Results of Proficiency Test
Vinyl Acetate Monomer
April 2011

Organised by: Institute for Interlaboratory Studies
                Spijkenisse, the Netherlands

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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 2010/2011, it was decided to continue the round robin for the analysis of Vinyl Acetate Monomer. In this interlaboratory study 28 laboratories in 21 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 guide 43 and ILAC-G13:2007. 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 Acid was obtained from a chemical producer. After homogenisation, 18 litre of the Vinyl Acetate Monomer was divided over 36 brown glass bottles of 500 mL and labelled #11115.
The homogeneity of the subsamples #11115 was checked by determination of Density in accordance with ASTM D4052:09 and water in accordance with ASTM D1364:07 on 4 stratified randomly selected samples.

	Density in kg/L	Water in mg/kg
sample #11115-1	0.93212	164
sample #11115-2	0.93218	182
sample #11115-3	0.93224	181
sample #11115-4	0.93218	193

table 1: homogeneity test results of subsamples #11115

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 in kg/L	Water in mg/kg
r (sample #11115)	0.00014	33.5
reference test	D4052:09	ASTM D1364:07
0.3*R (reference test)	0.00015	24.1

table 2: repeatabilities of subsamples #11115

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}= 40.3). Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 1 bottle of 500 mL Vinyl Acetate Monomer (sample #11115) was sent on April 5, 2011.

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 additional tests: Acetaldehyde, Acetone, Acidity, Density @ 20 $^{\circ}$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. 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; 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.
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 28 participants, 4 participants reported results after the final reporting date. Not all participants were able to report results for all the requested tests.
In total 264 numerical test results were reported. Observed were 16 outlying test results, which is 6.1% 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 Density and Distillation (IBP, Mid Boiling Point, DP and Boiling Range). In these cases the statistical evaluation should be used with due care.

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 range and therefore the reproducibility according to ASTMD2191:06 may not be reliable. When Horwitz is used for evaluation than this method is problematic.

Acetone: No numerical results were reported. Therefore no significant statistical conclusions were drawn. All reporting participants agreed on a result below 10.

Acidity: This determination (with and without Nitrogen purging) was very problematic. It is remarkable to find that the acidity after purging is not significantly lower than the acidity without purging.
In total three statistical outliers were observed. Both calculated reproducibilities, after rejection of the statistical outliers, are not at all in agreement with the requirements of ASTM D2086:08. 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.

Appearance: No analytical problems were observed. A new standardized method is available for Appearance since 2009, being ASTM E2680. According this method the appearance should be reported as 'pass' (or 'fail'). All the laboratories reported according this method, except three. Thirteen laboratories reported the appearance as pass. Five laboratories reported the appearance as Clear and Free from Suspended Matter (CFSM), four laboratories reported the appearance as Bright and Clear and one as Clear.

Density @ 20 ^{o}C: This determination is not problematic. One statistical outlier was observed and the calculated reproducibility is in good agreement with the requirements of ASTM D4052:09. This method describes the density for a range from 0.80 g/ml to 0.88 g/ml. However, the density of VAM is outside this range. The range of previous method, the ASTM D4052:02e, was 0.68 g/ml to 0.97 g/ml. Both methods have the same reproducibility.

Distillation: This determination is not problematic. In total five statistical outliers were observed. However, the calculated reproducibilities, after rejection of the statistical outliers, are all in good agreement with the requirements of ASTM D1078:05.

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

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

Methyl Acetate: This determination may be problematic. No statistical outliers were observed. But the calculated reproducibility is not in agreement with the requirements of the Horwitz equation.

Purity: Regretfully, no suitable reference method with precision data exists for this determination. Two statistical outliers are observed and compared with the spread found in the previous PT (iis09C03), the calculated reproducibility, after rejection of the statistical outlier, is large. Perhaps not all participants reported the purity on dry basis.

Water: This determination is not problematic. Three statistical outliers were observed. However, the calculated reproducibility, after rejection of the statistical outliers, is in full 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)
Acetaldehyde	mg/kg	16	25.73	18.78	80.00*
Acetone	mg/kg	17	< 10	n.a	n.a
Acidity (without N_2 purging)	mg/kg	18	24.4	15.8	6.0
Acidity (with N_2 purging)	mg/kg	12	24.9	10.8	6.0
Appearance	------	23	----	-----	-----
Density @ 20°C	kg/L	25	0.9320	0.0002	0.0005
Ethylacetate	mg/kg	20	186.8	45.9	38.1
Initial Boiling Point	ºC	19	72.41	0.24	1.13
50% recovery	ºC	17	72.70	0.00	0.50
Dry Point	ºC	19	72.83	0.20	0.78
Boiling Range	ºC	19	0.42	0.21	0.70
Inhibitor	mg/kg	19	4.24	1.26	1.00
Methyl Acetate	mg/kg	18	24.3	9.9	6.7
Purity	%M/M	23	99.97	0.03	unknown
Water	mg/kg	23	154.8	64.3	74.6

table 3: performance evaluation sample #11115

*see §4.1 (page 7)

4.3 EVALUATION OF THE PROFICIENCY TEST OF APRIL 2011 WITH PREVIOUS PTS

	April 2011	March 2010	March 2009
Number of rep. participants	28	21	26
Number of results reported	264	201	239
Statistical outliers	16	8	10
Percentage outliers	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:

