

Results of Proficiency Test

Methanol

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Organised by: Institute for Interlaboratory Studies
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CONTENTS

1	INTRODUCTION	3
2	SET UP.....	3
2.1	ACCREDITATION.....	3
2.2	PROTOCOL	3
2.3	CONFIDENTIALITY STATEMENT	4
2.4	SAMPLES	4
2.5	STABILITY OF THE SAMPLES	6
2.6	ANALYSES	6
3	RESULTS.....	7
3.1	STATISTICS.....	7
3.2	GRAPHICS.....	8
3.3	Z-SCORES.....	8
4	EVALUATION	8
4.1	EVALUATION PER TEST	9
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	13
4.3	COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2010 WITH PREVIOUS PT'S....	15

Appendices:

1.	Data and statistical results	16
2.	List of number of participants	72
3.	Abbreviations and literature	73

1 INTRODUCTION

Since 1996, a proficiency test for Methanol was organised every year by The Institute for Interlaboratory Studies. During the annual proficiency testing program 2010/2011, it was decided to continue the round robin for the analysis of Methanol in accordance with the latest applicable version of the IMPCA specification (latest version can be found and downloaded on www.impca.be). In the latest IMPCA specification of June 2010, the Sulphur determination ASTM D3961:98 as test method is mentioned. As ASTM D3961:98 was withdrawn in 2004 with no replacement, it was decided by the IMPCA group to extend the precision data of ASTM D5453 into the range below 1.0 mg/kg. Two samples were sent along with the regular PT samples. In this interlaboratory study 79 laboratories in 28 different countries have participated. See appendix 2 for a list of participants in alphabetical country order. In this report, the results of the proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory studies in Spijkenisse, The Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted. In this proficiency test, the participants received, depending on the registration, one or two samples of Methanol: 1*1L Methanol (labelled #1052) and/or 1*100 mL Methanol (labelled #1053) for UV only. With sample #1052, one vial of 8 mL labelled #S-01 (low) and one vial of 8 mL labelled #S-02 (high) was sent along especially for Sulphur determination. Sample #1052 was spiked with Acetone (15.2 mg/kg), Ethanol (25.3 mg/kg), Benzene (15.3 mg/kg), Toluene (19.8 mg/kg), Sodium Chloride (0.51 mg Cl/kg), Iron (0.024 mg/kg) and Trimethylamine (58 µg/kg). All materials used for spiking were >99% pure. The participants were requested to report rounded and unrounded results. The unrounded results were preferably used for the statistical evaluations.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO guide 43 and ILAC-G13:2007, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures 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 of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

2.3 CONFIDENTIALITY STATEMENT

All data present in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary 150 litre bulk material was provided by a Methanol producer. The bulk material was divided in two parts. The first batch of approx. 109 litre was spiked with the components listed in table 1:

<i>Component</i>	<i>Amount</i>
Acetone	1310 mg
Ethanol	2180 mg
Benzene	1320 mg
Toluene	1700 mg
Sodium Chloride	71.05 mg
Iron(III) Chloride	10.10 mg
Trimethylamine	5.02 mg

Table 1: components that were added to bulk material for sample #1052

After homogenisation in a pre-cleaned metal drum, this batch was divided over 105 brown glass bottles of 1L and labelled #1052.

The homogeneity of the subsamples #1052 was checked by determination of Density in accordance with ASTM D4052:02e1, Water content in accordance with ASTM E203:08, Chloride in accordance with IMPCA 002:98 and Toluene in accordance with an in house test method on 8 stratified random selected samples.

	<i>Density at 20°C in kg/L</i>	<i>Water in mg/kg</i>	<i>Chloride in mg/kg</i>	<i>Toluene in mg/kg</i>
sample #1052-1	0.79129	180	0.6	20
sample #1052-2	0.79129	160	0.6	20
sample #1052-3	0.79129	140	0.6	20
sample #1052-4	0.79129	150	0.6	20
sample #1052-5	0.79130	160	0.7	20
sample #1052-6	0.79130	180	0.6	20
sample #1052-7	0.79130	150	0.6	19
sample #1052-8	0.79129	170	0.7	20

Table 2: homogeneity tests of subsamples #1052

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

	Density at 15°C in kg/L	Water in mg/kg	Chloride in mg/kg	Toluene in mg/kg
r (sample #1052)	0.00002	41	0.1	1
reference test	ASTM D4052:02e1	ASTM E203:08	IMPCA002	Horwitz
0.3*R (reference test)	0.00015	81	0.1	2

Table 3: repeatabilities of the subsamples #1052

For the Sulphur determination, 2 bottles each with approx 1 litre Methanol were spiked with respective 0.50 mL and 1.00 mL of a Sulphur stock solution (394.5 mg Dibenzothiophene in 100mL Toluene). Both spiked Methanol samples were divided over 85 vials of 8 mL each and labelled resp. #S-01 (low) and #S-02 (high).

The homogeneity of the subsamples #S-01 and #S-02 were checked by determination of Sulphur in accordance with ASTM D5453:09 on 7 stratified random selected samples.

	Sulphur in mg/kg		Sulphur in mg/kg
sample #S-01-1	0.55	sample #S-02-1	0.86
sample #S-01-2	0.61	sample #S-02-2	0.82
sample #S-01-3	0.51	sample #S-02-3	0.86
sample #S-01-4	0.48	sample #S-02-4	0.79
sample #S-01-5	0.50	sample #S-02-5	0.94
sample #S-01-6	0.50	sample #S-02-6	0.84
sample #S-01-7	0.52	sample #S-02-7	0.89

Table 4: homogeneity tests of subsamples #S-01 and #S-02

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

	Sulphur in mg/kg		Sulphur in mg/kg
r (sample #S-01)	0.12	r (sample #S-02)	0.14
reference test	ASTM D5453:09	reference test	ASTM D5453:09
0.3*R (reference test)	0.10*	0.3*R (reference test)	0.15*

Table 5: repeatabilities of the subsamples #S-01 and #S-02

*) estimated through extrapolation from D5453:09. The actual reproducibility will be higher (see pages 63 and 65)

The third batch of approx. 7.5 litre of methanol was divided over 80 brown glass bottles of 100mL and labelled #1053.

The homogeneity of the subsamples #1053 was checked by determination of UV absorbance at 220, 250 and 268.5nm (using 5cm cells) according IMPCA004:08 on 8 stratified randomly selected samples.

	UV absorbance at 220 nm	UV absorbance at 250 nm	UV absorbance at 268.5 nm
sample #1053-1	1.252	0.162	0.047
sample #1053-2	1.201	0.157	0.043
sample #1053-3	1.202	0.156	0.043
sample #1053-4	1.232	0.155	0.043
sample #1053-5	1.221	0.156	0.043
sample #1053-6	1.229	0.155	0.042
sample #1053-7	1.218	0.154	0.042
sample #1053-8	1.220	0.158	0.043

Table 6: homogeneity tests of subsamples #1053

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

	UV at 220nm	UV at 250nm	UV at 268.5nm
r (sample #1053)	0.046	0.007	0.004
reference test	IMPCA004:06	IMPCA004:06	IMPCA004:06
0.3*R (reference test)	0.367	0.047	0.013

Table 7: repeatabilities of the subsamples #1053

Each calculated repeatability was equal or less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples #1052, #S-01, #S-02 and #1053 was assumed.

To the participants, depending on the registration, 1*1L bottle labelled #1052, 1*8 mL vial labelled #S-01, 1*8 mL vial labelled #S-02, and/or 1*100 mL bottle, labelled #1053 were sent on August 25, 2010.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were requested to determine Acidity, Anorganic Chloride, Appearance, Colour, Carbonisable Substances Pt/Co, Colour Pt/Co, Density @ 20°C, Distillation (IBP, 50% & DP), Acetone, Benzene, Ethanol, Water Miscibility, Nonvolatile Matter, Purity ("as is" and "on dry

basis"), Permanganate Time Test, Specific Gravity 20/20 °C/°C, Apparent Specific Gravity 20/20 °C/°C, Total Iron, Trimethylamine and Water (coulometric and titrimetric) on sample #952. On sample #S-01 and #S-02 was requested to determine Sulphur. On sample #1053 was requested to determine the UV absorbance at 300, 268.5, 250, 240, 230 and 220 nm.

To get comparable results, a detailed report form on which the units and the preferred test methods were printed, was sent together with each set of samples. In addition, a letter of instructions, and a SDS were added to the package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated per determination in appendix 1 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported results at that moment. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers, this check was repeated. In case a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nr.15 and 16).

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study.

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8.

The z-scores were calculated in accordance with:

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

To evaluate the performance of the participating laboratories the z-scores were calculated. Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare. Therefore the usual interpretation of z-scores is as follows:

$ z < 1$	good
$1 < z < 2$	satisfactory
$2 < z < 3$	questionable
$ z > 3$	unsatisfactory

4 EVALUATION

In this proficiency test, some major problems were encountered with despatch of the samples to the laboratories in Brazil, India, Malaysia, Mexico and Venezuela. Ten participants received the samples near, after the final reporting date, or not at all. In total, 14 participants reported after the deadline and 7 participants did not report any result at all. Not all participants were able to report all requested parameters. Finally, 73 participants did report 1353 numerical results.

Observed were 75 outlying results, which is 5.5% of the total of numerical results. In proficiency studies, outlier percentages of 3 - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. Not normal distributions were found for the following determinations: Anorganic Chloride, Carbonisable Substances, Colour Pt/Co, Density @ 20°C, Distillation (automatic and manual), NVM, Purity "as received", Purity "on dry basis", Benzene, Ethanol, PTT, Specific Gravity, Total Iron, Water (coulometric and titrimetric) and UV absorbance at 220nm. In these cases the statistical evaluation should be used with due care. From the Kernel Density graphs, one can see that this conclusion is not always justified.

Acidity: No analytical problems were observed. Only two statistical outliers were observed and the observed reproducibility, after rejection of the outliers, is in good agreement with the requirements of ASTM D1613:06.

Anorg. Chloride: This determination was problematic. Only one statistical outlier was detected. However, the calculated reproducibility after rejection of the statistical outlier, is not in agreement with the requirements of IMPCA002:98. The average recovery of the chloride content may be acceptable (0.60 mg/kg found and 0.51 mg/kg added).

Appearance: No analytical problems were observed. All labs agreed about the appearance of the sample #1052, which was bright, clear and free of suspended matter. The uniformity of reporting can be improved. A new standardized method is available for Appearance since 2009, being ASTM E2680. According this method the appearance should be reported as 'pass' (or 'fail').

Carbonisable Substances: This determination was problematic. Three statistical outliers were observed and the observed reproducibility, after rejection of the outliers, is not in agreement with the requirements of ASTM E346:08. The rounding of results may be the cause that the spread is relatively large.

Colour: No analytical problems were observed. No statistical outliers were observed and the observed reproducibility is in good agreement with the requirements of ASTM D1209:05e1.

Density @ 20°C: This determination was not problematic. Three statistical outliers were observed and the observed reproducibility, after rejection of the outliers, is in good agreement with the requirements of ASTM D4052:02e1.

Distillation: No analytical problems were observed for the automated and the manual mode as well. For the automated and manual mode in total, only three statistical outliers were detected. All calculated reproducibilities (IBP, MBP and DP for automated and manual mode) are, after rejection of the detected statistical outliers, in good agreement with the requirements for automated and manual modes of ASTM D1078:05. This may be remarkable, as several participants did obviously not correct for the theoretical mid boiling point of 64.5°C.

Water Miscibility: No analytical problems were observed. All laboratories, except one, reported the test as “pass” or “passes”. One laboratory reported “fail”.

NVM: No analytical problems were observed. Only two statistical outliers were observed and the observed reproducibility, after rejection of the statistical outliers, is in good agreement with the requirements of ASTM D1353:09.

Purity: For the purity “as received” and “on dry basis”, in total three statistical outliers were observed. The calculated reproducibilities after rejection of the statistical outliers, are in agreement with the calculated reproducibilities of the 2009 PT iis09C07 (for “as is” 0.017 vs 0.018 and for “dry basis” 0.020 vs 0.019).

Acetone: This determination was problematic. Two statistical outliers and three false positives were observed. The calculated reproducibility after rejection of the statistical outliers, is not at all in agreement with the strict reproducibility limits estimated using the Horwitz equation. However, the average recovery of Acetone (theoretical increment of 15.2 mg Acetone/kg) may be good: “less than 105%” (the actual blank Acetone content is unknown).

Benzene: This determination may be problematic for a number of laboratories. Two statistical outliers and three false negatives were observed. The calculated reproducibility after rejection of the statistical outliers, is in good agreement with the strict reproducibility limits estimated using the Horwitz equation. Also, the average recovery of Benzene (theoretical increment of 15.3 mg Benzene/kg) may be good: “less than 101%” (the actual blank Benzene content is unknown).

Ethanol: This determination may be problematic. Four statistical outliers were observed. Also, the calculated reproducibility after rejection of the statistical outliers, is not in agreement with the strict reproducibility limits estimated using the Horwitz equation. However, the average recovery of Ethanol (theoretical increment of 25.3 mg Ethanol/kg) may be good: “less than 116%” (the actual blank Ethanol content is unknown).

Toluene: This determination may be problematic. Two statistical outliers were observed and three “less than 10 mg/kg” results were reported. It should be noted that application of IMPCA001 to the letter indeed will result in a negative test result (no toluene detected). However, the calculated reproducibility after rejection of the statistical outliers, is in good agreement with the strict estimated reproducibility limits calculated using the Horwitz equation. Also, the average recovery of Toluene (theoretical increment of 19.8 mg Toluene/kg) may be good: “less than 99%” (the actual blank Toluene content is unknown).

PTT: All participants, except two, agreed on a result far above 60 minutes.

As it is unknown whether a Permanganate Time Test of >60 minutes is in the applicability range, it is therefore difficult to draw any conclusions. Therefore, no z-scores were calculated. Two statistical outliers were observed.

SG 20/20 °C: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers, is in good agreement with the requirements of ASTM D4052:02e1. The Specific Gravity is defined as: "*the ratio of the weight in Vacuum of a unit volume of a material at stated temperature to the weight in Vacuum of an equal volume of gas-free distilled water at a stated temperature*".

$$\text{SG } 20/20 \text{ } ^\circ\text{C} = (\text{density material at } 20 \text{ } ^\circ\text{C}) / (\text{density water at } 20 \text{ } ^\circ\text{C}).$$

ASG 20/20 °C: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers, is in good agreement with the requirements of ASTM D4052:02e1. The Apparent Specific Gravity is defined as: "*the ratio of the weight in air of a unit volume of a material at stated temperature to the weight in air of equal density of an equal volume of gas-free distilled water at a stated temperature*".

$$\text{SG Apparent } 20/20 \text{ } ^\circ\text{C} = (\text{density material at } 20 \text{ } ^\circ\text{C} - 0.00120) / (\text{density water at } 20 \text{ } ^\circ\text{C} - 0.00120).$$

SG General: When the Specific Gravities and Apparent Specific Gravities were calculated from the reported Densities, it was noticed that the reported results for the Specific Gravity 20/20 °C and Apparent Specific Gravity 20/20 °C are in line with the calculated results. Users of method ASTM D891 should be aware that this method results in Apparent Specific Gravity. To arrive at Specific Gravity or Density an additional conversion is necessary. The method provides the calculation formula.

Total Iron: This determination was problematic. Three statistical outliers and two false negatives were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM E394:09. However, the average recovery of Iron (theoretical increment of 0.024 mg Iron/kg) may be good: "less than 117%" (the actual blank Iron content is unknown).

TMA: This determination may be problematic. Two statistical outliers and one false negative were observed. The calculated reproducibility, after rejection of the statistical outliers, is not in agreement with the strict estimated reproducibility limits calculated using the Horwitz equation. The average recovery of the TMA (theoretical increment of 58.4 µg TMA/kg may be questionable, less than 64% (the actual blank TMA content is unknown)).

Water (coul.): This determination was very problematic. Six statistical outliers were observed. The calculated reproducibility even after rejection of the statistical outliers, is not at all in agreement with the strict requirements of ASTM E1064:08.

Water (titr.): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM E203:08.

Sulphur: As ASTM D3961:98 was withdrawn in 2004 with no replacement, it was decided by IMPCA to try and extend the application range for precision of ASTM D5453 to below 1 mg/kg. For this evaluation low sulphur level samples #S-01 and #S-02 were prepared. Both samples were spiked with dibenzothiophene: sample #S-01 to 0.502 mg Sulphur/kg and sample #S-02 to 1.007 mg Sulphur/kg. The participating laboratories were requested to determine and report the Sulphur content according both ASTM D5453 and ASTM D3961. The reported ASTM D5453 results for both samples appeared to be quite good. Only one statistical outlier was observed (for sample #S-02) and the calculated reproducibilities are both in full agreement with the extrapolated (!) requirements of ASTM D5453:09.

Regretfully, for the sulphur determination according ASTM D3961, no precision data is available, which makes direct evaluation of the test results difficult. However, the precision of the ASTM D3961 test results is smaller than the precision of the ASTM D5453 test results for sample #S-01 and somewhat larger for sample #S-02.

The average recovery of Sulphur on sample #S-01 (theoretical increment of 0.502 mg S/kg) may be good: “less than 123%” for ASTM D5453 and “less than 97%” for ASTM D3961. The actual blank Sulphur content is unknown. The average recovery of Sulphur on sample #S-02 (theoretical increment of 1.007 mg S/kg) may be good: “less than 99%” for ASTM D5453 and “less than 87%” for ASTM D3961. Again the actual blank Sulphur content is unknown.

UV-Absorbance: Sample #1053 was especially prepared for UV-absorbance testing. A split was made between the participants that used a 10mm and a 50mm cuvet. The determination was problematic for several laboratories. In total 24 statistical outliers were observed. The observed reproducibilities for UV at 250nm (50mm cuvet) and for 300nm and 268.5nm (10mm cuvet) were not in agreement with the requirements of IMPCA004:08. For UV at 240nm and 230nm no precision data are available. The other observed reproducibilities were all in agreement with IMPCA004:08.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM standards) are compared in the next tables.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as acetic acid	mg/kg	64	11.25	7.94	14.00
Anorganic Chloride as Cl	mg/kg	39	0.60	0.41	0.30
Carbonisable Substances	Pt/Co	48	6.0	6.4	5.0
Colour	Pt/Co	44	2.2	3.5	7.0
Density @ 20 °C	kg/L	60	0.79124	0.00022	0.00050
Initial Boiling Point (automatic)	°C	38	64.43	0.39	1.01
Mid Boiling Point (automatic)	°C	37	64.54	0.34	1.01
Dry Point (automatic)	°C	38	64.77	0.45	0.69
Initial Boiling Point (manual)	°C	27	64.39	0.17	0.69
Mid Boiling Point (manual)	°C	26	64.50	0.20	0.69
Dry Point (manual)	°C	25	64.77	0.25	0.84
Nonvolatile Matter	mg/100 mL	46	0.31	0.67	2.40
Purity	%M/M	46	99.977	0.018	unknown
Purity on dry basis	%M/M	48	99.986	0.019	unknown
Acetone	mg/kg	46	16.01	7.08	4.73
Benzene	mg/kg	32	15.50	2.23	4.60
Ethanol	mg/kg	50	29.55	9.66	7.95
Toluene	mg/kg	30	19.68	3.30	5.63
Permanganate Time Test	minutes	59	96.5	36.9	unknown
Specific Gravity 20/20 °C		56	0.79267	0.00017	0.00050
Apparent Spec.Grav.20/20 °C		34	0.79244	0.00027	0.00050
Total Iron as Fe	mg/kg	44	0.0281	0.0222	0.0151
Trimethylamine	mg/kg	9	37.4	13.4	9.7
Water (coulometric)	mg/kg	56	152.89	56.52	27.90
Water (titrimetric)	mg/kg	39	158.83	60.30	270.00

table 8: Reproducibilities for sample #1052

Parameter	unit	n	average	2.8 * sd	R (lit)
Sulphur (low) acc D5453	mg/kg	27	0.62	0.42	(0.40)
Sulphur (low) acc D3961	mg/kg	8	0.49	0.14	unknown
Sulphur (high) acc D5453	mg/kg	26	1.00	0.36	(0.58)
Sulphur (high) acc D3961	mg/kg	8	0.88	0.45	unknown

table 9: Reproducibilities for samples #S-01 and #S-02

Parameter	unit	n	average	2.8 * sd	R (lit)
UV absorbance at 300 nm (50 mm cell)		22	0.0101	0.0062	0.0151
UV absorbance at 268.5 nm (50 mm cell)		22	0.0398	0.0111	0.0108
UV absorbance at 250 nm (50 mm cell)		23	0.1413	0.0325	0.0143
UV absorbance at 240 nm (50 mm cell)		23	0.2430	0.0386	unknown
UV absorbance at 230 nm (50 mm cell)		23	0.5402	0.0832	unknown
UV absorbance at 220 nm (50 mm cell)		25	1.1352	0.1620	0.3076
UV absorbance at 300 nm (10 mm cell)		11	0.0019	0.0030	0.0005
UV absorbance at 268.5 nm (10 mm cell)		11	0.0077	0.0083	0.0022
UV absorbance at 250 nm (10 mm cell)		10	0.0274	0.0081	0.0078
UV absorbance at 240 nm (10 mm cell)		9	0.0477	0.0112	unknown
UV absorbance at 230 nm (10 mm cell)		9	0.1046	0.0268	unknown
UV absorbance at 220 nm (10 mm cell)		10	0.2242	0.0535	0.0643

table 10: Reproducibilities for sample #1053

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 SEPTEMBER 2010 WITH PREVIOUS PTS

	September 2010	September 2009	September 2008	September 2007
Number of reporting labs	73	59	60	48
Number of results reported	1353	782	748	619
Statistical outliers	75	41	24	28
Percentage outliers	5.5%	5.2%	3.2%	4.5%

table 11: comparison with previous proficiency tests.

