

Results of Proficiency Test Jet Fuel A1 September 2024

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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1 Introduction

Since 1995 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Jet Fuel A1 twice a year based on the latest version of the "Aviation Fuel Quality Requirements for Jointly Operated Systems (AFQRJOS)", sometimes referred to as the "Joint Fueling System Check List for Jet A-1". The interlaboratory study on Jet Fuel was extended with separate PTs for the determination of BOCLE, Particle Size Distribution, FAME, JFTOT and Particulate Contamination. During the annual proficiency testing program of 2024 it was decided to continue the round robin for the analysis of Jet Fuel A1.

In this interlaboratory study registered for participation:

- 158 laboratories in 69 countries for regular analyzes in Jet Fuel A1 iis24J02
- 31 laboratories in 19 countries on the BOCLE determination iis24J02BOCLE
- 68 laboratories in 31 countries on the Particle Size determination iis24J02PS
- 81 laboratories in 40 countries on the FAME determination iis24J02FAME
- 104 laboratories in 51 countries on the JFTOT determination iis24J02JF
- 68 laboratories in 38 countries on the Particulate Contamination determination iis24J02CP

In total 182 laboratories in 72 countries registered for participation in one or more proficiency tests, see appendix 5 for the number of participants per country. In this report the results of the Jet Fuel A1 proficiency tests are presented and discussed.

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 a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.

In this proficiency test the participants received, depending on the registration, from one up to seven different samples of Jet Fuel, see table below.

Sample ID	PT ID	Quantity	Purpose
#24150	iis24J02	2x 1 L	Regular analyzes
#24151	iis24J02BOCLE	1x 250 mL	BOCLE
#24152	iis24J02PS	1x 0.5 L	Particle Size Distribution
#24153	iis24J02FAME	1x 100 mL	FAME (low)
#24154	iis24J02FAME	1x 100 mL	FAME (high)
#24155	iis24J02JF	1x 1 L, 70% filled	JFTOT
#24156	iis24J02CP	4x 1 L	Particulate Contamination

Table 1: Jet Fuel samples used in PT iis24J02.

Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

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2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the Organisation 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 October 2024 (iis-protocol, version 4.0). 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

For the preparation of the sample for the regular analyzes in Jet Fuel A1 a batch of approximately 400 liters of Jet Fuel A1 was obtained from a third party. After homogenization 336 amber glass bottles of 1 L were filled and labelled #24150.

The homogeneity of the subsamples was checked by the determination of Density at 15 °C in accordance with ASTM D4052 on 16 stratified randomly selected subsamples.

	Density at 15 °C in kg/m³	
sample #24150-1	798.18	
sample #24150-2	798.18	
sample #24150-3	798.18	
sample #24150-4	798.19	
sample #24150-5	798.18	
sample #24150-6	798.18	
sample #24150-7	798.18	
sample #24150-8	798.19	
sample #24150-9	798.19	
sample #24150-10	798.18	
sample #24150-11	798.18	

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	Density at 15 °C in kg/m³
sample #24150-12	798.18
sample #24150-13	798.18
sample #24150-14	798.18
sample #24150-15	798.18
sample #24150-16	798.18

Table 2: homogeneity test results of subsamples #24150

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)	0.01
reference test method	ASTM D4052:22
0.3 x R (reference test method)	0.15

Table 3: evaluation of the repeatability of subsamples #24150

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

For the preparation of the sample for the BOCLE determination in Jet Fuel A1 a batch of approximately 10 liters of Jet Fuel A1 was obtained from a local refinery. After homogenization 50 amber glass bottles of 250 ml were filled and labelled #24151. The homogeneity of the subsamples was checked by the determination of Density at 15 °C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Density at 15 °C in kg/m³
sample #24151-1	811.62
sample #24151-2	811.62
sample #24151-3	811.62
sample #24151-4	811.62
sample #24151-5	811.62
sample #24151-6	811.62
sample #24151-7	811.62
sample #24151-8	811.62

Table 4: homogeneity test results of subsamples #24151

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.

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	Density at 15 °C in kg/m³
r (observed)	0.00
reference test method	ASTM D4052:22
0.3 x R (reference test method)	0.15

Table 5: evaluation of the repeatability of subsamples #24151

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

For the preparation of the sample for the Particle Size determination in Jet Fuel A1 a batch of approximately 200 liters of Jet Fuel A1 was obtained from a third party. After homogenization 80 amber glass bottles of 0.5 L were filled and labelled #24152. Each bottle was spiked with 1 mL of Lube Oil which contained suspended Arizona Dust before filling with Jet Fuel A1. The homogeneity of the subsamples was checked by the determination of Particle Size Distribution in accordance with IP565 on 8 stratified randomly selected subsamples.

	> 4 µm (c) counts/mL	> 6 µm (c) counts/mL
sample #24152-1	12460	4442
sample #24152-2	12870	4533
sample #24152-3	13494	4865
sample #24152-4	13453	4920
sample #24152-5	13928	4860
sample #24152-6	13531	4788
sample #24152-7	14003	5063
sample #24152-8	13492	4855

Table 6: homogeneity test results of subsamples #24152

From the above test results the relative standard deviations (RSD) were calculated and compared with 0.3 times the corresponding average relative standard deviation obtained from nineteen iis PTs of IP565 test data from 2014 - 2023 in agreement with the procedure of ISO13528, Annex B2 in the next table.

