Results of Proficiency Test
Vinyl Acetate Monomer
February 2013

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 2012/2013, it was decided to continue the round robin for the analysis of Vinyl Acetate Monomer. In this interlaboratory study 26 laboratories in 18 different countries have participated. See appendix 2 for the number of participants per country.
In this report, the results of the 2013 VAM 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 of VAM (0.5 litre bottle, labelled #13005) to the participants. 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 guide 43, ILAC-G13:2007 and IEC/ISO17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 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 amount of bulk material of Vinyl Acetate Monomer was obtained from a Belgian producer. After homogenisation, 25 litre of the Vinyl Acetate Monomer was divided over 50 brown glass bottles of 500 mL and labelled #13005.
The homogeneity of the subsamples #13005 was checked by determination of Density in accordance with ASTM D4052:11 and water in accordance with ASTM D1364:12 on 8 stratified randomly selected samples.

	Density @20°C in kg/L	Water in mg/kg
sample #13005-1	0.93217	55
sample #13005-2	0.93214	66
sample #13005-3	0.93225	76
sample #13005-4	0.93222	75
sample #13005-5	0.93222	73
sample #13005-6	0.93223	68
sample #13005-7	0.93226	78
sample #13005-8	0.93223	81

table 1: homogeneity test results of subsamples #13005

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°C in kg/L	Water in mg/kg
r (sample #13005)	0.00011	23
reference test	D4052:02e1	ASTM D1364:12
0.3*R (reference test)	0.00015	15

table 2: repeatabilities of subsamples #13005

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 D1364:12 (r_{D1364}= 25). Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 1 bottle of 500 mL Vinyl Acetate Monomer, labelled #13005, was sent on January 30, 2013.

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 (with and without N_2 purging), Appearance, Density @ 20°C, Distillation (IBP, 50% recovery, Dry Point, Boiling Range), Ethyl Acetate, Inhibitor, Methyl Acetate, Purity and Water.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website (www.iisnl.com).

A SDS and a form to confirm receipt of the samples were added to the sample 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

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 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. In case a data set does not have a normal distribution, the (results of 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 and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the 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 these 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; nos.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, some problems were encountered with despatch of the samples. Participants in Australia, Brazil, India, Thailand, United Arab Emirates and USA received the samples late or not at all due to problems at customs. Of the 26 participants, 5 participants reported results after the final reporting date and 2 laboratories had not received the sample still, due to customs clearance problems.
Not all participants were able to report results for all the requested tests. Finally, 24 participants reported in total 243 numerical results. Observed were 9 outlying test results, which is 3.7% 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 Acidity (without purging) and Distillation (IBP, 50% recovery, DP and Boiling Range). In these cases the statistical evaluation should be used with due care.

Acetaldehyde: This determination may be problematic. One statistical outlier was observed and the reproducibility is in agreement with ASTM D2191:12. However, this reproducibility is based on 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 #13005 and therefore the reproducibility according to ASTMD2191:12 may not be reliable. When the Horwitz estimate for the target reproducibility is used for evaluation then the conclusion is that this method was problematic.

Acetone: All of the reporting participants agreed on a result below 10 mg/kg. Therefore no significant statistical conclusions were drawn.

Acidity: This determination (with and without Nitrogen purging) may be very problematic. It is remarkable to find that the acidity after purging is not significantly lower than the acidity without purging.
Only one statistical outlier was observed. Both calculated reproducibilities, after rejection of the statistical outliers, are not at all in agreement with the requirements of ASTM D2086:12. One should keep in mind that the reproducibility of ASTM D2086 was determined with only two laboratories and therefore may not be very reliable (see note 5 in §13.2.2). ASTM D2086 leaves the solvent to be used optional, which

may be a source of spread. Also the required pre-titration of the solvent may be a source of spread.
When the Horwitz estimate for the target reproducibility is used for evaluation than the conclusion is that this method was problematic.

Appearance: No analytical problems were observed. All laboratories agreed about the appearance of the sample, although different ways of reporting were used. Seventeen laboratories reported the appearance as pass in agreement with ASTM E2680. Other laboratories reported Bright & Clear, Clear & Free and Clear & Free from Suspended Matter.

Density @ 20°C: This determination was 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 the reproducibility of 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 (Automated method).

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.

Inhibitor: This determination was problematic. One statistical outlier was observed.
(Hydroquinone)   The calculated reproducibility, after rejection of the statistical outlier is not in agreement with the requirements of ASTM D2193:12.

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

Purity: Regretfully, no reference method with precision data exists for this determination. No statistical outliers were observed. The calculated reproducibility is small in comparison with the spread found in the previous PT (iis11C03).

Water: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility is in agreement with the calculated reproducibility with the requirements of ASTM D1364:12.

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)
Acetaldehyde	mg/kg	16	25.7	10.9	80.0*
Acetone	mg/kg	16	< 10	n.e.	n.e.
Acidity (without N_2 purging)	mg/kg	15	19.6	12.0	6.0*
Acidity (with N_2 purging)	mg/kg	8	18.5	12.5	6.0*
Appearance	------	24	pass	-----	-----
Density @ 20°C	kg/L	24	0.9322	0.0002	0.0005
Distillation					
-Initial Boiling Point	ºC	19	72.45	0.20	1.13
-50% recovery	ºC	19	72.71	0.18	0.50
-Dry Point	ºC	18	72.81	0.22	0.78
-Boiling Range	ºC	18	0.35	0.24	0.69
Ethylacetate	mg/kg	19	138.3	31.2	29.5
Inhibitor	mg/kg	20	3.56	1.82	1.00
Methyl Acetate	mg/kg	14	9.8	3.8	3.1
Purity	%M/M	22	99.98	0.02	unknown
Water	mg/kg	20	72.1	52.9	50.9

table 3: performance evaluation sample #13005

*see §4.1 (page 8)

