Results of Proficiency Test
Vinyl Acetate Monomer
February 2019

Organised by: Institute for Interlaboratory Studies
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 2018/2019, 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 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 2019 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 0.5L of Vinyl Acetate Monomer, labelled #19001.
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 June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

Approximately 25 liter bulk material of Vinyl Acetate Monomer was obtained from a participating laboratory. After homogenization 43 amber glass bottles of 0.5L were filled and labelled #19001. 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 8 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in mg/kg
sample #19001-1	0.93234	62
sample #19001-2	0.93232	53
sample #19001-3	0.93232	54
sample #19001-4	0.93238	82
sample #19001-5	0.93236	60
sample #19001-6	0.93234	67
sample #19001-7	0.93236	65
sample #19001-8	0.93236	61

Table 1: homogeneity test results of subsamples #19001

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)	0.00006	25
reference test method	ISO12185:96	ASTM D1364:02(2012)
0.3 * R (reference test method)	0.00015	--
r (reference test method)	--	24

Table 2: evaluation of the repeatabilities of subsamples #19001

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 #19001, was sent on January 23, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of VAM packed in amber glass bottles 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 #19001; Acidity (without and/or with Nitrogen purge), Apparent Specific Gravity 20/20°C, Color Pt/Co, Density at 20°C, Distillation (IBP, 50% recovered, 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 sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and 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 June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

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

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. In this PT the criterion of ISO13528, paragraph 9.2.1 was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying 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 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:

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

4 EVALUATION

In this proficiency test, some problems were encountered with the dispatch of the samples. Three participants reported the test results after the final reporting date. Not all participants were able to report test results for all the requested tests. In total 27 participants reported 247 numerical test results. Observed were 11 outlying test 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 TEST

In this section, the reported test results are discussed per test. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

In the iis PT reports, ASTM test methods are referred to with a number (e.g. D1209) and an added designation for the year that the test 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 test result tables of appendix 1 only the test method number and year of adoption or revision (e.g. D1209:05) will be used.

Unfortunately, a suitable reference test method providing the precision data is not available for all determinations. For these the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

The VAM used in this PT was obtained from a participating laboratory. Some participants noticed that sample #19001 differs from the VAM samples these participants usually receive for analyses. After evaluation it is observed that the average of the Purity in this PT (99.878 %M/M) differs from the averages of the Purity found in previous iis PTs (for example 99.975 %M/M and 99.971 %M/M in iis18C01 and iis17C01 respectively).

Some participants have shared their observations about sample #19001 in the remarks of the test results. For example, one participant mentioned for the determination of acidity that an “abnormally large amount of titrant went to the titration”. Three participants mentioned some issues for the determination of Inhibitor as Hydroquinone and/or the determination of the Distillation Dry Point (DP). The DP is higher than usual and therefore the Distillation Range difficult to determine. Also for the parameters Acidity and Inhibitor as Hydroquinone is the variation in the test results large (Acidity: between 337 mg/kg and 956 mg/kg, Inhibitor as Hydroquinone: between <1 and 13.7 mg/kg). Therefore, it was decided not to calculate z-scores for Acidity, Inhibitor as Hydroquinone and Distillation Range.

Acidity: The determination without Nitrogen purging was problematic. Two statistical outliers were observed. As the calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D2086:08(2012), no z-scores were calculated (see also discussion above). 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)). No test results were received for the determination with Nitrogen purging.

Apparent 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 ISO12185:96.

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 ISO12185:96.

Distillation: The determination was not problematic for IBP and 50% recovered, however the determination was problematic for Dry Point and Distillation Range. In total one statistical outlier was observed. The calculated reproducibilities for both IBP and 50% recovered are after rejection of the statistical outlier in agreement with the requirements of ASTM D1078:11 (automated and manual mode). The calculated reproducibilities for Dry Point and Distillation Range are not in agreement with the requirements of ASTM D1078:11 (automated and manual mode). It was decided to calculate no z-scores for Distillation Range (see also discussion above).

Inhibitor (Hydroquinone): This determination was problematic. Two statistical outliers were observed. As the calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D2193:06(2012), no z-scores were calculated (see also discussion above).

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 (0.1266) was larger than observed in previous PTs (0.0127 and 0.0287 in iis18C01 and iis17C01 respectively) (see also discussion above).

Acetaldehyde: This determination 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: This determination was not problematic. All participants agreed on a “less than” test result. Therefore no z-scores were calculated.

Ethyl Acetate: 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.

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

Water: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers 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 or as declared by the estimated target reproducibility using the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, ISO test methods) or the estimated target reproducibility are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity (without N_2 purging)	mg/kg	9	814	70	(6)
Acidity (with N_2 purging)	mg/kg	0	n.a.	n.a.	n.a.
Apparent Specific Gravity 20/20°C		21	0.9340	0.0003	0.0005
Color Pt/Co		21	5.3	3.9	7
Density at 20°C	kg/L	25	0.9323	0.0003	0.0005
Distillation, Initial Boiling Point	°C	16	72.4	0.4	1.1
Distillation, 50% recovery	°C	15	72.7	0.2	0.5
Distillation, Dry Point	°C	16	73.3	1.1	0.8
Distillation, Range	°C	15	0.9	1.3	(0.8)
Inhibitor as Hydroquinone	mg/kg	16	2.6	4.9	(1.0)
Purity	%M/M	19	99.878	0.13	n.a.
Acetaldehyde	mg/kg	12	657	186	111
Acetone	mg/kg	11	<10	n.a.	n.a.
Ethyl Acetate	mg/kg	16	146	44	31
Methyl Acetate	mg/kg	15	28.0	9.4	7.6
Water	mg/kg	20	51.4	34.5	43.0

Table 3: reproducibilities of tests on sample #19001

Results between brackets should be used with due care

Without further statistical calculations, it could be concluded that for some tests there is a good compliance of the group of participating laboratories with the relevant reference test methods. The problematic tests have been discussed in paragraph 4 and 4.1.

