



## Standard Guide for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope—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.

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

[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 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 System in Petroleum Products 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](#)

[E1169 Practice for Conducting Ruggedness Tests](#)

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products—Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.94.01 on Task Group A: Quality Assurance and Quality Control.

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<sup>2</sup> 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/IEC Standards:<sup>3</sup>

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.

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 ASTM 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.

<sup>3</sup> Available from Available from Aerospace Industries Association of America, Inc. (AIA), 1000 Wilson Blvd., Suite 1700, Arlington, VA 22209-3928, <http://www.aiaaerospace.org>.

**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

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.

6.2.3.1 A running activity record may be useful to be maintained by the ILS Coordinator to overview the progress of the ILS activity.

6.2.4 *Statistician*—The Task Force should obtain the services of a statistician who is familiar with the protocols used in ASTM for precision calculations during the planning stage for input towards the design of the ILS, material selection, establishment of the statistical tools to be used, acceptance criteria, and testing protocol. This person may be a member of the Task Force. This person should also assist the task group in interpreting the results of the data analysis.

6.2.4.1 The statistical software available from D02 Committee can help in these calculations, but it is highly recommended that this work be done by, or under the oversight of the statistician engaged in 6.2.4.

6.3 *Basic Design*—The design should be kept as simple as possible in order to obtain estimates of within- and between-laboratory variability that are free of secondary effects. The basic design is represented by a two-way classification table in which the rows represent the laboratories, the columns represent the materials, and each cell (that is, the intersection of a row with a column) contains the test results obtained by a particular laboratory on a particular material. See Table 2 for illustration where x and y are the test results obtained on the same materials by the same operator from each laboratory.

6.3.1 Samples to be tested shall be presented as a single series of test samples, requiring only a single analysis per sample. Both the material identification and the corresponding duplicate shall be blinded such that the tester does not know the material identification of the test sample, nor its corresponding duplicate run. An example of a randomized series for the ILS design in Table 2 is presented in Table 3. If this single randomized series approach is not possible, then blind repeats of the same material(s) should be embedded among the samples provided. The blind samples should represent the materials used in the scope of the test method and concentration range for which the method is applicable. At the least, the blind duplicates should include a range of easy and difficult samples and include the full sample matrix in the sample set. By including only easy samples in blind testing or the full ILS for that matter, the resulting precision may demonstrate better repeatability than otherwise would be found in practice.

6.3.2 The randomness of testing samples is so important that this has to be a requirement, not just a good practice. The actual duplicates run for each test material shall be randomized within the series.

6.4 *Test Method*—A valid test—To establish initial precision statements for a new test method, a draft of the test method is required that has been developed in one or more competent laboratories and studied in a pilot ILS (see Section 8). This draft method should describe the test procedure in terms that the steps can be unequivocally followed in a typical laboratory by competent personnel with the knowledge of the materials and the property to be tested. The test conditions that affect the test results

**TABLE 2 Basic Design of an Interlaboratory Study**

Laboratory	Materials								
	A	B	C	D	E	F	G	H	I
A	x	x	x	x	x	x	x	x	x
	y	y	y	y	y	y	y	y	y
B	x	x	x	x	x	x	x	x	x
	y	y	y	y	y	y	y	y	y
C	x	x	x	x	x	x	x	x	x
	y	y	y	y	y	y	y	y	y
D	x	x	x	x	x	x	x	x	x
	y	y	y	y	y	y	y	y	y

**TABLE 3 Randomized Test Series**

Test Series	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Material	C	F	V	D	B	E	B	A	F	H	A	G	I	E	G	D	H	I

appreciably should be identified and the proper degree of control of sample handling and the test conditions be specified in the description of the test procedure. The test method should specify calibration procedure and its frequency and the format for reporting the test results.

**6.4.1 *Reevaluation of Existing Precision Statements***—To reevaluate existing precision statements for test methods, all aspects of the existing test method that are not intended to be revised shall be followed. Intended revisions to the test method shall be highlighted in the test method and explicitly stated to the participants.

**6.4.1.1 *ILS Execution Protocol***—The procedures and special instructions provided in the ILS protocol for participation shall not introduce restrictions or controls in the execution of the method that are not in the normal execution of the method being evaluated.

