

Results of Proficiency Test

Mono Ethylene Glycol (MEG)

October 2015

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 (MEG) every year. As part of the annual proficiency test program of 2015/2016, it was decided to continue this proficiency test on Mono Ethylene Glycol. In this interlaboratory study 63 laboratories in 26 different countries have participated. See appendix 2 for the number of participants per country. In this report the results of the 2015 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 an accredited lab. To get maximum information from this study it was decided to send 2 different samples:

	Bottle type	Tests requested
Sample #15200	1.0 L amber glass bottle	for all regular determinations on MEG
Sample #15201	0.1 L amber glass bottle	for UV transmittance 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 PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation 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 is electronically available through the iis internet site www.iisnl.com, from the FAQ page.

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 125 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, 78 amber glass bottles of 1.0 L were filled and labelled #15200.

The remainder of the batch was used for the UV sample, 78 amber glass bottles of 0.1 litre were filled. The bottles were closed with special screw caps with Teflon inner layer, and labelled #15201.

The homogeneity of the subsamples #15200 was checked by determination of Density in accordance with ASTM D4052, on 8 stratified randomly selected samples. The homogeneity of the sample #15201 was checked by determination UV Transmittance with nitrogen sparging at 220 nm in accordance with ASTM E2193-A on 7 stratified randomly selected samples.

	Density at 20°C in kg/l
Sample #15200-1	1.11331
Sample #15200-2	1.11330
Sample #15200-3	1.11331
Sample #15200-4	1.11330
Sample #15200-5	1.11331
Sample #15200-6	1.11331
Sample #15200-7	1.11331
Sample #15200-8	1.11331

table 2: homogeneity test results of subsamples #15200

	UV(220nm) in T%
Sample #15201-1	74.2
Sample #15201-2	74.8
Sample #15201-3	76.4
Sample #15201-4	75.9
Sample #15201-5	77.1
Sample #15201-6	74.5
Sample #15201-7	75.6

table 3: homogeneity test results of subsamples #15201

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 at 20°C in kg/l	UV(220nm) in T%
r (observed)	0.00001	2.96
0.3xR _(ISO12185:96)	0.00015	--
0.3xR _(ASTM E2193:08-A)	--	2.90

table 4: homogeneity evaluation of subsamples #15200 and #15201

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 #15200 and 1*100 mL bottle, labelled #15201), were sent on October 7, 2015.

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 content, Chloride as Cl, Colour Pt/Co (D1209,) Colour Pt/Co (D5386), Density at 20°C, Diethylene Glycol, Distillation (Initial Boiling Point, 50% recovered and Dry Point), Iron, Purity and Specific Gravity at 20/20°C and Water on sample #15200.

On sample #15201 was requested to determine UV Transmittance (at 350, 275, 250 and 220 nm).

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/.

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 via the data entry portal www.kpmd.co.uk./sgs-iis/. 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 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.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test (no.16). Stragglers are marked by D(0.05) for the Dixon's 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 averages and 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. Two participants reported after the deadline and ten participants did not report any test result at all. Not all participants were able to report all requested parameters. Finally, 53 participants did report 751 numerical test results. Observed were 14 outlying test results, which is 1.9% 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 reported results are discussed per test.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D1613) and an added designation for the year that the method was adopted or revised (e.g. D1613:06). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1613:06(2012)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D1613:06 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, see also paragraph 3.1.

- Acidity: The determination according ASTM E2679 may be very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the strict precision data of ASTM E2679:09. The determination according ASTM D1613 was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D1613:06(2012).
- Aldehydes: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM E2313:08.
- Appearance: A standardized method is available for Appearance since 2009, being ASTM E2680:09(2015). However, not all participants did report according this method. All participants agreed about the appearance of sample #15190 to be ‘clear and bright’, ‘clear and free of suspended matter’ or ‘pass’. Participants who used ASTM E2680 should report the Appearance as ‘pass’ (or ‘fail’). Thirty-one participants reported the appearance correctly as ‘pass’. Thirteen other laboratories reported the Appearance differently (e.g. Clear and Bright (C&B), CCFFSM, CFFSM).
- 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 problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM E2469:08a. The average recovery of the Chloride (theoretical increment of 0.057 mg Chloride/kg) may be satisfactory, less than 127% (the actual blank of Chloride content is unknown).

Colour D1209: 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(2011).

Colour D5386: The determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D5386:10.

Density: 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 ISO12185:96.

DEG: This determination was very problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM E2409:13.

Distillation: This determination was not problematic. No statistical outliers were observed. All three calculated reproducibilities are in good agreement with the requirements of ASTM D1078:11.
From the reported test results of the 50% recovered, it appears that seventeen participants obviously did not correct the results for barometric pressure and thermometer inaccuracy as described in ASTM D1078:11 (paragraph 11.1.3 and 11.1.4).

Iron: 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 ASTM E1615:08. The average recovery of the Iron (theoretical increment of 0.030 mg Iron/kg) may be satisfactory, less than 84% (the actual blank of the Iron content is unknown).

Purity: Regretfully, no reproducibility data for purity are mentioned in ASTM E2409:13. Therefore no significant conclusions were drawn. The calculated reproducibility of the 2015 PT is smaller than the reproducibility to the 2014 PT (0.031 vs 0.042).

Specific Gravity: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of E202:12.

Water: This determination was very problematic. No statistical outliers were observed. The calculated reproducibility 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 problematic. In total two statistical outliers were observed. Only the calculated reproducibility of UV at 350nm is in agreement with the requirements of ASTM E2193:08_method A. The calculated reproducibilities of UV at 275nm, 250nm and 220nm after rejection of the statistical outliers, are in not agreement with the requirements of ASTM E2193:08.

For method B, this determination may also be problematic. In total four statistical outliers were observed. The calculated reproducibilities of UV at 350nm and 275nm after rejection of the statistical outlier, are in agreement with the requirements of ASTM E2193:08_method B. The calculated reproducibilities of UV at 250nm and 220nm after rejection of the statistical outliers, are not at all 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	13	2.27	4.12	1.15
Acidity as Acetic Acid (D1613)	mg/kg	38	6.53	4.76	14.00
Aldehydes as Acetaldehyde	mg/kg	30	75.07	22.23	64.57
Appearance		44	Pass	n.a.	n.a.
Ash	%M/M	23	0.0002	0.0005	(0.0050)
Chloride as Cl	mg/kg	23	0.07	0.09	0.06
Colour D1209 manual	---	28	2.0	3.5	7.0
Colour D5386 automated	---	31	1.8	2.9	4.9
Density at 20°C	kg/L	43	1.1134	0.0003	0.0005
Diethylene Glycol	mg/kg	39	100.8	54.7	25.6
Initial Boiling Point	°C	41	197.0	0.8	3.1
50% recovered	°C	39	197.5	0.7	1.3
Dry Point	°C	39	198.0	1.1	2.1
Iron as Fe	mg/kg	39	0.025	0.022	0.027
Purity	%M/M	42	99.961	0.031	n.a.
Specific Gravity 20/20°C	---	42	1.1154	0.0003	0.0005
Water	mg/kg	51	196.7	101.4	33.6
UV Transmittance at 350 nm (N ₂)	%T	13	99.96	0.67	0.94
UV Transmittance at 275 nm (N ₂)	%T	11	96.42	1.60	1.10
UV Transmittance at 250 nm (N ₂)	%T	11	91.57	3.11	2.06
UV Transmittance at 220 nm (N ₂)	%T	13	78.47	17.97	9.68
UV Transmittance at 350 nm	%T	31	100.06	0.82	1.15
UV Transmittance at 275 nm	%T	33	99.68	2.29	2.11
UV Transmittance at 250 nm	%T	31	90.66	2.45	1.10
UV Transmittance at 220 nm	%T	31	70.42	6.09	4.05

table 5: reproducibilities of samples #15200 and #15201

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 2015 WITH PREVIOUS PTS

	October 2015	October 2014	October 2013	October 2012
Number of reporting labs	53	52	54	54
Number of results reported	751	766	785	838
Statistical outliers	14	31	40	48
Percentage outliers	1.9%	4.0%	5.1%	5.7%

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 2015	October 2014	October 2013	October 2012
Acidity as Acetic Acid (E2679)	--	--	--	++
Acidity as Acetic Acid (D1613)	++	++	n.a.	n.a.
Aldehydes as Acetaldehyde	++	+	++	(++)
Ash	(++)	(++)	(+/-)	(++)
Chloride as Cl	--	--	+/-	--
Colour D1209 manual	++	++	++	++
Colour D5368 automated	+	++	++	+
Density at 20°C	+	+	++	+
Diethylene Glycol	--	-	--	--
Initial Boiling Point	++	++	++	--
50% recovered	++	++	++	++
Dry Point	++	++	++	++
Iron as Fe	+	-	+/-	++
Purity	n.e.	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

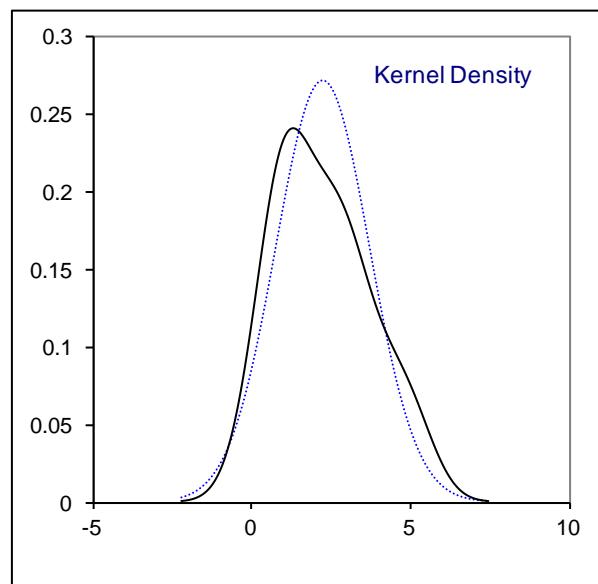
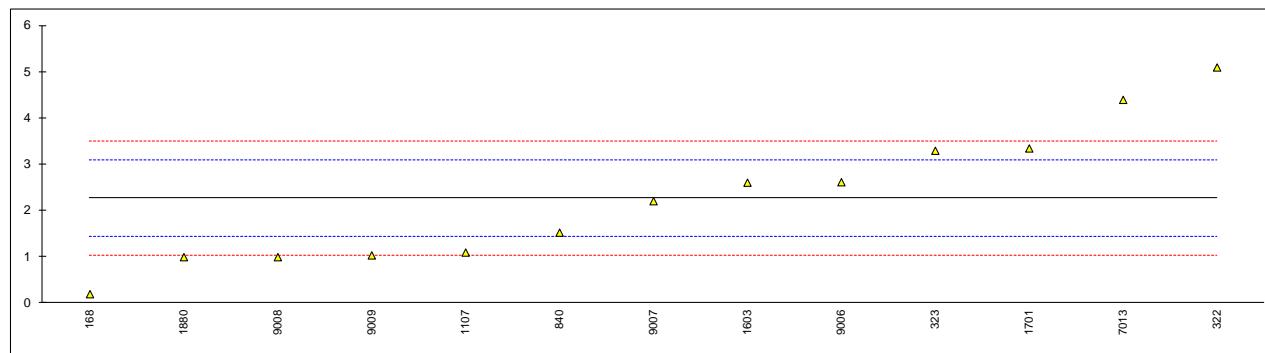
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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

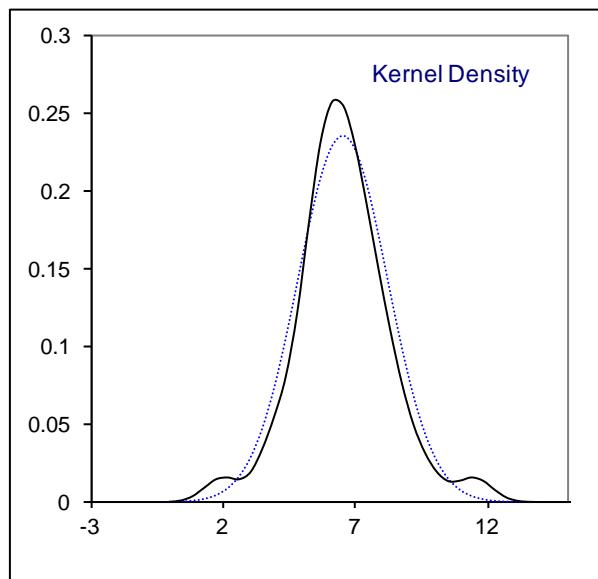
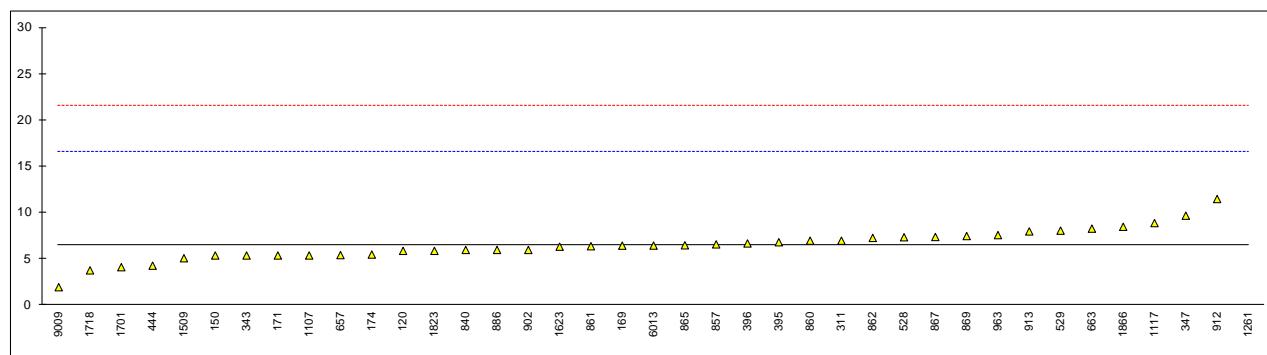
Determination of Acidity as Acetic Acid (E2679) on sample #15200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		----		----	
150		----		----	
168	E2679	0.2		-5.04	
169		----		----	
171		----		----	
174		----		----	
311		----		----	
322	E2679	5.1		6.92	
323	E2679	3.3		2.52	
343		----		----	
347		----		----	
370		----		----	
395		----		----	
396		----		----	
444		----		----	
528		----		----	
529		----		----	
557		----		----	
609		----		----	
610		----		----	
657		----		----	
663		----		----	
825		----		----	
840	E2679	1.53		-1.80	
857		----		----	
860		----		----	
861		----		----	
862		----		----	
865		----		----	
867		----		----	
869		----		----	
886		----		----	
902		----		----	
912		----		----	
913		----		----	
962		----		----	
963		----		----	
1101		----		----	
1107	E2679	1.1		-2.85	
1117		----		----	
1151		----		----	
1217		----		----	
1261		----		----	
1467		----		----	
1509		----		----	
1515		----		----	
1603	in house	2.61		0.84	
1623		----		----	
1701	E2679	3.35		2.65	
1718		----		----	
1823		----		----	
1866		----		----	
1880	E2679	1.0		-3.09	
1954		----		----	
1960		----		----	
2124		----		----	
6013		----		----	
7003		----		----	
7013	E2679	4.4		5.21	
9006	E2679	2.62		0.86	
9007	E2679	2.21		-0.14	
9008	E2679	1		-3.09	
9009	E2679	1.0383		-3.00	
	normality	OK			
	n	13			
	outliers	0			
	mean (n)	2.266			
	st.dev. (n)	1.4708			
	R(calc.)	4.118			
	R(E2679:09)	1.147			



