Results of Proficiency Test
Vinyl Acetate Monomer
February 2012

CONTENTS

Appendices:

1   INTRODUCTION

Since 2007, the Institute for Interlaboratory Studies organizes a proficiency test for the analysis of Vinyl Acetate Monomer (VAM). During the annual proficiency testing program 2011/2012, it was decided to continue the round robin for the analysis of Vinyl Acetate Monomer. In this interlaboratory study 25 laboratories in 19 different countries have participated. 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 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. It was decided to send one sample only. Analyses for fit-for-use and homogeneity were subcontracted to an accredited laboratory.
Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluations.

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 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and 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 January 2010 (iis-protocol, version 3.2).

2.3   CONFIDENTIALITY STATEMENT

All data present 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 amount of bulk material of Vinyl Acetate Monomer was obtained from a chemical producer. After homogenisation, 25 litre of the Vinyl Acetate Monomer was divided over 44 brown glass bottles of 500 mL and labelled #12005.
The homogeneity of the subsamples #12005 was checked by determination of Density in accordance with ASTM D4052:11 and water in accordance with ASTM D1364:07 on 8 stratified randomly selected samples.

	Density @20℃ in kg/L	Water in mg/kg
sample #12005-1	0.9322	147
sample #12005-2	0.9321	154
sample #12005-3	0.9322	147
sample #12005-4	0.9322	161
sample #12005-5	0.9322	133
sample #12005-6	0.9321	136
sample #12005-7	0.9322	140
sample #12005-8	0.9321	133

table 1: homogeneity test results of subsamples #12005

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities of the target methods or with the reproducibility calculate using the Horwitz equation in agreement with the procedure of ISO 13528, Annex B2 in the next table

	Density @20℃ in kg/L	Water in mg/kg
r (sample #12005)	0.00014	29
reference test	D4052:02e1	ASTM D1364:07
0.3*R (reference test)	0.00015	22

table 2: repeatabilities of subsamples #12005

The calculated repeatability for Density is in agreement with 0.3 times the corresponding reproducibility of the target method. The repeatability for Water is not in agreement with 0.3 times the corresponding reproducibility. However, the calculated repeatability for Water is in agreement with the repeatability limits of ASTM D1364:07 (r_{D1364}= 36). Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 1 bottle of 500 mL Vinyl Acetate Monomer (sample #12005) was sent on February 1, 2012.

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 asked to analyse according standard specification for VAM (ASTM D2190:07) plus additionally Acetaldehyde, Acetone, Acidity, Density @ 20°C, Distillation, Ethyl Acetate, Inhibitor, Purity and Water. To get comparable results, a detailed report form on which the units and the standard methods were printed, was sent together with each set of samples. Also a letter of instructions and a SDS were added to the package.

3   RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.
Directly after deadline, a reminder fax was sent to those laboratories that had not yet reported any 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 data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1   STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' January 2010 (iis-protocol, version 3.2)
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. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs 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. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs 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. 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 consequences for the evaluation of the test results.

Finally the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

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 method is for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nr.13 and 14).

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, 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 literature reproducibility by division with 2.8.

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 the fit-for-useness of the reported test result.

In case no literature reproducibility was available, other target values were used. In some cases literature repeatability is available; in other cases a reproducibility of a former iis proficiency test could be used and also the Horwitz equation can be used to estimate target reproducibility.

The z-scores were calculated according to:

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. The usual interpretation of z-scores is as follows:

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

4   EVALUATION

In this proficiency test, no serious problems were encountered with despatch of the samples. Of the 25 participants, 4 participants reported results after the final reporting date and one laboratory had not received the sample still, due to customs clearance problems.

Not all participants were able to report results for all the requested tests.

In total 257 numerical test results were reported. Observed were 8 outlying test results, which is 3.1% 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, not for all determinations a suitable standard test method exists with precision data. For these determinations, the spreads were compared against the strict spreads estimated from the Horwitz equation.

A not-normal distribution was found for Distillation (IBP, Mid Boiling Point, DP and Boiling Range). In these cases the statistical evaluation should be used with due care.

Acidity: The acidity determination without Nitrogen purging was very problematic and three groups of test results are visible. However the acidity determination with Nitrogen purging was not problematic.
The results of the individual laboratories (e.g. 171, 323, 657, 855, 859, 861, 865, 902 and 1429) show that the acidity without purging will be higher than the acidity with purging as expected. However, the average acidity without purging is not significantly different from the average acidity with purging. This may be explained by the group of four low test results in the determination without purging. These four low results have a large effect on the calculated average.
In total two statistical outliers were observed. Only the calculated reproducibility of the determination with purging, after rejection of the statistical outliers, is in agreement with the requirements of ASTM D2086:08.

Appearance: No analytical problems were observed. All laboratories agreed about the appearance of the sample, although different ways of reporting were used. Fourteen laboratories reported the appearance as pass in agreement with ASTM E2680. Other laboratories reported 'clear and free of suspended matter' or similar remarks.

Purity: Regretfully, no reference method with precision data exists for this determination. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is small in comparison with the spread found in the previous PT (iis11C15).

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

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

Acetone: Ony two numerical results were reported. Therefore no significant statistical conclusions were drawn. The majority of the reporting participants agreed on a result below 10 mg/kg.

Acetaldehyde: This determination may be problematic. No statistical outliers were observed and the reproducibility in agreement with ASTM D2191:06. However, the reproducibility is based upon an interlaboratory study on four samples of vinyl acetate containing 100, 200, 300, and 500 mg/kg acetaldehyde. This range is far above the measured concentration in sample #12005 and therefore the reproducibility according to ASTMD2191:06 may not be reliable. When the Horwitz estimate for the target reproducibility is used for evaluation than this method is problematic.

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

Density @ 20°C: This determination is not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D4052:02e1. The current version of this method ASTM D4052:11 only gives reproducibilities for the density range 0.71 g/ml to 0.88 g/ml, being valid for gasolines, distillates, basestocks and lubricating oils. Therefore this 2011 version is not applicable for VAM.

Distillation: This determination was not problematic. In total two statistical outliers were observed. The calculated reproducibilities, after rejection of the statistical outliers, are all in good agreement with the requirements of ASTM D1078:11 (Auto).

Water: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the calculated reproducibility with the requirements of ASTM D1364:07.

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, EN standards) are compared in the next table.

