Results of Proficiency Test
Acetone
September 2013

Organised by: Institute for Interlaboratory Studies
                Spijkenisse, the Netherlands

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1   INTRODUCTION

Since 1999, the Institute for Interlaboratory Studies organizes on regular basis a proficiency test for the analysis of Acetone. As part of the annual proficiency test program of 2013/2014, the Institute decided to continue this proficiency test on Acetone. In this interlaboratory study, 23 laboratories from 14 different countries have participated. See appendix 2 for the number of participants per country.
In this report, the results of the 2013 Acetone PT are presented and discussed.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. The analyses for fit-for-use and homogeneity determination were subcontracted. It was decided to send one sample Acetone (1*1L bottle, labelled #13162) to the participants. The participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Also, customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2), which can be downloaded from www.iisnl.com.

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 bulk material of Acetone was purchased from a local Chemical supplier. The approx. 35 litre bulk sample was homogenised in a pre-cleaned drum. After homogenisation, the bulk sample was divided over 32 brown glass bottles of one litre (labelled #13162). The homogeneity of the subsamples #13162 was checked by determination of the Density in accordance with ASTM D4052:11 on 4 stratified randomly selected samples.

	Density @20ºC in kg/L
sample #13162-1	0.79617
sample #13162-2	0.79617
sample #13162-3	0.79617
sample #13162-4	0.79617

Table 1: homogeneity test results of subsample #13162

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density @20ºC in kg/L
r (observed)	0.00000
reference method	D 4052:11
0.3 x R (ref. method)	0.00015

Table 2: repeatabilities of subsample #13162

The repeatability of the test results for Density on sample #13162 was in full agreement with the repeatability as required by the standard. Therefore, homogeneity of subsample #13162 was assumed.

One sample of acetone (a bottle of 1L, labelled #13162) was sent to each of the participating laboratories on August 21, 2013.

2.5 STABILITY OF THE SAMPLES

The stability of the Acetone, packed in the brown glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine: Acidity, Aldehydes, Appearance, Chloride as Cl, Colour as Pt/Co, Density @ 20°C, Specific gravity 25/25ºC, Distillation (IBP, MBP and DP), Water Miscibility, Nonvolatile Matter, Purity "as received", Purity on dry basis, Diacetonalcohol, Mesityloxide, Methanol, Permanganate Time Test @ 25°C, Refractive

Index @ 20°C and Water on sample #13162 in accordance with specification ASTM D329:13.

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 gathered. The original data are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to laboratories that had not reported 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' 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 to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty

failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; no.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, 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 target reproducibility (preferably taken from a standardized test method) by division with 2.8.
The z-scores were calculated in accordance with:

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

The $z_{(target)}$ scores are listed in the result tables in appendix 1.

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.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, 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 the dispatch of the samples to the Brazil and India, due to custom clearance problems. From the total of 23 participants, only two participants did not report any test result. In total 273 numerical results were reported. Observed were 7 outlying results, which is 2.6%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1   EVALUATION PER TEST

Not all original data sets proved to have a normal distribution. For Specific Gravity, IBP and MBP non-Gaussian distributions were found and therefore the statistical evaluation for these determinations should be used with care. In this section, the results are discussed per test.

Acidity: No analytical problems were observed. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1613:12.

Aldehydes: No conclusions could be drawn. Only three participants reported a numerical result. One of these laboratories reported a numerical result in accordance with an in house test method. Four other laboratories used test method ASTM D329:13 that describes a pass/fail test.

Appearance: No analytical problems were observed. All labs agreed about the appearance of sample #13162, which is bright, clear and free of suspended matter.

Chloride: It is hard to draw conclusions, because the chloride concentration was near or below the detection limit.

Colour: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1209:11.

Density @ 20°C: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D4052:11.

Specific Gravity 25/25°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D4052:11.

Distillation: This determination was not problematic. No statistical outliers were observed. All three calculated reproducibilities (for IBP, MBP and DP) were in agreement with the requirements of ASTM D1078:11. From the reported results of the Mid Boiling Point (automated), it was concluded that all participating laboratories, except one may have corrected the results properly for barometric pressure and thermometer accuracy as described in ASTM D1078:11.

Water Miscibility: This determination was not problematic. All laboratories reported this test pass. The analytical method described in ASTM D1722:09 is a pass/fail test.

NVM: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1353:13.

Purity as received: For this determination no statistical outliers were noticed. The calculated reproducibility is small in comparison with the 2011 PT "iis11C07" (0.043 vs 0.049).

Purity on DB: No statistical outliers were observed. The calculated reproducibility is large in comparison with the calculated reproducibility of the 2011 PT "iis11C07" (0.028 vs 0.010).

Diacetonalcohol: This determination may have been problematic. One statistical outlier is observed. However, the calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated requirements, calculated using the Horwitz equation.

Mesityloxide: This determination is not problematic. One statistical outlier is observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the estimated requirements, calculated using the Horwitz equation.

Methanol: This determination was problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated requirements, calculated using the Horwitz equation.

PTT:

This determination was not problematic. Only one statistical outlier was observed. All participants agreed on a result far above 30 minutes. When a statistical evaluation is performed on the actually reported results, the calculated reproducibility is not in agreement with the requirements of ASTM D1363:11. However, as it is unknown whether a Permanganate Time Test result of >100 minutes is in the applicability range, it is therefore difficult to draw any conclusions. Therefore, no z-scores were calculated.

Refractive index:

This test was not problematic. One statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D1218:12.

Water:

This determination was problematic for one laboratory. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D1364:12.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The 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)
Acidity as acetic acid	%M/M	21	0.0012	0.0008	0.0014
Aldehydes	---	7	n.a.	n.a.	n.a.
Appearance	---	17	pass	n.a.	n.a.
Chloride as Cl	mg/kg	6	<1	n.a.	n.a.
Colour Pt/Co	---	15	2.9	3.6	7.0
Density @ 20 °C	kg/L	19	0.7905	0.0002	0.0005
Specific Gravity 25/25ºC		12	0.7871	0.0004	0.0005
Initial Boiling Point	°C	19	55.92	0.36	0.87
Mid Boiling Point	°C	18	56.09	0.24	0.38
Dry Point	°C	19	56.33	0.38	0.60
Miscibility with water	---	18	pass	n.a.	n.a.
Nonvolatile Matter	mg/100 mL	15	0.39	0.93	2.11
Purity as received	%M/M	14	99.764	0.043	unknown
Purity on dry basis	%M/M	16	99.959	0.028	unknown
Diacetonalcohol	mg/kg	14	46.0	21.7	11.6
Mesityloxide	mg/kg	14	31.8	8.3	8.5
Methanol	mg/kg	13	317.9	76.7	59.8
Permanganate Time Test	min	15	108	55	(27)
Refractive Index	---	11	1.3587	0.0004	0.0005
Water	mg/kg	18	1987	278	270

Table 3: Reproducibilities for sample #13162

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2013 WITH PREVIOUS PT'S

	September 2013	September 2011	September 2009	September 2007
Number of reporting labs	21	18	19	18
Number of results reported	273	198	216	157
Statistical outliers	7	3	7	8
Percentage outliers	2.6%	1.5%	3.2%	5.1%

Table 4: comparison of summary data 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	September 2013	September 2011	September 2009	September 2007
Acidity as acetic acid	++	+	++	++
Chloride as Cl	n.e.	n.e.	n.e.	n.e.
Colour Pt/Co	++	++	++	++
Density @ 20 ^{o}C	++	++	++	++
Specific gravity 25/25ºC	+	n.e.	n.e.	n.e.
Distillation	++	++	++	++
Nonvolatile Matter	++	++	++	++
Diacetonalcohol	--	--	--	--
Mesityloxide	+/-	n.e.	+	n.e.
Methanol	-	+	++	n.e.
Permanganate Time Test	(--)	n.e.	n.e.	n.e.
Refractive Index	+	+/-	-	--
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 Acidity on sample #13162; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D1613	0.0019		1.43	
171	D1613	0.0011	C	-0.17	First reported 11
174	D1613	0.0010		-0.37	
311	D1613	0.0011		-0.17	
323	D1613	0.0011		-0.17	
343	D1613	0.0015		0.63	
345	D1613	0.00136	C	0.35	First reported 0.0001
349	D1613	0.001091		-0.19	
357	D1613	0.00091		-0.55	
444	D1613	0.00141		0.45	
445	D1613	0.0012		0.03	
551		-----		-----	
657	D1613	0.0010		-0.37	
840	D1613	0.00160		0.83	
886	D1613	0.0011		-0.17	
902	D1613	0.00085		-0.67	
912	D1613	0.0012		0.03	
913	D1613	0.00122		0.07	
963		-----		-----	
1016	D1613	0.0014		0.43	
1201	D1613	0.0012		0.03	
1429	D1613	0.0009		-0.57	
2493	D1613	0.0007566		-0.86	

normality	OK
n	21
outliers	0
mean (n)	0.00119
st.dev. (n)	0.000271
R(calc.)	0.00076
R(D1613:12)	0.00140

