Designation: D7778 – 15 (2022)ε1

An American National Standard

Standard Guide for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[1]

This standard is issued under the fixed designation D7778; 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.

ε[1] NOTE—Editorially corrected 6.2.2 in May 2022.

1. Scope

1.1 This guide describes the procedures for planning and conducting an interlaboratory study (ILS) of a test method used in Petroleum Products and Lubricants Committee D02 of ASTM for the purpose of estimating repeatability and reproducibility of the test method in accordance with ASTM Form and Style requirements.

1.2 This guide is concerned with the management of the ILS and intended to provide guidance for the planning stage and ensure the process, logistics, and tools are identified and agreed upon in advance.

1.2.1 Selection of the samples and the impact of sample selection on the final scope of the test method—both the range of materials covered in the scope and precision sections and the measurement range covered in the precision section—are important, and careful consideration needs to be given to these aspects before an ILS is launched.

1.3 This guide does not concern itself with the development of test methods but rather with gathering the information needed for a test method precision statement.

1.4 This guide is concerned with test methods which yield a single numerical figure as the test result, although the single figure may be the result of a calculation from a set of measurements.

1.5 This guide is designed for tests of properties that are stable, such that bulk samples can be homogenized and sub-samples can be prepared that will be identical and stable for the duration of the ILS testing period. This guide is not designed for unstable properties or for properties that make it difficult to obtain stable, representative samples for an ILS conducted at multiple laboratory sites.

1.6 This guide represents the suggested practices for a typical precision study. Individual subcommittees may modify this approach as their expertise directs.

1.7 This guide is voluntary, and thus, is not required for an approved precision study.

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization 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:*[2]

D130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination

D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants

D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

D6792 Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, and Lubricants Testing Laboratories

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E178 Practice for Dealing With Outlying Observations

E456 Terminology Relating to Quality and Statistics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

[1] This test method 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.

Current edition approved May 1, 2022. Published June 2022. Originally approved in 2012. Last previous edition approved in 2015 as D7778 – 15. DOI: 10.1520/D7778-15R22E01.

[2] 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.

E1169 Practice for Conducting Ruggedness Tests

2.2 *ISO/IEC Standards:*[3]

ISO/IEC Guide 30 Terms and Definitions Used in Connection with Reference Materials

ISO/IEC Guide 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions:*

3.1.1 *accuracy, n*—closeness of agreement between an individual test result and an accepted reference value. **E456**

3.1.2 *bias, n*—difference between the population mean of the test results and an accepted reference value. **E456**

3.1.3 *outlier, n*—observation that appears to deviate markedly from the other observations of the sample (also referred to as extreme result, outlying or doubtful observation, or aberrant value. **E178**

3.1.4 *precision, n*—closeness of agreement among test results obtained under prescribed conditions. **E456**

3.1.5 *reference material, n*—material or substance, one or more of whose property values are sufficiently homogenous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to other materials. **E177**

3.1.6 *repeatability, n*—quantitative expression of the random error associated with a single operator in a given laboratory obtaining repetitive results by applying the same test method with the same apparatus under constant operating conditions on identical test material within a short interval of time on the same day. It is defined as the difference between two such results at the 95% confidence level. **D6792, E177**

3.1.7 *reproducibility, n*—quantitative expression of the random error associated with different operators from different laboratories, using different apparatus, each obtaining a single result by applying the same method on an identical test sample. It is defined as the 95% confidence limit for the difference between two such single and independent results. **D6792, E177**

3.1.8 *trueness, n*—closeness of agreement between the population mean of the measurements or the test results and an accepted reference value. **E456**

4. Summary of Guide

4.1 The procedure presented in this guide consists of five basic steps:

(1) Planning an interlaboratory study (including objectives and expected outcome).

(2) Disclosure of plan details (participants, equipment, samples, logistics).

(3) Approval by membership.

(4) Guiding the testing phase of the study and collecting test data results.

(5) Post validation that the ILS was executed in accordance to the agreed plan.

[3] Available from Available from Aerospace Industries Association of America, Inc. (AIA), 1000 Wilson Blvd., Suite 1700, Arlington, VA 22209-3928, http://www.aia-aerospace.org.

4.1.1 This guide does not address the data analysis techniques. For details of such analysis techniques refer to Practice D6300 and E691, or other technically equivalent documents.

4.1.2 The relevant subcommittee is the sole arbiter of technical equivalence.

5. Significance and Use

5.1 All ASTM standard test methods are required to include statements on precision and bias. To obtain such data it is necessary to conduct an interlaboratory study. This document is designed to provide a brief overview of the steps necessary in an ILS and to suggest an appropriate sequence in carrying out these steps.

5.1.1 Qualitative tests such as Test Method D130 copper corrosion may not require an ILS.

6. Planning an Interlaboratory Study

6.1 Suggested steps in an ILS are given in Table 1.

6.2 Support for conducting the ILS is available from ASTM through its Interlaboratory Studies Office (ILS@astm.org). Individuals involved in conducting an ILS are listed below.

6.2.1 "Support" does not include financial support for new or alternate equipment or procedure beyond sampling and shipping materials to the participants.

6.2.2 A Task Force should be formed consisting at a minimum of the method developers, users, and statistician. Based on the objectives of the ILS, this task force should have the overall responsibility for the design, disclosure, and execution of the ILS. The Task Force is charged with the development of an ILS plan that includes scope and purpose, funding, staffing, participants (equipment structure), sample types, tests required, special instructions, reporting form, and sample distribution, participant protocol, and a questionnaire and statistical tools to be used, and criteria for acceptance of results.

6.2.3 An ILS Coordinator should be appointed as an overall person responsible for the distribution of materials and protocols to the laboratories, and receive the test results from the laboratories. Eventually this person should be responsible for all aspects of conducting the ILS, and writing the research report.

TABLE 1 Sequence of an Interlaboratory Study

Sequence	Procedure	Section Number in this Guide
1	Select ILS membership	6.2.2
2	Prepare Basic Design	6.3
3	Specify Test Method(s)	6.4
4	List Participating Laboratories	6.5
5	List Materials	6.6
6	Number of Test Results per Test Material per Lab	6.7
7	Provide Protocol	6.8
8	Ruggedness Study (Pilot Run)	7 and 8
9	Full Scale Run	9
10	Data Handling	10
11	Data Presentation	11
12	Prepare Study Report	12