Results of Proficiency Test
Mono Propylene Glycol (MPG)
October 2015

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

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1 INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies has organised a proficiency test for the analysis of Mono Propylene Glycol (MPG). As part of the annual proficiency test program of 2015/2016, the Institute for Interlaboratory Studies decided to continue this proficiency test on MPG. In this interlaboratory study, 25 laboratories in 14 different countries have participated. See appendix 2 for the number of participants per country. In this report the results of 2015 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to accredited lab. It was decided to send one sample (1* 0.5 L, labelled #15202) 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/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% 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 was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis internet site www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

One can with approximately 25 litre of MPG was obtained from a local trader.
This batch was spiked with Iron(III)chloride to find concentrations of approx. 0.05 mg Fe/kg and approx. 0.10 mg Cl/kg. From this batch were, after homogenisation, 44 amber glass bottles of 0.5L with inner and outer caps filled and labelled #15202. The homogeneity of the subsamples #15202 was checked by determination of Density at 20°C in accordance with ASTM D4052 and Iron in accordance with ASTM E202 on eight stratified randomly selected samples.

	Density at 20°C in kg/L	Iron in mg/kg
sample #15202-1	1.03611	0.036
sample #15202-2	1.03610	0.042
sample #15202-3	1.03612	0.038
sample #15202-4	1.03609	0.040
sample #15202-5	1.03611	0.038
sample #15202-6	1.03611	0.037
sample #15202-7	1.03611	0.038
sample #15202-8	1.03610	0.039

Table 1: homogeneity tests results of subsamples #15202

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

	Density at 20°C in kg/L	Iron in mg/kg
r (sample #15202)	0.00003	0.005
reference method	ISO12185:96	ASTM E202:05
$0.3xR_{(reference)}$	0.00015	0.021

Table 2: repeatabilities of subsamples #15202

Both calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the respective target method. Therefore, homogeneity of the subsamples #15202 was assumed.

One sample of MPG (0.5 L bottle, labelled #15202) was sent to each of the participating laboratories on October 07, 2015.

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, Density at 20°C, Dipropylene Glycol, Distillation (Initial Boiling Point, 50% recovered and Dry Point), Iron, Purity, Specific Gravity at 20/20°C/^{o}C and Water on sample #15202.
To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/.
A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch the results of the individual laboratories were gathered via the data entry portal www.kmpd.co.uk./sgs-iis/. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.
Directly after deadline, a reminder was sent to those laboratories that had not yet reported.
Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the (raw data of the) reported results.
Additional or corrected results have been used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ (iis-protocol, April 2014 version 3.3). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

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

According to ISO 5725 the original results per determination were submitted to Dixon’s and/or Grubbs' and/or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s

test (no.15). Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for each determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.13 and 14). Also a normal Gauss curve was projected over the Kernel Density Graph.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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

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

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

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

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

4 EVALUATION

In this proficiency test, some problems were encountered with the despatch of the samples. One participant reported the results after the final reporting date and two laboratories did not report any results at all. The 23 participants did report 207 numerical results. Observed were 4 outlying results, which is 1.9% 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 reported 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 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.

Unfortunately, a suitable standard test method, providing the precision data, is not available for chloride. For this test, the spread was compared against the spread estimated from the Horwitz equation.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

Acidity: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in 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 #15202 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: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility limits, calculated using the Horwitz equation. The average recovery of the Chloride (theoretical increment of 0.095 mg Chloride/kg) may be good, less than 112% (the actual blank of Chloride content is unknown).

Colour Pt/Co: 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 requirement of ASTM E202:12.

Density: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ISO12185:96.

DPG: 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.

Distillation: This determination was problematic. In total one statistical outlier was observed and one result was excluded as the reported result for IBP > 50%recovered, which is not possible. The calculated reproducibilities of IBP and 50% recovered are not at all in agreement with the requirements of ASTM E202:05, while the calculated reproducibility for Dry Point is in good agreement with the requirements of ASTM E202:05. From the reported test results of the 50% recovered, it appears that eight participants obviously did not correct the results for barometric pressure and thermometer inaccuracy. Standard ASTM E292:05 refers to ASTM D1078:11 for the conditions to use, see paragraph 11.1.3 and 11.1.4 of ASTM D1078:11

Iron: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM E202:05.
The average recovery of the Iron (theoretical increment of 0.050 mg Iron/kg) may be satisfactory, less than 80% (the actual blank of Iron content is unknown).

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.

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

Water: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of 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	21	0.0004	0.0005	0.0008
Appearance		22	pass	n.a.	n.a.
Chloride as Cl	mg/kg	7	0.11	0.05	0.07
Colour Pt/Co		15	2	2	7
Density at 20°C	kg/L	21	1.0362	0.0004	0.0005
Dipropylene Glycol	%M/M	15	0.008	0.006	0.140
Initial Boiling Point	°C	13	187.0	0.9	0.5
50% recovered	°C	13	187.3	0.8	0.4
Dry Point	°C	13	187.6	1.2	2.5
Iron	mg/kg	20	0.04	0.05	0.07
Purity	%M/M	21	99.97	0.06	0.17
Specific Gravity 20/20°C/°C		20	1.0381	0.0004	0.0005
Water	mg/kg	22	130.7	92.7	500.0

Table 3: reproducibilities of sample #15202

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 2015 WITH PREVIOUS PT

	October 2015	October 2013	October 2011	October 2009
Number of reporting labs	23	19	18	12
Number of results reported	207	189	185	113
Statistical outliers	4	5	6	9
Percentage outliers	1.9%	2.7%	3.2%	8.0%

