Results of Proficiency Test
Vinyl Acetate Monomer
February 2014

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 2013/2014, it was decided to continue the round robin for the analysis of Vinyl Acetate Monomer. In this interlaboratory study 24 laboratories in 18 different countries have participated. See appendix 2 for the number of participants per country.
In this report, the results of the 2014 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 #14004) 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 on 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 #14004.
The homogeneity of the subsamples #14004 was checked by determination of Density in accordance with ASTM D4052:11 and water in accordance with ASTM D1364:02(2012) on 8 stratified randomly selected samples.

	Density @20°C in kg/L	Water in mg/kg
sample #14004-1	0.93222	109
sample #14004-2	0.93220	104
sample #14004-3	0.93220	108
sample #14004-4	0.93221	107
sample #14004-5	0.93220	99
sample #14004-6	0.93220	104
sample #14004-7	0.93221	103
sample #14004-8	0.93220	113

table 1: homogeneity test results of subsamples #14004

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 calculated using the Horwitz equation in agreement with the procedure of ISO 13528, Annex B2 in table 2.

	Density @20°C in kg/L	Water in mg/kg
r (sample #14004)	0.00002	12
reference test	D4052:02e1	ASTM D1364:02(2012)
0.3*R (reference test)	0.00015	18.5

table 2: repeatabilities of subsamples #14004

The calculated repeatabilities for Density and water are in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 1 bottle of 500 mL Vinyl Acetate Monomer, labelled #14004, was sent on January 22, 2014.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were requested to determine according to the standard specification for VAM (ASTM D2190:07(2013)): Acetaldehyde, Acidity, Distillation (IBP, 50% recovery, Dry Point, Boiling Range), Inhibitor and Water, plus additionally Acidity with N_2 purging, Acetone, Appearance, Density @ 20°C, Ethyl Acetate, Methyl Acetate and Purity.

To get comparable results, a detailed report form, on which the units were prescribed, as well as 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 and by R(0.05) for the Rosner General ESD test (ref. 15). 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 on the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected 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 Brazil, India and Russia received the samples late. Of the 24 participants, 3 participants reported results after the final reporting date and 1 laboratory had not received the sample at the time the results were due.

Not all participants were able to report results for all the requested tests. Finally, 23 participants reported in total 240 numerical results. Observed were 9 outlying test results, which is 3.8% of the total of numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1   EVALUATION PER TEST

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

A not-normal distribution was found for Density, Distillation (IBP, 50% recovery, DP and Boiling Range) and Water. 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 D2191:06(2012). However, this reproducibility is based on an interlaboratory study with a range far above the measured concentration in sample #14004. Therefore the reproducibility according to ASTM D2191:06(2012) may not be applicable. Also, only three participants reported to have used ASTM D2191. When the Horwitz estimate is used as target for the evaluation, the determination may be problematic.

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

Acidity: This determination (with and without Nitrogen purging) may be problematic. As can be expected, the value determined with Nitrogen purging was somewhat lower, but not significantly.

Four statistical outliers were observed, of which one laboratory had high, outlying results, with and without Nitrogen purging.

Both calculated reproducibilities, after rejection of the statistical outliers, are not in agreement with the requirements of ASTM D2086:08(2012). Please note that the reproducibility of ASTM D2086:08(2012) was determined with only two laboratories (see note 5 in §13.2.2 of ASTM D2086:08(2012)). When compared to the spread found in the previous PT (iis13C03), the reproducibility value has improved for both tests.

Appearance: The determination was not problematic. All laboratories, but one, agreed on the appearance of the sample. Most laboratories reported "Pass" in agreement with ASTM E2680:09e1. Other laboratories reported Clear or Bright & Clear.

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.

Distillation: This determination may be problematic. In total two statistical outliers were observed. However, the calculated reproducibilities, after rejection of the statistical outliers, are all in good agreement with the requirements of ASTM D1078:11 (Automated method).

Regretfully it was noticed that two reporting laboratories did not correct sufficiently for either the thermometer deviation and/or the barometric pressure as described in ASTM D1078:11. When manually corrected to the theoretical boiling point as prescribed in the method (theoretical boiling point = 72.7°C), the calculated reproducibilities did not change significantly.

Ethyl Acetate: The determination of this impurity may be problematic. One statistical outlier was observed. However, the calculated reproducibility is in agreement with the requirements estimated from the Horwitz equation.

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

Methyl Acetate: The determination of this impurity may be problematic. No statistical outliers were observed. But two laboratories submitted a result <10, which is not used for evaluation, but appears to be a false negative result. The calculated reproducibility 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 does not differ significantly from the spread found in the previous PT (iis13C03).

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1364:02(2012).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The assigned values, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM, ISO, EN standards) are compared in the next table.

Parameter	unit	n	Mean	2.8 * sd	R (lit)
Acetaldehyde	mg/kg	16	29.1	14.9	80.0*
Acetone	mg/kg	15	<10	n.a.	n.a.
Acidity (without N_2 purging)	mg/kg	15	20.7	11.0	6.0*
Acidity (with N_2 purging)	mg/kg	9	17.1	11.1	6.0*
Appearance	------	23	Pass	-----	-----
Density @ 20°C	kg/L	23	0.9322	0.0002	0.0005
Distillation					
-Initial Boiling Point	ºC	16	72.44	0.20	1.13
-50% recovery	ºC	17	72.68	0.18	0.50
-Dry Point	ºC	17	72.80	0.26	0.78
-Boiling Range	ºC	16	0.36	0.23	0.69
Ethylacetate	mg/kg	17	157.6	25.8	33.0
Inhibitor	mg/kg	19	3.37	1.38	1.00
Methyl Acetate	mg/kg	16	78.2	29.2	18.2
Purity	%M/M	21	99.97	0.02	unknown
Water	mg/kg	23	86.6	57.2	55.8

table 3: performance evaluation sample #14004

*see §4.1 (page 8)

4.3   EVALUATION OF THE PROFICIENCY TEST OF FEBRUARY 2014 WITH PREVIOUS PTS

	February 2014	February 2013	February 2012	April 2011
Number of rep. participants	23	24	24	28
Number of results reported	240	243	257	264
Number of statistical outliers	9	9	8	16
Percentage outliers	3.8%	3.7%	3.1%	6.1%

