



Standard Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants¹

This standard is issued under the fixed designation D6300; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Both Research Report RR:D02-1007,² *Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants*² and the ISO 4259, benefitted greatly from more than 50 years of collaboration between ASTM and the Institute of Petroleum (IP) in the UK. The more recent work was documented by the IP and has become ISO 4259.

ISO 4259 encompasses both the determination of precision and the application of such precision data. In effect, it combines the type of information in RR:D02-1007² regarding the determination of the precision estimates and the type of information in Practice D3244 for the utilization of test data. The following practice, intended to replace RR:D02-1007,² differs slightly from related portions of the ISO standard.

1. Scope*

1.1 This practice covers the necessary preparations and planning for the conduct of interlaboratory programs for the development of estimates of precision (determinability, repeatability, and reproducibility) and of bias (absolute and relative), and further presents the standard phraseology for incorporating such information into standard test methods.

1.2 This practice is generally limited to homogeneous petroleum products, liquid fuels, and lubricants with which serious sampling problems (such as heterogeneity or instability) do not normally arise.

1.3 This practice may not be suitable for products with sampling problems as described in 1.2, solid or semisolid products such as petroleum coke, industrial pitches, paraffin waxes, greases, or solid lubricants when the heterogeneous properties of the substances create sampling problems. In such instances, consult a trained statistician.

1.4 *This international standard was developed in accordance with internationally recognized principles on standard-*

ization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:³

- D3244 Practice for Utilization of Test Data to Determine Conformance with Specifications
- D3606 Test Method for Determination of Benzene and Toluene in Spark Ignition Fuels by Gas Chromatography
- D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material
- D7915 Practice for Application of Generalized Extreme Studentized Deviate (GESD) Technique to Simultaneously Identify Multiple Outliers in a Data Set
- E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E456 Terminology Relating to Quality and Statistics
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

¹ This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.94 on Coordinating Subcommittee on Quality Assurance and Statistics.

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² Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1007. Contact ASTM Customer Service at service@astm.org.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard

2.2 ISO Standards:

ISO 4259 Petroleum Products-Determination and Application of Precision Data in Relation to Methods of Test⁴

3. Terminology

3.1 Definitions:

3.1.1 *analysis of variance (ANOVA)*, *n*—technique that enables the total variance of a method to be broken down into its component factors. **ISO 4259**

3.1.2 *bias*, *n*—the difference between the expectation of the test results and an accepted reference value.

3.1.2.1 *Discussion*—The term “expectation” is used in the context of statistics terminology, which implies it is a “statistical expectation.” **E177**

3.1.3 *between-method bias (relative bias)*, *n*—a quantitative expression for the mathematical correction that can statistically improve the degree of agreement between the expected values of two test methods which purport to measure the same property. **D6708**

3.1.4 *degrees of freedom*, *n*—the divisor used in the calculation of variance, one less than the number of independent results.

3.1.4.1 *Discussion*—This definition applies strictly only in the simplest cases. Complete definitions are beyond the scope of this practice. **ISO 4259**

3.1.5 *determinability*, *n*—a quantitative measure of the variability associated with the same operator in a given laboratory obtaining successive determined values using the same apparatus for a series of operations leading to a single result; it is defined as the difference between two such single determined values that would be exceeded about 5 % of the time (one case in 20 in the long run) in the normal and correct operation of the test method.

3.1.5.1 *Discussion*—This definition implies that two determined values, obtained under determinability conditions, which differ by more than the determinability value should be considered suspect. If an operator obtains more than two determinations, then it would usually be satisfactory to check the most discordant determination against the mean of the remainder, using determinability as the critical difference (1).⁵

3.1.6 *mean square*, *n*—in *analysis of variance*, sum of squares divided by the degrees of freedom. **ISO 4259**

3.1.7 *normal distribution*, *n*—the distribution that has the probability function *x*, such that, if *x* is any real number, the probability density is

$$f(x) = (1/\sigma)(2\pi)^{-1/2} \exp[-(x - \mu)^2/2\sigma^2] \quad (1)$$

NOTE 1— μ is the true value and σ is the standard deviation of the normal distribution ($\sigma > 0$). **ISO 4259**

3.1.8 *outlier*, *n*—a result far enough in magnitude from other results to be considered not a part of the set. **RR:D02–1007²**

3.1.9 *precision*, *n*—the degree of agreement between two or more results on the same property of identical test material. In

this practice, precision statements are framed in terms of *repeatability* and *reproducibility* of the test method.

3.1.9.1 *Discussion*—The testing conditions represented by repeatability and reproducibility should reflect the normal extremes of variability under which the test is commonly used. Repeatability conditions are those showing the least variation; reproducibility, the usual maximum degree of variability. Refer to the definitions of each of these terms for greater detail. **RR:D02–1007²**

3.1.10 *random error*, *n*—the chance variation encountered in all test work despite the closest control of variables. **RR:D02–1007²**

3.1.11 *repeatability (a.k.a. Repeatability Limit)*, *n*—a quantitative expression for the random error associated with the difference between two independent results obtained under repeatability conditions that would be exceeded about 5 % of the time (one case in 20 in the long run) in the normal and correct operation of the test method.

3.1.11.1 *Discussion*—Interpret as the limit value the absolute difference between two single test results obtained under repeatability conditions is expected to exceed with an approximate probability of 5 %.

3.1.11.2 *Discussion*—The difference is related to the repeatability standard deviation but it is not the standard deviation or its estimate.

3.1.11.3 *Discussion*—In 3.1.11 and 3.1.13, the term “probability” quantifies the likelihood of repeatability or reproducibility limit exceedance for the difference between a single pair of results obtained under the respective conditions. The “one case in 20 in the long run” in the parenthesis is not to be interpreted as one case in every 20, but it is over the long run. The long run concept can be illustrated using 10 cases out of 200, or 100 cases out of 2000, or 1000 cases in 20 000. The lowest numerical values of one case in 20 is used here.

3.1.11.4 *Discussion*—The “one case in 20” is a legacy term that was carried over from RR:D02-1007 in the original development of Practice D6300. **RR:D02–1007²**

3.1.12 *repeatability conditions*, *n*—conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time. **E177**

3.1.13 *reproducibility (a.k.a. Reproducibility Limit)*, *n*—a quantitative expression for the random error associated with the difference between two independent results obtained under reproducibility conditions that would be exceeded about 5 % of the time (one case in 20 in the long run) in the normal and correct operation of the test method.

3.1.13.1 *Discussion*—Interpret as the limit value the absolute difference between two single test results obtained under reproducibility conditions is expected to exceed with an approximate a probability of 5 %.

3.1.13.2 *Discussion*—The difference is related to the reproducibility standard deviation but is not the standard deviation or its estimate. **RR:D02–1007²**

3.1.13.3 *Discussion*—In those cases where the normal use of the test method does not involve sending a sample to a testing laboratory, either because it is an in-line test method or

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

⁵ The bold numbers in parentheses refers to the list of references at the end of this standard.