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 2015	October 2013	October 2011	October 2009
Acidity as Acetic Acid	+	+	++	++
Chloride as Cl	+	n.e.	++	++
Colour Pt/Co	++	++	++	++
Density at 20^{o}C	+	++	++	++
Dipropylene Glycol	++	++	++	++
Initial Boiling Point	--	-	-	-
50% recovered	--	+	--	--
Dry Point	++	++	++	++
Iron	+	++	++	++
Purity	++	++	++	++
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:

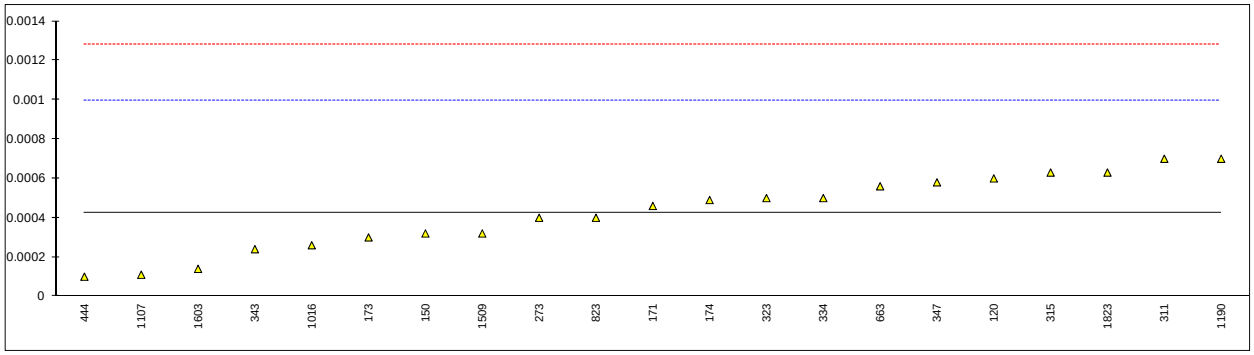
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

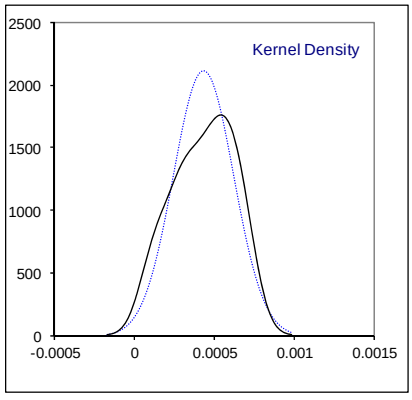
APPENDIX 1

Determination of Acidity as Acetic Acid on sample #15202; results in %M/M.

lab	method	value	mark	z(targ)	remarks
120	D1613	0.0006		0.61	
150	D1613	0.00032		-0.37	
171	D1613	0.00046	C	0.12	probably unit error, reported: 4.6 %M/M
173	D1613	0.0003		-0.44	
174	D1613	0.00049	C	0.22	first reported:4.9
273	D1613	0.0004		-0.09	
311	D1613	0.0007		0.96	
315	INH-101370	0.00063		0.71	
323	E202	0.0005		0.26	
334	D1613	0.0005		0.26	
343	INH-CM	0.00024		-0.65	
347	D1613	0.00058		0.54	
444	D1613	0.0001		-1.14	
446		-----		-----	
551		-----		-----	
558		-----		-----	
663	D1613	0.00056		0.47	
823	D1613	0.0004		-0.09	
902		-----		-----	
1016	D1613	0.00026		-0.58	
1107	E202	0.00011	C	-1.11	first reported:1.1
1190	D1613	0.0007		0.96	
1509	D1613	0.00032		-0.37	
1603	in house	0.00014		-1.00	
1823	D1613	0.00063		0.71	

normality	OK
n	21
outliers	0
mean (n)	0.00043
st.dev. (n)	0.000189
R(calc.)	0.00053
R(E202:05)	0.00080





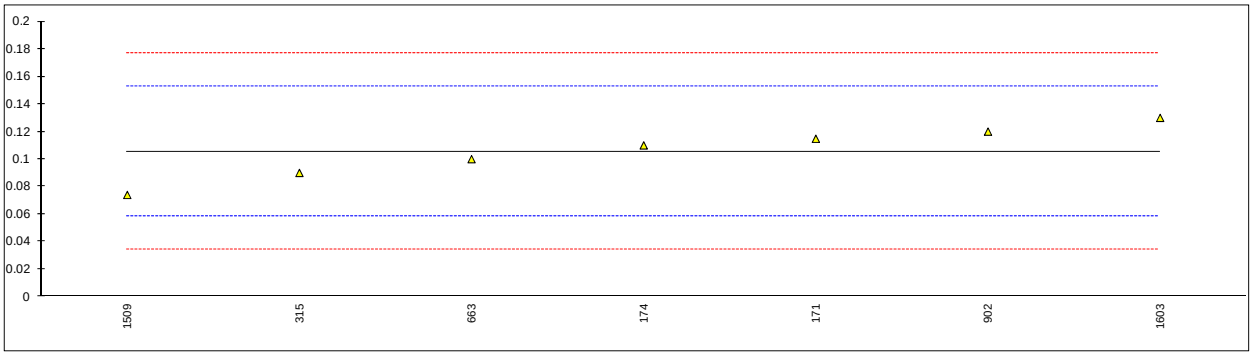
Determination of Appearance on sample #15202

