Results of Proficiency Test
Acetic Acid
February 2011

CONTENTS

Appendices:

1   INTRODUCTION

Since 2003, the Institute for Interlaboratory Studies organizes a proficiency test for Acetic Acid. During the annual proficiency test program of 2010/2011, it was decided to continue the proficiency test for the analysis of Acetic Acid. In this interlaboratory study 28 laboratories in 16 different countries have participated. See appendix 2 for the number of participants per country.

In this report, the results of the proficiency test are presented and discussed. This report is electronically available through the iis internet site http://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 (500 mL) spiked with Iron(III) Chloride and Sodium Sulphate. Analyses for fit-for-use and homogeneity were subcontracted.

Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluations.

2.1   ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO guide 43 and ILAC-G13:2007, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures 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 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 Acetic Acid was obtained from a chemical producer. The approximately 25 litres of Acetic Acid was spiked with 314.8 mg Iron(III)Chloride.6H_2O and 217.6 mg Sodium Sulphate. After homogenisation, this material was divided over 50 brown glass bottles of 0.5 L and labelled #11004.
The homogeneity of the subsamples #11004 was checked by determination of Chloride and Sulphate with an in-house test method on 8 stratified randomly selected samples.

	Chloride in mg/kg	*Sulphate in mg/kg*
sample #11004-1	4.3	6.1
sample #11004-2	4.5	6.2
sample #11004-3	4.3	5.6
sample #11004-4	4.2	6.2
sample #11004-5	4.3	6.3
sample #11004-6	4.3	6.1
sample #11004-7	4.2	6.2
sample #11004-8	4.3	6.2

table 1: homogeneity test results of subsamples #11004

From the above test results the repeatabilities were calculated and compared with 0.3 times the respective reproducibility calculated using the Horwitz equation in agreement with the procedure of ISO 13528, Annex B2 in the next table;

	Chloride in mg/kg	*Sulphate in mg/kg*
r (sample #11004)	0.26	0.61
target	Horwitz	Horwitz
0.3*R (target)	0.46	0.63

table 2: repeatabilities of subsamples #11004

The calculated repeatabilities were both in agreement with 0.3 times the corresponding target reproducibility. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 1* 0.5 litre (labelled #11004) was sent on February 2, 2011.

2.5 STABILITY OF THE SAMPLES

The stability of Acetic Acid, packed in an amber glass bottle, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6   ANALYSES

The participants were asked to determine Acetaldehyde, Chloride as Cl, Colour Pt/Co, Density @ 20 ^{o}C, Formic Acid, Freezing Point, Iron as Fe, Nonvolatile Matter, Purity (estimated from Freezing Point), Purity (titration), Sulphate as SO_4 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' (iis-protocol, version 3.2) of January 2010.

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, some serious problems were encountered with despatch of the samples to the laboratories in India and Malaysia. Three participants reported after the final reporting date, two of them received the samples after the reporting date.
In total 236 numerical results were reported by 28 participants. Observed were 10 outlying results, which is 4.2% of the total of numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1   EVALUATION PER TEST

In this section, the results are discussed per test.

For comparison of the results of this interlaboratory study, the requirements from the specification ASTM D3620:09 "Standard Specification for Glacial Acetic Acid" were used. Unfortunately, for many determinations this specification is referring to ASTM E302 "Standard Test Methods for Monobasic Organic Acids", which was withdrawn in 2001 with no replacement.

For the determination of the Purity by Titration, the method used for comparison is ASTM E301:94, which was also withdrawn, with no replacement, in 2001. However, no other useful standardised method is published yet.
The target reproducibility used for the determination of the Purity by Freezing Point is calculated from the values in table 1 and the target reproducibility both from ASTM E302. For the other determinations without any stated reproducibilities the observed spreads were compared with the strict spreads estimated from the Horwitz equation.

A not-normal distribution was found for Chloride, Colour Pt/Co, Density @ 20°C, Formic Acid, Freezing Point, Purity (estimated from Freezing Point) and water. In these cases the statistical evaluations should be used with due care.

Acetaldehyde: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outlier, is in good agreement with the requirements of ASTM D2191:06.

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #11004, which is bright, clear and free of suspended matter or pass in accordance with ASTM E2680.

Chloride: This determination was problematic for two laboratories. One false negative test result was observed. The calculated reproducibility is in good agreement with the estimated reproducibility limit, calculated using the Horwitz equation. The Chloride content before spiking is not known. However, based on the assumption of zero content before spiking, the average recovery was estimated to be max. 95%.

Colour: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1209:11.

Density @ 20 ^{o}C: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is in good agreement with the requirements of ASTM D4052:02e1.

Formic Acid: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility, after rejection of the statistical outliers, is in good agreement with ASTM D3546:11.

Freezing Point: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the obsolete method ASTM E302:95.

Iron as Fe: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM E394:09. The Iron content before spiking is not known. However, based on the assumption of zero content before spiking, the average recovery was estimated to be max. 96%.

Nonvolatile Matter: This determination may be problematic for one laboratory. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is in good agreement with the requirements of ASTM D1353:09.

Purity (from FP): Regretfully, no suitable reference method with precision data exists for this determination. Therefore, a target reproducibility was calculated out of table 1 of the obsolete ASTM E302:95 and the reproducibility data of the obsolete ASTM E302:95.
No analytical problems were observed. No statistical outliers were observed. The calculated reproducibility is in good agreement with the estimated reproducibility limits.

Purity (titration): This determination was not problematic. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is in good agreement with the requirements of the obsolete method ASTM E301:94.

Sulphate as SO4: This determination may be problematic. No statistical outliers were observed. However, one false negative test result was reported and the calculated reproducibility is not in agreement with the requirements of

the estimated reproducibility limits using the Horwitz equation. The Sulphate content before spiking is not known. However, based on the assumption of zero content before spiking, the average recovery was estimated to be max. 147%.

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is in good agreement with the requirements of the obsolete method ASTM E302:95.

