Results of Proficiency Test
Acetone
September 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 1999, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Acetone every two year. During the annual proficiency testing program of 2019/2020, it was decided to continue the proficiency tests for the analysis of Acetone in accordance with the latest applicable version of the specification ASTM D329.
In this interlaboratory study 27 laboratories in 15 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the 2019 proficiency test for Acetone 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 a one bottle of 1 liter filled with Acetone, labelled #19180.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. 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 Organization, 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

The necessary Acetone batch, approximately 50 liter, was obtained from a local supplier. After homogenization 50 amber glass bottles of 1L were filled and labelled #19180. The homogeneity of the subsamples was checked by determination of Water in accordance with ASTM E203 and Diacetone alcohol (DAA) in accordance with an in-house method on 4 stratified randomly selected samples.

	Water in mg/kg	Diacetone Alcohol in mg/kg
sample #19180-1	2400	29
sample #19180-2	2430	29
sample #19180-3	2470	29
sample #19180-4	2460	30

Table 1: homogeneity test results of subsamples #19180

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

	Water in mg/kg	Diacetone Alcohol in mg/kg
r (observed)	89	1.4
reference method	ASTM E203:16	Horwitz
0.3 x R (reference method)	234	2.4

Table 2: evaluation of homogeneity of subsamples #19180

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

To each of the participating laboratories one liter glass bottle, labelled #19180, was sent on September 4, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were requested to determine on sample #19180: Acidity as Acetic Acid, Aldehydes, Appearance, Chloride Inorganic as Cl, Color Pt/Co, Density at 20°C, Specific Gravity 20/20°C, Distillation (IBP, MBP, DP and distillation range), Miscibility with water, Nonvolatile Matter, Permanganate Time Test at 25°C, Purity on dry basis, Diacetone alcohol, Mesityloxide, Methanol, Refractive Index at 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 appropriate 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 reanalyzes). 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 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.

According to ISO5725 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. 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 them 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 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 or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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 (e.g. Horwitz). 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:

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

The $z_{(target)}$ 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 samples to Brazil, China and Thailand due to problems with custom clearance. Three participants did not report any test result and five other participants did report after the final reporting date. In total 256 numerical results were reported. Observed was 1 outlying test result, which is 0.4%. 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, 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 listed in appendix 3.

Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For the tests, that have no available precision data, the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D329) and an added designation for the year that the method was adopted or revised (e.g. D329:07). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D329:07(2013)). In the results tables of appendix 1 only the method number and year of adoption or revision e.g. D329:07 will be used.

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

Aldehydes: Three participants reported the test as pass, in accordance ASTM D329:07(2013), which describes a pass/fail test. Two other participants reported a numerical test result.

Appearance: This determination was not problematic. All laboratories agreed about the appearance, which is bright and clear (or Pass).

Chloride, Inorganic as Cl: The Chloride content was near or below the detection limit. Therefore, no z-scores were calculated.

Color Pt/Co: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1209:05(2019).

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

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

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

Water Miscibility: This determination was not problematic. All laboratories reported this test pass.

Nonvolatile Matter: The NVM content was near or below the detection limit. Therefore, no z-scores were calculated.

Permanganate Time Test at 25°C: This determination may not problematic. Almost all participants agreed on a result above 30 minutes. Therefore, no z-scores were calculated.

Purity on Dry Basis: In the determination of Purity no statistical outliers were observed. The calculated reproducibility is slightly higher than the calculated reproducibility of the 2017 PT iis17C10 (0.018 %MM vs 0.016 %M/M).

Diacetone Alcohol: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the estimated reproducibility using the Horwitz equation.

Mesityloxide: The Mesityloxide content was near or below the detection limit. Therefore, no z-scores were calculated.

Methanol: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility using the Horwitz equation.

Refractive index: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D1218:12(2016).

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is good in agreement with the requirements of ASTM E203:16.

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 laboratories that participated. The number of significant results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibilities derived from literature reference test methods (in casu ASTM and ISO test methods) or the estimated target reproducibility calculated by using the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acidity as Acetic Acid	mg/kg	22	12.6	9.8	14
Aldehydes		3	Pass	n.a.	n.a.
Appearance		22	Pass	n.a.	n.a.
Chloride as Cl	mg/kg	7	<1	n.a.	n.a.
Color Pt/Co		19	3.0	3.3	7
Density at 20°C	kg/L	21	0.7906	0.0002	0.0005
Specific Gravity 20/20°C		18	0.7920	0.0002	0.0005
Initial Boiling Point	°C	19	55.8	0.4	0.9
Mid Boiling Point	°C	19	56.1	0.3	0.4
Dry Point	°C	19	56.3	0.4	0.6

Parameter	unit	n	average	2.8 * sd	R (lit)
Miscibility with water		18	Pass	n.a.	n.a.
Nonvolatile Matter	mg/100 mL	17	<1	n.a.	n.a.
Permanganate Time Test at 25°C	minutes	17	>30	n.a.	n.a.
Purity on Dry Basis	%M/M	21	99.977	0.018	n.a.
Diacetone Alcohol	mg/kg	15	32.2	14.3	8.5
Mesityloxide	mg/kg	13	<10	n.a.	n.a.
Methanol	mg/kg	19	172	66	36
Refractive Index at 20°C		14	1.3588	0.0006	0.0005
Water	mg/kg	21	2470	378	780

Table 3: Reproducibilities of tests for sample #19180

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2019 WITH PREVIOUS PTS

	September 2019	September 2017	September 2015	September 2013	September 2011
Number of reporting labs	24	21	21	21	18
Number of test results	256	246	250	273	198
Number of statistical outliers	1	17	10	7	3
Percentage outliers	0.4%	6.9%	4.0%	2.6%	1.5%

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.