Kernel Density
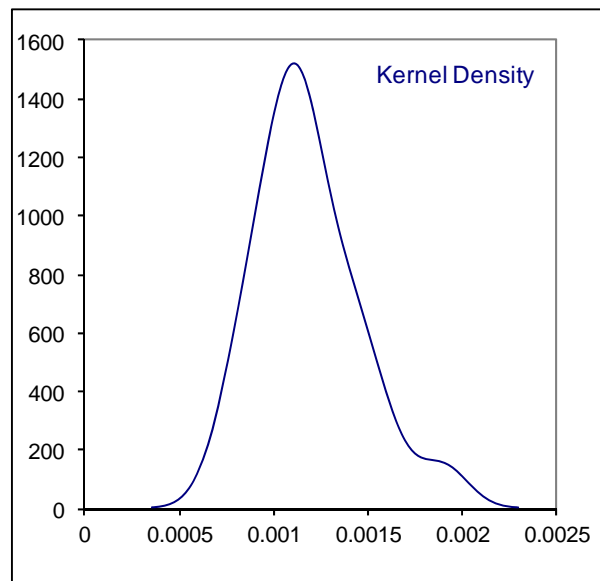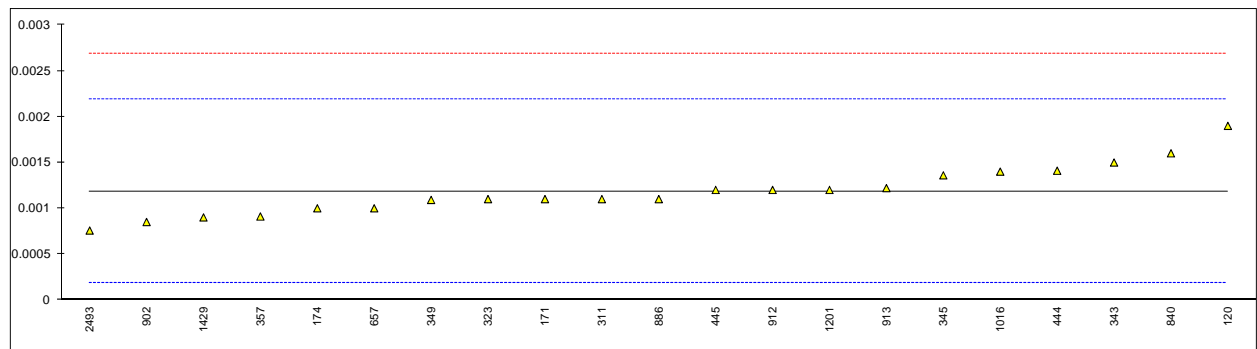
Determination of Aldehydes on sample #13162;

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171	D329Mod	1		-----	
174		-----		-----	
311	D329	Pass		-----	
323	D329	Pass		-----	
343		-----		-----	
345	E4714	49.1		-----	
349		-----		-----	
357	D329	Pass		-----	
444		-----		-----	
445		-----		-----	
551		-----		-----	
657		-----		-----	
840	D329	Pass		-----	
886		-----		-----	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1016		-----		-----	
1201		-----		-----	
1429		-----		-----	
2493	D329	36.8		-----	
	normality	n.a.			
	n	4			
	outliers	0			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D329:13)	n.a.			

Determination of Appearance on sample #13162;

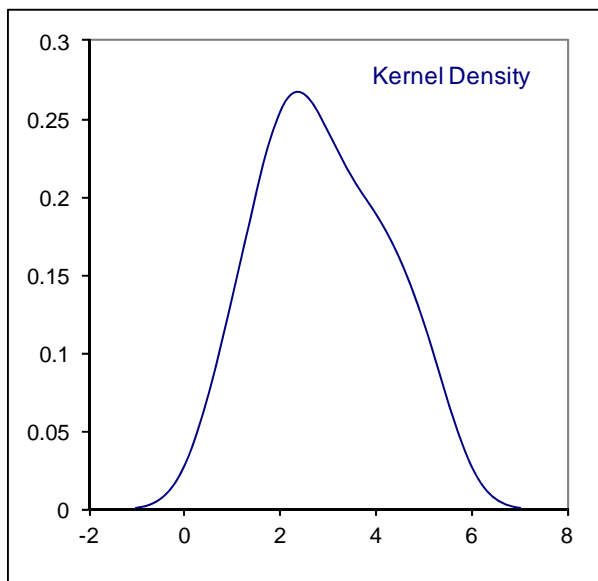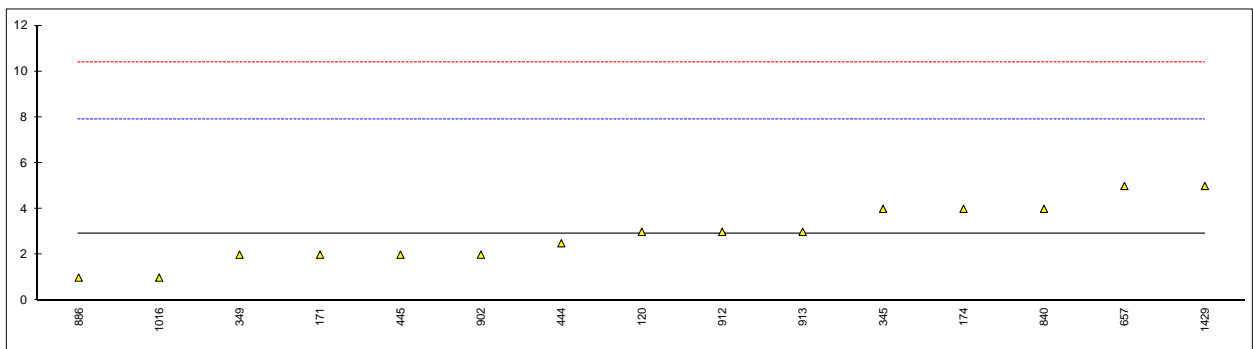| lab	method	value	mark	z(targ)	remarks
120	E2680	Pass		-----	
171	E2680	Pass		-----	
174	E2680	Pass		-----	
311	E2680	Pass		-----	
323	E2680	CFSM		-----	
343	E2680	Pass		-----	
345	Visual	Pass		-----	
349	E2680	Pass		-----	
357	E2680	Pass		-----	
444	E2680	Pass		-----	
445	E2680	Pass		-----	
551		-----		-----	
657	E2680	Pass		-----	
840	E2680	Pass		-----	
886		-----		-----	
902	E2680	Pass		-----	
912	E2680	Pass		-----	
913	E2680	Pass		-----	
963		-----		-----	
1016	in house	CFSM		-----	
1201	E2680	B&C		-----	
1429	E2680	B&C		-----	
2493		-----		-----	

Abbreviations:
B&C: bright and clear
CFSM: clear from suspended matter

Determination of Chloride as Cl on sample #13162; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171	Titrimetric	0.07		-----	
174	INH-IC	0.07		-----	
311	INH-158	<0.2		-----	
323	INH-008	<1		-----	
343		-----		-----	
345		-----		-----	
349		-----		-----	
357	Titrimetric	<0.5		-----	
444		-----		-----	
445		-----		-----	
551		-----		-----	
657		-----		-----	
840	IMPCA002	0.14		-----	
886		-----		-----	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1016		-----		-----	
1201		-----		-----	
1429		-----		-----	
2493		-----		-----	

normality	n.a.	
n	6	
outliers	0	
mean (n)	<1	
st.dev. (n)	n.a.	
R(calc.)	n.a.	
R(Horwitz)	n.a.	