	April 2011	March 2010	March 2009
Acetaldehyde	++	++	++
Acetone	n.e	n.e	n.e
Acidity as Acetic Acid	--	--	--
Density @ 20°C	++	++	++
Distillation	++	++	++
Ethyl Acetate	--	++	++
Inhibitor	-	--	--
Methyl Acetate	--	n.e	++
Purity	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 #11115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-256	22.3		-0.12	
173	INH-47	23.8		-0.07	
174		-----		-----	
311	D2191	17.35		-0.29	
323	D2191	<10		-----	False negative result?
347	INH-916	20		-0.20	
395	INH-47	27.110		0.05	
446	INH-47	38		0.43	
522		-----		-----	
551		-----		-----	
562		-----		-----	
613		-----		-----	
657	D2191	19		-0.24	
662	INH-40	27.20		0.05	
786	INH-004	27		0.04	
823	D2191	22		-0.13	
855	D2191	32		0.22	
859	D2191	31		0.18	
861	D2191	18		-0.27	
865	D2191	34		0.29	
887		-----		-----	
902		-----		-----	
913		-----		-----	
963	D2191	18		-0.27	
974		-----		-----	
1429	D2191	35	C	0.32	First reported 44
1449		-----		-----	
1622		-----		-----	

normality	OK	
n	16	
outliers	0	
mean (n)	25.74	
st.dev. (n)	6.708	
R(calc.)	18.78	
R(D2191:06)	80.00	Compare R(Horwitz) = 7.07
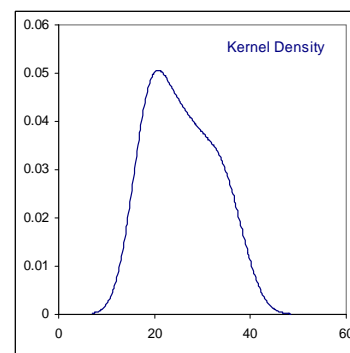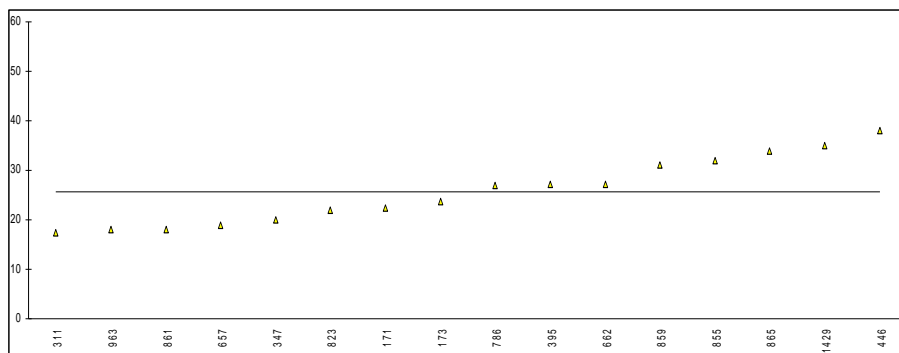
Determination of Acetone on sample #11115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-256	<1		-----	
173	INH-47	<10		-----	
174	D3545	<10		-----	
311	INH-122	<10		-----	
323	INH-067	<10		-----	
347	INH-916	<10		-----	
395	INH-47	<10		-----	
446		-----		-----	
522		-----		-----	
551		-----		-----	
562	INH-001	<10	C	-----	First reported < 0.001
613		-----		-----	
657		<10		-----	
662	INH-40	n.d.		-----	
786	INH-004	<10		-----	
823	D3545	<1		-----	
855	INH-065	<10		-----	
859	INH-065	<10		-----	
861		-----		-----	
865	INH-065	<10		-----	
887		-----		-----	
902		-----		-----	
913		-----		-----	
963	INH-009	<5		-----	
974		-----		-----	
1429	in house	<10		-----	
1449		-----		-----	
1622		-----		-----	

normality	n.e	
n	17	
outliers	0	
mean (n)	<10	
st.dev. (n)	n.e	
R(calc.)	n.e	
R(Horwitz)	n.e	

Determination of Acidity (without N_2 purging) on sample #11115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2086	19.3		-2.38	
173	INH-44	10		-6.72	
174	D2086	19.0		-2.52	
311	D2086	27	C	1.22	First reported 0.0027
323	D2086	33		4.02	
347	D2086	25		0.28	
395	INH-44	24.93		0.25	
446	INH-44	18.7		-2.66	
522		-----		-----	
551		-----		-----	
562	D2086	30	C	2.62	First reported 0.0030
613		-----		-----	
657	D2086	24.0		-0.18	
662		-----		-----	
786	D2086	25.6		0.56	
823	D2086	28		1.68	
855	D2086	25.0		0.28	
859	D2086	26		0.75	
861	D2086	27.2		1.31	
865	D2086	24.8		0.19	
887		-----		-----	
902		-----		-----	
913	D2086	18.5		-2.75	
963		-----		-----	
974	D2086	51	C,G(0.01)	12.42	First reported 0.0051
1429	D2086	33		4.02	
1449		-----		-----	
1622		-----		-----	