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

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

	September 2010	September 2009	September 2008	September 2007
Acidity as acetic acid	++	++	++	++
Chloride as Cl	--	++	++	--
Carbonisable Substances	--	--	--	--
Colour	++	++	++	++
Density @ 20 °C	++	++	++	++
Distillation (automatic)	++	++	++	++
Distillation (manual)	++	++	++	++
Nonvolatile Matter	++	++	++	++
Specific Gravity 20/20 °C	++	++	++	++
Total Iron	--	-	--	n.e. --
Water (coulometric)	--	--	--	--
Water (titrimetric)	++	++	++	++
Benzene	++	--	--	n.d.
Toluene	++	++	n.d.	n.d.
Acetone	--	--	n.e. --	--
Ethanol	--	+	-- +	+
UV absorbance 300nm *)	++	--	+ --	n.e. n.e.
UV absorbance 268.5 nm *)	+/-	--	--	n.e. n.e.
UV absorbance 250 nm *)	--	+/-	--	n.e. n.e.
UV absorbance 220 nm *)	++	++	++	n.e. n.e.

table 12: comparison determinations against the standard requirements

*) split-up into 50 mm and 10 mm cell results

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

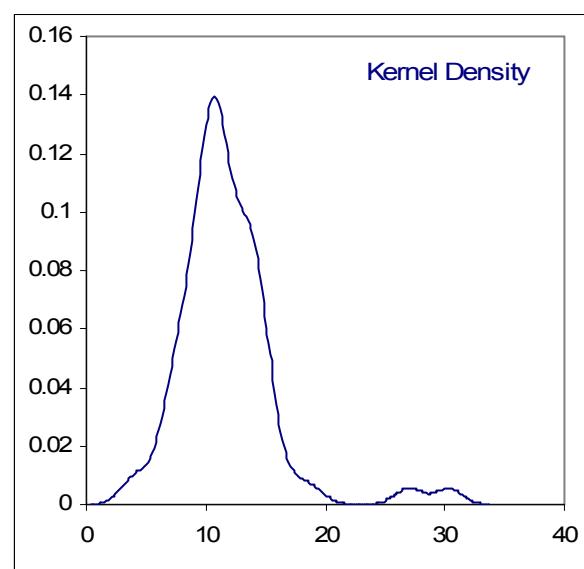
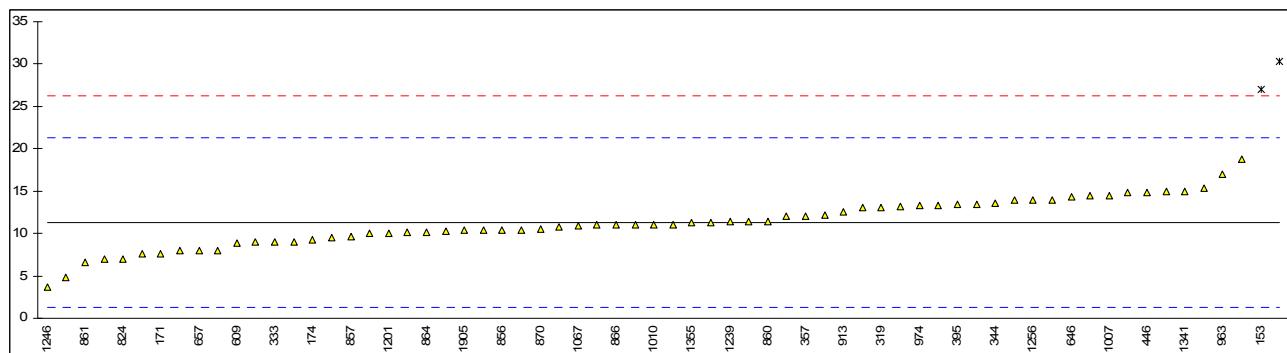
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

APPENDIX 1**Determination of Acidity as Acetic Acid on sample #1052; results in mg/kg**

lab	method	value	mark	z(targ)	remarks
53	D1613	11.37		0.02	
150	D1613	9		-0.45	
153	D1613	27	C,G(0.01)	3.15	reported 0.0027; probably in %M/M, converted by iis into mg/kg
171	D1613	7.6		-0.73	
174	D1613	9.3		-0.39	
311	D1613	11		-0.05	
316		----		----	
319	D1613	13		0.35	
323		----		----	
332	D1613	10		-0.25	
333	D1613	9		-0.45	
334		----		----	
342	D1613	13.2		0.39	
343	D1613	13.3	C	0.41	first reported 26.5
344	D1613	13.62		0.47	
345	D1613	14		0.55	
346		----		----	
347	D1613	14.4		0.63	
357	D1613	12.1		0.17	
395	D1613	13.46		0.44	
444	D1613	18.8	C	1.51	first reported 24.26
446	D1613	14.9		0.73	
494	D1613	9		-0.45	
497	D1613	15.3		0.81	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609	D1613	8.9		-0.47	
646	D1613	14.3		0.61	
657	D1613	8		-0.65	
663	D1613	9.5		-0.35	
823	D1613	7		-0.85	
824	D1613	7		-0.85	
825	D1613	8		-0.65	
840		----		----	
855	D1613	10.4		-0.17	
856	D1613	10.4		-0.17	
857	D1613	9.7		-0.31	
858	D1613	10.4		-0.17	
859	D1613	10.3		-0.19	
860	D1613	11.4		0.03	
861	D1613	6.6		-0.93	
862	D1613	12.2		0.19	
863	D1613	10.1		-0.23	
864	D1613	10.2		-0.21	
866	D1613	11.0		-0.05	
870	D1613	10.5		-0.15	
902	D1613	13.5		0.45	
912	D1613	15		0.75	
913	D1613	12.6		0.27	
963	D1613	17		1.15	
974	D1613	13.27		0.40	
1007	in house	14.5		0.65	
1009	D1613	11		-0.05	
1010	D1613	11		-0.05	
1016		----		----	
1029	D1613	14.89		0.73	
1041	D1613	11.31		0.01	
1067	D1613	10.9		-0.07	
1108		----		----	
1149	D1613	13		0.35	
1201	D1613	10		-0.25	
1204	D1613	10.7974		-0.09	
1221	D1613	12.087		0.17	
1239	D1613	11.35		0.02	
1246	D1613	3.7	C	-1.51	reported 0.00037; probably in %M/M, converted by iis into mg/kg
1256	D1613	14		0.55	
1341	D1613	15		0.75	
1342	D1613	11		-0.05	
1343		----		----	
1344	D1613	8		-0.65	
1354	D1613	7.6		-0.73	

1355	D1613	11.3	0.01
1510	D1613	14	0.55
1538		----	----
1615	D1613	4.78	-1.29
1826	D1613	30.3	3.81
1905	D1613	10.4	-0.17

normality OK
 n 64
 outliers 2
 mean (n) 11.25
 st.dev. (n) 2.837
 R(calc.) 7.94
 R(D1613:06) 14.00

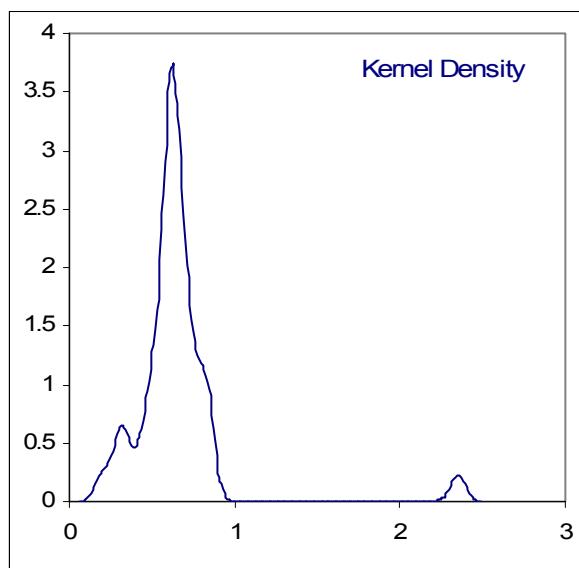
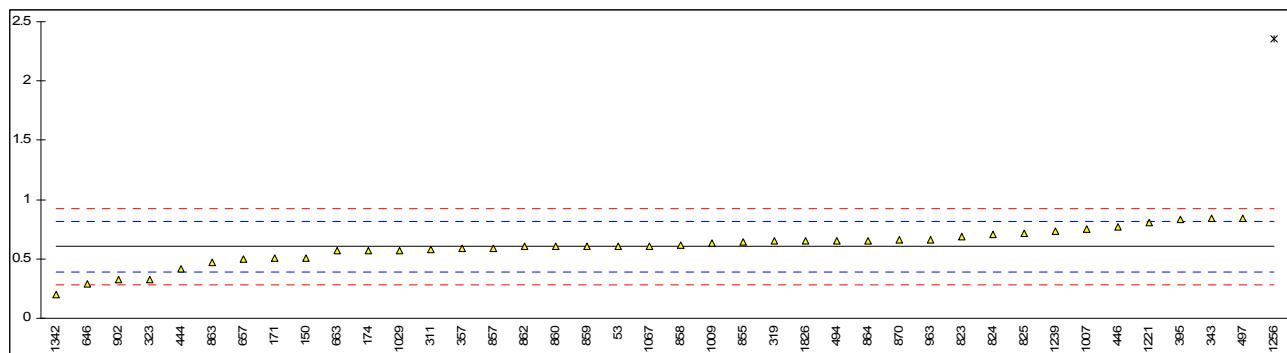


Determination of Anorganic Chloride as Cl on sample #1052; results in mg/kg

lab	method	value	mark	z(targ)	remarks
53	IMPCA002	0.61		0.05	
150	IMPCA002	0.51		-0.89	
153		----		----	
171	IMPCA002	0.51		-0.89	
174	IMPCA002	0.574		-0.29	
311	IMPCA002	0.58		-0.23	
316		----		----	
319	IMPCA002	0.65		0.42	
323	IMPCA002	0.33		-2.57	
332		----		----	
333		----		----	
334		----		----	
342		----		----	
343	IMPCA002	0.84		2.19	
344		----		----	
345		----		----	
346		----		----	
347		----		----	
357	IMPCA002	0.59		-0.14	
395	IMPCA002	0.834		2.14	
444	IMPCA002	0.418		-1.74	
446	IMPCA002	0.77		1.54	
494	IMPCA002	0.65		0.42	
497	IMPCA002	0.84		2.19	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609		----		----	
646	IMPCA002	0.29		-2.94	
657	IMPCA002	0.5		-0.98	
663	IMPCA002	0.57		-0.33	
823	IMPCA002	0.69		0.79	
824	IMPCA002	0.71		0.98	
825	IMPCA002	0.72		1.07	
840		----		----	
855	IMPCA002	0.64		0.33	
856		----		----	
857	IMPCA002	0.590		-0.14	
858	IMPCA002	0.62		0.14	
859	IMPCA002	0.61		0.05	
860	IMPCA002	0.61		0.05	
861		----		----	
862	IMPCA002	0.61		0.05	
863	IMPCA002	0.47		-1.26	
864	IMPCA002	0.65		0.42	
866		----		----	
870	IMPCA002	0.66		0.51	
902	IMPCA002	0.33		-2.57	
912		----		----	
913		----		----	
963	IMPCA002	0.66		0.51	
974		----		----	
1007	in house	0.75		1.35	
1009	in house	0.63		0.23	
1010	Turb.	>0.25		----	
1016		----		----	
1029	IMPCA002	0.575		-0.28	
1041		----		----	
1067	IMPCA002	0.61		0.05	
1108		----		----	
1149		----		----	
1201	IMPCA002	<1		----	
1204		----		----	
1221	IMPCA002	0.81		1.91	
1239	titrometric	0.73		1.17	
1246		----		----	
1256	IMPCA002	2.3518	G(0.01)	16.30	
1341		----		----	
1342	IMPCA002	0.2		-3.78	
1343		----		----	
1344		----		----	
1354		----		----	

1355		-----	-----
1510		-----	-----
1538		-----	-----
1615		-----	-----
1826	in house	0.65	0.42
1905		-----	-----

normality not OK
 n 39
 outliers 1 Spike
 mean (n) 0.60
 st.dev. (n) 0.147
 R(calc.) 0.41
 R(IMPCA002:98) 0.30



Determination of Appearance on sample #1052;

lab	method	value	mark	z(targ)	remarks
53	E2680	PASS	----		
150	E2680	PASS	----		
153		----			
171	E2680	C&B	----		
174	E2680	C&F	----		
311	INH-402	C&F	----		
316		----			
319	E2680	CFFSM	----		
323	E2680	PASS	----		
332	E2680	PASS	----		
333	E2680	C&B	----		
334		----			
342	E2680	PASS	----		
343	INH-1608	CFSM	----		
344	E2680	PASS	----		
345	E2680	PASS	----		
346		----			
347	E2680	PASS	----		
357	E2680	PASS	----		
395	E2680	PASS	----		
444	E2680	PASS	----		
446	IMPCA003	CFFSM	----		
494	E2680	PASS	----		
497	INH-1462	C&B	----		
528		----			
529		----			
551		----			
608		----			
609	E2680	PASS	----		
646	E2680	CFFSM	----		
657	E2680	CBSM	----		
663	E2680	C&B	----		
823	E2680	CFSM	----		
824	E2680	CFSM	----		
825	E2680	CFSM	----		
840		----			
855	E2680	CFFSM	----		
856	IMPCA003	CFFSM	----		
857	E2680	PASS	----		
858	E2680	PASS	----		
859	E2680	PASS	----		
860	E2680	PASS	----		
861	E2680	PASS	----		
862	E2680	PASS	----		
863	IMPCA003	CFFSM	----		
864	IMPCA003	CFFSM	----		
866	E2680	PASS	----		
870	E2680	CFFSM	----		
902	E2680	PASS	----		
912	E2680	CFFSM	----		
913	E2680	CFFSM	----		
963	E2680	PASS	----		
974	E2680	PASS	----		
1007	in house	PASS	----		
1009	in house	CFSM	----		
1010	IMPCA003	CFSM	----		
1016	INHOUSE	PASS	----		
1029	Visual	CLEAR	----		
1041	E2680	CFSM	----		
1067	IMPCA003	CFSM	----		
1108		----			
1149	E2680	C&B	----		
1201	E2680	C&B	----		
1204	IMPCA003	CWSF	----		
1221	IMPCA003	CFSM	----		
1239	E2680	PASS	----		
1246	IMPCA003	CFSM	----		
1256	E2680	C&B	----		
1341	IMPCA003	CFSM	----		
1342	E2680	CFSM	----		
1343		----			
1344	E2680	C&F	----		
1354	E2680	CFSM	----		

1355	E2680	CFSM	----
1510	VISUAL	CFFSM	----
1538		-----	----
1615	VISUAL	PASS	----
1826	E2680	PASS	----
1905	INH-232	CFSM	----
n		28	
	mean (n)	PASS	

Abbreviations:

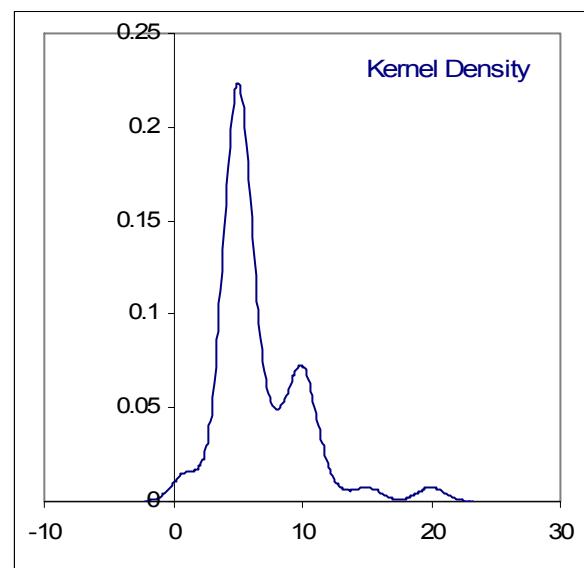
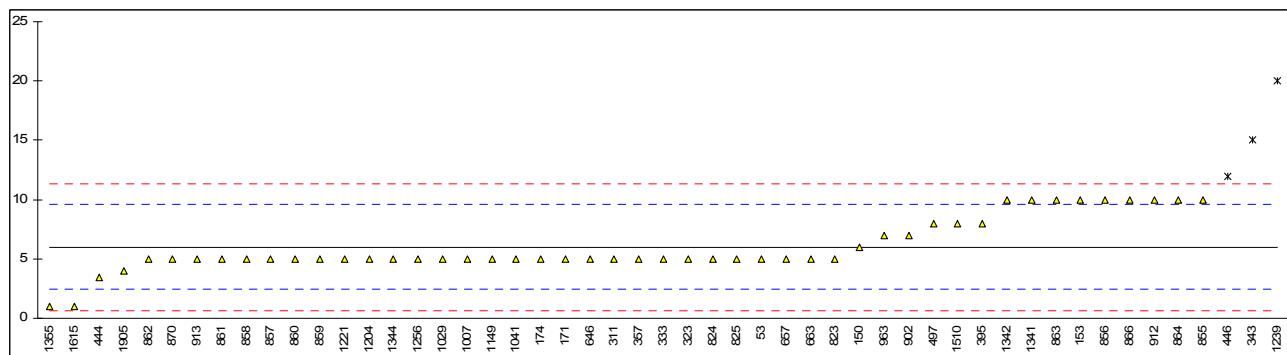
- C&B: bright and clear
CFFSM: clear and free from matter in suspension
CFSM: clear and free from suspended matter
C&F: clear and free
CWSF: clear with free particles

Determination of Carbonisable Substances Pt/Co on sample #1052;

lab	method	value	mark	z(targ)	remarks
53	E346	5		-0.56	
150	E346	6		0.00	
153	E346	10		2.24	
171	E346	5		-0.56	
174	E346	5		-0.56	
311	E346	5		-0.56	
316		----		----	
319	E346	<5		----	
323	E346	5		-0.56	
332		----		----	
333	E346	5		-0.56	
334		----		----	
342		----		----	
343	E346	15	G(0.05)	5.04	
344	E346	<30		----	
345		----		----	
346		----		----	
347		----		----	
357	E346	5		-0.56	
395	E346	8		1.12	
444	E346	3.4		-1.46	
446	E346	12	G(0.05)	3.36	
494		----		----	
497	E346	8		1.12	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609		----		----	
646	E346	5		-0.56	
657	E346	5		-0.56	
663	E346	5		-0.56	
823	E346	5		-0.56	
824	E346	5		-0.56	
825	E346	5		-0.56	
840		----		----	
855	E346	10		2.24	
856	E346	10		2.24	
857	E346	5		-0.56	
858	E346	5		-0.56	
859	E346	5		-0.56	
860	E346	5		-0.56	
861	E346	5		-0.56	
862	E346	5		-0.56	
863	E346	10		2.24	
864	E346	10		2.24	
866	E346	10		2.24	
870	E346	5		-0.56	
902	E346	7		0.56	
912	E346	10		2.24	
913	E346	5.0		-0.56	
963	E346	7		0.56	
974		----		----	
1007	in house	5		-0.56	
1009	E346	<30		----	
1010	E346	<5		----	
1016		----		----	
1029	E346	5		-0.56	
1041	E346	5		-0.56	
1067		----		----	
1108		----		----	
1149	E346	5		-0.56	
1201	E346	<5		----	
1204	E346	5		-0.56	
1221	E346	5		-0.56	
1239	E346	20	G(0.01)	7.84	
1246	E346	<30		----	
1256	E346	5		-0.56	
1341	E346	10		2.24	
1342	E346	10		2.24	
1343		----		----	
1344	E346	5		-0.56	
1354	E346	<1		<-2.80	False negative?

1355	E346	1	-2.80
1510	E346	8	1.12
1538		----	----
1615	E346	1	-2.80
1826		----	----
1905	E346	4	-1.12

normality not OK
 n 48
 outliers 3
 mean (n) 6.0
 st.dev. (n) 2.30
 R(calc.) 6.4
 R(E346:08) 5.0

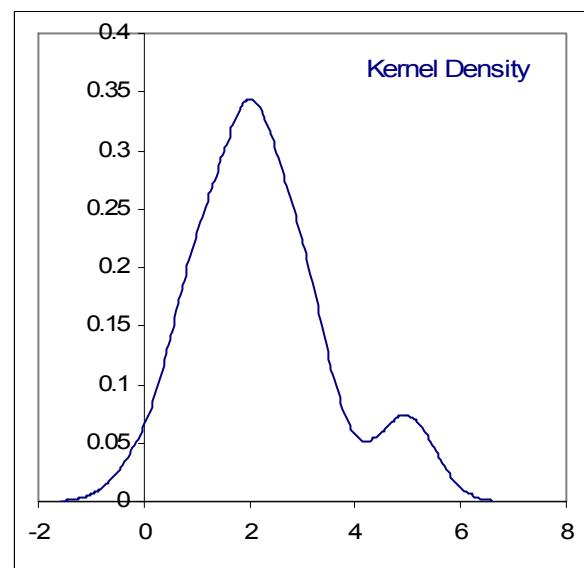
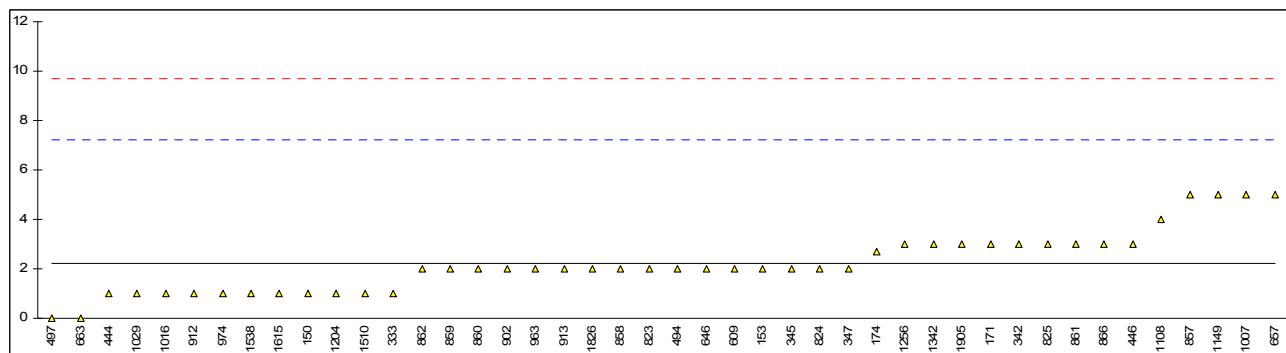


Determination of Colour as Pt/Co on sample #1052;

lab	method	value	mark	z(targ)	remarks
53	D1209	<5		----	
150	D1209	1		-0.48	
153	D1209	2		-0.08	
171	D1209	3		0.32	
174	D1209	2.7		0.20	
311	D1209	<5		----	
316		----		----	
319	D1209	<5		----	
323	D1209	<5		----	
332		----		----	
333	D1209	1		-0.48	
334		----		----	
342	D5386	3		0.32	
343		----		----	
344	d1209	<5		----	
345	D1209	2		-0.08	
346		----		----	
347	D1209	2		-0.08	
357	D1209	<5		----	
395	D1209	<5		----	
444	D5386	1		-0.48	
446	D1209	3		0.32	
494	D1209	2		-0.08	
497	D1209	0		-0.88	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609	D1209	2		-0.08	
646	D1209	2		-0.08	
657	D1209	5		1.12	
663	D1209	0		-0.88	
823	D1209	2		-0.08	
824	D1209	2		-0.08	
825	D1209	3		0.32	
840		----		----	
855	D1209	<5		----	
856	D1209	<5		----	
857	D1209	5		1.12	
858	D1209	2		-0.08	
859	D1209	2		-0.08	
860	D1209	2		-0.08	
861	D1209	3		0.32	
862	D1209	2		-0.08	
863	D1209	<5		----	
864	D1209	<5		----	
866	D1209	3		0.32	
870	D1209	<5		----	
902	D5386	2		-0.08	
912	D5386	1		-0.48	
913	D5386	2.0		-0.08	
963	D1209	2		-0.08	
974	D1209	1		-0.48	
1007	in house	5		1.12	
1009	D1209	<5		----	
1010	D1209	<5		----	
1016	D1209	1		-0.48	
1029	D1209	1		-0.48	
1041	D1209	<5		----	
1067	D1209	<5		----	
1108	D1209	4		0.72	
1149	D1209	5		1.12	
1201	D1209	<5		----	
1204	D1209	1		-0.48	
1221		----		----	
1239	D1209	<5		----	
1246	D1209	<5		----	
1256	D1209	3		0.32	
1341	D1209	<5		----	
1342	D1209	3		0.32	
1343		----		----	
1344	D1209	<5		----	
1354	D1209	<1		----	

