

**Results of Proficiency Test
Mono Propylene glycol (MPG)
October 2021**

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2004 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Mono Propylene glycol (MPG) in accordance with the latest version of ASTM E202 once every two year. During the annual proficiency testing program of 2021/2022 it was decided to continue the round robin for the analysis of Mono Propylene glycol.

In this interlaboratory study 23 laboratories in 16 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the Mono Propylene glycol 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 organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.

It was decided to send one sample MPG in a 0.5L glass bottle labelled #21195.

The 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/IEC17043:2010 (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 organization 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 June 2018 (iis-protocol, version 3.5). 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

A batch of approximately 30 liters of MPG was obtained from a local supplier. After homogenization 54 amber glass bottles of 0.5L were filled and labelled #21195. The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ASTM D4052 and Water in accordance with ASTM E1064 on 8 stratified randomly selected subsamples.

	Density at 20°C in kg/L	Water in mg/kg
sample #21195-1	1.03620	960
sample #21195-2	1.03621	980
sample #21195-3	1.03622	970
sample #21195-4	1.03622	990
sample #21195-5	1.03622	970
sample #21195-6	1.03622	980
sample #21195-7	1.03622	970
sample #21195-8	1.03621	1000

Table 1: homogeneity test results of subsamples #21195

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 20°C in kg/L	Water in mg/kg
r (observed)	0.00002	36
reference test method	ISO12185:96	ASTM E202:05
0.3 x R (reference test method)	0.00015	500

Table 2: evaluation of the repeatabilities of subsamples #21195

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample MPG labelled #21195 was sent on September 22, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

The participants were requested to determine: Acidity as Acetic Acid, Appearance, Inorganic Chloride as Cl, Color Pt/Co, Density at 20°C, Distillation (IBP, 50% recovered, Dry Point), Iron as Fe, Purity by GC as received, Dipropylene glycol, Specific Gravity at 20/20°C and Water.

It was explicitly requested to treat the sample as if it was a routine sample 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' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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 (when applicable) 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 reanalyzes). Additional or corrected test results are used for data analysis and the 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 organization 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 June 2018 (iis-protocol, version 3.5).

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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier tests can be used. 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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualize 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 or ISO 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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 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 is as follows:

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

4 EVALUATION

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. All participants reported test results of which three participants reported the test results after the extended reporting date. Not all laboratories were able to report all tests requested. In total 23 participants reported 191 numerical test results. Observed were 8 outlying test results, which is 4.2%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all 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.

4.1 EVALUATION PER TEST

In this section the reported test 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 explained in appendix 3.

In the iis PT reports ASTM test methods are referred to with a number and if appropriate an indication of sub test method (e.g. D1209) and an added designation for the year that the test method was adopted or revised (e.g. D1209:05). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1209:05(2019)). In the test results tables of appendix 1 only the method number (sub) and year of adoption or revision (e.g. D1209:05) will be used.

Acidity as Acetic Acid: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1613:17

Appearance: This determination was not problematic. All reporting participants, except one, agreed about the appearance; bright, clear and free of suspended matter or pass in accordance with ASTM E2680:16.

Inorganic Chloride as Cl: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM E2469:16.

Color Pt/Co: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D1209:05(2019) and ASTM D5386:16.

Density at 20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. No statistical outliers were observed over three distillation parameters. All three calculated reproducibilities are in agreement with the requirements of ASTM D1078:11(2019) automated and manual modes.

Iron: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility was very large. Therefore, no z-scores are calculated.

Purity by GC as received: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM E202:18.

Dipropylene glycol: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM E202:18.

Specific Gravity 20/20°C: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM E202:18.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM E202:05 and ASTM E1064:16.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference test methods (in casu ASTM and ISO test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acidity as Acetic Acid	%M/M	20	0.0011	0.0008	0.0014
Appearance		21	Pass	n.a.	n.a.
Inorganic Chloride as Cl	mg/kg	8	0.27	0.22	0.22
Color Pt/Co		15	2.5	2.3	7
Density at 20°C	kg/L	22	1.0362	0.0004	0.0005
Initial Boiling Point	°C	11	186.1	1.1	2.9
50% recovered	°C	11	187.3	0.6	1.3
Dry Point	°C	11	188.1	0.7	2.0
Iron as Fe	mg/kg	13	0.12	0.23	(0.07)
Purity by GC as received	%M/M	17	99.813	0.217	0.17
Dipropylene glycol	%M/M	14	0.037	0.035	0.14
Specific Gravity 20/20°C		19	1.0381	0.0002	0.0005
Water	mg/kg	22	1020	191	500

Table 3: reproducibilities of tests on sample #21195

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2021 WITH PREVIOUS PTS

	October 2021	October 2019	October 2017	October 2015	October 2013
Number of reporting labs	23	22	21	23	19
Number of test results	191	181	177	207	189
Number of statistical outliers	8	4	2	4	5
Percentage of statistical outliers	4.2%	2.2%	1.1%	1.9%	2.7%

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 compared to the requirements of the reference test methods. The conclusions are given the following table.