Parameter	unit	n	mean	2.8 * sd	R (lit)
Acidity (without N_2 purging)	mg/kg	18	20.3	18.3	6.0
Acidity (with N_2 purging)	mg/kg	10	20.2	6.5	6.0
Appearance	------	23	pass	-----	-----
Purity	%M/M	22	99.97	0.02	unknown
Methyl Acetate	mg/kg	15	6.5	1.7	2.2
Ethylacetate	mg/kg	20	167.0	32.9	34.6
Acetone	mg/kg	14	< 10	n.e.	n.e.
Acetaldehyde	mg/kg	19	31.4	25.8	80.0*
Inhibitor	mg/kg	21	4.45	2.90	1.00
Density @ 20°C	kg/L	24	0.9322	0.0002	0.0005
Initial Boiling Point	ºC	19	72.42	0.22	1.13
50% recovery	ºC	19	72.69	0.09	0.50
Dry Point	ºC	19	72.80	0.22	0.78
Boiling Range	ºC	20	0.38	0.18	0.70
Water	mg/kg	23	127.2	92.0	67.7

table 3: performance evaluation sample #12005

*see §4.1 (page 8)

4.3 EVALUATION OF THE PROFICIENCY TEST OF FEBRUARY 2012 WITH PREVIOUS PTS

	February 2012	April 2011	March 2010	March 2009
Number of rep. participants	24	28	21	26
Number of results reported	257	264	201	239
Number of statistical outliers	8	16	8	10
Percentage outliers	3.1%	6.1%	4.0%	4.2%

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 2012	*April 2011*	*March 2010*	*March 2009*
Acidity (no purging)	--	--	--	--
Acidity (with purging)	+/-	--	--	--
Purity	n.e.	n.e	n.e	n.e
Methyl Acetate	++	--	n.e	++
Ethyl Acetate	+/-	--	++	++
Acetone	n.e.	n.e	n.e	n.e
Acetaldehyde	++	++	++	++
Inhibitor	--	-	--	--
Density @ 20°C	++	++	++	++
Distillation	++	++	++	++
Water	-	+	--	++

table 5: comparison determinations against the reference standards

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 #12005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2086	19		-0.60	
173	INH-44	19		-0.60	
174	D2086	22.0		0.80	
311	D2086	11		-4.33	
323	D2086	11		-4.33	
395	INH-44	8.99		-5.27	
446	INH-44	11.7		-4.01	
522		-----		-----	
551		-----		-----	
562	D2086	26.6		2.95	
613		-----		-----	
657	D2086	21.43		0.53	
662		-----		-----	
786	D2086	23.3		1.41	
823	D2086	23		1.27	
855	D2086	20.4		0.05	
859	D2086	18.7		-0.74	
861	D2086	20.1		-0.09	
865	D2086	23.1		1.31	
902	D2086	21.9		0.75	
913		-----		-----	
963		-----		-----	
974		-----		-----	
1429	D2086	32.4		5.65	
7006	D2086	31.54		5.25	

normality	OK
n	18
outliers	0
mean (n)	20.29
st.dev. (n)	6.527
R(calc.)	18.27
R(D2086:08)	6.00

Kernel Density
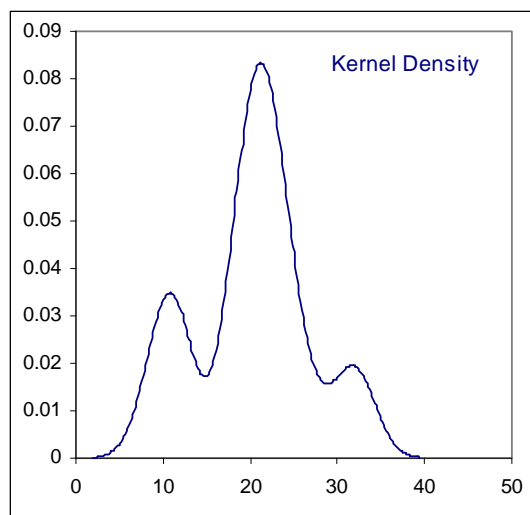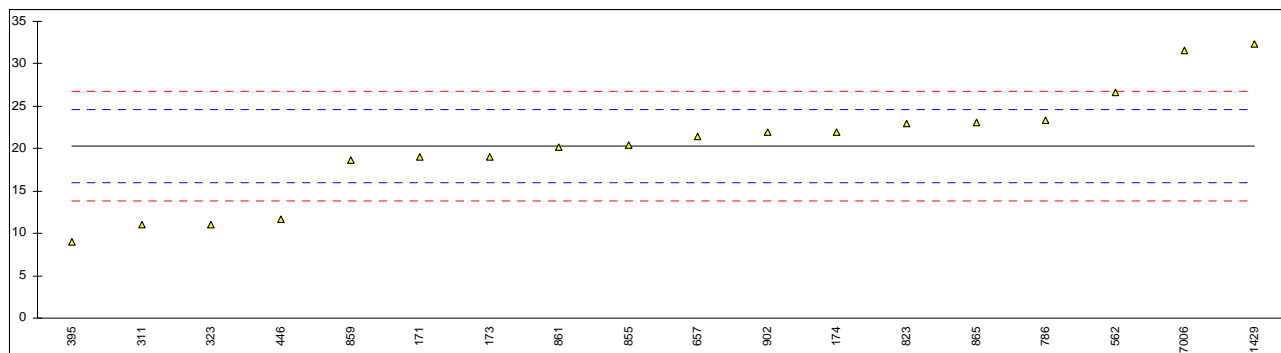
Determination of Acidity (with N_2 purging) on sample #12005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2086	18		-1.01	
173		-----		-----	
174		-----		-----	
311		-----		-----	
323	D2086	5	G(0.01)	-7.08	
395		-----		-----	
446		-----		-----	
522	INH-40	21.18		0.48	
551		-----		-----	
562		-----		-----	
613		-----		-----	
657	D2086	19.74		-0.20	
662	INH-40	580	G(0.01)	261.26	
786		-----		-----	
823	D2086	23		1.32	
855	D2086	17.5		-1.24	
859	D2086	17.4		-1.29	
861	D2086	18.4		-0.82	
865	D2086	22.1		0.90	
902	D2086	20.6		0.20	
913		-----		-----	
963		-----		-----	
974		-----		-----	
1429	D2086	23.7		1.65	
7006		-----		-----	

