## INTERNATIONAL STANDARD

Third edition 2006-08-01

# Petroleum products — Determination and application of precision data in relation to methods of test

Produits pétroliers — Détermination et application des valeurs de fidélité relatives aux méthodes d'essai



Reference number ISO 4259:2006(E)

## PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below

This document is a preview denerated by FLS

© ISO 2006

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office Case postale 56 • CH-1211 Geneva 20 Tel. + 41 22 749 01 11 Fax + 41 22 749 09 47 E-mail copyright@iso.org Web www.iso.org Published in Switzerland

## Contents

Forewo	ord	. v
Introdu	iction	vi
1	Scope	. 1
2	Normative references	. 1
3	Terms and Gefinitions	. 1
4	Stages in the planning of an inter-laboratory test programme for the determination of the precision of a test method	٨
4.1	General	
4.2	Preparing a draft method of test	5
4.3	Planning a pilot programme with at least two laboratories	
4.4	Planning the inter-laboratory programme	
4.5	Executing the inter-laboratory programme	. 5
5	Inspection of inter-laboratory results for uniformity and for outliers	. 6
5.1	General	. 6
5.2	Transformation of data	. 7
5.3	Tests for outliers	. 8
5.4	Rejection of complete data from a sample	11
5.5	Estimating missing or rejected values	12
5.6	Rejection test for outlying laboratories.	13
5.7	Estimating missing or rejected values Rejection test for outlying laboratories Confirmation of selected transformation	14
6	Analysis of variance, calculation and expression of precision estimates	14
6.1	General	14
6.2	Analysis of variance	14
6.3	Analysis of variance Expectation of mean squares and calculation of mecision estimates	17
6.4	Expression of precision estimates of a method effect	20
7	Significance of repeatability (r) and reproducibility (R)	21
7.1	General	21
7.2	Repeatability, r	21
7.3	Reproducibility, R	22
•	Chastifications 0	~ 4
8 8.1		24 วง
0.1	Aim of specifications	24
8.2 9	Construction of specifications limits in relation to precision	24 25
9 9.1	General	25
	General	23
9.2	Testing margin at the supplier Testing margin at the recipient	25
9.3 10	Dispute procedure	
	• •	
	A (normative) Determination of number of samples required	
	B (informative) Derivation of equation for calculating the number of samples required	
	C (normative) Notation and tests	31
Annex	D (normative) Example results of test for determination of bromine number and statistical tables	36
	E (normative) Types of dependence and corresponding transformations	
Annex	F (normative) Weighted linear regression analysis	49

## ISO 4259:2006(E)

Annex G (normative) Rules for rounding off results	. 56
Annex H (informative) Explanation of equations given in Clause 7	. 57
Annex I (informative) Specifications that relate to a specified degree of criticality	. 59
Bibliography	. 62

this document is a preview denerated by EKS

## 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 4259 was prepared by Technical Committee ISO/TC 28, Petroleum products and lubricants.

This third edition cancels and replaces the second edition (ISO 4259:1992), Clauses 1, 5, 7 C.7, E.2 and F.3 and subclauses 4.2, 5.2, 6.3.2, 6.3.3.1, 6.3.3.3, 6.4, 8.2, 10.2, 10.4 and 10.5, which have been technically revised. It also incorporates the Technical Congernum ISO 4259:1992/Cor.1:1993.



## 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 any given test method do 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 specific orrumstances.

precision for a method region objective measure of the degree of agreement expected pointed in the degree of agreement expected pointed of the object of the precision of the value (see 3.26). ISO 4259 makes reference to (SO 3534-2<sup>[11]</sup>, which gives a different definition of true value (see 3.26). ISO 4259 also refers to ISO 3534-2<sup>[11]</sup>, which gives a different definition of true value (see 5.2) for the purpose of estimating precision.

© ISO 2006 - All rights reserved

## Petroleum products — Determination and application of precision data in relation to methods of test

## 1 Scope

This International Standard covers the calculation of precision estimates and their application to specifications. In particular, it contains definitions of relevant statistical terms (Clause 3), the procedures to be adopted in the planning of an inter-laboratory test programme to determine the precision of a test method (Clause 4), the method of calculating the precision from the results of such a programme (Clauses 5 and 6), and the procedure to be followed in the interpretation of laboratory results in relation both to precision of the test methods and to the limits laid down in specifications (Clauses 7 to 10).

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

## 2 Normative references

The following referenced documents are indispensable for the application 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 5725-2:1994, 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

### 3.1

### analysis of variance

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

### 3.2

#### between-laboratory variance

element of the total variance attributable to the difference between the mean values of different laboratories

NOTE 1 When results obtained by more than one laboratory are compared, the scatter is usually wider than when the same number of tests are 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 mean values obtained by different laboratories.

NOTE 2 There is a corresponding definition for between-operator variance.

NOTE 3 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".