1355	D1209	<1	-----
1510	D1209	1	-0.48
1538	D1209	1	-0.48
1615	D1209	1	-0.48
1826	D1209	2	-0.08
1905	D1209	3	0.32

normality not OK
 n 44
 outliers 0
 mean (n) 2.2
 st.dev. (n) 1.25
 R(calc.) 3.5
 R(D1209:05e1) 7.0

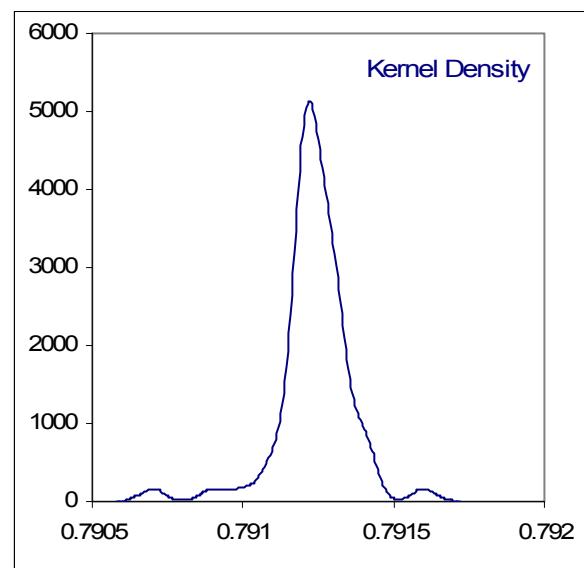
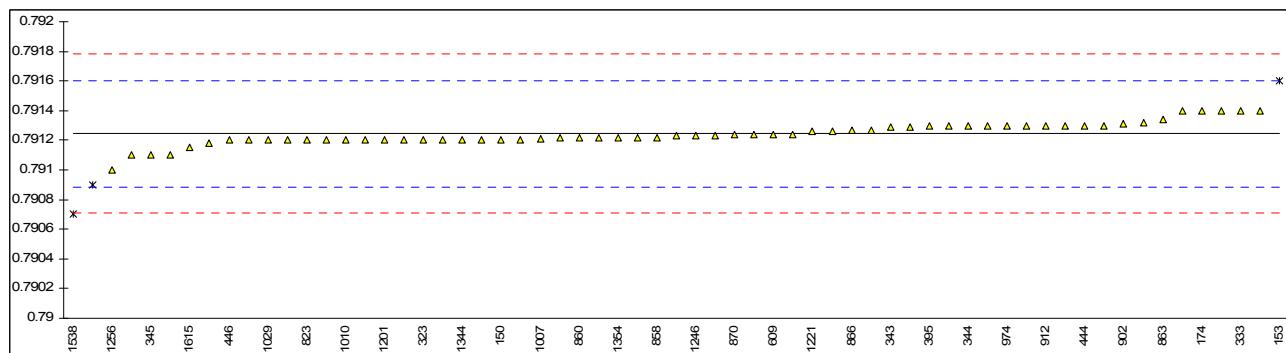


Determination of Density at 20 °C on sample #1052; results in kg/L

lab	method	value	mark	z(targ)	remarks
53	D4052	0.7913	C	0.31	first reported 791.26
150	D4052	0.7912		-0.25	
153	D4052	0.7916	G(0.01)	1.99	
171	D4052	0.7912		-0.25	
174	D4052	0.7914		0.87	
311		----		----	
316		----		----	
319		----		----	
323	D4052	0.7912	C	-0.25	first reported 0.7915
332	D4052	0.7914		0.87	
333	D4052	0.7914		0.87	
334	D4052	0.7914		0.87	
342	D4052	0.7911		-0.81	
343	D4052	0.79129	C	0.26	first reported 0.79178
344	D4052	0.7913		0.31	
345	D4052	0.7911		-0.81	
346	D1298	0.7909	G(0.05)	-1.93	
347	D4052	0.79132		0.43	
357	D4052	0.7911		-0.81	
395	D4052	0.7913		0.31	
444	D4052	0.7913		0.31	
446	D4052	0.7912		-0.25	
494	D4052	0.79118		-0.36	
497	D4052	0.79126		0.09	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609	D4052	0.79124		-0.02	
646		----		----	
657	D4052	0.7912		-0.25	
663	D4052	0.7912		-0.25	
823	D4052	0.7912		-0.25	
824	D4052	0.7912		-0.25	
825	D4052	0.7912		-0.25	
840		----		----	
855	D4052	0.79123		-0.08	
856		----		----	
857	D4052	0.79124		-0.02	
858	D4052	0.79122		-0.13	
859	D4052	0.79122		-0.13	
860	D4052	0.79122		-0.13	
861	D4052	0.79122		-0.13	
862	D4052	0.79124		-0.02	
863	D4052	0.79134		0.54	
864	D4052	0.79123		-0.08	
866	D4052	0.79127		0.15	
870	D4052	0.79124		-0.02	
902	D4052	0.79131		0.37	
912	D4052	0.7913		0.31	
913	D4052	0.79129		0.26	
963	D4052	0.7913		0.31	
974	D4052	0.7913		0.31	
1007	in house	0.79121		-0.19	
1009		----		----	
1010	D4052	0.7912		-0.25	
1016	D4052	0.7913	C	0.31	first reported 0.7908
1029	D4052	0.79120		-0.25	
1041		----		----	
1067	D4052	0.7913		0.31	
1108	D4052	0.79127		0.15	
1149		----		----	
1201	D4052	0.7912		-0.25	
1204	D4052	0.7913		0.31	
1221	D4052	0.79126		0.09	
1239		----		----	
1246	D4052	0.79123		-0.08	
1256	D4052	0.7910		-1.37	
1341		----		----	
1342	D4052	0.7912		-0.25	
1343		----		----	
1344	D4052	0.7912		-0.25	
1354	D4052	0.79122	C	-0.13	first reported 791.22

1355	D4052	0.79122	C	-0.13	first reported 791.22
1510	IP365	0.7914		0.87	
1538	D4052	0.7907	C,G(0.01)	-3.05	first reported 791.2
1615	D4052	0.79115		-0.53	
1826	D4052	0.7912	C	-0.25	first reported 791.2
1905	D4052	0.79120		-0.25	

normality not OK
 n 60
 outliers 3
 mean (n) 0.79124
 st.dev. (n) 0.000077
 R(calc.) 0.00022
 R(D4052:02e1) 0.00050



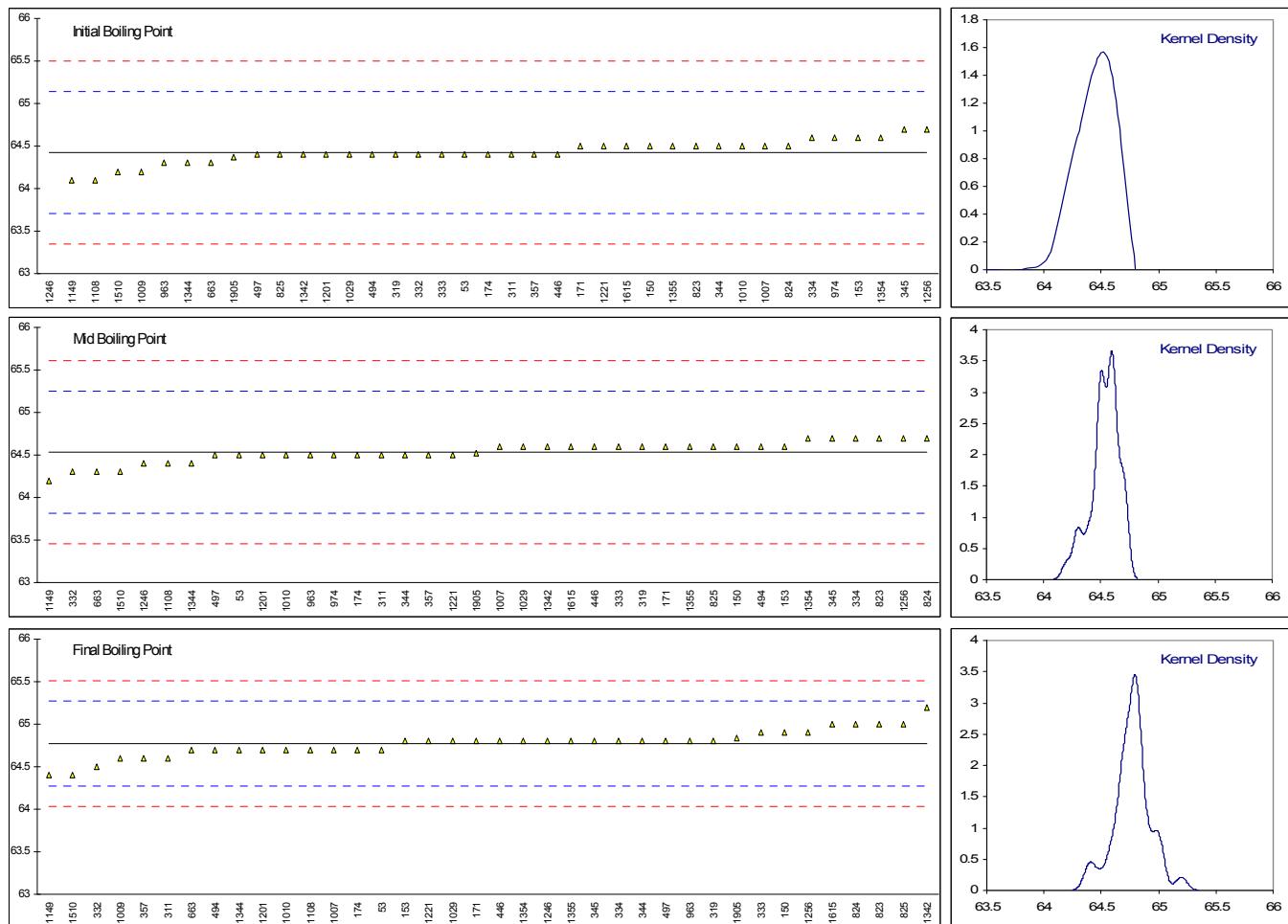
Determination of IBP, MBP and DP (automated) @ 760 mmHg on sample #1052; results in °C

lab	method	IBP	mark	z(targ)	MBP	mark	z(targ)	DP	mark	z(targ)	remarks
53	D1078	64.4		-0.08	64.5		-0.11	64.7		-0.29	
150	D1078	64.5		0.20	64.6		0.17	64.9		0.52	
153	D1078	64.6		0.48	64.6		0.17	64.8		0.11	
171	D1078	64.5		0.20	64.6		0.17	64.8		0.11	
174	D1078	64.4		-0.08	64.5		-0.11	64.7		-0.29	
311	D1078	64.4		-0.08	64.5		-0.11	64.6		-0.70	
316	----	----	----	----	----	----	----	----	----	----	
319	D1078	64.4		-0.08	64.6		0.17	64.8		0.11	
323	----	----	----	----	----	----	----	----	----	----	
332	D1078	64.4		-0.08	64.3		-0.66	64.5		-1.10	
333	D1078	64.4		-0.08	64.6		0.17	64.9		0.52	
334	D1078	64.6		0.48	64.7		0.45	64.8		0.11	
342	----	----	----	----	----	----	----	----	----	----	
343	----	----	----	----	----	----	----	----	----	----	
344	D1078	64.5		0.20	64.5		-0.11	64.8		0.11	
345	D1078	64.7		0.76	64.7		0.45	64.8		0.11	
346	----	----	----	----	----	----	----	----	----	----	
347	----	----	----	----	----	----	----	----	----	----	
357	D1078	64.4		-0.08	64.5		-0.11	64.6		-0.70	
395	----	----	----	----	----	----	----	----	----	----	
444	----	----	----	----	----	----	----	----	----	----	
446	D1078	64.4		-0.08	64.6		0.17	64.8		0.11	
494	D1078	64.4		-0.08	64.6		0.17	64.7		-0.29	
497	D1078	64.4		-0.08	64.5		-0.11	64.8		0.11	
528	----	----	----	----	----	----	----	----	----	----	
529	----	----	----	----	----	----	----	----	----	----	
551	----	----	----	----	----	----	----	----	----	----	
608	----	----	----	----	----	----	----	----	----	----	
609	----	----	----	----	----	----	----	----	----	----	
646	----	----	----	----	----	----	----	----	----	----	
657	----	----	----	----	----	----	----	----	----	----	
663	D1078	64.3		-0.36	64.3		-0.66	64.7		-0.29	
823	D1078	64.5		0.20	64.7		0.45	65.0		0.92	
824	D1078	64.5		0.20	64.7		0.45	65.0		0.92	
825	D1078	64.4		-0.08	64.6		0.17	65.0		0.92	
840	----	----	----	----	----	----	----	----	----	----	
855	----	----	----	----	----	----	----	----	----	----	
856	----	----	----	----	----	----	----	----	----	----	
857	----	----	----	----	----	----	----	----	----	----	
858	----	----	----	----	----	----	----	----	----	----	
859	----	----	----	----	----	----	----	----	----	----	
860	----	----	----	----	----	----	----	----	----	----	
861	----	----	----	----	----	----	----	----	----	----	
862	----	----	----	----	----	----	----	----	----	----	
863	----	----	----	----	----	----	----	----	----	----	
864	----	----	----	----	----	----	----	----	----	----	
866	----	----	----	----	----	----	----	----	----	----	
870	----	----	----	----	----	----	----	----	----	----	
902	----	----	----	----	----	----	----	----	----	----	
912	----	----	----	----	----	----	----	----	----	----	
913	----	----	----	----	----	----	----	----	----	----	
963	D1078	64.3		-0.36	64.5		-0.11	64.8		0.11	
974	D1078	64.6		0.48	64.5		-0.11	----		----	
1007	in house	64.5		0.20	64.6		0.17	64.7		-0.29	
1009	D1078	64.2		-0.64	----		----	64.6		-0.70	
1010	D1078	64.5		0.20	64.5		-0.11	64.7		-0.29	
1016	----	----	----	----	----	----	----	----	----	----	
1029	D1078	64.4		-0.08	64.6		0.17	64.8		0.11	
1041	----	----	----	----	----	----	----	----	----	----	
1067	----	----	----	----	----	----	----	----	----	----	
1108	D1078	64.1		-0.91	64.4		-0.38	64.7		-0.29	
1149	D1078	64.1		-0.91	64.2		-0.94	64.4		-1.50	
1201	D1078	64.4		-0.08	64.5		-0.11	64.7		-0.29	
1204	----	----	----	----	----	----	----	----	----	----	
1221	D1078	64.5		0.20	64.5		-0.11	64.8		0.11	
1239	----	----	----	----	----	----	----	----	----	----	
1246	D1078	0.4	G(0.01)	-178.37	64.4	excl.	-0.38	64.8		0.11	
1256	D1078	64.7		0.76	64.7		0.45	64.9		0.52	
1341	----	----	----	----	----	----	----	----	----	----	
1342	D1078	64.4		-0.08	64.6		0.17	65.2		1.73	
1343	----	----	----	----	----	----	----	----	----	----	
1344	D1078	64.3		-0.36	64.4		-0.38	64.7		-0.29	
1354	D1078	64.6		0.48	64.7		0.45	64.8		0.11	
1355	D1078	64.5		0.20	64.6		0.17	64.8		0.11	
1510	D1078	64.2		-0.64	64.3		-0.66	64.4		-1.50	

1538		-----								
1615	D1078	64.5	0.20	64.6	0.17	65.0	0.92			
1826		-----		-----		-----	-----			
1905	D1078	64.37	-0.16	64.52	-0.05	64.84	0.27			
normality		not OK		not OK		not OK				
n		38		37		38				
outliers		1		0	(+1 excl.)	0				
mean (n)		64.43		64.54		64.77				
st.dev. (n)		0.138		0.123		0.160				
R(calc.)		0.39		0.34		0.45				
R(D1078:05-A)		1.01		1.01		0.69				

Results after manual correction by iis for Mid Boiling Point (not present in below graphs)

lab	method	IBP	mark	z(targ)	MBP	mark	z(targ)	DP	mark	z(targ)	remarks
332	D1078	64.6			64.5			64.7		-0.26	
334	D1078	64.4			64.5			64.6		-0.66	
345	D1078	64.5			64.5			64.6		-0.66	
663	D1078	64.5			64.5			64.9		0.55	
823	D1078	64.3			64.5			64.8		0.14	
824	D1078	64.3			64.5			64.8		0.14	
1149	D1078	64.4			64.5			64.7		-0.26	
1256	D1078	64.5			64.5			64.7		-0.26	
1354	D1078	64.4			64.5			64.6		-0.66	
1510	D1078	64.4			64.5			64.6		-0.66	
normality		not OK			not OK			not OK			
n		38			38			38			
outliers		1			0			0			
mean (n)		64.42			64.53			64.76			
st.dev. (n)		0.102			0.060			0.129			
R(calc.)		0.29			0.17			0.36			
R(D1078:05-A)		1.01			1.01			0.69			



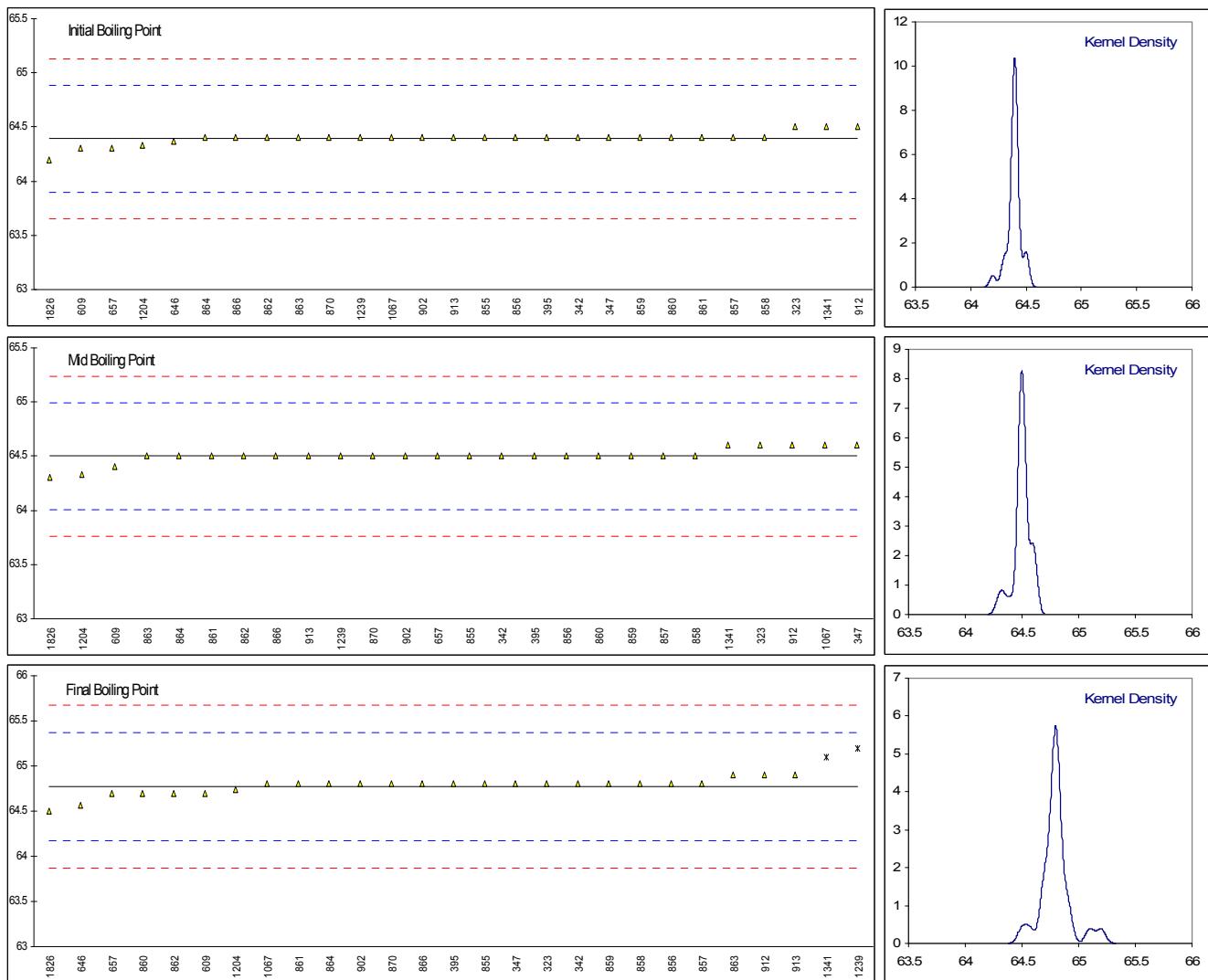
Determination of IBP, MBP and DP (manual) @ 760 mmHg on sample #1052; results in °C

Lab	method	IBP	mark	z(targ)	MBP	mark	z(targ)	DP	mark	z(targ)	Remarks
53		----		----			----			----	
150		----		----			----			----	
153		----		----			----			----	
171		----		----			----			----	
174		----		----			----			----	
311		----		----			----			----	
316		----		----			----			----	
319		----		----			----			----	
323	D1078	64.5		0.44	64.6		0.40	64.8		0.09	
332		----		----			----			----	
333		----		----			----			----	
334		----		----			----			----	
342	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
343		----		----			----			----	
344		----		----			----			----	
345		----		----			----			----	
346		----		----			----			----	
347	D1078	64.4		0.03	64.6		0.40	64.8		0.09	
357		----		----			----			----	
395	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
444		----		----			----			----	
446		----		----			----			----	
494		----		----			----			----	
497		----		----			----			----	
528		----		----			----			----	
529		----		----			----			----	
551		----		----			----			----	
608		----		----			----			----	
609	D1078	64.3		-0.38	64.4		-0.41	64.7		-0.24	
646	D1078	64.37		-0.09	----		----	64.57		-0.67	
657	D1078	64.3		-0.38	64.5		-0.01	64.7		-0.24	
663		----		----			----			----	
823		----		----			----			----	
824		----		----			----			----	
825		----		----			----			----	
840		----		----			----			----	
855	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
856	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
857	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
858	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
859	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
860	D1078	64.4		0.03	64.5		-0.01	64.7		-0.24	
861	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
862	D1078	64.4		0.03	64.5		-0.01	64.7		-0.24	
863	D1078	64.4		0.03	64.5		-0.01	64.9		0.42	
864	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
866	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
870	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
902	D1078	64.4		0.03	64.5		-0.01	64.8		0.09	
912	D1078	64.5		0.44	64.6		0.40	64.9		0.42	
913	D1078	64.4		0.03	64.5		-0.01	64.9		0.42	
963		----		----			----			----	
974		----		----			----			----	
1007		----		----			----			----	
1009		----		----			----			----	
1010		----		----			----			----	
1016		----		----			----			----	
1029		----		----			----			----	
1041		----		----			----			----	
1067	D1078	64.4		0.03	64.6		0.40	64.8		0.09	
1108		----		----			----			----	
1149		----		----			----			----	
1201		----		----			----			----	
1204	D1078	64.334		-0.24	64.334		-0.68	64.734		-0.13	
1221		----		----			----			----	
1239	D1078	64.4		0.03	64.5		-0.01	65.2	G(0.05)	1.42	
1246		----		----			----			----	
1256		----		----			----			----	
1341	D1078	64.5		0.44	64.6		0.40	65.1	G(0.05)	1.09	
1342		----		----			----			----	
1343		----		----			----			----	
1344		----		----			----			----	
1354		----		----			----			----	
1355		----		----			----			----	
1510		----		----			----			----	

