Results of Proficiency Test
Vinyl Acetate Monomer (VAM)
February 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 2007 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Vinyl Acetate Monomer (VAM) in accordance with the latest version of ASTM D2190 every year. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of Vinyl Acetate Monomer.

In this interlaboratory study 24 laboratories in 16 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the Vinyl Acetate Monomer 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 Vinyl Acetate Monomer in a 0.5 liter amber glass bottle labelled #22001.
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 of Vinyl Acetate Monomer was obtained from a participating laboratory. After homogenization 44 amber glass bottles of 0.5 liter were filled and labelled #22001.
The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ASTM D12185 and Water in accordance with ASTM E203 on 8 stratified randomly selected subsamples.

	Density at 20°C in kg/L	Water in mg/kg
sample #22001-1	0.93216	120
sample #22001-2	0.93216	120
sample #22001-3	0.93217	110
sample #22001-4	0.93216	110
sample #22001-5	0.93217	120
sample #22001-6	0.93216	120
sample #22001-7	0.93216	120
sample #22001-8	0.93217	120

Table 1: homogeneity test results of subsamples #22001

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the 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)	0.00001	13.0
reference test method	ISO12185:96	D1364:22
0.3 x R (reference test method)	0.00015	19.5

Table 2: evaluation of the repeatabilities of subsamples #22001

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

To each of the participating laboratories one sample Vinyl Acetate Monomer labelled #22001 was sent on January 12, 2022. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Vinyl Acetate Monomer 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: Acidity without 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 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 the original test results are placed under 'Remarks' in the 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO 13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier test can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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. Therefore, the usual interpretation of z-scores is as follows:

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

4 EVALUATION

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with one week. One participant reported test results after the extended final reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.

In total 21 participants reported 225 numerical test results. Observed were 8 outlying test results, which is 3.6%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all 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 in appendix 1. 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 tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

Acidity without Nitrogen purge: This determination was problematic. One statistical outlier was observed. Due to the large variation in test results, no z-scores are calculated.

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.

Color Pt/Co: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good 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 good agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. In total two statistical outliers were observed over four parameters. All calculated reproducibilities after rejection of the statistical outliers are in agreement with the requirements of the automated mode of ASTM D1078:11(2019).

Inhibitor as 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 D2193:22.

Purity by GC: This determination may not be problematic. Two statistical outliers were observed. The calculated reproducibility (0.0130) was smaller in comparison with the calculated reproducibility of the previous PT (0.0232 in iis21C01). Regretfully, no reference test method with precision data exists for this determination. Therefore, no z-scores are calculated.

Acetaldehyde: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated with 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 are calculated.

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

Methyl Acetate: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with 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 D1364:22.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference test methods (in casu ASTM and ISO test methods) or estimated using the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity without N_2 purge	mg/kg	11	24.1	17.2	(6)
Apparent Specific Gravity 20/20°C		19	0.9339	0.0002	0.0005
Color Pt/Co		15	3.0	2.8	7
Density at 20°C	kg/L	20	0.9322	0.0002	0.0005
Initial Boiling Point	°C	15	72.5	0.3	1.1
50% recovery	°C	13	72.7	0.2	0.5
Dry Point	°C	15	72.9	0.3	0.8
Range	°C	15	0.4	0.5	0.7
Inhibitor as Hydroquinone	mg/kg	20	2.4	1.2	1.0
Purity by GC	%M/M	15	99.976	0.013	n.a.
Acetaldehyde	mg/kg	12	27.8	15.8	7.5
Acetone	mg/kg	9	<10	n.e.	n.e.
Ethyl Acetate	mg/kg	14	124	31	27
Methyl Acetate	mg/kg	12	14.7	6.0	4.4
Water	mg/kg	18	90	41	57

Table 3: reproducibilities of tests on sample #22001

Results between brackets no z-scores are calculated

Without further statistical calculations, it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2022 WITH PREVIOUS PTS

	February 2022	February 2021	February 2020	February 2019	February 2018
Number of reporting laboratories	21	22	20	27	25
Number of test results	225	225	214	247	282
Number of statistical outliers	8	11	10	11	10
Percentage of statistical outliers	3.6%	4.9%	4.7%	4.5%	3.5%

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 tests was compared against the requirements of the reference test methods. The conclusions are given in the following table.

Parameter	February 2022	February 2021	February 2020	February 2019	February 2018
Acidity without N_2 purge	(--)	--	--	(--)	-
Apparent Specific Gravity 20/20°C	++	++	++	+	++
Color Pt/Co	++	++	++	+	++
Density at 20°C	++	++	++	+	++
Distillation	++	++	++	+/-	++
Inhibitor as Hydroquinone	-	+	--	(--)	-
Acetaldehyde	--	+	-	-	--
Acetone	n.e.	n.e.	n.e.	n.e.	n.e.
Ethyl Acetate	-	-	-	-	++
Methyl Acetate	-	-	-	-	+
Water	+	+	+	+	+

Table 5: comparison determinations against the reference test methods
Results between brackets no z-scores are calculated

The following performance categories were used:

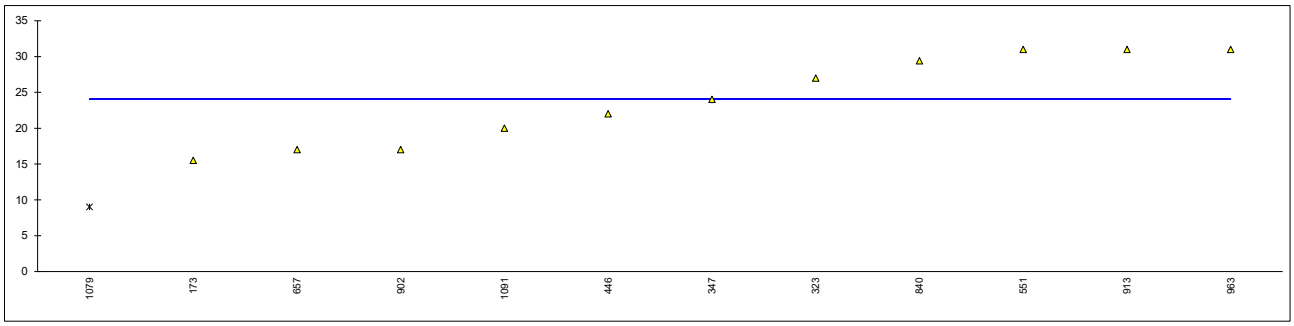
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

