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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.

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.

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 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

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:*

[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 D02.94.01 on Task Group A: Quality Assurance and Quality Control.

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[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.

[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.

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

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.

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.

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

6.2.4.1 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*—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 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.

TABLE 2 Basic Design of an Interlaboratory Study

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

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 1 mg/kg up to a limit of 15 mg/kg, the ILS should include samples from as close to zero as possible, ranging up to, perhaps, 25 mg/kg or 30 mg/kg purposely exceeding the limit of 15 mg/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.

TABLE 4 ILS Protocol Plan

Subcommittee:
ILS Title:

Activity	Description	Status
A. Design		
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. Implementation		
1. Communication to Sub-Committee	Subcommittee review and notificiation 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.	

6.8.7 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.

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.

7.5 Further discussion on ruggedness testing can be found in Guide E1169.

8. Pilot Run of ILS

8.1 Before investing time and resources in a full scale ILS, it is usually wise to conduct a pilot run with only one or perhaps two material(s) and three or four laboratories to determine whether the test method as well as the protocol and all the ILS procedures are clear and to serve as a familiarization procedure for those without sufficient experience with the test method.

8.1.1 This is the last chance for the proponent of the test method to make any technical changes in the proposed test method. Any improvement in wording for clarity, any modification to sample conditioning, and new limits on test parameters, have to be incorporated into the test method at this stage, prior to the ILS. Once the ILS is completed, if there is a need to clarify or modify the procedures technically, it can cause the results from the ILS to become invalid, requiring conducting another new ILS.

8.2 The results of this pilot run also give the Task Group an indication on how well each laboratory will perform in following the protocol of the ILS and whether there are any difficulties in the testing protocols or data submission procedure.

9. Full Scale Run of the ILS

9.1 *Sample Preparation and Labeling*—If it is possible, enough of each material to supply a quantity of 50% more than the minimum amount needed by the number of laboratories involved in the ILS should be prepared. The containers should be labeled with code letter (for the type of material) and code number (for sequential container), allocated by the ILS coordinator.

9.1.1 Suitable containers should be selected for each type of material and the quantity to be shipped. Consult various ASTM standards regarding the best container materials for the oil industry products. See Practice D5854.

9.2 *Randomization*—The specified number of test units to each laboratory, should be allocated for each material using a random number table, or a suitable computerized randomization based on random numbers. See example in 6.3.1.

9.3 *Shipping*—All test units should be packaged properly to arrive in the desired condition at their destinations. When the test material is sensitive to the conditions to which it is exposed (light, heat, humidity, etc.), special directions for opening the package on a label outside the package should be placed on the container. The name of the person coordinating the laboratory participation in the ILS should be clearly indicated on the label.

9.3.1 Follow the U. S. Department of Transportation requirements for shipping materials, hazardous or not, or the appropriate transportation requirements for other countries. In case of air shipment, comply with all IATA requirements.

9.3.2 As the ILS progresses, a laboratory may find that it needs extra test units to replace the defective or improper test units. In such cases the ILS Coordinator needs to decide whether to send a set of replacement units, replace the misused units, or do nothing.

NOTE 1—Often a third party handling and shipping contractor can be utilized for the steps and instructions in 9.1 through 9.3.2.

10. Data Handling

10.1 The analysis and treatment of the ILS test results have three purposes: to determine whether the collected data are consistent to form the basis for a test method precision statement, to investigate and act on any data considered inconsistent, and to obtain the precision statistics on which the precision statement can be based. Since such an analysis can be invalidated by the presence of severe outliers, it is necessary to first examine the consistency of the data.

10.1.1 Data handling should be done by the ILS coordinator.

10.1.2 It is useful for the ILS participants to fill out a predetermined spreadsheet for calculation of the statistics using D2PP software.

10.1.3 Each participant shall provide all information requested. This might include instrumentation details and the calibration material and frequency.

10.2 *Data Inspection*—Upon receipt of the returned data sheets the ILS coordinator should examine the data immediately to detect unusual data points or other deficiencies that should be questioned but not yet discarded.