	> 4 µm (c)	> 6 µm (c)
RSD% (observed)	4	4
reference method	iis PTs	iis PTs
0.3 x RSD% (reference method)	5	6

Table 7: evaluation of the relative standard deviations of subsamples #24152

The calculated relative standard deviations are in agreement with 0.3 times the corresponding average relative standard deviation obtained from the previous iis PTs. Therefore, homogeneity of the subsamples was assumed.

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For the preparation of the samples for the FAME determination in Jet Fuel A1 it was decided to prepare two different PT samples: one with a low(er) and one with a high(er) level of FAME. For the low(er) level of FAME a batch of approximately 10 liters of Jet Fuel A1 was made available from a local refinery. After homogenization 95 amber glass bottles of 100 mL were filled and labelled #24153.

For the high(er) level of FAME a batch of approximately 10 liters of Jet Fuel A1 was made available from a local refinery. After homogenization 95 amber glass bottles of 100 mL were filled and labelled #24154.

The homogeneity of the subsamples #24153 and #24154 was checked by the determination of FAME in accordance with method IP585 on 8 stratified randomly selected subsamples.

	FAME in mg/kg #24153	FAME in mg/kg #24154
sample 1	13.3	51.7
sample 2	13.7	51.6
sample 3	13.4	52.2
sample 4	13.4	52.0
sample 5	13.9	51.7
sample 6	13.7	51.7
sample 7	13.7	52.3
sample 8	13.8	51.2

Table 8: homogeneity test results of subsamples #24153 and #24154

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

	FAME in mg/kg #24153	FAME in mg/kg #24154
r (observed)	0.6	1.0
reference test method	IP585:21	IP585:21
0.3 x R (reference test method)	1.3	4.2

Table 9: evaluation of the repeatabilities of subsamples #24153 and #24154

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

For the preparation of the sample for the JFTOT determination in Jet Fuel A1 a batch of approximately 180 liters of Jet Fuel was made positive. After homogenization 125 amber glass bottles of 1 L were filled at a level of 70% and labelled #24155.

The homogeneity of the subsamples was checked by the determination of Density at 15 °C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

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	Density at 15 °C in kg/m³
sample #24155-1	798.35
sample #24155-2	798.36
sample #24155-3	798.36
sample #24155-4	798.36
sample #24155-5	798.36
sample #24155-6	798.36
sample #24155-7	798.36
sample #24155-8	798.35

Table 10: homogeneity test results of subsamples #24155

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)	0.01
reference test method	ASTM D4052:22
0.3 x R (reference test method)	0.15

Table 11: evaluation of the repeatability of subsamples #24155

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

For the preparation of the sample for Particulate Contamination determination in Jet Fuel A1 a batch of approximately 400 liters of Jet Fuel A1 was obtained from a third party. After homogenization 330 amber glass bottles of 1 L were filled and labelled #24156. The homogeneity of the subsamples was checked by the determination of Density at 15 °C in accordance with ASTM D4052 on 14 stratified randomly selected subsamples.

	Density at 15 °C in kg/m³
sample #24156-1	798.36
sample #24156-2	798.36
sample #24156-3	798.36
sample #24156-4	798.36
sample #24156-5	798.36
sample #24156-6	798.36
sample #24156-7	798.36
sample #24156-8	798.32
sample #24156-9	798.36
sample #24156-10	798.36

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	Density at 15 °C in kg/m³
sample #24156-11	798.36
sample #24156-12	798.36
sample #24156-13	798.36
sample #24156-14	798.36

Table 12: homogeneity test results of subsamples #24156

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)	0.03
reference test method	ASTM D4052:22
0.3 x R (reference test method)	0.15

Table 13: evaluation of the repeatability of subsamples #24156

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

Depending on the registration of the participant the appropriate set of PT samples was sent on Augustus 14, 2024. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Jet Fuel A1 packed in the amber glass bottles was checked. The type of bottle was chosen in accordance with ASTM D4306:20. The material has been found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine on sample #24150: Appearance, Total Acidity, Aromatics by FIA, Mono Aromatics (MAH) by HPLC, Di Aromatics (DAH) by HPLC, Total Aromatics by HPLC (in %M/M and %V/V), Color Saybolt (automated and manual), Copper Corrosion 2 hrs. at 100 °C, Density at 15 °C, Distillation at 760 mmHg (IBP, Temperature at 10%, 50%, 90% recovered and FBP, Distillation Residue and Distillation Loss), Existent Gum (unwashed), Flash Point, Freezing Point, Kinematic Viscosity at -20 °C,

Mercaptan Sulfur as S, MSEP, Naphthalenes, Smoke Point, Specific Energy (Net) on Sulfur free basis and Total Sulfur.

On sample #24151 it was requested to determine: Wear Scar Diameter.

On sample #24152 it was requested to determine: Particle Size Distribution in counts/mL for \geq 4, \geq 6, \geq 14, \geq 25 and \geq 30 μ m and scale number for \geq 4, \geq 6 and \geq 14. Some extra information was asked about the equipment used for Particle Size Distribution.

On samples #24153 and #24154 it was requested to determine: FAME content.

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On sample #24155 it was requested to determine: Copper and JFTOT.

On sample #24156 it was requested to determine: Particulate Contamination.

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 Organisation 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 October 2024 (iis-protocol, version 4.0).

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,

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

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

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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 its proficiency tests.

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

The z-scores were calculated according to:

```
z_{\text{(target)}} = \text{(test result - 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. Therefore, the usual interpretation of z-scores is as follows:

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

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