lab	method	value	mark	z(targ)	remarks
120	Visual	Pass		-----	
150	E2680	Pass		-----	
171	E2680	Pass		-----	
173	E2680	Pass		-----	
174	E2680	Pass		-----	
273	Visual	Bright and Clear		-----	
311	E2680	Pass		-----	
315	INH-402	Bright and Clear		-----	
323	Visual	Clear & Bright		-----	
334		-----		-----	
343	E2680	Pass		-----	
347	E2680	Pass		-----	
444	E2680	Pass		-----	
446	E2680	Pass		-----	
551		-----		-----	
558		-----		-----	
663	Visual	Pass		-----	
823	Visual	Pass		-----	
902	E2680	Pass		-----	
1016	in house	Pass		-----	
1107	Visual	Clear		-----	
1190	Visual	Pass		-----	
1509	E2680	Pass		-----	
1603	Visual	Pass		-----	
1823	E2680	Pass		-----	
	normality	n.a.			
	n	22			
	mean (n)	Pass			

Determination of Chloride as Cl on sample #15202; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
120		-----		-----	
150		-----		-----	
171	E2469	0.11483		0.39	
173	INH-0221	<0.5		-----	
174	E2469	0.11		0.19	
273		-----		-----	
311	INH-158	<0.2		-----	
315	INH-158	0.090		-0.66	
323	E2469	<0.1		-----	
334		-----		-----	
343	INH-CM	<0.5		-----	
347		-----		-----	
444		-----		-----	
446	INH-3221	<1		-----	
551		-----		-----	
558		-----		-----	
663	INH-100867	0.10		-0.23	
823		-----		-----	
902	E2469	0.12		0.61	
1016		-----		-----	
1107	in house	<0.2		-----	
1190	in house	<10		-----	
1509	in house	0.074		-1.33	
1603	in house	0.130		1.03	
1823		-----		-----	

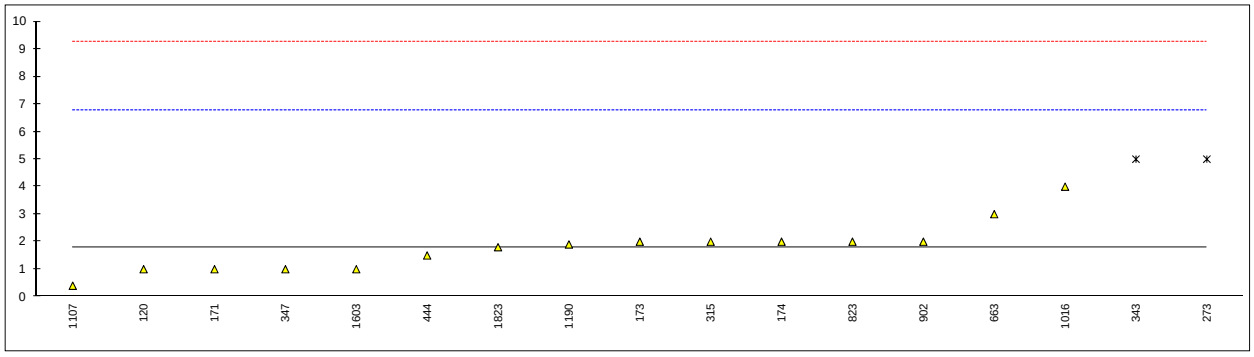
normality	unknown	spike	%rec
n	7		
outliers	0		
mean (n)	0.106	0.095	<112%
st.dev. (n)	0.0191		
R(calc.)	0.053		
R(Horwitz)	0.066		

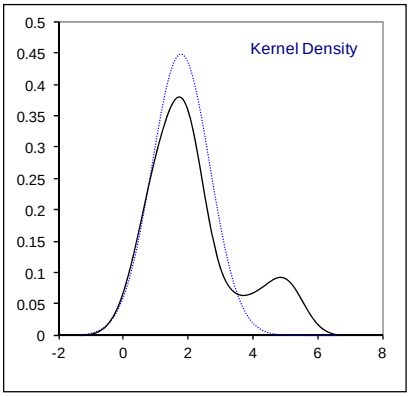


Determination of Colour Pt/Co on sample #15202

lab	method	value	mark	z(targ)	remarks
120	D1209	1		-0.31	
150	D5386	<1		-----	
171	E202	1		-0.31	
173	D1209	2		0.09	
174	D1209	2		0.09	
273	D1209	5	DG(0.05)	1.29	
311	D1209	<5		-----	
315	D5386	2.0		0.09	
323	D1209	<5		-----	
334		-----		-----	
343	D5386	5	DG(0.05)	1.29	
347	D5386	1		-0.31	
444	D5386	1.5		-0.11	
446	D1209	<5		-----	
551		-----		-----	
558		-----		-----	
663	D1209	3		0.49	
823	D5386	2		0.09	
902	D5386	2		0.09	
1016	D1209	4		0.89	
1107	D5386	0.4		-0.55	
1190	D1209	1.9		0.05	
1509	D1209	<5		-----	
1603	in house	1		-0.31	
1823	D5386	1.8		0.01	

normality	suspect
n	15
outliers	2
mean (n)	1.8
st.dev. (n)	0.90
R(calc.)	2.5
R(E202:12)	7.0