4.3 EVALUATION OF THE PROFICIENCY TEST OF FEBRUARY 2019 WITH PREVIOUS PTS

	February 2019	February 2018	February 2017	February 2016	February 2015
Number of reporting labs	27	25	23	25	20
Number of results reported	247	282	266	275	253
Number of statistical outliers	11	10	8	9	2
Percentage outliers	4.5%	3.5%	3.0%	3.3%	0.8%

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 2019	February 2018	February 2017	February 2016	February 2015
Acidity (without N_2 purging)	(--) [1)]	-	--	--	--
Acidity (with N_2 purging)	n.a.	--	--	--	--
Apparent Specific Gravity	+	++	++	++	++
Color Pt/Co	+	++	++	++	+
Density at 20°C	+	++	++	++	++
Distillation	+/-	++	++	++	++
Inhibitor as Hydroquinone	(--) [1)]	-	+/-	-	-
Purity	(--) [2)]	(++) [2)]	(--) [2)]	(+) [2)]	(+) [2)]
Acetaldehyde	-	--	-	+/-	-
Acetone	n.e.	n.e.	-	n.e.	-
Ethyl Acetate	-	++	+	+/-	-
Methyl Acetate	-	+	-	+	-
Water	+	+	++	++	+

Table 5: comparison determinations against the reference test methods

1) Results between brackets should be used with due care

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

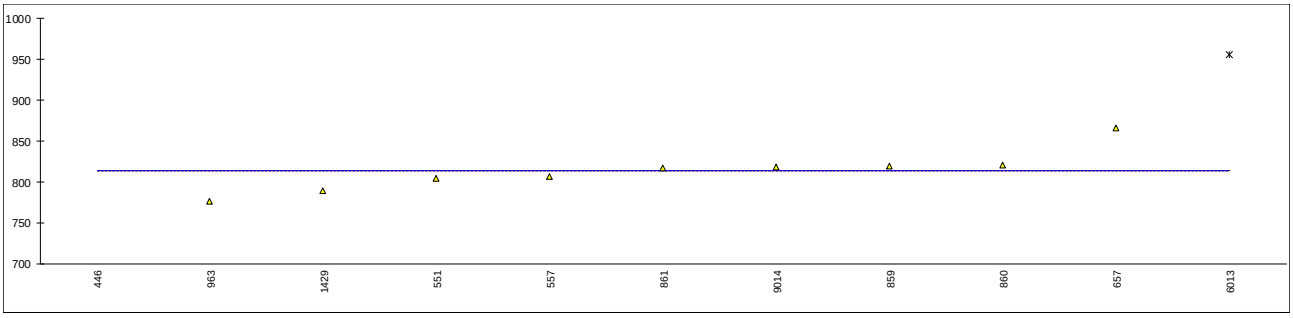
The following performance categories were used:

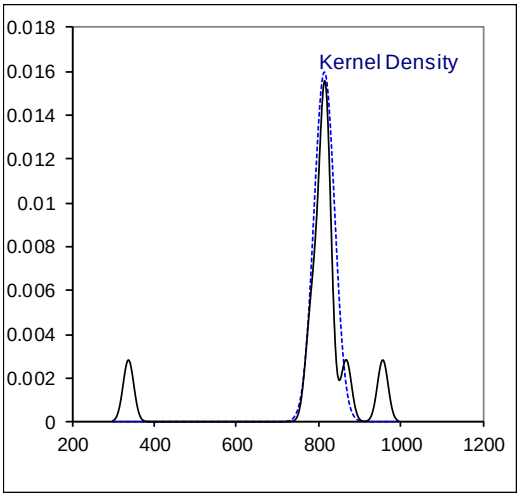
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Acidity (without N_2 purge) on sample #19001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173		-----		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
337		-----		-----	
347		-----		-----	
391		-----		-----	
446	D2086	337	G(0.01)	-----	
522		-----		-----	
528		-----		-----	
541		-----		-----	
551	D2086	805.1		-----	
557	D2086	807.8851782		-----	
657	D2086	866.96		-----	
840		-----		-----	
859	D2086	820		-----	
860	D2086	821		-----	
861	D2086	818		-----	
872		-----		-----	
913		-----		-----	
963	D2086	777		-----	
974		-----		-----	
1091		-----		-----	
1429	D2086	790		-----	
6013	D2086	956	G(0.01)	-----	
9014	D2086	818.47	C	-----	first reported 0.7639 mg/kg
	normality	not OK			
	n	9			
	outliers	2			
	mean (n)	813.82			
	st.dev. (n)	24.968			
	R(calc.)	69.91			
	st.dev.(D2086:08)	(2.143)			
	R(D2086:08)	(6)			





Determination of Acidity (with N_2 purge) on sample #19001; results in mg/kg

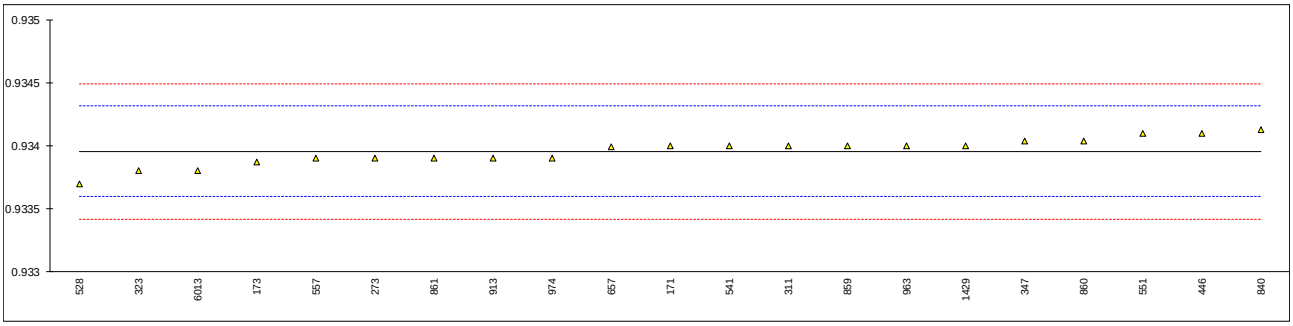
lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173		-----		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
337		-----		-----	
347		-----		-----	
391		-----		-----	
446		-----		-----	
522		-----		-----	
528		-----		-----	
541		-----		-----	
551		-----		-----	
557		-----		-----	
657		-----		-----	
840		-----		-----	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872		-----		-----	
913		-----		-----	
963		-----		-----	
974		-----		-----	
1091		-----		-----	
1429		-----		-----	
6013		-----		-----	
9014		-----		-----	

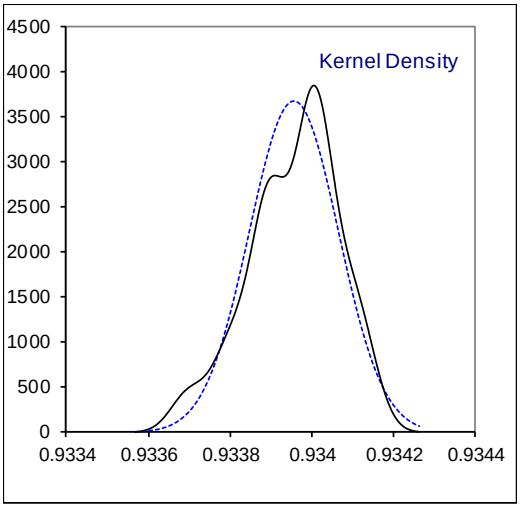
No test results were reported.

