Results of Proficiency Test Aviation Gasoline March 2020

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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1 Introduction

Since 2011 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Aviation Gasoline grade 100LL based on the scope of the latest specification of ASTM D910 and DefStan 91-090 once every two years. During the annual proficiency testing program of 2019/2020, it was decided to continue the round robin for the analysis of Aviation Gasoline 100LL.

In this interlaboratory study 13 laboratories from 13 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of this proficiency test for Aviation Gasoline 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 analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two identical samples of one liter each of Aviation Gasoline labelled #20025 to the participants. 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 a 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.

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2.4 SAMPLES

A batch of approximately 80 liters of Aviation Gasoline grade 100LL was obtained from a local supplier. After homogenization 58 amber glass bottles of 1 liter were filled and labelled #20025. The homogeneity of the subsamples was checked by determination of Density at 15°C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/m³
sample #20025-1	717.02
sample #20025-2	717.02
sample #20025-3	717.05
sample #20025-4	717.05
sample #20025-5	717.04
sample #20025-6	717.07
sample #20025-7	717.08
sample #20025-8	717.07

Table 1: homogeneity test results of subsamples #20025

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15°C in kg/m³
r(observed)	0.063
reference test method	ISO12185:96
0.3 * R(reference test method)	0.45

Table 2: evaluation of repeatability of subsamples #20025

The calculated repeatability was in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories two bottles of 1L of Aviation Gasoline, both labelled #20025, were sent on February 26, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Aviation Gasoline 100LL packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

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2.6 ANALYZES

The participants were requested to determine on the Aviation Gasoline sample #20025: Aromatics by FIA (%M/M and %V/V), Mono, Di and Total Aromatics by HPLC, Color, Copper Corrosion 2hrs at 100°C, Density at 15°C, Distillation at 760 mmHg (IBP, Temperature at 10, 40, 50, 90% evaporated, and FBP), Existent Gum (solvent washed), Freezing Point, Heat of Combustion (Net), Lead as Pb, Lead as Tetra Ethyl Lead, Lead Precipitate, Potential Gum, Sulfur, Water reaction (volume change), MON and Lean mixture Aviation rating.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' 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 reanalysis). 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.

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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.

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The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used. When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

During the execution of this proficiency test some problems occurred. Due to COVID-19 outbreak 2 participants were not able to report because of restricted dispatch and 1 participant, who did receive the samples, did not report any test results. Not all laboratories were able to perform all analyzes requested. Finally, 10 participants reported 149 numerical test results. Observed were 10 outlying test results, which is 6.7%. 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 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 methods are also in the tables together with the reported test results. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports, ASTM methods are referred to with a number e.g. D873 and an added designation for the year that the method was adopted or revised e.g. D873:12. If applicable, a designation in parentheses is added to designate the year of reapproval e.g. D873:12(2018). In the results tables of appendix 1 only the method number and year of adoption or revision e.g. D873:12 will be used.

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Sample #20025

Mono Aromatics (MAH) by HPLC: 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 D6379:11(2019).

<u>Di Aromatics (DAH) by HPLC:</u> This determination was not problematic. Most of the reporting participants agreed on a level <0.10 %M/M.

<u>Total Aromatics by HPLC:</u> This determination was not problematic. One statistical outlier was observed over two parameters (%M/M and %V/V). The calculated reproducibilities after rejection of the statistical outlier are in agreement with the requirements of ASTM D6379:11(2019).

Aromatics by FIA: This determination was not problematic. Results were reported in %V/V only. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1319:19.

<u>Color:</u> This determination was not problematic. Most of the reporting participants reported the color as "Blue".

<u>Copper Corrosion:</u> This determination was not problematic. All of the reporting participants agreed on a result of 1 (1a/1b).

<u>Density at 15°C:</u> 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 ISO12185:96.