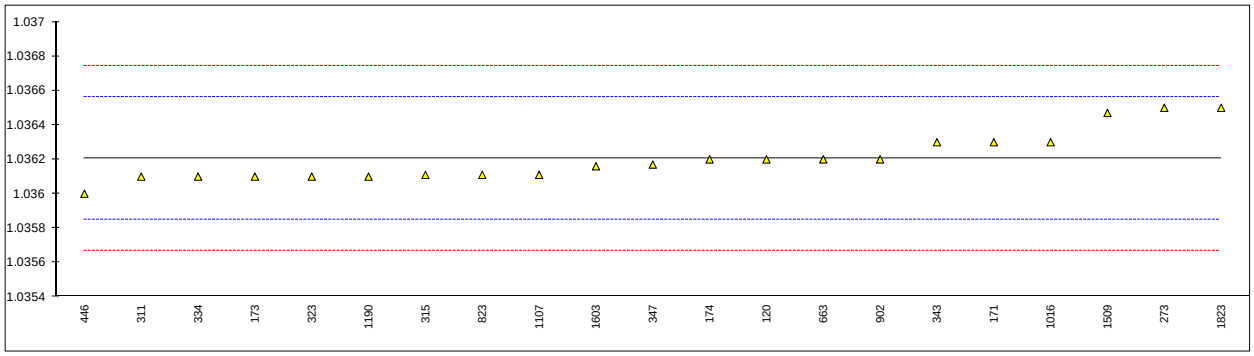


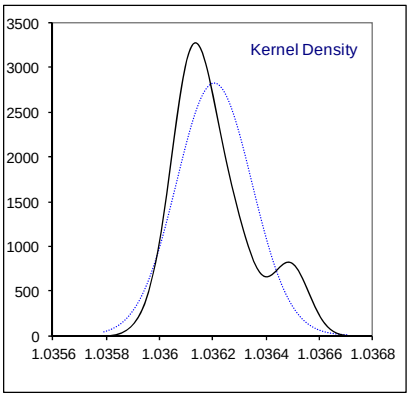


Determination of Density at 20°C on sample #15202; results in kg/L.

lab	method	value	mark	z(targ)	remarks
120	D4052	1.0362		-0.03	
150		-----		-----	
171	ISO12185	1.0363		0.53	
173	D4052	1.0361		-0.59	
174	D4052	1.0362		-0.03	
273	D4052	1.0365		1.65	
311	D4052	1.0361		-0.59	
315	D4052	1.03611		-0.54	
323	D4052	1.0361		-0.59	
334	ISO12185	1.0361		-0.59	
343	ISO12185	1.0363		0.53	
347	D4052	1.03617		-0.20	
444		-----		-----	
446	D4052	1.036		-1.15	
551		-----		-----	
558		-----		-----	
663	D4052	1.0362		-0.03	
823	ISO12185	1.03611		-0.54	
902	D4052	1.0362		-0.03	
1016	D4052	1.0363		0.53	
1107	D4052	1.03611		-0.54	
1190	D4052	1.0361		-0.59	
1509	D4052	1.03647		1.48	
1603	in house	1.03616		-0.26	
1823	D4052	1.0365		1.65	

normality	suspect
n	21
outliers	0
mean (n)	1.03621
st.dev. (n)	0.000141
R(calc.)	0.00040
R(ISO12185:96)	0.00050

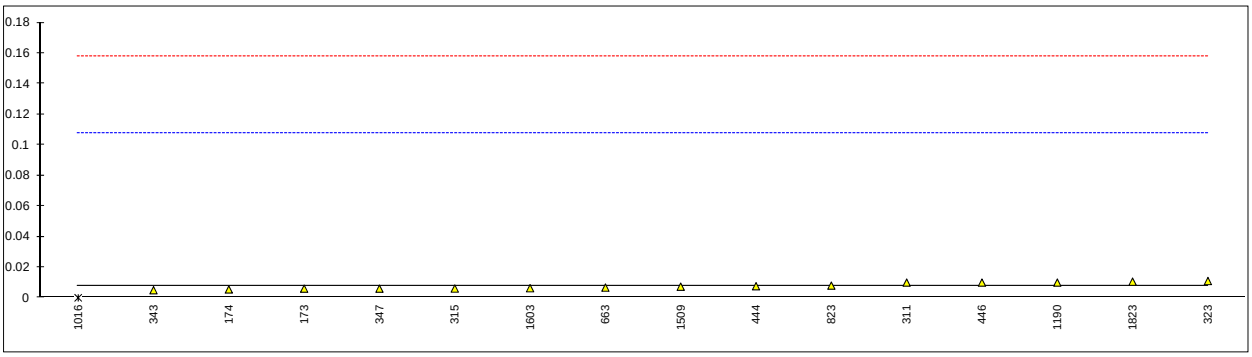


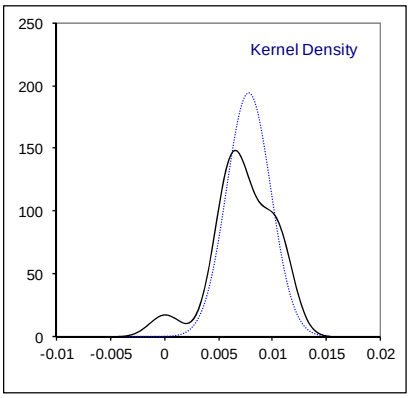


Determination of Dipropylene Glycol on sample #15202; results in %M/M.

lab	method	value	mark	z(targ)	remarks
120	E2409	<0.01		-----	
150		-----		-----	
171		-----		-----	
173	INH-0540	0.006		-0.03	
174	E2409	0.0055		-0.04	
273		-----		-----	
311	INH-103	0.010		0.05	
315	INH-100687	0.0061		-0.03	
323	E202	0.0110		0.07	
334		-----		-----	
343	INH-CM	0.005		-0.05	
347	E2409	0.0060		-0.03	
444	INH-CM	0.0076		0.00	
446	INH-130	0.01		0.05	
551		-----		-----	
558		-----		-----	
663	INH-100687	0.00669		-0.02	
823	E202	0.008		0.01	
902		-----		-----	
1016	E202	0	ex	-0.15	result excluded, zero is not a real value
1107		-----		-----	
1190	in house	0.01		0.05	
1509	E202	0.0073		-0.01	
1603	in house	0.0063		-0.03	
1823	E2409	0.0106		0.06	

normality	OK
n	15
outliers	0 + 1 excl.
mean (n)	0.0077
st.dev. (n)	0.00205
R(calc.)	0.0057
R(E202:12)	0.1400





Determination of Distillation: IBP, 50% recovered, Dry Point on sample #15202; results in °C.

