NIST Standard Reference Materials: Supporting Metrology and Traceability for the Forensic Science Community

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The ability to measure precisely and consistently is the Great Enabler of modern technology. The reliability, safety and interoperability of today's devices and systems, the foundation of international trade and global manufacturing, even the veracity of evidence presented in court can be traced to the fact that a volt is measured by the same standard everywhere across the power grid, that a gram in a laboratory in Leipzig is identical to one on an assembly line in Lansing, that a degree Celsius is a degree Celsius whether measured in a clean room or a court room.

We stand on this pinnacle thanks to more than a century of determined effort by thousands of talented men and women, many associated with organizations such as ASTM International and the National Institute of Standards and Technology (NIST). A non-regulatory agency of the U.S. Department of Commerce, NIST was established by Congress as the National Bureau of Standards in 1901 to improve the competitiveness of American industry by setting and maintaining the Nation's measurement standards and advancing the measurement science that underpins those standards. Within this charter, NIST's mission has expanded to promote U.S. innovation and industrial competitiveness by advancing measurement science, standards, and technology in ways that enhance economic security and improve our quality of life. Today, the Institute and its 3 000 employees -- among them many of the world's leading scientists and engineers, including Nobel laureates, and more than 1 500 annual guest researchers -- enjoy a worldwide reputation as both a premier metrology research team and an unbiased referee in disputes involving measurements and standards.

NIST Standard Reference Materials®

NIST continually defines and refines physical and chemical measurements. It pushes back the limits of accuracy and precision - developing innovative methods for measuring time, length, mass, energy, electrical current, resistance, chemical composition, and the other quantities on which science, technology and commerce depend. Just as important, the Institute transfers the results of its research to those who rely most on metrology by providing reference data, calibration services, and reference materials. These services support disciplines as varied as industrial materials production, environmental analysis, radiation threat detection, and health measurements. They enable manufacturers, scientific and technical organizations, Government agencies, academia, and commercial concerns to perform required measurements accurately and consistently.

Among NIST's measurement services programs, that of developing and providing reference materials is the largest.[1] The International Vocabulary of Metrology (VIM) published by the International Standards Organization (ISO) defines a reference material (RM) as one or more materials or substances whose properties are sufficiently homogeneous and well established to be used for calibrating apparatuses, assessing measurement methods, or assigning values to materials. The VIM defines Certified Reference Material (CRM) as an RM, one or more of whose property values are certified as traceable 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 [1]. A NIST Standard Reference Material® (SRM) is a CRM that meets NIST-specific certification criteria and is issued with a certificate of analysis that details its characteristics and provides information on its appropriate uses. NIST certificates of analysis contain certified values and uncertainties in which NIST has the highest confidence because it has fully investigated or accounted for all known or suspected sources of bias.

NIST issued "Standard Samples" almost immediately after its creation. In 1910 it began to use the SRM name and current numbering system. Since issuing its first SRM (*SRM - 1 Argillaceous Limestone*), the Institute has developed more than 4 900 SRMs, many of which have been replaced, updated, or discontinued as the world's measurement requirements have changed. Today, NIST's active catalog offers over 1 300 SRMs, and 30 to 40 new or updated materials are added each year.

<u>NIST SRM Development and Production</u>

The need for ever more precise standards of measurement is insatiable, propelled by rapid advancements in technology and manufacturing processes, the expanding rules of international commerce, and pressing requirements in crucial areas such as homeland security. In response, NIST has devised a process that brings together its diverse resources to meet the challenge. Oversight responsibility for the process is provided by NIST's Measurement Services Advisory Group (MSAG), comprising the directors of NIST's laboratories involved with measurement services. NIST's technical labs assess customer needs, set SRM priorities and perform the technical work. NIST's Measurement Services Division (MSD) performs the business, administrative, and product support functions necessary to deliver SRMs to customers. These functions include specialized material processing, packaging, labeling, pricing, warehousing, sales and distribution services for all SRM products. And NIST's Statistical Engineering Division assists in the design of sampling and measurement strategies, consults on issues involving certified values and uncertainty estimation, and develops standardized statistical design and analysis templates used by the labs for SRM projects.

There are three phases to SRM development. The first is research - the constant effort to discover ways to entice nature to give up its secrets and ongoing investment in advanced

[1] See http://www.nist.gov/srm.

facilities, equipment and the best people. Without these, NIST would be unprepared when the need for an SRM or other measurement service arises. Research activities are funded primarily by the annual Congressional appropriation process.

