Results of Proficiency Test
Vinyl Acetate Monomer
February 2016

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 2007, a proficiency test for Vinyl Acetate Monomer (VAM) is organised every year by the Institute for Interlaboratory Studies. During the planning of the annual proficiency testing program 2015/2016, it was decided to continue the round robin for the analysis of Vinyl Acetate Monomer. The proficiency test on Vinyl Acetate Monomer has been organised in accordance with the latest applicable version of the ASTM D2190 specification and a number of additional tests requested by some participants
In this interlaboratory study, 26 laboratories in 19 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2016 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Analysis for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. It was decided to send one sample (1* 0.5 L of Vinyl Acetate Monomer, labelled #16010). Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Also customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation 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 April 2014 (iis-protocol, version 3.3).
The protocol can be downloaded from 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

The necessary bulk material for sample #16010 was obtained from a local trader. The approximately 25 litre bulk sample was homogenised in a pre-cleaned drum and 50 amber glass bottles of 0.5L were filled and labelled #16010. The homogeneity of these subsamples was checked by determination of Density in accordance with ASTM D4052 and Water in accordance with ASTM D1364 on 7 stratified randomly selected samples.

	Density at 20°C in kg/L	*Water in mg/kg*
sample #16010-1	0.93215	65
sample #16010-2	0.93214	47
sample #16010-3	0.93214	52
sample #16010-4	0.93214	50
sample #16010-5	0.93215	47
sample #16010-6	0.93215	50
sample #16010-7	0.93215	56

Table 1: homogeneity test results of subsamples #16010

From the test results of table 1, the repeatabilities were calculated and compared with the corresponding repeatability or with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 20°C in kg/L	*Water in mg/kg*
r (observed)	0.00001	18
reference test method	ISO12185:96	ASTM D1364:02(2012)
0.3*R (reference test method)	0.00015	--
r (reference test method)	--	22

Table 2: repeatabilities of subsamples #16010

The calculated repeatabilities for density and water are in agreement with resp. 0.3 times the corresponding reproducibility and repeatability of the reference test method. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 1 bottle of 0.5 L Vinyl Acetate Monomer, labelled #16010, was sent on January 27, 2016.

2.5 STABILITY OF THE SAMPLES

The stability of Vinyl Acetate Monomer, packed in the brown glass bottles was checked. The material has been found stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine according to the standard specification for Vinyl Acetate Monomer (ASTM D2190:07(2013)): Acetaldehyde, Acidity, Apparent Specific Gravity 20/20°C, Distillation (IBP, 50% recovery, Dry Point, Boiling Range), Inhibitor as Hydroquinone and Water, plus additionally Acidity with N_2 purging, Acetone, Colour Pt/Co, Density at 20°C, Ethyl Acetate, Methyl Acetate and Purity.
To get comparable results, a detailed report form on which the units were prescribed as well as the preferred test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. A SDS and a form to confirm receipt of the samples were added to the package.

3 RESULTS

During five weeks after sample despatch, the 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 represented by the code numbers.
Directly after the deadline, a reminder was sent to those laboratories that did not report results at that moment.
Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.
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 organisation of this proficiency test is described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 the original results per determination were submitted subsequently to Dixon, Grubbs and/or Rosner General ESD outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner General ESD test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.
Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

3.2 GRAPHICS

In order to visualise 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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. 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, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study.

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility of former iis proficiency tests could be used.

The z-scores were calculated in accordance with:

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

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

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 results are fit-for-use.

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

In this proficiency test, some problems were encountered with dispatch of the samples. Participants in Brazil received the samples late. Of the 26 participants, one participant reported the test results after the final reporting date and one other participant did not report any test result at all.

Not all participants were able to report test results for all the requested tests. Finally, 25 participants reported in total 275 numerical results. Observed were 9 outlying test results, which is 3.3% of the total of numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the reported results are discussed per test.
Unfortunately, a suitable standard test method, providing the precision data, is not available for all determinations. For the tests, that have no available precision data, the spreads were compared against the spreads estimated from the Horwitz equation.
In the iis PT reports, ASTM methods are referred to with a number (e.g. D2086) and an added designation for the year that the method was adopted or revised (e.g. D2086:08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086:08(2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

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.

Acidity: This determination (with and without Nitrogen purging) was problematic. The acidity value determined with Nitrogen purging was not different to the acidity determination without Nitrogen purging.
In total two statistical outliers were observed. Both calculated reproducibilities after rejection of the statistical outliers are not in agreement with the requirements of ASTM D2086:08(2012).
Please note that the reproducibility of ASTM D2086:08(2012) was determined with only two laboratories (see note 5 in §13.2.2 of ASTM D2086:08(2012)). When compared to the spread found in the previous PT (iis15C01), the observed reproducibility did improve only for Acidity purged with nitrogen.

Apparent Specific Gravity 20/20°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D4052:02e1.

Colour Pt/Co: The determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1209:05(2012).

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

Distillation: This determination was not problematic. No statistical outliers were observed. The calculated reproducibilities are all in good agreement with the requirements of ASTM D1078:11 (Automated method).

Inhibitor (Hydroquinone): This determination was problematic. One statistical outlier was observed The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D2193:06(2012).

Purity: Regretfully, no reference method with precision data exists for this determination. No statistical outliers were observed. When compared to the spread found in the previous PT (iis15C01), the reproducibility value has slightly improved.

Acetaldehyde: The determination of this impurity may be problematic for a number of laboratories. Two statistical outliers were observed. However the reproducibility after rejection of the statistical outliers is in agreement with the requirements estimated from the Horwitz equation.

Acetone: All participants agreed on a result less than 10 mg/kg, which was near or below the detection limit of the test methods used, therefore no significant conclusions were drawn.

Ethyl Acetate: The determination of this impurity may not be problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements estimated from the Horwitz equation.

Methyl Acetate: The determination of this impurity may not be problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements estimated from the Horwitz equation.

Water: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good 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 standard and the reproducibility as found for the group of participating laboratories. The assigned values, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM, ISO standards) are compared in the next table.

Parameter	unit	n	Mean	2.8 * sd	R (lit)
Acidity (without N_2 purging)	mg/kg	14	22.4	13.7	6.0
Acidity (with N_2 purging)	mg/kg	8	22.0	10.0	6.0
Apparent Specific Gravity 20/20°C		21	0.9339	0.0003	0.0005
Colour Pt/Co		16	3.7	3.0	7.0
Density at 20°C	kg/L	24	0.9322	0.0003	0.0005
Distillation, Initial Boiling Point	ºC	18	72.5	0.2	1.1
Distillation, 50% recovery	ºC	18	72.7	0.2	0.5
Distillation, Dry Point	ºC	18	72.8	0.2	0.8
Distillation, Boiling Range	ºC	18	0.4	0.3	0.7
Inhibitor as Hydroquinone	mg/kg	20	3.2	1.2	1.0
Purity	%M/M	19	99.978	0.011	(0.013)
Acetaldehyde	mg/kg	16	32.0	8.7	8.5
Acetone	mg/kg	15	<10	n.a.	n.a.
Ethyl Acetate	mg/kg	17	135.5	26.9	29.0
Methyl Acetate	mg/kg	9	4.5	1.3	1.6
Water	mg/kg	23	57.0	26.3	45.3

