Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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Fitness for Purpose: Overview of ASTM Guides for Quality Assurance of Data from Nuclear Analytical Laboratories

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Abstract

Analytical laboratories in the nuclear industry must meet stringent requirements for the quality of their measurements. This is particularly true with measurements used for safeguards or nuclear material accountancy, but also for product assay and process control. Expectations for the nuclear industry go above and beyond those described in the ISO/IEC 17025:2017 standard that specifies general requirements for the competence of testing and calibration laboratories and is used as a basis for laboratory accreditation. Method uncertainty, for example, is more stringently controlled in the nuclear industry than in many other fields.

Reliance on international consensus standards is increasing due to the benefits they can provide, such as time savings in method development and consistency of results. Analytical test methods from ASTM International and the International Organization for Standardization (ISO) are well known and widely utilized. However, ASTM International has also developed a series of guides that describe best practices for ensuring the quality assurance (QA) of data from nuclear laboratories. These standards provide guidance on QA programs, qualification of measurement methods, preparation of working reference materials, calibration for nuclear measurement methods, and measurement system quality control (QC) programs. A key concept espoused in these standards is the concept of "fitness for purpose"; that is, ensuring that a measurement process produces data that enables correct decision-making by an end user for a specified purpose. Implicit in the concept is that the method has the appropriate level of rigor to meet data quality objectives – "appropriate" being neither too little nor too much rigor.

This paper presents an overview of the seven guides developed by ASTM International to address QA needs for nuclear analytical laboratories, including recent updates that have been completed or are currently in progress. These guides can be used to build a framework for ensuring the quality of data generated by these laboratories.

Introduction

Analytical chemistry measurements in nuclear facilities for safeguards, accountancy, product assay, process control, waste management, and decommissioning require strict QC and QA measures. For example, in the U.S. Department of Energy (DOE), DOE Order 474.2 specifies requirements for nuclear material control and accountability (NMC&A).[1] These requirements include a documented measurement control program which "must assure the quality of measurements made for MC&A purposes"; establishment of key measurement points; traceability and proper use of standards; control of measurement uncertainty; and a requirement that "Measurement methods are qualified, formally documented, periodically validated, and approved".

Similarly, ISO/IEC 17025:2017 specifies requirements for selection, verification and validation of methods.[2] This international standard:

- Recommends the use of "methods published either in international, regional or national standards";
- Requires laboratories to verify that they can properly perform the methods they intend to use; and
- Calls for validation of non-standard methods.

Standards development organizations (SDOs) such as ASTM International and the International Organization for Standardization (ISO) provide vehicles for the development of consensus standards that are used throughout the nuclear industry worldwide. These SDOs adhere to the World Trade Organization's (WTO) principles of international standards development: openness, transparency, impartiality and consensus, effectiveness and relevance, coherence, and development dimension.[3] These principles are achieved by:

- Bringing together people with a diversity of backgrounds, expertise, and knowledge;
- Providing a balanced representation of interests;
- Strict balloting and due process requirements to enhance quality; and
- Using a working group format to promote open discussion.

A previous paper reviewed practices and test methods from ASTM International and ISO pertinent to NMC&A measurements.[4] This paper provides a review of the seven ASTM International guides that address various aspects of qualification of measurements. Unlike standard practices and test methods, ASTM guides typically offer direction by providing information, or a series of options, used to determine a course of action, as opposed to a step-by-step procedure. The seven guides reviewed below are under the jurisdiction of ASTM International Subcommittee C26.08 on Quality Assurance, Statistical Applications, and Reference Materials, which originally developed them from 1986-1996 and continues to maintain and update them.

Interrelationship of the ASTM C26.08 Standard Guides

Figure 1, which is included in most of the C26.08 standard guides discussed in this paper, illustrates how they provide guidance for the typical process by which a measurement method is selected, validated, qualified, and then utilized under proper measurement control. The figure illustrates the steps in the process and also the importance of calibration and reference materials throughout the process. It is noteworthy that ASTM C1009[5], the guide for QA programs in nuclear laboratories, is not included in Figure 1. It is necessary to have a suitable QA program in place prior to method selection and validation. Since that precedes the process selection, validation, qualification and utilization, it is not included in Figure 1 below.