	October 2021	October 2019	October 2017	October 2015	October 2013
Acidity as Acetic Acid	+	+	+	+	+
Inorganic Chloride as Cl	+/-	n.e.	--	+	n.e.
Color Pt/Co	++	++	++	++	++
Density at 20°C	+	+	+/-	+	++
Initial Boiling Point	++	++	--	--	-
50% recovered	++	+	--	--	+
Dry Point	++	+	++	++	++
Iron as Fe	(--)	+	(--)	+	++
Purity by GC as received	-	++	(--)	++	++
Dipropylene glycol	++	++	++	++	++
Specific Gravity 20/20°C	++	+	+/-	+	++
Water	++	++	++	++	++

Table 5: comparison determinations against the reference test methods

Results between brackets should be used with due care

The following performance categories in above table were used:

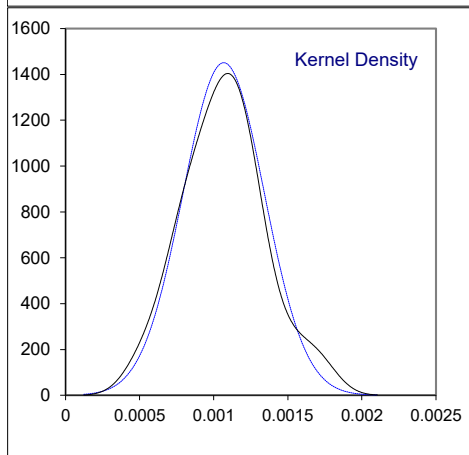
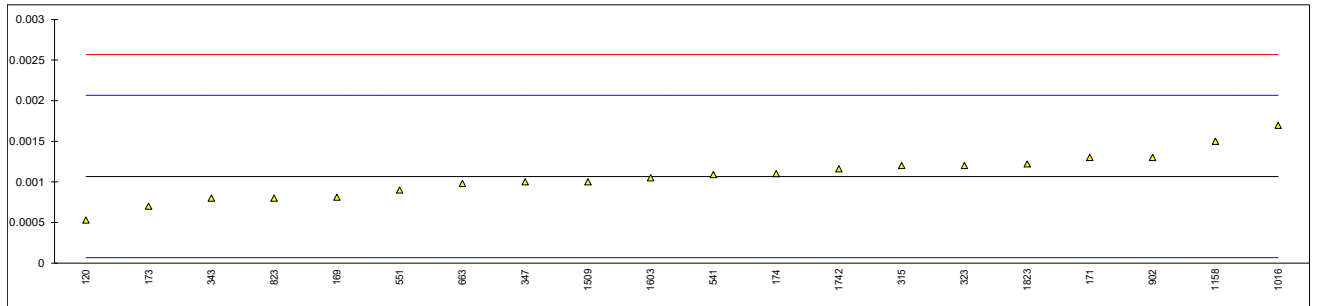
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Acidity as Acetic Acid on sample #21195; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	D1613	0.00053		-1.07	
169	D1613	0.00081		-0.51	
171	D1613	0.0013	C	0.47	Reported 13%M/M
173	E202	0.0007		-0.73	
174	D1613	0.0011		0.07	
315	D1613	0.0012		0.27	
323	D1613	0.0012	C	0.27	First reported 12%M/M
334		----		----	
343	INH-2196	0.0008		-0.53	
347	D1613	0.0010		-0.13	
444		----		----	
446		----		----	
541	D1613	0.00109		0.05	
551	D1613	0.0009		-0.33	
663	D1613	0.00098		-0.17	
823	D1613	0.0008		-0.53	
902	D1613	0.0013		0.47	
1016	D1613	0.0016959		1.26	
1158	D1613	0.0015		0.87	
1509	D1613	0.0010		-0.13	
1603	In house	0.00105		-0.03	
1742	E2679	0.00116		0.19	
1823	D1613	0.00122		0.31	

normality OK
 n 20
 outliers 0
 mean (n) 0.001067
 st.dev. (n) 0.0002750
 R(calc.) 0.000770
 st.dev.(D1613:17) 0.0005
 R(D1613:17) 0.0014

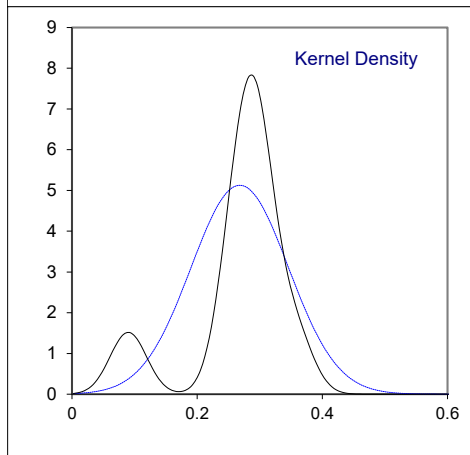
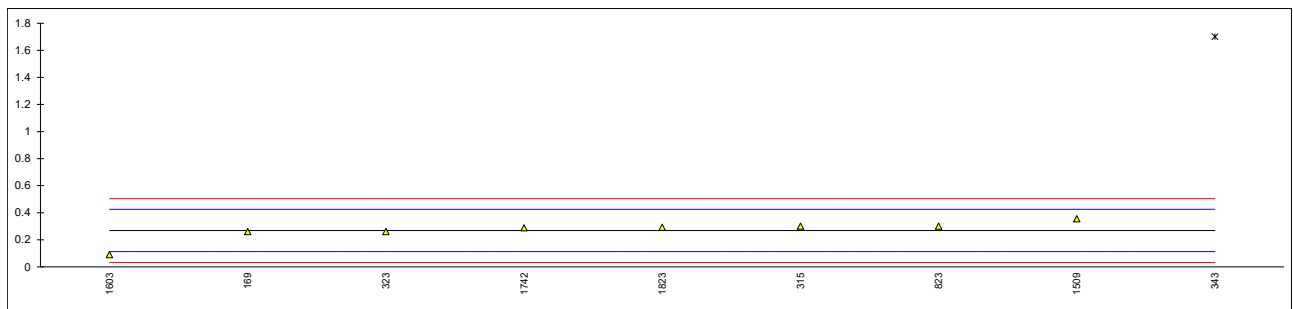