Table 3: performance evaluation sample #16010

between brackets is compared against the reproducibility of the previous round

4.3 EVALUATION OF THE PROFICIENCY TEST OF FEBRUARY 2016 WITH PREVIOUS PTS

	February 2016	*February 2015*	*February 2014*	*February 2013*
Number of rep. participants	25	20	23	24
Number of results reported	275	253	240	243
Number of statistical outliers	9	2	9	9
Percentage outliers	3.3%	0.8%	3.8%	3.7%

Table 4: evaluation 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 standards. The conclusions are given the following table:

	February 2016	*February 2015*	*February 2014*	*February 2013*
Acidity (no purging)	--	--	-	--
Acidity (with purging)	--	--	-	--
Apparent Spec.Gravity	++	++	n.e.	n.e.
Colour Pt/Co	++	+	n.e.	n.e.
Density at 20^{o}C	++	++	++	++
Distillation	++	++	++	++
Inhibitor as Hydroquinone	-	-	-	-
Purity	(+)	(+)	n.e.	n.e.
Acetaldehyde	+/-	-	++	++
Acetone	n.e.	-	n.e.	n.e.
Ethyl Acetate	+/-	-	++	+/-
Methyl Acetate	+	-	-	-
Water	++	+	+/-	+/-

Table 5: comparison determinations against the reference standards
between brackets is compared against the reproducibility of the previous round

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

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

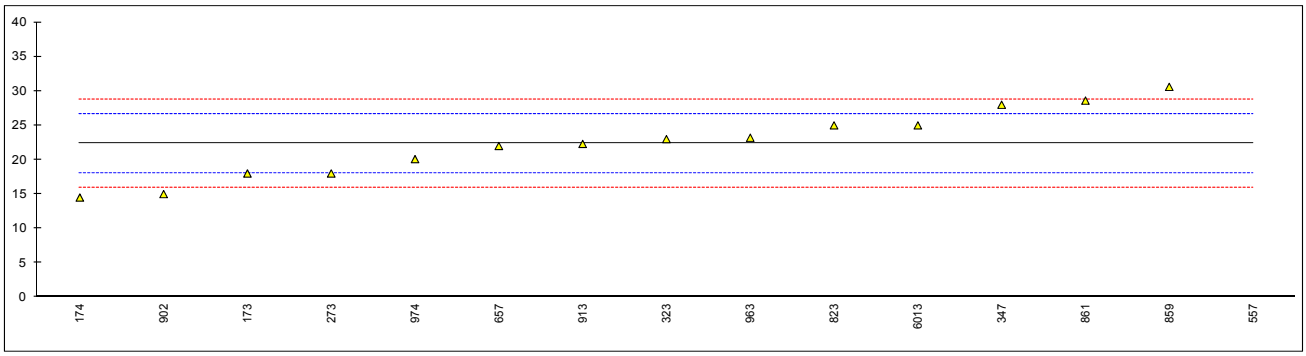
APPENDIX 1

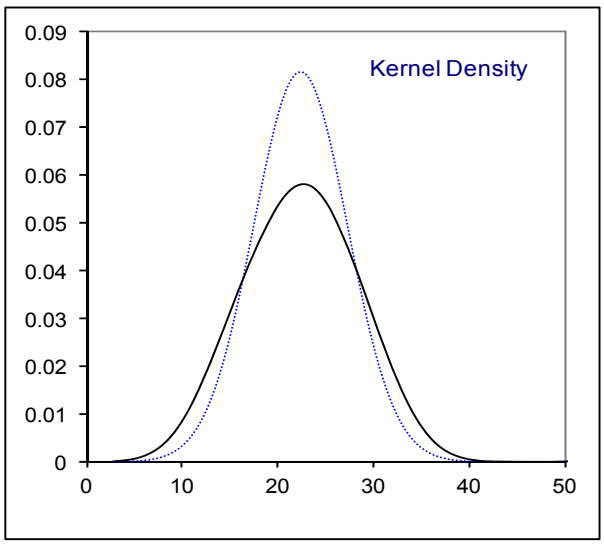
Determination of Acidity (without N_2 purging) on sample #16010; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173	INH-44	18		-2.04	
174	D2086	14.5		-3.68	
273	D1613	18		-2.04	
311		-----		-----	
323	D2086	23		0.29	
337		-----		-----	
347	D2086	28		2.62	
391		-----		-----	
395		-----		-----	
522		-----		-----	
551		-----		-----	
557	D2086	61.842387	G(0.01)	18.42	
657	D2086	22		-0.18	
663		-----		-----	
823	D2086	25	C	1.22	First reported 37
859	D2086	30.6		3.84	
861	D2086	28.6		2.90	
902	D2086	15		-3.44	
913	D2086	22.3		-0.04	
963	D2086	23.2		0.38	
974	D2086	20.09		-1.07	
1107		-----		-----	
1429		-----		-----	
6013	D2086	25	C	1.22	Reported 0.0025mg/kg, probably unit error
7006		-----		-----	

normality	OK
n	14
outliers	1
mean (n)	22.38
st.dev. (n)	4.906
R(calc.)	13.74
R(D2086:08)	6.00

Compare R(iis15C01) = 13.00



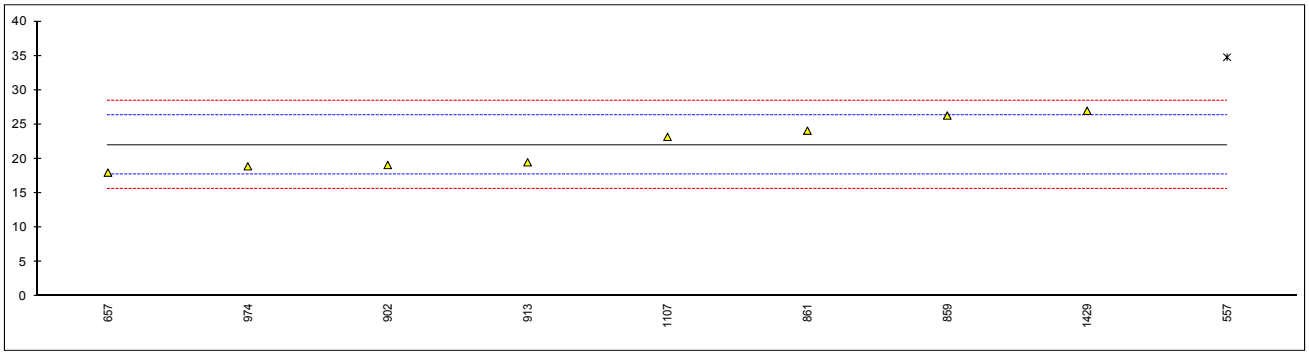


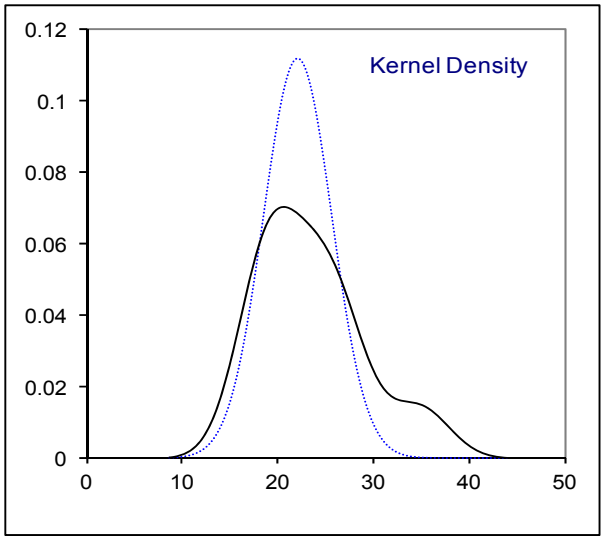
Determination of Acidity (with N_2 purging) on sample #16010; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173		-----		-----	
174		-----		-----	
273		-----		-----	
311		-----		-----	
323		-----		-----	
337		-----		-----	
347		-----		-----	
391		-----		-----	
395		-----		-----	
522		-----		-----	
551		-----		-----	
557	D2086	34.786345	G(0.05)	5.96	
657	D2086	18		-1.87	
663		-----		-----	
823		-----		-----	
859	D2086	26.3		2.00	
861	D2086	24.1		0.97	
902	D2086	19.1	C	-1.36	First reported 11.1
913	D2086	19.5		-1.17	
963		-----		-----	
974	D2086	18.91		-1.45	
1107	D2086	23.2		0.55	
1429	D2086	27		2.33	
6013		-----		-----	
7006		-----		-----	

	normality	OK
	n	8
	outliers	1
	mean (n)	22.01
	st.dev. (n)	3.577
	R(calc.)	10.01
	R(D2086:08)	6.00