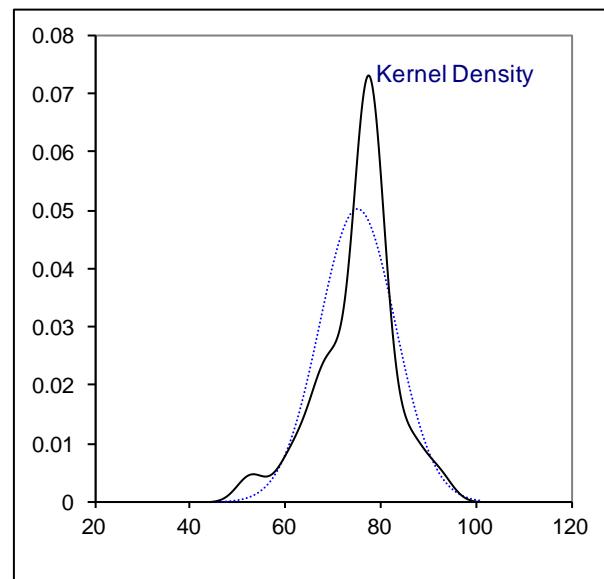
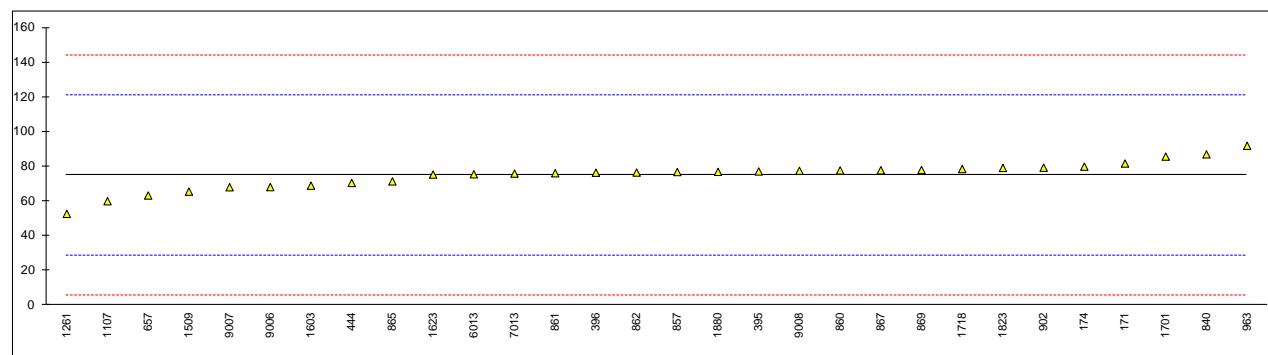
Determination of Acidity as Acetic Acid (D1613) on sample #15200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	D1613	5.9		-0.13	
150	D1613	5.4		-0.23	
168		----		----	
169	D1613	6.46		-0.01	
171	D1613	5.4		-0.23	
174	D1613	5.5		-0.21	
311	D1613	7		0.09	
322		----		----	
323		----		----	
343	D1613	5.4		-0.23	
347	D1613	9.7		0.63	
370		----		----	
395	D1613	6.83		0.06	
396	D1613	6.7		0.03	
444	D1613	4.3	C	-0.45	first reported:1.8
528	D1613	7.37		0.17	
529	D1613	8.084		0.31	
557		----		----	
609		----		----	
610		----		----	
657	D1613	5.44		-0.22	
663	D1613	8.3		0.35	
825		----		----	
840	D1613	6.0		-0.11	
857	D1613	6.6		0.01	
860	D1613	7.0		0.09	
861	D1613	6.4		-0.03	
862	D1613	7.3		0.15	
865	D1613	6.5		-0.01	
867	D1613	7.4		0.17	
869	D1613	7.5		0.19	
886	D1613	6		-0.11	
902	D1613	6		-0.11	
912	D1613	11.5		0.99	
913	D1613	8.0		0.29	
962		----		----	
963	D1613	7.6		0.21	
1101		----		----	
1107	D1613	5.4		-0.23	
1117	D1613	8.9		0.47	
1151		----		----	
1217		----		----	
1261	D1613	110	R(0.01)	20.69	
1467		----		----	
1509	D1613	5.1		-0.29	
1515		----		----	
1603		----		----	
1623	D1613	6.35		-0.04	
1701	D1613	4.135		-0.48	
1718	D1613	3.79		-0.55	
1823	D1613	5.9		-0.13	
1866	D1613	8.50		0.39	
1880		----		----	
1954		----		----	
1960		----		----	
2124		----		----	
6013	D1613	6.47		-0.01	
7003		----		----	
7013		----		----	
9006		----		----	
9007		----		----	
9008		----		----	
9009	D1613	1.9723		-0.91	
	normality	suspect			
	n	38			
	outliers	1			
	mean (n)	6.53			
	st.dev. (n)	1.699			
	R(calc.)	4.76			
	R(D1613:06)	14.00			



Determination of Aldehydes as Acetaldehyde on sample #15200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		----			
150		----			
168		----			
169		----			
171	E2313	81.8		0.29	
174	E2313	80		0.21	
311	E2313	>50			
322		----			
323	E2313	>50			
343		----			
347		----			
370		----			
395	E2313	77.23		0.09	
396	E2313	76.5		0.06	
444	E2313	70.6	C	-0.19	first reported:0
528		----			
529		----			
557		----			
609		----			
610		----			
657	E2313	63.36		-0.51	
663		----			
825		----			
840	E2313	87.07		0.52	
857	E2313	76.9		0.08	
860	E2313	77.8		0.12	
861	E2313	76.2		0.05	
862	E2313	76.6		0.07	
865	E2313	71.5		-0.15	
867	E2313	77.9		0.12	
869	E2313	78.10		0.13	
886		----			
902	E2313	79.4		0.19	
912		----			
913		----			
962		----			
963	E2313	92.02		0.74	
1101		----			
1107	E2313	60.07		-0.65	
1117		----			
1151		----			
1217		----			
1261	E2313	52.79		-0.97	
1467		----			
1509	E2313	65.60		-0.41	
1515		----			
1603	in house	69.02		-0.26	
1623	E2313	75.44		0.02	
1701	INH-502	85.79		0.46	
1718	E2313	78.62		0.15	
1823	E2313	79.24		0.18	
1866		----			
1880	E2313	77.0		0.08	
1954		----			
1960		----			
2124		----			
6013	E2313	75.6		0.02	
7003		----			
7013	E2313	76.0		0.04	
9006	E2313	68.2		-0.30	
9007	E2313	68.12		-0.30	
9008	E2313	77.61		0.11	
9009		----			
	normality	suspect			
	n	30			
	outliers	0			
	mean (n)	75.07			
	st.dev. (n)	7.938			
	R(calc.)	22.23			
	R(E2313:08)	64.57			



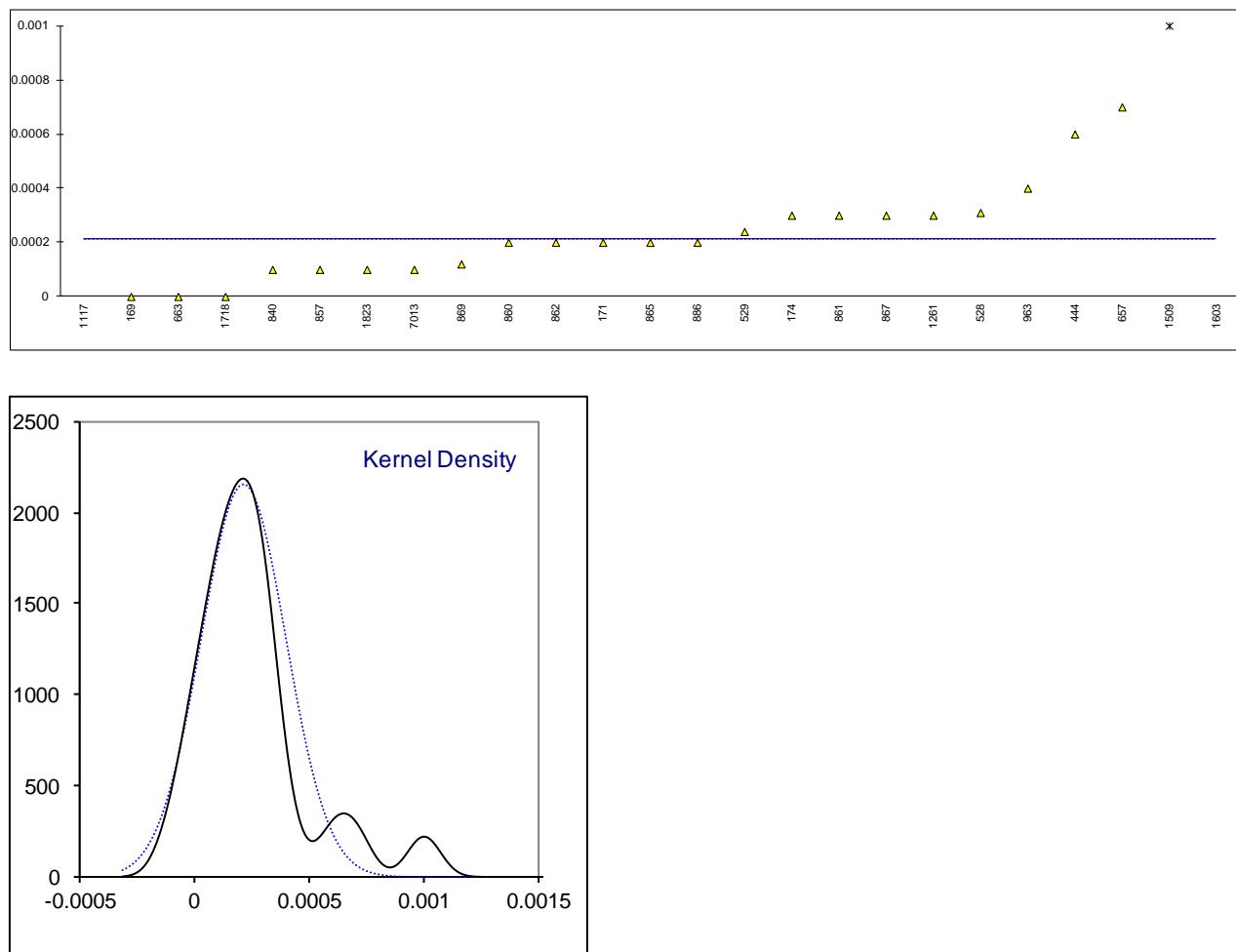
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Determination of Appearance on sample #15200;

lab	method	value	mark	z(targ)	remarks
120	Visual	Pass	-----		
150	E2680	Pass	-----		
168	E2680	Clear&Bright	-----		
169	D4176	C&FSM	-----		
171	E2680	Pass	-----		
174	E2680	Pass	-----		
311	E2680	Pass	-----		
322	E2680	Pass	-----		
323	E2680	Clear&Bright	-----		
343	E2680	Pass	-----		
347	E2680	Pass	-----		
370	E2680	Pass	-----		
395	E2680	Pass	-----		
396		-----	-----		
444	E2680	Pass	-----		
528	E2680	Pass	-----		
529	E2680	Pass	-----		
557		-----	-----		
609		-----	-----		
610		-----	-----		
657	E2680	Pass	-----		
663	Visual	Pass	-----		
825		-----	-----		
840	E2680	Pass	-----		
857	E2680	Pass	-----		
860	E2680	Pass	-----		
861	Visual	Bright&Clear	-----		
862	E2680	Pass	-----		
865	E2680	Pass	-----		
867	Visual	Bright&Clear	-----		
869	Visual	Clear&Bright	-----		
886		-----	-----		
902	E2680	Pass	-----		
912	E2680	Pass	-----		
913	Visual	Pass	-----		
962		-----	-----		
963	E2680	Pass	-----		
1101		-----	-----		
1107	Visual	Clear	-----		
1117	D4176	on spec	-----		
1151		-----	-----		
1217		-----	-----		
1261		-----	-----		
1467		-----	-----		
1509	E2680	Pass	-----		
1515		Pass	-----		
1603	Visual	Clear	-----		
1623	Visual	CCFFSM	-----		
1701	Visual	CFFSM	-----		
1718	D4176	Pass	-----		
1823		-----	-----		
1866		Pass	-----		
1880		-----	-----		
1954		-----	-----		
1960		-----	-----		
2124		Clear&Bright	-----		
6013	D4176	Clear	-----		
7003		Pass	-----		
7013		-----	-----		
9006	E2680	Pass	-----		
9007		-----	-----		
9008	E2680	Pass	-----		
9009		-----	-----		
normality		n.a.			
n		44			
mean (n)		Pass			

