

Institute for Interlaboratory Studies

Results of Proficiency Test Aviation Gasoline March 2022

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 the analysis of Aviation Gasoline based on the scope of the latest version of ASTM D910 and DefStan 91-090 once every two years. During the annual proficiency testing program of 2021/2022, it was decided to continue the round robin for the analysis of Aviation Gasoline.

In this interlaboratory study 18 laboratories from 15 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Aviation Gasoline 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 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 #22040.

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 70 liters of Aviation Gasoline grade 100LL was obtained from a local supplier. After homogenization 58 amber glass bottles of 1 L were filled and labelled #22040.

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 #22040-1	717.38
sample #22040-2	717.37
sample #22040-3	717.38
sample #22040-4	717.40
sample #22040-5	717.40
sample #22040-6	717.37
sample #22040-7	717.40
sample #22040-8	717.40

Table 1: homogeneity test results of subsamples #22040

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)	0.04
reference test method	ISO12185:96
0.3 x R (reference test method)	0.45

Table 2: evaluation of repeatability of subsamples #22040

The calculated repeatability is 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 with Aviation Gasoline labelled #22040, were sent on March 2, 2022. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Aviation Gasoline grade 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: Appearance, Aromatics by FIA, Mono, Di and Total Aromatics (%M/M and %V/V) 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, FBP and Residue), Existent Gum, 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 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 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.

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 test 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 F(0.01) for the Rosner's test. Stragglers are marked by F(0.01) for the Dixon's test, by F(0.01) 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 (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.

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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 (derived from e.g. ISO or ASTM test methods), 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)}} = (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. 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**

In this proficiency test no problems were encountered with the dispatch of the samples. All participants were able to report in time. Not all participants were able to perform all tests requested.

Finally, 18 participants reported 205 numerical test results. Observed was 1 outlying test result, which is 0.5%. 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.

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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 methods are also in the tables together with the original data in appendix 1. 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 (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 test results tables of appendix 1 only the method number and year of adoption or revision (e.g. D873:12) will be used.

<u>Appearance</u>: This determination was not problematic. All of the reporting participants reported the appearance as pass, C&B or 1.

<u>Aromatics by FIA</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1319:20a.

Mono Aromatics (MAH) by HPLC: This determination may not be problematic. Only three test results were reported. The calculated reproducibility is in agreement with the requirements of ASTM D6379:21e1.

<u>Di Aromatics (DAH) by HPLC</u>: Only two participants reported a test result. Therefore, no z-scores are calculated.

<u>Total Aromatics by HPLC (%M/M)</u>: This determination may not be problematic. Only three test results were reported and are in line with each other.

<u>Total Aromatics by HPLC (%V/V)</u>: This determination was very problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements of ASTM D6379:21e1.

<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. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

<u>Distillation</u>: This determination was not problematic. No statistical outliers were observed. All calculated reproducibilities are in agreement with the requirements of ASTM D86:20b automated mode.

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

observed. The calculated reproducibility is in agreement with the

requirements of ASTM D381:22.

<u>Freezing Point</u>: 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 D910:21 and

DefStan 91-090:2019.

<u>Heat of Combustion</u>: This determination was very problematic. No statistical outliers were

observed. The calculated reproducibility is not at all in agreement with the

requirements of ASTM D3338:20a.

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

observed. The calculated reproducibility is not at all in agreement with the

requirements of ASTM D3341:16.

<u>Lead as TEL</u>: Only one laboratory reported a test result. Therefore, no z-scores are

calculated.

Lead Precipitate: This determination was not problematic. Almost all of the reporting

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

<u>Potential Gum</u>: This determination was problematic. No statistical outliers were observed.

The calculated reproducibility is not in agreement with the requirements of

ASTM D873:12(2018).

Sulfur: 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. Therefore, no z-scores are calculated.

Water reaction, volume change: This determination is not problematic. All of the reporting

participants agreed on a value less than 1 mL. Therefore, no z-scores are

calculated.

MON and Lean mixture Aviation rating: 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

D2700:19.

Only three participants read the Lean Mixture Aviation rating from table 8 in

ASTM D2700:19, based on the MON test result.