Compare R(iis15C01) = 13.20

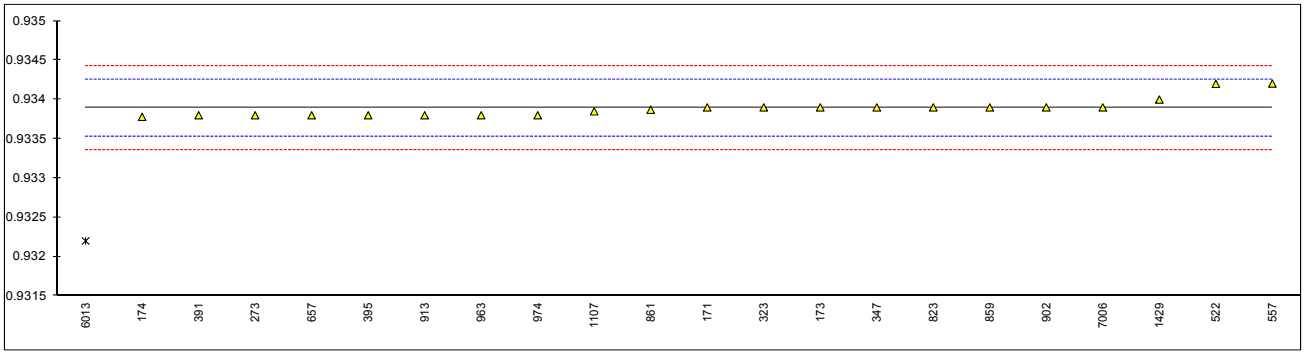


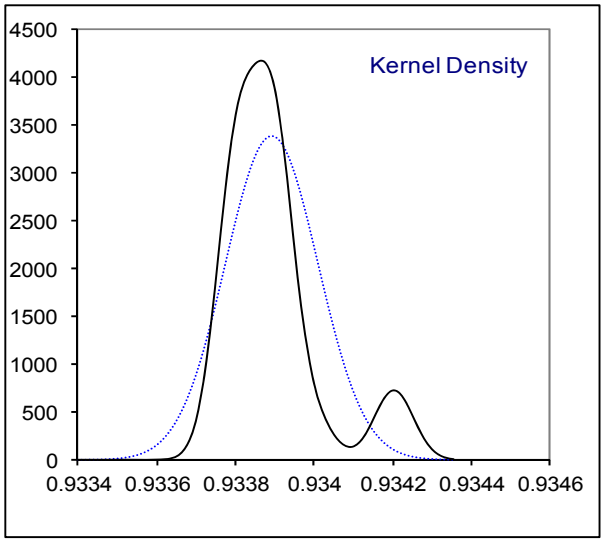


Determination of App. Specific Gravity 20/20 °C on sample #16010;

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9339		0.05	
173	D4052	0.9339		0.05	
174	D4052	0.93378		-0.62	
273	D4052	0.9338		-0.51	
311		-----		-----	
323	D4052	0.9339		0.05	
337		-----		-----	
347	D4052	0.9339		0.05	
391	D4052	0.9338		-0.51	
395	D4052	0.9338		-0.51	
522	D4052	0.9342		1.73	
551		-----		-----	
557	D4052	0.93420375		1.75	
657	D4052	0.93380		-0.51	
663		-----		-----	
823	D4052	0.93390		0.05	
859	D4052	0.9339		0.05	
861	D4052	0.93387		-0.12	
902	D4052	0.9339		0.05	
913	D4052	0.9338		-0.51	
963	D4052	0.9338		-0.51	
974	D4052	0.9338		-0.51	
1107	D4052	0.93385		-0.23	
1429	D4052	0.9340		0.61	
6013	D4052	0.9322	R(0.01)	-9.47	
7006	D4052	0.9339		0.05	

normality	not OK
n	21
outliers	1
mean (n)	0.93389
st.dev. (n)	0.000118
R(calc.)	0.00033
R(D4052:02e1)	0.00050

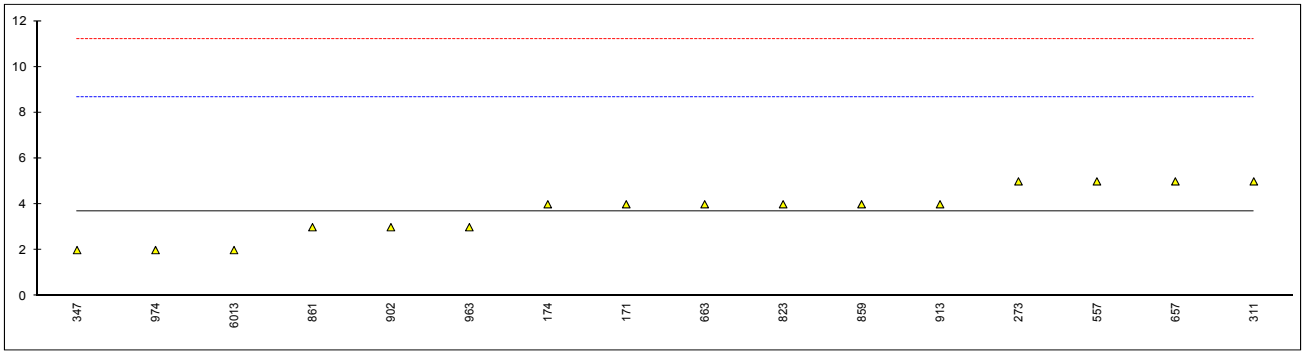


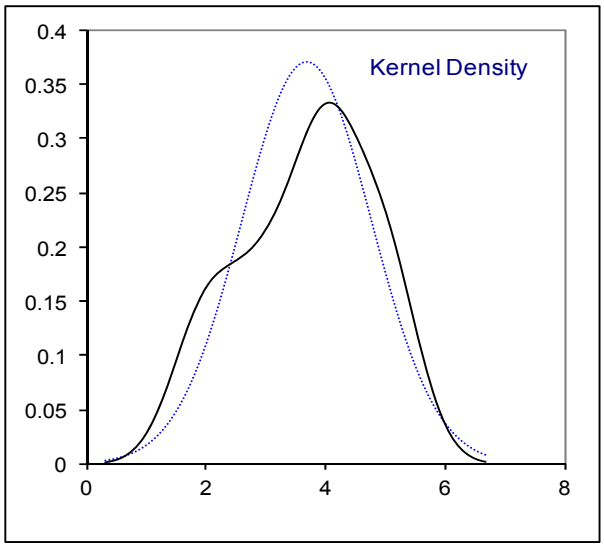


Determination of Colour Pt/Co on sample #16010;

