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Standard Guide for Planning, Carrying Out, and Reporting Traceable Chemical Analyses of Metals, Ores, and Related Materials[1]

This standard is issued under the fixed designation E 2053; 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 sets a protocol for generating and reporting measurements that are traceable to SI units or Certified Reference Materials in laboratories that serve the metals industries.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the application of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 135 Standard Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[2]

2.2 *Other Documents:*

ISO/IEC 17025:1999—General Requirements for the Competence of Testing and Calibration Laboratories[3]

ISO Guide 30—Terms and Definitions Used in Connection with Reference Materials[3]

International Vocabulary of Basic and General Terms in Metrology [VIM]; ISO: 2nd ed., 1993[3]

3. Terminology

3.1 *Definitions*—Except as defined as follows, for definitions of terms used in this guide, refer to Terminology E 135.

3.2 *certified reference material*—reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which established its traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence. (ISO Guide 30:1992)

3.3 *traceability*—property of the result of a measurement or the value of a standard whereby it can be related, with a stated uncertainty, to stated references, usually national or international standards, through an unbroken chain of comparisons. (ISO Guide 30:1992)

3.4 *Uncertainty (of measurement)*—parameter, associated with the result of a measurement, that characterizes the dispersion of values that could reasonably be attributed to the measured. (International Vocabulary of Basic and General Terms in Metrology [VIM] (ISO: 2nd ed., 1993)

3.5 *work plan*—a documented procedure intended for use by a laboratory to meet the measurement traceability requirements of a defined need.

4. Summary of Guide

4.1 A client shall prepare a request for traceable measurements and submit it to an ISO 17025 compliant laboratory for review and acceptance.

4.2 The laboratory shall prepare a work plan to meet the requirements and obtain concurrence with the client.

4.3 The laboratory shall make the measurements and issue a report.

5. Significance and Use

5.1 This guide establishes basic requirements which should be met by laboratories that generate and report test results that are traceable to SI units or to certified reference materials that are traceable to SI units. Traceability of chemical measurements is important because it provides a uniform basis for the comparison of results from different measurement systems and because it relates those results to our current knowledge of physical laws.

5.2 Many laboratories comply with ISO 17025 and participate in proficiency testing programs. Laboratories that are connected to the same accreditation bodies and proficiency test providers can be expected to report statistically similar results on the same sample. However, some test methods and some certified reference materials are not supported with data traceable to SI units. Therefore it is possible that fully compliant laboratories that are not connected to the same providers might report statistically different results if they used

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[2] *Annual Book of ASTM Standards*, Vol 03.05.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

the same, non-traceable test method on the same sample. This problem could be alleviated if competent laboratories used test methods and certified reference materials that were traceable to SI units.

5.3 Although some standard test methods and certified reference materials provide evidence of traceability to SI units, many others do not. Therefore, all laboratories cannot be expected to universally meet all requests for traceable analyses until the traceability of more test methods and certified reference materials is recognized through appropriate documentation.

5.4 The primary significance of this guide is that it establishes a consensus that, in order for a laboratory to generate traceable measurements, it must (*1*) have a clear understanding of the needs of the user of the traceable measurements, (*2*) comply with the internationally accepted quality-system requirements included in ISO 17025, (*3*) use test methods which have been shown to be traceable to SI units, and (*4*) be able to demonstrate that the laboratory was in statistical control at the time the measurements were made.

6. Procedure

6.1 The client shall prepare a request for traceable measurements and submit it to an ISO 17025 compliant laboratory for review and acceptance, as follows.

6.1.1 The request shall identify the source, sampling history, configuration, and approximate composition of the test materials, the traceable measurements to be made with their required maximum uncertainties, and identify special requirements, such as the need to certify that the material tested complies with a specific product specification or that traceability must be made to a specific standard or certified reference material. It shall also specify if any non-traceable measurements are to be included as part of the project.

6.1.2 The client shall select an ISO 17025 compliant laboratory and submit it to the laboratory for review. (Note 1)

NOTE 1—By reference, this guide incorporates the laboratory quality system requirements of ISO 17025. A laboratory that does not comply with ISO 17025 is considered to be noncompliant with this guide.

6.1.3 The laboratory shall review the request, work with the client to clarify the understanding of the need, and inform the client of its willingness and capability to proceed.

6.2 The laboratory shall prepare a work plan to meet the requirements and obtain concurrence with the client, as follows.

6.2.1 The work plan shall identify protocols for sample receipt, chain of custody, and sample preparation. It shall also identify all test methods to be used. All test methods used for traceable measurements shall have associated documentation showing their complete traceability to SI units. Traceability can be demonstrated either by direct measurement, use of pure materials which were assayed using methods traceable to SI units, or use of certified reference materials which were certified in a manner traceable to SI units. Not all of these approaches can be applied to all analytical measurement systems. If traceable standard methods are not available, the laboratory may propose to validate new analytical methods for this week. The work plan shall also document how non-traceable measurements, if required, are to be carried out.

6.2.2 The work plan shall describe the anticipated measurement uncertainty for the project to ensure that the estimated uncertainties in the final report values do not exceed the client's specified requirements.

6.2.3 The work plan shall identify matrix-matched certified reference materials or pure chemicals to be carried along with the traceable measurements. It shall also specify the maximum estimated uncertainties to be permitted in making these measurements. The laboratory staff shall make sure that maximum estimated uncertainties are attainable and that the published uncertainties associated with these materials are small enough to permit the attainment of the expected value (Note 2).

NOTE 2—The laboratory's ability to meet these anticipated uncertainties will indicate that the measurement uncertainties in the measuring laboratory were reasonable (3.4) while the traceable measurements were being made.

6.2.4 The work plan shall define the contents of the final report.

6.2.5 The work plan shall be reviewed and approved for presentation to the client by the laboratory's management. That review should include projected time and cost schedules, as needed, to meet local business needs.

6.2.6 The work plan shall be presented to the client for review. If revisions are needed, those revisions shall be reviewed and approved by the laboratory's management before being presented to the client for final acceptance. The work plan shall be accepted by the client, after which the laboratory can begin to carry out the work.

6.3 The laboratory shall make the measurements and issue a report, as follows:

6.3.1 The laboratory shall make measurements and document them in accordance with the work plan and ISO 17025. If, in the course of making the measurements, it becomes necessary to deviate from the work plan in any way that will affect the client or the client's use of the data, the laboratory shall inform the client. If necessary, the work plan shall be revised and approved as described in accordance with 6.2.

6.3.2 When the work is complete, staff shall prepare and issue a written report which shall include all of the material and measurement traceability documentation described in the work plan. In the event that the work included any method validation (6.1.1), those results shall be included. The report shall also comply with the reporting requirements of ISO 17025 (Note 3). It shall clearly differentiate between traceable and non-traceable measurements, if any were specified.

NOTE 3—It is recommended, but not required, that all copies of the original work plan be destroyed. Since the original work plan covered what was expected to occur and the final report describes what actually occurred, retaining the planning document may precipitate post-completion confusion about the integrity of the final report.

7. Keywords

7.1 certified reference materials; guide; measurement traceability