Determination of Apparent Specific Gravity 20/20°C on sample #19001;

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9340		0.25	
173	D4052	0.93387		-0.48	
273	D4052	0.9339		-0.31	
311	D4052	0.9340		0.25	
323	ISO12185	0.9338		-0.87	
337		-----		-----	
347	D4052	0.93404		0.47	
391		-----		-----	
446	D4052	0.9341		0.81	
522		-----		-----	
528	D4052	0.9337		-1.43	
541	D4052	0.93400		0.25	
551	D4052	0.9341		0.81	
557	D4052	0.9339		-0.31	
657	D4052	0.93399		0.19	
840	D4052	0.93413		0.98	
859	D4052	0.9340		0.25	
860	D4052	0.93404		0.47	
861	D4052	0.9339		-0.31	
872		-----		-----	
913	D4052	0.9339		-0.31	
963	ISO12185	0.9340		0.25	
974	D4052	0.9339		-0.31	
1091		-----		-----	
1429	D4052	0.9340		0.25	
6013	D4052	0.9338		-0.87	
9014		-----		-----	

normality	OK
n	21
outliers	0
mean (n)	0.93396
st.dev. (n)	0.000109
R(calc.)	0.00030
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

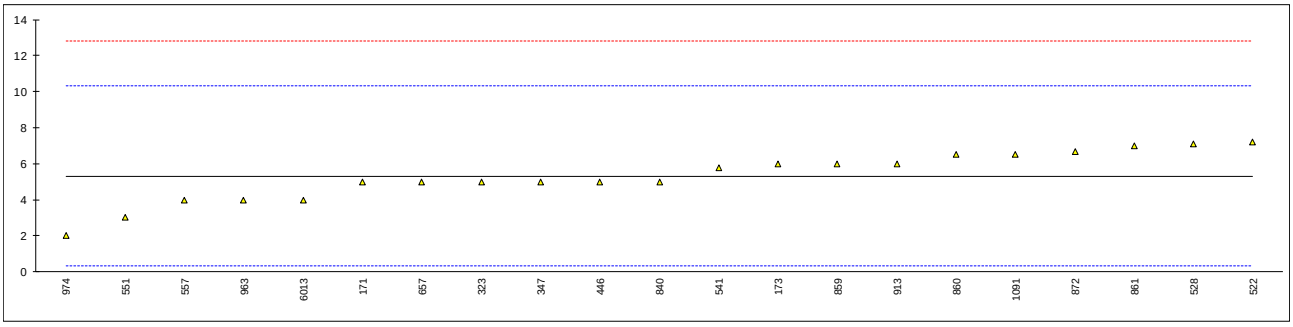


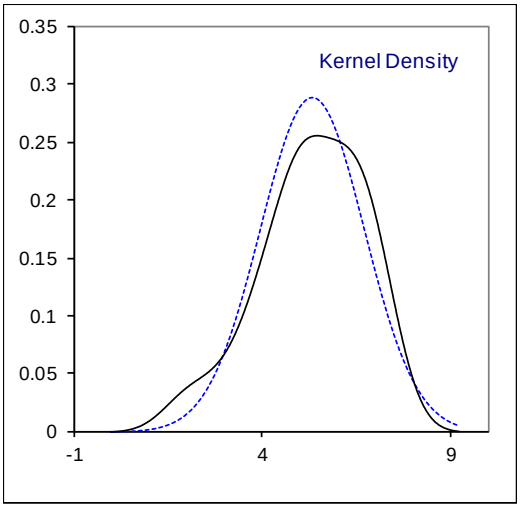


Determination of Color Pt/Co on sample #19001;

lab	method	value	mark	z(targ)	remarks
171	D1209	5		-0.13	
173	D5386	6.0		0.27	
273		-----		-----	
311	D1209	<5		-----	
323	D1209	5		-0.13	
337		-----		-----	
347	D5386	5		-0.13	
391		-----		-----	
446	D5386	5		-0.13	
522		7.2		0.75	
528	D1209	7.1		0.71	
541	D5386	5.8		0.19	
551	D1209	3		-0.93	
557	D1209	4		-0.53	
657	D1209	5		-0.13	
840	D1209	5		-0.13	
859	D1209	6		0.27	
860	D5386	6.5		0.47	
861	D1209	7		0.67	
872	D5386	6.7		0.55	
913	D5386	6		0.27	
963	D1209	4		-0.53	
974	D5386	2		-1.33	
1091	D5386	6.5		0.47	
1429	D1209	Less than 5		-----	
6013	D5386	4		-0.53	
9014		-----		-----	

normality	OK
n	21
outliers	0
mean (n)	5.32
st.dev. (n)	1.380
R(calc.)	3.86
st.dev.(D1209:05)	2.500
R(D1209:05)	7

