Results of Proficiency Test
Mono Propylene Glycol (MPG)
October 2013

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

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Report: iis13C11

January 2014

CONTENTS

Appendices:

1 INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies has organised a proficiency test for the analysis of Monopropylene Glycol (MPG). As part of the annual proficiency test program of 2013/2014, the Institute decided to continue this proficiency test on MPG. In this interlaboratory study, 19 laboratories in 11 different countries have participated. See appendix 2 for the number of participants per country. In this report the results of 2013 MPG proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted. It was decided to send one sample (1* 500 mL, labelled #13196) 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 accordance with ISO/IEC 17043:2010, since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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), which can be downloaded from www.iisnl.com.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

One can with approximately 25 litre of MPG was obtained from a local trader. After homogenisation, the material was divided over 50 amber glass bottles of 500 mL with inner and outer caps and labelled #13196. The homogeneity of the subsamples #13196 was checked by determination of Density @ 20°C in accordance with ASTM D 4052:02e1 on eight stratified randomly selected samples.

	Density @ 20°C in kg/L
sample #13196-1	1.03611
sample #13196-2	1.03612
sample #13196-3	1.03612
sample #13196-4	1.03610
sample #13196-5	1.03610
sample #13196-6	1.03610
sample #13196-7	1.03610
sample #13196-8	1.03611

Table 1: homogeneity tests results of subsamples #13196

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
r (sample #13196)	0.00002
reference method	ASTM D4052:02e1
$0.3xR_{(reference)}$	0.00015

Table 2: repeatabilities of subsamples #13196

The calculated repeatability for Density is in agreement with 0.3 times the corresponding reproducibility of the target method. Therefore, homogeneity of the subsamples #13196 was assumed.

One sample of MPG (500 ml bottle, labelled #13196) was sent to each of the participating laboratories on October 9, 2013.

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/^{o}C and Water on sample #13196.
To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website (www.iisnl.com).
A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were 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 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.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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 can be found on the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.13 and 14).

3.3 Z-SCORES

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

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8.
The z-scores were calculated in accordance with:

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

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

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate the fit-for-useness of the reported test result.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. The usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test, no problems were encountered with the despatch of the samples. One participant reported the results after the final reporting date. All participants reported results. The 19 participants did report 189 numerical results. Observed were 5 outlying results, which is 2.7% 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. The latest standardized method available for analysis of Ethylene Glycols and Propylene Glycols is ASTM E202:2012. This version was not used for the evaluation of all tests, as no precision data are mentioned for Propylene Glycols in this test method. Therefore the previous version ASTM E202:2005 was used for the evaluation of Acidity, Distillation (IBP, 50% recovered, Dry Point), Iron and Water.
Not all data sets proved to have a normal distribution. A not normal distribution was found for Colour Pt/Co, Density and Specific Gravity. For these determinations the statistical evaluation should be used with due care.

Acidity: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM E202:05.

Appearance: A new standardized method is available for Appearance since 2009, being ASTM E2680. According to this method, the appearance should be reported as ‘pass’ (or ‘fail’). All laboratories, except five, reported the appearance as pass.

Chloride: Regretfully, the consensus value of the group may be near or below the limit of detection of the test methods used. Therefore no significant conclusions were drawn.

Colour Pt/Co: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirement of ASTM E202:12.

Distillation: This determination was problematic. In total two statistical outliers were observed. However the calculated reproducibility of IBP is not in agreement with the requirements of ASTM E202:05, while the calculated reproducibility for 50% Recovered and Dry Point is in good agreement with the requirements of ASTM E202:05. Regretfully, it was noticed that six of the eleven reporting laboratories did not correct sufficiently for either the thermometer deviation and/or the barometric pressure as prescribed by ASTM. When manually corrected to the theoretical boiling point as prescribed in ASTM D1078 (theoretical boiling point = 187.6ºC), the calculated reproducibilities for IBP, 50% recovered and DP are all in good agreement.

Iron: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM E202:05.

Purity: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM E202:12.

DPG: 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 E202:12.

Density: 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 D4052:02e1.

Specific Gravity: 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 E202:12.

Water: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is 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	18	0.0005	0.0006	0.0008
Appearance		18	pass	n.a.	n.a.
Chloride as Cl	mg/kg	5	0.12	n.a.	(0.07)*
Colour Pt/Co		14	2	2	7
Initial Boiling Point	°C	10	187.1	0.6	0.5
50% recovered	°C	10	187.5	0.3	0.4
Dry Point	°C	11	187.9	0.9	2.5
Iron	mg/kg	15	0.05	0.05	0.07
Purity	%M/M	17	99.95	0.06	0.17
Dipropylene Glycol	%M/M	15	0.0350	0.0269	0.1400
Density @ 20^{o}C	kg/L	16	1.0362	0.0002	0.0005
Specific Gravity 20/20°C/^{o}C		16	1.0380	0.0002	0.0005
Water	%M/M	19	0.01	0.01	0.05

Table 3: reproducibilities of sample #13196

*values between brackets may be near or below limit of detection

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 2013 WITH PREVIOUS PT

	October 2013	October 2011	October 2009	October 2007
Number of reporting labs	19	18	12	11
Number of results reported	189	185	113	106
Statistical outliers	5	6	9	6
Percentage outliers	2.7%	3.2%	8.0%	5.7%

Table 4: 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 checked against the requirements of the respective standards. The conclusions are given the following table:

Determination	October 2013	October 2011	October 2009	October 2007
Acidity as Acetic Acid	+	++	++	++
Chloride as Cl	n.e.	++	++	++
Colour Pt/Co	++	++	++	++
Initial Boiling Point	-	-	-	--
50% recovered	+	--	--	n.e.
Dry Point	++	++	++	++
Iron	++	++	++	--
Purity	++	++	++	++
Dipropylene Glycol	++	++	++	++
Density @ 20^{o}C	++	++	++	++
Specific Gravity 20/20°C/^{o}C	++	++	++	++
Water	++	++	++	++