normality	OK	
n	18	
outliers	1	
mean (n)	24.39	
st.dev. (n)	5.646	
R(calc.)	15.81	
R(D2086:08)	6.00	
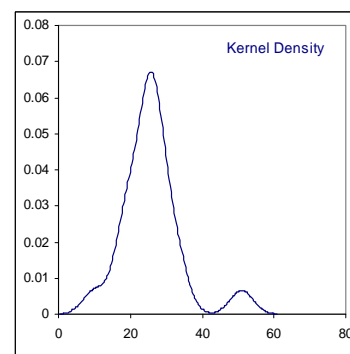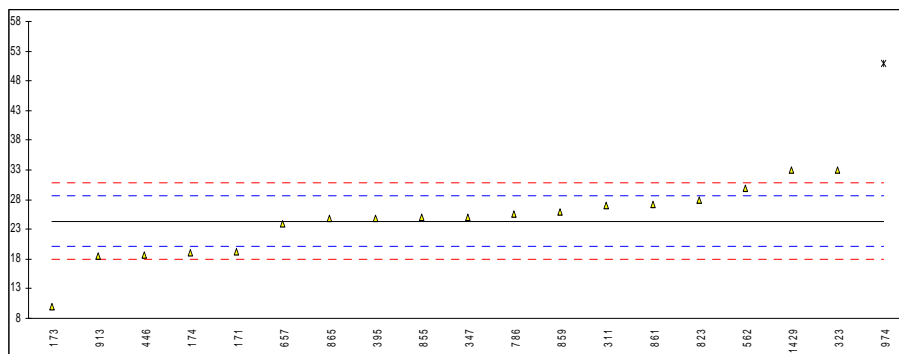
Determination of Acidity (with N_2 purging) on sample #11115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171		-----		-----	
173		-----		-----	
174		-----		-----	
311	D2086	25	C	0.03	First reported 0.0025
323	D2086	8	DG(0.05)	-7.91	
347		-----		-----	
395		-----		-----	
446		-----		-----	
522	INH-40	29.3		2.03	
551		-----		-----	
562		-----		-----	
613		-----		-----	
657	D2086	19.7		-2.45	
662	INH-40	27		0.96	
786		-----		-----	
823	D2086	17		-3.71	
855	D2086	24.1		-0.39	
859	D2086	24		-0.44	
861	D2086	24.3		-0.30	
865	D2086	22.9		-0.95	
887		-----		-----	
902	D2086	11	DG(0.05)	-6.51	
913		-----		-----	
963		-----		-----	
974	D2086	28	C	1.43	First reported 0.0028
1429	D2086	28		1.43	
1449	D2086	30	C	2.36	First reported 260
1622		-----		-----	

normality	OK
n	12
outliers	2
mean (n)	24.94
st.dev. (n)	3.864
R(calc.)	10.82
R(D2086:08)	6.00
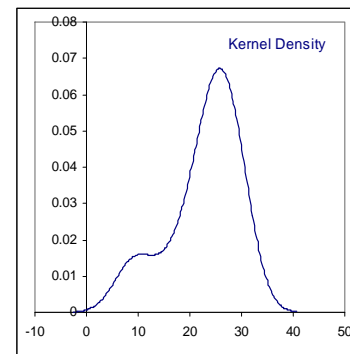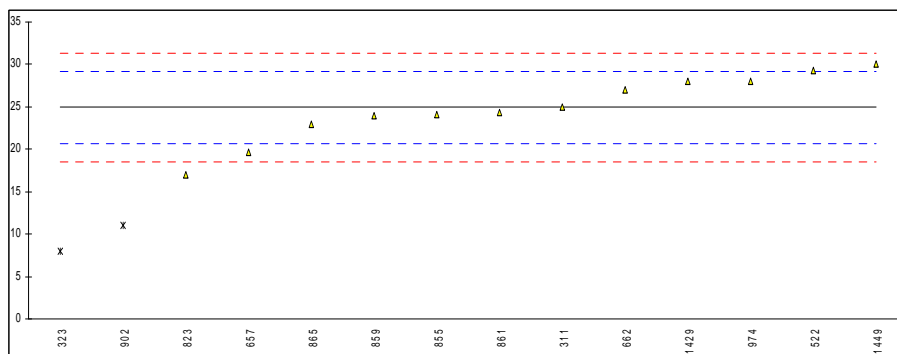
Determination of Appearance on sample #11115;

| lab	method	value	mark	z(targ)	remarks
171	E2680	B&C		-----	
173	E2680	CFSM		-----	
174	E2680	PASS		-----	
311	E2680	PASS		-----	
323	E2680	CFFSM		-----	
347	E2680	PASS		-----	
395	E2680	PASS		-----	
446	INH-500	PASS		-----	
522	visual	CFSM		-----	
551		-----		-----	
562	E2680	PASS		-----	
613	INH-40	B&C		-----	
657	E2680	PASS		-----	
662	E2680	CLEAR		-----	
786	E2680	PASS		-----	
823	E2680	PASS		-----	
855	E2680	PASS		-----	
859	E2680	PASS		-----	
861	E2680	B&C		-----	
865	E2680	PASS		-----	
887		-----		-----	
902	E2680	CFFSM		-----	
913	E2680	CFFSM		-----	
963	E2680	PASS		-----	
974		-----		-----	
1429	E2680	B&C		-----	
1449		-----		-----	
1622		-----		-----	