Determination of Colour as Pt/Co on sample #13162;

lab	method	value	mark	z(targ)	remarks
120	D1209	3		0.04	
171	D1209	2		-0.36	
174	D1209	4		0.44	
311	D1209	<5		-----	
323	D1209	<5		-----	
343	D1209	L5		-----	
345	D1209	4		0.44	
349	D5386	2		-0.36	
357	D1209	<5		-----	
444	D5386	2.5		-0.16	
445	D1209	2		-0.36	
551		-----		-----	
657	D1209	5		0.84	
840	D1209	4		0.44	
886	D1209	1		-0.76	
902	D1209	2		-0.36	
912	D5386	3		0.04	
913	D1209	3		0.04	
963		-----		-----	
1016	D1209	1		-0.76	
1201	D1209	<5		-----	
1429	D1209	5		0.84	
2493		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	2.9
st.dev. (n)	1.28
R(calc.)	3.6
R(D1209:11)	7.0

Kernel Density

Determination of Density @ 20°C on sample #13162; results in kg/L

lab	method	value	mark	z(targ)	remarks
120	D4052	0.79047		0.06	
171	D4052	0.7904		-0.34	
174	D4052	0.7903		-0.90	
311	D4052	0.79040		-0.34	
323	D4052	0.7904		-0.34	
343	D4052	0.7904		-0.34	
345	D4052	0.7905		0.22	
349		-----		-----	
357	D4052	0.79046		0.00	
444	D4052	0.79047		0.06	
445	D4052	0.7905		0.22	
551		-----		-----	
657	D4052	0.79047		0.06	
840	D4052	0.79043		-0.17	
886	D4052	0.7904		-0.34	
902	D4052	0.79054		0.45	
912	D4052	0.7905		0.22	
913	D4052	0.7905		0.22	
963		-----		-----	
1016	D4052	0.7906		0.78	
1201	D4052	0.7905		0.22	
1429	D4052	0.7905		0.22	
2493	INH-872	0.793	G(0.01)	14.22	

normality	OK
n	19
outliers	1
mean (n)	0.79046
st.dev. (n)	0.000067
R(calc.)	0.00019
R(D4052:11)	0.00050
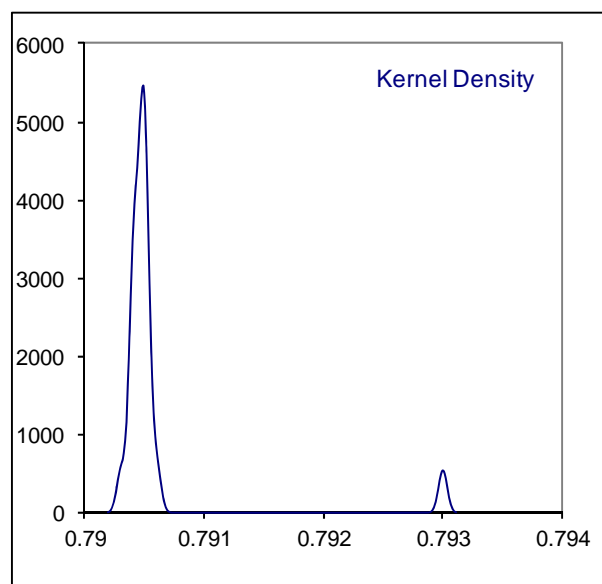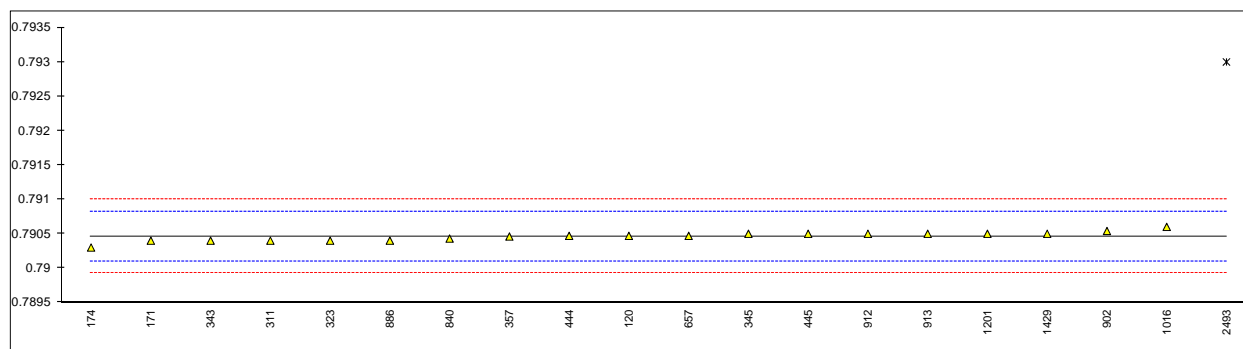
Determination of Specific Gravity 25/25 °C on sample #13162;

lab	method	value	mark	z(targ)	remarks
120	D4052	0.78711		0.19	
171	D4052	0.7869		-0.99	
174	D4052	0.7868	C	-1.55	First reported 0.7880
311		-----		-----	
323		-----		-----	
343	D4052	0.7874		1.81	
345		-----		-----	
349		-----		-----	
357	D4052	0.78713		0.30	
444		-----		-----	
445		-----		-----	
551		-----		-----	
657	D4052	0.7871		0.13	
840	D4052	0.78705		-0.15	
886		-----		-----	
902	D4052	0.78713		0.30	
912	D4052	0.7871		0.13	
913	D4052	0.7871		0.13	
963		-----		-----	
1016		-----		-----	
1201	D4052	0.7871		0.13	
1429	D4052	0.7870		-0.43	
2493		-----		-----	

normality	not OK	
n	12	
outliers	0	
mean (n)	0.78708	
st.dev. (n)	0.000144	
R(calc.)	0.00040	
R(D4052:11)	0.00050	
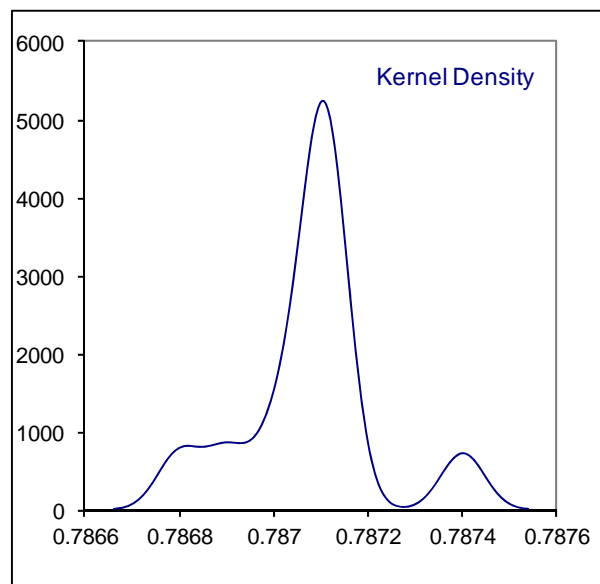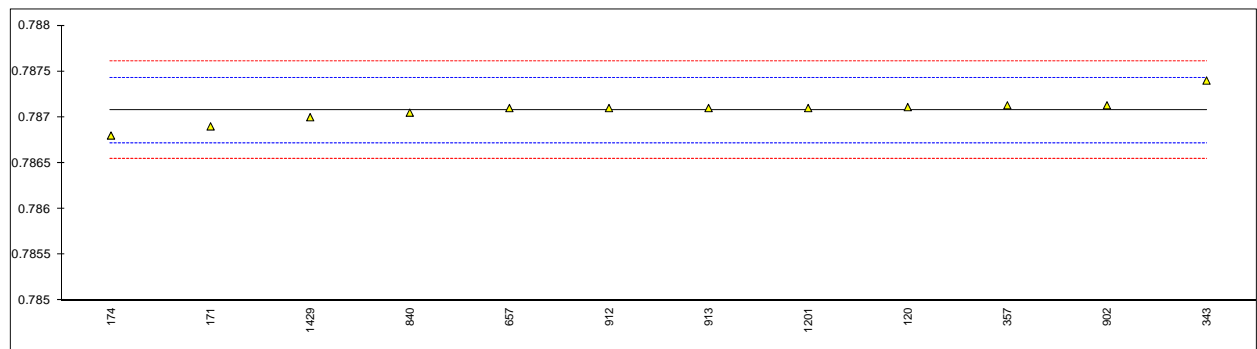
Determination of IBP, MBP and DP @ 760 mmHg on sample #13162; results in °C