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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 methods are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Appearance		8	Pass	n.a.	n.a.
Aromatics by FIA	%V/V	9	17.21	2.81	2.87
Mono Aromatics (MAH) by HPLC	%M/M	3	21.95	1.33	1.28
Di Aromatics (DAH) by HPLC	%M/M	2	n.e.	n.e.	n.e.
Total Aromatics by HPLC	%M/M	3	22.09	1.97	1.34
Total Aromatics by HPLC	%V/V	6	17.77	2.32	1.07
Color		5	Blue	n.a.	n.a.
Copper Corrosion 2 hrs at 100 °C		16	1 (1a/1b)	n.a.	n.a.
Density at 15 °C	kg/m³	17	717.6	0.7	1.5
Initial Boiling Point	°C	17	35.4	3.3	4.7
Temp. at 10 % evaporated	°C	17	61.4	3.0	4.4
Temp. at 40 % evaporated	°C	17	97.4	2.0	4.0
Temp. at 50 % evaporated	°C	17	104.8	1.1	3.4
Temp. at 90 % evaporated	°C	17	130.1	1.5	4.7
Final Boiling Point	°C	17	156.0	1.7	7.1
Existent Gum	mg/100mL	8	0.8	0.8	2.2
Freezing Point	°C	12	<-58	n.e.	n.e.
Heat of Combustion (Net)	MJ/kg	9	43.569	0.106	0.046
Lead as Pb	g Pb/L	7	0.537	0.060	0.028
Lead as Tetra Ethyl Lead	mL TEL/L	1	n.e.	n.e.	n.e.
Lead Precipitate	mg/100mL	5	<1	n.e.	n.e.
Potential Gum	mg/100mL	5	2.03	3.33	3
Sulfur	mg/kg	9	0.8	1.6	(0.4)
Water reaction, volume change	mL	11	<1	n.a.	n.a.
MON		7	103.1	1.3	2

Table 3: reproducibilities of tests on sample #22040

For results between brackets no z-scores are calculated

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

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4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2022 WITH PREVIOUS PTS

	March 2022	March 2020	April 2018	April 2016	April 2014
Number of reporting laboratories	18	10	14	20	17
Number of test results	205	149	159	211	193
Number of statistical outliers	1	10	3	3	9
Percentage of statistical outliers	0.5%	6.7%	1.9%	1.4%	4.7%

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 in the following table.

Parameter	March 2022	March 2020	April 2018	April 2016	April 2014
Aromatics by FIA	+/-	+	n.e.	n.e.	n.e.
Aromatics by HPLC	-	++	n.e.		-
Density at 15 °C	++	++	++	++	+
Distillation at 760 mmHg	++	++	++	++	+
Existent Gum	++	++	++	++	++
Heat of Combustion (Net)		++			
Lead as Pb		-		-	
Lead as Tetra Ethyl Lead	n.e.	+/-	n.e.	-	
Potential Gum	-	+	++	+/-	n.e.
Sulfur	()	n.e.	n.e.	n.e.	n.e.
MON	+	+/-	+/-	-	+

Table 5: comparison determinations to the reference test methods

For results between brackets no z-scores are calculated

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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APPENDIX 1

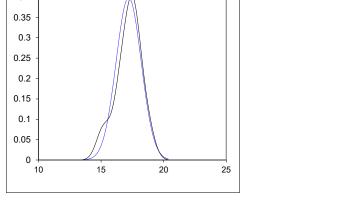
Determination of Appearance on sample #22040

lab	method	Value	z(targ)	remarks
62	D4176	CB / pass		
150				
235	D4176	1		
334				
365	D4176	Pass		
496				
631	D4176	Pass		
1016				
1141	Inhouse	Clear, bright and visually free from solid matter		
1150				
1299	D4176	CL&BR		
1316				
1521	D4176	Clear, bright liquid without solid particles		
1538	D4176	1		
1581				
1650				
1741				
6384				
		_		
	n	8		
	mean (n)	Pass / 1		

