

Irish Standard I.S. EN 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)

 $\ensuremath{\mathbb O}$  CEN 2017  $\hfill No copying without NSAI permission except as permitted by copyright law.$ 

#### I.S. EN ISO 4259-1:2017

Incorporating amendments/corrigenda/National Annexes issued since publication:

The National Standards Authority of Ireland (NSAI) produces the following categories of formal documents:

I.S. xxx: Irish Standard – national specification based on the consensus of an expert panel and subject to public consultation.

S.R. xxx: Standard Recommendation — recommendation based on the consensus of an expert panel and subject to public consultation.

SWiFT xxx: A rapidly developed recommendatory document based on the consensus of the participants of an NSAI workshop.

This document replaces/revises/consolidates the NSAI adoption of the document(s) indicated on the CEN/CENELEC cover/Foreword and the following National document(s):

*NOTE: The date of any NSAI previous adoption may not match the date of its original CEN/CENELEC document.* 

*This document is based on:* EN ISO 4259-1:2017 *Published:* 2017-12-06

This document was published under the authority of the NSAI and comes into effect on:

2017-12-24

ICS number:

75.080

NOTE: If blank see CEN/CENELEC cover page

NSAI	T +353 1 807 3800	Sales:	
1 Swift Square,	F +353 1 807 3838	T +353 1 857 6730	
Northwood, Santry	E standards@nsai.ie	F +353 1 857 6729	
Dublin 9	W NSAI.ie	W standards.ie	

Údarás um Chaighdeáin Náisiúnta na hÉireann

#### **National Foreword**

I.S. EN ISO 4259-1:2017 is the adopted Irish version of the European Document EN 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)

This document does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

For relationships with other publications refer to the NSAI web store.

#### Compliance with this document does not of itself confer immunity from legal obligations.

*In line with international standards practice the decimal point is shown as a comma (,) throughout this document.* 

This is a free page sample. Access the full version online.

This page is intentionally left blank

## EUROPEAN STANDARD NORME EUROPÉENNE

## EN ISO 4259-1

# 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.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION COMITÉ EUROPÉEN DE NORMALISATION EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

This is a free page sample. Access the full version online. I.S. EN ISO 4259-1:2017

EN ISO 4259-1:2017 (E)

Contents	Page
European foreword	

## **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.

This document supersedes EN ISO 4259:2006.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

#### **Endorsement notice**

The text of ISO 4259-1:2017 has been approved by CEN as EN ISO 4259-1:2017 without any modification.

This is a free page sample. Access the full version online.

This page is intentionally left blank

# INTERNATIONAL STANDARD

## ISO 4259-1

First 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

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

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



Reference number ISO 4259-1:2017(E) ISO 4259-1:2017(E)



#### © ISO 2017, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

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 www.iso.org

Page

## Contents

Forew	ord		<b>v</b>		
Introd	uction		vi		
1	Scope				
2	Norm	ative references	1		
3					
	Terms and definitions				
4	Stages in the planning of an interlaboratory study for the determination of the precision of a test method				
	4.1	General			
	4.2	Preparing a draft method of test	5		
	4.3	Planning a pilot study with at least two laboratories	5		
	4.4	Planning the ILS	5		
	4.5	Executing the ILS			
		tical treatment of ILS results			
	5.1	General recommendation			
	5.2 5.3	Pre-screen using GESD technique Transformation of data and outlier tests			
	5.5	5.3.1 General			
		5.3.2 Outlier identification after pre-screening			
		5.3.3 Uniformity of repeatability			
		5.3.4 Uniformity of reproducibility			
	5.4	Rejection of complete data (from all laboratories) for a sample			
	5.5	Estimating missing or rejected values			
		5.5.1 One of the two repeat values missing or rejected	12		
		5.5.2 Both repeat values missing or rejected			
	5.6	Rejection test for outlying laboratories			
	5.7	Confirmation of selected transformation 5.7.1 General			
		5.7.2 Identification of excessively influential sample(s)			
6	Analy	sis of variance, calculation and expression of precision estimates			
0	6.1	General			
	6.2	Analysis of variance			
		6.2.1 Forming the sums of squares for the laboratories × samples interaction			
		sum of squares			
		<ul><li>6.2.2 Forming the sum of squares for the exact analysis of variance</li><li>6.2.3 Degrees of freedom</li></ul>			
		<ul><li>6.2.5 Degrees of freedom.</li><li>6.2.4 Mean squares and analysis of variance</li></ul>	15 15		
	6.3	Expectation of mean squares and calculation of precision estimates			
	010	6.3.1 Expectation of mean squares with no estimated values			
		6.3.2 Expectation of mean squares with estimated values			
		6.3.3 Calculation of precision estimates			
	6.4	Expression of precision estimates of a method of test			
	6.5	Specification of scope for the test method			
7		tio			
Annex	A (nor	mative) Determination of number of samples required	21		
Annex		ormative) <b>Derivation of formula for estimating the number of laboratories and</b> les required to meet minimum 30 degrees of freedom	23		
Annex	<b>C</b> (nor	mative) Notation and tests			
	<b>D</b> (noi	rmative) Illustration of procedures using ILS results for Bromine Number and			
	statis	tical tables			

## This is a free page sample. Access the full version online. I.S. EN ISO 4259-1:2017

### ISO 4259-1:2017(E)

Annex E (normative) Types of dependence and corresponding transformations	49
Annex F (normative) Weighted linear regression analysis	55
Annex G (normative) Rules for rounding	62
Annex H (normative) GESD technique to simultaneously identify multiple outliers in a data set	64
Annex I (informative) Glossary	72
Bibliography	75

## 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 <a href="https://www.iso.org/directives">www.iso.org/directives</a>).

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 <a href="https://www.iso.org/patents">www.iso.org/patents</a>).

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: <a href="http://www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

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 <u>Annex I</u> in this document.

# 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.

#### 2 Normative references

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 <u>https://www.iso.org/obp</u>
- IEC Electropedia: available at <u>http://www.electropedia.org/</u>

#### 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



This is a free preview. Purchase the entire publication at the link below:

**Product Page** 

S Looking for additional Standards? Visit Intertek Inform Infostore

> Learn about LexConnect, All Jurisdictions, Standards referenced in Australian legislation