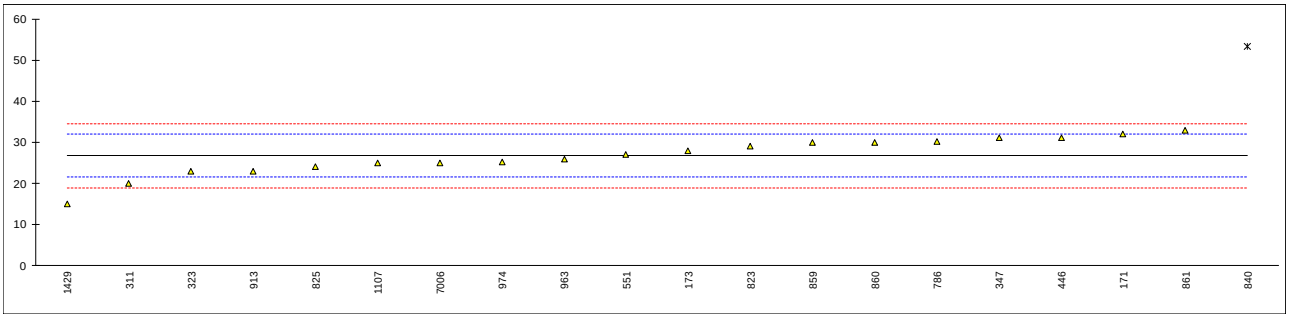


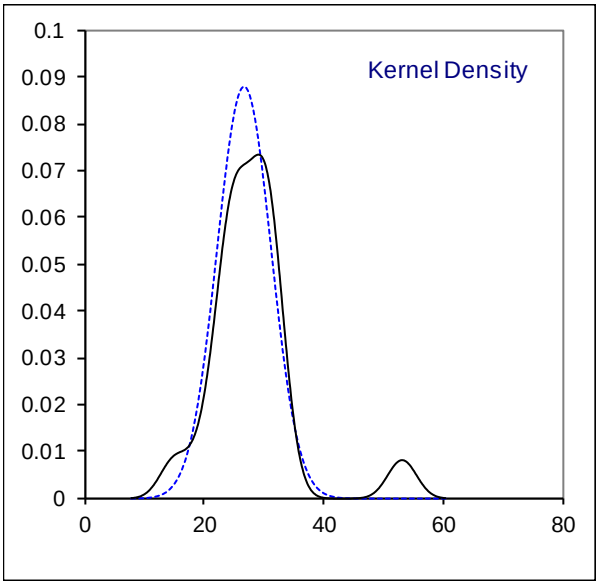


Determination of Acetaldehyde on sample #18001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-0001	32		2.03	
173	INH-47	28		0.50	
174		-----		-----	
273		-----		-----	
311	INH-122	20		-2.57	
323	INH-067	23		-1.42	
347	INH-096	31		1.65	
391		-----		-----	
446	INH-102582	31		1.65	
522		-----		-----	
551	INH-1355	27		0.11	
557		-----		-----	
657		-----		-----	
786	INH-88	30	C	1.38	First reported 47
823	INH-021	29		0.88	
825	INH-021	24		-1.04	
840	INH-001	53.3	R(0.01)	10.20	
859	SH/T1628.2	30		1.26	
860	SH/T1628.2	30		1.26	
861	SH/T1628.2	33		2.42	
913		23		-1.42	
963	In house	25.9		-0.31	
974	INH-102582	25.2	C	-0.58	First reported 7
1107	In house	25		-0.65	
1429		15		-4.49	
7006		25.0		-0.65	

normality	OK
n	19
outliers	1
mean (n)	26.71
st.dev. (n)	4.544
R(calc.)	12.72
st.dev.(Horwitz)	2.606
R(Horwitz)	7.30