Determination of Appearance on sample #21195;

lab	method	value	mark	z(targ)	remarks
120	D4176	Pass		----	
169	Visual	Pass		----	
171	E2680	pass		----	
173	E2680	Pass		----	
174	Visual	Clear & Free		----	
315	E2680	pass		----	
323	D2679	clear and bright		----	
334	Visual	clear & bright		----	
343	E2680	PASS		----	
347	E2680	Pass		----	
444	E2680	Pass		----	
446	E2680	Pass		----	
541	E2680	pass		----	
551	E2680	Pass		----	
663	Visual	Pass		----	
823	E2680	Pass		----	
902	E2680	PASS		----	
1016	Visual	Pass		----	
1158		----		----	
1509	E2680	Clear & FFSM		----	
1603	Visual	PASS		----	
1742	E2680	Fail		----	
1823	D4176	Pass		----	
	n	21			
	mean	Pass (Bright & Clear)	1		Fail

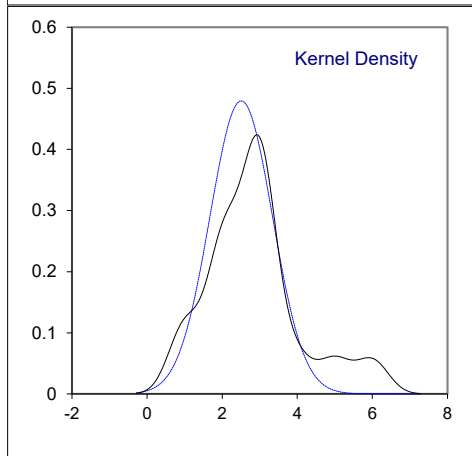
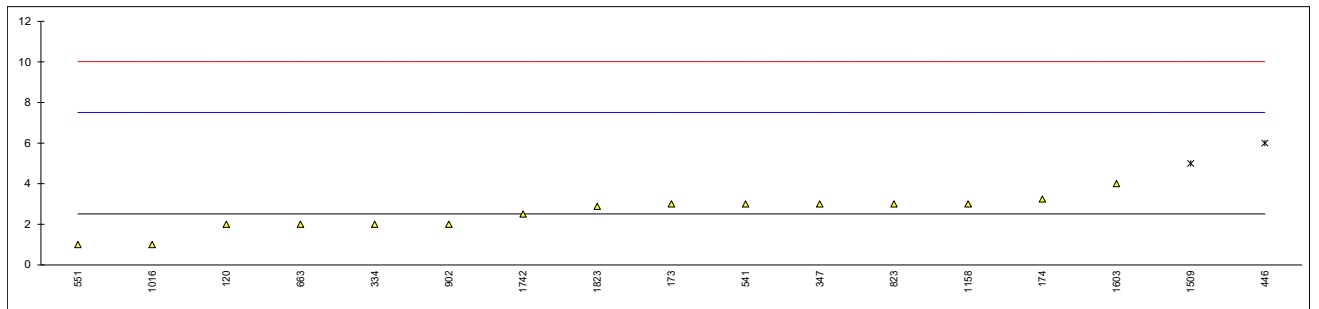
Determination of Inorganic Chloride as Cl on sample #21195; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120		----		----	
169	E2469	0.26		-0.10	
171	E2469	<0.5		----	
173	INH-0221	>0.5		----	
174		----		----	
315	INH-158	0.3		0.41	
323	E2469	0.26		-0.10	
334	INH-96001	<0.5		----	
343	INH-2195	1.7	G(0.01)	18.27	
347		----		----	
444		----		----	
446		----		----	
541		----		----	
551		----		----	
663		----		----	
823	E2469	0.30		0.41	
902		----		----	
1016		----		----	
1158		----		----	
1509	E2469	0.3550		1.11	
1603	In house	0.09		-2.27	
1742	E2469	0.288		0.25	
1823	E2469	0.2920		0.30	
	normality	not OK			
	n	8			
	outliers	1			
	mean (n)	0.2681			
	st.dev. (n)	0.07783			
	R(calc.)	0.2179			
	st.dev.(E2469:16)	0.07838			
	R(E2469:16)	0.2195			



