

Results of Proficiency Test

Mono Ethylene Glycol (MEG)

October 2014

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 1994, the Institute for Interlaboratory Studies organised a proficiency test for the analysis of Mono Ethylene Glycol every year. As part of the annual proficiency test program of 2014/2015, the Institute decided to continue this proficiency test on Mono Ethylene Glycol. In this interlaboratory study 58 laboratories in 26 different countries have participated. See appendix 2 for the number of participants per country. In this report the results of the 2014 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. Sample analyses for fit-for-use and homogeneity testing were subcontracted. To get maximum information from this study it was decided to send 2 different samples:

	Bottle type	Tests requested
Sample #14200	1.0 L amber glass bottle	for all regular determinations on MEG
Sample #14201	0.1 L amber glass bottle	for UV transmittance and Water only

table 1: type of samples

The participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. This PT falls under the accredited scope. 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 was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website <http://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, approximately 110 litre of MEG polyester grade was obtained from a local production plant. The bulk material was transferred to a precleaned 200 litre drum. From this batch, after homogenization, 90 amber glass bottles of 1.0 L were filled and labelled #14200.

The remainder of the batch was used for the UV sample, 97 amber glass bottles of 0.1 litre were filled. The bottles were closed with special screw caps with Teflon inner layer, and labelled #14201.

The homogeneity of the subsamples #14200 was checked by determination of Density in accordance with ASTM D4052, on 8 stratified randomly selected samples. The homogeneity of the sample #14201 was checked by determination UV Transmittance without nitrogen sparging at 220 nm in accordance with ASTM E2193 on 7 stratified randomly selected samples.

	Density @ 20°C in kg/l
Sample #14200-1	1.11333
Sample #14200-2	1.11333
Sample #14200-3	1.11333
Sample #14200-4	1.11333
Sample #14200-5	1.11333
Sample #14200-6	1.11333
Sample #14200-7	1.11333
Sample #14200-8	1.11333

table 2: homogeneity test results of subsamples #14200

	UV(220nm) in T%
Sample #14201-1	92.9
Sample #14201-2	91.9
Sample #14201-3	92.3
Sample #14201-4	91.9
Sample #14201-5	90.9
Sample #14201-6	93.7
Sample #14201-7	93.6

table 3: homogeneity test results of subsamples #14201

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

	Density @ 20°C in kg/l	UV(220nm) in T%
r (observed)	0.00000	2.98
0.3xR _(ASTMD4052:02e1)	0.00015	--
0.3xR _(ASTM E2193:08-A)	--	2.90

table 4: homogeneity evaluation of subsamples #14200 and #14201

Each calculated repeatability was equal or less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the samples was assumed.

To each of the participating laboratories 2 bottles (1*1 L bottle, labelled #14200 and 1*100 mL bottle, labelled #14201), were sent on October 8, 2014.

2.5 STABILITY OF THE SAMPLES

The stability of the Mono Ethylene Glycol, packed in amber glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine Acidity as Acetic Acid (E2679 and D1613), Aldehydes as Acetaldehyde, Appearance, Ash, Chloride as Cl, Colour (D5386), Colour Pt/Co (D1209) Density at 20°C, Diethylene Glycol, Distillation (Initial Boiling Point, 50% recovered and Dry Point), Iron, Purity and Specific Gravity @ 20/20°C on sample #14200. On sample #14201 was requested to determine UV Transmittance (at 350, 275, 250 and 220 nm) and Water.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The detailed report form was also 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.

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 (raw data of the) reported results.

Additional or corrected results have been 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, April 2014 version 3.3). 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 a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care. In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and Rosner 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 and by R(0.01) for the Rosner General ESD test (see appendix 3, no.16). Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner's 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 them 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 each 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; nos.14 and 15). Also a normal Gauss curve was projected over the Kernel Density Graph.

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 whether the reported test result is fit-for-use.
The z-scores were calculated in accordance with:

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

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

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare.
Therefore the usual interpretation of z-scores maybe as follows:

- $|z| < 1$ good
- $1 < |z| < 2$ satisfactory
- $2 < |z| < 3$ questionable
- $3 < |z|$ unsatisfactory

4 EVALUATION

In this interlaboratory study, problems with sample despatch were encountered due to several reasons. Six participants reported after the deadline and 6 participants did not report any result at all. Not all participants were able to report all requested parameters. Finally, 52 participants did report 766 numerical results. Observed were 31 outlying results, which is 4.0% 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 sample and per test. The specified test methods and requirements were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the reported data. The abbreviations, used in these tables, are listed in appendix 3.

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.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care.

Acidity: The determination according ASTM E2679 may be very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the strict precision data of ASTM E2679:09.

The determination according ASTM D1613 was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1613:06(2012).

Aldehydes: This determination is not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM E2313:08.

Appearance: No analytical problems were observed. All participants, except one, agreed about the appearance of sample #14200, which was bright, clear and free of suspended matter (= pass).

Ash: The consensus value is below the application range (0.001 – 0.180 %M/M) of ASTM D482:13. Therefore no significant conclusions were drawn.

Chloride: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM E2469:08a.

Colour Pt/Co: The determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D1209:05(2012).

Colour D5386: The determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility is in good agreement with the requirements of ASTM D5386:10.

Density: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4052:02e1. D4052:11 is applicable only for the density range 0.71 g/ml to 0.88 g/ml, being valid for gasoline's, distillates, base stocks and lubricating oils. Therefore this 2011 version is may be not applicable for MEG.

- DEG: This determination was problematic. Four statistical outliers were observed and one false negative test result was observed. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM E2409:13.
- Distillation: This determination was not problematic. In total three statistical outliers (from one lab) were observed. All three calculated reproducibilities after rejection of the statistical outliers are in good agreement with the requirements of ASTM D1078:11.
- Iron: This determination was problematic at this low Iron concentration level. One statistical outlier and two test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM E1615:08.
- Purity: Regretfully, no reproducibility data for purity are mentioned in ASTM E2409:13. Therefore no significant conclusions were drawn. A bimodal distribution is observed probably caused by reporting the result as "as received" or "on dry basis".
- Specific Gravity: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of E202:12.
- Water: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM E1064:12.
- UV: The reported test results were split up into method A (sparged with nitrogen) and method B (not sparged with nitrogen). Both sets of test results were evaluated separately.
For method A, this determination was not problematic. In total three statistical outliers were observed. The calculated reproducibilities of UV at 350nm, 250nm and 220 nm after rejection of the statistical outliers, are in agreement with the requirements of ASTM E2193:08. However, the calculated reproducibility of UV at 275nm is not in agreement with the requirements of ASTM E2193:08_method A.
For method B, this determination was not problematic. In total six statistical outliers were observed. The calculated reproducibilities of UV at 350nm, 275nm, 250nm and 220nm, after rejection of the statistical outliers, are in agreement with the requirements of ASTM E2193:08_method B.

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 table:

Parameter	unit	n	average	$2.8 * s_{dR}$	R (lit.)
Acidity as Acetic Acid (E2679)	mg/kg	9	1.39	2.32	0.70
Acidity as Acetic Acid (D1613)	mg/kg	45	6.31	5.59	14.0
Aldehydes as Acetaldehyde	mg/kg	33	31.02	15.80	26.68
Appearance		44	Pass	n.a.	n.a.
Ash	%M/M	24	0.0004	0.0008	(0.0050)
Chloride as Cl	mg/kg	29	0.04	0.08	0.03
Colour Pt/Co ASTM D1209	---	33	2.0	3.1	7.0
Colour ASTM D5386	---	29	1.5	1.8	4.8
Density at 20°C	kg/L	48	1.1133	0.0003	0.0005
Diethylene Glycol	mg/kg	39	85.0	29.0	21.6
Initial Boiling Point	°C	39	197.1	0.7	3.1
50% recovered	°C	39	197.6	0.7	1.4
Dry Point	°C	39	198.1	0.8	2.1
Iron as Fe	mg/kg	32	0.010	0.017	0.011
Purity	%M/M	42	99.958	0.042	n.a.
Specific Gravity 20/20°C	---	46	1.1153	0.0003	0.0005
Water	mg/kg	43	183.4	115.0	31.4
UV Transmittance at 350 nm (N ₂)	%T	15	100.10	0.60	0.94
UV Transmittance at 275 nm (N ₂)	%T	16	99.17	1.31	1.10
UV Transmittance at 250 nm (N ₂)	%T	16	97.76	2.18	2.06
UV Transmittance at 220 nm (N ₂)	%T	14	91.81	5.29	9.68
UV Transmittance at 350 nm	%T	25	100.06	0.50	1.15
UV Transmittance at 275 nm	%T	27	99.25	0.99	2.11
UV Transmittance at 250 nm	%T	25	95.76	1.11	1.10
UV Transmittance at 220 nm	%T	27	78.30	2.70	4.05

table 5: reproducibilities of samples #14200 and #14201

Results between brackets were below the application range of the method, therefore results should be evaluated with care

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 tests that are problematic have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2014 WITH PREVIOUS PTS

	October 2014	October 2013	October 2012	October 2011
Number of reporting labs	52	54	54	63
Number of results reported	766	785	838	927
Statistical outliers	31	40	48	42
Percentage outliers	4.0%	5.1%	5.7%	4.5%

table 6: comparison of statistical summary parameters 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 in the following table:

	October 2014	October 2013	October 2012	October 2011
Acidity as Acetic Acid (E2679)	--	--	++	-
Acidity as Acetic Acid (D1613)	++	n.a.	n.a.	n.a.
Aldehydes as Acetaldehyde	+	++	(++)	++
Ash	(++)	(+/-)	(++)	(++)
Chloride as Cl	--	+/-	--	+/-
Colour Pt/Co ASTM D1209	++	++	++	-
Colour ASTM D5368	++	++	+	--
Density at 20°C	+	++	+	++
Diethylene Glycol	-	--	--	+/-
Initial Boiling Point	++	++	--	++
50% recovered	++	++	++	++
Dry Point	++	++	++	++
Iron as Fe	-	+/-	++	++
Purity	n.a.	--	--	++
Specific Gravity 20/20°C	+	++	+	++
Water	--	--	+/-	++
UV Transmittance at 350 nm	+	++	++	--
UV Transmittance at 275 nm	-	++	++	--
UV Transmittance at 250 nm	-	+/-	-	--
UV Transmittance at 220 nm	++	+	-	-

table 7: comparison determinations against the standard

Results between brackets were below the application range of the method, therefore results should be evaluated with care

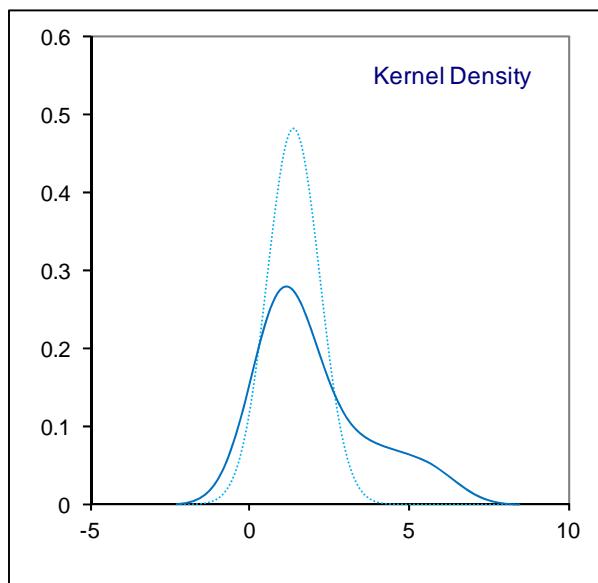
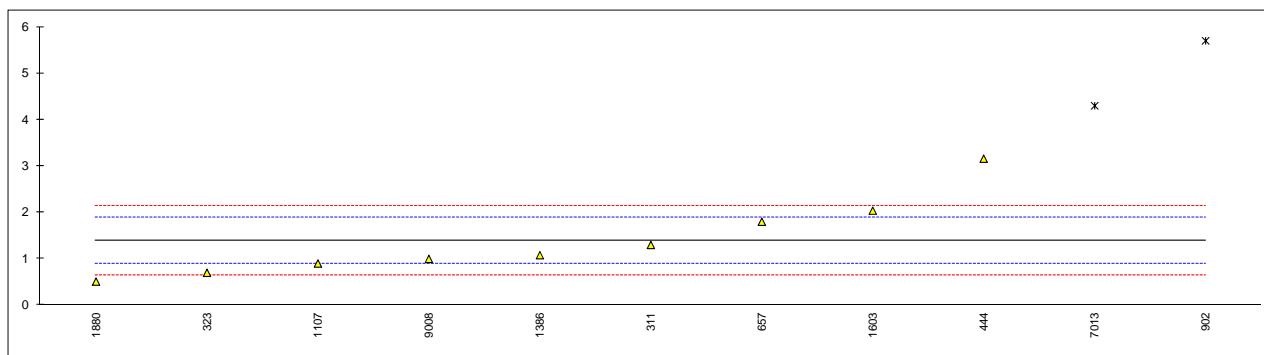
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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