lab	method	IBP	mark	z(targ)	MBP	mark	z(targ)	DP	mark	z(targ)	remarks
120	D1078-A	55.7		-0.71	56.1		0.05	56.3		-0.16	
171	D1078-A	56.0		0.25	56.1		0.05	56.2		-0.63	
174	D1078-A	56.0		0.25	56.1		0.05	56.2		-0.63	
311	D1078-A	56.0		0.25	56.1		0.05	56.3		-0.16	
323	D1078-M	55.9		-0.07	56.1		0.05	56.3		-0.16	
343	D1078-A	55.9		-0.07	56.1		0.05	56.3		-0.16	
345	D1078-A	55.6		-1.03	55.8	see §4.1	-2.14	56.0		-1.55	
349		-----		-----	-----		-----	-----		-----	
357	D1078-A	56.0		0.25	56.1		0.05	56.3		-0.16	
444	D1078	55.8		-0.39	-----		-----	56.6		1.23	
445	D1078-M	55.98		0.19	56.08		-0.10	56.39		0.26	
551		-----		-----	-----		-----	-----		-----	
657	D1078-M	56.0		0.25	56.1		0.05	56.4		0.30	
840	D1078-A	55.84		-0.26	56.10		0.05	56.27		-0.30	
886	D1078	56.0		0.25	56.2		0.78	56.4		0.30	
902	D1078-M	55.9		-0.07	56.2		0.78	56.4		0.30	
912	D1078-M	56.0		0.25	56.1		0.05	56.4		0.30	
913	D1078-M	55.9		-0.07	56.1		0.05	56.5		0.77	
963		-----		-----	-----		-----	-----		-----	
1016	D1078-A	56.2		0.89	56.2		0.78	56.5		0.77	
1201	D1078-A	55.9		-0.07	56.1		0.05	56.4		0.30	
1429	D1078-A	55.9		-0.07	56.0		-0.68	56.2		-0.63	
2493		-----		-----	-----		-----	-----		-----	
	normality	not OK			not OK			OK			
	n	19			18			19			
	outliers	0			0			0			
	mean (n)	55.92			56.09			56.33			
	st.dev. (n)	0.129			0.087			0.135			
	R(calc.)	0.36			0.24			0.38			
	R(D1078:11-A)	0.87			0.38			0.60			
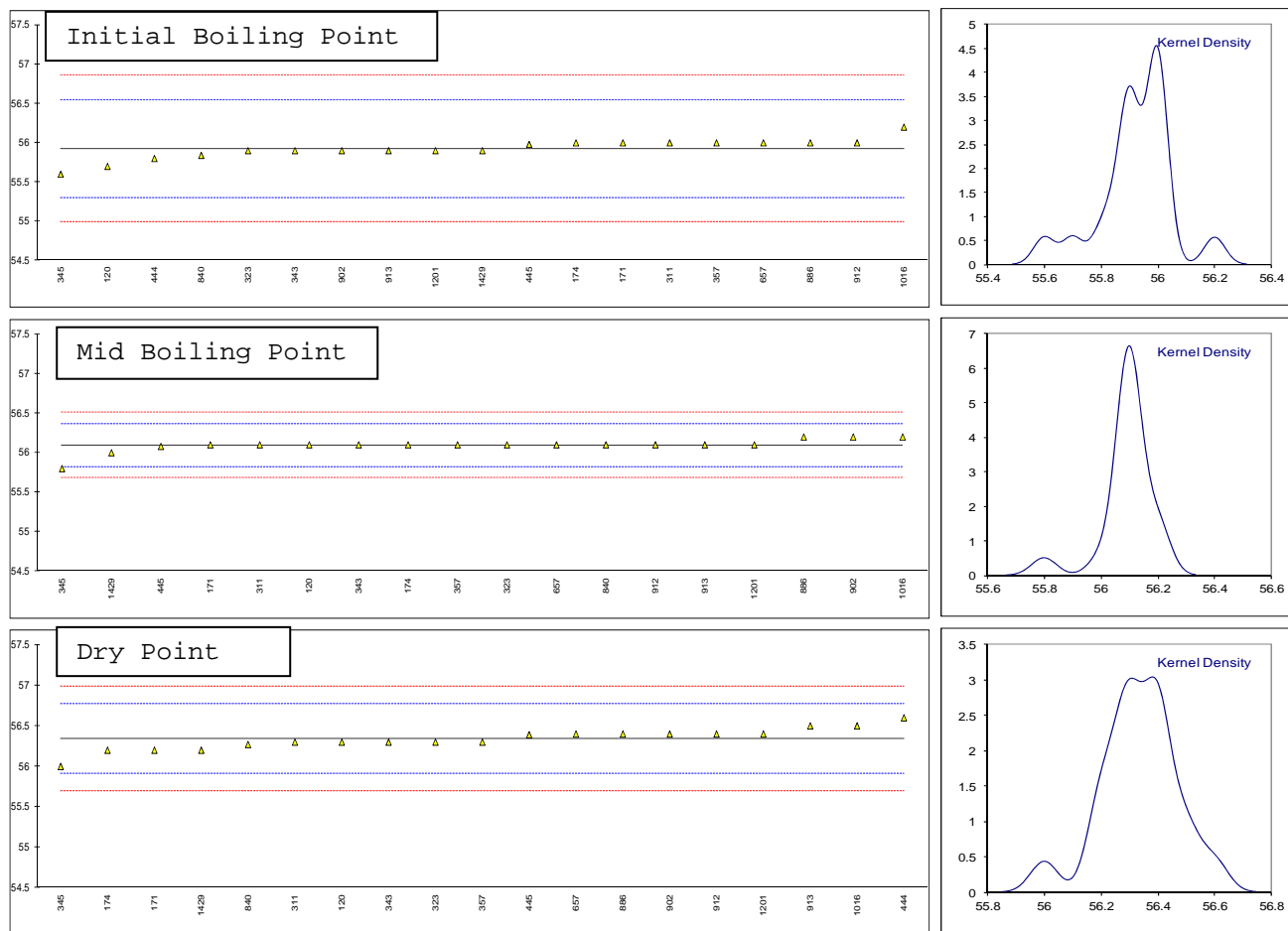
Determination of Miscibility with Water on sample #13162;

lab	method	value	mark	z(targ)	remarks
120	D1722	Pass		-----	
171	D1722	Pass		-----	
174	D1722	Pass		-----	
311	D1722	Pass		-----	
323	D1722	Pass		-----	
343		-----		-----	
345	D1722	Pass		-----	
349		-----		-----	
357	D1722	Pass		-----	
444	D1722	Pass		-----	
445	D1722	Pass		-----	
551		-----		-----	
657	D1722	Pass		-----	
840	D1722	Pass		-----	
886		-----		-----	
902	D1722	Pass		-----	
912	D1722	Pass		-----	
913	D1722	Pass		-----	
963		-----		-----	
1016	D1722	Pass		-----	
1201	D1722	Pass		-----	
1429	D1722	Pass		-----	
2493	D1722	Pass		-----	

normality	n.a.
n	18
outliers	0
mean (n)	Pass
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D1722:09)	n.a.