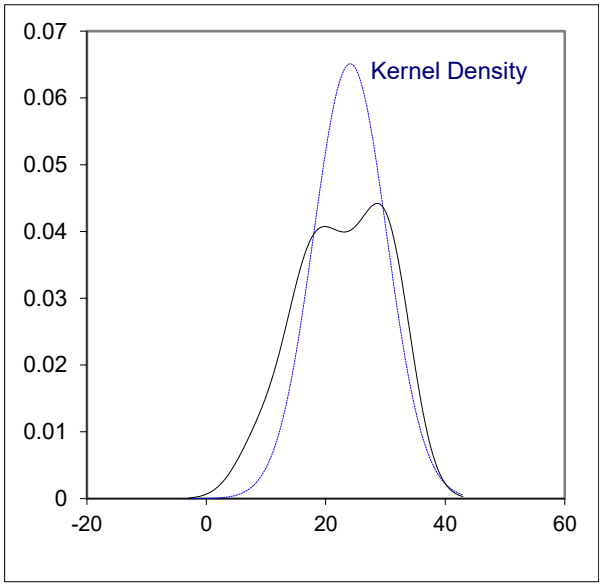
APPENDIX 1

Determination of Acidity without N_2 purge on sample #22001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173	INH-44	15.5		-----	
273		-----		-----	
323	D2086	27		-----	
347	D2086	24		-----	
391		-----		-----	
396		-----		-----	
446	INH-40	22		-----	
522		-----		-----	
541		-----		-----	
551	D2086	31		-----	
657	D2086	17		-----	
840	D2086	29.4		-----	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902	D2086	17		-----	
913	D2086	31		-----	
963	D2086	31		-----	
974		-----		-----	
1079	D2086	9	G(0.05)	-----	
1091	D2086	20		-----	
1429		-----		-----	
6262		-----		-----	

normality	OK
n	11
outliers	1
mean (n)	24.08
st.dev. (n)	6.126
R(calc.)	17.15
st.dev.(D2086:22)	(2.143)
R(D2086:22)	(6)

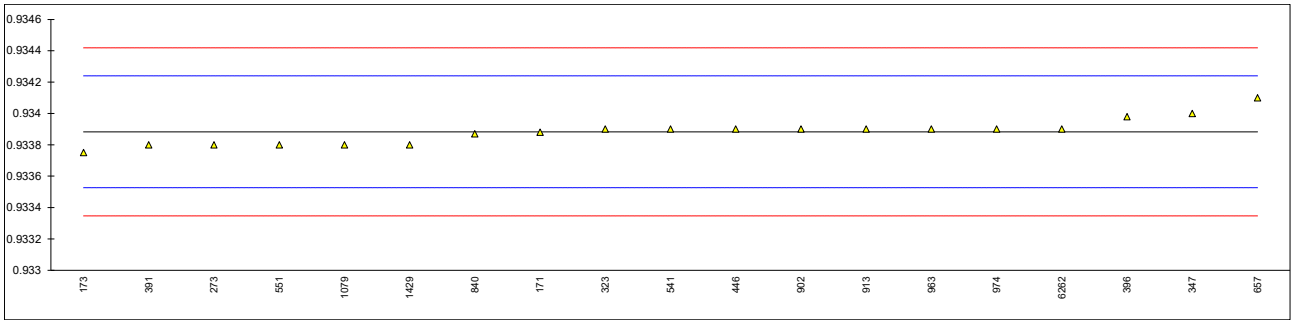


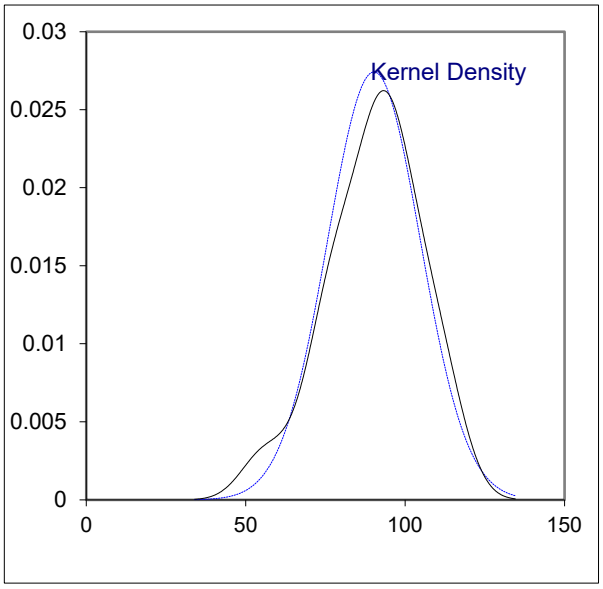


Determination of Apparent Specific Gravity 20/20°C on sample #22001;

lab	method	value	mark	z(targ)	remarks
171	D4052	0.93388		-0.02	
173	D4052	0.93375		-0.75	
273	D4052	0.9338		-0.47	
323	D4052	0.9339		0.09	
347	D4052	0.9340		0.65	
391	ISO12185	0.9338		-0.47	
396	D4052	0.93398		0.54	
446	D4052	0.9339		0.09	
522		-----		-----	
541	D4052	0.93390		0.09	
551	D4052	0.9338		-0.47	
657	D4052	0.9341		1.21	
840	D4052	0.93387		-0.07	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902	D4052	0.9339		0.09	
913	ISO12185	0.9339		0.09	
963	ISO12185	0.9339		0.09	
974	D4052	0.9339		0.09	
1079	ISO12185	0.9338		-0.47	
1091		-----		-----	
1429	D4052	0.9338		-0.47	
6262	D4052	0.9339		0.09	

normality	suspect
n	19
outliers	0
mean (n)	0.93388
st.dev. (n)	0.000083
R(calc.)	0.00023
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