Quality Assurance Program Guidance: ASTM C1009

The nuclear analytical laboratory needs to consider a number of quality requirements for its overall operation. ISO 9001 provides requirements for quality management systems in organizations.[6] ASME NQA-1 provides the framework for quality assurance programs for nuclear facilities.[7] ISO/IEC 17025 provides requirements to demonstrate the competence of a testing or calibration laboratory.[2] ASTM C1009 provides guidance specific to nuclear analytical laboratories for the development of a QA program that considers the requirements in NQA-1, ISO/IEC 17025, and ISO 9001. This relationship is illustrated in Figure 2. In fact, ASTM C1009 contains a cross-reference table between these four documents. It describes, in the context of the nuclear analytical laboratory, the basic elements of a laboratory QA program such as organization; training and qualification; procedures; control of records, procurement, measuring equipment, and measurements; and addressing deficiencies and corrective actions.

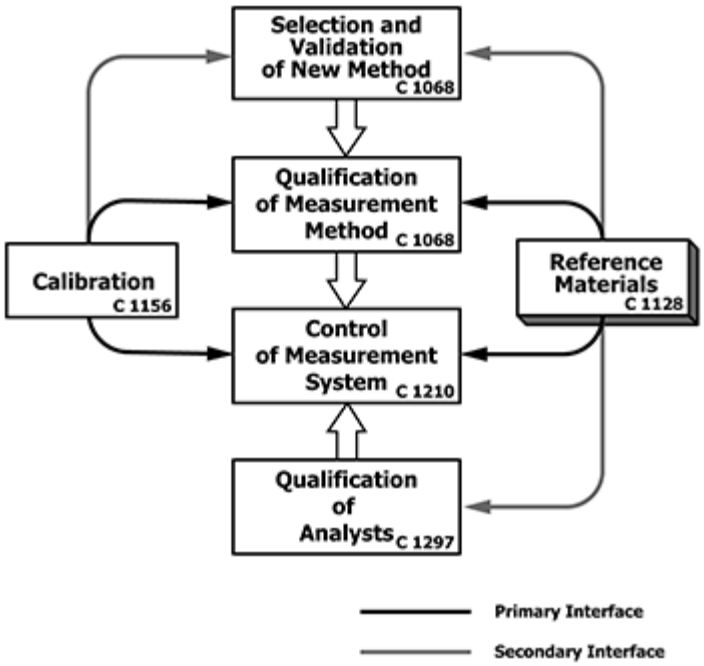

Fig. 1 – Interrelationship of ASTM C26.08 Guides for Method Selection, Qualification and Measurement Control[5]