Determination of Nonvolatile Matter on sample #13162; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
120	D1353	<0.1		-----	
171	D1353	0.03		-0.48	
174	D1353	0.5		0.15	
311	D1353	<1		-----	
323	D1353	1		0.81	
343	D1353	<0.1		-----	
345	D1353	0		-0.52	
349		-----		-----	
357	D1353	< 1		-----	
444	D1353	0.2		-0.25	
445	D1353	1		0.81	
551		-----		-----	
657	D1353	0.3		-0.12	
840	D1353	0.4		0.01	
886	D1353	0.3		-0.12	
902	D1353	0.3		-0.12	
912	D1353	0.8		0.55	
913	D1353	0.601		0.28	
963		-----		-----	
1016	D1353	0.1		-0.38	
1201	D1353	0.3		-0.12	
1429	D1353	0		-0.52	
2493		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	0.39
st.dev. (n)	0.333
R(calc.)	0.93
R(D1353:13)	2.11

Kernel Density
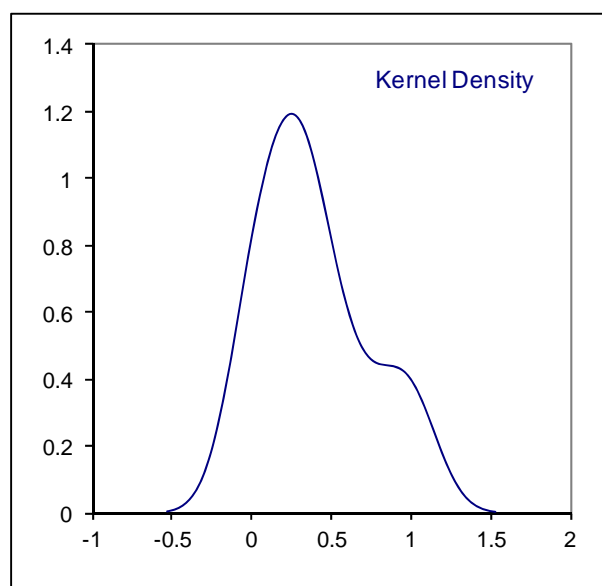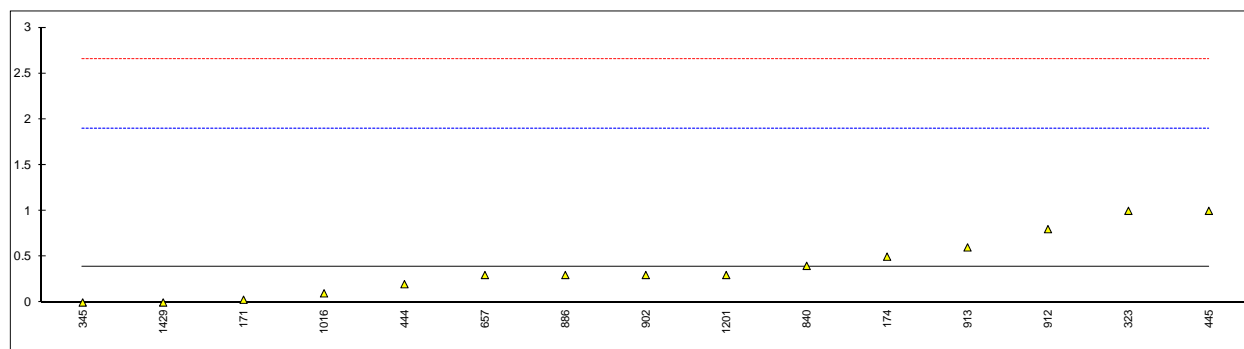
Determination of Purity "as received" on sample #13162, results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171		99.78		-----	
174	D6438	99.754		-----	
311		-----		-----	
323	INH-020	99.75		-----	
343	in house	99.7724	C	-----	First reported 99.971
345	E4714	99.7432		-----	
349		-----		-----	
357	INH-054	99.766		-----	
444	INH-001	99.7538		-----	
445		-----		-----	
551		-----		-----	
657	Calc.	99.7606		-----	
840	INH-005	99.762		-----	
886	D329	99.79		-----	
902	INH-125	99.74		-----	
912	in house	99.78		-----	
913	D2804	99.784		-----	
963		-----		-----	
1016		-----		-----	
1201		99.76		-----	
1429		-----		-----	
2493		-----		-----	

|		
normality	OK	
n	14	
outliers	0	
mean (n)	99.7640	
st.dev. (n)	0.01545	
R(calc.)	0.0433	
R(lit)	unknown	Compare R(iis11C07) = 0.0485
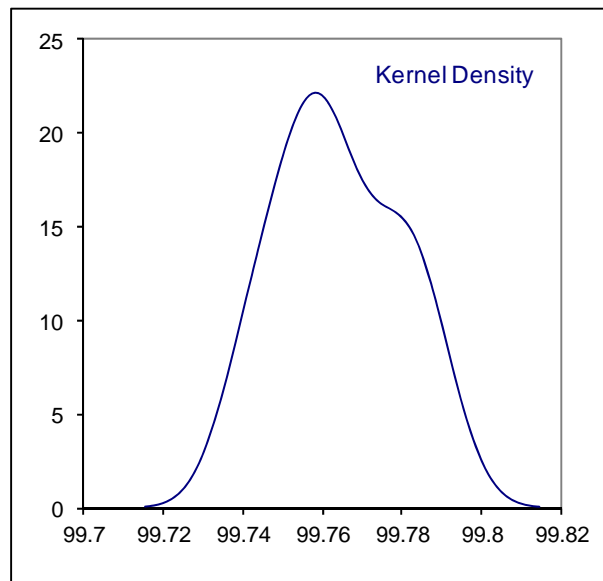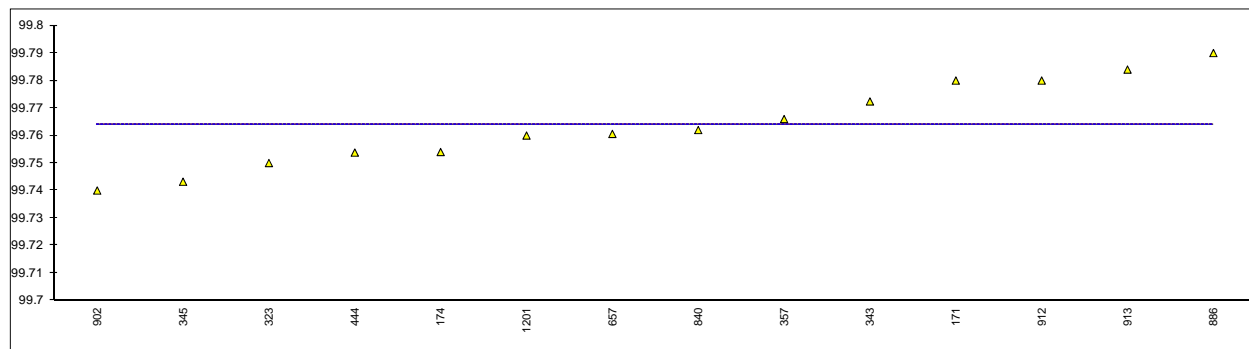
Determination of Purity on Dry Basis on sample #13162, results in %M/M

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171		99.98		-----	
174	D6438	99.958		-----	
311	INH-394	99.953	C	-----	First reported 99.94
323	INH-020	99.95		-----	
343	in house	99.971	C	-----	First reported 99.7724
345	E7414	99.9504		-----	
349	INH-034	99.9562		-----	
357	INH-054	99.964		-----	
444	INH-001	99.9530		-----	
445		-----		-----	
551		-----		-----	
657	INH-009	99.9556		-----	
840	INH-005	99.958		-----	
886		-----		-----	
902	INH-125	99.95		-----	
912	in house	99.97		-----	
913	D2804	99.971		-----	
963		-----		-----	
1016	DIN55687	99.9408		-----	
1201		99.96		-----	
1429		-----		-----	
2493		-----		-----	