4.3   EVALUTION OF THE PROFICIENCY TEST OF FEBRUARY 2013 WITH PREVIOUS PTS

	February 2013	February 2012	April 2011	March 2010
Number of rep. participants	24	24	28	21
Number of results reported	243	257	264	201
Number of statistical outliers	9	8	16	8
Percentage outliers	3.7%	3.1%	6.1%	4.0%

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 2013	February 2012	April 2011	March 2010
Acetaldehyde	++	++	++	++
Acetone	n.e.	n.e.	n.e	n.e
Acidity (no purging)	--	--	--	--
Acidity (with purging)	--	+/-	--	--
Density @ 20°C	++	++	++	++
Distillation	++	++	++	++
Ethyl Acetate	+/-	+/-	--	++
Inhibitor	-	--	-	--
Methyl Acetate	-	++	--	n.e
Purity	n.e.	n.e.	n.e	n.e
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 Acetaldehyde on sample #13005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-GC	25.5		-0.01	
173	D2191	26.31		0.02	
174	D3545	20.2		-0.19	
311	INH-122	25		-0.02	
315		-----		-----	
323	INH-067	23		-0.09	
347	INH-96	49	G(0.01)	0.82	
395	INH-47	19.55		-0.21	
446		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
657	D2191	32.37		0.23	
662	INH-40	25.4		-0.01	
786	INH-004	29		0.12	
823	D2191	26		0.01	
855	D2191	30		0.15	
859	D2191	31.2		0.19	
861	INH-1628	26		0.01	
865	D2191	27		0.05	
902		-----		-----	
913		-----		-----	
963	D2191	19		-0.23	
974		-----		-----	
1107		-----		-----	
1429	INH-CM	25		-0.02	

normality	OK	
n	16	
outliers	1	
mean (n)	25.66	
st.dev. (n)	3.896	
R(calc.)	10.91	
R(D2191:12)	80.00	Compare R(Horwitz) = 7.05
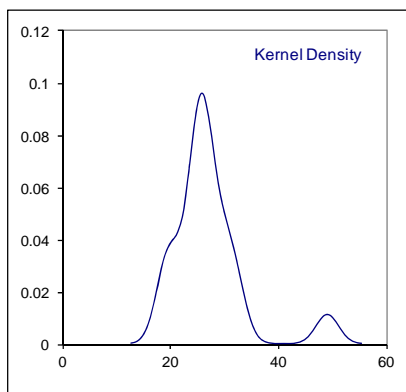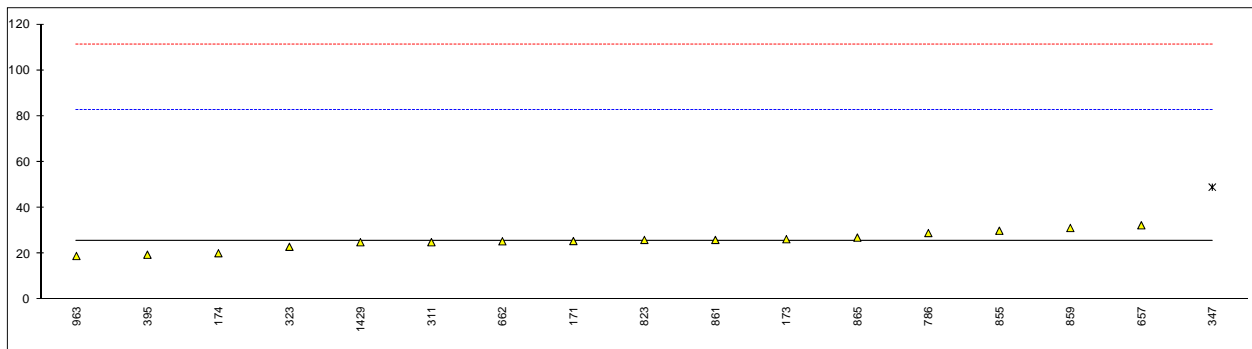
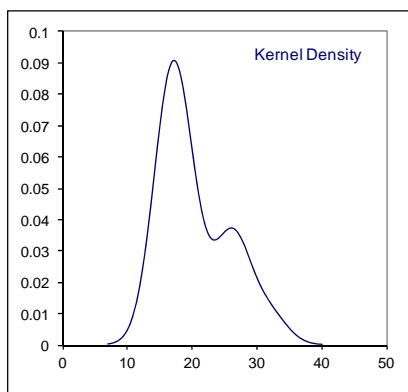Determination of Acetone on sample #13005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-GC	<10		-----	
173	INH-257	<0.01		-----	
174	D3545	<10		-----	
311	INH-122	<10		-----	
315		-----		-----	
323	INH-067	<10		-----	
347	INH-96	<10		-----	
395		-----		-----	
446		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
657	INH-0047	<10		-----	
662	INH-40	3.6		-----	
786	INH-004	<10		-----	
823	D3545	<5		-----	
855	INH-1628	<10		-----	
859	INH-1628	<10		-----	
861		-----		-----	
865	INH-1628	<10		-----	
902		-----		-----	
913		-----		-----	
963	INH-009	8		-----	
974		-----		-----	
1107	in house	<10		-----	
1429	INH-CM	<10		-----	
	normality	n.e			
	n	16			
	outliers	0			
	mean (n)	<10			
	st.dev. (n)	n.e			
	R(calc.)	n.e			
	R(Horwitz)	n.e			