<u>Distillation:</u> This determination was not problematic. Six statistical outliers were observed and six other test results were excluded over six parameters. All calculated reproducibilities after rejection of the suspect data are in agreement with the requirements of ASTM D86:19 automatic mode.

Existent Gum: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D381:19.

Freezing Point: This determination was not problematic. Almost all reporting participants agreed on a result below -58°C. The value of -58°C is the upper limit for freezing point according to the product specification ASTM D910:20 and DefStan 91-090:2015.

Heat of Combustion: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D3338:09(2014e2).

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<u>Lead as Pb:</u> This determination was problematic. No statistical outliers were observed.

However, the calculated reproducibility is not in agreement with the

requirements of ASTM D3341:16.

<u>Lead as TEL:</u> This determination may not be problematic. Only three laboratories

reported a test result. No statistical outliers were observed. The calculated

TEL values by iis correspond to the reported test results from the

participants. The estimated reproducibility of the calculated results is in full

agreement with the requirements of ASTM D3341:16.

<u>Lead precipitate:</u> This determination was not problematic. All of the reporting participants

agreed on a value less than 1 mg/100mL.

Potential Gum: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in agreement with the

requirements of ASTM D873:12(2018).

<u>Sulfur:</u> This determination was not problematic. All reporting participants agreed on

a result below of near the minimal application level of 3 mg/kg of ASTM

D2622:16.

Water reaction, volume change: This determination may not be problematic. Most of the

reporting participants agreed on a value less than 0.5 mL.

 $\underline{\mathsf{MON}}$ and Lean mixture Aviation rating: This determination was not problematic. No statistical

outliers were observed. The calculated reproducibility is in full agreement

with the requirements of ASTM D2700:19.

Only three participants read the Lean mixture Aviation rating from table 8 in ASTM D2700:19, based on the MON test result. Two readouts were correct and one participant had possibly applied linear interpolation from table 8,

see appendix 1.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Mono Aromatics (MAH) by HPLC	%M/M	3	21.61	0.47	2.03
Di Aromatics (DAH) by HPLC	%M/M	3	<0.10	n.e.	n.e.
Total Aromatics by HPLC	%M/M	4	21.72	0.65	2.17
Total Aromatics by HPLC	%V/V	5	17.43	1.44	1.87
Aromatics by FIA	%V/V	6	17.85	2.20	2.97

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Parameter	unit	n	average	2.8 * sd	R(lit)
Color		4	Accept/Blue	n.a.	n.a.
Copper Corrosion 2 hrs at 100°C	rating	10	1 (1a/1b)	n.a.	n.a.
Density at 15°C	kg/m³	9	717.0	0.2	1.5
Initial Boiling Point	°C	8	34.8	3.9	4.7
Temp. at 10% evaporated	°C	8	60.6	3.0	4.5
Temp. at 40% evaporated	°C	8	97.1	1.1	4.0
Temp. at 50% evaporated	°C	8	104.6	0.7	3.4
Temp. at 90% evaporated	°C	8	129.8	1.5	4.7
Final Boiling Point	°C	8	156.1	2.3	7.1
Existent Gum (solvent washed)	mg/100mL	8	0.5	1.0	2.2
Freezing Point	°C	7	<-58	n.e.	n.e.
Heat of Combustion (Net)	MJ/kg	5	43.549	0.023	0.046
Lead as Pb	g Pb/L	5	0.540	0.039	0.028
Lead as Tetra Ethyl Lead	mL TEL/L	3	0.508	0.025	0.027
Lead Precipitate	mg/100mL	7	<1	n.e.	n.e.
Potential Gum	mg/100mL	9	0.93	2.16	3
Sulfur	mg/kg	10	<3	n.e.	n.e.
Water reaction, volume change	mL	7	<0.5	n.a.	n.a.
MON		7	102.3	1.8	2

Table 3: performance evaluation sample #20025

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2020 WITH PREVIOUS PTS

	March 2020	April 2018	April 2016	April 2014	April 2013
Number of reporting laboratories	10	14	20	17	17
Number of test results	149	159	211	193	209
Number of statistical outliers	10	3	3	9	6
Percentage of statistical outliers	6.7%	1.9%	1.4%	4.7%	2.9%

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 in the following table.

