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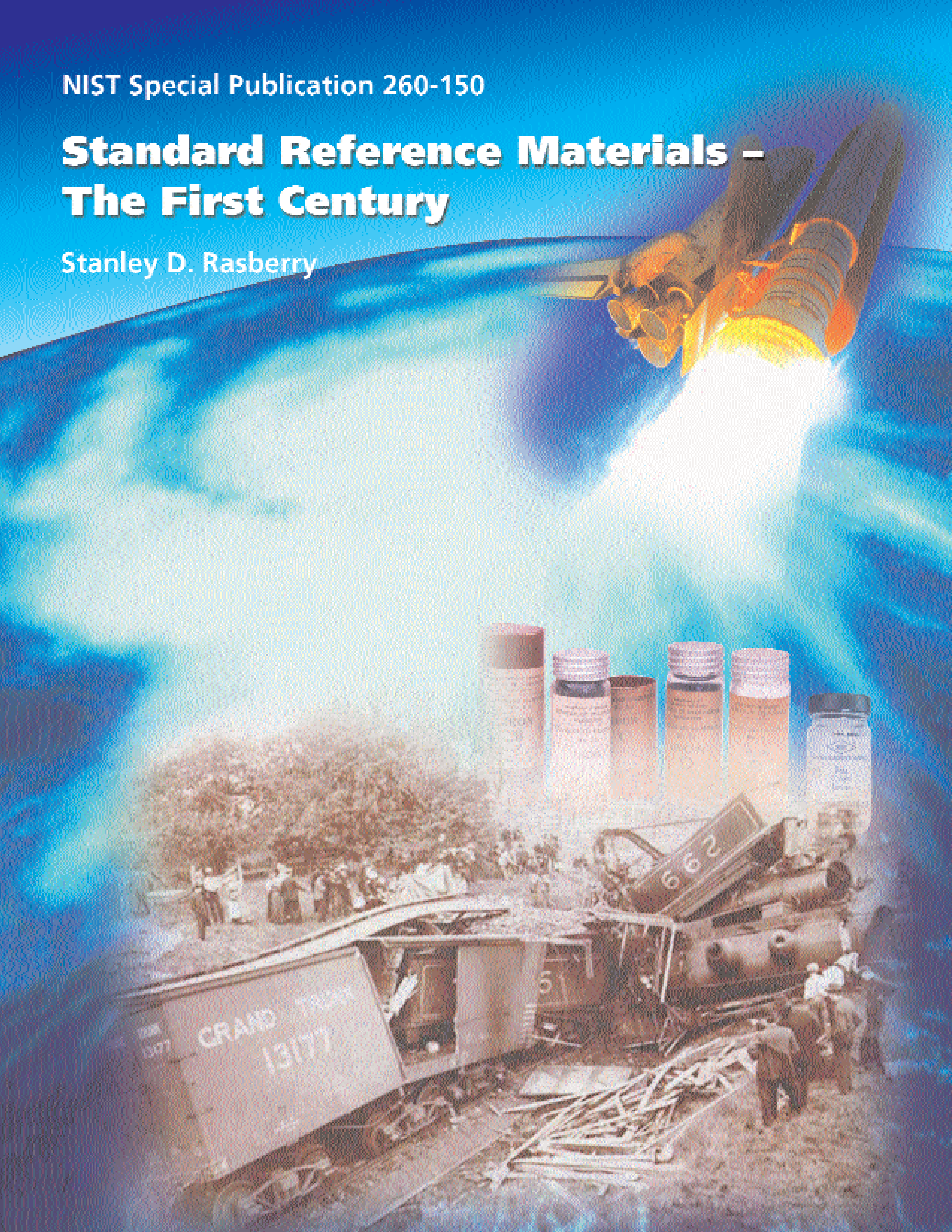
Standard Reference Materials – The First Century

Stanley D. Rasberry



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Standard Reference Materials - The First Century

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Foreword

Standard Reference Materials have played a major role in the history of the National Institute of Standards and Technology (NIST), as well as its predecessor organization, the National Bureau of Standards (NBS). Standard Reference Materials (SRMs) were one of the first tangible outputs from the nation's investment in improved measurement standards and technology that was started at the beginning of the 20th century. As NBS evolved over the last hundred years in terms of scientific capability and fields of work, SRMs have taken on new forms and new roles in ensuring that our Nation is second to none in measurement capability.

No one is more familiar with the history of this important program than Stanley Rasberry, longtime Chief of the program as well as a major developer of SRMs himself. In this retrospective, Raspberry captures the spirit and importance of the program for analysts, researchers and technologists everywhere. The reader will find this lively exposition both informative and enlightening about of NIST's most important programs.

John Rumble
August 2002

Acknowledgment

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Summary

Over the course of its first one hundred years, the National Institute of Standards and Technology (NIST) has made numerous contributions to advancing the science and practice of analytical chemistry. Contributions to fundamental constants and reference data, such as determination of the Faraday, Avagadro's number, and atomic masses, began at almost the beginning of this institution when it was formed in 1901. Instrumentation development, improvement, and reproducible methods for their use have also been an important part of the NIST effort.

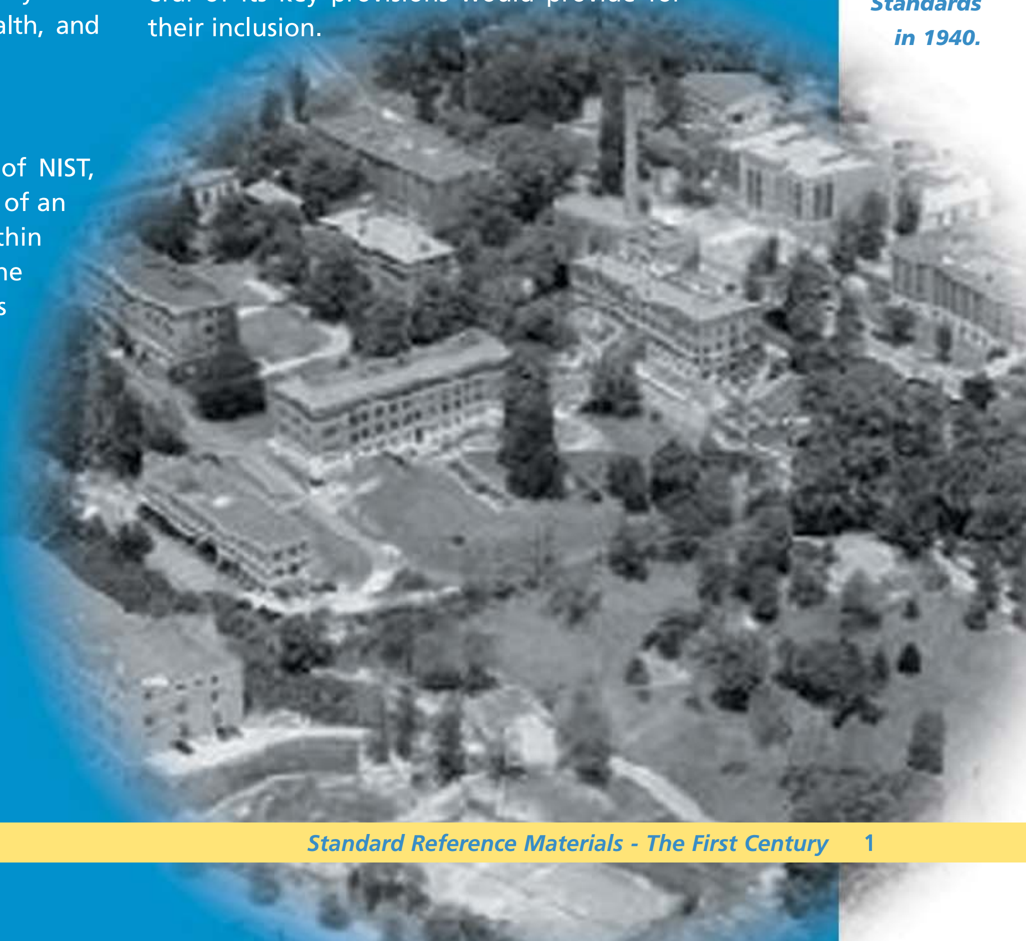
This publication describes what may be the organization's most important and certainly its most unique contribution; namely, certified reference materials. Ultimately these certified reference materials would become known at NIST as standard reference materials (SRMs). This contribution has now been mirrored around the world with reference materials being certified in at least 25 countries and routinely applied in more than twice that number. The result has been more accurate analyses of materials that impact our safety, health, and well-being.

Background

While celebrating the first century of NIST, we must note that the contributions of an organization can only originate within the minds and then hard work of the people of that organization. This is important to remember because in a work as short as this one, it is quite impossible to give fair recognition to the thousands who have produced the contributions. It is those people who are remembered whenever NIST or "the Bureau" are mentioned.

The term Bureau is appropriate to cover the first 87% of the century. In 1901, the agency originated as the National Bureau of Standards (NBS), but had its name abridged to simply Bureau of Standards in 1903. "National" was restored to the name in 1934, to differentiate it from the many state-level bureaus of standards which had been established. There was no further change until 1988, when the current name National Institute of Standards and Technology was received. While the name has changed three times, the character of the place has formed largely around one theme. That theme is to support the development of accurate measurements essential to science and technology. Almost as a corollary, work on the measurements has led to advancements in the technologies themselves.

The mission of the new agency was defined in its "organic act" legislated by the U.S. Congress. While that very brief act makes no specific mention of reference materials, several of its key provisions would provide for their inclusion.



Aerial view of the National Bureau of Standards in 1940.

The entire charge to the new agency was found in six provisions:

- custody of the standards;
- comparison of the standards used in scientific investigations, engineering, manufacturing, commerce, and educational institutions with the standards adopted by or recognized by the Government;
- construction, when necessary, of standards, their multiples and subdivisions;
- testing and calibration of standard measuring apparatus;
- solution of problems which arise in connection with standards; and,
- determination of physical constants and the properties of materials, when such data are of great importance and are not to be obtained of sufficient accuracy elsewhere.

