

Results of Proficiency Test
Monopropylene glycol
October 2017

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

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1 INTRODUCTION

Since 1995, the Institute for Interlaboratory Studies (iis) organized once every two years a proficiency test for Monopropylene glycol (MPG). During the annual proficiency testing program 2017/2018, it was decided to continue the round robin for the analysis of MPG. In this interlaboratory study, 21 laboratories from 14 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2017 MPG proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send 1 sample (1 * 0.5 L glass bottle, labelled #17208). Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation 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 March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

One can with approximately 25 litre of MPG was obtained from a local trader. After homogenisation 48 amber glass bottles of 0.5 litre were filled and labelled #17208. The homogeneity of the subsamples #17208 was checked by determination of the Density in accordance with ASTM D4052 and by determination of Water in accordance ASTM E1064 on 8 stratified randomly selected samples.

	<i>Density at 20°C in kg/L</i>	<i>Water in mg/kg</i>
sample #17208-1	1.03641	2150
sample #17208-2	1.03642	2160
sample #17208-3	1.03642	2150
sample #17208-4	1.03642	2170
sample #17208-5	1.03640	2150
sample #17208-6	1.03641	2160
sample #17208-7	1.03641	2160
sample #17208-8	1.03641	2150

Table 1: homogeneity test results of subsamples #17208.

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

	<i>Density at 20°C in kg/L</i>	<i>Water in mg/kg</i>
r (observed)	0.00002	21
reference test method	ISO12185:96	ASTM E202:05
0.3 x R (ref. test method)	0.00015	150

Table 2: evaluation of the repeatabilities of subsamples #17208.

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

To each of the participating laboratories 1 bottle was sent (one bottle of 0.5 L, labelled #17208), on October 4, 2017. An SDS was added to the sample package

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were asked to determine on sample #17208: Acidity, Appearance, Inorganic Chloride, Color, Density, Distillation, Iron, Purity, Dipropylene Glycol, Specific Gravity at 20/20°C and Water.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

Shortly after the deadline, the available test results were screened for suspect data. A test 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 reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

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 March 2017 (iis-protocol, version 3.4).

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are 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 reference test method. 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. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised

to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

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 some problems were encountered with the dispatch of the sample to the laboratory in P.R. of China. All participants reported the test results in time. Not all participants were able to report all requested parameters. Finally, 21 laboratories did report 177 numerical test results. Observed were 2 outlying test results, which is 1.1%. In proficiency studies outlier percentages of 3 - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

In this section, the results are discussed per test.

The test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

The latest standardized method available for analysis of Ethylene Glycols and Propylene Glycols is ASTM E202:2012. This version was not used for a number of tests because 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.

Acidity: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the

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

Appearance: A standardized method is available for Appearance since 2009, being ASTM E2680:09(2015). However, not all participants did report according this method. All participants agreed about the appearance of sample #17208 to be 'clear and bright', 'clear and free of suspended matter' or 'pass'. According ASTM E2680, the appearance should be reported as 'pass' (or 'fail').

Chloride, Inorganic: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM E2469:16. Chloride was not (artificially) added to this sample.

Colour Pt/Co: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM E202:12 and ASTM E5386:16.

Density: This determination was not problematic. No statistical outliers were observed, but one test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ISO12185:96.

Distillation: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibilities of the Initial Boiling Point and 50% recovered are not in agreement with the requirements of ASTM E202:05. The calculated reproducibility of the Dry Point is in agreement with the requirements of ASTM E202:05.

Iron: This determination was problematic. The reported test results appear to be bimodally distributed. Therefore, no significant conclusions were drawn. Iron was not (artificial) added to this sample

Purity: This determination was problematic. The reported test results appear to be trimodally distributed. Therefore, no significant conclusions were drawn.

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

Specific Gravity 20/20°C: This determination was not problematic. No statistical outliers were observed, but one test result was excluded. However, the calculated reproducibility, after rejection of the suspect data is in full agreement with the requirements of E202:12.

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

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acetic Acid	%M/M	16	0.0005	0.0005	0.0008
Appearance		18	Pass	n.a.	n.a.
Chloride, inorganic as Cl	mg/kg	9	0.75	0.30	0.09
Colour Pt/Co		19	4.9	2.9	7
Density at 20°C	kg/L	18	1.0365	0.0005	0.0005
Initial Boiling Point	°C	10	184.8	1.3	0.5
50% recovered	°C	10	187.3	0.7	0.4
Dry Point	°C	10	188.2	1.2	2.5
Iron	mg/kg	10	0.32	0.93	(0.07)
Purity	%M/M	17	(99.636)	(0.597)	(0.17)
Dipropylene Glycol	%M/M	15	0.053	0.069	0.14
Specific Gravity 20/20°C		18	1.0384	0.0005	0.0005
Water	mg/kg	21	2224	201	500

Table 3: reproducibilities of sample #17208

Results between brackets were not subject for evaluation as the group was divided bimodal (or trimodal), therefore evaluation should be done with due care

Without further statistical calculations it can be concluded that for only some components there is a good compliance of the group of participating laboratories with the relevant reference test methods. The components that are problematic have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE OCTOBER 2017 PROFICIENCY TEST WITH PREVIOUS PTS

	October 2017	October 2015	October 2013	October 2011
Number of reporting labs	21	23	19	18
Number of results reported	177	207	189	185
Statistical outliers	2	4	5	6
Percentage outliers	1.1%	1.9%	2.7%	3.2%

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 compared against the requirements of the respective reference test methods. The conclusions are given the following table:

	October 2017	October 2015	October 2013	October 2011
Acidity as Acetic Acid	+	+	+	++
Chloride as Cl	--	+	n.e.	++
Colour Pt/Co	++	++	++	++
Density at 20°C	+/-	+	++	++
Initial Boiling Point	--	--	-	-
50% recovered	--	--	+	--
Dry Point	++	++	++	++
Iron	(--)	+	++	++
Purity	(--)	++	++	++
Dipropylene Glycol	++	++	++	++
Specific Gravity 20/20°C/°C	+/-	+	++	++
Water	++	++	++	++