APPENDIX 1

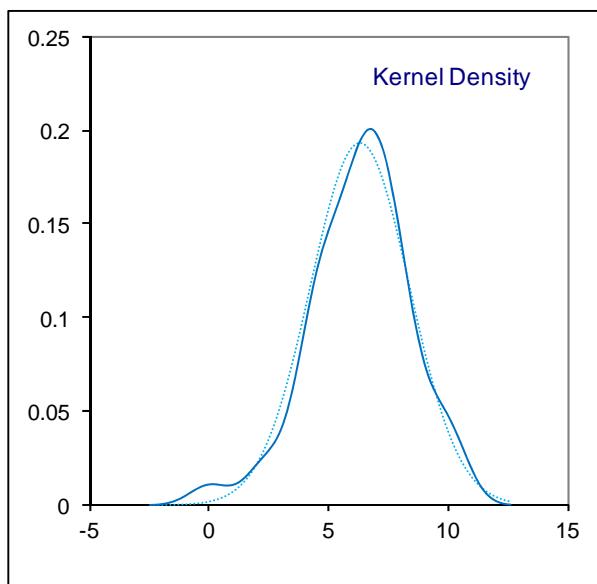
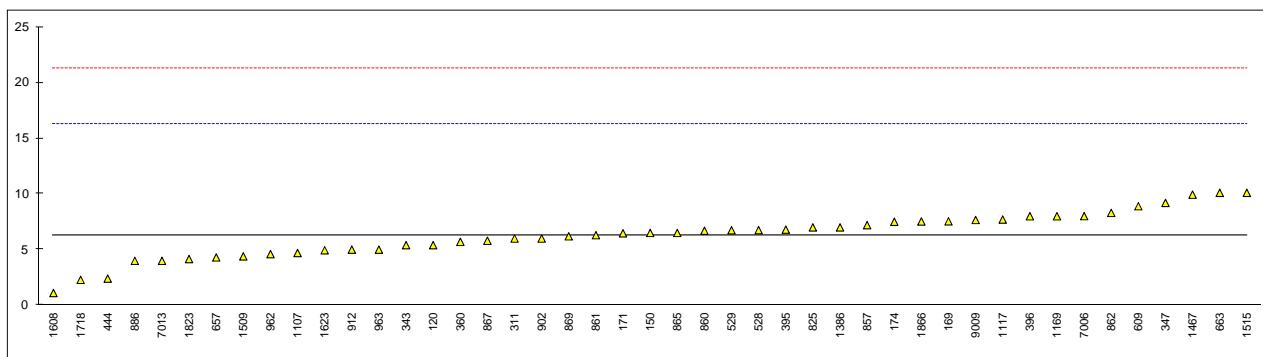
Determination of Acidity as Acetic Acid (E2679) on sample #14200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		----		----	
150		----		----	
169		----		----	
171		----		----	
174		----		----	
311	E2679	1.3		-0.35	
323	E2679	0.7		-2.74	
343		----		----	
347		----		----	
360		----		----	
370		----		----	
395		----		----	
396		----		----	
444	E2679	3.16		7.07	
528		----		----	
529		----		----	
551		----		----	
557		----		----	
558		----		----	
609		----		----	
657	E2679	1.8		1.64	
663		----		----	
825		----		----	
857		----		----	
860		----		----	
861		----		----	
862		----		----	
865		----		----	
867		----		----	
869		----		----	
886		----		----	
902	E2679	5.7	DG(0.05)	17.19	
912		----		----	
913		----		----	
962		----		----	
963		----		----	
1101		----		----	
1107	E202	0.9		-1.94	
1117		----		----	
1151		----		----	
1169		----		----	
1217		----		----	
1386	E2679	1.08		-1.23	
1467		----		----	
1509		----		----	
1515		----		----	
1603	in house	2.04		2.60	
1608		----		----	
1623		----		----	
1718		----		----	
1823		----		----	
1866		----		----	
1880	E2679	0.51		-3.50	
1960		----		----	
7006		----		----	
7013	E2679	4.3	DG(0.05)	11.61	
9008	E2679	1		-1.55	
9009		----		----	
					<u>Only E2679:09 data:</u>
	normality	not OK			not OK
	n	9			7
	outliers	2			2
	mean (n)	1.388			1.364
	st.dev. (n)	0.8269			0.8947
	R(calc.)	2.315			2.505
	R(E2679:09)	0.702			0.690



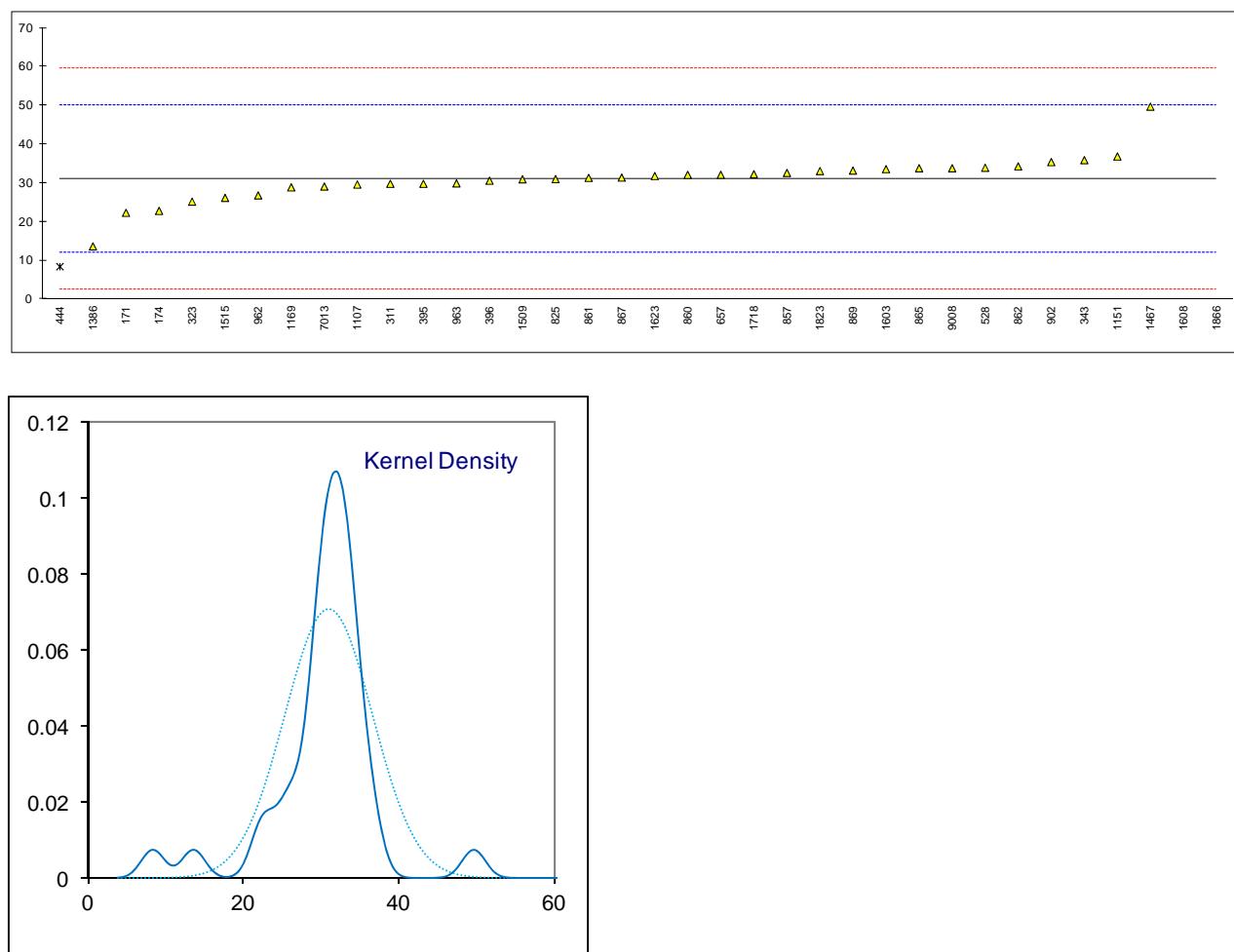
Determination of Acidity as Acetic Acid (D1613) on sample #14200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	D1613	5.4		-0.18	
150	D1613	6.5		0.04	
169	D1613	7.5431		0.25	
171	D1613	6.467		0.03	
174	D1613	7.5		0.24	
311	D1613	6		-0.06	
323		----		----	
343	D1613	5.4		-0.18	
347	D1613	9.2		0.58	
360	D1613	5.7		-0.12	
370		----		----	
395	D1613	6.8		0.10	
396	D1613	8		0.34	
444	D1613	2.40		-0.78	
528	D1613	6.757		0.09	
529	D1613	6.7375		0.09	
551		----		----	
557		----		----	
558		----		----	
609	D1613	8.9		0.52	
657	D1613	4.3		-0.40	
663	D1613	10.1		0.76	
825	D1613	7		0.14	
857	D1613	7.2		0.18	
860	D1613	6.7		0.08	
861	D1613	6.3		0.00	
862	D1613	8.3		0.40	
865	D1613	6.5		0.04	
867	D1613	5.8		-0.10	
869	D1613	6.2		-0.02	
886	D1613	4		-0.46	
902	D1613	6		-0.06	
912	D1613	5		-0.26	
913		----		----	
962	D1613	4.6		-0.34	
963	D1613	5.0		-0.26	
1101		----		----	
1107	D1613	4.7		-0.32	
1117	D1613	7.7		0.28	
1151		----		----	
1169	D1613	8		0.34	
1217		----		----	
1386	D1613	7.0		0.14	
1467	D1613	9.94		0.73	
1509	D1613	4.4		-0.38	
1515	D1613	10.1		0.76	
1603		----		----	
1608	D1613	1.104	C	-1.04	Probably unit error, reported 0.0001104 mg/kg
1623	D1613	4.94		-0.27	
1718	D1613	2.29		-0.80	
1823	D1613	4.15		-0.43	
1866	D1613	7.53		0.24	
1880		----		----	
1960		----		----	
7006	D1613	8.01		0.34	
7013	D1613	4.0		-0.46	
9008		----		----	
9009	D1613	7.666		0.27	
	normality	OK			
	n	45			
	outliers	0			
	mean (n)	6.31			
	st.dev. (n)	1.996			
	R(calc.)	5.59			
	R(D1613:06)	14.00			



Determination of Aldehydes as Acetaldehyde on sample #14200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		----		----	
150		----		----	
169		----		----	
171	E2313	22.31038		-0.91	
174	E2313	22.8		-0.86	
311	E2313	29.8		-0.13	
323	E2313	25.2		-0.61	
343	E2313	35.9		0.51	
347		----		----	
360		----		----	
370		----		----	
395	E2313	29.8		-0.13	
396	E2313	30.6		-0.04	
444	E2313	8.4	C,R(0.05)	-2.37	First reported:0
528	E2313	33.923		0.30	
529		----		----	
551		----		----	
557		----		----	
558		----		----	
609		----		----	
657	E2313	32.13		0.12	
663		----		----	
825	E2313	31		0.00	
857	E2313	32.6		0.17	
860	E2313	32.1		0.11	
861	E2313	31.33		0.03	
862	E2313	34.3		0.34	
865	E2313	33.8		0.29	
867	E2313	31.4		0.04	
869	E2313	33.2		0.23	
886		----		----	
902	E2313	35.4		0.46	
912		----		----	
913		----		----	
962	E2313	26.8		-0.44	
963	E2313	29.9		-0.12	
1101		----		----	
1107	E2313	29.6		-0.15	
1117		----		----	
1151	E2313	36.845		0.61	
1169	E2313	28.88		-0.22	
1217		----		----	
1386	E2313	13.63		-1.82	
1467	E2313	49.7		1.96	
1509	E2313	30.95		-0.01	
1515	E2313	26.155		-0.51	
1603	in house	33.58		0.27	
1608	E2313	152.7	R(0.01)	12.77	
1623	INH-012	31.81		0.08	
1718	E2313	32.27		0.13	
1823	E2313	33.087		0.22	
1866	E2313	156	R(0.01)	13.11	
1880		----		----	
1960		----		----	
7006		----		----	
7013	E2313	29.1	C	-0.20	First reported: 19.834
9008	E2313	33.8		0.29	
9009		----		----	
	normality	not OK			
	n	33			
	outliers	3			
	mean (n)	31.021			
	st.dev. (n)	5.6438			
	R(calc.)	15.803			
	R(E2313:08)	26.684			



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Determination of Appearance on sample #14200;

lab	method	value	mark	z(targ)	remarks
120	E2680	Pass	-----		
150	E2680	Pass	-----		
169	E2680	Pass	-----		
171	E2680	C&F	-----		
174	E2680	Pass	-----		
311	E2680	Pass	-----		
323	E2680	CFFMIS	-----		
343		Pass	-----		
347	E2680	Pass	-----		
360	E2680	C&B	-----		
370	E2680	Pass	-----		
395	E2680	Pass	-----		
396	E2680	Pass	-----		
444		Pass	-----		
528	E2680	Pass	-----		
529	E2680	Pass	-----		
551		-----	-----		
557		-----	-----		
558		-----	-----		
609	E2680	Pass	-----		
657	E2680	Pass	-----		
663	Visual	Pass	-----		
825		Pass	-----		
857	E2680	Pass	-----		
860	E2680	Pass	-----		
861	Visual	C&B	-----		
862	E2680	Pass	-----		
865	Visual	Pass	-----		
867	Visual	C&B	-----		
869	Visual	C&B	-----		
886		-----	-----		
902	E2680	Pass	-----		
912	E2680	Pass	-----		
913		-----	-----		
962		Pass	-----		
963	E2680	Pass	-----		
1101		-----	-----		
1107	Visual	C&B	-----		
1117	D4176	Pass	-----		
1151		-----	-----		
1169		Pass	-----		
1217		-----	-----		
1386	E2680	Pass	-----		
1467		Pass	-----		
1509	E2680	Pass	-----		
1515	D4176	Pass	-----		
1603	in house	CFP	-----		
1608		Pass	-----		
1623	D2090	Clear	-----		
1718	D4176	CFFSM	-----		
1823	D4176	Fail	-----	Two brown spots in it	
1866		Clear	-----		
1880	E2680	Pass	-----		
1960		-----	-----		
7006		-----	-----		
7013		-----	-----		
9008		-----	-----		
9009		-----	-----		
normality		n.a.			
n		44			
outliers		1 (fail)			
mean (n)		Pass			