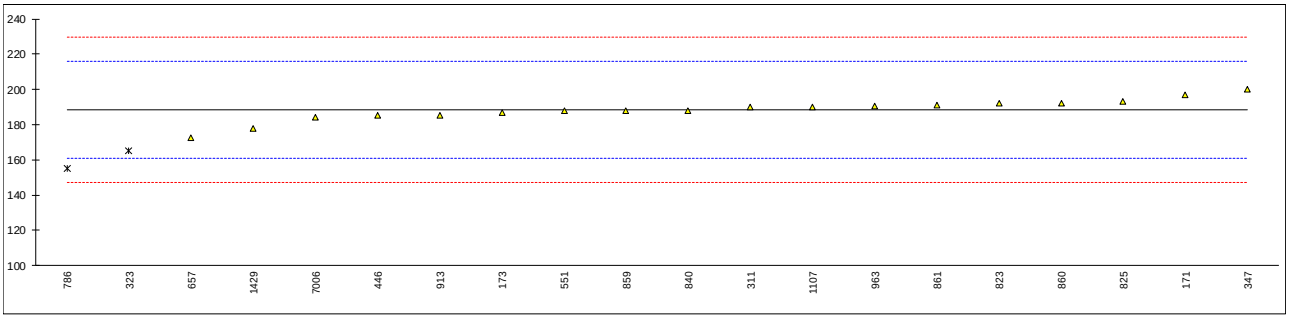
Determination of Acetone on sample #18001, results in mg/kg

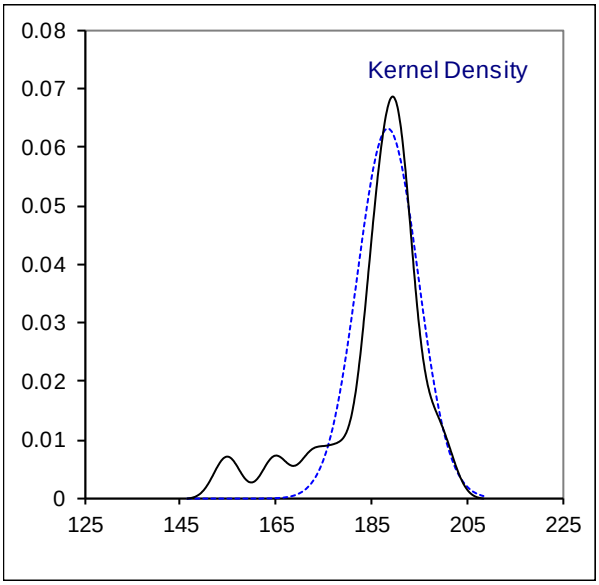
lab	method	value	mark	z(targ)	remarks
171	INH-0001	<5		-----	
173		-----		-----	
174		-----		-----	
273		-----		-----	
311	INH-122	<10		-----	
323	INH-067	<10		-----	
347		-----		-----	
391		-----		-----	
446		-----		-----	
522		-----		-----	
551	INH-1355	Less than 5		-----	
557		-----		-----	
657	INH-047	< 10		-----	
786	INH-88	<10		-----	
823	INH-021	<10		-----	
825	INH-021	<10		-----	
840	INH-001	<5		-----	
859	SH/T1628.2	<5		-----	
860	SH/T1628.2	<10		-----	
861	SH/T1628.2	<5		-----	
913		ND		-----	
963		-----		-----	
974	INH-102582	<10		-----	
1107		-----		-----	
1429		< 5		-----	
7006		-----		-----	

Determination of Ethyl Acetate on sample #18001, results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-0001	197		0.63	
173	INH-47	187		-0.10	
174		-----		-----	
273		-----		-----	
311	INH-122	190		0.12	
323	INH-067	165	R(0.05)	-1.71	
347	INH-096	200		0.85	
391		-----		-----	
446	INH-102582	185		-0.25	
522		-----		-----	
551	INH-1355	188		-0.03	
557		-----		-----	
657	INH-047	172.6		-1.15	
786	INH-88	155	R(0.05)	-2.44	
823	INH-021	192		0.26	
825	INH-021	193		0.34	
840	INH-001	188.1		-0.02	
859	SH/T1628.2	188		-0.03	
860	SH/T1628.2	192		0.26	
861	SH/T1628.2	191		0.19	
913		185		-0.25	
963	In house	190.6		0.16	
974	INH-102582	<10		<-13.02	False negative test result?
1107	In house	190		0.12	
1429		178		-0.76	
7006		184		-0.32	

normality	suspect
n	18
outliers	2
mean (n)	188.41
st.dev. (n)	6.301
R(calc.)	17.64
st.dev.(Horwitz)	13.702
R(Horwitz)	38.37