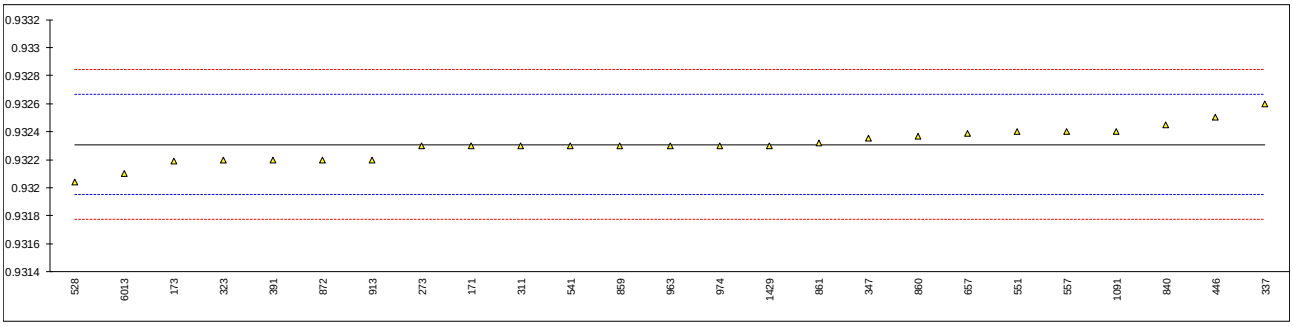


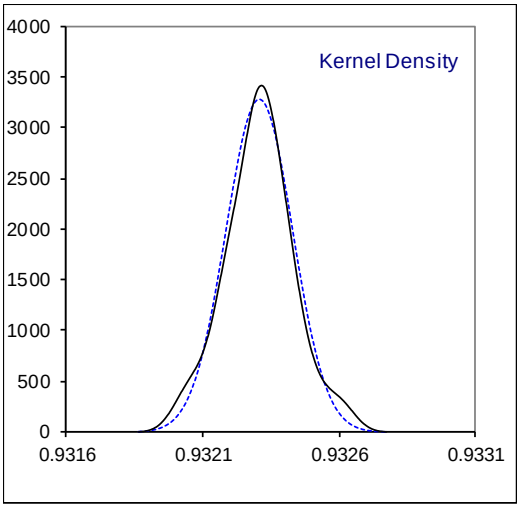


Determination of Density at 20°C on sample #19001; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9323		-0.05	
173	D4052	0.93219		-0.66	
273	D4052	0.9323		-0.05	
311	D4052	0.9323		-0.05	
323	ISO12185	0.9322		-0.61	
337	ISO12185	0.9326		1.63	
347	D4052	0.93235		0.23	
391	ISO12185	0.9322		-0.61	
446	D4052	0.9325		1.07	
522		-----		-----	
528	D4052	0.93204		-1.50	
541	D4052	0.93230		-0.05	
551	D4052	0.9324		0.51	
557	D4052	0.9324		0.51	
657	D4052	0.93239		0.46	
840	D4052	0.93245		0.79	
859	D4052	0.9323		-0.05	
860	D4052	0.93237		0.35	
861	D4052	0.93232		0.07	
872	D4052	0.9322		-0.61	
913	D4052	0.9322		-0.61	
963	ISO12185	0.9323		-0.05	
974	D4052	0.9323		-0.05	
1091	D4052	0.9324		0.51	
1429	D4052	0.9323		-0.05	
6013	ISO12185	0.9321		-1.17	
9014		-----		-----	

normality	OK
n	25
outliers	0
mean (n)	0.93231
st.dev. (n)	0.000122
R(calc.)	0.00034
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

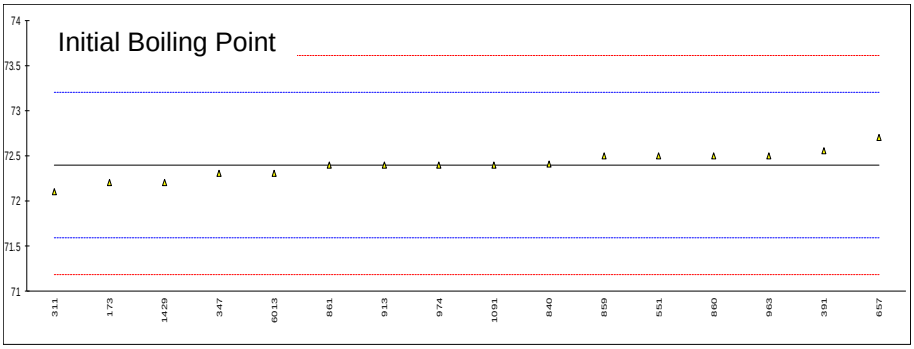


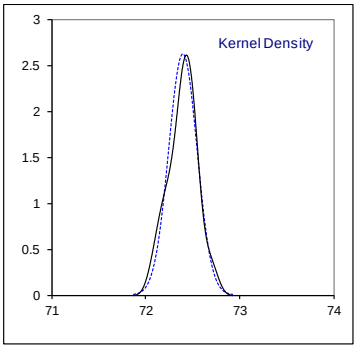


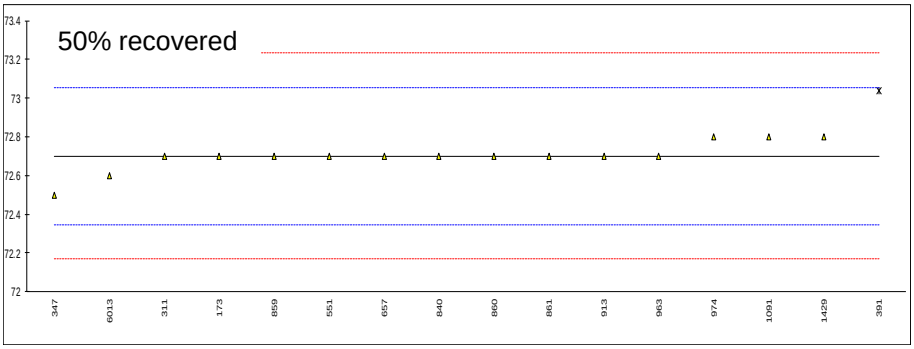
Determination of Distillation on sample #19001; results in °C

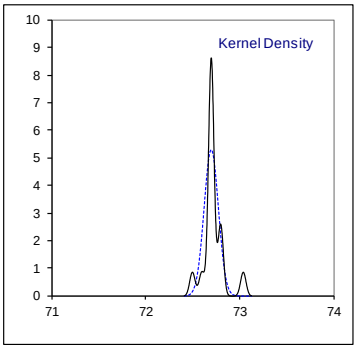
lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	DP	mark	z(targ)	range	mark	z(targ)
171		-----		-----	-----		-----	-----		-----	-----		-----
173	D1078-A	72.2		-0.49	72.7		0.00	73.2		-0.41	1.0		-----
273		-----		-----	-----		-----	-----		-----	-----		-----
311	D1078-A	72.1		-0.74	72.7		0.00	73.4		0.31	1.3		-----
323		-----		-----	-----		-----	-----		-----	-----		-----
337		-----		-----	-----		-----	-----		-----	-----		-----
347	D1078-A	72.3		-0.24	72.5		-1.13	73.3		-0.05	1.0		-----
391	D1078-A	72.56		0.40	73.04	G5	1.92	74.04		2.59	-----		-----
446		-----		-----	-----		-----	-----		-----	-----		-----
522		-----		-----	-----		-----	-----		-----	-----		-----
528		-----		-----	-----		-----	-----		-----	-----		-----
541		-----		-----	-----		-----	-----		-----	-----		-----
551	D1078	72.5		0.25	72.7		0.00	73.0		-1.12	0.5		-----
557		-----		-----	-----		-----	-----		-----	-----		-----
657	D1078-M	72.7		0.75	72.7		0.00	73.3		-0.05	0.6		-----
840	D1078-A	72.41		0.03	72.70		0.00	73.39		0.27	0.98		-----
859	D1078-M	72.5		0.25	72.7		0.00	72.9		-1.48	0.4		-----
860	D1078-M	72.5		0.25	72.7		0.00	72.9		-1.48	0.4		-----
861	D1078-M	72.4		0.00	72.7		0.00	72.9		-1.48	0.5		-----
872		-----		-----	-----		-----	-----		-----	-----		-----
913	D1078-M	72.4		0.00	72.7		0.00	73.0		-1.12	0.6		-----
963	D1078-M	72.5		0.25	72.7		0.00	73.3		-0.05	0.8		-----
974	D1078-A	72.4		0.00	72.8		0.56	73.0		-1.12	0.5		-----
1091		72.4		0.00	72.8		0.56	74.1		2.80	1.7		-----
1429		72.2		-0.49	72.8		0.56	74.0		2.45	1.8		-----
6013		72.3		-0.24	72.6		-0.56	73.3		-0.05	1.0		-----
9014		-----		-----	-----		-----	-----		-----	-----		-----
	normality	OK			not OK			suspect			OK		
	n	16			15			16			15		
	outliers	0			1			0			0		
	mean (n)	72.40			72.70			73.31			0.87		
	st.dev. (n)	0.152			0.076			0.404			0.448		
	R(calc.)	0.43			0.21			1.13			1.25		
	st.dev.(D1078-A:11)	0.403			0.177			0.280			(0.271)		
	R(D1078-A:11)	1.13			0.50			0.78			(0.76)		
	compare												
	R(D1078-M:11)	0.77			0.47			0.95			(0.68)		