1538	----	----	----	----	----	----	----
1615	----	----	----	----	----	----	----
1826	D1078	64.2	-0.78	64.3	-0.82	64.5	-0.90
1905	----	----	----	----	----	----	----
normality	not OK		not OK		not OK		
n	27		26		25		
outliers	0		0		2		
mean (n)	64.39		64.50		64.77		
st.dev. (n)	0.060		0.071		0.091		
R(calc.)	0.17		0.20		0.25		
R(D1078:05-M)	0.69		0.69		0.84		

Results after manual correction by iis for Mid Boiling Point (not present in below graphs)

lab	method	IBP	mark	z(targ)	MBP	mark	z(targ)	DP	mark	z(targ)	remarks
1204	D1078	64.5		0.38	64.5		-0.06	64.9		0.38	
1826	D1078	64.4		-0.03	64.5		-0.06	64.7		-0.29	
normality		not OK			not OK			not OK			
n		27			26			25			
outliers		0			0			2			
mean (n)		64.41			64.52			64.79			
st.dev. (n)		0.048			0.046			0.076			
R(calc.)		0.13			0.13			0.21			
R(D1078:05-M)		0.69			0.69			0.84			



Determination of Water Miscibility on sample #1052;

lab	method	value	mark	z(targ)	remarks
53	D1722	PASS	-----		
150	D1722	PASS	-----		
153	D1722	PASS	-----		
171	D1722	PASS	-----		
174	D1722	PASS	-----		
311	D1722	PASS	-----		
316		-----	-----		
319	D1722	PASS	-----		
323	D1722	PASS	-----		
332	D1722	PASS	-----		
333	D1722	PASS	-----		
334		-----	-----		
342	D1722	PASS	-----		
343	D1722	PASS	-----		
344	D1722	PASS	-----		
345	D1722	PASS	-----		
346		-----	-----		
347	D1722	PASS	-----		
357	D1722	PASS	-----		
395	D1722	PASS	-----		
444	D1722	PASS	-----		
446	D1722	PASS	-----		
494	D1722	PASS	-----		
497	D1722	PASS	-----		
528		-----	-----		
529		-----	-----		
551		-----	-----		
608		-----	-----		
609	D1722	PASS	-----		
646	D1722	FAIL	-----		
657	D1722	PASS	-----		
663	D1722	PASS	-----		
823	D1722	PASS	-----		
824	D1722	PASS	-----		
825	D1722	PASS	-----		
840		-----	-----		
855	D1722	PASS	-----		
856	D1722	PASS	-----		
857	D1722	PASS	-----		
858	D1722	PASS	-----		
859	D1722	PASS	-----		
860	D1722	PASS	-----		
861	D1722	PASS	-----		
862	D1722	PASS	-----		
863	D1722	PASS	-----		
864	D1722	PASS	-----		
866	D1722	PASS	-----		
870	D1722	PASS	-----		
902	D1722	PASS	-----		
912	D1722	PASS	-----		
913	D1722	PASS	-----		
963	D1722	PASS	-----		
974	D1722	PASS	-----		
1007	in house	PASS	-----		
1009	D1722	PASS	-----		
1010	D1722	PASS	-----		
1016	D1722	PASS	-----		
1029	D1722	PASS	-----		
1041	D1722	PASS	-----		
1067	D1722	PASS	-----		
1108		-----	-----		
1149	D1722	PASS	-----		
1201	D1722	PASS	-----		
1204	D1722	PASS	-----		
1221		-----	-----		
1239	D1722	PASS	-----		
1246		-----	-----		
1256	D1722	PASS	-----		
1341	D1722	PASS	-----		
1342	D1722	PASS	-----		
1343		-----	-----		
1344	D1722	PASS	-----		
1354	D1722	PASS	-----		

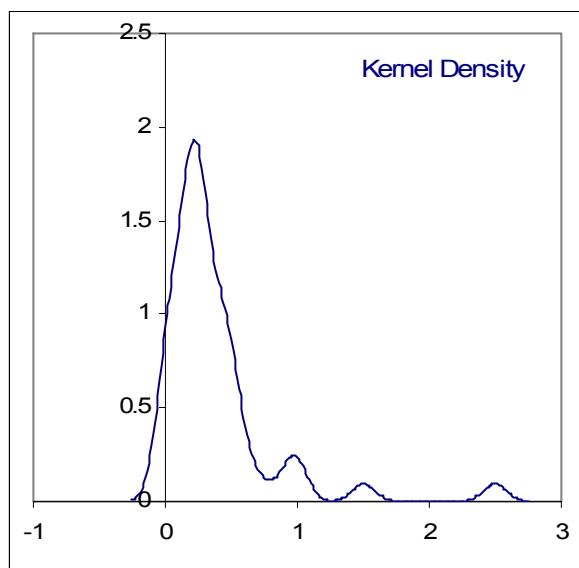
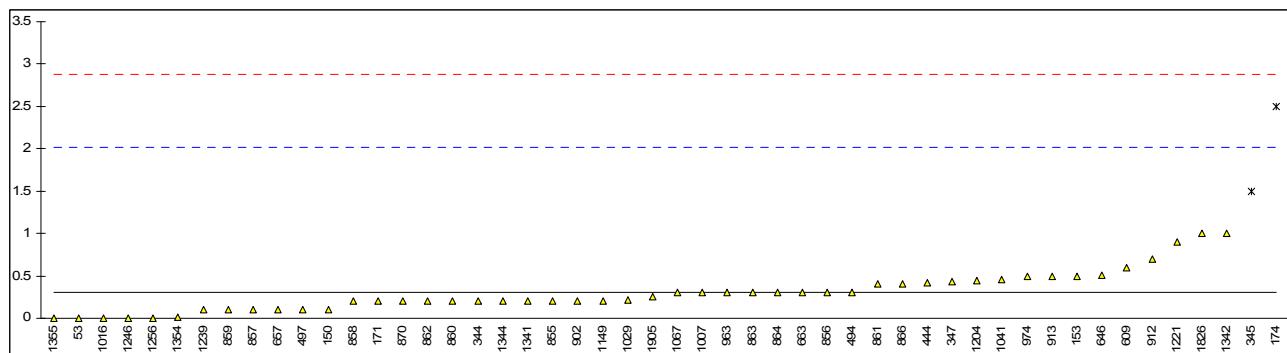
1355	D1722	PASS	-----
1510	D1722	PASS	-----
1538		-----	-----
1615	D1722	PASS	-----
1826	D1722	PASS	-----
1905	D1722	PASS	-----

n	65
mean (n)	PASS
R(D1722:09)	n.a.

Determination of Nonvolatile Matter on sample #1052; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
53	D1353	0	excl.	-0.37	zero is not a true value
150	D1353	0.1		-0.25	
153	D1353	0.5		0.22	
171	D1353	0.2		-0.13	
174	D1353	2.5	G(0.01)	2.55	
311	D1353	<1		----	
316		----		----	
319	D1353	<8		----	
323	D1353	<1		----	
332		----		----	
333	D1353	<0.1		----	
334		----		----	
342		----		----	
343		----		----	
344	D1353	0.2		-0.13	
345	D1353	1.5	G(0.01)	1.38	
346		----		----	
347	D1353	0.43		0.13	
357	D1353	<1		----	
395		----		----	
444	D1353	0.424		0.13	
446		----		----	
494	D1353	0.3		-0.02	
497	D1353	0.1		-0.25	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609	D1353	0.6		0.33	
646	D1353	0.51		0.23	
657	D1353	0.1		-0.25	
663	D1353	0.3		-0.02	
823		----		----	
824		----		----	
825		----		----	
840		----		----	
855	D1353	0.2		-0.13	
856	D1353	0.3		-0.02	
857	D1353	0.1		-0.25	
858	D1353	0.2		-0.13	
859	D1353	0.1		-0.25	
860	D1353	0.2		-0.13	
861	D1353	0.4		0.10	
862	D1353	0.2		-0.13	
863	D1353	0.3		-0.02	
864	D1353	0.3		-0.02	
866	D1353	0.4		0.10	
870	D1353	0.2		-0.13	
902	D1353	0.2		-0.13	
912	D1353	0.7		0.45	
913	D1353	0.5		0.22	
963	D1353	0.3		-0.02	
974	D1353	0.5		0.22	
1007	in house	0.3		-0.02	
1009	D1353	<1		----	
1010	D1353	<0.8		----	
1016	D1353	0.0002		-0.37	
1029	D1353	0.22		-0.11	
1041	D1353	0.46		0.17	
1067	D1353	0.3		-0.02	
1108		----		----	
1149	D1353	0.20		-0.13	
1201	D1353	<1		----	
1204	D1353	0.45		0.16	
1221	D1353	0.9		0.68	
1239	D1353	0.1		-0.25	
1246	D1353	0.0004		-0.37	
1256	D1353	0.0026		-0.36	
1341	D1353	0.2		-0.13	
1342	D1353	1		0.80	
1343		----		----	
1344	D1353	0.2		-0.13	
1354	D1353	0.010		-0.36	

1355	D1353	0	excl.	-0.37	zero is not a true value
1510		----		----	
1538		----		----	
1615	D1353	<1.0		----	
1826	D1353	1		0.80	
1905	D1353	0.25		-0.08	
 normality not OK					
n		46			
outliers		2			
mean (n)		0.31			
st.dev. (n)		0.238			
R(calc.)		0.67			
R(D1353:09)		2.40			



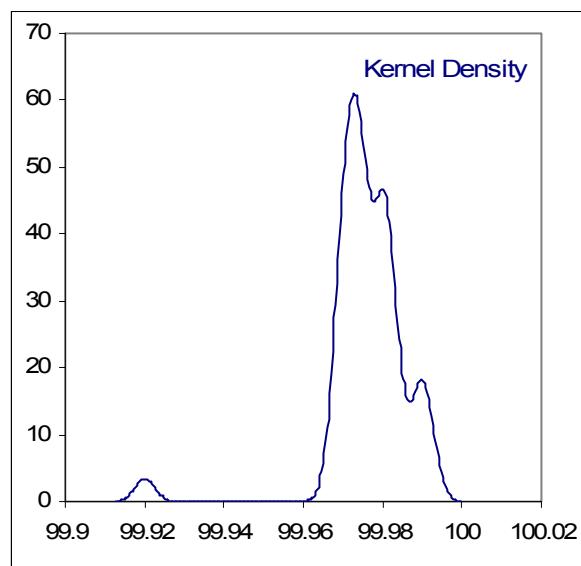
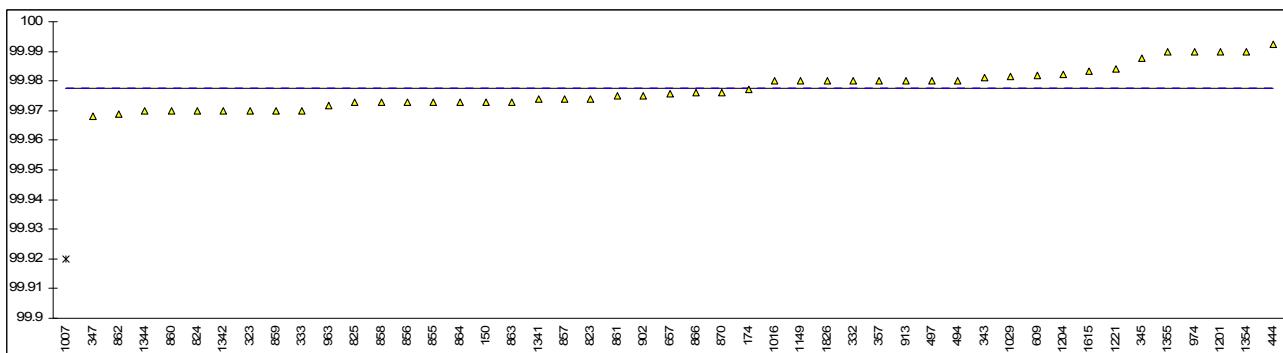
Determination of Purity "as received" on sample #1052; results in %M/M

lab	method	value	mark	z(targ)	remarks
53		----		----	
150		99.973		----	
153		----		----	
171		----		----	
174	IMPCA001	99.977		----	
311		----		----	
316		----		----	
319		----		----	
323	inh-064	99.97		----	
332		99.98		----	
333		99.97		----	
334		----		----	
342		----		----	
343	calc.	99.981		----	
344		----		----	
345	inh-40	99.9875		----	
346		----		----	
347	IMPCA001	99.968		----	
357		99.98		----	
395		----		----	
444		99.9924		----	
446		----		----	
494		99.98		----	
497	INH1460	99.98		----	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609	calc.	99.982		----	
646		----		----	
657	calc.	99.9756		----	
663		----		----	
823	IMPCA001	99.974		----	
824		99.97		----	
825		99.973		----	
840		----		----	
855	IMPCA001	99.973		----	
856	IMPCA001	99.973		----	
857		99.974		----	
858	IMPCA001Mod.	99.973		----	
859		99.970		----	
860		99.970		----	
861	IMPCA001	99.975		----	
862		99.969		----	
863	IMPCA001	99.973		----	
864	IMPCA001	99.973		----	
866	IMPCA001	99.976		----	
870	IMPCA001	99.976		----	
902		99.975		----	
912		----		----	
913	IMPCA001	99.98		----	
963	IMPCA001Mod.	99.9716		----	
974	IMPCA001	99.99		----	
1007	in house	99.92	G(0.01)	----	
1009		----		----	
1010		----		----	
1016		99.98		----	
1029		99.98135		----	
1041		----		----	
1067		----		----	
1108		----		----	
1149		99.98		----	
1201		99.99		----	
1204	calc.	99.9821		----	
1221		99.984		----	
1239		----		----	
1246		----		----	
1256		----		----	
1341		99.9738		----	
1342		99.97		----	
1343		----		----	
1344		99.97		----	
1354		99.99		----	

1355	99.99
1510	----
1538	----
1615	calc.
1826	in house
1905	----

normality	not OK
n	46
outliers	1
mean (n)	99.977
st.dev. (n)	0.0066
R(calc.)	0.018
R(lit.)	unknown

Compare R(iis09C07) = 0.017



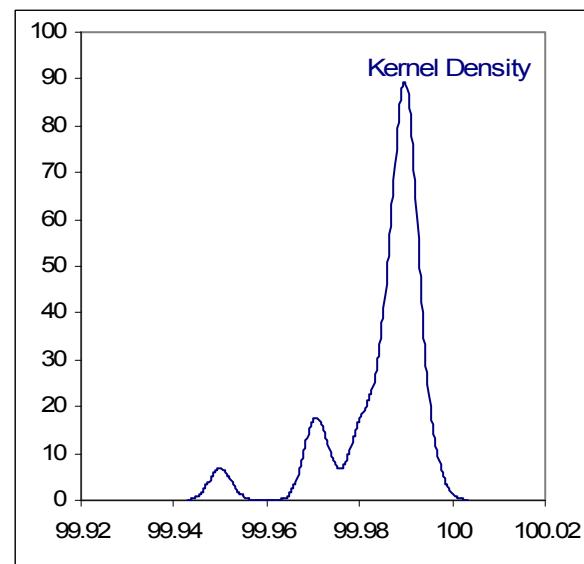
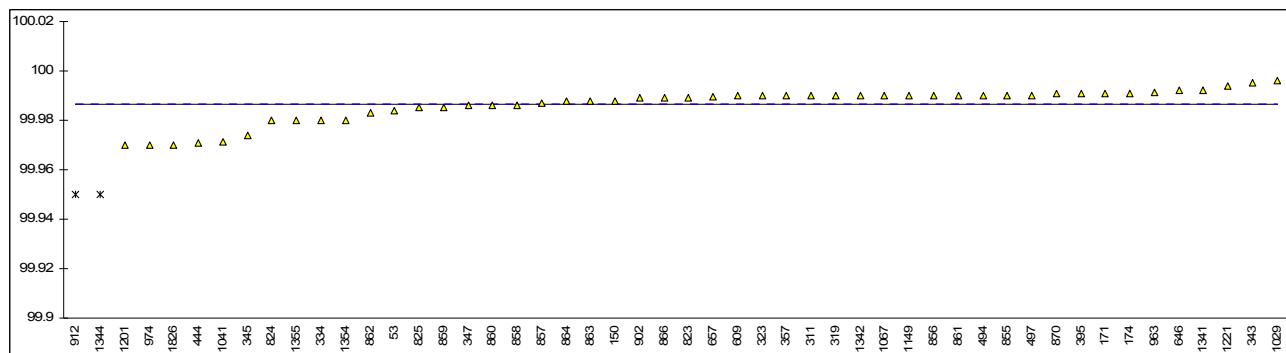
Determination of Purity on dry basis on sample #1052; results in %M/M

lab	method	value	mark	z(targ)	remarks
53	IMPCA001	99.984		----	
150	IMPCA001	99.988		----	
153		-----		-----	
171	IMPCA001	99.991		----	
174	IMPCA001	99.991		----	
311	IMPCA001	99.99		----	
316		-----		-----	
319	IMPCA001	99.99		----	
323	IMPCA001	99.99		----	
332		-----		-----	
333		-----		-----	
334	IMPCA001	99.98		----	
342		-----		-----	
343	IMPCA001	99.995		----	
344		-----		-----	
345	INH-40	99.9738		----	
346		-----		-----	
347	IMPCA001	99.986		----	
357	IMPCA001	99.99		----	
395	IMPCA001	99.991		----	
444	IMPCA001	99.9707		----	
446		-----		-----	
494	IMPCA001	99.99		----	
497	IMPCA001	99.99		----	
528		-----		-----	
529		-----		-----	
551		-----		-----	
608		-----		-----	
609	IMPCA001	99.99		----	
646	IMPCA001	99.992		----	
657	IMPCA001	99.9896		----	
663		-----		-----	
823	IMPCA001	99.989		----	
824	IMPCA001	99.98		----	
825	IMPCA001	99.985		----	
840		-----		-----	
855	IMPCA001	99.990		----	
856	IMPCA001	99.990		----	
857	IMPCA001	99.987		----	
858	IMPCA001	99.986		----	
859	IMPCA001	99.985		----	
860	IMPCA001	99.986		----	
861	IMPCA001	99.990		----	
862	IMPCA001	99.983		----	
863	IMPCA001	99.988		----	
864	IMPCA001	99.988		----	
866	IMPCA001	99.989		----	
870	IMPCA001	99.991		----	
902	IMPCA001	99.989		----	
912	IMPCA001	99.95	G(0.01)	----	
913		-----		-----	
963	IMPCA001	99.9913		----	
974	IMPCA001	99.97		----	
1007		-----		-----	
1009		-----		-----	
1010	IMPCA001	>99.99		----	
1016		-----		-----	
1029	IMPCA001	99.99617		----	
1041	IMPCA001	99.9714		----	
1067	IMPCA001	99.99		----	
1108		-----		-----	
1149	IMPCA001	99.99		----	
1201	IMPCA001	99.97		----	
1204		-----		-----	
1221	IMPCA001	99.994		----	
1239	IMPCA001	>99.9		----	
1246		-----		-----	
1256		-----		-----	
1341	IMPCA001	99.9921		----	
1342	IMPCA001	99.99		----	
1343		-----		-----	
1344	IMPCA001	99.95	G(0.01)	----	
1354	IMPCA001	99.98		----	

1355	IMPCA001	99.98	-----
1510		-----	-----
1538		-----	-----
1615		-----	-----
1826	in house	99.97	-----
1905		-----	-----

normality	not OK
n	48
outliers	2
mean (n)	99.986
st.dev. (n)	0.0069
R(calc.)	0.019
R(lit.)	unknown

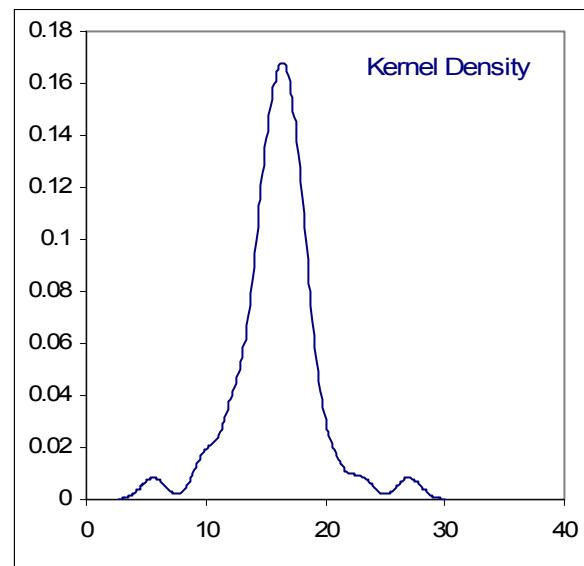
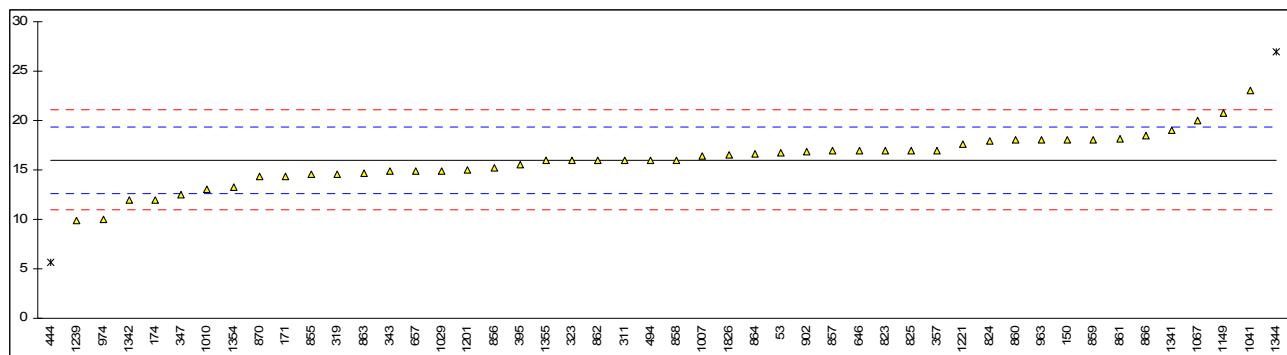
Compare R(iis09C07) = 0.020



