Institute for
Interlaboratory Studies

Results of Proficiency Test
Vinyl Acetate Monomer (VAM)
February 2023

Organized 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 the analysis of Vinyl Acetate Monomer (VAM) in accordance with the latest version of ASTM D2190 every year. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of Vinyl Acetate Monomer.

In this interlaboratory study 23 laboratories in 16 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 #23001.
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 third party. After homogenization 40 amber glass bottles of 0.5 liter were filled and labelled #23001. 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 subsamples.

	Density at 20 °C in kg/L	Water in mg/kg
sample #23001-1	0.93216	210
sample #23001-2	0.93216	222
sample #23001-3	0.93218	221
sample #23001-4	0.93216	202
sample #23001-5	0.93216	207
sample #23001-6	0.93217	212
sample #23001-7	0.93216	208
sample #23001-8	0.93217	209

Table 1: homogeneity test results of subsamples #23001

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)	0.00002	19.3
reference test method	ISO12185:96	D1364:22
0.3 x R (reference test method)	0.00015	26.2

Table 2: evaluation of the repeatabilities of subsamples #23001

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 of 0.5 L Vinyl Acetate Monomer labelled #23001 was sent on January 11, 2023. 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. Therefore, the reporting time on the data entry portal was extended with one week. Four participants reported the test results after the extended reporting date and one other participant did not report any test results. Not all participants were able to report all tests requested.

In total 22 participants reported 226 numerical test results. Observed were 8 outlying test results, which is 3.5%. 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.

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.

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). When a method has been reapproved an “R” will be added and the year of approval (e.g. D1209:05R19).

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. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier 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 D1209:05R19.

Density at 20 °C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. In total two statistical outliers were observed and two other test results were excluded over four parameters. All calculated reproducibilities after rejection of the suspect data are in agreement with the requirements of the automated mode of ASTM D1078:11R19.
From the reported results of the 50% recovered, it appears that three participants probably did not correct the results for barometric pressure and thermometer inaccuracy as described in test method ASTM D1078 in paragraph 11.

Inhibitor as Hydroquinone: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D2193:22.

Purity by GC: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility was larger in comparison with the calculated reproducibility of the previous PT iis22C01 (resp. 0.0331 and 0.0130). Regretfully, no reference test method with precision data exists for this determination. Therefore, no z-scores are calculated.

Acetaldehyde: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Acetone: This determination was not problematic. Almost all participants agreed on a test result near or below the detection limit. Therefore, no z-scores are calculated.

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

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

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D1364:22.

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 methods are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity without N_2 purge	mg/kg	11	38.3	16.7	(6)
Apparent Spec. Gravity 20/20 °C		20	0.9339	0.0002	0.0005
Color Pt/Co		17	3.4	3.8	7
Density at 20 °C	kg/L	20	0.9322	0.0002	0.0005
Initial Boiling Point	°C	13	72.4	0.2	1.1
50% recovery	°C	13	72.7	0.2	0.5
Dry Point	°C	13	72.8	0.3	0.8
Distillation Range	°C	13	0.4	0.3	0.7
Inhibitor as Hydroquinone	mg/kg	21	2.6	1.0	1.0
Purity by GC	%M/M	19	99.963	0.033	n.a.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acetaldehyde	mg/kg	13	34.9	9.4	9.2
Acetone	mg/kg	12	<10	n.e.	n.e.
Ethyl Acetate	mg/kg	14	154	29	32
Methyl Acetate	mg/kg	11	10.7	3.6	3.4
Water	mg/kg	17	220	61	89

Table 3: reproducibilities of tests on sample #23001

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 2023 WITH PREVIOUS PTS

	February 2023	February 2022	February 2021	February 2020	February 2019
Number of reporting laboratories	22	21	22	20	27
Number of test results	226	225	225	214	247
Number of statistical outliers	8	8	11	10	11
Percentage of statistical outliers	3.5%	3.6%	4.9%	4.7%	4.5%

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 to the requirements of the reference test methods. The conclusions are given in the following table.

Parameter	February 2023	February 2022	February 2021	February 2020	February 2019
Acidity without N_2 purge	(--)	(--)	--	--	(--)
Apparent Spec. 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 to the reference test methods

For results between brackets no z-scores are calculated.

The following performance categories were used:

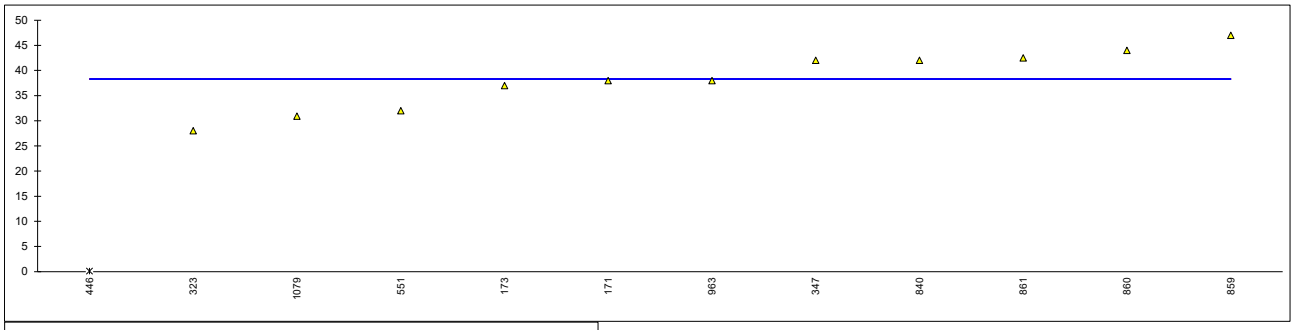
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.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

