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Standard Practice for Conducting Proficiency Tests in the Chemical Analysis of Metals, Ores, and Related Materials[1]

This standard is issued under the fixed designation E2027; 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 practice provides direction for organizing and conducting proficiency test programs in analytical chemistry for metals, ores, and related materials. It is consistent with ISO Guide 43 and Guide E1301. It does not address the selection and use of proficiency testing schemes by accrediting bodies.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E826 Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry

E1187 Terminology Relating to Conformity Assessment (Withdrawn 2006)[3]

E1301 Guide for Proficiency Testing by Interlaboratory Comparisons (Withdrawn 2012)[3]

E1724 Guide for Testing and Certification of Metal, Ore, and Metal-Related Reference Materials (Withdrawn 2010)[3]

2.2 *ISO Standards:*[4]

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

ISO Guide 43 Proficiency Testing by Interlaboratory Comparisons

ISO Guide 9000 Quality Management and Quality System Elements

3. Terminology

3.1 *Definitions*—For formal definitions related to laboratory accreditation, Terminology E1187 applies.

3.2 For other definitions of terms used in the practice, refer to Terminology E135.

4. Significance and Use

4.1 This practice sets the basic requirements for proficiency test programs in the chemical analysis of metals, ores, and related materials. It does not set specific procedural requirements, but does establish a framework for particular programs, including those with either small or large numbers of participants. (**Warning—**The data from proficiency testing programs must never be used to assign certification values to the materials used in the program. The elements of a properly conceived and implemented certification program are described in detail in Guide E1724.)

4.2 Most accreditation bodies require that laboratories participate regularly in proficiency testing programs that they have accepted for the purpose. Therefore, it is essential that each program comply with accepted principles including technical requirements, statistical procedures (see Annex A1), and quality management (see Annex A2).

5. Types of Proficiency Testing

5.1 Proficiency testing techniques vary depending on the nature of the test item, the method in use and the number of laboratories participating. The most common approach involves randomly selected sub-samples from a source of material being distributed simultaneously to participating testing laboratories for concurrent testing. It is essential that all of the material from which the participants' test materials are taken be sufficiently homogeneous so that any results later identified as outliers should not be attributed to any significant test item variability. After completion of the testing, the results are returned to the coordinating body, and compared with the assigned value(s) or to the mean and standard deviations obtained from a statistical analysis of the data to give an indication of the performance of the individual laboratories and the group as a whole.

5.2 In some cases, separate portions of previously certified reference materials are circulated.

[1] This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.22 on Laboratory Quality.

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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] The last approved version of this historical standard is referenced on www.astm.org.

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

6. Organization and Design

6.1 *Framework:*

6.1.1 The design stage of any proficiency testing program requires the input of technical experts, statisticians and a program coordinator to ensure its success and smooth operation.

6.1.2 The coordinator, in consultation with these other personnel, develops a program appropriate to the particular proficiency test. A proficiency test program shall be designed to avoid any confusion about its objectives. A plan shall be established and documented (see Annex A2) before the start of the program and shall include the following information:

6.1.2.1 The name and the address of the organization conducting the proficiency program,

6.1.2.2 The name and address of the coordinator and other personnel involved in the design and operation of the proficiency program,

6.1.2.3 The nature and the purpose of the proficiency program,

6.1.2.4 A procedure for the manner in which the participants are selected, or criteria that need to be met before participation is allowed,

6.1.2.5 The name and address of the laboratory or laboratories performing the various parts of the program (for example, sampling, sample processing, homogeneity testing and assigning values) and a description of the market to be served,

6.1.2.6 The nature of the test material(s) and test(s) selected, as well as a short description of the considerations underlying these choices,

6.1.2.7 A description of the manner in which the test materials are obtained, processed, checked and transported,

6.1.2.8 The time schedule for the various phases of the proficiency testing,

6.1.2.9 The expected initial and target dates or deadlines of the proficiency program including the date(s) for the testing to be conducted by the participants,