The development phase begins when customer needs and market assessments indicate that an SRM is required to address barriers to innovation or the competitiveness of U.S. industry. Development is triggered when the NIST technical labs are satisfied that a broad customer base will benefit from the SRM and that the benefit is really something that NIST should provide. NIST involvement is usually indicated when U.S. industry must be able to link measurements of quantities to the International System of Units (the SI), which NIST's measurement services are specifically designed to do. This traceability leads to worldwide acceptance of U.S. product measurements and specifications, and the technical design of an SRM certification effort is defined by the degree of accuracy required by the intended user community. Many commercial and manufacturing applications, for example, do not require state-of-the-art measurements at the ultimate limits of NIST capability. Accurately matching measurements to customer requirements saves unnecessary costs on all sides and results in SRMs that are practical and affordable.

Once NIST labs are satisfied that an SRM is warranted, the development of a prototype material is undertaken. The purpose of prototype development is to ensure that NIST's measurement expertise can be transferred through a stable artifact. Suitable sources of the specific material are investigated, keeping in mind the required range for the intended measured quantities (the *measurands*) and the target levels of uncertainty in the resulting certified values. Measurement methods both for assessing material homogeneity and for assigning certified values and uncertainties to the measurands are tested.

Cost is an important consideration in any SRM program. Development efforts are funded in part by a service development surcharge that is collected through the sale of NIST's current SRMs in stock. Partnerships with other federal agencies and standards organizations are also used to fine tune the measurement design to meet customer needs while controlling costs. Special programs such as the NIST Traceable Reference Material[2] program provide a mechanism for linking commercially produced reference materials to NIST primary standards.

When all research and development questions have been answered and NIST is confident that a candidate material can be obtained and characterized to meet the technical specifications of the SRM design, *production* begins. Under the rules if its current Congressional authorization bill, NIST recovers all SRM production costs through the sale of these products to end users. The Institute has decided that the production phase should take no longer than three years, and that production costs should be recovered through sales within five years. So the unit price of an SRM is set by the production costs, operation surcharges, and the projected sales rate. This ensures that SRM development funds are recovered in a reasonable period and so are available for new high-priority SRM projects. It is a system that has led to an effective process for identifying the highest priorities of the user community and channeling funding and resources

[2] See: http://ts.nist.gov/ts/htdocs/230/232/COMMON/NTRM.HTM

where they are needed most.

Using this process, NIST produces and sells over 30 000 SRM units per year with value of over $8 million.

A Case Study: NIST Standard Bullets and Casings

NIST has served as technical consultant to the criminal justice system since 1912 and, in fact, trained the FBI's first forensic scientists when the Bureau opened its Crime Laboratory in 1924. In 1971 NIST established its Office of Law Enforcement Standards (OLES) to provide law enforcement and public safety agencies with measurements, standards and technical guidance.

OLES is particularly active in the area of forensic sciences, developing tools and techniques that help investigators collect, manage and analyze material from crime scenes in ways that meet evidentiary requirements established by the courts. Among these tools are a number of SRMs, including materials for:

- calibrating instruments and methods used to measure blood-alcohol levels
- verifying methods used to detect drugs of abuse in urine and hair
- identifying residues of additives in smokeless gunpowder and residues of ignitable liquids used by arsonists, and
- standardizing forensic DNA analyses and providing quality control of polymerase chain reaction (PCR) procedures and the sequencing of human mitochondrial DNA (mtDNA).[3]

Among the forensic SRM efforts that have drawn the most recent attention is the NIST Standard Bullets and Casings project.

The Federal Bureau of Investigation (FBI) reports that, in 2004, firearms were used in the commission of more than 1.2 million crimes in the United States. This makes solving firearm-related crimes a primary national concern, and ballistic comparisons that match ammunition to firearms are among the most useful tools in this effort.

When a firearm is discharged, the gun barrel striates the surface of the bullet and the gun's firing pin, breech face, and ejector imprint the bullet casing with distinct markings. These markings comprise a "bullet signature" and a "casing signature" that are unique to each firearm. By analyzing these signatures, forensic examiners can connect a firearm to a criminal act. In the early 1990s, the Bureau of Alcohol, Tobacco and Firearms (ATF) and the FBI each established its own system for imaging the signatures of bullets and casings found at crime scenes, analyzing those images, and storing them in a centralized database. State and local law enforcement agencies can image bullets and casings and access the central database through the Integrated Ballistics Identification System (IBIS), enabling them to compare bullets and casings found at different crime scenes, establish connections, and build cases against suspects.

[3] Information on NIST's SRMs related to the Forensic Sciences can be found at https://srmors.nist.gov/tables/view_table.cfm?table=115-1.htm

In 1997 ATF and the FBI agreed to create a single system and national database of bullet and casing signatures, the National Integrated Ballistics Information Network (NIBIN). Two key requirements for this effort were to unify ballistics measurements among the hundreds of forensic laboratories nationwide and to establish a reliable method for tracing measurements and ensuring their accuracy through the system. Based on its long-standing research in realizing the SI unit of length, NIST undertook the task of helping the criminal justice community harmonize ballistics measurements nationwide. NIST Standard Bullets and NIST Standard Casings are being developed to help fulfill these requirements.