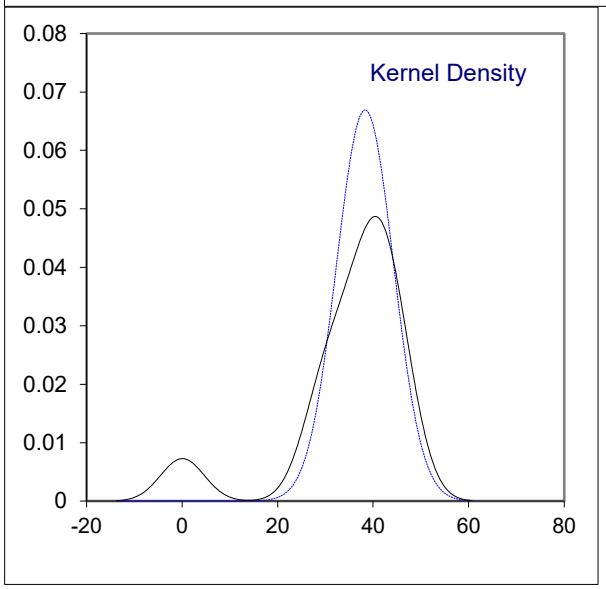
APPENDIX 1

Determination of Acidity without N_2 purge on sample #23001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2086	38		-----	
173	INH-44	37		-----	
273		-----		-----	
323	D2086	28		-----	
347	D2086	42		-----	
357		-----		-----	
391		-----		-----	
396		-----		-----	
446	D2086	0.088	G(0.01)	-----	Possibly a false negative test result?
522		-----		-----	
541		-----		-----	
551	D2086	32	C	-----	first reported 64
657		-----		-----	
840	D2086	42.0		-----	
859	D2086	47		-----	
860	D2086	44		-----	
861	D2086	42.5		-----	
913		-----		-----	
963	D2086	38		-----	
974		-----		-----	
1079	D2086	30.92		-----	
1429	D2086	<10	C	-----	first reported <0.001 mg/kg, Possibly a false negative test result?
6262		-----		-----	

normality	OK
n	11
outliers	1
mean (n)	38.31
st.dev. (n)	5.961
R(calc.)	16.69
st.dev.(D2086:22)	(2.143)
R(D2086:22)	(6)

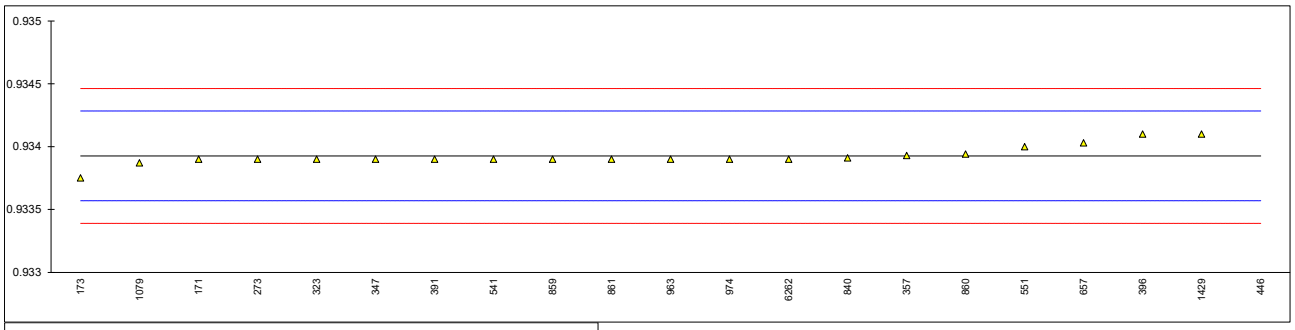


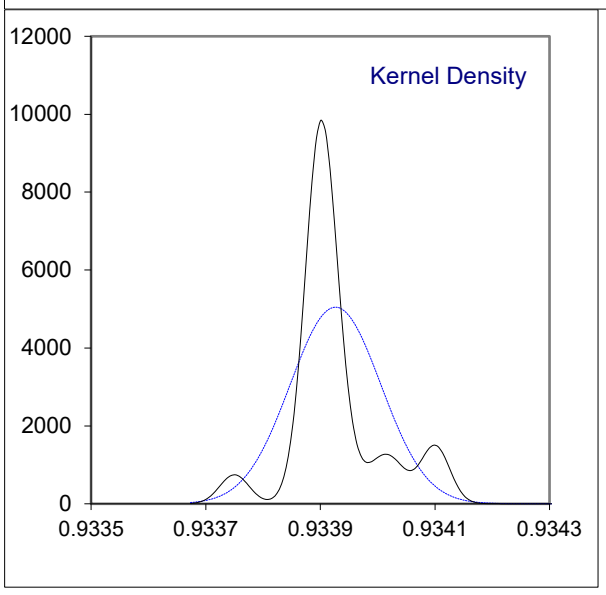


Determination of Apparent Specific Gravity 20/20 °C on sample #23001;

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9339		-0.15	
173	D4052	0.93375		-0.99	
273	D4052	0.9339		-0.15	
323	D4052	0.9339		-0.15	
347	D4052	0.9339	C	-0.15	first reported 0.9335
357	ISO12185	0.93393		0.02	
391	ISO12185	0.9339		-0.15	
396	D4052	0.9341		0.97	
446	D4052	0.9356	G(0.01)	9.37	
522		-----		-----	
541	D4052	0.9339		-0.15	
551	D4052	0.9340		0.41	
657	D4052	0.93403		0.58	
840	D4052	0.93391		-0.09	
859	D4052	0.9339		-0.15	
860	D4052	0.93394		0.08	
861	D4052	0.9339		-0.15	
913		-----		-----	
963	ISO12185	0.9339		-0.15	
974	D4052	0.9339		-0.15	
1079	ISO12185	0.93387		-0.32	
1429	ISO12185	0.9341		0.97	
6262	D4052	0.9339		-0.15	

normality	suspect
n	20
outliers	1
mean (n)	0.93393
st.dev. (n)	0.000079
R(calc.)	0.00022
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