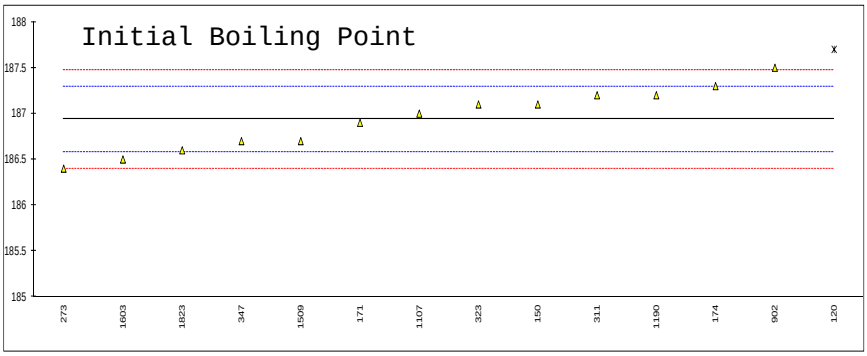
lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)	remarks
120	D1078	187.7	ex	4.26	187.3		-0.16	187.3		-0.32	
150	D1078	187.1		0.90	187.6		1.94	187.6		0.02	
171	D1078	186.9		-0.22	187.2		-0.86	187.4		-0.21	
173		-----		-----	-----		-----	-----		-----	
174	D1078	187.3		2.02	187.6		1.94	187.7		0.13	
273	D1078	186.4		-3.02	-----		-----	186.9		-0.77	
311	D1078	187.2		1.46	187.6		1.94	187.9		0.35	
315		-----		-----	-----		-----	-----		-----	
323	D1078	187.1		0.90	187.4		0.54	187.8		0.24	
334		-----		-----	-----		-----	-----		-----	
343		-----		-----	-----		-----	-----		-----	
347	D1078	186.7		-1.34	187.3		-0.16	187.4		-0.21	
444		-----		-----	-----		-----	-----		-----	
446		-----		-----	-----		-----	-----		-----	
551		-----		-----	-----		-----	-----		-----	
558		-----		-----	-----		-----	-----		-----	
663		-----		-----	-----		-----	-----		-----	
823		-----		-----	-----		-----	-----		-----	
902	D1078	187.5		3.14	187.7		2.64	187.8		0.24	
1016		-----		-----	-----		-----	-----		-----	
1107	D1078	187.0		0.34	187.0		-2.26	189.5	G(0.05)	2.15	
1190	D1078	187.2		1.46	187.6		1.94	188.0		0.47	
1509	D1078	186.7		-1.34	187.0		-2.26	188.2		0.69	
1603	in house	186.5		-2.46	186.7		-4.36	186.7		-0.99	
1823	D1078	186.6		-1.90	187.2		-0.86	187.9		0.35	
	normality	OK			OK			OK			
	n	13			13			13			
	outliers	0 (+1 excl)			0			1			
	mean (n)	186.94			187.32			187.58			
	st.dev. (n)	0.336			0.300			0.434			
	R(calc.)	0.94			0.84			1.21			
	R(E202:05)	0.50			0.40			2.50			
	Compare R(D1078:11)	2.92			1.28			2.01			

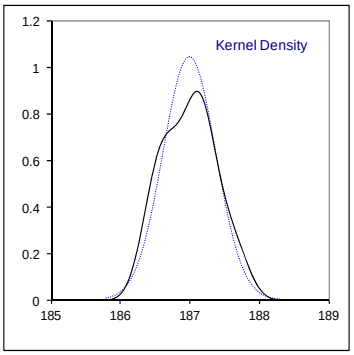
Lab 120: IBP excluded as report result is larger than 50%recovered (typo error?)

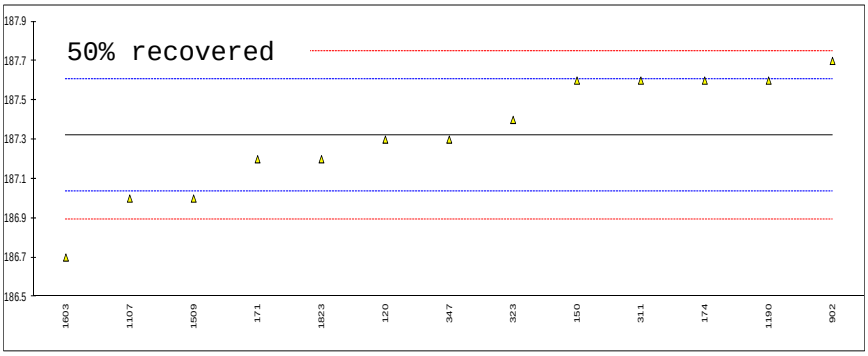
Several laboratories did not correct for theoretical mid boiling point (187.6). Results after manual correction:

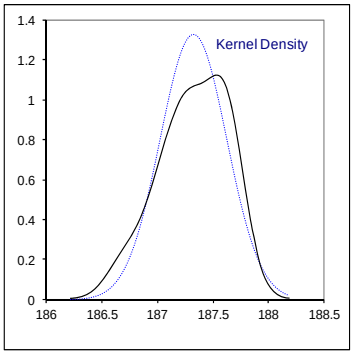
lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)	remarks
120	D1078	188.0	ex	4.48	187.6		-0.05	187.6		-0.25	
171	D1078	187.3		0.56	187.6		-0.05	187.8		-0.03	
323	D1078	187.3		0.56	187.6		-0.05	188.0		0.20	
347	D1078	187.0		-1.12	187.6		-0.05	187.7		-0.14	
1107	D1078	187.6		2.24	187.6		-0.05	190.1		2.55	
1509	D1078	187.3		0.56	187.6		-0.05	188.8		1.09	
1603	in house	187.4		1.12	187.6		-0.05	187.6		-0.25	
1823	D1078	187.0		-1.12	187.6		-0.05	188.3		0.53	
	normality	not OK			not OK			not OK			
	n	13			13			13			
	outliers	0 (+1 excl)			0			1			
	mean (n)	187.20			187.61			187.82			
	st.dev. (n)	0.297			0.028			0.436			
	R(calc.)	0.83			0.08			1.22			
	R(E202:05)	0.50			0.40			2.50			

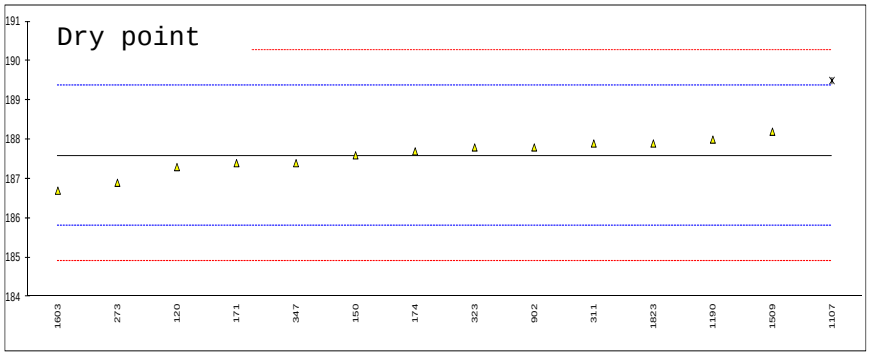
Before corrections:

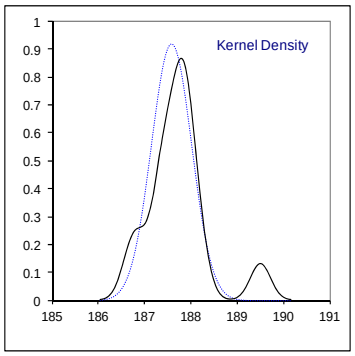








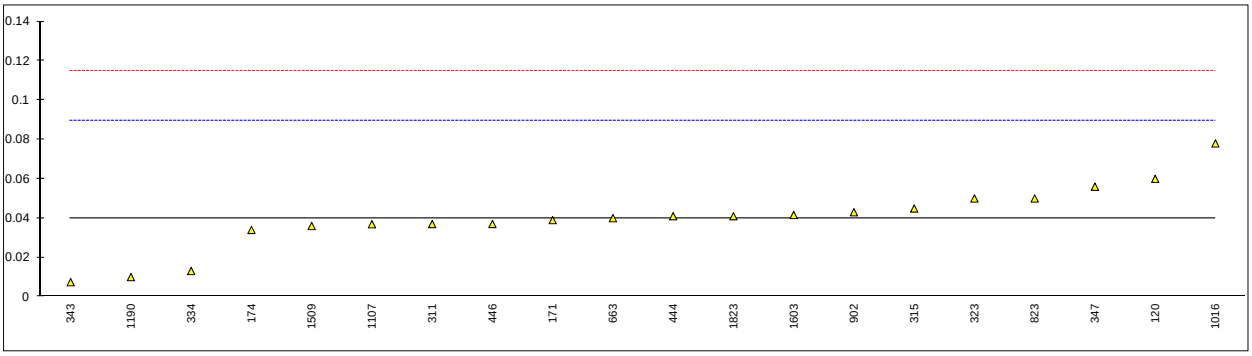


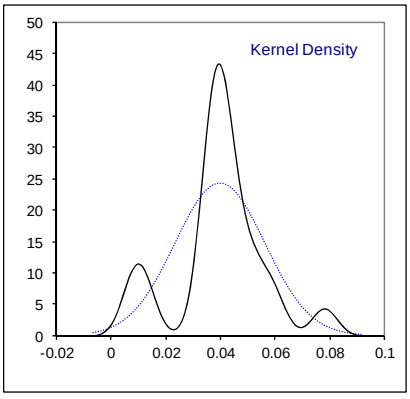


Determination of Iron as Fe on sample #15202; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
120	D1615	0.06		0.81	
150	E394	<0.1		-----	
171	E1615	0.039		-0.03	
173	INH-0290	<0.1		-----	
174	D1615	0.034		-0.23	
273		-----		-----	
311	D1615	0.037		-0.11	
315	E202	0.0449		0.20	
323	E1615	0.050		0.41	
334	E202	0.0131		-1.07	
343	D1615	0.0074		-1.30	
347	E394	0.056		0.65	
444	E202	0.041		0.05	
446	E202	0.037		-0.11	
551		-----		-----	
558		-----		-----	
663	E394	0.040		0.01	
823	E394	0.05		0.41	
902	E394	0.043		0.13	
1016	NEN6966	0.078		1.53	
1107	E202	0.0369		-0.12	
1190	E202	0.01		-1.19	
1509	E394	0.036		-0.15	
1603	in house	0.0416		0.07	
1823	D1615	0.041		0.05	

	normality	suspect	spike	%rec.
	n	20		
	outliers	0		
	mean (n)	0.0398	0.050	<80%
	st.dev. (n)	0.01639		
	R(calc.)	0.0459		
	R(E202:05)	0.0700		