normality	OK
n	10
outliers	2
mean (n)	20.16
st.dev. (n)	2.3149
R(calc.)	6.48
R(D2086:08)	6.00
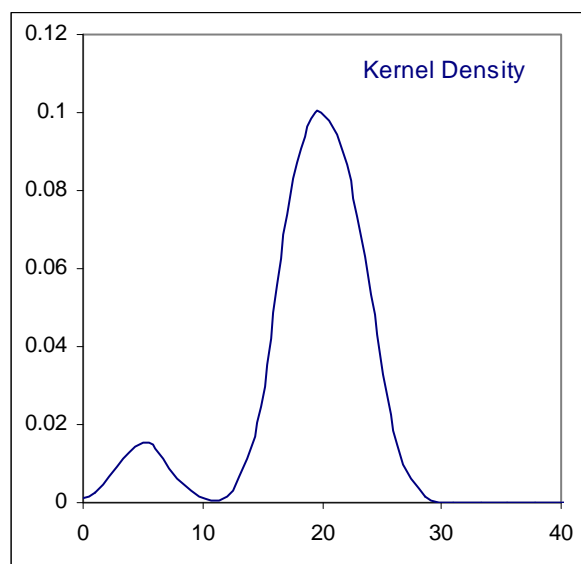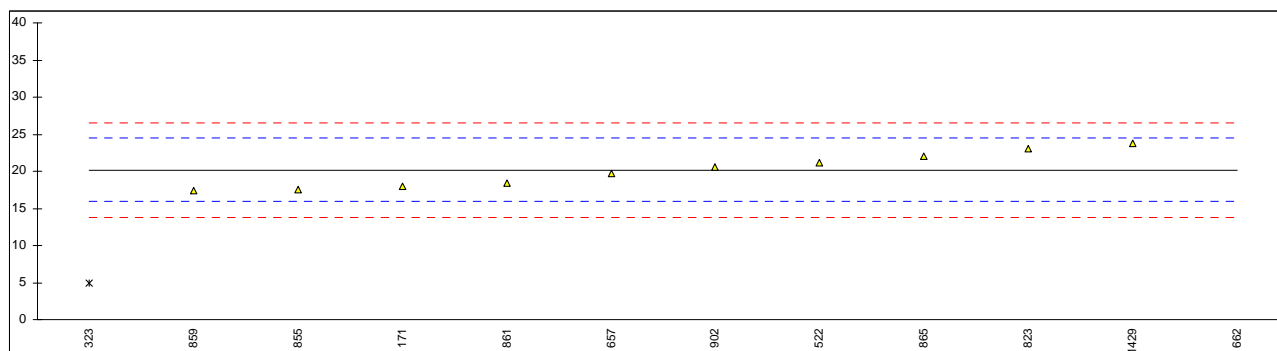
Determination of Appearance on sample #12005;

lab	method	value	mark	z(targ)	remarks
171	E2680	C&F		-----	
173	E2680	CFSM		-----	
174	E2680	C&F		-----	
311	E2680	pass		-----	
323	E2680	CFSM		-----	
395	E2680	pass		-----	
446	INH-500	pass		-----	
522	INH-40	CFSM		-----	
551		-----		-----	
562	E2680	pass		-----	
613	INH-40	B&C		-----	
657	E2680	pass		-----	
662	E2680	B&C		-----	
786	E2680	pass		-----	
823	E2680	pass		-----	
855	E2680	pass		-----	
859	E2680	pass		-----	
861	E2680	pass		-----	
865	E2680	pass		-----	
902	E2680	pass		-----	
913	E2680	CFSM		-----	
963	E2680	pass		-----	
974	E2680	pass		-----	
1429	E2680	B&C		-----	
7006		-----		-----	

C&F	= Clear and free	
CFSM	= Clear and free from suspended matter	
B&C	= Bright and clear	

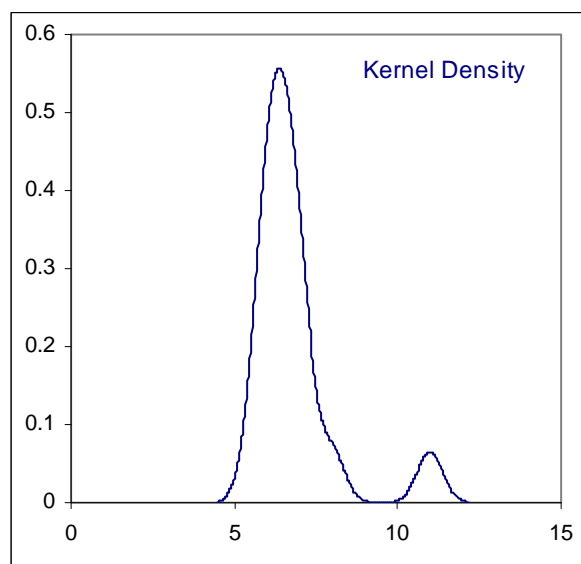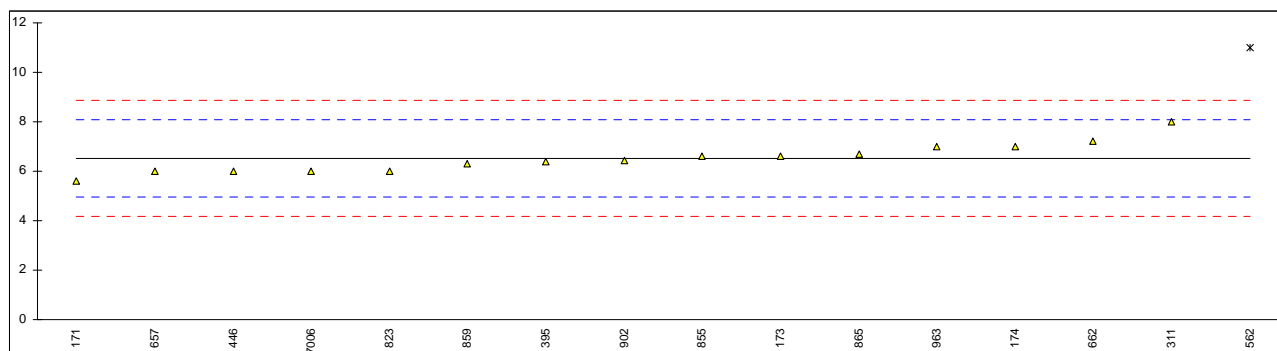## Determination of Purity on sample #12005; results in % M/M

lab	method	value	mark	z(targ)	remarks
171	INH-256	99.967		-----	
173	INH-47	99.98		-----	
174	D3545	99.965		-----	
311	INH-122	99.98		-----	
323	INH-067	99.97		-----	
395	INH-257	99.976		-----	
446		-----		-----	
522	INH-40	99.98		-----	
551		-----		-----	
562	INH-001	99.96		-----	
613	INH-40	99.972	C	-----	first reported 99.997
657	INH-047	99.9740		-----	
662	INH-40	99.90	G(0.01)	-----	
786	INH-004	99.96		-----	
823	D3545	99.971		-----	
855	INH-1628	99.976		-----	on dry basis
859	INH-1628	99.971		-----	
861	INH-065	99.965		-----	
865	INH-1628	99.966		-----	
902	INH-22	99.969		-----	
913	INH-256	99.97		-----	
963	INH-009	99.97		-----	
974	INH-03	99.972		-----	
1429	in house	99.9635		-----	
7006	INH-2	99.97		-----	