Determination	September 2019	September 2017	September 2015	September 2013	September 2011
Acidity as Acetic Acid	+	++	++	++	+
Chloride as Cl	n.e.	n.e.	n.e.	n.e.	n.e.
Color Pt/Co	++	++	++	++	++
Density at 20°C	++	++	++	++	++
Specific gravity 20/20°C	++	++	++	+	n.e.
Distillation	+	++	++	++	++
Nonvolatile Matter	n.e.	(--)	(--)	++	++
Permanganate Time Test at 25°C	n.e.	(--)	(--)	(--)	n.e.
Diacetone Alcohol	-	--	-	--	--

Determination	September 2019	September 2017	September 2015	September 2013	September 2011
Mesityloxide	n.e.	--	-	+/-	n.e.
Methanol	--	--	--	-	+
Refractive Index at 20°C	+/-	+	+/-	+	+/-
Water	++	-	-	+/-	--

Table 5: comparison determinations against the respective reference test methods

The following performance categories were used:

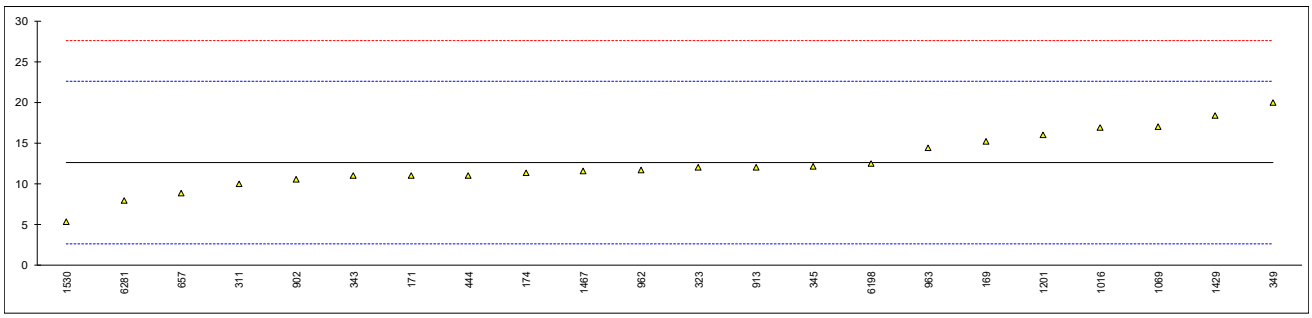
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

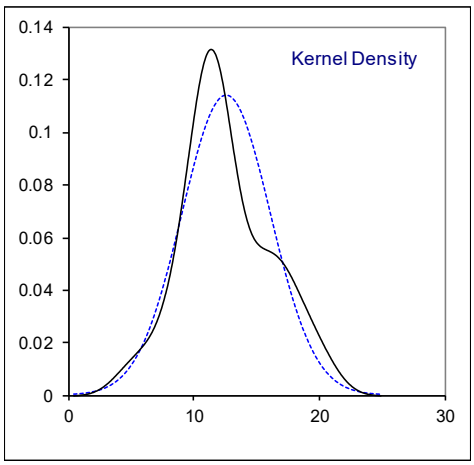
APPENDIX 1

Determination of Acidity as Acetic Acid on sample #19180; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	D1613	15.17		0.52	
171	D1613	11		-0.32	
174	D1613	11.4		-0.24	
311	D1613	10		-0.52	
323	D1613	12		-0.12	
343	D1613	11.0		-0.32	
345	D1613	12.1		-0.10	
349	D1613	20		1.48	
444	D1613	11.05		-0.31	
551		-----		-----	
557		-----		-----	
657	D1613	8.9		-0.74	
886		-----		-----	
902	D1613	10.6		-0.40	
913	D1613	12		-0.12	
962	D1613	11.64		-0.19	
963	D1613	14.4		0.36	
1016	D1613	16.9		0.86	
1069	D1613	17		0.88	
1201	D1613	16		0.68	
1429	D1613	18.4		1.16	
1467	D1613	11.600	C	-0.20	First reported 11.336
1530	D1613	5.36		-1.45	
1669	D1613	<10		-----	
6198	D1613	12.5		-0.02	
6262		-----		-----	
6281	D1613	8		-0.92	

normality	OK
n	22
outliers	0
mean (n)	12.592
st.dev. (n)	3.5020
R(calc.)	9.806
st.dev.(D1613:17)	5
R(D1613:17)	14





Determination of Aldehydes on sample #19180;

lab	method	value	mark	z(targ)	remarks
169	D329	Pass		-----	
171		-----		-----	
174		-----		-----	
311		-----		-----	
323	D329	PASS		-----	
343	D329	PASS		-----	
345		-----		-----	
349		-----		-----	
444		-----		-----	
551		-----		-----	
557		-----		-----	
657		-----		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1016		-----		-----	
1069	D329	29.3		-----	Possibly false positive test result? Test method D329 is a pass/fail test
1201		-----		-----	
1429		-----		-----	
1467		-----		-----	
1530		-----		-----	
1669	GC	35		-----	
6198		-----		-----	
6262		-----		-----	
6281		-----		-----	
	n	3			
	outliers	2			
	mean (n)	Pass			

Determination of Appearance on sample #19180;

lab	method	value	mark	z(targ)	remarks
169	Visual	BC&FSM		-----	
171	E2680	Pass		-----	
174	Visual	Clear and Free		-----	
311	E2680	pass		-----	
323	E2680	Clear Bright Liquid		-----	
343	E2680	PASS		-----	
345	Visual	PASS		-----	
349	E2680	PASS		-----	
444	Visual	Pass		-----	
551		-----		-----	
557		-----		-----	
657	E2680	Pass		-----	
886		-----		-----	
902	E2680	Pass		-----	
913	E2680	Pass		-----	
962		PASS		-----	
963	E2680	Pass		-----	
1016		Pass		-----	
1069	D4176	PASS		-----	
1201	Visual	Clear & Bright		-----	
1429	E2680	Pass		-----	
1467	Visual	Clear colourless Liquid		-----	
1530	Visual	confirm		-----	
1669	D4176	Pass		-----	
6198		-----		-----	
6262		-----		-----	
6281	Visual	Clear&bright		-----	
	n	22			
	mean (n)	Pass			

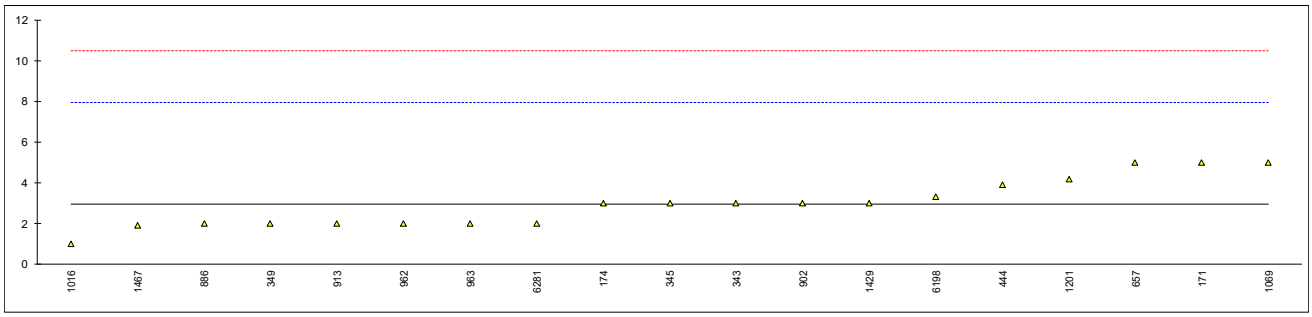
Determination of Chloride, Inorganic as Cl on sample #19180; results in mg/kg