Table 5: comparison determinations against the standard

Results between brackets were not subject for evaluation as the group was divided bimodal (or trimodal), therefore evaluation should be done with due care

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

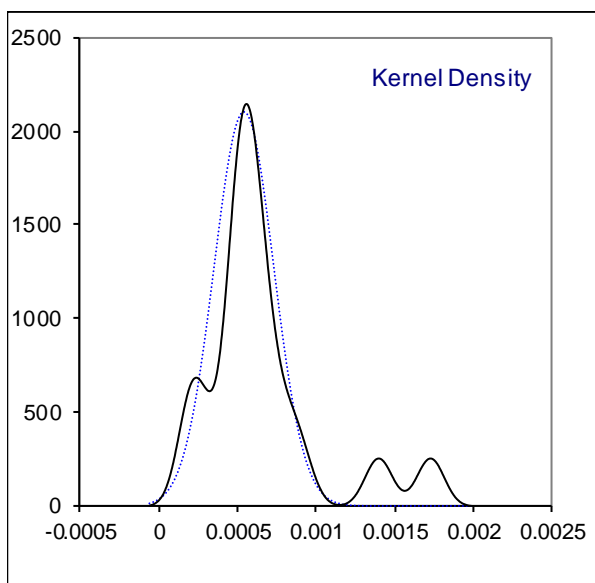
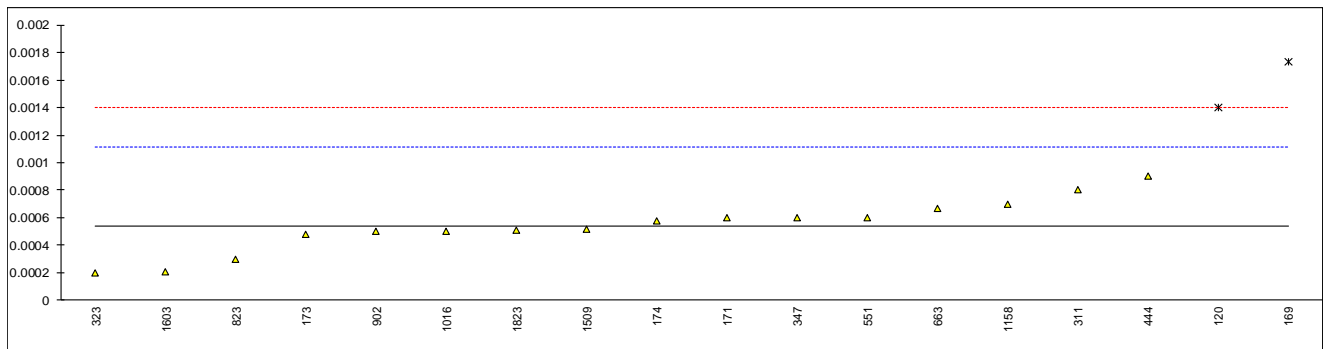
- ++: group performed much better than the reference test methods
- + : group performed better than the reference test methods
- +/-: group performance equals the reference test methods
- : group performed worse than the reference test methods
- : group performed much worse than the reference test methods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

APPENDIX 1

Determination of Acidity as Acetic Acid on sample #17208; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D1613	0.0014	C,G(0.01)	3.00	First reported 0.0025
169	D1613	0.00173	G(0.05)	4.16	
171	D1613	0.0006		0.20	
173	E202	0.00048		-0.22	
174	D1613	0.00058		0.13	
311	D1613	0.0008		0.90	
323	E202	0.0002		-1.20	
334		----		----	
347	D1613	0.0006		0.20	
444	D1613	0.0009		1.25	
446		----		----	
551	D1613	0.0006		0.20	
663	D1613	0.00067		0.45	
823	D1613	0.0003		-0.85	
902	D1613	0.0005		-0.15	
1016	D1613	0.0005		-0.15	
1158	D1613	0.0007		0.55	
1509	D1613	0.00052		-0.08	
1603	In house	0.00021		-1.16	
1823	INH-2015	0.00051		-0.11	
2458		----		----	

normality OK
n 16
outliers 2
mean (n) 0.00054
st.dev. (n) 0.000190
R(calc.) 0.00053
st.dev.(E202:05) 0.000286
R(E202:05) 0.0008

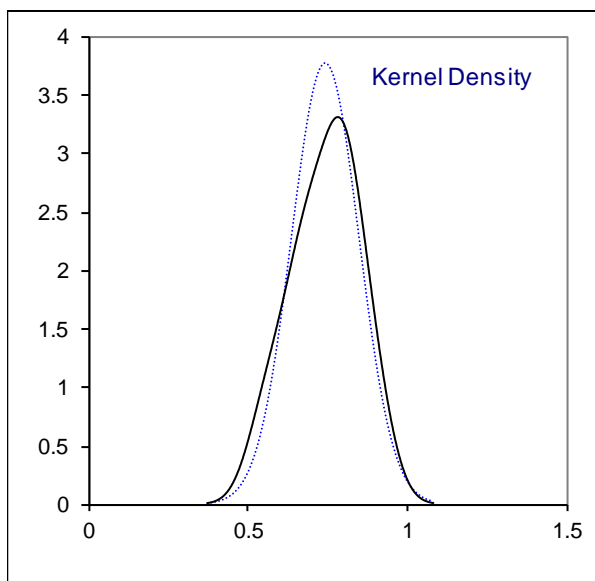
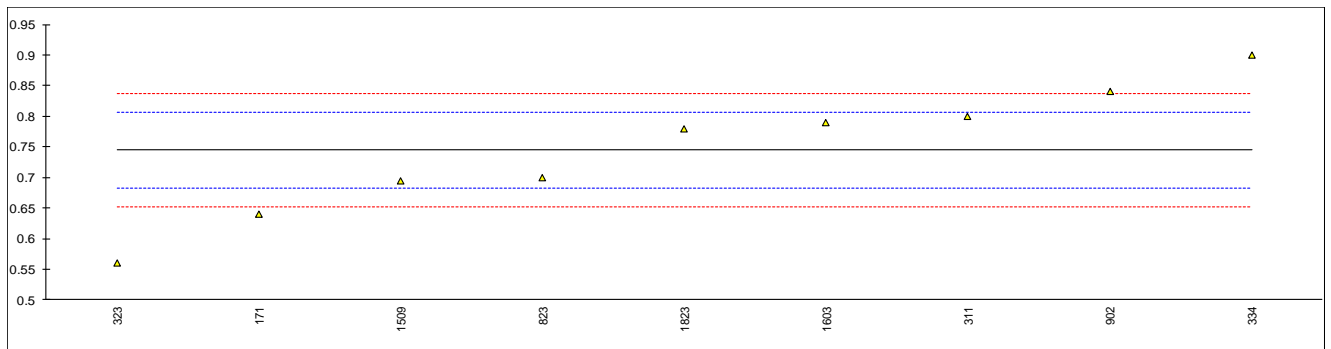