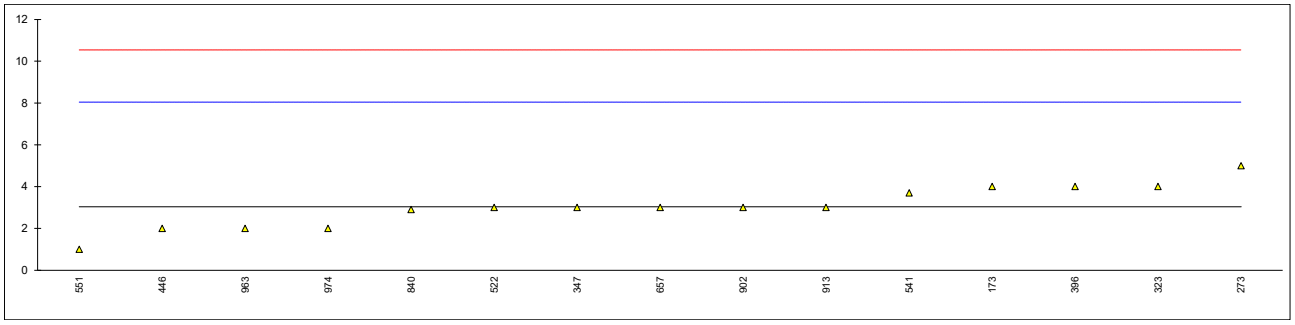


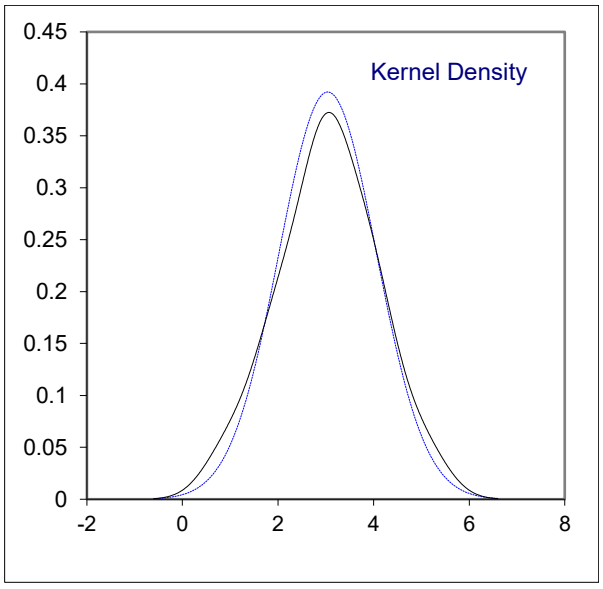


Determination of Color Pt/Co on sample #22001;

lab	method	value	mark	z(targ)	remarks
171	D1209	<5		-----	
173	D1209	4		0.38	
273	D1209	5		0.78	
323	D5386	4		0.38	
347	D5386	3		-0.02	
391	D1209	<5		-----	
396	D5386	4		0.38	
446	D5386	2		-0.42	
522	D1209	3		-0.02	
541	D5386	3.7		0.26	
551	D1209	1		-0.82	
657	D1209	3		-0.02	
840	D5386	2.9		-0.06	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902	D1209	3		-0.02	
913	D1209	3		-0.02	
963	D1209	2		-0.42	
974	D5386	2		-0.42	
1079	D1209	<5		-----	
1091	D5386	<5		-----	
1429	D1209	<5		-----	
6262	D1209	<5		-----	

normality	OK
n	15
outliers	0
mean (n)	3.04
st.dev. (n)	1.017
R(calc.)	2.85
st.dev.(D1209:05)	2.500
R(D1209:05)	7

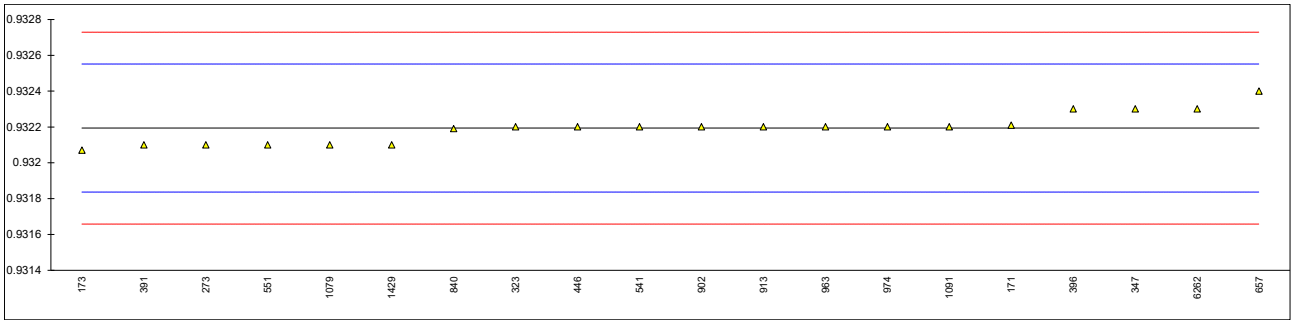


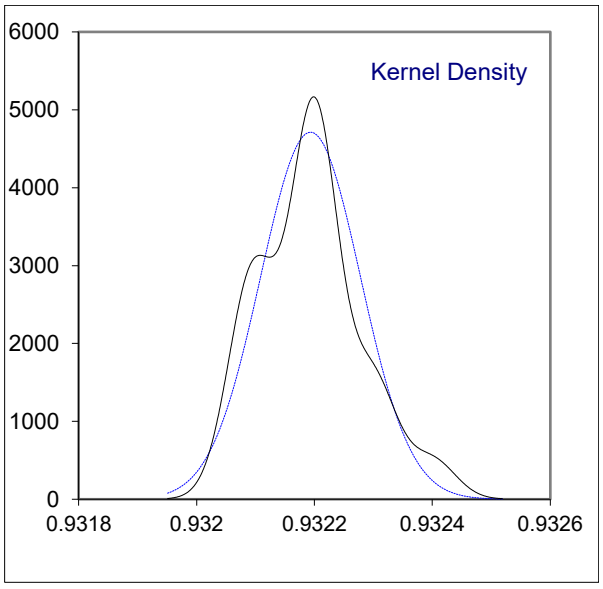


Determination of Density at 20°C on sample #22001; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.93221		0.09	
173	D4052	0.93207		-0.69	
273	D4052	0.9321		-0.52	
323	D4052	0.9322		0.04	
347	D4052	0.9323		0.60	
391	ISO12185	0.9321		-0.52	
396	D4052	0.9323		0.60	
446	D4052	0.9322		0.04	
522		-----		-----	
541	D4052	0.93220		0.04	
551	D4052	0.9321		-0.52	
657	D4052	0.9324		1.16	
840	D4052	0.93219		-0.02	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902	D4052	0.9322		0.04	
913	D4052	0.9322		0.04	
963	ISO12185	0.9322		0.04	
974	D4052	0.9322		0.04	
1079	ISO12185	0.9321		-0.52	
1091	D4052	0.9322		0.04	
1429	D4052	0.9321		-0.52	
6262	D4052	0.9323		0.60	