Determination of Acetone content on sample #1052; results in mg/kg

lab	method	value	mark	z(targ)	remarks
53	IMPCA001	16.75		0.44	
150	IMPCA001	18		1.18	
153		----		----	
171	IMPCA001	14.3		-1.02	
174	IMPCA001	12		-2.38	
311	IMPCA001	16		-0.01	
316		----		----	
319	IMPCA001	14.6		-0.84	
323	IMPCA001	16		-0.01	
332		----		----	
333		----		----	
334		----		----	
342		----		----	
343	IMPCA001	14.9		-0.66	
344	D1612	<30		----	
345		----		----	
346		----		----	
347	IMPCA001	12.5		-2.08	
357	IMPCA001	17		0.58	
395	IMPCA001	15.51		-0.30	
444	IMPCA001	5.6	C,G(0.05)	-6.17	first reported 4.17
446		----		----	
494	IMPCA001	16		-0.01	
497		----		----	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609	E346	<30		----	
646	IMPCA001	17		0.58	
657	IMPCA001	14.91		-0.65	
663		----		----	
823	IMPCA001	17		0.58	
824	IMPCA001	17.9		1.12	
825	IMPCA001	17		0.58	
840		----		----	
855	IMPCA001	14.6		-0.84	
856	IMPCA001	15.2		-0.48	
857	IMPCA001	17		0.58	
858	IMPCA001	16		-0.01	
859	IMPCA001	18		1.18	
860	IMPCA001	18		1.18	
861	IMPCA001	18.2		1.29	
862	IMPCA001	16		-0.01	
863	IMPCA001	14.7		-0.78	
864	IMPCA001	16.6		0.35	
866	IMPCA001	18.5		1.47	
870	IMPCA001	14.3		-1.02	
902	IMPCA001	16.8		0.47	
912		----		----	
913		----		----	
963	IMPCA001	18		1.18	
974	IMPCA001	10		-3.56	
1007	in house	16.4		0.23	
1009		----	C	----	first reported 32
1010	IMPCA001	13		-1.79	
1016		----		----	
1029	IMPCA001	14.94		-0.64	
1041	IMPCA001	22.99		4.13	
1067	IMPCA001	20		2.36	
1108		----		----	
1149	IMPCA001	20.74		2.80	
1201	IMPCA001	15		-0.60	
1204	D1612	>30		>8.29	False positive
1221	IMPCA001	17.66		0.97	
1239	IMPCA001	9.9		-3.62	
1246	E346	>30		>8.29	False positive
1256		----		----	
1341	IMPCA001	19.0024		1.77	
1342	IMPCA001	12		-2.38	
1343		----		----	
1344	IMPCA001	27	G(0.05)	6.51	
1354	IMPCA001	13.28		-1.62	

1355	IMPCA001	15.99	-0.01	
1510		----	----	
1538		----	----	
1615	D1612	>30	>8.29	False positive
1826	in house	16.5	0.29	
1905	INH-232	<30	----	
	normality	OK		
	n	46		
	outliers	2		
	mean (n)	16.01	<u>Spike</u>	
	st.dev. (n)	2.527		
	R(calc.)	7.08		
	R(Horwitz)	4.73		

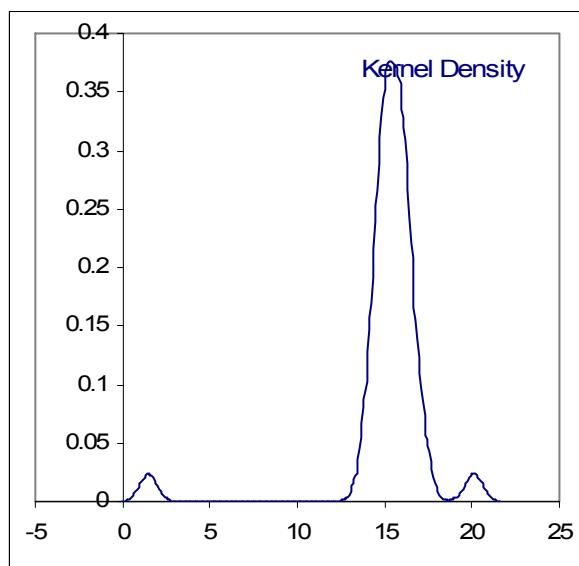
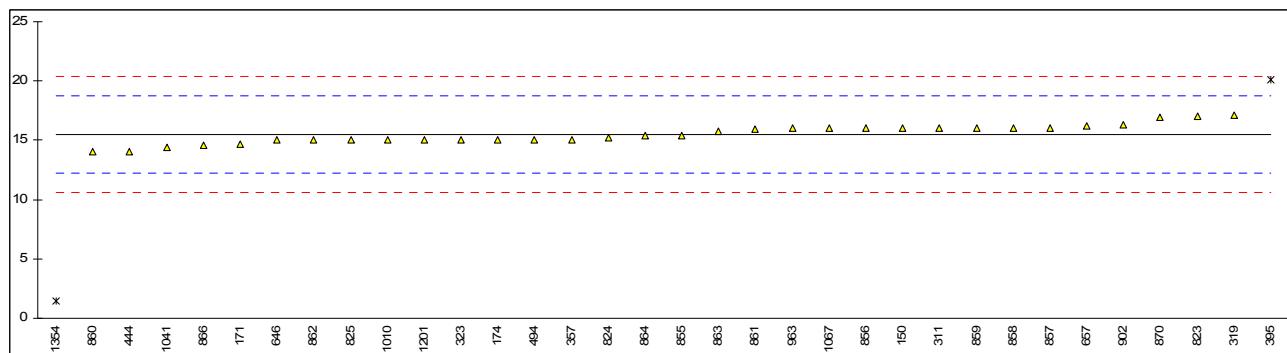


Determination of Benzene content on sample #1052; results in mg/kg

lab	method	value	mark	z(targ)	remarks
53		----		----	
150	IMPCA001	16		0.31	
153		----		----	
171	IMPCA001	14.7		-0.48	
174	IMPCA001	15		-0.30	
311	IMPCA001	16		0.31	
316		----		----	
319	INH-064	17.1		0.98	
323	inh-064	15		-0.30	
332		----		----	
333		----		----	
334		----		----	
342		----		----	
343		----		----	
344		----		----	
345		----		----	
346		----		----	
347		----		----	
357	IMPCA001	15		-0.30	
395	IMPCA001	20.11	G(0.01)	2.81	
444	IMPCA001	14.0	C	-0.91	first reported 6.95
446		----		----	
494	IMPCA001	15		-0.30	
497		----		----	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609		----		----	
646	IMPCA001	15		-0.30	
657	IMPCA001	16.17		0.41	
663		----		----	
823	IMPCA001	17		0.92	
824	IMPCA001	15.2		-0.18	
825	IMPCA001	15		-0.30	
840		----		----	
855	IMPCA001	15.4		-0.06	
856	IMPCA001	16.0		0.31	
857	IMPCA001	16		0.31	
858	IMPCA001	16		0.31	
859	IMPCA001	16		0.31	
860	IMPCA001	14		-0.91	
861	IMPCA001	15.9		0.25	
862	IMPCA001	15		-0.30	
863	INH-043	15.8		0.19	
864	IMPCA001	15.4		-0.06	
866	IMPCA001	14.6		-0.55	
870	IMPCA001	16.9		0.86	
902	IMPCA001	16.3		0.49	
912		----		----	
913		----		----	
963	IMPCA001	16		0.31	
974		----		----	
1007		----		----	
1009		----		----	
1010	IMPCA001	15		-0.30	
1016		----		----	
1029		----		----	
1041	IMPCA001	14.37		-0.69	
1067	IMPCA001	16		0.31	
1108		----		----	
1149		----		----	
1201	IMPCA001	15		-0.30	
1204		----		----	
1221		----		----	
1239		----		----	
1246		----		----	
1256		----		----	
1341		----		----	
1342	IMPCA001	<10		<-3.35	False negative
1343		----		----	
1344	IMPCA001	<10		<-3.35	False negative
1354	IMPCA001	1.47	G(0.01)	-8.55	

1355		-----	
1510		-----	
1538		-----	
1615		-----	
1826	in house	<1	<-8.83 False negative
1905		-----	

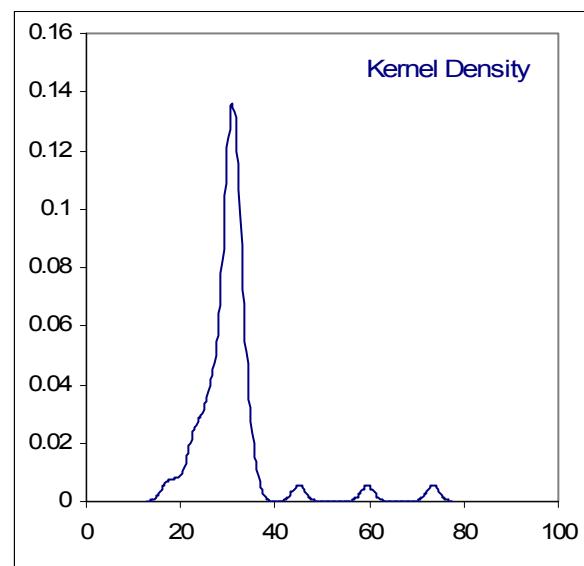
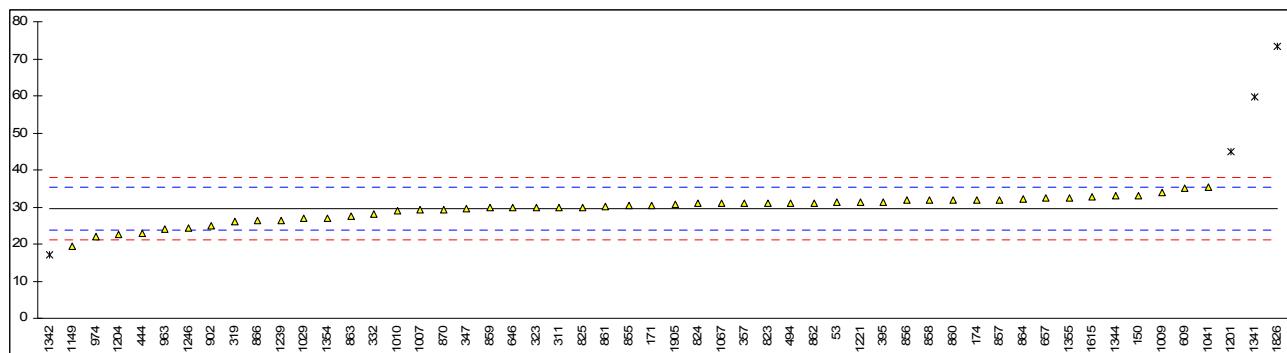
normality	not OK
n	32
outliers	2
mean (n)	15.50
st.dev. (n)	0.797
R(calc.)	2.23
R(Horwitz)	4.60

Spike

Determination of Ethanol content on sample #1052; results in mg/kg

lab	method	value	mark	z(targ)	remarks
53	IMPCA001	31.22		0.59	
150	IMPCA001	33	C	1.22	first reported 39
153		----		----	
171	IMPCA001	30.4		0.30	
174	IMPCA001	32		0.86	
311	IMPCA001	30		0.16	
316		----		----	
319	IMPCA001	26.2		-1.18	
323	IMPCA001	30		0.16	
332	IMPCA001	28		-0.54	
333		----		----	
334		----		----	
342		----		----	
343	IMPCA001	<50		----	
344		----		----	
345		----		----	
346		----		----	
347	IMPCA001	29.5		-0.02	
357	IMPCA001	31		0.51	
395	IMPCA001	31.32		0.62	
444	IMPCA001	22.8		-2.38	
446		----		----	
494	IMPCA001	31		0.51	
497		----		----	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609	E346	35		1.92	
646	IMPCA001	30		0.16	
657	IMPCA001	32.41		1.01	
663		----		----	
823	IMPCA001	31		0.51	
824	IMPCA001	30.9		0.48	
825	IMPCA001	30		0.16	
840		----		----	
855	IMPCA001	30.3		0.27	
856	IMPCA001	31.9		0.83	
857	IMPCA001	32		0.86	
858	IMPCA001	32		0.86	
859	IMPCA001	30		0.16	
860	IMPCA001	32		0.86	
861	IMPCA001	30.1		0.20	
862	IMPCA001	31		0.51	
863	IMPCA001	27.6		-0.68	
864	IMPCA001	32.3		0.97	
866	IMPCA001	26.3		-1.14	
870	IMPCA001	29.4		-0.05	
902	IMPCA001	25		-1.60	
912		----		----	
913		----		----	
963	IMPCA001	24		-1.95	
974	IMPCA001	22		-2.66	
1007	in house	29.4		-0.05	
1009	IMPCA001	34		1.57	
1010	IMPCA001	29		-0.19	
1016		----		----	
1029	IMPCA001	27.03		-0.89	
1041	IMPCA001	35.27		2.02	
1067	IMPCA001	31		0.51	
1108		----		----	
1149	IMPCA001	19.36		-3.59	
1201	IMPCA001	45	G(0.01)	5.44	
1204	E346	22.47		-2.49	
1221	IMPCA001	31.32		0.62	
1239	IMPCA001	26.5		-1.07	
1246	E346	24.21		-1.88	
1256		----		----	
1341	IMPCA001	59.6566	G(0.01)	10.60	
1342	IMPCA001	17	G(0.05)	-4.42	
1343		----		----	
1344	IMPCA001	33		1.22	
1354	IMPCA001	27.05		-0.88	

1355	IMPCA001	32.45		1.02
1510		----		----
1538		----		----
1615	IMPCA001	32.85		1.16
1826	in house	73.4	G(0.01)	15.44
1905	in house	30.7		0.41
	normality	not OK		
	n	50		
	outliers	4	Spike	
	mean (n)	29.55		25.3
	st.dev. (n)	3.448		
	R(calc.)	9.66		
	R(Horwitz)	7.95		

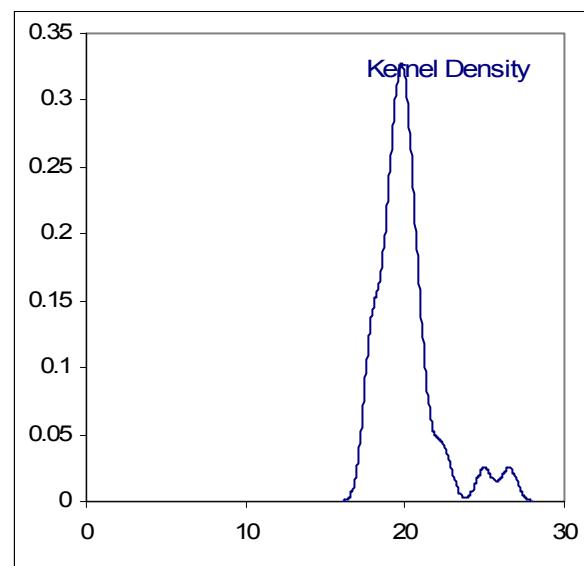
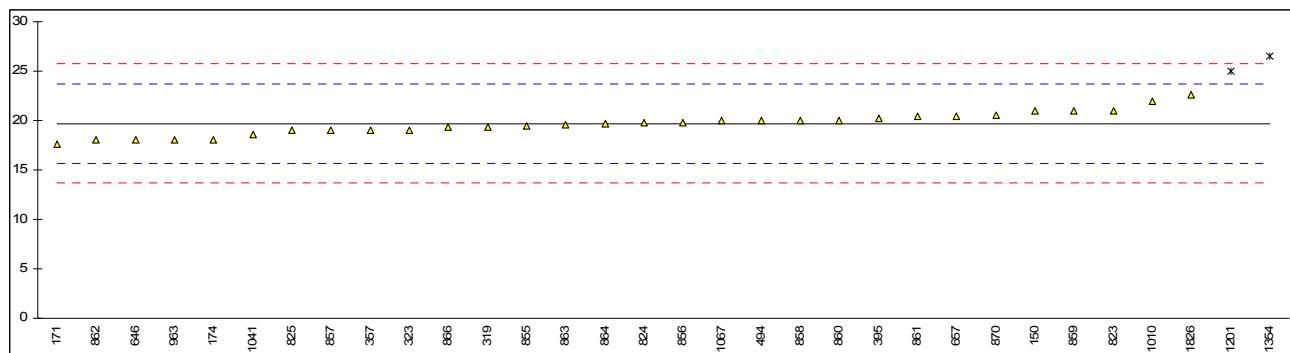


Determination of Toluene content on sample #1052; results in mg/kg

lab	method	value	mark	z(targ)	remarks
53		----		----	
150	IMPCA001	21		0.65	
153		----		----	
171	IMPCA001	17.6		-1.04	
174	IMPCA001	18		-0.84	
311	IMPCA001	<10		<-4.81	False negative
316		----		----	
319	IMPCA001	19.4		-0.14	
323	inh-064	19		-0.34	
332		----		----	
333		----		----	
334		----		----	
342		----		----	
343		----		----	
344		----		----	
345		----		----	
346		----		----	
347		----		----	
357	IMPCA001	19		-0.34	
395	IMPCA001	20.22		0.27	
444		----		----	
446		----		----	
494	IMPCA001	20		0.16	
497		----		----	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609		----		----	
646	IMPCA001	18		-0.84	
657	IMPCA001	20.44		0.38	
663		----		----	
823	IMPCA001	21		0.65	
824	IMPCA001	19.8		0.06	
825	IMPCA001	19		-0.34	
840		----		----	
855	IMPCA001	19.5		-0.09	
856	IMPCA001	19.8		0.06	
857	IMPCA001	19		-0.34	
858	IMPCA001	20		0.16	
859	IMPCA001	21		0.65	
860	IMPCA001	20		0.16	
861	IMPCA001	20.4		0.36	
862	IMPCA001	18		-0.84	
863	INH-043	19.6		-0.04	
864	IMPCA001	19.7		0.01	
866	IMPCA001	19.3		-0.19	
870	IMPCA001	20.5		0.41	
902		----		----	
912		----		----	
913		----		----	
963	IMPCA001	18		-0.84	
974		----		----	
1007		----		----	
1009		----		----	
1010	IMPCA001	22		1.15	
1016		----		----	
1029		----		----	
1041	IMPCA001	18.64		-0.52	
1067	IMPCA001	20		0.16	
1108		----		----	
1149		----		----	
1201	IMPCA001	25	G(0.01)	2.64	
1204		----		----	
1221		----		----	
1239		----		----	
1246		----		----	
1256		----		----	
1341		----		----	
1342	IMPCA001	<10		<-4.81	False negative
1343		----		----	
1344	IMPCA001	<10		<-4.81	False negative
1354	IMPCA001	26.54	G(0.01)	3.41	

1355		-----	
1510		-----	
1538		-----	
1615		-----	
1826	in house	22.6	1.45
1905		-----	

normality OK
 n 30
 outliers 2 **Spike**
 mean (n) 19.68 19.8
 st.dev. (n) 1.181
 R(calc.) 3.30
 R(Horwitz) 5.63



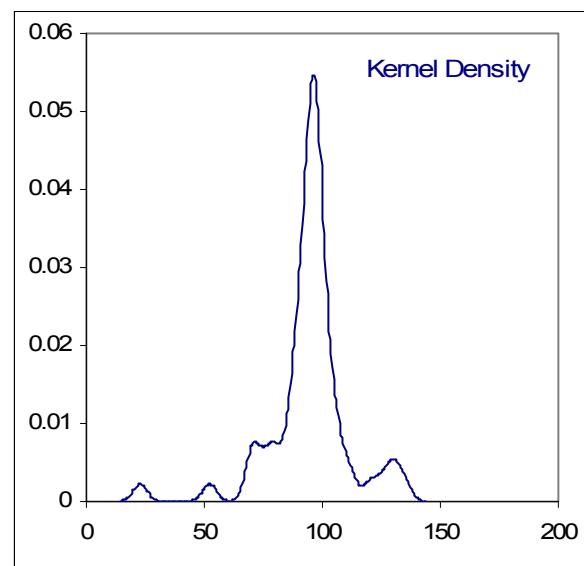
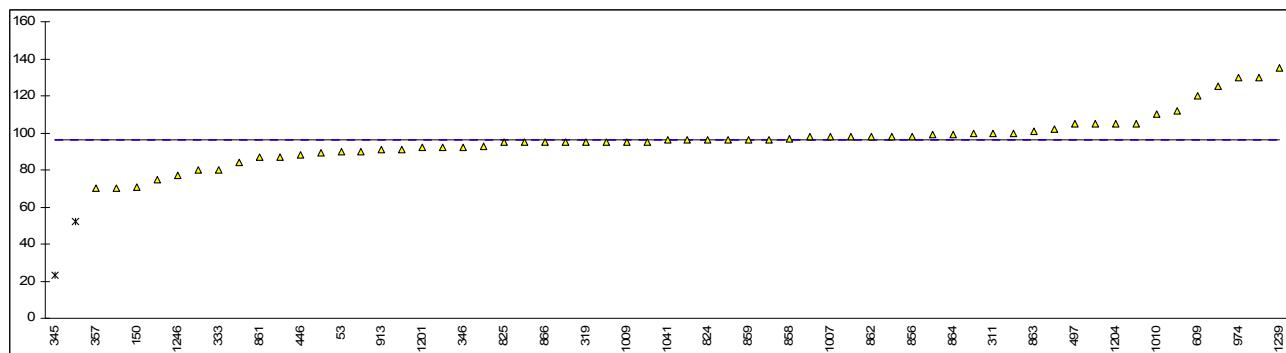
Determination of Permanganate Time Test @ 15°C on sample #1052; results in minutes

lab	method	value	mark	z(targ)	remarks
53	D1363	90		----	
150	D1363	71		----	
153	D1363	70		----	
171	D1363	95		----	
174	D1363	98		----	
311	D1363	100		----	
316		----		----	
319	D1363	95		----	
323		----		----	
332		----		----	
333	D1363	80		----	
334		----		----	
342	D1363	96		----	
343	D1363	>50		----	
344	D1362	52.15	G(0.01)	----	
345	D1363	23	G(0.01)	----	
346	D1363	92		----	
347	D1363	100		----	
357	D1363	70		----	
395	D1363	130		----	
444	D1363	95		----	
446	D1363	88		----	
494	D1363	91		----	
497	D1363	105		----	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609	D1363	120		----	
646	D1363	102		----	
657	D1363	112		----	
663	D1363	75		----	
823	D1363	105		----	
824	D1363	96		----	
825	D1363	95		----	
840		----		----	
855	D1363	98		----	
856	D1363	98		----	
857	D1363	96		----	
858	D1363	97		----	
859	D1363	96		----	
860	D1363	96		----	
861	D1363	87		----	
862	D1363	98		----	
863	D1363	101		----	
864	D1363	99		----	
866	D1363	95		----	
870	D1363	98		----	
902	D1363	84		----	
912	D1363	95		----	
913	D1363	91.0		----	
963	D1363	>50		----	
974	D1363	130		----	
1007	in house	98		----	
1009	D1363	95		----	
1010	D1363	110		----	
1016		----		----	
1029	D1363	100		----	
1041	D1363	96		----	
1067	D1363	99		----	
1108		----		----	
1149	D1363	92		----	
1201	D1363	92		----	
1204	D1363	105		----	
1221	D1363	87		----	
1239	D1363	135		----	
1246	D1363	77		----	
1256	D1363	>30		----	
1341	D1363	125		----	
1342	D1363	105		----	
1343		----		----	
1344	D1363	95		----	
1354	D1363	80		----	

