

Results of Proficiency Test  
Mono Propylene Glycol (MPG)  
October 2009

Organised by: Institute for Interlaboratory Studies (i.i.s.)  
Spijkenisse, the Netherlands

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## **1 INTRODUCTION**

Since 2004, the Institute for Interlaboratory Studies organises a proficiency test for the analysis of Monopropylene Glycol (MPG). As part of the annual proficiency test program of 2009/2010, the Institute decided to continue this proficiency test on MPG. In this international interlaboratory study, 15 laboratories in 12 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 (i.i.s.) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to send one sample (1\* 500 mL, labelled #0976) to the participants. The participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

### **2.1 ACCREDITATION**

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO 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 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (i.i.s.-protocol, version 3.1) of November 2008.

### **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

One can with approximately 25 litre of MPG was obtained from a local trader. This material was spiked with 13.25 mg Sodiumchloride. After homogenisation, the material was divided over 43 amber glass bottles of 500 mL with inner and outer caps and labelled #0976. The homogeneity of the subsamples #0976 was checked by determination of Density @ 20 °C in accordance with ASTM D 4052:02e1 and Chloride as Cl<sup>-</sup> in accordance with an in house test method on 4 stratified random selected samples.

	Density @ 20 °C in kg/L	Chloride in mg/kg
sample #0976-1	1.03978	0.33
sample #0976-2	1.03973	0.32
sample #0976-3	1.03971	0.33
sample #0976-4	1.03976	0.34

Table 1: homogeneity tests of subsamples #0976

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

	Density @ 20 °C in kg/L	Chloride in mg/kg
r (sample #0976)	0.00009	0.02
reference method	ASTM D 4052:02e1	Horwitz
0.3xR <sub>(reference)</sub>	0.00015	0.02

Table 2: repeatabilities of subsamples #0976

The repeatability of the homogeneity tests for Density @20°C was in agreement with 0.3 times the reproducibility required by ASTM D 4052:02e1.

For the determination of Chloride in MPG no reference method exists. So, the calculated repeatability was compared with 0.3 times the estimated reproducibility calculated using the Horwitz equation and was found in agreement.

Therefore, homogeneity of the subsamples #0976 was assumed.

One sample of MPG (500 mL of sample #0976) was sent to each of the participating laboratories on September 30, 2009.

## 2.5 STABILITY OF THE SAMPLES

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

## 2.6 ANALYSES

The participants were requested to determine Acidity as Acetic Acid, Appearance, Chloride as Cl, Colour Pt/Co, Distillation @ 760 mmHg (Initial Boiling Point, 50% recovered and Dry Point), Iron, Purity, Dipropylene Glycol, Density @ 20 °C, Specific Gravity @ 20/20 °C/°C and Water on sample #0976. To get comparable results a detailed report form, on which the units and the standard methods were printed, was sent together with each set of samples. Also a letter of instructions and a SDS were added to the package.

## 3 RESULTS

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

Directly after deadline, a reminder fax was sent to those laboratories that had not yet reported any results at that moment.

Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

### 3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of November 2008 (i.i.s.-protocol, version 3.1).

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.13 and 14).

### 3.3 Z-SCORES

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

As it was decided to evaluate the performance of the participants against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8.

The z-scores were calculated in accordance with:

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

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

## 4 EVALUATION

In this proficiency test, no problems were encountered with the despatch of the samples. Three participants reported the results after the final reporting date. All participants, except one reported results. The 12 participants did report 113 numerical results.

Observed were 9 outlying results, which is 8.0 % of the 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 all data sets proved to have a normal distribution. Not a normal distribution was found for Distillation (Dry Point) and Density. For these determinations the statistical evaluation should be used with due care.

Acidity: This determination is not problematic. One statistical outlier was observed and the calculated reproducibility is in good agreement with the requirements of ASTM E 202:05.

Appearance: No analytical problems were observed. All labs agreed about the appearance of the sample #0976, 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').

Chloride: This determination is problematic for one laboratory. One statistical outlier was observed. The calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the estimated requirements calculated using the Horwitz equation. Although the Chloride content in the blank is not known, the average recovery of the Chloride compared to the theoretical increment of 0.32 mg Cl/kg is good (<106%).

Colour Pt/Co: This determination is not problematic. One statistical outlier was observed and the calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the requirement of ASTM E202:05.

Distillation: This determination was problematic. No statistical outliers were observed. The calculated reproducibilities of the IBP and 50% recovered are respectively almost and not in agreement with the requirements of ASTM E202:05, while the calculated reproducibility for DP is in good agreement with the requirements of ASTM E 202:05. Regretfully, it was noticed that five of the seven reporting laboratories did not correct for barometric pressure as prescribed in ASTM D1078 (theoretical boiling point = 187.6°C). When manually corrected for barometric pressure the calculated reproducibilities for 50% recovered and DP are in agreement, while only the calculated reproducibility for IBP is not in agreement.

Iron: This determination is not problematic. Two statistical outliers were observed. The calculated reproducibility is, after rejection of the

statistical outliers, in good agreement with the requirements of ASTM E202:05.

Purity on dry basis: This determination is not problematic. Only one statistical outlier was observed. The calculated reproducibility is, after rejection of the statistical outlier, in good agreement with ASTM E202:05.

DPG: This determination is problematic for only one laboratory that reported a false negative result. No statistical outliers were observed. The calculated reproducibility is, after exclusion of one result, in good agreement with the requirements of ASTM E202:05.

Density: The determination is not problematic. One statistical outlier was observed. The calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the requirements of ASTM D4052:02e1.

Specific Gravity: The determination is problematic for one laboratory. One statistical outlier was observed. The calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the requirements of ASTM E202:05.

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the requirements of ASTM E202:05.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acetic Acid	%M/M	9	0.00052	0.00044	0.00080
Chloride as Cl	mg/kg	4	0.34	0.09	0.18
Colour Pt/Co	---	6	2.5	1.6	7.0
Initial Boiling Point	°C	7	186.80	0.54	0.50
50% recovered	°C	7	187.37	0.60	0.40
Dry Point	°C	7	188.29	1.24	2.50
Iron	mg/kg	7	0.176	0.031	0.070
Purity	%M/M	11	99.898	0.048	0.170
Dipropylene Glycol	%M/M	10	0.0727	0.0716	0.1400
Density @ 20°C	kg/L	12	1.03611	0.00028	0.00050
Specific Gravity 20/20°C/°C	---	11	1.03797	0.00030	0.00050
Water	%M/M	12	0.0171	0.0081	0.0500

Table 4: reproducibilities of sample #0976

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

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2009 WITH PREVIOUS PT

	October 2009	October 2007	October 2006	October 2005
Number of reporting labs	12	11	10	11
Number of results reported	113	106	108	134
Statistical outliers	9	6	0	8
Percentage outliers	8.0%	5.7%	0.0%	6.0%

Table 5: comparison with previous proficiency tests.