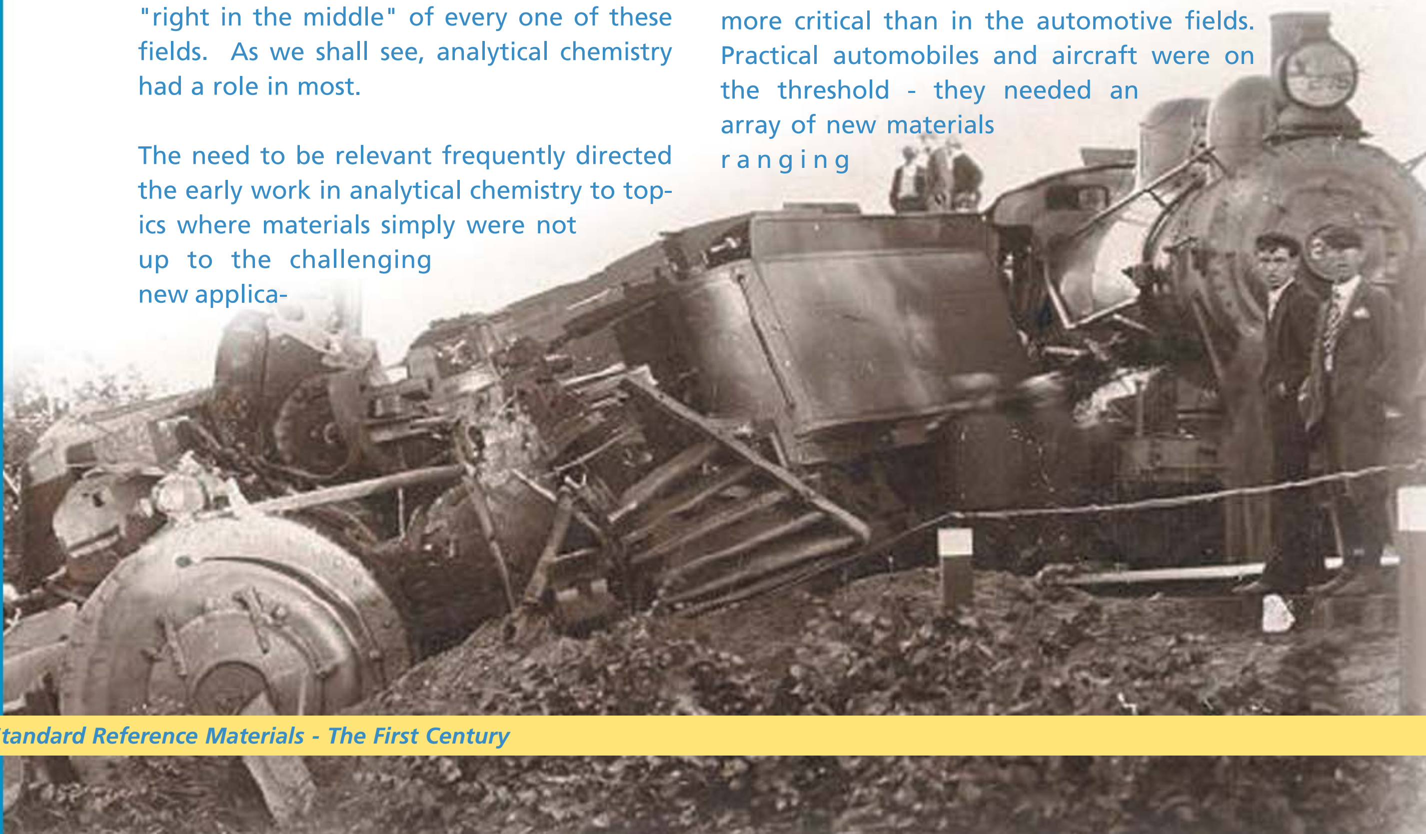
The Bureau could have emerged at no more opportune time. It was the dawn of virtually every technology that we know today - automobiles, airplanes, modern ships and locomotives, steel construction, motion pictures, practical radio, and subsequently, television, space craft, computers, and information technology. In the early days, the Bureau was "right in the middle" of every one of these fields. As we shall see, analytical chemistry had a role in most.

The need to be relevant frequently directed the early work in analytical chemistry to topics where materials simply were not up to the challenging new applications. With these enormous needs in mind, the Congress of the United States gave careful deliberation to the staffing of the new Bureau. Initially, it decided it would not be sufficient to have a director, a physicist and two assistant physicists; a chemist and two assistant chemists also would be allocated. Later, in 1901, the expense of constructing the new laboratory and the scarcity of funds caused the Congress to cancel the two assistant chemist positions. Thus in its first year, the young agency began trade-offs between staff and facilities that last to this day.

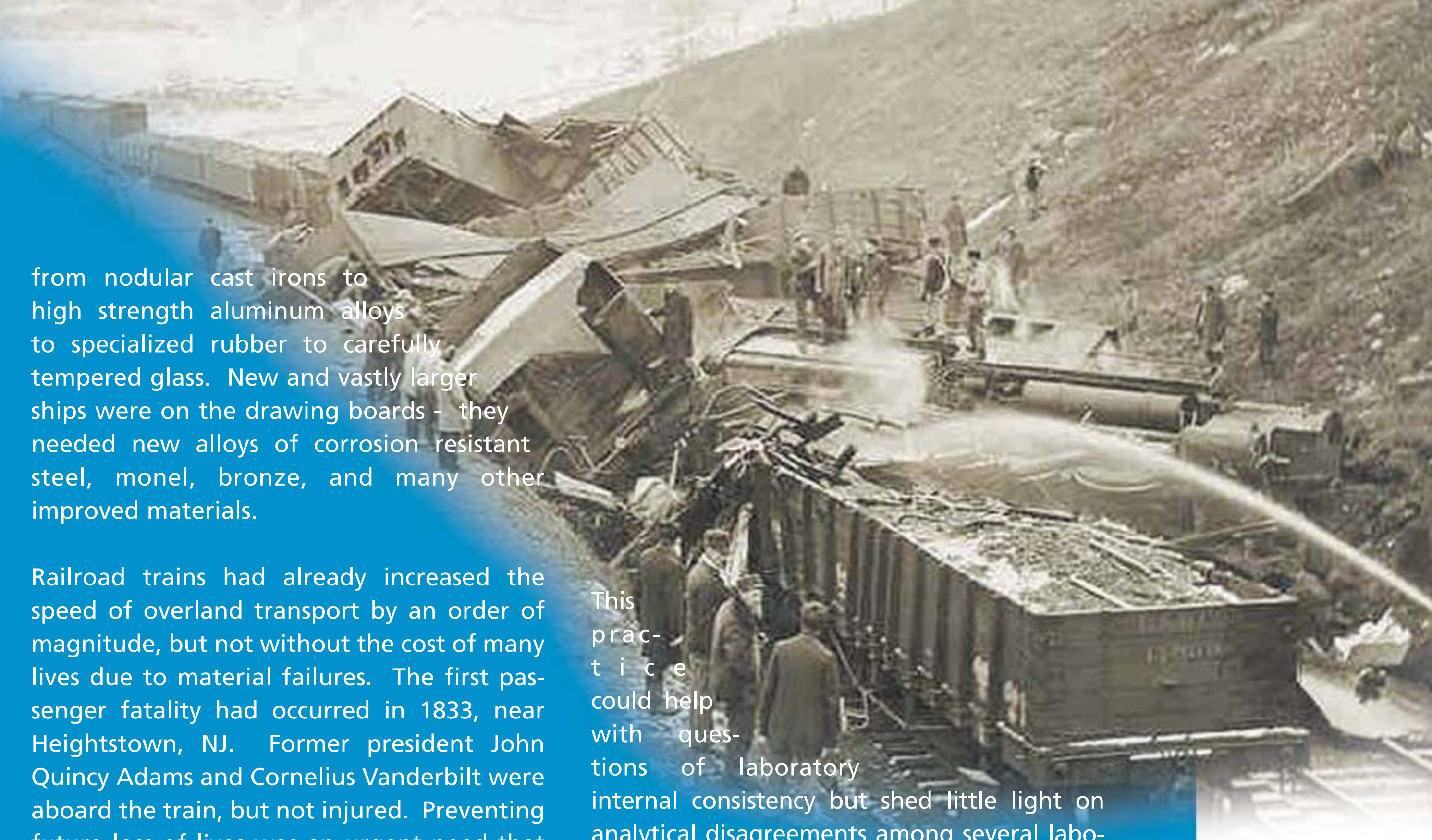
An Urgent Need

Many of the greatest technical challenges of the early twentieth century were related to materials and their performance. Construction of skyscrapers and suspension bridges would require new and stronger alloys of steel, and better quality control for Portland cement used in concrete. Tungsten alloy performance would become critical to vacuum tubes for lighting and electronics. Copper alloys would figure heavily in wiring for communications and in valves and fittings with nearly endless configurations.

Perhaps the material concerns were nowhere more critical than in the automotive fields. Practical automobiles and aircraft were on the threshold - they needed an array of new materials ranging



Broken axles and car wheels caused numerous derailments.



D&H Railroad wreck at Richmondville, N.Y. 1/29/1909.

from nodular cast irons to high strength aluminum alloys to specialized rubber to carefully tempered glass. New and vastly larger ships were on the drawing boards - they needed new alloys of corrosion resistant steel, monel, bronze, and many other improved materials.

Railroad trains had already increased the speed of overland transport by an order of magnitude, but not without the cost of many lives due to material failures. The first passenger fatality had occurred in 1833, near Heightstown, NJ. Former president John Quincy Adams and Cornelius Vanderbilt were aboard the train, but not injured. Preventing future loss of lives was an urgent need that pressed the Bureau into the new venture of certifying reference materials.

In 1905, the American Foundrymen's Association approached the Bureau to see if it would assume leadership of a new work the Association had recently begun. The Association was trying to solve the problem of rail car derailments due to the fracturing of cast iron wheels. Appropriate alloys had been found and Association research showed that they would cure the problem. However, the chemical laboratories at the various foundries that supplied materials to the railroads could not analyze the materials with sufficiently consistent accuracy to provide ongoing quality assurance. The problem as the foundrymen defined it was to have a source of accurately analyzed materials having compositions at and bracketing the compositions of alloys known to be acceptable. Those "standardizing" materials could then be used by the foundries to maintain their analyses in control.

At the time, chemists throughout the world were expected to develop and maintain their own lots of materials that could be used as benchmarks for calibrating or testing analyses. This practice could help with questions of laboratory internal consistency but shed little light on analytical disagreements among several laboratories. Methods were practically limited to those based immediately on first principles where standards could be physical. This still left open questions of completeness of separation, stoichiometry, purity of reagents, and other issues. Evaluating the accuracy of newly emerging instrumental methods would produce even greater need for certified reference materials to serve, first as accuracy benchmarks, and then as calibrators for quality assurance into the future.

The Bureau accepted the challenge and set to work - but not alone. In fact, this very first reference material project set a precedent for cooperative efforts that continue to this day. Included in that precedent is the idea that projects will be started only on demonstrated need and demand of the technical community. Furthermore, priority will be assigned to those projects where cooperation of the requesters is assured. This has helped the Bureau select worthy projects over the years. The cooperation has included provision or preparation of materials and contribution of data to the certification campaign. In the case of the first ever project, cooperation with the American Foundrymen's Association extended to all three of these aspects. The analytical work was carried out in several companies that were members of the Association, and the Association provided to the Bureau an industrial research associate to do the analytical work at NBS. The young chemist, John Cain, would become a full member of the staff, about twenty years later.

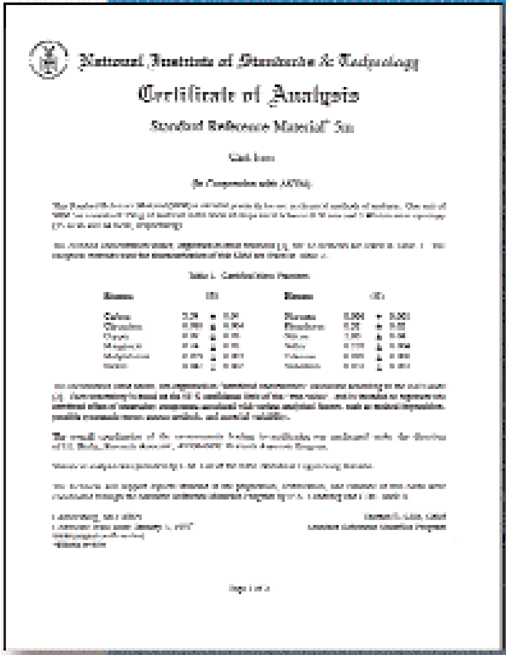
When the first project was completed in 1906, four materials were placed on sale together with their certificates of analyses to serve as the needed benchmarks. They consisted of bottles of cast iron chips which were labeled "Standardizing Iron Sample A," through "Standardizing Iron Sample D." Their worth was quickly realized in improved cast iron rail car wheels and an improved safety record for the railroads.