Dermination of Acidity (without N$_2$ purging) on sample #13005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2086	16.5		-1.46	
173	INH-44	32	G(0.05)	5.78	
174	D2086	17.8		-0.85	
311	D2086	16		-1.69	
315		-----		-----	
323	D2086	18		-0.76	
347	D2086	16		-1.69	
395	INH-44	25.93		2.95	
446		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
657	D2086	24.75		2.39	
662		-----		-----	
786	D2086	27		3.44	
823		-----		-----	
855	D2086	17.0		-1.22	
859	D2086	18.0		-0.76	
861	D2086	20.3		0.32	
865	D2086	16.5		-1.46	
902		-----		-----	
913		-----		-----	
963	D2086	27		3.44	
974		-----		-----	
1107	D2086	15		-2.16	
1429	D2086	18.5		-0.52	

|				
	normality	not OK		
	n	15		
	outliers	1		
	mean (n)	19.62		
	st.dev. (n)	4.303		
	R(calc.)	12.05		
	R(D2086:12)	6.00		Compare R(Horwitz) = 5.62
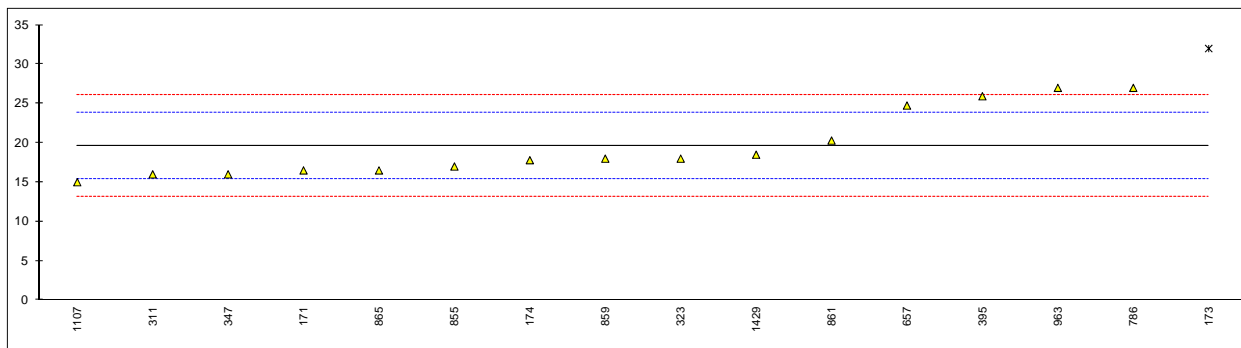
Determination of Acidity (with N_2 purging) on sample #13005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173		-----		-----	
174		-----		-----	
311		-----		-----	
315		-----		-----	
323		-----		-----	
347		-----		-----	
395		-----		-----	
446		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
657		-----		-----	
662		-----		-----	
786		-----		-----	
823	D2086	23		2.12	
855	D2086	16.3		-1.01	
859	D2086	17.2		-0.59	
861	D2086	18.9		0.20	
865	D2086	16.0		-1.15	
902		-----		-----	
913		-----		-----	
963	D2086	27		3.98	
974		-----		-----	
1107	D2086	16		-1.15	
1429	D2086	13.3		-2.41	
	normality	OK			
	n	8			
	outliers	0			
	mean (n)	18.46			
	st.dev. (n)	4.452			
	R(calc.)	12.46			
	R(D2086:12)	6.00		Compare R(Horwitz) = 5.33	
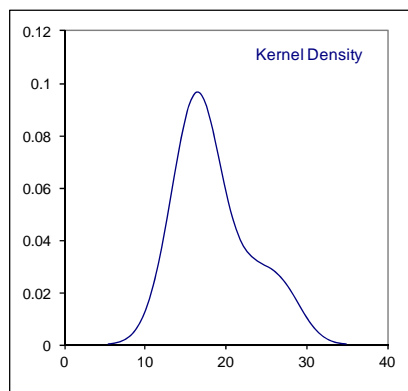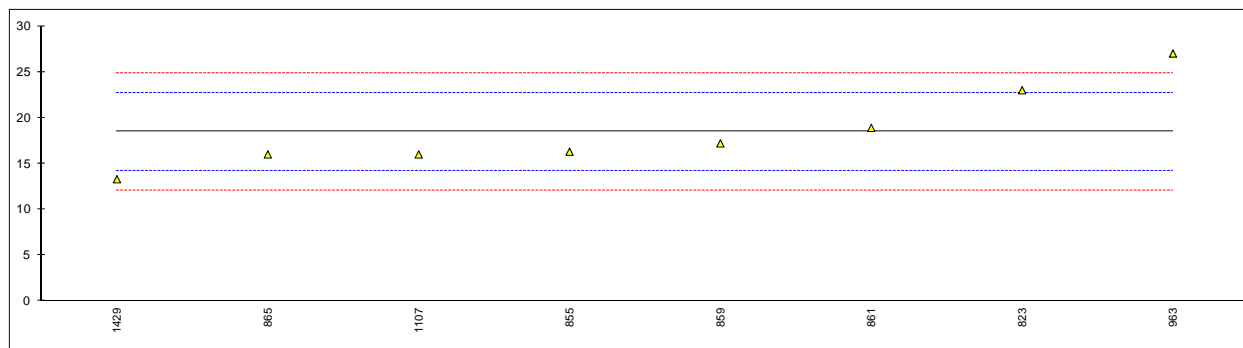
Determination of Appearance on sample #13005;

lab	method	value	mark	z(targ)	remarks
171	E2680	Pass		-----	
173	Visual	CFSM		-----	
174	E2680	C&F		-----	
311	E2680	Pass		-----	
315	E2680	Pass		-----	
323	E2680	CFFSM		-----	
347	E2680	Pass		-----	
395	E2680	Pass		-----	
446	E2680	Pass		-----	
522	Visual	CFSM		-----	
551		-----		-----	
613	INH-40	B&C		-----	
657	E2680	Pass		-----	
662	E2680	Pass		-----	
786	E2680	Pass		-----	
823	E2680	Pass		-----	
855	E2680	Pass		-----	
859	E2680	Pass		-----	
861	E2680	B&C		-----	
865	E2680	Pass		-----	
902	E2680	Pass		-----	
913		-----		-----	
963	E2680	Pass		-----	
974	E2680	Pass		-----	
1107	E2680	Pass		-----	
1429	E2680	B&C		-----	
	n	24			
	mean (n)	Pass			