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Parameter	March 2020	April 2018	April 2016	April 2014	April 2013
Aromatics by HPLC	++	n.e.		-	
Aromatics by FIA	+	n.e.	n.e.	n.e.	n.e.
Density at 15°C	++	++	++	+	++
Distillation at 760 mmHg	++	++	++	+	+
Existent Gum (solvent washed)	++	++	++	++	+/-
Heat of Combustion (Net)	++				
Lead as Pb	-		-		
Lead as Tetra Ethyl Lead	+/-	n.e.	-		
Potential Gum	+	++	+/-	n.e.	+
Sulfur	n.e.	n.e.	n.e.	n.e.	n.e.
MON and LMA rating 1)	+/-	+/-	-	+	

Table 5: comparison determinations against the reference test methods

In the table above 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

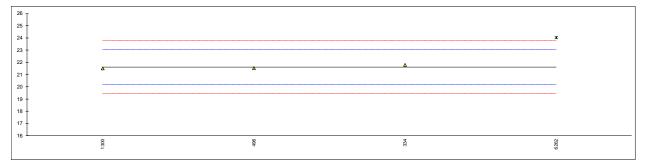
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¹⁾ Lean mixture Aviation (LMA) rating as of 2020

APPENDIX 1

Determination of Mono Aromatics (MAH) by HPLC on sample #20025; results in %M/M

lab	method	value	mark	z(targ)	remarks
62					
150					
334	D6379	21.8		0.26	
496	D6379	21.53		-0.11	
631					
1016					
1299					
1300	D6379	21.495		-0.16	
1521					
1720					
1741					
6041					
6262	EN12916	24.026	D(0.05)	3.34	
	normality	unknown			
	n	3			
	outliers	1			
	mean (n)	21.608			
	st.dev. (n)	0.1669			
	R(calc.)	0.467			
	st.dev.(D6379:11)	0.7239			
	R(D6379:11)	2.027			



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Determination of Di Aromatics (DAH) by HPLC on sample #20025; results in %M/M

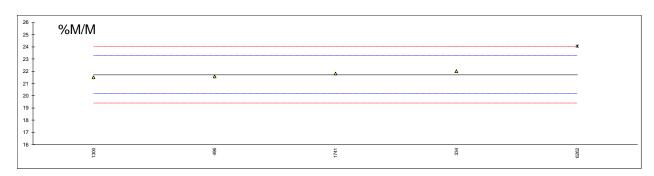
lab	method	value	mark z(targ)	remarks
62				
150				
334	D6379	0.2		
496	D6379	0.04		
631				
1016				
1299				
1300	D6379	<0.1		
1521				
1720				
1741				
6041				
6262	EN12916	0.032		
	n	3		
	mean (n)	<0.10		application range ASTM D6379:11: 0.10 - 6.64 %M/M

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Determination of Total Aromatics by HPLC on sample #20025; results in %M/M and %V/V

lab	method	%M/M	mark	z(targ)	%V/V	mark	z(targ)	
62								
150	D6379				16.6		-1.25	
334	D6379	22.0		0.36	17.8		0.55	
496	D6379	21.57		-0.19	17.63		0.29	
631								
1016								
1299								
1300	D6379	21.495		-0.29	17.29		-0.22	
1521								
1720								
1741	D6379	21.81		0.12	17.85		0.62	
6041								
6262	EN12916	24.058	C,D(0.05)	3.02		W		
	normality n outliers mean (n) st.dev. (n) R(calc.) st.dev.(D6379:11) R(D6379:11)	unknown 4 1 21.719 0.2307 0.646 0.7737 2.166			unknown 5 0 17.434 0.5152 1.443 0.6682 1.871			