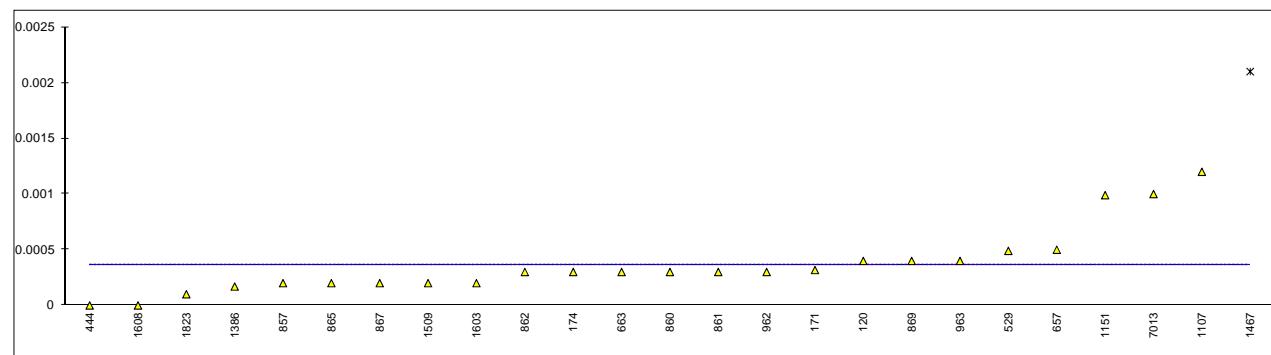
B&C = Bright and clear

(C)FFSM = Clear and free from suspended matter

C&F = Clear and Free

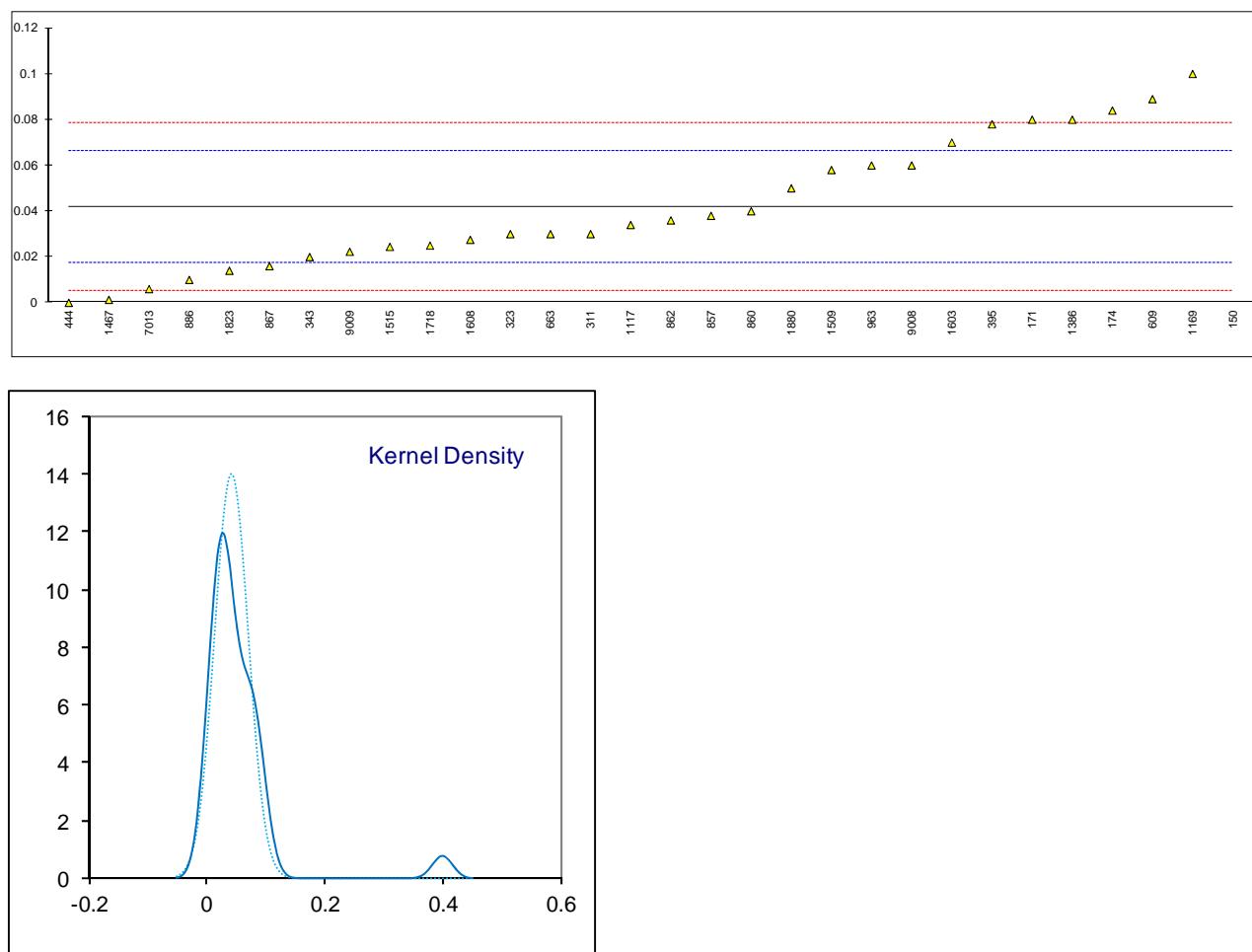
Determination of Ash on sample #14200; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D482	0.0004		----	
150	D482	<0.001		----	
169	D482	<0.0001		----	
171	D482	0.000317903		----	
174	D482	0.0003		----	
311	D482	<0.0001		----	
323	D482	<0.01		----	
343	D482	<0.001		----	
347	D482	<0.001		----	
360	D482	<0.001		----	
370		----		----	
395		----		----	
396		----		----	
444	D482	0.0		----	
528		----		----	
529	D482	0.00049		----	
551		----		----	
557		----		----	
558		----		----	
609		----		----	
657	D482	0.0005		----	
663	D482	0.0003		----	
825		----		----	
857	D482	0.0002		----	
860	D482	0.0003		----	
861	D482	0.0003		----	
862	D482	0.0003		----	
865	D482	0.0002		----	
867	D482	0.0002		----	
869	D482	0.0004		----	
886	D482	<0.001		----	
902	D482	<0.001		----	
912	D482	<0.001		----	
913		----		----	
962	D482	0.0003		----	
963	D482	0.0004		----	
1101		----		----	
1107	D482	0.0012		----	
1117	D482	<0.001		----	
1151	D482	0.00099		----	
1169		----		----	
1217		----		----	
1386	D482	0.00017		----	
1467	D482	0.0021	R(0.01)	----	False positive test result?
1509	D482	0.0002		----	
1515		----		----	
1603	in house	0.0002	C	----	First reported:0.00469
1608	D482	0.000		----	
1623	D482	<0.001		----	
1718	D482	<0.001		----	
1823	D482	0.0001		----	
1866		----		----	
1880		----		----	
1960		----		----	
7006		----		----	
7013	D482	0.0010		----	
9008		----		----	
9009		----		----	
normality		not OK			
n		24			
outliers		1			
mean (n)		0.00037			
st.dev. (n)		0.000300			
R(calc.)		0.00084			
R(D482:13)		(0.00500)			Application range: 0.001 – 0.180 %M/M



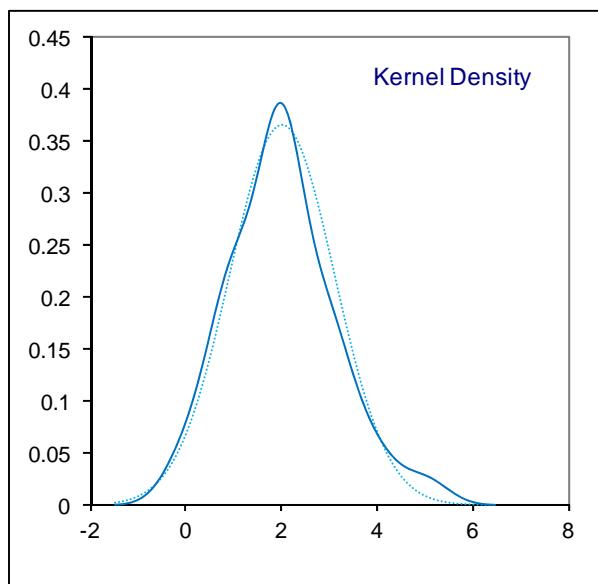
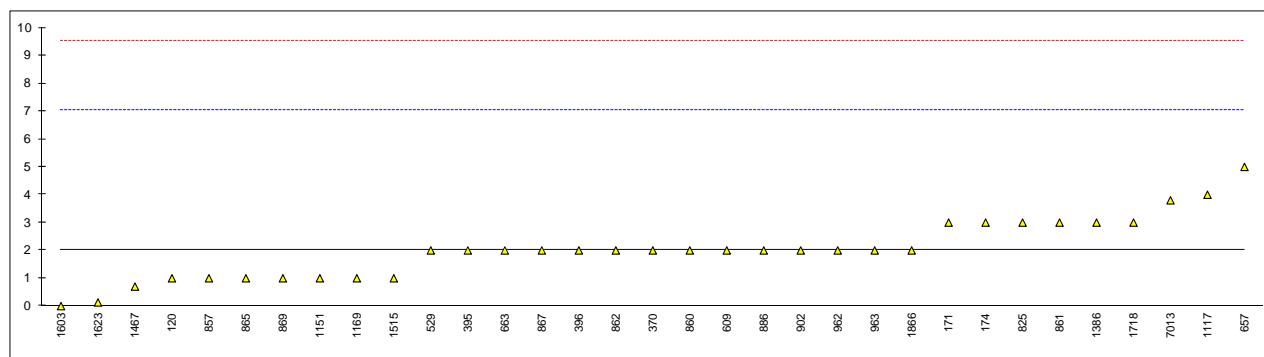
Determination of Chloride as Cl on sample #14200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	INH-0221	<0.5	----	----	
150	D7359	0.4	C,R(0.01)	29.28	First reported:0.2
169		----		----	
171	E2469	0.08		3.12	
174	E2469	0.084		3.45	
311	E2469	0.03		-0.97	
323	E2469	0.03		-0.97	
343	E2469	0.02		-1.79	
347		----		----	
360		----		----	
370		----		----	
395	E2469	0.078		2.96	
396		----		----	
444	E2469	0.0		-3.42	
528		----		----	
529		----		----	
551		----		----	
557		----		----	
558		----		----	
609	E2469	0.089		3.85	
657		----		----	
663	INH-101876	0.03		-0.97	
825		----		----	
857	E2469	0.038		-0.31	
860	IMPCA002	0.04		-0.15	
861		----		----	
862	E2469	0.036		-0.48	
865	INH-001	<0.5		----	
867	E2469	0.016		-2.11	
869		----		----	
886	in house	0.01		-2.60	
902	E2469	<0.05		----	
912		----		----	
913		----		----	
962		----		----	
963	E2469	0.06		1.48	
1101		----		----	
1107	in house	<0.2		----	
1117	E2469	0.034		-0.64	
1151		----		----	
1169	E2469	0.1		4.75	
1217		----		----	
1386	E2469	0.08		3.12	
1467	in house	0.0013		-3.31	
1509	E2469	0.058		1.32	
1515	E2469	0.02445		-1.42	
1603	in house	0.07		2.30	
1608	E2469	0.0275		-1.17	
1623		----		----	
1718	E2469	0.025		-1.38	
1823	INH-2901	0.014		-2.28	
1866		----		----	
1880	E2469	0.050		0.67	
1960		----		----	
7006		----		----	
7013	E2469	0.006		-2.93	
9008	E2469	0.06		1.48	
9009	E2469	0.0223		-1.60	
					<u>Only E2469 data:</u>
	normality	OK		OK	
	n	29		23	
	outliers	1		0	
	mean (n)	0.0418		0.0456	
	st.dev. (n)	0.02848		0.02862	
	R(calc.)	0.0797		0.0801	
	R(E2469:08a)	0.0342		0.0373	



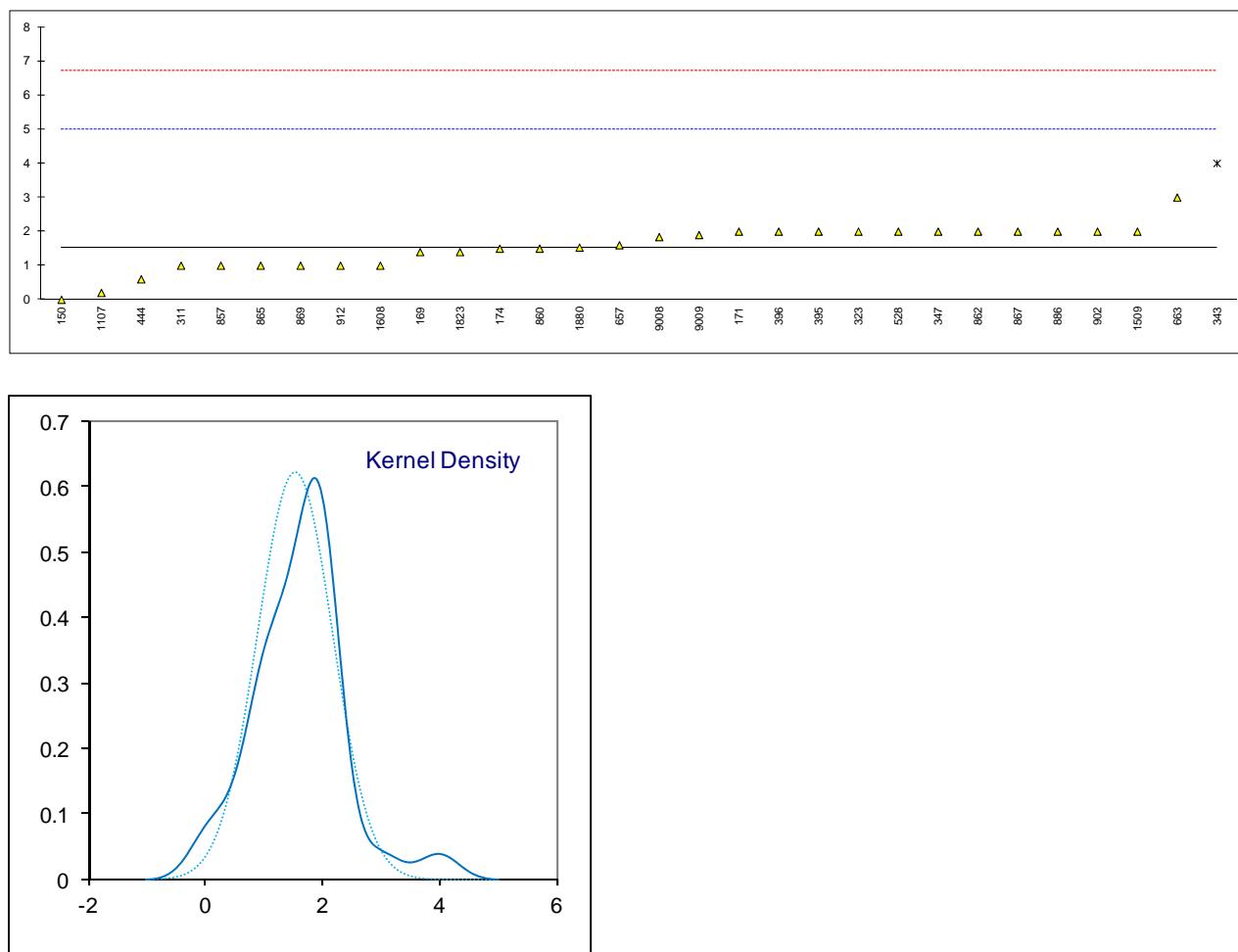
Determination of Colour Pt/Co (D1209) on sample #14200;

lab	method	value	mark	z(targ)	remarks
120	D1209	1		-0.41	
150		----		----	
169		----		----	
171	D1209	3		0.39	
174	D1209	3		0.39	
311	D1209	<5		----	
323	D1209	<5		----	
343		----		----	
347		----		----	
360		----		----	
370	D1209	2		-0.01	
395	D1209	2		-0.01	
396	D1209	2		-0.01	
444		----		----	
528		----		----	
529	D1209	2		-0.01	
551		----		----	
557		----		----	
558		----		----	
609	D1209	2		-0.01	
657	D1209	5		1.19	
663	D1209	2		-0.01	
825	D1209	3		0.39	
857	D1209	1		-0.41	
860	D1209	2		-0.01	
861	D1209	3		0.39	
862	D1209	2		-0.01	
865	D1209	1		-0.41	
867	D1209	2		-0.01	
869	D1209	1		-0.41	
886	D1209	2		-0.01	
902	D1209	2		-0.01	
912		----		----	
913		----		----	
962	D1209	2		-0.01	
963	D1209	2		-0.01	
1101		----		----	
1107	D1209	<5		----	
1117	D1209	4		0.79	
1151	D1209	1		-0.41	
1169	D1209	1		-0.41	
1217		----		----	
1386	D1209	3		0.39	
1467	D1209	0.7		-0.53	
1509	D1209	<5		----	
1515	D1209	1		-0.41	
1603	in house	0		-0.81	
1608		----		----	
1623	D1209	0.13		-0.76	
1718	D1209	3		0.39	
1823		----		----	
1866	D1209	2		-0.01	
1880		----		----	
1960		----		----	
7006		----		----	
7013	D1209	3.8		0.71	
9008		----		----	
9009		----		----	
normality					
n					
outliers					
mean (n)					
st.dev. (n)					
R(calc.)					
R(D1209:05)					