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Determination of Aromatics by FIA on sample #22040; results in %V/V

lab	method	value	mark	z(targ)	remarks				
62	D1319	17.38		0.17					
150 235	D1319	 17.913		0.69					
334	סוטוש	17.913		0.09					
365									
496	D1319	18.70		1.46					
631	D1319	17.45		0.24					
1016 1141									
1150									
1299	D1319	17.8		0.58					
1316	D4040	40.5							
1521 1538	D1319	16.5 		-0.69 					
1581									
1650	D1319	15.21		-1.95					
1741	D1319	17.30		0.09					
6384	D1319	16.6		-0.59					
	normality	suspect							
	n	9							
	outliers	0							
	mean (n) st.dev. (n)	17.206 1.0021							
	R(calc.)	2.806							
	st.dev.(D1319:20a)	1.0242							
	R(D1319:20a)	2.868							
22 T									
20 +									
18 -							Δ	Δ	Δ
16 +	Δ	Δ				Δ			
10 +	Δ								
14 -									
12 +									
10									
10 -	1650	6384	Š		62	631	1299	235	496
0.45			7						
0.4 -	K	ernel Density							
0.35 -	// \\								
	// \								
0.3 -	//								
0.25 -									
0.2 -	//								
0.2									



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Determination of Mono Aromatics (MAH) by HPLC on sample #22040; results in %M/M

lab	method	value	mark	z(targ)	remarks
62	·				
150					
235					
334	D6379	22.5		1.19	
365	D0070				
496	D6379	21.74		-0.47	
631					
1016					
1141					
1150 1299					
1316					
1521					
1538					
1581					
1650					
1741	D6379	21.624		-0.72	
6384	D0070	21.024		-0.72	
0004					
	normality	unknown			
	n 	3			
	outliers	0			
	mean (n)	21.955			
	st.dev. (n)	0.4758			
	R(calc.)	1.332 0.4572			
	st.dev.(D6379:21e1)	1.280			
	R(D6379:21e1)	1.200			
24 T					
23 +					Δ
22 +	Δ				Δ
21					
20 +					
19 +					
18 +					
17					

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Determination of Di Aromatics (DAH) by HPLC on sample #22040; results in %M/M

lab	method	value	mark	z(targ)	remarks
62					
150					
235					
334	D6379	0.4			
365					
496	D6379	0.01			
631					
1016					
1141					
1150					
1299					
1316					
1521					
1538					
1581					
1650					
1741					
6384					

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Determination of Total Aromatics by HPLC on sample #22040; results in % M/M

lab	method	value	mark	z(targ)	remarks
62					
150					
235					
334	D6379	22.9		1.68	
365	D0070				
496	D6379	21.75		-0.71	
631					
1016					
1141 1150					
1299					
1316					
1521					
1538					
1581					
1650					
1741	D6379	21.627		-0.97	
6384					
	normality n outliers mean (n) st.dev. (n) R(calc.) st.dev.(D6379:21e1) R(D6379:21e1)	unknown 3 0 22.092 0.7022 1.966 0.4803 1.345			
24 T					
23 -					Δ
22 -					Δ
	Δ				<u> </u>
21 -					
20 -					
19 -					

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Determination of Total Aromatics by HPLC on sample #22040; results in %V/V

lab	method	value	mark z(targ)	remarks		
62						
150						
235						
334	D6379	18.5	1.91			
365	D0070	47.70				
496	D6379	17.79	0.06			
631 1016	IP436	17.308	 -1.20			
1141	17430	17.300	-1.20			
1150						
1299						
1316	D6379	16.5	-3.31			
1521	50070					
1538						
1581						
1650						
1741	D6379	17.710	-0.15			
6384	D6379	18.8	2.69			
	normality	unknown				
	n	6				
	outliers	0				
	mean (n)	17.768 0.8275				
	st.dev. (n) R(calc.)	2.317				
	st.dev.(D6379:21e1)	0.3832				
	R(D6379:21e1)	1.073				
	N(D0379.2161)	1.073				
19.5 T						
19.5						
						Δ
18.5 -					Δ	
18 +			Δ	Δ		
17.5 -		Δ				
17 +						
16.5 +	Δ					
16 +						
15.5 -						
15	1316	1016	1741	964	334	6384
	5	6	4	₹	м	63