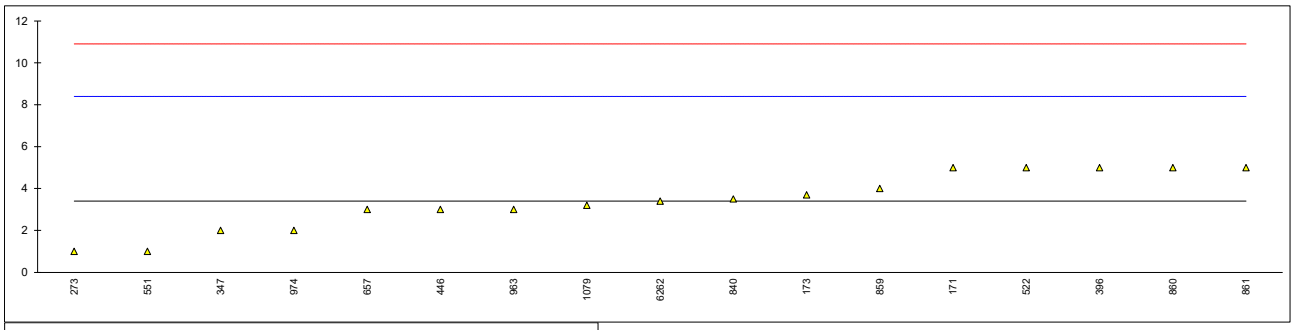


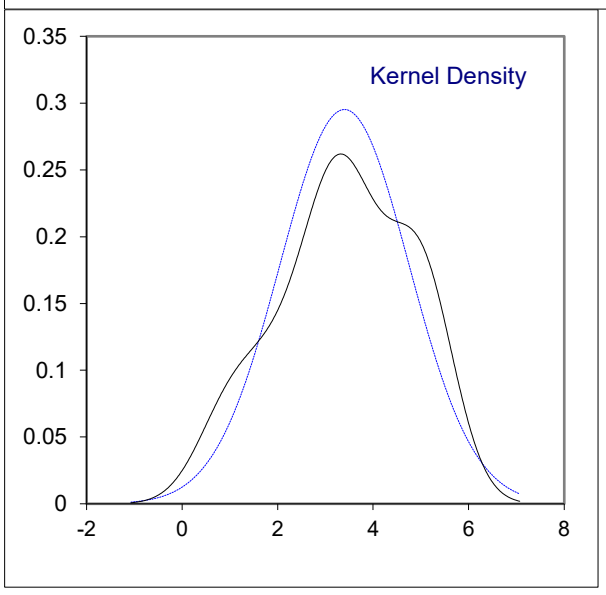


Determination of Color Pt/Co on sample #23001;

lab	method	value	mark	z(targ)	remarks
171	D1209	5		0.64	
173	D5386	3.70		0.12	
273	D1209	1		-0.96	
323	D1209	<5		-----	
347	D5386	2		-0.56	
357	D1209	< 5		-----	
391	D1209	<5		-----	
396	D5386	5		0.64	
446	D5386	3		-0.16	
522	D1209	5		0.64	
541		-----		-----	
551	D1209	1		-0.96	
657	D1209	3		-0.16	
840	D5386	3.5		0.04	
859	D1209	4		0.24	
860	D1209	5		0.64	
861	D1209	5		0.64	
913		-----		-----	
963	D1209	3		-0.16	
974	D1209	2		-0.56	
1079	D5386	3.2		-0.08	
1429	D1209	<5		-----	
6262	D1209	3.4		0.00	

normality	OK
n	17
outliers	0
mean (n)	3.40
st.dev. (n)	1.351
R(calc.)	3.78
st.dev.(D1209:05R19)	2.500
R(D1209:05R19)	7

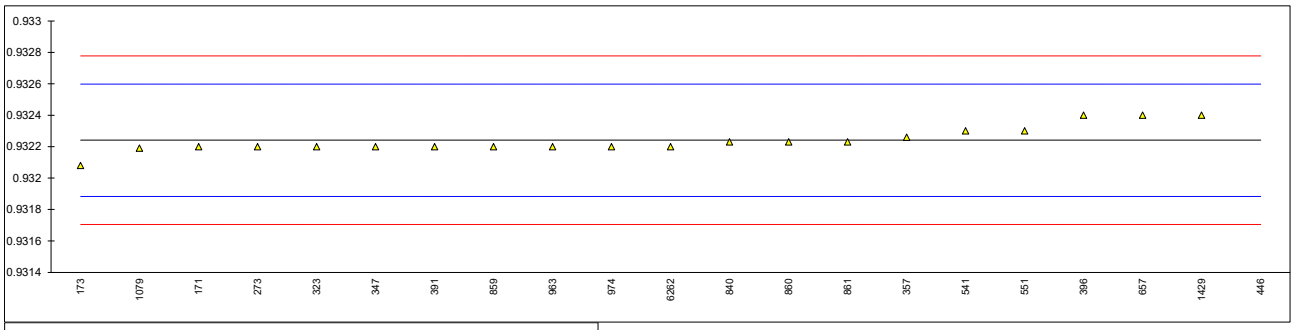


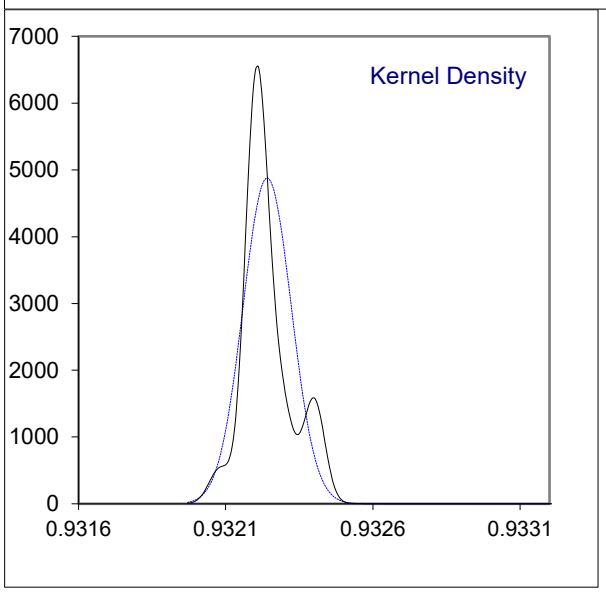


Determination of Density at 20 °C on sample #23001; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9322		-0.23	
173	D4052	0.93208		-0.90	
273	D4052	0.9322		-0.23	
323	D4052	0.9322		-0.23	
347	D4052	0.9322		-0.23	
357	ISO12185	0.93226		0.11	
391	ISO12185	0.9322		-0.23	
396	D4052	0.9324		0.89	
446	D4052	0.9339	G(0.01)	9.29	
522		-----		-----	
541	D4052	0.9323		0.33	
551	D4052	0.9323		0.33	
657	D4052	0.9324		0.89	
840	D4052	0.93223		-0.06	
859	D4052	0.9322		-0.23	
860	D4052	0.93223		-0.06	
861	D4052	0.93223		-0.06	
913		-----		-----	
963	ISO12185	0.9322		-0.23	
974	D4052	0.9322		-0.23	
1079	ISO12185	0.93219		-0.29	
1429	ISO12185	0.9324		0.89	
6262	D4052	0.9322		-0.23	

normality	OK
n	20
outliers	1
mean (n)	0.93224
st.dev. (n)	0.000082
R(calc.)	0.00023
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

