Results of Proficiency Test
Vinyl Acetate Monomer (VAM)
February 2020

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 2019/2020 it was decided to continue the round robin for the analysis of VAM based on the scope of the latest specification of ASTM D2190.
In this interlaboratory study 24 laboratories in 17 different countries registered for participation. See appendix 2 for the number of participants per country.
In this report the results of the 2020 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 analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample of 0.5 L Vinyl Acetate Monomer labelled #20001.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on 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

A batch of approximately 25 liters bulk material of Vinyl Acetate Monomer was obtained from a participating laboratory. After homogenization 42 amber glass bottles of 0.5 liter were filled and labelled #20001. The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ASTM D4052 and Water in accordance with ASTM E203 on 8 stratified randomly selected samples.

	Density at 20°C in kg/L	Water in mg/kg
sample #20001-1	0.93218	120
sample #20001-2	0.93220	120
sample #20001-3	0.93219	120
sample #20001-4	0.93219	130
sample #20001-5	0.93219	120
sample #20001-6	0.93220	140
sample #20001-7	0.93222	140
sample #20001-8	0.93220	120

Table 1: homogeneity test results of subsamples #20001

From the above test results the repeatabilities were calculated and compared with 0.3 times the reproducibility or the repeatability of the corresponding reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 20°C in kg/L	Water in mg/kg
r (observed)	0.00003	26
reference test method	ISO12185:96	ASTM D1364:02(2012)
0.3 * R (ref. test method)	0.00015	---
r (reference test method)	---	34

Table 2: evaluation of the repeatabilities of subsamples #20001

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

To each of the participating laboratories one bottle of 0.5 liter VAM labelled #20001 was sent on January 15, 2020. 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 ANALYZES

The participants were requested to determine on sample #20001: 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 and Impurities (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 that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

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

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

3.1 STATISTICS

The protocol followed in the 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 ISO5725 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 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 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 target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.
When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

z(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 no problems were encountered with the dispatch of the samples. Four participants did not report any test results at all and one participant reported test results after the final reporting date. Not all participants were able to report test results for all the requested tests. Twenty participants reported 214 numerical test results. Observed were 10 statistically outlying test results, which is 4.7%. 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 reported test results. The abbreviations, used in these tables, are explained 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(2019). 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.

Sample #20001

Acidity: The determination without Nitrogen purge was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all 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).
Only one test result was received for the determination with Nitrogen purge. Therefore, no z-scores could be calculated.

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. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D1209:05(2019).

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 all distillation parameters; IBP, 50% recovered, Dry Point and Distillation Range. In total five statistical outliers were observed and two other test results were excluded. The calculated reproducibilities after rejection of the suspect data are in agreement with the requirements of ASTM D1078:11(2019) (automated and manual mode).

Inhibitor as Hydroquinone: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D2193:06(2012).

Purity by GC: 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 (0.0247) was much less than in previous PT (0.1266 in iis19C01) and more in line with the 2018 PT (0.0127 in iis18C01).

Acetaldehyde: This determination may be problematic. Three 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. Most participants agreed on a test result less than 10 mg/kg. Therefore, no z-scores were calculated.

Ethyl 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.

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. No statistical outliers were observed. The calculated reproducibility 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 purge	mg/kg	9	27.1	13.2	6
Acidity with N_2 purge	mg/kg	1	n.e.	n.e.	n.e.
Apparent Specific Gravity 20/20°C		17	0.9339	0.0002	0.0005
Color Pt/Co		16	3.0	3.2	7
Density at 20°C	kg/L	18	0.9322	0.0002	0.0005
Distillation, Initial Boiling Point	°C	14	72.4	0.4	1.1
Distillation, 50% recovery	°C	13	72.7	0.1	0.5
Distillation, Dry Point	°C	12	72.9	0.3	0.8
Distillation, Range	°C	14	0.5	0.6	0.7
Inhibitor as Hydroquinone	mg/kg	17	2.6	2.0	1.0
Purity by GC	%M/M	17	99.972	0.025	n.a.
Acetaldehyde	mg/kg	9	31.0	11.8	8.3
Acetone	mg/kg	7	<10	n.e.	n.e.
Ethyl Acetate	mg/kg	12	134	55	29
Methyl Acetate	mg/kg	12	22.9	10.5	6.4
Water	mg/kg	18	105	49	62

Table 3: performance evaluation sample #20001

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 4.1.

4.3 EVALUATION OF THE PROFICIENCY TEST OF FEBRUARY 2020 WITH PREVIOUS PTS

	February 2020	February 2019	February 2018	February 2017	February 2016
Number of reporting laboratories	20	27	25	23	25
Number of test results	214	247	282	266	275
Number of statistical outliers	10	11	10	8	9
Percentage statistical outliers	4.7%	4.5%	3.5%	3.0%	3.3%

Table 4: comparison with 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 in the following table.

Parameter	February 2020	February 2019	February 2018	February 2017	February 2016
Acidity without N_2 purge	--	(--) [1]	-	--	--
Acidity with N_2 purge	n.e.	n.e.	--	--	--
Apparent Specific Gravity 20/20°C	++	+	++	++	++
Color Pt/Co	++	+	++	++	++
Density at 20°C	++	+	++	++	++
Distillation	++	+/-	++	++	++
Inhibitor as Hydroquinone	--	(--) [1]	-	+/-	-
Purity by GC	(++) [2]	(--) [2]	(++) [2]	(--) [2]	(+) [2]
Acetaldehyde	-	-	--	-	+/-
Acetone	n.e.	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

In the table above the following performance categories were used:

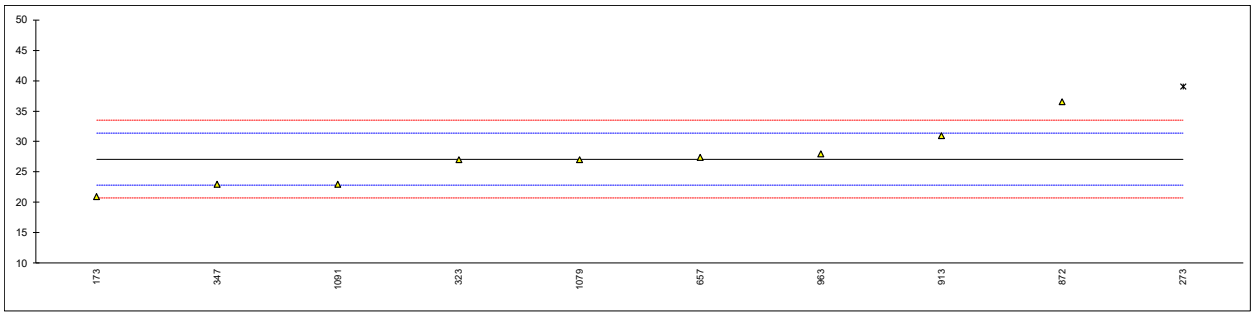
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

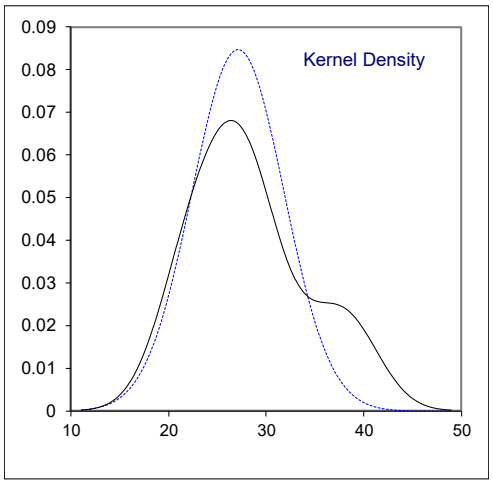
APPENDIX 1

Determination of Acidity without N_2 purge on sample #20001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173	INH-44	21		-2.85	
273	INH-40	39	C,G(0.05)	5.55	first reported 385
311		-----		-----	
323	D2086	27		-0.05	
347	D2086	23	C	-1.92	first reported 8
391		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551		-----		-----	
657	D2086	27.4		0.14	
840		-----		-----	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872	D2086	36.58		4.42	
913	D2086	31		1.82	
963	D2086	28		0.42	
974		-----		-----	
1079	D2086	27.0		-0.05	
1091	D2086	23.0		-1.92	
1429		-----		-----	
6262		-----		-----	

normality	suspect
n	9
outliers	1
mean (n)	27.11
st.dev. (n)	4.701
R(calc.)	13.16
st.dev.(D2086:08)	2.143
R(D2086:08)	6





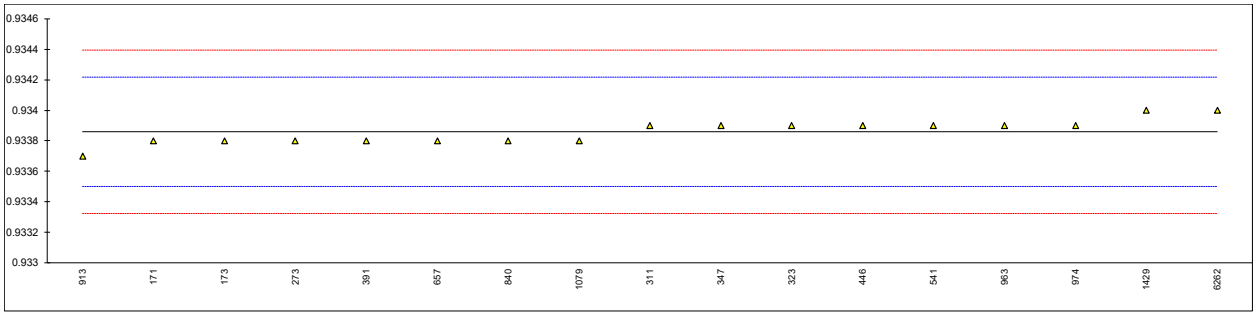
Determination of Acidity with N_2 purge on sample #20001; results in mg/kg

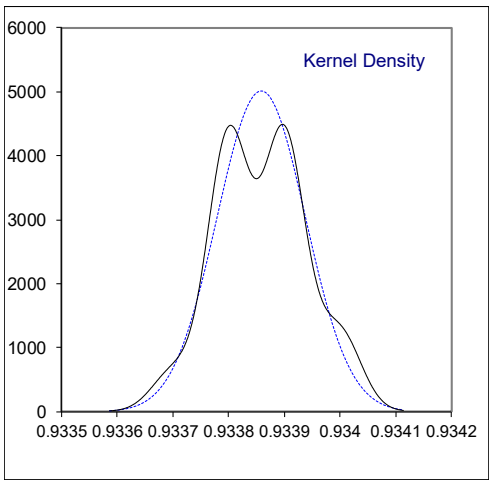
lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173		-----		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
347		-----		-----	
391		-----		-----	
446	D2086	24		-----	
522		-----		-----	
541		-----		-----	
551		-----		-----	
657		-----		-----	
840		-----		-----	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872		-----		-----	
913		-----		-----	
963		-----		-----	
974		-----		-----	
1079		-----		-----	
1091		-----		-----	
1429		-----		-----	
6262		-----		-----	

Determination of Apparent Specific Gravity 20/20°C on sample #20001;

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9338		-0.33	
173	D4052	0.9338		-0.33	
273	D4052	0.9338		-0.33	
311	D4052	0.9339		0.23	
323	ISO12185	0.9339		0.23	
347	D4052	0.9339		0.23	
391	ISO12185	0.9338		-0.33	
446	D4052	0.9339		0.23	
522		-----		-----	
541	D4052	0.9339		0.23	
551		-----		-----	
657	D4052	0.9338		-0.33	
840	D4052	0.93380		-0.33	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872		-----		-----	
913	D4052	0.9337		-0.89	
963	ISO12185	0.9339		0.23	
974	D4052	0.9339		0.23	
1079	ISO12185	0.9338		-0.33	
1091		-----		-----	
1429	ISO12185	0.9340		0.79	
6262	D4052	0.9340		0.79	

normality	OK
n	17
outliers	0
mean (n)	0.93386
st.dev. (n)	0.000080
R(calc.)	0.00022
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