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

Determination of Density @ 20 °C on sample #13005; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9321		-0.56	
173	D4052	0.93217		-0.17	
174	D4052	0.9321		-0.56	
311	D4052	0.9322		0.00	
315	D4052	0.9322		0.00	
323	D4052	0.9323		0.56	
347	D4052	0.9322		0.00	
395	D4052	0.9322		0.00	
446	D4052	0.9322		0.00	
522	INH-40	0.9324		1.12	
551		-----		-----	
613	D4052	0.93224		0.22	
657	D4052	0.93213		-0.39	
662	D4052	0.93209		-0.62	
786	D4052	0.9321		-0.56	
823	D4052	0.9322		0.00	
855	D4052	0.93218		-0.11	
859	D4052	0.93221		0.06	
861	D4052	0.9322		0.00	
865	D4052	0.93224		0.22	
902	D4052	0.93224		0.22	
913		-----		-----	
963	D4052	0.9321		-0.56	
974	D4052	0.9321		-0.56	
1107	D4052	0.9323		0.56	
1429	D4052	0.9324		1.12	

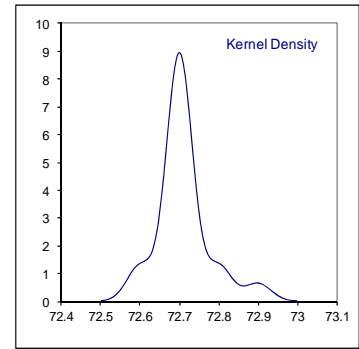|	
normality	OK
n	24
outliers	0
mean (n)	0.93220
st.dev. (n)	0.000087
R(calc.)	0.00024
R(D4052:02e1)	0.00050
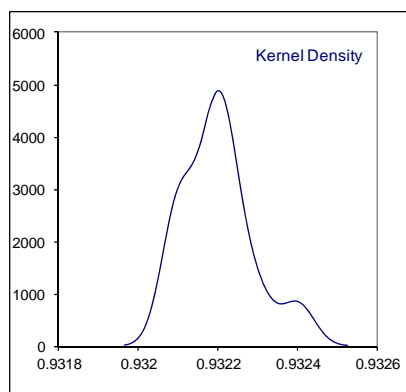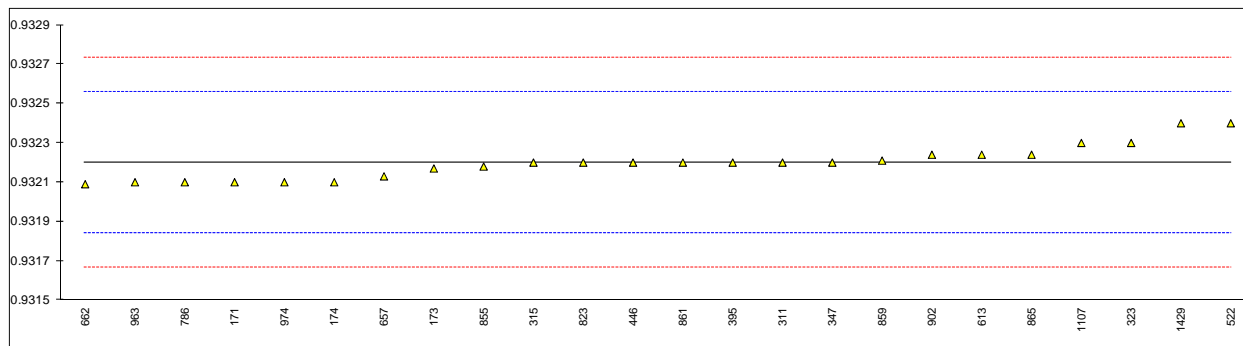
Determination of Distillation on sample #13005; results in °C

lab	method	IBP	mark	50% rec.*	mark	DP	mark	range	mark
171	D1078	72.5		72.7		72.8		0.3	
173	D1078	72.6		72.8		72.8		0.2	
174	D1078	72.5		72.7		72.8		0.3	
311	D1078	72.4		72.7		72.7		0.3	
315		-----		-----		-----		-----	
323	D1078	72.5		72.7		72.9		0.4	
347	D1078	72.5		72.7		72.9		0.4	
395	D1078	72.4		72.6		72.8		0.4	
446	INH-45	72.5		72.9		73.0		0.5	
522		-----		-----		-----		-----	
551		-----		-----		-----		-----	
613		-----		-----		-----		-----	
657	D1078	72.4		72.7		73.2	G(0.01)	0.8	G(0.01)
662	D1078	72.6		72.7		72.8		0.2	
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	72.4		72.7		72.8		0.4	
861	D1078	72.4		72.7		72.8		0.4	
865	D1078	72.4		72.7		72.8		0.4	
902	D1078	72.4		72.8		72.9		0.5	
913		-----		-----		-----		-----	
963	D1078	72.4		72.7		72.7		0.3	
974		-----		-----		-----		-----	
1107		-----		-----		-----		-----	
1429	D1078	72.4		72.7		72.7		0.3	
	normality	not OK		not OK		not OK		not OK	
	n	19		19		18		18	
	outliers	0		0		1		1	
	mean (n)	72.45		72.71		72.81		0.35	
	st.dev. (n)	0.070		0.066		0.080		0.086	
	R(calc.)	0.20		0.18		0.22		0.24	
	R(D1078:11)	1.13		0.50		0.78		0.69	