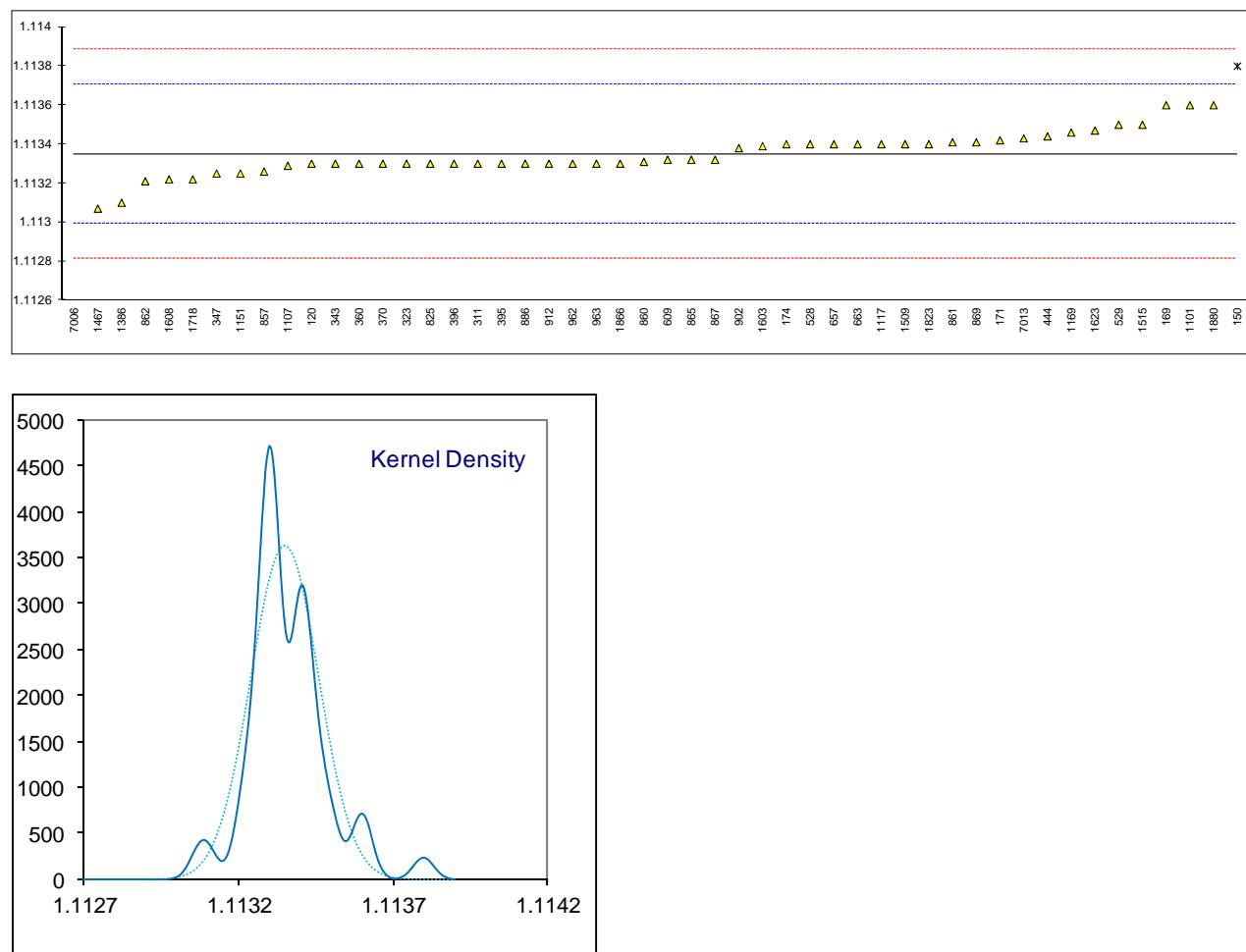
Determination of Colour Pt/Co (D5386) on sample #14200;

lab	method	value	mark	z(targ)	remarks
120		----		----	
150	D5386	0		-0.89	
169	D5386	1.4		-0.08	
171	D5386	2		0.27	
174	D5386	1.5		-0.02	
311	D5386	1		-0.31	
323	D5386	2		0.27	
343	D5386	4	R(0.05)	1.43	
347	D5386	2		0.27	
360		----		----	
370		----		----	
395	D5386	2		0.27	
396	D5386	2		0.27	
444	D5386	0.6		-0.54	
528	D5386	2.0		0.27	
529		----		----	
551		----		----	
557		----		----	
558		----		----	
609		----		----	
657	D5386	1.6		0.04	
663	D5386	3		0.85	
825		----		----	
857	D5386	1		-0.31	
860	D5386	1.5		-0.02	
861		----		----	
862	D5386	2		0.27	
865	D5386	1		-0.31	
867	D5386	2		0.27	
869	D5386	1		-0.31	
886	D5386	2		0.27	
902	D5386	2		0.27	
912	D5386	1		-0.31	
913		----		----	
962		----		----	
963		----		----	
1101		----		----	
1107	D5386	0.2		-0.77	
1117		----		----	
1151		----		----	
1169		----		----	
1217		----		----	
1386		----		----	
1467		----		----	
1509	D5386	2		0.27	
1515		----		----	
1603		----		----	
1608	D5386	1		-0.31	
1623		----		----	
1718		----		----	
1823	D5386	1.4		-0.08	
1866		----		----	
1880	D5386	1.53		0.00	
1960		----		----	
7006		----		----	
7013		----		----	
9008	D5386	1.84		0.18	
9009	D5386	1.9		0.21	
	normality	OK			
	n	29			
	outliers	1			
	mean (n)	1.53			
	st.dev. (n)	0.642			
	R(calc.)	1.80			
	R(D5386:10)	4.84			



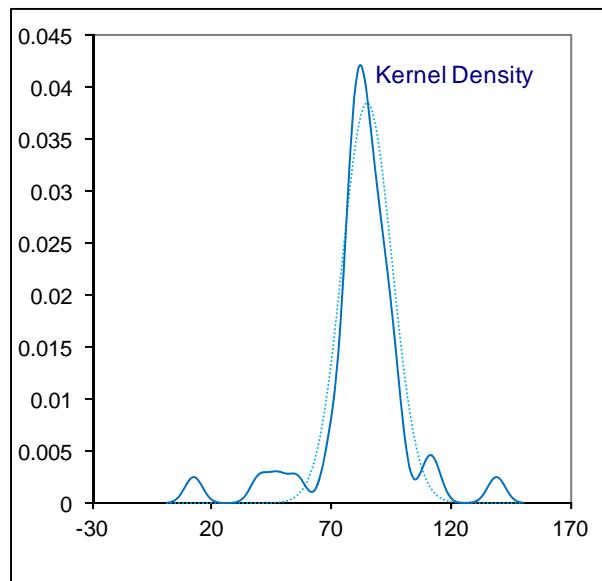
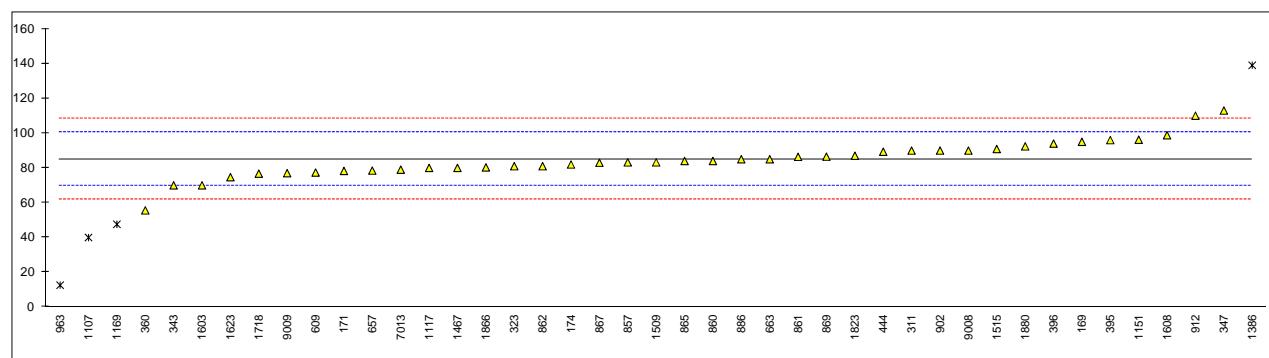
Determination of Density at 20 °C on sample #14200; results in kg/L

lab	method	value	mark	z(targ)	remarks
120	D4052	1.1133		-0.27	
150	D4052	1.1138	R(0.01)	2.53	
169	D4052	1.1136		1.41	
171	D4052	1.11342		0.40	
174	D4052	1.1134		0.29	
311	D4052	1.1133		-0.27	
323	D4052	1.1133		-0.27	
343	D4052	1.1133		-0.27	
347	D4052	1.11325		-0.55	
360	D4052	1.1133		-0.27	
370	D4052	1.1133		-0.27	
395	D4052	1.1133		-0.27	
396	D4052	1.1133		-0.27	
444	D4052	1.11344		0.51	
528	D4052	1.1134		0.29	
529	D4052	1.1135		0.85	
551		-----		-----	
557		-----		-----	
558		-----		-----	
609	D4052	1.11332		-0.16	
657	D4052	1.11340		0.29	
663	D4052	1.1134	C	0.29	First reported:1113.4
825	D4052	1.1133		-0.27	
857	D4052	1.11326		-0.50	
860	D4052	1.11331		-0.22	
861	D4052	1.11341		0.34	
862	D4052	1.11321		-0.78	
865	D4052	1.11332		-0.16	
867	D4052	1.11332		-0.16	
869	D4052	1.11341		0.34	
886	D4052	1.1133		-0.27	
902	D4052	1.11338		0.17	
912	D4052	1.1133		-0.27	
913		-----		-----	
962	D4052	1.1133		-0.27	
963	D4052	1.1133		-0.27	
1101	ISO12185	1.1136	C	1.41	First reported:0.0011136
1107	D4052	1.11329		-0.33	
1117	D4052	1.1134		0.29	
1151	D4052	1.11325		-0.55	
1169	D4052	1.11346		0.62	
1217		-----		-----	
1386	D4052	1.1131		-1.39	
1467	D4052	1.11307		-1.56	
1509	D4052	1.1134		0.29	
1515	D4052	1.1135		0.85	
1603	in house	1.11339		0.23	
1608	D4052	1.11322		-0.72	
1623	D4052	1.11347		0.68	
1718	D4052	1.11322		-0.72	
1823	D4052	1.1134		0.29	
1866	D4052	1.1133		-0.27	
1880	D4052	1.11360		1.41	
1960		-----		-----	
7006	D4052	1.11215	R(0.01)	-6.71	
7013	D4052	1.11343		0.45	
9008		-----		-----	
9009		-----		-----	
	normality	OK			
	n	48			
	outliers	2			
	mean (n)	1.11335			
	st.dev. (n)	0.000110			
	R(calc.)	0.00031			
	R(D4052:02e1)	0.00050			