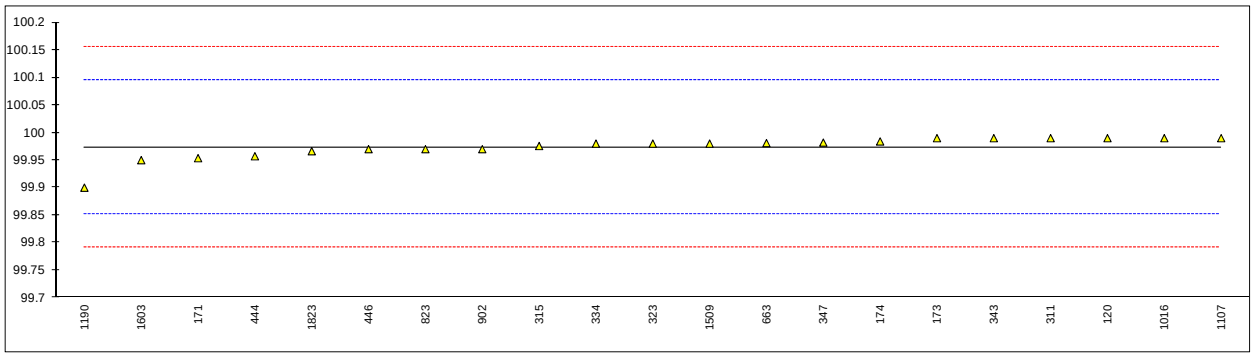


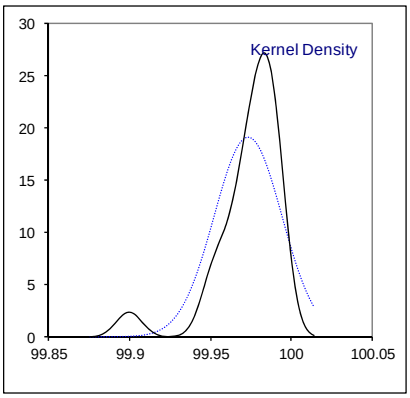


Determination of Purity on sample #15202; results in %M/M.

lab	method	value	mark	z(targ)	remarks
120	E2409	99.99		0.27	
150		-----		-----	
171	E2409	99.9536		-0.32	
173	INH-0540	99.99		0.27	
174	E2409	99.984		0.18	
273		-----		-----	
311	INH-103	99.99		0.27	
315	INH-100687	99.9757		0.04	
323	E2409	99.98		0.11	
334	INH-103	99.98		0.11	
343	INH-CM	99.99		0.27	
347	E2409	99.9819		0.14	
444	INH-CM	99.9571		-0.27	
446	INH-130	99.97		-0.05	
551		-----		-----	
558		-----		-----	
663	INH-100687	99.981		0.13	
823	E202	99.97		-0.05	
902	INH-72	99.97		-0.05	
1016	E202	99.990		0.27	
1107		99.99		0.27	
1190	in house	99.9		-1.21	
1509	E202	99.980		0.11	
1603	in house	99.95		-0.38	
1823	E2409	99.9663		-0.12	

normality	not OK
n	21
outliers	0
mean (n)	99.973
st.dev. (n)	0.0209
R(calc.)	0.058
R(E202:12)	0.170

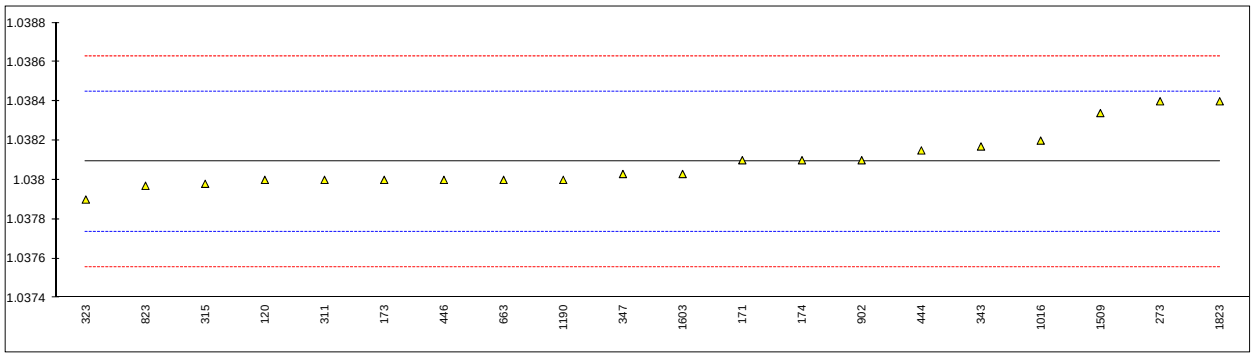


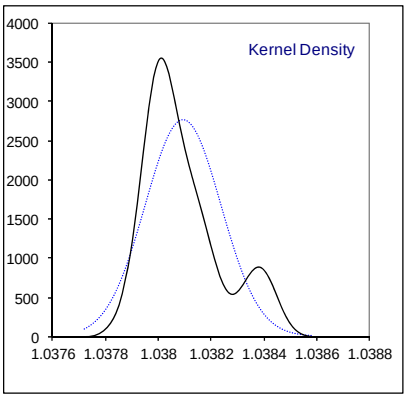


Determination of Specific Gravity 20/20°C/°C on sample #15202;