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Determination of Color on sample #22040

lab	method	value	mark	z(targ)	remarks
62	Visual	blue			
150	D2392	Acceptable			
235					
334					
365					
496					
631	Visual	Blue			
1016					
1141	Visual	Blue			
1150					
1299	D2392	Acceptable			
1316					
1521	D2392	blue			
1538					
1581					
1650					
1741	IP569	Blue 2.9			
6384					
	n	5			
	n moon (n)	Blue			
	mean (n)	Diue			

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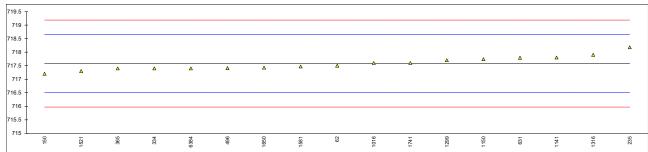
Determination of Copper Corrosion 2hrs at 100 °C on sample #22040

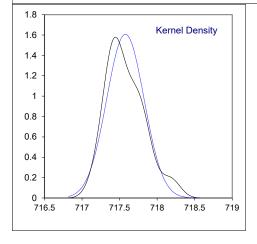
lab	method	value	mark z(targ)	remarks
62	D130	1a		
150	D130	1a		
235	D130	1b		
334	D130	1b		
365	IP154	1a		
496	ISO2160	1a		
631	D130	1a		
1016	D130	1a		
1141	D130	Class 1		
1150	ISO2160	1b		
1299	D130	1A		
1316	D130	1a		
1521	D130	1		
1538				
1581				
1650	D130	1a		
1741	D130	1a		
6384	D130	1a		
	n	16		
	mean (n)	1 (1a/1b)		

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Determination of Density at 15 °C on sample #22040; results in kg/m³

lab	method	value	mark	z(targ)	remarks
62	D4052	717.5		-0.14	
150	D4052	717.2		-0.70	
235	ISO12185	718.18		1.12	
334	ISO12185	717.4		-0.33	
365	IP365	717.4		-0.33	
496	ISO12185	717.41		-0.31	
631	D4052	717.79		0.40	
1016	D4052	717.6		0.04	
1141	D4052	717.8		0.42	
1150	ISO3675	717.74		0.30	
1299	D4052	717.7	С	0.23	First reported 710.3
1316	D4052	717.9		0.60	
1521	D4052	717.3		-0.52	
1538					
1581	ISO12185	717.47		-0.20	
1650	ISO12185	717.43		-0.28	
1741	ISO12185	717.6		0.04	
6384	D4052	717.4		-0.33	
	normality	OK			
	n	17			
	outliers	0			
	mean (n)	717.578			
	st.dev. (n)	0.2482			
	R(calc.)	0.6949			
	st.dev.(ISO12185:96)	0.536			
	R(ISO12185:96)	1.5			
	. (1.55.2.30.00)				





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Determination of Distillation at 760 mmHg on sample #22040; results in °C