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 2014	February 2013	February 2012	April 2011
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	-	-	++	--
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 #14004; results in mg/kg

lab	method	value	Mark	z(targ)	Remarks
171	INH-0001	30.4		0.05	
173	INH-47	30.90		0.06	
174	D3545	33		0.14	
311	INH-122	20		-0.32	
315		-----		-----	
323	INH-49	27		-0.07	
347	INH-096	33		0.14	
395	INH-47	29.21		0.00	
446		-----		-----	
522	INH-40	26.5		-0.09	
551		-----		-----	
613		-----		-----	
657		-----		-----	
663	INH-40	35.8		0.23	
786	INH-004	67.06	G(0.01)	1.33	
823	D2191	34		0.17	
859	D2191	30		0.03	
861	INH-065	29		0.00	
902	INH-22	28		-0.04	
913		-----		-----	
963	D2191	30		0.03	
974		-----		-----	
1107	in house	34		0.17	
1429	in house	15		-0.49	
	normality	OK			
	n	16			
	outliers	1			
	mean (n)	29.11			
	st.dev. (n)	5.316			
	R(calc.)	14.88			
	R(D2191:06)	80.00			Compare R(Horwitz) = 7.85

Kernel Density
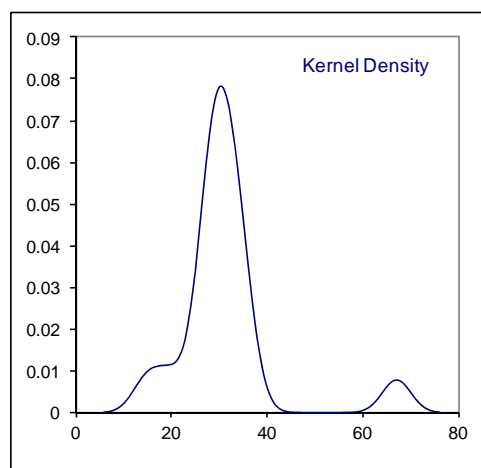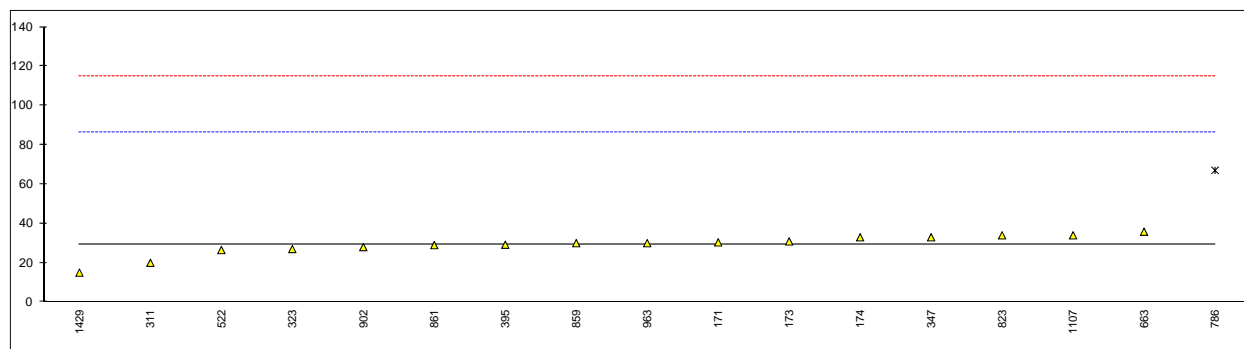
Determination of Acetone on sample #14004, results in mg/kg

lab	method	value	mark	z(targ)	Remarks
171	INH-0001	<5		-----	
173	INH-47	0		-----	
174	D3545	<10		-----	
311	INH-122	<10		-----	
315		-----		-----	
323	INH-49	<10		-----	
347	INH-096	<10		-----	
395		-----		-----	
446		-----		-----	
522		-----		-----	
551	D3545	<10		-----	
613		-----		-----	
657	in house	<10		-----	
663	INH-40	0.0		-----	
786	INH-004	<10		-----	
823	D3545	<5		-----	
859	INH-1628	<10		-----	
861		-----		-----	
902	INH-22	<10		-----	
913		-----		-----	
963	INH-009	<10		-----	
974		-----		-----	
1107		-----		-----	
1429	in house	<5		-----	
	normality	n.a.			
	n	15			
	outliers	0			
	mean (n)	<10			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	n.a.			

Determination of Acidity (without N_2 purging) on sample #14004; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
171	D2086	23	C	1.10	First reported: 33
173	INH-44	16		-2.17	
174	D2086	23		1.10	
311	D2086	24		1.56	
315		-----		-----	
323	D2086	18		-1.24	
347	D2086	22		0.63	
395	INH-44	35.9	G(0.05)	7.12	
446	INH-44	19		-0.77	
522		-----		-----	
551	D2086	27		2.96	
613		-----		-----	
657	D2086	18.2		-1.14	
663		-----		-----	
786		-----		-----	
823	D2086	24		1.56	
859	D2086	15.6		-2.36	
861	D2086	16.8		-1.80	
902	D2086	20.2		-0.21	
913		-----		-----	
963	D2086	16.0		-2.17	
974	D2086	27		2.96	
1107		-----		-----	
1429	D2086	54	G(0.01)	15.56	