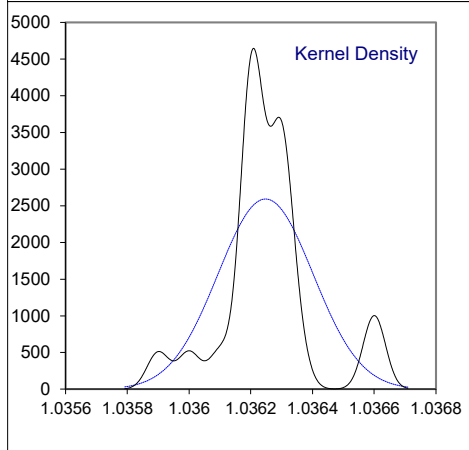
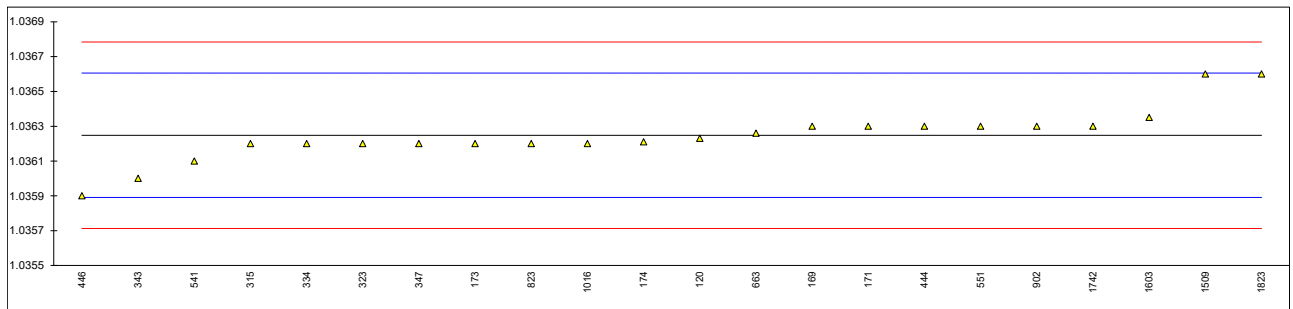
Determination of Color Pt/Co on sample #21195;

lab	method	value	mark	z(targ)	remarks
120	D1209	2		-0.20	
169	D1209	<5		----	
171	D5386	<5		----	
173	D1209	3		0.20	
174	D5386	3.24		0.29	
315	D1209	<5		----	
323	D1209	< 5		----	
334	D1209	2		-0.20	
343		----		----	
347	D5386	3		0.20	
444	D5386	<5		----	
446	D1209	6	DG(0.05)	1.40	
541	D5386	3		0.20	
551	D1209	1		-0.60	
663	D5386	2		-0.20	
823	D5386	3		0.20	
902		2		-0.20	
1016	D1209	1		-0.60	
1158	D1209	3		0.20	
1509	D1209	5	DG(0.05)	1.00	
1603	In house	4		0.60	
1742	D1209	2.5		0.00	
1823	D5386	2.88		0.15	
normality		OK			
n		15			
outliers		2			
mean (n)		2.508			
st.dev. (n)		0.8320			
R(calc.)		2.330			
st.dev.(D1209:05)		2.5			
R(D1209:05)		7			
Compare					
R(D5386:16)		4.988			



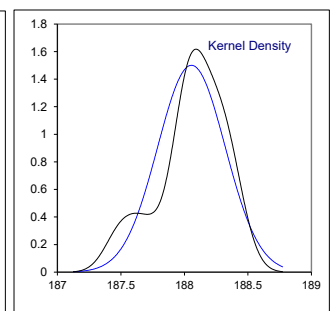
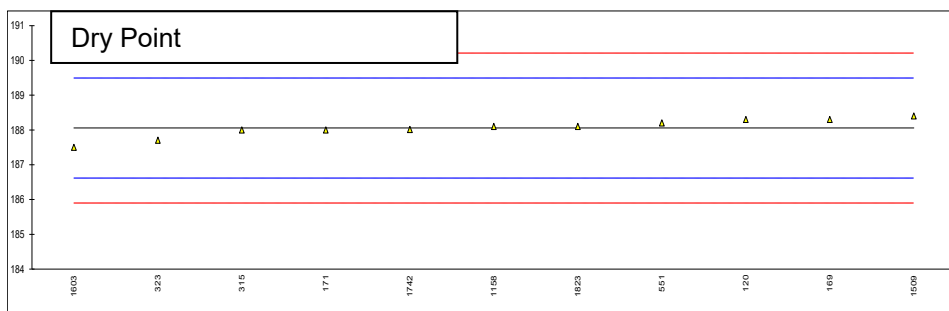
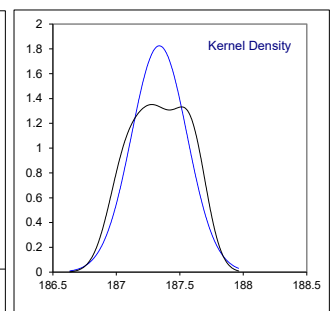
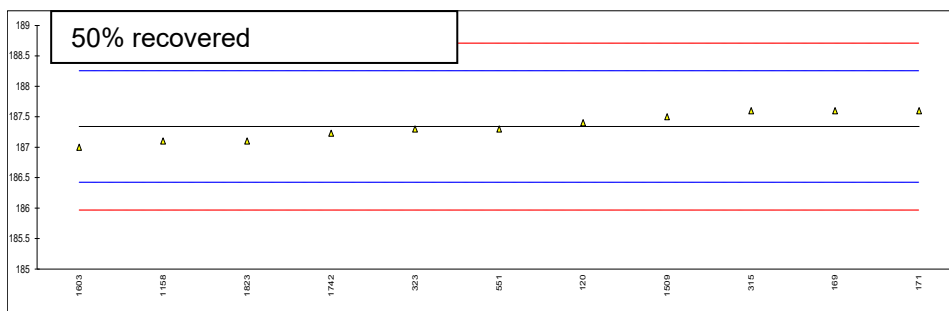
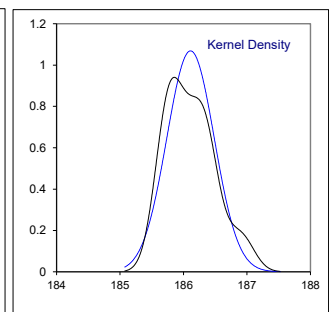
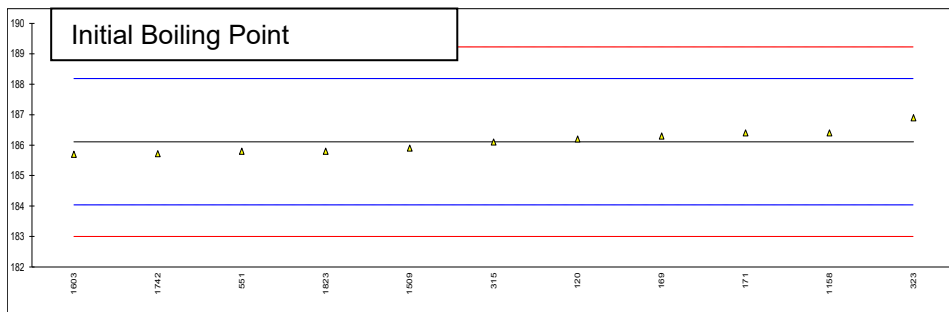
Determination of Density at 20°C on sample #21195; results in kg/L