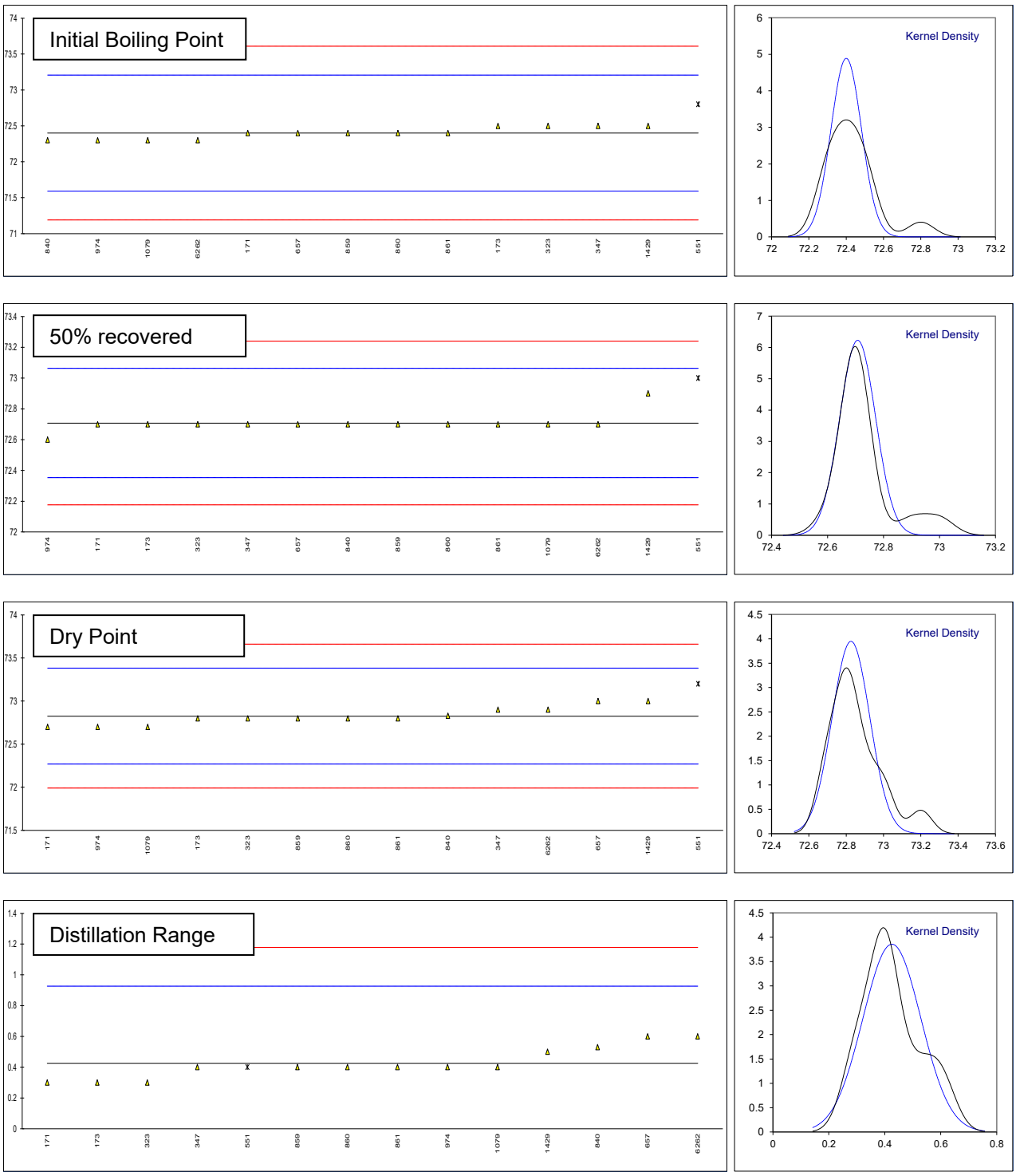




Determination of Distillation on sample #23001; results in °C

lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	DP	mark	z(targ)	range	mark	z(targ)
171	D1078-A	72.4		0.00	72.7		-0.04	72.7		-0.45	0.3		-0.50
173	D1078-A	72.5		0.25	72.7		-0.04	72.8		-0.09	0.3		-0.50
273		-----		-----	-----		-----	-----		-----	-----		-----
323	D1078-A	72.5		0.25	72.7		-0.04	72.8		-0.09	0.3		-0.50
347	D1078-A	72.5		0.25	72.7		-0.04	72.9		0.27	0.4		-0.10
357		-----		-----	-----		-----	-----		-----	-----		-----
391		-----		-----	-----		-----	-----		-----	-----		-----
396		-----		-----	-----		-----	-----		-----	-----		-----
446		-----		-----	-----		-----	-----		-----	-----		-----
522		-----		-----	-----		-----	-----		-----	-----		-----
541		-----		-----	-----		-----	-----		-----	-----		-----
551	D1078-A	72.8	G(1)	0.99	73.0	G(1)	1.65	73.2	ex	1.35	0.4	ex	-0.10
657	D1078-A	72.4		0.00	72.7		-0.04	73.0		0.63	0.6		0.70
840	D1078-A	72.30		-0.25	72.70		-0.04	72.83		0.02	0.53		0.42
859	D1078-M	72.4		0.00	72.7		-0.04	72.8		-0.09	0.4		-0.10
860	D1078-M	72.4		0.00	72.7		-0.04	72.8		-0.09	0.4		-0.10
861	D1078	72.4		0.00	72.7		-0.04	72.8		-0.09	0.4		-0.10
913		-----		-----	-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----	-----		-----
974	D1078-A	72.3		-0.25	72.6		-0.61	72.7		-0.45	0.4		-0.10
1079	D1078-A	72.3		-0.25	72.7		-0.04	72.7		-0.45	0.4		-0.10
1429	D1078-A	72.5		0.25	72.9		1.08	73.0		0.63	0.5		0.30
6262	D1078-A	72.3		-0.25	72.7		-0.04	72.9		0.27	0.6		0.70
normality		OK			not OK			OK			OK		
n		13			13			13			13		
outliers		1			1			0 +1ex			0 +1ex		
mean (n)		72.40			72.71			72.83			0.43		
st.dev. (n)		0.082			0.064			0.101			0.103		
R(calc.)		0.23			0.18			0.28			0.29		
st.dev.(D1078-A:11R19)		0.403			0.177			0.278			0.251		
R(D1078-A:11R19)		1.13			0.50			0.78			0.70		
Compare													
R(D1078-M:11R19)		0.78			0.47			0.95			0.64		