*Theoretical mid boiling point = 72.7 °C

z-scores of distillation

lab	IBP	50% rec	DP	range
171	0.12	-0.06	-0.02	-0.20
173	0.37	0.50	-0.02	-0.61
174	0.12	-0.06	-0.02	-0.20
311	-0.13	-0.06	-0.38	-0.20
315	-----	-----	-----	-----
323	0.12	-0.06	0.34	0.20
347	0.12	-0.06	0.34	0.20
395	-0.13	-0.62	-0.02	0.20
446	0.12	1.07	0.70	0.61
522	-----	-----	-----	-----
551	-----	-----	-----	-----
613	-----	-----	-----	-----
657	-0.13	-0.06	1.42	1.82
662	0.37	-0.06	-0.02	-0.61
786	-0.13	-0.62	-0.38	-0.20
823	-0.13	-0.06	-0.02	0.20
855	0.12	-0.06	-0.02	-0.20
859	-0.13	-0.06	-0.02	0.20
861	-0.13	-0.06	-0.02	0.20
865	-0.13	-0.06	-0.02	0.20
902	-0.13	0.50	0.34	0.61
913	-----	-----	-----	-----
963	-0.13	-0.06	-0.38	-0.20
974	-----	-----	-----	-----
1107	-----	-----	-----	-----
1429	-0.13	-0.06	-0.38	-0.20

IBP

Kernel Density

50% recov

Kernel Density

Dry point

Kernel Density

Distillation range

Kernel Density
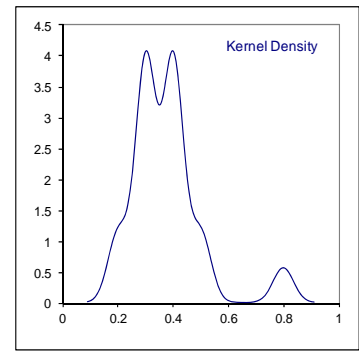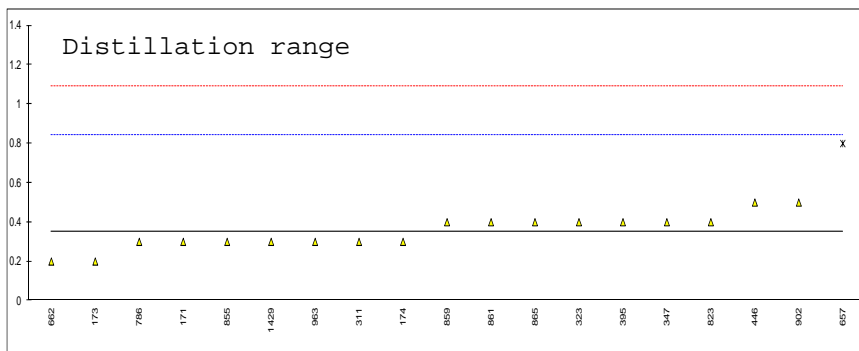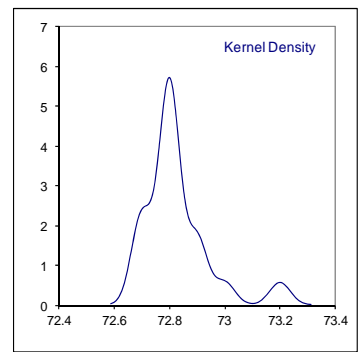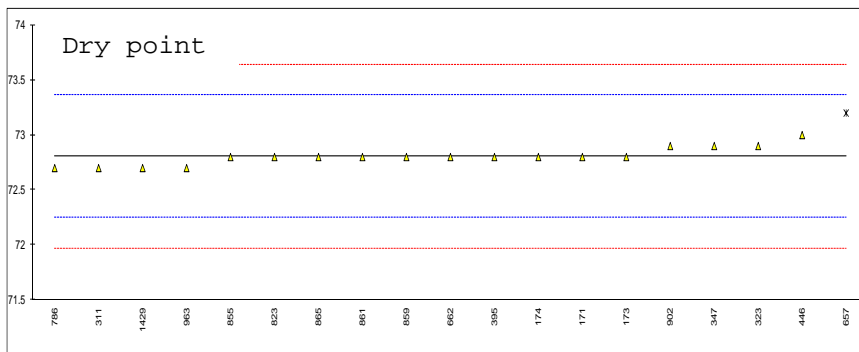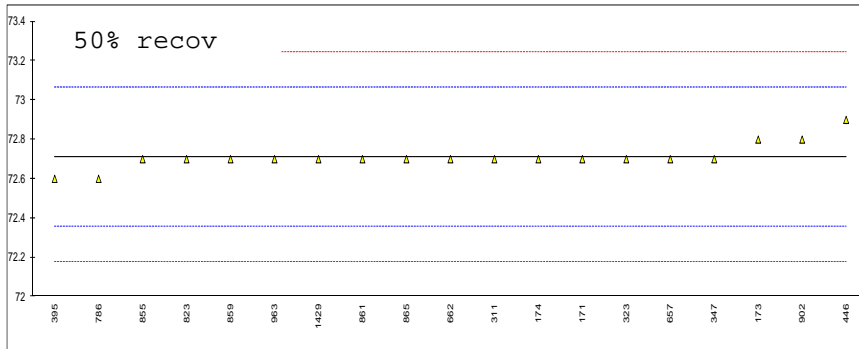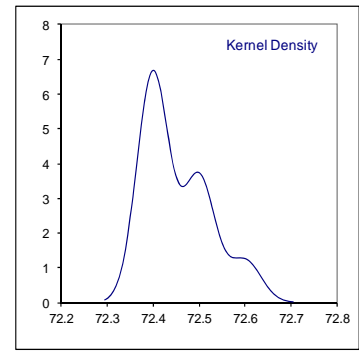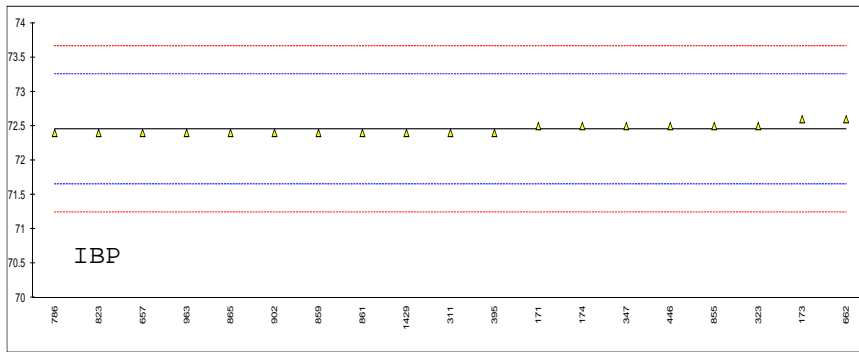
Determination of Ethyl Acetate on sample #13005, results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-GC	145.5		0.68	
173	INH-257	138.93		0.06	
174	D3545	133		-0.50	
311	INH-122	160		2.06	
315		-----		-----	
323	INH-067	130		-0.79	
347	INH-96	142		0.35	
395	INH-47	123.99		-1.36	
446	INH-47	138		-0.03	
522		-----		-----	
551		-----		-----	
613		-----		-----	
657	INH-0047	142		0.35	
662	INH-40	139.0		0.06	
786	INH-004	114		-2.31	
823	D3545	137		-0.12	
855	INH-1628	140.1		0.17	
859	INH-1628	153.5		1.44	
861		-----		-----	
865	INH-1628	142		0.35	
902		-----		-----	
913		-----		-----	
963	INH-009	139		0.06	
974	INH-03	151		1.20	
1107	in house	119		-1.83	
1429	INH-CM	140		0.16	