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 average results per sample, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM standards) are compared in the next tables.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acetaldehyde	mg/kg	9	11.0	6.3	80
Appearance		26	pass	n.a.	n.a.
Chloride	mg/kg	14	4.1	0.9	1.5
Colour	Pt/Co	24	11.3	4.6	7.0
Density @ 20°C	kg/L	25	1.0493	0.0002	0.0005
Formic Acid	mg/kg	16	17.2	44.8	360
Freezing Point	°C	24	16.45	0.11	0.25
Iron as Fe	mg/kg	23	2.2	0.4	1.0
Nonvolatile Matter	mg/100 mL	18	2.0	1.8	2.4
Purity (Freezing Point)	%M/M	26	99.90	0.06	0.13
Purity (Titration)	%M/M	10	99.89	0.12	0.54
Sulphate as SO_4	mg/kg	10	6.3	3.6	2.1
Water	%M/M	27	0.047	0.010	0.050

table 3: Reproducibilities for sample #11004

Without further statistical calculations, it can be concluded that for almost all tests there is a good compliance of the group of participating laboratories with the relevant standards. The tests that are problematic have been discussed in paragraph 4.1.

4.3   COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2011 WITH PREVIOUS PTS

	Febr. 2011	*March 2010*	*February 2008*	*March 2007*
Number of rep. participants	28	26	28	25
Number of results reported	236	193	197	181
Statistical outliers	10	8	7	17
Percentage outliers	4.2%	4.2%	3.6%	9.4%

table 4: comparison with previous proficiency tests.

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

Determination	*Febr. 2011*	*March 2010*	*February 2008*	*March 2007*
Acetaldehyde	++	++	++	++
Chloride	++	++	++	++
Colour	++	++	++	++
Density @ 20 ^{o}C	++	++	++	++
Formic Acid	++	++	++	++
Freezing Point	++	++	++	++
Iron as Fe	++	--	-	++
Nonvolatile matter	++	++	+	++
Purity (Freezing point)	++	++	++	++
Purity (Titration)	++	++	++	++
Sulphate as SO_4	--	--	n.e.	n.e.
Water	++	++	++	++

table 5: comparison determinations against the standard

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 #11004; results in mg/kg
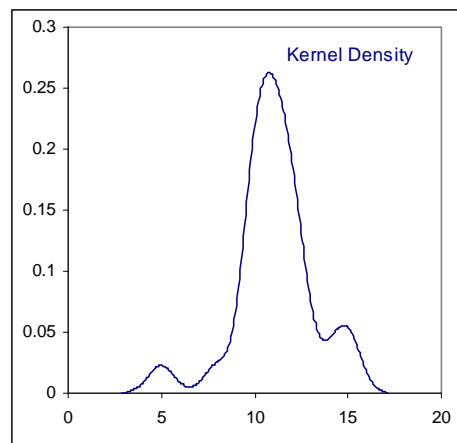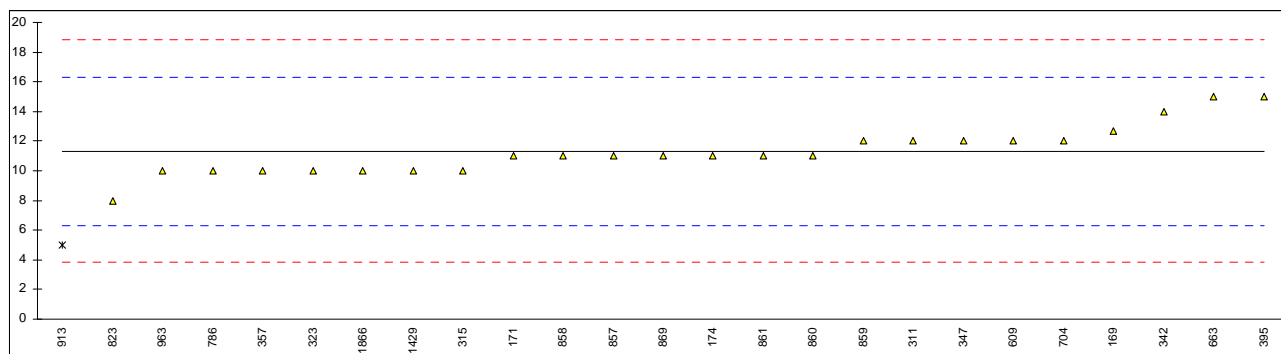
lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171		-----		-----	
174		-----		-----	
311	D2191	9		-0.07	
315		-----		-----	
319	INH-5033	<1		-----	
323	D2191	<10		-----	
342		-----		-----	
347		-----		-----	
357	INH-052	<50		-----	
359	INH-052	<50		-----	
395	INH-157	10		-0.04	
609	D2191	15.03		0.14	
663		-----		-----	
704	D2191	38.8	DG(0.01)	0.97	
786	INH-005	<10		-----	
823		-----		-----	
857	D2191	9		-0.07	
858	D2191	10		-0.04	
859	D2191	11		0.00	
860	D2191	9		-0.07	
861	D2191	14		0.10	
869	D2191	12		0.03	
913		-----		-----	
963	D2191	47.5	DG(0.01)	1.28	
1429		-----		-----	
1649	GC	<10		-----	
1866		-----		-----	

normality	OK
n	9
outliers	2
mean (n)	11.0
st.dev. (n)	2.24
R(calc.)	6.3
R(D2191:06)	80

Kernel Density
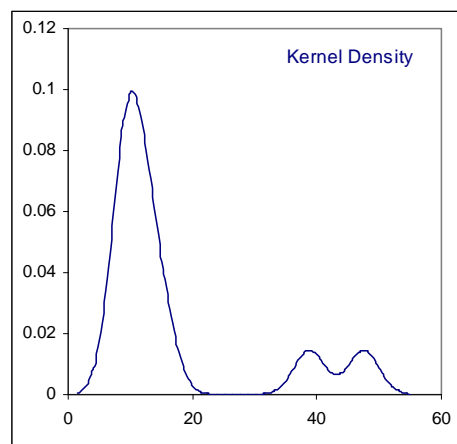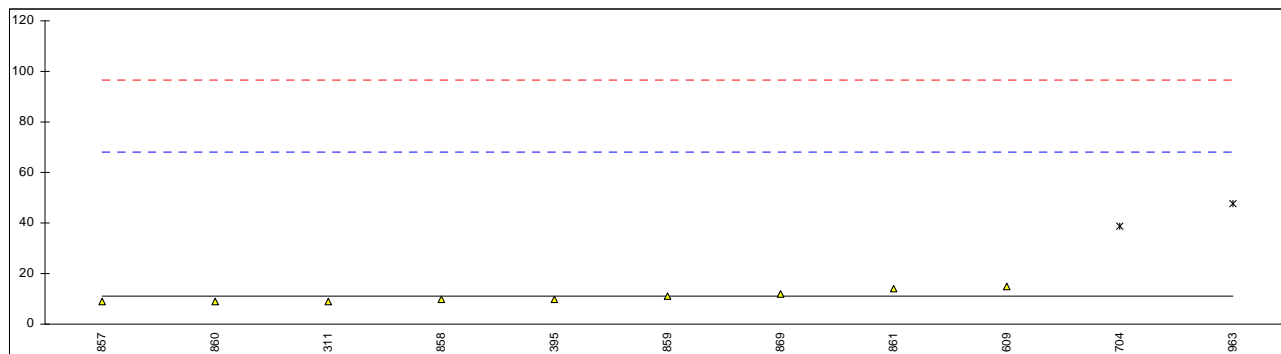
Determination of Appearance on sample #11004;