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

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

Determination	October 2009	October 2007	October 2006	October 2005
Acidity as Acetic Acid	++	++	++	++
Chloride as Cl	++	++	++	--
Colour Pt/Co	++	++	++	++
Initial Boiling Point	-	--	--	--
50% recovered	--	n.e.	n.e.	n.e.
Dry Point	++	++	++	+
Iron	++	--	--	++
Purity	++	++	++	+/-
Dipropylene Glycol	++	++	++	++
Density @ 20°C	++	++	++	++
Specific Gravity 20/20°C/°C	++	++	++	++
Water	++	++	++	++

Table 6: comparison determinations against the standard

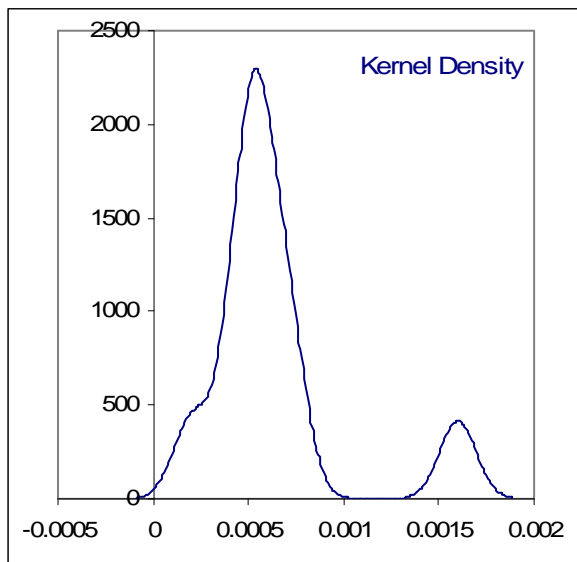
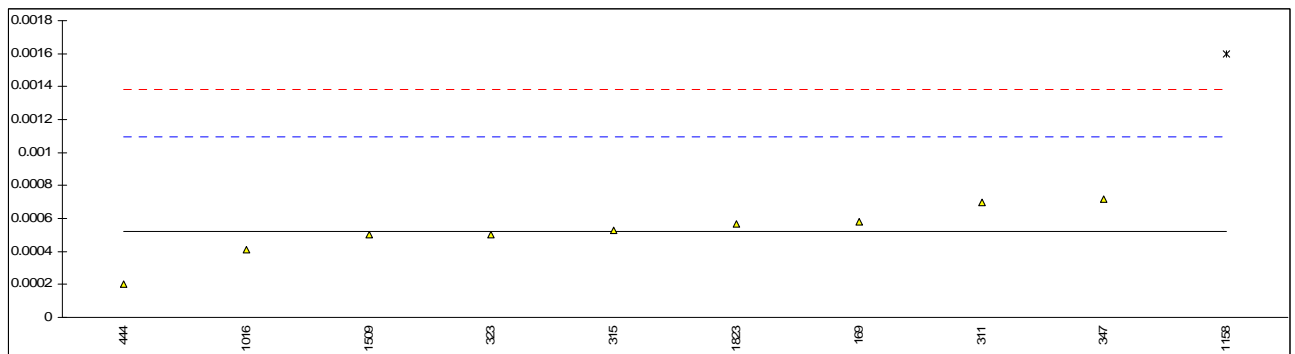
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

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

**APPENDIX 1**

Determination of Acidity as Acetic Acid on sample #0976; results in %M/M.

lab	method	value	mark	z(targ)	remarks
169	D1613	0.000578		0.19	
311	D1613	0.0007		0.62	
315	D1613	0.00053		0.02	
323	D1613	0.0005		-0.08	
334		-----		-----	
347	D1613	0.00072		0.69	
444	INH-370	0.0002		-1.13	
446		-----		-----	
913		-----		-----	
1016	D1613	0.00041		-0.40	
1059		-----		-----	
1158	D1613	0.0016	G(0.01)	3.77	
1509	D1613	0.00050		-0.08	
1603		-----		-----	
1823	D1613	0.00057		0.16	
	normality	OK			
	n	9			
	outliers	1			
	mean (n)	0.00052			
	st.dev. (n)	0.000156			
	R(calc.)	0.00044			
	R(E202:05)	0.00080			



## Determination of Appearance on sample #0976.

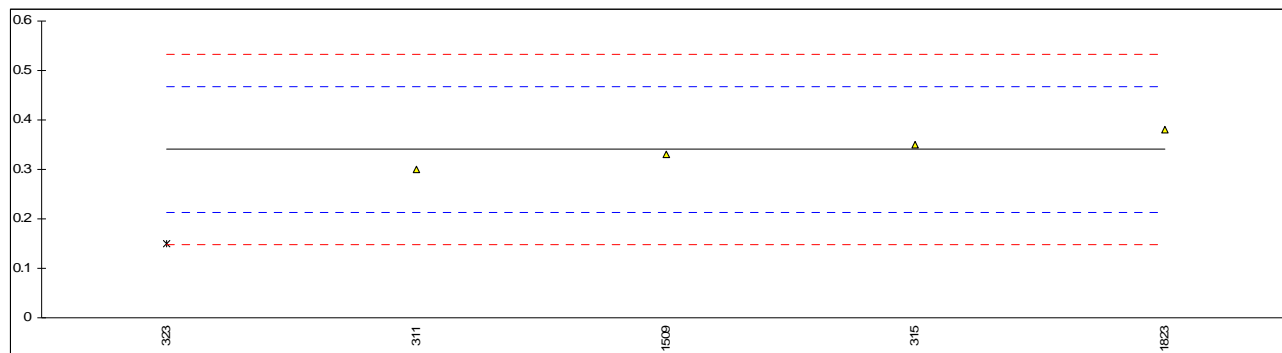
lab	method	value	mark	z(targ)	remarks
169	E2680	CFSM		----	
311	E2680	PASS		----	
315	INH-402	PASS		----	
323	E2680	PASSES		----	
334		----		----	
347	E2680	CFSM		----	
444	VISUAL	PASS		----	
446	D4670	CFFMIS		----	
913		----		----	
1016	E2680	PASS		----	
1059		----		----	
1158		----		----	
1509	D4176	CFSM		----	
1603		----		----	
1823	E2680	PASS		----	