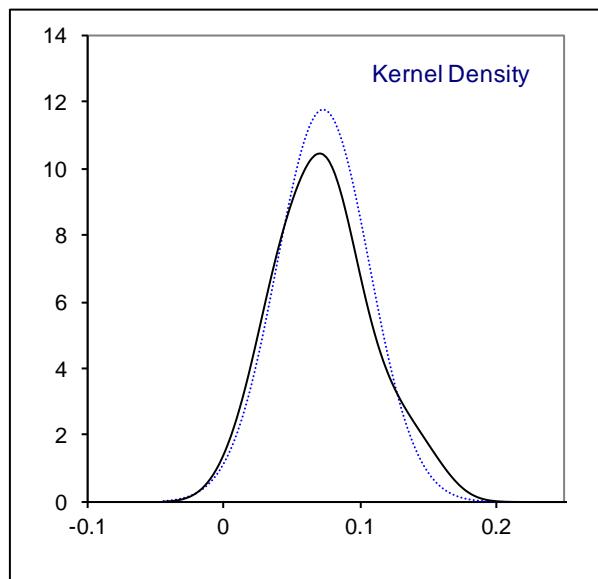
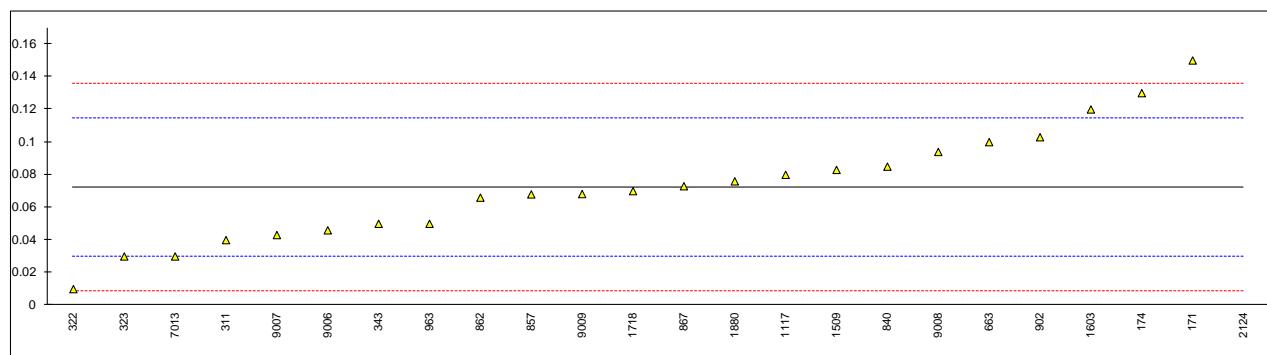
Determination of Ash on sample #15200; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D482	<0.001	----		
150	D482	<0.001	----		
168		----	----		
169	D482	0.000	----		
171	D482	0.0002	----		
174	D482	0.0003	----		
311	D482	<0.001	----		
322		----	----		
323	D482	<0.001	----		
343	D482	<0.001	----		
347	D482	<0.0010	----		
370		----	----		
395		----	----		
396		----	----		
444	D482	0.0006	----		
528	D482	0.00031	----		
529	D482	0.00024	----		
557		----	----		
609		----	----		
610		----	----		
657	D482	0.0007	----		
663	D482	0.000	----		
825		----	----		
840	D482	0.0001	----		
857	D482	0.0001	----		
860	D482	0.0002	----		
861	D482	0.0003	----		
862	D482	0.0002	----		
865	D482	0.0002	----		
867	D482	0.0003	----		
869	D482	0.00012	----		
886	D482	0.0002	----		
902	D482	<0.001	----		
912	D482	<0.001	----		
913		----	----		
962		----	----		
963	D482	0.0004	----		
1101		----	----		
1107		----	----		
1117	D482	-0.0001	----		
1151		----	----		
1217		----	----		
1261	D482	0.0003	----		
1467		----	----		
1509	D482	0.0010	R(0.01)	----	
1515		----	----		
1603	in house	0.0040	R(0.01)	----	false positive test result?
1623	D482	<0.001	----		
1701		----	----		
1718	D482	0.0000	----		
1823	D482	0.0001	----		
1866		----	----		
1880		----	----		
1954		----	----		
1960		----	----		
2124		----	----		
6013	D482	<0.001	----		
7003		----	----		
7013	D482	0.0001	----		
9006		----	----		
9007		----	----		
9008		----	----		
9009		----	----		
normality					
n		suspect			
outliers					
mean (n)		23			
st.dev. (n)		2			
R(calc.)		0.00021			
R(D482:13)		0.000185			
		0.00052			
		(0.00500)			
Application range: 0.001 – 0.180% M/M					



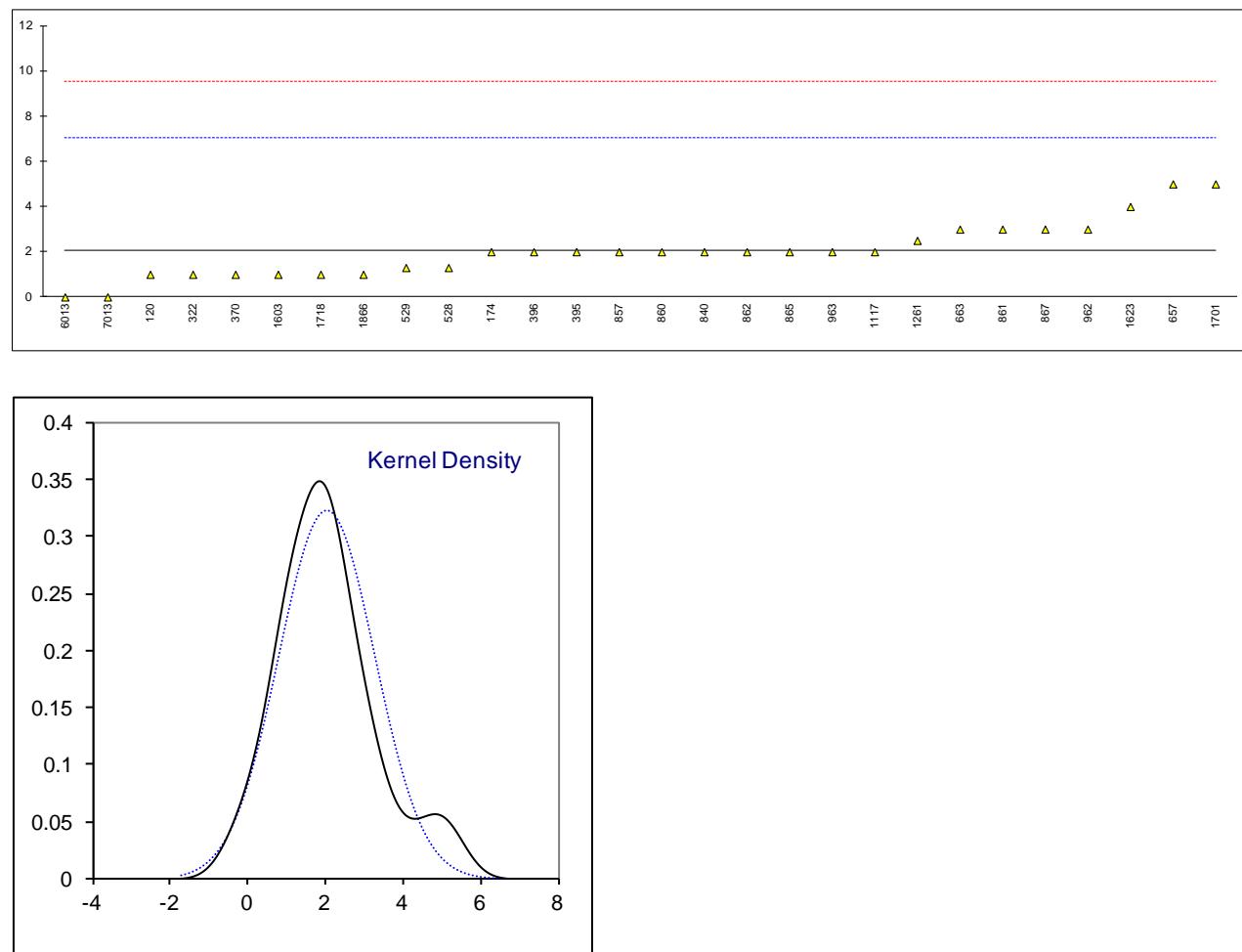
Determination of Chloride as Cl on sample #15200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		----		----	
150		----		----	
168		----		----	
169		----		----	
171	E2469	0.15	C	3.67	first reported:0.17
174	E2469	0.13		2.72	
311	E2469	0.04		-1.53	
322	E2469	0.01		-2.95	
323	E2469	0.03		-2.00	
343	E2469	0.05		-1.06	
347		----		----	
370		----		----	
395		----		----	
396		----		----	
444		----		----	
528		----		----	
529		----		----	
557		----		----	
609		----		----	
610		----		----	
657		----		----	
663	INH-101867	0.10		1.30	
825		----		----	
840	IMPCA002	0.085	C	0.60	first reported:0.17
857	E2469	0.068		-0.21	
860		----		----	
861		----		----	
862	E2469	0.066		-0.30	
865	INH-001	<0.1		----	
867	E2469	0.073		0.03	
869		----		----	
886		----		----	
902	E2469	0.103	C	1.45	first reported:0.21
912		----		----	
913		----		----	
962		----		----	
963	E2469	0.05		-1.06	
1101		----		----	
1107	in house	<0.2		----	
1117	E2469	0.08		0.36	
1151		----		----	
1217		----		----	
1261		----		----	
1467		----		----	
1509	E2469	0.083		0.50	
1515		----		----	
1603	in house	0.120		2.25	
1623		----		----	
1701		----		----	
1718	E2469	0.070		-0.11	
1823		----		----	
1866	E2469	<0.1		----	
1880	E2469	0.076		0.17	
1954		----		----	
1960		----		----	
2124	D4327	1.0	R(0.01)	43.83	
6013	INH-472	<0.2		----	
7003		----		----	
7013	INH-635	0.03		-2.00	
9006	E2469	0.046		-1.25	
9007	E2469	0.0431		-1.38	
9008	E2469	0.094		1.02	
9009	E2469	0.0682		-0.20	
<u>Only ASTM E2469 data:</u>					
normality	OK	<u>spike</u>		OK	
n	23			19	
outliers	1			0	
mean (n)	0.0724	0.057	<127% rec	0.0700	
st.dev. (n)	0.03391			0.03350	
R(calc.)	0.0949			0.0938	
R(E2469:08a)	0.0593			0.0573	



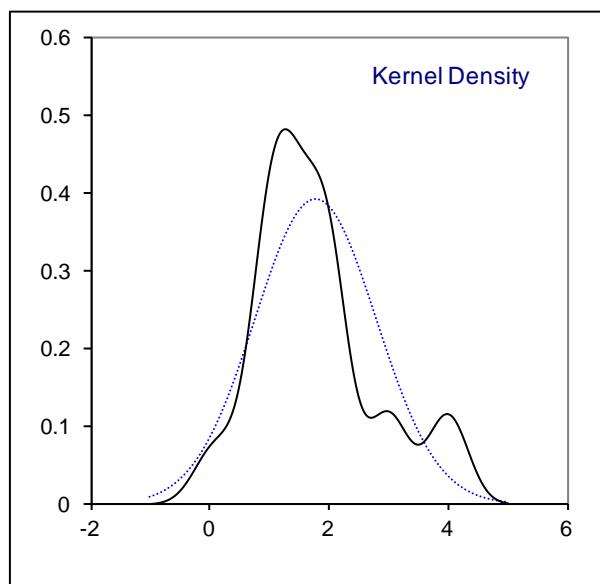
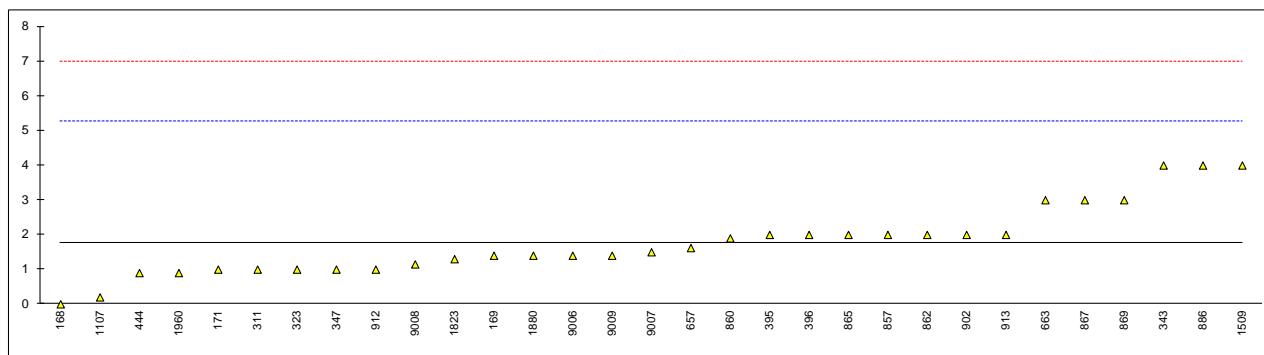
Determination of Colour Pt/Co manual (D1209) on sample #15200;

lab	method	value	mark	z(targ)	remarks
120	D1209	1		-0.42	
150		----		----	
168		----		----	
169	D1209	<5		----	
171		----		----	
174	D1209	2		-0.02	
311	D1209	<5		----	
322	D1209	1		-0.42	
323	D1209	<5		----	
343		----		----	
347		----		----	
370	D1209	1		-0.42	
395	D1209	2		-0.02	
396	D1209	2		-0.02	
444		----		----	
528	D1209	1.3		-0.30	
529	D1209	1.3		-0.30	
557		----		----	
609		----		----	
610		----		----	
657	D1209	5		1.18	
663	D1209	3		0.38	
825		----		----	
840	D1209	2		-0.02	
857	D1209	2		-0.02	
860	D1209	2		-0.02	
861	D1209	3		0.38	
862	D1209	2		-0.02	
865	D1209	2		-0.02	
867	D1209	3		0.38	
869		----		----	
886		----		----	
902		----		----	
912		----		----	
913		----		----	
962	D1209	3		0.38	
963	D1209	2		-0.02	
1101		----		----	
1107		----		----	
1117	D1209	2		-0.02	
1151		----		----	
1217		----		----	
1261	D1209	2.5		0.18	
1467		----		----	
1509	D1209	<5		----	
1515		----		----	
1603	in house	1		-0.42	
1623	D1209	4		0.78	
1701	D1209	5		1.18	
1718	D1209	1		-0.42	
1823		----		----	
1866	D1209	1		-0.42	
1880		----		----	
1954		----		----	
1960		----		----	
2124		----		----	
6013	D1209	0		-0.82	
7003		----		----	
7013	D1209	0		-0.82	
9006		----		----	
9007		----		----	
9008		----		----	
9009		----		----	
normality					
n					
outliers					
mean (n)					
st.dev. (n)					
R(calc.)					
R(D1209:05)					