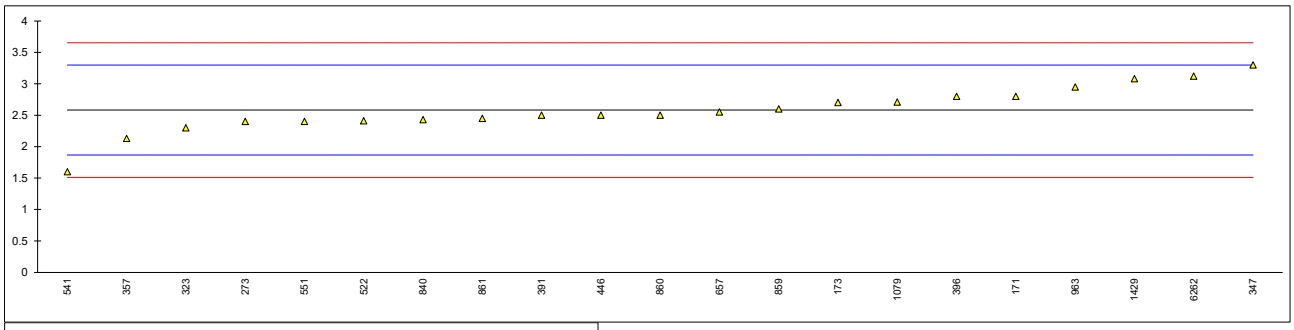
Lab 551 excluded for Dry Point and Distillation Range as statistical outliers in corresponding distillation parameters

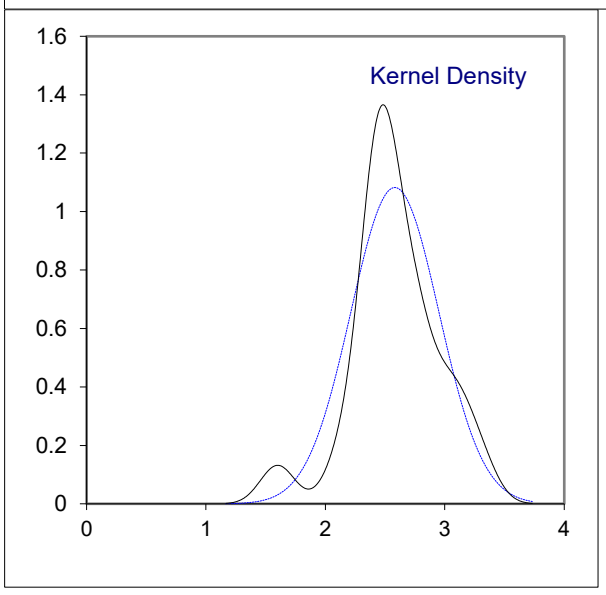

Initial Boiling Point
Kernel Density
50% recovered
Kernel Density
Dry Point
Kernel Density
Distillation Range
Kernel Density

Determination of Inhibitor as Hydroquinone on sample #23001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2193	2.8		0.61	
173	D2193	2.70		0.33	
273	INH-40	2.4		-0.51	
323	D2193	2.3		-0.79	
347	INH-097	3.3		2.01	
357	D2193	2.13		-1.27	
391	D2193	2.5		-0.23	
396	D2193	2.8		0.61	
446	D2193	2.5		-0.23	
522	INH-40	2.41		-0.48	
541	D2193	1.6		-2.75	
551	D2193	2.4	C	-0.51	first reported 1.6
657	D2193	2.55		-0.09	
840	D2193	2.43	C	-0.43	first reported 5.43
859	D2193	2.6		0.05	
860	D2193	2.5		-0.23	
861	D2193	2.45		-0.37	
913		-----		-----	
963	INH-8309	2.95		1.03	
974		-----		-----	
1079	In house	2.71		0.36	
1429	D2193	3.08		1.39	
6262	D2193	3.12		1.51	

normality	suspect
n	21
outliers	0
mean (n)	2.58
st.dev. (n)	0.369
R(calc.)	1.03
st.dev.(D2193:22)	0.357
R(D2193:22)	1.0