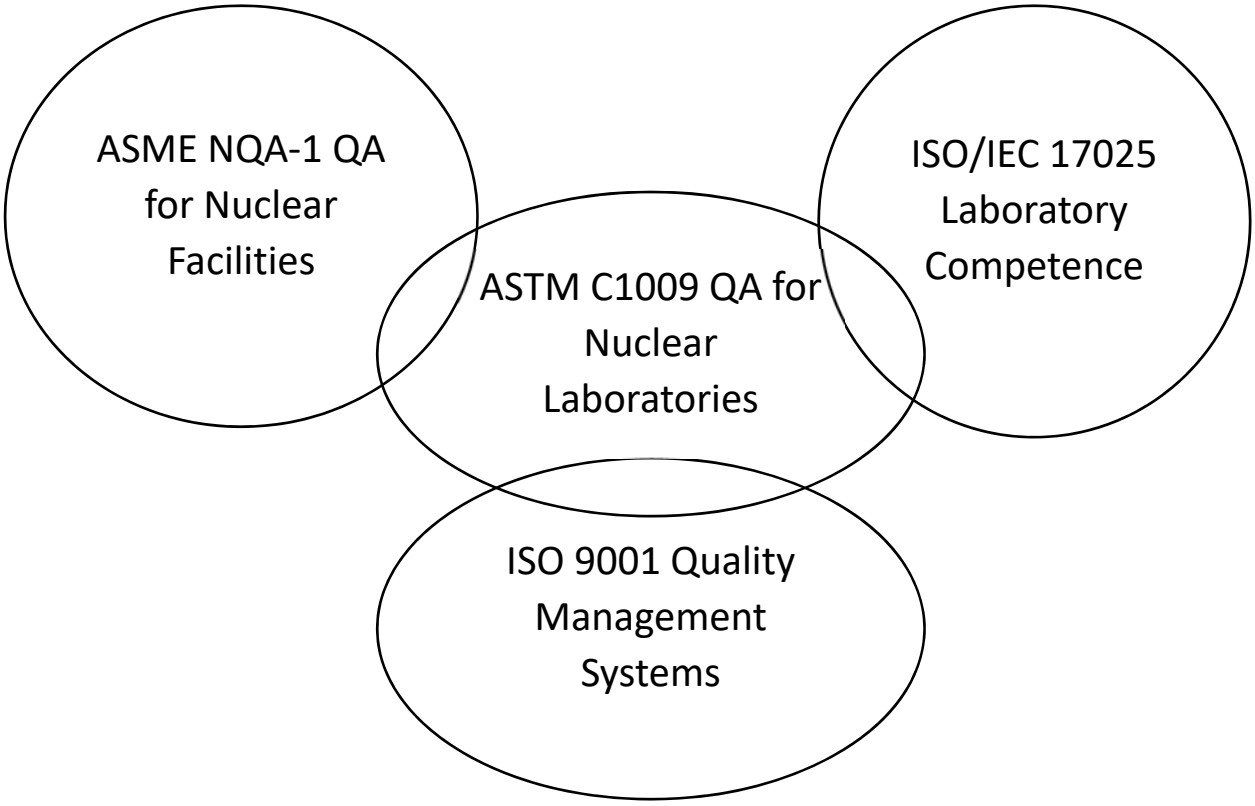

Fig. 2 – Interrelationship of ASTM C1009 with Key Quality Standards

When properly used, ASTM C1009 offers a framework for the development of those planned and systematic actions needed to provide confidence that the laboratory's mission is conducted in an acceptable and consistent manner. The laboratory's QA program becomes formal and visible through documentation that prescribes the applicable QA requirements and describes how they are implemented.

Since the most recent revision of ASTM C1009 in 2013, NQA-1, ISO 9001 and ISO/IEC 17025 have all been revised. The current versions of ISO 9001 and ISO/IEC 17025 are much more performance-based than their predecessors. ASTM Subcommittee C26.08 is in the process of updating ASTM C1009 to improve its harmonization with these revised standards.

The Concept of Fitness for Purpose: ASTM C1068

ASTM C1068, *Standard Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry*, provides guidance for demonstrating that a measurement method is fit for its intended purpose.[8] The concept of "fitness for purpose" is based on the International Union of Pure and Applied Chemistry's *Compendium of Analytical Nomenclature – Definitive Rules*,[9] and is defined as the "degree to which data produced by a measurement process enables a user to make technically and administratively correct decisions for a stated purpose." Put another way, it is the usability of the results produced by the measurement method that determine whether it is fit for the intended purpose. If, for example, the uncertainty of a given method is too large, it may not be possible to determine whether a data point falls within a limit or a specification, and the results may not be usable. This can be particularly important for measurement methods used for safeguards, accountancy, or nuclear safety applications.

ASTM C1068 contains a checklist to evaluate the fitness for purpose of a measurement method. While the checklist was developed primarily for safeguards and nuclear safety methods, it can be utilized to evaluate fitness for purpose for other methods. Fitness for purpose considers the needs to be met; qualification requirements; traceability to the International System of Units (SI); adequately calculated measurement uncertainty; appropriate QA and QC; and conformance to the current version of international standards.

ASTM C1068 was last revised in 2015, prior to the most recent (2017) revision of ISO/IEC 17025, and is now being reviewed for needed updates. For example, the standard uses the terms "validation" and "qualification" while ISO/IEC 17025:2017 uses "verification" and "validation"; the 2005 version used only "validation" in a manner similar to how it is used in ASTM C1068. Thus, harmonization of how these terms are used is a potential improvement opportunity for ASTM C1068.

Measurement System Quality Control: ASTM C1210

ASTM C1210 provides guidance for establishing a measurement system QC program.[10] The focus of the guide is on the technical aspects such as what constitutes a valid QC sample; analysis of those samples; and evaluating data from QC samples. Consideration is also given to measurement system calibration, ongoing qualification of a measurement system (noting that initial qualification is described in ASTM C1068), and maintenance of measurement systems.