normality	OK	
n	16	
outliers	0	
mean (n)	99.9588	
st.dev. (n)	0.01012	
R(calc.)	0.0283	
R(lit)	unknown	Compare R(iis11C07) = 0.0099
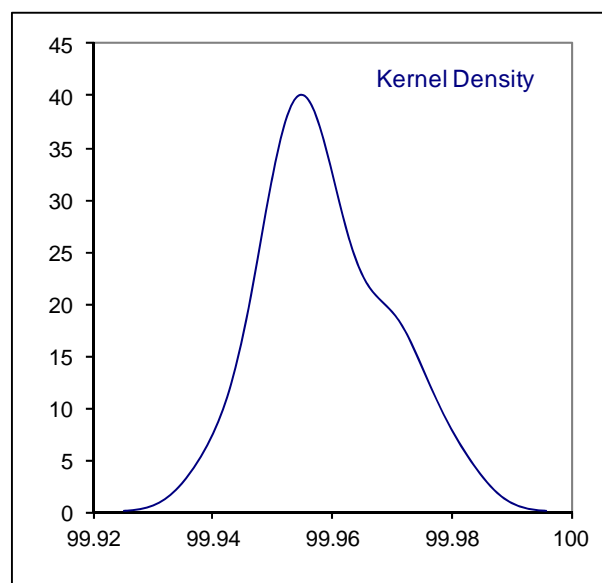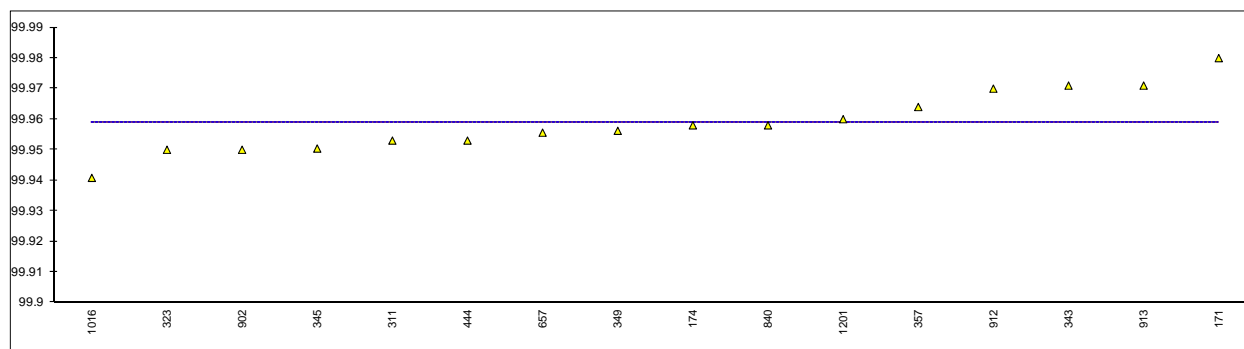
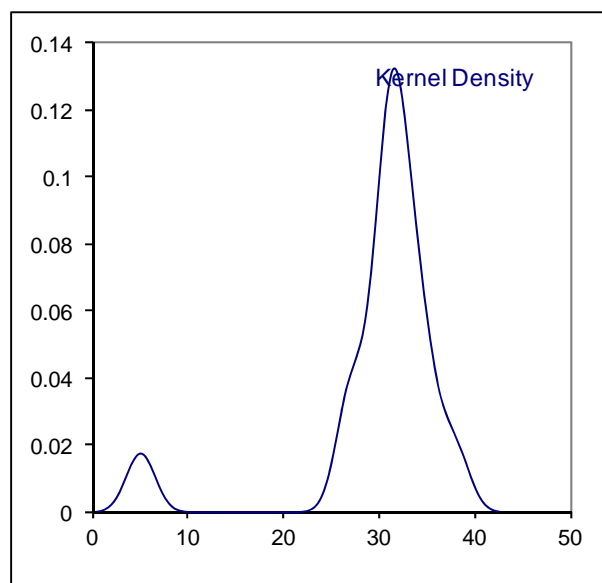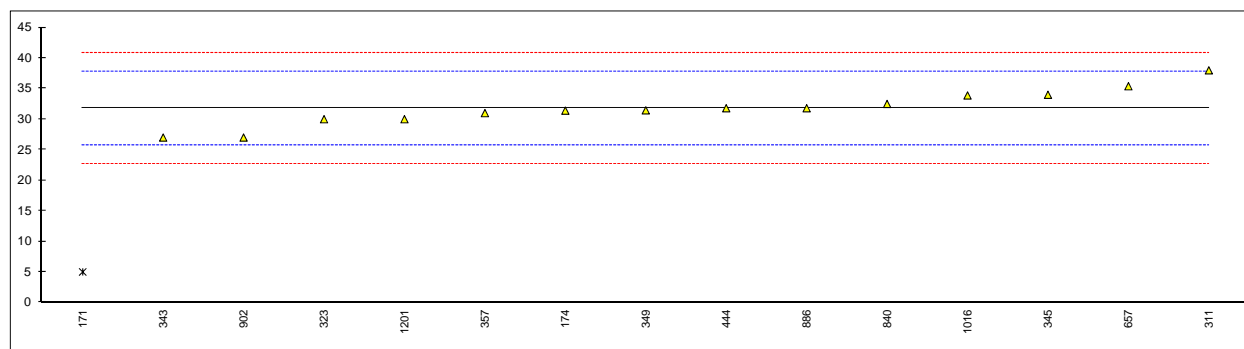## Determination of Diacetonalcohol on sample #13162, results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171		11	G(0.05)	-8.46	
174	D6438	50.9		1.20	
311	INH-394	51		1.22	
323	INH-020	33		-3.13	
343	in house	35		-2.65	
345	E4714	56.2		2.48	
349	INH-034	39.87		-1.47	
357	INH-054	50		0.98	
444	INH-001	52.2		1.51	
445		-----		-----	
551		-----		-----	
657	INH-009	42.3		-0.88	
840	INH-005	41.2		-1.15	
886	INH-119	59.2		3.21	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1016	DIN55687	46.48	C	0.13	First reported 0.045
1201		40		-1.44	
1429		-----		-----	
2493		46.0		0.01	

normality	OK
n	14
outliers	1
mean (n)	45.95
st.dev. (n)	7.757
R(calc.)	21.72
R(Horwitz)	11.57
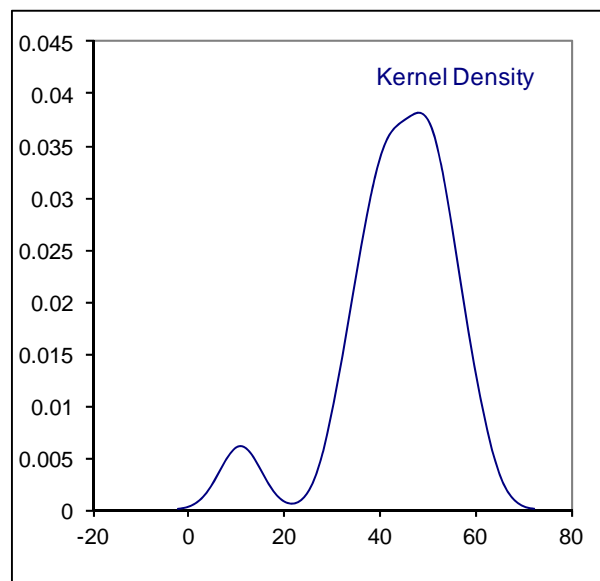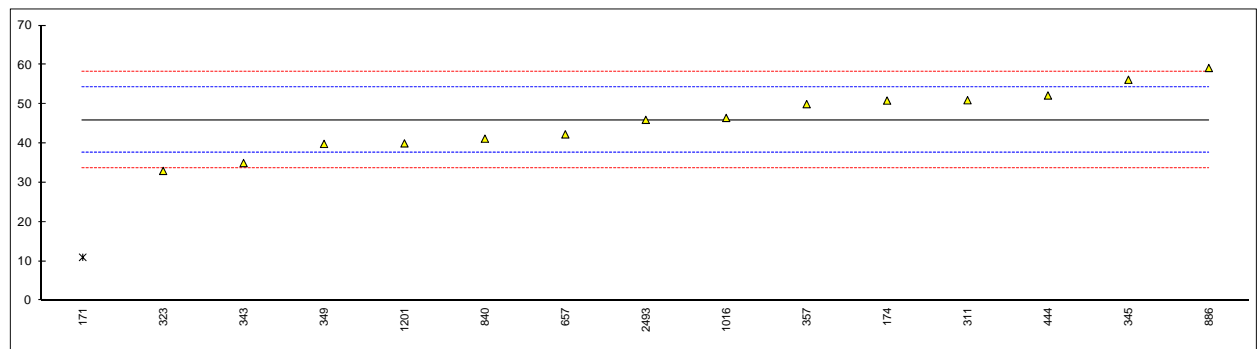
Determination of Mesityloxide on sample #13162, results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171		5	G(0.01)	-8.87	
174	D6438	31.4		-0.13	
311	INH-394	38		2.05	
323	INH-020	30		-0.60	
343	in house	27		-1.59	
345	E4714	34	C	0.73	First reported 42.8
349	INH-034	31.45		-0.12	
357	INH-054	31		-0.27	
444	INH-001	31.8		0.00	
445		-----		-----	
551		-----		-----	
657	INH-009	35.4		1.19	
840	INH-005	32.5		0.23	
886	INH-119	31.8		0.00	
902	INH-125	27		-1.59	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1016	DIN55687	33.89	C	0.69	First reported 0.034
1201		30		-0.60	
1429		-----		-----	
2493		-----		-----	

normality	OK
n	14
outliers	1
mean (n)	31.80
st.dev. (n)	2.969
R(calc.)	8.31
R(Horwitz)	8.46