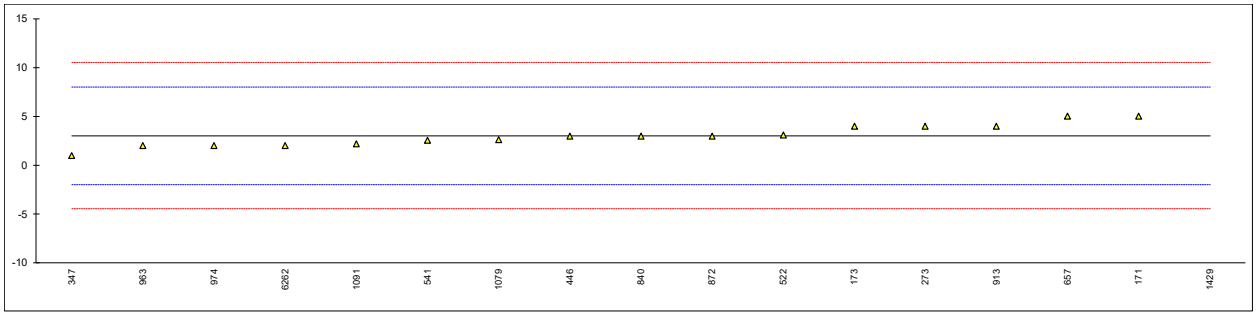


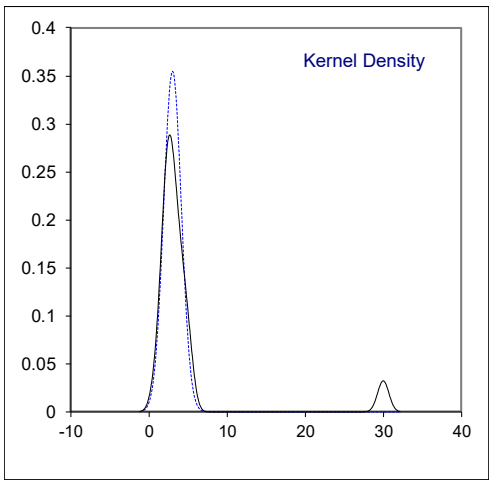


Determination of Color Pt/Co on sample #20001;

lab	method	value	mark	z(targ)	remarks
171	D1209	5		0.79	
173	D5386	4		0.39	
273	D5386	4		0.39	
311	D1209	<5		-----	
323	D1209	<5		-----	
347	D5386	1		-0.81	
391	D1209	<5		-----	
446	D5386	3		-0.01	
522		3.1		0.03	
541	D5386	2.53		-0.20	
551		-----		-----	
657	D1209	5		0.79	
840	D1209	3		-0.01	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872	D1209	3		-0.01	
913	D5386	4		0.39	
963	D1209	2		-0.41	
974	D5386	2		-0.41	
1079	D5386	2.6		-0.17	
1091	D5386	2.2		-0.33	
1429	D1209	30	G(0.01)	10.79	
6262	D1209	2.0		-0.41	

normality	OK
n	16
outliers	1
mean (n)	3.03
st.dev. (n)	1.126
R(calc.)	3.15
st.dev.(D1209:05)	2.500
R(D1209:05)	7

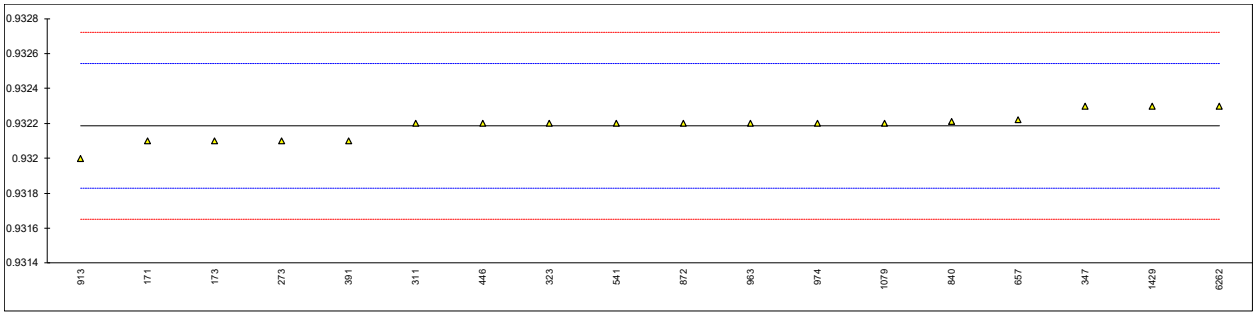


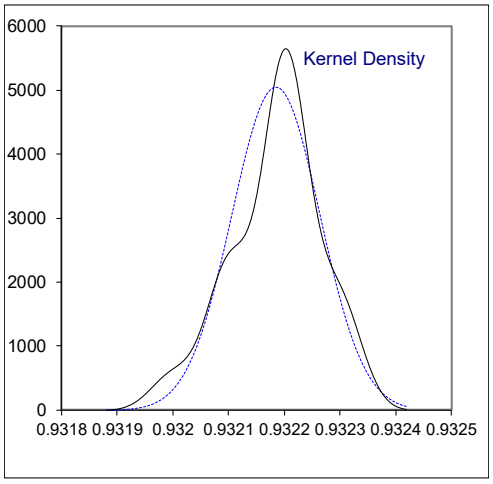


Determination of Density at 20°C on sample #20001; results in kg/L

lab	method	value	Mark	z(targ)	remarks
171	D4052	0.9321		-0.48	
173	D4052	0.9321		-0.48	
273	D4052	0.9321		-0.48	
311	D4052	0.9322		0.08	
323	ISO12185	0.9322		0.08	
347	D4052	0.9323		0.64	
391	ISO12185	0.9321		-0.48	
446	D4052	0.9322		0.08	
522		-----		-----	
541	D4052	0.9322		0.08	
551		-----		-----	
657	D4052	0.93222		0.20	
840	D4052	0.93221		0.14	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872	D4052	0.9322		0.08	
913	D4052	0.9320		-1.04	
963	ISO12185	0.9322		0.08	
974	D4052	0.9322		0.08	
1079	ISO12185	0.9322		0.08	
1091		-----		-----	
1429	ISO12185	0.9323		0.64	
6262	D4052	0.9323		0.64	

normality	OK
N	18
Outliers	0
mean (n)	0.93219
st.dev. (n)	0.000079
R(calc.)	0.00022
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005



