

DIN EN ISO 4259-1:2021-05 (E)

Petroleum and related products - Precision of measurement methods and results - Part 1: Determination of precision data in relation to methods of test (ISO 4259-1:2017 + Amd 1:2019 + Amd 2:2020)

Contents	Page
European foreword	4
A₁ European foreword to Amendment A₁	5
A₂ European foreword to Amendment A₂	6
Foreword	7
A₁ Foreword to Amendment A₁	8
A₂ Foreword to Amendment A₂	9
Introduction	10
1 Scope	11
2 Normative references	11
3 Terms and definitions	11
4 Stages in the planning of an interlaboratory study for the determination of the precision of a test method	14
4.1 General	14
4.2 Preparing a draft method of test	15
4.3 Planning a pilot study with at least two laboratories	15
4.4 Planning the ILS	15
4.5 Executing the ILS	16
5 Statistical treatment of ILS results	17
5.1 General recommendation	17
5.2 Pre-screen using GESD technique	17
5.3 Transformation of data and outlier tests	18
5.3.1 General	18
5.3.2 Outlier identification after pre-screening	21
5.3.3 Uniformity of repeatability	21
5.3.4 Uniformity of reproducibility	21
5.4 Rejection of complete data (from all laboratories) for a sample	21
5.5 Estimating missing or rejected values	22
5.5.1 One of the two repeat values missing or rejected	22
5.5.2 Both repeat values missing or rejected	22
5.6 Rejection test for outlying laboratories	22
5.7 Confirmation of selected transformation	23
5.7.1 General	23
5.7.2 Identification of excessively influential sample(s)	23
6 Analysis of variance, calculation and expression of precision estimates	24
6.1 General	24
6.2 Analysis of variance	24
6.2.1 Forming the sums of squares for the laboratories × samples interaction sum of squares	24
6.2.2 Forming the sum of squares for the exact analysis of variance	25
6.2.3 Degrees of freedom	25
6.2.4 Mean squares and analysis of variance	25
6.3 Expectation of mean squares and calculation of precision estimates	25
6.3.1 Expectation of mean squares with no estimated values	25
6.3.2 Expectation of mean squares with estimated values	26
6.3.3 Calculation of precision estimates	27
6.4 Expression of precision estimates of a method of test	28
6.5 Specification of scope for the test method	29
A₁ 6.6 Reporting limits instruction for the test method A₁	30

7	<i>R/r</i> ratio	30
	Annex A (normative) Determination of number of samples required	31
	Annex B (informative) Derivation of formula for estimating the number of laboratories and samples required to meet minimum 30 degrees of freedom	33
	Annex C (normative) Notation and tests	35
	Annex D (normative) Illustration of procedures using ILS results for Bromine Number and statistical tables	40
	Annex E (normative) Types of dependence and corresponding transformations	59
	Annex F (normative) Weighted linear regression analysis	65
	Annex G (normative) Rules for rounding	72
	Annex H (normative) GESD technique to simultaneously identify multiple outliers in a data set ..	74
	Annex I (informative) Glossary	82
	Bibliography	85