Lab 6262 first reported 0 at %M/M and withdraw test result 24.058 at %V/V

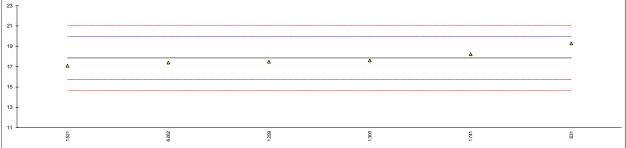




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Determination of Aromatics by FIA on sample #20025; results in %M/M and %V/V

lab	method	%M/M	mark	z(targ)	%V/V	mark	z(targ)	
62								
150								
334								
496								
631	D1319				19.27		1.34	
1016								
1299	D1319				17.5		-0.32	
1300	D1319				17.60		-0.23	
1521	D1319				17.1		-0.70	
1720								
1741	D1319				18.2		0.33	
6041								
6262	D1319				17.4		-0.42	
	normality				unknown			
	n	0			6			
	outliers				0			
	mean (n)				17.845			
	st.dev. (n)				0.7860			
	R(calc.)				2.201			
	st.dev.(D1319:19)				1.0622			
	R(D1319:19)				2.974			



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Determination of Color on sample #20025

lab	method	value	mark	z(targ)	remarks
62					
150	D2392	Acceptable			
334					
496					
631					
1016					
1299					
1300	D2392	Blue			
1521	D2392	blue			also by using method IP569: 2,8 blue
1720					
1741	IP569	Blue			Lovibond units: Blue 2,9 / Yellow 0,9 / Red 0 / Neutral 0,5
6041					
6262					
	n	4			
	mean (n)	Acceptable/Blue			

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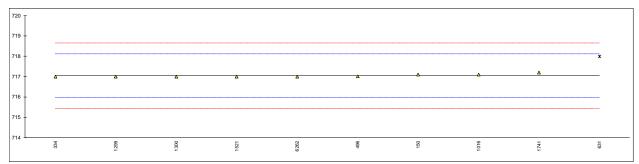
Determination of Copper Corrosion 2hrs at 100°C on sample #20025

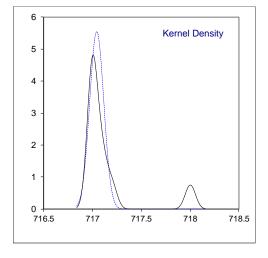
lab	method	value	mark	z(targ)	remarks
62					
150	D130	1a			
334	D130	1A			
496	D130	1a			
631	D130	1a			
1016	D130	1A			
1299	D130	1B			
1300	D130	1			
1521	D130	1			
1720					
1741	ISO2160	1a			
6041					
6262	D130	1A			
	n	10			
	mean (n)	1 (1a/1b)			

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Determination of Density at 15°C on sample #20025; results in kg/m³

lab	method	value	mark	z(targ)	remarks
62	<u> </u>				
150	D4052	717.1		0.10	
334	ISO12185	717.0		-0.09	
496	ISO12185	717.01		-0.07	
631	D4052	718.0	D(0.01)	1.78	
1016	D4052	717.1		0.10	
1299	D4052	717.0		-0.09	
1300	ISO12185	717.0		-0.09	
1521	D4052	717.0		-0.09	
1720					
1741	ISO12185	717.2		0.29	
6041					
6262	D4052	717.0		-0.09	
	normality	not OK			
	n	9			
	outliers	1			
	mean (n)	717.046			
	st.dev. (n)	0.0720			
	R(calc.)	0.201			
	st.dev.(ISO12185:96)	0.5357			
	R(ISO12185:96)	1.5			
	N(13012103.90)	1.5			





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Determination of Distillation at 760 mmHg on sample #20025; results in °C