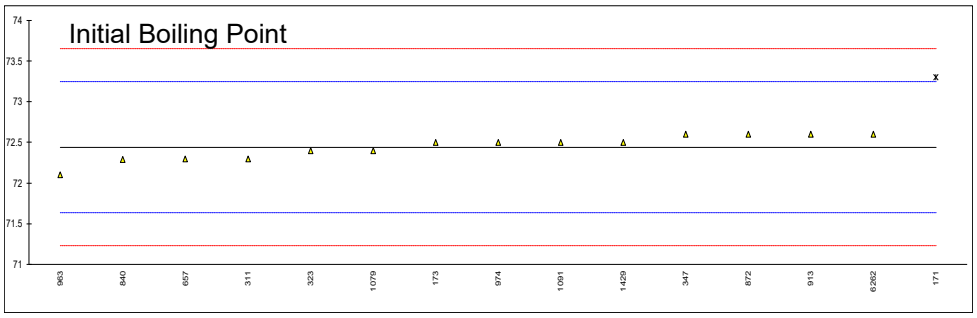


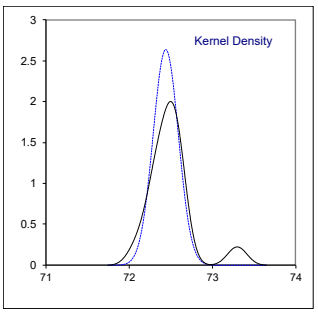
Determination of Distillation on sample #20001; results in °C

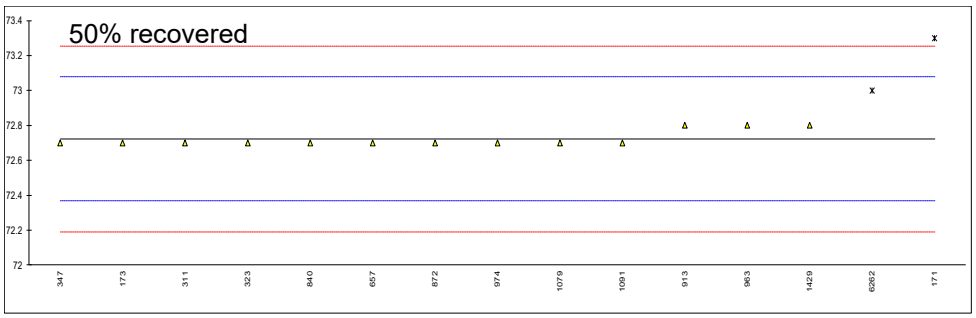
lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	DP	mark	z(targ)	range	mark	z(targ)
171	D1078-A	73.3	G(1)	2.13	73.3	G(1)	3.25	73.4	ex	1.87	0.1	ex	-1.59
173	D1078-A	72.5		0.14	72.7		-0.13	72.9		0.07	0.4		-0.41
273		-----		-----	-----		-----	-----		-----	-----		-----
311	D1078-A	72.3		-0.35	72.7		-0.13	72.8		-0.29	0.5		-0.02
323	D1078-A	72.4		-0.10	72.7		-0.13	73.3	DG(5)	1.51	0.9		1.55
347	D1078-A	72.6		0.39	72.7		-0.13	72.9		0.07	0.3		-0.81
391		-----		-----	-----		-----	-----		-----	-----		-----
446		-----		-----	-----		-----	-----		-----	-----		-----
522		-----		-----	-----		-----	-----		-----	-----		-----
541		-----		-----	-----		-----	-----		-----	-----		-----
551		-----		-----	-----		-----	-----		-----	-----		-----
657	D1078-M	72.3		-0.35	72.7		-0.13	73.1		0.79	0.8		1.16
840	D1078-A	72.29		-0.38	72.70		-0.13	72.76		-0.43	0.47		-0.14
859		-----		-----	-----		-----	-----		-----	-----		-----
860		-----		-----	-----		-----	-----		-----	-----		-----
861		-----		-----	-----		-----	-----		-----	-----		-----
872	D1078-M	72.6		0.39	72.7		-0.13	72.8		-0.29	0.20		-1.20
913	D1078-M	72.6		0.39	72.8		0.43	73.4	DG(5)	1.87	0.8		1.16
963		72.1		-0.85	72.8		0.43	72.9		0.07	0.8		1.16
974	D1078-A	72.5		0.14	72.7		-0.13	72.8		-0.29	0.3		-0.81
1079	D1078-A	72.4		-0.10	72.7		-0.13	72.8		-0.29	0.4		-0.41
1091		72.5		0.14	72.7		-0.13	72.8		-0.29	0.3		-0.81
1429	D1078	72.5		0.14	72.8		0.43	72.9		0.07	0.4		-0.41
6262	D1078-A	72.6		0.39	73.0	G(1)	1.56	73.1		0.79	0.5		-0.02
	normality	OK			suspect			suspect			OK		
	n	14			13			12			14		
	outliers	1			2			2 +1ex			0 +1ex		
	mean (n)	72.44			72.72			72.88			0.51		
	st.dev. (n)	0.151			0.044			0.115			0.227		
	R(calc.)	0.42			0.12			0.32			0.64		
	st.dev.(D1078-A:11)	0.404			0.177			0.279			0.254		
	R(D1078-A:11)	1.13			0.50			0.78			0.71		
Compare													
	R(D1078-M:11)	0.78			0.47			0.95			0.64		