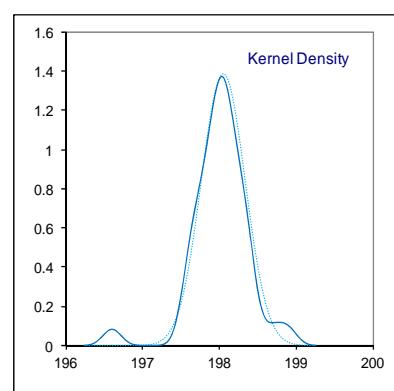
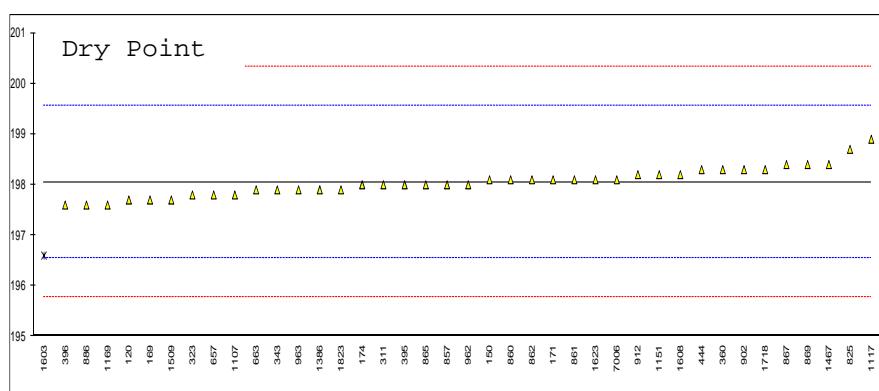
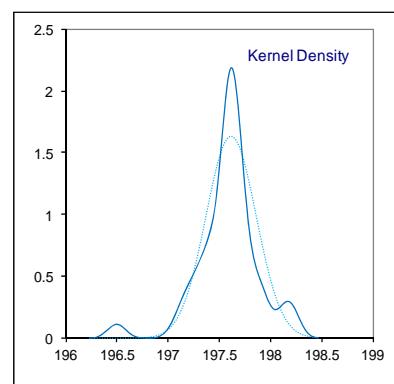
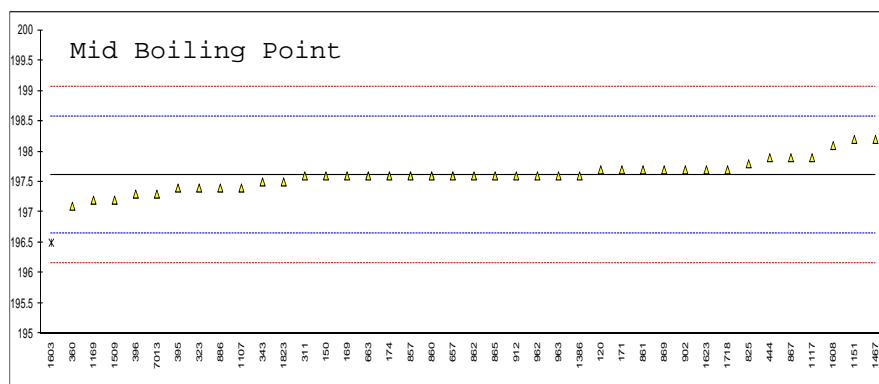
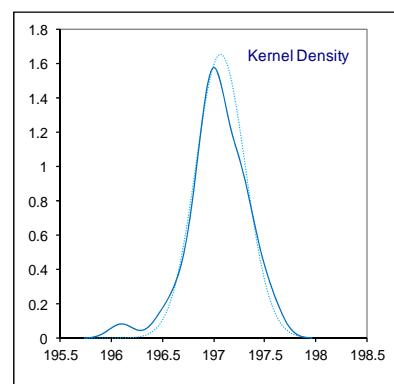
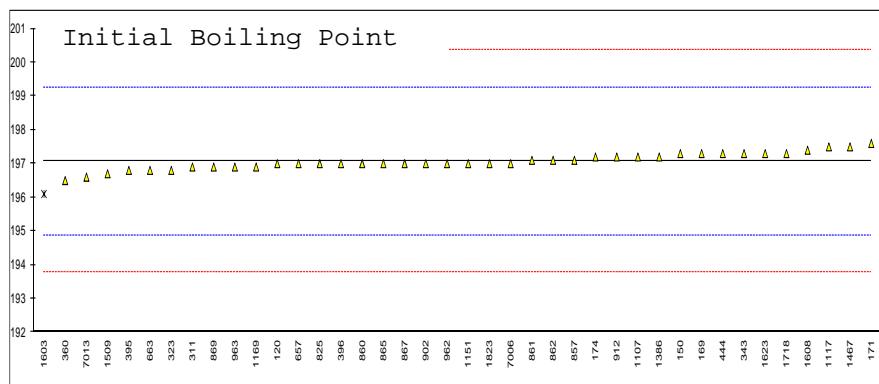
Determination of Diethylene Glycol on sample #14200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		----		----	
150		----		----	
169	E2409	95		1.30	
171	E2409	78.27		-0.87	
174	E2409	82		-0.39	
311	E2409	90		0.65	
323	E2409	81		-0.52	
343	E2409	70		-1.94	
347	E2409	113		3.63	
360	E2409	55.6		-3.81	
370		----		----	
395	E2409	96		1.43	
396	E2409	94		1.17	
444	E2409	89.3	C	0.56	First reported:0.018
528		----		----	
529		----		----	
551		----		----	
557		----		----	
558		----		----	
609	E2409	77.33		-0.99	
657	E2409	78.5		-0.84	
663	E2409	85.0		0.00	
825		----		----	
857	E2409	83.2		-0.23	
860	E2409	84		-0.13	
861	E2409	86.4		0.18	
862	E2409	81		-0.52	
865	E2409	84		-0.13	
867	E2409	83		-0.26	
869	E2409	86.5		0.20	
886	E2409	85.0		0.00	
902	E2409	90		0.65	
912	E2409	110		3.24	
913		----		----	
962		----		----	
963	E2409	12.6	R(0.01)	-9.37	
1101		----	W	-----	Result withdrawn, wrong parameter was reported
1107	in house	40	R(0.05)	-5.83	
1117	E2409	80		-0.65	
1151	E2409	96.2		1.45	
1169	E2409	47.6	R(0.05)	-4.84	
1217		----		----	
1386	E2409	139	R(0.01)	7.00	
1467	E2409	80		-0.65	
1509	E2409	83.2		-0.23	
1515	E2409	90.9		0.77	
1603	in house	70		-1.94	
1608	E2409	98.80		1.79	
1623	E2409	74.67		-1.34	
1718	E2409	76.7		-1.07	
1823	E2409	87		0.26	
1866	E2409	80.32		-0.60	
1880	E2409	92.4		0.96	
1960		----		----	
7006	E202	<50		<-4.42	False negative test result?
7013	E2409	79		-0.77	
9008	E2409	90		0.65	
9009	E2409	77		-1.03	
	normality	not OK			
	n	39			
	outliers	4			
	mean (n)	84.98			
	st.dev. (n)	10.362			
	R(calc.)	29.01			
	R(E2409:13)	21.62			Application range: 5 – 3000 mg/kg



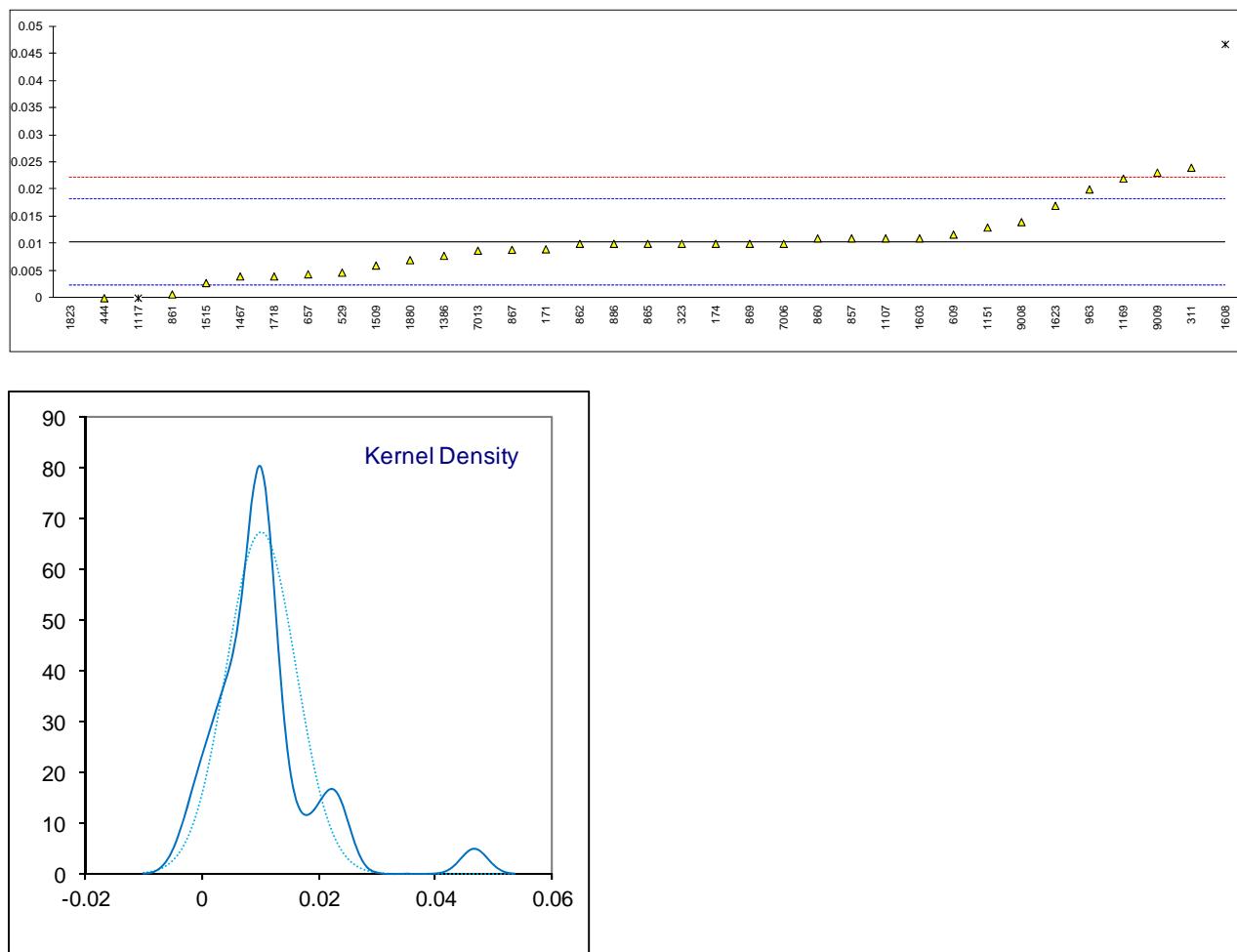
Determination of Distillation: IBP, 50% recovered, Dry Point on sample #14200; results in °C

lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)	remarks
120	D1078	197.0		-0.06	197.7		0.18	197.7		-0.47	
150	D1078	197.3		0.21	197.6		-0.03	198.1		0.06	
169	D1078	197.3		0.21	197.6		-0.03	197.7		-0.47	
171	D1078	197.6		0.48	197.7		0.18	198.1		0.06	
174	D1078	197.2		0.12	197.6		-0.03	198.0		-0.07	
311	D1078	196.9		-0.15	197.6		-0.03	198.0		-0.07	
323	D1078	196.8		-0.25	197.4		-0.45	197.8		-0.34	
343	D1078	197.3		0.21	197.5		-0.24	197.9		-0.20	
347		----		----	----		----	----		----	
360	D1078	196.5		-0.52	197.1		-1.07	198.3		0.33	
370		----		----	----		----	----		----	
395	D1078	196.8		-0.25	197.4		-0.45	198.0		-0.07	
396	D1078	197.0		-0.06	197.3		-0.65	197.6		-0.60	
444	D1078	197.3		0.21	197.9		0.59	198.3		0.33	
528		----		----	----		----	----		----	
529		----		----	----		----	----		----	
551		----		----	----		----	----		----	
557		----		----	----		----	----		----	
558		----		----	----		----	----		----	
609		----		----	----		----	----		----	
657	D1078	197.0		-0.06	197.6		-0.03	197.8		-0.34	
663	D1078	196.8		-0.25	197.6		-0.03	197.9		-0.20	
825	D1078	197.0		-0.06	197.8		0.38	198.7		0.85	
857	D1078	197.1		0.03	197.6		-0.03	198.0		-0.07	
860	D1078	197.0		-0.06	197.6		-0.03	198.1		0.06	
861	D1078	197.1		0.03	197.7		0.18	198.1		0.06	
862	D1078	197.1		0.03	197.6		-0.03	198.1		0.06	
865	D1078	197.0		-0.06	197.6		-0.03	198.0		-0.07	
867	D1078	197.0		-0.06	197.9		0.59	198.4		0.46	
869	D1078	196.9		-0.15	197.7		0.18	198.4		0.46	
886		----		----	197.4		-0.45	197.6		-0.60	
902	D1078	197.0		-0.06	197.7		0.18	198.3		0.33	
912	D1078	197.2		0.12	197.6		-0.03	198.2		0.19	
913		----		----	----		----	----		----	
962	D1078	197.0		-0.06	197.6		-0.03	198.0		-0.07	
963	D1078	196.9		-0.15	197.6		-0.03	197.9		-0.20	
1101		----		----	----		----	----		----	
1107	D1078	197.2		0.12	197.4		-0.45	197.8		-0.34	
1117	D1078	197.5		0.39	197.9		0.59	198.9		1.12	
1151	D1078	197.0		-0.06	198.2		1.21	198.2		0.19	
1169	D1078	196.9		-0.15	197.2		-0.86	197.6		-0.60	
1217		----		----	----		----	----		----	
1386	D1078	197.2		0.12	197.6		-0.03	197.9		-0.20	
1467	D1078	197.5		0.39	198.2		1.21	198.4		0.46	
1509	D1078	196.7		-0.34	197.2		-0.86	197.7		-0.47	
1515		----		----	----		----	----		----	
1603	in house	196.1	R(0.05)	-0.88	196.5	R(0.01)	-2.31	196.6	R(0.01)	-1.92	
1608	D1078	197.4		0.30	198.1		1.01	198.2		0.19	
1623	D1078	197.3		0.21	197.7		0.18	198.1		0.06	
1718	D1078	197.3		0.21	197.7		0.18	198.3		0.33	
1823	D1078	197.0		-0.06	197.5		-0.24	197.9		-0.20	
1866		----		----	----		----	----		----	
1880		----		----	----		----	----		----	
1960		----		----	----		----	----		----	
7006	D1078	197.0		-0.06	----		----	198.1		0.06	
7013	D1078	196.6		-0.43	197.3		-0.65	----		----	
9008		----		----	----		----	----		----	
9009		----		----	----		----	----		----	
normality		OK		OK			OK				
n		39		39			39				
outliers		1		1			1				
mean (n)		197.07		197.62			198.05				
st.dev. (n)		0.2419		0.244			0.287				
R(calc.)		0.68		0.68			0.80				
R(D1078:11)		3.07		1.35			2.12				