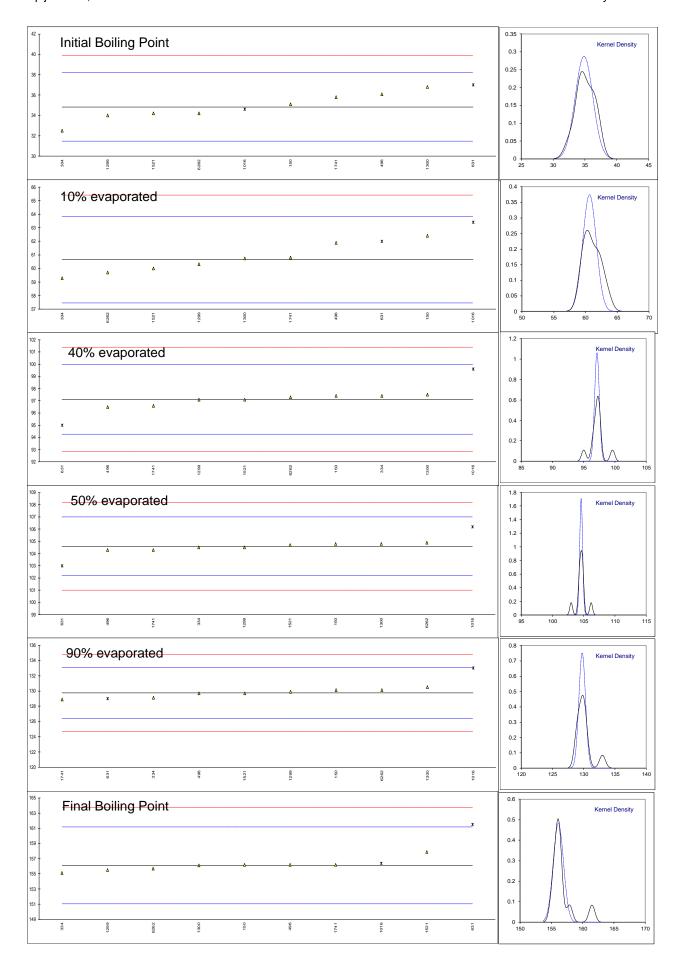
lab	method	IBP	10%	40%	50%	90%	FBP	residue
62								
150	D86-automated	35.1	62.4	97.4	104.8	130.1	156.2	1.0
334	D86-automated	32.5	59.3	97.4	104.5	129.1	155.1	0.9
496	D86-automated	36.1	61.9	96.5	104.3	129.7	156.2	1.0
631	D86-manual	37.0 ex	62.0 ex	95.0 D(0.05)	103.0 D(0.01)	129.0 ex	161.5 (D0.05)	0.4
1016		34.6 ex	63.4 ex	99.6 D(0.01)	106.2 D(0.01)	133.0 D(0.05)	156.4 ex	1.0
1299	D86-automated	34.0	60.3	97.1	104.5	129.9	155.5	1.0
1300	D86-automated	36.8	60.75	97.5	104.8	130.5	156.1	1.0
1521	D86-automated	34.2	60.0	97.1	104.7	129.7	157.9	0.5
1720								
1741		35.8	60.8	96.6	104.3	128.9	156.2	0.6
6041								
6262	D86-automated	34.2	59.7	97.3	104.9	130.1	155.7	1.0
	normality	OK	OK	OK	OK	OK	not OK	
	n	8	8	8	8	8	8	
	outliers	0 +2ex	0 +2ex	2	2	1 +1ex	1 +1ex	
	mean (n)	34.84	60.64	97.11	104.60	129.75	156.11	
	st.dev. (n)	1.385	1.064	0.376	0.233	0.532	0.827	
	R(calc.)	3.88	2.98	1.05	0.65	1.49	2.32	
	st.dev.(D86-A:19)	1.679	1.588	1.422	1.201	1.681	2.536	
	R(D86-A:19)	4.7	4.45	3.98	3.36	4.71	7.1	