Table 5: comparison determinations against the standard

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

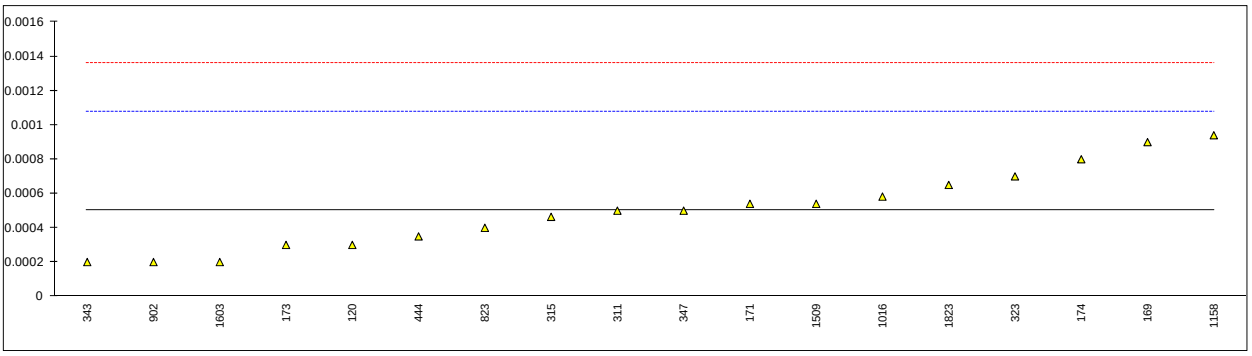
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

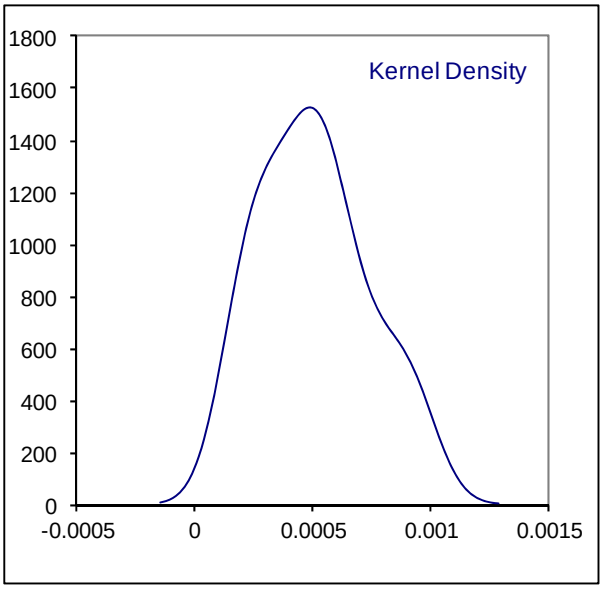
APPENDIX 1

Determination of Acidity as Acetic Acid on sample #13196; results in %M/M.

lab	method	value	mark	z(targ)	remarks
120	D1613	0.0003	C	-0.71	First reported as 0.003
169	D1613	0.0009		1.39	
171	E2679	0.00054	C	0.13	Reported as 5.4 (probably unit error)
173	D1613	0.0003	C	-0.71	Reported as 3 (probably unit error)
174	D1613	0.0008		1.04	
311	D1613	0.0005		-0.01	
315	INH-570	0.000464		-0.14	
323	E2679	0.0007		0.69	
343	INH-CM	0.0002		-1.06	
347	D1613	0.0005		-0.01	
444	E202	0.00035		-0.54	
446		-----		-----	
823	D1613	0.0004		-0.36	
902	E2679	0.0002		-1.06	
1016	D1613	0.000582		0.27	
1158	E202	0.00094		1.53	
1509	D1613	0.00054		0.13	
1603	in house	0.0002		-1.06	
1823	D1613	0.00065		0.51	

normality	OK
n	18
outliers	0
mean (n)	0.00050
st.dev. (n)	0.000230
R(calc.)	0.00064
R(E202:05)	0.00080





Determination of Appearance on sample #13196

lab	method	value	mark	z(targ)	remarks
120	E2680	Pass		-----	
169	E2680	BC&FSM		-----	
171	E2680	Pass		-----	
173	Visual	CFSM		-----	
174	E2680	Pass		-----	
311	E2680	Pass		-----	
315	INH-402	C&B		-----	
323	E2680	Pass		-----	
343	E2680	Pass		-----	
347	E2680	Pass		-----	
444	E2680	Pass		-----	
446	E2680	Pass		-----	
823	E2680	Pass		-----	
902	E2680	Pass		-----	
1016	in house	C&B		-----	
1158		-----		-----	
1509	E2680	Pass		-----	
1603	in house	Clear liquid		-----	
1823	E2680	Pass		-----	

normality	n.a.
n	18
outliers	n.a.
mean (n)	pass
st.dev. (n)	n.a.
R(calc.)	n.a.
R(E2680:09)	n.a.

CF = Clear and Free
B&C = Bright and Clear
CFSM = Clear Free of Suspended Matter

Determination of Chloride as Cl on sample #13196; results in mg/kg.

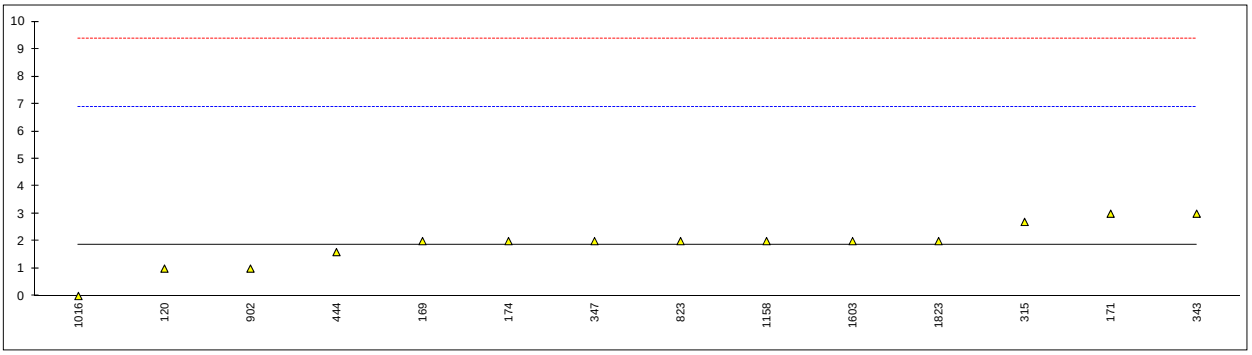
lab	method	value	mark	z(targ)	remarks
120	INH-0221	<0.5		-----	
169		-----		-----	
171		-----		-----	
173	INH-0221	<1		-----	
174	E2469	0.081		-----	
311	INH-158	<0.2		-----	
315	INH-158	0.13		-----	
323		-----		-----	
343	INH-CM	<0.5		-----	
347		-----		-----	
444		-----		-----	
446	INH-3221	<1		-----	
823		-----		-----	
902		-----		-----	
1016		-----		-----	
1158		-----		-----	
1509	in house	0.023		-----	
1603	in house	0.25		-----	
1823	INH-1677	0.12		-----	
	normality	OK			
	n	5			
	outliers	0			
	mean (n)	0.121			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(Horwitz)	(0.074)*			