CFSM = Clear free of suspended matter

CFFMIS = Clear free form miscellaneous in suspension

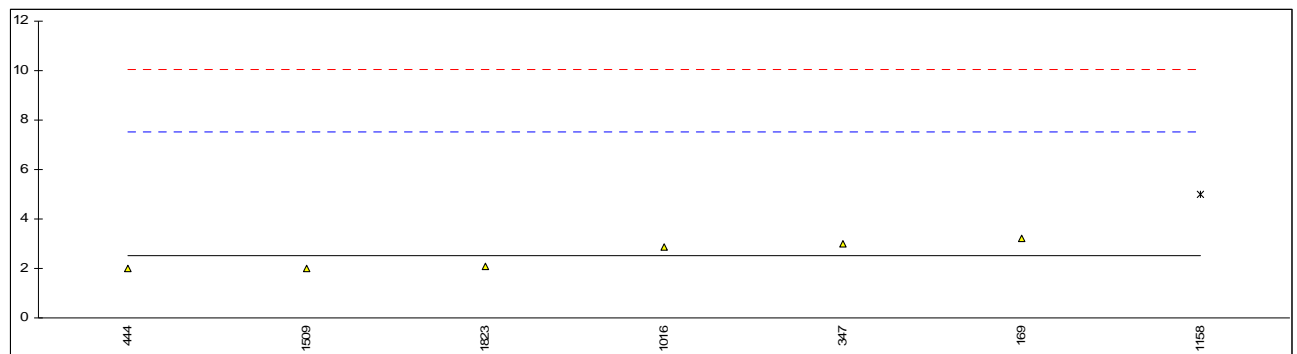
Determination of Chloride as Cl on sample #0976; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
169		----		----	
311	INH-158	0.3		-0.63	
315	INH-158	0.351		0.17	
323	E2469	0.15	C,G(0.05)	-2.97	First reported 0.65
334		----		----	
347		----		----	
444	INH-867	<0.5		----	
446		----		----	
913		----		----	
1016		----		----	
1059		----		----	
1158		----		----	
1509	INHOUSE	0.330		-0.16	
1603		----		----	
1823	INHOUSE	0.380		0.62	
	normality	n.a.			
	n	4			
	outliers	1	<u>Spike</u>		
	mean (n)	0.34	0.32		<106% recovered
	st.dev. (n)	0.034			
	R(calc.)	0.09			
	R(Horwitz)	0.18			



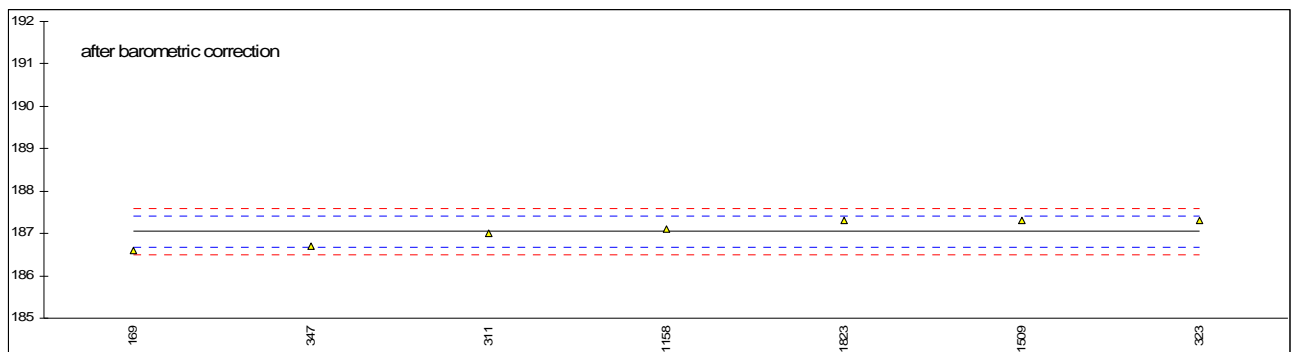
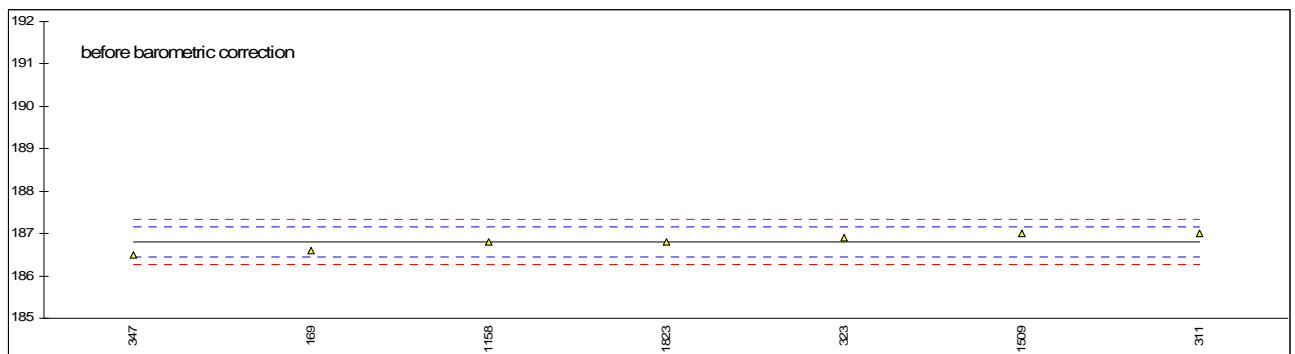
Determination of Colour Pt/Co on sample #0976;

lab	method	value	mark	z(targ)	remarks
169	D5386	3.2		0.27	
311	D1209	<5		----	
315	D1209	<5		----	
323	D1209	<5		----	
334		----		----	
347	D1209	3		0.19	
444	D5386	2		-0.21	
446	D1209	<5		----	
913		----		----	
1016	D1209	2.89		0.14	
1059		----		----	
1158	D1209	5	D(0.05)	0.99	
1509	D1209	2		-0.21	
1603		----		----	
1823	D5386	2.1		-0.17	
normality		OK			
n		6			
outliers		1			
mean (n)		2.5			
st.dev. (n)		0.56			
R(calc.)		1.6			
R(E202:05)		7.0			