When the initial lots of materials were exhausted, they were replaced with new lots of nearly equal composition. Of course, the new lots were slightly different, thus new certification campaigns were required to provide new certificates of analysis. Also different was the labeling system. By the time the second lots were prepared, the cast irons were part of a larger program of reference materials, which by about 1910, had become known as "Standard Samples." "Standardizing Iron Sample B" became "Standard Sample No. 4a - Iron B." The numbers 3, 4, 5, and 6 were never assigned without the "a" designation, owing perhaps to the originals having been issued with letter designations. Standard sample numbers 1 and 2 had been assigned to other materials, so they were not available to the cast irons. Two of the original formulations of cast iron have been renewed at every exhaustion since. For Cast Iron C, there have now been 14 lots prepared over the past 95 years.

Success Inspires Imitation

The success of the cast iron reference materials led to expansion of the concept into new material types. Certification work on several other alloys, iron ores, and copper slags began the same year as the cast irons were issued. All these activities caught the interest of the steel producers. A member of the Bureau's Visiting Committee, Albert Ladd Colby, was a leading authority on metallurgy and he urged the Bureau to extend its success with cast iron into the field of steel. Perhaps because it was smaller then, the Bureau was quite

Standard Reference Material (SRM) 5m is the fourteenth lot (thirteenth renewal) of Standardized Iron Sample C, and has in recent years, been certified in cooperation with ASTM. Note the "C" designation has been dropped.



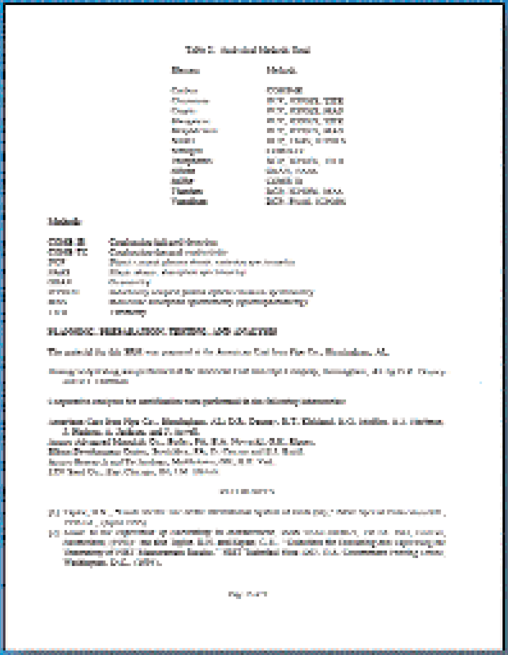
National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 5m

Cast Iron

(In Cooperation with ASTM)



agile in starting new projects and new cooperative ventures. It was able to start the new effort together with the Association of American Steel Manufacturers in 1907. A series of 17 steel standard samples emerged and started NBS-NIST on a path of support to the US steel industry that has spanned nine decades. At the beginning of the effort, the chief of the Chemistry Division was Dr. William Noyes, and the analytical work at the Bureau was done by John Cain and three other chemists: Witmer, Isham, and Waters, all of whom were probably industrial research associates.

By 1911, the catalog of reference materials had grown to 25 entries, all in support of chemical analysis. More importantly, the interest of the Nation's chemists was growing too, with observation of the utility of the "Standard Samples" wherever they were available. Clearly more types of materials would be needed and they were on the way with the cooperation of the American Chemical Society, and later the Portland Cement Association, the Copper Development Association, and numerous other groups.

Table 1. The First 14 Metal Reference Materials and their First Renewals (with numbers)

—	Standardized Iron Sample A	1906	—	Standardized Steel Bessemer 0.4	1907
SS 3	White Iron	1958	SS 10a	Bessemer Steel 0.4% C	1911
—	Standardized Iron Sample B	1906	—	Standardized Steel B.O.H. 0.2	1908
SS 4a	Cast Iron B	1910	SS 11a	Basic Open Hearth Steel 0.2% C	1911
—	Standardized Iron Sample C	1906	—	Standardized Steel B.O.H. 0.4	1908
SS 5a	Cast Iron C	1910	SS 12a	Basic Open Hearth Steel 0.4% C	1911
—	Standardized Iron Sample D	1906	—	Standardized Steel B.O.H. 0.6	1908
SS 6a	Cast Iron D	1910	SS 13a	Basic Open Hearth Steel 0.6% C	1911
SS 7	Iron E	1917	—	Standardized Steel B.O.H. 0.8	1908
SS 7b	Cast Iron (High Phosphorus)	1926	SS 14a	Basic Open Hearth Steel 0.8% C	1911
—	Standardized Steel Bessemer 0.1	1907	—	Standardized Steel B.O.H. 0.1	1908
SS 8a	Bessemer Steel 0.1% C	1911	SS 15a	Basic Open Hearth Steel 0.1% C	1911
—	Standardized Steel Bessemer 0.2	1907	—	Standardized Steel B.O.H. 1.0	1908
SS 9a	Bessemer Steel 0.2% C	1911	SS 16a	Basic Open Hearth Steel 1.0% C	1911

The Early Catalog

While the new concept of standard samples was sure to expand to many new types, that expansion did not produce a rational numbering system. From 1906 until 1910, numbers were not assigned and the term standard sample was not used. To help examine

Table 2. Other Examples Selected From the First 100 Standard Sample Numbers

SS 1	Argillaceous Limestone	1910	SS 43	Zinc (M.P. or F.P.Circ.66)	1915?
SS 2	Zinc Ore	1919	SS 44	Aluminum (M.P. or F.P. Circ.66)	1915?
SS 17	Sucrose (Stoichiometry)	1912	SS 45	Copper (M.P. or F.P.Circ.66)	1915?
SS 19-23	A series of carbon steels	1910 - 1920	SS 46	Portland Cement Sieve Test	1915?
SS 24	Vanadium Steel 0.15% V	1910	SS 47	Portland Cement Sieve Test	1915?
SS 25	Manganese Ore	1910	SS 48	Benzoic Acid (Acidimetric)	1919?
SS 26	Crescent Iron Ore	1910	SS 49	Lead (Freezing Point)	1915?
SS 27	Sibley Iron Ore	1910	SS 50	Cr - W - V Steel	1921
SS 28	Norrie Iron Ore	1910	SS 52	Cast Bronze	1921
SS 29	Magnetite Iron Ore	1910	SS 53	Lead-base Bearing Metal	1921
SS 30	Chrome-Vanadium Steel	1912	SS 54	Tin-base Bearing Metal	1923
SS 31	Chrome-Tungsten Steel	1912	SS 57	Silicon Metal	1924
SS 32	Chrome-Nickel Steel	1912	SS 58	Ferrosilicon	1924
SS 37	Sheet Brass	1914	SS 60	Ferrovanadium	1924
SS 38	Napthalene (Heat of Combustion)	1912	SS 80	Soda-lime Glass	1927
SS 39	Benzoic Acid (Heat of Comb.)	1912	SS 83	Arsenic Trioxide(Reductiometric)	1927
SS 40	Sodium Oxalate (Oxidimetry)	1924	SS 85	Aluminum Alloy (Duralumin)	1943
SS 42	Tin (M.P. or F.P.Circ.66)	1915?	SS 98	Plastic Clay	1931

some of the materials comprising the first 100 certified, two tables are presented. In Table 1, the initial 14 metal reference materials are listed, together with the designations of their first renewals. Table 2 lists other examples drawn from the first 100 standard sample numbers.

It is not clear that SS 7 Iron E was ever produced as a "Standardized Iron E." The earliest available certificate is for SS 7 without an "a" designation indicating that it may represent the first time the material was produced. Other metal standard samples were produced in the 1910 to 1912 time period, as is indicated in Table 2. It is also interesting to note that the elapsed time before first renewal was typically only three to four years, indicating heavy usage.

It is not surprising that limestone was an early standard sample - it was useful as a refractory liner in metals production. The first material to be certified that was completely unrelated to metal production was sucrose as a calibrating material for polarimeters. "SS 2" was reserved for zinc ore as early as 1910, although the first archived certificate is dated 1919. Early additions to the catalog included primary chemicals to assist in calibrating titrations, and several standard samples for physical chemistry, especially for calibrating calorimeters and thermometers.

From the information in Table 2, it would appear that there was a slowing of reference material production during World War I. At the time significant portions of the Bureau

Table 3. NBS Programs and Staff (Noting Source of University Degree) - June 1904

Division I - Weights & Measures, Thermometry, Optics, Engineering Instruments		
Louis Fischer	Columbia University	Weights & Measures
Llewelyn Hoxton	University of Virginia	Weights & Measures
Roy Ferner	University of Wisconsin	Weights & Measures
Nathan Osborne	Michigan School of Mines	Weights & Measures
Charles Waidner	Johns Hopkins University	Heat & Thermometry
George Burgess	University of Paris	Heat & Thermometry
Hobart Dickinson	Clark University	Heat & Thermometry
Samuel Stratton	University of Illinois	Optics + Director, NBS
Frederick Bates	University of Nebraska	Optics
Perley Nutting	Cornell University	Optics
Albert Merrill	Massachusetts Institute of Technology	Engineering Instruments
Division II - Electricity		
Frank Wolff	Johns Hopkins University	Resistance & Emf
Francis Cady	Massachusetts Institute of Technology	Resistance & Emf
George Middlekauf	Johns Hopkins University	Resistance & Emf
Karl Guthe	University of Michigan	Magnetism & Absolute Current
Edward Rosa	Wesleyan University	Inductance & Capacity
Ernest Dorsey	Johns Hopkins University	Inductance & Capacity
Frederick Grover	Wesleyan University	Inductance & Capacity
Morton Lloyd	University of Pennsylvania	Electrical measuring instruments
Herbert Brooks	Ohio State University	Electrical measuring instruments
C. E. Reid	Purdue University	Electrical measuring instruments
Franklin Durston	Wesleyan University	Electrical measuring instruments
Edward Hyde	Johns Hopkins University	Photometry
Charles Sponsler	Pennsylvania State Collage	Engineering Plant
Division III - Chemistry		
William Noyes	Johns Hopkins University	
Henry Stokes	Johns Hopkins University	

staff were redirected to the war effort, a precursor to the much larger dedication of personnel (approximately 4000) to supporting the military in World War II. Shortly after the first war, "SS 85" was reserved for the aluminum alloy "Duralumin" even though it was not certified until 1943.

An Unintended Consequence of Success in Reference Materials

The early work on Standard Samples at the Bureau had a profound impact on the later work of the agency. The U.S. Congress had initially seen the agency as being primarily dedicated to construction and maintenance of the physical standards of measurement. It seems quite clear that by looking at the earliest staff and their work of the Bureau (see Table 3), that there was no plan to emphasize studies in chemical and material fields further than what was needed to support programs in instrumentation and metrology.