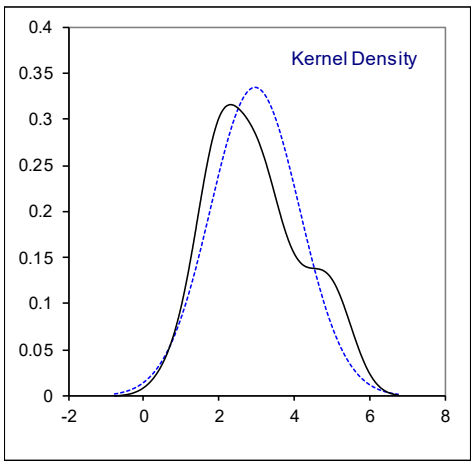
lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171	IMPCA002	0.4		-----	
174		-----		-----	
311	INH-158	<0.2		-----	
323	E2469	<0.25		-----	
343		-----		-----	
345		-----		-----	
349		-----		-----	
444	INH-009	<5		-----	
551		-----		-----	
557		-----		-----	
657		-----		-----	
886		-----		-----	
902	IMPCA002	0.03		-----	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1016		-----		-----	
1069	D7359	0.26		-----	
1201	IMPCA002	0.06		-----	
1429	ISO6227	0.48		-----	
1467		-----		-----	
1530		-----		-----	
1669		-----		-----	
6198		-----		-----	
6262		-----		-----	
6281		-----		-----	
	n	7			
	mean (n)	<1			

Determination of Color as Pt/Co on sample #19180;

lab	method	value	mark	z(targ)	remarks
169	D1209	<5.0		-----	
171	D1209	5		0.81	
174	D5386	3.0		0.01	
311	D1209	<5		-----	
323	D1209	<5		-----	
343	D1209	3		0.01	
345	D1209	3		0.01	
349	D5386	2		-0.39	
444	D5386	3.9		0.37	
551		-----		-----	
557		-----		-----	
657	D1209	5		0.81	
886	D1209	2		-0.39	
902	D5386	3		0.01	
913	D5386	2		-0.39	
962	D1209	2		-0.39	
963	D1209	2		-0.39	
1016	D1209	1		-0.79	
1069	D1209	5		0.81	
1201	D5386	4.2		0.49	
1429	D1209	3		0.01	
1467	D1209	1.9		-0.43	
1530	D1209	< 3		-----	
1669	D1209	<5		-----	
6198	D5386	3.3		0.13	
6262		-----		-----	
6281	D1209	2		-0.39	

normality	OK
n	19
outliers	0
mean (n)	2.96
st.dev. (n)	1.191
R(calc.)	3.33
st.dev.(D1209:05)	2.5
R(D1209:05)	7

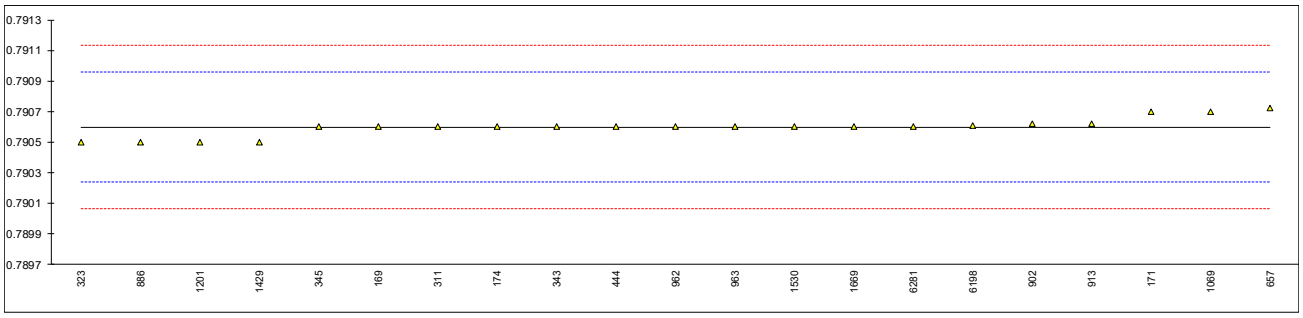


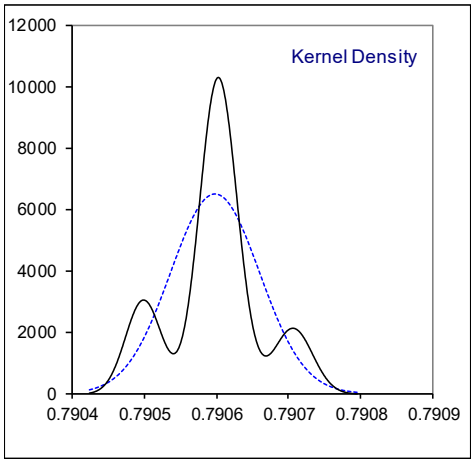


Determination of Density at 20°C on sample #19180; results in kg/L

lab	method	value	mark	z(targ)	remarks
169	D4052	0.7906		0.01	
171	D4052	0.7907		0.57	
174	D4052	0.79060		0.01	
311	D4052	0.7906		0.01	
323	D4052	0.7905		-0.55	
343	D4052	0.7906		0.01	
345	D4052	0.7906		0.01	
349		-----		-----	
444	D4052	0.7906		0.01	
551		-----		-----	
557		-----		-----	
657	D4052	0.79072		0.68	
886	D4052	0.7905		-0.55	
902	ISO12185	0.79062		0.12	
913	D4052	0.79062		0.12	
962	ISO12185	0.7906		0.01	
963	ISO12185	0.7906		0.01	
1016		-----		-----	
1069	D4052	0.7907		0.57	
1201	D4052	0.7905		-0.55	
1429	D4052	0.7905		-0.55	
1467		-----		-----	
1530	D4052	0.7906	C	0.01	First reported 790.6 kg/L
1669	D4052	0.7906	C	0.01	First reported 0.7921
6198	D4052	0.79061		0.06	
6262		-----		-----	
6281	D4052	0.7906		0.01	

normality	OK
n	21
outliers	0
mean (n)	0.79060
st.dev. (n)	0.000061
R(calc.)	0.00017
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