Determination of Iron as Fe on sample #14200; results in mg/kg

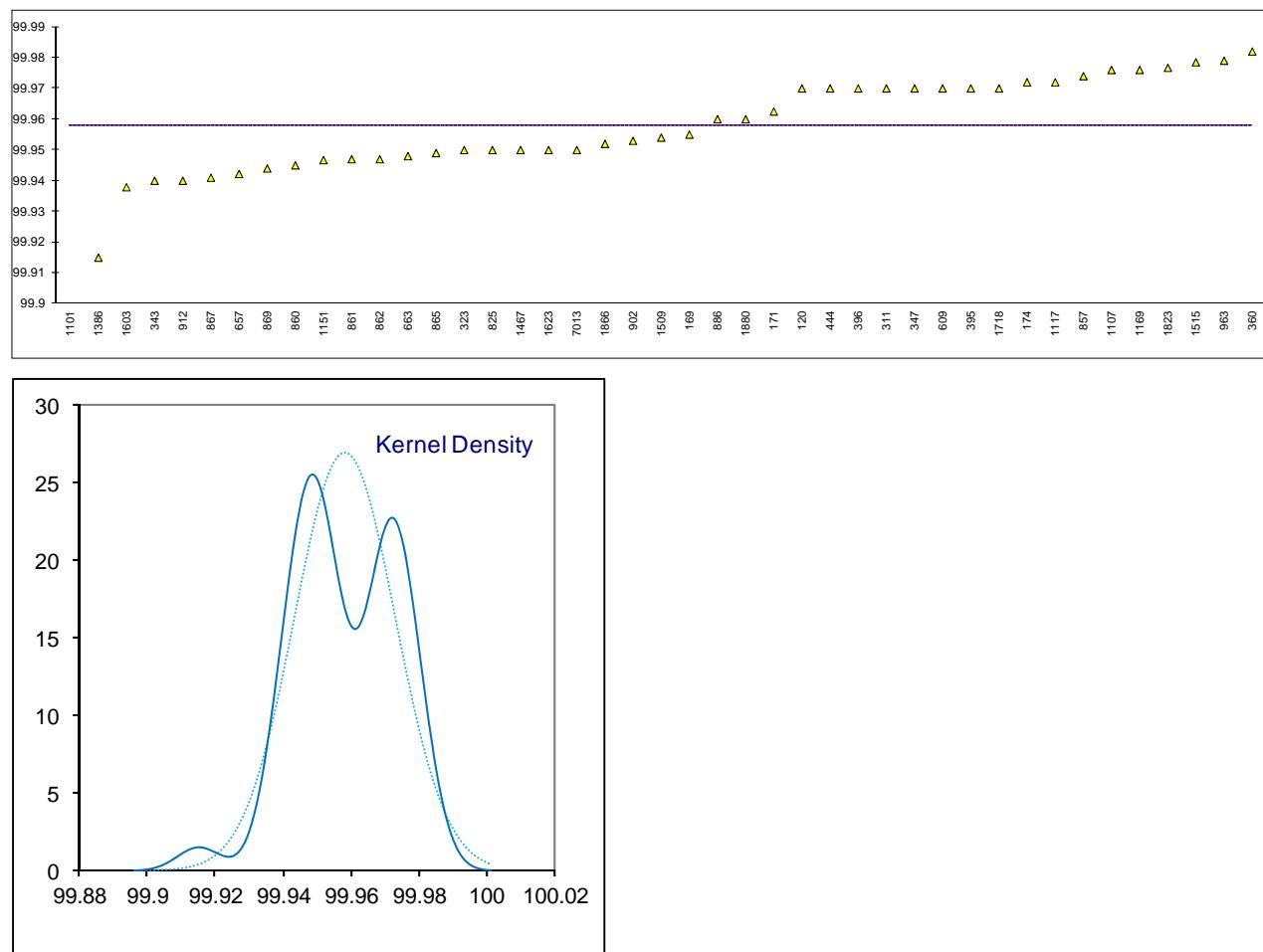
lab	method	value	mark	z(targ)	
120		----		----	
150	E394	<0.01		----	
169		----		----	
171	E1615	0.009		-0.31	
174	E1615	0.010		-0.05	
311	E1615	0.024		3.49	
323	E1615	0.010		-0.05	
343	E1615	<0.010		----	
347	E394	<0.01		----	
360		----		----	
370		----		----	
395		----		----	
396		----		----	
444	E1615	0.00		-2.58	
528		-----		-----	
529	E1615	0.0047		-1.39	
551		----		----	
557		----		----	
558		----		----	
609	E1615	0.01169		0.37	
657	E1615	0.0044		-1.47	
663	E394	<0.01		----	
825		----		----	
857	E1615	0.011		0.20	
860	E394	0.011		0.20	
861	E394	0.0007		-2.41	
862	E1615	0.010		-0.05	
865	E394	0.010		-0.05	
867	E1615	0.0089		-0.33	
869	E394	0.010		-0.05	
886	E202	0.01		-0.05	
902	E1615	<0.01		----	
912		----		----	
913		----		----	
962		----		----	
963	E394	0.02		2.48	
1101		----		----	
1107	E394	0.011		0.20	
1117	E394	0	ex	-2.58	Result excluded, zero is not real value
1151	E394	0.013		0.70	
1169	E1615	0.022		2.98	
1217		----		----	
1386	E1615	0.0078		-0.61	
1467	E1615	0.004		-1.57	
1509	E394	0.006		-1.07	
1515	E394	0.00278		-1.88	
1603	in house	0.011		0.20	
1608	E1615	0.0467	G(0.01)	9.23	
1623	E202	0.017		1.72	
1718	E394	0.004		-1.57	
1823	E394	-0.003	ex	-3.34	Result excluded, true test result can't be negative
1866	E1615	<0.05		----	
1880	E1615	0.007		-0.81	
1960		----		----	
7006	E394	0.01		-0.05	
7013	E394	0.00874		-0.37	
9008	E1615	0.014		0.96	
9009	E1615	0.0231		3.26	
	normality	OK			
	n	32			
	outliers	1 + 2 excl.			
	mean (n)	0.0102			
	st.dev. (n)	0.00594			
	R(calc.)	0.0166			
	R(E1615:08)	0.0111			



Determination of Purity as received on sample #14200; results in %M/M

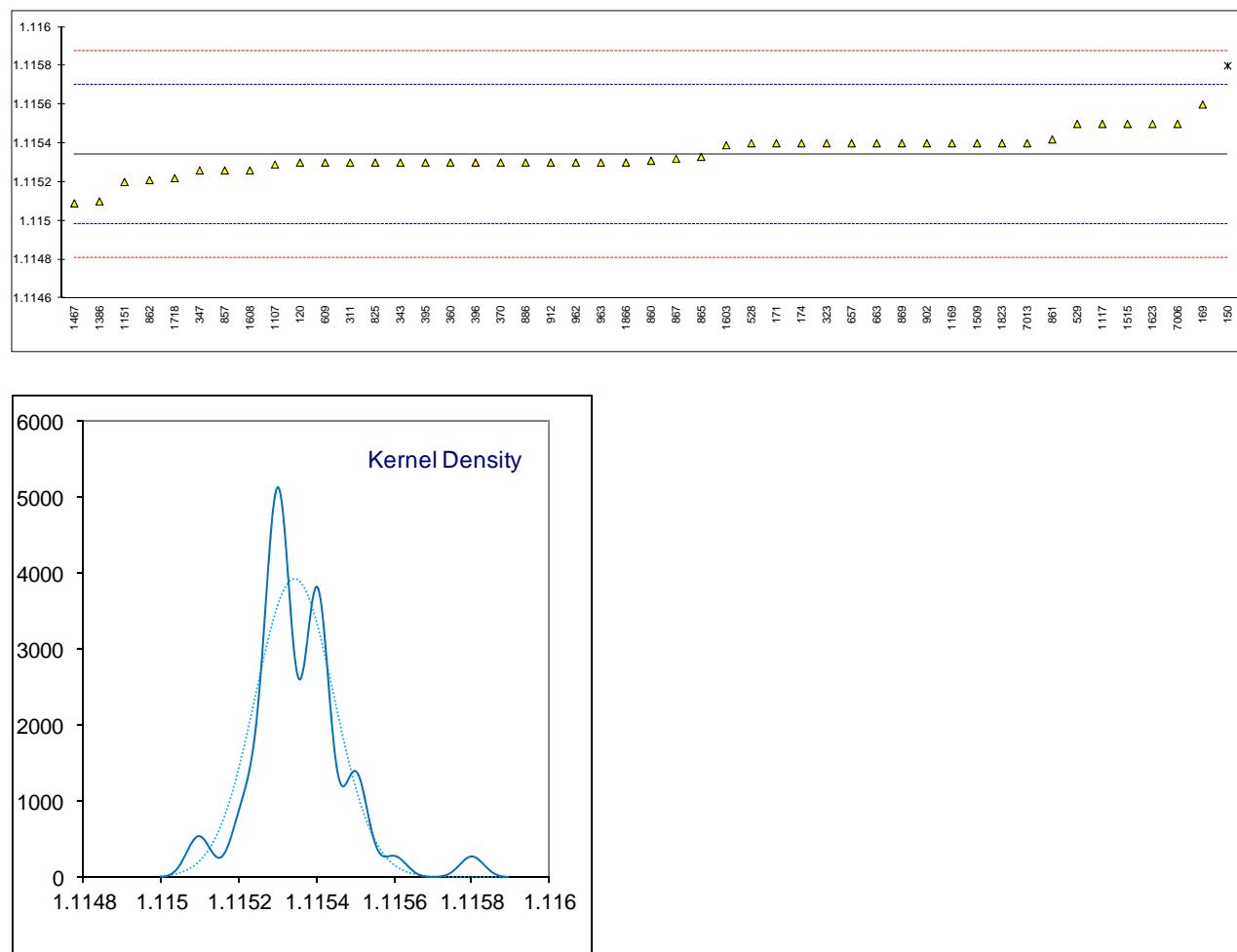
lab	method	value	mark	z(targ)	remarks
120	E202	99.97		----	
150		----		----	
169	E202	99.955		----	
171	E2409	99.9625		----	
174	E2409	99.972		----	
311	in house	99.97		----	
323	E2409	99.95		----	
343		99.94		----	
347	E2409	99.97		----	
360	E2409	99.982		----	
370		----		----	
395	E2409	99.97		----	
396	E2409	99.97		----	
444	E2409	99.97		----	
528		----		----	
529		----		----	
551		----		----	
557		----		----	
558		----		----	
609	E2409	99.97		----	
657	E2409	99.9422		----	
663	E2409	99.948		----	
825	E2409	99.95		----	
857	E2409	99.974		----	
860	E2409	99.945		----	
861	E2409	99.947		----	
862	E202	99.947		----	
865	E2409	99.949		----	
867	E2409	99.941		----	
869	E2409	99.944		----	
886	E2409	99.96		----	
902	E2409	99.953		----	
912		99.94		----	
913		----		----	
962		----		----	
963	E2409	99.979		----	
1101	E202	89.3	R(0.01)	----	
1107	in house	99.976		----	
1117	E2409	99.972		----	
1151	E202	99.9467		----	
1169		99.976		----	
1217		----		----	
1386	E2409	99.915		----	
1467		99.95		----	
1509	E2409	99.954		----	
1515	E2409	99.97851		----	
1603	in house	99.9379		----	
1608		----		----	
1623	E2409	99.95		----	
1718	E2409	99.970		----	
1823	E2409	99.9767	C	----	First reported:99.7766
1866	in house	99.952		----	
1880		99.960		----	
1960		----		----	
7006		----		----	
7013		99.95		----	
9008		----		----	
9009		----		----	
				Group 1	Group 2
normality	OK			OK	OK
n	42			17	24
outliers	1			0	0
mean (n)	99.9580			99.9733	99.9489
st.dev. (n)	0.01484			0.00397	0.00648
R(calc.)	0.0415			0.0111	0.01815
R(lit)	unknown			unknown	unknown

Compare R(iis13C10) = 0.0551



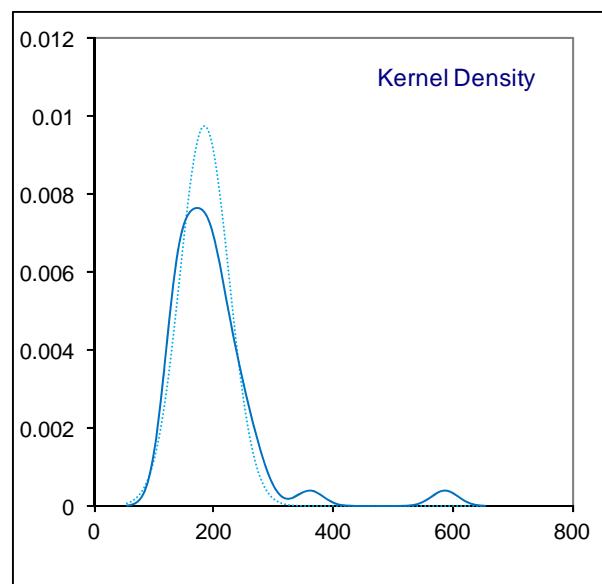
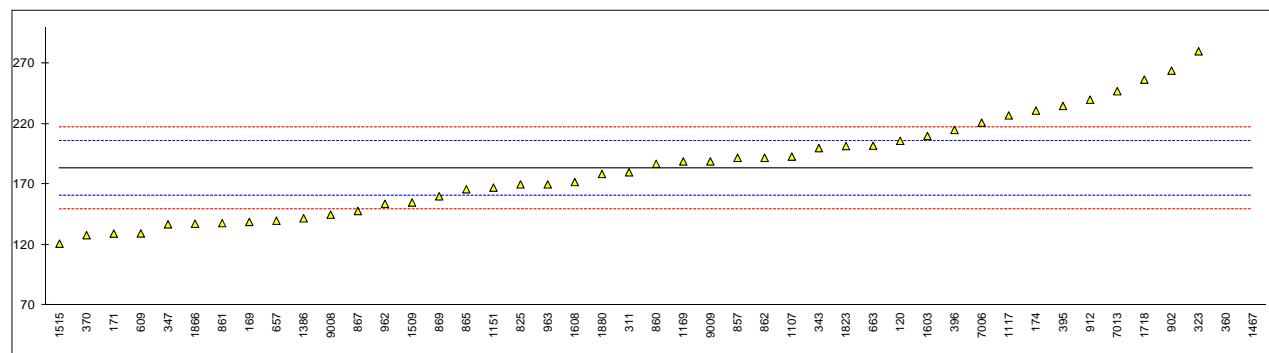
Determination of Specific Gravity 20/20°C on sample #14200;

lab	method	value	mark	z(targ)	remarks
120	D4052	1.1153		-0.24	
150	D4052	1.1158	R(0.01)	2.56	
169	D4052	1.1156		1.44	
171	D4052	1.1154		0.32	
174	D4052	1.1154		0.32	
311	D4052	1.1153		-0.24	
323	E202	1.1154		0.32	
343	E202	1.1153		-0.24	
347	D4052	1.11526		-0.46	
360	D4052	1.1153		-0.24	
370	D4052	1.1153		-0.24	
395	D4052	1.1153		-0.24	
396	E202	1.1153		-0.24	
444		-----		-----	
528	D4052	1.1154		0.32	
529	E202	1.1155		0.88	
551		-----		-----	
557		-----		-----	
558		-----		-----	
609	D4052	1.1153		-0.24	
657	D4052	1.1154		0.32	
663	D4052	1.1154	C	0.32	First reported:1115.4
825	D4052	1.1153		-0.24	
857	D4052	1.11526		-0.46	
860	D4052	1.11531		-0.18	
861	D4052	1.11542		0.43	
862	D4052	1.11521		-0.74	
865	E202	1.11533		-0.07	
867	D4052	1.11532		-0.13	
869	D4052	1.1154		0.32	
886	D4052	1.1153		-0.24	
902	D4052	1.1154		0.32	
912	D4052	1.1153		-0.24	
913		-----		-----	
962	E202	1.1153		-0.24	
963	D4052	1.1153		-0.24	
1101		-----		-----	
1107	E202	1.11529		-0.29	
1117	D4052	1.1155		0.88	
1151	D4052	1.1152		-0.80	
1169	E202	1.1154		0.32	
1217		-----		-----	
1386	E202	1.1151		-1.36	
1467	E202	1.11509		-1.41	
1509	D4052	1.1154		0.32	
1515	D4052	1.1155		0.88	
1603	in house	1.11539		0.27	
1608	E202	1.11526		-0.46	
1623	E202	1.1155		0.88	
1718	D4052	1.11522		-0.69	
1823	D4052	1.1154		0.32	
1866	E202	1.1153		-0.24	
1880		-----		-----	
1960		-----		-----	
7006	D4052	1.1155		0.88	
7013	E202	1.1154		0.32	
9008		-----		-----	
9009		-----		-----	
normality					
n					
outliers					
mean (n)					
st.dev. (n)					
R(calc.)					
R(E202:12)					