Despite the initial lack of agency emphasis on materials efforts, the great success of the Standard Samples work weighed heavily in steering most of the growth of the new agency into the direction of solving practical material problems and, in some cases, using cutting edge instrumentation and methods. Later amendments to the agency's organic act recognized the Bureau's contributions to materials characterization and development, and by 1950 provided specific authority to certify and distribute reference materials as the leading U.S. authority.

William Meggers applied spectroscopy to physical and chemical measurements.

Launching Domestic Industries

Research at the Bureau occasionally provided initiative for the production of a new SRM, and very often provided the tools needed to certify materials with a reduced uncertainty. There are also cases where the production of an SRM inspired the start of an industry new to the United States. A striking example of that occurred during World War I. Before that war, the Bureau was distributing standard samples of sugar for three important applications: calibrating saccharimeters; calibrating calorimeters used in measuring the heat content of fuels and for use in differentiating bacteria in medical laboratory tests. Germany was the source of the pure sugars that the Bureau characterized, certified, and sold.

When the war broke out and the materials were no longer available, the Bureau had to produce its own pure sucrose and dextrose. The German patents and production literature were written so obliquely to protect proprietary rights from other producers that reconstruction of the production processes required almost completely original research. The results were well worth the effort because the output was not only Bureau Standard Samples, but also the technology for producing low cost dextrose that launched a new domestic industry for American sugar producers and corn farmers.

The connection with instrument manufacturers is perhaps less direct; but nevertheless, just as real. The ideas developed at the Bureau to solve all manner of analytical problems were frequently blended with the work of instrument makers to either create new instruments or impact the progress in developments of existing ones. Perhaps the tradition started with recruitment from 1901 to 1904, of the first 26 professional staff members, seven of whom were from the Johns Hopkins University. Johns Hopkins was at the time a international leader in spectroscopic technology, so perhaps it was fitting that the entire Chemistry Division professional staff (2) were from Hopkins - William Noyes and Henry Stokes. It was Noyes who first produced atomic mass data at the Bureau, reporting in 1907, the weights for several elements, including hydrogen at 1.00783.

In 1905, William Coblentz joined the staff and would serve the Bureau for the next 40 years. By 1914, William Meggers had also come from Johns Hopkins to join the staff, and would contribute for 52 years to a wide variety of spectroscopic techniques that would later find their way into commercial production.

Just as many of the early instrumental techniques for chemical analysis found their basis in spectroscopy, so grew the need for reference materials. This resulted from most spectrochemical techniques requiring reference materials as calibrants.

Early Connections - Lasting Patterns

One of the most interesting aspects found in studying the early technical efforts of the

The Challenger flight STS-6 provided (National Aeronautics and Space Administration) the 10µm spheres for SRM 1960.

Bureau is the degree to which they established enduring programs of reference material production. Some of these cases are so obvious as to require no further explication, for example the program in cast iron standard samples setting a 95-year-long pattern that lasts to the present. Some others are less obvious but no less interesting:

- **Radioactivity** - In 1911 Marie Curie prepared, the first standard for activity as a sealed glass tube containing weighed amounts of radium and radium salts and characterized for its gamma ray count rate. The standard was accepted as an international standard and was maintained at the BIPM[1] France. A similar tube was prepared and calibrated with the one at BIPM for delivery to the Bureau in 1913, becoming our Nation's first standard for radioactivity. This would serve as the start of a program that would see the Bureau develop reference materials to accommodate every aspect of radioactivity measurement.

  An interesting repayment occurred in 1921, for Mme. Curie's earlier generosity to the scientific community. By that time, she was in need of additional radium to pursue her investigations, but the material was too expensive for her resources. On hearing of her plight, a group of American women banded together and raised the funds necessary to purchase 1g of radium for Mme. Curie. The material was certified by the Bureau for purity and activity and was presented to the famous scientist by President Warren Harding.

  Today, NIST has more than seventy radioactivity SRMs in the catalog. These cover a wide range of applications including certified activity for radiopharmaceuticals, alpha particle point sources, and gamma ray point sources.

- **Aviation/NASA** - From the first days of aviation, NBS took an active part in developing the technology. Wind tunnels were quickly constructed to develop improved airfoils and a special laboratory building (the Dynamometer Building) was constructed to research the weakest link in the whole enterprise, the engines. Absolutely the highest power-to-weight ratio was needed and that meant higher compression and extreme demands on fuels. Reference materials were issued for isooctane and n-heptane to serve as quality controls for the emerging aviation fuel industry. Calorimetry reference materials were vital in designing the best fuels for piston, jet and rocket engines.

  In 1915 the National Advisory Committee for Aeronautics (NACA) was formed, the Bureau had a leading place in the committee. The other agencies, including the Army and Navy, did not have aeronautical facilities, so the laboratory work fell to the Bureau. In late 1932, President-elect Roosevelt proposed folding NACA into the Bureau. That proposal was never carried out but in 1946, Hugh Dryden, one of the nation's top theoretical aerodynamisists, left the Bureau to direct all research at NACA. Subsequently he led the transformation of NACA into NASA in 1958.

  Later, reference materials of many types contributed to aerospace development. Aluminum alloys, and high-strength alloys were needed for skins and airframes. High temperature alloys were needed for jet and rocket engine hot-section components. NDE (non-destructive evaluation) reference materials were needed for monitoring part reliability in service. Every reference material in support of electronics technologies has a carry-over application in aerospace.

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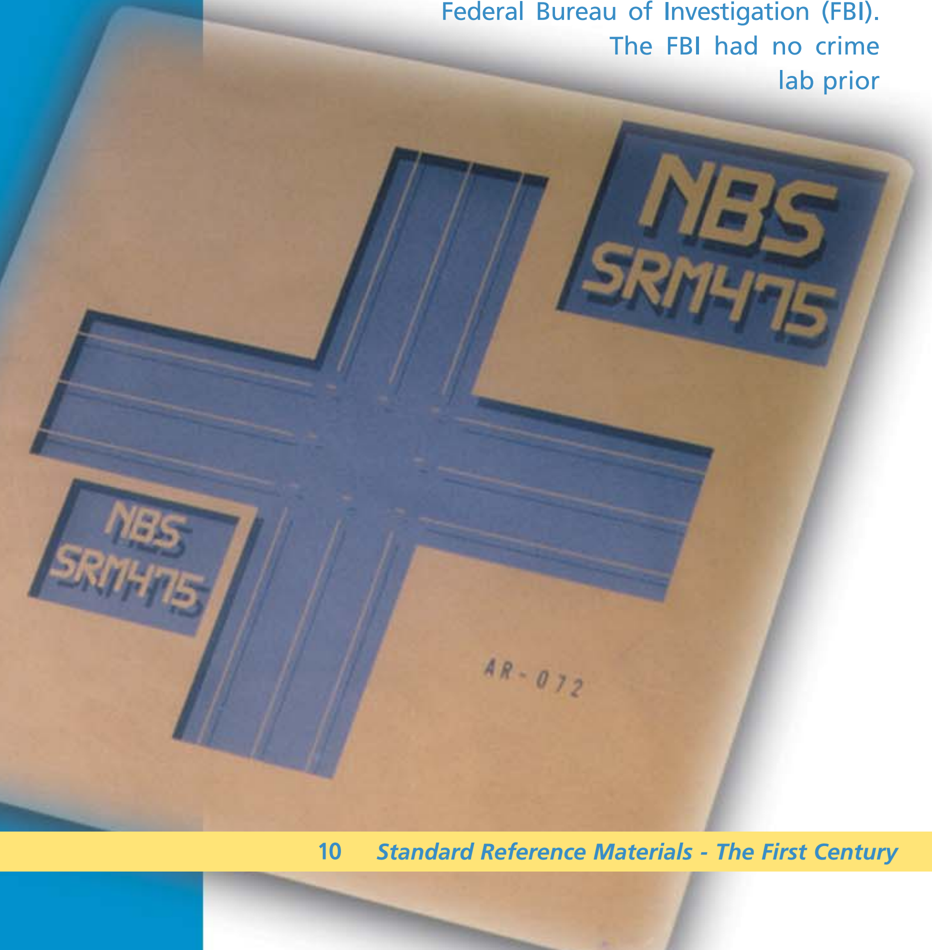
[1] The Bureau International des Poids and Measures (BIPM) is located in Sevres, a suburb of Paris. It has the task of ensuring worldwide unifacation of physical measurements.

In the mid-1980s, the Standard Reference Materials Program organized the certification campaign for the first commercialized material produced in space - a length standard at microscopic scale. Perhaps it was the long-term connections between the two agencies that gave NASA the confidence in NBS to carry out the novel project. The material certified was SRM 1960 Nominal 10µm Diameter Polystyrene Spheres (certified as 9.89µm ± 0.04µm). John Vanderhoff of Lehigh U. and Dale Kornfeld of NASA had teamed together to produce spheres in "Monodisperse Latex Reactors" aboard five missions in 1982 and 1983. The missions were STS 3-4 and STS 6-8, with the spheres for SRM 1960 being produced aboard the Challenger's STS 6 mission. After the Challenger disaster, George Uriano (SRM chief, 1979 - 1983) served as a Congressional staff member in the investigation of the tragedy.

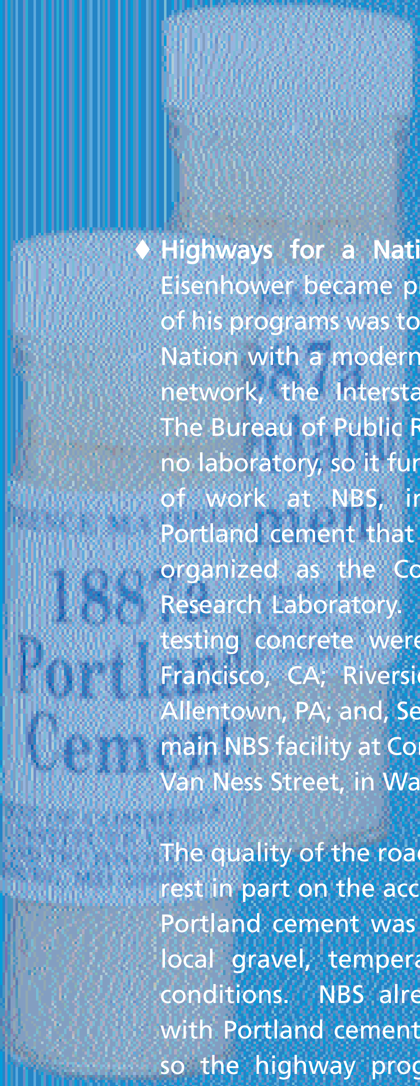
♦ **Forensic "Signatures"** - Just as one could say that NASA was a Bureau spin-off, the same is true for the crime laboratory at the Federal Bureau of Investigation (FBI). The FBI had no crime lab prior to 1932, when the Bureau assisted in its development. Before that time, the Bureau did laboratory work as a service to the FBI, including the famous Lindbergh kidnaping case in 1932. The Bureau had significant impact on the case through the handwriting analysis of Wilmer Souder who conducted forensic investigations for the Bureau from 1913 until 1954.