Determination of Methanol on sample #13162, results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
171		116	G(0.01)	-9.45	
174	D6438	331		0.61	
311	INH-394	358		1.88	
323	INH-020	323		0.24	
343	in house	<350	C	-----	First reported 232
345	E4714	347.6		1.39	
349	INH-034	366.18		2.26	
357	INH-054	279		-1.82	
444	INH-001	289.4		-1.33	
445		-----		-----	
551		-----		-----	
657	INH-009	316		-0.09	
840	INH-005	293.8		-1.13	
886	INH-119	287.7		-1.41	
902		-----		-----	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1016	DIN55687	322.4	C	0.21	First reported 0.324
1201		310		-0.37	
1429		-----		-----	
2493		309	C	-0.42	First reported 241

|		
normality	OK	
n	13	
outliers	1	
mean (n)	317.93	
st.dev. (n)	27.390	
R(calc.)	76.69	
R(Horwitz)	59.84	
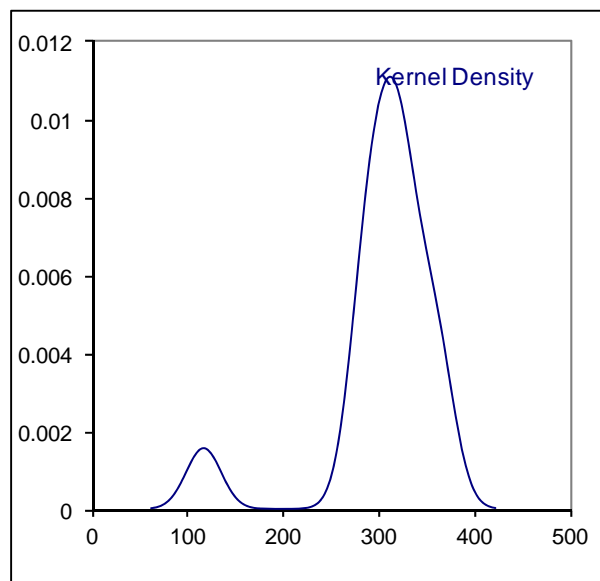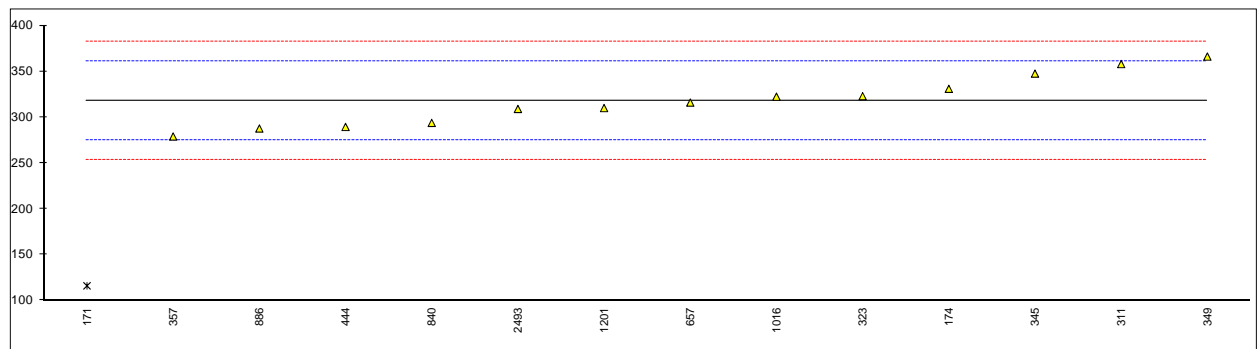
Determination of Permanganate Time Test @ 25°C on sample #13162; results in minutes

lab	method	value	mark	z(targ)	remarks
120	D1363	270	G(0.01)	-----	
171		-----		-----	
174	D1363	92		-----	
311	D1363	108		-----	
323	D1363Mod.	110		-----	
343	D1363	<150		-----	
345	E4714	155		-----	
349		-----		-----	
357	D1363	109		-----	
444	D1363	90	C	-----	First reported 30
445	D1363	105		-----	
551		-----		-----	
657	D1363	118		-----	
840	D1363	112		-----	
886	D1363	60-90		-----	
902	D1363	104		-----	
912	D1363	120		-----	
913	D1363	115		-----	
963		-----		-----	
1016	D1363	127		-----	
1201	D1363	88		-----	
1429	D1363	68.20		-----	
2493		-----		-----	

normality	OK
n	15
outliers	1
mean (n)	108
st.dev. (n)	19.8
R(calc.)	55
R(D1363:11)	(27)

Kernel Density
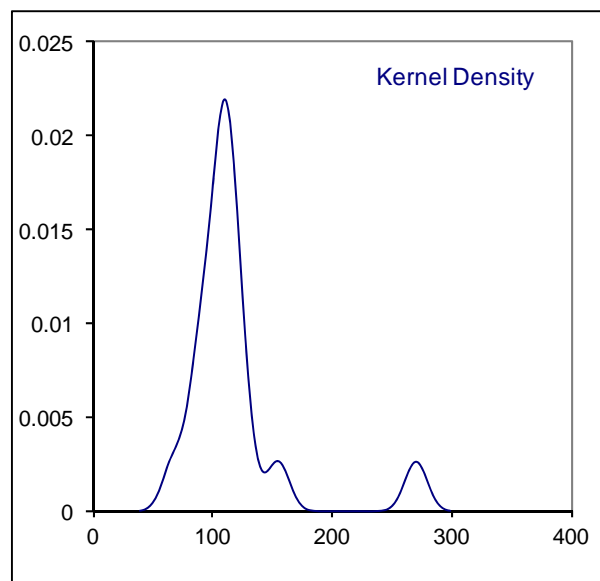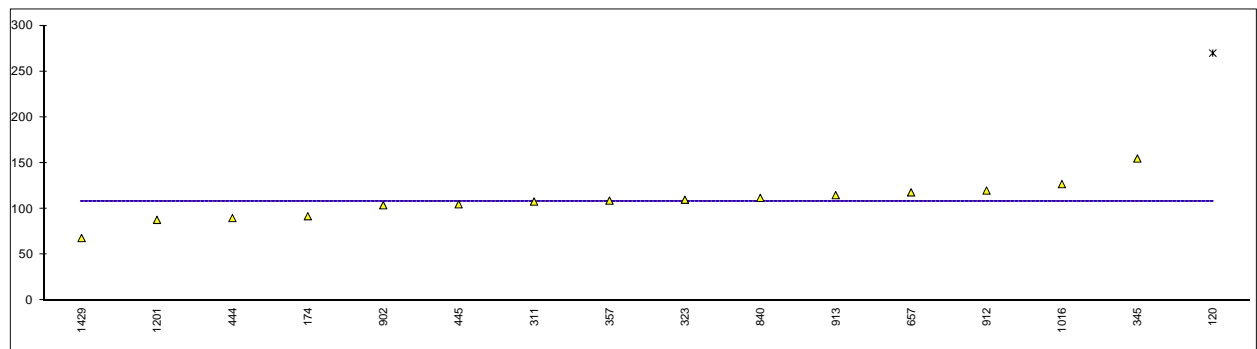
Determination of Refractive Index @ 20°C on sample #13162;

lab	method	value	mark	z(targ)	remarks
120	D1218	1.3587		-0.09	
171	D1218	1.3588		0.47	
174	D1218	1.3586		-0.65	
311	D1218	1.3587		-0.09	
323	D1218	1.35860		-0.65	
343		-----		-----	
345		-----		-----	
349		-----		-----	
357	D1218	1.3587		-0.09	
444		-----		-----	
445		-----		-----	
551		-----		-----	
657	D1218	1.3588		0.47	
840	D1218	1.3590		1.59	
886		-----		-----	
902	D1218	1.35884		0.69	
912		-----		-----	
913		-----		-----	
963		-----		-----	
1016	D1218	1.3586		-0.65	
1201	D1218	1.3599	G(0.01)	6.63	
1429	D1218	1.35854		-0.99	
2493		-----		-----	

normality	OK	
n	11	
outliers	1	
mean (n)	1.35872	
st.dev. (n)	0.000135	
R(calc.)	0.00038	
R(D1218:12)	0.00050	