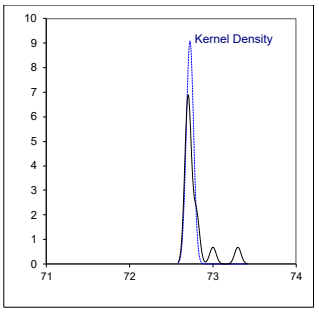
Lab 171 excluded for Dry Point and Distillation Range as statistical outliers in corresponding distillation parameters

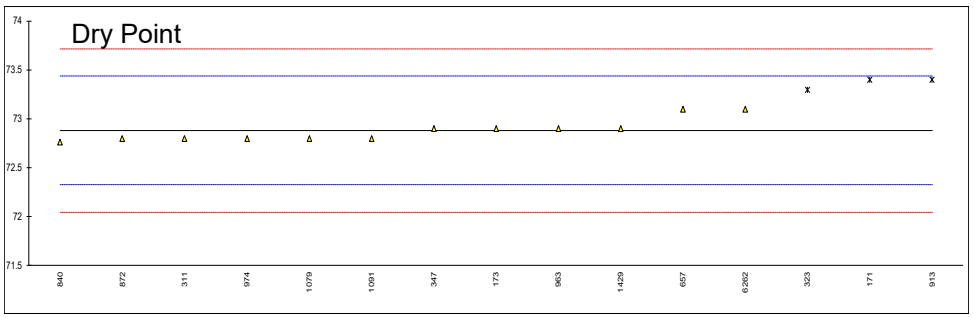
G(1) = outlier in Grubbs' outlier test
DG(5) = straggler in Double Grubbs' outlier test

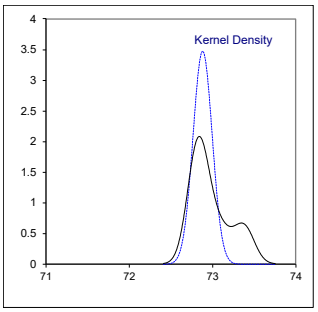

Initial Boiling Point
963 840 657 311 323 1079 173 974 1091 1429 347 872 913 6262 171

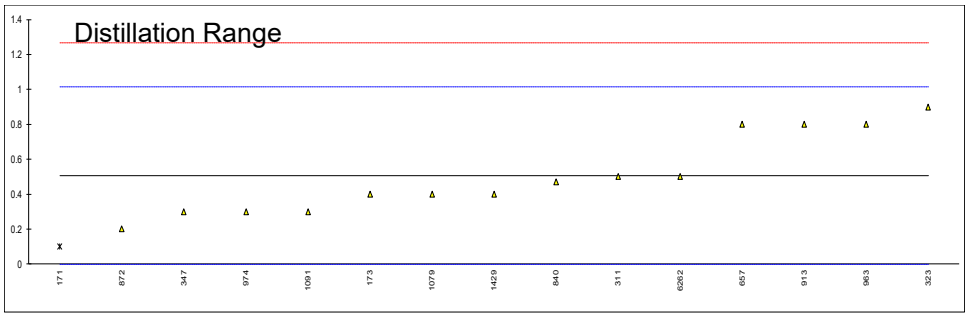

Kernel Density

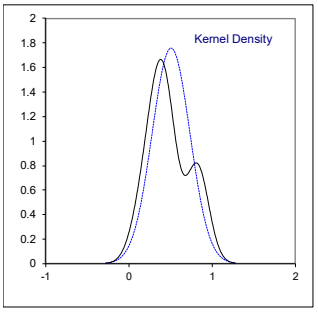

50% recovered
347 173 311 323 840 657 872 974 1079 1091 913 963 1429 6262 171


Kernel Density


Dry Point
840 872 311 974 1079 1091 347 173 963 1429 657 6262 323 171 913


Kernel Density

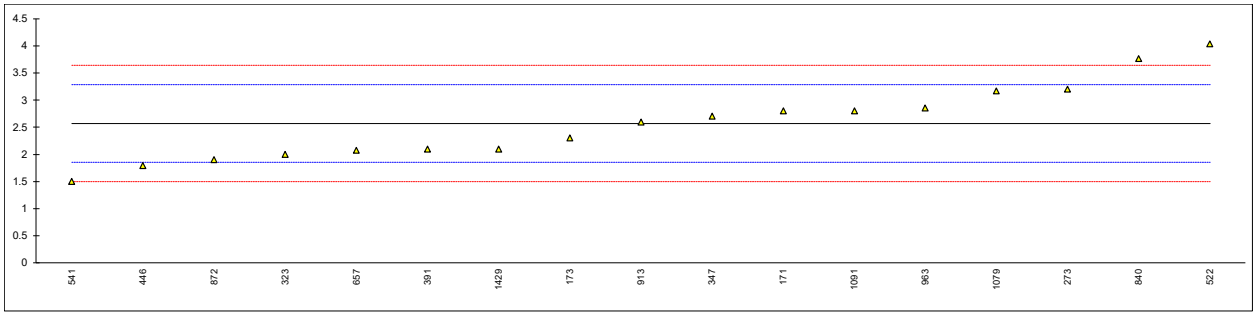

Distillation Range
171 872 347 974 1091 173 1079 1429 840 311 6262 657 913 963 323