**6.4.1.2 *Variables***—All variables in the test method that are intended to be variables shall be left as variables in the ILS. To remove, control, or set limits on factors that are not intended to be controlled in the normal usage of the test method in the execution of the ILS will result in the precision being overly optimistic, and as such precision statements thus generated will likely be unattainable by users in the normal usage of the method.

**6.5 *Laboratories***—An ILS should ideally include enough laboratories to produce a reproducibility estimate with at least 30 degrees of freedom. See Practice **D6300** for further guidance. It is important that a reasonable cross section of the population of qualified laboratories be represented in the ILS. Special attention is required to ensure that at least 75% of the participants represent product manufacturer laboratories or third party commercial independent testing laboratories, or both. Take care to ensure that the population is random and not biased towards special interest participants such as equipment vendors. Vendor participation shall be managed carefully so as not to bias the data population.

**6.5.1** The final statement of precision of a test method shall not be based on fewer than six laboratories with acceptable test results for each material. More than 7 or 8 laboratories should participate, preferably a minimum of 10 laboratories in case some data is unacceptable and data from one or more laboratories has to be excluded from the data evaluation.

**6.5.2** Any laboratory considered qualified to run the test routinely should be encouraged to participate in the ILS. In order to obtain an adequate number of participating laboratories, the ILS study should be publicized through trade magazines (if feasible), meetings, broadcast through pertinent ASTM committees, etc.

**6.5.3** “Qualified” above implies proper laboratory facilities and testing equipment, competent operators, familiar with the test method, a reputation for reliable testing work, and sufficient time and interest to perform the task.

**6.5.3.1** A good gauge of a laboratories competence can be estimated by its compliance with ISO 17025 or Practice **D6792** laboratory quality management standards.

**6.5.4** If a laboratory is not familiar with the test method, it should do several runs on available materials so all the difficulties are worked out before participating in the actual ILS.

**6.5.4.1** Analysts and laboratories new to the Test Method should be encouraged to document any aspects of the Test Method that are unclear or ambiguous so the wording of the Test Method, especially the procedure, can be clarified to remove ambiguities.

**6.5.5** The ILS should not be restricted to a group of laboratories judged to be exceptionally qualified and equipped for the ILS. This can lead to extremely precise data which cannot be replicated later on when the test method is used in general laboratories. Precision estimates in a standard test method should reflect real-world situation existing at the time of the ILS.

**6.6 *Materials***—If possible, different materials having the same property being investigated should be included in the ILS, preferably at different levels of concentrations. Different dilutions of the same material or compound to be assayed can be considered as “different materials” for the purposes of this guide.

**6.6.1** The number and types of materials to be included in an ILS will depend on a number of considerations including the range of the levels in the class of materials to be tested and likely relation of precision to level over the range, the number of different types of materials to which the test method is to be applied, the difficulty and expense involved in obtaining, processing, and distributing samples, the difficulty of, length of time required for, and expense of performing the test, the commercial or legal need for obtaining a reliable and comprehensive estimate of precision and the uncertainty on any one of these points.

**6.6.2** For example, if it is already known that the precision is either constant or proportional to the average level over the range of values of interest, a smaller number of materials will be needed than if it is merely known that the precision is different at different levels. In such cases ruggedness test (see **7.1**) or the preliminary pilot program (see Section **8**) can help settle some of these questions, and may often result in the saving of considerable time and expense in the full ILS.

**6.6.3** An ILS of a test method should include at least three materials representing different test levels, and for development of a broadly applicable precision statement six or more materials should be included in the study. The number of different materials used in the ILS will be dictated by the scope of the method.

**6.6.3.1** The range of materials in the ILS shall cover the range of materials identified in the scope of the Test Method. The broader the range of materials identified in the scope of the Test Method, the larger number of samples will be needed in the ILS.

6.6.3.2 The range in concentration or level of the parameter being tested should start at the lowest level obtainable or reasonably expected in commerce, ranging to a level that exceeds the expected specification limit. For example, if the test is for sulfur, and the expected range could be from  $\pm 1 \text{ mg/mg/kg-kg}$  up to a limit of  $\pm 15 \text{ mg/mg/kg-kg}$ , the ILS should include samples from as close to zero as possible, ranging up to, perhaps,  $2525 \text{ mg/kg}$  or  $3030 \text{ mg/mg/kg-kg}$  purposely exceeding the limit of  $\pm 15 \text{ mg/mg/kg-kg}$ .