normality	OK	
n	15	
outliers	2	
mean (n)	20.65	
st.dev. (n)	3.923	
R(calc.)	10.98	
R(D2086:08)	6.00	Compare R(iis13C03) = 12.05
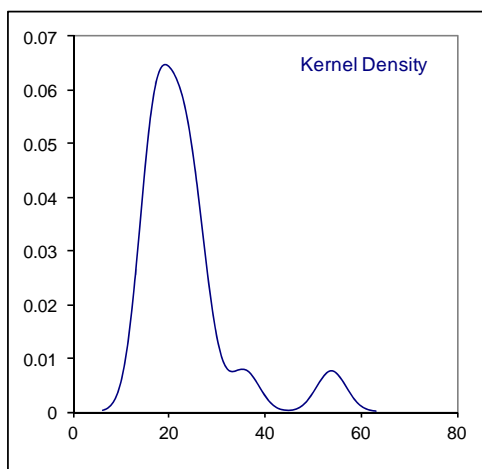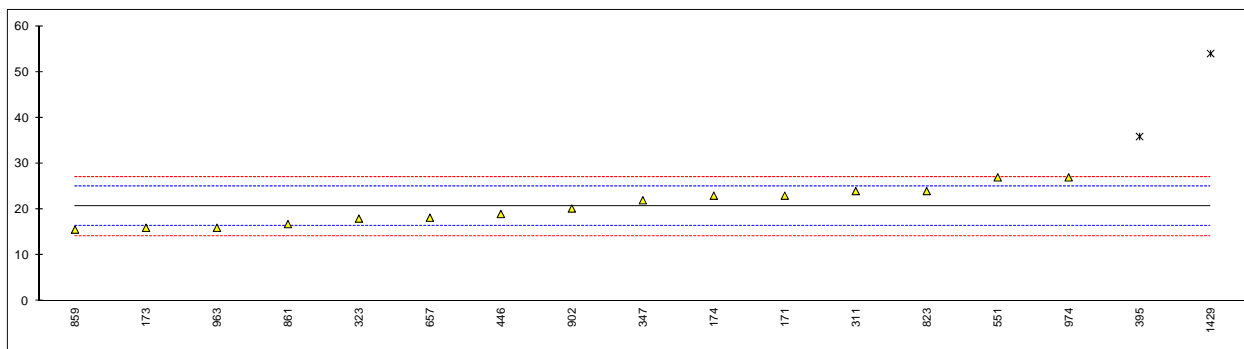
Determination of Acidity (with N_2 purging) on sample #14004; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
171		-----		-----	
173		-----		-----	
174		-----		-----	
311	D2086	20		1.36	
315		-----		-----	
323		-----		-----	
347		-----		-----	
395		-----		-----	
446		-----		-----	
522		-----		-----	
551		-----		-----	
613		-----		-----	
657	D2086	10.8		-2.93	
663	INH-40	38.2	C,G(0.01)	9.86	First reported: 35.3
786	D2086	15.6		-0.69	
823		-----		-----	
859	D2086	15.0		-0.97	
861	D2086	14.2		-1.34	
902	D2086	20.4		1.55	
913		-----		-----	
963	D2086	15.5		-0.74	
974	D2086	24		3.23	
1107	D2086	18.2		0.52	
1429	D2086	52	G(0.05)	16.30	

normality	OK	
n	9	
outliers	2	
mean (n)	17.08	
st.dev. (n)	3.960	
R(calc.)	11.09	
R(D2086:08)	6.00	Compare R(iis13C03) = 12.46

Kernel Density
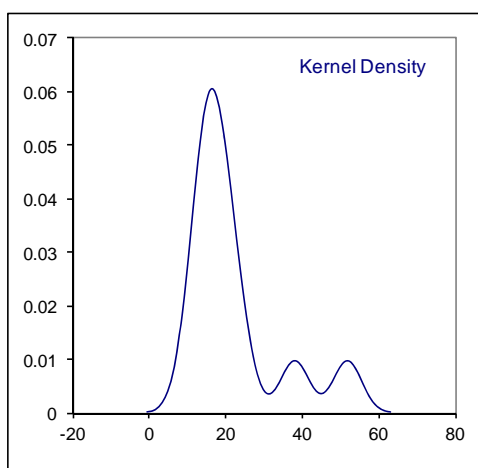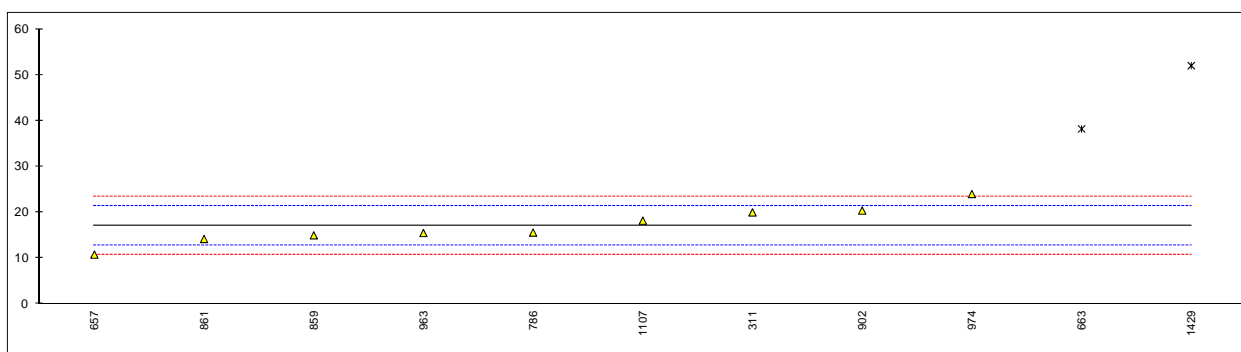
Determination of Appearance on sample #14004;

lab	method	value	mark	z(targ)	Remarks
171	E2680	Pass		-----	
173	E2680	Pass		-----	
174	E2680	Pass		-----	
311	E2680	Pass		-----	
315	E2680	B&C		-----	
323	E2680	B&C		-----	
347	E2680	Pass		-----	
395	E2680	Pass		-----	
446	INH-500	Pass		-----	
522	visual	Pass		-----	
551	E2680	Pass		-----	
613	INH-40	B&C		-----	
657	E2680	Pass		-----	
663	E2680	Pass		-----	
786	E2680	Pass		-----	
823	E2680	Pass		-----	
859	E2680	Pass		-----	
861	E2680	B&C		-----	
902	E2680	Pass		-----	
913		-----		-----	
963	E2680	Pass		-----	
974	E2680	Pass		-----	
1107	E2680	Clear		-----	
1429	E2680	Fail		-----	False negative?
	normality	n.a.			
	n	23			
	outliers	n.a.			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(E2680)	n.a.			