Determination of Appearance on sample #17208;

lab	method	value	mark	z(targ)	remarks
120	D4176	Pass, Clear and Br		-----	
169	Visual	Pass		-----	
171	E2680	passes		-----	
173	E2680	pass		-----	
174	Visual	clear & free		-----	
311	E2680	pass		-----	
323	Visual	C&B		-----	
334		-----		-----	
347	E2680	PASS		-----	
444	E2680	Pass		-----	
446	E2680	Pass		-----	
551	E2680	Pass		-----	
663	Visual	Bright & Clear		-----	
823	E2680	Pass		-----	
902	E2680	Pass		-----	
1016	In house	Pass		-----	
1158		-----		-----	
1509	E2680	Pass		-----	
1603	In house	PASS		-----	
1823	D4176	pass		-----	
2458		-----		-----	
	normality	unknown			
	n	18			
	outliers	n.a.			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	st.dev.(.)	n.a.			
	R()	n.a.			

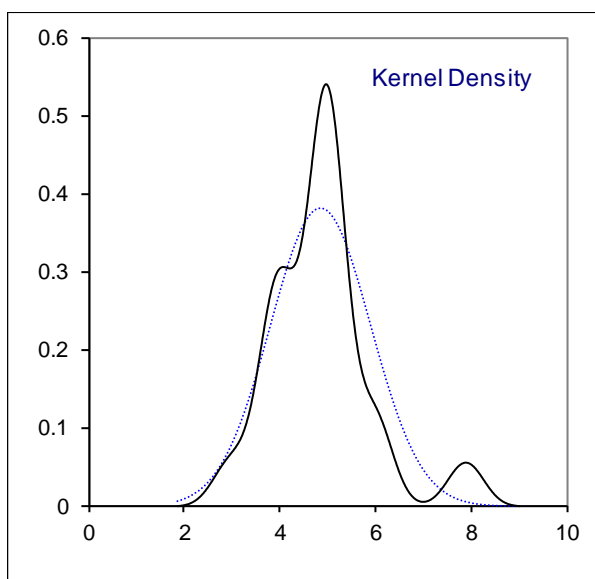
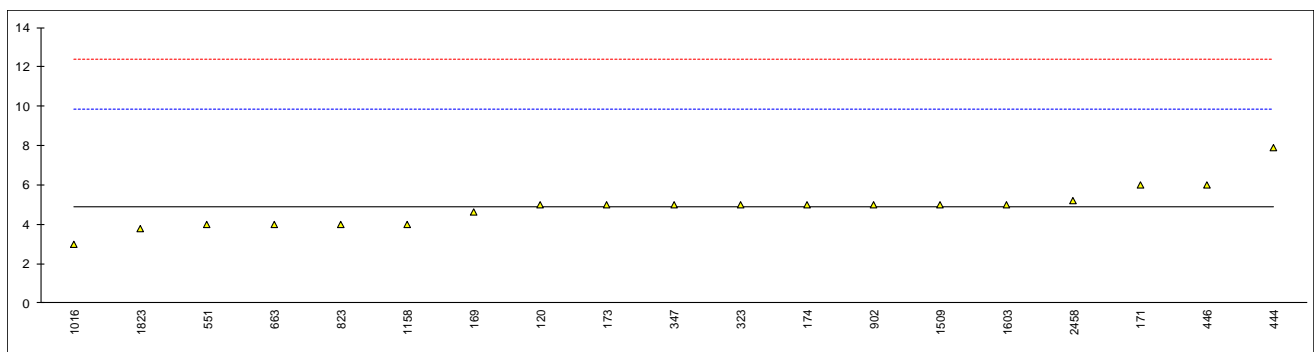
Determination of Chloride, Inorganic as Cl on sample #17208; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		----		----	
169		----		----	
171	E2469	0.64		-3.40	
173	INH-221	>0.5		----	
174	INH-221	>0.5		----	
311	INH-158	0.8		1.78	
323	E2469	0.56		-6.00	
334		0.9		5.02	
347		----		----	
444		----		----	
446	INH-CM3221	>10		>300.00	False positive test result?
551		----		----	
663		----		----	
823	E2469	0.70		-1.46	
902	E2469	0.84		3.08	
1016		----		----	
1158		----		----	
1509	INH-CM	0.695		-1.62	
1603	In house	0.79		1.46	
1823	INH-1677	0.78		1.13	
2458		----		----	
normality		OK			
n		9			
outliers		0			
mean (n)		0.745			
st.dev. (n)		0.1055			
R(calc.)		0.295			
st.dev.(E2469:16)		0.0309			
R(E2469:16)		0.086			



Determination of Colour Pt/Co on sample #17208;