normality	OK	
n	22	
outliers	1	
mean (n)	99.97033	
st.dev. (n)	0.00583	
R(calc.)	0.016325	
R(target)	unknown	Compare R(iis11C15) = 0.0298
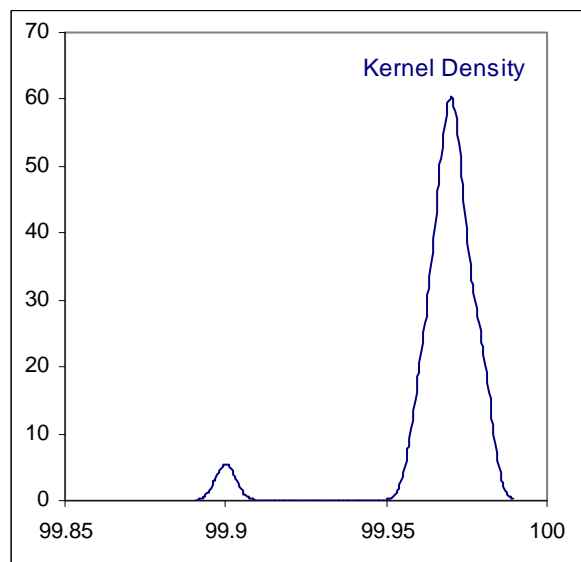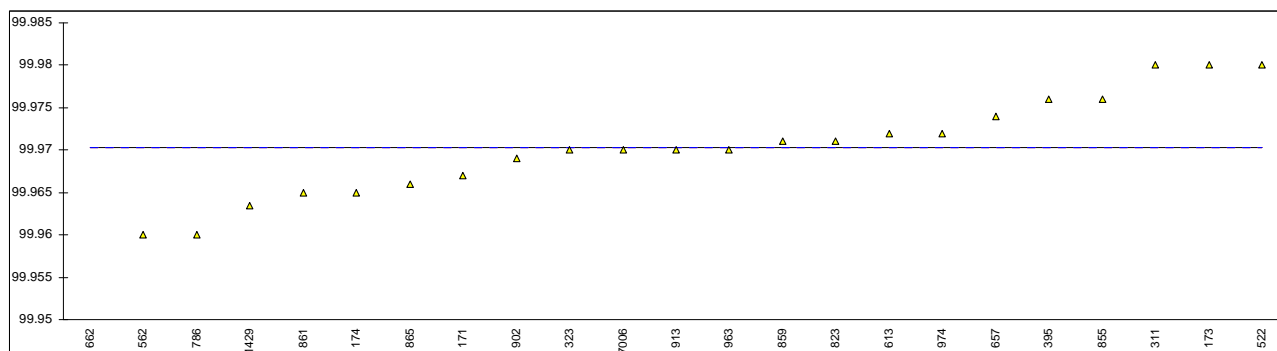
Determination of Methyl Acetate on sample #12005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-256	5.6		-1.17	
173	INH-47	6.6		0.10	
174	D3545	7.0		0.61	
311	INH-122	8		1.88	
323	INH-067	<10		-----	
395	INH-47	6.4		-0.16	
446	INH-47	6		-0.66	
522		-----		-----	
551		-----		-----	
562	INH-001	11	G(0.01)	5.69	
613		-----		-----	
657	INH-047	6		-0.66	
662	INH-40	7.20		0.86	
786	INH-004	<10		-----	
823	D3545	6		-0.66	
855	INH-1628	6.6		0.10	
859	INH-1628	6.3		-0.28	
861		-----		-----	
865	INH-1628	6.7		0.22	
902	INH-22	6.45		-0.09	
913		-----		-----	
963	INH-009	7		0.61	
974		-----		-----	
1429	in house	<10		-----	
7006	INH-2	6.0		-0.66	

normality	OK
n	15
outliers	1
mean (n)	6.52
st.dev. (n)	0.607
R(calc.)	1.699
R(Horwitz)	2.20

Determination of Ethyl Acetate on sample #12005, results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-256	173.3		0.51	
173	INH-47	163.6		-0.28	
174	D3545	191.0		1.94	
311	INH-122	180	C	1.05	first reported 195
323	INH-067	149		-1.46	
395	INH-47	176.9		0.80	
446	INH-47	162		-0.41	
522		-----		-----	
551		-----		-----	
562	INH-001	169		0.16	
613		-----		-----	
657	INH-047	170		0.24	
662	INH-40	166.92		-0.01	
786	INH-004	187		1.61	
823	D3545	164		-0.24	
855	INH-1628	166.8		-0.02	
859	INH-1628	167.0		0.00	
861		-----		-----	
865	INH-1628	175		0.64	
902	INH-22	158.6		-0.68	
913		-----		-----	
963	INH-009	163		-0.33	
974	INH-03	143	C	-1.94	first reported 129
1429	in house	152.4		-1.18	
7006	INH-2	162.0		-0.41	

normality	OK	
n	20	
outliers	0	
mean (n)	167.03	
st.dev. (n)	11.765	
R(calc.)	32.94	
R(Horwitz)	34.63	
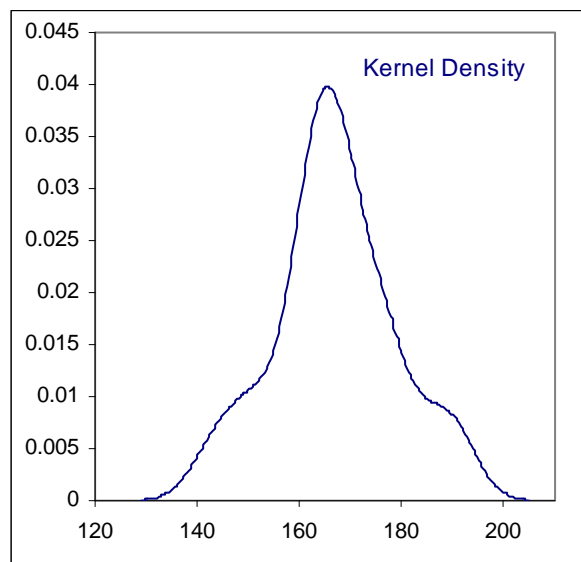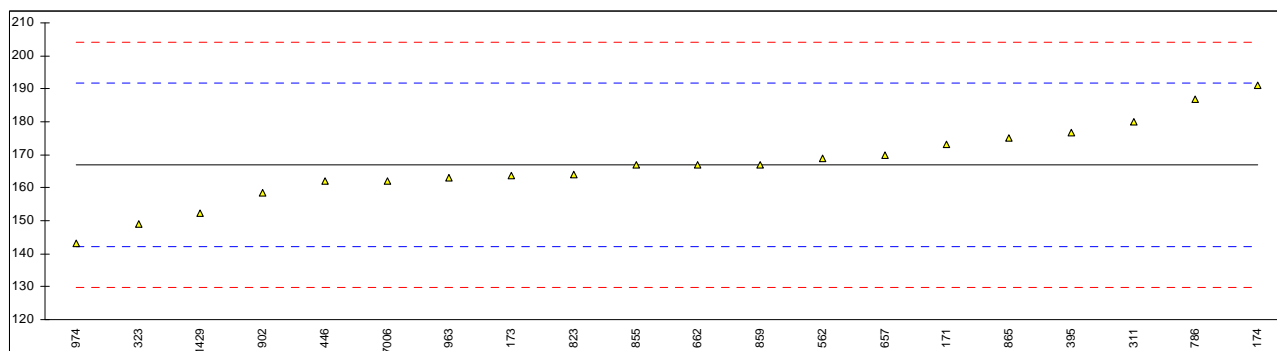
Determination of Acetone on sample #12005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-256	<10		-----	
173	INH-47	<0.01		-----	
174	D3545	<5		-----	
311	INH-122	0.3		-----	
323	INH-067	<10		-----	
395		-----		-----	
446		-----		-----	
522		-----		-----	
551		-----		-----	
562	INH-001	<10		-----	
613		-----		-----	
657	GC	<10		-----	
662	INH-40	n.d.		-----	
786	INH-004	<10		-----	
823	D3545	<5		-----	
855	INH-1628	<10		-----	
859	INH-1628	<10		-----	
861		-----		-----	
865	INH-1628	<10		-----	
902	INH-22	99.52		-----	false positive?
913		-----		-----	
963	INH-009	<5		-----	
974		-----		-----	
1429	in house	<10		-----	
7006		-----		-----	