B&C   = Bright and clear

Determination of Density @ 20 °C on sample #14004; results in kg/L

lab	method	value	mark	z(targ)	Remarks
171	D4052	0.9322		0.01	
173	D4052	0.93221		0.07	
174	D4052	0.9322		0.01	
311	D4052	0.9322		0.01	
315	D4052	0.9322		0.01	
323	D4052	0.9322		0.01	
347	D4052	0.9322		0.01	
395	D4052	0.9322		0.01	
446	D4052	0.9322		0.01	
522	D4052	0.9322		0.01	
551	D4052	0.9321		-0.55	
613	INH-40	0.93224		0.24	
657	D4052	0.93211		-0.49	
663	D4052	0.9321		-0.55	
786	D4052	0.9321		-0.55	
823	D4052	0.9323		0.57	
859	D4052	0.93219		-0.04	
861	D4052	0.93219		-0.04	
902	D4052	0.9322		0.01	
913		-----		-----	
963	D4052	0.9321		-0.55	
974	D4052	0.9322		0.01	
1107	D4052	0.9324		1.13	
1429	D4052	0.9323		0.57	

normality	not OK
n	23
outliers	0
mean (n)	0.93220
st.dev. (n)	0.000071
R(calc.)	0.00020
R(D4052:02e1)	0.00050
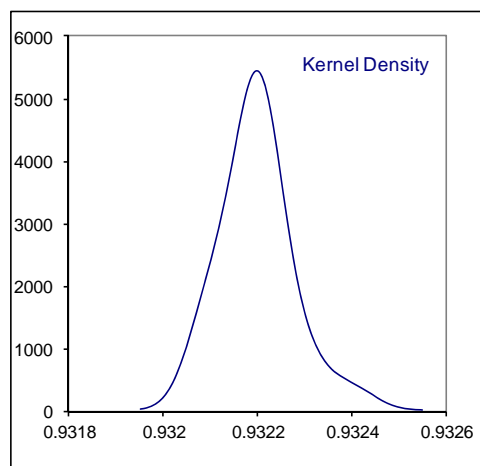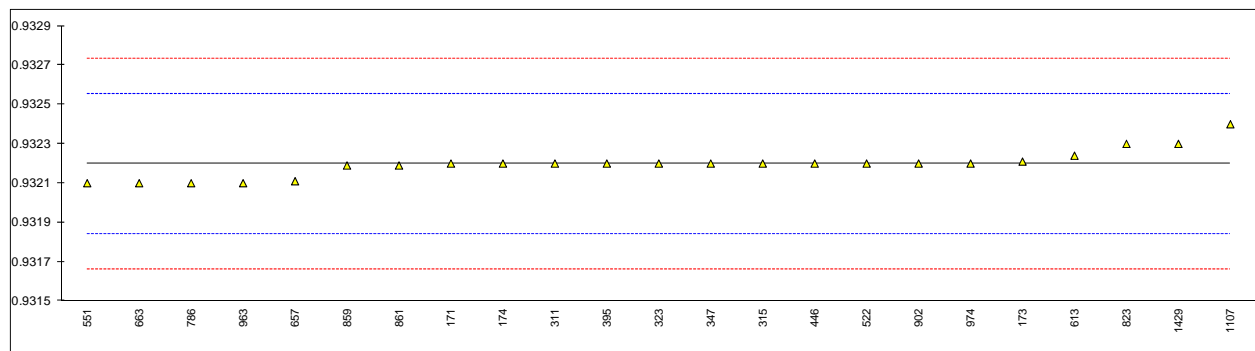
Determination of Distillation on sample #14004; results in ºC

lab	method	IBP	mark	50% rec.*	mark	DP	mark	range	mark
171	D1078	72.5		72.7		72.8		0.3	
173	D1078	72.8		73.0	D(0.01)	73.0		0.2	
174	D1078	72.6	C**	72.7		73.0		0.4	C**
311	D1078	72.3		72.7		72.8		0.5	
315		-----		-----		-----		-----	
323	D1078	72.5		72.7		72.9		0.4	
347	D1078	72.4		72.7		72.8		0.4	
395	D1078	72.5		72.7		72.8		0.3	
446	D1078	72.4		72.7		72.7		0.3	
522		-----		-----		-----		-----	
551	D1078	72.4		72.5		72.8		0.4	
613		-----		-----		-----		-----	
657	D1078	72.5		72.7		72.7		0.2	
663		-----		-----		-----		-----	
786	D1078	72.4		72.6		72.6		0.2	
823		-----		-----		-----		-----	
859	D1078	72.4		72.7		72.8		0.4	
861	D1078	72.4		72.7		72.8		0.4	
902	D1078	72.4		72.6		72.7		0.3	
913		-----		-----		-----		-----	
963	D1078	72.4		72.7		72.8		0.4	
974	D1078	72.1		72.8		72.9		0.8	D(0.01)
1107	D1078	72.5		72.7		72.9		0.4	
1429	D1078	72.4		72.7		72.8		0.4	
	normality	not OK		not OK		not OK		not OK	
	n	18		17		18		17	
	outliers	0		1		0		1	
	mean (n)	72.44		72.68		72.81		0.35	
	st.dev. (n)	0.138		0.064		0.102		0.087	
	R(calc.)	0.39		0.18		0.29		0.24	
	R(D1078:11)	1.13		0.50		0.78		0.69	

*) Theoretical mid boiling point = 72.7 ºC

**) Lab 174 first reported for IBP: 72.3 and for Distillation range: 0.7

z-scores of Distillation on sample #14004

lab	IBP	50% rec.*	DP	range
171	0.15	0.10	-0.04	-0.19
173	0.89	1.79	0.68	-0.60
174	0.40	0.10	0.68	0.21
311	-0.34	0.10	-0.04	0.62
315	-----	-----	-----	-----
323	0.15	0.10	0.32	0.21
347	-0.10	0.10	-0.04	0.21
395	0.15	0.10	-0.04	-0.19
446	-0.10	0.10	-0.40	-0.19
522	-----	-----	-----	-----
551	-0.10	-1.03	-0.04	0.21
613	-----	-----	-----	-----
657	0.15	0.10	-0.40	-0.60
663	-----	-----	-----	-----
786	-0.10	-0.46	-0.76	-0.60
823	-----	-----	-----	-----
859	-0.10	0.10	-0.04	0.21
861	-0.10	0.10	-0.04	0.21
902	-0.10	-0.46	-0.40	-0.19
913	-----	-----	-----	-----
963	-0.10	0.10	-0.04	0.21
974	-0.84	0.66	0.32	1.83
1107	0.15	0.10	0.32	0.21
1429	-0.10	0.10	-0.04	0.21