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

Determination of Density @ 20 ^{o}C on sample #11115; results in kg/L

lab	method	value	mark	z(targ)	remarks
171	D4052	0.9321		-0.51	
173	D4052	0.9322		0.05	
174	D4052	0.9322		0.05	
311	D4052	0.9322		0.05	
323	D4052	0.9323		0.61	
347	D4052	0.9322		0.05	
395	D4052	0.9321		-0.51	
446	INH-76	0.9322		0.05	
522		-----		-----	
551		-----		-----	
562	D4052	0.9315	G(0.01)	-3.87	
613	D4052	0.93220		0.05	
657	D4052	0.93210		-0.51	
662	D4052	0.9322		0.05	
786	D4052	0.9321		-0.51	
823	D4052	0.9321		-0.51	
855	D4052	0.93220		0.05	
859	D4052	0.93219		0.00	
861	D4052	0.93215		-0.23	
865	D4052	0.93221		0.11	
887	D4052	0.93223		0.22	
902	D4052	0.93220		0.05	
913	D4052	0.93217		-0.11	
963	D4052	0.9322		0.05	
974	D4052	0.9323		0.61	
1429	D4052	0.9323		0.61	
1449	D4052	0.9321		-0.51	
1622	D4052	0.9323		0.61	

|		
normality	not OK	
n	25	
outliers	1	
mean (n)	0.93219	
st.dev. (n)	0.000065	
R(calc.)	0.00018	
R(D4052:09)	0.00050	Compare R(D4052:02e1) = 0.0005
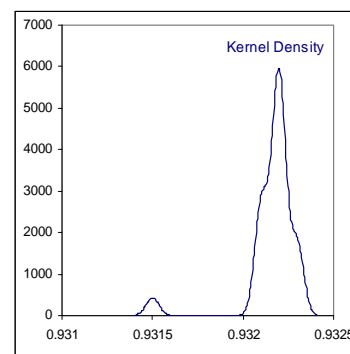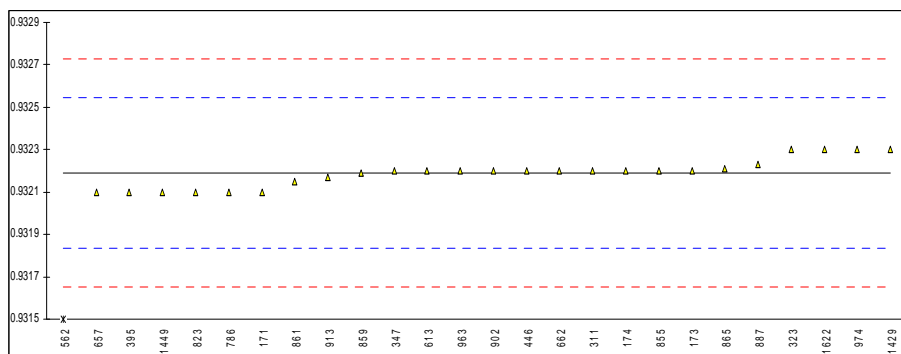
Determination of Distillation on sample #11115; results in ºC

lab	method	IBP	mark	50% rec.	mark	DP	mark	range	mark
171	D1078	72.4		72.7		72.8		0.4	
173	D1078	72.4		72.7		72.8		0.4	
174	D1078	72.6		72.8	G(0.01)	72.9		0.3	
311	D1078	72.2		72.7		72.8		0.6	
323	D1078	72.5		72.7		72.9		0.4	
347	D1078	72.5		72.7		72.9		0.4	
395	D1078	72.4		72.7		72.8		0.4	
446	INH-45	68.1	G(0.01)	-----		73.2	G(0.01)	5.1	G(0.01)
522		-----		-----		-----		-----	
551		-----		-----		-----		-----	
562	D1078	72.3		72.6	G(0.01)	72.7		0.4	
613		-----		-----		-----		-----	
657	D1078	72.3		72.7		72.7		0.4	
662		-----		-----		-----		-----	
786	D1078	72.50		72.70		72.80		0.30	
823	D1078	72.4		72.7		72.8		0.4	
855	D1078	72.4		72.7		72.8		0.4	
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		72.9		0.5	
887		-----		-----		-----		-----	
902	D1078	72.5		72.7		72.9		0.4	
913	D1078	72.4		72.7		72.8		0.4	
963	D1078	72.4		72.7		73.0		0.6	
974		-----		-----		-----		-----	
1429	D1078	72.4		72.7		72.8		0.4	
1449		-----		-----		-----		-----	
1622		-----		-----		-----		-----	
	normality	not OK		not OK		not OK		not OK	
	n	19		17		19		19	
	outliers	1		2		1		1	
	mean (n)	72.41		72.70		72.83		0.42	
	st.dev. (n)	0.088		0.000		0.073		0.076	
	R(calc.)	0.24		0.00		0.20		0.21	
	R(D1078:05)	1.13		0.50		0.78		0.70	

