DIN EN ISO 4259-1:2026-10 (E)

Petroleum and related products - Precision of measurement methods and results - Part 1: Determination of precision data in relation to methods of test (ISO 4259-1:2026)

Contents

Page

Foreword ..... **v**

Introduction ..... **vi**

1 Scope **1**

2 Normative references **1**

3 Terms and definitions **1**

4 Stages in the planning of an interlaboratory study for the determination of the precision of a test method **4**
4.1 General 4
4.2 Preparing a draft method of test 4
4.3 Planning a pilot study with at least two laboratories 5
4.4 Planning the ILS 5
4.5 Executing the ILS 6

5 Statistical treatment of ILS results **7**
5.1 General 7
5.2 Pre-screen using GESD technique 7
5.3 Transformation of data and outlier tests 8
5.3.1 General 8
5.3.2 Outlier identification after pre-screening 11
5.3.3 Uniformity of repeatability 11
5.3.4 Uniformity of reproducibility 11
5.4 Rejection of complete data (from all laboratories) for a sample 11
5.5 Estimating missing or rejected values 12
5.5.1 One of the two repeat values missing or rejected 12
5.5.2 Both repeat values missing or rejected 12
5.6 Rejection test for outlying laboratories 12
5.7 Confirmation of selected transformation 13
5.7.1 General 13
5.7.2 Identification of excessively influential sample(s) 13

6 Analysis of variance, calculation and expression of precision estimates **14**
6.1 General 14
6.2 Analysis of variance 14
6.2.1 Forming the sums of squares for the laboratories multiplied by samples interaction sum of squares 14
6.2.2 Forming the sum of squares for the exact analysis of variance 15
6.2.3 Degrees of freedom 15
6.2.4 Mean squares and analysis of variance 15
6.3 Expectation of mean squares and calculation of precision estimates 15
6.3.1 Expectation of mean squares with no estimated values 15
6.3.2 Expectation of mean squares with estimated values 16
6.3.3 Calculation of precision estimates 17
6.4 Expression of precision estimates of a method of test 18
6.5 Specification of scope for the test method 19
6.6 Reporting limits instruction for the test method 20

7 *R/r* ratio **20**

Annex A (normative) **Determination of number of samples required** **21**

Annex B (informative) **Derivation of formula for estimating the number of laboratories and samples required to meet minimum 30 degrees of freedom** **23**

Annex C (normative) **Notation and tests** **25**
Annex D (normative) **Illustration of procedures using ILS results for the Bromine Number and statistical tables** **30**
Annex E (normative) **Types of dependence and corresponding transformations** **50**
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** **63**
Annex I (informative) **Glossary** **71**
Bibliography **75**