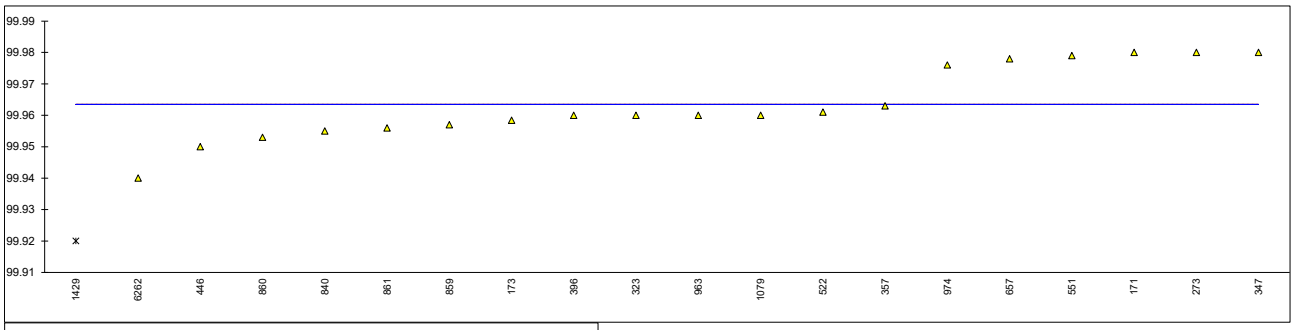


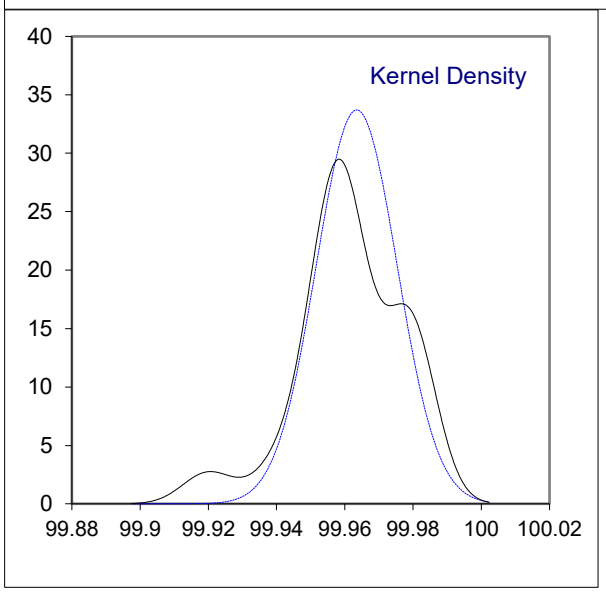


Determination of Purity by GC on sample #23001; results in %M/M

lab	method	value	mark	z(targ)	remarks
171	INH-01-12	99.98		-----	
173	INH-47	99.9584		-----	
273	INH-12A	99.98		-----	
323	INH-067	99.96		-----	
347	INH-096	99.98		-----	
357	INH-052	99.963		-----	
391		-----		-----	
396	D3545	99.96		-----	
446	INH-102582	99.95		-----	
522	INH-102582	99.961		-----	
541		-----		-----	
551	INH-1355	99.979		-----	
657	INH-0047	99.9780		-----	
840	D3545	99.955		-----	
859	SH/T1628.2	99.957		-----	
860	SH/T1628.2	99.953		-----	
861	SH/T1628.2	99.956		-----	
913		-----		-----	
963	INH-8124	99.96		-----	
974	INH-40	99.976		-----	
1079	In house	99.96		-----	
1429		99.92	G(0.05)	-----	
6262		99.94		-----	

	normality	OK
	n	19
	outliers	1
	mean (n)	99.9635
	st.dev. (n)	0.01183
	R(calc.)	0.0331
	st.dev.(lit)	n.a.
	R(lit)	n.a.
Compare		
	R(iis22C01)	0.0130

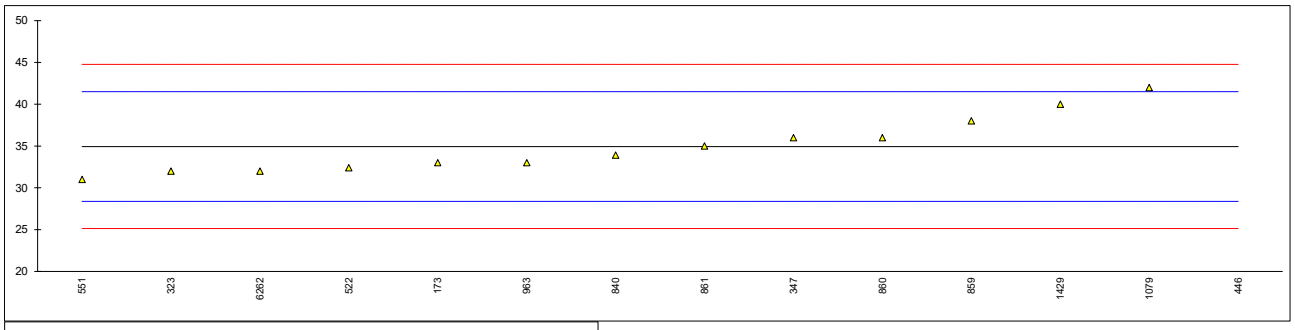


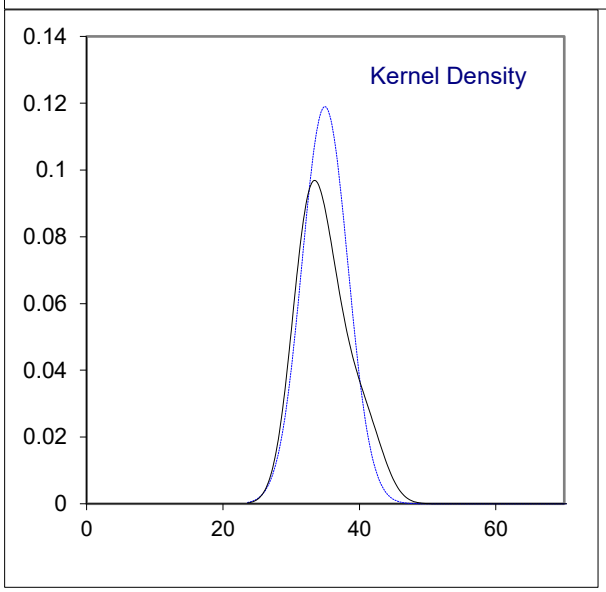


Determination of Acetaldehyde on sample #23001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01-12	<100		-----	
173	INH-47	33		-0.59	
273		-----		-----	
323	INH-067	32		-0.90	
347	INH-096	36		0.32	
357		-----		-----	
391		-----		-----	
396		-----		-----	
446	INH-102582	98	G(0.01)	19.25	
522	INH-102582	32.4		-0.78	
541		-----		-----	
551	INH-1355	31		-1.20	
657		-----		-----	
840	D3545	33.9		-0.32	
859	SH/T1628.2	38		0.93	
860	SH/T1628.2	36		0.32	
861	SH/T1628.2	35		0.02	
913		-----		-----	
963	INH-8124	33		-0.59	
974		-----		-----	
1079	In house	42		2.15	
1429		40	C	1.54	first reported 46
6262		32		-0.90	

normality	OK
n	13
outliers	1
mean (n)	34.95
st.dev. (n)	3.353
R(calc.)	9.39
st.dev.(Horwitz)	3.275
R(Horwitz)	9.17