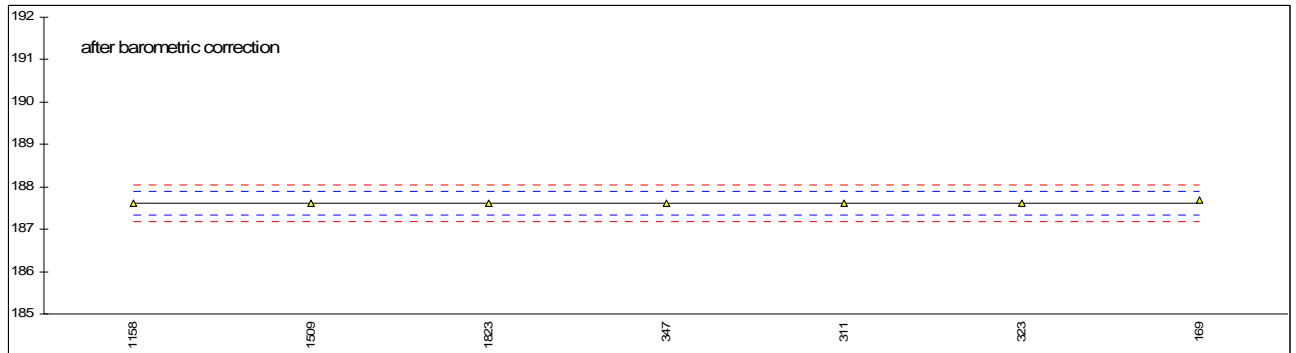
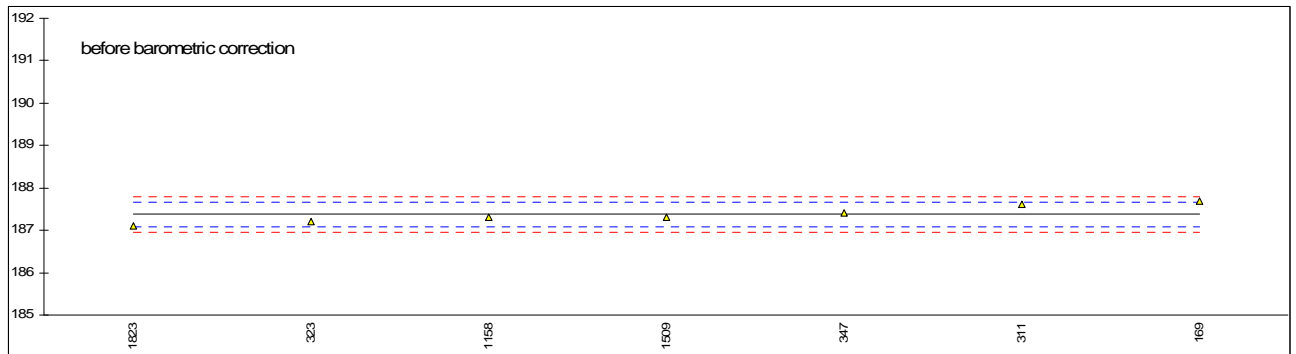
Determination of Initial Boiling Point @ 760 mmHg on sample #0976; results in °C.

lab	method	value	mark	z(targ)	value after manual correction	mark	z(targ)	remarks	
169	D1078	186.6		-1.12	186.6		-2.48		
311	D1078	187.0		1.12	187.0		-0.24		
315		----		----	----		----		
323	D1078	186.9		0.56	187.3		1.44		
334		----		----	----		----		
347	D1078	186.5		-1.68	186.7		-1.92		
444		----		----	----		----		
446		----		----	----		----		
913		----		----	----		----		
1016		----		----	----		----		
1059		----		----	----		----		
1158	D1078	186.8		0.00	187.1		0.32		
1509	D1078	187.0		1.12	187.3		1.44		
1603		----		----	----		----		
1823	D1078	186.8		0.00	187.3		1.44		
normality		OK			OK				
n		7			7				
outliers		0			0				
mean (n)		186.80			187.04				
st.dev. (n)		0.191			0.294				
R(calc.)		0.54			0.82				
R(E202:05)		0.50			0.50				



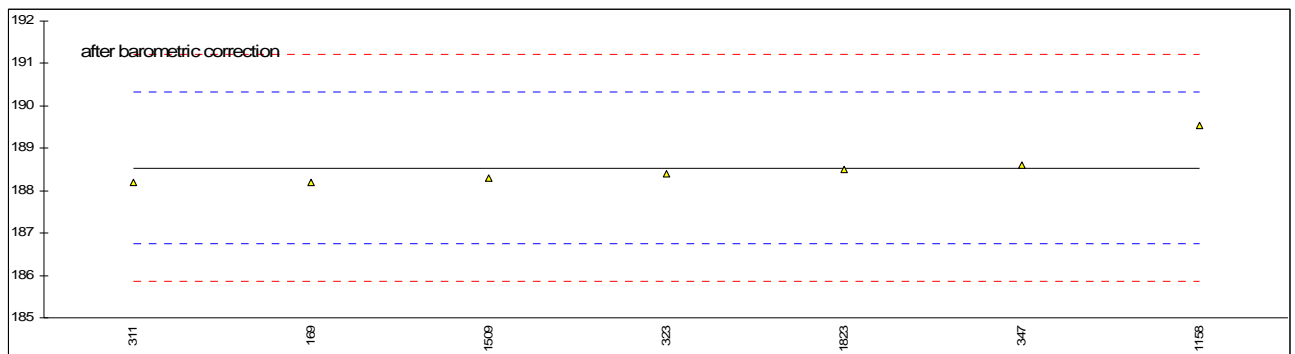
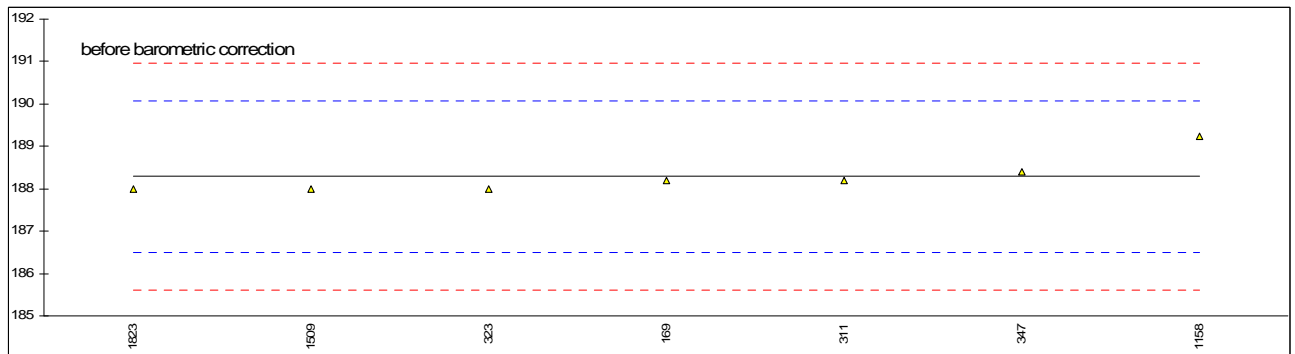
Determination of 50% recovered @ 760 mmHg on sample #0976; results in °C