1355	D1363	89	-----
1510		-----	-----
1538		-----	-----
1615	D1363	>60	-----
1826	D1363	93	-----
1905	D1363	90	-----

normality not OK
 n 59
 outliers 2
 mean (n) 96.5
 st.dev. (n) 13.17
 R(calc.) 36.9
 R(D1363:06) unknown

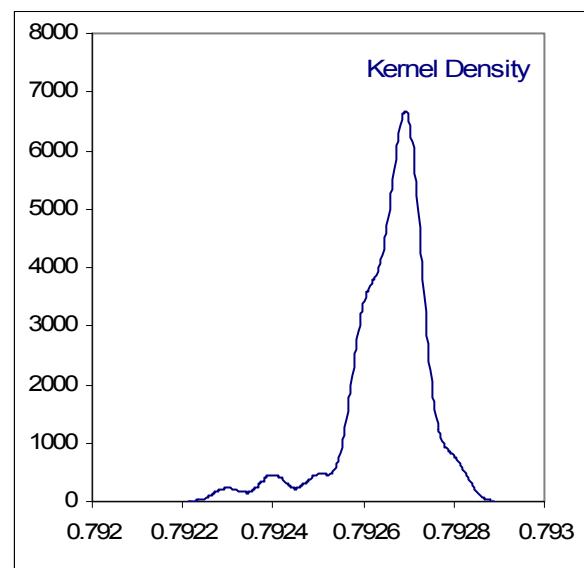
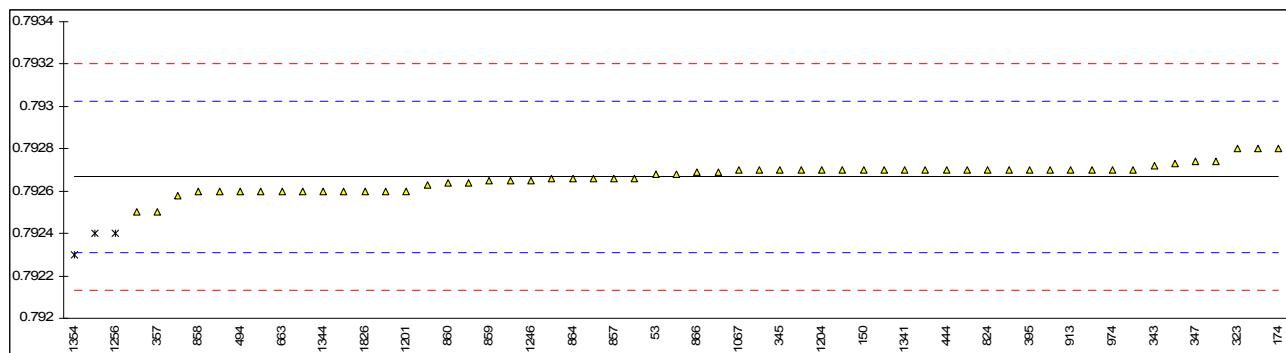
Compare R(iis09C07) = 33.0 at a mean of 106.2



Determination of Specific Gravity 20/20 °C/°C on sample #1052;

lab	method	value	mark	z(targ)	remarks
53		0.79268		0.07	
150	D4052	0.7927		0.18	
153		-----		-----	
171	D4052	0.79268		0.07	
174	D4052	0.7928		0.74	
311		0.7927		0.18	
316		-----		-----	
319	D4052	0.79264		-0.16	
323	D891A	0.7928		0.74	
332		-----		-----	
333		0.7928		0.74	
334		-----		-----	
342	D4052	0.7925		-0.94	
343		0.79272	C	0.29	first reported 0.79321
344		0.7927		0.18	
345		0.7927		0.18	
346		-----		-----	
347	D4052	0.79274		0.40	
357	D4052	0.7925		-0.94	
395	D4052	0.7927		0.18	
444		0.7927		0.18	
446	D4052	0.7926		-0.38	
494		0.7926		-0.38	
497	INH-1401	0.79269		0.12	
528		-----		-----	
529		-----		-----	
551		-----		-----	
608		-----		-----	
609	D4052	0.7927		0.18	
646		-----		-----	
657	D4052	0.7927		0.18	
663	D4052	0.7926		-0.38	
823	D4052	0.7926		-0.38	
824	D4052	0.7927		0.18	
825	D4052	0.7927		0.18	
840		-----		-----	
855	D4052cal.	0.79266		-0.05	
856		-----		-----	
857		0.79266		-0.05	
858	D4052	0.7926		-0.38	
859	D4052	0.79265		-0.10	
860	D4052	0.79264		-0.16	
861	D4052	0.79265		-0.10	
862	D4052	0.79266		-0.05	
863	D4052cal.	0.79273		0.35	
864	D4052cal.	0.79266		-0.05	
866	D4052	0.79269		0.12	
870	D4052cal.	0.79266		-0.05	
902	D4052	0.79274		0.40	
912	D4052	0.7927		0.18	
913	D4052	0.7927		0.18	
963	D4052	0.7927		0.18	
974	D4052	0.7927		0.18	
1007	in house	0.7927		0.18	
1009	D4052	0.7926		-0.38	
1010		0.7927		0.18	
1016		-----		-----	
1029	D4052	0.79263		-0.21	
1041		-----		-----	
1067		0.7927		0.18	
1108		-----		-----	
1149		-----		-----	
1201		0.7926		-0.38	
1204	D4052	0.7927		0.18	
1221	D4052	0.79270		0.18	
1239		0.7926		-0.38	
1246		0.79265		-0.10	
1256		0.7924	G(0.01)	-1.50	
1341	D4052	0.7927		0.18	
1342	D4052	0.7924	G(0.05)	-1.50	
1343		-----		-----	
1344		0.7926		-0.38	
1354	D4052	0.7923	G(0.01)	-2.06	

1355	D4052	0.7926	C	-0.38	first reported 0.7923
1510		-----		-----	
1538		-----		-----	
1615	D4052	0.79258		-0.49	
1826		0.7926		-0.38	
1905		-----		-----	
 normality not OK					
n		56			
outliers		3			
mean (n)		0.79267			
st.dev. (n)		0.000062			
R(calc.)		0.00017			
R(D4052:02e1)		0.00050			

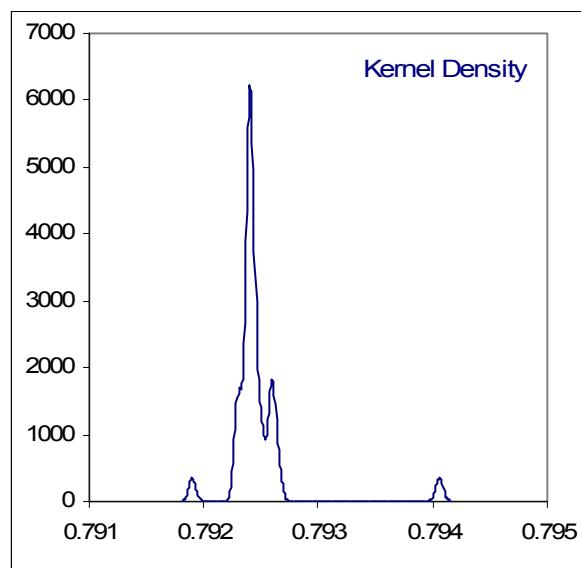
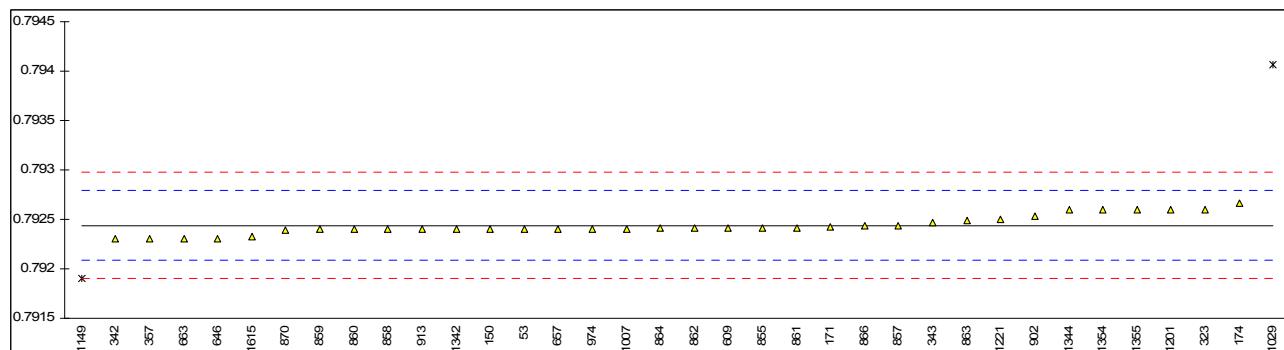


Determination of Specific Gravity, Apparent 20/20 °C/°C on sample #1052;

lab	method	value	mark	z(targ)	remarks
53		0.7924		-0.22	
150	D4052	0.7924	C	-0.22	first reported 0.7915
153		-----		-----	
171	D4052	0.79242		-0.11	
174	D4052	0.79266		1.24	
311		-----		-----	
316		-----		-----	
319		-----		-----	
323	D891A	0.7926		0.90	
332		-----		-----	
333		-----		-----	
334		-----		-----	
342	D4052	0.7923		-0.78	
343		0.79247	C	0.17	first reported 0.79296
344		-----		-----	
345		-----		-----	
346		-----		-----	
347		-----		-----	
357	D4052calc	0.7923		-0.78	
395		-----		-----	
444		-----		-----	
446		-----		-----	
494		-----		-----	
497		-----		-----	
528		-----		-----	
529		-----		-----	
551		-----		-----	
608		-----		-----	
609	D4052	0.79241		-0.16	
646	D891	0.7923		-0.78	
657	D4052	0.7924		-0.22	
663	D4052	0.7923		-0.78	
823		-----		-----	
824		-----		-----	
825		-----		-----	
840		-----		-----	
855	D4052cal.	0.79241		-0.16	
856		-----		-----	
857		0.79244		0.01	
858	D891	0.79240		-0.22	
859	D891	0.79240		-0.22	
860	D891	0.79240		-0.22	
861		0.79241		-0.16	
862	D891	0.79241		-0.16	
863	D4052cal.	0.79249		0.29	
864	D4052cal.	0.79241		-0.16	
866	D4052	0.79244		0.01	
870	D4052cal.	0.79239		-0.27	
902	D4052	0.79253		0.51	
912		-----		-----	
913	D891	0.7924		-0.22	
963		-----		-----	
974	D4052	0.7924		-0.22	
1007	in house	0.7924		-0.22	
1009		-----		-----	
1010		-----		-----	
1016		-----		-----	
1029		0.79406	G(0.01)	9.08	
1041		-----		-----	
1067		-----		-----	
1108		-----		-----	
1149		0.7919	G(0.01)	-3.02	
1201		0.7926		0.90	
1204		-----		-----	
1221		0.7925		0.34	
1239		-----		-----	
1246		-----		-----	
1256		-----		-----	
1341		-----		-----	
1342	D4052	0.7924		-0.22	
1343		-----		-----	
1344		0.7926		0.90	
1354	D4052	0.7926		0.90	

1355	D4052	0.7926	0.90
1510		-----	-----
1538		-----	-----
1615	D4052calc	0.79233	-0.61
1826		-----	-----
1905		-----	-----

normality not OK
 n 34
 outliers 2
 mean (n) 0.79244
 st.dev. (n) 0.000096
 R(calc.) 0.00027
 R(D4052:02e1) 0.00050

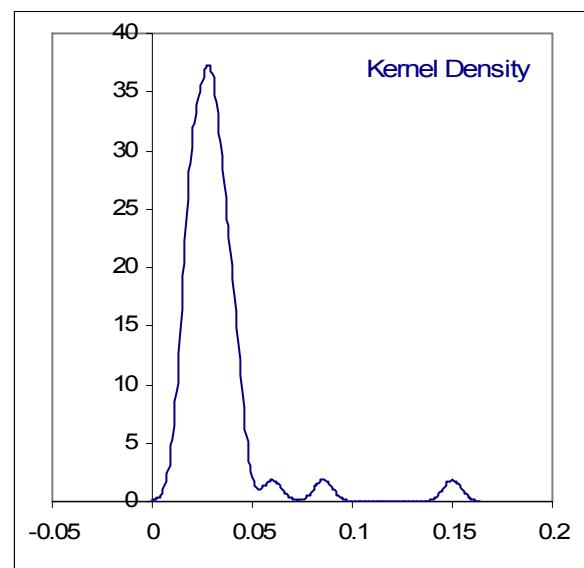
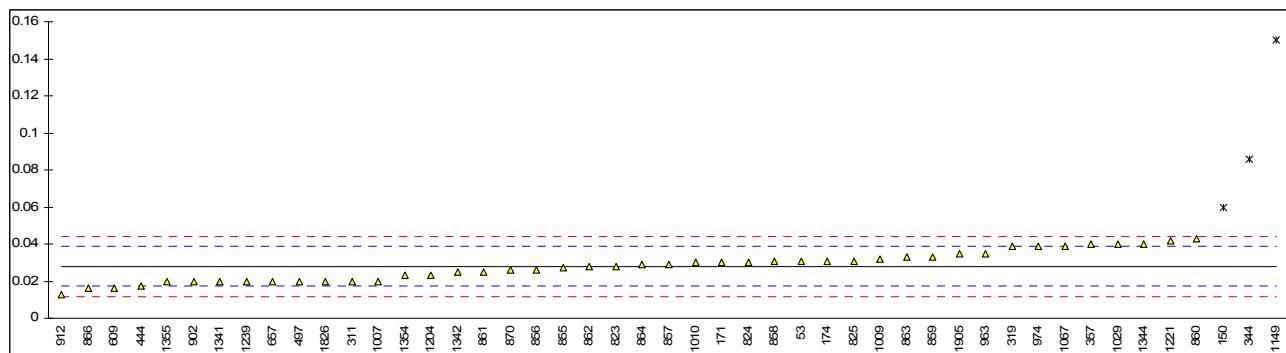


Determination of Total Iron as Fe on sample #1052; results in mg/kg

lab	method	value	mark	z(targ)	remarks
53	E394	0.031		0.54	
150	E394	0.06	G(0.05)	5.91	
153		----		----	
171	E394	0.03		0.36	
174	E394	0.031		0.54	
311	E394	0.02	C	-1.49	first reported 0.07
316		----		----	
319	E394	0.0387		1.97	
323		----		----	
332		----		----	
333		----		----	
334		----		----	
342		----		----	
343		----		----	
344	E394	0.0856	G(0.01)	10.64	
345		----		----	
346		----		----	
347		----		----	
357	E394	0.04		2.21	
395		----		----	
444	E394	0.0175		-1.95	
446		----		----	
494		----		----	
497	E394	0.02		-1.49	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609	E394	0.016		-2.23	
646	E394	<0.01		<-3.36	False negative
657	E394	0.02		-1.49	
663		----		----	
823	E394	0.028		-0.01	
824	E394	0.03		0.36	
825	E394	0.031		0.54	
840		----		----	
855	E394	0.027		-0.20	
856	E394	0.026		-0.38	
857	E394	0.029		0.17	
858	E394	0.031		0.54	
859	E394	0.033		0.91	
860	E394	0.043		2.76	
861	E394	0.025		-0.57	
862	E394	0.028		-0.01	
863	E394	0.033		0.91	
864	E394	0.029		0.17	
866	E394	0.016		-2.23	
870	E394	0.026		-0.38	
902	E394	0.0198		-1.53	
912	E394	0.013		-2.79	
913	E394	<0.01		<-3.36	False negative
963	E394	0.035		1.28	
974	E394	0.039		2.02	
1007	in house	0.02		-1.49	
1009	E394	0.032		0.73	
1010	E394	0.03		0.36	
1016		----		----	
1029	E394	0.04		2.21	
1041		----		----	
1067	E394	0.039		2.02	
1108		----		----	
1149	ICP	0.15	G(0.01)	22.55	
1201		----		----	
1204	E394	0.023104		-0.92	
1221	E394	0.042		2.58	
1239	E394	0.02		-1.49	
1246		----		----	
1256		----		----	
1341	E394	0.02		-1.49	
1342	E394	0.025		-0.57	
1343		----		----	
1344	E394	0.04		2.21	
1354	E394	0.023		-0.94	

1355	in house	0.0197	-1.55
1510		-----	-----
1538		-----	-----
1615		-----	-----
1826	E394	0.02	-1.49
1905	E394	0.035	1.28

normality not OK
 n 44
 outliers 3 Spike
 mean (n) 0.0281
 st.dev. (n) 0.00793
 R(calc.) 0.0222
 R(E394:09) 0.0151



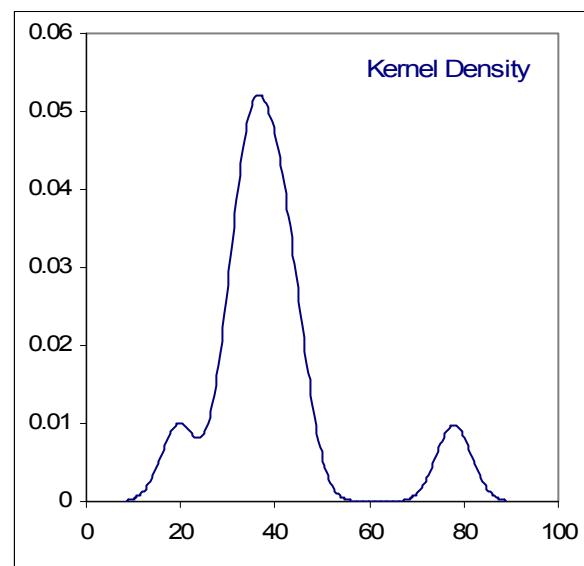
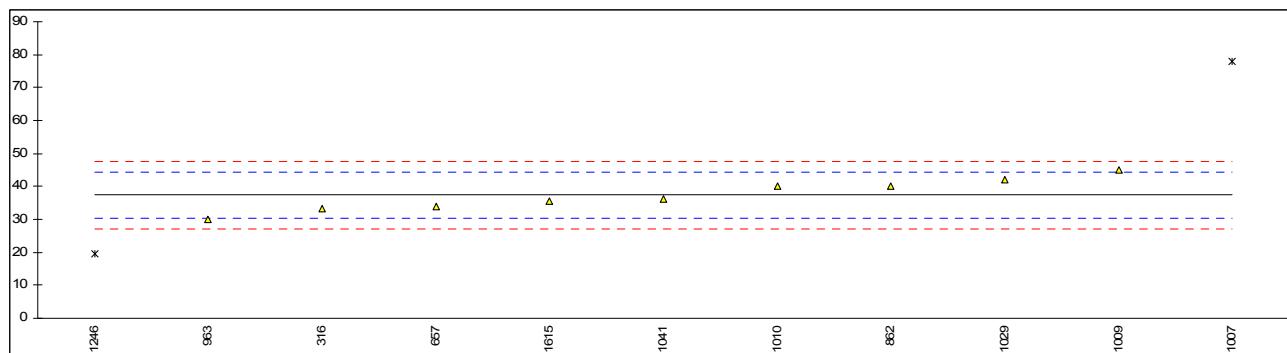
Determination of Trimethylamine on sample #1052; results in µg/kg

lab	method	value	mark	z(targ)	remarks
53		----		----	
150		----		----	
153		----		----	
171		----		----	
174		----		----	
311		----		----	
316	inh-d	33.3		-1.17	
319		----		----	
323		----		----	
332		----		----	
333		----		----	
334		----		----	
342		----		----	
343		----		----	
344		----		----	
345		----		----	
346		----		----	
347		----		----	
357		----		----	
395		----		----	
444		----		----	
446		----		----	
494		----		----	
497		----		----	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609		----		----	
646		----		----	
657	E346	34		-0.97	
663		----		----	
823		----		----	
824		----		----	
825		----		----	
840		----		----	
855		----		----	
856		----		----	
857		----		----	
858		----		----	
859		----		----	
860		----		----	
861		----		----	
862	E346	40		0.76	
863		----		----	
864		----		----	
866		----		----	
870		----		----	
902		----		----	
912		----		----	
913		----		----	
963	E346	30		-2.12	
974		----		----	
1007	in house	77.9	G(0.01)	11.69	
1009	E346	45		2.20	
1010	inhouse	40		0.76	
1016		----		----	
1029	E346	42.19		1.39	
1041	DIN51405	36.21		-0.33	
1067		----		----	
1108		----		----	
1149		----		----	
1201	E346	<10		<-7.89	False negative
1204		----		----	
1221		----		----	
1239		----		----	
1246	E346	19.52	G(0.01)	-5.15	
1256		----		----	
1341		----		----	
1342		----		----	
1343		----		----	
1344		----		----	
1354		----		----	

1355	-----	-----			
1510	-----	-----			
1538	-----	-----			
1615	E346	35.562	C	-0.52	first reported <10
1826	-----	-----	-----	-----	-----
1905	-----	-----	-----	-----	-----

normality	OK
n	9
outliers	2
mean (n)	37.4
st.dev. (n)	4.77
R(calc.)	13.4
R(Horwitz)	9.7