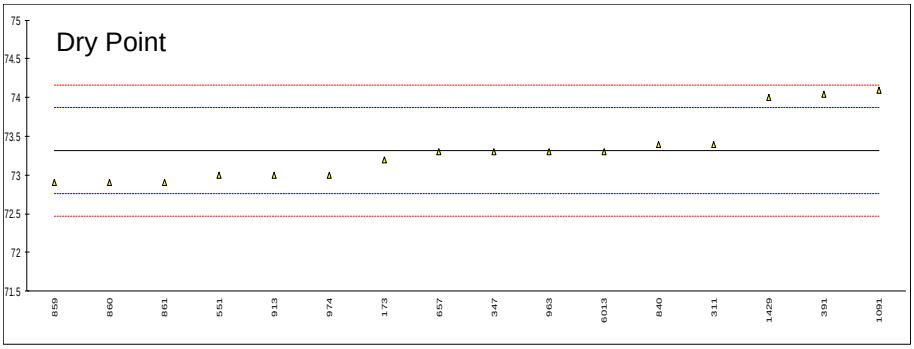
G5 = G(0.05) = straggler in Grubbs' outlier test

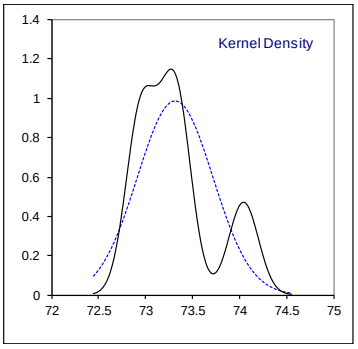

Initial Boiling Point
74
73.5
73
72.5
72
71.5
71
311 173 1429 347 6013 861 913 974 1091 840 859 551 860 963 391 657

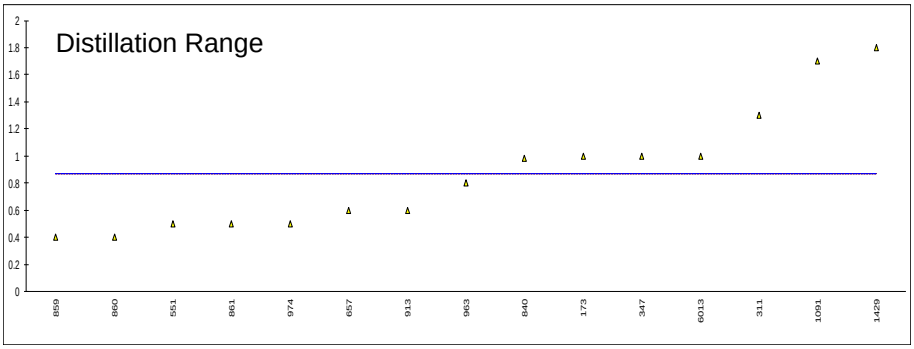

Kernel Density
3
2.5
2
1.5
1
0.5
0
71 72 73 74

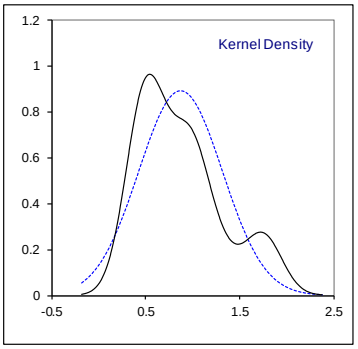

50% recovered
73.4
73.2
73
72.8
72.6
72.4
72.2
72
347 6013 311 173 859 551 657 840 860 861 913 963 974 1091 1429 391


Kernel Density
10
9
8
7
6
5
4
3
2
1
0
71 72 73 74


Dry Point
75
74.5
74
73.5
73
72.5
72
71.5
859 860 861 551 913 974 173 657 347 963 6013 840 311 1429 391 1091


Kernel Density
1.4
1.2
1
0.8
0.6
0.4
0.2
0
72 72.5 73 73.5 74 74.5 75

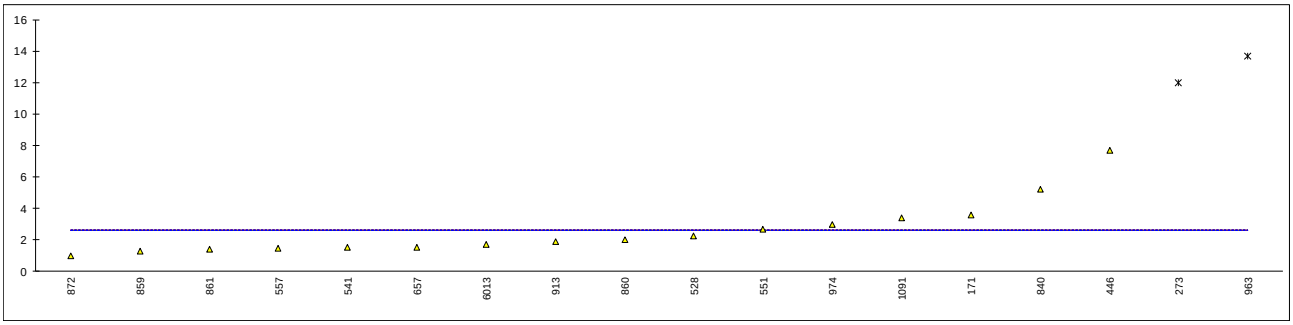

Distillation Range
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
859 860 551 861 974 657 913 963 840 173 347 6013 311 1091 1429