Quality control is an important aspect of an overall measurement control program for a measurement system. Additional aspects of the overall measurement control program, such as sampling plans and storage, records considerations, personnel training and qualification, are described in ANSI N15.51.[11] Some of these aspects are addressed in ASTM C1009 or other C26.08 guides. A task group within ASTM C26.08 has reviewed ANSI N15.51 as well as other documents and has identified opportunities for improvement. ASTM C1210 will remain focused on QC but will include additional

information on acceptance criteria and proficiency testing, as well as improve harmonization of terms with ISO/IEC 17025:2017 as appropriate.

Working Reference Materials: ASTM C1128

Reference materials are the linchpin for ensuring traceability of a measurement method to the SI. As shown in Figure 1, reference materials are used throughout the lifecycle of a measurement method, from its selection to its verification, validation and qualification, and then the routine use of the method. ISO has developed a number of documents related to the development of reference materials; in particular, ISO 17034 provides for the competence of reference material producers[12], and ISO Guide 80 provides guidance for the use of quality control materials,[13] which are not metrologically traceable and have limited uses in nuclear analytical laboratories. While certified reference materials (CRM) are at the top of the reference material hierarchy, there is a class of reference materials that are not certified but that go through a comparable qualification process. For nuclear analytical laboratories, these are known as working reference materials (WRM) and the process for preparing them is described in ASTM C1128.[14]

A detailed description of the most recent revisions to ASTM C1128 has been provided in a separate paper.[15] A WRM is typically traceable to the SI through a CRM and can be produced by an analytical laboratory to meet its own internal needs. WRMs can serve to reduce the routine uses of CRMs, provide more exact matrix matching, or be adapted for a specific concentration range.

Development of a course on the use of WRMs based on ASTM C1128 has been described in a separate paper.[16] The course is currently in the final stages of development. Information obtained during development of the course has identified opportunities for future improvements to ASTM C1128.

The Last Three: ASTM C1156, ASTM C1297, and ASTM C1215

Calibration, like reference materials, is a vital function throughout the lifecycle of a measurement method, and is required by NQA-1, ANSI N15.51, and other standards. ASTM C1156 provides guidance for establishing calibration for nuclear measurement methods.[17] The guidance encompasses the content of calibration procedures; calibration standards (such as a CRM or, in some instances, a WRM); control of calibrated equipment; documentation; and general considerations. Last updated in 2018, there are no current plans for further revisions to this guide.

Qualification of laboratory analysts is a key component of the overall training and qualification requirements of NQA-1 and ANSI N15.51. ASTM C1297 addresses the qualification of analysts for measuring chemical analysis or physical measurements of nuclear materials.[18] The guide emphasizes the method-specific aspects of training and qualification, including the demonstration of proficiency (e.g., by analysis of reference materials or QC samples) and statistical tests that can be applied to the evaluation of analyst data.

The seventh C26.08 guide, ASTM C1215,[19] is not shown in Figure 1. It provides guidance for precision and bias statements in nuclear test methods. This guide is primarily used in the development of ASTM test methods for the nuclear fuel cycle since ASTM International requires test methods to provide a statement of precision and bias. The guide acknowledges some of the difficulties in performing inter-laboratory studies involving nuclear materials (e.g., inability to transport samples to participating laboratories, lack of reference materials in some instances). It provides discussion on statistical concepts and sources of variation; confidence intervals; error models; and representative versus random sampling. The guide is currently in the process of being revised.

Conclusion

The guides developed and maintained by ASTM International Subcommittee C26.08 provide information for nuclear analytical laboratories in establishing a comprehensive quality assurance program, including administrative program requirements; selection, verification, validation, and qualification of nuclear measurement methods; quality control; calibration; use of reference materials including proper preparation of working reference materials; and qualification of laboratory analysts. These guides, originally developed over a ten-year period (1986-1996), have been updated as nuclear measurement science and associated requirements have evolved. Four of the seven guides are in some phase of revision presently. A small but dedicated group of subject matter experts (SMEs) from around the world is participating in these revision efforts. SMEs who participate in ASTM, ISO, ANSI, and other standards development organizations are the backbone of ensuring consistency in nuclear analytical laboratory measurements worldwide.

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