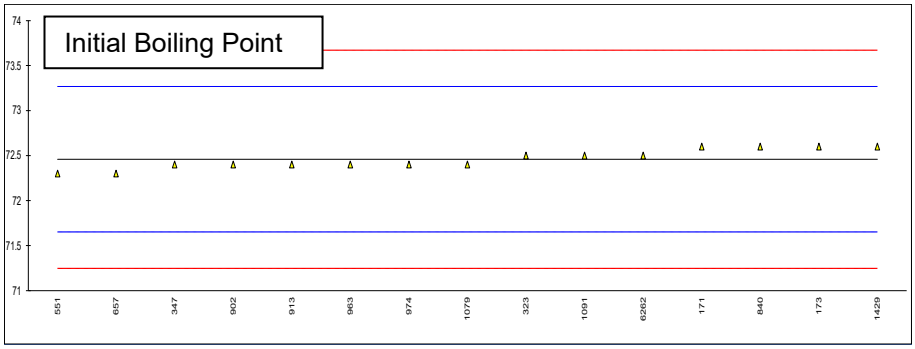
normality	OK
n	20
outliers	0
mean (n)	0.93219
st.dev. (n)	0.000085
R(calc.)	0.00024
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

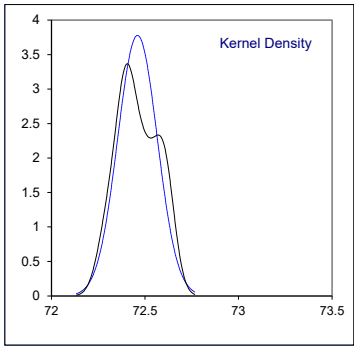


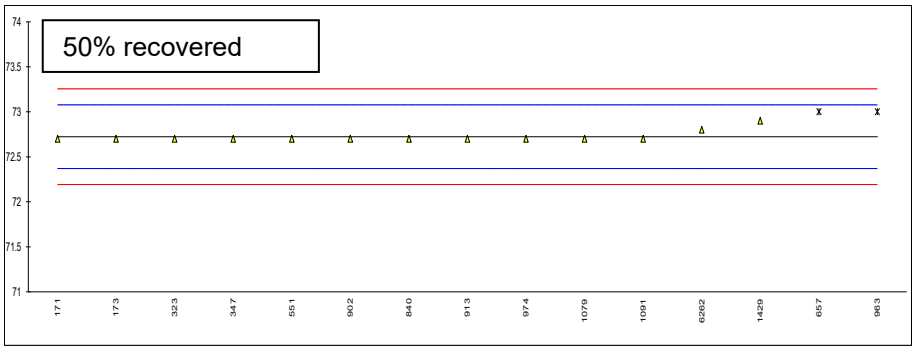


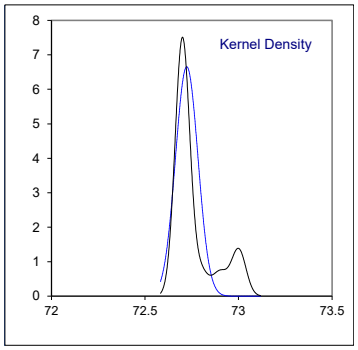
Determination of Distillation on sample #22001; results in °C

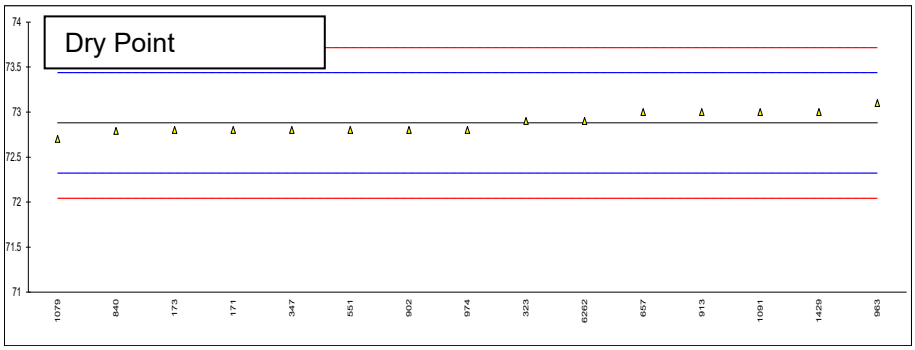
lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	DP	mark	z(targ)	range	mark	z(targ)
171	D1078-automated	72.6		0.35	72.7		-0.13	72.8		-0.28	0.2		-0.88
173	D1078-automated	72.6		0.35	72.7		-0.13	72.8		-0.28	0.2		-0.88
273		-----		-----	-----		-----	-----		-----	-----		-----
323	D1078-automated	72.5		0.10	72.7		-0.13	72.9		0.07	0.4		-0.08
347	D1078-automated	72.4		-0.15	72.7		-0.13	72.8		-0.28	0.4		-0.08
391		-----		-----	-----		-----	-----		-----	-----		-----
396		-----		-----	-----		-----	-----		-----	-----		-----
446		-----		-----	-----		-----	-----		-----	-----		-----
522		-----		-----	-----		-----	-----		-----	-----		-----
541		-----		-----	-----		-----	-----		-----	-----		-----
551	D1078-automated	72.3		-0.40	72.7		-0.13	72.8		-0.28	0.5		0.32
657	D1078-automated	72.3		-0.40	73.0	DG5	1.56	73.0		0.43	0.7		1.12
840	D1078-automated	72.60		0.35	72.70		-0.13	72.79		-0.32	0.19		-0.92
859		-----		-----	-----		-----	-----		-----	-----		-----
860		-----		-----	-----		-----	-----		-----	-----		-----
861		-----		-----	-----		-----	-----		-----	-----		-----
902	D1078-automated	72.4		-0.15	72.7		-0.13	72.8		-0.28	0.4		-0.08
913	D1078-automated	72.4		-0.15	72.7		-0.13	73.0		0.43	0.6		0.72
963	D1078-automated	72.4		-0.15	73.0	DG5	1.56	73.1		0.79	0.7		1.12
974	D1078-automated	72.4		-0.15	72.7		-0.13	72.8		-0.28	0.4		-0.08
1079	D1078-automated	72.4		-0.15	72.7		-0.13	72.7		-0.64	0.3		-0.48
1091		72.5		0.10	72.7		-0.13	73.0		0.43	0.5		0.32
1429	D1078-automated	72.6		0.35	72.9		1.00	73.0		0.43	0.4		-0.08
6262	D1078-automated	72.5		0.10	72.8		0.43	72.9		0.07	0.4		-0.08
	normality	OK			not OK			OK			OK		
	n	15			13			15			15		
	outliers	0			2			0			0		
	mean (n)	72.46			72.72			72.87			0.42		
	st.dev. (n)	0.106			0.060			0.115			0.162		
	R(calc.)	0.30			0.18			0.28			0.45		
	st.dev.(D1078-A:11)	0.404			0.177			0.279			0.250		
	R(D1078-A:11)	1.13			0.50			0.78			0.70		