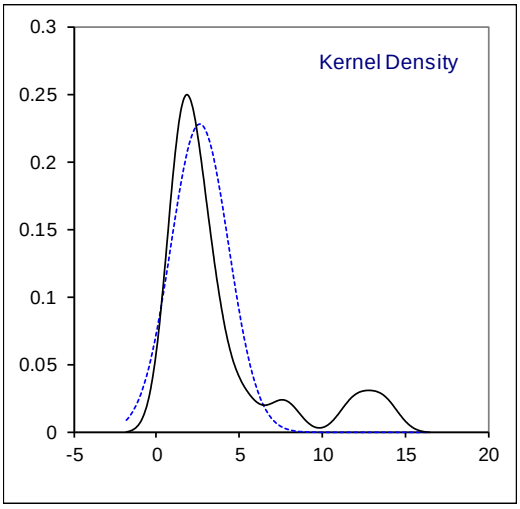

Kernel Density
1.2
1
0.8
0.6
0.4
0.2
0
-0.5 0.5 1.5 2.5

Determination of Inhibitor as Hydroquinone on sample #19001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2193	3.6		-----	
173		-----		-----	
273	D2193	12.0	G(0.01)	-----	
311		-----		-----	
323		-----		-----	
337		-----		-----	
347		-----		-----	
391		-----		-----	
446	D2193	7.7		-----	
522		-----		-----	
528	D2193	2.266		-----	
541	D2193	1.51		-----	
551	D2193	2.7		-----	
557	D2193	1.453346		-----	
657	D2193	1.5287		-----	
840	D2193	5.23		-----	
859	D2193	1.3		-----	
860	D2193	2.0		-----	
861	D2193	1.4		-----	
872	INH-9955	1.0		-----	
913	D2193	1.9		-----	
963	D2193	13.7	G(0.01)	-----	
974	INH-2401	3		-----	
1091	D2193	3.4	C	-----	first reported 13.4 with an in-house method
1429	D2193	< 1		-----	possibly false negative test result?
6013	D2193	1.7		-----	
9014		-----		-----	

normality	not OK
n	16
outliers	2
mean (n)	2.61
st.dev. (n)	1.748
R(calc.)	4.90
st.dev.(D2193:06)	(0.357)
R(D2193:06)	(1.0)

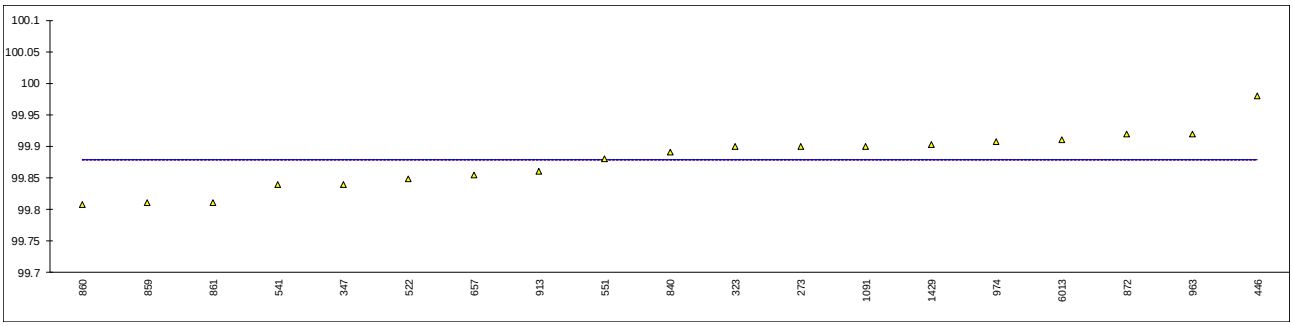


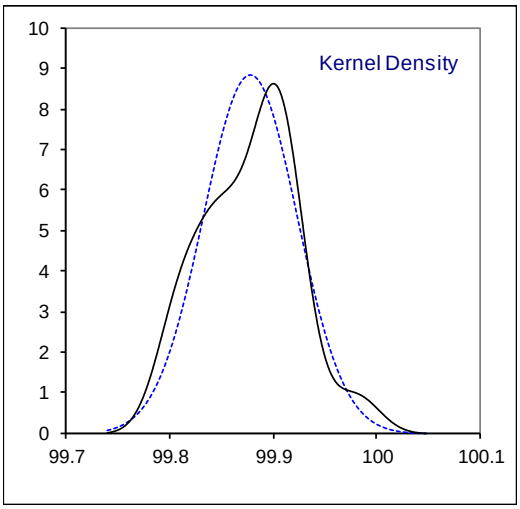


Determination of Purity on sample #19001; results in %M/M

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173		-----		-----	
273	INH-102582	99.90		-----	
311		-----		-----	
323		99.90		-----	
337		-----		-----	
347	INH-096	99.84		-----	
391		-----		-----	
446	INH-102582	99.98		-----	
522		99.849		-----	
528		-----		-----	
541	INH-102582	99.84	C	-----	first reported 99.830
551	INH-1355	99.88		-----	
557		-----		-----	
657	INH-0047	99.8551		-----	
840	D3545	99.890		-----	
859	SH/T1628.2	99.81		-----	
860	SH/T1628.2	99.808		-----	
861	SH/T1628.2	99.81		-----	
872	INH-9955	99.92		-----	
913		99.86		-----	
963	INH-8124	99.92		-----	
974	INH-2401	99.908		-----	
1091	In house	99.90		-----	
1429	In house	99.903		-----	
6013		99.91		-----	
9014		-----		-----	

normality	OK
n	19
outliers	0
mean (n)	99.8781
st.dev. (n)	0.04522
R(calc.)	0.1266
st.dev.(lit)	n.a.
R(lit)	n.a.
compare	
R(iis18C01)	0.0127
R(iis17C01)	0.0287