z-scores of distillation

lab	method	IBP	50% rec	DP	range
171	D1078	-0.03	0.00	-0.09	-0.06
173	D1078	-0.03	0.00	-0.09	-0.06
174	D1078	0.47	0.56	0.26	-0.46
311	D1078	-0.52	0.00	-0.09	0.74
323	D1078	0.22	0.00	0.26	-0.06
347	D1078	0.22	0.00	0.26	-0.06
395	D1078	-0.03	0.00	-0.09	-0.06
446	INH-45	-10.68	-----	1.34	18.72
522		-----	-----	-----	-----
551		-----	-----	-----	-----
562	D1078	-0.27	-0.56	-0.45	-0.06
613		-----	-----	-----	-----
657	D1078	-0.27	0.00	-0.45	-0.06
662		-----	-----	-----	-----
786	D1078	0.22	0.00	-0.09	-0.46
823	D1078	-0.03	0.00	-0.09	-0.06
855	D1078	-0.03	0.00	-0.09	-0.06
859	D1078	-0.03	0.00	-0.09	-0.06
861	D1078	-0.03	0.00	-0.09	-0.06
865	D1078	-0.03	0.00	0.26	0.34
887		-----	-----	-----	-----
902	D1078	0.22	0.00	0.26	-0.06
913	D1078	-0.03	0.00	-0.09	-0.06
963	D1078	-0.03	0.00	0.62	0.74
974		-----	-----	-----	-----
1429	D1078	-0.03	0.00	-0.09	-0.06
1449		-----	-----	-----	-----
1622		-----	-----	-----	-----
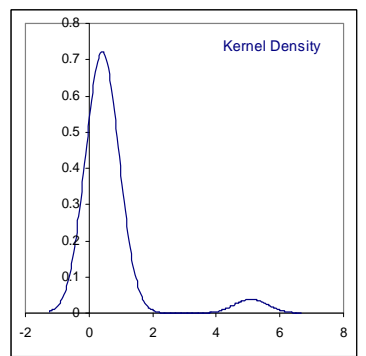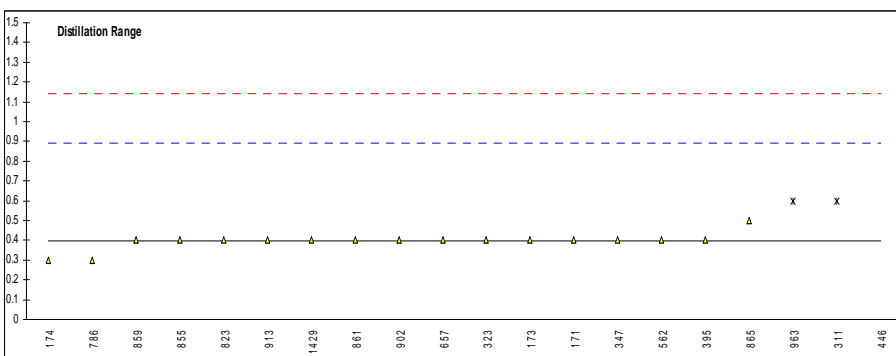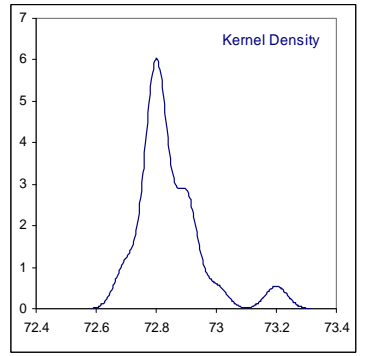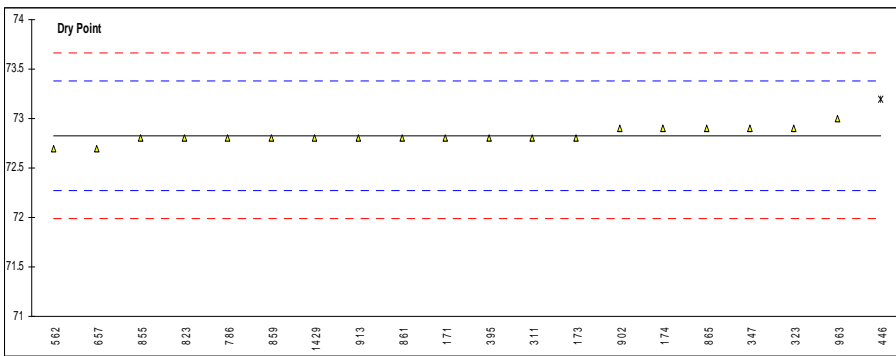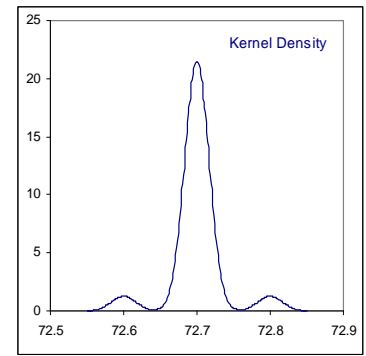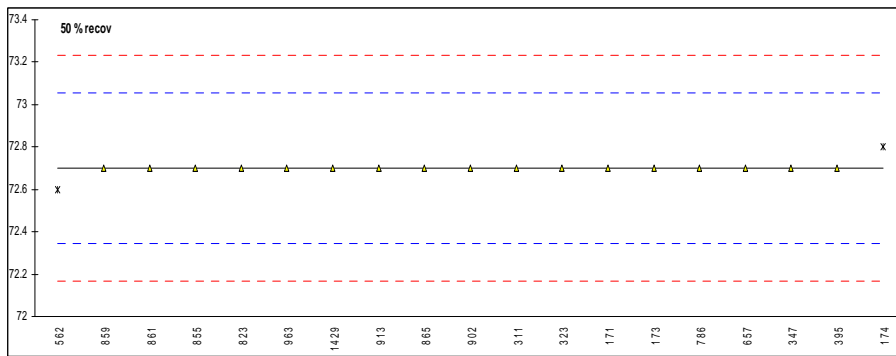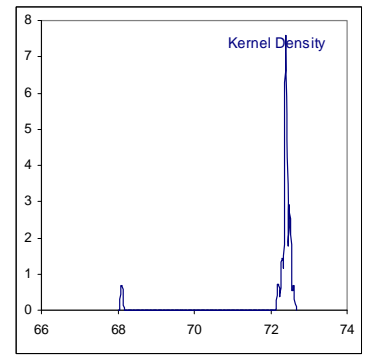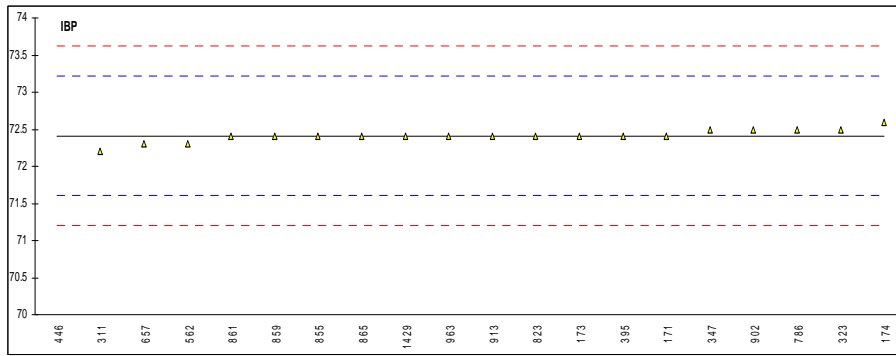
Determination of Ethyl Acetate on sample #11115, results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-256	187.5		0.05	
173	INH-47	221.6		2.56	
174	D3545	201		1.05	
311	INH-122	196.4		0.71	
323	INH-067	170		-1.23	
347	INH-916	180		-0.50	
395	INH-47	188.843		0.15	
446	INH-47	262	G(0.01)	5.53	
522		-----		-----	
551		-----		-----	
562	INH-001	211	C	1.78	First reported 0.0211
613		-----		-----	
657		192		0.38	
662	INH-40	199.84		0.96	
786	INH-004	165		-1.60	
823	D3545	180		-0.50	
855	INH-065	181.8		-0.37	
859	INH-065	201.2		1.06	
861		-----		-----	
865	INH-065	188		0.09	
887		-----		-----	
902	INH-22	184.5	C	-0.17	First reported 240.7
913		156		-2.26	
963	INH-009	166		-1.53	
974		170		-1.23	
1429	in house	195		0.60	
1449		-----		-----	
1622		-----		-----	