	normality	n.e.
	n	14
	outliers	1
	mean (n)	<10
	st.dev. (n)	n.e.
	R(calc.)	n.e.
	R(Horwitz)	n.e.

Determination of Acetaldehyde on sample #12005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-256	28.5		-0.10	
173	INH-47	28.6		-0.10	
174		-----		-----	
311	INH-122	34		0.09	
323	INH-067	22		-0.33	
395	INH-47	37.9		0.23	
446		-----		-----	
522	INH-40	10.9		-0.72	
551		-----		-----	
562	INH-001	30		-0.05	
613		-----		-----	
657	D2191	38.27		0.24	
662	INH-40	51.77		0.71	
786	INH-004	32		0.02	
823	D2191	30		-0.05	
855	D2191	38		0.23	
859	D2191	33		0.06	
861	INH-065	26		-0.19	
865	D2191	38		0.23	
902	INH-22	25.18		-0.22	
913		-----		-----	
963	D2191	30		-0.05	
974		-----		-----	
1429	in house	18.2		-0.46	
7006	INH-2	44.0		0.44	

normality	OK
n	19
outliers	0
mean (n)	31.39
st.dev. (n)	9.228
R(calc.)	25.84
R(D2191:06)	80.00

Compare R(Horwitz) = 8.37
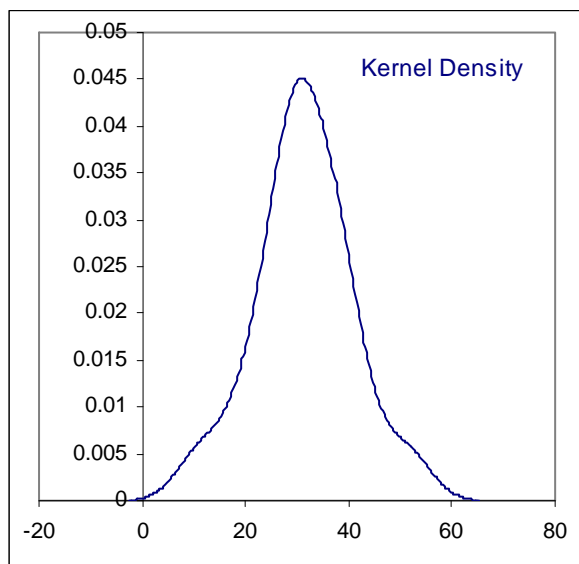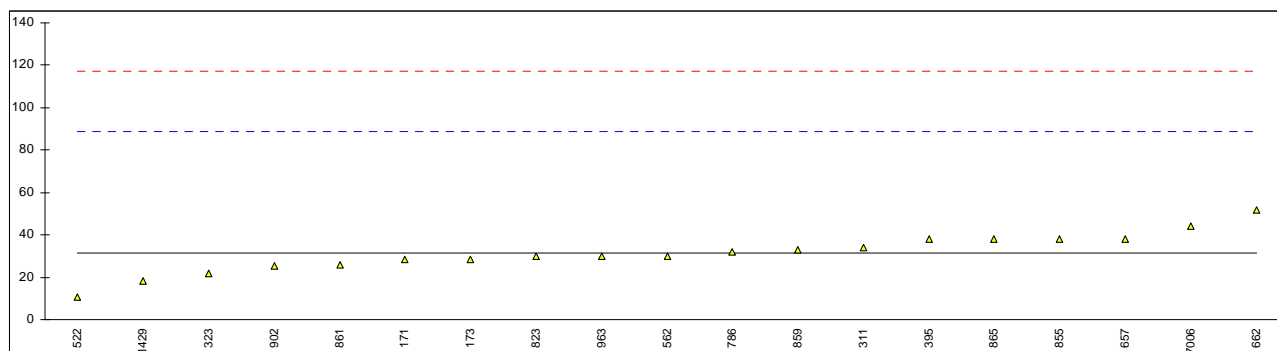
Determination of Inhibitor (Hydroquinone) on sample #12005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2193	5.2		2.11	
173	INH-43	5.7		3.51	
174	D2193	5.0		1.55	
311	D2193	3.9		-1.53	
323	D2193	5.3		2.39	
395	INH-43	5.04		1.66	
446		-----		-----	
522	INH-40	3.5		-2.65	
551		-----		-----	
562	D2193	3.39		-2.96	
613	INH-40	3.13		-3.69	
657	D2193	4.0		-1.25	
662		-----		-----	
786	D2193	3.9		-1.53	
823	D2193	6.2		4.91	
855	D2193	4.62		0.48	
859	D2193	4.26		-0.53	
861	D2193	4.11		-0.95	
865	D2193	4.55		0.29	
902		-----		-----	
913	D2193	4.1		-0.97	
963	D2193	2.7		-4.89	
974	D2193	3.21		-3.47	
1429	D2193	6.75		6.45	
7006	D2193	4.84		1.10	