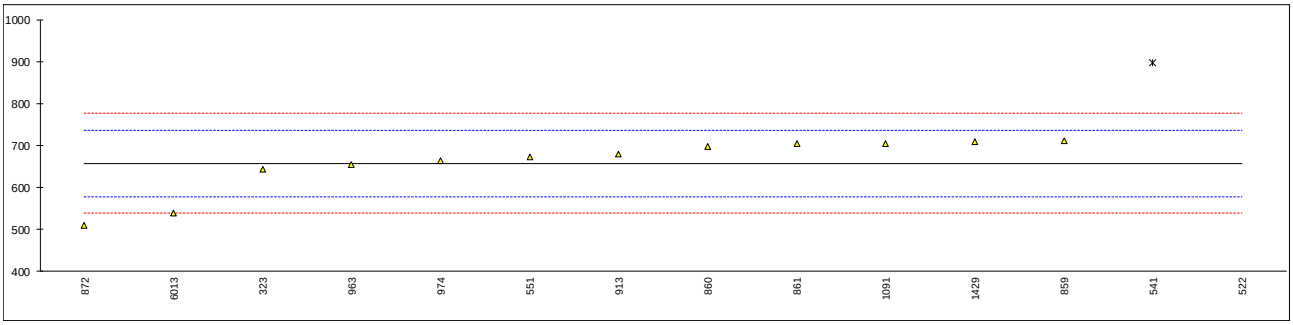


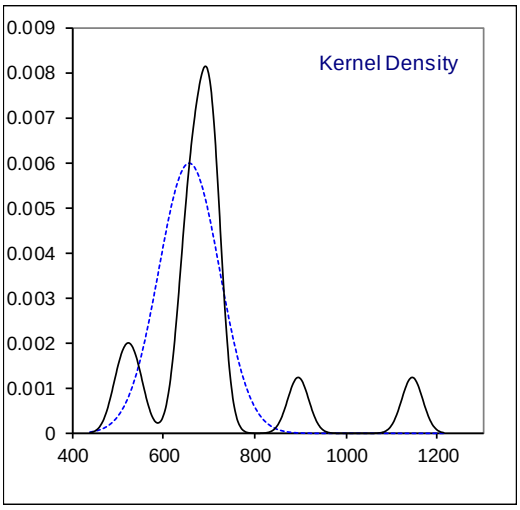


Determination of Acetaldehyde on sample #19001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173		-----		-----	
273		-----		-----	
311		-----		-----	
323		643		-0.36	
337		-----		-----	
347		-----		-----	
391		-----		-----	
446	INH-102582	>220		-----	
522		1145.7	G(0.01)	12.33	
528		-----		-----	
541	INH-102582	896.0	G(0.01)	6.03	
551	INH-1355	673		0.40	
557		-----		-----	
657		-----		-----	
840		-----		-----	
859	SH/T1628.2	710		1.33	
860	SH/T1628.2	698		1.03	
861	SH/T1628.2	705		1.20	
872	INH-9955	509		-3.75	
913		680		0.57	
963	INH-8124	654		-0.08	
974	INH-2401	663		0.14	
1091	In house	705		1.20	
1429	In house	709		1.30	
6013		539		-2.99	
9014		-----		-----	

normality	suspect
n	12
outliers	2
mean (n)	657.33
st.dev. (n)	66.507
R(calc.)	186.22
st.dev.(Horwitz)	39.608
R(Horwitz)	110.90





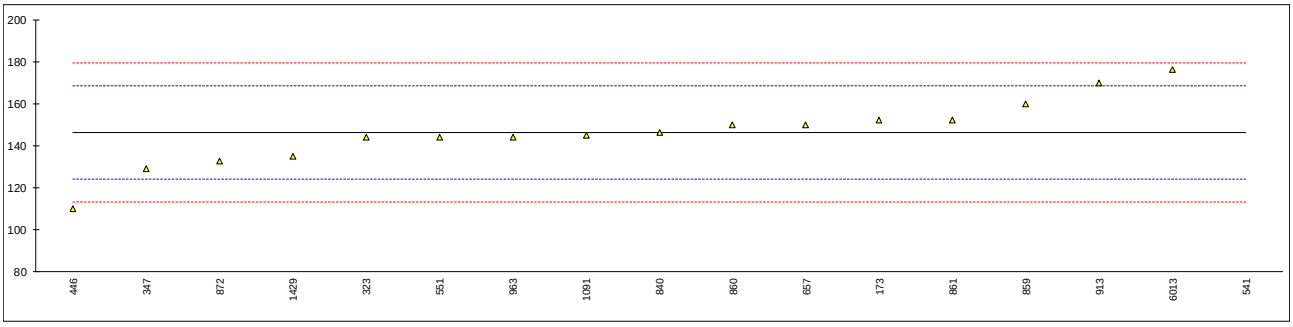
Determination of Acetone on sample #19001, results in mg/kg

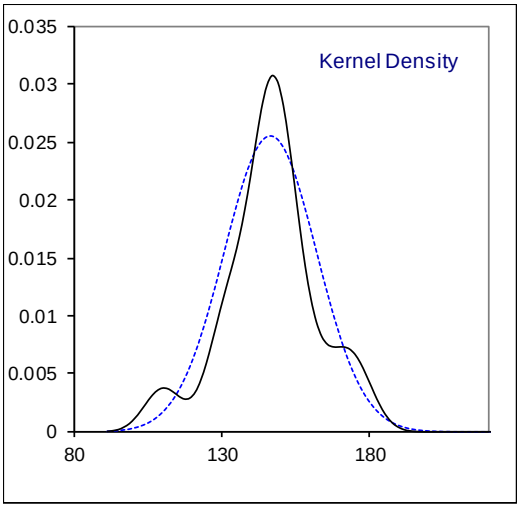
lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173		-----		-----	
273		-----		-----	
311		-----		-----	
323		<10		-----	
337		-----		-----	
347	INH-096	<10		-----	
391		-----		-----	
446		-----		-----	
522		-----		-----	
528		-----		-----	
541		-----		-----	
551	INH-1355	<5		-----	
557		-----		-----	
657	INH-0047	< 10		-----	
840		-----		-----	
859	SH/T1628.2	<10		-----	
860	SH/T1628.2	<10		-----	
861	SH/T1628.2	<10		-----	
872	INH-9955	<10		-----	
913		<5		-----	
963		-----		-----	
974		-----		-----	
1091		-----		-----	
1429	In house	Less than 5		-----	
6013		<1		-----	
9014		-----		-----	
	n	11			
	mean (n)	<10			

Determination of Ethyl Acetate on sample #19001, results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173	INH-47	152		0.52	
273		-----		-----	
311		-----		-----	
323		144		-0.20	
337		-----		-----	
347	INH-096	129		-1.56	
391		-----		-----	
446	INH-102582	110		-3.28	
522		-----		-----	
528		-----		-----	
541	INH-102582	546.5	G(0.01)	36.22	
551	INH-1355	144		-0.20	
557		-----		-----	
657	INH-0047	150.09		0.35	
840	D3545	146.5		0.02	
859	SH/T1628.2	160		1.24	
860	SH/T1628.2	150		0.34	
861	SH/T1628.2	152		0.52	
872	INH-9955	132.6		-1.24	
913		170		2.15	
963	INH-8124	144		-0.20	
974		-----		-----	
1091	In house	145		-0.11	
1429	In house	135		-1.02	
6013		176		2.69	
9014		-----		-----	

normality	suspect
n	16
outliers	1
mean (n)	146.26
st.dev. (n)	15.651
R(calc.)	43.82
st.dev.(Horwitz)	11.050
R(Horwitz)	30.94