|		
normality	OK	
n	20	
outliers	1	
mean (n)	186.78	
st.dev. (n)	16.403	
R(calc.)	45.93	
R(Horwitz)	38.08	Compare R(D3545:06) = 900 mg/kg at concentration 1000 mg/kg
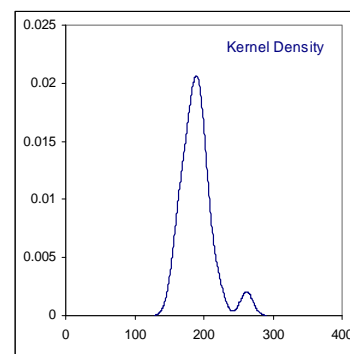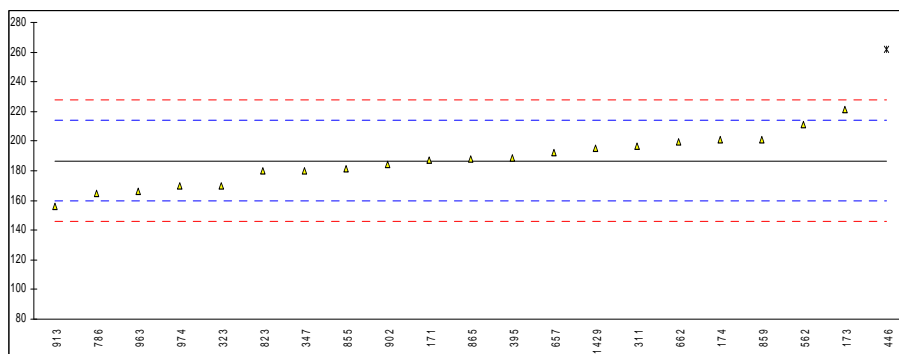
Determination of Inhibitor (Hydroquinone) on sample #11115; results in mg/kg