Most forensic signatures are not in handwriting. Perhaps more important are bullet lead markings, broken glass matching, breathalyzer tests, drugs of abuse in urine, and DNA profiling. Over the past 60 years, NBS/NIST has certified reference materials for all these signatures.

♦ **Proximity Fuse/Electronics** - A major NBS contribution to the ordinance effort for World War II was the development of the proximity fuse that caused shells and bombs to explode at a predetermined point above the ground. By the 1950s, all such direct military work was transferred out of the agency. However, a great deal of the technical work associated with the fuses was to miniaturize and harden the associated electronic circuitry. The fundamental parts of that work stayed at NBS and stimulated the development of a number of SRMs.

Today, the SRMs related to electronics cover a wide array from the composition of solder alloys to the transmission properties of complex optoelectronic devices. Perhaps most basic are those for the conductivity and resistance of a variety of metals and especially silicon. Residual resistivity ratio SRMs are also available for silicon. Dimensional metrology is so critical to semiconductor manufacture quality assurance that NIST has developed photomask linewidth artifacts, such as SRM 475, for calibrating optical and scanning electron microscopes, and has developed a range of thin film thickness SRMs for ellipsometry.



- **Highways for a Nation** - When Dwight Eisenhower became president in 1953, one of his programs was to link every part of the Nation with a modern high-speed highway network, the Interstate Highway System. The Bureau of Public Roads at the time had no laboratory, so it funded significant levels of work at NBS, including efforts on Portland cement that would eventually be organized as the Concrete and Cement Research Laboratory. Field laboratories for testing concrete were maintained in San Francisco, CA; Riverside, CA; Denver, CO; Allentown, PA; and, Seattle, WA besides the main NBS facility at Connecticut Avenue and Van Ness Street, in Washington, D.C.

  The quality of the roads constructed would rest in part on the accuracy with which the Portland cement was formulated to meet local gravel, temperature, and humidity conditions. NBS already had experience with Portland cement reference materials, so the highway program provided need and opportunity to expand the SRM catalog with more than a dozen new offerings for cement composition, particle size, and rheology.

- **Consumer Protection/Health** - While the Bureau has produced many services and publications of interest to consumers, it has never been primarily a consumer products laboratory. Indeed, if described beyond being a metrology laboratory, the more comfortable niche would be as an industrially-oriented laboratory. When the Consumer Product Safety Commission (CPSC) was formed in the 1960s, it had no laboratory and depended entirely upon NBS for laboratory support. During the 1970s, the level of work had increased sufficiently that CPSC could start its own laboratory with the help and transfer of many NBS staff members.

  What remains at NIST today are the complex metrology issues associated with health-related measurements in such areas as the environment, clinical chemistry, pharmaceutical measurements, food labeling, and nutritional studies. The general SRM categories for these are listed in Table 7. For these fields, NIST has excelled in providing hundreds of well-selected and characterized SRMs. Environmental natural matrix materials are covered with a wide variety of solids, liquids, and gases being covered for both inorganic and organic constituents. More than 35 different SRMs serve to validate clinical laboratory determinations. Most analytical instruments found in pharmaceutical laboratories have one or more SRMs available to provide measurement traceability. Some of the newest SRM work has been dedicated to food analysis, with about 30 types now available.

- **A Team Approach** - From the earliest days of cooperation with the American Foundrymen's Association, the Bureau has built on the success of steady growth in cooperation with technical societies and standards organizations. Perhaps no part of Government is as welcome in the activities of such bodies. This means NIST is usually the agency most likely to be trusted as an "honest technical broker" of ideas and programs. Even a partial list of cooperating organizations would need to include the American Society for Testing and Materials (ASTM), the Society of Automotive Engineers (SAE), the Institute for Electrical and Electronic Engineering (IEEE), the

American Chemical Society (ACS), the American Ceramic Society (ACS), the Institute for Textile and Color Chemists (ITCC), the American Iron and Steel Institute (AISI), the Copper Development Association (CDA), the College of American Pathologists (CAP), and the American Association of Clinical Chemistry (AACC). NIST has had joint programs to develop or share SRMs with every one of these organizations and many more besides. See Appendix A.

Continuing Efforts

Between the two World Wars, Standard Sample activities grew slowly, but steadily. Industrial needs for materials for chemical analysis were the major, almost exclusive, impetus. Starting in World War II the needs for Standard Samples began to grow and change.

As a primary national laboratory for materials research, NBS contributed to the Manhattan Project through uranium studies. Among other accomplishments, NBS scientists carried out pioneering work in the separation of uranium isotopes and developed Standard Samples for determining the isotopic composition of materials containing uranium and plutonium. These materials continue to serve the country today for the accurate assay of reactor fuels.

After the war, breakthroughs in the fields of electronics, polymer research, and the spread of spectrometric instruments brought new demands for reference materials. By the 1950s, special hydrocarbon blends were available for calorimetry and Standard Samples were being certified for such properties as pH, melting point, and radioactivity. New high temperature and super strength metal alloys were needed to meet the demands of innovations in jet aircraft and rockets.

The 50s and 60s saw an acceleration of efforts to certify reference materials with 582 types available in the catalog in 1969. It was during this period that the Bureau began to transfer some of its efforts in reference materials to other institutions. This will be discussed in more detail in the section: "Closing the Cycle." During the 1960s, NBS realized that SRMs [2] were becoming increasingly important to industry and that industrial demand would continue to grow. The Bureau also recognized the potential contribution SRMs could make in solving measurement problems in emerging areas of national need such as clinical and environmental chemistry.

To appreciate the place and importance of SRMs in the work of the Bureau at the midpoint of its centennial requires a closer look at events impacting the agency around the middle of the 20th century.

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[2] The term “Standard Reference Materials,” and the acronym “SRM” were introduced into use by NBS in 1965. The term and acronym were later registered with the U.S. Patent and Trademark Office.

Mid-century Turmoil

Two separate, initially very damaging, tribulations fell upon the Bureau between 1948 and 1952. Eventually, the agency would recover from these two blows with a firmer resolve and an even stronger sense of mission. Before that happened, two directors would face disgrace and the budget would suffer serious decline. One of the strongest elements of the Bureau's program was its standard samples activity, and we will see how it was a major contributor to restoring both fiscal and public relations health to NBS.

Edward Condon, fourth director of NBS, was perhaps its most eminent director in terms of scientific achievement. He was a noted theoretician with great insight in atomic and nuclear physics, and provided great service to the Nation during World War II. Prior to leading NBS, he worked at Los Alamos on development of the bomb and served with Chairman Lyman Briggs (third NBS director) on Committee S-1 to directly advise President Roosevelt on atomic and nuclear issues, including the decision to build the atomic bomb.

After the war, he continued to advise President Truman and the Congress. As part of that advice, he strongly advocated the formation of the Atomic Energy Commission to move control of nuclear activities from the military to the civilian sector. He became a spokesman for peaceful uses of the atom to benefit the whole world. Neither of these positions were popular with the military, which had a strong post-war victory standing, or with the House Unamerican Activities Committee. In 1948, that committee charged Condon with being a risk to National security. No charges were ever proved, but the committee denied the director opportunity to address the charges and he left NBS in 1951 to become the director of research at Corning Glass. In all, it was one of the sadder episodes of "witch hunting" seen in our country.

The second turmoil also began in 1948 but it peaked in 1952, thus impacting the fifth director, Allen Astin. This cloud over the Bureau arose from the assignment to test many kinds of commercial products. One of these, a battery additive named AD-X2, was found by NBS to be ineffective. Less controlled tests in other places showed some promise for the additive, so the developer (and more significantly, his congressman) attempted to discredit NBS results. Astin was well versed in electrical engineering and stood behind the results of his technical staff through a firestorm of congressional hearings and unmeasured personal attacks. Finally, the Secretary of Commerce fired Astin. Now, it was the turn of the technical staff to stand behind their director. Hundreds told the President that they would go if Astin went. Based on additional data from other sources and the unanimous advice of the NBS Visiting Committee, the Secretary retained Dr. Astin as director and he served with distinction until retirement in 1968, following

(top) Aerial view of Gaithersburg Campus.
(bottom) Aerial view of Boulder Campus.

Table 4. NBS Funding for Selected Years

Year	Appropriation ($ millions)	Comment
1946	3.6	Condon era begins, need for war $ replacement
1948	7.1	House Unamerican Activities Comm. / Condon
1950	8.5	Working Capital Fund (WCF) authorized
1952	7.8	Astin era begins, peak of AD-X2 affair
1954	5.7	Kelly report will shape future budgets
1956	7.4	Budget still below 1949 level of $ 8.7 million
1957	8.4 + 2.8 WCF = 11.2	WCF implemented, 10-yr funding crisis ended
1958	9.7 + 2.5 WCF = 12.2	Sputnik launched
1960	~ 20	First impact of Sputnik on budget
1970	> 40	Final budget developed by Dr. Astin

completion of the Boulder laboratory (1954) and of the Gaithersburg facility (1966).

The two episodes could only adversely affect the budgets from 1952 through 1956. More than 90% of NBS funding during the war years was from defense sources and that was being reduced in peacetime. Turmoil had struck just when it was not needed. Table 4 provides a snapshot of the seriousness of the situation. In reading the table, it is important to note that the budget process is slow and usually follows impacts by about two years. Dr. Astin's first appropriation from Congress was for 1954, covering July 1, 1953 through June 30, 1954, and at $5.7 million was the smallest in the previous seven years.

A New Beginning

One response of the Secretary of Commerce to the AD-X2 difficulty was to commission a high- level assessment of the work and mission of NBS. The study was chaired by Mervin Kelly, president of Bell Telephone Laboratories. On October 15, 1953, the committee issued recommendations that would

Table 5. Kelly Committee Report - October 15, 1953

Increase

1. Basic Programs
2. Facilities and Space for Basic Programs
6. Use of Bureau Expertise by Other Agencies of Government
9. Support of Standard Samples Program

Decrease

4. Weapons Related Work, and Transfer it to the Defense Department
7. Repetitive Test Operations (Including Routine Product Testing)

Administrative

3. Revise Use of Associate Directors
5. Continue DoD and AEC use of NBS for Non-weapons Work
8. Transfer to Doc a Significant Portion of Product Testing Work
10. Form Technical Advisory Groups with Members from 8 S&T Societies

shape the Bureau for the next 35 years. The report would become known as the Kelly Report and it singled out two parts of Bureau work to praise: basic research in metrology; and, the value of the standard samples program. The Kelly Report contained ten recommendations that are grouped into three categories and presented in Table 5, with the numbers indicating the sequence of the recommendations as found in the report.