normality	OK
n	21
outliers	0
mean (n)	4.447
st.dev. (n)	1.0366
R(calc.)	2.903
R(D2193:06)	1.000
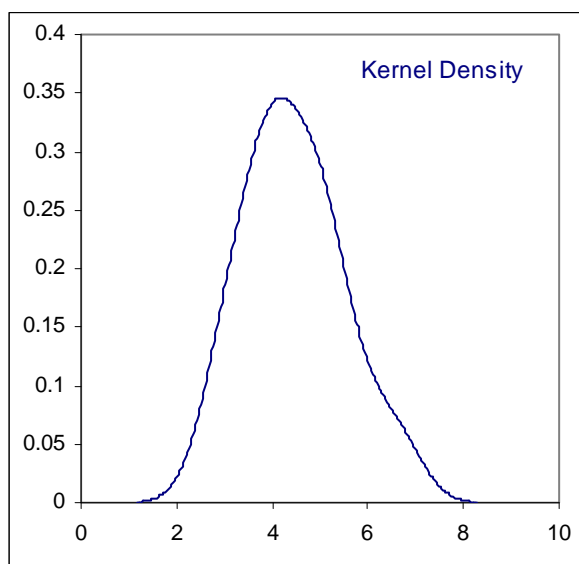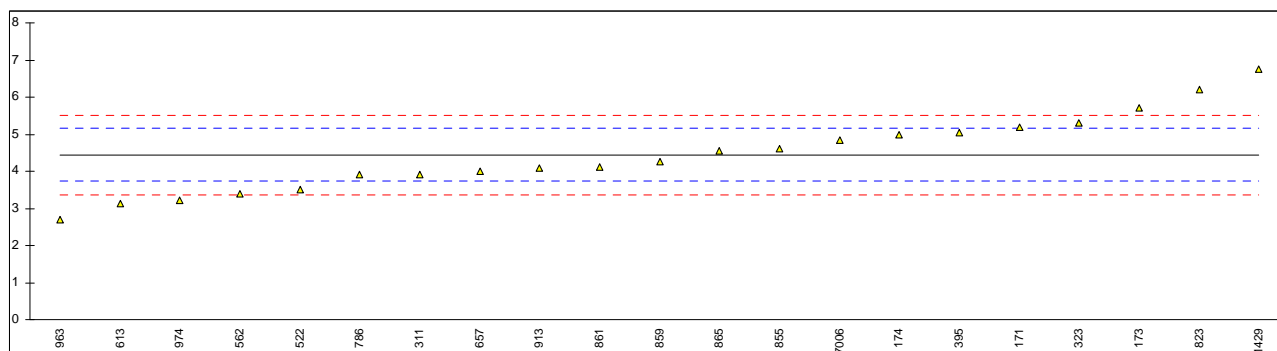
Determination of Density @ 20 °C on sample #12005; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9323		0.60	
173	D4052	0.93218		-0.07	
174	D4052	0.9322		0.04	
311	D4052	0.93218		-0.07	
323	D4052	0.9321		-0.52	
395	D4052	0.9321		-0.52	
446	D4052	0.9320		-1.08	
522	INH-40	0.9324		1.16	
551		-----		-----	
562	D4052	0.9322		0.04	
613	D4052	0.93224		0.27	
657	D4052	0.9321		-0.52	
662	D4052	0.9322		0.04	
786	D4052	0.9321		-0.52	
823	D4052	0.9322		0.04	
855	D4052	0.93216		-0.18	
859	D4052	0.93222		0.16	
861	D4052	0.93215		-0.24	
865	D4052	0.93224		0.27	
902	D4052	0.93224		0.27	
913	D4052	0.9322		0.04	
963	D4052	0.9322		0.04	
974	D4052	0.9323		0.60	
1429	D4052	0.9322		0.04	
7006	D4052	0.9322		0.04	

normality	OK
n	24
outliers	0
mean (n)	0.93219
st.dev. (n)	0.000080
R(calc.)	0.00023
R(D4052:02e1)	0.00050
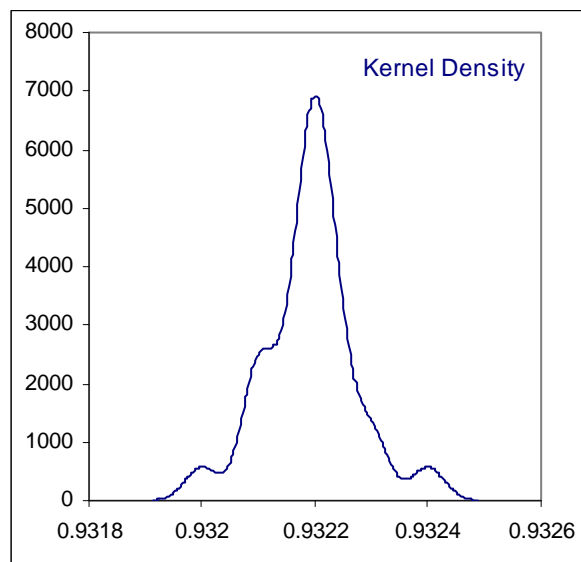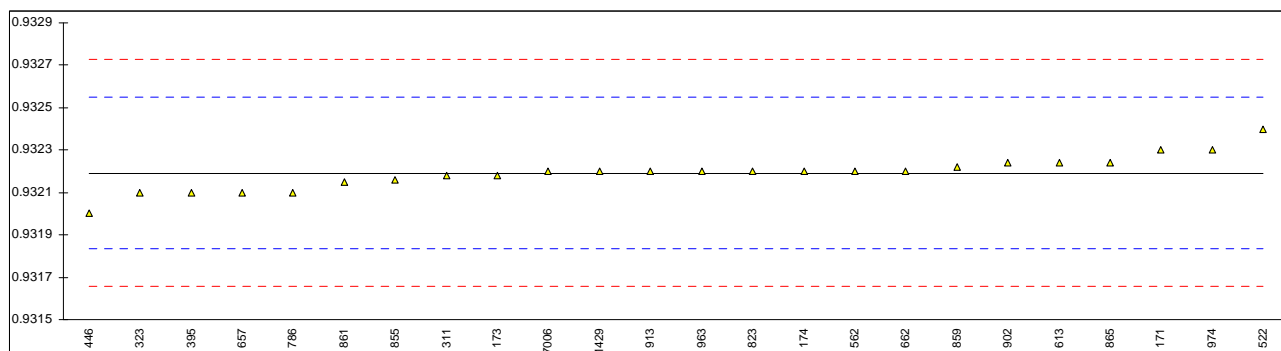
Determination of Distillation on sample #12005; results in ºC