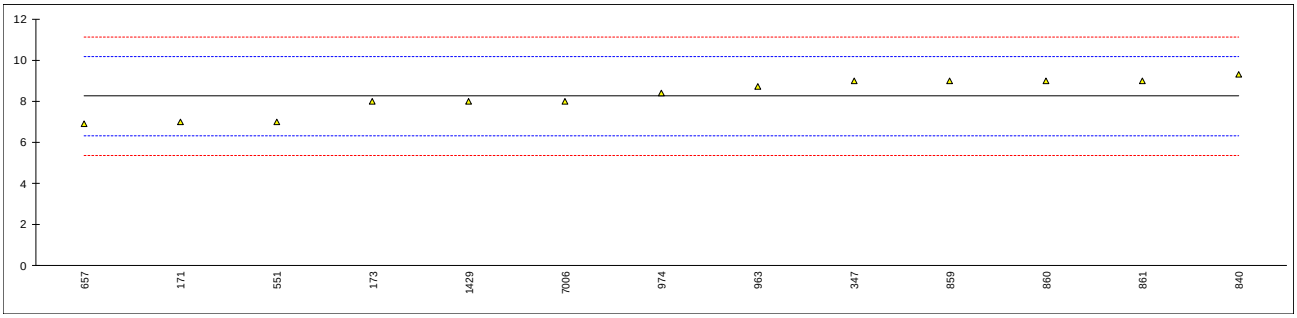


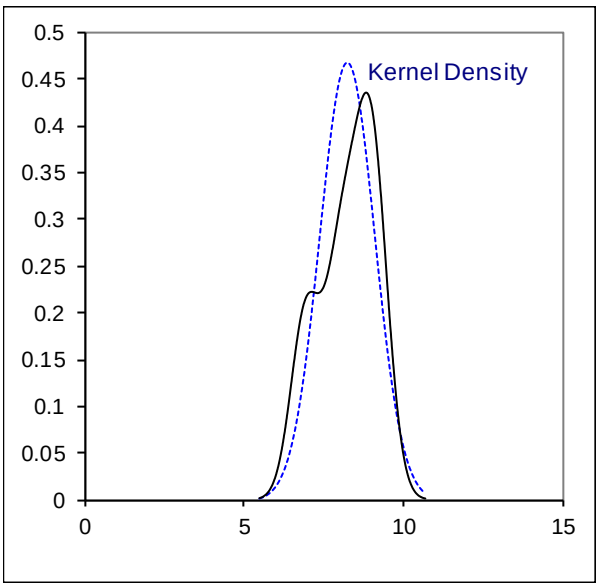


Determination of Methyl Acetate on sample #18001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-0001	7		-1.30	
173	INH-47	8		-0.26	
174		-----		-----	
273		-----		-----	
311	INH-122	<10		-----	
323	INH-067	<10		-----	
347	INH-096	9		0.78	
391		-----		-----	
446		-----		-----	
522		-----		-----	
551	INH-1355	7		-1.30	
557		-----		-----	
657	INH-047	6.9		-1.41	
786	INH-88	<10		-----	
823	INH-021	<10		-----	
825	INH-021	<10		-----	
840	INH-001	9.3		1.09	
859	SH/T1628.2	9		0.78	
860	SH/T1628.2	9		0.78	
861	SH/T1628.2	9		0.78	
913		< 5		<-3.39	False negative test result?
963	In house	8.7		0.46	
974	INH-102582	8.4	C	0.15	First reported 37
1107	In house	<10		-----	
1429		8		-0.26	
7006		8.0		-0.26	

normality	OK
n	13
outliers	0
mean (n)	8.25
st.dev. (n)	0.852
R(calc.)	2.39
st.dev.(Horwitz)	0.961
R(Horwitz)	2.69