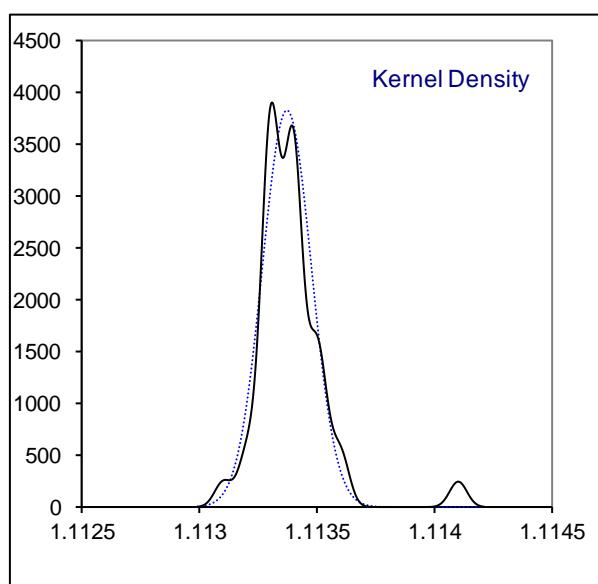
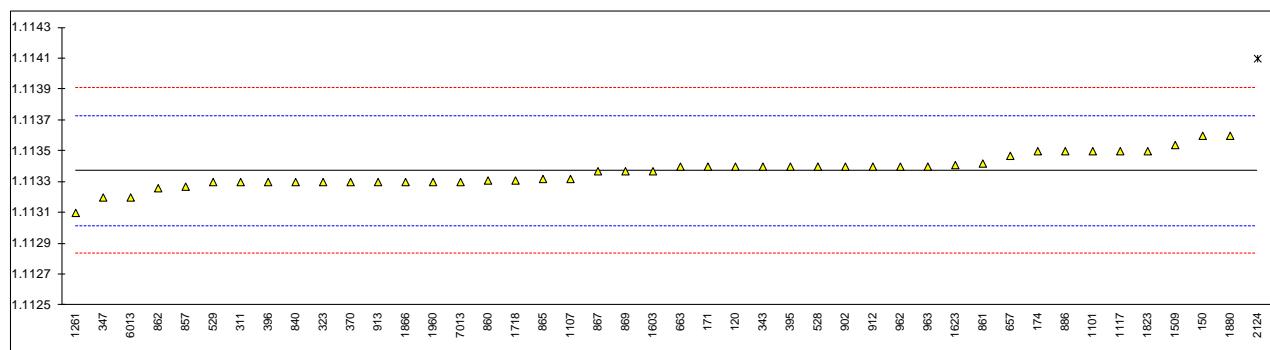
Determination of Colour Pt/Co automated (D5386) on sample #15200;

lab	method	value	mark	z(targ)	remarks
120		----		----	
150	D5386	<1		----	
168	D5386	0		-1.02	
169	D5386	1.4		-0.22	
171	D1209	1		-0.45	
174		----		----	
311	D5386	1		-0.45	
322		----		----	
323	D5386	1		-0.45	
343	D5386	4		1.28	
347	D5386	1		-0.45	
370		----		----	
395	D5386	2		0.13	
396	D5386	2		0.13	
444	D5386	0.9		-0.50	
528		----		----	
529		----		----	
557		----		----	
609		----		----	
610		----		----	
657	D5386	1.62		-0.09	
663	D5386	3		0.70	
825		----		----	
840		----		----	
857	D1209	2		0.13	
860	D5386	1.9		0.07	
861		----		----	
862	D5386	2		0.13	
865	D1209	2		0.13	
867	D5386	3		0.70	
869	D5386	3		0.70	
886	D5386	4		1.28	
902	D5386	2		0.13	
912	D5386	1		-0.45	
913	D5386	2		0.13	
962		----		----	
963		----		----	
1101		----		----	
1107	D5386	0.2		-0.91	
1117		----		----	
1151		----		----	
1217		----		----	
1261		----		----	
1467		----		----	
1509	D5386	4		1.28	
1515		----		----	
1603		----		----	
1623		----		----	
1701		----		----	
1718		----		----	
1823	D5386	1.3		-0.27	
1866		----		----	
1880	D5386	1.40		-0.22	
1954		----		----	
1960	D5386	0.9		-0.50	
2124		----		----	
6013		----		----	
7003		----		----	
7013		----		----	
9006	D5386	1.4		-0.22	
9007	D5386	1.5		-0.16	
9008	D5386	1.15		-0.36	
9009	D5386	1.4		-0.22	
	normality	OK			
	n	31			
	outliers	0			
	mean (n)	1.78			
	st.dev. (n)	1.019			
	R(calc.)	2.85			
	R(D5386:10)	4.87			



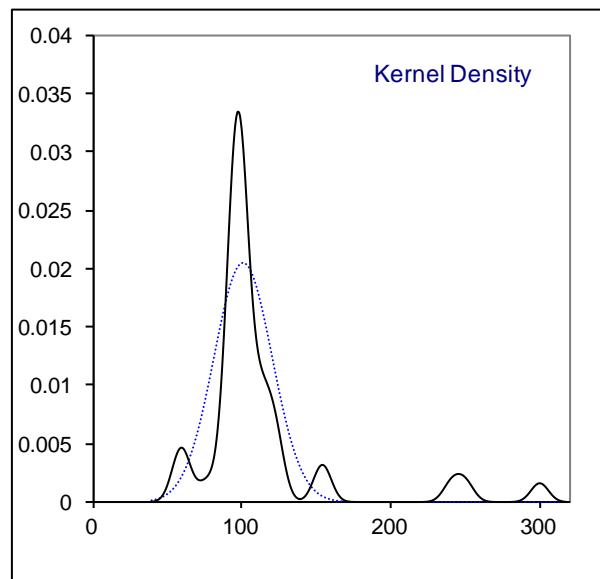
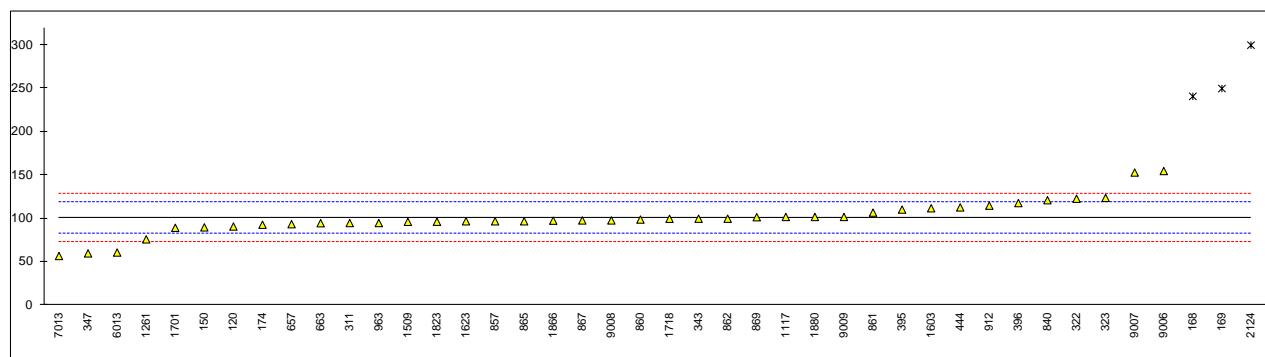
Determination of Density at 20 °C on sample #15200; results in kg/L

lab	method	value	mark	z(targ)	remarks
120	D4052	1.1134		0.17	
150	D4052	1.1136		1.29	
168		----		----	
169		----		----	
171	D4052	1.1134		0.17	
174	D4052	1.1135		0.73	
311	D4052	1.1133		-0.39	
322		----		----	
323	D4052	1.1133		-0.39	
343	D4052	1.1134		0.17	
347	D4052	1.1132		-0.95	
370	D4052	1.1133		-0.39	
395	D4052	1.1134		0.17	
396	D4052	1.1133		-0.39	
444		----		----	
528	D4052	1.1134		0.17	
529	D4052	1.1133		-0.39	
557		----		----	
609		----		----	
610		----		----	
657	D4052	1.11347		0.56	
663	D4052	1.1134		0.17	
825		----		----	
840	D4052	1.1133		-0.39	
857	D4052	1.11327		-0.56	
860	D4052	1.11331		-0.34	
861	D4052	1.11342		0.28	
862	D4052	1.11326		-0.62	
865	D4052	1.11332		-0.28	
867	D4052	1.11337		0.00	
869	D4052	1.11337		0.00	
886	D4052	1.1135		0.73	
902	D4052	1.1134		0.17	
912	D4052	1.1134		0.17	
913	D4052	1.1133		-0.39	
962	D4052	1.1134		0.17	
963	D4052	1.1134		0.17	
1101	ISO12185	1.1135		0.73	
1107	D4052	1.11332		-0.28	
1117	D4052	1.1135		0.73	
1151		----		----	
1217		----		----	
1261	D4052	1.1131		-1.51	
1467		----		----	
1509	D4052	1.11354		0.95	
1515		----		----	
1603	in house	1.11337		0.00	
1623	D4052	1.11341		0.22	
1701		----		----	
1718	D4052	1.11331		-0.34	
1823	D4052	1.1135		0.73	
1866	D4052	1.1133		-0.39	
1880	D4052	1.11360		1.29	
1954		----		----	
1960	D4052	1.1133		-0.39	
2124	D5002	1.1141	R(0.01)	4.09	
6013	ISO12185	1.1132		-0.95	
7003		----		----	
7013	D4052	1.1133		-0.39	
9006		----		----	
9007		----		----	
9008		----		----	
9009		----		----	
normality					
n					
outliers					
mean (n)					
st.dev. (n)					
R(calc.)					
R(ISO12185:96)					



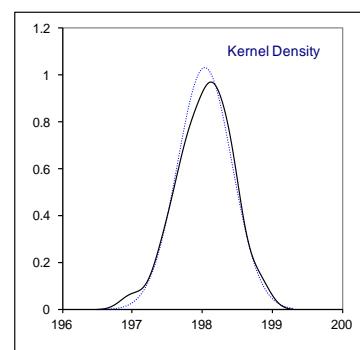
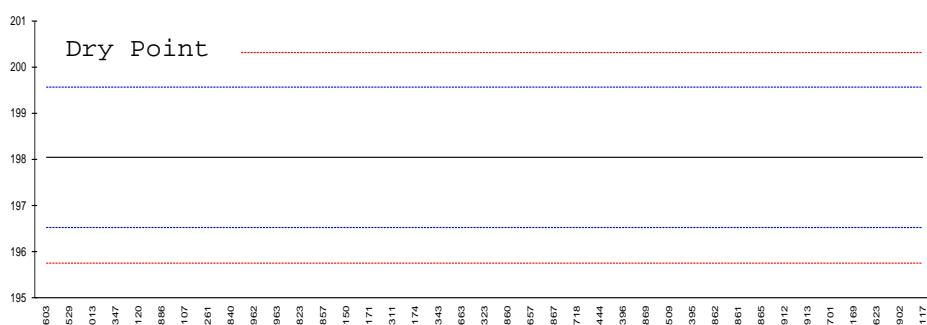
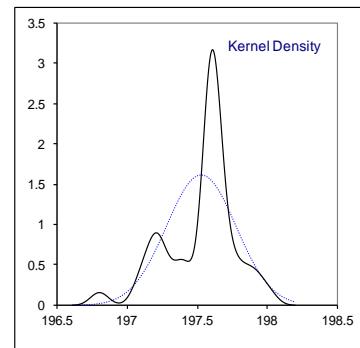
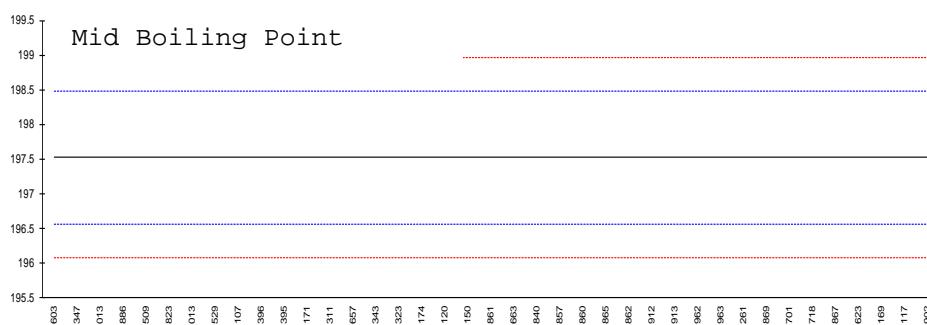
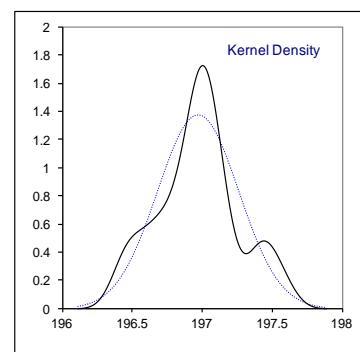
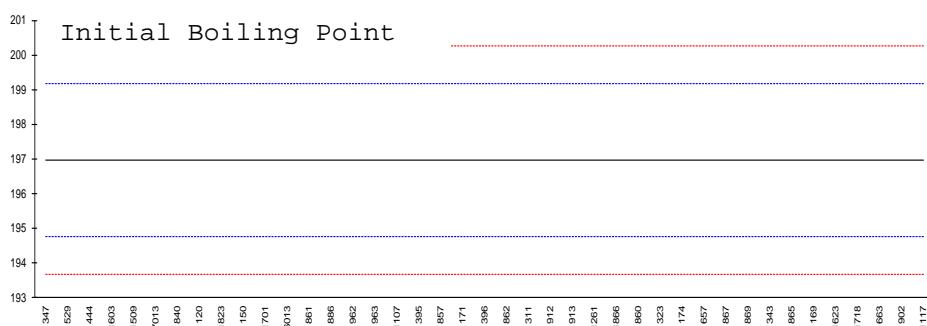
Determination of Diethylene Glycol on sample #15200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	E2409	91	C	-1.07	first reported:9.1
150	E2409	90.0		-1.18	
168	E2409	241	R(0.01)	15.30	probably unit error, reported after correction 0.00000241
169	E2409	250	C,R(0.01)	16.28	first reported:0.025
171		----		----	
174	E2409	93		-0.85	
311	E2409	95		-0.64	
322	E2409	123		2.42	
323	E2409	124		2.53	
343	E2409	100	C	-0.09	first reported:0.01
347	E2409	60	C	-4.46	first reported:0.0060
370		----		----	
395	E2409	110.5		1.06	
396	E2409	118		1.87	
444	E2409	113	C	1.33	first reported:668
528		----		----	
529		----		----	
557		----		----	
609		----		----	
610		----		----	
657	E2409	93.65		-0.78	
663	E2409	94.8	C	-0.66	first reported:55.7
825		----		----	
840	E2409	121.3		2.23	
857	E2409	97		-0.42	
860	E2409	99		-0.20	
861	E2409	107.0		0.67	
862	E2409	100		-0.09	
865	E2409	97		-0.42	
867	E2409	98		-0.31	
869	E2409	101.7		0.10	
886		----		----	
902		----		----	
912	E2409	115		1.55	
913	E2409	<10		<-9.91	false negative test result?
962		----		----	
963	E2409	95		-0.64	
1101	E2409	<100		----	
1107		----		----	
1117	E2409	102		0.13	
1151		----		----	
1217		----		----	
1261	E2409	76.17		-2.69	
1467		----		----	
1509	E2409	96.4		-0.48	
1515		----		----	
1603	in house	112		1.22	
1623	E2409	96.99		-0.42	
1701	E2409	89.5	C	-1.24	first reported:59.5
1718	E2409	99.9		-0.10	
1823	E2409	96.5		-0.47	
1866	E2409	97.72		-0.34	
1880	E2409	102.0		0.13	
1954		----		----	
1960		----		----	
2124	in house	300	R(0.01)	21.74	
6013	in house	61		-4.35	
7003		----		----	
7013	E2409	57	C	-4.78	first reported:154
9006	E2409	154.946		5.91	
9007	E2409	153.204		5.72	
9008	E2409	98		-0.31	
9009	E2409	102	C	0.13	probably unit error, reported 0.0102 mg/kg
	normality	not OK			
	n	39			
	outliers	3			
	mean (n)	100.83			
	st.dev. (n)	19.520			
	R(calc.)	54.66			
	R(E2409:13)	25.65			