Results after manual correction for Mid-Boiling Point (=72.7°C)

lab	method	IBP	z-score	50% rec.*	z-score	DP	z-score
173	D1078	72.5	0.17	72.7	0.03	72.7	-0.38
551	D1078	72.6	0.41	72.7	0.03	73.0	0.70
	normality	not OK		not OK		not OK	
	n	18		18		18	
	outliers	0		0		0	
	mean (n)	72.43		72.69		72.81	
	st.dev. (n)	0.114		0.042		0.106	
	R(calc.)	0.32		0.12		0.30	
	R(D1078:11)	1.13		0.50		0.78	
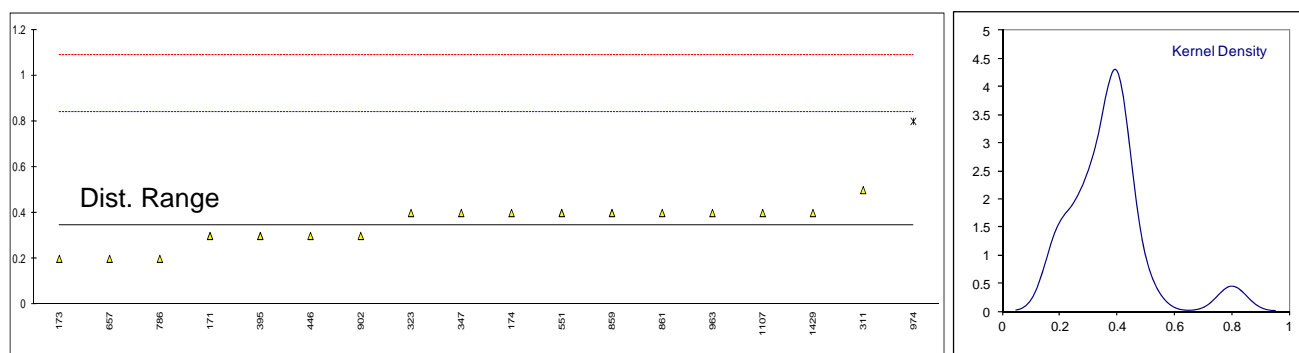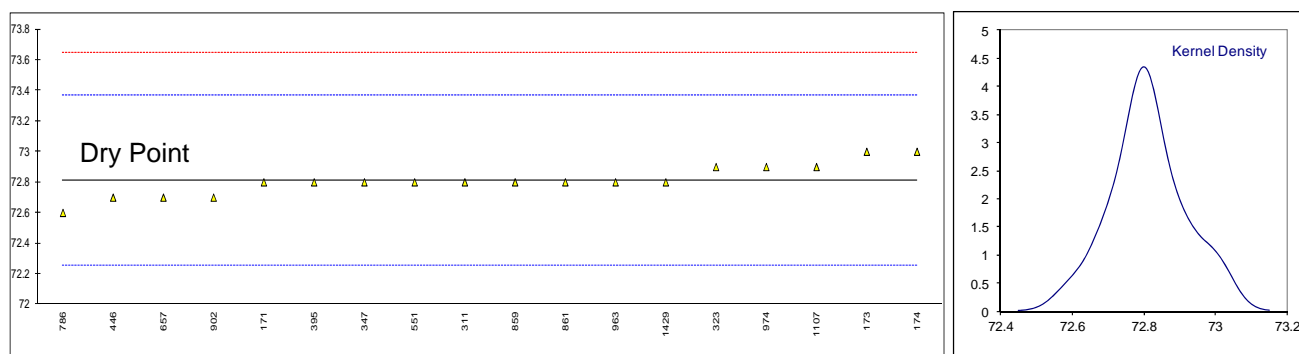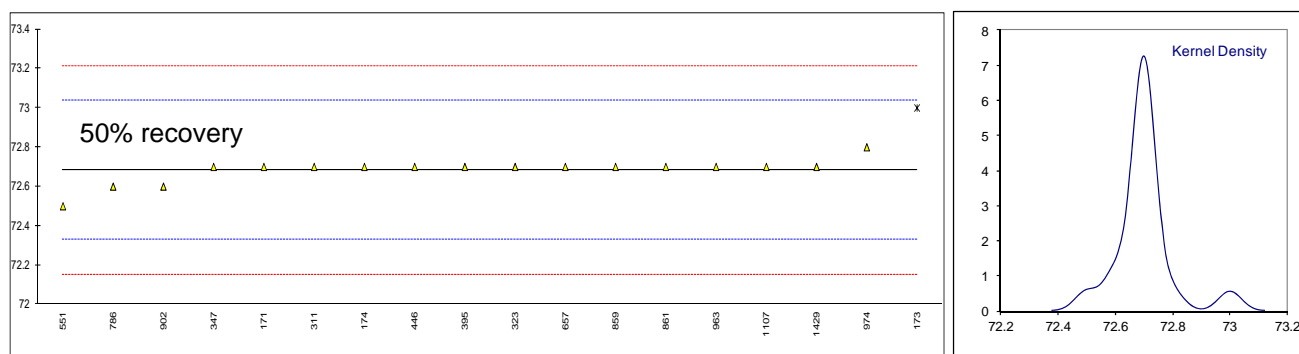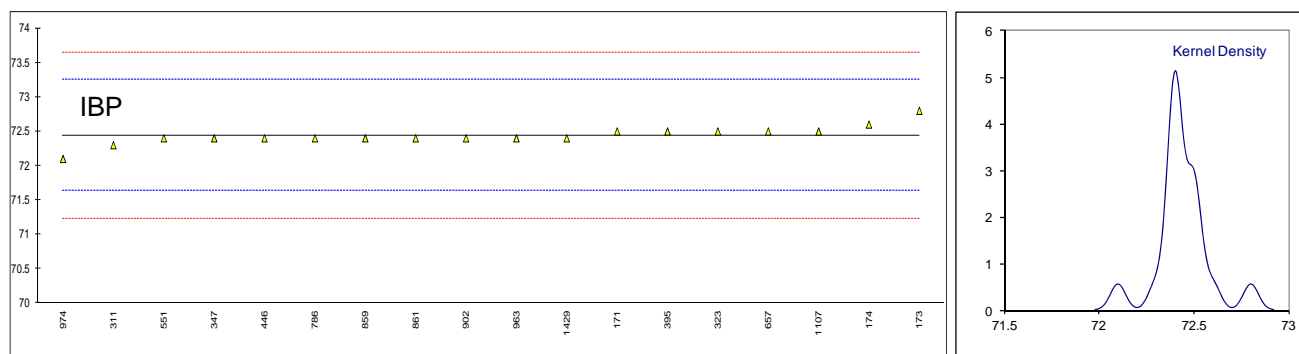
Determination of Ethyl Acetate on sample #14004, results in mg/kg