6.1.2.10 For ongoing programs, the frequency at which test materials are distributed,

6.1.2.11 Information on methods or procedures which participants may need to use to perform the tests or measurements (ASTM test methods, laboratory standard procedures/methods, etc.),

6.1.2.12 An outline of the statistical analysis to be used including the determination of assigned value(s) and any outlier detection techniques,

6.1.2.13 The basis for performance evaluation techniques, and

6.1.2.14 A description of the extent to which the test results, and the conclusions that will be based on the outcome of the proficiency tests, are to be made public.

6.2 *Staff:*

6.2.1 The staff shall include, or collaborate closely with, those holding adequate qualifications and experience in the design, implementation and reporting of interlaboratory comparisons. They shall possess appropriate technical, statistical and administrative skills.

6.2.2 The operation of specific interlaboratory comparisons requires the guidance of persons with detailed technical knowledge and experience of the test methods involved. To this end the coordinator shall enlist some professionals to act as an advisory group. The functions of this advisory group may be to:

6.2.2.1 Develop and review procedures for the planning execution, analysis, reporting and monitoring the effectiveness of the proficiency testing program,

6.2.2.2 Identify and evaluate interlaboratory comparisons organized by other bodies,

6.2.2.3 Evaluate proficiency test results of participating laboratories,

6.2.2.4 Provide advice to any body assessing the technical competence of participating laboratories, both on the results obtained during a proficiency test program, and how those results should be used with other aspects of laboratory evaluations,

6.2.2.5 Provide advice to participants who appear to experience problems, and

6.2.2.6 Resolve disputes between the coordinator and participants.

6.3 *Data Processing Equipment*—Equipment shall be adequate to conduct all necessary data entry and statistical analyses and provide timely and valid results. Procedures for checking data entry shall be implemented and all software shall be verified, supported and backed up. The storage and security of data files shall be controlled.

6.4 *Statistical Design:*

6.4.1 The statistical model and data analysis techniques to be used shall be documented together with a short description of the background to their selection. Further details of common statistical procedures and treatment of proficiency testing data are discussed in Annex A1.

6.4.2 Careful consideration shall be given to the following matters and their interactions: the repeatability and reproducibility of the test(s) involved; the smallest differences to be detected between participating laboratories at a desired confidence level; the number of participating laboratories; the number of samples to be tested and the number of repeat tests or measurements to be carried out on each sample; the procedures to be used to estimate the assigned value; procedures to be used to identify outliers; and, potential bias in the test methods employed.

6.5 *Test Materials Preparation:*

6.5.1 Preparation of test materials may either be outsourced or undertaken by the coordinator. The organization preparing the test material shall have demonstrable competence to do so.

6.5.2 Any conditions relating to the test materials that may affect the integrity of the interlaboratory comparison, such as homogeneity, stability, possible damage in transit and effects of ambient conditions shall be considered.

6.5.3 The test materials or materials to be distributed in the program shall be similar in nature to those routinely tested by participating laboratories.

6.5.4 The number of test materials to be distributed may depend on whether there is a requirement to cover a range of compositions.

6.5.5 The assigned value(s) shall not be disclosed to the participants until after the results have been collated. However, in some cases it may be appropriate to advise target ranges prior to testing.

6.5.6 Consideration may be given to preparation of additional test materials other than those needed for the proficiency test program. Surplus test materials may be useful as quality control materials, test samples for interlaboratory tests of new test methods, or training aids for laboratories after results from participants have been evaluated.

6.6 *Test Materials Management:*

6.6.1 Procedures for sampling, randomizing, transporting, receiving, identifying, labelling, storing and handling of test materials shall be documented.