NIST Standard Bullet SRM 2460 is in its final certificate review and user acceptance testing. The SRM consists of both a physical standard and a virtual standard. The physical material is the bullet itself, the tool for verifying the accuracy of imaging equipment and signature comparisons [2]. The virtual standard is a set of six digitized bullet profile signatures that provides the information used to machine the bullet signatures on the physical standard [3-4]. The digitized profiles serve as the template for producing the physical SRM bullets. The SRM is intended for use in calibrating IBIS equipment and verifying ballistic measurements.

At the outset of the project, NIST established four basic technical requirements for its standard bullets:

- **Bullet surface characteristics** - The shape, size, material and color of the physical standard bullets must be as close as possible to those of real bullets.
- **Repeatability and reproducibility -** The signatures on the SRM standard bullets must show such a high degree of repeatability and reproducibility that when the bullets are distributed nationwide for instrument calibrations, they function as a single bullet.
- **Measurement traceability -** NIST's measurements of the SRM bullet signatures must be traceable to the SI unit of length. Bullet signature measurements performed at local laboratories must be traceable to measurements of SRMs maintained at the ATF and FBI central laboratories.
- **Information technology** - The bullet signatures for the SRM standard bullets must be digitized so that they can be used to produce identical standard bullets at any time.

The development phase of the work began in 1998, when NIST's Precision Engineering and Manufacturing Metrology Divisions machined the first two prototype standard bullets, each bearing six reference bullet signatures. Tests showed the signatures on the manufactured bullets to be highly uniform and reproducible. From 1998 to 2001, NIST collected comments on these prototype standard bullets from firearm examiners at ATF and the FBI, and improved both the design and manufacturing process for the physical standard bullets. Further work was necessary to establish a traceable measurement parameter that would account for the small differences between the 2D virtual standard and its 3D realization on the standard bullets.

The development phase of this work continued with the ATF National Laboratory Center and the FBI Central Laboratory each providing three master bullets, which had been fired from different guns under standardized firing conditions. NIST's Surface Calibration Laboratory used a commercial stylus instrument to perform a profile measurement of each bullet, and the resulting set of six digitized two-dimensional bullet signatures was stored in a NIST computer as the 2D virtual bullet signature standard.

To produce the SRM, the virtual bullet signature standards were used to control the tool path of a numerically controlled diamond turning machine at the NIST Instrument Shop. Six bullet signatures were machined on each of the SRM 2460 standard bullets [3]. The material is OFHC copper rod with about a 1 mm thick pure-copper coating. After machining, the NIST Metallurgy Division applied a commercial corrosion protection to the surface of the SRM bullets.

Figure 1. A NIST SRM 2460 standard bullet is mounted on a blue stub. One of the six highly uniform, land engraved areas (trapezoidal in shape) is clearly visible.

A similar process is being used to develop and manufacture standard casings from five master casings supplied by the ATF National Laboratory Center. From these masters, two different manufacturers produced 21 prototype standard casings. IBIS was used to test these prototypes, with comparisons made between markings from firing pins, ejectors, and breech faces. The results demonstrated a high degree of reproducibility and close agreement between compared images. Based on these initial results, NIST is improving the design in preparation for fabrication and distribution.

Summary

NIST Standard Reference Materials have long played a central role in industry and commerce, and more recently in areas such a criminal justice, public safety and homeland security. NIST has earned this role by consistently matching design, development and production to customer needs, incorporating innovation, ensuring quality, and providing outstanding follow-up support. And seeing the growing need for reference materials - in industry, commerce, and beyond - NIST is already developing capabilities that will lead to the next generation of measurement standards.

[1] International Vocabulary of Basic and General Terms in Metrology (VIM), 2nd Edition; BIPM/IEC/IFCC/ISO/IUPAC/IUPAP/OIML, International Organization for Standardization (ISO), 1993.

[2] Song, J., Vorburger, T. and Ols, M., "Establishment of A Virtual/Physical Standard for Bullet Signature Measurements," Proc. 2001 NCSL, Washington, D.C., August 2001.

[3] Song, J., Whitenton, E., Kelley, D., Clary, R., Ma, L., Ballou, S. and Ols, M., "SRM 2460/2461 Standard Bullets and Casings Project", J. Res. Natl. Inst. Stand. Technol. 109, 6, 2004, pp 533-541.

[4] Ma, L., Song, J., Whitenton, E., Zheng, A., Vorburger, V. and Zhou, J., "NIST Bullet Signature Measurement System for SRM (Standard Reference Material) 2460 Standard Bullets", J. Forensic Sci., 49, 4, July 2004, pp 649-659.

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