lab	method	value	mark	z(targ)	Remarks
171	INH-0001	162.6		0.43	
173	INH-47	155.22		-0.20	
174	D3545	160	C	0.21	First reported: 210
311	INH-122	160		0.21	
315		-----		-----	
323	INH-49	145		-1.07	
347	INH-096	153		-0.39	
395	INH-47	153.76		-0.32	
446	INH-47	141		-1.41	
522		-----		-----	
551	D3545	147.5		-0.86	
613		-----		-----	
657	in house	162		0.38	
663	INH-40	176.6		1.62	
786	INH-004	108.06	G(0.01)	-4.21	
823	D3545	160		0.21	
859	INH-1628	169.0		0.97	
861		-----		-----	
902	INH-22	155		-0.22	
913		-----		-----	
963	INH-009	151		-0.56	
974		-----		-----	
1107	in house	171		1.14	
1429	in house	156		-0.13	

normality	OK	
n	17	
outliers	1	
mean (n)	157.57	
st.dev. (n)	9.229	
R(calc.)	25.84	
R(Horwitz)	32.96	
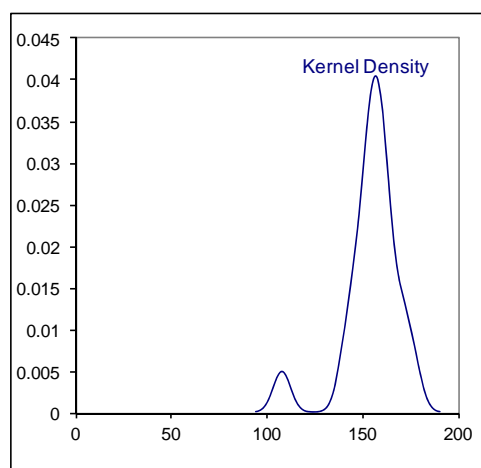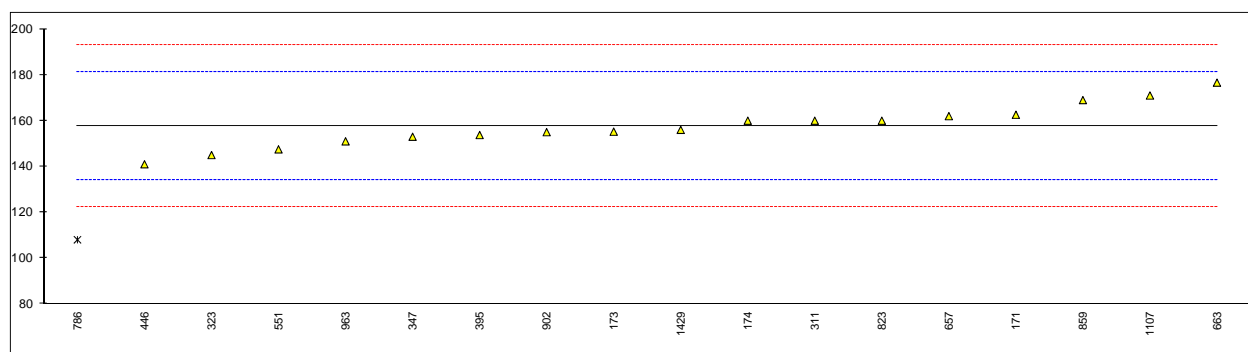
Determination of Inhibitor (Hydroquinone) on sample #14004; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D2193	4.2	C	2.33	First reported: 4.6
173	INH-43	2.06		-3.66	
174	D2193	3.8		1.21	
311	D2193	3.3		-0.19	
315		-----		-----	
323	D2193	3.4		0.09	
347	D2193	3.3		-0.19	
395	INH-43	3.21		-0.44	
446		-----		-----	
522	INH-40	3.1		-0.75	
551	D2193	3.4		0.09	
613		-----		-----	
657	D2193	3.35		-0.05	
663	D2193	2.75		-1.73	
786	D2193	3.72		0.99	
823	D2193	3.1		-0.75	
859	D2193	3.52		0.43	
861	D2193	3.33		-0.10	
902	D2193	3.4		0.09	
913		-----		-----	
963	D2193	4.0		1.77	
974	D2193	2.956		-1.15	
1107	D2193	4.07		1.97	
1429	in house	11.15	G(0.01)	21.79	