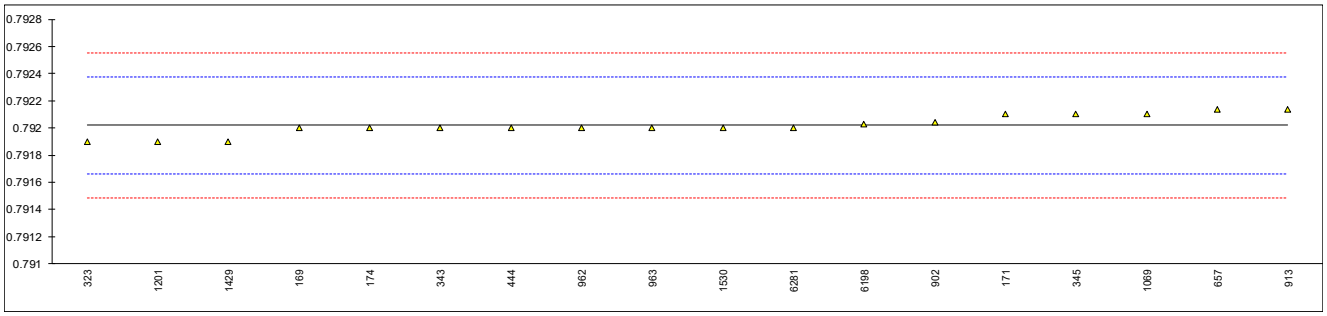


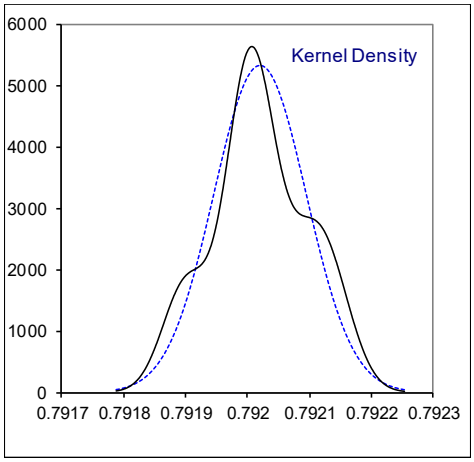


Determination of Specific Gravity 20/20°C on sample #19180;

lab	method	value	mark	z(targ)	remarks
169	D4052	0.7920		-0.11	
171	D4052	0.7921		0.45	
174	D4052	0.7920		-0.11	
311		-----		-----	
323	D4052	0.7919		-0.67	
343	D4052	0.7920		-0.11	
345	D4052	0.7921		0.45	
349		-----		-----	
444	D4052	0.7920		-0.11	
551		-----		-----	
557		-----		-----	
657	D4052	0.79214		0.68	
886		-----		-----	
902	ISO12185	0.79204		0.12	
913	D4052	0.79214		0.68	
962	ISO12185	0.7920		-0.11	
963	ISO12185	0.7920		-0.11	
1016		-----		-----	
1069	D4052	0.7921		0.45	
1201	D4052	0.7919		-0.67	
1429	D4052	0.7919		-0.67	
1467		-----		-----	
1530	D4052	0.7920	C	-0.11	First reported 792.0
1669		-----		-----	
6198	D4052	0.79203		0.06	
6262		-----		-----	
6281	D4052	0.7920		-0.11	

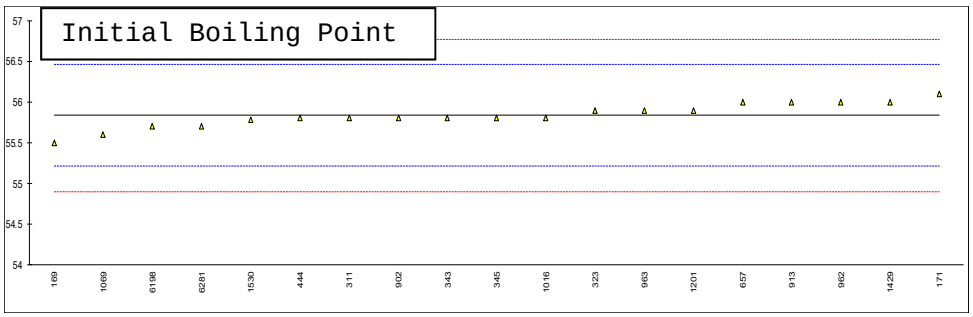
normality	OK
n	18
outliers	0
mean (n)	0.79202
st.dev. (n)	0.000075
R(calc.)	0.00021
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

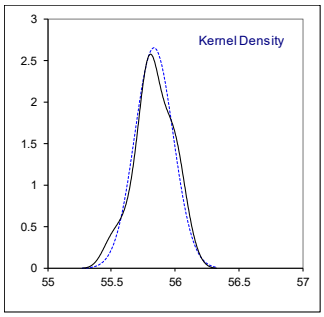


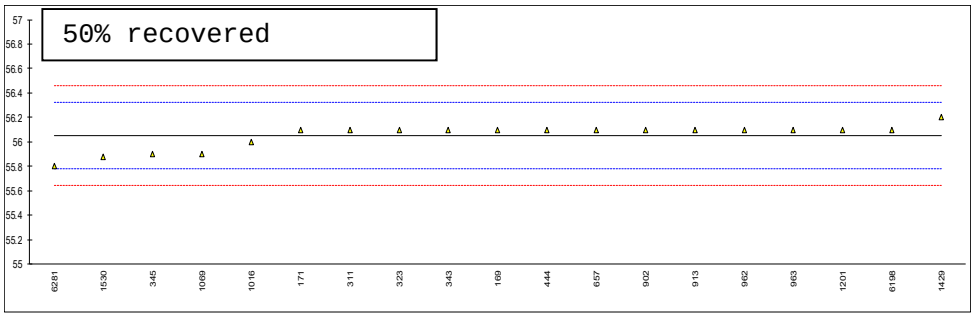


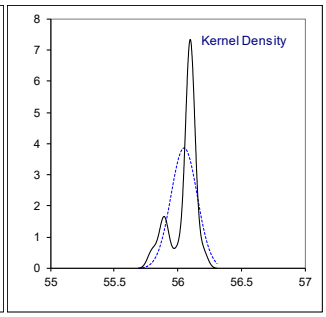
Determination of IBP, 50% recovered and DP at 760 mmHg on sample #19180; results in °C