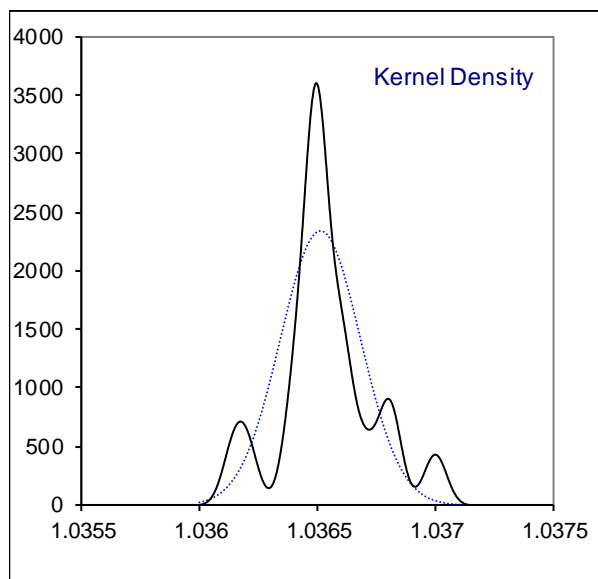
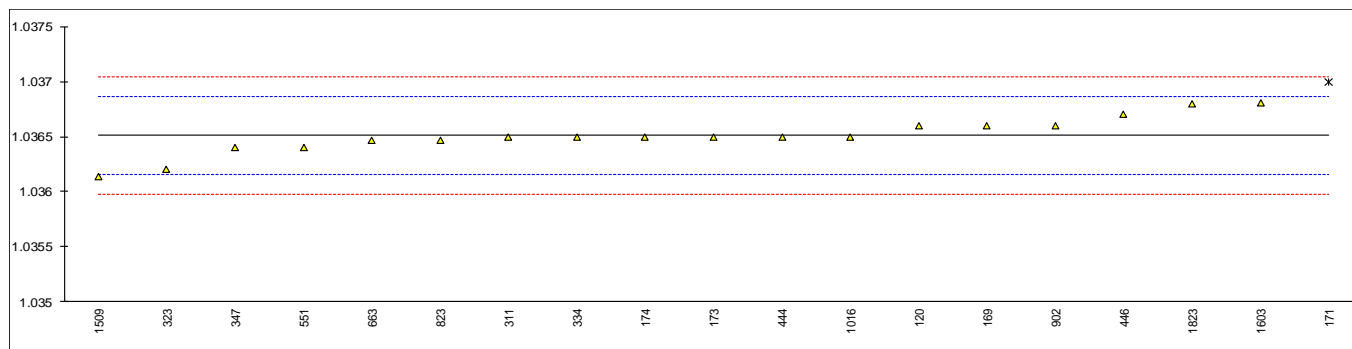
lab	method	value	mark	z(targ)	remarks
120	D1209	5		0.05	
169	D5386	4.6		-0.11	
171	D5386	6		0.45	
173	D1209	5		0.05	
174	D1209	5		0.05	
311	E202	<5		-----	
323	E202	5		0.05	
334		-----		-----	
347	D5386	5		0.05	
444	D5386	7.9		1.21	
446	D5386	6		0.45	
551	D5386	4		-0.35	
663	D1209	4		-0.35	
823	D5386	4		-0.35	
902	D5386	5		0.05	
1016	D1209	3		-0.75	
1158	D1209	4		-0.35	
1509	E202	5		0.05	
1603	In house	5		0.05	
1823	D5386	3.8		-0.43	
2458	ISO6271	5.2		0.13	
normality		not OK			
n		19			
outliers		0			
mean (n)		4.87			
st.dev. (n)		1.047			
R(calc.)		2.93			
st.dev.(E202:12)		2.5			
R(E202:12)		7			
Compare					
R(E5386:16)		5.4			



Determination of Density at 20°C on sample #17208; results in kg/L

lab	method	value	mark	z(targ)	remarks
120	D4052	1.0366	C	0.50	First reported 1036.6 kg/L
169	D4052	1.0366		0.50	
171	D4052	1.037	ex	2.74	Test result excluded for being rounded too far
173	D4052	1.0365		-0.06	
174	D4052	1.0365		-0.06	
311	D4052	1.0365		-0.06	
323	ISO12185	1.0362		-1.74	
334	ISO12185	1.0365		-0.06	
347	D4052	1.0364		-0.62	
444	D4052	1.0365		-0.06	
446	D4052	1.0367		1.06	
551	D4052	1.0364		-0.62	
663	D4052	1.03647		-0.23	
823	D4052	1.03647		-0.23	
902	D4052	1.0366		0.50	
1016	D4052	1.0365		-0.06	
1158		----		----	
1509	D4052	1.03614		-2.08	
1603	In house	1.03681		1.68	
1823	D4052	1.0368		1.62	
2458		----		----	

