

SLOVENSKI STANDARD

SIST EN ISO 4259-1:2018

01-februar-2018

Nadomešča:
SIST EN ISO 4259:2006

Nafta in sorodni proizvodi - Natančnost merilnih metod in rezultatov - 1. del: Določevanje natančnosti preskusnih metod (ISO 4259-1:2017)

Petroleum and related products - Precision of measurement methods and results - Part 1: Determination of precision data in relation to methods of test (ISO 4259-1:2017)

Mineralölerzeugnisse - Präzision von Messverfahren und Ergebnissen - Teil 1: Bestimmung der Werte für die Präzision von Prüfverfahren (ISO 4259-1:2017)

Produits pétroliers - Fidélité des méthodes de mesure et des résultats - Partie 1: Détermination des valeurs de fidélité relatives aux méthodes d'essai (ISO 4259-1:2017)

Ta slovenski standard je istoveten z: EN ISO 4259-1:2017

ICS:

75.080	Naftni proizvodi na splošno	Petroleum products in general
75.180.30	Oprema za merjenje prostornine in merjenje	Volumetric equipment and measurements

SIST EN ISO 4259-1:2018 en,fr,de

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EUROPEAN STANDARD

EN ISO 4259-1

NORME EUROPÉENNE

EUROPÄISCHE NORM

December 2017

ICS 75.080

Supersedes EN ISO 4259:2006

English Version

Petroleum and related products - Precision of
measurement methods and results - Part 1: Determination
of precision data in relation to methods of test (ISO 4259-
1:2017)

Produits pétroliers et connexes - Fidélité des méthodes
de mesure et de leurs résultats - Partie 1:
Détermination des valeurs de fidélité relatives aux
méthodes d'essai (ISO 4259-1:2017)

Mineralölerzeugnisse - Präzision von Messverfahren
und Ergebnissen - Teil 1: Bestimmung der Werte für
die Präzision von Prüfverfahren (ISO 4259-1:2017)

This European Standard was approved by CEN on 27 October 2017.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN ISO 4259-1:2017) has been prepared by Technical Committee ISO/TC 28 "Petroleum and related products, fuels and lubricants from natural or synthetic sources" in collaboration with Technical Committee CEN/TC 19 "Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin" the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2018, and conflicting national standards shall be withdrawn at the latest by June 2018.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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INTERNATIONAL
STANDARDISO
4259-1First edition
2017-11

**Petroleum and related products —
Precision of measurement methods
and results —****Part 1:
Determination of precision data in
relation to methods of test**

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Produits pétroliers — Fidélité des méthodes de mesure et des résultats —

Partie 1: Détermination des valeurs de fidélité relatives aux méthodes d'essai

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Reference number
ISO 4259-1:2017(E)

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*.

This first edition of ISO 4259-1, together with ISO 4259-2, cancels and replaces ISO 4259, which has been technically revised.

A list of all parts in the ISO 4259 series can be found on the ISO website.

ISO 4259-1:2017(E)**Introduction**

For purposes of quality control and to check compliance with specifications, the properties of commercial petroleum products are assessed by standard laboratory test methods. Two or more measurements of the same property of a specific sample by a specific test method, or, by different test methods that purport to measure the same property, will not usually give exactly the same result. It is, therefore, necessary to take proper account of this fact, by arriving at statistically based estimates of the precision for a method, i.e. an objective measure of the degree of agreement expected between two or more results obtained in specified circumstances.

This document makes reference to ISO 3534-2[1], which gives a different definition of true value (see 3.23). This document also refers to ISO 5725-2. The latter is required in particular and unusual circumstances (see 5.3.1) for the purpose of estimating precision.

The two parts of ISO 4259 encompass both the derivation of precision estimates and the application of precision data. They combine the information in ASTM D6300[2] regarding the determination of the precision estimates and the information in ASTM D3244[3] for the utilization of test data.

A glossary of the variables used in this document and ISO 4259-2 is included as [Annex I](#) in this document.

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Petroleum and related products — Precision of measurement methods and results —

Part 1:

Determination of precision data in relation to methods of test

1 Scope

This document specifies the methodology for the design of an Interlaboratory Study (ILS) and calculation of precision estimates of a test method specified by the study. In particular, it defines the relevant statistical terms ([Clause 3](#)), the procedures to be adopted in the planning of ILS to determine the precision of a test method ([Clause 4](#)), and the method of calculating the precision from the results of such a study ([Clauses 5](#) and [6](#)).