lab	method	value	mark	z(targ)	remarks
171	D5386	4		0.13	
173	INH-46	<5		-----	
174	D1209	4		0.13	
273	D1209	5		0.53	
311	D1209	5		0.53	
323	D1209	<5		-----	
337		-----		-----	
347	D5386	2		-0.68	
391	D1209	<5		-----	
395	D1209	<5		-----	
522		-----		-----	
551		-----		-----	
557	D1209	5		0.53	
657	D1209	5		0.53	
663	D1209	4		0.13	
823	D1209	4		0.13	
859	D1209	4		0.13	
861	D1209	3		-0.28	
902	D5386	3		-0.28	
913	D5386	4		0.13	
963	D1209	3		-0.28	
974	D5386	2		-0.68	
1107	D1209	<5		-----	
1429	D1209	<5		-----	
6013	D1209	2		-0.68	
7006		-----		-----	

normality	OK
n	16
outliers	0
mean (n)	3.7
st.dev. (n)	1.08
R(calc.)	3.0
R(D1209:05)	7.0

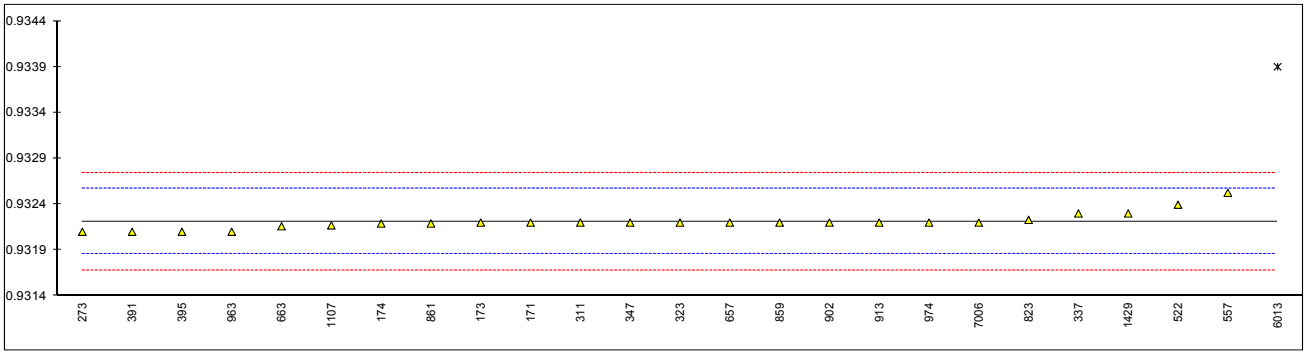


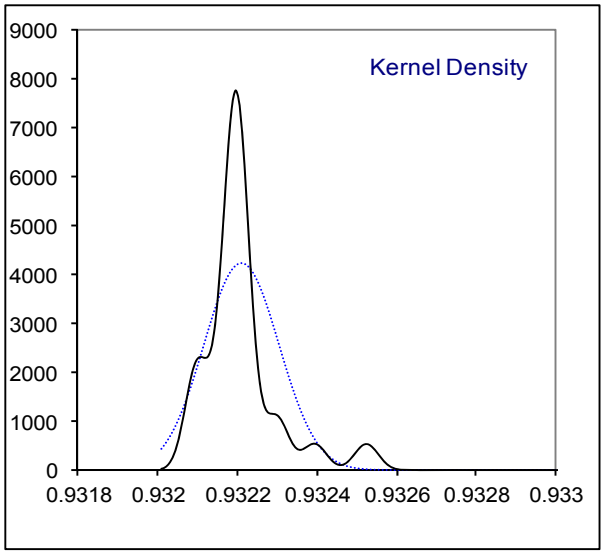


Determination of Density at 20 °C on sample #16010; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9322		-0.06	
173	D4052	0.9322		-0.06	
174	D4052	0.93219		-0.12	
273	D4052	0.9321		-0.62	
311	D4052	0.9322		-0.06	
323	D4052	0.9322		-0.06	
337	D4052	0.9323		0.50	
347	D4052	0.9322		-0.06	
391	D4052	0.9321		-0.62	
395	D4052	0.9321		-0.62	
522	D4052	0.932395		1.03	
551		-----		-----	
557	D4052	0.932525		1.76	
657	D4052	0.93220		-0.06	
663	D4052	0.93216		-0.29	
823	ISO12185	0.93223		0.11	
859	D4052	0.9322		-0.06	
861	D4052	0.93219		-0.12	
902	D4052	0.9322		-0.06	
913	D4052	0.9322		-0.06	
963	ISO12185	0.9321		-0.62	
974	D4052	0.9322		-0.06	
1107	D4052	0.93217		-0.23	
1429	D4052	0.9323		0.50	
6013	D4052	0.9339	R(0.01)	9.46	
7006	D4052	0.9322		-0.06	

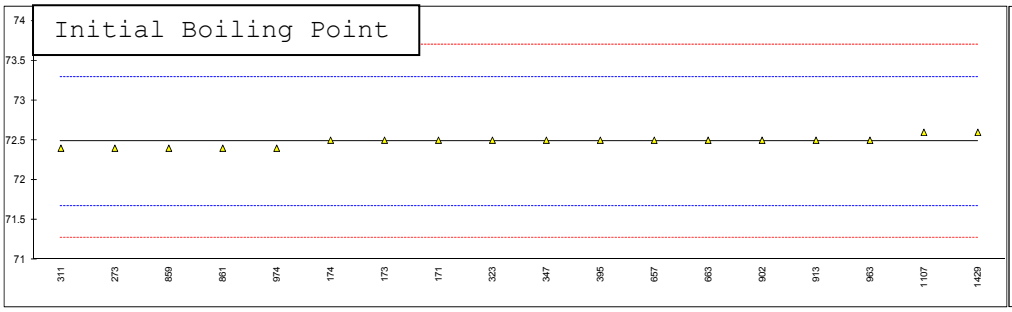
normality	not OK
n	24
outliers	1
mean (n)	0.93221
st.dev. (n)	0.000094
R(calc.)	0.00026
R(ISO12185:96)	0.00050

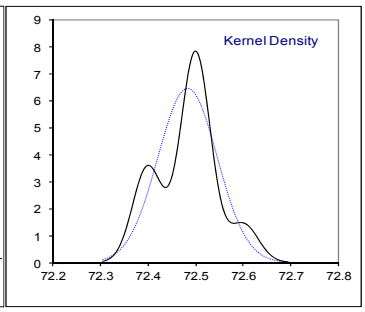


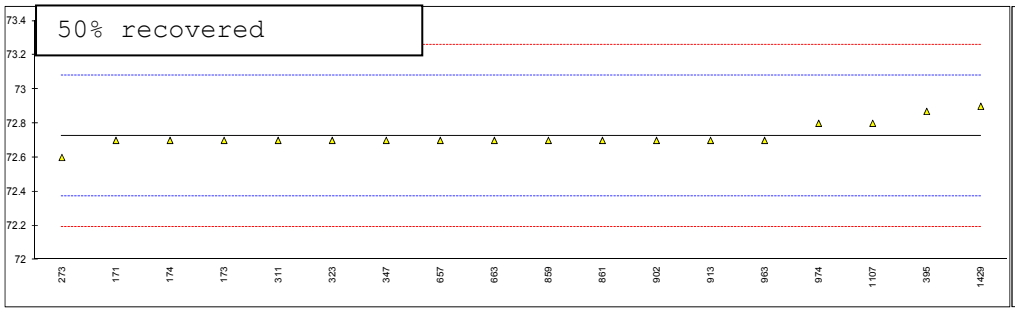


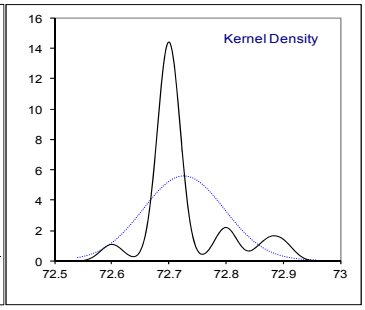
Determination of Distillation on sample #16010; results in ºC