lab	method	value	mark	z(targ)	value after manual correction	mark	z(targ)	remarks
169	D1078	187.7		2.30	187.7		0.60	
311	D1078	187.6		1.60	187.6		-0.10	
315		----		----	----		----	
323	D1078	187.2		-1.20	187.6		-0.10	
334		----		----	----		----	
347	D1078	187.4		0.20	187.6		-0.10	
444		----		----	----		----	
446		----		----	----		----	
913		----		----	----		----	
1016		----		----	----		----	
1059		----		----	----		----	
1158	D1078	187.3		-0.50	187.6		-0.10	
1509	D1078	187.3		-0.50	187.6		-0.10	
1603		----		----	----		----	
1823	D1078	187.1		-1.90	187.6		-0.10	
	normality	OK			not OK			
	n	7			7			
	outliers	0			0			
	mean (n)	187.37			187.61			Theoretical point = 187.6°C
	st.dev. (n)	0.214			0.038			
	R(calc.)	0.60			0.11			
	R(E202:05)	0.40			0.40			



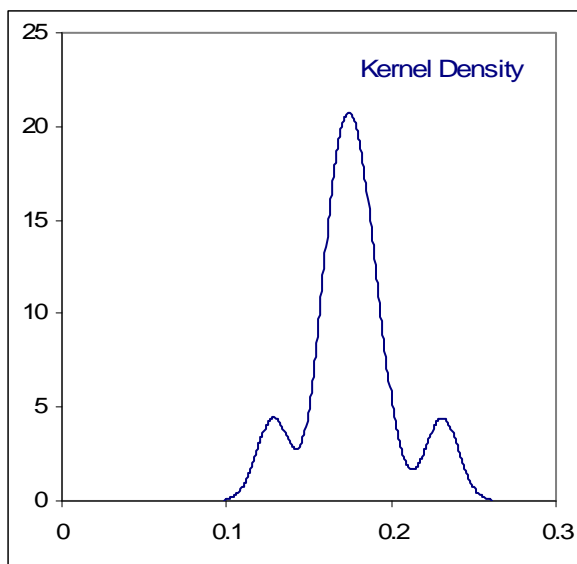
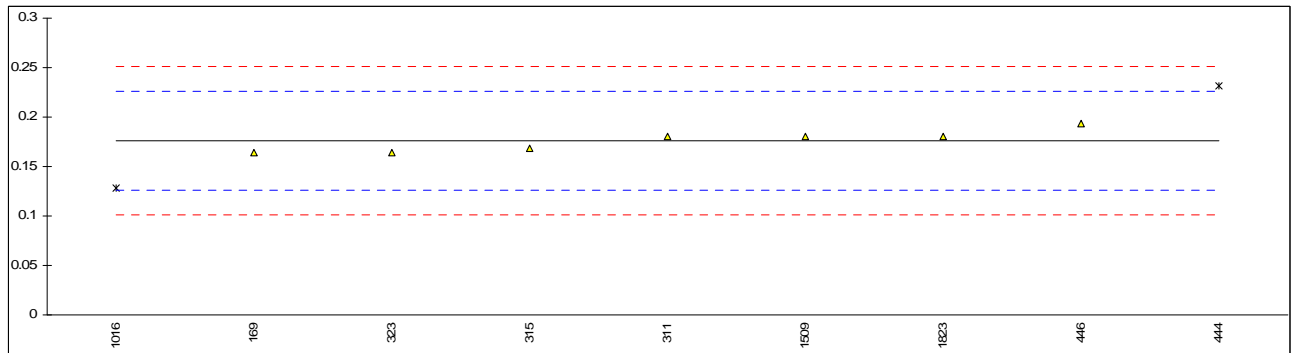
Determination of Dry Point @ 760 mmHg on sample #0976; results in °C.

lab	method	value	mark	z(targ)	value after manual correction	mark	z(targ)	remarks
169	D1078	188.2		-0.10	188.2		-0.37	
311	D1078	188.2		-0.10	188.2		-0.37	
315		----		----	----		----	
323	D1078	188.0		-0.33	188.4		-0.15	
334		----		----	----		----	
347	D1078	188.4		0.12	188.6		0.07	
444		----		----	----		----	
446		----		----	----		----	
913		----		----	----		----	
1016		----		----	----		----	
1059		----		----	----		----	
1158	D1078	189.233		1.06	189.533		1.12	
1509	D1078	188.0		-0.33	188.3		-0.26	
1603		----		----	----		----	
1823	D1078	188.0		-0.33	188.5		-0.04	
	normality	OK			not OK			
	n	7			7			
	outliers	0			0			
	mean (n)	188.29			188.53			
	st.dev. (n)	0.442			0.465			
	R(calc.)	1.24			1.30			
	R(E202:05)	2.50			2.50			