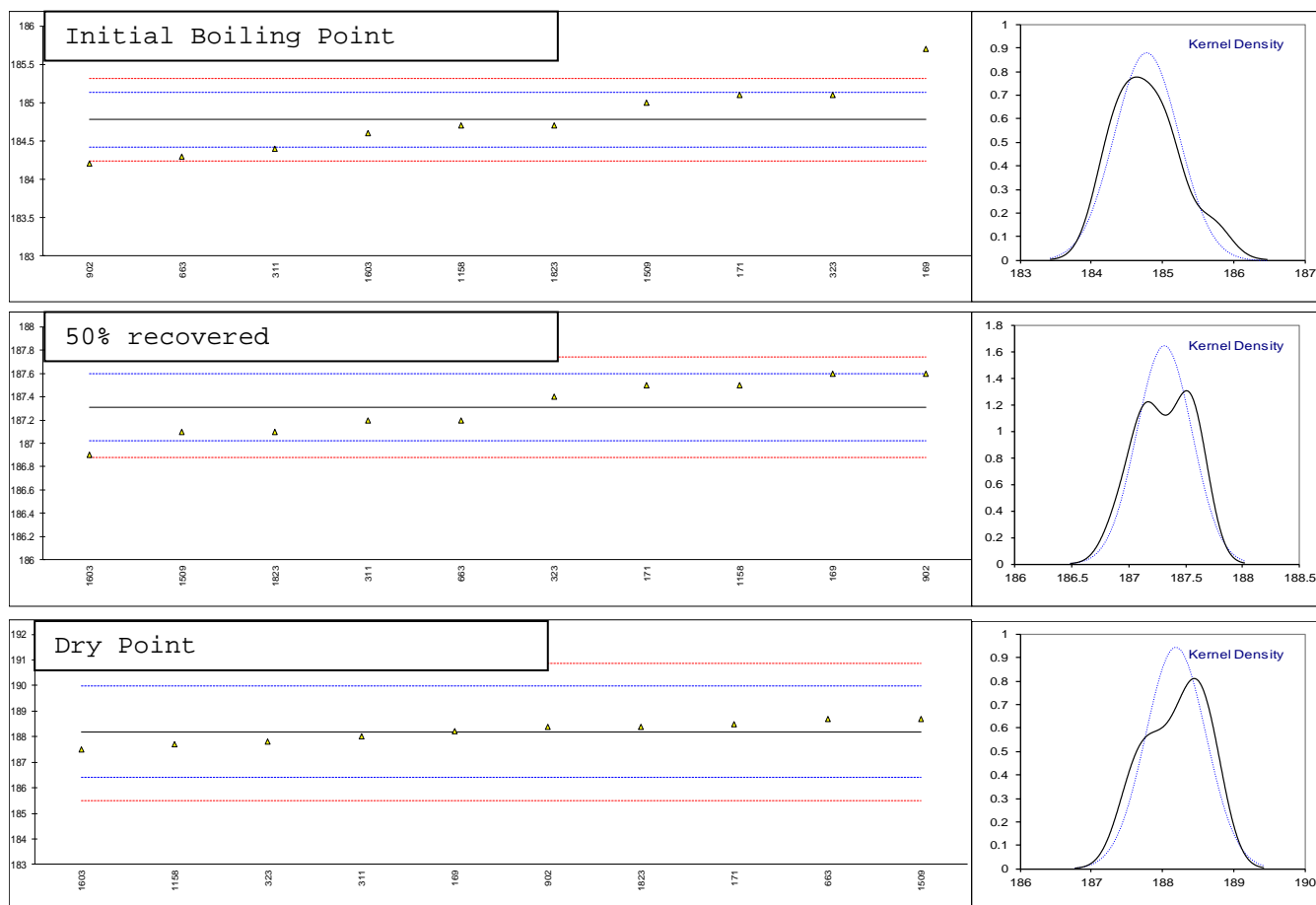
normality OK
n 18
outliers 0 (+1 excl)
mean (n) 1.03651
st.dev. (n) 0.000171
R(calc.) 0.00048
st.dev.(ISO12185:96) 0.000179
R(ISO12185:96) 0.0005



Determination of Distillation: IBP, 50% recovered, Dry Point on sample #17208; results in °C

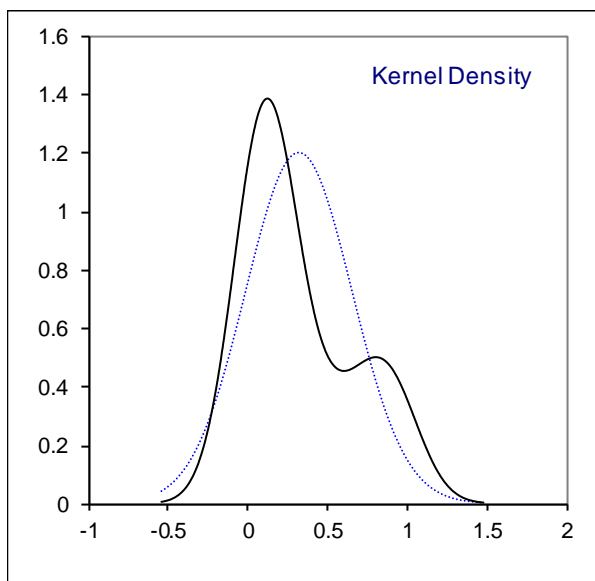
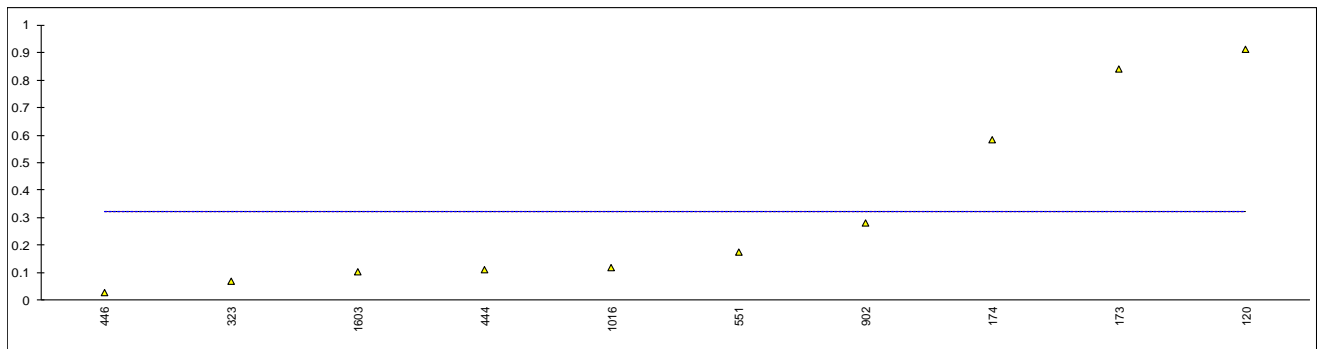
lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)	remarks
120		----		----	----		----	----		----	
169	D1078	185.7		5.15	187.6		2.03	188.2		0.01	
171	D1078	185.1		1.79	187.5		1.33	188.5		0.35	
173		----		----	----		----	----		----	
174		----		----	----		----	----		----	
311	E202	184.4		-2.13	187.2		-0.77	188.0		-0.21	
323	E202	185.1		1.79	187.4		0.63	187.8		-0.44	
334		----		----	----		----	----		----	
347		----		----	----		----	----		----	
444		----		----	----		----	----		----	
446		----		----	----		----	----		----	
551		----		----	----		----	----		----	
663	D1078	184.3		-2.69	187.2		-0.77	188.7		0.57	
823		----		----	----		----	----		----	
902	D1078	184.2		-3.25	187.6		2.03	188.4		0.24	
1016		----		----	----		----	----		----	
1158	D1078	184.7		-0.45	187.5		1.33	187.7		-0.55	
1509	D1078	185.0		1.23	187.1	C	-1.47	188.7		0.57	
1603	In house	184.6		-1.01	186.9		-2.87	187.5		-0.77	
1823	D1078	184.7		-0.45	187.1		-1.47	188.4		0.24	
2458		----		----	----		----	----		----	
normality		OK			OK			OK			
n		10			10			10			
outliers		0			0			0			
mean (n)		184.78			187.31			188.19			
st.dev. (n)		0.454			0.242			0.423			
R(calc.)		1.27			0.68			1.18			
st.dev.(E202:05)		0.179			0.143			0.893			
R(E202:05)		0.5			0.4			2.5			