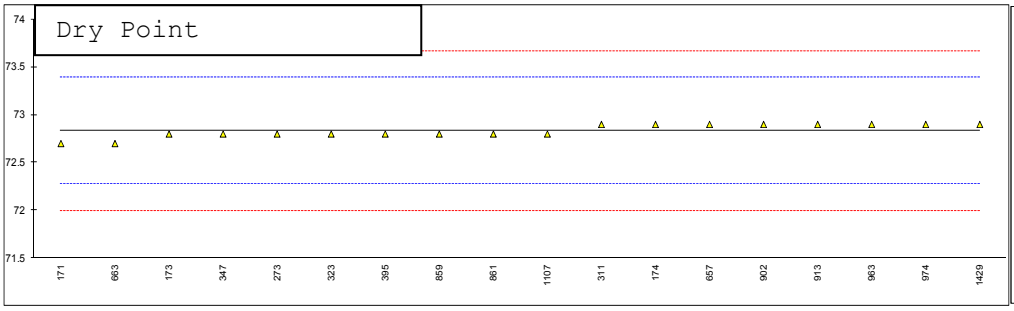
lab	method	IBP	mark	z(targ)	50% rec.	mark	z(targ)	DP	mark	z(targ)	range	mark	z(targ)
171	D1078	72.5		0.04	72.7		-0.15	72.7		-0.48	0.2		-0.63
173	D1078	72.5		0.04	72.7		-0.15	72.8		-0.12	0.3		-0.22
174	D1078	72.5		0.04	72.7		-0.15	72.9		0.24	0.4		0.18
273	D1078	72.4		-0.21	72.6		-0.71	72.8		-0.12	0.4		0.18
311	D1078	72.4		-0.21	72.7		-0.15	72.9		0.24	0.5		0.58
323	D1078	72.5		0.04	72.7		-0.15	72.8		-0.12	0.3		-0.22
337		-----		-----	-----		-----	-----		-----	-----		-----
347	D1078	72.5		0.04	72.7		-0.15	72.8		-0.12	0.3		-0.22
391		-----		-----	-----		-----	-----		-----	-----		-----
395	D1078	72.5		0.04	72.87		0.81	72.8		-0.12	0.4		0.18
522		-----		-----	-----		-----	-----		-----	-----		-----
551		-----		-----	-----		-----	-----		-----	-----		-----
557		-----		-----	-----		-----	-----		-----	-----		-----
657	D1078	72.5		0.04	72.7		-0.15	72.9		0.24	0.4		0.18
663	D1078	72.50		0.04	72.70		-0.15	72.70		-0.48	0.20		-0.63
823		-----		-----	-----		-----	-----		-----	-----		-----
859	D1078	72.4		-0.21	72.7		-0.15	72.8		-0.12	0.4		0.18
861	D1078	72.4		-0.21	72.7		-0.15	72.8		-0.12	0.4		0.18
902	D1078	72.5		0.04	72.7		-0.15	72.9		0.24	0.4		0.18
913	D1078	72.5		0.04	72.7		-0.15	72.9		0.24	0.4		0.18
963	D1078	72.5		0.04	72.7		-0.15	72.9		0.24	0.4		0.18
974	D1078	72.4		-0.21	72.8		0.42	72.9		0.24	0.5		0.58
1107	D1078	72.6		0.29	72.8		0.42	72.8		-0.12	0.2		-0.63
1429	D1078	72.6		0.29	72.9		0.98	72.9		0.24	0.3		-0.22
6013		-----		-----	-----		-----	-----		-----	-----		-----
7006		-----		-----	-----		-----	-----		-----	-----		-----
	normality	OK			not OK			OK			OK		
	n	18			18			18			18		
	outliers	0			0			0			0		
	mean (n)	72.48			72.73			72.83			0.36		
	st.dev. (n)	0.062			0.071			0.069			0.092		
	R(calc.)	0.17			0.20			0.19			0.26		
	R(D1078:11)	1.13			0.50			0.78			0.69		