Spike

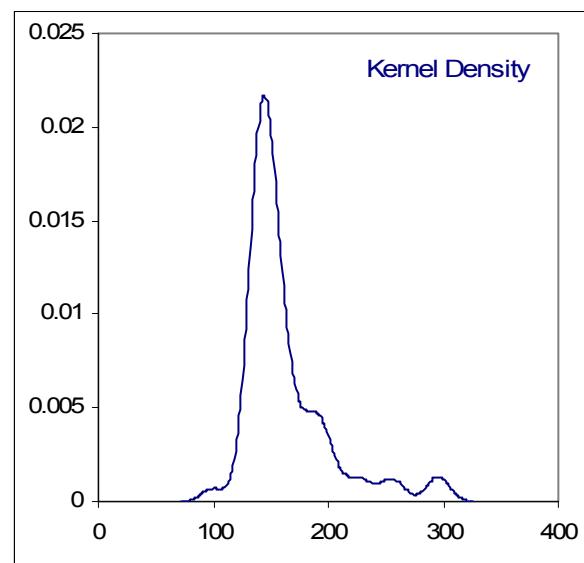
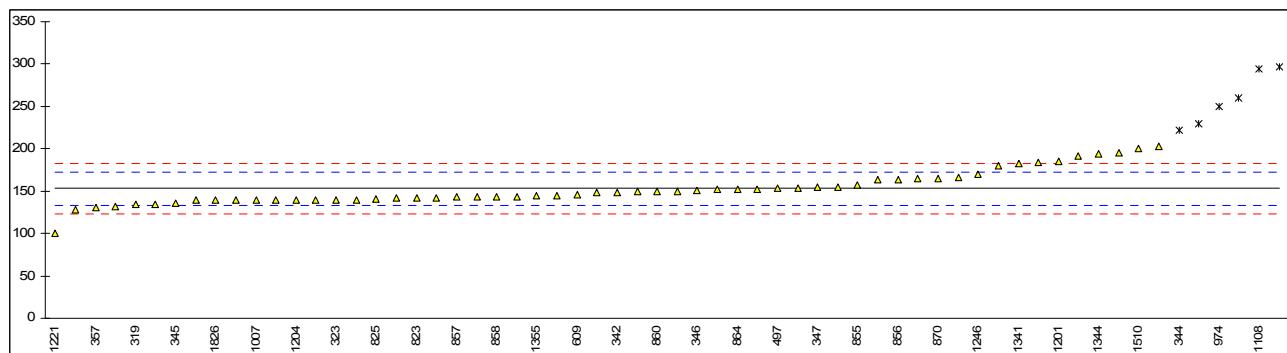


Determination of Water content (coulometric) on sample #1052; results in mg/kg

lab	method	value	mark	z(targ)	remarks
53	E1064	141.4		-1.23	
150	E1064	145		-0.84	
153		----		----	
171		----		----	
174		----		----	
311	E1064	140		-1.38	
316		----		----	
319	E1064	134	C	-2.02	first reported 0.013
323	E1064	140		-1.38	
332		----		----	
333	E1064	260	G(0.05)	11.47	
334	E1064	140		-1.38	
342	E1064	149		-0.42	
343	E1064	143		-1.06	
344	E1064	222	DG(0.05)	7.40	
345	E1064	136		-1.81	
346	E1064	150.8		-0.22	
347	E1064	154.7		0.19	
357	E1064	131		-2.34	
395	E1064	191.07		4.09	
444		----		----	
446	E1064	150		-0.31	
494	E1064	135		-1.92	
497	E1064	154		0.12	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609	E1064	145.6		-0.78	
646	E1064	230	DG(0.05)	8.26	
657	E1064	140		-1.38	
663	E1064	152		-0.10	
823	E1064	142		-1.17	
824	E1064	142		-1.17	
825	E1064	141		-1.27	
840		----		----	
855	E1064	157		0.44	
856	E1064	163		1.08	
857	E1064	142.8		-1.08	
858	E1064	143		-1.06	
859	E1064	139		-1.49	
860	E1064	150		-0.31	
861	E1064	152		-0.10	
862	E1064	143		-1.06	
863	E1064	154		0.12	
864	E1064	152		-0.10	
866	E1064	131.8		-2.26	
870	E1064	165		1.30	
902	E1064	155		0.23	
912		----		----	
913		----		----	
963	E1064	184		3.33	
974	E1064	250	G(0.05)	10.40	
1007	in house	139.6		-1.42	
1009	E1064	150		-0.31	
1010	E1064	127.5		-2.72	
1016	E1064	164.9		1.29	
1029	E1064	148.2		-0.50	
1041	ISO12937	195.33		4.55	
1067	E1064	297	G(0.05)	15.43	
1108	E1064	294	G(0.01)	15.11	
1149	E1064	163.00		1.08	
1201	E1064	185		3.44	
1204	E1064	140.0		-1.38	
1221	E1064	100.0		-5.66	
1239		----		----	
1246	E1064	170	C	1.83	first reported 0.0170
1256		----		----	
1341	E1064	183.1		3.24	
1342	E1064	203		5.37	
1343		----		----	
1344	E1064	194		4.40	
1354	E1064	139.09		-1.48	

1355	E1064	144.16	-0.93
1510	E1064	200	5.05
1538		----	----
1615	E1064	166.67	1.48
1826	E1064	139	-1.49
1905	E1064	180	2.90

normality not OK
 n 56
 outliers 6
 mean (n) 152.89
 st.dev. (n) 20.185
 R(calc.) 56.52
 R(E1064:08) 27.90

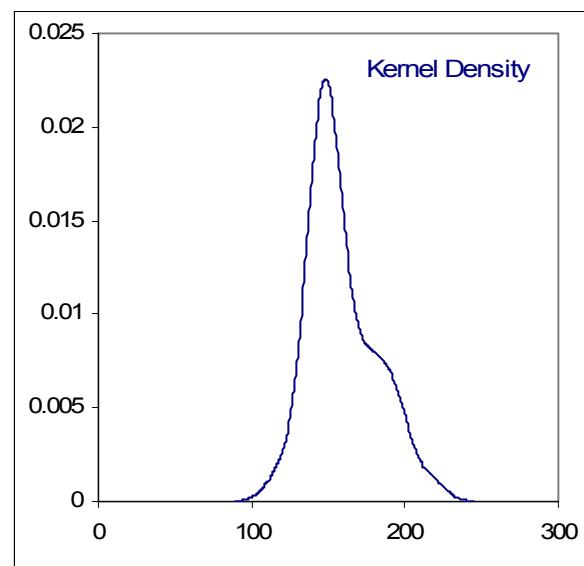
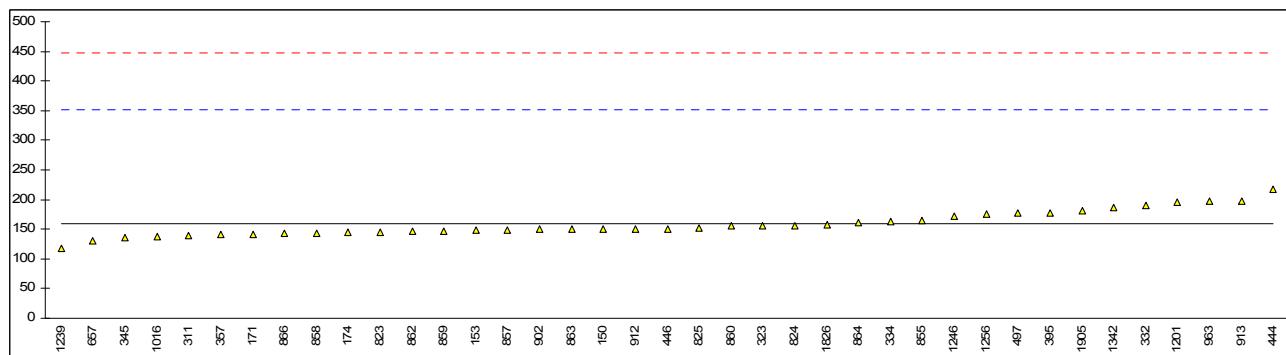


Determination of Water content (titrimetric) on sample #1052; results in mg/kg

lab	method	value	mark	z(targ)	remarks
53		----		----	
150	E203	150		-0.09	
153	E203	148	C	-0.11	first reported 0.0148
171	E203	142		-0.17	
174	E203	145		-0.14	
311	E203	140		-0.20	
316		----		----	
319		----		----	
323	E203	156		-0.03	
332	E203	190		0.32	
333		----		----	
334	E203	163		0.04	
342		----		----	
343		----		----	
344		----		----	
345	E203	135		-0.25	
346		----		----	
347		----		----	
357	E203	142		-0.17	
395	E203	178.09		0.20	
444	E203	217		0.60	
446	E203	150		-0.09	
494		----		----	
497	E203	178		0.20	
528		----		----	
529		----		----	
551		----		----	
608		----		----	
609		----		----	
646		----		----	
657	E203	130		-0.30	
663		----		----	
823	E203	145		-0.14	
824	E203	156		-0.03	
825	E203	152		-0.07	
840		----		----	
855	D1364	165		0.06	
856		----		----	
857	E203	148.2		-0.11	
858	E203	144		-0.15	
859	E203	146		-0.13	
860	E203	155		-0.04	
861		----		----	
862	E203	146		-0.13	
863	D1364	150		-0.09	
864	D1364	161		0.02	
866	E203	143.2		-0.16	
870		----		----	
902	E203	150		-0.09	
912	E203	150		-0.09	
913	E203	198		0.41	
963	E203	197		0.40	
974		----		----	
1007		----		----	
1009		----		----	
1010		----		----	
1016	D1364	138		-0.22	
1029		----		----	
1041		----		----	
1067		----		----	
1108		----		----	
1149		----		----	
1201	E203	195		0.38	
1204		----		----	
1221		----		----	
1239	E203	117		-0.43	
1246	E203	173	C	0.15	first reported 0.0173
1256	E203	175	C	0.17	first reported 0.020
1341		----		----	
1342	E203	187		0.29	
1343		----		----	
1344		----		----	
1354		----		----	

1355		-----	
1510		-----	
1538		-----	
1615		-----	
1826	E203	157	-0.02
1905	E203	182	0.24

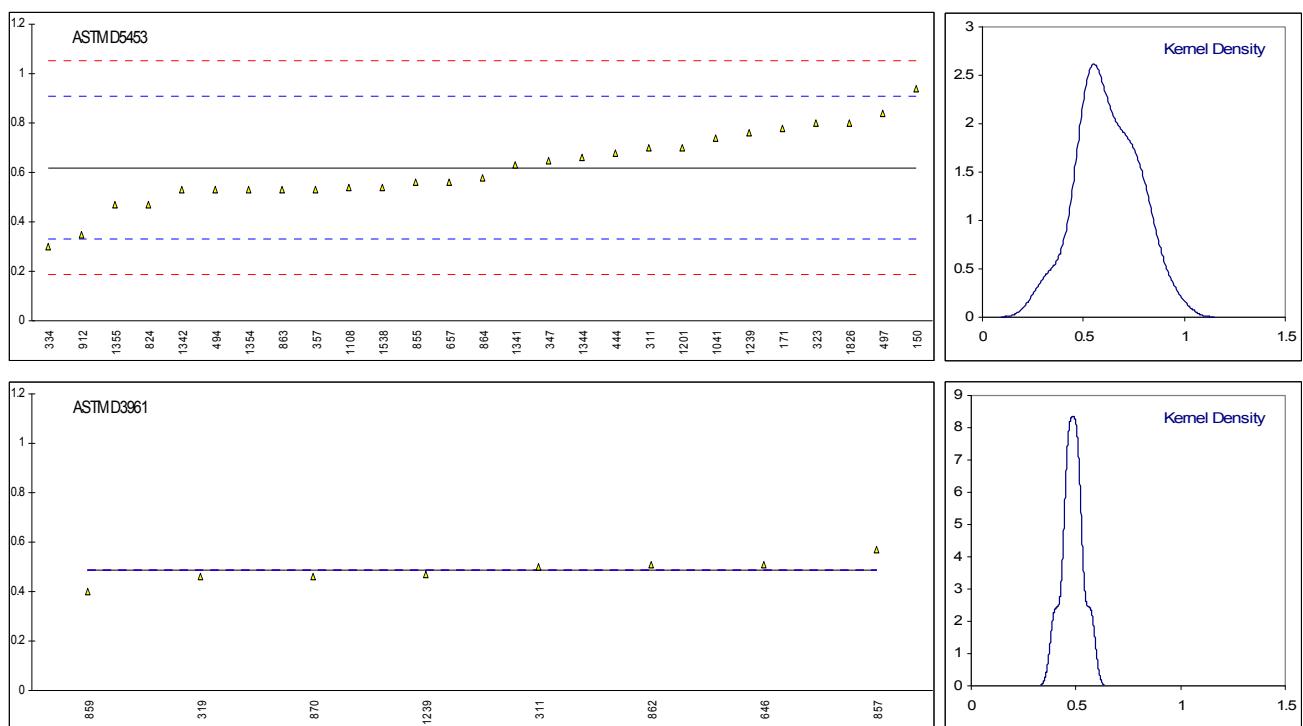
normality not OK
 n 39
 outliers 0
 mean (n) 158.83
 st.dev. (n) 21.537
 R(calc.) 60.30
 R(E203:08) 270.00



Determination of Sulphur (low) on sample #S-01, results in mg/kg

lab	method	D5453	mark	z(targ)	D3961	mark	z(targ)	Other	mark	z(targ)
53		----		----	----		----	----		----
150	D5453	0.94		2.23	----		----	----		----
153		----		----	----		----	----		----
171	D5453	0.78		1.12	----		----	----		----
174		----		----	----		----	----		----
311	D5453	0.7		0.56	0.5		----	----		----
316		----		----	----		----	----		----
319		----		0.46	----		----	----		----
323	D5453	0.8		1.26	----		----	----		----
332		----		----	----		----	----		----
333		----		----	----		----	0.3	NFM07059	----
334	D5453	0.3		-2.21	----		----	----		----
342		----		----	----		----	----		----
343		----		----	----		----	----		----
344		----		----	----		----	----		----
345		----		----	----		----	----		----
346		----		----	----		----	----		----
347	D5453	0.65		0.22	----		----	----		----
357	D5453	0.53		-0.61	----		----	----		----
395		----		----	----		----	----		----
444	D5453	0.68		0.43	----		----	----		----
446		----		----	----		----	----		----
494	D5453	0.53		-0.61	----		----	----		----
497	D5453	0.84		1.53	----		----	----		----
528		----		----	----		----	----		----
529		----		----	----		----	----		----
551		----		----	----		----	----		----
608		----		----	----		----	----		----
609		----		----	----		----	----		----
646		----		0.51	----		----	----		----
657	D5453	0.56		-0.41	----		----	----		----
663		----		----	----		----	----		----
823		----		----	----		----	----		----
824	D5453	0.47		-1.03	----		----	----		----
825		----		----	----		----	----		----
840		----		----	----		----	----		----
855	D5453	0.56		-0.41	----		----	----		----
856		----		----	----		----	----		----
857		----		0.57	----		----	----		----
858		----		----	----		----	----		----
859		----		0.4	----		----	----		----
860		----		----	----		----	----		----
861		----		----	----		----	----		----
862		----		0.51	----		----	----		----
863	D5453	0.53		-0.61	----		----	----		----
864	D5453	0.58		-0.27	----		----	----		----
866		----		----	----		----	----		----
870		----		0.46	----		----	----		----
902		----		----	----		----	----		----
912	D5453	0.35		-1.86	----		----	----		----
913		----		----	----		----	----		----
963		----		----	----		----	0.96	D3120	----
974		----		----	----		----	<0.5	in house	----
1007		----		----	----		----	----		----
1009		----		----	----		----	----		----
1010		----		----	----		----	<0.5	SIEVERS	----
1016		----		----	----		----	0.30	ISO 20846	----
1029		----		----	----		----	----		----
1041	D5453	0.74		0.84	----		----	----		----
1067		----		----	----		----	----		----
1108	D5453	0.54	C, fr 1.2	-0.54	----		----	0.17	ICP	----
1149		----		----	----		----	----		----
1201	D5453	0.7		0.56	----		----	----		----
1204		----		----	----		----	----		----
1221		----		----	----		----	----		----
1239	D5453	0.76		0.98	0.47	----	----	----		----
1246		----		----	----		----	----		----
1256		----		----	----		----	----		----
1341	D5453	0.63		0.08	----		----	----		----
1342	D5453	0.53		-0.61	----		----	----		----
1343		----		----	----		----	----		----
1344	D5453	0.66		0.29	----		----	----		----
1354	D5453	0.53		-0.61	----		----	----		----

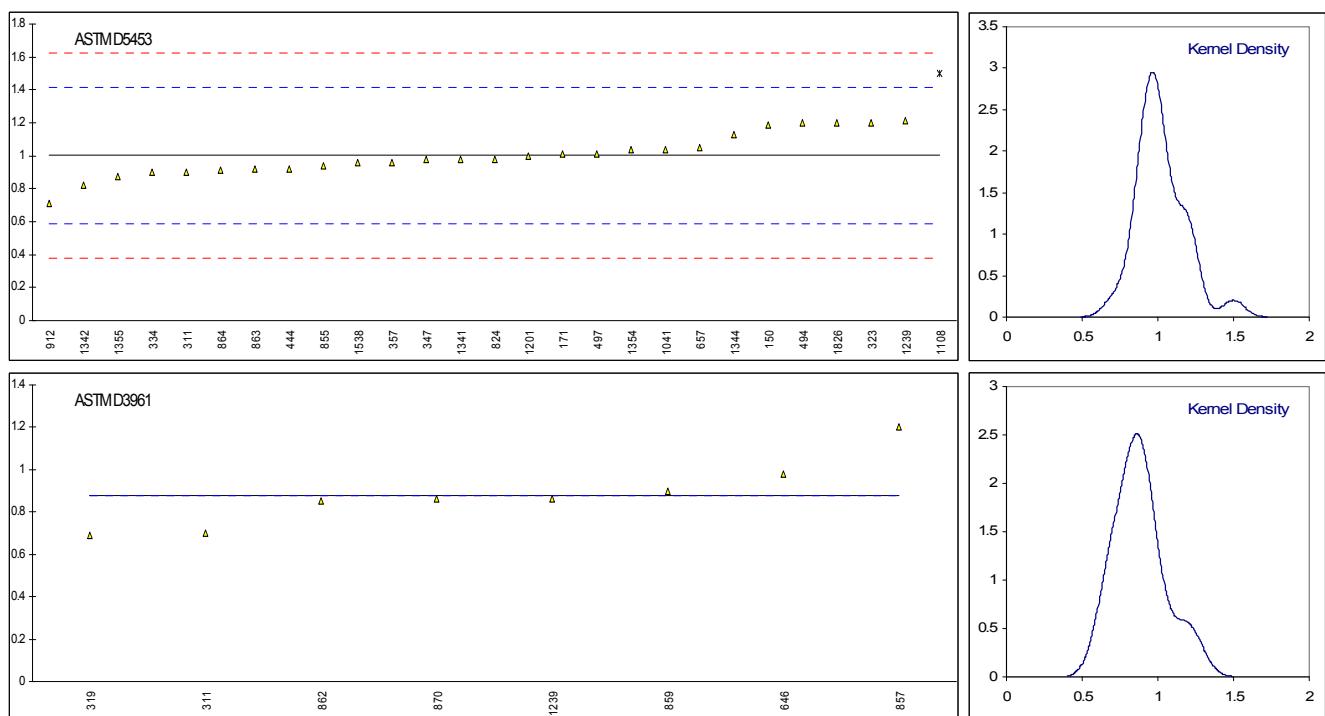
1355	D5453	0.470	-1.03	----	----	----	----
1510		----	----	----	----	----	----
1538	D5453	0.54	-0.54	----	----	----	----
1615		----	----	----	----	----	----
1826	D5453	0.8	1.26	----	----	----	----
1905		----	----	----	----	----	----
normality		OK		OK		n.a.	
n		27		8		4	
outliers		0	<u>Spike</u>	0	<u>Spike</u>	0	
mean (n)		0.619	0.502	< 123%	0.485	0.502	< 97%
st.dev. (n)		0.1489			0.0499		
R(calc.)		0.417			0.140		
RSD _R		67%			0.29%		
R(lit)		(0.404)	D5453	unknown	D3961	unknown	



Determination of Sulphur (High) on sample #S-02, results in mg/kg

lab	method	D5453	mark	z(targ)	D3961	mark	z(targ)	Other	mark	z(targ)
53		----		----	----		----	----		----
150	D5453	1.19		0.91	----		----	----		----
153		----		----	----		----	----		----
171	D5453	1.01		0.04	----		----	----		----
174		----		----	----		----	----		----
311	D5453	0.9		-0.49	0.7		----	----		----
316		----		----	----		----	----		----
319		----		0.69			----	----		----
323	D5453	1.2	C, fr. 1.4	0.96	----		----	----		----
332		----		----	----		----	----		----
333		----		----	----		----	0.9	NFM07059	----
334	D5453	0.9		-0.49	----		----	----		----
342		----		----	----		----	----		----
343		----		----	----		----	----		----
344		----		----	----		----	----		----
345		----		----	----		----	----		----
346		----		----	----		----	----		----
347	D5453	0.98		-0.10	----		----	----		----
357	D5453	0.96		-0.20	----		----	----		----
395		----		----	----		----	----		----
444	D5453	0.92		-0.39	----		----	----		----
446		----		----	----		----	----		----
494	D5453	1.20		0.96	----		----	----		----
497	D5453	1.01		0.04	----		----	----		----
528		----		----	----		----	----		----
529		----		----	----		----	----		----
551		----		----	----		----	----		----
608		----		----	----		----	----		----
609		----		----	----		----	----		----
646		----		0.98			----	----		----
657	D5453	1.05		0.23	----		----	----		----
663		----		----	----		----	----		----
823		----		----	----		----	----		----
824	D5453	0.98		-0.10	----		----	----		----
825		----		----	----		----	----		----
840		----		----	----		----	----		----
855	D5453	0.94		-0.30	----		----	----		----
856		----		----	----		----	----		----
857		----		1.20	----		----	----		----
858		----		----	----		----	----		----
859		----		0.9	----		----	----		----
860		----		----	----		----	----		----
861		----		----	----		----	----		----
862		----		0.85	----		----	----		----
863	D5453	0.92		-0.39	----		----	----		----
864	D5453	0.91		-0.44	----		----	----		----
866		----		----	----		----	----		----
870		----		0.86	----		----	----		----
902		----		----	----		----	----		----
912	D5453	0.71		-1.41	----		----	----		----
913		----		----	----		----	----		----
963		----		----	----		----	----		----
974		----		----	----		----	----		----
1007		----		----	----		<0.5	in house		----
1009		----		----	----		----	----		----
1010		----		----	----		<0.5	SIEVERS		----
1016		----		----	----		0.58	ISO 20846		----
1029		----		----	----		----	----		----
1041	D5453	1.04		0.19	----		----	----		----
1067		----		----	----		----	----		----
1108	D5453	1.5	C,G(0.05)	2.41	----		----	----		----
1149		----		----	----		0.07	ICP		----
1201	D5453	1.0		-0.01	----		----	----		----
1204		----		----	----		----	----		----
1221		----		----	----		----	----		----
1239	D5453	1.21		1.01	0.86		----	----		----
1246		----		----	----		----	----		----
1256		----		----	----		----	----		----
1341	D5453	0.98		-0.10	----		----	----		----
1342	D5453	0.82		-0.88	----		----	----		----
1343		----		----	----		----	----		----
1344	D5453	1.13		0.62	----		----	----		----
1354	D5453	1.04		0.19	----		----	----		----