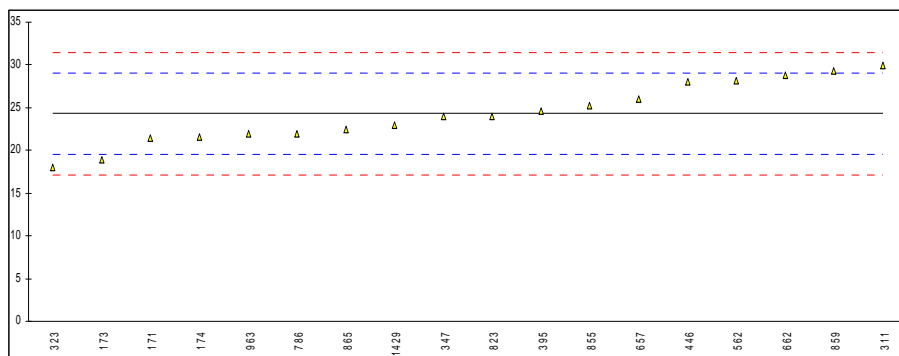| lab	method	value	mark	z(targ)	remarks
171	D2193	4.1		-0.38	
173	INH-43	4.5		0.74	
174	D2193	5.0		2.14	
311	D2193	4.55		0.88	
323		-----		-----	
347	D2193	4.5	C	0.74	First reported 5.5
395	INH-43	4.67		1.22	
446		-----		-----	
522	INH-40	4.12		-0.32	
551		-----		-----	
562	D2193	3.95		-0.80	
613	INH-40	3.6		-1.78	
657	D2193	3.47		-2.14	
662		-----		-----	
786	D2193	4.18		-0.16	
823	D2193	3.8		-1.22	
855	D2193	4.34		0.29	
859	D2193	4.49		0.71	
861	D2193	4.49		0.71	
865	D2193	4.02		-0.60	
887		-----		-----	
902	D2193	4.2		-0.10	
913	D2193	3.5		-2.06	
963		-----		-----	
974	D2193	6.01	G(0.05)	4.97	
1429	D2193	5.0	C	2.14	First reported 7.59
1449		-----		-----	
1622		-----		-----	

normality	OK
n	19
outliers	1
mean (n)	4.236
st.dev. (n)	0.4485
R(calc.)	1.256
R(D2193:06)	1.000
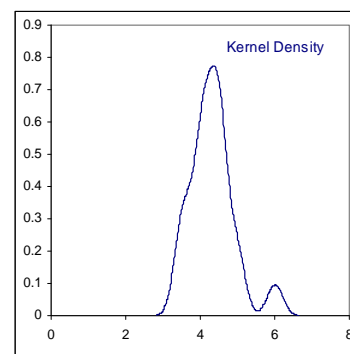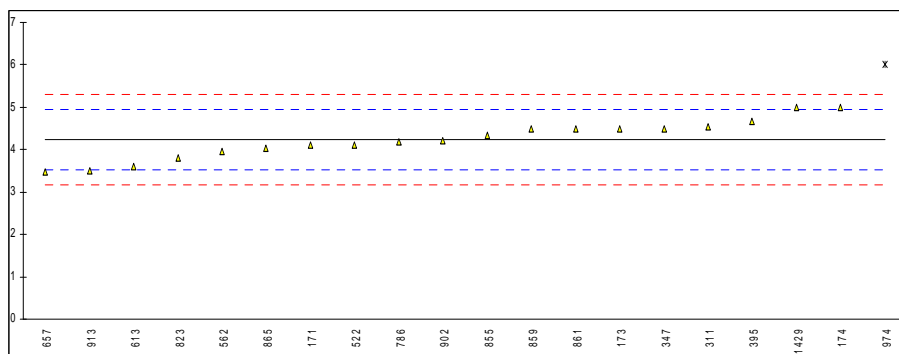
Determination of Methyl Acetate on sample #11115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-256	21.4		-1.20	
173	INH-47	18.9		-2.24	
174	D3545	21.5	C	-1.16	First reported 215
311	INH-122	29.9		2.33	
323	INH-067	18		-2.62	
347	INH-916	24		-0.12	
395	INH-47	24.613		0.13	
446	INH-47	28		1.54	
522		-----		-----	
551		-----		-----	
562	INH-001	28.2	C	1.63	First reported 0.00282
613		-----		-----	
657		26		0.71	
662	INH-40	28.78		1.87	
786	INH-004	22		-0.95	
823	D3545	24		-0.12	
855	INH-065	25.2		0.38	
859	INH-065	29.3		2.08	
861		-----		-----	
865	INH-065	22.4		-0.79	
887		-----		-----	
902		-----		-----	
913		-----		-----	
963	INH-009	22		-0.95	
974		-----		-----	
1429	in house	23		-0.54	
1449		-----		-----	
1622		-----		-----	

normality	OK
n	18
outliers	0
mean (n)	24.29
st.dev. (n)	3.517
R(calc.)	9.85
R(Horwitz)	6.73
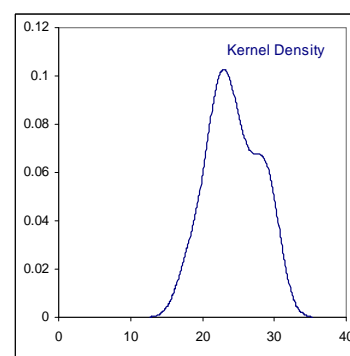
Determination of Purity on sample #11115; results in % M/M