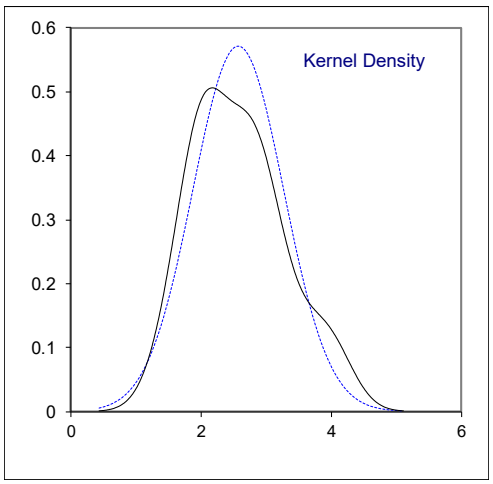

Kernel Density

Determination of Inhibitor as Hydroquinone on sample #20001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2193	2.8		0.64	
173	D2193	2.3		-0.76	
273	1B-2A2.40-1u	3.2		1.76	
311		-----		-----	
323	D2193	2		-1.60	
347	D2193	2.7		0.36	
391	D2193	2.1		-1.32	
446	D2193	1.8		-2.16	
522	IB-2A2.40-1	4.04		4.11	
541	D2193	1.50		-3.00	
551		-----		-----	
657	D2193	2.08		-1.37	
840	D2193	3.76		3.33	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872	INH-88	1.9		-1.88	
913	D2193	2.6		0.08	
963	D2193	2.86		0.81	
974		-----		-----	
1079	JIS6724	3.167		1.67	
1091	In house	2.8		0.64	
1429	D2193	2.1		-1.32	
6262		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	2.57
st.dev. (n)	0.698
R(calc.)	1.96
st.dev.(D2193:06)	0.357
R(D2193:06)	1.0

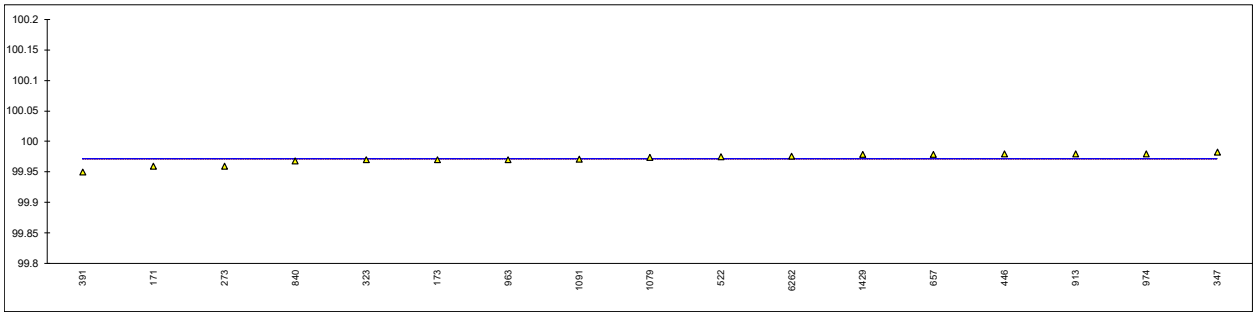


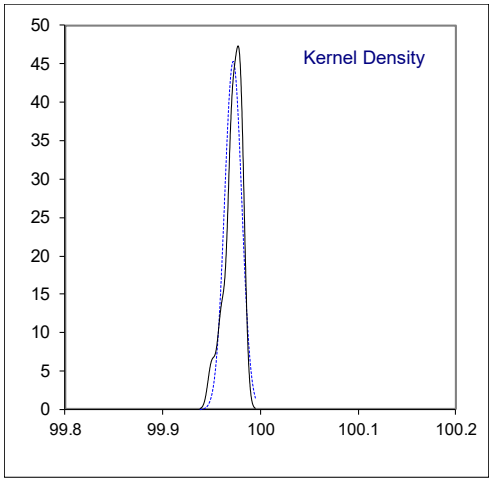


Determination of Purity by GC on sample #20001; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	INH-12	99.96		-----	
173	INH-257	99.97		-----	
273	INH-12A	99.96		-----	
311		-----		-----	
323	In house	99.97		-----	
347	OINH-096	99.983		-----	
391	INH-257	99.95		-----	
446	INH-102582	99.98		-----	
522	INH-102582	99.975		-----	
541		-----		-----	
551		-----		-----	
657	INH-0047	99.9790		-----	
840	D3545Mod.	99.968		-----	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872		-----		-----	
913	INH-12345	99.98		-----	
963	INH-8124	99.97		-----	
974	INH-2401	99.98		-----	
1079	In house	99.9741		-----	
1091	In house	99.971		-----	
1429		99.9788		-----	
6262		99.9763		-----	

	normality	suspect
	n	17
	outliers	0
	mean (n)	99.9721
	st.dev. (n)	0.00881
	R(calc.)	0.0247
	st.dev.(lit)	n.a.
	R(lit)	n.a.
Compare		
	R(iis19C01)	0.1266
	R(iis18C01)	0.0127