lab	method	value	mark	z(targ)	remarks
169	E2680	PASS		-----	
171	E2680	CF		-----	
174	E2680	CF		-----	
311	E2680	PASS		-----	
315	inh-402	CBFFSM		-----	
319	INH-5036	CLEAR		-----	
323	E2680	CFFSM		-----	
342	E2680	PASS		-----	
347	E2680	PASS		-----	
357	E2680	PASS		-----	
359	E2680	PASS		-----	
395	E2680	PASS		-----	
609	INH111	CFMI		-----	
663	E2680	PASS		-----	
704	E2680	PASS		-----	
786	E2680	PASS		-----	
823	E2680	PASS		-----	
857	E2680	PASS		-----	
858	E2680	PASS		-----	
859	E2680	PASS		-----	
860	E2680	PASS		-----	
861	E2680	PASS		-----	
869	E2680	PASS		-----	
913	E2680	CFSM		-----	
963	E2680	PASS		-----	
1429	VISUAL	BC		-----	
1649		-----		-----	
1866		-----		-----	

|		
	normality	n.a.
	n	n.a.
	outliers	n.a.
	mean (n)	n.a.
	st.dev. (n)	n.a.
	R(calc.)	n.a.
	R(lit.)	unknown

Determination of Chloride as Cl on sample #11004; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171		-----		-----	
174		-----		-----	
311	INH-158	4.3		0.48	
315	INH-158	4.385		0.64	
319	INH-5039	<1		<-5.81	false negative?
323	INH-008	4.2		0.29	
342		-----		-----	
347		-----		-----	
357	INH-709	4.3		0.48	
359	INH-709	4.4		0.67	
395		-----		-----	
609		-----		-----	
663		-----		-----	
704	INH-19814	4		-0.09	
786	INH-19814	<4	C	<-0.09	first reported 3
823	INH-279	4.2		0.29	
857	INH-001	4.08		0.06	
858	INH-012	4.0		-0.09	
859	INH-012	4.0		-0.09	
860	INH-012	4.1		0.10	
861	INH-001	4		-0.09	
869	INH-001	3.5		-1.04	
913		-----		-----	
963		-----		-----	
1429	INHOUSE	0.2	G(0.01)	-7.33	
1649	turb.	3.2		-1.62	
1866		-----		-----	

normality	not OK			
n	14			
outliers	1	Spike:		
mean (n)	4.05	4.28		(recovery <95%)
st.dev. (n)	0.333			
R(calc.)	0.93			
R(Horwitz)	1.47			
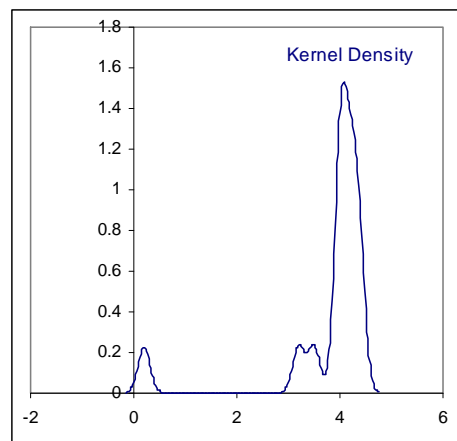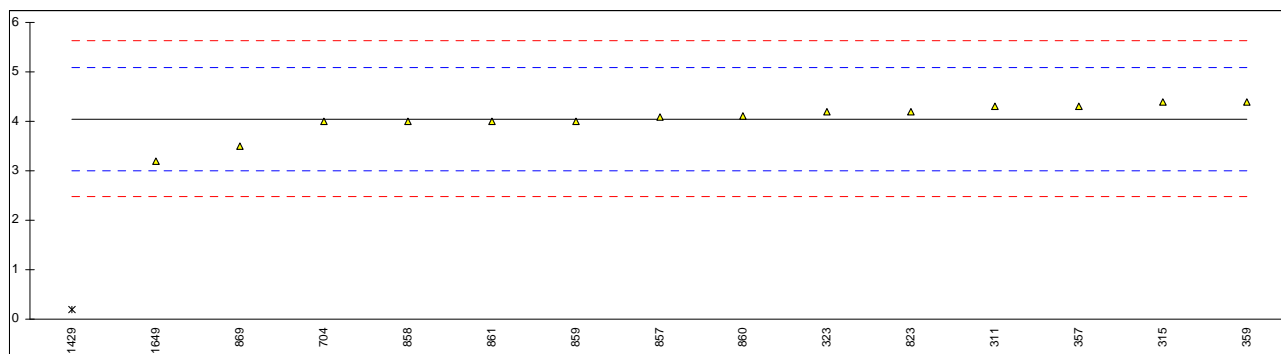
Determination of Colour Pt/Co on sample #11004

lab	method	value	mark	z(targ)	remarks
169	D5386	12.7		0.55	
171	D5386	11		-0.13	
174	E302	11		-0.13	
311	E302	12		0.27	
315	D1209	10		-0.53	
319		-----		-----	
323	D1209	10		-0.53	
342	D5386	14		1.07	
347	D1209	12		0.27	
357	D1209	10		-0.53	
359	D1209	5-10		-----	OFF HUE
395	D1209	15		1.47	
609	D1209	12		0.27	
663	D1209	15		1.47	
704	D1209	12		0.27	
786	D1209	10		-0.53	
823	E302	8		-1.33	
857	E302	11		-0.13	
858	E302	11		-0.13	
859	E302	12		0.27	
860	D5386	11		-0.13	
861	E302	11		-0.13	
869	E302	11		-0.13	
913	E302	5.0	G(0.05)	-2.53	
963	E302	10		-0.53	
1429	D6667	10		-0.53	
1649		-----		-----	
1866	E302	10		-0.53	

normality	not OK
n	24
outliers	1
mean (n)	11.3
st.dev. (n)	1.65
R(calc.)	4.6
R(D1209:11)	7.0