Determination of Distillation: IBP, 50% recovered, Dry Point on sample #15200; results in °C

lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)	remarks
120	D1078	196.7		-0.25	197.6		0.16	197.6		-0.58	
150	D1078	196.8		-0.16	197.6		0.16	197.9		-0.18	
168	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
169	D1078	197.4		0.39	197.9		0.78	198.5		0.61	
171	D1078	197.0		0.03	197.4		-0.26	197.9		-0.18	
174	D1078	197.1		0.12	197.6		0.16	198.0		-0.05	
311	D1078	197.0		0.03	197.6		0.16	197.9		-0.18	
322	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
323	D1078	197.1		0.12	197.6		0.16	198.1		0.08	
343	D1078	197.2		0.21	197.6		0.16	198.1		0.08	
347	D1078	196.4		-0.52	197.1		-0.88	197.5		-0.71	
370	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
395	D1078	197.0		0.03	197.4		-0.26	198.3		0.35	
396	D1078	197.0		0.03	197.4		-0.26	198.2		0.21	
444	D1078	196.5		-0.43	-----		-----	198.2		0.21	
528	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
529	D1078	196.45		-0.48	197.25		-0.57	197.4		-0.84	
557	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
609	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
610	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
657	D1078	197.1		0.12	197.6		0.16	198.1		0.08	
663	D1078	197.5		0.48	197.6		0.16	198.1		0.08	
825	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
840	D1078	196.68		-0.27	197.60		0.16	197.77		-0.35	
857	D1078	197.0		0.03	197.6		0.16	197.9		-0.18	
860	D1078	197.1		0.12	197.6		0.16	198.1		0.08	
861	D1078	196.9		-0.07	197.6		0.16	198.4		0.48	
862	D1078	197.0		0.03	197.6		0.16	198.4		0.48	
865	D1078	197.2		0.21	197.6		0.16	198.4		0.48	
867	D1078	197.1		0.12	197.8		0.57	198.1		0.08	
869	D1078	197.1		0.12	197.7		0.36	198.2		0.21	
886	D1078	196.9		-0.07	197.2		-0.67	197.6		-0.58	
902	D1078	197.5		0.48	198.0		0.99	198.8		1.01	
912	D1078	197.0		0.03	197.6		0.16	198.4		0.48	
913	D1078	197.0		0.03	197.6		0.16	198.4		0.48	
962	D1078	196.9		-0.07	197.6		0.16	197.8		-0.31	
963	D1078	196.9		-0.07	197.6		0.16	197.8		-0.31	
1101	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
1107	D1078	196.9		-0.07	197.3		-0.47	197.7		-0.45	
1117	D1078	197.6		0.57	197.9		0.78	198.8		1.01	
1151	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
1217	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
1261	D1078	197		0.03	197.6		0.16	197.7		-0.45	
1467	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
1509	D1078	196.6		-0.34	197.2		-0.67	198.2		0.21	
1515	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
1603	in house	196.5		-0.43	196.8		-1.50	197.0		-1.37	
1623	D1078	197.4		0.39	197.8		0.57	198.5		0.61	
1701	D1078	196.8		-0.16	197.7		0.36	198.4		0.48	
1718	D1078	197.4		0.39	197.7		0.36	198.1		0.08	
1823	D1078	196.7		-0.25	197.2		-0.67	197.8		-0.31	
1866	D1078	197		0.03	-----		-----	-----	-----	-----	
1880	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
1954	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
1960	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
2124	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
6013	D1078	196.8		-0.16	197.1		-0.88	197.4		-0.84	
7003	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
7013	D1078	196.6		-0.34	197.2		-0.67	-----		-----	
9006	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
9007	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
9008	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
9009	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	
normality	OK				OK			OK			
n	41				39			39			
outliers	0				0			0			
mean (n)	196.97				197.52			198.04			
st.dev. (n)	0.291				0.248			0.387			
R(calc.)	0.81				0.69			1.08			
R(D1078:11)	3.07				1.35			2.12			

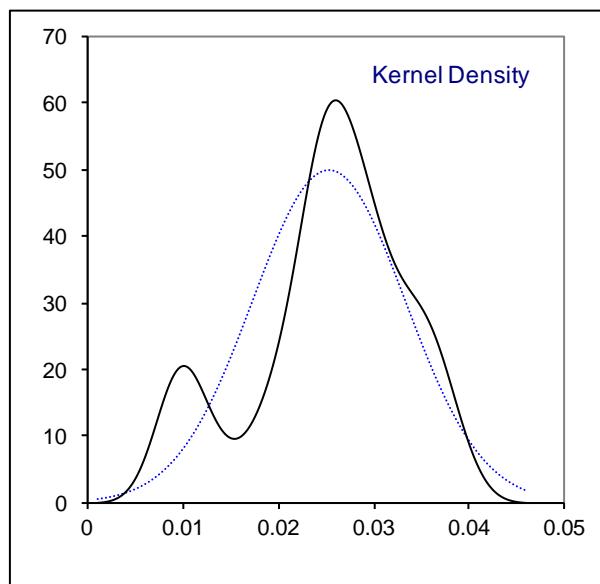
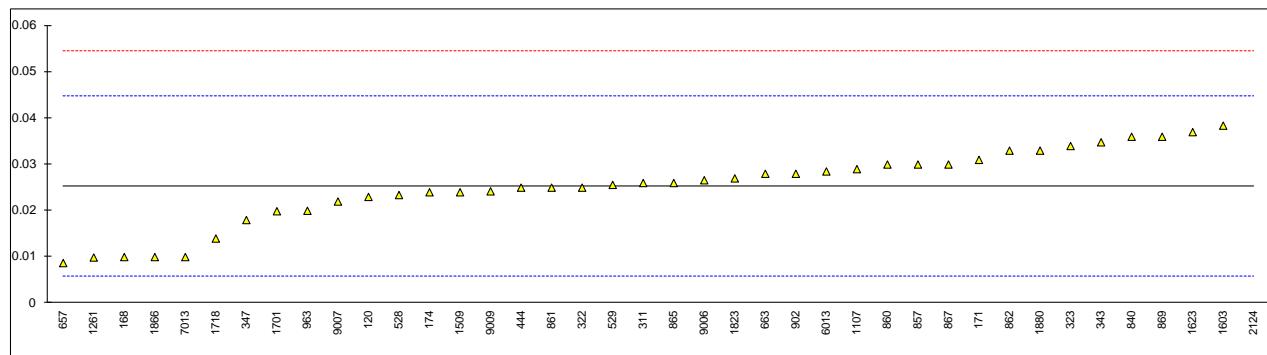


Several laboratories did not correct for theoretical mid boiling point (197.6). Results after manual correction:

lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)	remarks
169	D1078	197.1		0.05	197.6		0.01	198.2		0.50	
171	D1078	197.2		0.14	197.6		0.01	198.1		0.00	
347	D1078	196.9		-0.14	197.6		0.01	198.2		0.11	
395	D1078	197.2		0.14	197.6		0.01	198.5		0.50	
396	D1078	197.2		0.14	197.6		0.01	198.4		0.37	
529	D1078	196.8		-0.23	197.6		0.01	197.75		-0.49	
867	D1078	196.9		-0.14	197.6		0.01	197.9		-0.29	
886	D1078	197.3		0.23	197.6		0.01	198.0		-0.16	
902	D1078	197.1		0.05	197.6		0.01	198.4		0.37	
1107	D1078	197.2		0.14	197.6		0.01	198.0		-0.16	
1117	D1078	197.3		0.23	197.6		0.01	197.5		-0.82	
1509	D1078	197.0		-0.05	197.6		0.01	198.2		0.11	
1603	in house	197.3		0.23	197.6		0.01	197.8		-0.42	
1623	D1078	197.2		0.14	197.6		0.01	198.3		0.24	
1823	D1078	197.1		0.05	197.6		0.01	198.2		0.50	
6013	D1078	197.3		0.23	197.6		0.01	197.9		-0.29	
7013	D1078	197.0		-0.05	197.6		0.01	----		----	
		OK			not OK			OK			
		41			39			39			
		0			0			0			
		197.05			197.60			198.12			
		0.202			0.071			0.257			
		0.57			0.20			0.72			
		3.07			1.35			2.12			