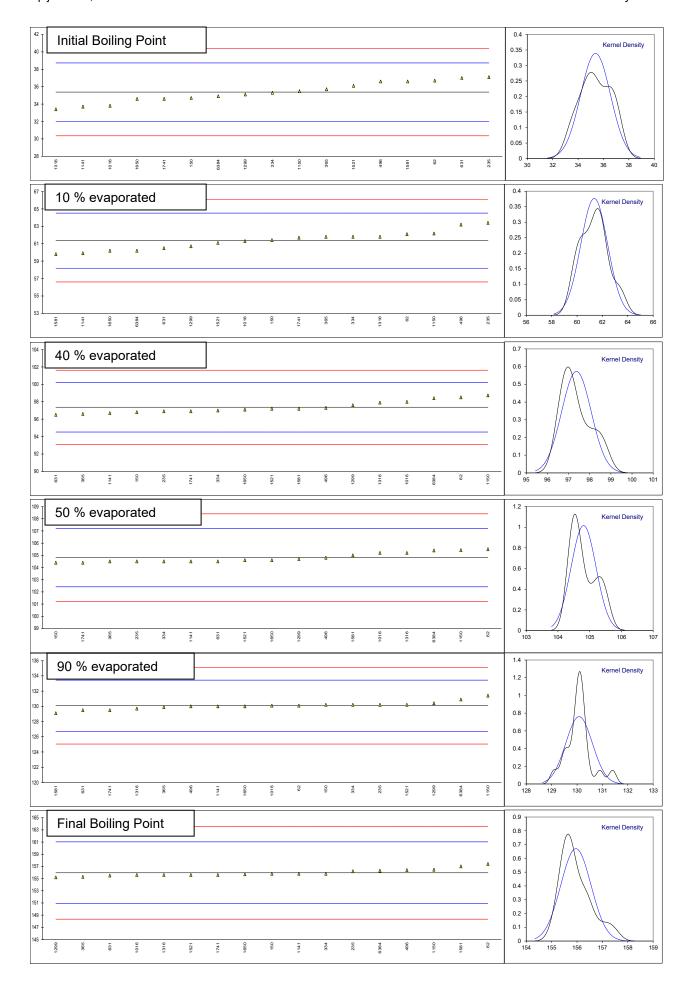
lab	method	IBP	10%	40%	50%	90%	FBP	residue
62	D86-automated	36.7	62.1	98.5	105.5	130.1 C	157.4	0.9
150	D86-automated	34.7	61.4	96.8	104.4	130.2	155.8	
235	D86-automated	37.1	63.4	96.9	104.5	130.2	156.2	1.0
334	D86-automated	35.3	61.8	97.0	104.5	130.2	155.8	1.1
365	IP123-automated	35.7	61.8	96.6	104.5	129.9	155.3	1.0
496	D86-automated	36.6	63.2	97.3	104.8	130.0	156.4	1.0
631	D86-manual	37.0	60.5	96.5	104.5	129.5	155.5 C	0.6
1016	D86-automated	33.8	61.3	98.0	105.2	130.1	155.6	1.0
1141	D86-automated	33.7	59.9	96.7	104.5	130.0	155.8	1.1
1150	ISO3405-automated	35.47	62.17	98.73	105.43	131.4 C	156.47	0.97
1299	D86-automated	35.1	60.7	97.6	104.7	130.4	155.2	1.0
1316	D86-automated	33.4	61.8	97.9	105.2	129.7	155.6	0.5
1521	D86-automated	36.1	61.1	97.2	104.6	130.2	155.6	0.6
1538								
1581		36.6	59.8	97.2	105	129.1	157	0.1
1650	D86-automated	34.6	60.2	97.1	104.6	130.0	155.7	0.9
1741		34.6	61.7	96.9	104.4	129.5	155.6	0.8
6384	D86-automated	34.9	60.2	98.4	105.4	130.9	156.3	1
	normality	OK	OK	OK	OK	suspect	suspect	
	n	17	17	17	17	17	17	
	outliers	0	0	0	0	0	0	
	mean (n)	35.37	61.36	97.37	104.81	130.08	155.96	
	st.dev. (n)	1.177	1.059	0.696	0.393	0.525	0.595	
	R(calc.)	3.30	2.97	1.95	1.10	1.47	1.67	
	st.dev.(D86-A:20b)	1.679	1.585	1.421	1.200	1.680	2.536	
	R(D86-A:20b)	4.7	4.44	3.98	3.36	4.70	7.1	