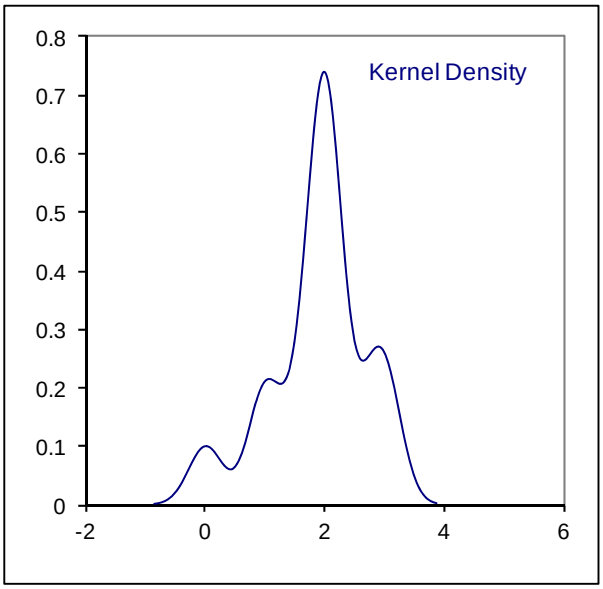
*values between brackets may be near or below limit of detection

Determination of Colour Pt/Co on sample #13196

lab	method	value	mark	z(targ)	remarks
120	D1209	1		-0.35	
169	D5386	2.0		0.05	
171	D1209	3		0.45	
173	D1209	<5		-----	
174	D1209	2		0.05	
311	D1209	<5		-----	
315	D5386	2.7		0.33	
323	D1209	<5		-----	
343	D5386	3		0.45	
347	D5386	2		0.05	
444	E202	1.6		-0.11	
446	D1209	<5		-----	
823	D5386	2		0.05	
902	D5386	1		-0.35	
1016	D1209	0		-0.75	
1158	E202	2		0.05	
1509	D1209	<5		-----	
1603	in house	2		0.05	
1823	D5386	2.0		0.05	

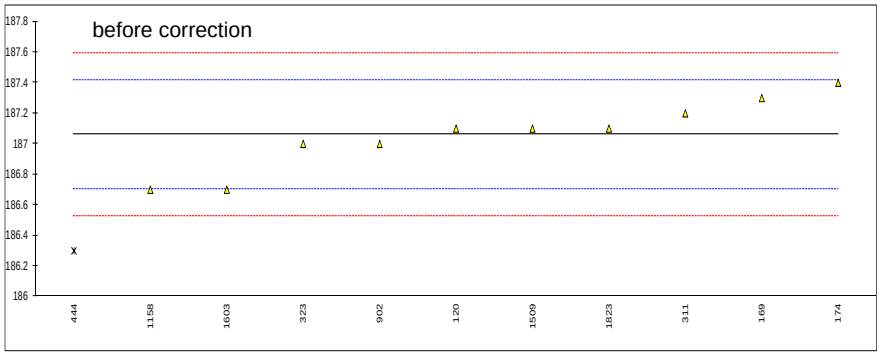
normality	not OK
n	14
outliers	0
mean (n)	1.9
st.dev. (n)	0.81
R(calc.)	2.3
R(E202:12)	7.0

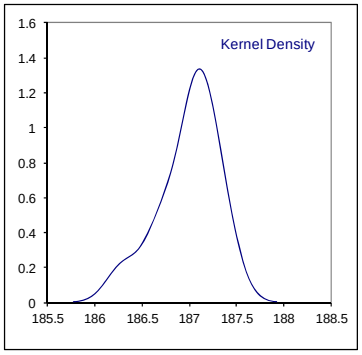


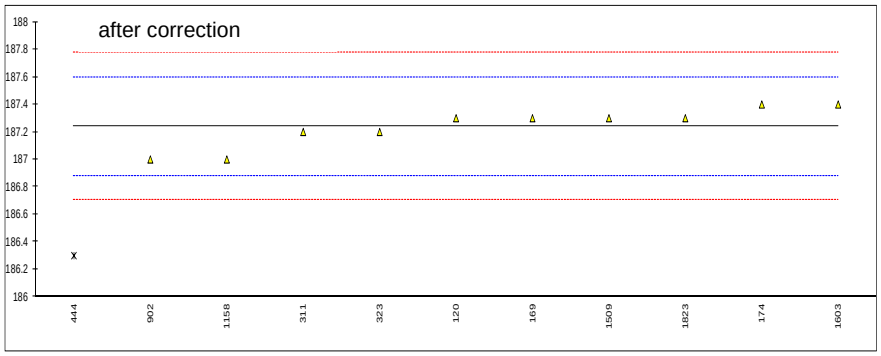


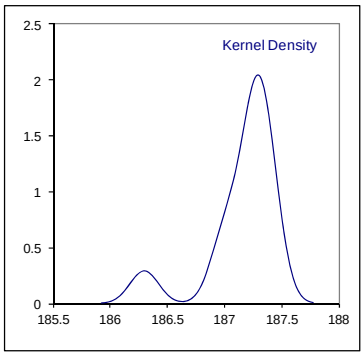
Determination of Initial Boiling Point @ 760 mmHg on sample #13196; results in °C.

lab	method	value	mark	z(targ)	value after corr.	mark	z(targ)	remarks
120	D1078	187.1		0.22	187.3	C	0.34	
169	D1078	187.3		1.34	187.3		0.34	
171		-----		-----	-----		-----	
173		-----		-----	-----		-----	
174	D1078	187.4		1.90	187.4		0.90	
311	D1078	187.2		0.78	187.2		-0.22	
315		-----		-----	-----		-----	
323	D1078	187.0		-0.34	187.2	C	-0.22	
343		-----		-----	-----		-----	
347		-----		-----	-----		-----	
444	E202	186.3	G(0.05)	-4.26	186.3	G(0.01)	-5.26	
446		-----		-----	-----		-----	
823		-----		-----	-----		-----	
902	D1078	187.0		-0.34	187.0		-1.34	
1016		-----		-----	-----		-----	
1158	E202	186.7		-2.02	187.0	C	-1.34	
1509	D1078	187.1		0.22	187.3	C	0.34	
1603	in house	186.7		-2.02	187.4	C	0.90	
1823	D1078	187.1		0.22	187.3	C	0.34	
	normality	OK			not OK			
	n	10			10			
	outliers	1			1			
	mean (n)	187.06			187.24			
	st.dev. (n)	0.227			0.143			
	R(calc.)	0.64			0.40			
	R(E202:05)	0.50			0.50			Compare R(D1078:11) = 2.92