Kernel Density

Determination of Density @ 20 ^{o}C on sample #11004; results in kg/L

lab	method	value	mark	z(targ)	remarks
169	D4052	1.0490	G(0.01)	-1.51	
171	D4052	1.0493		0.17	
174	D4052	1.0494		0.73	
311	D4052	1.0493		0.17	
315	D4052	1.0493		0.17	
319		-----		-----	
323	D4052	1.0492		-0.39	
342	D4052	1.0492		-0.39	
347	D4052	1.04929		0.12	
357	D4052	1.0492		-0.39	
359	D4052	1.0492		-0.39	
395	D4052	1.0492		-0.39	
609	D4052	1.04922		-0.28	
663	D4052	1.0493		0.17	
704	D4052	1.0493		0.17	
786	D4052	1.0493		0.17	
823	D4052	1.04927		0.00	
857	D4052	1.04925		-0.11	
858	D4052	1.04924		-0.16	
859	D4052	1.04925		-0.11	
860	D4052	1.04922		-0.28	
861	D4052	1.04926		-0.05	
869	D4052	1.04935		0.45	
913	D4052	1.0494		0.73	
963	D4052	1.0492		-0.39	
1429	D4052	1.0493		0.17	
1649		-----		-----	
1866	D4052	1.04929		0.12	

|	
normality	not OK
n	25
outliers	1
mean (n)	1.04927
st.dev. (n)	0.000059
R(calc.)	0.00017
R(D4052:02e1)	0.00050

Kernel Density
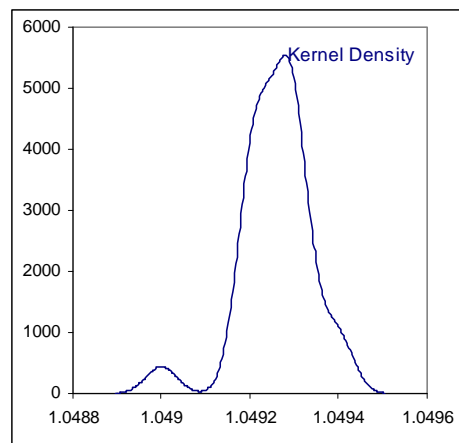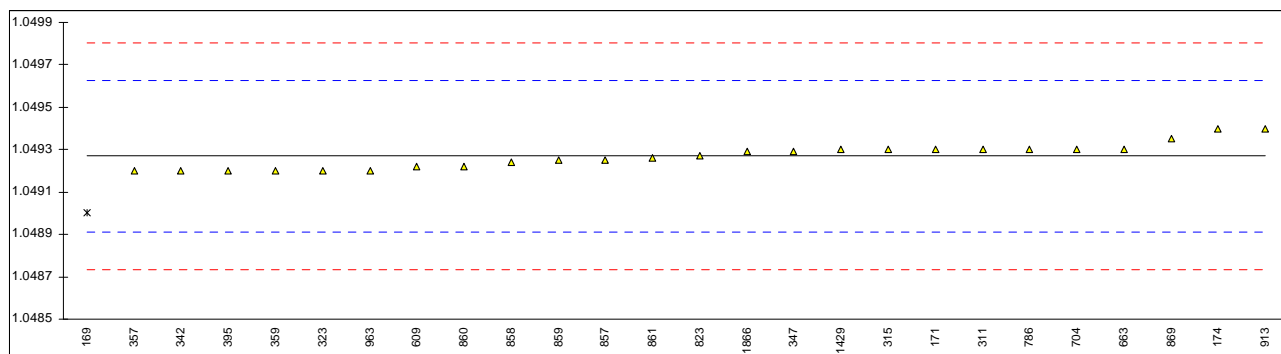
Determination of Formic Acid on sample #11004; results in mg/kg
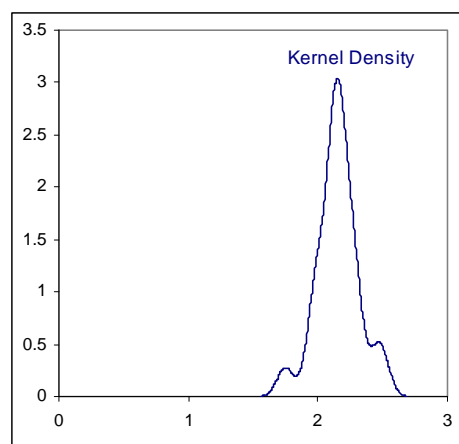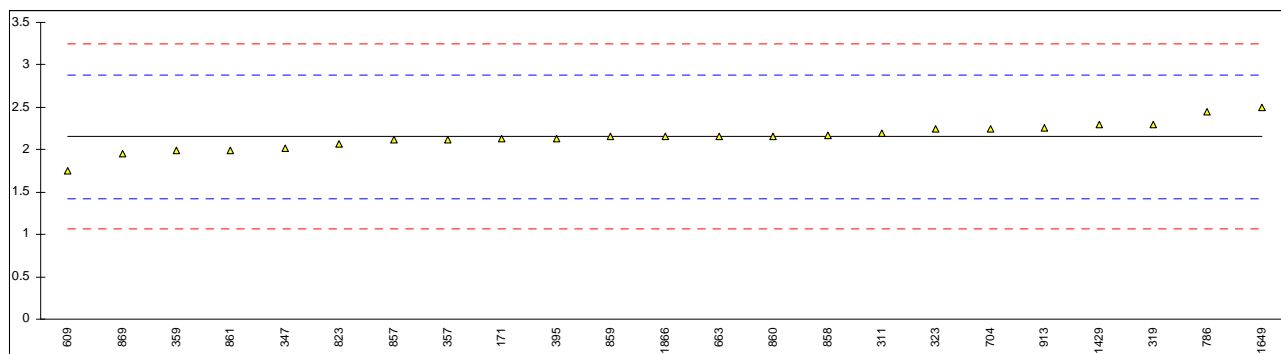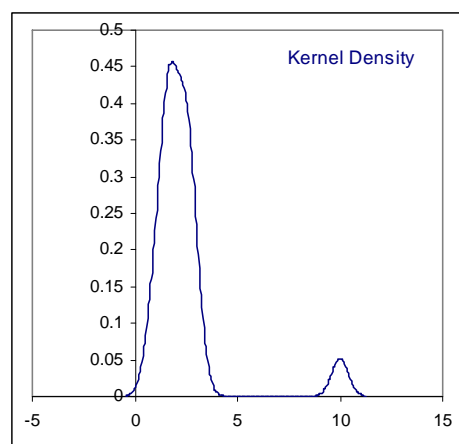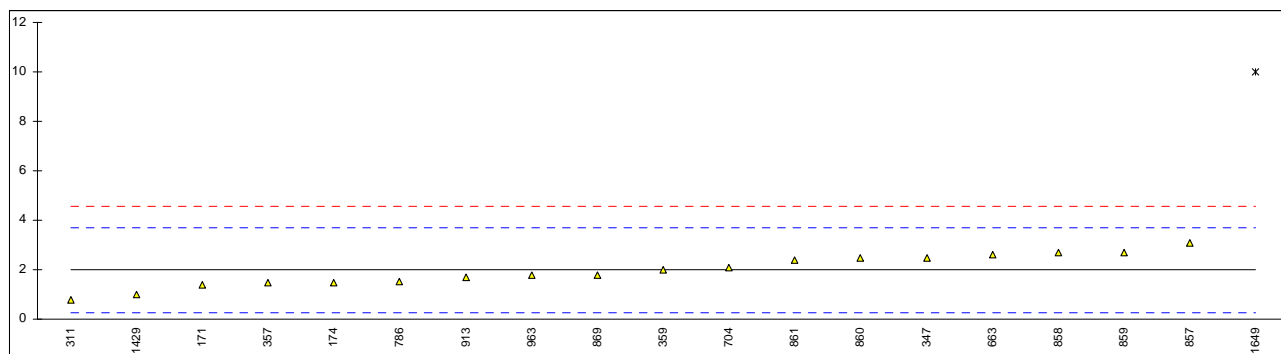
lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171		-----		-----	
174		-----		-----	
311	D3546	11		-0.05	
315		-----		-----	
319	INH-5032	11		-0.05	
323	D3546	70		0.41	
342		-----		-----	
347	D3546	30		0.10	
357	D3546	18		0.01	
359	D3546	26		0.07	
395		-----		-----	
609	D3546	16.1		-0.01	
663		-----		-----	
704	D3546	152	G(0.01)	1.05	
786	INH-19814	163	C,G(0.05)	1.13	first reported 63
823		-----		-----	
857	D3546	7.0		-0.08	
858	D3546	8.6		-0.07	
859	D3546	6.9		-0.08	
860	D3546	9		-0.06	
861	D3546	9		-0.06	
869	D3546	9		-0.06	
913		-----		-----	
963	D3546	25		0.06	
1429	INH-23	3.9		-0.10	
1649	GC	15		-0.02	
1866		-----		-----	