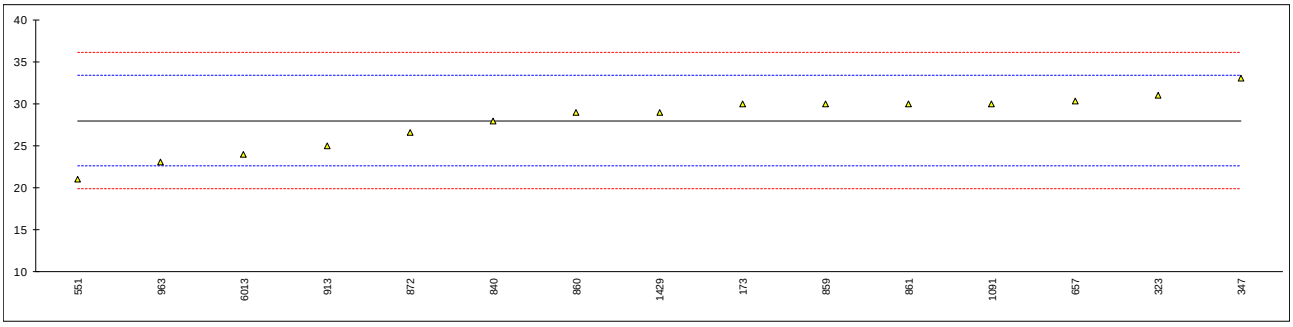


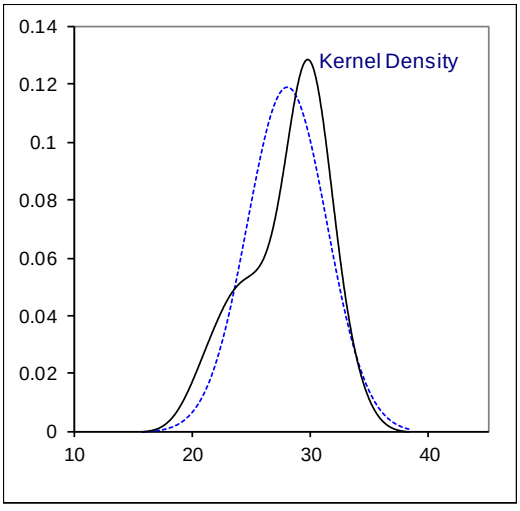


Determination of Methyl Acetate on sample #19001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173	INH-47	30		0.74	
273		-----		-----	
311		-----		-----	
323		31		1.11	
337		-----		-----	
347	INH-096	33		1.85	
391		-----		-----	
446		-----		-----	
522		-----		-----	
528		-----		-----	
541		-----		-----	
551	INH-1355	21		-2.58	
557		-----		-----	
657	INH-0047	30.29		0.85	
840	D3545	27.9		-0.03	
859	SH/T1628.2	30		0.74	
860	SH/T1628.2	29		0.37	
861	SH/T1628.2	30		0.74	
872	INH-9955	26.6		-0.51	
913		25		-1.10	
963	INH-8124	23		-1.84	
974		-----		-----	
1091	In house	30		0.74	
1429	In house	29		0.37	
6013		24		-1.47	
9014		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	27.99
st.dev. (n)	3.362
R(calc.)	9.41
st.dev.(Horwitz)	2.712
R(Horwitz)	7.59



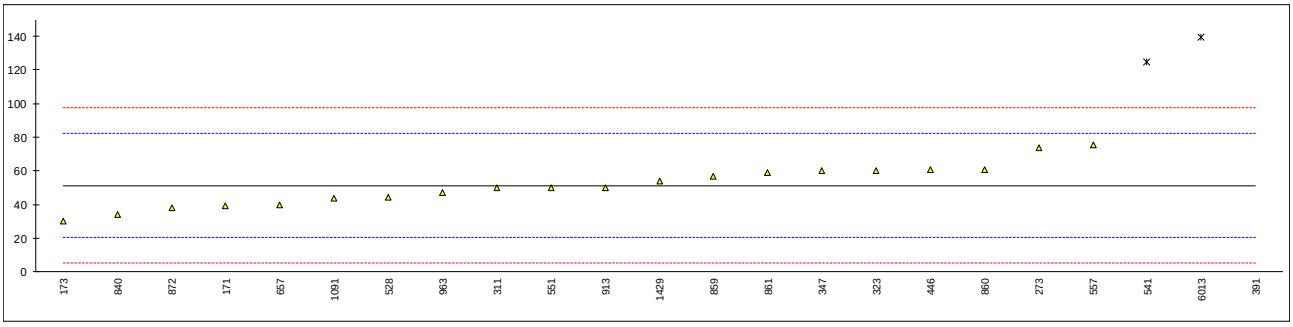


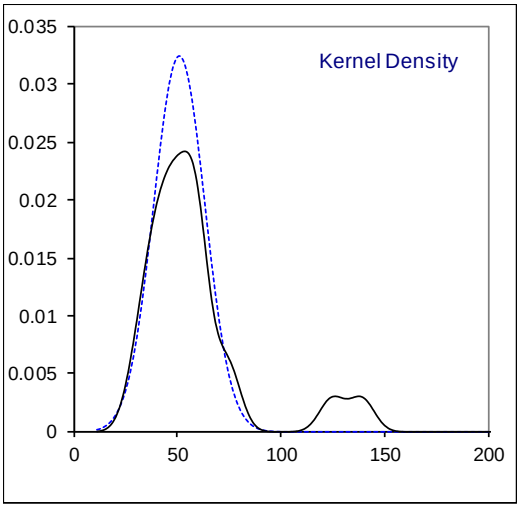
Determination of Water, titrimetric on sample #19001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	39		-0.81	
173	E203	30		-1.39	
273	E203	74		1.47	
311	E203	50		-0.09	
323	D1364	60		0.56	
337		-----		-----	
347	D1364	60		0.56	
391	D1364	300	G(0.01)	16.18	
446	D1364	61		0.63	
522		-----		-----	
528	D1364	44.55		-0.45	
541	D1364	125	C,G(0.01)	4.79	first reported 0.025 mg/kg
551	D1364	50		-0.09	
557	D1364	75.58550		1.57	
657	E1064	39.7		-0.76	
840	D1364	34.0		-1.13	
859	D1364	57		0.37	
860	D1364	61		0.63	
861	D1364	59		0.50	
872	D1364	38		-0.87	
913	E1064	50		-0.09	
963	D1364	47		-0.29	
974		-----		-----	
1091	D1364	44		-0.48	
1429	D1364	54		0.17	
6013	D1364	139.3	G(0.01)	5.72	
9014		-----		-----	

	normality	OK
	n	20
	outliers	3
	mean (n)	51.39
	st.dev. (n)	12.315
	R(calc.)	34.48
	st.dev.(D1364:02)	15.362
	R(D1364:02)	43.01

range 100 - 1000 mg/kg





APPENDIX 2

Number of participants per country

1 lab in ARGENTINA
2 labs in BELGIUM
2 labs in BRAZIL
3 labs in CHINA, People's Republic
1 lab in FRANCE
1 lab in INDIA
1 lab in ITALY
2 labs 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 SPAIN
2 labs in TURKEY
1 lab in UNITED ARAB EMIRATES
2 labs in UNITED KINGDOM
2 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	= possibly an error in calculations
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, June 2018
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)