normality	OK
n	19
outliers	1
mean (n)	3.367
st.dev. (n)	0.4923
R(calc.)	1.379
R(D2193:06)	1.000
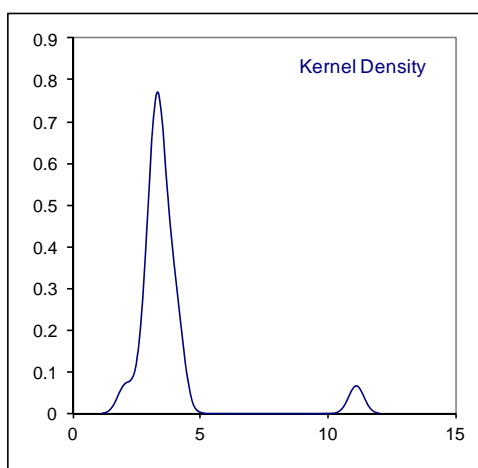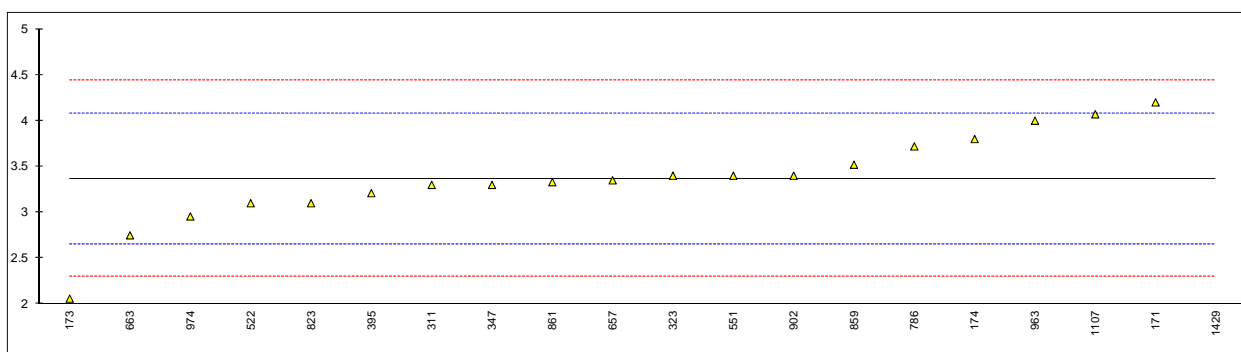
Determination of Methyl Acetate on sample #14004; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	INH-0001	69.1		-1.40	
173	INH-47	82.21		0.62	
174	D3545	88		1.51	
311	INH-122	90		1.82	
315		-----		-----	
323	INH-49	67		-1.72	
347	INH-096	81		0.44	
395	INH-47	62.76		-2.37	
446	INH-47	91		1.98	
522		-----		-----	
551	D3545	70.7		-1.15	
613		-----		-----	
657	in house	86		1.21	
663	INH-40	88.5		1.59	
786	INH-004	55.73		-3.46	
823	D3545	80		0.28	
859	INH-1628	79.7		0.24	
861		-----		-----	
902	INH-22	83		0.74	
913		-----		-----	
963	INH-009	<10		< -10.50	False negative?
974		-----		-----	
1107	in house	<10		< -10.50	False negative?
1429	in house	76		-0.33	

normality	OK
n	16
outliers	0
mean (n)	78.17
st.dev. (n)	10.428
R(calc.)	29.20
R(Horwitz)	18.17
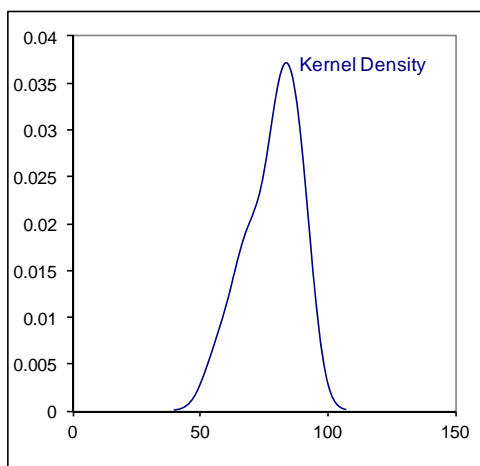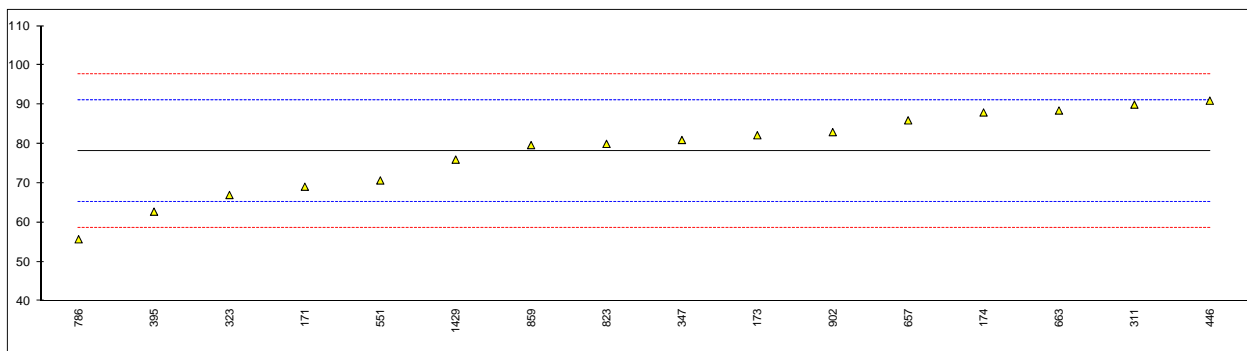
Determination of Purity on sample #14004; results in % M/M

lab	method	value	mark	z(targ)	remarks
171	INH-0001	99.97		-----	
173	INH-47	99.9729		-----	
174	D3545	99.958		-----	
311	INH-122	99.97		-----	
315		-----		-----	
323	INH-49	99.96		-----	
347	INH-096	99.97		-----	
395	INH-257	99.963		-----	
446	INH-257	99.960		-----	
522	INH-40	99.97		-----	
551	D3545	99.9747		-----	
613	INH-40	99.978		-----	
657	INH-0047	99.9633		-----	
663	INH-40	99.95		-----	
786	INH-004	99.9666		-----	
823	D3545	99.96		-----	
859	INH-1628	99.96		-----	
861	INH-065	99.965		-----	
902	INH-22	99.97		-----	
913		-----		-----	
963	INH-009	99.96		-----	
974		-----		-----	
1107	in house	99.96		-----	
1429	in house	99.975		-----	

