



Institute for
Interlaboratory Studies

**Results of Proficiency Test
Gasoil - ASTM (winter)
September 2024**

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: Mrs. A. Ouwerkerk, BSc
Correctors: Mrs E.R. Montenij-Bos & Mr. R.J. Starink, BSc
Approved by: Mr. R.J. Starink, BSc

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CONTENTS

1 INTRODUCTION 3

2 SET UP..... 3

2.1 ACCREDITATION..... 3

2.2 PROTOCOL 4

2.3 CONFIDENTIALITY STATEMENT 4

2.4 SAMPLES 4

2.5 STABILITY OF THE SAMPLES 7

2.6 ANALYZES 7

3 RESULTS..... 7

3.1 STATISTICS 8

3.2 GRAPHICS 9

3.3 Z-SCORES..... 9

4 EVALUATION 10

4.1 EVALUATION PER SAMPLE AND PER TEST 10

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES 14

4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2024 WITH PREVIOUS PTS 15

Appendices:

1. Data, statistical and graphical results..... 18

2. Number of participants per country112

3. Abbreviations and literature113

1 INTRODUCTION

Since 1994 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Gasoil twice a year. One round is based on the latest version of EN590 and the other round on the latest version of ASTM D975. During the annual proficiency testing program of 2024 it was decided to continue the round robin for the analysis of Gasoil winter quality in accordance with the latest version of ASTM D975.

In this interlaboratory study registered for participation:

- 178 laboratories in 76 countries for regular analyzes in Gasoil iis24G06ASTM
- 38 laboratories in 27 countries on the Cetane Number analyzes iis24G06CN
- 58 laboratories in 37 countries on the Total Contamination analyzes iis24G06TC
- 49 laboratories in 29 countries on the Oxidation Stability analyzes iis24G06OX

In total 183 laboratories in 76 countries registered for participation in one or more proficiency tests, see appendix 2 for the number of participants per country. In this report the results of these Gasoil ASTM (winter) 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 four different samples of Gasoil, see table below.

Sample ID	PT ID	Quantity	Purpose
#24170	iis24G06ASTM	1x 1 L + 1x 0.5 L	Regular analyzes
#24171	iis24G06CN	4x 1 L	Cetane Number & DCN
#24172	iis24G06TC	1x 1 L	Total Contamination
#24173	iis24G06OX	1x 1 L	Oxidation Stability

Table 1: Gasoil samples used in PT iis24G06

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

2.1 ACCREDITATION

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

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, 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 Gasoil a batch of approximately 400 liters of a winter grade Gasoil was obtained from a local supplier. After homogenization 200 amber glass bottles of 1 L and 200 amber glass bottles of 0.5 L were filled and both were labelled #24170.

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/m ³
sample #24170-1	840.16
sample #24170-2	840.17
sample #24170-3	840.16
sample #24170-4	840.17
sample #24170-5	840.17
sample #24170-6	840.17
sample #24170-7	840.17
sample #24170-8	840.17
sample #24170-9	840.17
sample #24170-10	840.18
sample #24170-11	840.17
sample #24170-12	840.18
sample #24170-13	840.17
sample #24170-14	840.17
sample #24170-15	840.17
sample #24170-16	840.17

Table 2: homogeneity test results of subsamples #24170

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/m ³
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 #24170

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 analyzes of Cetane Number and DCN a batch of approximately 400 liters of a winter grade Gasoil was obtained from a local supplier. After homogenization 250 amber glass bottles of 1 L were filled and labelled #24171.

The homogeneity of the subsamples was checked by the determination of Density at 15 °C in accordance with ASTM D4052 on 10 stratified randomly selected subsamples.

	Density at 15 °C in kg/m ³
sample #24171-1	840.17
sample #24171-2	840.18
sample #24171-3	840.18
sample #24171-4	840.18
sample #24171-5	840.17
sample #24171-6	840.18
sample #24171-7	840.18
sample #24171-8	840.18
sample #24171-9	840.18
sample #24171-10	840.18

Table 4: homogeneity test results of subsamples #24171

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/m ³
r (observed)	0.01
reference test method	ASTM D4052:22
0.3 x R (reference test method)	0.15

Table 5: evaluation of the repeatability of subsamples #24171

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 analyzes of Total Contamination in Gasoil a batch of approximately 150 liters of Gasoil was obtained from a local supplier. By means of a calibrated pipette, a defined volume of freshly prepared and well shaken Arizona Dust material in an oil suspension was added to an empty glass bottle. The addition was checked by weighing the bottle before and after addition. In total 80 bottles were prepared and subsequently filled up with 1 L from this batch of Gasoil and homogenized. The subsamples were labelled #24172.

For the preparation of the sample for the analyzes of Oxidation Stability in Gasoil a batch of approximately 100 liters of Gasoil was obtained from a local supplier. The batch was made positive for Oxidation Stability by adding copper rods for a while to enhance the oxidation of Gasoil. After homogenization 85 amber glass bottles of 1 L were filled and labelled #24173. 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/m ³
sample #24173-1	836.12
sample #24173-2	836.12
sample #24173-3	836.12
sample #24173-4	836.12
sample #24173-5	836.12
sample #24173-6	836.12
sample #24173-7	836.12
sample #24173-8	836.12

Table 6: homogeneity test results of subsamples #24173

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/m ³
r (observed)	0.00
reference test method	ASTM D4052:22
0.3 x R (reference test method)	0.15

Table 7: evaluation of the repeatability of subsamples #24173

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 August 28, 2024. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

The participants were requested to determine on sample #24170: Total Acid Number, API Gravity, Aromatics by FIA without oxygenates correction, Ash content, Calculated Cetane Index (two and four variables), Cloud Point, Cold Filter Plugging Point (CFPP), Color ASTM, Conradson Carbon Residue on 10% residue, Ramsbottom Carbon Residue on 10% distillation residue, Copper Corrosion 3 hrs at 50 °C, Density at 15 °C, Distillation at 760 mmHg (IBP, 10%, 50%, 90%, 95% recovered, FBP, Volume at 250 °C and 350 °C, Distillation Residue), FAME content, Flash Point PMcc, Kinematic Viscosity at 40 °C, Lubricity by HFRR at 60 °C, Nitrogen, Pour Point (Manual and/or Automated), Total Sulfur, Water and Water and Sediment (D2709 and D1796).

On sample #24171 it was requested to determine: Cetane Number, Derived Cetane Number (DCN, D6890 and D7668) and Indicated Cetane Number (ICN).

On sample #24172 it was requested to determine: Particulate Contamination (D6217) and Total Contamination (EN12662).

On sample #24173 it was requested to determine: Oxidation Stability (Filterable Insolubles, Adherent Insolubles and Total Insolubles).

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 Organization, 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, 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 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 (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 z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

The $Z_{(\text{target})}$ scores are listed in the 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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Address: Malledijk 18, P.O. Box 200, 3200 AE Spijkensisse, The Netherlands
Telephone number: +31 (0)88 214 45 41
Email address: nl.iis@sgs.com
Website: www.iisnl.com

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