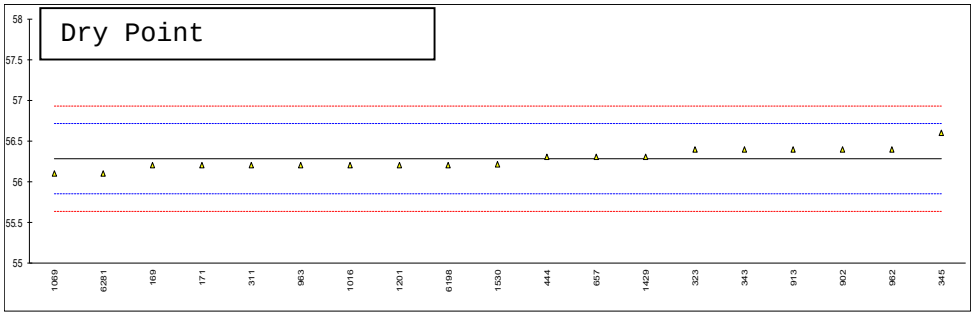
lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	DP	mark	z(targ)	range
169	D1078-automated	55.5		-1.08	56.1		0.35	56.2		-0.37	0.7
171	D1078-automated	56.1		0.85	56.1		0.35	56.2		-0.37	0.2
174		-----		-----	-----		-----	-----		-----	-----
311	D1078-automated	55.8		-0.12	56.1		0.35	56.2		-0.37	0.4
323	D1078-manual	55.9		0.21	56.1		0.35	56.4		0.56	0.5
343	D1078-automated	55.8		-0.12	56.1		0.35	56.4		0.56	0.6
345	D1078-automated	55.8		-0.12	55.9		-1.11	56.6		1.49	0.8
349		-----		-----	-----		-----	-----		-----	-----
444	D1078-automated	55.8		-0.12	56.1		0.35	56.3		0.09	0.5
551		-----		-----	-----		-----	-----		-----	-----
557		-----		-----	-----		-----	-----		-----	-----
657	D1078-automated	56.0		0.53	56.1		0.35	56.3		0.09	0.3
886		-----		-----	-----		-----	-----		-----	-----
902	D1078-automated	55.8		-0.12	56.1		0.35	56.4		0.56	0.6
913	D1078-manual	56.0		0.53	56.1		0.35	56.4		0.56	0.4
962	D1078-automated	56.0		0.53	56.1		0.35	56.4		0.56	0.4
963	D1078-automated	55.9		0.21	56.1		0.35	56.2		-0.37	0.3
1016		55.8		-0.12	56.0		-0.38	56.2		-0.37	-----
1069	D1078-automated	55.6		-0.76	55.9		-1.11	56.1		-0.84	0.5
1201	D1078-automated	55.9		0.21	56.1		0.35	56.2		-0.37	0.3
1429	D1078-automated	56.0		0.53	56.2		1.09	56.3		0.09	0.3
1467		-----		-----	-----		-----	-----		-----	-----
1530	D1078-manual	55.78		-0.18	55.88		-1.25	56.22		-0.28	0.4
1669		-----		-----	-----		-----	-----		-----	-----
6198	D1078	55.7		-0.44	56.1		0.35	56.2		-0.37	0.5
6262		-----		-----	-----		-----	-----		-----	-----
6281	D1078	55.7		-0.44	55.8		-1.84	56.1		-0.84	0.4
	normality	OK			OK			OK			
	n	19			19			19			
	outliers	0			0			0			
	mean (n)	55.84			56.05			56.28			
	st.dev. (n)	0.150			0.104			0.127			
	R(calc.)	0.42			0.29			0.35			
	st.dev.(D1078-A:11)	0.311			0.137			0.215			
	R(D1078-A:11)	0.87			0.38			0.60			
Compare											
	R(D1078-M:11)	0.60			0.36			0.73			

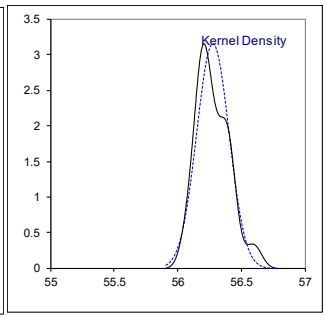

Initial Boiling Point
57
56.5
56
55.5
55
54.5
54
169 1069 6198 6281 1530 444 311 902 343 345 1016 323 963 1201 657 913 962 1429 171


Kernel Density
3
2.5
2
1.5
1
0.5
0
55 55.5 56 56.5 57


50% recovered
57
56.8
56.6
56.4
56.2
56
55.8
55.6
55.4
55.2
55
6281 1530 345 1069 1016 171 311 323 343 169 444 657 902 913 962 963 1201 6198 1429


Kernel Density
8
7
6
5
4
3
2
1
0
55 55.5 56 56.5 57


Dry Point
58
57.5
57
56.5
56
55.5
55
1069 6281 169 171 311 963 1016 1201 6198 1530 444 657 1429 323 343 913 902 962 345


Kernel Density
3.5
3
2.5
2
1.5
1
0.5
0
55 55.5 56 56.5 57

Determination of Miscibility with Water (Hydrocarbons) on sample #19180;

lab	method	value	mark	z(targ)	remarks
169	D1722	Pass		-----	
171	D1722	Pass		-----	
174	D1722	Pass		-----	
311	D1722	pass		-----	
323	D1722	PASS		-----	
343		-----		-----	
345	D1722	PASS		-----	
349		-----		-----	
444	D1722	Pass		-----	
551		-----		-----	
557		-----		-----	
657	D1722	Pass		-----	
886		-----		-----	
902	D1722	Pass		-----	
913	D1722	Pass		-----	
962	D1722	PASS		-----	
963	D1722	Pass		-----	
1016	D1722	Pass		-----	
1069	D1722	passes test		-----	
1201	D1722	Pass		-----	
1429	D1722	Passes test		-----	
1467		-----		-----	
1530	D1722	pass		-----	
1669		-----		-----	
6198	D1722	pass		-----	
6262		-----		-----	
6281		-----		-----	
	n	18			
	mean (n)	Pass			

Determination of Nonvolatile Matter on sample #19180; results in mg/100 mL

lab	method	value	mark	z(targ)	remarks
169	D1353	0.0		-----	
171	D1353	0.2		-----	
174	D1353	0.3		-----	
311	D1353	<1		-----	
323	D1353	<1		-----	
343		-----		-----	
345	D1353	0.5		-----	
349		-----		-----	
444	D1353	0		-----	
551		-----		-----	
557		-----		-----	
657	D1353	0.3		-----	
886		-----		-----	
902	D1353	0.1		-----	
913	D1353	<1		-----	
962	D1353	0.3		-----	
963	D1353	0.2		-----	
1016	D1353	0.5		-----	
1069	D1353	<10		-----	
1201	D1353	0		-----	
1429	D1353	Less than 1		-----	
1467	D1353	0.4	C	-----	First reported 4
1530	D1353	< 3		-----	
1669	D1353	<10		-----	
6198	D1353	<1		-----	
6262		-----		-----	
6281		-----		-----	
	n	17			
	mean (n)	<1			