Lab 631 IBP, 10% and 90% test results excluded as statistical outliers in other related distillation parameters Lab 1016 IBP, 10% and FBP test results excluded as statistical outliers in other related distillation parameters

z-scores

lab	IBP	10%	40%	50%	90%	FBP
62						
150	0.16	1.11	0.20	0.17	0.21	0.03
334	-1.39	-0.85	0.20	-0.08	-0.39	-0.40
496	0.75	0.79	-0.43	-0.25	-0.03	0.03
631	1.29	0.85	-1.49	-1.33	-0.45	2.12
1016	-0.14	1.74	1.75	1.33	1.93	0.11
1299	-0.50	-0.22	-0.01	-0.08	0.09	-0.24
1300	1.17	0.07	0.27	0.17	0.45	0.00
1521	-0.38	-0.41	-0.01	0.08	-0.03	0.70
1720						
1741	0.57	0.10	-0.36	-0.25	-0.51	0.03
6041						
6262	-0.38	-0.59	0.13	0.25	0.21	-0.16

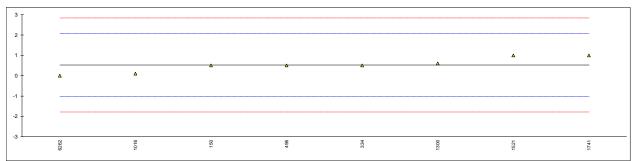
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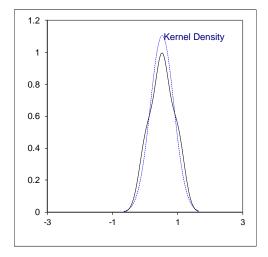


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Determination of Existent Gum (solvent washed) on sample #20025; results in mg/100mL

lab	method	value	mark z(targ)	remarks
62				
150	D381	0.5	-0.03	
334	D381	0.5	-0.03	
496	D381	0.5	-0.03	
631	D381	<1		
1016	D381	0.1	-0.55	
1299	D381	<1		
1300	D381	0.6	0.10	
1521	D381	1	0.62	
1720				
1741	D381	1.0	0.62	
6041				
6262	D381	0	-0.68	
	normality	unknown		
	n	8		
	outliers	0		
	mean (n)	0.52		
	st.dev. (n)	0.362		
	R(calc.)	1.01		
	st.dev.(D381:19)	0.770		
	R(D381:19)	2.16		





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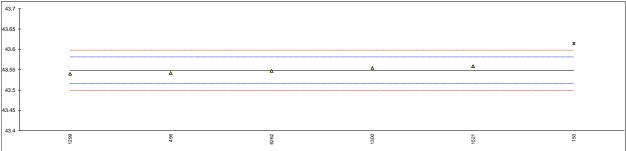
Determination of Freezing Point on sample #20025; results in °C

lab	method	value	mark	z(targ)	remarks
62					
150	D2386	<-70			
334					
496	D2386	<-76			
631	D5972	<-58.0			
1016	D2386	-58			
1299	D2386	<-65.0			
1300	D2386	<-70			
1521	D2386	<-61,5			
1720					
1741	D2386	<-65			
6041					
6262					
	n	7			
	mean (n)	<-58			

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Determination of Heat of Combustion (Net) on sample #20025; results in MJ/kg

lab	method	value	mark	z(targ)	remarks
62					
150	D3338	43.614	D(0.01)	3.99	
334					
496	D3338	43.542		-0.39	
631					
1016					
1299	D3338	43.54		-0.51	
1300	D3338	43.5543		0.36	
1521	D3338	43.559		0.64	
1720					
1741					
6041					
6262	D3338	43.547	С	-0.09	first reported 7670.62 MJ/kg
	normality	unknown			
	n	5			
	outliers	1			
	mean (n)	43.5485			
	st.dev. (n)	0.00807			
	R(calc.)	0.0226			
	st.dev.(D3338:09)	0.01643			
	R(D3338:09)	0.0460			