| lab	method	IBP	mark	50% rec.	mark	DP	mark	range	mark
171	D1078	72.4		72.7		72.7		0.3	
173	D1078	72.5		72.7		72.8		0.3	
174	D1078	72.4	C	72.7		72.9		0.5	C
311	D1078	72.5		72.7		72.8		0.3	
323	D1078	72.5		72.7		72.9		0.4	
395	D1078	72.5		72.7		72.9		0.4	
446	INH-45	72.4		72.7		72.8		0.4	
522		-----		-----		-----		-----	
551		-----		-----		-----		-----	
562	D1078	72.2		72.6		72.6		0.4	
613		-----		-----		-----		-----	
657	D1078	72.5		72.7		72.9		0.4	
662		-----		-----		-----		-----	
786	D1078	72.4		72.6		72.7		0.3	
823	D1078	72.4		72.7		72.8		0.4	
855	D1078	72.5		72.7		72.8		0.3	
859	D1078	72.5		72.7		72.8		0.3	
861	D1078	72.4		72.7		72.8		0.4	
865	D1078	72.4		72.7		72.8		0.4	
902	D1078	72.4		72.7		72.8		0.4	
913		72.4		72.7		72.8		0.4	
963	D1078	72.3		72.7		72.8		0.5	
974		-----		-----		-----		-----	
1429	D1078	72.4		72.7		72.7		0.3	
7006	D86	71.8	G(0.01)	72.1	G(0.01)	-----		0.4	
	normality	not OK		not OK		not OK		not OK	
	n	19		19		19		20	
	outliers	1		1		0		0	
	mean (n)	72.421		72.689		72.795		0.375	
	st.dev. (n)	0.0787		0.0315		0.0780		0.0639	
	R(calc.)	0.220		0.088		0.218		0.179	
	R(D1078:11-A)	1.130		0.496		0.779		0.696	

* laboratory 174 first reported 72.2 / 72.7 / 72.9 / 0.7

z-scores of distillation

| lab	IBP	50% rec	DP	range
171	-0.05	0.06	-0.34	-0.30
173	0.20	0.06	0.02	-0.30
174	-0.05	0.06	0.38	0.50
311	0.20	0.06	0.02	-0.30
323	0.20	0.06	0.38	0.10
395	0.20	0.06	0.38	0.10
446	-0.05	0.06	0.02	0.10
522	-----	-----	-----	-----
551	-----	-----	-----	-----
562	-0.55	-0.50	-0.70	0.10
613	-----	-----	-----	-----
657	0.20	0.06	0.38	0.10
662	-----	-----	-----	-----
786	-0.05	-0.50	-0.34	-0.30
823	-0.05	0.06	0.02	0.10
855	0.20	0.06	0.02	-0.30
859	0.20	0.06	0.02	-0.30
861	-0.05	0.06	0.02	0.10
865	-0.05	0.06	0.02	0.10
902	-0.05	0.06	0.02	0.10
913	-0.05	0.06	0.02	0.10
963	-0.30	0.06	0.02	0.50
974	-----	-----	-----	-----
1429	-0.05	0.06	-0.34	-0.30
7006	-1.54	-3.32	-----	0.10
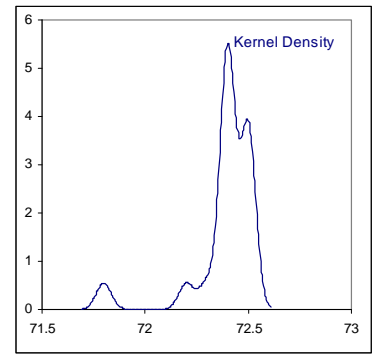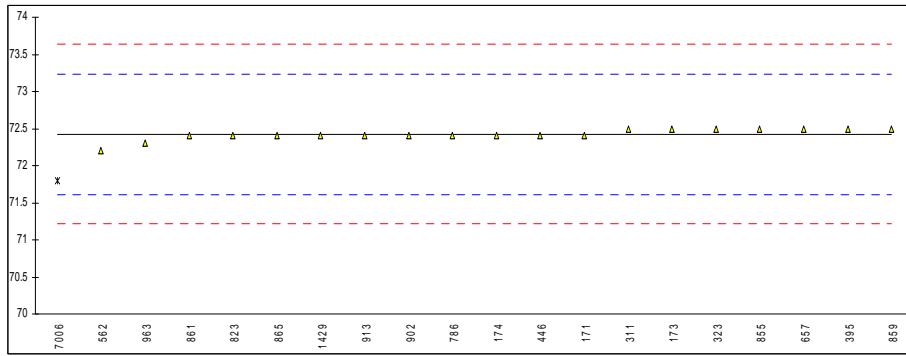
ibp
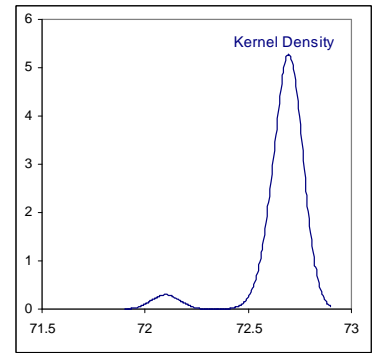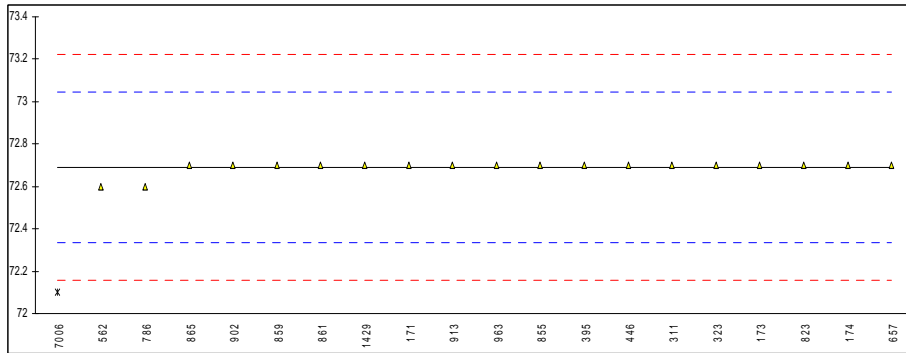
mid point (50% rec.)
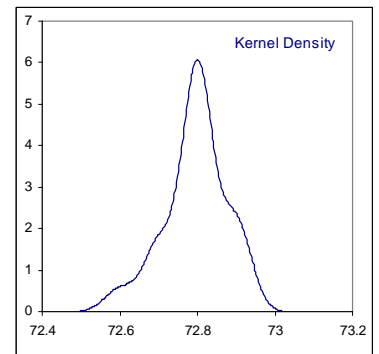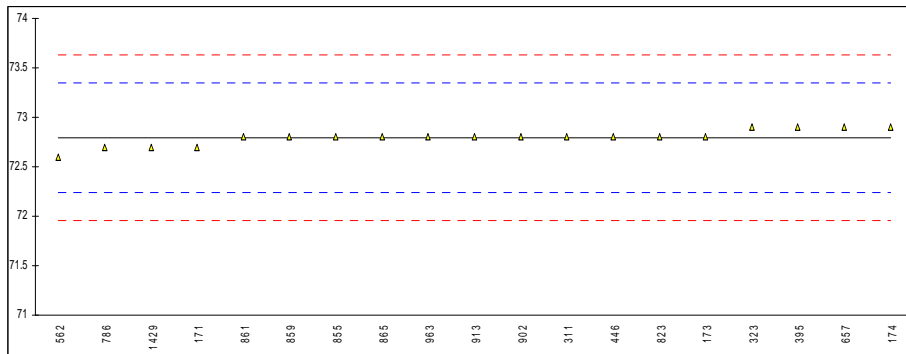
dry point
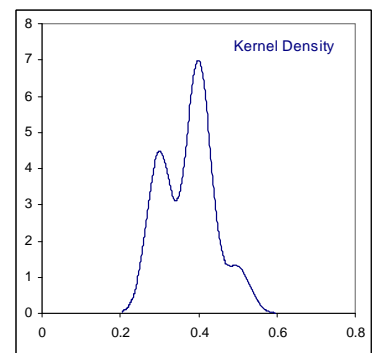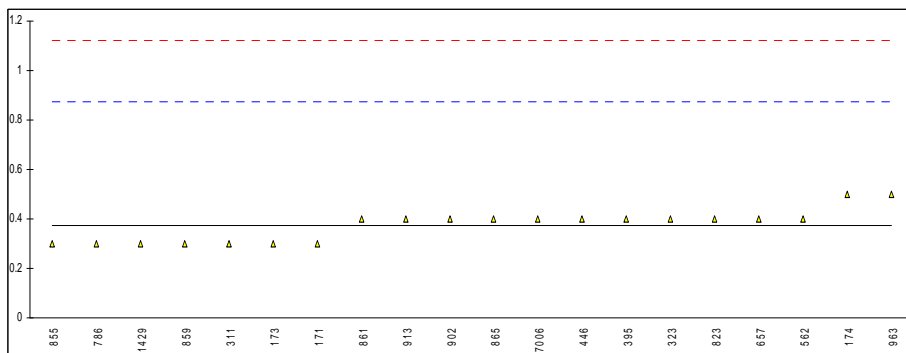
distillation range