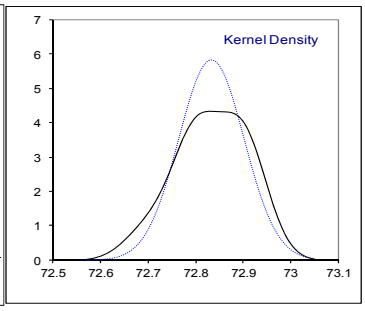








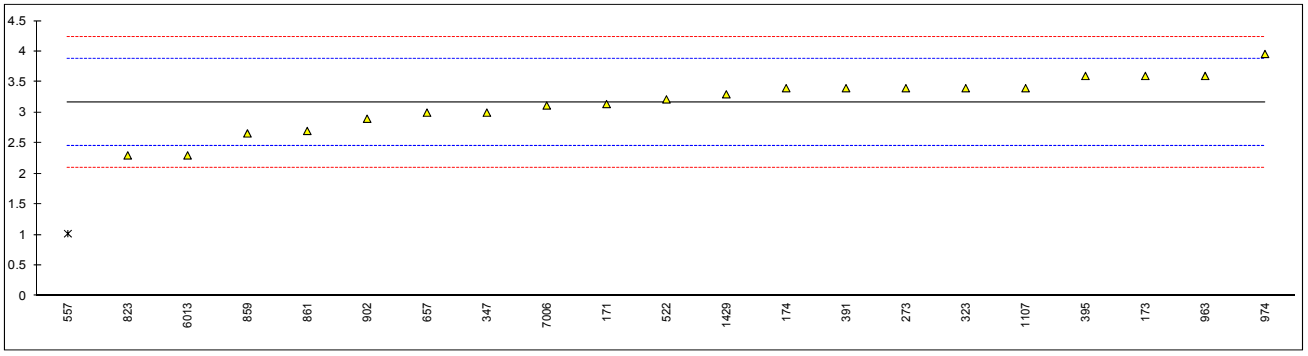


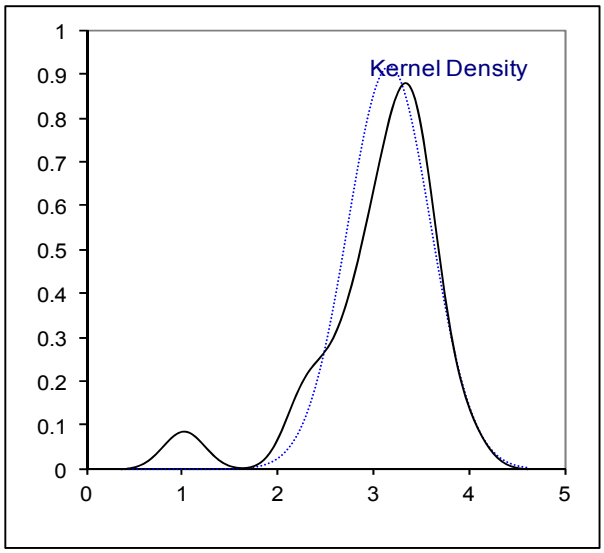


Determination of Inhibitor as Hydroquinone on sample #16010; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-43	3.14		-0.08	
173	INH-43	3.6		1.20	
174	INH-29	3.4		0.64	
273	D2193	3.4		0.64	
311		-----		-----	
323	D2193	3.4		0.64	
337		-----		-----	
347	D2193	3.0		-0.48	
391	D2193	3.4		0.64	
395	INH-43	3.6		1.20	
522	INH-40	3.2166		0.13	
551		-----		-----	
557	D2193	1.0188522	R(0.01)	-6.02	
657	D2193	3.0		-0.48	
663		-----		-----	
823	D2193	2.3		-2.44	
859	D2193	2.66		-1.43	
861	D2193	2.70		-1.32	
902	D2193	2.9		-0.76	
913		-----		-----	
963	D2193	3.6		1.20	
974	D2193	3.96		2.21	
1107	In house	3.4		0.64	
1429	D2193	3.3		0.36	
6013	D2193	2.3		-2.44	
7006	D2193	3.117		-0.15	

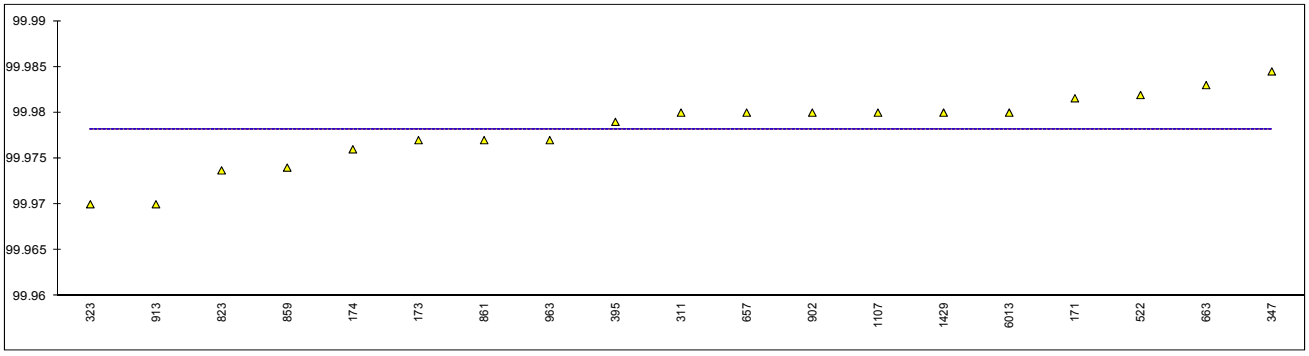
normality	OK
n	20
outliers	1
mean (n)	3.17
st.dev. (n)	0.435
R(calc.)	1.22
R(D2193:06)	1.00

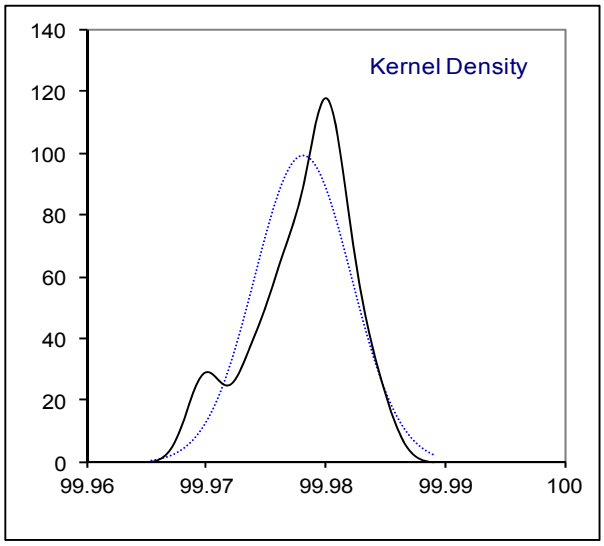




Determination of Purity on sample #16010; results in % M/M

lab	method	value	mark	z(targ)	remarks
171	INH-0001	99.981564		-----	
173	INH-257	99.977		-----	
174	INH-1B	99.976		-----	
273		-----		-----	
311	INH-122	99.98		-----	
323	INH-067	99.97		-----	
337		-----		-----	
347	INH-96	99.9845		-----	
391		-----		-----	
395	INH-257	99.979		-----	
522	INH-40	99.98192		-----	
551		-----		-----	
557		-----		-----	
657	INH-0047	99.98		-----	
663	INH-102582	99.983		-----	
823	INH-021	99.9737		-----	
859	SH/T1628.2	99.974		-----	
861	SH/T1628.2	99.977		-----	
902	INH-22	99.98		-----	
913	INH-12345	99.97		-----	
963	INH-009	99.977		-----	
974		-----		-----	
1107	In house	99.98		-----	
1429	In house	99.980		-----	
6013	In house	99.98		-----	
7006		-----		-----	
	normality	OK			
	n	19			
	outliers	0			
	mean (n)	99.9781			
	st.dev. (n)	0.00401			
	R(calc.)	0.0112			
	R(lit.)	unknown			Compare R(iis15C01) = 0.0127