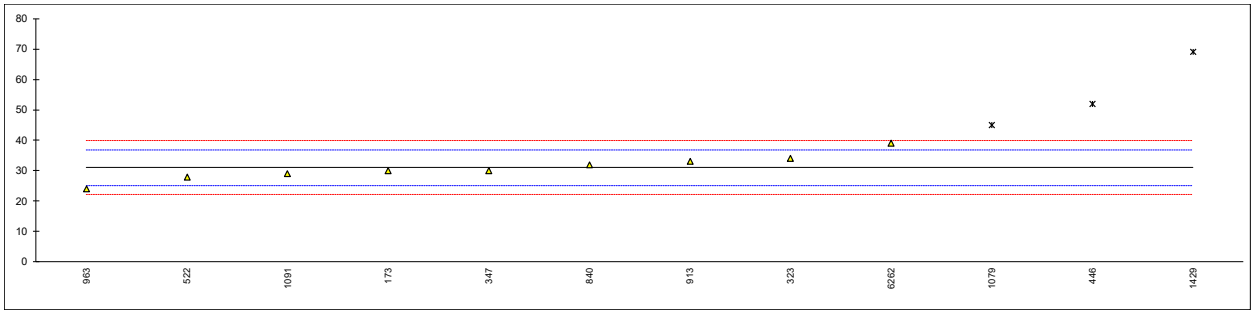


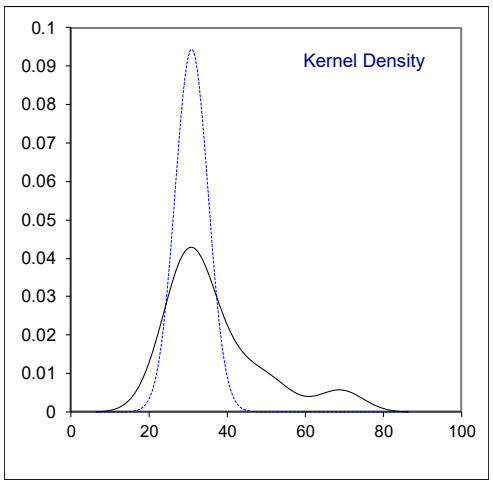


Determination of Acetaldehyde on sample #20001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173	INH-47	30		-0.33	
273		-----		-----	
311		-----		-----	
323	In house	34		1.02	
347	INH-096	30		-0.33	
391		-----		-----	
446	INH-102582	52	DG(0.05)	7.11	
522	INH-102582	27.9		-1.04	
541		-----		-----	
551		-----		-----	
657		-----		-----	
840	D3545Mod.	32		0.34	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872		-----		-----	
913	INH-12345	33		0.68	
963	INH-8124	24	C	-2.36	first reported 18
974		-----		-----	
1079	In house	45	C,DG(0.05)	4.74	first reported 51
1091	In house	29		-0.67	
1429		69	G(0.05)	12.85	
6262		39	C	2.71	first reported 19

normality	suspect
n	9
outliers	3
mean (n)	30.99
st.dev. (n)	4.222
R(calc.)	11.82
st.dev.(Horwitz)	2.957
R(Horwitz)	8.28





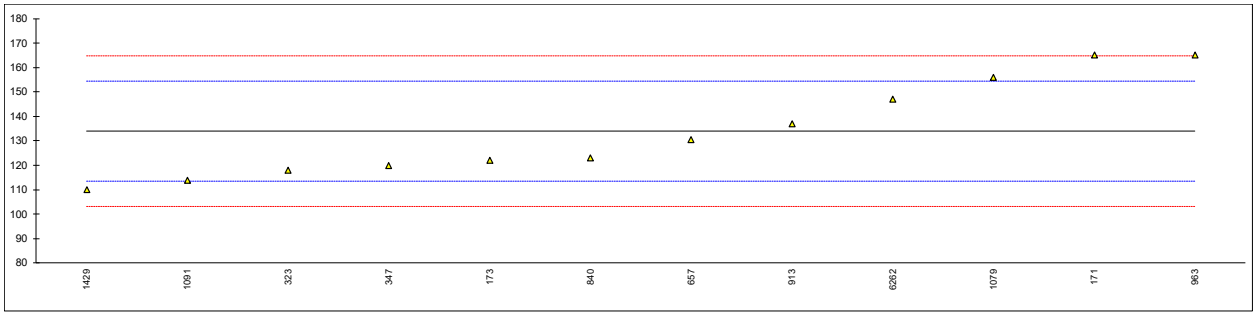
Determination of Acetone on sample #20001, results in mg/kg

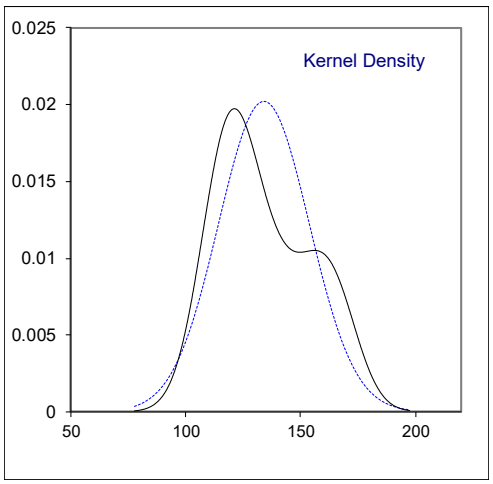
lab	method	value	mark	z(targ)	remarks
171	INH-12	<10		-----	
173		-----		-----	
273		-----		-----	
311		-----		-----	
323	In house	< 10		-----	
347		-----		-----	
391		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551		-----		-----	
657	INH-0047	< 10		-----	
840	D3545Mod.	<10		-----	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872		-----		-----	
913	INH-12345	12		-----	
963		-----		-----	
974		-----		-----	
1079	In house	8		-----	
1091		-----		-----	
1429		8		-----	
6262		<10		-----	
	n	7			
	mean (n)	<10			

Determination of Ethyl Acetate on sample #20001, results in mg/kg

lab	method	value	mark	z(targ)	Remarks
171	INH-12	165		3.03	
173	INH-47	122		-1.17	
273		-----		-----	
311		-----		-----	
323	In house	118		-1.56	
347	INH-096	120		-1.36	
391		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551		-----		-----	
657	INH-0047	130.40		-0.35	
840	D3545Mod.	123		-1.07	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872		-----		-----	
913	INH-12345	137		0.30	
963	INH-8124	165		3.03	
974		-----		-----	
1079	In house	156		2.15	
1091	In house	114		-1.95	
1429		110		-2.34	
6262		147	C	1.27	first reported 75

normality	OK
n	12
outliers	0
mean (n)	133.95
st.dev. (n)	19.748
R(calc.)	55.30
st.dev.(Horwitz)	10.255
R(Horwitz)	28.71