Determination of Permanganate Time Test at 25°C on sample #19180; results in minutes

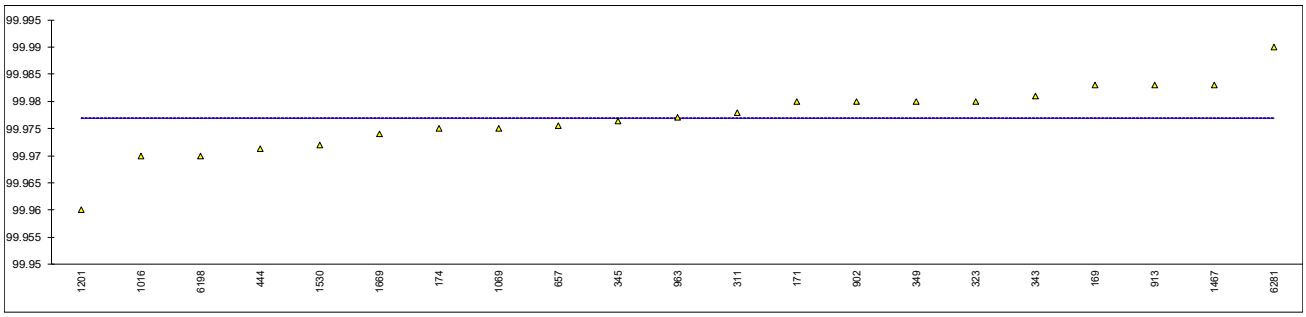
lab	method	value	mark	z(targ)	remarks
169	D1363	>120		-----	
171	D1363	>120		-----	
174	D1363	>120		-----	
311	D1363	>240		-----	
323	D1363	>25		-----	According test method minimum test period is 30 minutes.
343		-----		-----	
345	D1363	270		-----	
349		-----		-----	
444	D1363	>180		-----	
551		-----		-----	
557		-----		-----	
657	D1363	240		-----	
886		-----		-----	
902	D1363	>150		-----	
913	D1363	>30		-----	
962	D1363	>30		-----	
963	D1363	>30		-----	
1016	D1363	>300		-----	
1069	D1363	515		-----	
1201	D1363	>240		-----	
1429		-----		-----	
1467	D1363	360		-----	
1530		-----		-----	
1669	D1363	180		-----	
6198	D1363	>120		-----	
6262		-----		-----	
6281		-----		-----	
	n	17			
	mean (n)	>30			

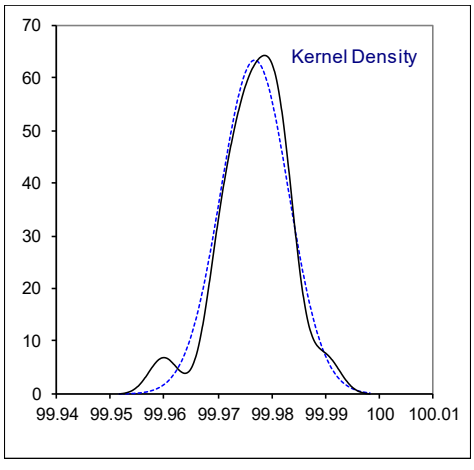
Determination of Purity by GC on Dry Basis on sample #19180, results in %M/M

lab	method	value	mark	z(targ)	remarks
169	INH-ACS	99.983		-----	
171	INH-001	99.98		-----	
174	INH-ACS	99.975		-----	
311	INH-394	99.978		-----	
323	INH-020	99.98		-----	
343	INH-1858	99.981		-----	
345		99.9763		-----	
349	INH-034	99.98		-----	
444	INH-001	99.9713		-----	
551		-----		-----	
557		-----		-----	
657	INH-009Mod.	99.9755		-----	
886		-----		-----	
902	INH-125	99.980		-----	
913	INH-SOP	99.983		-----	
962		-----		-----	
963	INH-012	99.977		-----	
1016	DIN55687	99.97		-----	
1069	In house	99.975		-----	
1201	In house	99.96		-----	
1429		-----		-----	
1467		99.983	C	-----	First reported 99.7439
1530		99.972		-----	
1669	In house	99.974		-----	
6198		99.97		-----	
6262		-----		-----	
6281		99.99		-----	

normality	suspect
n	21
outliers	0
mean (n)	99.97687
st.dev. (n)	0.006301
R(calc.)	0.01764
st.dev.(lit)	unknown
R(lit)	unknown