10.3 For extended calculations it is usually necessary to retain extra significant digits in order to ensure that statistically important information is not lost in calculation by rounding off too soon. As a general rule, at least two more digits in the averages than in the reported test results, and at least three significant figures in the standard deviations should be retained.

10.4 The test results received from the laboratories are usually best arranged in rows and columns as shown in Table 2. Each column contains the data obtained from all laboratories for one material, and each row contains the data from one laboratory for all materials. The test results from one laboratory for one material constitutes a cell. It helps in the interpretation of the data to arrange the materials in increasing order of the measured values.

10.5 The results reported by the laboratories should be examined for cells with unusually extreme values. Look for the signs of mislabeling of the test samples such that the test results for one material are reported as belonging to another material.

10.6 The laboratory reports should also be checked looking for deviations from either the test method or the test protocol.

10.7 If the investigation disclosed no clerical, sampling or procedural errors, the unusual data should be retained, and the precision statistics based on them should be published. If, on the other hand, a cause is found during the investigation, the Task Force or the statistician shall decide whether to eliminate that result set, ask the lab to rerun the particular sample set, or ignore the incident.

10.8 When a large number of laboratories have participated in the ILS and no cause for the unusually extreme cell values have been found during the investigation, it may be appropriate to exclude these cells from the data analysis if agreed to between the ILS Coordinator and the statistician. Generally speaking no more than five percent of the ILS data should not be rejected otherwise it will likely lead to the presentation of precision data that the test method cannot deliver in routine operation. Any discarded data must be accompanied by a statement detailing the rationale, which may or may not be included in the a priori agreed upon set of criteria.

10.9 For test methods where the precision depends on the level of the test results, the variability of the reported results is expected to be different from sample to sample. The method of analysis in ASTM D02 Committee on Petroleum Products and Lubricants (Practice D6300) addresses this situation via a suitable data transformation.

10.10 After transformation, check all data for outliers using Cochran test for uniformity of repeatability and Hawkins test for uniformity of reproducibility.

10.11 At this stage an analysis of variance is carried out and finally the precision estimates derived. Full details with worked examples are given in Practice D6300. A final validation step is required where the Task group carries out a “health check” of the precision statements including (where possible) a comparison to previous precision statements, if available.

10.12 The software for calculating the precision estimates in compliance with Practice D6300 is posted on the ASTM D02 website under “Committee Documents’.

10.13 If alternate test methods are used for determining the same parameter, the data should be checked using Practice D6708.

11. Data Presentation

11.1 After the outlier rejection and calculation of precision statistics, the results may be presented in the precision and bias section of a test method as:

Repeatability limit = xxxxx

Reproducibility limit = xxxxx

At analyte concentration level yyy (units zz) in mmm materials.

11.2 The range for which xxxx is valid must also be listed in order to prevent unintentional extrapolation of the precision estimates, which can lead to misrepresentation of test method precision.

12. ILS Research Report

12.1 ASTM requires an ILS Research Report be prepared at the conclusion of an ILS and publication of a new or revision of an existing test method. This research report is prepared by the ILS Coordinator and should contain:

(1) An Executive Summary of ILS Objectives and Findings

(2) The ILS Approval Plan and Approval Confirmation
(3) Introduction and Reason for the ILS
(4) A List of Participating Laboratories
(5) A List of Materials Used
(6) ILS Protocol
(7) Experimental Procedure Including Calibration
(8) Lab Test Results with Materials Identified
(9) Precision Estimates
(10) Conclusions

12.1.1 A copy of the test method and the statistical analysis should also be attached to the research report.

12.1.2 Research report format is available on the ASTM website (http://www.astm.org/ILS/researchreports.html).

12.2 This research report is submitted to the ILS coordinator in ASTM office and the D02 Committee Staff Manager, and is assigned a report number. The report is cited in the test method in the precision and bias section.

12.3 The following footnote style is used in the precision and bias section of the test method: "The results of the cooperative test program, from which these values have been derived, are filed at ASTM Headquarters as RR-D02-XXXX".