|	
normality	OK
n	19
outliers	0
mean (n)	138.32
st.dev. (n)	11.131
R(calc.)	31.17
R(Horwitz)	29.51

Compare R(3545:06) = 900 mg/kg at concentration 1000 mg/kg
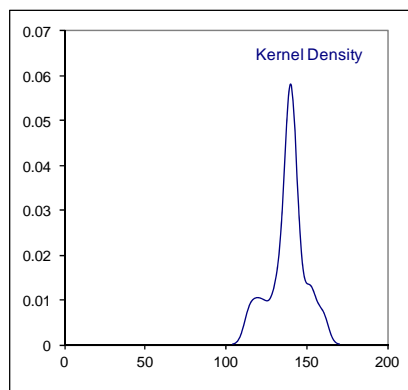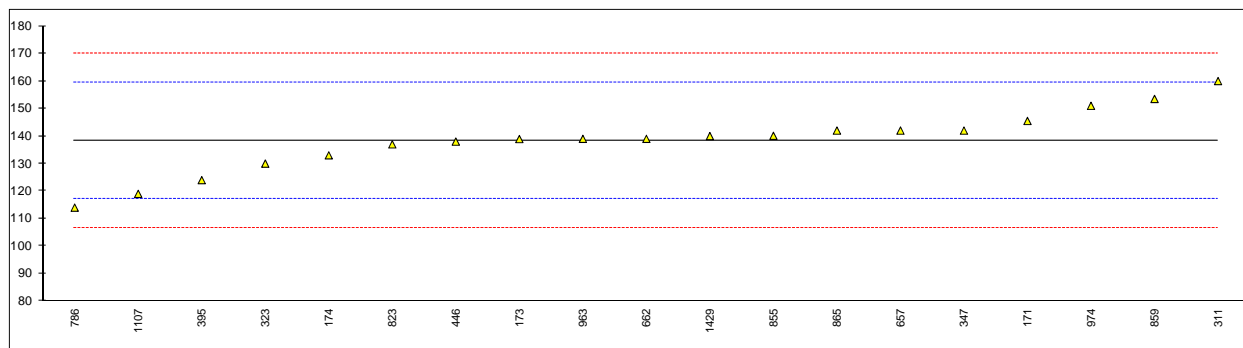
Determination of Inhibitor (Hydroquinone) on sample #13005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2193	4.6		2.90	
173	D2193	3.85		0.80	
174	D2193	4.3		2.06	
311	D2193	3.9		0.94	
315		-----		-----	
323	D2193	3.3		-0.74	
347	INH-97	4.6		2.90	
395	INH-43	3.14		-1.18	
446		-----		-----	
522	INH-40	0.37	G(0.01)	-8.94	
551		-----		-----	
613	INH-40	2.41		-3.23	
657	D2193	4.28		2.01	
662	D2193	2.49		-3.00	
786	D2193	3.5		-0.18	
823	D2193	2.6		-2.70	
855	D2193	3.65		0.24	
859	D2193	3.92		1.00	
861	D2193	3.46		-0.29	
865	D2193	4.0		1.22	
902		-----		-----	
913		-----		-----	
963	D2193	2.9		-1.86	
974	D2193	3.36		-0.57	
1107	in house	3.8		0.66	
1429	D2193	3.2		-1.02	