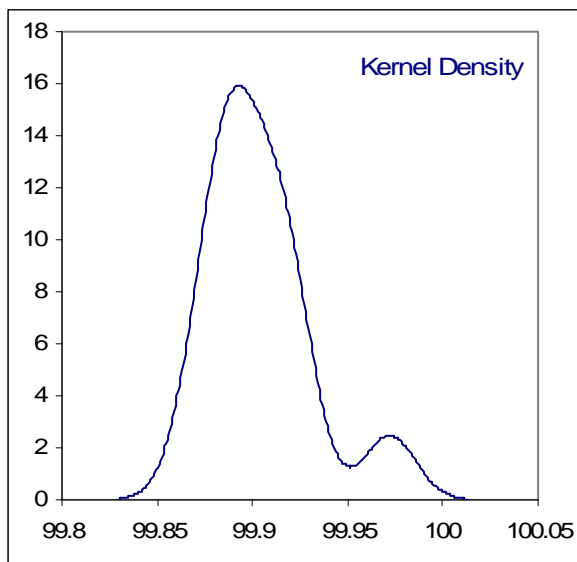
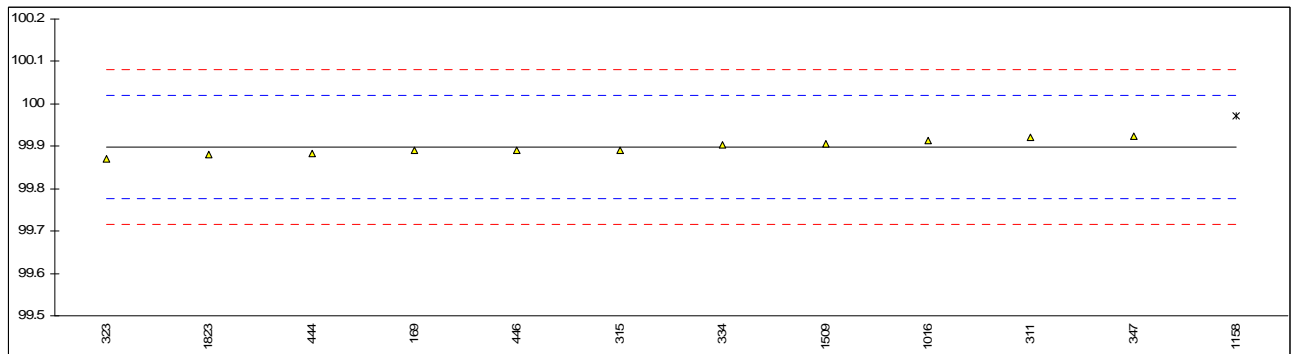
Determination of Iron as Fe on sample #0976; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
169	E1615	0.1637		-0.48	
311	E202	0.18		0.17	
315	E202	0.168		-0.31	
323	E1615	0.164		-0.47	
334		----		----	
347		----		----	
444	E202	0.231	DG(0.05)	2.21	
446	E202	0.194	C	0.73	First reported 0.0002
913		----		----	
1016	NEN0966	0.1285	DG(0.05)	-1.89	
1059		----		----	
1158		----		----	
1509	E202	0.180		0.17	
1603		----		----	
1823	E202	0.18		0.17	
normality		OK			
n		7			
outliers		2			
mean (n)		0.176			
st.dev. (n)		0.0110			
R(calc.)		0.031			
R(E202:05)		0.070			



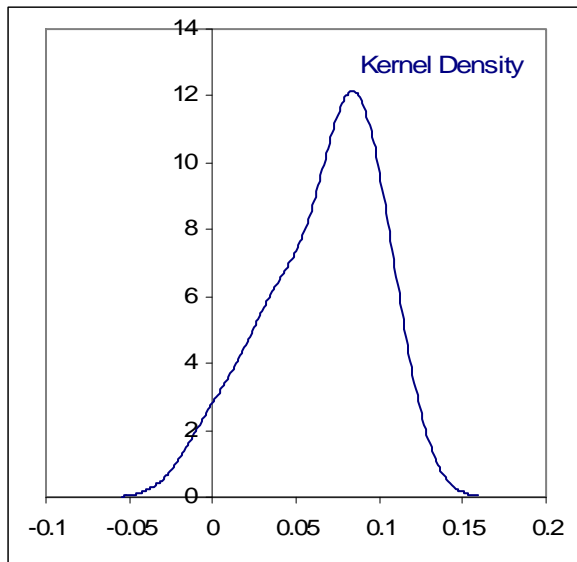
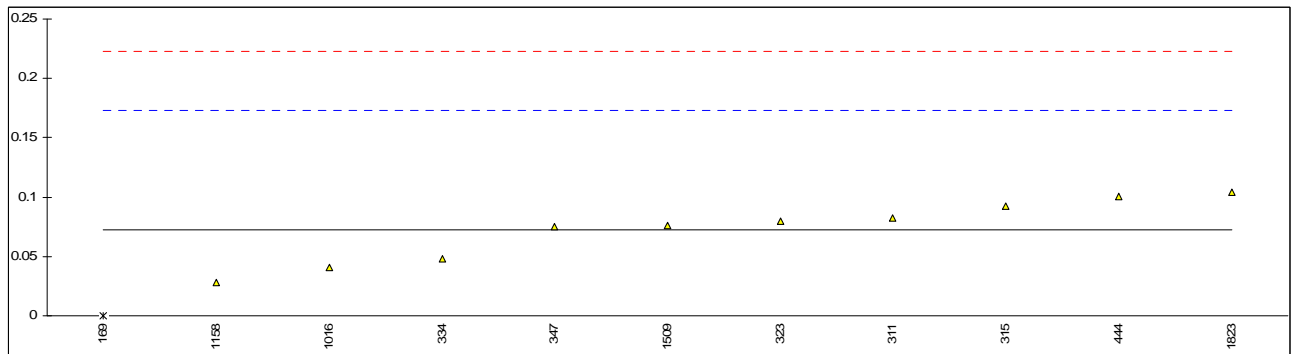
Determination of Purity on sample #0976; results in %M/M.

lab	method	value	mark	z(targ)	remarks
169	E202	99.8895		-0.13	
311	E202	99.92		0.37	
315	INHOUSE	99.891		-0.11	
323	E202	99.87		-0.45	
334	INH-130	99.9037		0.10	
347	E202	99.923		0.42	
444	INH-687	99.884		-0.22	
446	INH-130	99.89		-0.12	
913		----		----	
1016	INHOUSE	99.914		0.27	
1059		----		----	
1158	INH-003	99.9722	G(0.05)	1.23	
1509	E202	99.907		0.16	
1603		----		----	
1823	E2409	99.881		-0.27	
normality		OK			
n		11			
outliers		1			
mean (n)		99.898			
st.dev. (n)		0.0171			
R(calc.)		0.048			
R(E202:05)		0.170			



