

# 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)

<b>Contents</b>		<b>Page</b>
European foreword .....		4
[A <sub>1</sub> ] European foreword to Amendment [A <sub>1</sub> ] .....		5
[A <sub>2</sub> ] European foreword to Amendment [A <sub>2</sub> ] .....		6
Foreword .....		7
[A <sub>1</sub> ] Foreword to Amendment [A <sub>1</sub> ] .....		8
[A <sub>2</sub> ] Foreword to Amendment [A <sub>2</sub> ] .....		9
Introduction .....		10
<b>1</b>	<b>Scope</b> .....	<b>11</b>
<b>2</b>	<b>Normative references</b> .....	<b>11</b>
<b>3</b>	<b>Terms and definitions</b> .....	<b>11</b>
<b>4</b>	<b>Stages in the planning of an interlaboratory study for the determination of the precision of a test method</b> .....	<b>14</b>
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
<b>5</b>	<b>Statistical treatment of ILS results</b> .....	<b>17</b>
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
<b>6</b>	<b>Analysis of variance, calculation and expression of precision estimates</b> .....	<b>24</b>
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
[A <sub>1</sub> ] 6.6	Reporting limits instruction for the test method [A <sub>1</sub> ] .....	30

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