6.6.2 Where bulk material is prepared for a proficiency test, it shall be sufficiently homogeneous for each test parameter so that all laboratories will receive test materials that do not differ significantly in the parameters to be measured. The coordinator shall clearly state the procedure used to establish the homogeneity of the test item (see A1.4). Homogeneity testing shall be conducted prior to the dispatch of the test materials to the participating laboratories.

6.6.3 Where applicable, the coordinating laboratory shall also provide evidence that the test materials are sufficiently stable to ensure that they will not undergo any significant change throughout the conduct of the proficiency test. When unstable analytes need to be assessed, it may be necessary for the coordinating organization to prescribe a date by which the testing shall be completed, including required special pretesting procedures.

6.6.4 Coordinators shall consider any hazards that the test materials might pose and take appropriate action to advise any party that might be at risk (for example, test material distributors, testing laboratories, etc.).

6.7 *Choice of Test Method:*

6.7.1 The coordinator may instruct participants to use a specified test method. Such test methods are usually nationally or internationally-accepted standard test methods, and will have been validated by an appropriate procedure (for example, collaborative trial).

6.7.2 Participants may be able to use the test method of their choice, which is consistent with routine procedures used in their laboratories. Where participants are free to use a test method of their own choice, coordinators shall request details of the test methods used to allow, where appropriate, the use of participants' results to compare and comment on the test methods.

7. Operation and Reporting

7.1 *Coordination and Documentation*—The day-to-day operation of a program shall be the responsibility of a coordinator. All practices and procedures shall be documented. These may be incorporated in, or supplemented by, a quality manual (see Annex A2).

7.2 *Instructions:*

7.2.1 Detailed instructions covering all aspects of the program that should be followed by the participating laboratories shall be provided. These may be provided, for example, as an integral part of a program protocol.

7.2.2 Instructions shall include details concerning factors that could influence the testing of the supplied materials. Such factors shall include qualifications of operators, nature of the materials, equipment status, selection of test procedures and timing of testing.

7.2.3 Specific instructions on the recording and reporting of test or calibration results shall also be supplied (for example, units, number of significant figures, reporting basis, result deadlines, etc.).

7.2.4 Participants shall be advised to treat proficiency testing items as if they were routine tests (unless there are some special requirements in the design of the proficiency test which may require departure from this principle). They shall also be advised to avoid collusion with other participants.

7.2.5 Participants shall be advised to ensure that their laboratory capabilities are compatible with the protocols and test samples provided by the programs. Incompatibility between the program and its participants' capabilities can lead to inappropriate indicators of poor performance.

7.3 *Packaging and Transportation:*

7.3.1 The coordinator of the program shall ensure that packaging and methods of transport are adequate and able to protect the stability and characteristics of the test materials. There may be certain restrictions on transportation such as dangerous goods regulations, or customs requirements. In some cases, the laboratories themselves also take responsibility for the transport of the items, particularly in sequential measurement comparisons programs.

7.3.2 All appropriate customs declaration forms shall be completed by the coordinator to ensure that delays in customs clearance are minimized. The program shall comply with national and international regulations applicable to test item transport.

7.4 *Data Analysis and Records:*

7.4.1 The results received from the participating laboratories shall be entered and analyzed and then reported as soon as practicable. It is essential that procedures are implemented to check the validity of data entry and transfers and subsequent statistical analysis. Data sheets, computer back-up files, printouts, graphs, etc., shall be retained for a specified period.

7.4.2 Data analysis shall generate summary measures and performance statistics and associated information consistent with the program's statistical model and objectives. The influence of extreme results on summary statistics shall be minimized by the use of outlier detection tests to identify and then omit them or, preferably, by the use of robust statistics. Annex A1 contains some broad suggestions for statistical evaluations.

7.4.3 Program coordinators shall have documented criteria for dealing with test results that may be inappropriate for proficiency evaluations. For example, it is recommended that for analytes for which the test material has been shown not to be sufficiently homogeneous or stable for the purposes of a proficiency test, no grading or scoring shall be given for those analytes.