normality	OK	
n	21	
outliers	0	
mean (n)	99.9655	
st.dev. (n)	0.00699	
R(calc.)	0.0196	
R(lit.)	unknown	Compare R(iis13C03) = 0.0172
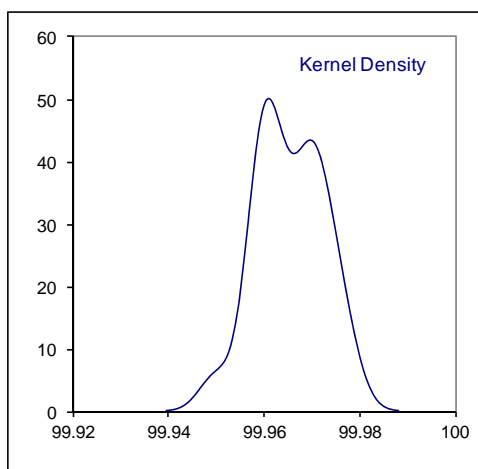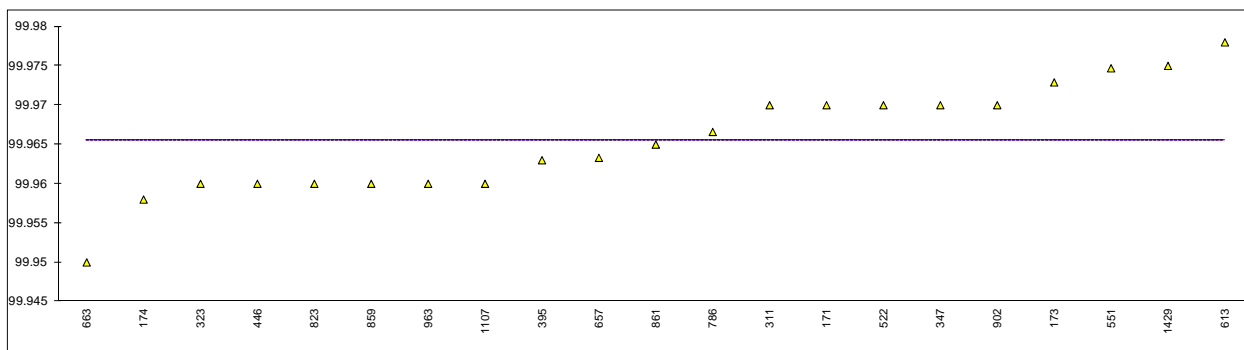
Determination of Water on sample #14004; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D1364	89		0.12	
173	E203	70		-0.83	
174	D1364	85		-0.08	
311	D1364	70		-0.83	
315	D1364	80		-0.33	
323	D1364	138		2.58	
347	D1364	82		-0.23	
395	D1364	89.37		0.14	
446	D1364	84	C	-0.13	First reported: 0.0084
522	INH-40	90		0.17	
551	D1364	105		0.93	
613	INH-40	92		0.27	
657	E1064	82.6		-0.20	
663	E203	118.0		1.58	
786	D1364	86		-0.03	
823	D1364	87		0.02	
859	D1364	62		-1.23	
861	E1064	54		-1.63	
902	D1364	81.8		-0.24	
913		-----		-----	
963	D1364	92		0.27	
974	D1364	57.8	C	-1.44	First reported: 578
1107	D1364	127		2.03	
1429	D1364	68		-0.93	

normality	not OK
n	23
outliers	0
mean (n)	86.55
st.dev. (n)	20.438
R(calc.)	57.23
R(D1364:02)	55.82
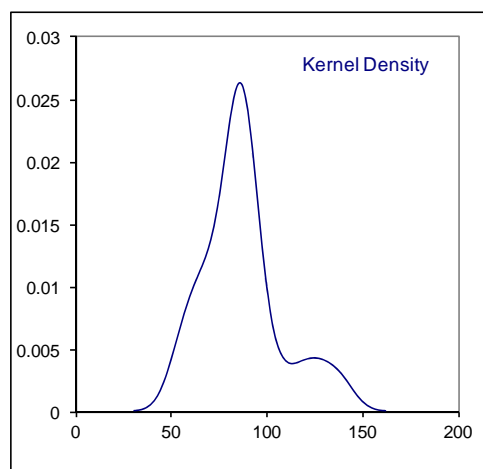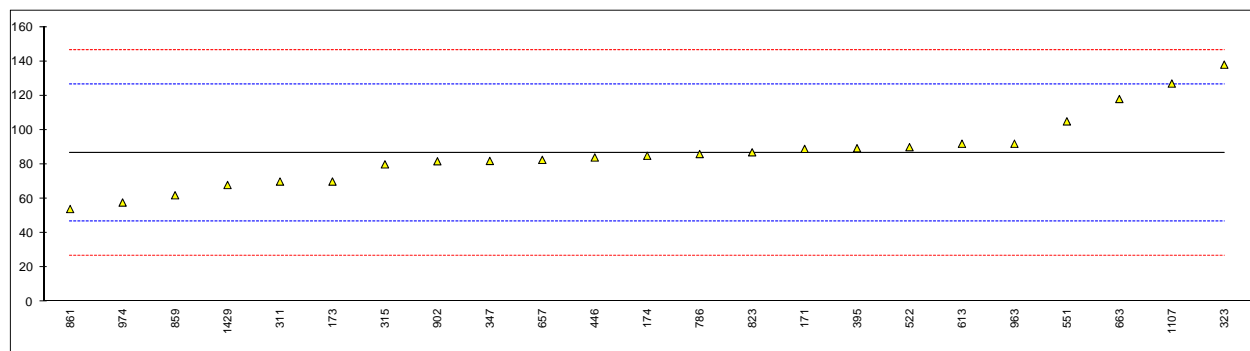
APPENDIX 2

Number of participants per country

1 lab in AUSTRALIA

2 labs in BELGIUM

1 lab in BRAZIL

2 labs in CHINA, People's Republic

1 lab in INDIA

1 lab in ITALY

1 lab in MEXICO

2 labs in NETHERLANDS

1 lab in RUSSIAN FEDERATION

1 lab in SAUDI ARABIA

1 lab in SINGAPORE

1 lab in SOUTH KOREA

1 lab in SPAIN

1 lab in THAILAND

1 lab in TURKEY

1 lab in UNITED ARAB EMIRATES

2 labs in UNITED KINGDOM

3 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C   = final result after checking of first reported suspect result
D(0.01)   = outlier in Dixon's outlier test
D(0.05)   = straggler in Dixon's outlier test
G(0.01)   = outlier in Grubbs' outlier test
G(0.05)   = straggler in Grubbs' outlier test
DG(0.01)   = outlier in Double Grubbs' outlier test
DG(0.05)   = straggler in Double Grubbs' outlier test
E   = error in calculations
ex   = excluded from calculations
n.e.   = not evaluated
wd   = withdrawn method

Literature:

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3   ASTM E1301:89
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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.
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15   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics,
            25(2), pp. 165-172, (1983)