normality	not OK
n	16
outliers	2
mean (n)	17.2
st.dev. (n)	15.99
R(calc.)	44.8
R(D3546:11)	360

Kernel Density
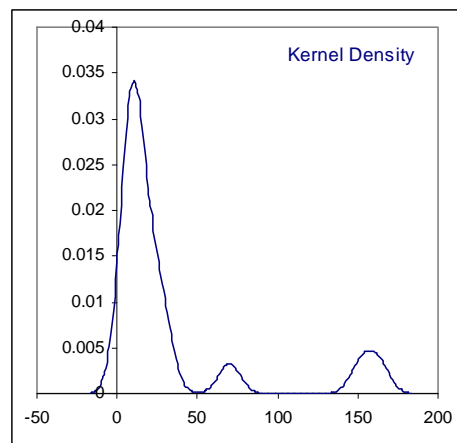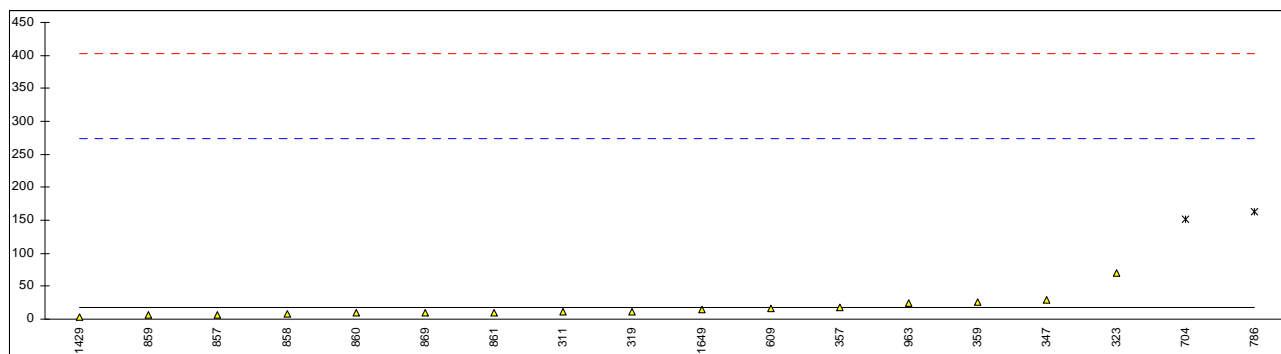
Determination of Freezing Point on sample #11004; results in °C

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171	E302	16.5		0.58	
174	E302	16.5		0.58	
311	E302	16.50		0.58	
315	D1493	16.48		0.36	
319		-----		-----	
323	D1493	16.45		0.02	
342	E302	16.45		0.02	
347	E302	16.45		0.02	
357	E302	16.45		0.02	
359	E302	16.50		0.58	
395	INH-124	16.4		-0.54	
609	inh70013	16.40		-0.54	
663	D6875	16.40		-0.54	
704	INH-61	16.40		-0.54	
786	E302	16.5		0.58	
823	E302	16.42		-0.31	
857	E302	16.48		0.36	
858	E302	16.46		0.14	
859	E302	16.46		0.14	
860	E302	16.45		0.02	
861	E302	16.40		-0.54	
869	E302	16.40		-0.54	
913	E302	16.4		-0.54	
963	E302	16.40		-0.54	
1429	E302	16.5		0.58	
1649		-----		-----	
1866		-----		-----	

normality	not OK
n	24
outliers	0
mean (n)	16.448
st.dev. (n)	0.0405
R(calc.)	0.114
R(E302:95)	0.250