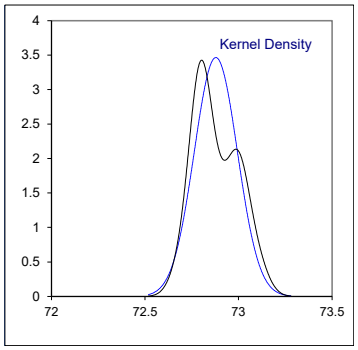

Initial Boiling Point
74
73.5
73
72.5
72
71.5
71
551 657 347 902 913 963 974 1079 323 1091 6262 171 840 173 1429

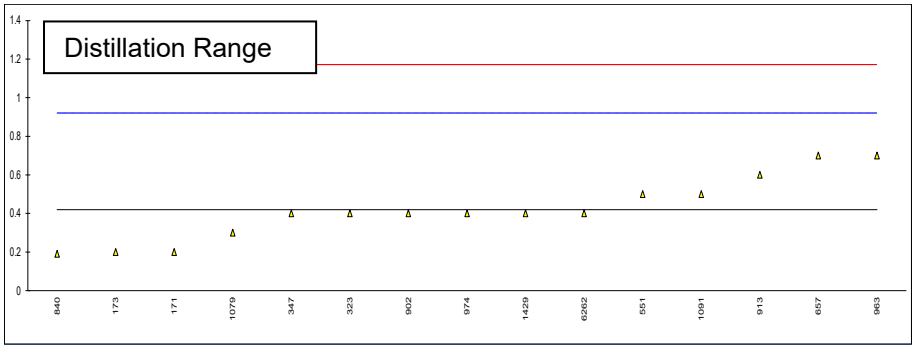

Kernel Density
4
3.5
3
2.5
2
1.5
1
0.5
0
72 72.5 73 73.5

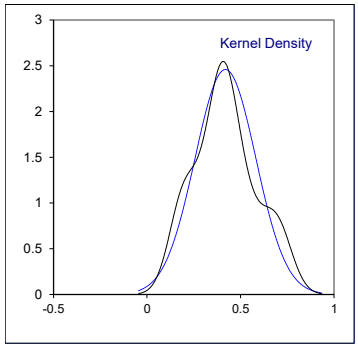

50% recovered
74
73.5
73
72.5
72
71.5
71
171 173 323 347 551 902 840 913 974 1079 1091 6262 1429 657 963


Kernel Density
8
7
6
5
4
3
2
1
0
72 72.5 73 73.5


Dry Point
74
73.5
73
72.5
72
71.5
71
1079 840 173 171 347 551 902 974 323 6262 657 913 1091 1429 963


Kernel Density
4
3.5
3
2.5
2
1.5
1
0.5
0
72 72.5 73 73.5

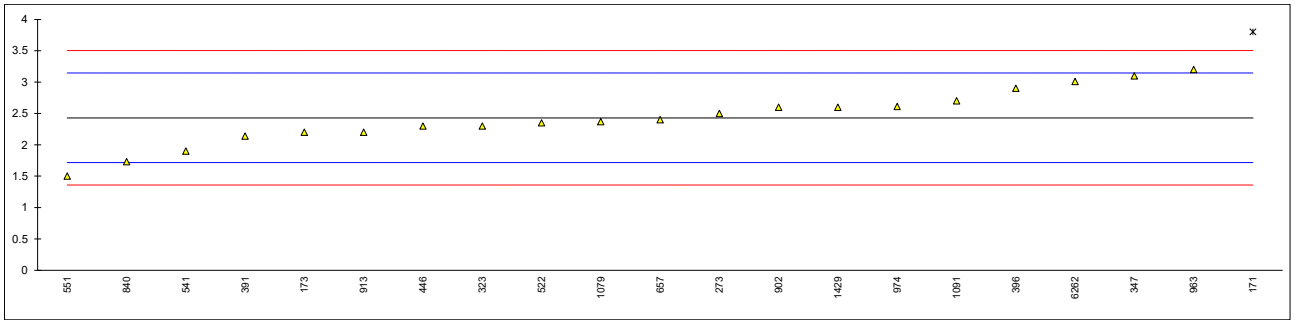

Distillation Range
1.4
1.2
1
0.8
0.6
0.4
0.2
0
840 173 171 1079 347 323 902 974 1429 6262 551 1091 913 657 963

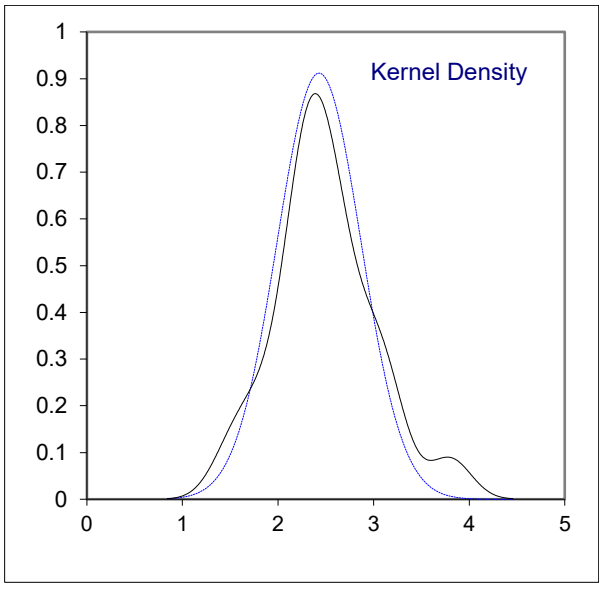

Kernel Density
3
2.5
2
1.5
1
0.5
0
-0.5 0 0.5 1

Determination of Inhibitor as Hydroquinone on sample #22001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2193	3.8	G(0.05)	3.83	
173	D2193	2.2	C	-0.65	first reported 4.0
273	INH-40	2.50		0.19	
323	D2193	2.3		-0.37	
347	D2193	3.1		1.87	
391	D2193	2.14		-0.81	
396	D2193	2.9		1.31	
446	INH-40	2.3		-0.37	
522	INH-40-1	2.35		-0.23	
541	D2193	1.90		-1.49	
551	D2193	1.5		-2.61	
657	D2193	2.4		-0.09	
840	D2193	1.73		-1.96	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902	D2193	2.6		0.47	
913	D2193	2.2		-0.65	
963	INH-8309	3.2		2.15	
974	D2193	2.61		0.50	
1079	JIS6724	2.37		-0.17	
1091	In house	2.7		0.75	
1429	D2193	2.6		0.47	
6262	D2193	3.01		1.62	

normality	OK
n	20
outliers	1
mean (n)	2.43
st.dev. (n)	0.438
R(calc.)	1.23
st.dev.(D2193:22)	0.357
R(D2193:22)	1.0