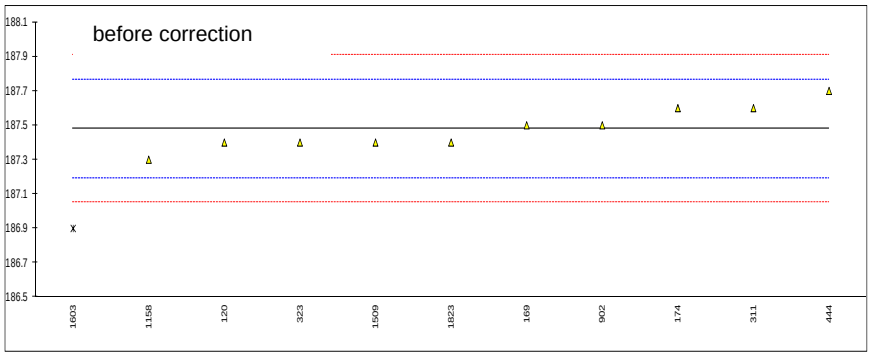


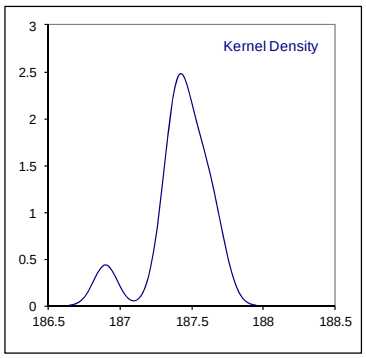


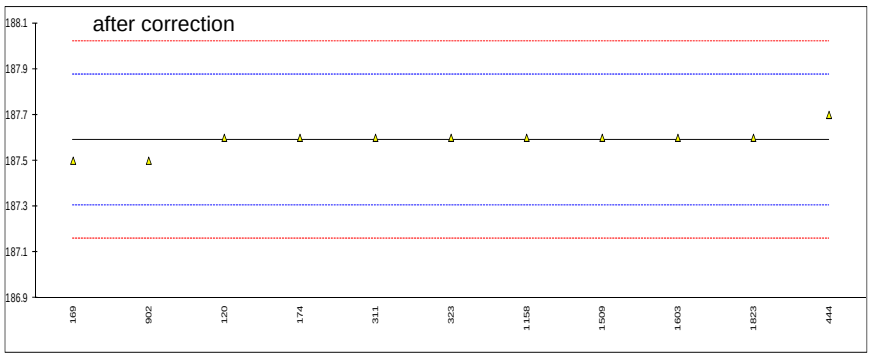
Determination of 50% recovered @ 760 mmHg on sample #13196; results in °C

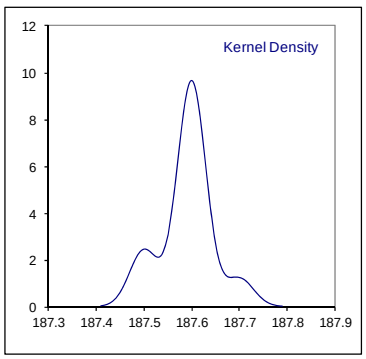
lab	method	value	mark	z(targ)	value after corr.	mark	z(targ)	remarks
120	D1078	187.4		-0.56	187.6	C	0.06	
169	D1078	187.5		0.14	187.5		-0.64	
171		-----		-----	-----		-----	
173		-----		-----	-----		-----	
174	D1078	187.6		0.84	187.6		0.06	
311	D1078	187.6		0.84	187.6		0.06	
315		-----		-----	-----		-----	
323	D1078	187.4		-0.56	187.6	C	0.06	
343		-----		-----	-----		-----	
347		-----		-----	-----		-----	
444	E202	187.7		1.54	187.7		0.76	
446		-----		-----	-----		-----	
823		-----		-----	-----		-----	
902	D1078	187.5		0.14	187.5		-0.64	
1016		-----		-----	-----		-----	
1158	E202	187.3		-1.26	187.6	C	0.06	
1509	D1078	187.4		-0.56	187.6	C	0.06	
1603	in house	186.9	G(0.05)	-4.06	187.6	C	0.06	
1823	D1078	187.4		-0.56	187.6	C	0.06	
	normality	OK			not OK			
	n	10			11			
	outliers	1			0			
	mean (n)	187.48			187.59*			
	st.dev. (n)	0.123			0.054			
	R(calc.)	0.34			0.15			
	R(E202:05)	0.40			0.40			Compare R(D1078:11) = 1.28