1355	D5453	0.875	-0.61	----	----	----	----
1510		----	----	----	----	----	----
1538	D5453	0.96	-0.20	----	----	----	----
1615		----	----	----	----	----	----
1826	D5453	1.2	0.96	----	----	----	----
1905		----	----	----	----	----	----
normality		OK		OK		n.a.	
n		26		8		3	
outliers		1	<u>Spike</u>	0	<u>Spike</u>	0	
mean (n)		1.001	1.007	< 99%	0.880	1.007	< 87%
st.dev. (n)		0.1271			0.1617		
R(calc.)		0.356			0.453		
RSD _R		36%			51%		
R(lit)		(0.580)	D5453		unknown	D3961	unknown



Determination of UV Absorbance (50 mm Cuvette) on sample #1053

lab	method	300nm	mark	268.5nm	mark	250nm	mark	240nm	mark	230nm	mark	220nm	mark
150	IMPCA004	0.008	C	0.038	C	0.140	C	----	----	----	----	1.185	C
171		----		----		----		----		----		----	
311	IMPCA004	0.012	C	0.043	C	0.172	G(5)	0.252		0.564		1.177	C
319		----		----		----		----		----		----	
323	IMPCA004	0.010		0.044	C	0.167	C	----	----	----	----	1.286	C
334	IMPCA004	0.011		0.040		0.164		0.273		0.590		1.234	
342		----		----		----		----		----		----	
347		----		----		----		----		----		----	
357	IMPCA004	0.012		0.043		0.155		0.260		0.564		1.183	
395		----		----		----		----		----		----	
444	IMPCA004	0.000	G(1)	0.027	G(5)	0.133		0.230		0.601		1.110	
609		----		----		----		----		----		----	
657	IMPCA004	0.008		0.038		0.135		0.245		0.551		1.132	
823		----		----		----		----		----		----	
824		----		----		----		----		----		----	
825		----		----		----		----		----		----	
840		----		----		----		----		----		----	
855	IMPCA004	0.008		0.036		0.124		0.228		0.513		1.072	
856	IMPCA004	0.010		0.038		0.133		0.234		0.505		1.073	
857	IMPCA004	0.0112		0.0434		0.1484		0.2592		0.5601		1.1274	
858	IMPCA004	0.0108		0.0413		0.1482		0.2513		0.5432		1.1027	
859	IMPCA004	0.0131		0.0443		0.1489		0.2634		0.5620		1.1304	
860	IMPCA004	0.010		0.040		0.140		0.243		0.533		1.133	
861	IMPCA004	0.008		0.037		0.136		0.233		0.514		1.061	
862	IMPCA004	0.010		0.039		0.148		0.245		0.534		1.086	
863	IMPCA004	0.013		0.046		0.140		0.238		0.510		1.102	
864	IMPCA004	0.010		0.038		0.131		0.238		0.522		1.091	
866	IMPCA004	0.014		0.046		0.142		0.239		0.519		1.094	
870	IMPCA004	0.007		0.033		0.121		0.222		0.497		1.088	
963	IMPCA004	0.0081	C	0.0327	C	0.143	C	0.255	C	0.589		1.244	C
974		----		----		----		----		----		----	
1007		----		----		----		----		----		----	
1010	IMPCA004	0.012		0.041		0.149		0.243		0.530		1.134	
1041	IMPCA004	0.005	C	0.033	C	0.139	C	0.236	C	0.512	C	1.133	C
1067	IMPCA004	0.010		0.041		0.141		0.253		0.561		1.180	
1201	IMPCA004	0	G(1)	0	G(1)	0.102	G(5)	0.188	G(5)	0.393	G(1)	0.616	G(1)
1246	IMPCA004	-0.033	C,G(1)	-0.001	C,G(5)	0.109	CG(5)	0.229	C	0.542	C	1.140	C
1342		----		----		----		----		----		----	
1343		----		----		----		----		----		----	
1344		----		----		----		----		----		----	
1354		----		----		----		----		----		----	
1826		----		----		----		----		----		----	
1905	IMPCA004	0.000	G(1)	0.0240	G(5)	0.1240		0.2190		0.5075		1.0815	
<hr/>													
normality		OK		OK		OK		OK		OK		not OK	
n		22		22		23		23		23		25	
outliers		4		4		3		1		1		1	
mean (n)		0.0101		0.0398		0.1413		0.2430		0.5402		1.1352	
st.dev. (n)		0.00222		0.00398		0.01160		0.01377		0.02970		0.05786	
R(calc.)		0.0062		0.0111		0.0325		0.0386		0.0832		0.1620	
R(IMPCA004:08)		0.0151		0.0108		0.0143		unknown		unknown		0.3076	

G(1) = Grubbs's outlier G(0.01)

G(5) = Grubbs's outlier G(0.05)

Lab 150 first reported results for a 10mm cuvette

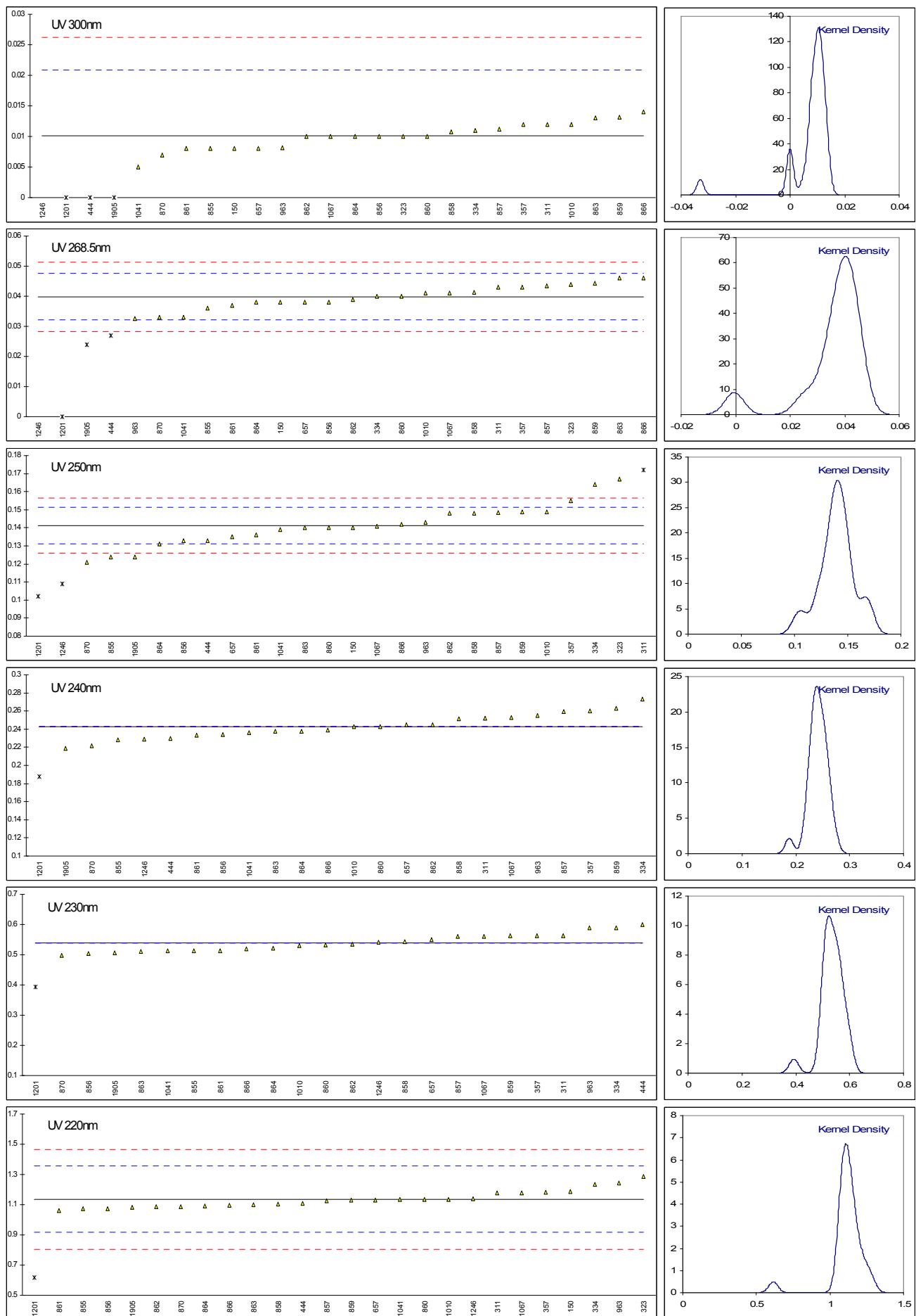
Lab 311 first reported 0.031@300nm; 0.062@268.5nm and 1.241@220nm

Lab 323 first reported 0.040@268.5nm; 0.160@250nm and 1.255@220nm

Lab 963 first reported 0.86499@300nm; 0.8934@268.5nm; 0.99557@250nm; 1.00776@240nm and 2.0205@220nm

Lab 1041 first reported -0.021@300nm; 0.007@268.5nm; 0.112@250nm; 0.210@240nm; 0.486@230nm and 1.107@220nm

Lab 1246 first reported 2.699@300nm; 2.700@268.5nm; 2.543@250nm; 2.423@240nm; 2.162@230nm and 1.485@220nm

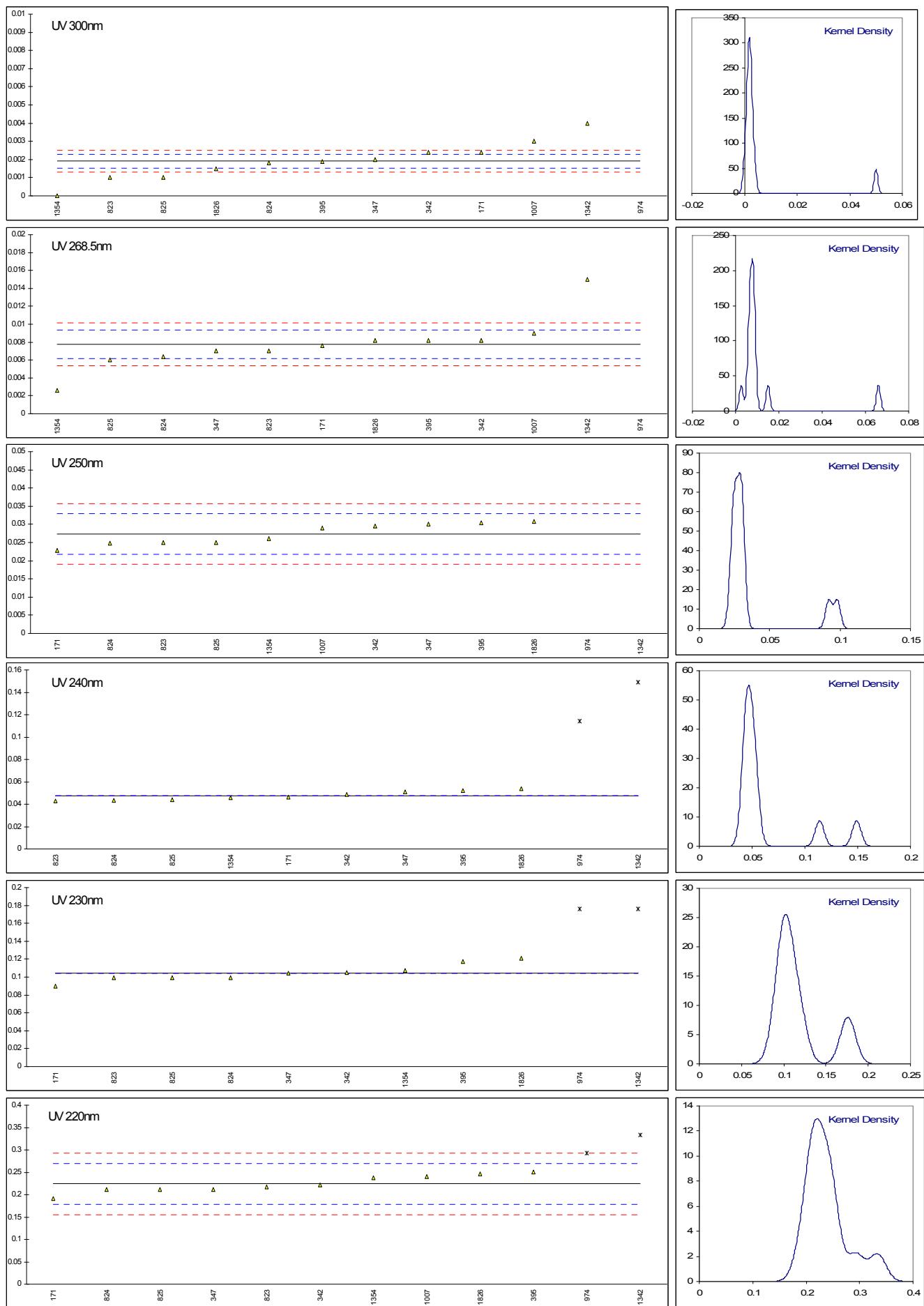


Determination of UV Absorbance (10 mm Cuvette) on sample #1053

lab	method	300nm	mark	268.5nm	mark	250nm	mark	240nm	mark	230nm	mark	220nm	mark
150		----		----		----		----		----		----	
171	IMPCA004	0.0024		0.0076		0.0228		0.0462		0.0902		0.1911	
311		----		----		----		----		----		----	
319		----		----		----		----		----		----	
323		----		----		----		----		----		----	
334		----		----		----		----		----		----	
342	IMPCA004	0.0024		0.0082		0.0296		0.0489		0.1050		0.2223	
347	IMPCA004	0.002		0.007		0.030		0.051		0.104		0.212	
357		----		----		----		----		----		----	
395	IMPCA004	0.0019		0.0082		0.0304		0.0520		0.1172		0.2511	
444		----		----		----		----		----		----	
609		----		----		----		----		----		----	
657		----		----		----		----		----		----	
823	IMPCA004	0.001		0.007		0.025		0.043		0.099		0.217	
824	IMPCA004	0.0018		0.0064		0.0249		0.0437		0.0993		0.2116	
825	IMPCA004	0.001		0.006		0.025		0.044		0.099		0.212	
840		----		----		----		----		----		----	
855		----		----		----		----		----		----	
856		----		----		----		----		----		----	
857		----		----		----		----		----		----	
858		----		----		----		----		----		----	
859		----		----		----		----		----		----	
860		----		----		----		----		----		----	
861		----		----		----		----		----		----	
862		----		----		----		----		----		----	
863		----		----		----		----		----		----	
864		----		----		----		----		----		----	
866		----		----		----		----		----		----	
870		----		----		----		----		----		----	
963		----		----		----		----		----		----	
974	IMPCA004	0.050	G(1)	0.066	G(1)	0.092	G(1)	0.114	G(1)	0.176	G(1)	0.293	G(5)
1007	in house	0.003		0.009		0.029		----		----		0.240	
1010		----		----		----		----		----		----	
1041		----		----		----		----		----		----	
1067		----		----		----		----		----		----	
1201		----		----		----		----		----		----	
1246		----		----		----		----		----		----	
1342	IMPCA004	0.004		0.015		0.098	G(1)	0.149	G(5)	0.176	G(1)	0.334	G(5)
1343		----		----		----		----		----		----	
1344		----		----		----		----		----		----	
1354	IMPCA004	0.00		0.0026		0.026		0.046		0.107		0.238	
1826	IMPCA004	0.0015		0.0082		0.0308		0.0541		0.121		0.247	
1905		----		----		----		----		----		----	
normality	OK		not OK		OK		OK		OK		OK		
n	11		11		10		9		9		10		
outliers	1		1		2		2		2		2		
mean (n)	0.0019		0.0077		0.0274		0.0477		0.1046		0.2242		
st.dev. (n)	0.00107		0.00295		0.00290		0.00401		0.00957		0.01912		
R(calc.)	0.0030		0.0083		0.0081		0.0112		0.0268		0.0535		
R(IMPCA004:08)	0.0005		0.0022		0.0078		unknown		unknown		0.0643		

G(1) = Grubbs's outlier G(0.01)

G(5) = Grubbs's outlier G(0.05)



Other UV details

lab	UV Curve	Sample pass	Component (Benzene)	remarks
150	not smooth	FAIL	present	
171	----	FAIL	9.6	reported also the presence of styrene
311	FAIL	FAIL	8.7	reported also the presence of n-amylalcohol
319	----	----	----	
323	FAIL	FAIL	present	reported also the presence of MEK and ethanol
334	not smooth	FAIL	----	
342	----	----	----	
347	----	FAIL	----	
357	not smooth	FAIL	9	
395	not smooth	FAIL	----	
444	FAIL	FAIL	8.9	reported also the presence of MEK and tridecane
609	----	----	----	
657	not smooth	FAIL	10.29	
823	----	----	----	
824	not smooth	FAIL	----	
825	----	----	----	
840	----	----	----	
855	not smooth	FAIL	8.9	
856	not smooth	FAIL	10.0	
857	not smooth	FAIL	9	reported also the presence of toluene
858	not smooth	FAIL	9	reported also the presence of toluene
859	not smooth	FAIL	9.8	reported also the presence of toluene
860	not smooth	FAIL	8.7	reported also the presence of toluene
861	not smooth	FAIL	9.2	reported also the presence of toluene
862	not smooth	FAIL	8.8	reported also the presence of toluene
863	not smooth	FAIL	10.2	
864	not smooth	FAIL	9.1	
866	not smooth	FAIL	9.2	
870	not smooth	FAIL	9.0	
963	not smooth	FAIL	----	reported the presence of MEK
974	----	PASS	----	
1007	----	----	----	
1010	not smooth	FAIL	10	reported also the presence of MEK and toluene
1041	----	PASS	----	
1067	not smooth	FAIL	----	
1201	not smooth	FAIL	----	
1246	smooth	PASS	----	
1342	----	----	----	
1343	----	----	----	
1344	----	----	----	
1354	not smooth	FAIL	1.46	reported also MEK, ethanol, n-butanol and toluene
1826	not smooth	FAIL	present	reported also the presence of toluene
1905	not smooth	FAIL	----	

z-scores UV absorbance

lab	50mm Cuvette						10mm Cuvette					
	300nm	268.5nm	250nm	240nm	230nm	220nm	300nm	268.5nm	250nm	240nm	230nm	220nm
150	-0.38	-0.47	-0.26	----	----	0.43	----	----	----	----	----	----
171	----	----	----	----	----	----	0.48	-0.19	-4.61	----	----	-1.44
311	0.36	0.83	6.02	----	----	0.36	----	----	----	----	----	----
319	----	----	----	----	----	----	----	----	----	----	----	----
323	-0.01	1.09	5.04	----	----	1.30	----	----	----	----	----	----
334	0.18	0.05	4.45	----	----	0.85	----	----	----	----	----	----
342	----	----	----	----	----	----	0.48	0.61	2.28	----	----	-0.08
347	----	----	----	----	----	----	0.09	-0.99	2.69	----	----	-0.53
357	0.36	0.83	2.68	----	----	0.41	----	----	----	----	----	----
395	----	----	----	----	----	----	-0.01	0.61	3.09	----	----	1.17
444	-1.87	-3.32	-1.63	----	----	-0.22	----	----	----	----	----	----
609	----	----	----	----	----	----	----	----	----	----	----	----
657	-0.38	-0.47	-1.24	----	----	-0.03	----	----	----	----	----	----
823	----	----	----	----	----	----	-0.89	-0.99	-2.38	----	----	-0.31
824	----	----	----	----	----	----	-0.11	-1.79	-2.48	----	----	-0.55
825	----	----	----	----	----	----	-0.89	-2.33	-2.38	----	----	-0.53
840	----	----	----	----	----	----	----	----	----	----	----	----
855	-0.38	-0.99	-3.40	----	----	-0.54	----	----	----	----	----	----
856	-0.01	-0.47	-1.63	----	----	-0.53	----	----	----	----	----	----
857	0.21	0.93	1.39	----	----	-0.07	----	----	----	----	----	----
858	0.14	0.39	1.35	----	----	-0.28	----	----	----	----	----	----
859	0.57	1.17	1.49	----	----	-0.04	----	----	----	----	----	----
860	-0.01	0.05	-0.26	----	----	-0.02	----	----	----	----	----	----
861	-0.38	-0.73	-1.04	----	----	-0.64	----	----	----	----	----	----
862	-0.01	-0.21	1.31	----	----	-0.42	----	----	----	----	----	----
863	0.55	1.61	-0.26	----	----	-0.29	----	----	----	----	----	----
864	-0.01	-0.47	-2.03	----	----	-0.38	----	----	----	----	----	----
866	0.73	1.61	0.13	----	----	-0.35	----	----	----	----	----	----
870	-0.57	-1.77	-3.99	----	----	-0.41	----	----	----	----	----	----
963	-0.36	-1.84	0.33	----	----	0.94	----	----	----	----	----	----
974	----	----	----	----	----	----	47.02	77.71	65.53	----	----	2.99
1007	----	----	----	----	----	----	1.07	1.67	1.67	----	----	0.69
1010	0.36	0.31	1.51	----	----	-0.01	----	----	----	----	----	----
1041	-0.94	-1.77	-0.46	----	----	-0.02	----	----	----	----	----	----
1067	-0.01	0.31	-0.06	----	----	0.39	----	----	----	----	----	----
1201	-1.87	-10.33	-7.71	----	----	-4.46	----	----	----	----	----	----
1246	-7.99	-10.59	-6.34	----	----	0.04	----	----	----	----	----	----
1342	----	----	----	----	----	----	2.04	9.68	71.61	----	----	4.78
1343	----	----	----	----	----	----	----	----	----	----	----	----
1344	----	----	----	----	----	----	----	----	----	----	----	----
1354	----	----	----	----	----	----	-1.87	-6.86	-1.37	----	----	0.60
1826	----	----	----	----	----	----	-0.40	0.61	3.50	----	----	0.99
1905	-1.87	-4.10	-3.40	----	----	-0.46	----	----	----	----	----	----

APPENDIX 2**List of number of participants per country**

1 lab in BELGIUM
1 lab in BRAZIL
3 labs in CANADA
1 lab in CHILE
1 lab in FINLAND
3 labs in FRANCE
3 labs in GERMANY
1 lab in GREECE
2 labs in INDIA
1 lab in ITALY
3 labs in KOREA
4 labs in MALAYSIA
2 labs in MEXICO
2 labs in NEW ZEALAND
1 lab in NORWAY
13 labs in P.R. of CHINA
1 lab in POLAND
2 labs in SAUDI ARABIA
2 labs in SINGAPORE
6 labs in SPAIN
1 lab in THAILAND
7 labs in THE NETHERLANDS
1 lab in TURKEY
1 lab in U.A.E.
10 labs in U.S.A.
3 labs in UNITED KINGDOM
2 labs 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
E	= error in calculations
U	= reported in different unit
Ex	= excluded from calculations
n.a.	= not applicable
W	= result withdrawn on request of participant

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
- 2 ASTM E178-02
- 3 ASTM E1301-03
- 4 ISO 5725-86
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- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
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- 11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 IMPCA Methanol Reference Specifications, IMPCA, Brussels, January 2008.
- 14 ASTM E346-03e1
- 15 Analytical Methods Committee Technical brief, No4 January 2001.
- 16 The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>).