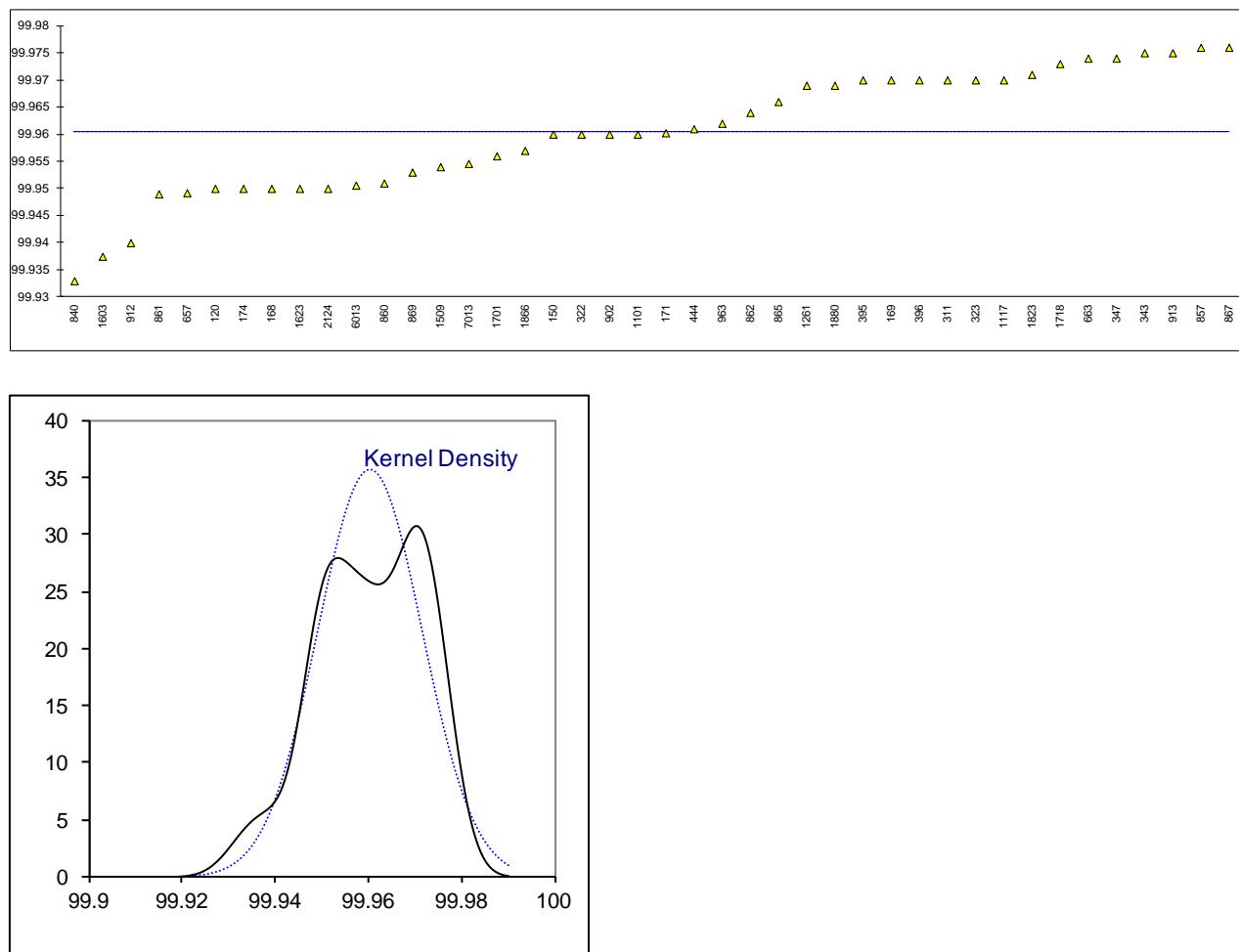
Determination of Iron as Fe on sample #15200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	E1615	0.023		-0.23	
150	E394	<0.1		-----	
168	E1615	0.01	C	-1.56	first reported:0.008
169		-----		-----	
171	E1615	0.031		0.59	
174	E1615	0.024		-0.13	
311	E1615	0.026		0.08	
322	E1615	0.025		-0.02	
323	E1615	0.034		0.90	
343	E1615	0.0348	C	0.98	first reported:0.0524
347	E394	0.018		-0.74	
370		-----		-----	
395	E394	<0.01		-----	
396	E1615	<0.01		-----	
444	E1615	0.025		-0.02	
528	E1615	0.0234		-0.19	
529	E1615	0.0256		0.04	
557		-----		-----	
609		-----		-----	
610		-----		-----	
657	E1615	0.0087		-1.69	
663	E394	0.028		0.28	
825		-----		-----	
840	E394	0.036		1.10	
857	E1615	0.030		0.49	
860	E394	0.030		0.49	
861	E394	0.025		-0.02	
862	E1615	0.033		0.80	
865	E394	0.026		0.08	
867	E1615	0.030		0.49	
869	E394	0.036		1.10	
886		-----		-----	
902	E1615	0.028		0.28	
912		-----		-----	
913	E1615	<0.01		-----	
962		-----		-----	
963	E394	0.02		-0.54	
1101		-----		-----	
1107	E1615	0.0290		0.39	
1117	E394	<0.01		-----	
1151		-----		-----	
1217		-----		-----	
1261	E394	0.0099		-1.57	
1467		-----		-----	
1509	E394	0.024		-0.13	
1515		-----		-----	
1603	in house	0.0384		1.35	
1623	E202	0.037		1.20	
1701	E394	0.0199		-0.55	
1718	E394	0.014		-1.15	
1823	E394	0.027		0.18	
1866	E1615	0.01		-1.56	
1880	E1615	0.033		0.80	
1954		-----		-----	
1960		-----		-----	
2124	ISO11885	0.1	R(0.01)	7.65	
6013	E1615	0.0285		0.33	
7003		-----		-----	
7013	E394	0.01		-1.56	
9006	E1615	0.0266		0.14	
9007	E1615	0.022		-0.33	
9008		-----		-----	
9009	E1615	0.02420		-0.11	
normality					
n					
outliers					
mean (n)					
st.dev. (n)					
R(calc.)					
R(E1615:08)					
Spike					
<84% rec					



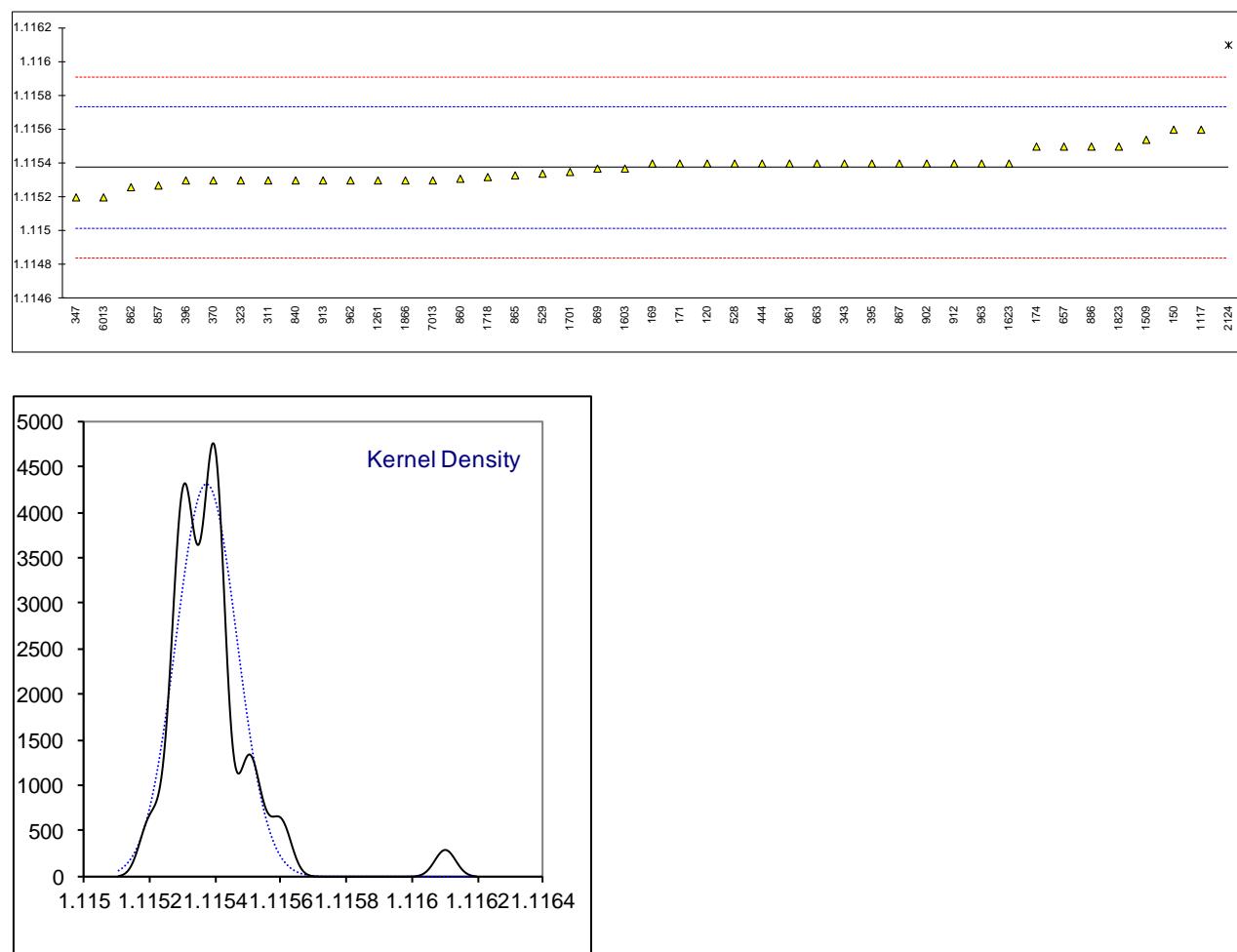
Determination of Purity as received on sample #15200; results in %M/M

lab	method	value	mark	z(targ)	remarks	
120	E2409	99.950		----		
150	E2409	99.96		----		
168	E2409	99.95		----		
169	E2409	99.97		----		
171		99.960251		----		
174	E2409	99.950		----		
311	E2409	99.97		----		
322	E2409	99.96		----		
323	E2409	99.97		----		
343	E2409	99.975		----		
347	E2409	99.974		----		
370		----		----		
395	E2409	99.97		----		
396	E2409	99.97		----		
444	E2409	99.961	C	----	first reported:99.906	
528		----		----		
529		----		----		
557		----		----		
609		----		----		
610		----		----		
657	E2409	99.9492		----		
663	E2409	99.974		----		
825		----		----		
840	E2409	99.933		----		
857	E2409	99.976		----		
860	E2409	99.951		----		
861	E2409	99.949		----		
862	E202	99.964		----		
865	E2409	99.966		----		
867	E2409	99.976		----		
869	E2409	99.953		----		
886		----		----		
902	E2409	99.96		----		
912	E2409	99.94		----		
913	E2409	99.975		----		
962		----		----		
963	E2409	99.962		----		
1101	E2409	99.96		----		
1107		----		----		
1117	E2409	99.97		----		
1151		----		----		
1217		----		----		
1261	E202	99.969		----		
1467		----		----		
1509	E2409	99.954		----		
1515		----		----		
1603	in house	99.9375		----		
1623	E2409	99.95		----		
1701	E2409	99.956		----		
1718	E2409	99.973		----		
1823	E2409	99.9710		----		
1866	E2409	99.957		----		
1880		99.969		----		
1954		----		----		
1960		----		----		
2124	in house	99.95		----		
6013	in house	99.9506		----		
7003		----		----		
7013	E2409	99.9546		----		
9006		----		----		
9007		----		----		
9008		----		----		
9009		----		----		
normality		OK				
n		42				
outliers		0				
mean (n)		99.9605				
st.dev. (n)		0.01119				
R(calc.)		0.0313				
R(lit)		unknown				
compare R(iis14C09) = 0.0415						



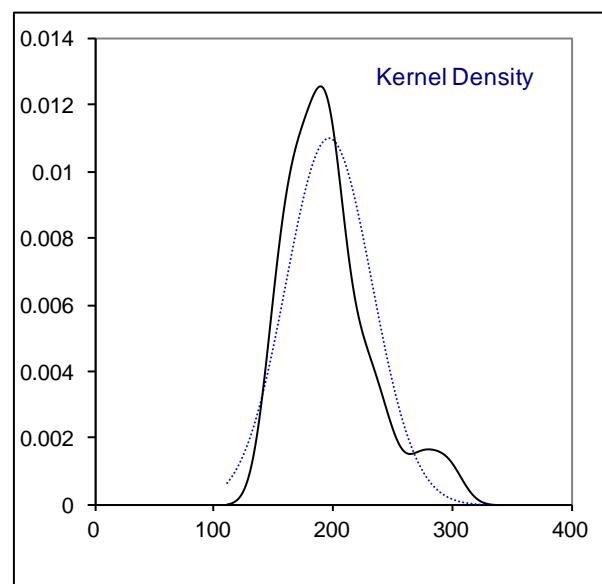
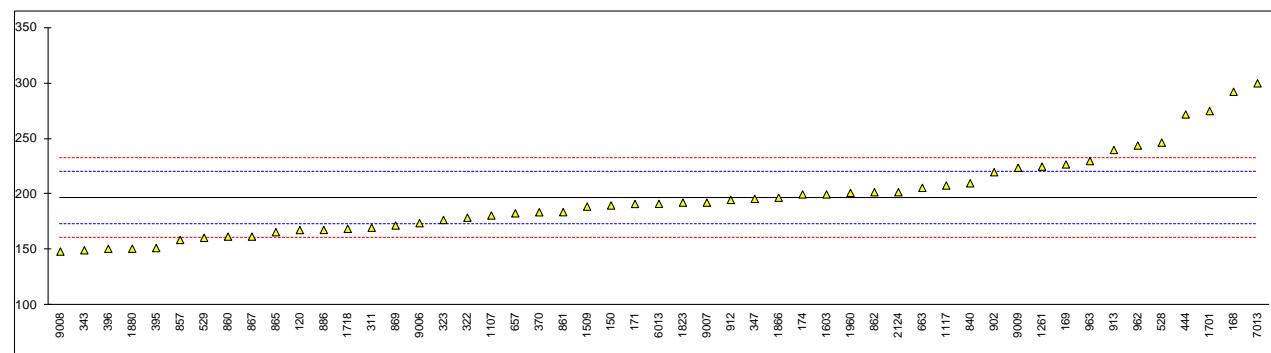
Determination of Specific Gravity 20/20°C on sample #15200;

lab	method	value	mark	z(targ)	remarks
120	D4052	1.1154		0.15	
150	D4052	1.1156		1.27	
168		----		----	
169	D4052	1.1154		0.15	
171	D4052	1.1154	C	0.15	first reported:99.960251
174	D4052	1.1155		0.71	
311	D4052	1.1153		-0.41	
322		----		----	
323	D4052	1.1153		-0.41	
343	D4052	1.1154		0.15	
347	D4052	1.1152		-0.97	
370	E202	1.1153		-0.41	
395	D4052	1.1154		0.15	
396	E202	1.1153		-0.41	
444	D4052	1.1154		0.15	
528	D4052	1.1154		0.15	
529	D4052	1.11534		-0.18	
557		----		----	
609		----		----	
610		----		----	
657	D4052	1.1155		0.71	
663	D4052	1.1154		0.15	
825		----		----	
840	D4052	1.1153		-0.41	
857	D4052	1.11527		-0.58	
860	D4052	1.11531		-0.35	
861	D4052	1.1154		0.15	
862	D4052	1.11526		-0.63	
865	D4052	1.11533		-0.24	
867	D4052	1.1154		0.15	
869	E202	1.11537		-0.02	
886	D4052	1.1155		0.71	
902	D4052	1.1154		0.15	
912	D4052	1.1154		0.15	
913	D4052	1.1153		-0.41	
962	D4052	1.1153		-0.41	
963	D4052	1.1154		0.15	
1101		----		----	
1107		----		----	
1117	D4052	1.1156		1.27	
1151		----		----	
1217		----		----	
1261	E202	1.1153		-0.41	
1467		----		----	
1509	D4052	1.11554		0.94	
1515		----		----	
1603	in house	1.11537		-0.02	
1623	E202	1.1154		0.15	
1701	D4052	1.11535		-0.13	
1718	D4052	1.11532		-0.30	
1823	D4052	1.1155		0.71	
1866	D4052	1.1153		-0.41	
1880		----		----	
1954		----		----	
1960		----		----	
2124		1.1161	R(0.01)	4.07	
6013	D4052	1.1152		-0.97	
7003		----		----	
7013	D4052	1.1153		-0.41	
9006		----		----	
9007		----		----	
9008		----		----	
9009		----		----	
normality					
n					
outliers					
mean (n)					
st.dev. (n)					
R(calc.)					
R(E202:12)					