* theoretical mid boiling point = 187.6°C



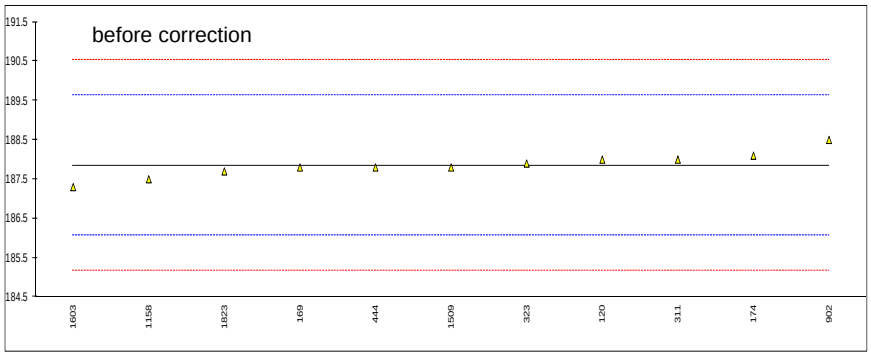


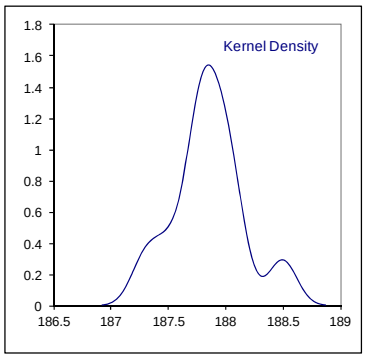


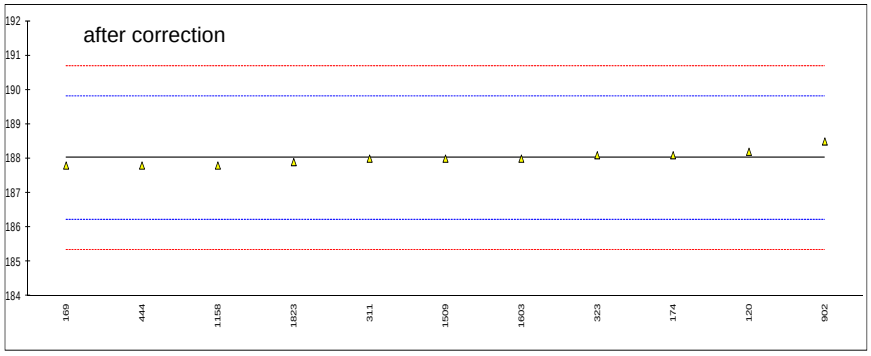


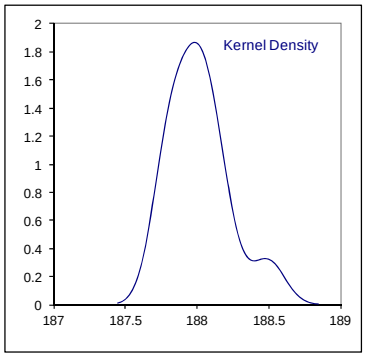
Determination of Dry Point @ 760 mmHg on sample #13196; results in °C

lab	method	value	mark	z(targ)	value after corr.	mark	z(targ)	remarks
120	D1078	188.0		0.16	188.2	C	0.20	
169	D1078	187.8		-0.06	187.8		-0.24	
171		-----		-----	-----		-----	
173		-----		-----	-----		-----	
174	D1078	188.1		0.27	188.1		0.09	
311	D1078	188.0		0.16	188.0		-0.02	
315		-----		-----	-----		-----	
323	D1078	187.9		0.05	188.1	C	0.09	
343		-----		-----	-----		-----	
347		-----		-----	-----		-----	
444	E202	187.8		-0.06	187.8		-0.24	
446		-----		-----	-----		-----	
823		-----		-----	-----		-----	
902	D1078	188.5		0.72	188.5		0.54	
1016		-----		-----	-----		-----	
1158	E202	187.5		-0.40	187.8	C	-0.24	
1509	D1078	187.8		-0.06	188.0	C	-0.02	
1603	in house	187.3		-0.62	188.0	C	-0.02	
1823	D1078	187.7		-0.17	187.9	C	-0.13	
	normality	OK			OK			
	n	11			11			
	outliers	0			0			
	mean (n)	187.85			188.02			
	st.dev. (n)	0.314			0.209			
	R(calc.)	0.88			0.58			
	R(E202:05)	2.50			2.50			Compare R(D1078:11) = 2.01





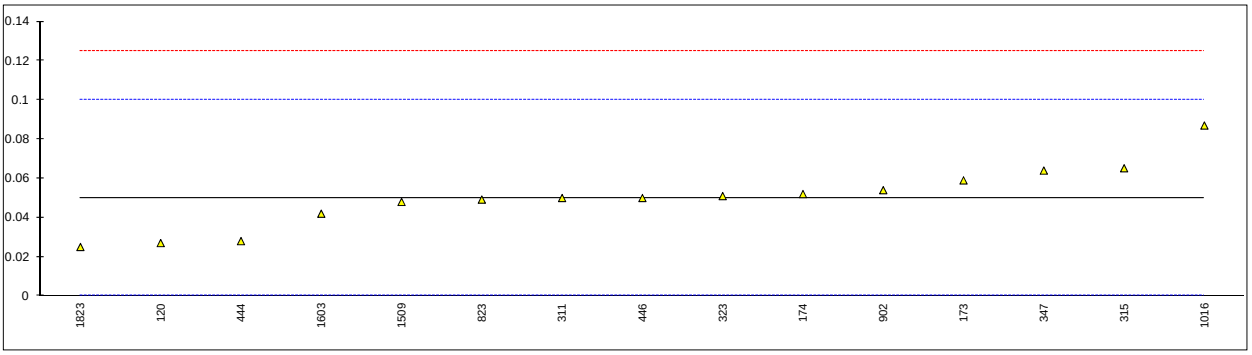


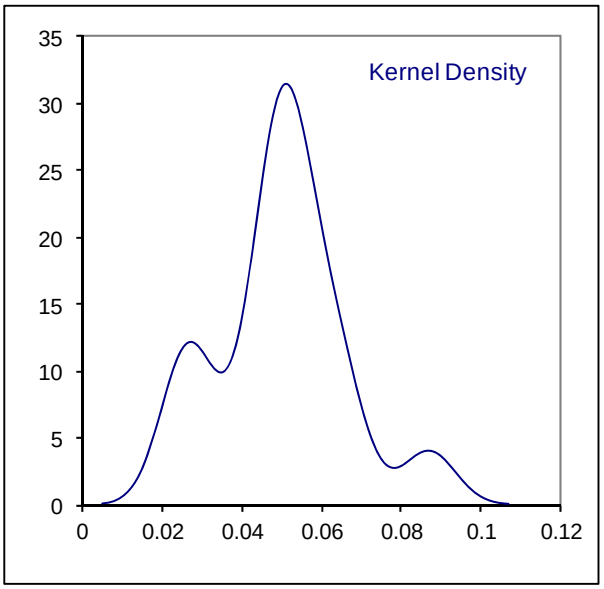


Determination of Iron as Fe on sample #13196; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
120	INH-0290	0.027		-0.92	
169		-----		-----	
171		-----		-----	
173	INH-0290	0.059		0.36	
174	E1615	0.052		0.08	
311	E1615	0.050		0.00	
315	E202	0.0652		0.60	
323	E1615	0.051		0.04	
343		-----		-----	
347	E394	0.064		0.56	
444	E202	0.028	C	-0.88	First reported as 0.0042
446	E202	0.050		0.00	
823	E202	0.0492		-0.04	
902	E1615	0.054		0.16	
1016	NEN6966	0.087		1.48	
1158		-----		-----	
1509	E394	0.048		-0.08	
1603	in house	0.0420		-0.32	
1823	E1615	0.025		-1.00	

normality	OK
n	15
outliers	0
mean (n)	0.050
st.dev. (n)	0.0160
R(calc.)	0.045
R(E202:05)	0.070