lab	method	value	mark	z(targ)	remarks
120	D4052	1.03623		-0.10	
169	D4052	1.0363		0.29	
171	D4052	1.0363		0.29	
173	D4052	1.0362		-0.27	
174	D4052	1.03621		-0.21	
315	D4052	1.0362		-0.27	
323	D4052	1.0362		-0.27	
334	ISO12185	1.0362		-0.27	
343	D4052	1.036		-1.39	
347	D4052	1.0362		-0.27	
444	D4052	1.0363		0.29	
446	D4052	1.0359		-1.95	
541	D4052	1.0361		-0.83	
551	D4052	1.0363		0.29	
663	D4052	1.03626		0.07	
823	D4052	1.0362		-0.27	
902	D4052	1.0363		0.29	
1016	D4052	1.0362		-0.27	
1158		----		----	
1509	D4052	1.03660		1.97	
1603	In house	1.03635		0.57	
1742	ISO12185	1.0363		0.29	
1823	D4052	1.0366		1.97	
normality		not OK			
n		22			
outliers		0			
mean (n)		1.036248			
st.dev. (n)		0.0001538			
R(calc.)		0.000431			
st.dev.(ISO12185:96)					
)		0.0001786			
R(ISO12185:96)		0.0005			



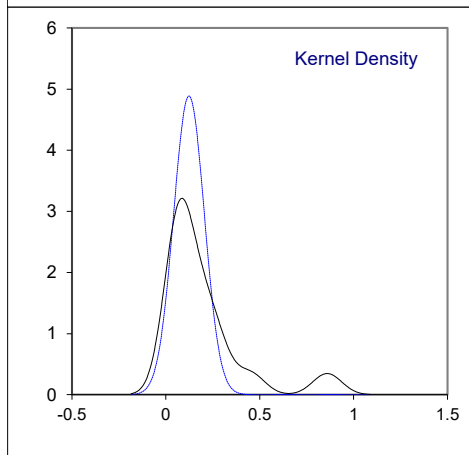
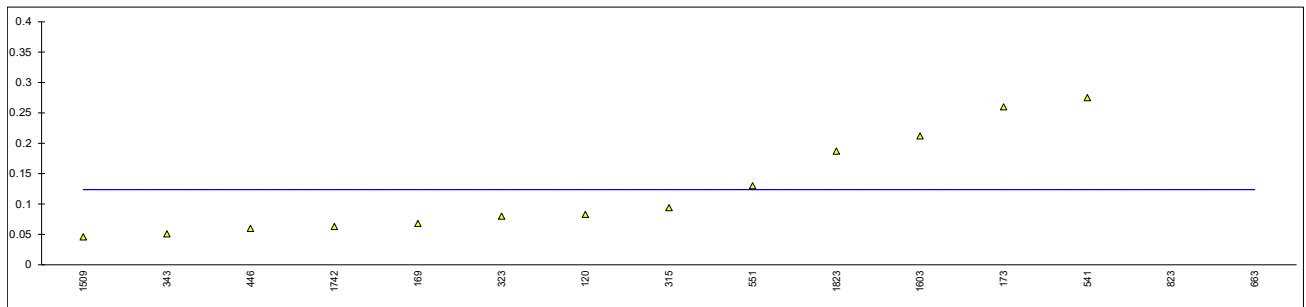
Determination of Distillation: IBP, 50% recovered, Dry Point on sample #21195; results in °C

lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)
120	D1078-automated	186.2		0.09	187.4		0.13	188.3		0.34
169	D1078-automated	186.3		0.18	187.6		0.57	188.3		0.34
171	D1078-automated	186.4		0.28	187.6		0.57	188		-0.08
173		----		----	----		----	----		----
174		----		----	----		----	----		----
315	D1078-automated	186.1		-0.01	187.6		0.57	188.0		-0.08
323	D1078	186.9		0.76	187.3		-0.09	187.7		-0.49
334		----		----	----		----	----		----
343		----		----	----		----	----		----
347		----		----	----		----	----		----
444		----		----	----		----	----		----
446		----		----	----		----	----		----
541		----		----	----		----	----		----
551	D1078-automated	185.8		-0.30	187.3		-0.09	188.2		0.20
663		----		----	----		----	----		----
823		----		----	----		----	----		----
902		----		----	----		----	----		----
1016		----		----	----		----	----		----
1158	D1078-manual	186.4		0.28	187.1		-0.52	188.1		0.06
1509	D1078-automated	185.9		-0.20	187.5		0.35	188.4		0.48
1603	In house	185.7		-0.40	187.0		-0.74	187.5		-0.77
1742	D1078-automated	185.72		-0.38	187.23		-0.24	188.01		-0.06
1823	D1078	185.8		-0.30	187.1		-0.52	188.1		0.06
normality		OK			OK			OK		
n		11			11			11		
outliers		0			0			0		
mean (n)		186.11			187.34			188.06		
st.dev. (n)		0.373			0.219			0.266		
R(calc.)		1.05			0.61			0.74		
st.dev.(D1078-A:11)		1.037			0.457			0.719		
R(D1078-A:11)		2.90			1.28			2.01		
Compare										
R(D1078-M:11)		1.99			1.21			2.45		



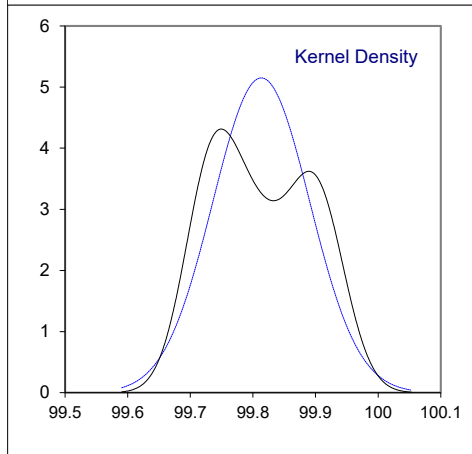
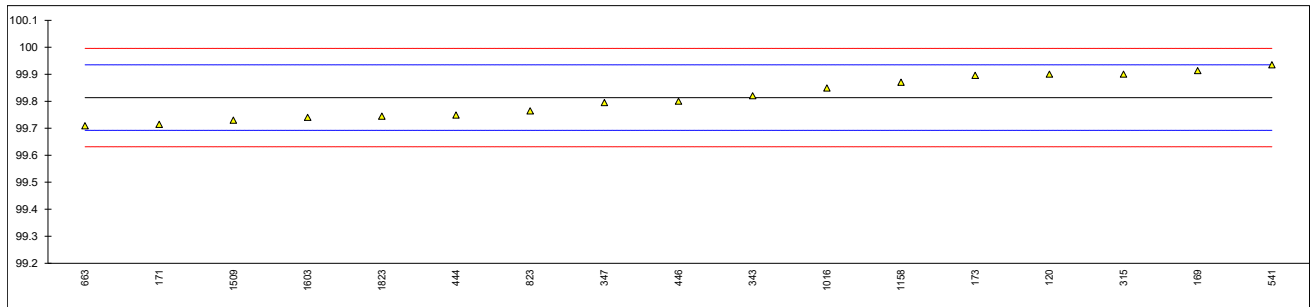
Determination of Iron as Fe on sample #21195; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	E394	0.083		----	
169	E1615	0.068		----	
171		----		----	
173	E394	0.26	C	----	First reported 0.32
174		----		----	
315	E1615	0.094		----	
323	E1615	0.080		----	
334		----		----	
343	E1615	0.051		----	
347		----		----	
444		----		----	
446	INH-2290	0.06		----	
541	E394	0.275		----	
551	E394	0.13		----	
663	E394	0.86	C,G(0.01)	----	First reported 0.86
823	E394	0.45	C,G(0.05)	----	First reported 0.41
902		----		----	
1016	NEN6966	<0.20	C	----	First reported 0.655
1158		----		----	
1509	E394	0.046		----	
1603	In house	0.212	C	----	First reported 0.731
1742	In house	0.063		----	
1823	D1615	0.187		----	
normality		OK			
n		13			
outliers		2			
mean (n)		0.1238			
st.dev. (n)		0.08161			
R(calc.)		0.2285			
		(0.02385			
st.dev.(E394:15))			
R(E394:15)		(0.0668)			