Lab 1509: first reported 188.1



Determination of Iron as Fe on sample #17208; results in mg/kg

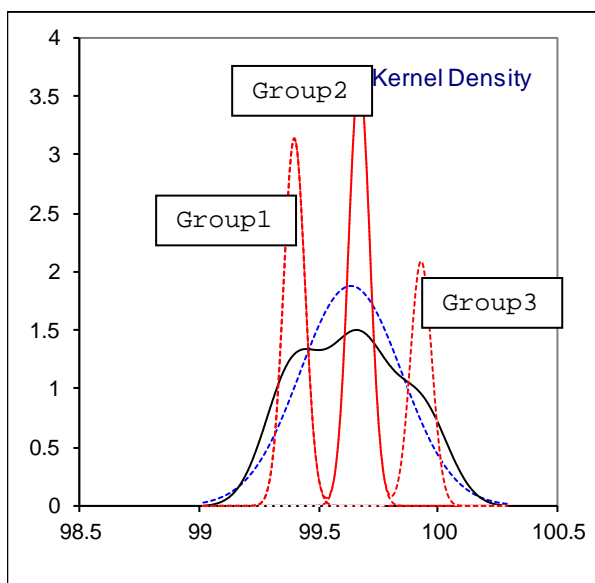
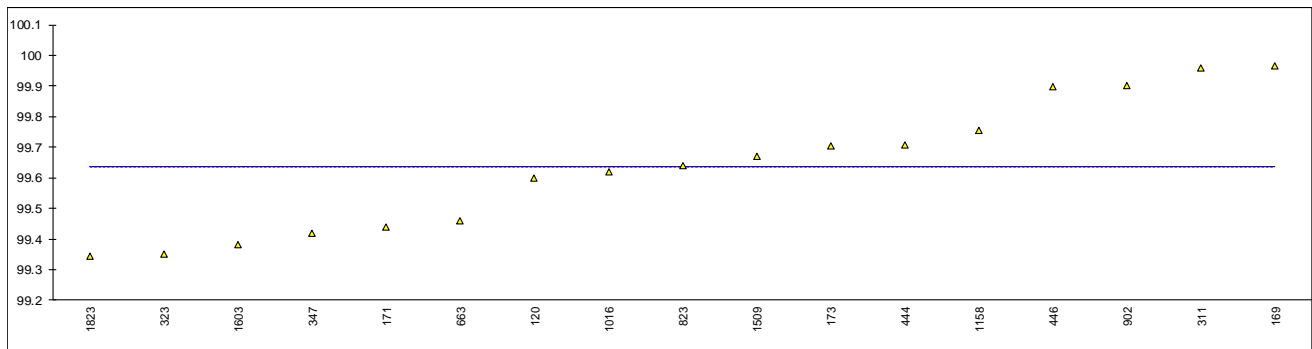
lab	method	value	mark	z(targ)	remarks
120	E394	0.911		----	
169		----		----	
171		>0.200		----	
173	E394	0.84		----	
174	E1615	0.584		----	
311		----		----	Sample gets hazy after adding water, determination impossible
323	E202	0.068		----	
334		----		----	
347		----		----	
444	E202	0.111		----	
446	E202	0.028		----	
551	E394	0.175		----	
663		----		----	
823		----		----	
902	E394	0.28		----	
1016	NEN6966	0.1185		----	
1158		----		----	
1509		----	W	----	Result withdrawn, reported 0.785
1603	In house	0.105		----	
1823		----		----	
2458		----		----	
normality		suspect			
n		10			
outliers		0			
mean (n)		0.322			
st.dev. (n)		0.3319			
R(calc.)		0.929			
st.dev.(E202:05)		(0.025)			
R(E202:05)		(0.07)			



Determination of Purity on sample #17208; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	E202	99.60		----	
169	INH-CM	99.966		----	
171	E2409	99.44		----	
173	INH-540	99.704		----	
174				----	
311	INH-103	99.96		----	
323	E202	99.35		----	
334				----	
347	E2409	99.42		----	
444	E2409	99.708		----	
446	INH-CM130	99.897		----	
551				----	
663	E2409	99.461		----	
823	E2409	99.6400		----	
902	INH-72	99.90		----	
1016	E202	99.62		----	
1158	INH-003	99.756		----	
1509	E202	99.6709		----	
1603	In house	99.38		----	
1823	INH-2015	99.3447		----	
2458				----	