normality	OK
n	20
outliers	1
mean (n)	3.563
st.dev. (n)	0.6517
R(calc.)	1.825
R(D2193:12)	1.000
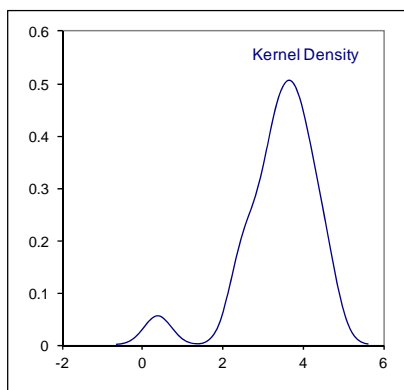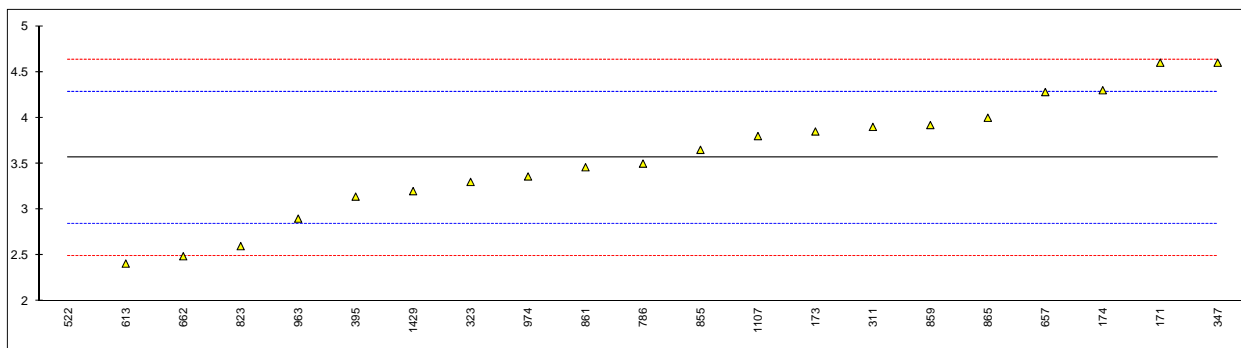
Determination of Methyl Acetate on sample #13005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-GC	8.4		-1.29	
173	INH-257	10.36		0.47	
174	D3545	8.0		-1.65	
311	INH-122	13		2.83	
315		-----		-----	
323	INH-067	<10		-----	
347	INH-96	<10		-----	
395	INH-47	5.35	G(0.05)	-4.02	
446	INH-47	9		-0.75	
522		-----		-----	
551		-----		-----	
613		-----		-----	
657	INH-0047	10		0.14	
662	INH-40	9.7		-0.13	
786	INH-004	10		0.14	
823	D3545	8		-1.65	
855	INH-1628	9.1		-0.66	
859	INH-1628	11.2		1.22	
861		-----		-----	
865	INH-1628	10		0.14	
902		-----		-----	
913		-----		-----	
963	INH-009	11		1.04	
974		-----		-----	
1107	in house	<10		-----	
1429	INH-CM	10		0.14	

normality	OK
n	14
outliers	1
mean (n)	9.84
st.dev. (n)	1.349
R(calc.)	3.78
R(Horwitz)	3.12
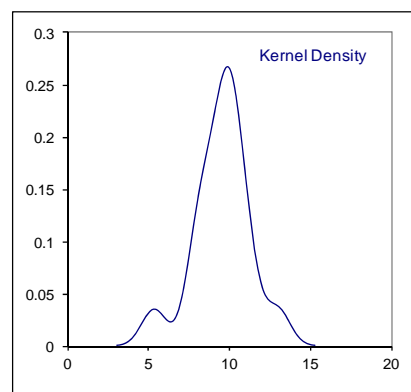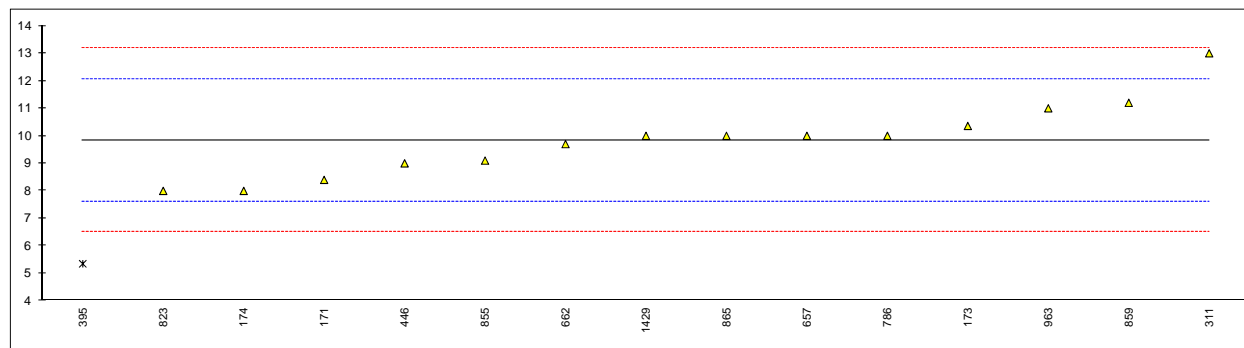
Determination of Purity on sample #13005; results in % M/M

lab	method	value	mark	z(targ)	remarks
171	INH-GC	99.974		-----	
173	INH-257	99.9816		-----	
174	D3545	99.974		-----	
311	INH-122	99.98		-----	
315		-----		-----	
323	INH-067	99.97		-----	
347	INH-96	99.981		-----	
395	INH-257	99.979		-----	
446	INH-257	99.975		-----	
522	INH-40	99.99		-----	
551		-----		-----	
613	INH-40	99.983		-----	
657	INH-0047	99.9778		-----	
662	INH-40	99.97		-----	
786	INH-004	99.98		-----	
823	D3545	99.977		-----	
855	INH-1628	99.970		-----	
859	INH-1628	99.978		-----	
861	INH-1628	99.972		-----	
865	INH-1628	99.978		-----	
902		-----		-----	
913		-----		-----	
963	INH-009	99.97		-----	
974	INH-03	99.976		-----	
1107	in house	99.96		-----	
1429	INH-CM	99.98		-----	