Compare R(iis17C10) = 0.01610

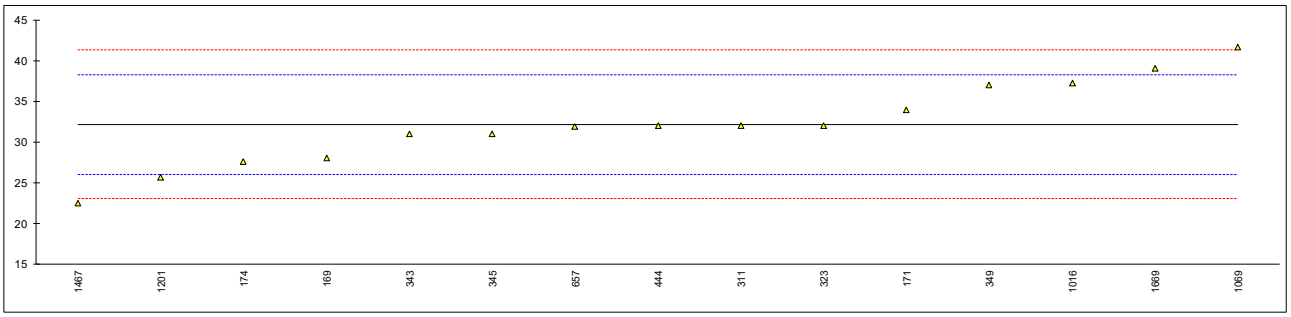


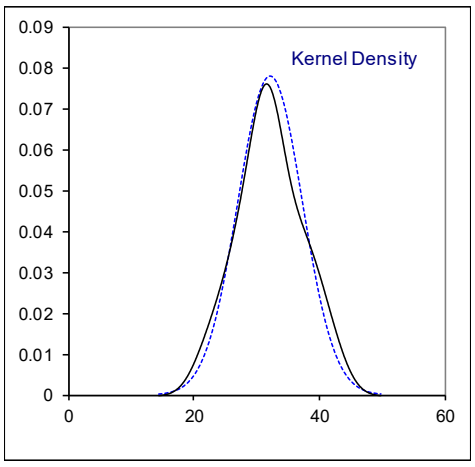


Determination of Diacetone Alcohol on sample #19180, results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	INH-ACS	28.0		-1.37	
171	INH-001	34		0.60	
174	INH-ACS	27.6		-1.50	
311	INH-394	32		-0.06	
323	INH-020	32		-0.06	
343	INH-1862	31		-0.38	
345		31		-0.38	
349	INH-034	37		1.58	
444	INH-001	31.97		-0.07	
551		-----		-----	
557		-----		-----	
657	INH-009Mod.	31.9		-0.09	
886		-----		-----	
902		-----		-----	
913		-----		-----	
962		-----		-----	
963		-----		-----	
1016	DIN55687	37.198		1.65	
1069	In house	41.7		3.12	
1201	In house	25.7		-2.12	
1429		-----		-----	
1467		22.55		-3.15	
1530		-----		-----	
1669	In house	39		2.24	
6198		-----		-----	
6262		-----		-----	
6281		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	32.175
st.dev. (n)	5.1061
R(calc.)	14.297
st.dev.(Horwitz)	3.0530
R(Horwitz)	8.548





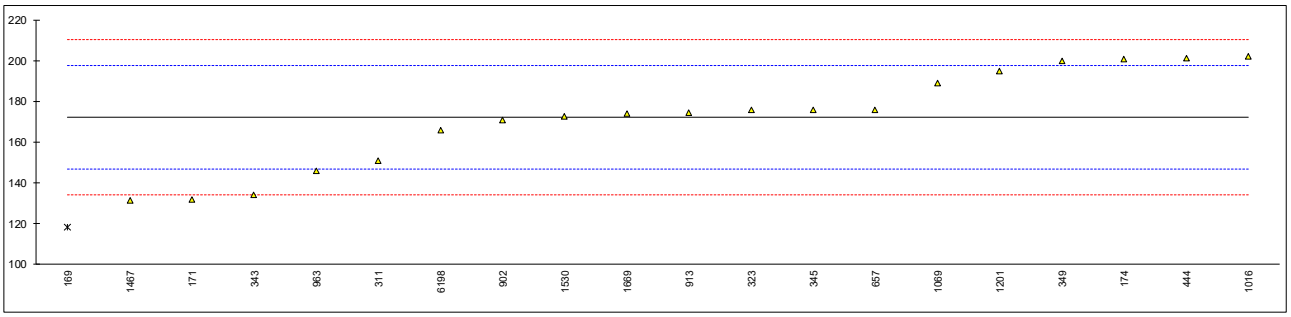
Determination of Mesityloxide on sample #19180, results in mg/kg

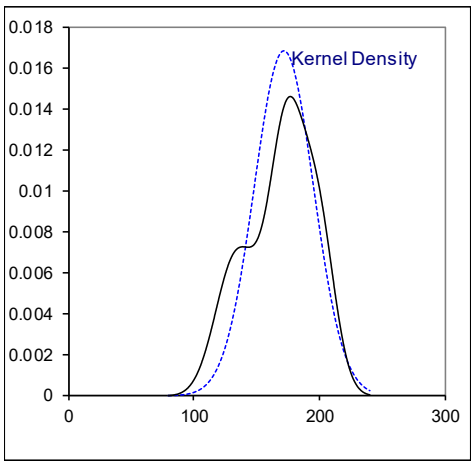
lab	method	value	mark	z(targ)	remarks
169		-----		-----	
171		-----		-----	
174		-----		-----	
311	INH-394	<1		-----	
323	INH-020	<5		-----	
343	INH-1862	<5		-----	
345		<1		-----	
349	INH-034	0		-----	
444	INH-001	0		-----	
551		-----		-----	
557		-----		-----	
657	INH-009Mod.	N.D.		-----	
886		-----		-----	
902		-----		-----	
913		-----		-----	
962		-----		-----	
963	INH-012	<10		-----	
1016	DIN55687	0.765		-----	
1069	In house	0.2		-----	
1201	In house	0		-----	
1429		-----		-----	
1467		1.03		-----	
1530		-----		-----	
1669	In house	<5		-----	
6198		-----		-----	
6262		-----		-----	
6281		-----		-----	
	n	13			
	mean (n)	<10			

Determination of Methanol on sample #19180, results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	INH-ACS	118.1	R(0.05)	-4.25	
171	INH-001	132		-3.15	
174	INH-ACS	200.7		2.26	
311	INH-394	151		-1.66	
323	INH-020	176		0.32	
343	INH-1862	134		-3.00	
345		176		0.32	
349	INH-034	200		2.21	
444	INH-001	201		2.29	
551		-----		-----	
557		-----		-----	
657	INH-009Mod.	176		0.32	
886		-----		-----	
902	INH-125	171		-0.08	
913	INH-SOP	174.5		0.20	
962		-----		-----	
963	INH-012	146		-2.05	
1016	DIN55687	202.110		2.37	
1069	In house	189		1.34	
1201	In house	194.9		1.81	
1429		-----		-----	
1467		131.3025		-3.21	
1530		172.5		0.04	
1669	In house	174		0.16	
6198		166		-0.47	
6262		-----		-----	
6281		-----		-----	

normality	OK
n	19
outliers	1
mean (n)	172.00
st.dev. (n)	23.666
R(calc.)	66.27
st.dev.(Horwitz)	12.681
R(Horwitz)	35.51

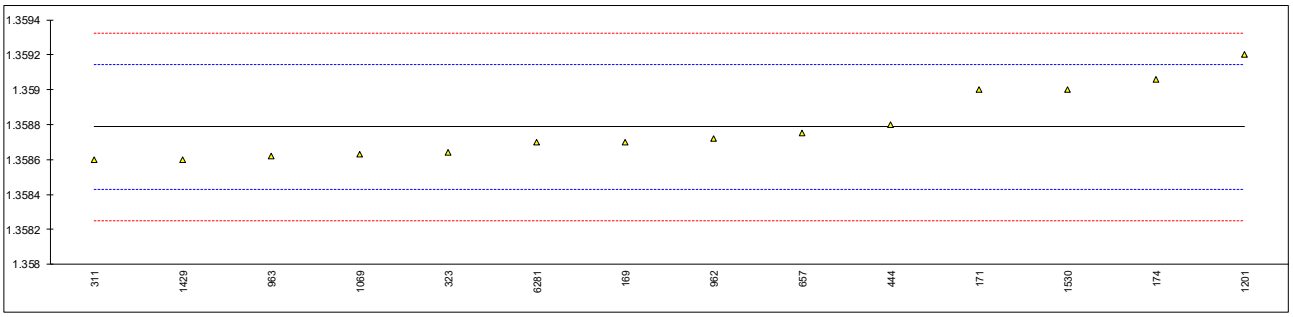


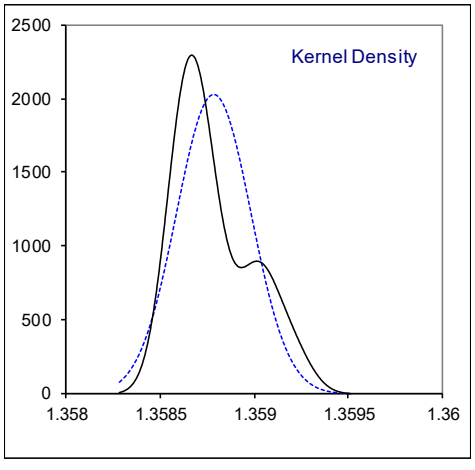


Determination of Refractive Index at 20°C on sample #19180;