lab	method	value	mark	z(targ)	remarks
171	INH-256	99.959		-----	
173	INH-47	99.60	G(0.01)	-----	
174	D3545	99.961		-----	
311	INH-122	99.974		-----	
323	INH-067	99.96		-----	
347	INH-916	99.977		-----	
395	INH-257	99.957		-----	
446	INH-257	99.948		-----	
522	INH-40	99.978		-----	
551		-----		-----	
562	INH-001	99.98		-----	
613	INH-40	99.973		-----	
657		99.9651		-----	
662	INH-40	99.94		-----	
786	INH-004	99.954		-----	
823	D3545	99.964		-----	
855	INH-065	99.975		-----	On dry basis
859	INH-065	99.974		-----	
861	INH-065	99.96		-----	
865	INH-065	99.975		-----	
887	in house	99.96		-----	
902	INH-22	99.92	C,G(0.01)	-----	First reported 99.935
913	D3545	99.97		-----	
963	INH-009	99.97		-----	
974		99.98		-----	
1429	in house	99.9738		-----	
1449		-----		-----	
1622		-----		-----	

|		
normality	OK	
n	23	
outliers	2	
mean (n)	99.9664	
st.dev. (n)	0.01065	
R(calc.)	0.0298	
R(Lit)	unknown	Compare R(iis09C03) = 0.023
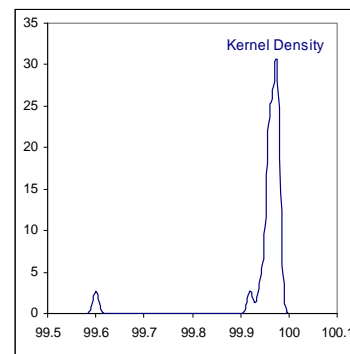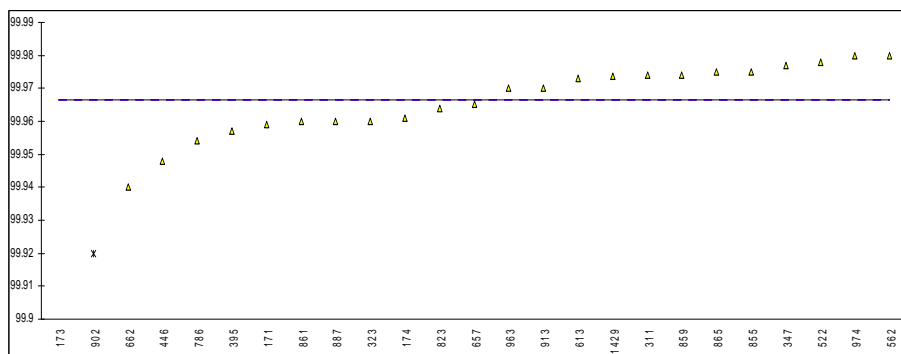
Determination of Water on sample #11115; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	154		-0.03	
173	E203	132.5		-0.84	
174	D1364	150		-0.18	
311	D1364	160	C	0.20	First reported 0.016
323	D1364	154		-0.03	
347	D1364	137		-0.67	
395	INH-63	154.62		-0.01	
446	INH-63	155		0.01	
522	INH-40	185.6		1.16	
551		-----		-----	
562	E203	157		0.08	
613		-----		-----	
657	D1364	156.6		0.07	
662	INH-40	310	G(0.05)	5.82	
786	D1364	220.8		2.48	
823	D1364	126		-1.08	
855	D1364	147		-0.29	
859	D1364	140		-0.55	
861	E1604	170		0.57	
865	D1364	139		-0.59	
887	D1364	153		-0.07	
902	D1364	162		0.27	
913	D1364	175		0.76	
963	D1364	134		-0.78	
974	D1364	110.5		-1.66	
1429	D1364	186		1.17	
1449	D1364	260	C, G(0.01)	3.95	First reported 30
1622	D1364	46	G(0.05)	-4.08	

normality	OK
n	23
outliers	3
mean (n)	154.77
st.dev. (n)	22.962
R(calc.)	64.29
R(D1364:07)	74.64
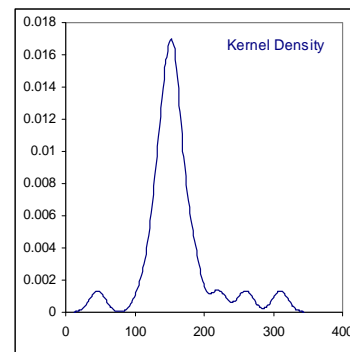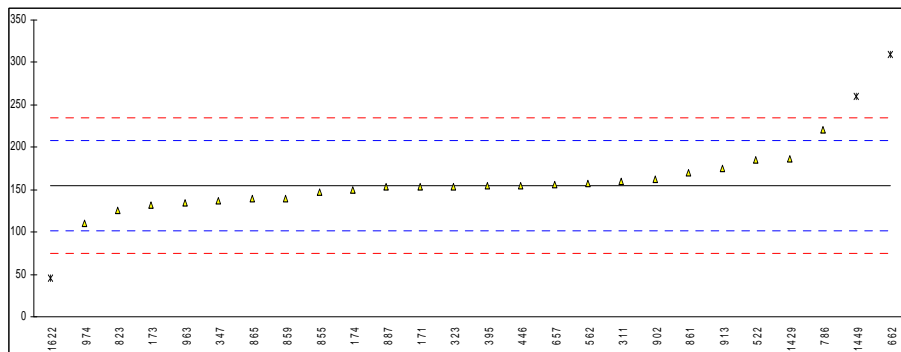
APPENDIX 2

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

2 labs in INDONESIA

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 TAIWAN R.O.C.

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/)