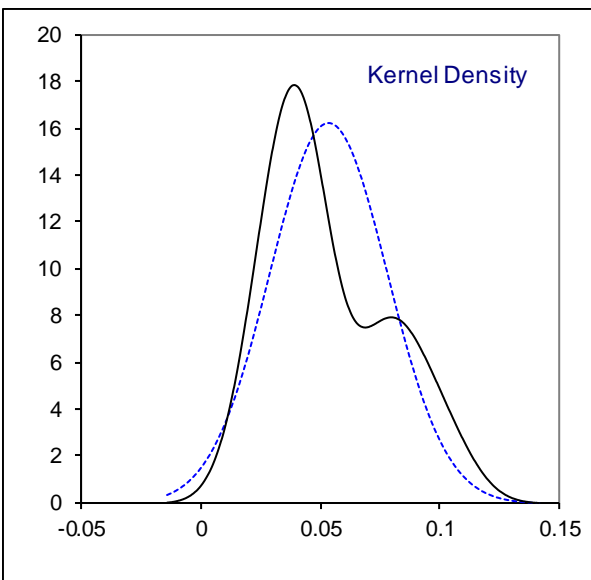
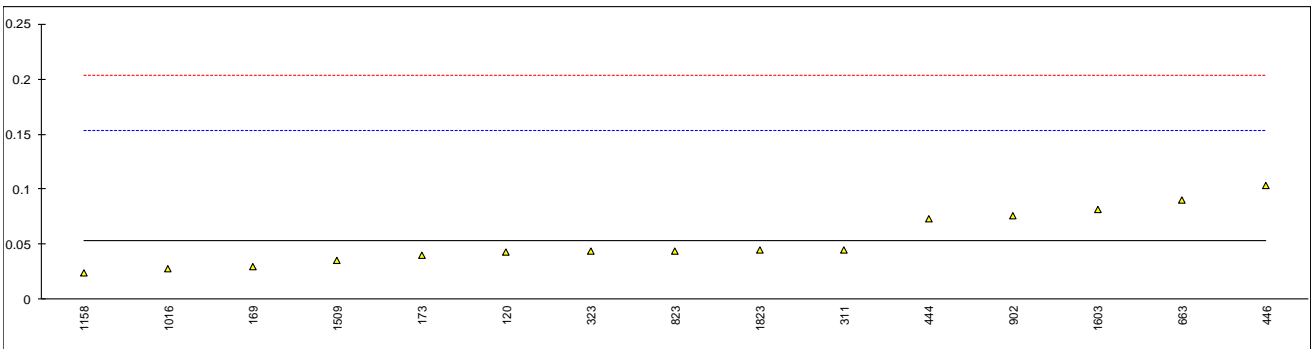
	Group 1	Group 2	Group 3
normality	OK	OK	not OK
n	6	7	4
outliers	0	0	0
mean (n)	99.3993	99.6713	99.9307
st.dev. (n)	0.04832	0.05520	0.03734
R(calc.)	0.1353	0.1546	0.1045
st.dev.(E202:12)	0.06071	0.06071	0.06071
R(E202:12)	0.17	0.17	0.17



Determination of Dipropylene Glycol on sample #17208; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	E202	0.04281		-0.21	
169	INH-CM	0.03		-0.47	
171		----		----	
173	INH-540	0.0398		-0.27	
174		----		----	
311	INH-103	0.045		-0.17	
323	E202	0.0438		-0.19	
334		----		----	
347	E2409	<0.01		----	
444	E2409	0.0728		0.39	
446	INH-CM130	0.103		0.99	
551		----		----	
663	E2409	0.090		0.73	
823	E2409	0.0440		-0.19	
902	INH-72	0.0755		0.44	
1016	E202	0.028		-0.51	
1158	INH-003	0.0243		-0.58	
1509	E202	0.0352		-0.36	
1603	In house	0.0820		0.57	
1823	INH-2015	0.0446		-0.18	
2458		----		----	

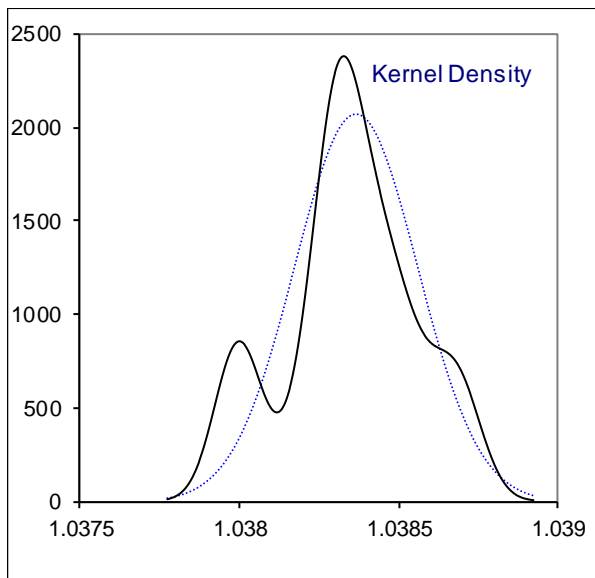
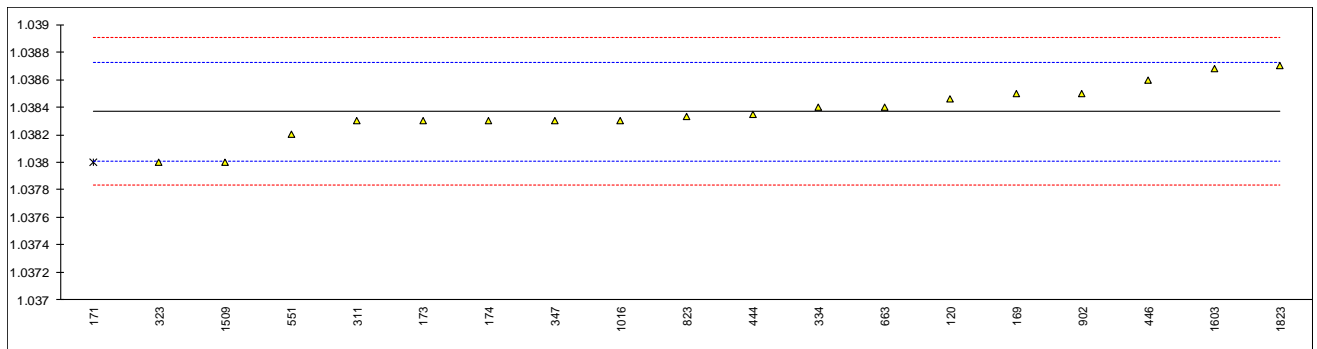
normality OK
 n 15
 outliers 0
 mean (n) 0.05339
 st.dev. (n) 0.024610
 R(calc.) 0.06891
 st.dev.(E202:12) 0.05
 R(E202:12) 0.14