6.6.4 The materials involved in any one ILS should differ primarily only in the level of property measured by the test method. When it is known or suspected that different classes of materials will exhibit different levels of precision when tested by the proposed test method, consideration should be given to conducting separate interlaboratory studies for each class of material. However, in real-world this may be difficult to arrange; hence it should be considered to arrange an ILS using both similar materials and a collection of different materials. When an ILS is conducted only on one type of samples, this should be noted in the precision section that the data was collected in such a manner and mixing of sample types may affect the individual laboratory's precision.

6.6.5 Each material in an ILS should be made to be or selected to be as homogenous as possible prior to its subdivision into test units or test specimens. Distribution of the test specimen should be done among the laboratories by randomization. Homogeneity must be tested/confirmed on random sub-samples prior to sample shipment by methods that can detect non-homogeneity, for example, density or other simple tests.

6.6.6 It may be convenient to use established standard reference materials in the ILS since their homogeneity has been demonstrated. It will also help in establishing the bias of the test method after the ILS is completed.

6.6.7 The materials used in the ILS shall be homogenous and stable, and not volatilize, degrade, gel, evaporate, form sediment, or otherwise change between dispatch and receipt in Practices **D4306** and **D5854**.

6.6.8 The containers used for shipping shall not react with or get degraded by the samples. Appropriate containers for different types of materials are suggested in Practice **D5854**.

#### 6.7 *Number of Test Results per Test Material Samples:*

6.7.1 In the design of an ILS a sufficient total number of test results on each sample shall be specified to obtain a good estimate of the measure of repeatability standard deviation. In many cases, the standard deviation will be a function of the property level being measured. Although it is suggested in Practice **E691** to limit the replicates to three for chemical tests and three or four for a physical or optical test, Committee D02's standard protocol for statistical calculations of precision permits analysis of duplicate samples.

6.7.2 Generally, the time and effort invested in an ILS is better spent on examining more samples across more laboratories than on recording a large number of test results per sample within a few laboratories. The aim is to get a broad consensus of the laboratory community rather just a narrow view of a small segment of the industry.

#### 6.8 *Protocol of an ILS:*

6.8.1 The ILS protocol begins with the development of a plan (see **Table 4** for requirements of the protocol plan) developed by the ILS Task Force. The plan requires approval from the Task Force and notification to the subcommittee members. This is then recorded in the subcommittee meeting minutes. An example of a protocol for an ILS is shown in **Fig. X1.2**.

6.8.2 The ILS Task Force Coordinator shall send a letter to all participating laboratory contacts. (An example of such a letter is given in **Fig. X1.1**.) This letter should include the name, address, telephone number, and e-mail address of the Coordinator. Laboratories should be encouraged to contact the Coordinator regarding any questions about the ILS. The letter should also include the ILS plan outlining the complete details of the ILS.

6.8.3 The ILS Task Force shall determine sample analysis protocol, including sample amount, time and measurement time.

6.8.4 The specific version of the test method being studied should be clearly identified. If the test method allows several options in apparatus or procedure, the protocol should specify which option or options have been selected for the ILS. Test units and test data sheets must be provided for each option. An example of such a data return form is given in **Fig. X1.3**.

6.8.5 When special calibration procedures are required before every determination or every test result, they should be described specifically in the test method.

6.8.6 Any special circumstances that must be addressed in implementing the repeatability conditions, such as the period of time between obtaining the successive test results for the same material; that is, not less than in normal testing and not so long as to likely permit significant changes in the test material, equipment, or environment.

6.8.7 Also specified should be the care, handling, and conditioning of the materials to be tested, where appropriate.

6.8.8 Different orders in which the samples are to be run should be suggested for different groups of laboratories in the ILS so as to randomize any possible effects of all laboratories running in the same sequence should be suggested.

