

Petroleum and related products - Precision of measurement methods and results - Part 4: Use of statistical control charts to validate 'in-statistical-control' status for the execution of a standard test method in a single laboratory (ISO 4259-4:2021, Corrected version 2023-10)

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

<p>See Eesti standard EVS-EN ISO 4259-4:2022 sisaldab Euroopa standardi EN ISO 4259-4:2022 ingliskeelset teksti.</p> <p>Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas</p> <p>Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 15.06.2022.</p> <p>Standard on kättesaadav Eesti Standardimis- ja Akrediteerimiskeskusest.</p>	<p>This Estonian standard EVS-EN ISO 4259-4:2022 consists of the English text of the European standard EN ISO 4259-4:2022.</p> <p>This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation and Accreditation.</p> <p>Date of Availability of the European standard is 15.06.2022.</p> <p>The standard is available from the Estonian Centre for Standardisation and Accreditation.</p>
---	---

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile [standardiosakond@evs.ee](mailto:standardiosakond@evs.ee).

ICS 75.080

Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardimis- ja Akrediteerimiskeskusele  
Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardimis- ja Akrediteerimiskeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardimis- ja Akrediteerimiskeskusega:  
Koduleht [www.evs.ee](http://www.evs.ee); telefon 605 5050; e-post [info@evs.ee](mailto:info@evs.ee)

The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation and Accreditation  
No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation and Accreditation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation and Accreditation:

Homepage [www.evs.ee](http://www.evs.ee); phone +372 605 5050; e-mail [info@evs.ee](mailto:info@evs.ee)

English Version

**Petroleum and related products - Precision of measurement methods and results - Part 4: Use of statistical control charts to validate 'in-statistical-control' status for the execution of a standard test method in a single laboratory (ISO 4259-4:2021, Corrected version 2023-10)**

Produits pétroliers et connexes - Fidélité des méthodes de mesure et de leurs résultats - Partie 4: Utilisation de cartes de contrôle statistique pour valider l'état 'sous maîtrise statistique' pour l'exécution d'une méthode d'essai normalisée dans un seul laboratoire (ISO 4259-4:2021, Version corrigée 2023-10)

Mineralölerzeugnisse - Präzision von Messverfahren und Ergebnissen - Teil 4: Verwendung von Kontrollkarten zur Validierung des Status der statistischen Kontrolle bei der Durchführung von genormten Prüfverfahren in einem einzelnen Labor (ISO 4259-4:2021, korrigierte Fassung 2023-10)

This European Standard was approved by CEN on 30 November 2021.

This European Standard was corrected and reissued by the CEN-CENELEC Management Centre on 15 November 2023.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye 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**

## European foreword

This document (EN ISO 4259-4:2022) 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 December 2022, and conflicting national standards shall be withdrawn at the latest by December 2022.

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.

Any feedback and questions on this document should be directed to the users' national standards body/national committee. A complete listing of these bodies can be found on the CEN website.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## Endorsement notice

The text of ISO 4259-4:2021, Corrected version 2023-10 has been approved by CEN as EN ISO 4259-4:2022 without any modification.

# Contents

Page

<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms, definitions, symbols and abbreviated terms</b> .....	<b>1</b>
3.1 Specific terms and definitions .....	2
3.2 Symbols and abbreviated terms.....	2
<b>4 Statistical control in the execution of a standard test method by a laboratory</b> .....	<b>3</b>
4.1 General.....	3
4.2 Control chart description.....	4
4.2.1 General.....	4
4.2.2 I- and MR-charts.....	4
4.2.3 I-chart sensitivity enhancement strategy .....	4
4.2.4 In-statistical-control conditions .....	5
4.3 Control chart work process.....	5
4.3.1 General.....	5
4.3.2 Stage 1 of control chart work process.....	5
4.3.3 Stage 2 of control chart work process.....	9
4.4 QC material batch change transition.....	11
4.4.1 General.....	11
4.4.2 Procedure 1, concurrent testing .....	12
4.4.3 Procedure 2, Q-chart.....	12
4.4.4 Procedure 3, dynamically updated I-chart with EWMA .....	12
<b>5 Guidance for insufficient variation or non-normal data</b> .....	<b>13</b>
5.1 General requirement.....	13
5.2 How to deal with insufficient variation or non-normal data .....	13
5.2.1 Insufficient variation.....	13
5.2.2 Non-normal data.....	14
<b>Annex A (informative) Details of the control chart work process</b> .....	<b>15</b>
<b>Annex B (normative) Check procedures</b> .....	<b>34</b>
<b>Bibliography</b> .....	<b>36</b>