Lab 62 first reported 131.4 Lab 631 first reported 158.5 Lab 1150 first reported 132.43

z-scores

lab	IBP	10%	40%	50%	90%	FBP
62	0.79	0.47	0.79	0.58	0.01	0.57
150	-0.40	0.03	-0.40	-0.34	0.07	-0.06
235	1.03	1.29	-0.33	-0.26	0.07	0.10
334	-0.04	0.28	-0.26	-0.26	0.07	-0.06
365	0.19	0.28	-0.54	-0.26	-0.11	-0.26
496	0.73	1.16	-0.05	-0.01	-0.05	0.17
631	0.97	-0.54	-0.61	-0.26	-0.35	-0.18
1016	-0.94	-0.04	0.44	0.33	0.01	-0.14
1141	-1.00	-0.92	-0.47	-0.26	-0.05	-0.06
1150	0.06	0.51	0.96	0.52	0.78	0.20
1299	-0.16	-0.41	0.16	-0.09	0.19	-0.30
1316	-1.18	0.28	0.37	0.33	-0.23	-0.14
1521	0.43	-0.16	-0.12	-0.17	0.07	-0.14
1538						
1581	0.73	-0.98	-0.12	0.16	-0.58	0.41
1650	-0.46	-0.73	-0.19	-0.17	-0.05	-0.10
1741	-0.46	0.22	-0.33	-0.34	-0.35	-0.14
6384	-0.28	-0.73	0.72	0.49	0.49	0.14

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Determination of Existent Gum on sample #22040; results in mg/100mL

lab	method	value	mark	z(targ)	remarks			
62	D381	<1						
150	D381	<0.5						
235 334	D381	0.4		-0.51 0.26				
334 365	D381 IP131	1 <1		0.26				
496	IFISI							
631	D381	<1						
1016	D381	<1						
1141	D381	0.6		-0.26				
1150	ISO6246	0.4		-0.51				
1299	D381	1.0		0.26				
1316 1521	D381 D381	<1 1		0.26				
1538	D381	<1 <1		0.20				
1581	D001							
1650	D381	1.2		0.51				
1741	D381	8.0		0.00				
6384								
	normality n	unknown 8						
	outliers	0						
	mean (n)	0.800						
	st.dev. (n)	0.3024						
	R(calc.)	0.847						
	st.dev.(D381:22)	0.7824						
	R(D381:22)	2.191						
3.5 _T								
3 +								
2.5 +								
2 +								
1.5 -								
1 +					Δ	Δ	Δ	Δ
0.5 +		Δ		Δ				
	Δ							
0	235	4		1741	334	1289	1521	1650
1.4			\neg \mid					
	<u></u>	Cernel Density						
1.2 -								
	/ /\`	\						
1 -	/ / \	()						
0.8 -		//						
0.0		//						
0.6		//						
0.0	//	//						
0.4 -		\\						
	//	//						
0.2 -	//	1						
0 -0.5	0 0.5 1	1.5						
-0.5	U U.5 T	1.0	_					

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Determination of Freezing Point on sample #22040; results in °C

lab	method	value	mark z(tai	rg)	remarks
62			-		
150					
235	D2386	<-70			
334					
365					
496	D2386	<-76			
631	D5972	<-58			
1016	D2386	<-65.0			
1141	D2386	< -60			
1150					
1299	D2386	<-65.0			
1316	D7153	<-80			
1521	D7153	< -60,0			
1538	D5972	<-80			
1581	D2386	-55.6			Possibly a false positive test result?
1650	D2386	< -65			
1741	D2386	<-65			
6384	D2386	<-70,0			
	n	12			
	n mean (n)	<-58			
	mean (n)	\- 50			

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Determination of Heat of Combustion (Net) on sample #22040; results in MJ/kg

lab	method	value	mark	z(targ)	remarks				
62	D3338	43.553		-0.99					
150									
235	D3338	43.532		-2.27					
334 365									
496									
631	D3338	43.536		-2.02					
1016									
1141	D4529	43.616		2.85					
1150 1299	D3338	 43.61		2.48					
1316	D3330	45.01		2.40					
1521	D3338	43.579		0.60					
1538									
1581	D2220	42.004		2.45					
1650 1741	D3338 D3338	43.621 43.546		3.15 -1.41					
6384	D3338	43.53		-2.39					
	normality	OK							
	n outliers	9 0							
	mean (n)	43.5692							
	st.dev. (n)	0.03784							
	R(calc.)	0.1060							
	st.dev.(D3338:20a) R(D3338:20a)	0.01643 0.046							
	K(D3336.20a)	0.040							
43.64 _T									
43.62 -									_
43.6 -							Δ	Δ	
43.58 -						Δ			
43.56 -					Δ				
43.54 -	Δ		Δ						
43.52 -									
43.5	235	631	-		62	Ξ.	<u> </u>		9
	6384	8	1741			1521	1299	141	1650
12 —			¬						
	K	ernel Density							
10 -									
	/ X								
8 -	/ / \								
	// \								
6 -									
		\ \							
4 -									
	//								
2 -	//	//							
		//							
0	<u> </u>		⅃ ┃						
43.45	43.5 43.55 43.6	43.65	13.7						