Kernel Density
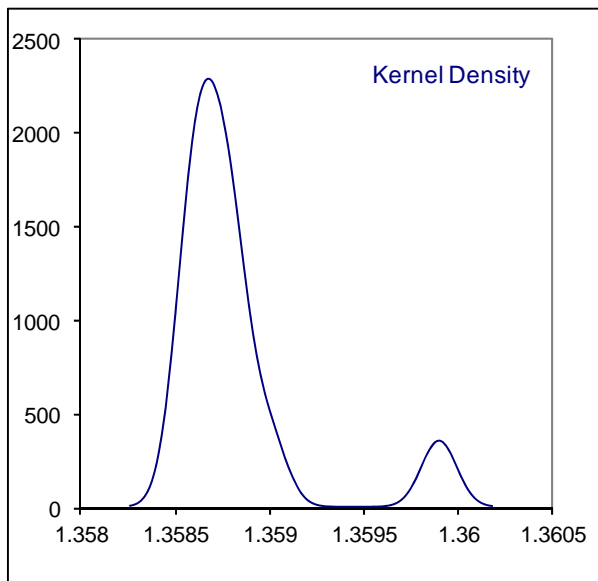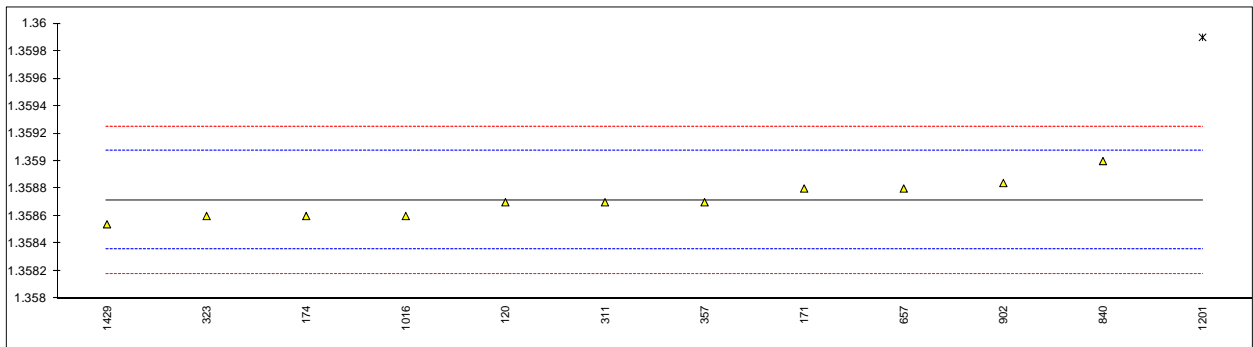
Determination of Water on sample #13162; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	D1364	2527	G(0.01)	5.60	
171	D1364	1896		-0.95	
174	D1364	2037		0.52	
311	D1364	1970		-0.18	
323	D1364	2052		0.67	
343	E203	1982		-0.05	
345	D1364	2072		0.88	
349	D1364	1894		-0.97	
357	D1364	1985		-0.02	
444	E203	1992		0.05	
445	D1364	2210		2.31	
551		-----		-----	
657		-----		-----	
840	D1364	1942		-0.47	
886	D1364	2047		0.62	
902	D1364	2151		1.70	
912	D1364	1853		-1.39	
913	D1364	1870		-1.22	
963		-----		-----	
1016	D1364	1836.5	C	-1.56	First reported 0.18365
1201	D1364	2000		0.13	
1429		-----		-----	
2493	D1364	1980		-0.07	

normality	OK
n	18
outliers	1
mean (n)	1987.2
st.dev. (n)	99.41
R(calc.)	278.3
R(D1364:12)	270.0
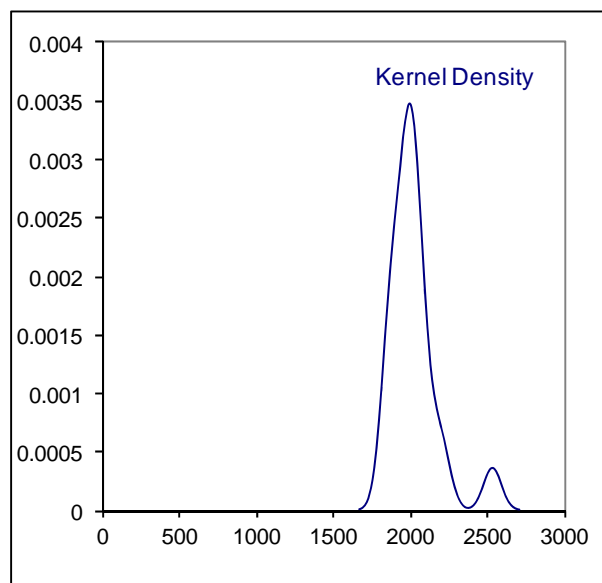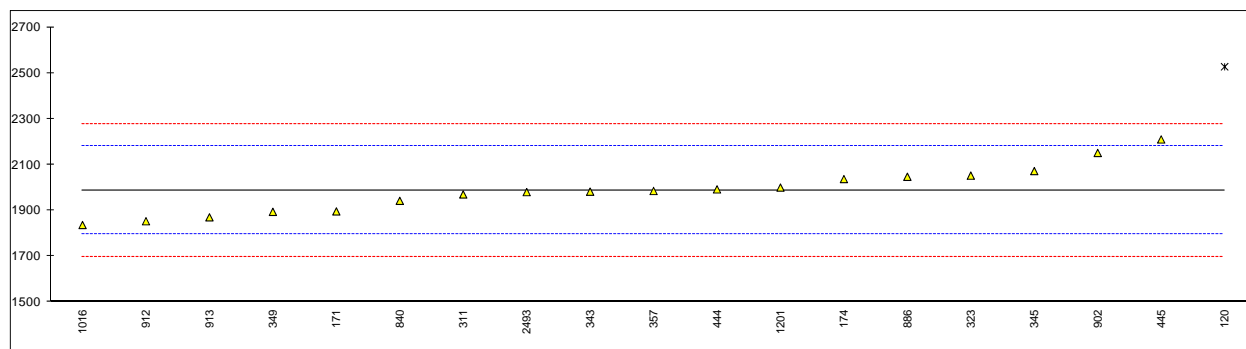
APPENDIX 2

Number of participants per country

   1 lab in BELGIUM
   1 lab in BRAZIL
   1 lab in FINLAND
   1 lab in HUNGARY
  2 labs in INDIA
  3 labs in NETHERLANDS, THE
   1 lab in SAUDI ARABIA
   1 lab in SINGAPORE
  3 labs in SPAIN
   1 lab in TAIWAN R.O.C.
   1 lab in TURKEY
  3 labs in U.S.A.
  3 labs in UNITED KINGDOM
   1 lab in VIETNAM

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   = reported in wrong unit
ex   = excluded from calculations
n.a.   = not applicable
n.d.   = not detected

Literature:

1   i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
2   ASTM E178-02
3   ASTM E1301-03
4   ISO 5725-86
5   ISO 5725, parts 1-6, 1994
6   M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
7   W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
8   IP 367/84
9   DIN 38402 T41/42
10   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
11   J.N. Miller, Analyst, 118, 455, (1993)
12   IMPCA Methanol Reference Specifications, IMPCA, Brussels, December 2010.
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/).