13. ILS Research Report Balloting

13.1 It is recommended that the ILS Research Report accompany the Subcommittee ballot of the new or revised standard. It is acceptable to list a contact where the Research Report may be obtained. Negatives and comments pertaining to the precision and bias statements, or other aspects of the Research Report, are adjudicated according to the regulations governing Subcommittee ballots. Approval of the precision and bias statements in the standard denotes approval of the Research Report by the referencing footnote in the precision section, when there are no other negatives on the Research Report. It is not necessary to ballot the Research Report separately if it is referenced in a standard. If the Subcommittee chooses to approve the report separately by letter balloting the groups involved, then the same balloting requirements apply. If the Research Report is balloted separately, the corresponding standard ballot action approval will be contingent upon approval of the Research report.

13.2 Research Reports once approved at the Subcommittee level, are not normally balloted at the D02 Committee level.

14. Keywords

14.1 calibration; ILS; interlaboratory study; precision; repeatability; reproducibility; ruggedness test

APPENDIX

(Nonmandatory Information)

X1. EXAMPLES

X1.1 See examples of letters and forms in Figs. X1.1-X1.4.

Date: mm/dd/yyyy
From: (ILS Coordinator's Name)
Address: (Mailing address)
Contact: Phone number xxx-xxx-xxxx, and e-mail address xxx@xxx.xx
To: Laboratory Contact

In separate mail, we are sending you a set of samples for use in the ASTM Interlaboratory Study in which you had volunteered to participate. This ILS is being undertaken to (a) establish the precision of a new test method or (2) to revise the existing precision estimates for an existing test method or for an additional type of material(s) for this test method, or both.

Information on the sample materials and expected analytes in them, where available, are provided. A copy of the new method or modification of an existing test method is also attached.

Please follow the protocol and order given in the attachment. Report all results using the attached form. Please indicate all deviations from the test method during the ILS analysis on the report form. A questionnaire on the conditions used during analysis is enclosed; that should also be completed and returned along with the ILS results.

It is important that all laboratories follow this protocol for obtaining the best precision estimates from this ILS.

Please send the test report along with the other requested documentation back to the ILS Coordinator at the address given above. Your help in participating in this ILS is very much appreciated.

If you have any questions regarding this ILS, please contact the ILS Coordinator.

FIG. X1.1 Example of a Letter from the ILS Coordinator to the Participating Laboratories

In addition to the instructions provided to the laboratories beyond those included in the test method or procedure should include:
- Any special conditions or precautions to be observed
- The scheduling and timing for testing the materials or specimens
- Detailed procedures for handling, storing, and disposal of the test materials or specimens
- Special features with respect to the test method
- Reporting of the data to a specified number of significant figures
- Where to submit the data
- Special information to be reported for the benefit of the round robin analysis

1. Follow good laboratory practice when handling samples. Some samples may have very low levels of analyte. All samples should be handled with extreme care to avoid contamination. The samples should remain sealed and should be opened only when ready for analysis.

2. The samples are to be analyzed by following the ASTM Test Method Dxxxx (or the draft method) without any deviation.

3. All information (for example, calibration material and frequency, instrumentation details, etc.) requested in the questionnaire need to be provided.

4. Testing shall be performed under normal conditions using an operator with good experience. A change of operator, if necessary, should be between the sample segments and definitely not during replicate analysis.

5. Approximate concentration range for the analyte may be provided for the samples, if required. Should a sample be found outside its expected range, reanalyze using an appropriate calibration curve.

6. Use a quality control sample each day of testing. Use a known control of your own or create a separate control sample from the stock solution.

7. Performance check samples, if part of the sample set, are representative of the sample types analyzed, are homogenous and stable under the expected shipping and storage conditions, and have an accepted reference value established by an certificate of analysis, multiple analyses against a Standard Reference Material (SRM), or other high value multiple analysis technique.

8. Trial runs shall not be performed on the distributed samples. If training is necessary, use other materials.

9. The report form should be used for documenting unusual occurrences, difficulties, changes in operator (if absolutely necessary), and other comments regarding the performance of the analysis. Also, please provide all requested information on the form.