lab	method	value	mark	z(targ)	remarks
169	D1218	1.3587		-0.49	
171	D1218	1.3590	C	1.19	First reported 1.3567
174	D1218	1.35906		1.53	
311	D1218	1.3586		-1.05	
323	D1218	1.35864		-0.82	
343		-----		-----	
345		-----		-----	
349		-----		-----	
444	D1218	1.3588		0.07	
551		-----		-----	
557		-----		-----	
657	D1218	1.35875		-0.21	
886		-----		-----	
902		-----		-----	
913		-----		-----	
962	D1218	1.35872		-0.37	
963	D1218	1.35862		-0.93	
1016		-----		-----	
1069	D1218	1.35863		-0.88	
1201	D1218	1.3592	C	2.31	First reported 1.3597
1429	D1218	1.3586		-1.05	
1467		-----		-----	
1530	D1218	1.3590		1.19	
1669		-----		-----	
6198		-----		-----	
6262		-----		-----	
6281	D1218	1.358697		-0.50	

normality	OK
n	14
outliers	0
mean (n)	1.35879
st.dev. (n)	0.000196
R(calc.)	0.00055
st.dev.(D1218:12)	0.000179
R(D1218:12)	0.0005

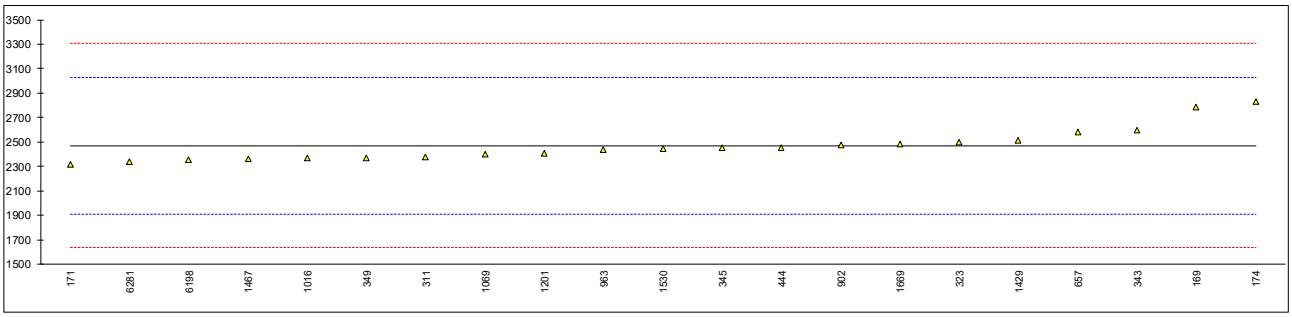


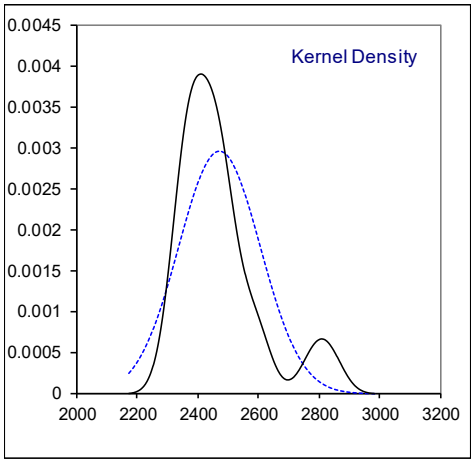


Determination of Water on sample #19180; results in mg/kg

lab	method	value	mark	z(targ)	remarks
169	E203	2783		1.12	
171	E203	2319		-0.54	
174	E203	2834		1.31	
311	E203	2380		-0.32	
323	E203	2500	C	0.11	First reported 0.250 mg/kg
343	D1364	2595		0.45	
345	D1364	2450		-0.07	
349	D1364	2372		-0.35	
444	E203	2450		-0.07	
551		-----		-----	
557		-----		-----	
657	E203	2585		0.41	
886		-----		-----	
902	E203	2476		0.02	
913		-----		-----	
962		-----		-----	
963	E203	2440		-0.11	
1016	D1364	2370		-0.36	
1069	D1364	2400		-0.25	
1201	E203	2408		-0.22	
1429	E203	2514		0.16	
1467	E203	2363		-0.38	
1530	EN15692	2449		-0.08	
1669	D1364	2480		0.04	
6198	D1364	2358		-0.40	
6262		-----		-----	
6281	D1364	2342.2	C	-0.46	First reported 0.23422 mg/kg

normality	not OK
n	21
outliers	0
mean (n)	2469.91
st.dev. (n)	134.908
R(calc.)	377.74
st.dev.(E203:16)	278.571
R(E203:16)	780





APPENDIX 2

Number of participants per country

2 labs in BELGIUM
2 labs in BRAZIL
1 lab in CHINA P.R. of
1 lab in FINLAND
1 lab in GERMANY
1 lab in INDIA
3 labs in NETHERLANDS
3 labs in SAUDI ARABIA
1 lab in SINGAPORE
4 labs in SPAIN
1 lab in TAIWAN R.O.C.
1 lab in THAILAND
1 lab in TURKEY
2 labs in UNITED KINGDOM
3 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	= possibly an error in calculations
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
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ASTM E178:02
3 ASTM E1301:03
4 ISO 5725:86
5 ISO 5725, parts 1-6, 1994
6 ISO 13528:15
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 IMPCA Methanol Reference Specifications, IMPCA, Brussels, December 2015.
14 Analytical Methods Committee Technical brief, No 4, January 2001
15 P.J. Lothian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
16 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
17 Horwitz, W and Albert, R, J. AOAC Int, 79, 3, 589, (1996)