Determination of Water on sample #12005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	E203	114		-0.55	
173	E203	91.0		-1.50	
174	D1364	121		-0.26	
311	D1364	170		1.77	
323	D1364	170		1.77	
395	D1364	98.41		-1.19	
446	INH-63	133		0.24	
522	INH-40	143.22		0.66	
551		-----		-----	
562	E203	177		2.06	
613		-----		-----	
657	E1064	143		0.65	
662	INH-40	150		0.94	
786	D1364	136		0.36	
823	D1364	117		-0.42	
855	D1364	104		-0.96	
859	D1364	94		-1.37	
861	E1064	91		-1.50	
865	D1364	104		-0.96	
902	D1364	90		-1.54	
913	D1364	180	C	2.18	reported in a different unit 0.018
963	D1364	103		-1.00	
974	D1364	126		-0.05	
1429	D1364	188		2.52	
7006	D1364	82.0		-1.87	

normality	OK
n	23
outliers	0
mean (n)	127.20
st.dev. (n)	32.839
R(calc.)	91.95
R(D1364:07)	67.67

Kernel Density
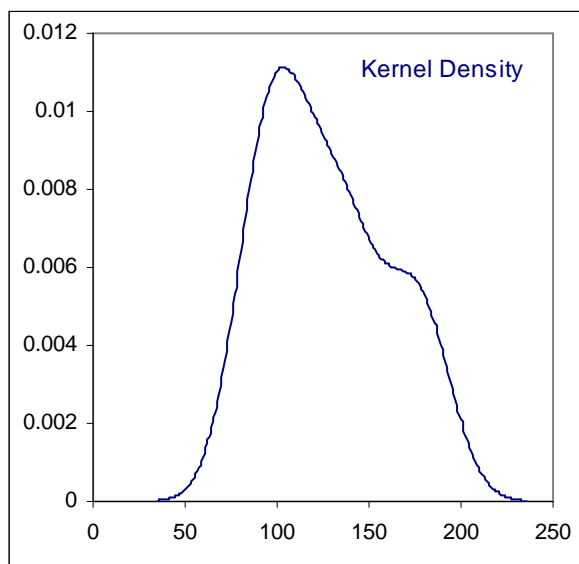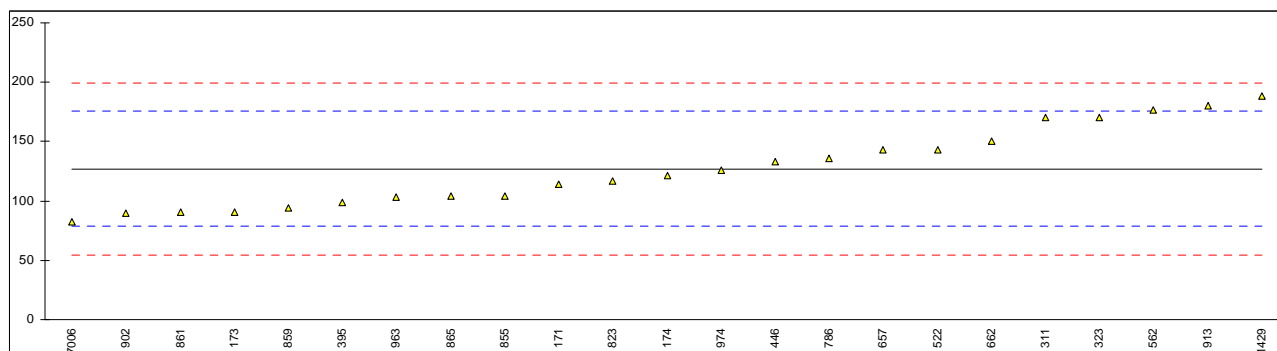
APPENDIX 2

Number of participants per country

1 lab in AUSTRALIA

1 lab in BELGIUM

1 lab in BRAZIL

1 lab in CHILE

1 lab in INDIA

1 lab in IRAN

1 lab in ITALY

1 lab in KOREA

1 lab in MEXICO

4 labs in P.R. of CHINA

1 lab in RUSSIA

1 lab in SAUDI ARABIA

1 lab in SINGAPORE

1 lab in THAILAND

1 lab in THE NETHERLANDS

1 lab in TURKEY

1 lab in U.A.E.

3 labs in U.S.A.

2 labs in UNITED KINGDOM

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
E	= error in calculations
ex	= excluded from calculations
n.e.	= not evaluated
wd	= withdrawn method

Literature:

1	i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
2	ASTM E178:89
3	ASTM E1301:89
4	ISO 5725:86
5	ISO 5725, parts 1-6, 1994
6	ISO13528-05
7	M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8	W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9	IP 367:84
10	DIN 38402 T41/42
11	P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12	J.N. Miller, Analyst, 118, 455, (1993)
13	Analytical Methods Committee Technical brief, No4 January 2001
14	The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson (see http://www.rsc.org/suppdata/an/b2/b205600n/)