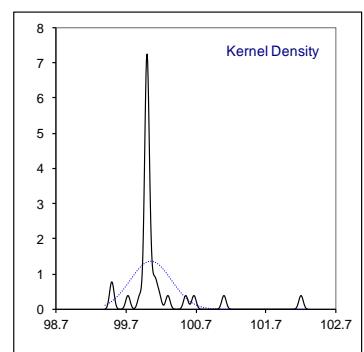
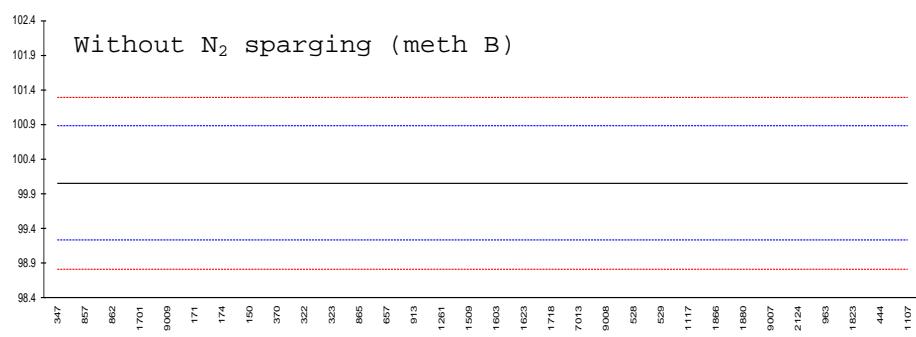
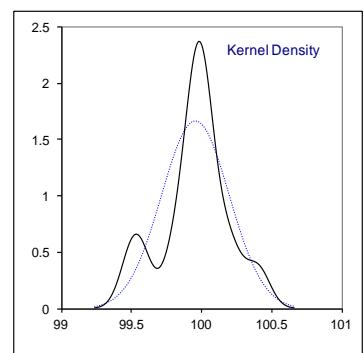
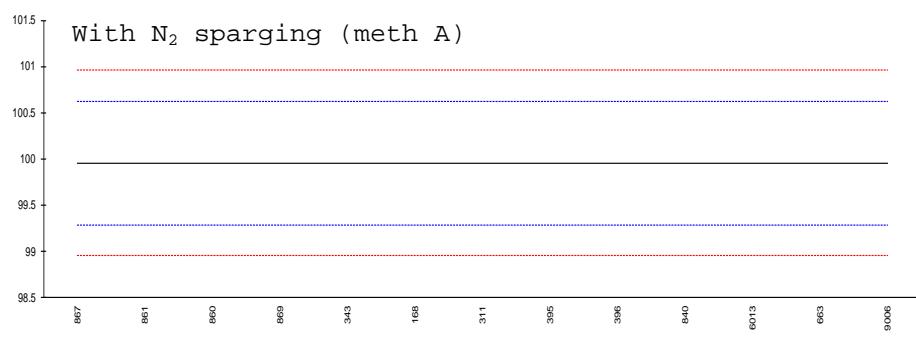
Determination of Water, coulometric KF titration on sample #15200; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	E1064	167.9		-2.40	
150	E1064	190		-0.56	
168	E1064	292.4		7.97	
169	E1064	227		2.52	
171	E1064	191.4		-0.44	
174	E1064	200		0.28	
311	E1064	170		-2.22	
322	E1064	179		-1.47	
323	E1064	177		-1.64	
343	E1064	149.7		-3.91	
347	E1064	196		-0.06	
370	E1064	183.9		-1.06	
395	E1064	151.68		-3.75	
396	E1064	151		-3.80	
444	E1064	272		6.27	
528	E1064	246.6		4.16	
529	E1064	160.87		-2.98	
557		----		----	
609		----		----	
610		----		----	
657	E1064	182.95		-1.14	
663	E1064	205.9		0.77	
825		----		----	
840	E1064	210		1.11	
857	E1064	159		-3.14	
860	E1064	162		-2.89	
861	E1064	184		-1.06	
862	E1064	202		0.44	
865	E1064	166		-2.55	
867	E1064	162		-2.89	
869	E1064	172		-2.06	
886	E1064	168		-2.39	
902	E1064	220		1.94	
912	E1064	195		-0.14	
913	E1064	240		3.61	
962	E1064	244		3.94	
963	E1064	230		2.77	
1101		----		----	
1107	E1064	181	C	-1.31	first reported:0.0181
1117	E1064	208		0.94	
1151		----		----	
1217		----		----	
1261	E1064	225		2.36	
1467		----		----	
1509	E1064	189		-0.64	
1515		----		----	
1603	in house	200		0.28	
1623		----		----	
1701	E203	275	C	6.52	first reported:255
1718	E1064	169.0		-2.30	
1823	E1064	192.6		-0.34	
1866	E1064	197		0.03	
1880	E1064	151.1		-3.80	
1954		----		----	
1960	D4928	201.33		0.39	
2124	in house	202		0.44	
6013	E1064	191.5		-0.43	
7003		----		----	
7013	E1064	300		8.60	
9006	E1064	174.1		-1.88	
9007	E1064	192.6		-0.34	
9008	E1064	148.5		-4.01	
9009	E1064	224	C	2.27	probably unit error, reported:0.0224
	normality	not OK			
	n	51			
	outliers	0			
	mean (n)	196.69			
	st.dev. (n)	36.203			
	R(calc.)	101.37			
	R(E1064:12)	33.63			



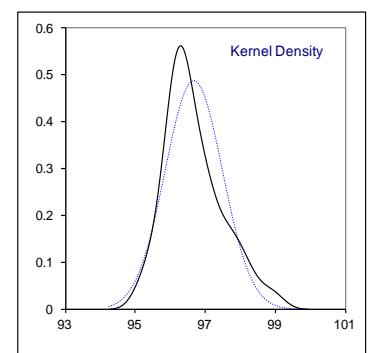
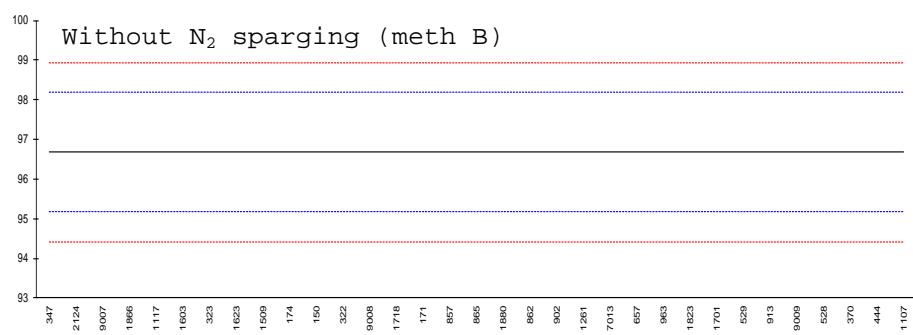
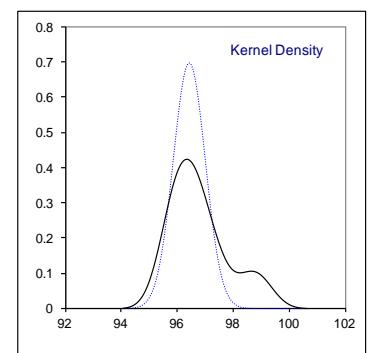
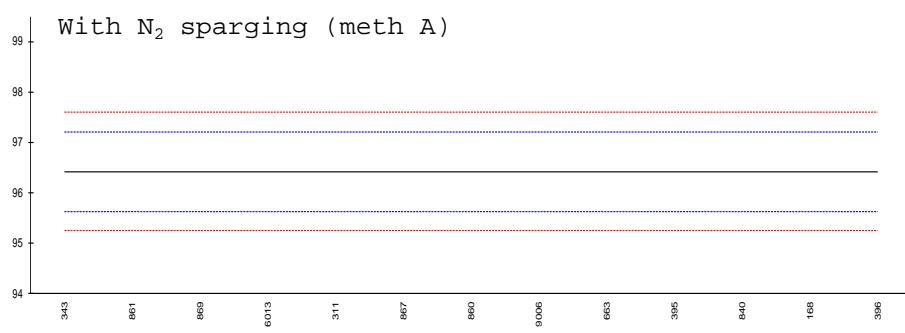
Determination of UV Transmittance at 350 nm on sample #15201; results in %Transmittance

lab	Method	Method A	mark	z(targ)	Method B	mark	z(targ)	remarks
120		----		----	----		----	
150	E2193-B	----		----	100.00		-0.14	
168	E2193-A	100.0		0.13	----		----	
169		----		----	----		----	
171	E2193-B	----		----	99.9999		-0.14	
174	E2193-B	----		----	100.00		-0.14	
311	E2193-A	100		0.13	----		----	
322	E2193-B	----		----	100		-0.14	
323	E2193-B	----		----	100.0		-0.14	
343	E2193-A	99.92		-0.11	----		----	
347	E2193-B	----		----	99.5		-1.35	
370	E2193-B	----		----	100.0		-0.14	
395	E2193-A	100		0.13	----		----	
396	E2193-A	100.0		0.13	----		----	
444	E2193-B	----		----	101.1	C	2.53	first reported:101.45
528	E2193-B	----		----	100.015		-0.10	
529	E2193-B	----		----	100.044	C	-0.03	first reported:71.911
557		----		----	----		----	
609		----		----	----		----	
610		----		----	----		----	
657	E2193-B	----		----	100.00		-0.14	
663	E2193-A	100.22		0.79	----		----	
825		----		----	----		----	
840	E2193-A	100.00		0.13	----		----	
857	E2193-B	----		----	99.5		-1.35	
860	E2193-A	99.8		-0.47	----		----	
861	E2193-A	99.57		-1.16	----		----	
862	E2193-B	----		----	99.73		-0.79	
865	E2193-B	----		----	100.0		-0.14	
867	E2193-A	99.5		-1.37	----		----	
869	E2193-A	99.9		-0.17	----		----	
886		----		----	----		----	
902	E2193-B	----		----	>100.0		----	
912		----		----	----		----	
913	E2193-B	----		----	100.00		-0.14	
962		----		----	----		----	
963	E2193-B	----		----	100.555		1.21	
1101		----		----	----		----	
1107	E2193-B	----		----	102.2	G(0.01)	5.20	
1117	E2193-B	----		----	100.05		-0.02	
1151		----		----	----		----	
1217		----		----	----		----	
1261	INH-577A	----		----	100		-0.14	
1467		----		----	----		----	
1509	E2193-B	----		----	100.00		-0.14	
1515		----		----	----		----	
1603	in house	----		----	100		-0.14	
1623	E2193-B	----		----	100.0		-0.14	
1701	E2193-B	----		----	99.9		-0.38	
1718	E2193-B	----		----	100.00		-0.14	
1823	E2193-B	----		----	100.672	C	1.49	first reported:100.039
1866	E2193-B	----		----	100.11		0.13	
1880	E2193-B	----		----	100.12		0.15	
1954		----		----	----		----	
1960		----		----	----		----	
2124	E2193-B	----		----	100.3		0.59	
6013	E2193-A	100.129		0.51	----		----	
7003		----		----	----		----	
7013	E2193-B	----		----	100.0		-0.14	
9006	E2193-A	100.401		1.33	----		----	
9007	E2193	----		----	100.176		0.29	
9008	E2193	----		----	100		-0.14	
9009	E2193-B	----		----	99.992		-0.16	
normality		OK		not OK				
n		13		31				
outliers		0		1				
mean (n)		99.957		100.057				
st.dev. (n)		0.2403		0.2946				
R(calc.)		0.673		0.825				
R(E2193:08)		0.936		1.154				



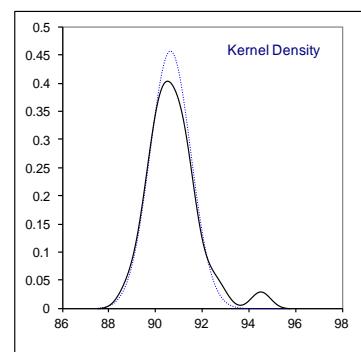
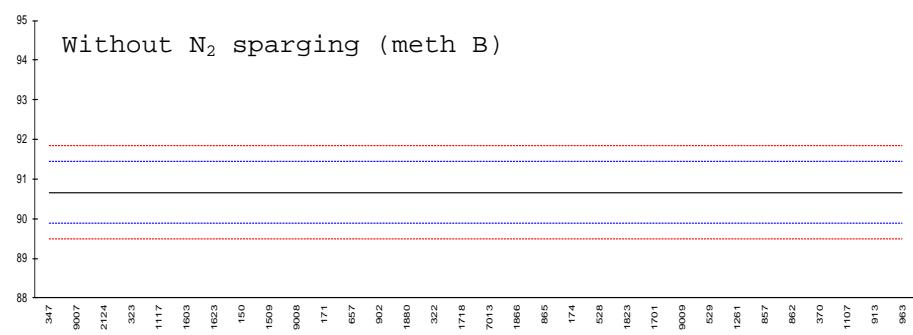
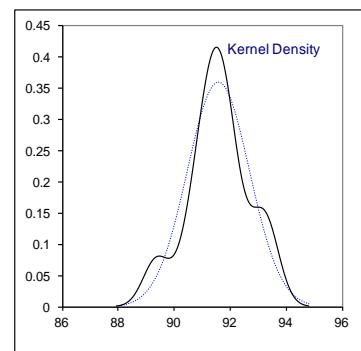
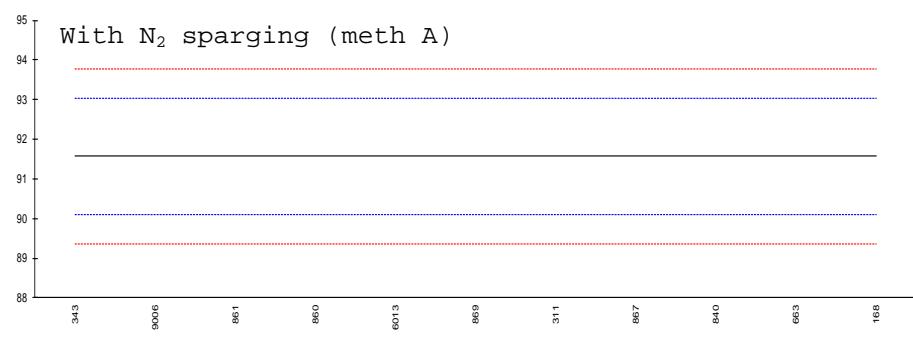
Determination of UV Transmittance at 275 nm on sample #15201; results in %Transmittance