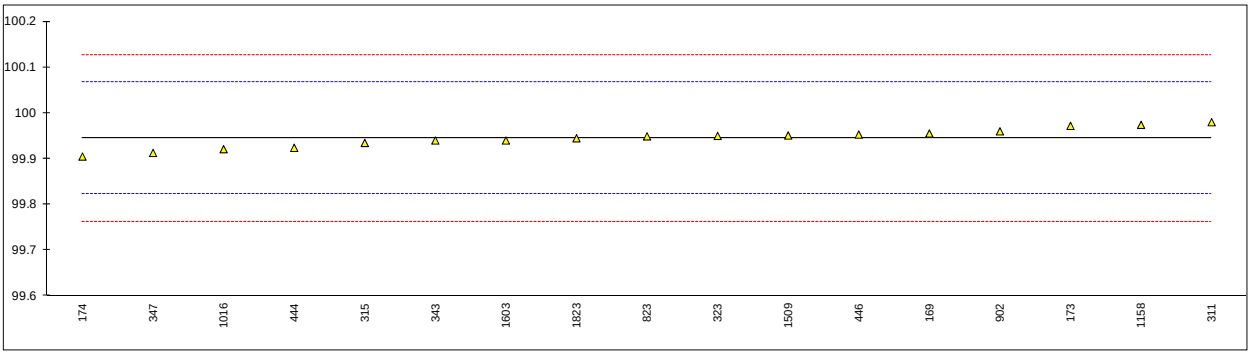


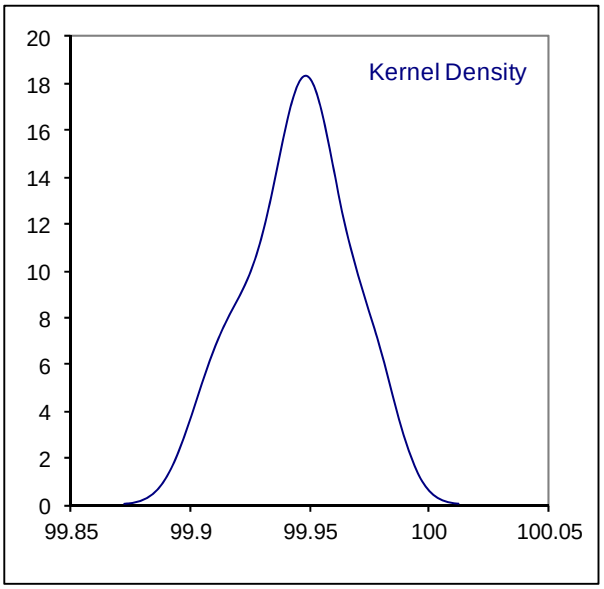


Determination of Purity on sample #13196; results in %M/M.

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
169	INH-0540	99.9553		0.17	
171		-----		-----	
173	INH-0540	99.971928		0.44	
174	E2409	99.905		-0.66	
311	INH-103	99.98		0.57	
315	INH-687	99.9346		-0.17	
323	in house	99.95		0.08	
343	INH-CM	99.940	C	-0.08	First reported as 99.82
347	E202	99.9128		-0.53	
444	E202	99.924		-0.35	
446	INH-130	99.953		0.13	
823	E202	99.9491		0.07	
902	INH-72	99.96		0.25	
1016	E202	99.921		-0.40	
1158	INH-003	99.9742		0.48	
1509	E202	99.951		0.10	
1603	in house	99.94		-0.08	
1823		99.9449		0.00	

normality	OK
n	17
outliers	0
mean (n)	99.945
st.dev. (n)	0.0211
R(calc.)	0.059
R(E202:12)	0.170

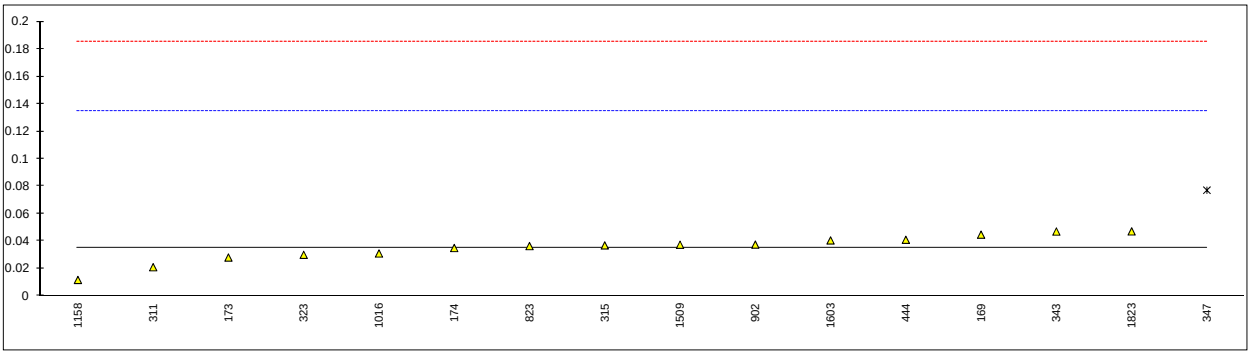


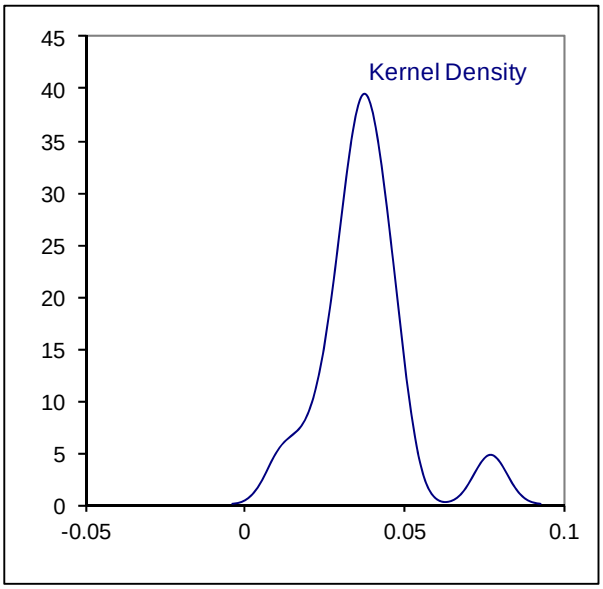


Determination of Dipropylene Glycol on sample #13196; results in %M/M.