It would be overreaching to say that NBS Standard Samples rescued the Bureau at the century's mid-point, but it would be fair to say that they had made a very impressive mark and again had a prime role in shaping the Bureau's new mission. New emphasis was given to moving the development of standard samples beyond chemistry alone. The new approach started slowly. Then, in 1964, the Office of Standard Reference Materials was established and given the responsibility for directing all SRM activities. Table 6 provides a list of the leaders for reference material activities both before and after creation of the new office. Previously the individual technical divisions had managed separate components of the SRM program with coordination through the Analytical Chemistry Division. With the establishment of a new

Table 6. Leaders of the Standard Sample/ SRM Production Activities at NIST 1905 - 2001

Year	Name	Title	Organization
1905	John Cain	Research Associate	Chemistry Division
1918	Gustave Lundell	Chief	Analytical Methods & Standard Samples
1940	Harry Bright	Chief	Metal & Ore Analysis, Standard Samples
1950	Harry Bright and Bourdon Scribner	Chief Chief	Analytical Chemistry Spectrochemistry
1960	John Hague Bourdon Scribner	Chief Chief	Analytical Chemistry Spectrochemistry
1964	Wayne Meinke	Chief	Office of Standard Reference Materials
1969	J. Paul Cali	Chief	Office of Standard Reference Materials
1979	George Uriano	Chief	Office of Standard Reference Materials
1983	Stanley Rasberry	Chief	Office of Standard Reference Materials
1991	William Reed	Chief	Standard Reference Materials Program
1994	Thomas Gills	Chief	Standard Reference Materials Program
2000	Nancy Trahey-Bale	Chief	Standard Reference Materials Program
2002	John Rumble Jr.	Chief	Standard Reference Materials Program

office, a number of new thrusts were developed. New program areas were identified and initiated, including the start of what was to become a major effort in developing SRMs for clinical chemistry. For a list of some of the key SRMs beyond the first 100, see Appendix B.

Through the 1970s, the program had its most productive decade in terms of the development of both numbers and types of SRMs. Over 600 new SRMs were certified during that period and about 250 were discontinued. By 1979, 1,060 types appeared in the catalog. Typical of the research activity into new SRM types in this period was a whole array of environmental natural matrix materials certified for inorganic constituents. Leading the list, and arguably the most important materials of their era, were SRM 1571 Orchard Leaves, SRM 1645 River Sediment, and SRM 1648 Urban Particulate Matter. John Taylor directed the certification of these materials. The river sediment was prepared from material dredged from the Indiana Harbor Canal, near Gary, Indiana. Heavily loaded with toxic metals the material served as the initial benchmark for environmental studies in the field. It is also important to note from an analytical perspective, that these were the first environmental matrix materials to receive extensive application of the new isotope dilution mass spectrometry method developed by NBS and which was also used to good effect in the certification of SRM 909 Human Serum.

During the 1980s, the many advances in inorganic environmental reference materials were extended by the addition of SRM certifications for organic analytes of environmental health concern. Some of these included PCBs in human serum, in oil, and in sediments. Also made available were SRMs with certified values for a variety of forms of dioxin, polynuclear aromatic hydrocarbons, halocarbons, chlorinated pesticides, and other priority pollutants.

Food-based SRMs began coming into the inventory in the 70s and 80s, with the introduction of such materials as wheat flour, rice flour, and freeze-dried bovine liver, oyster tissue, and spinach leaves. The SRM efforts toward better coverage of food types really have seen the most progress in the 1990s, with work being dedicated to certifying mixed diet food materials, infant formula, and individual foodstuffs for vitamin content. Efforts along these lines are sure to continue into NIST's next century.

Today's Catalog

As impressive as is the history of SRM production and certification, perhaps even more impressive is the work summarized in the current catalog and in the work program for future certification. Table 7 provides the category names that cover the approximately 1400 SRMs and other reference materials available in the current SRM catalog, available under the NIST home page on the World Wide Web as well as in printed form.

Wide dissemination of information on availability, including exhibits at 5 to 10 conferences each year, helps the program distribute more than 35,000 units of material to about 10,000 customers. Approximately one-third of each year's sales are delivered abroad.

Table 7. SRM Categories for 2001

Health and Clinical
Industrial Hygiene
Environmental
Food and Agriculture
High Purity Materials
Industrial Materials
Engineering Materials
Physical Properties
Radioactivity

Closing the Cycle

As many types of reference materials matured and NIST interests turned to other technologies, efforts have been made to find institutions willing to receive transfer of the materials and responsibilities. This is essential to permit reallocation of scarce resources to fund new efforts.

During the 1950s and 60s, some of the first of these transfers began to take place. The extensive hydrocarbon blend project was transferred to the American Petroleum Institute. Color and fading SRMs were transferred to the American Association of Textile Chemists and Colorists, and viscosity materials went to Carnegie-Mellon University. During the 1980s, in part for security reasons, the SRMs for uranium and plutonium were transferred to the New Brunswick Laboratory of the US Department of Energy located at Argonne National Laboratory. At the beginning of the 1990s, NIST was closing out its work on rubber compounding so about a dozen types of rubber compounding SRMs were transferred to ASTM. These are a few of many examples of "passing the baton" on reference materials that NIST can no longer support.

Not all the transfer efforts have involved actual movement of the materials. Most of the metal materials that have figured so heavily in the early development of the Standard sample and SRM programs, have remained at NIST, but much of the certification effort on renewal lots is only undertaken by laboratories organized in cooperation with ASTM. It is highly likely that the trend will be for NIST to continue to seek partners in industry, universities, or other government agencies to provide homes for SRMs and RMs being closed out at NIST.

Benefit/Cost

The annual cost each year for all types of measurements in the United States is counted in the hundreds of billions of dollars. A small view of the scale and vigor of the activity in analytical chemistry alone is seen each year at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy where about 30,000 conferees gather, representing perhaps a tenth of their colleagues working in the field. Those who have attended this conference for many decades will recognize the many changes in the field that have brought significant improvements in effectiveness and efficiency to most types of chemical analysis. The driving forces behind these changes have included the rapid and far-reaching development of instrumentation, the improvement of analytical methods, and the availability of Standard Reference Materials from NIST and secondary suppliers.

It is not hard to calculate that an SRM contribution to measurement improvement, even as small as 0.1%, would save companies who measure hundreds of millions of dollars each year - easily justifying the approximately $10 million spent annually to buy SRMs. Client demand for ever increased numbers of types provides a qualitative indicator of the program benefits. The demand requires careful prioritization for it greatly exceeds each year's SRM budget. Table 8 summarizes the approximate number of SRM types available at ten-year intervals. The table data are not exact, as no hard data were recorded for most of the intervals. However, several sources have been studied and the values are probably dependable with an uncertainty of less than 10% of each value. Reasons for the ambiguity include discontinuation of SRMs (with perhaps 1000 types having been discontinued over the history of the program) and an uncertain number of materials listed in catalogs being out of stock at a given time. Additionally, SRMs have not been numbered sequentially, complicating historical counts.

What is harder to quantitate is the social contribution made by SRMs. In a study by the Research Triangle Institute on the economic impact of SRMs for sulfur in fossil fuels, three economists led by Sheila Martin determined that the group of SRMs had a benefit/cost ratio of 113, and a social rate of return of more than 1,050%. Similar conclusions have been reached at other times and in other studies with regard to a variety of SRMs. While not as quantifiable, there are major benefits to people dependent on good clinical measurements; for example, the child whose epileptic seizures are better controlled by measurements improved with SRMs. In another example, cholesterol measurement uncertainties have been reduced from 20% in the 1970s to 5% in the 1990s, again with the help of SRMs. It is clear that customers' demands for more and better SRMs are unrelenting. Despite the common complaint "SRMs cost too much," customers continue to buy them in quantities adequate to fund future certification programs.

Table 8. Approximate Number of SRM types Available by Decade

Year	Number	Year	Number
1910	20	1960	400
1920	50	1970	600
1930	90	1980	1100
1940	200	1990	1200
1950	250	2000	1300

Collaboration Around the World

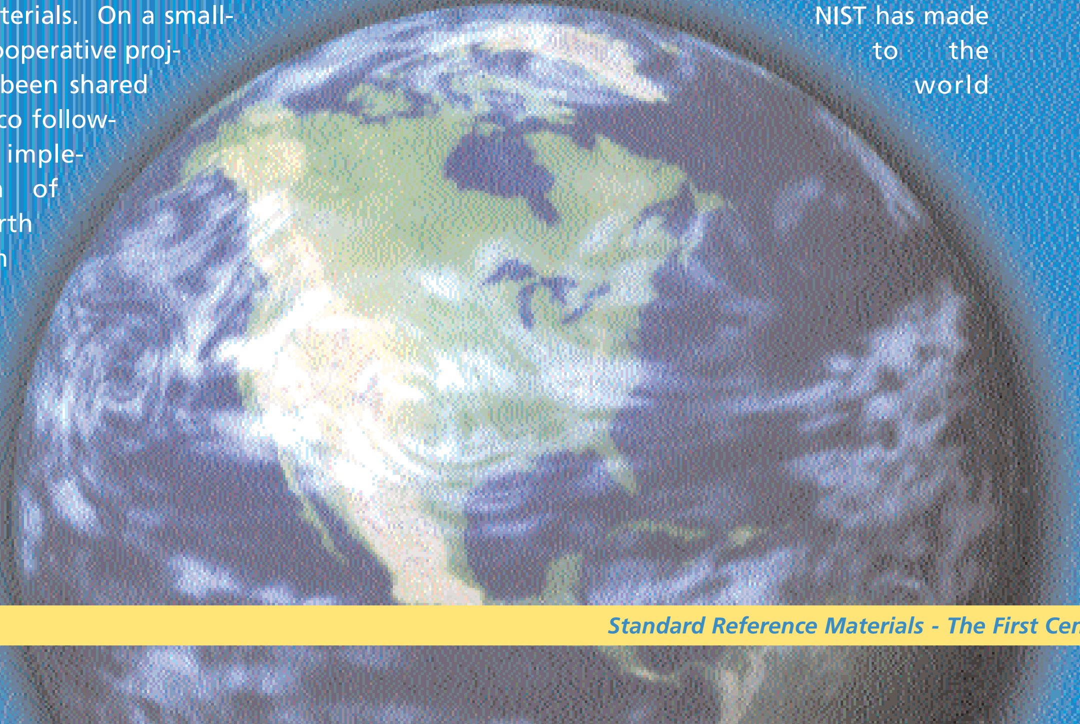
Especially over the last five decades, the Bureau has nurtured strong ties for international cooperation. More recently, about 25 years ago, the International Organization for Standardization (ISO) accepted as a Council Committee, a group to work on reference materials. The committee was first referred to as REMPA and very quickly had a change in name to ISO Committee on Reference Materials (REMCO). Strong impetus for its founding and early success was supplied by NBS staff members such as Bill Andrus, Paul Cali, and George Uriano. REMCO has become the focus for defining the terminology and practices of reference material certification and use.

NIST has participated in many bilateral and multilateral cooperations. Some of these have had as a goal, certification of a specific SRM or groups of SRMs, while others have been oriented toward helping other countries get started in the field. As an example of the latter, NIST hosted approximately two hundred chemists from China over the first fifteen years following renormalization of relations between the two countries. Analysis of SRMs was part of the effort for many of the chemists. Some of them, on return to China, have established a very vital program in production of reference materials. On a smaller scale, cooperative projects have been shared with Mexico following the implementation of the North American Free Trade Agreement and agreements for cooperation in metrology. Additional efforts have been carried out with France, the United Kingdom, Germany, Japan, Egypt, Poland, and several other countries.

Some of the most effective collaborations are those aimed at gathering data to characterize proposed new reference materials. In recent years these have included many countries contributing to the development of SRMs. A few examples would include the certification of infant formula and mixed diet SRMs, and also a large suite of food RMs from Canada. Other efforts have included contributions from most of the world's industrialized countries. An earlier example occurred in the late 1970s. NBS partnered with 70 individuals from 22 organizations in the U.S. copper industry and the ASTM to develop a series of unalloyed copper SRMs. Robert Michaelis, Jerry Hust, and Lynus Barnes directed the effort which received not only a large measure of U.S. industrial support, but also the contribution of data from Canada, South Africa, South America, and the European contributors.