Kernel Density
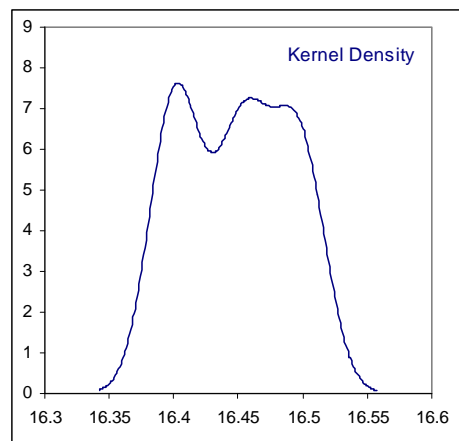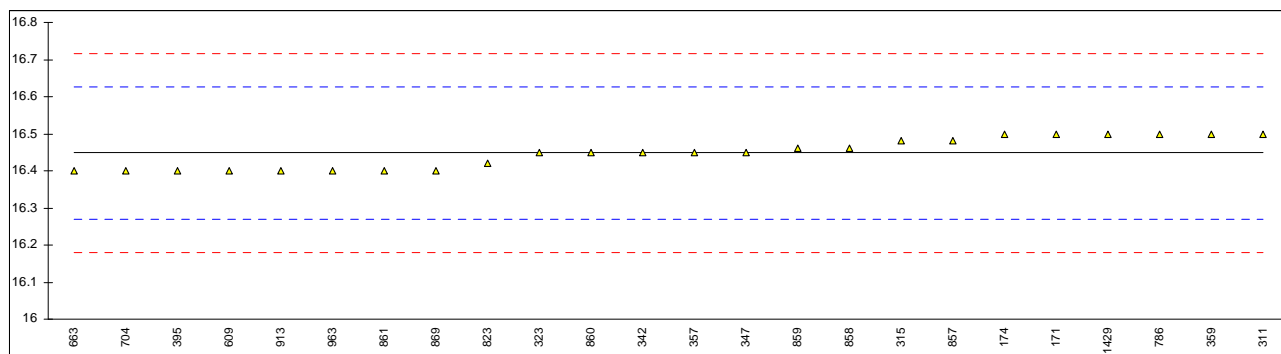
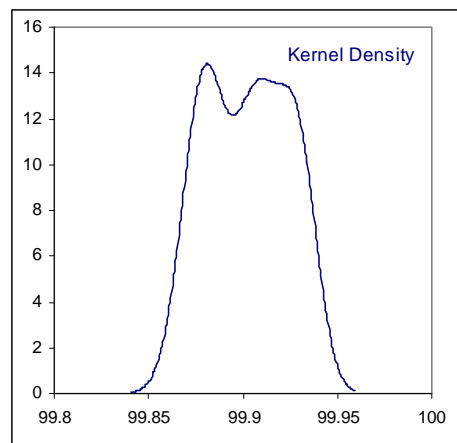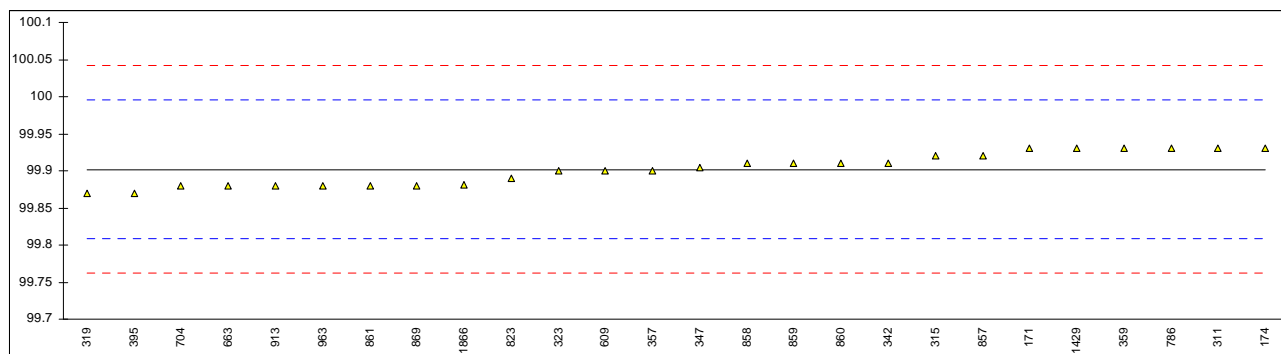## Determination of Iron as Fe on sample #11004; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171	E394	2.127		-0.07	
174		-----		-----	
311	E394	2.19		0.10	
315		-----		-----	
319	E394	2.3		0.40	
323	E394	2.24		0.24	
342		-----		-----	
347	E394	2.02	C	-0.37	first reported 2.52
357	D3620	2.12		-0.09	
359	D3620	1.99		-0.45	
395	E394	2.132		-0.06	
609	E394	1.75		-1.11	
663	E394	2.15		-0.01	
704	E394	2.247		0.26	
786	E394	2.45	C	0.81	first reported 1.600
823	E394	2.07		-0.23	
857	E394	2.12		-0.09	
858	E394	2.17		0.04	
859	E394	2.15		-0.01	
860	E394	2.16		0.02	
861	E394	1.99		-0.45	
869	E394	1.954		-0.55	
913	E394	2.26		0.29	
963		-----		-----	
1429	E394	2.3		0.40	
1649	Phot.	2.5		0.95	
1866	E394	2.15		-0.01	

	normality	OK			
	n	23			
	outliers	0	spike:		
	mean (n)	2.154	2.25		recovery <96%
	st.dev. (n)	0.1602			
	R(calc.)	0.448			
	R(E394:09)	1.021			