lab	method	value	mark	z(targ)	remarks
120	D4052	1.0380		-0.52	
150		-----		-----	
171	D4052	1.0381		0.04	
173	D4052	1.0380		-0.52	
174	D4052	1.0381		0.04	
273	D4052	1.0384		1.72	
311	D4052	1.0380		-0.52	
315	D4052	1.03798		-0.64	
323	D4052	1.0379		-1.08	
334		-----		-----	
343	D4052	1.03817		0.43	
347	D4052	1.03803		-0.36	
444	D4052	1.03815		0.32	
446	D4052	1.038		-0.52	
551		-----		-----	
558		-----		-----	
663	D4052	1.0380		-0.52	
823	D4052	1.03797		-0.69	
902	D4052	1.0381		0.04	
1016	D4052	1.0382		0.60	
1107		-----		-----	
1190	D4052	1.0380		-0.52	
1509	D4052	1.03834		1.38	
1603	in house	1.03803		-0.36	
1823	D4052	1.0384		1.72	

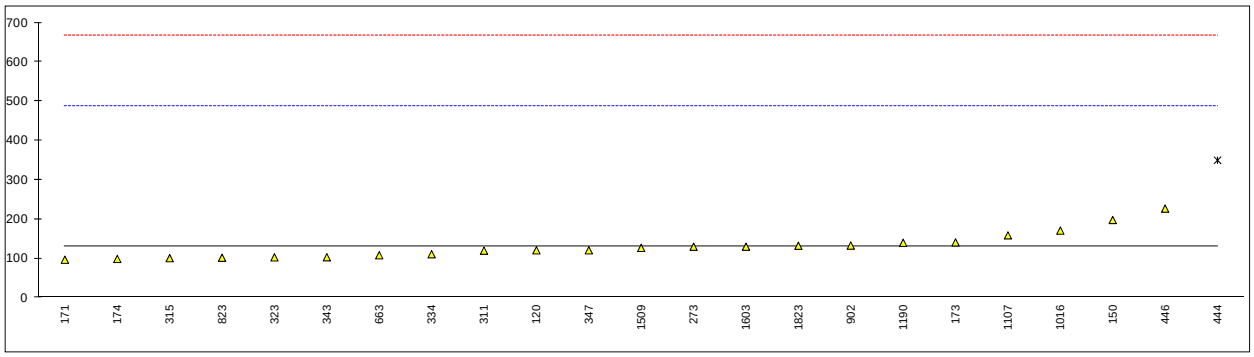
normality	suspect
n	20
outliers	0
mean (n)	1.03809
st.dev. (n)	0.000144
R(calc.)	0.00040
R(E202:12)	0.00050

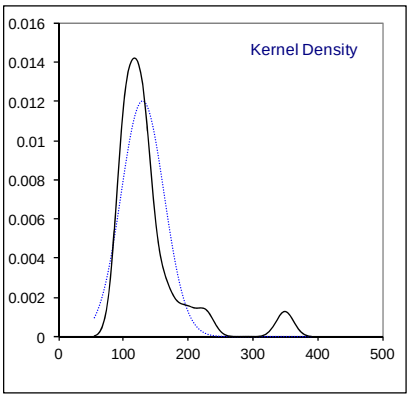




Determination of Water, coulometric KF titration on sample #15202; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	E1064	121	C	-0.05	first reported:0.01
150	E1064	198		0.38	
171	E1064	97.0		-0.19	
173	E203	141		0.06	
174	E1064	99		-0.18	
273	E1064	130		0.00	
311	E1064	120	C	-0.06	first reported:0.012
315	E1064	101		-0.17	
323	E1064	103		-0.16	
334	E1064	111		-0.11	
343	E1064	103.3	C	-0.15	first reported:0.01033
347	E1064	121		-0.05	
444	E203	350	G(0.01)	1.23	
446	E203	227		0.54	
551		-----		-----	
558		-----		-----	
663	E1064	108.3		-0.13	
823	E203	102		-0.16	
902	E1064	133		0.01	
1016	D1364	171		0.23	
1107	E202	159	C	0.16	first reported:0.016
1190	D1364	140		0.05	
1509	E203	127		-0.02	
1603	in house	130		0.00	
1823	E203	132.5		0.01	
	normality	not OK			
	n	22			
	outliers	1			
	mean (n)	130.69			
	st.dev. (n)	33.095			
	R(calc.)	92.67			
	R(E202:05)	500.00			Compare R(E1064:12) = 22.35





APPENDIX 2

Number of participants per country

2 labs in	BELGIUM
2 labs in	BRAZIL
1 lab in	CHINA, People's Republic
1 lab in	FRANCE
1 lab in	GERMANY
3 labs in	NETHERLANDS
1 lab in	SINGAPORE
1 lab in	SOUTH AFRICA
1 lab in	SOUTH KOREA
2 labs in	SPAIN
1 lab in	THAILAND
1 lab in	TURKEY
2 labs in	UNITED KINGDOM
6 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
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner 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, April 2014

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3 ASTM E1301-89

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

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9 DIN 38402 T41/42

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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.

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15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)