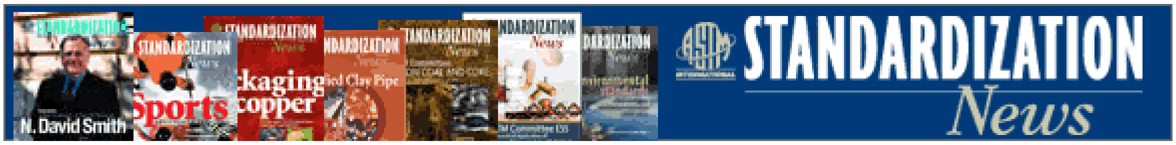

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John Sieber is a research chemist in the NIST Analytical Chemistry Division. A specialist in x-ray fluorescence spectrometry, he is a member of ASTM Committees F40 on Declarable Substances in Materials and E01 on Analytical Chemistry of Metals, Ores, and Related Materials. Dr. Sieber has been employed by NIST for more than 16 years.

Creating Standard Reference Materials for Testing Declarable Substances in Materials

by John R. Sieber

The mission of the National Institute of Standards and Technology is "to develop and promote measurement, standards, and technology to enhance productivity, facilitate trade, and improve the quality of life." Better measurements facilitate trade by helping producers and consumers agree on the properties of a product. Better measurements improve the quality of life when producers and consumers can minimize hazardous and suspect constituents to help prevent them from entering the environment. Measurements are improved by developing more rigorous test methods and the reference materials to validate test methods.

To learn what measurements must be improved, NIST must interact with experts from industry, academia, regulatory bodies, and other informed parties. Organizations such as ASTM International are among the best places to find these experts. By taking part in ASTM activities, NIST scientists learn first-hand what metrology issues are important to the private sector. They learn how the private sector measures the properties of their products and what difficulties are encountered. Together, both parties can prioritize the issues and work toward solutions.

NIST representatives combine what they learn with information from other sources to propose scientific research programs and reference material development projects intended to provide the private sector with better tools for chemical metrology. For this reason, NIST has participated in the creation of new ASTM Committee F40 on Declarable Substances in Materials.

The members of Committee F40 have created a committee focused on the technical difficulties faced by the private sector as they cope with the accelerating promulgation of regulations such as the European Union's Restriction of Hazardous Substances (RoHS) Directive. Committee F40 is somewhat unique among ASTM technical committees in that its scope does not limit it to any particular industry, analytical technique, compound, type of material, or product. The committee is interested in the targeted substances within whatever raw materials and intermediate materials are used in consumer products and their components. Committee F40 intends to create methods, guides, and practices for the metals, plastics and polymers, and semiconductor and electronics industries as well as any other industry that contributes to consumer products.

Many industry sectors already have standards writing bodies working on test methods, product specifications, etc., but not necessarily for the purpose of conforming to RoHS or related regulations. To succeed, F40 must cooperate with other ASTM committees to enumerate and prioritize the needs for test methods and reference materials. The speed with which F40 was created and the variety of organizations represented by the more than 100 international members are strong evidence of the scope of the regulations and the force of their impact on the private sector. It is this breadth of membership that makes an ASTM committee such as F40 a good source of information for NIST.

The Development of SRMs

Arguably, consensus-based committees are a source of relatively unbiased information. In an ASTM committee all members have an equal voice. Therefore, programmatic decisions made by NIST on the basis of said information can be made with a higher degree of confidence that they are correctly targeted to help the majority of committee members. To obtain funding for a project, especially the development of a Standard Reference Material®, the NIST scientist who champions it must create a proposal that clearly demonstrates the need for the investment.

Key elements of a proposal include a statement of the work to be performed, the technical impact of the project, the economic impact of the problem and the expected solutions, the human impact of the project (e.g., healthcare), information about the expected users of the SRM, and the resources available for the project. Much of the required information can only be obtained from industry experts. In some cases, the experimental design of an SRM project includes work to be carried out in private sector laboratories. The private sector shares in the workload and financial burden of methods research and reference materials programs through the direct involvement of its experts. Those experts design and carry out experiments in direct cooperation with NIST scientists.

The prioritization of technical needs and identification of materials represent the beginning of the process of SRM development. In the area of chemical metrology, the NIST Chemical Science and Technology Laboratory prioritizes and carries out the development of SRMs proposed by CSTL scientists. Typically, a proposal for a new SRM (i.e., a material with which NIST has no prior experience) would include a request for development funds with which to study the materials and select the best ones for further development. Good candidate materials must have a homogeneous distribution of constituents of interest, have concentration levels appropriate for the validation of methods, and be available in sufficient quantity to meet the anticipated demand. In addition, the initial development phase must include an evaluation of available test methods. Because certification requires measurements by NIST, the measurement capability must be demonstrated before a new SRM can go into production. Methods must determine each constituent or property with sufficient accuracy to meet the need. The definition of sufficient accuracy depends strongly on the method validation and reporting requirements of the users of an SRM.

SRMs Both Planned and Under Way

With regard to substances of interest to Committee F40, the initial focus is on hexavalent chromium, arsenic, cadmium, mercury, lead, and certain flame retardants containing bromine. Members of Committee F40 have already requested SRMs for these important elements and compounds in polymers, alloys, and coatings. An effort is already under way to identify sources of poly(vinyl chloride) and acrylonitrile butadiene styrene. Existing SRMs for metals, minerals, and environmental materials are being evaluated for their suitability. Some may already be certified for constituents of interest while others may contain useful concentrations that can be certified.

NIST capabilities for the determination of hexavalent chromium, arsenic, cadmium, mercury, lead, and flame retardants are at various stages of development. CSTL has long recognized the importance of these constituents. Efforts are continually being made to improve the state of the art of chemical metrology for these elements and compounds. Elemental analysis methods of high accuracy have been available for years.

Noteworthy among these are isotope dilution mass spectrometry methods for total chromium, lead, cadmium, and a recently developed method for mercury. These elements plus arsenic can also be determined using inductively coupled plasma mass spectrometry, inductively coupled plasma atomic emission spectrometry, instrumental neutron activation analysis, X-ray fluorescence spectrometry, and glow discharge atomic emission spectrometry. Speciation methods are under development. Some have been realized, including one for the determination of methylmercury. Methods for the determination of individual flame retardant compounds have recently been developed and are currently being used to certify these compounds in a variety of matrices.

The mention of analytical test methods brings the discussion back to the NIST mission statement.

Improving measurements is often accomplished by developing test methods. As a member of an ASTM committee, the NIST representative participates in the methods development activities of the committee. This includes participation in ruggedness tests and interlaboratory studies to establish the performance characteristics of a draft test method. In certain cases, NIST has supported special round robin programs to establish the general level of capability in private sector labs and to demonstrate a technical problem that NIST could help solve. NIST has significant resources for the development of new test methods. In fact, the development of new tools for chemical metrology is at the core of NIST activities.

The activities and issues discussed in this article help to define the role of a NIST scientist who interacts with ASTM International committees. Information sharing enables NIST to learn about the technical needs of the private sector. NIST members support the development of ASTM methods through participation in method writing and performance testing. NIST has the resources to develop new methods and new technology to remove barriers to technical advancement and trade. NIST and private sector scientists identify, plan, and carry out SRM development activities. These interactions occur because ASTM committees are recognized as important sources of information about the technical issues facing U.S. industry. //