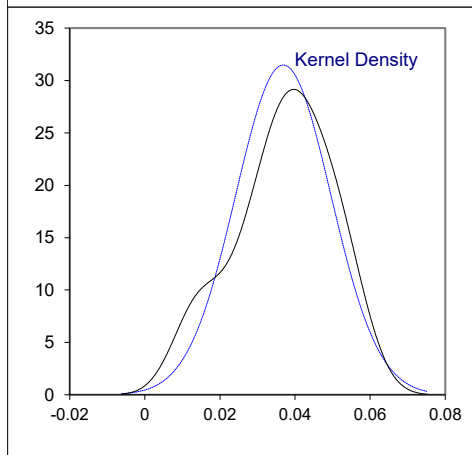
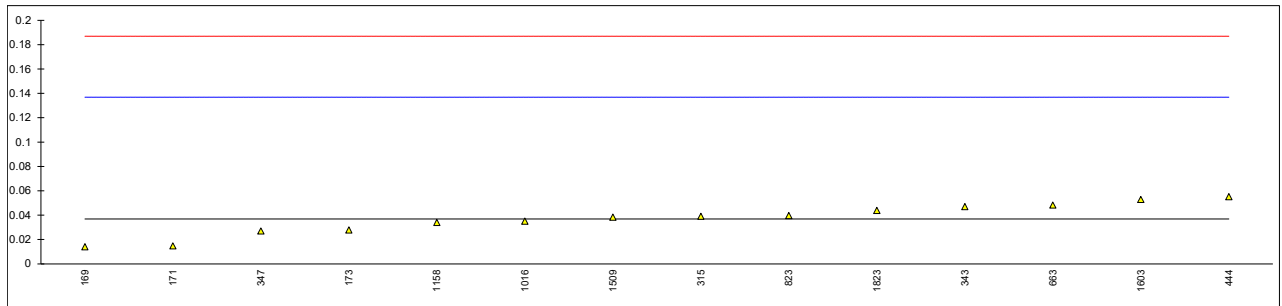
Determination of Purity by GC as received on sample #21195; results in %M/M

lab	method	value	mark	z(targ)	remarks
120	E2409	99.90		1.43	
169	E2409	99.9133		1.64	
171	E2409	99.7146		-1.63	
173	INH-0540	99.8957		1.35	
174		----		----	
315	INH-103	99.90		1.43	
323		----		----	
334		----		----	
343	INH-1321	99.82		0.11	
347	INH-177	99.795		-0.30	
444	INH-100687	99.749		-1.06	
446	INH-2290	99.80		-0.22	
541	INH-100687	99.935		2.00	
551		----		----	
663	INH-100687	99.709		-1.72	
823	E2409	99.764		-0.81	
902		----		----	
1016	E202	99.849		0.59	
1158	In house	99.87		0.93	
1509	E202	99.730		-1.37	
1603	In house	99.74		-1.21	
1742		----	W	----	Test result withdrawn, reported 99.3
1823	E202	99.7443		-1.14	
normality		OK			
n		17			
outliers		0			
mean (n)		99.8135			
st.dev. (n)		0.07743			
R(calc.)		0.2168			
st.dev.(E202:18)		0.06071			
R(E202:18)		0.17			



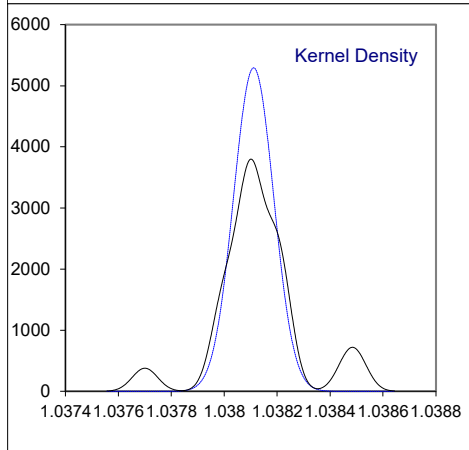
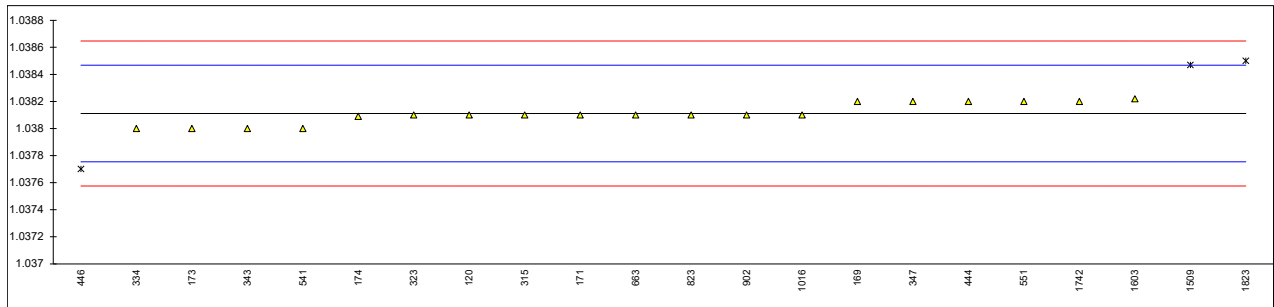
Determination of Dipropylene glycol on sample #21195; results in %M/M

lab	method	value	mark	z(targ)	remarks
120		----		----	
169	E2409	0.01396		-0.46	
171	E2409	0.0147		-0.44	
173	INH-0540	0.0278		-0.18	
174		----		----	
315	INH-103	0.039		0.04	
323		----		----	
334		----		----	
343	INH-1321	0.047		0.20	
347	INH-177	0.027		-0.20	
444	INH-100687	0.0551		0.36	
446		----		----	
541	INH-100687	<0.0023		----	
551		----		----	
663		0.04822		0.23	
823	E2409	0.0396		0.05	
902		----		----	
1016	E202	0.035		-0.04	
1158	In house	0.034		-0.06	
1509	E202	0.0383		0.03	
1603	In house	0.0528		0.32	
1742		----	W	----	Test result withdrawn, reported 0.00049
1823	E202	0.0439		0.14	
normality		OK			
n		14			
outliers		0			
mean (n)		0.03688			
st.dev. (n)		0.012676			
R(calc.)		0.035492			
st.dev.(E202:18)		0.0500			
R(E202:18)		0.14			