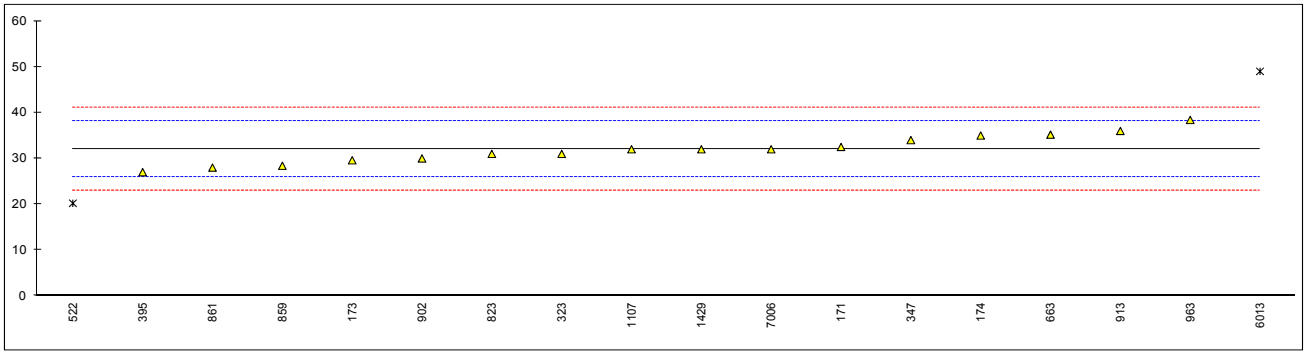


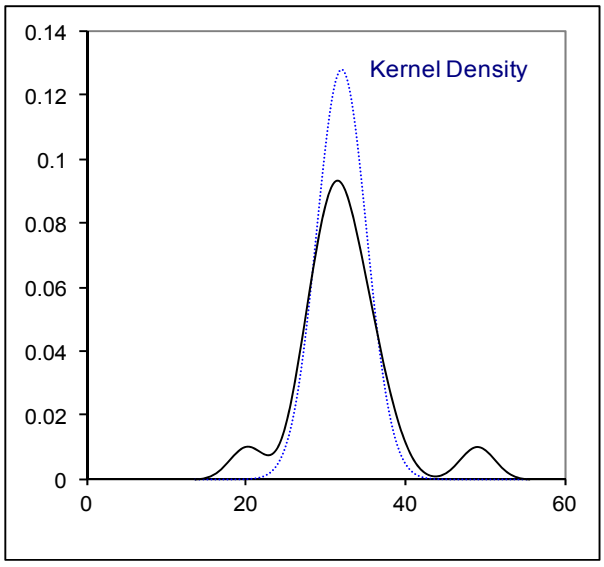


Determination of Acetaldehyde on sample #16010; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-0001	32.505		0.16	
173	INH-47	29.6		-0.79	
174	INH-1B	35		0.98	
273		-----		-----	
311		-----		-----	
323	INH-067	31		-0.33	
337		-----		-----	
347	INH-96	34		0.66	
391		-----		-----	
395	INH-257	27.0		-1.65	
522	INH-40	20.2166	G(0.05)	-3.88	
551		-----		-----	
557		-----		-----	
657		-----		-----	
663	INH-102582	35.2	C	1.05	First reported 0
823	INH-021	31		-0.33	
859	SH/T1628.2	28.4		-1.19	
861	SH/T1628.2	28		-1.32	
902	INH-22	30		-0.66	
913	INH-12345	36		1.31	
963	INH-009	38.4		2.10	
974		-----		-----	
1107	In house	32		0.00	
1429	In house	32		0.00	
6013	In house	49	G(0.05)	5.59	
7006		32		0.00	

normality	OK
n	16
outliers	2
mean (n)	32.01
st.dev. (n)	3.122
R(calc.)	8.74
R(Horwitz)	8.51





Determination of Acetone on sample #16010, results in mg/kg

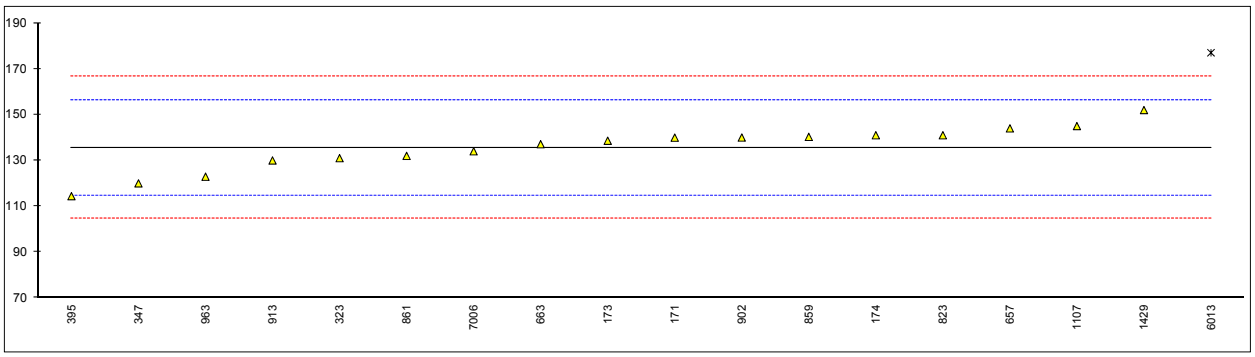
lab	method	value	mark	z(targ)	remarks
171	INH-0001	<1		-----	
173	INH-47	<10		-----	
174	INH-1B	<10		-----	
273		-----		-----	
311		-----		-----	
323	INH-067	<5		-----	
337		-----		-----	
347	INH-96	<10		-----	
391		-----		-----	
395		-----		-----	
522		-----		-----	
551		-----		-----	
557		-----		-----	
657	INH-0047	<10		-----	
663	INH-102582	0		-----	
823	INH-021	0		-----	
859	SH/T1628.2	1.1		-----	
861	SH/T1628.2	1		-----	
902	INH-22	<10		-----	
913	INH-12345	<10		-----	
963	INH-009	2.2		-----	
974		-----		-----	
1107	In house	0		-----	
1429	In house	<5		-----	
6013		-----		-----	
7006		-----		-----	

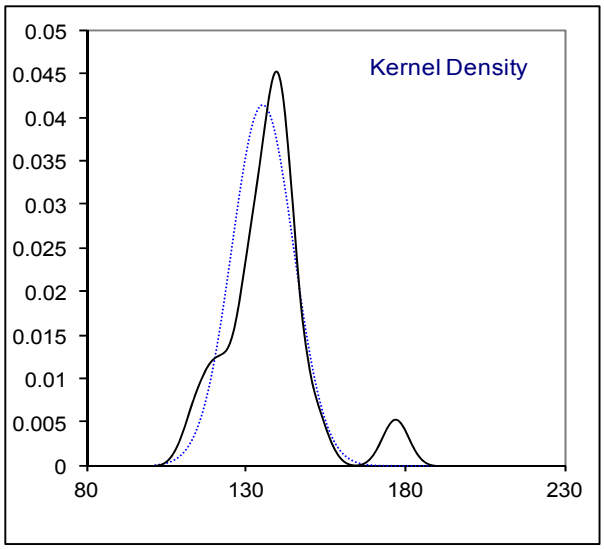
normality	n.a.
n	15
outliers	n.a.
mean (n)	<10
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit)	n.a.

Determination of Ethyl Acetate on sample #16010, results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-0001	139.96		0.43	
173	INH-47	138.6		0.30	
174	INH-1B	141		0.53	
273		-----		-----	
311		-----		-----	
323	INH-067	131		-0.43	
337		-----		-----	
347	INH-96	120		-1.50	
391		-----		-----	
395	INH-257	114.4		-2.04	
522		-----		-----	
551		-----		-----	
557		-----		-----	
657	INH-0047	144		0.82	
663	INH-102582	137.1		0.16	
823	INH-021	141	C	0.53	First reported 4
859	SH/T1628.2	140.3		0.46	
861	SH/T1628.2	132		-0.34	
902	INH-22	140		0.44	
913	INH-12345	130		-0.53	
963	INH-009	122.9		-1.22	
974		-----		-----	
1107	In house	145		0.92	
1429	In house	152		1.59	
6013	In house	177	G(0.05)	4.01	
7006		134		-0.14	

normality	OK
n	17
outliers	1
mean (n)	135.49
st.dev. (n)	9.617
R(calc.)	26.93
R(Horwitz)	28.99