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Determination of Lead as Pb on sample #22040; results in g Pb/L

lab	method	value	mark	z(targ)	remarks			
62	D3341	0.530	-	-0.71				
150								
235								
334 365								
496								
631								
1016	D5059-A	0.516		-2.11				
1141 1150								
1299	D5059-A	0.52		-1.71				
1316	ISO3830	0.526		-1.11				
1521	D3341	0.542		0.49				
1538 1581								
1650	IP352	0.578		4.09				
1741	D3341	0.548		1.09				
6384								
	normality	unknown						
	n	7						
	outliers	0						
	mean (n)	0.5371						
	st.dev. (n) R(calc.)	0.02132 0.0597						
	st.dev.(D3341:16)	0.01000						
	R(D3341:16)	0.0280						
0.6								
0.58 +								Δ
0.56								
0.54						Δ	Δ	
			Δ		Δ			
0.52 +	Δ	Δ						
0.5								
0.48								
	1016	1299	1316		62	1521	1741	1650
25 —								
-		Kernel Density						
		rtornor Bonoky						
20 -	\sim							
	$/ \wedge \rangle$							
15 -	// \\	\						
	// \							
10	// \	()						
10 -		\ \						
5 -	/	4						
	/							
0	,		⊣					
0.4	0.45 0.5 0.5	55 0.6	0.65					

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Determination of Lead as Tetra Ethyl Lead on sample #22040; results in mL TEL/L

lab	method	value	mark	z(targ)	remarks
62					
150					
235					
334					
365					
496					
631					
1016					
1141					
1150					
1299					
1316					
1521	D3341	0.512			
1538					
1581					
1650					
1741					
6384					

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Determination of Lead Precipitate on sample #22040; results in mg/100mL

lab	method	value	mark	z(targ)	remarks
62					
150					
235					
334					
365					
496					
631					
1016	D873	<1			
1141					
1150					
1299	D873	0			
1316	D873	0			
1521	D873	< 1			
1538					
1581					
1650					
1741	D873	0.2			
6384	D873	3.7			Possibly a false positive test result?
		_			
	n	5			
	mean (n)	<1			

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1521

Determination of Potential Gum on sample #22040; results in mg/100mL

lab	method	value	mark z(targ)	remarks		_
62						
150						
235						
334						
365						
496						
631	D873	<1				
1016	D873	<1				
1141						
1150						
1299	D873	2.1	0.07			
1316	D873	4.0	1.84			
1521	D873	1	-0.96			
1538						
1581						
1650						
1741	D873	1.2	-0.77			
6384	D873	1.85	-0.17			
	normality	unknown				
	n	5				
	outliers	0				
	mean (n)	2.030				
	st.dev. (n)	1.1904				
	R(calc.)	3.333				
	st.dev.(D873:12)	1.0714				
	R(D873:12)	3				
	(20.0.12)	· ·				
6 T						
5 +						
4 +					Δ	
3 +						
2 +						
1 - T				Δ		
1 +	Δ	Δ				