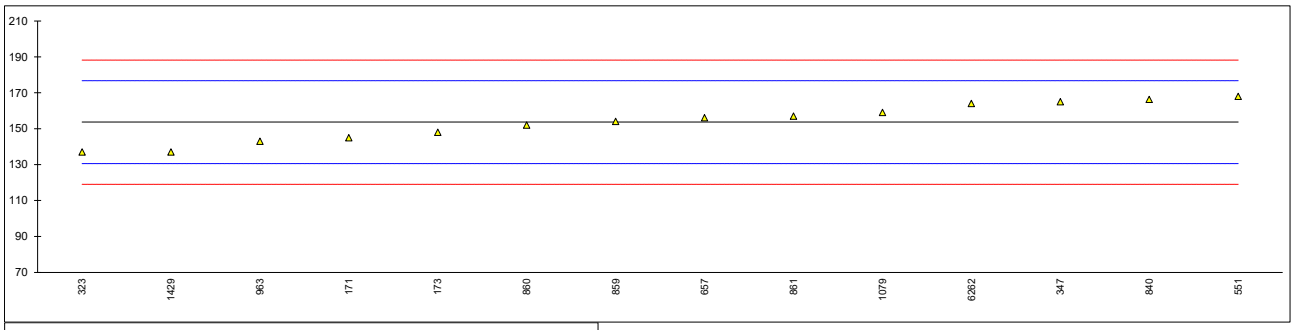
Determination of Acetone on sample #23001; results in mg/kg

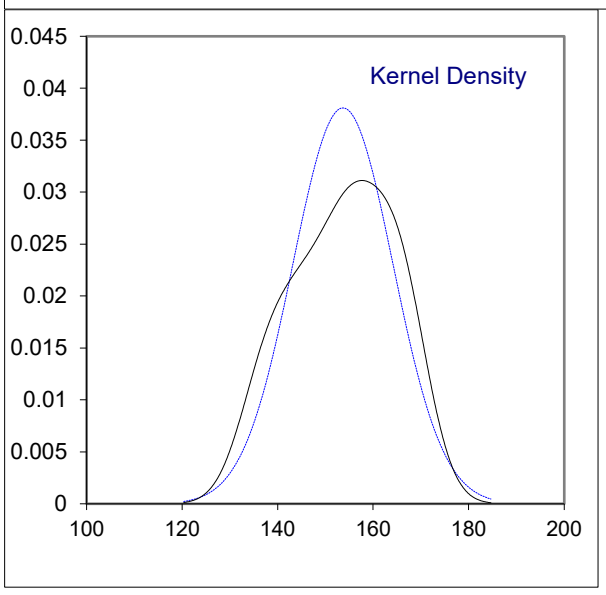
lab	method	value	mark	z(targ)	remarks
171	INH-01-12	<100		-----	
173		-----		-----	
273		-----		-----	
323	INH-067	<10		-----	
347	INH-096	<10		-----	
357		-----		-----	
391		-----		-----	
396		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551	INH-1355	<10		-----	
657	INH-0047	<10		-----	
840	D3545	<10		-----	
859	SH/T1628.2	<10		-----	
860	SH/T1628.2	<10		-----	
861	SH/T1628.2	<10		-----	
913		-----		-----	
963	INH-8124	<10		-----	
974		-----		-----	
1079	In house	3		-----	
1429		<5		-----	
6262		<10	C	-----	first reported 147
	n	12			
	mean (n)	<10			

Determination of Ethyl Acetate on sample #23001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01-12	145	C	-0.75	first reported 131
173	INH-47	148		-0.49	
273		-----		-----	
323	INH-067	137		-1.45	
347	OINH-096	165		0.98	
357		-----		-----	
391		-----		-----	
396		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551	INH-1355	168		1.24	
657	INH-0047	156.05		0.21	
840	D3545	166.3		1.10	
859	SH/T1628.2	154		0.03	
860	SH/T1628.2	152		-0.14	
861	SH/T1628.2	157		0.29	
913		-----		-----	
963	INH-8124	143		-0.93	
974		-----		-----	
1079	In house	159		0.46	
1429		137	C	-1.45	first reported 290
6262		164		0.90	

normality	OK
n	14
outliers	0
mean (n)	153.67
st.dev. (n)	10.470
R(calc.)	29.31
st.dev.(Horwitz)	11.524
R(Horwitz)	32.27