Conclusion

The aim of this brief account has been to highlight a few of the many contributions that NBS-NIST has made to the world



through the certification and distribution of reference materials. The selection of examples cited only begin to probe the surface and cannot do real justice to the program in its entirety over almost 100 years. Many chemists will, however, relate to the great feeling of success that comes when analyzing an SRM from NIST and obtaining the certified values or discovering a disagreement, thereby preventing the propagation of an error!

Standard Samples and SRMs have been among the most widely known and distributed measurement artifacts of all the NBS/NIST contributions, with several million units having been circulated in the first 100 years. Few are the scientist around the world who have not heard of SRMs and the value they provide for validating a method or calibrating an instrument. By promoting and sharing the ideas and technologies of reference material development, the Bureau has been a good neighbor in the world community.

Standard Samples and SRMs have represented some of the finest efforts produced by NIST. By successfully solving material-related problems and bringing attention to those successes, the work on reference materials has had a major effect on the development of the early Bureau's selection of work programs and ultimately its character as a world-class metrology organization. It has been an exciting century for the field of reference materials in support of analytical chemistry and the other sciences. Perhaps it will be seen in another 100 years as a worthy introduction to the even greater things that are yet to come for NIST SRMs.

For those inclined to speculate about the future, a few of the author's notions are tabulated in Appendix C. The only certainty about them is that they are uncertain. They are most likely to be seen in retrospect as too conservative.

Appendix A Cooperation With ASTM in Standard Reference Material Development

The NBS/NIST reference materials program has, since it began to develop reference materials in 1904, benefited from close cooperation with many of the nation's finest technical societies and associations. It is clear today that without this cooperation, the program would either never have started or would be vastly different and probably greatly diminished in size and scope. None of the alliances has been more far reaching and had greater impact than the one with the American Society for Testing and Materials (ASTM). Starting before the second world war, the cooperation has arranged for the shared development of more than 1000 Standard Samples and Standard Reference Materials. Formal Research Associate Programs have posted ASTM staff at NIST to participate directly in the preparation and measurement of candidate materials, and, perhaps even more importantly, to arrange for the technical assistance of many contributors from hundreds of industrial laboratories. Today, ASTM E01.94, Subcommittee on Development of Reference Materials for the Chemical Analysis of Metals, Metal-Bearing Ores and Related Materials, meets semiannually to prioritize requests to NIST for new and renewal reference materials and to cooperate in their development.

In 1987, the author prepared a list of selected instances where SRMs are specified for use in ASTM technical standards. The list is by no means complete (for example ASTM D02.SC3, Subcommittee on Elemental Analysis, recently included SRM 1848 "Lubricant Additive Package" in new standards for motor oil test methods) but is given below to indicate the scope of SRM inclusion in ASTM standards.

During its first century, NIST has provided technical staff support to ASTM, filling as many as 800 committee assignments in some years. Several staff members have served as directors on the ASTM Board. The former chief of the Standard Reference Materials Program, Nancy Trahey-Bale, has been one of those, having served as treasurer and ultimately as chairman of the Board of Directors for ASTM in 1993.

Selected Examples of SRM Incorporation in ASTM Standards (as of 1987)

ASTM STANDARD	TEST	NIST SRM
C 204	Cement Fineness (Blaine)	114n Portland Cement
C 115	Cement Fineness (Wagner)	114n Portland Cement
C 430	Sieve Residue	114n Portland Cement
C 336	Glass Annealing and Strain Points	711 and 717 Glass Viscosity
C 338	Glass Softening Points	711 and 717 Glass Viscosity
C 657	Glass Electrical Resistivity	624 Glass Electrical Resistivity
C 770	Glass Stress Optical Coefficient	708 and 709 Glass
C 829	Glass Liquidus Temperature	773 Glass Liquidus Temperature
D 1238	Melt Flow Rate (Plastic)	1475 and 1476 Polyethylene
D 1505	Density (Plastic)	1475 and 1476 Polyethylene
D 1434	Gas Transmission Rate (Volumetric)	1470 Polyester Film
D 1434	Gas Transmission Rate (Manometric)	1470 Polyester Film
D 3985	Gas Transmission Rate (Coulometric)	1470 Polyester Film
D 1646	Mooney Viscosity	388m Butyl Rubber
D 2268	Chemical Analysis of Fuels	1816a Isooctane
D 3177	Sulfur Analysis in Coal and Coke	2682-2685 Sulfur in Coal
D 4239	Sulfur Analysis in Coal and Coke	2682-2685 Sulfur in Coal
D 2795	Analysis of Coal and Coke Ash	Soda Feldspar
E 27	Analysis of Zinc and Zinc Alloys	94c Zinc Base Alloy
E 129	Analysis of Thermionic Nickel	671-673 Nickel Oxide
E 322	Analysis of Steels and Cast Irons	numerous steel/iron SRMs cited
E 539	Analysis of 6Al-4V Titanium Alloy	173 and 654a Titanium Alloy
E 162	Flame Spread Index	1002c Surface Flammability
E 648	Critical Radiant Flux	1012 Flooring Radiant Panel
F 746	Pitting and Crevice Corrosion	1890-1891 Crevice Corrosion

Appendix B Key NBS Reference Materials Beyond the First 100

Numbering of Standard Samples and Standard Reference Materials has been assigned according to several different systems during the first century of production and certification. Almost all of the first 100 SRMs were completed before 1930 and assigned numbers more or less in sequence, with the exception of the first four "standard samples" as was discussed in the main text. Occasionally a number was reserved for a certain SRM, such as 85 for aluminum alloy (Duralumin) first issued in 1943, that had a delayed issue or was not issued at all. In a few cases, numbers for unissued materials and SRMs that were issued but discontinued have been reused for different materials.

Current practice retires the numbers of discontinued materials. Typically, when a given lot of material for an SRM is exhausted and replaced with a new lot, new certification measurements and a new certificate are required. The new lot of the SRM retains its numerical designation and a lower case letter is appended or changed to denote the lot. For example, SRM 916a Bilirubin is the first reissue (second material lot) of SRM 916 Bilirubin, while SRM 955b Lead in Blood is the third lot of that material.

The table that follows sketches the development of SRM types over time by listing the date of initial issue for selected key materials. The list is intended only to be representative as it includes but a few percent of all materials issued. Selection for the list typically indicates the SRM is an early member of a given material type.

Selected Key NBS/NIST Reference Materials Beyond the First 100

SS/SRM	TITLE	DATE	APPLICATION
102	Silica Brick	1932	Composition
103	Chrome Refractory	1934	Composition
112	Silicon Carbide	1937	Composition
113	Zinc Concentrate	1941	Composition
119	Chromel P	1935	Thermometry
120	Phosphate Rock	1939	Composition
136	Potassium Dichromate	1948	Oxidimetric
140	Benzoic Acid	1942	Microcombustion
147	Triphenyl Phosphate	1969	Microanalytical
154	Titanium Dioxide	1943	Composition
160	Stainless Steel 19Cr-9Ni-3Mo	1949	Composition
162	Ni-Cu Alloy	1949	Composition
164	Mn-Al Bronze	1951	Composition
165	Glass Sand	1948	Composition
173	Titanium Base Alloy (6Al-4V)	1957	Composition

(Continuation of Table)
Selected Key NBS/NIST Reference Materials Beyond the First 100

SS/SRM	TITLE	DATE	APPLICATION
187	Borax pH Standard	1947	pH Standard
303	Burnt Sienna	1944	Color
330	Copper Ore Mill Heads	1973	Composition
331	Copper Ore Mill Tails	1973	Composition
343	Stainless Steel 16Cr-2Ni	1962	Composition
349	Waspalloy	1959	Composition
475	Optical Microscope Linewidth Measure	1981	Length
480	Electron Microprobe Standard (W-20Mo)	1968	Composition
482	Gold-Copper Wires for Microprobe	1969	Composition
484	Scanning Electron Microscope Magnication	1977	Length
485	Austenite in Ferrite	1970	Composition
592	Hydrocarbon Blend No. 1	1961	Composition
600	Bauxite	1988	Composition
601	Spectrographic Aluminum	1951	Composition
610	Trace Elements in Glass Matrix	1970	Composition
620	Soda Lime Flat Glass	1972	Composition
621	Soda Lime Container Glass	1975	Composition
623	Borosilicate Glass	1976	Composition
633	Portland Cement	1974	Composition
640	Silicon Powder for X-ray Diffraction	1974	Lattice Pattern
671	Nickel Oxide	1960	Composition
674	X-ray Powder Diffraction	1983	Intensity
679	Brick Clay	1987	Composition
680	High Purity Platinum	1967	Composition
702	Light Sensitive Plastic Chip	1966	Fading
705	Polystyrene	1963	Molecular Weight
740	Zinc Freezing Point	1970	Temperature
762	Magnetic Moment - Nickel Disk	2000	Magnetic Moment
767	Superconductive Fixed Point	1974	Temperature
772	Magnetic Moment - Nickel Sphere	1978	Magnetic Moment
781	Molybdenum - Heat Capacity	1977	Heat Capacity
870	Column Performance for LC	2000	Efficiency
877	Chiral Selectivity for LC	2000	Selectivity
909	Human Serum (Clinical)	1980	Composition
911	Cholesterol (Clinical)	1967	Composition
912	Urea (Clinical)	1968	Composition
913	Uric Acid	1968	Composition
914	Creatinine	1968	Composition
916	Bilirubin	1971	Composition
927	Bovine Serum Albumin (Total Protein)	1977	Composition

(Continuation of Table)

Selected Key NBS/NIST Reference Materials Beyond the First 100

SS/SRM	TITLE	DATE	APPLICATION
930	Glass Filters for Spectrophotometry	1971	Transmittance
934	Clinical Laboratory Thermometer	1974	Temperature
945	Plutonium Metal	1971	Assay
946	Plutonium Isotopic	1971	Composition
955	Lead in Blood	1984	Composition
968	Fat Soluble Vitamins in Human Serum	1989	Composition
987	Assay-Isotopic Strontium	1971	Assay & Composition
1001	X-ray Film Step Tablet	1973	Optical Density
1003	Calibrated Glass Spheres	1965	Length
1008	Photographic Step Tablets	1971	Optical Density
1010	Microcopy Resolution Test Charts	1963	Resolution
1013	Portland Cement	1962	Composition
1083	Wear Metals in Lubricating Oil	1985	Composition
1244	Inconel 600	1984	Composition
1246	Incoloy 800	1984	Composition
1261	AISI 4340 Steel	1970	Composition
1321	Coating Thickness - Nonmagnetic on Steel	1988	Length
1387	Coating Weight - Gold on Nickel	1985	Length
1400	Bone Ash	1992	Composition
1470	Polyester Film - Gas Transmission	1978	Transmission
1475	Linear Polyethylene	1969	Molecular Weight
1479	Polystyrene	1981	Molecular Weight
1491	Aromatic Hydrocarbons in Hexane/Toluene	1989	Composition
1492	Chlorinated Pesticide in Hexane	1989	Composition
1511	Multi-Drugs of Abuse in Freeze-dried Urine	1994	Composition
1515	Apple Leaves	1991	Composition
1521	Boron-doped Silicon Slices for Resistivity Meas.	1978	Resistance
1523	Silicon Resistivity for Eddy Current Testers	1985	Resistance
1544	Fatty Acids & Cholesterol in Frozen Diet	1996	Composition
1546	Meat Homogenate	2000	Composition
1547	Peach Leaves	1991	Composition
1548	Total Diet	1990	Composition
1549	Non-fat Milk Powder	1984	Composition
1563	Cholestrol & Fat-Soluble Vitamins in Coconut Oil	1987	Composition
1566	Oyster Tissue	1979	Composition
1567	Wheat Flour	1978	Composition
1568	Rice Flour	1978	Composition
1570	Trace Elements in Spinach	1976	Composition
1571	Orchard Leaves	1971	Composition
1577	Bovine Liver	1972	Composition