Kernel Density

Determination of Nonvolatile Matter on sample #11004; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171	D1353	1.4		-0.68	
174	D1353	1.5		-0.56	
311	D1353	0.8		-1.38	
315		-----		-----	
319	INH-5041	<2		-----	
323		-----		-----	
342		-----		-----	
347	D1353	2.5		0.61	
357	D1353	1.5		-0.56	
359	D1353	2.0		0.02	
395		-----		-----	
609		-----		-----	
663	D1353	2.6		0.72	
704	D1353	2.1		0.14	
786	D1353	1.53		-0.52	
823		-----		-----	
857	D1353	3.1		1.31	
858	D1353	2.7		0.84	
859	D1353	2.7		0.84	
860	D1353	2.5		0.61	
861	D1353	2.4		0.49	
869	D1353	1.8		-0.21	
913	D1353	1.7		-0.33	
963	D1353	1.8		-0.21	
1429	D1353	1		-1.14	
1649	USP	10	G(0.01)	9.36	
1866		-----		-----	

```
normality      OK
n              18
outliers       1
mean (n)       1.979
st.dev. (n)    0.6372
R(calc.)       1.784
R(D1353:09)    2.400
```


Kernel Density

Determination of Purity (estimated from the Freezing Point) on sample #11004; results in %M/M

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171	E302	99.93		0.60	
174	E302	99.93		0.60	
311	E302	99.93		0.60	
315	E302	99.92		0.38	
319	ISO1392	99.87		-0.69	
323	E302	99.90		-0.05	
342	E302	99.91		0.17	
347	E302	99.905		0.06	
357	E302	99.90		-0.05	
359	E302	99.93		0.60	
395	INH-124	99.87		-0.69	
609	inh70014	99.90		-0.05	
663	E302	99.88		-0.48	
704	E302	99.88		-0.48	
786	E302	99.93		0.60	
823	E302	99.89		-0.26	
857	E302	99.92		0.38	
858	E302	99.91		0.17	
859	E302	99.91		0.17	
860	E302	99.91		0.17	
861	E302	99.88		-0.48	
869	E302	99.88		-0.48	
913	E302	99.88		-0.48	
963	E302	99.88		-0.48	
1429	E302	99.93		0.60	
1649		-----		-----	
1866	GC	99.8811		-0.45	

|	
normality	not OK
n	26
outliers	0
mean (n)	99.902
st.dev. (n)	0.0211
R(calc.)	0.059
R(E302:95)	0.130

Kernel Density

Determination of Purity (by titration) on sample #11004; results in %M/M

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171	E301	99.88		-0.07	
174		-----		-----	
311	E301	99.97		0.40	
315		-----		-----	
319		-----		-----	
323	E301	99.88		-0.07	
342		-----		-----	
347		-----		-----	
357		-----		-----	
359		-----		-----	
395		-----		-----	
609		-----		-----	
663	INH-576	99.88		-0.07	
704		-----		-----	
786		-----		-----	
823		-----		-----	
857	E301	99.922		0.15	
858	E301	99.927		0.18	
859		-----		-----	
860		-----		-----	
861	E301	99.87		-0.12	
869		-----		-----	
913	E301	99.90		0.04	
963	E301	99.90		0.04	
1429		-----		-----	
1649	ISO753-2	99.8		-0.48	
1866	E301	99.645	G(0.01)	-1.29	

|	
normality	OK
n	10
outliers	1
mean (n)	99.893
st.dev. (n)	0.0444
R(calc.)	0.124
R(E301:94)	0.540

Kernel Density
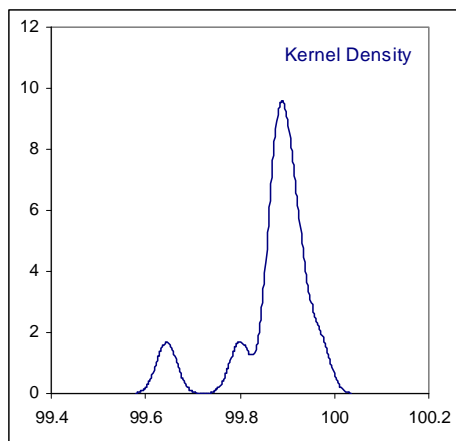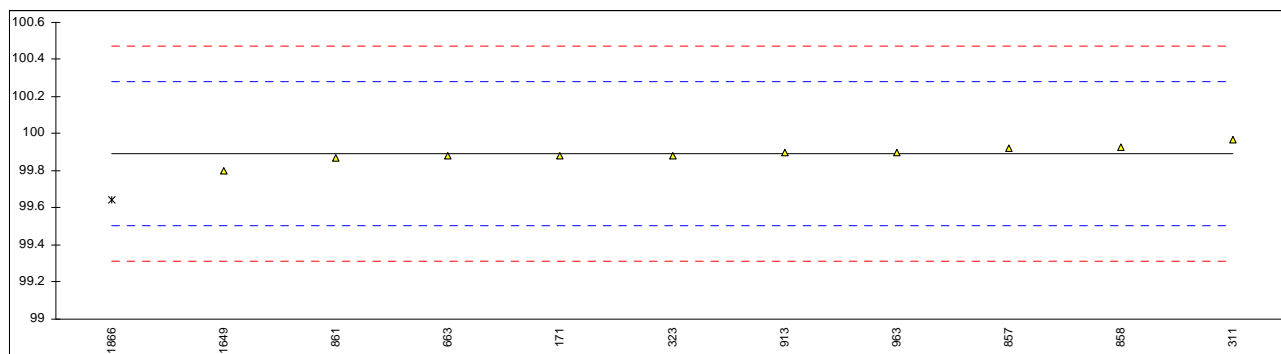
Determination of Sulphate as SO_4 on sample #11004, results in mg/kg

lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171		-----		-----	
174		-----		-----	
311		-----		-----	
315		-----		-----	
319	INH-5037	8		2.26	
323		-----		-----	
342		-----		-----	
347		-----		-----	
357	INH-857	5		-1.68	
359	INH-857	5		-1.68	
395		-----		-----	
609		-----		-----	
663		-----		-----	
704	INH-19814	4.8		-1.94	
786	INH-19814	5.0		-1.68	
823		-----		-----	
857	INH-70021	7		0.94	
858	INH-70021	7		0.94	
859	INH-70021	7		0.94	
860	INH-9728	6		-0.37	
861		-----		-----	
869	INH-70021	8		2.26	
913		-----		-----	
963		-----		-----	
1429		-----		-----	
1649	TURB.	<0.3		<-7.85	false negative?
1866		-----		-----	