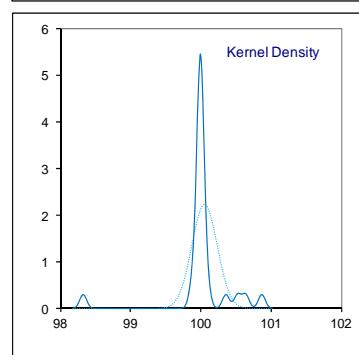
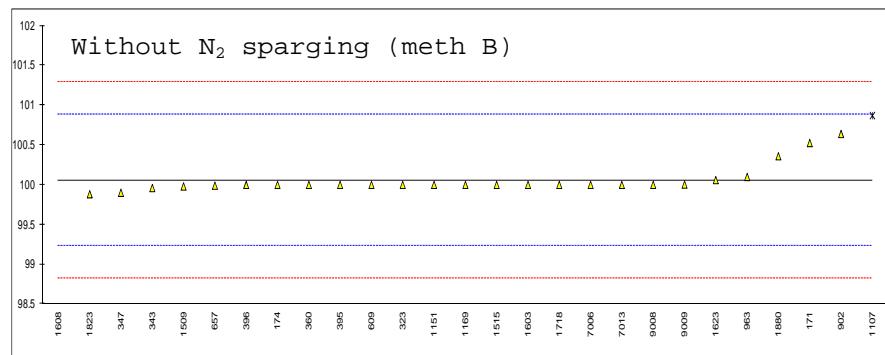
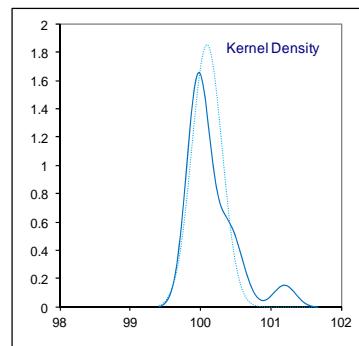
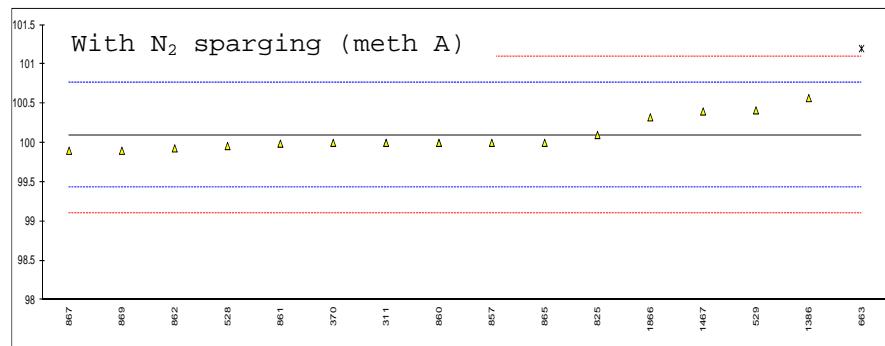
Determination of Water on sample #14201; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	E1064	206		2.02	
150		----		----	
169	E1064	139		-3.96	
171	E1064	129.35567		-4.82	
174	E1064	231		4.25	
311	E1064	180		-0.30	
323	E1064	280		8.63	
343	E1064	200		1.48	
347	E1064	137		-4.14	
360	E1064	359.9	R(0.01)	15.76	
370	E1064	128		-4.95	
395	E1064	234.8		4.59	
396	E1064	215		2.82	
444		----		----	
528		----		----	
529		----		----	
551		----		----	
557		----		----	
558		----		----	
609	E1064	129.47		-4.81	
657	E1064	140		-3.87	
663	E1064	202		1.66	
825	E1064	170		-1.20	
857	E1064	192		0.77	
860	E1064	187		0.32	
861	E1064	138		-4.05	
862	E1064	192		0.77	
865	E1064	166		-1.55	
867	E1064	148		-3.16	
869	E1064	160.2		-2.07	
886		----		----	
902	E1064	264.0		7.20	
912	E1064	240		5.06	
913		----		----	
962	E1064	154		-2.62	
963	E1064	170		-1.20	
1101		----		----	
1107	E1064	193		0.86	
1117	D4672	227		3.89	
1151	E1064	167.3		-1.44	
1169	E1064	189		0.50	
1217		----		----	
1386	E1064	142		-3.70	
1467	E1064	586	R(0.01)	35.95	
1509	E1064	155		-2.53	
1515	E1064	121		-5.57	
1603	in house	210		2.38	
1608	E1064	172.0		-1.02	
1623		----	W	-----	Result withdrawn, first reported:0.02468
1718	E1064	256.6		6.54	
1823	E1064	201.7		1.64	
1866	E1064	137.5		-4.10	
1880	E1064	178.6		-0.43	
1960		----		----	
7006	E203	221.0		3.36	
7013	E1064	247	C	5.68	First reported:0.02468
9008	E1064	145		-3.43	
9009	E1064	189		0.50	
	normality	OK			
	n	43			
	outliers	2			
	mean (n)	183.38			
	st.dev. (n)	41.072			
	R(calc.)	115.00			
	R(E1064:12)	31.36			



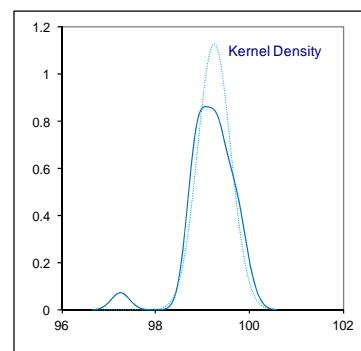
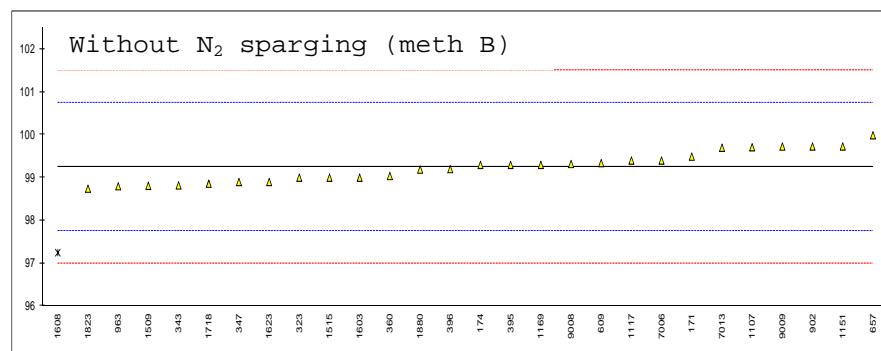
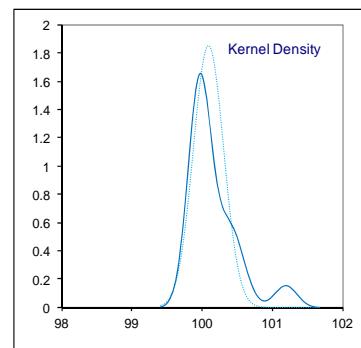
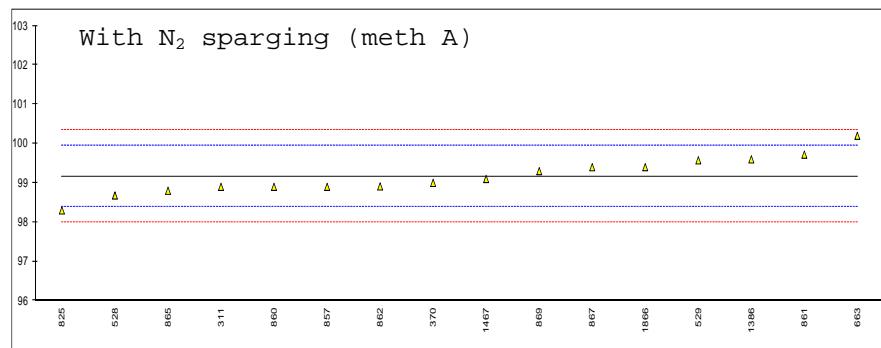
Determination of UV Transmittance at 350 nm on sample #14201; results in %Transmittance

lab	method	Meth A	mark	z(targ)	Meth B	mark	z(targ)	remarks
120		----		----	----		----	
150		----		----	----		----	
169		----		----	----		----	
171	E2193-B	----		----	100.5260	C	1.14	First reported method A
174	E2193-B	----		----	100.0		-0.14	
311	E2193-A	100.0		-0.30	----		----	
323	E2193-B	----		----	100.0	C	-0.14	First reported method A
343	E2193	----		----	99.96	C	-0.23	First reported:78.03
347	E2193-B	----		----	99.9		-0.38	
360	E2193-B	----		----	100.00		-0.14	
370	E2193-A	100		-0.30	----		----	
395	E2193-B	----		----	100		-0.14	
396	E2193-B	----		----	100.0		-0.14	
444		----		----	----		----	
528	E2193-A	99.96	C	-0.42	----		----	First reported:89.39
529	E2193-A	100.414		0.94	----		----	
551		----		----	----		----	
557		----		----	----		----	
558		----		----	----		----	
609	E2193-B	----		----	100.000		-0.14	
657	E2193-B	----		----	99.99		-0.16	
663	E2193-A	101.2	C,G(0.01)	3.29	----		----	First reported:94.89
825	E2193-A	100.1		0.00	----		----	
857	E2193-A	100.0		-0.30	----		----	
860	E2193-A	100.0		-0.30	----		----	
861	E2193-A	99.99		-0.33	----		----	
862	E2193-A	99.93		-0.51	----		----	
865	E2193-A	100.0		-0.30	----		----	
867	E2193-A	99.9		-0.60	----		----	
869	E2193-A	99.9		-0.60	----		----	
886		----		----	----		----	
902	E2193-B	----		----	100.64	C	1.42	First reported: method A
912		----		----	----		----	
913		----		----	----		----	
962		----		----	----		----	
963	E2193-B	----		----	100.1		0.11	
1101		----		----	----		----	
1107	E2193-B	----		----	100.87	R(0.01)	1.97	
1117	E2193-B	----		----	>99.9		----	
1151	E2193	----		----	100.0		-0.14	
1169	E2193-B	----		----	100	C	-0.14	First reported: method A, 79.6
1217		----		----	----		----	
1386	E2193-A	100.57		1.41	----		----	
1467	E2193-A	100.4		0.90	----		----	
1509	E2193-B	----		----	99.98		-0.18	
1515	E2193-B	----		----	100.0	C	-0.14	Results mixed up
1603	in house	----		----	100		-0.14	
1608	E2193-B	----		----	98.333	R(0.01)	-4.18	
1623	E2193-B	----		----	100.06		0.01	
1718	E2193	----		----	100.00		-0.14	
1823	E2193-B	----		----	99.882		-0.42	
1866	E2193-A	100.325		0.68	----		----	
1880	E2193	----		----	100.36		0.74	
1960		----		----	----		----	
7006	E2193-B	----		----	100		-0.14	
7013	E2193	----		----	100.0		-0.14	
9008	E2193-B	----		----	100.00	C	-0.14	First reported method A
9009	E2193-B	----		----	100.004	C	-0.13	First reported method A
<hr/>								
normality	suspect			not OK				
n	15			25				
outliers	1			2				
mean (n)	100.099			100.056				
st.dev. (n)	0.2156			0.1799				
R(calc.)	0.604			0.504				
R(E2193:08)	0.936			1.154				



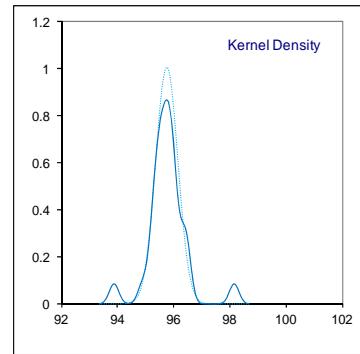
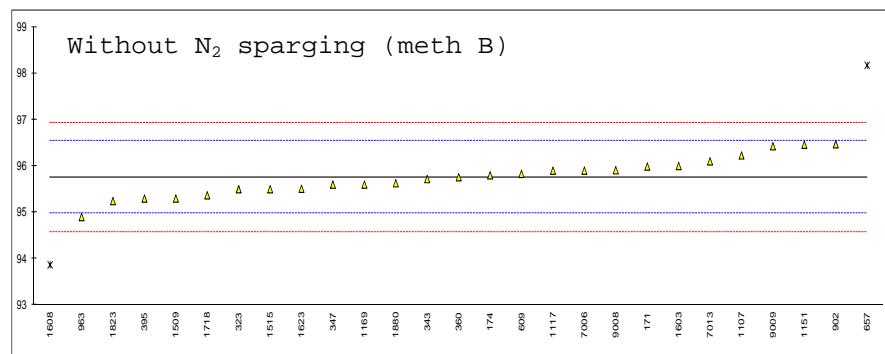
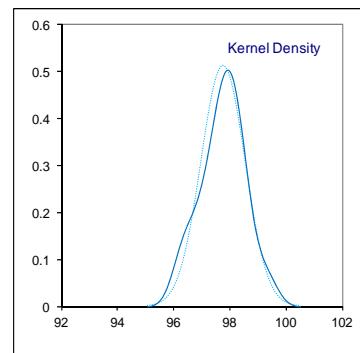
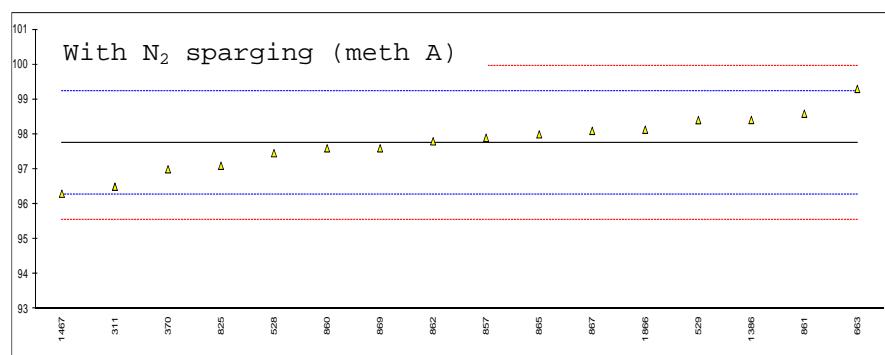
Determination of UV Transmittance at 275 nm on sample #14201; results in %Transmittance