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Determination of Lead as Pb on sample #20025; results in g Pb/L

lab	method	value	mark z(targ)	remarks	
62					
150	1000000	0.500	4.07		
334 496	ISO3830	0.526	-1.37		
631					
1016					
1299	D5059-A	0.56	2.02		
1300	D3341	0.5436	0.39		
1521	D5059-A	0.527	-1.27		
1720					
1741	D3341	0.5420	0.23		
6041					
6262					
	normality	unknown			
	n	5			
	outliers	0			
	mean (n)	0.5397			
	st.dev. (n)	0.01398			
	R(calc.)	0.0391			
	st.dev.(D3341:16)	0.01003			
	R(D3341:16)	0.0281			
0.58					
0.56					^
0.55					-
0.54			4	Δ	
0.53		Δ			
0.52	Δ	Δ			
0.51					
0.5					
0.49					
0.48	334	521	741	88	588

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Determination of Lead as Tetra Ethyl Lead on sample #20025; results in mL TEL/L

lab	method	value	mark z(t	targ)	calculated by iis	remarks	
62							
150							
334							
496							
631 1016							
1299							
1300	D3341	0.5142		0.60	0.514		
1521	D5059- meth.A	0.498		1.07	0.499		
1720	Doods mem./				0.400		
1741	D3341	0.513		0.47	0.513		
6041							
6262							
	normality	unknown					
	n	3					
	outliers	0					
	mean (n)	0.5084					
	st.dev. (n)	0.00903					
	R(calc.) st.dev.(D3341:16)	0.0253 0.00972					
	R(D3341:16)	0.00972					
	N(D3541.10)	0.0272					
0.55 т							
0.55							
0.53							
0.51				Δ			Δ
	Δ						
0.49							

0.47							
0.45				- 7			00:

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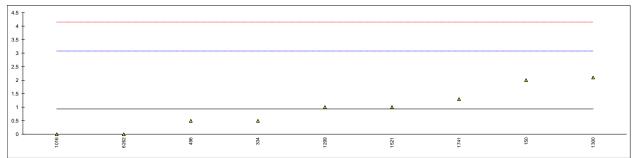
Determination of Lead Precipitate on sample #20025; results in mg/100mL

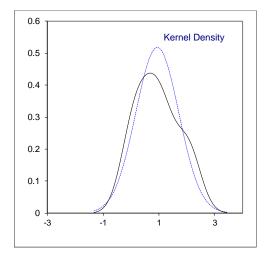
lab	method	value	mark	z(targ)	remarks
62					
150	D873	<1			
334	D873	0			
496					
631					
1016	D873	0.3			
1299	D873	0			
1300	D873	<1			
1521	D873	< 1			
1720					
1741	D873	0.2			
6041					
6262					
	n	7			
	mean (n)	<1			

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Determination of Potential Gum on sample #20025; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
62					
150	D873	2		1.00	
334	D873	0.5		-0.40	
496	D873	0.5		-0.40	
631					
1016	D873	0.0		-0.87	
1299	D873	1		0.06	
1300	D873	2.1		1.09	
1521	D873	1		0.06	
1720					
1741	D873	1.3		0.34	
6041					
6262	D873	0		-0.87	
	normality	OK			
	n	9			
	outliers	0			
	mean (n)	0.933			
	st.dev. (n)	0.7714			
	R(calc.)	2.160			
	st.dev.(D873:12)	1.0714			
	R(D873:12)	3			
	,				





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Determination of Sulfur on sample #20025; results in mg/kg

lab	method	value	mark	z(targ)	remarks
62					
150	D2622	<3			
334	ISO20846	<3	С		first reported 4
496	D2622	0.6			•
631	D7039	<3			
1016	D2622	<3			
1299	D2622	1.3			
1300	D5453	0.7			
1521	D5453	< 3,0			
1720					
1741	D5453	0.21			
6041					
6262	ISO20846	0			
	n	10			
	mean (n)	<3			Application range: 3 – 46000 mg/kg