6384

1299

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Determination of Sulfur on sample #22040; results in mg/kg

lab	method	value	mark	z(targ)	remark	S			
62	D5453	1							
150	D2622	<3.0							
235	D5453	0.172							
334	ISO20846	<3							
365	ISO20846	0.53							
496	D7000								
631	D7039	<3							
1016	D2622	1.25							
1141 1150	D5453 ISO20884	1 1.998							
1299	D2622	<3							
1316	D2022								
1521	D2622	< 3,0							
1538	DZQZZ								
1581	ISO20846	0.75							
1650	D5453	0.4							
1741	D5453	0.2							
6384									
	normality	OK							
	n	9							
	outliers	0							
	mean (n)	0.811							
	st.dev. (n)	0.5816							
	R(calc.)	1.629							
	st.dev.(D2622:16)	(0.1290)							
	R(D2622:16)	(0.361)			Applicat	ion range : >3	Bmg/kg		
2.5 _T									
2 +									Δ
1.5 -									
								Δ	
1 +						Δ	Δ		
			Δ		Δ				
0.5 +		Δ	<u> </u>						
0	Δ								
0	1741	1650	365		1581	95	1141	1016	1150
							·	·	
0.8			\neg $ $						
0.7 -	K	ernel Density							
0.7	\wedge								
0.6 -	^ \								
0.0	// \\								
0.5 -	// \\								
	// \\								
0.4 -	// \\								
	// \\								
0.3 -	// \\								
0.2 -	//								
0.2	/	\							
0.1 -	/								
	/								
0 1		-	-						
-2	-1 0 1	2 3	4						
			_						

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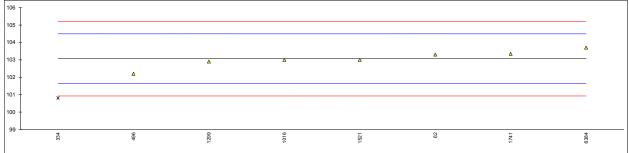
Determination of Water reaction, volume change on sample #22040; results in mL

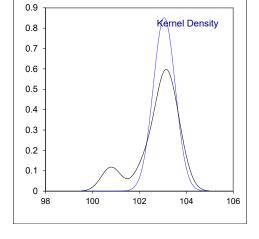
lab	method	value	mark	z(targ)	remarks
62					
150					
235					
334	D1094	0.5			
365					
496					
631	D1094	<0.5			
1016	D1094	<1			
1141	D1094	0.5			
1150					
1299	D1094	0.5			
1316	D1094	0			
1521	D1094	0.0			
1538	D1094	0.5			
1581					
1650	D1094	< 0.5			
1741	D1094	1			
6384	D1094	0.0			
	n	11			
	mean (n)	<1			

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Determination of MON and Lean mixture Aviation rating on sample #22040

			•	•	Lean mixture	Lean mixture	
lab	method	MON	mark	z(targ)	Aviation rating	Aviation rating by iis	remarks
62	D2700	103.3		0.33			
150							
235							
334	D2700	100.8	C,G(0.05)	-3.17			fr. 101.1
365							
496	D2700	102.2		-1.21			
631							
1016	D2700	103.0		-0.09			
1141							
1150	D						
1299	D2700	102.9		-0.23	108.8	108.8	
1316	D0700	400.0			400.4	100.1	
1521	D2700	103.0		-0.09	109.1	109.1	
1538 1581							
1650							
1741	D2700	103.34		0.39			
6384	D2700 D2700	103.34		0.39	110.95	111.0	
0004	D2700	100.7		0.00	110.00	1 111.0	
	normality	unknown					
	n	7					
	outliers	1					
	mean (n)	103.063					
	st.dev. (n)	0.4687					
	R(calc.)	1.312					
	st.dev.(D2700:19)	0.7143					
	R(D2700:19)	2					
	•						
6 T							
5 -							
´							





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APPENDIX 2

Number of participants per country

- 1 lab in BULGARIA
- 1 lab in CANADA
- 1 lab in FRANCE
- 1 lab in GERMANY
- 1 lab in GREECE
- 1 lab in IRELAND
- 1 lab in MACEDONIA
- 1 lab in MAURITIUS
- 1 lab in NETHERLANDS
- 1 lab in PHILIPPINES
- 2 labs in POLAND
- 2 labs in SERBIA
- 2 labs in SPAIN
- 1 lab in SWEDEN
- 1 lab in UNITED STATES OF AMERICA

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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 = 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

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- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 7 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 8 J.N. Miller, Analyst, <u>118</u>, 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, <u>127</u>, 1359-1364, (2002)
- 11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
- Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)

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