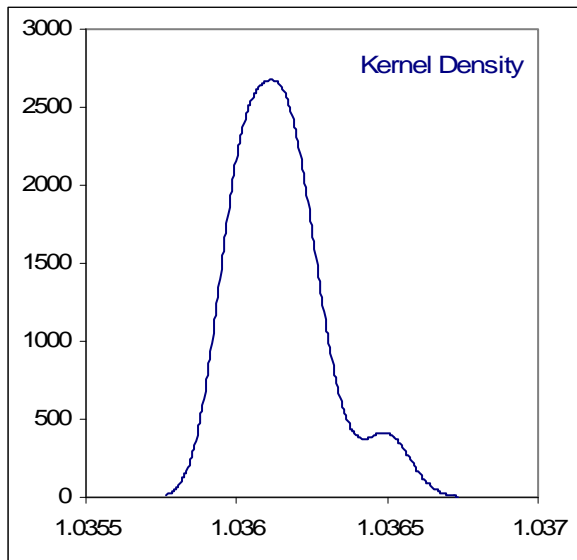
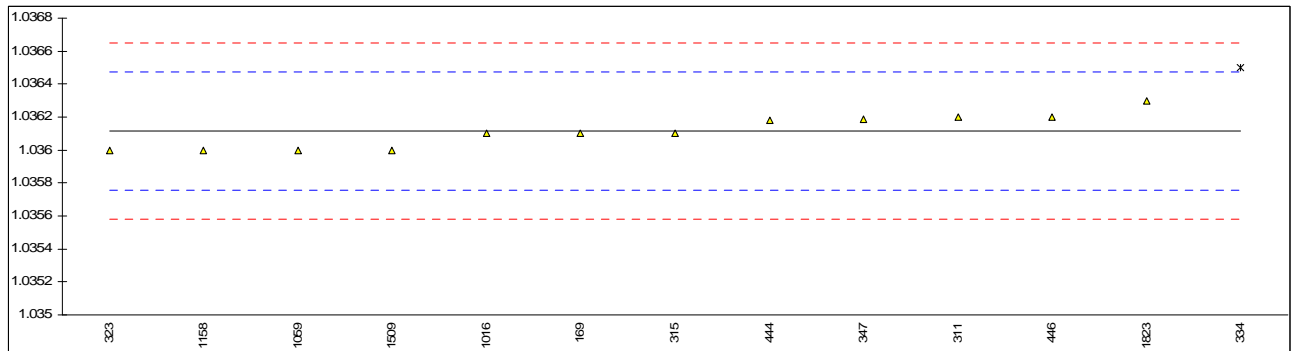
Determination of Dipropylene Glycol on sample #0976; results in %M/M.

lab	method	value	mark	z(targ)	remarks
169	E202	0.0000	ex	-1.45	Result excluded, not a real result. False negative?
311	E202	0.082		0.19	
315	INHOUSE	0.0924		0.39	
323	E202	0.08		0.15	
334	INH-130	0.0484		-0.49	
347	E202	0.075		0.05	
444	INH-687	0.1002		0.55	
446		-----		-----	
913		-----		-----	
1016	INHOUSE	0.041		-0.63	
1059		-----		-----	
1158	INH-003	0.0278		-0.90	
1509	E202	0.076		0.07	
1603		-----		-----	
1823	E2409	0.104		0.63	
normality		OK			
n		10			
outliers		1			
mean (n)		0.0727			
st.dev. (n)		0.02559			
R(calc.)		0.0716			
R(E202:05)		0.1400			



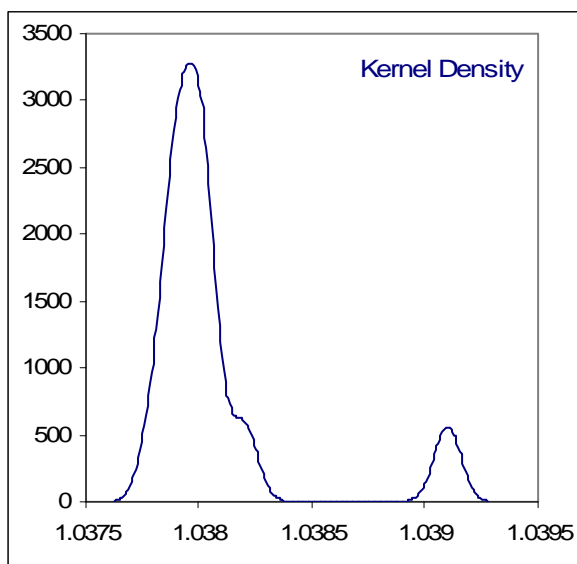
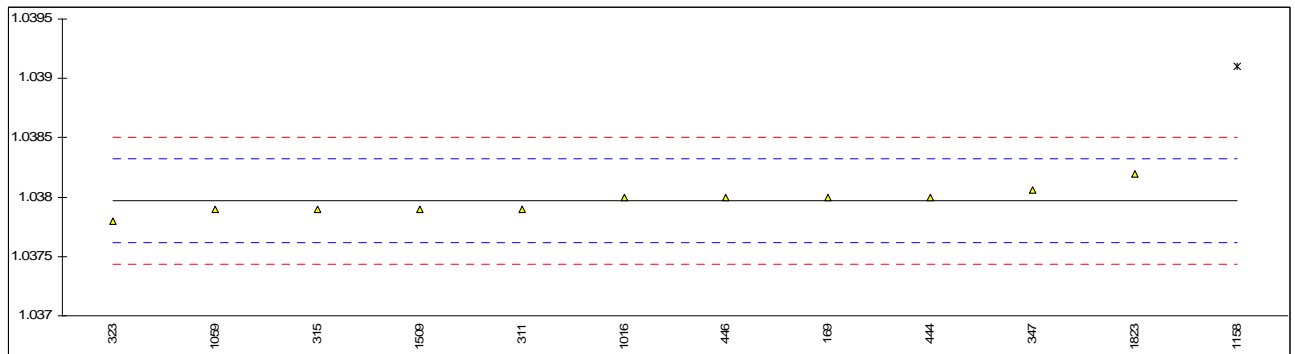
Determination of Density @ 20 °C on sample #0976; results in kg/L.