Determination of Specific Gravity 20/20°C on sample #17208;

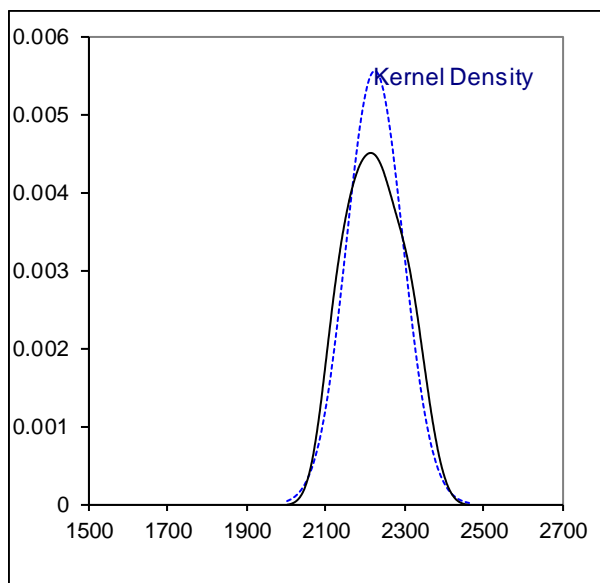
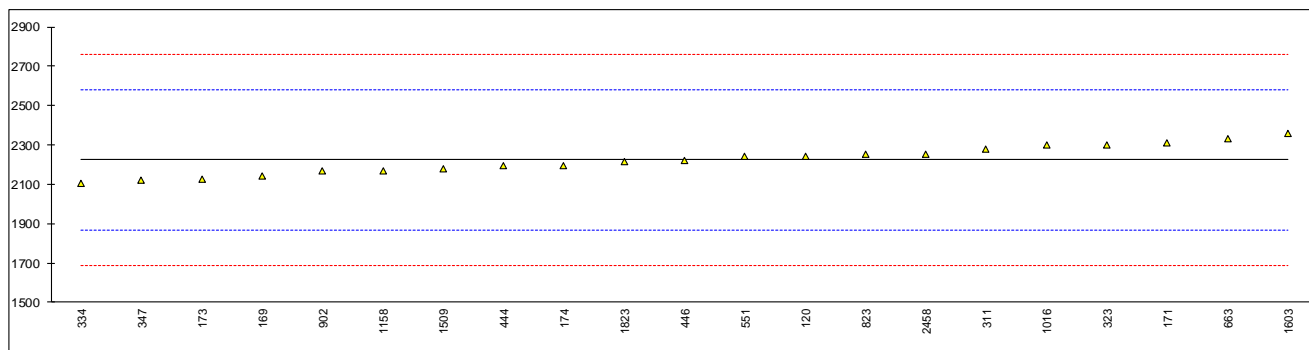
lab	method	value	mark	z(targ)	remarks
120	D4052	1.03846		0.52	
169	D4052	1.0385		0.74	
171	D4052	1.038	ex	-2.06	Test result excluded for being rounded too far
173	D4052	1.0383		-0.38	
174	D4052	1.0383		-0.38	
311	D4052	1.0383		-0.38	
323	E202	1.0380		-2.06	
334	D4052	1.0384		0.18	
347	D4052	1.0383		-0.38	
444	D4052	1.03835		-0.10	
446	D4052	1.0386		1.30	
551	D4052	1.0382		-0.94	
663	D4052	1.038401		0.19	
823	D4052	1.03833		-0.21	
902	D4052	1.0385		0.74	
1016	D4052	1.0383		-0.38	
1158		----		----	
1509	D4052	1.0380		-2.06	
1603	In house	1.03868		1.75	
1823	D4052	1.0387		1.86	
2458		----		----	

normality OK
n 18
outliers 0 (+1 excl)
mean (n) 1.03837
st.dev. (n) 0.000193
R(calc.) 0.00054
st.dev.(E202:12) 0.000179
R(E202:12) 0.0005



Determination of Water on sample #17208; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	E202	2240.3		0.09	
169	E1064	2144		-0.45	
171	E1064	2310		0.48	
173	E203	2128		-0.54	
174	E1064	2194		-0.17	
311	E1064	2280	C	0.31	First reported 0.228
323	E202	2303		0.44	
334	E1064	2107		-0.66	
347	E1064	2123	C	-0.57	First reported 0.2123
444	E1064	2193		-0.17	
446	E203	2220		-0.02	
551	E1064	2240		0.09	
663	E1064	2330.5		0.60	
823	E1064	2251		0.15	
902	E1064	2170		-0.30	
1016	D1364	2300		0.42	
1158	E203	2170	C	-0.30	First reported 0.217
1509	E203	2177		-0.26	
1603	In house	2358		0.75	
1823	E203	2218	C	-0.03	First reported 222
2458	ISO12937	2251	C	0.15	First reported 0.2251
normality		OK			
n		21			
outliers		0			
mean (n)		2224.2			
st.dev. (n)		71.86			
R(calc.)		201.2			
st.dev.(E202:05)		178.57			
R(E202:05)		500			



APPENDIX 2

Number of participants per country

1 lab in BELGIUM

1 lab in BRAZIL

1 lab in CHINA, People's Republic

1 lab in FRANCE

2 labs in GERMANY

2 labs in NETHERLANDS

1 lab in ROMANIA

1 lab in SINGAPORE

1 lab in SOUTH KOREA

1 lab in SPAIN

1 lab in THAILAND

1 lab in TURKEY

2 labs in UNITED KINGDOM

5 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
ex	= test result excluded from the statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
- 2 ASTM E178:16
- 3 ASTM E1301:95(2003)
- 4 ISO 5725:86 (1994)
- 5 ISO 5725, parts 1-6, 1994
- 6 ISO 13528:05
- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical brief, No4 January 2001.
- 14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst 2002, 127, 1359-1364, (2002)
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)
- 16 Horwitz, W and Albert, R, J. AOAC Int, 79, 3, 589, (1996)