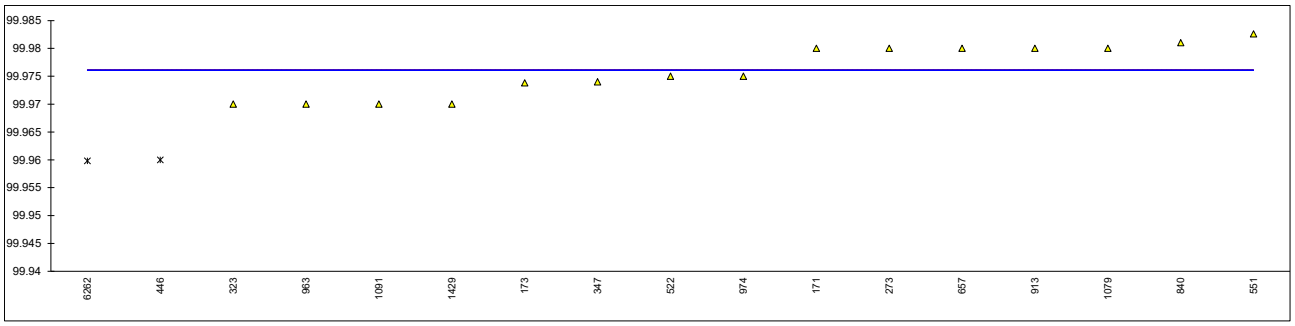


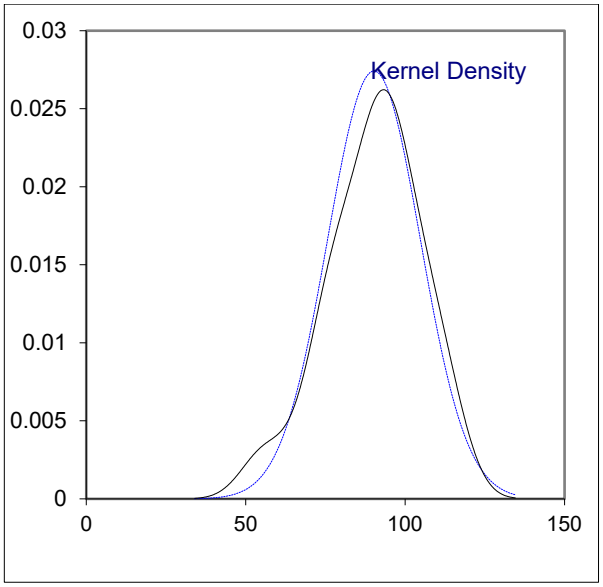


Determination of Purity by GC on sample #22001; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	INH-0001	99.98		-----	
173	INH-257	99.9738		-----	
273	INH-12A	99.98		-----	
323	INH-067	99.97		-----	
347	INH-096	99.974		-----	
391		-----		-----	
396		-----		-----	
446	INH-10285	99.96	G(0.05)	-----	
522	DOWM 102582	99.975		-----	
541		-----		-----	
551	INH-1355	99.9826		-----	
657	INH-0047	99.98		-----	
840	D3545	99.981		-----	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902		-----		-----	
913	D3545	99.98		-----	
963	INH-8124	99.97		-----	
974	INH-40	99.975	C	-----	first reported 99.935
1079	In house	99.98		-----	
1091	In house	99.97		-----	
1429	In house	99.97		-----	
6262		99.9598	G(0.05)	-----	

	normality	OK
	n	15
	outliers	2
	mean (n)	99.9761
	st.dev. (n)	0.00465
	R(calc.)	0.0130
	st.dev.(lit)	n.a.
	R(lit)	n.a.
Compare		
	R(iis21C01)	0.0232