normality	OK	
n	22	
outliers	0	
mean (n)	99.9762	
st.dev. (n)	0.00615	
R(calc.)	0.0172	
R(Lit)	unknown	Compare R(iis11C03) = 0.0298
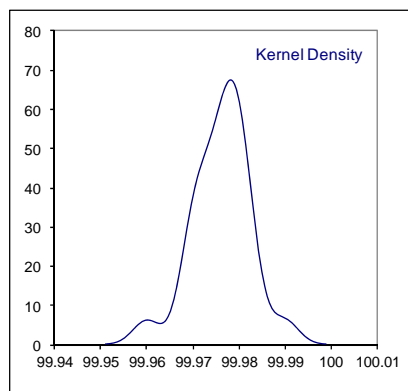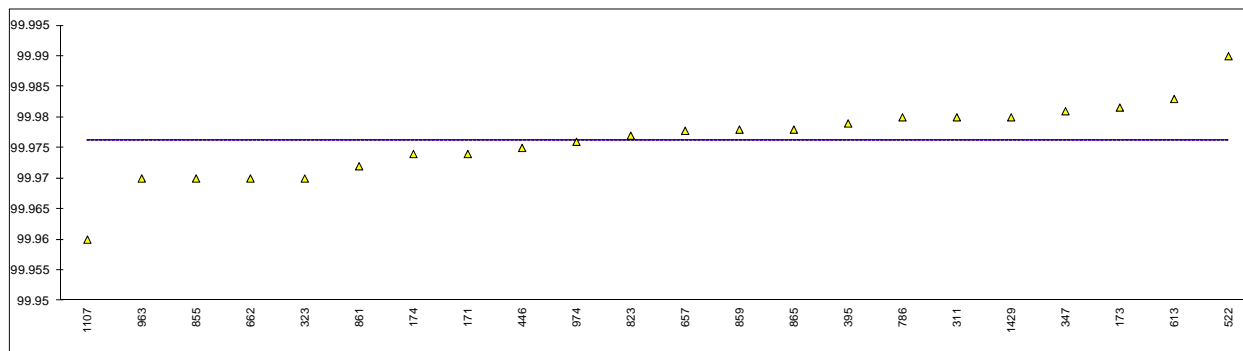
Determination of Water on sample #13005; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	72.5		0.02	
173	E203	48		-1.32	
174	D1364	96		1.32	
311	D1364	83		0.60	
315	D1364	64.75		-0.40	
323	D1364	96		1.32	
347	D1364	33		-2.15	
395	D1364	57.45		-0.80	
446	INH-63	60		-0.66	
522	INH-40	136	DG(0.05)	3.51	
551		-----		-----	
613		-----		-----	
657	E1064	91.01		1.04	
662	D1364	98		1.43	
786	D1364	58		-0.77	
823	D1364	62		-0.55	
855	D1364	81.2		0.50	
859	D1364	84		0.66	
861	E1064	71		-0.06	
865	D1364	80		0.44	
902	D1364	57.25		-0.81	
913		-----		-----	
963	D1364	50		-1.21	
974	D1364	155	DG(0.05)	4.56	
1107	D1364	224	G(0.01)	8.35	
1429	D1364	98.1		1.43	

normality	OK	
n	20	
outliers	3	
mean (n)	72.06	
st.dev. (n)	18.902	
R(calc.)	52.92	
R(D1364:12)	50.93	

Kernel Density
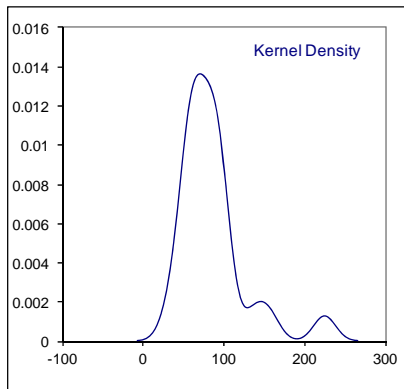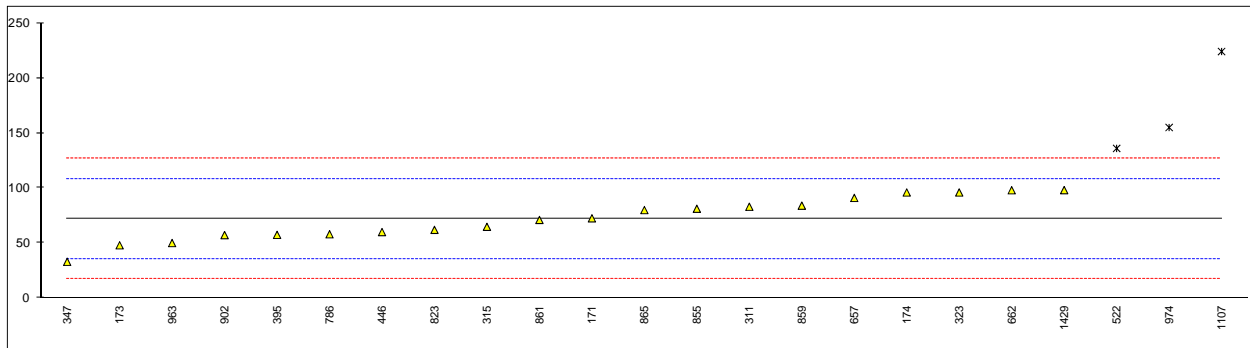
APPENDIX 2

Number of participants per country

1 lab in	AUSTRALIA
2 labs in	BELGIUM
1 lab in	BRAZIL
1 lab in	INDIA
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	SPAIN
1 lab in	THAILAND
2 labs 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/)