lab	method	Meth A	mark	z(targ)	Meth B	mark	z(targ)	remarks
120		----		----	----		----	
150		----		----	----		----	
169		----		----	----		----	
171	E2193-B	----		----	99.4922	C	0.32	First reported method A
174	E2193-B	----		----	99.3		0.07	
311	E2193-A	98.9		-0.68	----		----	
323	E2193-B	----		----	99.0	C	-0.33	First reported method A
343	E2193	----		----	98.82	C	-0.57	First reported method:95.72
347	E2193-B	----		----	98.9		-0.46	
360	E2193-B	----		----	99.04		-0.28	
370	E2193-A	99		-0.43	----		----	
395	E2193-B	----		----	99.3		0.07	
396	E2193-B	----		----	99.2		-0.06	
444		----		----	----		----	
528	E2193-A	98.68	C	-1.24	----		----	First reported method:97.46
529	E2193-A	99.575		1.04	----		----	
551		----		----	----		----	
557		----		----	----		----	
558		----		----	----		----	
609	E2193-B	----		----	99.340		0.12	
657	E2193-B	----		----	99.99		0.99	
663	E2193-A	100.2	C	2.63	----		----	First reported method:99.27
825	E2193-A	98.3		-2.21	----		----	
857	E2193-A	98.9		-0.68	----		----	
860	E2193-A	98.9		-0.68	----		----	
861	E2193-A	99.72		1.41	----		----	
862	E2193-A	98.91		-0.66	----		----	
865	E2193-A	98.8		-0.94	----		----	
867	E2193-A	99.4		0.59	----		----	
869	E2193-A	99.3		0.34	----		----	
886		----		----	----		----	
902	E2193-B	----		----	99.73	C	0.64	First reported method A
912		----		----	----		----	
913		----		----	----		----	
962		----		----	----		----	
963	E2193-B	----		----	98.8		-0.60	
1101		----		----	----		----	
1107	E2193-B	----		----	99.71		0.61	
1117	E2193-B	----		----	99.4		0.20	
1151	E2193	----		----	99.73		0.64	
1169	E2193-B	----		----	99.3	C	0.07	First reported method A, 95.6
1217		----		----	----		----	
1386	E2193-A	99.60		1.10	----		----	
1467	E2193-A	99.1		-0.17	----		----	
1509	E2193-B	----		----	98.81		-0.58	
1515	E2193-B	----		----	99.0	C	-0.33	results mixed up
1603	in house	----		----	99		-0.33	
1608	E2193-B	----		----	97.248	R(0.01)	-2.66	
1623	E2193-B	----		----	98.90		-0.46	
1718	E2193-B	----		----	98.86		-0.52	
1823	E2193-B	----		----	98.744		-0.67	
1866	E2193-A	99.402		0.60	----		----	
1880	E2193	----		----	99.19		-0.08	
1960		----		----	----		----	
7006	E2193-B	----		----	99.4		0.20	
7013	E2193	----		----	99.7		0.60	
9008	E2193-B	----		----	99.32	C	0.10	First reported method A
9009	E2193-B	----		----	99.728	C	0.64	First reported method A
normality								
n		OK			OK			
n		16			27			
outliers		0			1			
mean (n)		99.168			99.248			
st.dev. (n)		0.4683			0.3534			
R(calc.)		1.311			0.989			
R(E2193:08)		1.098			2.105			



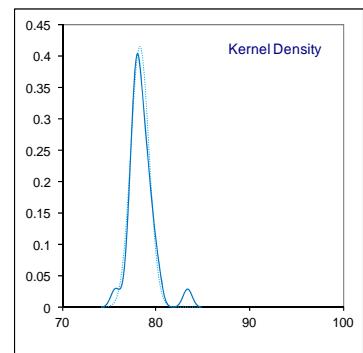
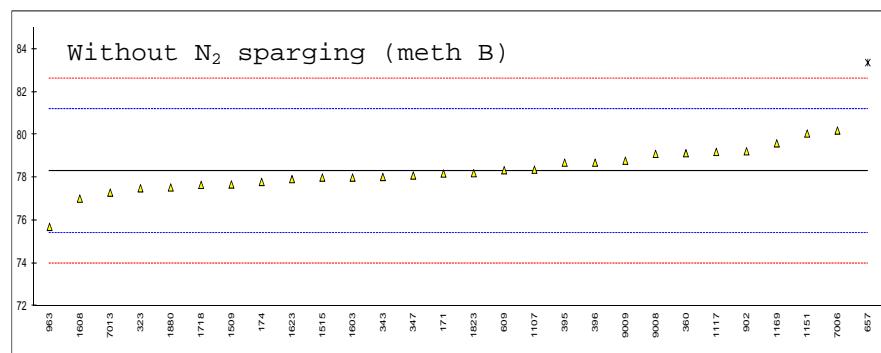
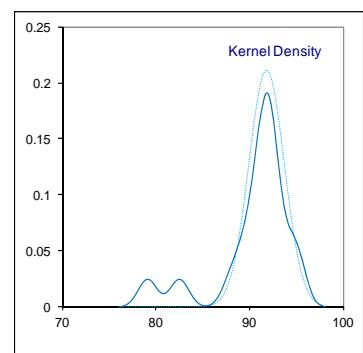
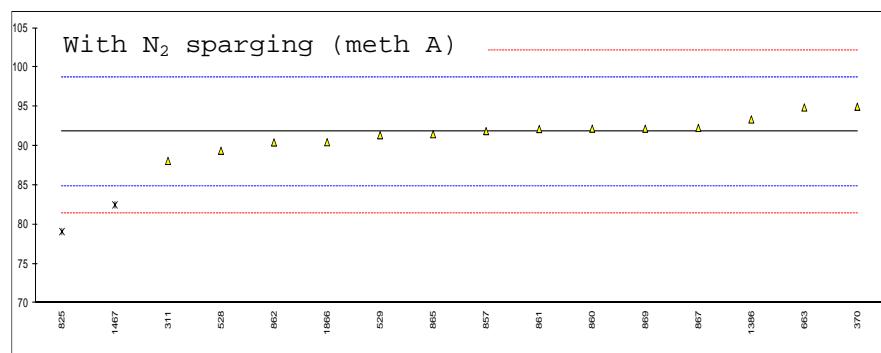
Determination of UV Transmittance at 250 nm on sample #14201; results in %Transmittance

lab	method	Meth A	mark	z(targ)	Meth B	mark	z(targ)	remarks
120		----		----	----		----	
150		----		----	----		----	
169		----		----	----		----	
171	E2193-B	----		----	95.9900	C	0.59	First reported method A
174	E2193-B	----		----	95.8		0.11	
311	E2193-A	96.5		-1.71	----		----	
323	E2193-B	----		----	95.5	C	-0.66	First reported method A,
343	E2193	----		----	95.72	C	-0.10	First reported method:98.82
347	E2193-B	----		----	95.6		-0.40	
360	E2193-B	----		----	95.76		0.00	
370	E2193-A	97		-1.03	----		----	
395	E2193-B	----		----	95.3		-1.16	
396		----		----	----		----	
444		----		----	----		----	
528	E2193-A	97.46	C	-0.41	----		----	First reported method:98.68
529	E2193-A	98.407		0.87	----		----	
551		----		----	----		----	
557		----		----	----		----	
558		----		----	----		----	
609	E2193-B	----		----	95.832		0.19	
657	E2193-B	----		----	98.17	R(0.01)	6.13	
663	E2193-A	99.3	C	2.09	----		----	First reported method:100.23
825	E2193-A	97.1		-0.90	----		----	
857	E2193-A	97.9		0.19	----		----	
860	E2193-A	97.6		-0.22	----		----	
861	E2193-A	98.59		1.12	----		----	
862	E2193-A	97.80		0.05	----		----	
865	E2193-A	98.0		0.32	----		----	
867	E2193-A	98.1		0.46	----		----	
869	E2193-A	97.6		-0.22	----		----	
886		----		----	----		----	
902	E2193-B	----		----	96.47	C	1.81	First reported method A
912		----		----	----		----	
913		----		----	----		----	
962		----		----	----		----	
963	E2193-B	----		----	94.9		-2.18	
1101		----		----	----		----	
1107	E2193-B	----		----	96.23		1.20	
1117	E2193-B	----		----	95.9		0.36	
1151	E2193	----		----	96.46		1.78	
1169	E2193-B	----		----	95.6	C	-0.40	First reported method A, 99.3
1217		----		----	----		----	
1386	E2193-A	98.41		0.88	----		----	
1467	E2193-A	96.3		-1.98	----		----	
1509	E2193-B	----		----	95.30		-1.16	
1515	E2193-B	----		----	95.5	C	-0.66	Results mixed up
1603	in house	----		----	96		0.61	
1608	E2193-B	----		----	93.869	R(0.01)	-4.80	
1623	E2193-B	----		----	95.51		-0.63	
1718	E2193	----		----	95.37		-0.99	
1823	E2193-B	----		----	95.245		-1.30	
1866	E2193-A	98.132		0.50	----		----	
1880	E2193	----		----	95.63		-0.33	
1960		----		----	----		----	
7006	E2193-B	----		----	95.9		0.36	
7013	E2193	----		----	96.1		0.87	
9008	E2193-B	----		----	95.91	C	0.39	First reported method A
9009	E2193-B	----		----	96.429	C	1.70	First reported method A
normality		OK		OK				
n		16		25				
outliers		0		2				
mean (n)		97.762		95.758				
st.dev. (n)		0.7792		0.3982				
R(calc.)		2.182		1.115				
R(E2193:08)		2.063		1.102				



Determination of UV Transmittance at 220 nm on sample #14201; results in %Transmittance

lab	method	Meth A	mark	z(targ)	Meth B	mark	z(targ)	remarks
120		----		----	----		----	
150		----		----	----		----	
169		----		----	----		----	
171	E2193-B	----		----	78.1902	C	-0.08	First reported method A
174	E2193-B	----		----	77.8		-0.35	
311	E2193-A	88.1		-1.07	----		----	
323	E2193-B	----		----	77.5	C	-0.55	First reported method A
343	E2193	----		----	78.03	C	-0.19	First reported:99.96
347	E2193-B	----		----	78.1		-0.14	
360	E2193-B	----		----	79.14		0.58	
370	E2193-A	95	C	0.92	----		----	First reported:94
395	E2193-B	----		----	78.7		0.28	
396	E2193-B	----		----	78.7		0.28	
444		----		----	----		----	
528	E2193-A	89.39	C	-0.70	----		----	First reported:99.96
529	E2193-A	91.373		-0.13	----		----	
551		----		----	----		----	
557		----		----	----		----	
558		----		----	----		----	
609	E2193-B	----		----	78.346		0.03	
657	E2193-B	----		----	83.37	R(0.01)	3.51	
663	E2193-A	94.9	C	0.89	----		----	First reported:101.18
825	E2193-A	79.1	G(0.05)	-3.68	----		----	
857	E2193-A	91.9	C	0.03	----		----	First reported:92.9
860	E2193-A	92.2		0.11	----		----	
861	E2193-A	92.16		0.10	----		----	
862	E2193-A	90.45	C	-0.39	----		----	First reported:92.45
865	E2193-A	91.5		-0.09	----		----	
867	E2193-A	92.3		0.14	----		----	
869	E2193-A	92.2		0.11	----		----	
886		----		----	----		----	
902	E2193-B	----		----	79.23	C	0.64	First reported method A
912		----		----	----		----	
913		----		----	----		----	
962		----		----	----		----	
963	E2193-B	----		----	75.7		-1.80	
1101		----		----	----		----	
1107	E2193-B	----		----	78.37		0.05	
1117	E2193-B	----		----	79.2		0.62	
1151	E2193	----		----	80.06		1.22	
1169	E2193-B	----		----	79.6	C	0.90	First reported method A, 100
1217		----		----	----		----	
1386	E2193-A	93.38		0.45	----		----	
1467	E2193-A	82.5	G(0.05)	-2.69	----		----	
1509	E2193-B	----		----	77.68		-0.43	
1515	E2193-B	----		----	78.0	C	-0.21	Results mixed up
1603	in house	----		----	78		-0.21	
1608	E2193-B	----		----	77.019		-0.89	
1623	E2193-B	----		----	77.93		-0.26	
1718	E2193	----		----	77.66		-0.44	
1823	E2193-B	----		----	78.207		-0.06	
1866	E2193-A	90.479		-0.38	----		----	
1880	E2193	----		----	77.54		-0.53	
1960		----		----	----		----	
7006	E2193-B	----		----	80.2		1.31	
7013	E2193	----		----	77.3		-0.69	
9008	E2193-B	----		----	79.11	C	0.56	First reported method A
9009	E2193-B	----		----	78.788	C	0.34	First reported method A
	normality	OK			suspect			
	n	14			27			
	outliers	2			1			
	mean (n)	91.809			78.300			
	st.dev. (n)	1.8891			0.9630			
	R(calc.)	5.289			2.696			
	R(E2193:08)	9.682			4.047			



APPENDIX 2**Number of participants per country**

1 lab in AUSTRALIA
1 lab in AUSTRIA
2 labs in BELGIUM
3 labs in BRAZIL
1 lab in BULGARIA
1 lab in CANADA
9 labs in CHINA, People's Republic
1 lab in GERMANY
2 labs in INDIA
2 labs in IRAN, Islamic Republic of
2 labs in ITALY
2 labs in KUWAIT
1 lab in LITHUANIA
2 labs in MALAYSIA
2 labs in MEXICO
2 labs in NETHERLANDS
7 labs in SAUDI ARABIA
3 labs in SINGAPORE
1 lab in SOUTH KOREA
2 labs in SPAIN
1 lab in TAIWAN
1 lab in THAILAND
1 lab in TURKEY
1 lab in UNITED KINGDOM
7 labs in UNITED STATES OF AMERICA
1 lab in VENEZUELA

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
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
on db	= on dry basis
ex	= excluded from calculations
E	= probably error in calculations
U	= probably reported in different unit
n.a.	= not applicable
fr.	= first reported
W	= withdrawn on request of the participant

Literature:

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- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 W. Horwitz and R. Albert, J. AOAC Int., Vol. 79, 3, p. 589, (1996)
- 14 Analytical Methods Committee Technical brief, No4 January 2001.
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- 16 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)