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Determination of Water reaction, volume change on sample #20025; results in mL

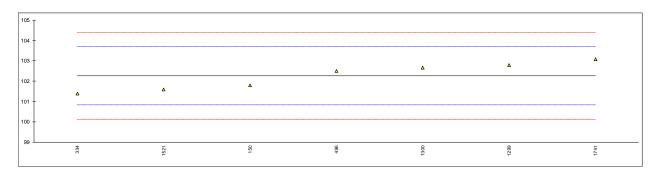
lab	method	value	mark z(targ)	remarks
62				
150	D1094	1.0		
334	D1094	0		
496	D1094	0		
631	D1094	<0.5		
1016	D1094	0		
1299	D1094	1.0		
1300	D1094	<0.5		
1521	D1094	0.0		
1720				
1741	D1094	<0,5		
6041				
6262				
		_		
	n	7		
	mean (n)	<0.5		

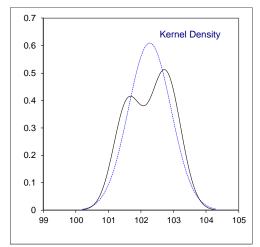
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Determination of MON and Lean mixture Aviation rating on sample #20025

	•	•			Lean mixture Aviation	Lean mixture Aviation	•
lab	method	MON	mark	z(targ)	rating	rating by iis	remarks
62							
150	D2700	101.8		-0.65			
334	D2700	101.4		-1.21			
496	D2700	102.5		0.33			
631							
1016							
1299	D2700	102.8		0.75	108.6	108.6	
1300	D2700	102.66		0.56	108.172	108.3	
1521	D2700	101.6		-0.93	105.3	105.3	
1720							
1741	D2700	103.08		1.14			
6041							
6262							
	!!						
	normality	unknown 7					
	n outliere						
	outliers	0					
	mean (n)	102.26					
	st.dev. (n) R(calc.)	0.654 1.83					
	` '						
	st.dev.(D2700:19) R(D2700:19)	0.714 2					
	K(D2100:19)	4					

Lab 1300 has a different readout of the Lean mixture Aviation rating than iis. According to ASTM D2700 paragraph 19.1.1.2. and 19.2.2. iis first rounded the MON test result to one decimal: 102.7. Then the readout was applied from table 8 where the midpoint was taken between 108.01 and 108.55 and rounded to one decimal: 108.3. Possibly Lab 1300 applied linear interpolation: at 102.4 is the value 107.48 and at 102.8 is the value 108.55. Linear interpolation on the test result 102.66 then gives the value 108.1755 which is in line with the reported value of the participant, but not in line with ASTM D2700.





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APPENDIX 2

Number of participants per country

- 1 lab in CANADA
- 1 lab in UNITED STATES OF AMERICA
- 1 lab in FRANCE
- 1 lab in GERMANY
- 1 lab in PHILIPPINES
- 1 lab in NETHERLANDS
- 1 lab in SPAIN
- 1 lab in ESTONIA
- 1 lab in POLAND
- 1 lab in SUDAN
- 1 lab in SERBIA
- 1 lab in CHINA, People's Republic
- 1 lab in BELGIUM

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APPENDIX 3

Abbreviations

C = final test result after checking of first reported suspect test result

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \\ DG(0.05) &= \text{straggler in Double Grubbs' outlier test} \\ \end{array}$

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

fr. = first reported
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
SDS = Safety Data Sheet

Literature

- iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ASTM E178:02
- 3 ASTM E1301:03
- 4 ISO5725:86
- 5 ISO5725, parts 1-6, 1994
- 6 ISO13528:05
- 7 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Man of the AOAC, (1975)
- 9 IP367:84
- 10 DIN38402 T41/42
- 11 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 12 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
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- 14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, <u>127</u>, 1359-1364 (2002)
- Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)

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