lab	Method	Method A	mark	z(targ)	Method B	mark	z(targ)	remarks
120		----		----	----		----	
150	E2193-B	----		----	96.26		-0.55	
168	E2193-A	98.498	C,DG(0.05)	5.29	----		----	first reported:98.558
169		----		----	----		----	
171	E2193-B	----		----	96.3897		-0.38	
174	E2193-B	----		----	96.20		-0.63	
311	E2193-A	96.3		-0.31	----		----	
322	E2193-B	----		----	96.3		-0.50	
323	E2193-B	----		----	96.1		-0.77	
343	E2193-A	95.69		-1.87	----		----	
347	E2193-B	----		----	95.3		-1.83	
370	E2193-B	----		----	98.1		1.89	
395	E2193-A	97.2		1.98	----		----	
396	E2193-A	99.0	C,DG(0.05)	6.57	----		----	first reported: 97.4
444	E2193-B	----		----	98.25	C	2.09	first reported: 99.05
528	E2193-B	----		----	97.720		1.39	
529	E2193-B	----		----	97.253	C	0.77	first reported:91.298
557		----		----	----		----	
609		----		----	----		----	
610		----		----	----		----	
657	E2193-B	----		96.83			0.20	
663	E2193-A	96.77		0.89	----		----	
825		----		----	----		----	
840	E2193-A	97.42		2.54	----		----	
857	E2193-B	----		----	96.4		-0.37	
860	E2193-A	96.5		0.20	----		----	
861	E2193-A	95.76		-1.69	----		----	
862	E2193-B	----		----	96.42		-0.34	
865	E2193-B	----		----	96.4		-0.37	
867	E2193-A	96.4		-0.06	----		----	
869	E2193-A	95.8		-1.59	----		----	
886		----		----	----		----	
902	E2193-B	----		96.8			0.16	
912		----		----	----		----	
913	E2193-B	----		97.68			1.34	
962		----		----	----		----	
963	E2193-B	----		97.026			0.47	
1101		----		----	----		----	
1107	E2193-B	----		98.9			2.96	
1117	E2193-B	----		95.95			-0.97	
1151		----		----	----		----	
1217		----		----	----		----	
1261	INH-577A	----		96.8			0.16	
1467		----		----	----		----	
1509	E2193-B	----		96.11			-0.75	
1515		----		----	----		----	
1603	in house	----		96			-0.90	
1623	E2193-B	----		96.1			-0.77	
1701	E2193-B	----		97.15			0.63	
1718	E2193-B	----		96.38			-0.39	
1823	E2193-B	----		97.142	C	0.62	first reported:94.957	
1866	E2193-B	----		95.88			-1.06	
1880	E2193-B	----		96.40			-0.37	
1954		----		----	----		----	
1960		----		----	----		----	
2124	E2193-B	----		95.4			-1.70	
6013	E2193-A	96.116		-0.78	----		----	
7003		----		----	----		----	
7013	E2193-B	----		96.8			0.16	
9006	E2193-A	96.686		0.67	----		----	
9007	E2193	----		95.842			-1.11	
9008	E2193	----		96.34			-0.45	
9009	E2193-B	----		97.693			1.35	
normality		OK		OK				
n		11		33				
outliers		2		0				
mean (n)		96.422		96.676				
st.dev. (n)		0.5717		0.8194				
R(calc.)		1.601		2.294				
R(E2193:08)		1.098		2.105				



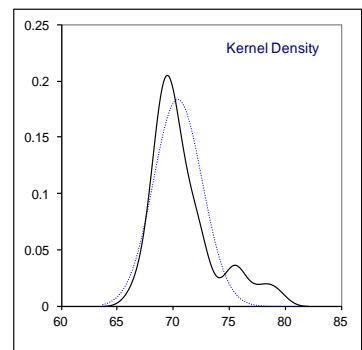
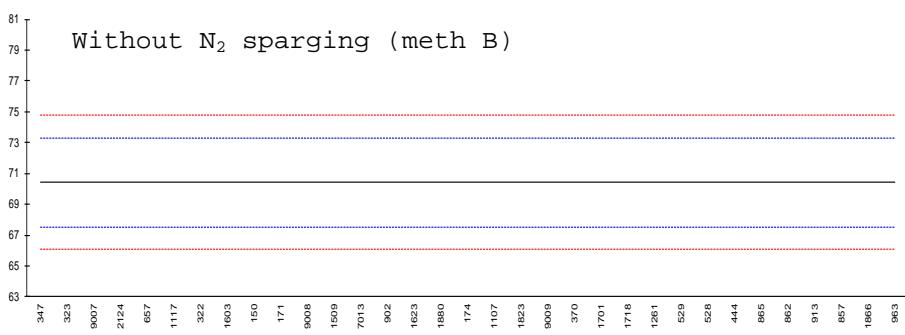
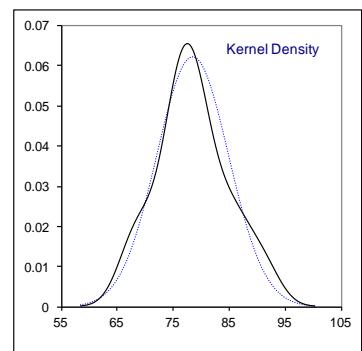
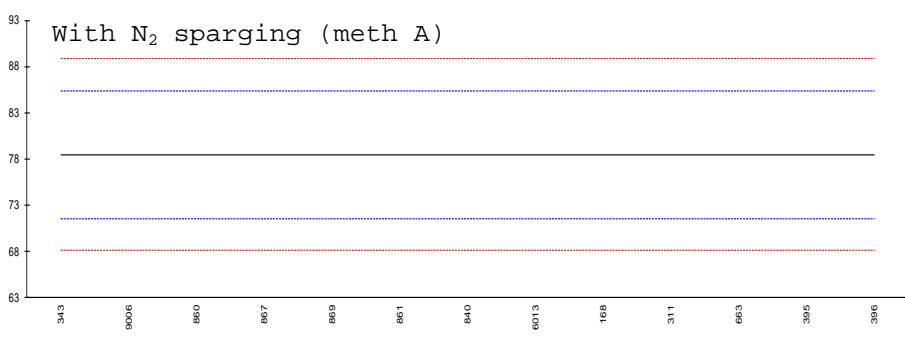
Determination of UV Transmittance at 250 nm on sample #15201; results in %Transmittance

lab	Method	Method A	mark	z(targ)	Method B	mark	z(targ)	remarks
120		----		----	----		----	first reported:93.446
150	E2193-B	----		----	90.04		-1.58	
168	E2193-A	93.3655	C	2.44	----		----	
169		----		----	----		----	
171	E2193-B	----		----	90.3444		-0.81	
174	E2193-B	----		----	90.95		0.73	
311	E2193-A	91.8		0.32	----		----	
322	E2193-B	----		----	90.5		-0.41	
323	E2193-B	----		----	89.7		-2.45	
343	E2193-A	89.36		-3.00	----		----	
347	E2193-B	----		----	88.8		-4.73	
370	E2193-B	----		----	91.8		2.89	
395	E2193-A	----		----	----		----	
396	E2193-A	----		----	----		----	
444	E2193-B	----		----	----		----	
528	E2193-B	----		----	91.067		1.03	
529	E2193-B	----		----	91.298	C	1.61	first reported:97.253
557		----		----	----		----	
609		----		----	----		----	
610		----		----	----		----	
657	E2193-B	----		----	90.36		-0.77	
663	E2193-A	93.05		2.01	----		----	
825		----		----	----		----	
840	E2193-A	92.21	C	0.87	----		----	first reported:94.76
857	E2193-B	----		----	91.6		2.38	
860	E2193-A	91.1		-0.63	----		----	
861	E2193-A	91.06		-0.69	----		----	
862	E2193-B	----		----	91.67		2.56	
865	E2193-B	----		----	90.8		0.35	
867	E2193-A	91.8		0.32	----		----	
869	E2193-A	91.5		-0.09	----		----	
886		----		----	----		----	
902	E2193-B	----		----	90.4		-0.67	
912		----		----	----		----	
913	E2193-B	----		----	92.77	C	5.35	first reported:94.77
962		----		----	----		----	
963	E2193-B	----		----	94.541	C,G(0.01)	9.85	first reported:93.541
1101		----		----	----		----	
1107	E2193-B	----		----	92.3		4.16	
1117	E2193-B	----		----	89.70		-2.45	
1151		----		----	----		----	
1217		----		----	----		----	
1261	INH-577A	----		----	91.3		1.62	
1467		----		----	----		----	
1509	E2193-B	----		----	90.04		-1.58	
1515		----		----	----		----	
1603	in house	----		----	90		-1.68	
1623	E2193-B	----		----	90.0		-1.68	
1701	E2193-B	----		----	91.20		1.36	
1718	E2193-B	----		----	90.61		-0.13	
1823	E2193-B	----		----	91.162	C	1.27	first reported:88.866
1866	E2193-B	----		----	90.77		0.27	
1880	E2193-B	----		----	90.48		-0.46	
1954		----		----	----		----	
1960		----		----	----		----	
2124	E2193-B	----		----	89.5		-2.95	
6013	E2193-A	91.455		-0.15	----		----	
7003		----		----	----		----	
7013	E2193-B	----		----	90.7		0.09	
9006	E2193-A	90.539		-1.40	----		----	
9007	E2193	----		----	89.334		-3.38	
9008	E2193	----		----	90.13		-1.35	
9009	E2193-B	----		----	91.226		1.43	
normality		OK			OK			
n		11			31			
outliers		0			1			
mean (n)		91.567			90.663			
st.dev. (n)		1.1125			0.8747			
R(calc.)		3.115			2.449			
R(E2193:08)		2.063			1.102			



Determination of UV Transmittance at 220 nm on sample #15201; results in %Transmittance

lab	Method	Method A	mark	z(targ)	Method B	mark	z(targ)	remarks
120		----		----	----		----	
150	E2193-B	----		----	69.04		-0.96	
168	E2193-A	80.230		0.51	----		----	
169		----		----	----		----	
171	E2193-B	----		----	69.2122		-0.84	
174	E2193-B	----		----	70.07		-0.24	
311	E2193-A	80.7		0.64	----		----	
322	E2193-B	----		----	69.0		-0.98	
323	E2193-B	----		----	67.9		-1.75	
343	E2193-A	67.72		-3.11	----		----	
347	E2193-B	----		----	66.7		-2.58	
370	E2193-B	----		----	70.6		0.12	
395	E2193-A	86.5	C	2.32	----		----	first reported:84.5
396	E2193-A	91.0	C	3.62	----		----	first reported:84.3
444	E2193-B	----		----	72.4	C	1.37	first reported:80.15
528	E2193-B	----		----	72.034		1.11	
529	E2193-B	----		----	71.911	C	1.03	first reported:100.044
557		----		----	----		----	
609		----		----	----		----	
610		----		----	----		----	
657	E2193-B	----		----	68.87		-1.07	
663	E2193-A	84.71		1.80	----		----	
825		----		----	----		----	
840	E2193-A	78.12	C	-0.10	----		----	first reported:86.44
857	E2193-B	----		----	75.7		3.65	
860	E2193-A	74.8		-1.06	----		----	
861	E2193-A	77.04		-0.41	----		----	
862	E2193-B	----		----	75.11		3.24	
865	E2193-B	----		----	72.8		1.64	
867	E2193-A	75.2		-0.95	----		----	
869	E2193-A	75.8		-0.77	----		----	
886		----		----	----		----	
902	E2193-B	----		----	69.5		-0.64	
912		----		----	----		----	
913	E2193-B	----		----	75.65		3.62	
962		----		----	----		----	
963	E2193-B	----		----	79.131	DG(0.05)	6.02	
1101		----		----	----		----	
1107	E2193-B	----		----	70.2		-0.15	
1117	E2193-B	----		----	68.99		-0.99	
1151		----		----	----		----	
1217		----		----	----		----	
1261	INH-577A	----		----	71.9		1.02	
1467		----		----	----		----	
1509	E2193-B	----		----	69.32		-0.76	
1515		----		----	----		----	
1603	in house	----		----	69		-0.98	
1623	E2193-B	----		----	69.6		-0.57	
1701	E2193-B	----		----	70.78		0.25	
1718	E2193-B	----		----	71.15		0.50	
1823	E2193-B	----		----	70.222	C	-0.14	first reported:65.090
1866	E2193-B	----		----	77.82	DG(0.05)	5.12	
1880	E2193-B	----		----	69.87		-0.38	
1954		----		----	----		----	
1960		----		----	----		----	
2124	E2193-B	----		----	68.4		-1.40	
6013	E2193-A	78.595		0.04	----		----	
7003		----		----	----		----	
7013	E2193-B	----		----	69.4		-0.71	
9006	E2193-A	69.708		-2.53	----		----	
9007	E2193	----		----	68.078		-1.62	
9008	E2193	----		----	69.24		-0.82	
9009	E2193-B	----		----	70.465		0.03	
normality		OK		not OK				
n		13		31				
outliers		0		2				
mean (n)		78.471		70.423				
st.dev. (n)		6.4181		2.1741				
R(calc.)		17.971		6.087				
R(E2193:08)		9.682		4.047				



APPENDIX 2**Number of participants per country**

2 labs in AUSTRALIA

1 lab in AUSTRIA

2 labs in BELGIUM

1 lab in BRAZIL

3 labs in CANADA

8 labs in CHINA, People's Republic

1 lab in GERMANY

4 labs in INDIA

2 labs in IRAN, Islamic Republic of

2 labs in ITALY

2 labs in KUWAIT

1 lab in LITHUANIA

3 labs in MALAYSIA

2 labs in MEXICO

3 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

2 labs in TURKEY

1 lab in UNITED KINGDOM

6 labs in UNITED STATES OF AMERICA

1 lab in VENEZUELA

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
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

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