lab	method	Value	mark	z(targ)	remarks
120		-----		-----	
169	INH-0540	0.0447		0.19	
171		-----		-----	
173	INH-0540	0.02807		-0.14	
174	E2409	0.035		0.00	
311	INH-103	0.021		-0.28	
315	INH-687	0.0369		0.04	
323	in house	0.03		-0.10	
343	INH-CM	0.047	C	0.24	First reported as 0.175
347	E202	0.0771	G(0.05)	0.84	
444	E202	0.041		0.12	
446		-----		-----	
823	E202	0.0364		0.03	
902	INH-72	0.0375		0.05	
1016	E202	0.031		-0.08	
1158	INH-003	0.0117		-0.47	
1509	E202	0.0374		0.05	
1603	in house	0.0405		0.11	
1823	INH-33	0.0471		0.24	

normality	OK
n	15
outliers	1
mean (n)	0.0350
st.dev. (n)	0.00962
R(calc.)	0.0269
R(E202:12)	0.1400

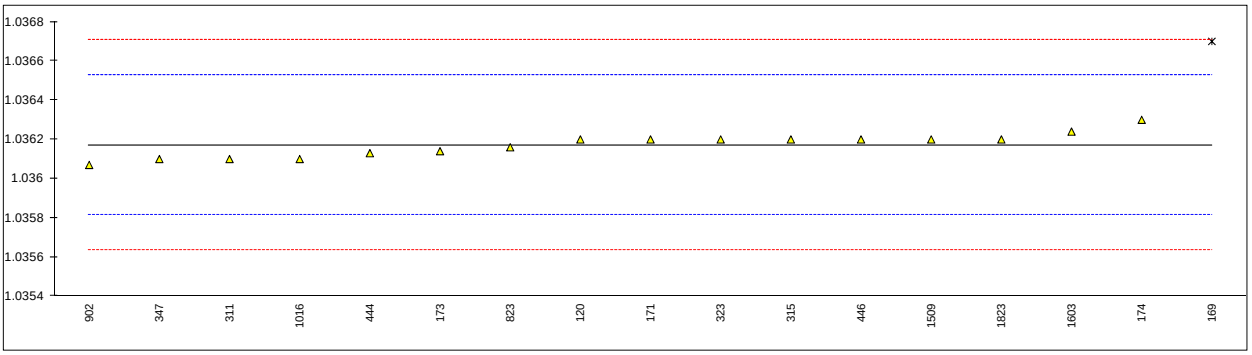


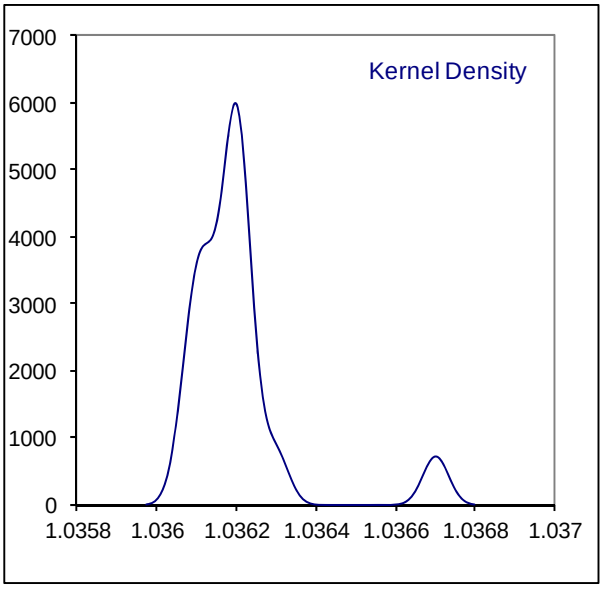


Determination of Density @ 20°C on sample #13196; results in kg/L.

lab	method	value	mark	z(targ)	remarks
120	D4052	1.0362		0.16	
169	D4052	1.0367	G(0.01)	2.96	
171	D4052	1.0362		0.16	
173	D4052	1.03614		-0.17	
174	D4052	1.0363		0.72	
311	D4052	1.0361		-0.40	
315	D4052	1.0362		0.16	
323	D4052	1.0362		0.16	
343		-----		-----	
347	D4052	1.0361		-0.40	
444	D4052	1.03613		-0.23	
446	D4052	1.0362		0.16	
823	D4052	1.03616		-0.06	
902	D4052	1.03607		-0.57	
1016	D4052	1.0361		-0.40	
1158		-----		-----	
1509	D4052	1.0362		0.16	
1603	in house	1.03624		0.39	
1823	D4052	1.0362		0.16	

normality	not OK
n	16
outliers	1
mean (n)	1.03617
st.dev. (n)	0.000061
R(calc.)	0.00017
R(D4052:02e1)	0.00050

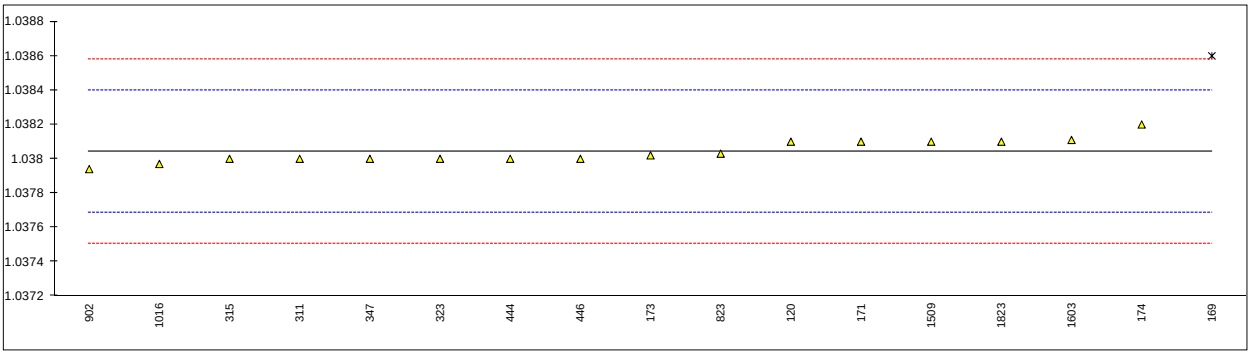


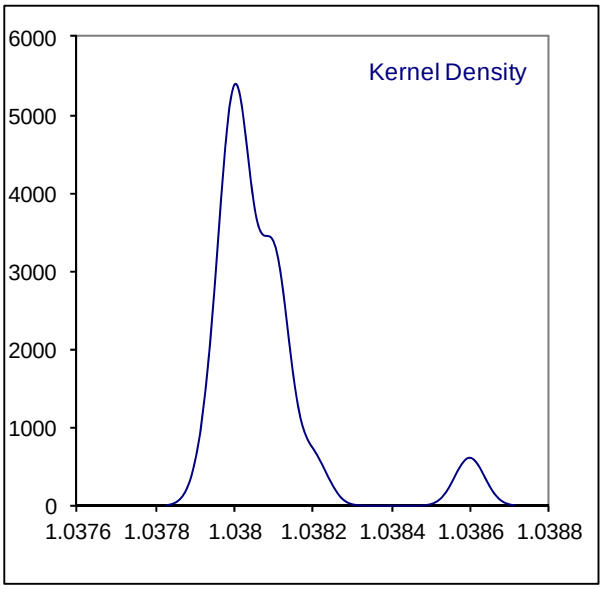


Determination of Specific Gravity 20/20°C/°C on sample #13196;