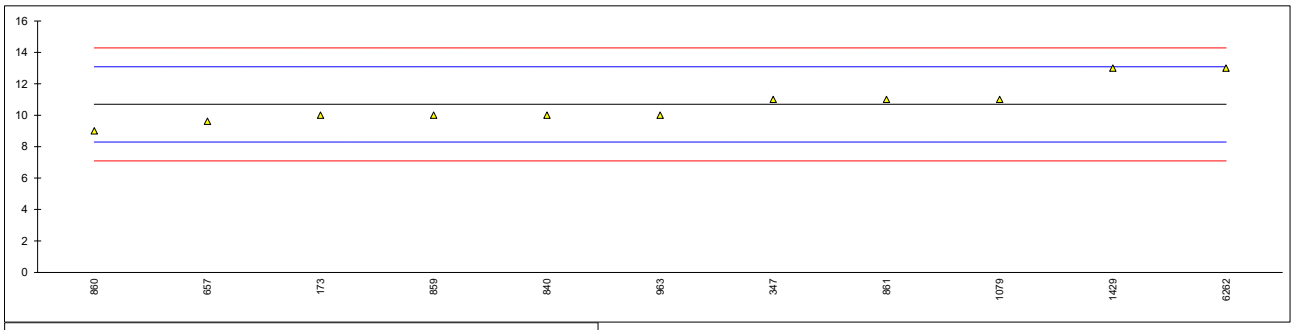


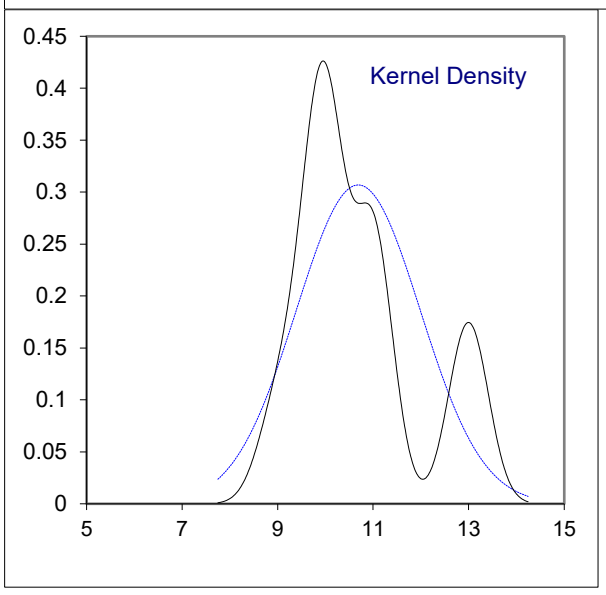


Determination of Methyl Acetate on sample #23001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-01-12	<100		-----	
173	INH-47	10		-0.58	
273		-----		-----	
323	INH-067	<10		-----	
347	INH-096	11		0.26	
357		-----		-----	
391		-----		-----	
396		-----		-----	
446		-----		-----	
522		-----		-----	
541		-----		-----	
551	INH-1355	<10		-----	
657	INH-0047	9.61		-0.90	
840	D3545	10.0		-0.58	
859	SH/T1628.2	10		-0.58	
860	SH/T1628.2	9		-1.41	
861	SH/T1628.2	11		0.26	
913		-----		-----	
963	INH-8124	10		-0.58	
974		-----		-----	
1079	In house	11		0.26	
1429		13		1.93	
6262		13		1.93	

normality	OK
n	11
outliers	0
mean (n)	10.69
st.dev. (n)	1.300
R(calc.)	3.64
st.dev.(Horwitz)	1.198
R(Horwitz)	3.35

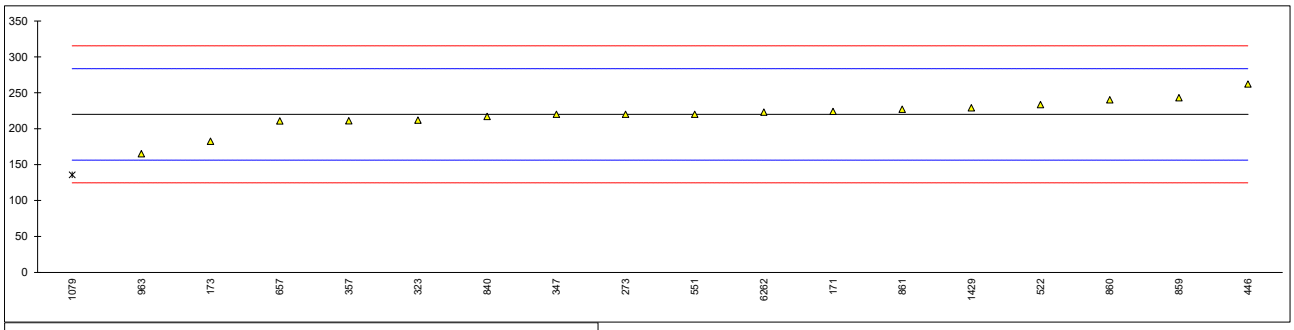


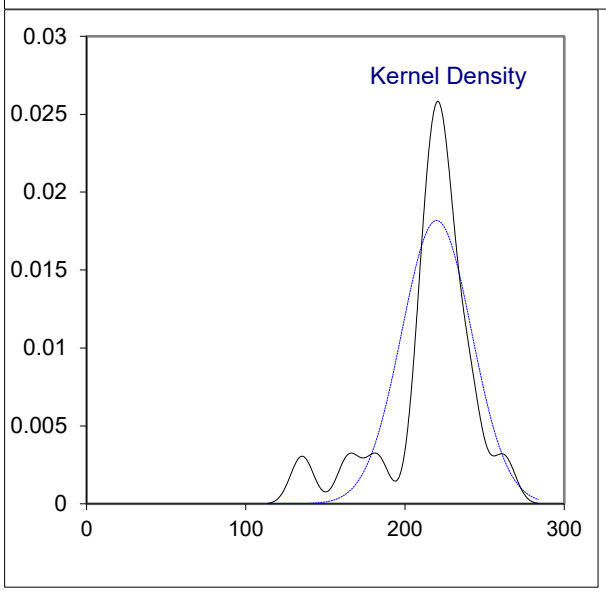


Determination of Water on sample #23001; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	224		0.13	
173	E203	182.5		-1.18	
273	E1064	220		0.00	
323	D1364	212		-0.25	
347	D1364	220		0.00	
357	E203	211		-0.28	
391		-----		-----	
396		-----		-----	
446	D1364	262		1.32	
522	E203	233.5		0.43	
541		-----		-----	
551	D1364	220		0.00	
657	E1064	210.6		-0.30	
840	D1364	217		-0.09	
859	D1364	243		0.72	
860	D1364	240		0.63	
861	D1364	227		0.22	
913		-----		-----	
963	D1364	165		-1.73	
974		-----		-----	
1079	E203	135.5	G(0.05)	-2.66	
1429	D1364	229		0.28	
6262	D1364	223		0.10	

normality	not OK
n	17
outliers	1
mean (n)	219.98
st.dev. (n)	21.943
R(calc.)	61.44
st.dev.(D1364:22)	31.782
R(D1364:22)	88.99





APPENDIX 2

Number of participants per country

1 lab in	ARGENTINA
3 labs in	BELGIUM
1 lab in	BRAZIL
3 labs in	CHINA, People's Republic
1 lab in	FINLAND
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	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)/D1	= outlier in Dixon's outlier test
D(0.05)/D5	= straggler in Dixon's outlier test
G(0.01)/G1	= outlier in Grubbs' outlier test
G(0.05)/G5	= straggler in Grubbs' outlier test
DG(0.01)/DG1	= outlier in Double Grubbs' outlier test
DG(0.05)/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

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12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)