(Continuation of Table)
Selected Key NBS/NIST Reference Materials Beyond the First 100

SS/SRM	TITLE	DATE	APPLICATION
1579	Powdered Lead Based Paint	1973	Composition
1580	Organics in Shale Oil	1980	Composition
1581	Polychlorinated Biphenyls in Oils	1982	Composition
1582	Petroleum Crude Oil	1984	Composition
1583	Clorinated Pesticides in 2,2,4-trimethylpentane	1985	Composition
1584	Priority Pollutant Phenols in Methanol	1984	Composition
1588	Organics in Cod Liver Oil	1989	Composition
1589	Polychlorinated Biphenyls in Human Serum	1985	Composition
1590	Stabilized Wine	1980	Alcohol Content
1598	Inorganic Constituents in Bovine Serum	1989	Composition
1599	Anticonvulsant Drug Level Assay	1982	Assay
1601	Carbon Dioxide in Nitrogen	1973	Composition
1604	Oxygen in Nitrogen	1968	Composition
1610	Hydrocarbon in Air	1969	Composition
1614	Dioxin	1985	Composition
1617	Sulfur in Kerosine	1988	Composition
1619	Sulfur in Residual Fuel Oil	1981	Composition
1625	Sulfur Dioxide Permeation Tube	1970	Composition
1630	Trace Mercury in Coal	1971	Composition
1633	Trace Elements in Coal Fly Ash	1974	Composition
1634	Trace Elements in Fuel Oil	1978	Composition
1636	Lead in Reference Fuel (Gasoline)	1975	Composition
1640	Trace Elements in Natural Water	1997	Composition
1641	Mercury in Water	1974	Composition
1643	Trace Elements in Water	1977	Composition
1645	River Sediment	1978	Composition
1646	Estuarine Sediment	1982	Composition
1647	Priority Pollutant PAHs	1981	Composition
1648	Urban Particulate Matter	1978	Composition
1649	Urban Dust / Organics	1982	Composition
1650	Diesel Particulate Matter	1985	Composition
1659	Methane in Air	1976	Composition
1661	Sulfur Dioxide in Nitrogen	1976	Composition
1669	Propane in Air	1973	Composition
1677	Carbon Monoxide in Air	1974	Composition
1683	Nitric Oxide in Nitrogen	1974	Composition
1745	Indium Freezing Point	1998	Temperature
1761	Low Alloy Steel	1985	Composition
1866	Bulk Asbestos - Common	1988	Composition
1895	Nickel Microhardness - Knoop	1984	Hardness

(Continuation of Table)
Selected Key NBS/NIST Reference Materials Beyond the First 100

SS/SRM	TITLE	DATE	APPLICATION
1896	Nickel Microhardness - Vickers	1992	Hardness
1920	Near Infrared Reflectance Wavelength	1986	Wavelength
1921	Infrared Transmission Wavelength	1984	Wavelength
1922	Liquid Refractive Index - Mineral Oil	1999	Refractive Index
1930	Glass Filters for Spectrophotometry	1987	Transmittance
1941	Organics in Marine Sediment	1989	Composition
1945	Organics in Whale Blubber	1994	Composition
1951	Cholesterol in Human Serum (Frozen)	1988	Composition
1960	Nominal 10 Fm Dia. Polystyrene Spheres	1985	Length
1968	Gallium Melting Point	1977	Temperature
1974	Organics in Mussel Tissue	1990	Composition
2003	Aluminum on Glass - First Surface Mirror	1971	Reflectance
2030	Glass Filters for Transmittance	1976	Transmittance
2031	Metal-on-Quartz Filters Spectrophotometry	1984	Transmittance
2069	Scanning Electron Microscope Performance	1983	Efficiency
2071	Sinusoidal Roughness Specimen	1989	Roughness
2084	CMM Probe Performance	1994	Length
2092	Low-Energy Charpy V-Notch Test	1989	Energy
2109	Chromium (VI) Speciation Stan'rd Solution	1992	Composition
2121	Spectrometric Standard Solutions	1984	Composition
2135	Nickel/Cromium Thin-Film Depth Profile	1985	Length
2261	Chlorinated Pesticides in Hexane	1992	Composition
2287	Ethanol in Reference Gasoline	1995	Composition
2294	Reformulated Gasoline	1998	Composition
2381	Morphine & Codine in Freeze-dried Urine	1992	Composition
2383	Baby Food Composite	1997	Composition
2390	DNA Profiling Standard	1992	Structure
2392	Mitochondrial DNA Sequencing - Human	1999	Structure
2415	Battery Lead	1991	Composition
2416	Bullet Lead	1988	Composition
2524	Optical Fiber Chromatic Dispersion	1997	Dispersion
2526	(111) p-Type Silicon Resistivity	1983	Resistance
2531	Ellipsometric Parameters - SiO_2 on Silicon	1992	Length
2541	Silicon Resistivity - 0.01 ohm · cm Level	1997	Resistance
2556	Used Auto Catalyst	1993	Composition
2567	Catalyst Package for Lubricant Oxidation	1992	Composition
2570	Lead Paint Film	1999	Composition
2583	Trace Elements in Indoor Dust	1998	Composition
2670	Toxic Metals in Freeze-dried Urine	1993	Composition
2676	Metals on Filter Media	1975	Composition

(Continuation of Table)

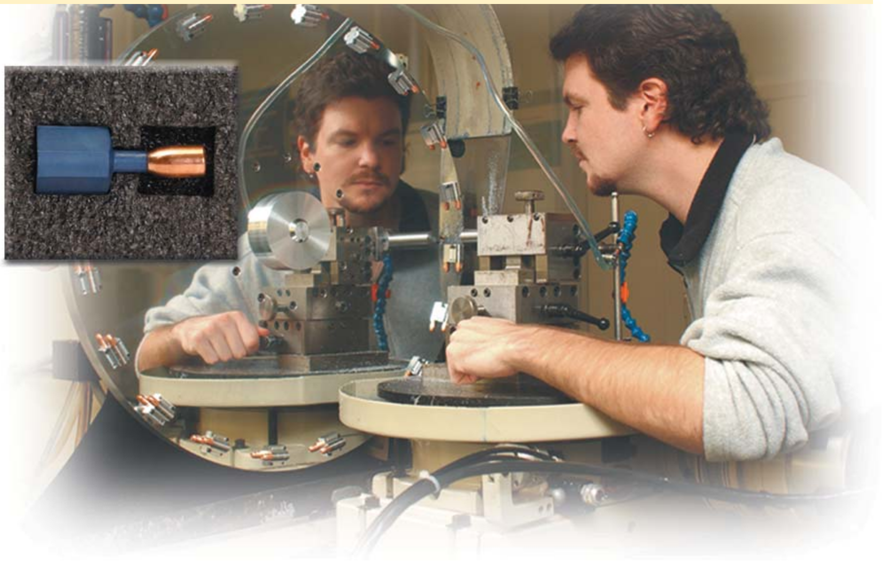
Selected Key NBS/NIST Reference Materials Beyond the First 100

SS/SRM	TITLE	DATE	APPLICATION
2682	Sulfur in Coal	1982	Composition
2694	Simulated Rainwater	1985	Composition
2710	Montana Soil	1992	Composition
2712	Lead in Reference Fuel (Gasoline)	1988	Composition
2782	Industrial Sludge	1998	Composition
2810	Rockwell C Scale Hardness - Low Range	1998	Hardness
2811	Rockwell C Scale Hardness - Mid Range	1998	Hardness
2812	Rockwell C Scale Hardness - High Range	1998	Hardness
3087	Metal on Filter Media	1990	Composition
3101-69	Element Solution for Spectrometry	1986	Composition
3171	Multielement Mix A Standard Solution	1988	Composition
3190	Aqueous Electrolytic Conductivity	1990	Conductivity
3200	Magnetic Tape - Computer Amplitude	1969	Signal Amplitude
4200B	Point Source Radioactivity	1965	Radioactivity
4201	Gamma Ray Niobium-94	1965	Radioactivity
4203B	Cobalt-60 Gamma Ray Point Source	1967	Radioactivity
4350	Environmental Radioactivity - River Sediment	1975	Radioactivity
4351	Environmental Radioactivity - Human Lung	1982	Radioactivity
4352	Environmental Radioactivity - Human Liver	1982	Radioactivity
4353	Environmental Radioactivity - Rocky Flats Soil	1981	Radioactivity
4355	Environmental Radioactivity - Peruvian Soil	1982	Radioactivity
4356	Ashed Bone	2000	Radioactivity
4357	Environmental Radioactivity - Ocean Sediment	1997	Radioactivity
4408-27	Radiopharmaceuticals (Short Half Lives)	1960's	Radioactivity

Appendix C Past and Future Driving Forces on Reference Material Production

The following table is the author's attempt to crystalize the most powerful driving forces on the selection and production of reference materials over the first century of NIST operation. On less stable ground, guesses are made at how new forces may develop over the course of the next century, thus shaping the SRM program in years to come.

TIME FRAME	DRIVING FORCES	SS/SRM RESPONSES
1901 - 1924	Automotive Age Explodes	Metals, Ores, and Cement
	Industrial Progress	Thermometry/Calorimetry
	WW I	Sucrose
1925 - 1949	Depression	Primary Chemicals
	Industrial Scale-up	Glass and Ceramics
	WW II	Aluminum Alloys
1950 - 1974	Korea	High Strength Alloys
	Space Race	High Temperature Alloys
	Vietnam	Metrology & Semiconductor
1975 - 2000	Human Needs	Environmental & ClinicalFood &
Info Age Explodes	Food & Agriculture	
	Economic Competition	Fuels, Oils & Engine Wear
Speculation:		
2001 - 2024	Biotech Revolution	New Suite of DNA
	Infrastructure Reconstruction	Lab on Chip Validators
		Many SRMs become NTRMs (NIST traceable reference materials)
2025 - 2049	New (Fusion?) Energy Age	Ultrastrength Materials
	Nanotechnology Breakthroughs	Nanometrology
2050 - 2074	Large-scale Environmental Reconstruction	Ultra Complex Natural Matrix Materials
2075 - 2100	Space Colonization	New Fuels, Foods, Materials



NIST's Numerically Controlled Diamond Turning Machine used to manufacture RM 8240 - Standard Bullets with identical signatures.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