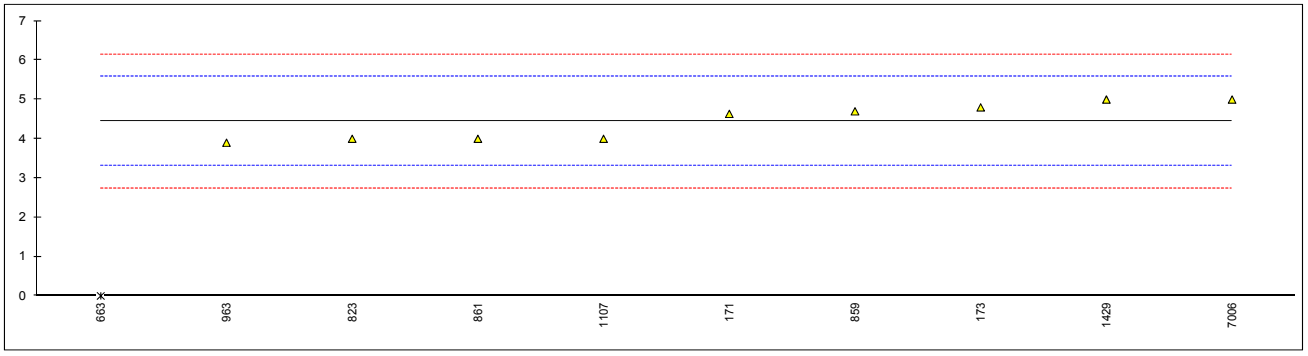


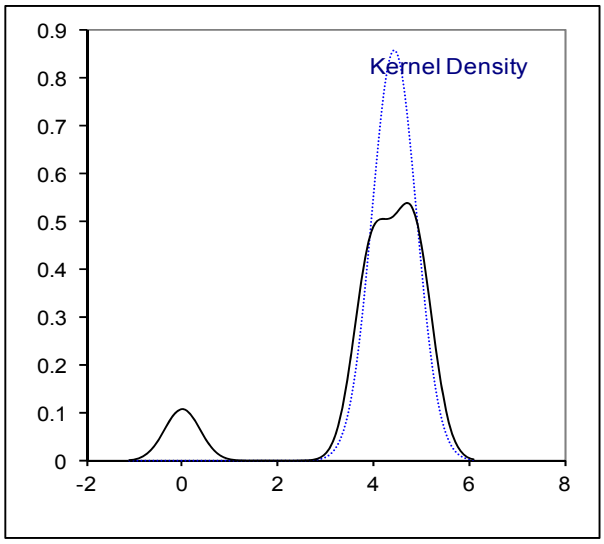


Determination of Methyl Acetate on sample #16010; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-0001	4.635		0.33	
173	INH-47	4.8		0.62	
174	INH-1B	<10		-----	
273		-----		-----	
311		-----		-----	
323	INH-067	<5		-----	
337		-----		-----	
347	INH-96	<20		-----	
391		-----		-----	
395		-----		-----	
522		-----		-----	
551		-----		-----	
557		-----		-----	
657	INH-0047	<10		-----	
663	INH-102582	0	ex	-7.82	Result excluded as zero is not a real result
823	INH-021	4	C	-0.79	First reported 141
859	SH/T1628.2	4.7		0.44	
861	SH/T1628.2	4		-0.79	
902	INH-22	<10		-----	
913		-----		-----	
963	INH-009	3.9		-0.96	
974		-----		-----	
1107	In house	4		-0.79	
1429	In house	5		0.97	
6013		-----		-----	
7006		5		0.97	

normality	OK
n	9
outliers	0 (+1 excl)
mean (n)	4.45
st.dev. (n)	0.466
R(calc.)	1.30
R(Horwitz)	1.59

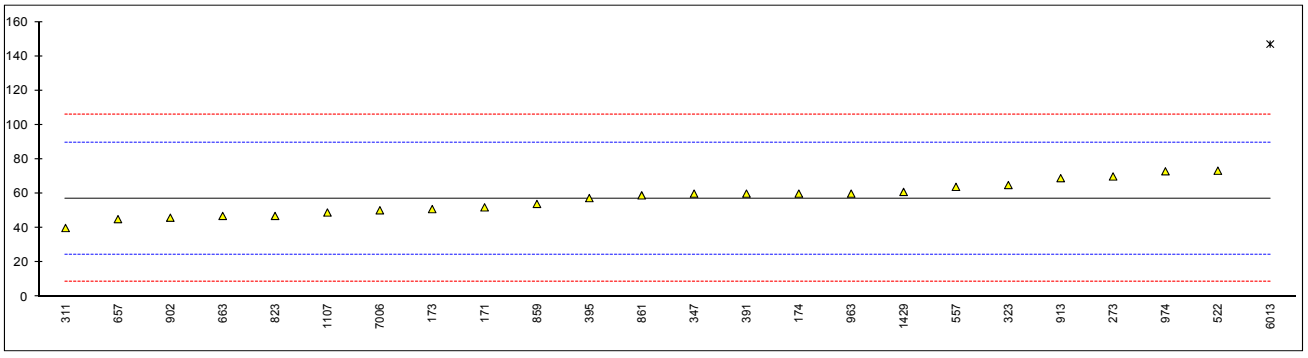


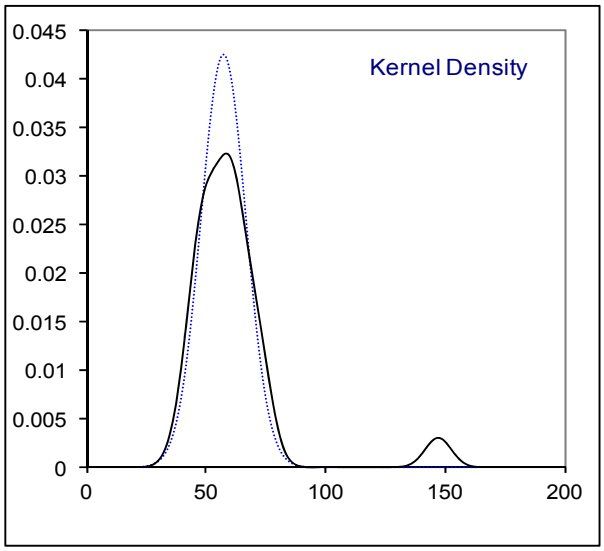


Determination of Water, titrimetric on sample #16010; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	E203	52		-0.31	
173	D1364	51		-0.38	
174	D1364	60		0.18	
273	E203	70		0.80	
311	D1364	40		-1.06	
323	D1364	65		0.49	
337		-----		-----	
347	D1364	60		0.18	
391	E203	60		0.18	
395	D1364	57.4		0.02	
522	D1364	73.3366		1.00	
551		-----		-----	
557	D1364	63.938		0.42	
657	E1064	45.15		-0.74	
663	E203	47		-0.62	
823	D1364	47		-0.62	
859	D1364	54		-0.19	
861	D1364	59		0.12	
902	D1364	46		-0.69	
913	D1364	69		0.74	
963	D1364	60		0.18	
974	D1364	73		0.98	
1107	D1364	49		-0.50	
1429	D1364	61		0.24	
6013	D1364	147	R(0.01)	5.55	
7006	E203	50.3		-0.42	

normality	OK
n	23
outliers	1
mean (n)	57.09
st.dev. (n)	9.374
R(calc.)	26.25
R(D1364:02)	45.34





APPENDIX 2

Number of participants per country

2 labs in BELGIUM
2 labs in BRAZIL
2 labs in CHINA, People's Republic
1 lab in FRANCE
1 lab in INDIA
1 lab in IRAN, Islamic Republic of
2 labs in ITALY
1 lab in MEXICO
1 lab in NETHERLANDS
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SOUTH AFRICA
1 lab in SOUTH KOREA
1 lab in SPAIN
1 lab in THAILAND
2 labs in TURKEY
1 lab in UNITED ARAB EMIRATES
1 lab in UNITED KINGDOM
3 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect 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 outlier test
R(0.05)	= straggler in Rosner outlier test
E	= probably error in calculations
ex	= test result excluded from calculations
n.a.	= not applicable
SDS	= safety data sheet
wd	= withdrawn method

Literature:

1	iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
2	ASTM E178-02
3	ASTM E1301-03
4	ISO 5725, parts 1-6, 1994
5	ISO13528-05
6	M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
7	W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
8	IP 367:84
9	DIN 38402 T41/42
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