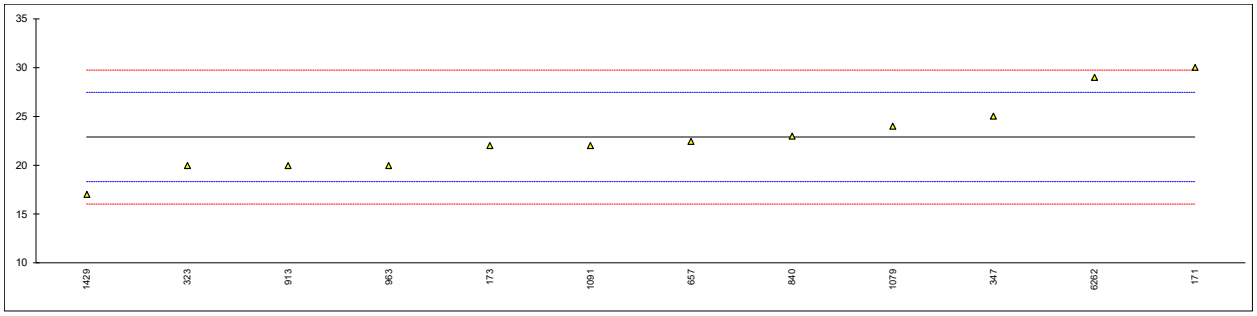


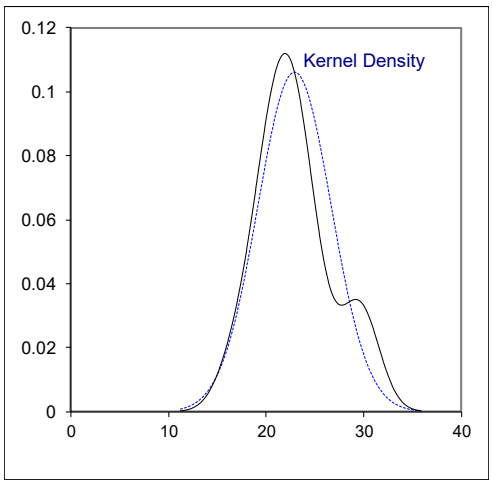


Determination of Methyl Acetate on sample #20001; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
171	INH-12	30		3.12	
173	INH-47	22		-0.38	
273		-----		-----	
311		-----		-----	
323	In house	20		-1.26	
347	INH-096	25		0.93	
391		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551		-----		-----	
657	INH-0047	22.45		-0.18	
840	D3545Mod.	23		0.06	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872		-----		-----	
913	INH-12345	20		-1.26	
963	INH-8124	20		-1.26	
974		-----		-----	
1079	In house	24		0.49	
1091	In house	22		-0.38	
1429		17		-2.57	
6262		29		2.68	

normality	OK
n	12
outliers	0
mean (n)	22.87
st.dev. (n)	3.755
R(calc.)	10.51
st.dev.(Horwitz)	2.285
R(Horwitz)	6.40

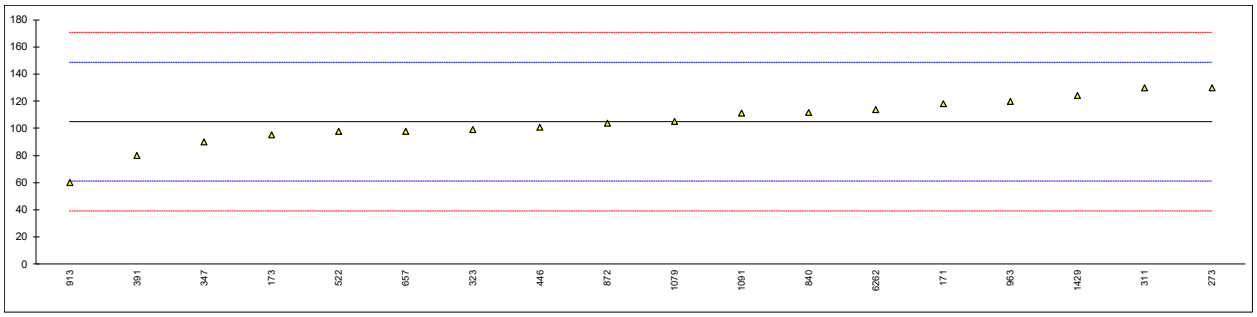


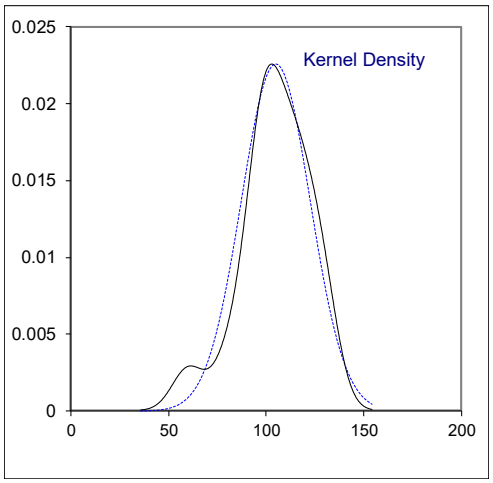


Determination of Water on sample #20001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	118		0.60	
173	E203	95		-0.45	
273	E203	130		1.14	
311	E203	130		1.14	
323	D1364	99		-0.27	
347	D1364	90		-0.68	
391	E203	80	C	-1.13	first reported 180
446	D1364	101		-0.18	
522	E203	97.7		-0.33	
541		-----		-----	
551		-----		-----	
657	E1064	97.8		-0.32	
840	D1364	111.8		0.31	
859		-----		-----	
860		-----		-----	
861		-----		-----	
872	D1364	104		-0.04	
913	E203	60		-2.05	
963	D1364	120		0.69	
974		-----		-----	
1079	D1364	105		0.00	
1091	D1364	111		0.28	
1429	D1364	124		0.87	
6262	D1364	114		0.41	

	normality	suspect
	n	18
	outliers	0
	mean (n)	104.91
	st.dev. (n)	17.659
	R(calc.)	49.44
	st.dev.(D1364:02)	21.948
	R(D1364:02)	61.45
Compare		
	R(E203)	780





APPENDIX 2

Number of participants per country

1 lab in	ARGENTINA
4 labs in	BELGIUM
1 lab in	BRAZIL
3 labs in	CHINA, People's Republic
1 lab in	INDIA
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
1 lab in	SPAIN
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 ISO5725:05
5 ISO5725, 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)
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