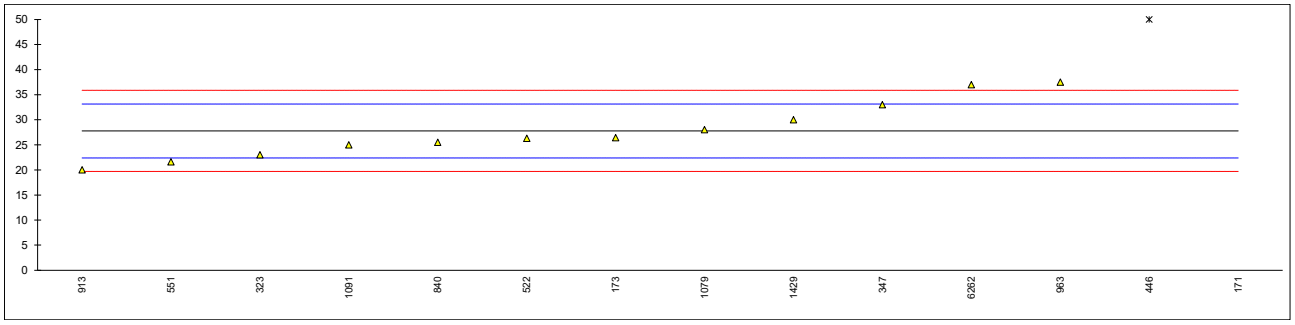


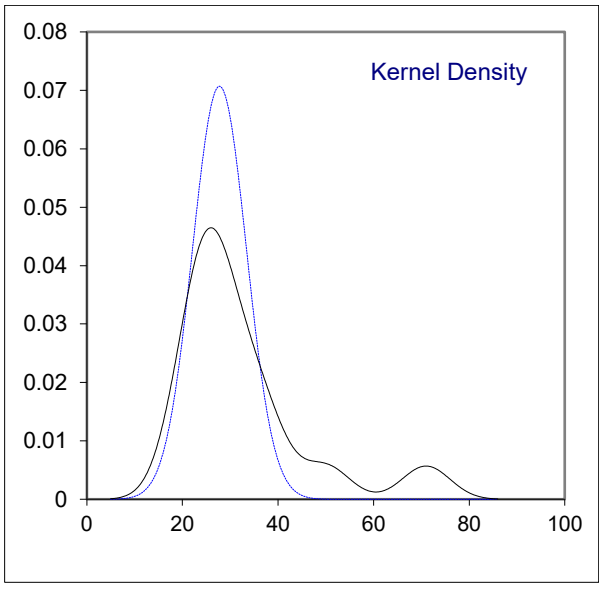


Determination of Acetaldehyde on sample #22001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-001	71	G(0.01)	16.04	
173	INH-47	26.41		-0.51	
273		-----		-----	
323	INH-067	23		-1.77	
347	INH-096	33		1.94	
391		-----		-----	
396		-----		-----	
446	INH-10282	50	G(0.05)	8.25	
522	DOWM 102582	26.3		-0.55	
541		-----		-----	
551	INH-1355	21.6		-2.29	
657		-----		-----	
840	D3545	25.5		-0.84	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902		-----		-----	
913	D3545	20		-2.89	
963	INH-8124	37.5		3.61	
974		-----		-----	
1079	In house	28		0.08	
1091	In house	25		-1.03	
1429	In house	30		0.83	
6262		37		3.42	

normality	OK
n	12
outliers	2
mean (n)	27.78
st.dev. (n)	5.643
R(calc.)	15.80
st.dev.(Horwitz)	2.695
R(Horwitz)	7.54





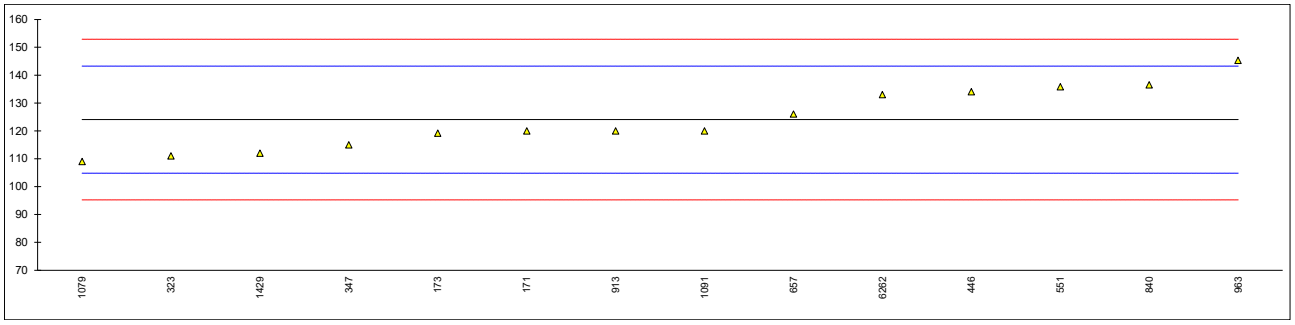
Determination of Acetone on sample #22001; results in mg/kg

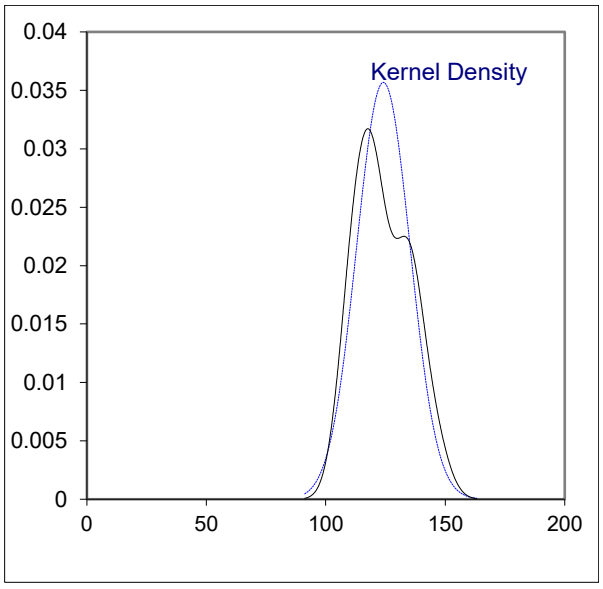
lab	method	value	mark	z(targ)	remarks
171	INH-001	<20		-----	
173		-----		-----	
273		-----		-----	
323	INH-067	< 10		-----	
347	INH-096	<10		-----	
391		-----		-----	
396		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551	INH-1355	1.7		-----	
657	INH-0047	3		-----	
840	D3545	<10		-----	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902		-----		-----	
913	D3545	<10		-----	
963		-----		-----	
974		-----		-----	
1079	In house	0		-----	
1091		-----		-----	
1429	In house	<1		-----	
6262		<10		-----	
	n	9			
	mean (n)	<10			

Determination of Ethyl Acetate on sample #22001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-001	120		-0.42	
173	INH-47	119.16		-0.51	
273		-----		-----	
323	INH-067	111		-1.36	
347	INH-096	115		-0.94	
391		-----		-----	
396		-----		-----	
446	INH-10282	134		1.04	
522		-----		-----	
541		-----		-----	
551	INH-1355	135.8		1.22	
657	INH-0047	126		0.20	
840	D3545	136.5		1.30	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902		-----		-----	
913	D3545	120		-0.42	
963	INH-8124	145.3		2.21	
974		-----		-----	
1079	In house	109		-1.57	
1091	In house	120		-0.42	
1429	In house	112		-1.25	
6262		133		0.93	

normality	OK
n	14
outliers	0
mean (n)	124.05
st.dev. (n)	11.179
R(calc.)	31.30
st.dev.(Horwitz)	9.607
R(Horwitz)	26.90