10. The apparatus to be used in the ILS must be in good working order, and be assembled and operated according to the manufacturer's recommendations and using manufacturer approved supplies and consumables.

11. The reagents to be used in the method are to be of the quality specified in the test method. Always use a blank sample to check for contamination. The blanks are prepared by an individual laboratory.

12. Be sure the sample size and instrument parameters are used as recommended in the test method. Note all exceptions on the ILS report form.

13. The apparatus measurement capability is affected by instrument maintenance quality, laboratory environment/protocols, equipment age, and other factors. Requiring ILS participants to modify their "normal" operating environment or performing extended maintenance may not be practical. Therefore, before analyzing the ILS samples, each instrument/apparatus must be qualified for the determination intended. Instrument/calibration qualification is accomplished by calibrating the analyzer for the range of samples to be measured using the applicable proposed test method requirements or the instrument manufacturer's recommendations, or both, and completing a successful analysis of the provided performance check sample prior to the commencement of the analysis session.

14. After the above initial calibration and obtaining acceptable check sample results, sample testing can begin. An analysis session shall consist of a sequential analysis of the samples in duplicate, in the order specified in the ILS report form.

15. After an elapsed time period of at least 8 hours but less than 72 hours, return the apparatus operating conditions (if required) to those used for calibration and data collection in the first analysis session. During the day 2 analysis session also calibration and check samples analysis shall be performed before starting the analysis of ILS samples.

16. Use the ILS report form to document all unusual occurrences, difficulties, changes in operator (if necessary), and other comments regarding the performance of the analysis. Provide all requested information, such as, sample size, check sample results, and basic apparatus operational settings, etc.

17. Primary communications are to be carried out regarding this ILS with the Task Group ILS Coordinator.

18. The Task Group reserves the right to reject data that is reported late or is generated with significant deviations from the test method.

FIG. X1.2 Example of a Protocol for an Interlaboratory Study

LABORATORY:
CONTACT:
PHONE NUMBER:
E-MAIL:

SAMPLE #	ANALYSIS # 1	ANALYSIS # 2
A		
B		
C		
D		

Report the results to x significant figures.

FIG. X1.3 Example of an ILS Return Form

LABORATORY:
CONTACT:
PHONE NUMBER:
E-MAIL:

I. APPARATUS
1. Instrument Manufacturer:
2. Instrument Model:
3. Other relevant parameters:

II. REAGENTS AND MATERIALS
4. Source of reference standards used:
5. Number of reference standards used;
6. Reference value for above standards;
7. Other relevant parameters:

III. CALIBRATION AND VERIFICATION
8. Were all timers, temperature measuring devices, balances, etc. used properly calibrated?
9. Calibration materials used:
10. Concentration range of calibration materials:
11. Check standards used:
12. Concentration range of check standards:

IV. QUALITY CONTROL/QUALITY ASSURANCE
13. Was a QC/QA sample analyzed with the ILS set of samples?
14. What was the QC/QA sample used?
15. Frequency of QC/QA analysis:
16. Precision based on QC analysis: Mean: Standard Deviation:

V. GENERAL INFORMATION
17. Did the lab personnel follow the test method (including the procedure) exactly as written when analyzing the ILS samples?
18. If “No”, list and describe the deviations from the test method supplied.
19. Did the lab personnel follow the ILS protocol instructions exactly as written that provide supplementary information on how the samples to be analyzed?
20. If “No”, list and describe the deviations from the protocol.
21. Other comments that the lab would like to add that were not asked in the questionnaire so far

RETURN THIS RESPONSE FORM ALONG WITH THE TEST RESULTS TO THE ILS COORDINATOR.

FIG. X1.4 Example of an ILS Questionnaire

SUMMARY OF CHANGES

Subcommittee D02.94 has identified the location of selected changes to this standard since the last issue (D7778 – 12) that may impact the use of this standard. (Approved April 1, 2015.)

(1) Revised subsection 6.4 and added new subsections 6.4.1, 6.4.1.1, and 6.4.1.2 to clarify the requirement for an ILS to reevaluate precision for an existing method to strictly follow the existing methodology.