The procedures in this document have been designed specifically for petroleum and petroleum related products, which are normally considered as homogeneous. However, the procedures described in this document can also be applied to other types of homogeneous products. Careful investigations are necessary before applying this document to products for which the assumption of homogeneity can be questioned.

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2 Normative references

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The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

analysis of variance

ANOVA

technique that enables the total variance of a method to be broken down into its component factors

3.2

accepted reference value

ARV

agreed-upon reference value for a specific property of a material determined using an accepted reference method and protocol, e.g. derived from an ILS

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3.3 between laboratory variance

component of the total variance attributable to the difference between the means of different laboratories

Note 1 to entry: When results obtained by more than one laboratory are compared, the scatter is usually wider than when the same number of tests is carried out by a single laboratory, and there is some variation between means obtained by different laboratories. These give rise to the between laboratory variance which is that component of the overall variance due to the difference in the means obtained by different laboratories.

Note 2 to entry: There is a corresponding definition for between operator variance.

Note 3 to entry: The term “between laboratory” is often shortened to “laboratory” when used to qualify representative parameters of the dispersion of the population of results, for example as “laboratory variance”.

3.4 bias

<of a test method> difference between the population mean of test results from a very large number of different laboratories for the property of a material obtained using a specific test method versus the accepted reference value for the property where this is available

Note 1 to entry: See Note 1 to entry in [3.13](#) for an interpretation of “population mean of test results”.

3.5 blind coding

assignment of a different number to each sample so that no other identification or information on any sample is given to the operator

3.6 check sample

sample taken at the place where a product is exchanged, i.e. where the responsibility for the product quality passes from the supplier to the recipient

3.7 degrees of freedom

divisor used in the calculation of variance

Note 1 to entry: The definition applies strictly only in the simplest cases. Definitions for more complex cases are beyond the scope of this document.

3.8 determination

process of carrying out the series of operations specified in a test method, whereby a single value is obtained

3.9 interlaboratory study ILS

study specifically designed to estimate the repeatability and reproducibility of a standard test method achieved at a fixed point in time by multiple laboratories through the statistical analysis of their test results obtained on aliquots prepared from multiple materials

3.10 known value

quantitative value for a property that can be theoretically derived or calculated by the preparation of the sample

Note 1 to entry: The known value does not always exist, for example for empirical tests such as flash point.

3.11 mean

sum of a set of results divided by the number of results

3.12**mean square**

sum of squares divided by the degrees of freedom

3.13**normal distribution**

probability distribution of a continuous random variable, x , such that, if x is any real number, the probability density is as shown in Formula (1):

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{1}{2}\left(\frac{x-\mu}{\sigma}\right)^2\right], -\infty < x < \infty \quad (1)$$

Note 1 to entry: In the context of modelling a distribution of test results, μ is the population mean, or true value (see 3.23) of the property as determined by a specific test method; σ is the standard deviation of the normal distribution used to describe the distribution of an infinite number of test results obtained using the same test method by an infinite number of laboratories ($\sigma > 0$).

3.14**operator**

person who normally and regularly carries out a particular test

3.15**outlier**

result far enough in magnitude from other results to be considered not a part of the set

3.16**precision**

closeness of agreement between the results obtained by applying the same test procedure several times on essentially the same materials and under prescribed conditions

Note 1 to entry: The smaller the random part of the experimental error, the more precise the procedure.

3.17**random error**

component of measurement error that in replicate measurements varies in an unpredictable manner

3.18**repeatability**

limiting value for the difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, within a short interval of time, under the same test conditions, that is expected to be exceeded with a probability of 5% due to random variation

Note 1 to entry: Same test conditions are to be considered as same operator, same apparatus, same calibration and same laboratory.

Note 2 to entry: The representative parameter for the dispersion of the population that can be associated with these results is repeatability standard deviation or repeatability variance. Repeatability refers to the maximum difference attributable to random variation between two results obtained under the state of minimum random variability. Therefore, the period of time during which repeat results are to be obtained should be short enough to exclude time dependent variation, for example, variation caused by environmental changes, or variation associated with multiple calibrations".

Note 3 to entry: The term "repeatability" is not to be confused with the terms "between repeats" or "repeats".