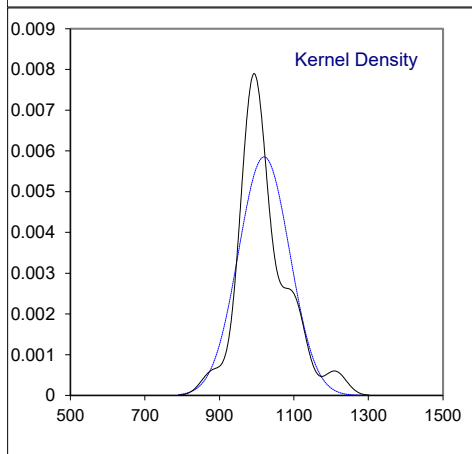
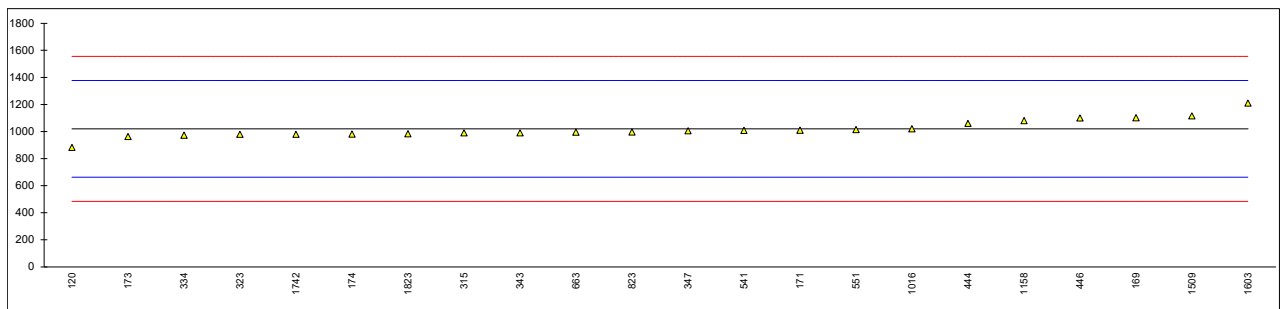
Determination of Specific Gravity at 20/20°C on sample #21195;

lab	method	value	mark	z(targ)	remarks
120	D4052	1.03810		-0.06	
169	D4052	1.0382		0.50	
171	D4052	1.0381		-0.06	
173	D4052	1.0380		-0.62	
174	D4052	1.03809		-0.12	
315	D4052	1.0381		-0.06	
323	D4052	1.0381		-0.06	
334	D4052	1.038		-0.62	
343	D4052	1.038		-0.62	
347	D4052	1.0382		0.50	
444	D4052	1.0382		0.50	
446	D4052	1.0377	R(0.01)	-2.30	
541	D4052	1.0380		-0.62	
551	D4052	1.0382		0.50	
663	D4052	1.0381		-0.06	
823	ISO12185	1.0381		-0.06	
902	D4052	1.0381		-0.06	
1016	D4052	1.0381		-0.06	
1158		----			
1509	D4052	1.03847	R(0.01)	2.01	
1603	In house	1.03822		0.61	
1742	D4052	1.0382		0.50	
1823	D4052	1.0385	R(0.01)	2.18	
normality		OK			
n		19			
outliers		3			
mean (n)		1.03811			
st.dev. (n)		0.000075			
R(calc.)		0.00021			
st.dev.(E202:18)		0.000179			
R(E202:18)		0.0005			



Determination of Water on sample #21195; results in mg/kg

lab	method	value	mark	z(targ)	remarks
120	E1064	882.6		-0.77	
169	E1064	1101.64		0.46	
171	E1064	1010		-0.06	
173	E203	963		-0.32	
174	E1064	981		-0.22	
315	E1064	990		-0.17	
323	E1064	979		-0.23	
334	E1064	973		-0.26	
343	E1064	990		-0.17	
347	INH-176	1005		-0.08	
444	E203	1060		0.22	
446	E203	1100		0.45	
541	E1064	1008.5		-0.06	
551	E1064	1014.4		-0.03	
663	E1064	995.05		-0.14	
823	E1064	997		-0.13	
902		----		----	
1016	D1364	1020		0.00	
1158	E203	1080		0.34	
1509	E203	1115.5	C	0.54	First reported 0.1116 mg/kg
1603	In house	1210		1.06	
1742	E1064	979		-0.23	
1823	E1064	983.00		-0.21	
	normality	not OK			
	n	22			
	outliers	0			
	mean (n)	1019.90			
	st.dev. (n)	68.143			
	R(calc.)	190.80			
	st.dev.(E202:05)	178.571			
	R(E202:05)	500			
Compare	R(E1064:16)	174.40			



APPENDIX 2

Number of participants per country

1 lab in	ARGENTINA
1 lab in	BELGIUM
1 lab in	BRAZIL
1 lab in	CHINA, People's Republic
1 lab in	FINLAND
1 lab in	FRANCE
1 lab in	GERMANY
1 lab in	KOREA, Republic of
2 labs in	NETHERLANDS
1 lab in	ROMANIA
1 lab in	SINGAPORE
2 labs 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
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
- 5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 8 J.N. Miller, Analyst, 118, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
- 10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
- 11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)