lab	method	value	mark	z(targ)	remarks
120	D4052	1.0381		0.33	
169	D4052	1.0386	G(0.01)	3.13	
171	D4052	1.0381		0.33	
173	D4052	1.03802		-0.12	
174	D4052	1.0382		0.89	
311	D4052	1.0380		-0.23	
315	D4052	1.0380		-0.23	
323	D4052	1.0380		-0.23	
343		-----		-----	
347	D4052	1.0380		-0.23	
444	E202	1.0380		-0.23	
446	D4052	1.0380		-0.23	
823	D4052	1.03803		-0.07	
902	D4052	1.03794		-0.57	
1016	D4052	1.03797		-0.40	
1158		-----		-----	
1509	D4052	1.0381		0.33	
1603	in house	1.03811		0.38	
1823	D4052	1.0381		0.33	

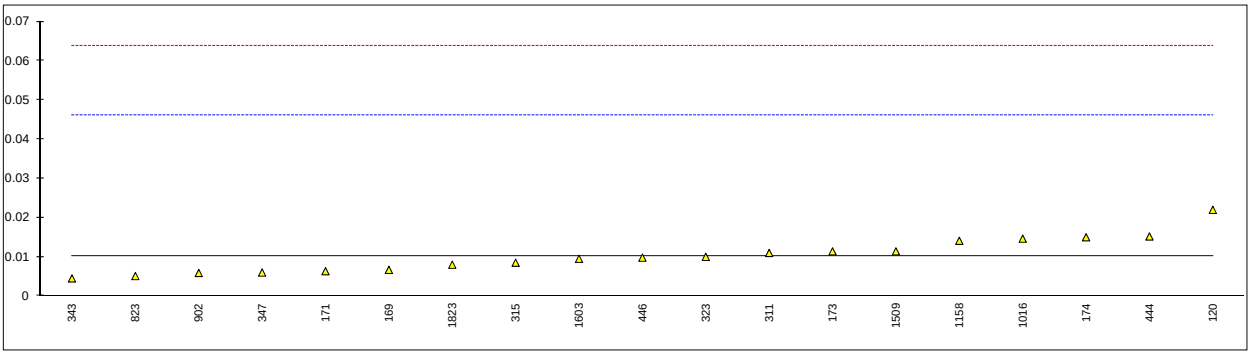
normality	not OK
n	16
outliers	1
mean (n)	1.03804
st.dev. (n)	0.000068
R(calc.)	0.00019
R(E202:12)	0.00050

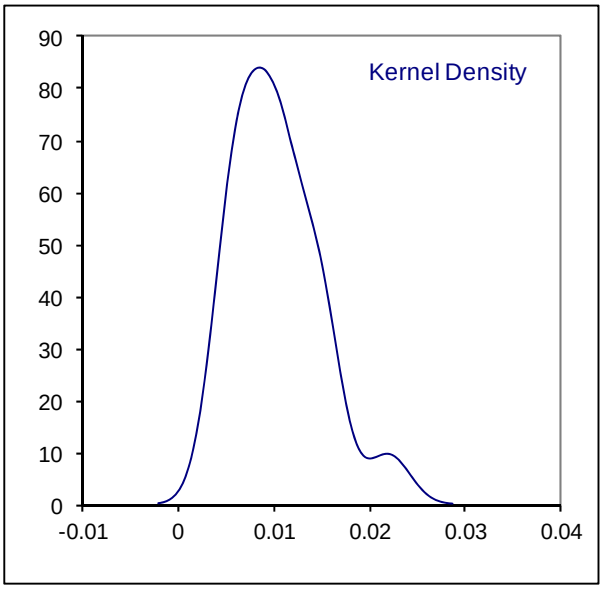




Determination of Water on sample #13196; results in %M/M.

lab	method	value	mark	z(targ)	remarks
120	E1064	0.0220		0.66	
169	E1064	0.0067		-0.20	
171	E203	0.00635		-0.22	
173	E202	0.0114		0.06	
174	E203	0.015	C	0.26	First reported as 0.0486
311	D1364	0.011		0.04	
315	E203	0.0085		-0.10	
323	E1064	0.010		-0.02	
343	E1064	0.0045		-0.32	
347	E1064	0.0060		-0.24	
444	E202	0.0152		0.28	
446	E203	0.0098		-0.03	
823	E1064	0.0051		-0.29	
902	E1064	0.0059		-0.24	
1016	E202	0.01465		0.25	
1158	E203	0.0141		0.21	
1509	E203	0.0114		0.06	
1603	in house	0.0095		-0.04	
1823	INH-33	0.008		-0.13	
	normality	OK			
	n	19			
	outliers	0			
	mean (n)	0.0103			
	st.dev. (n)	0.00444			
	R(calc.)	0.0124			
	R(E202:05)	0.0500			Compare R(E1064) = 0.0016%M/M





APPENDIX 2

Number of participants per country

1 lab in BELGIUM
1 lab in CHINA, P.R.
1 lab in GERMANY
3 labs in NETHERLANDS
1 lab in ROMANIA
1 lab in SINGAPORE
1 lab in SOUTH KOREA
2 labs in SPAIN
1 lab in TURKEY
2 labs in UNITED KINGDOM
5 labs in UNITED STATES OF AMERICA

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, January 2010

2 ASTM E178-89

3 ASTM E1301-89

4 ISO 13528-05

4 ISO 5725-86

5 ISO 5725, parts 1-6, 1994

6 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)

7 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)

8 IP 367/84

9 DIN 38402 T41/42

10 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)

11 J.N. Miller, Analyst, 118, 455, (1993)

12 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/).