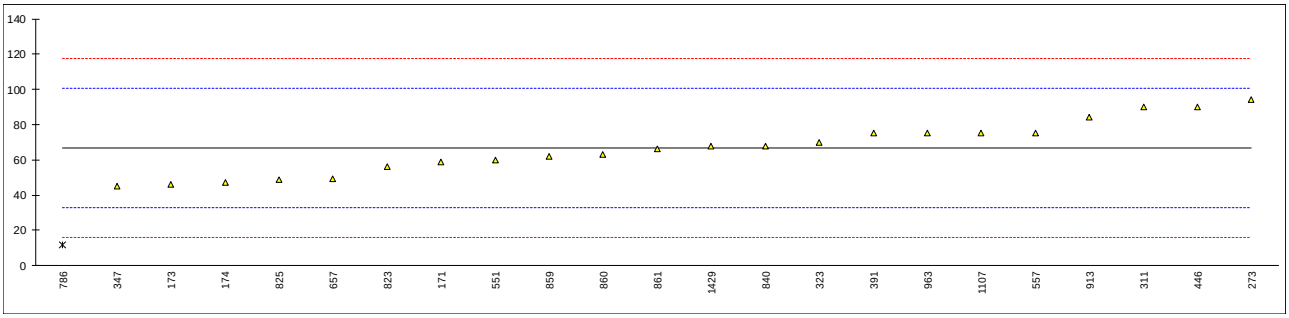


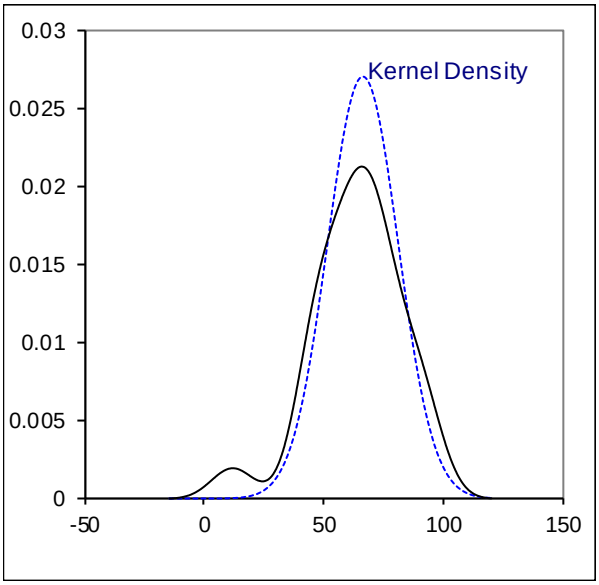


Determination of Water, titrimetric on sample #18001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		59		-0.44	
173	E203	46		-1.18	
174	E203	47		-1.12	
273	E203	94	C	1.56	First reported 140
311	D1364	90		1.34	
323	D1364	70		0.19	
347	D1364	45		-1.24	
391	D1364	75		0.48	
446	D1364	90		1.34	
522		-----		-----	
551	D1364	60		-0.38	
557	D1364	75.319		0.50	
657	E1064	49.05		-1.01	
786	D1364	12	G(0.05)	-3.12	
823	D1364	56		-0.61	
825	D1364	49		-1.01	
840	D1364	68		0.08	
859	D1364	62		-0.26	
860	D1364	63		-0.21	
861	D1364	66		-0.04	
913	D1364	84		0.99	
963	D1364	75		0.48	
974		-----		-----	
1107	D1364	75		0.48	
1429	D1364	67.6		0.06	
7006		-----		-----	

normality	OK
n	22
outliers	1
mean (n)	66.63
st.dev. (n)	14.787
R(calc.)	41.40
st.dev.(D1364:02)	17.492
R(D1364:02)	48.98





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 INDIA
1 lab in IRAN, Islamic Republic of
1 lab 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
2 labs in SOUTH KOREA
1 lab in SPAIN
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 E178:02
3 ASTM E1301:03
4 ISO 5725:05
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)