## 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](http://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](http://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 of 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 [www.iso.org/iso/foreword.html](http://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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

This corrected version of ISO 4259-4:2021 incorporates the following corrections:

- in [4.3.3.1](#), the first sentence in 10<sup>th</sup> bullet has been corrected to read : "If out of statistical control is not confirmed, exclude the initial out of control result and use only the re-analysis result if the initial out of control result exceeds the control limit by an amount  $> 0,25 s_{\text{chart}}$  or there is MR upper control limit exceedance associate with either the original or the retest result."

## Introduction

In the current global business environment, measurement data ‘trustworthiness’ is a key business driver and an implicit expectation from customers and regulatory entities. Data trustworthiness means the data quality meets expectations and is ‘fit-for-use’. Trustworthy data can only be produced by measurement systems that are demonstrated to be stable and are under common cause variation only.

This document describes the applications of specific statistical control charts selected from those that are widely used by the manufacturing sector for the purpose of monitoring and demonstrating the in-statistical-control status of a laboratory in the execution of a standardized test method to produce trustworthy data.

In ISO 4259-2<sup>[9]</sup>, the requirement for assessment of product quality conformance to specification, is to be interpreted that each laboratory’s test result is obtained from a test method that is in-statistical-control in terms of precision and bias, to be substantiated by in-house statistical quality control (SQC) charts or other equivalent statistical techniques. While in-house techniques are used by many laboratories for test method quality assurance, standardization on how to establish in-statistical-control is necessary to ensure consistency in application of ISO 4259-2<sup>[9]</sup>. Addressing the aforementioned necessity is the motivation of this document, which is based on ASTM D6299<sup>[1]</sup>.

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

Part 4:

## Use of statistical control charts to validate 'in-statistical-control' status for the execution of a standard test method in a single laboratory

### 1 Scope

This document specifies the process and methodology for the construction, operation, and maintenance of statistical control charts to assess if a laboratory's execution of a standard test method is in-statistical-control and how to establish and validate the 'in-statistical-control' status.

It specifies control charts that are most appropriate for ISO/TC 28 test methods where the dominant common cause variation is associated with the long term, multiple operator conditions. The control charts specified for determination of in-statistical-control are: individual (I), moving range of 2 ( $MR_2$ ), and either the exponentially weighted moving average (EWMA) or zone-based run rules [similar to Western Electric (WE) run rules<sup>[3]</sup>] as sensitivity enhancement strategy to support the I-chart.

The procedures in this document have been primarily designed for numerical results obtained from testing of control samples prepared from a homogenous source of petroleum and related products in a manner that preserves the homogeneity of properties of interest between control samples. If the test method permits, a certified reference material (CRM) sample is used as a control sample provided the sample composition is representative of the material being tested and is not a pure compound; if this is done then the laboratory best establishes its own mean for the CRM sample.

This document is applicable to properties of interest that are (known to be) stable over time, and for data sets with sufficient resolution to support validation of the assumption that the data distribution can be approximately represented by the normal (Gaussian) model. Mitigating strategies are suggested for situations where the assumption cannot be validated.

### 2 Normative references

The following documents are referred to in the text in such a way that some of their content support 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 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*

### 3 Terms, definitions, symbols and abbreviated terms

For the purposes of this document, the terms and definitions given in ISO 4259-1 and the following apply.

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

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