lab	method	value	mark	z(targ)	remarks
169	D4052	1.0361		-0.08	
311	D4052	1.0362		0.48	
315	D4052	1.0361		-0.08	
323	D4052	1.0360		-0.64	
334	D4052	1.0365	G(0.05)	2.16	
347	D4052	1.03619		0.43	
444	D4052	1.03618		0.37	
446	D4052	1.0362		0.48	
913		-----		-----	
1016	D4052	1.0361		-0.08	
1059	D4052	1.0360		-0.64	
1158	D891	1.036	C	-0.64	First reported 1.037
1509	D4052	1.0360		-0.64	
1603		-----		-----	
1823	D4052	1.0363		1.04	
normality		OK			
n		12			
outliers		1			
mean (n)		1.03611			
st.dev. (n)		0.000101			
R(calc.)		0.00028			
R(D4052:02e1)		0.00050			



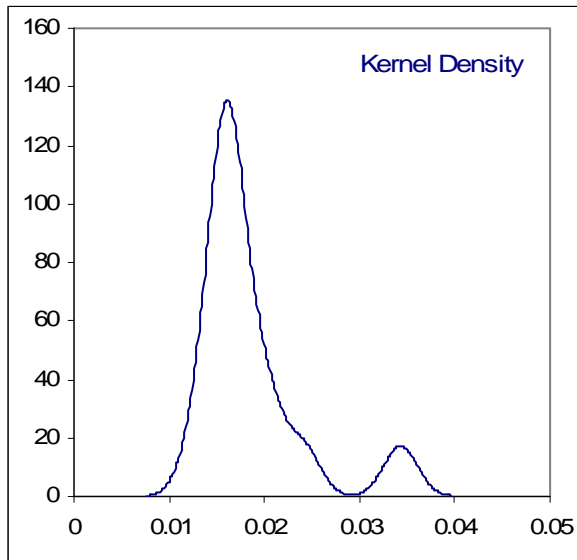
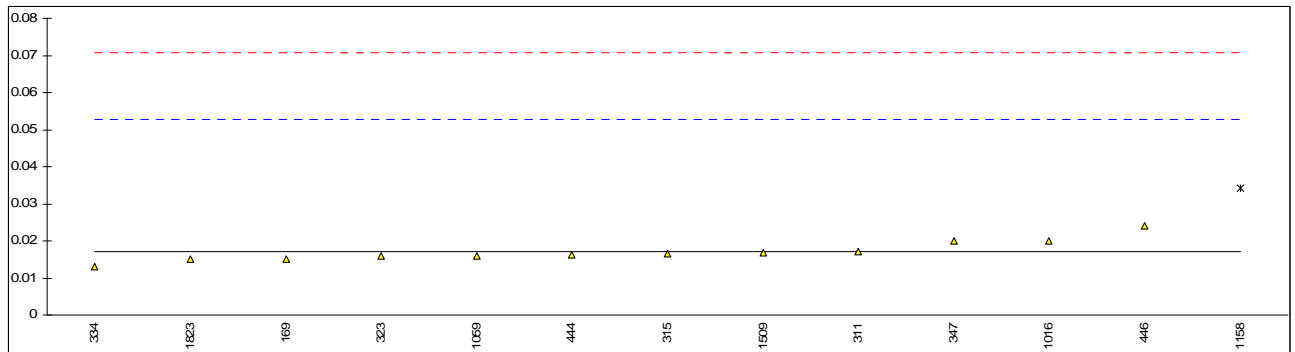
Determination of Specific Gravity 20/20 °C/°C on sample #0976;

lab	method	value	mark	z(targ)	remarks
169	D4052	1.0380		0.17	
311	D4052	1.0379		-0.39	
315	D4052	1.0379		-0.39	
323	D4052	1.0378		-0.95	
334		----		----	
347	D4052	1.03806		0.51	
444	D4052	1.0380		0.17	
446	D4052	1.0380		0.17	
913		----		----	
1016	D4052	1.0380		0.17	
1059	D4052	1.0379		-0.39	
1158	D891	1.0391	G(0.01)	6.33	
1509	D4052	1.0379		-0.39	
1603		----		----	
1823	D4052	1.0382		1.29	
normality		OK			
n		11			
outliers		1			
mean (n)		1.03797			
st.dev. (n)		0.000106			
R(calc.)		0.00030			
R(E202:05)		0.00050			



Determination of Water on sample #0976; results in %M/M.

lab	method	value	mark	z(targ)	remarks
169	E1064	0.0151		-0.11	
311	E1064	0.017		-0.01	
315	E1064	0.0165		-0.04	
323	E1064	0.016		-0.06	
334	E1064	0.0130		-0.23	
347	E1064	0.0200		0.16	
444	E203	0.0162		-0.05	
446	E203	0.0240		0.38	
913		-----		-----	
1016	D1364	0.020		0.16	
1059	E1064	0.016		-0.06	
1158	E203	0.0343	G(0.01)	0.96	
1509	E203	0.0168		-0.02	
1603		-----		-----	
1823	E1064	0.0151		-0.11	
normality		not OK			
n		12			
outliers		1			
mean (n)		0.0171			
st.dev. (n)		0.00291			
R(calc.)		0.0081			
R(E202:05)		0.0500			



## **APPENDIX 2**

### **List of number of participants**

- 1 laboratory in BELGIUM
- 1 laboratory in FRANCE
- 1 laboratory in GERMANY
- 1 laboratory in INDIA
- 1 laboratory in P.R.of CHINA
- 1 laboratory in ROMANIA
- 1 laboratory in SINGAPORE
- 1 laboratory in SLOVENIA
- 1 laboratory in SPAIN
- 3 laboratories in THE NETHERLANDS
- 1 laboratory in U.S.A.
- 2 laboratories in UNITED KINGDOM

## APPENDIX 3

### Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
E	= error in calculations
ex	= excluded from calculations
n.a.	= not applicable

### Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, November 2003
- 2 ASTM E178-89
- 3 ASTM E1301-89
- 4 ISO 13528-05
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- 5 ISO 5725, parts 1-6, 1994
- 6 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 7 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 8 IP 367/84
- 9 DIN 38402 T41/42
- 10 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 11 J.N. Miller, Analyst, 118, 455, (1993)
- 12 W. Horwitz and R. Albert, J. AOAC Int., Vol. 79, 3, p. 589, (1996)
- 13 Analytical Methods Committee Technical brief, No4 January 2001.
- 14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>).