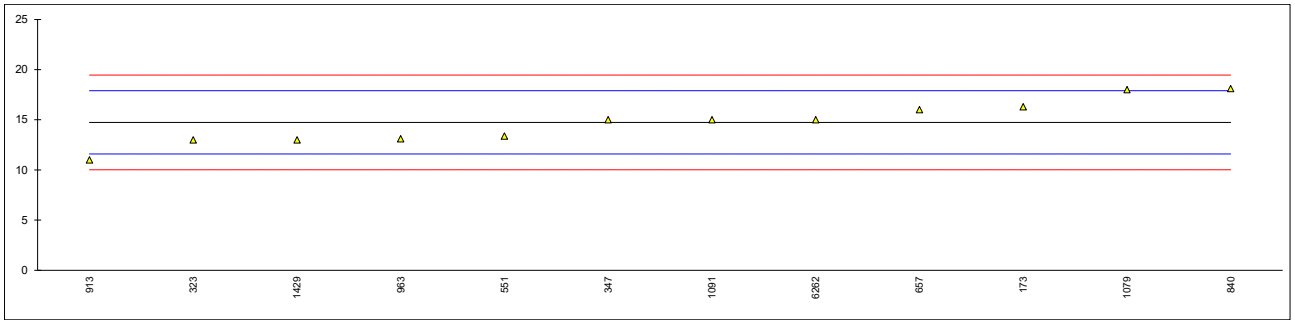


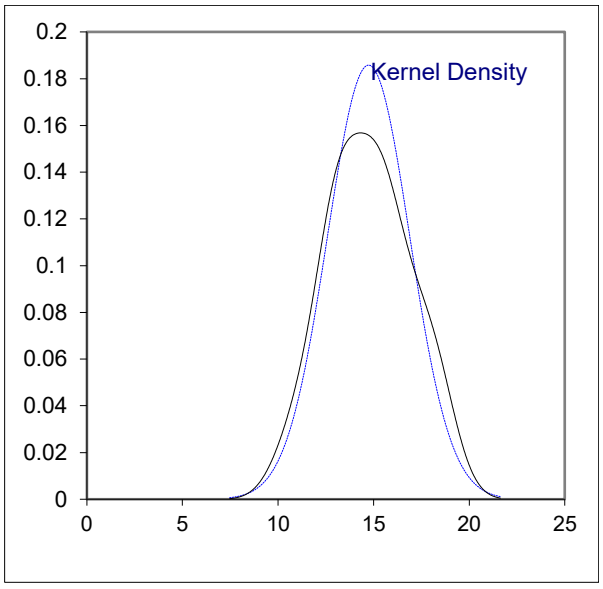


Determination of Methyl Acetate on sample #22001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-001	<20		-----	
173	INH-47	16.29		0.99	
273		-----		-----	
323	INH-067	13		-1.11	
347	INH-096	15		0.17	
391		-----		-----	
396		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551	INH-1355	13.37		-0.87	
657	INH-0047	16		0.80	
840	D3545	18.1		2.14	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902		-----		-----	
913	D3545	11		-2.38	
963	INH-8124	13.1		-1.04	
974		-----		-----	
1079	In house	18		2.07	
1091	In house	15		0.17	
1429	In house	13		-1.11	
6262		15		0.17	

normality	OK
n	12
outliers	0
mean (n)	14.74
st.dev. (n)	2.147
R(calc.)	6.01
st.dev.(Horwitz)	1.573
R(Horwitz)	4.40

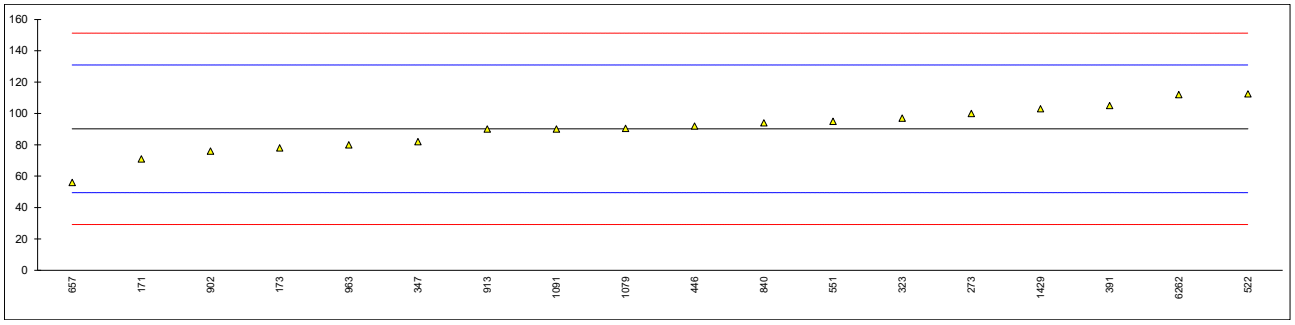


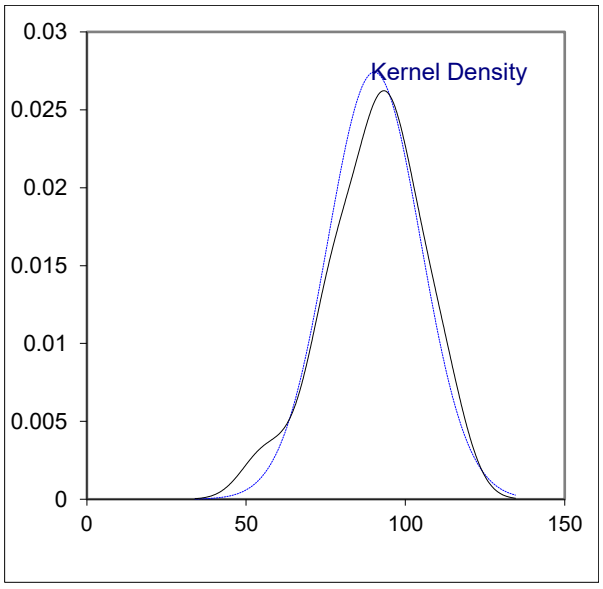


Determination of Water on sample #22001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	E203	71		-0.94	
173	E203	78		-0.60	
273	E203	100		0.48	
323	D1364	97		0.33	
347	D1364	82		-0.40	
391	D1364	105		0.73	
396		-----		-----	
446	D1364	92		0.09	
522	E203	112.5		1.09	
541		-----		-----	
551	D1364	95		0.23	
657	E1064	56		-1.68	
840	D1364	94		0.19	
859		-----		-----	
860		-----		-----	
861		-----		-----	
902	D1364	76		-0.70	
913	D1364	90		-0.01	
963	D1364	80		-0.50	
974		-----		-----	
1079	E203	90.5		0.01	
1091	D1364	90		-0.01	
1429	D1364	103		0.63	
6262	D1364	112		1.07	

normality	OK
n	18
outliers	0
mean (n)	90.22
st.dev. (n)	14.549
R(calc.)	40.74
st.dev.(D1364:22)	20.354
R(D1364:22)	56.99





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
2 labs in	ITALY
1 lab in	MEXICO
1 lab in	SAUDI ARABIA
1 lab in	SINGAPORE
1 lab in	SOUTH AFRICA
1 lab in	SPAIN
1 lab 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) or D1	= outlier in Dixon's outlier test
D(0.05) or D5	= straggler in Dixon's outlier test
G(0.01) or G1	= outlier in Grubbs' outlier test
G(0.05) or G5	= straggler in Grubbs' outlier test
DG(0.01) or DG1	= outlier in Double Grubbs' outlier test
DG(0.05) or DG5	= 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	= calculation difference between reported test result and result calculated by iis
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
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
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6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)