normality	OK		
n	10		
outliers	0	spike:	
mean (n)	6.28	4.28	recovery <147%
st.dev. (n)	1.276		
R(calc.)	3.57		
R(Horwitz)	2.13		
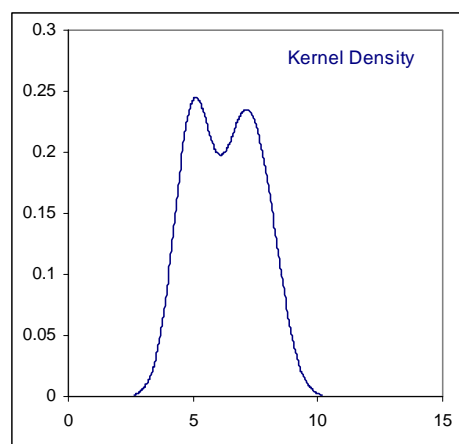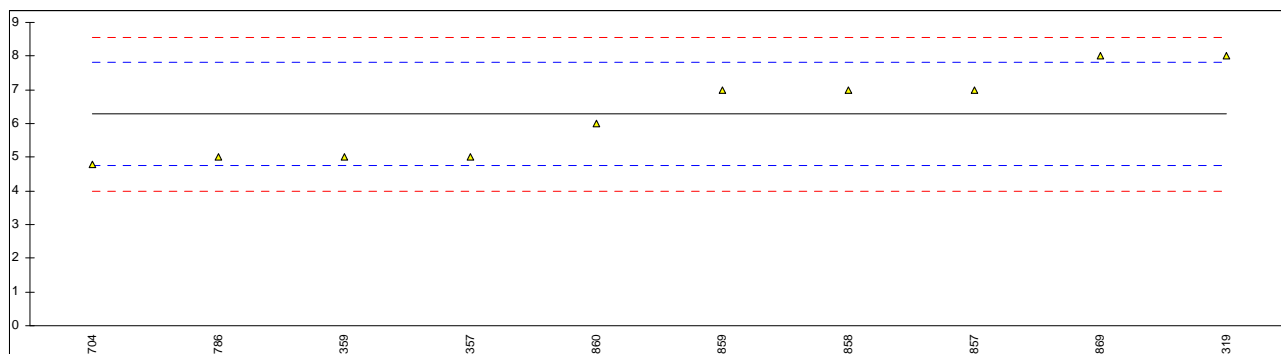
Determination of Water on sample #11004, results in %M/M

lab	method	value	mark	z(targ)	remarks
169	E1064	0.0452		-0.08	
171	E302	0.0452	C	-0.08	first reported 0.0616
174	E302	0.0452	C	-0.08	first reported 0.0604
311	E302	0.049		0.13	
315	E203	0.04735		0.04	
319	INH-5008	0.05		0.19	
323	E203	0.052		0.30	
342	E1064	0.0452		-0.08	
347	E1064	0.0447		-0.11	
357	E1064	0.046		-0.04	
359	E203	0.045		-0.09	
395	E1064	0.04603		-0.04	
609	D1364	0.048		0.08	
663	E1064	0.045		-0.09	
704	E302	0.043		-0.20	
786	E1064	0.0453		-0.08	
823	E302	0.047		0.02	
857	E302	0.0457		-0.05	
858	E302	0.0448		-0.10	
859	E302	0.0436		-0.17	
860	E1064	0.045		-0.09	
861	E302	0.0434		-0.18	
869	E302	0.0511		0.25	
913	E302	0.054		0.41	
963	E302	0.04		-0.37	
1429	D1364	0.047		0.02	
1649	KF-titr.	0.056		0.52	
1866	E302	0.066	G(0.01)	1.08	

normality	not OK
n	27
outliers	1
mean (n)	0.0467
st.dev. (n)	0.00348
R(calc.)	0.0097
R(E302:95)	0.0500

Kernel Density
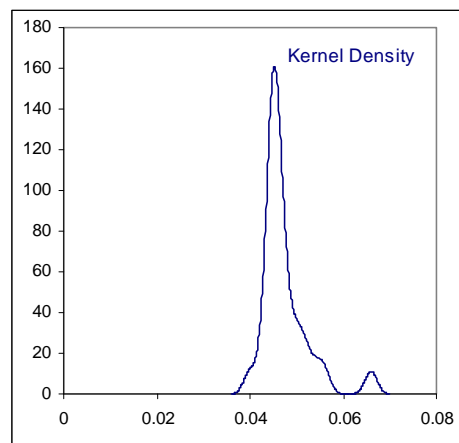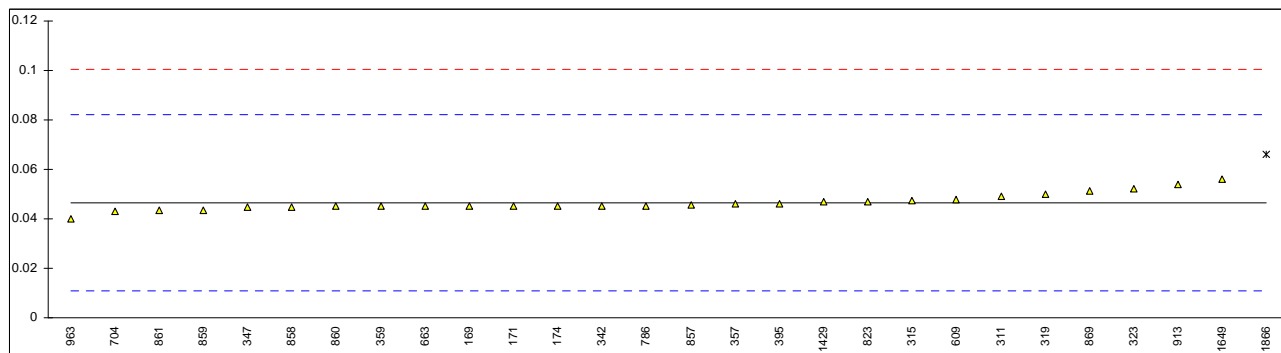
APPENDIX 2

Number of participants per country

1 lab in	AUSTRIA
1 lab in	BELGIUM
2 labs in	FINLAND
1 lab in	INDIA
1 lab in	ITALY
1 lab in	KOREA
1 lab in	MALAYSIA
6 labs in	P.R. of CHINA
1 lab in	RUSSIA
2 labs in	SAUDI ARABIA
2 labs in	SPAIN
1 lab in	THAILAND
3 labs in	THE NETHERLANDS
3 labs in	U.S.A.
1 lab in	UKRAINE
1 lab 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
U   = error in reporting unit
ex   = excluded from calculations
n.a.   = not applicable
wd   = withdrawn method

Literature:

1   iis 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/)