6.8.9 A report form should be provided with the ILS, to help in getting the results in a uniform fashion for later data handling. Information regarding the significant digits to be used in reporting the results should be provided. It is recommended that where possible a significant digit one more than required by the test method should be used.

6.8.10 Laboratories should keep a record or a log of any special events that happen during any phase of ILS testing. This account should be sent to the ILS Coordinator. It can provide a valuable source of information when dealing with unusual data and in making improvements to the test method in future revisions.

TABLE 4 ILS Protocol Plan

Subcommittee:			
ILS Title:			
	Activity	Description	Status
<b>A. Design</b>			
1.	Purpose	Describe the purpose of the ILS (precision, analyzer evaluation, etc.)	_____
2.	Funding	Describe how the ILS will be funded.	_____
3.	Staffing	Identify the ILS leader and the team.	_____
4.	Participants	Identify the participant laboratories in the ILS. Define participation criteria and balance of participation.	_____
5.	Equipment	Identify the models and make of analyzer(s) involved in the ILS.	_____
6.	Samples Types	Define the product(s) to be analyzed.	_____
7.	Tests	Define test method(s) for analysis and special requirements, if any.	_____
8.	Special Instructions	Define any special instructions for sample handling, multiple analysis, etc.	_____
9.	Reporting Form	Develop the reporting form for participants for data and unusual observations.	_____
10.	Statistical Tools	Define what statistical tools will be applied to the data.	_____
11.	Criteria for Data Acceptance	Define what criteria will be used for rejection or acceptance of the data.	_____
12.	Participants' Questionnaire	All information (e.g., calibration material and frequency, instrumentation details, etc.) requested in the questionnaire need to be provided.	_____
<b>B. Implementation</b>			
1.	Communication to Sub-Committee	Subcommittee review and notification of the ILS plan	_____
2.	Solicit Interested Participant	Seek interested participants. Ensure criteria based on the plan are maintained.	_____
3.	Sample Preparation and Shipment	Identify source(s) and sample preparation site.	_____
4.	Pilot Run	Define a pre-ILS as a pilot run if necessary.	_____
5.	Full Scale Run	Conduct the ILS.	_____
6.	Preliminary Data Review	Based on criteria and established statistics.	_____
7.	Application of Statistics	Run statistic.	_____
8.	Data Presentation	Presentation of the data to the Subcommittee for review and approval.	_____
9.	ILS Report	Prepare and submit the final ILS report to ASTM.	_____

6.8.11 A questionnaire should be also sent requesting information on specific aspects of the apparatus, reagents and materials including supplies used, calibration procedures, quality control procedure, etc. Such information can eventually help in establishing the general conditions used in the application of the proposed test method. An example of such a questionnaire is given in Fig. X1.4.

**7. Ruggedness Test**

7.1 Often it is desirable to initiate an ILS first with a ruggedness study. The purpose of a ruggedness test is to find the variables (experimental factors) that strongly influence the measurements provided by the test method, and to determine how closely these variables need to be controlled. Ruggedness tests may not determine the optimum conditions for the test method.

7.2 A ruggedness test is conducted by making systematic changes in the variables associated with the test method and observing the size of the associated changes in the test method results. Generally, the designs (systematic plans of experimentation) associated with ruggedness test are taken from the field of statistics.

7.3 The ruggedness test of a test method should precede an interlaboratory study. The latter should be the final proof test for determining the precision of the test method. If a ruggedness test has not been run to determine, and subsequently to restrict the allowable ranges of the critical variables in the test method, then the precision resulting from the round robin may be poor. It may not be known what went wrong, or how to fix the problem. The ruggedness test, by studying the influence of the test method variables and by indicating the need for selective tightening of test method specifications, helps avoid such situations. The use of ruggedness tests encourages the orderly and optimal development of a test method.

7.4 Ruggedness testing may be done within a single laboratory so the effects of the variables are easier to see. Only the effect of changes in the test method variables from high to low levels needs to be determined. Numerous variables such as temperature, pressure, relative humidity, etc. may need to be studied. The influence of these changes is best studied under the short-term, high-precision conditions found in a single competent laboratory.

7.4.1 For brand new tests methods, there is considerable value in having two or three laboratories participate in the ruggedness study to help reveal significant issues or factors that the original developing laboratory did not consider.