ASTM Subcommittee E13.19 on Chromatography

John V. Hinshaw, Serveron Corp., Hillsboro, Oregon, USA.

The American Society for Testing and Materials (ASTM) is an international forum for the development and promulgation of consensus standards. In this month's "GC Connections," John Hinshaw writes about the role that ASTM International plays in chromatography standardization.

Standards play a crucial role in chromatography and other fields of endeavour that bring together academic, commercial and consumer concerns. In this context, a standard defines agreed-upon procedures, test methods, product descriptions, classifications, guides or nomenclature definitions. Recognized standards provide a common framework and a benchmark to which diverse groups can refer. A standard for testing detectors, for example, is useful to manufacturers for evaluation and quality control and to chromatographers for comparison between manufacturers, as well as for internal performance qualification. The same is true for columns, inlets, data-handling systems or any other instrument used in chromatography. Nomenclature standards enable chromatographers to understand each other through agreed-upon terms and expressions. Rapid and effective international communication — essential to the advancement of chromatographic science — would be greatly hindered without these standards. The usefulness of standards extends far beyond laboratories into the areas of jurisprudence and consumer affairs, as well.

Standards Organizations

A large number of organizations promulgate standards. Among them, the International Organization for Standardization (ISO, Geneva, Switzerland), the International Union for Pure and Applied Chemistry (IUPAC, Research Triangle Park, North Carolina, USA), the US Environmental Protection Agency (EPA, Washington, District of Columbia, USA), the *US Pharmacopeia* (USP, Rockville, Maryland, USA), and the American Society for Testing and Materials (ASTM International, West Conshohocken, Pennsylvania, USA) should be familiar to most *LC•GC Europe* readers as organizations that issue standards that involve chromatography in some form. ISO, for example, maintains 82 chromatography standards that specify analytical methods for a wide variety of materials, as well as the ISO 9000 quality management and ISO 14000 environmental management standards for which it is perhaps best known. IUPAC publishes the international standard for chromatography nomenclature and continues to issue periodic updates.[1] EPA publishes analytical standards for air, water and soil analysis. USP's *US Pharmacopeia–National Formulary*, which is revised annually, is the bible to pharmaceutical companies for all aspects of drug and related pharmaceutical products.

ASTM International: As excerpted from the ASTM Mission Statement found on its website at http://www.astm.org, ASTM International is a "…developer and provider of voluntary consensus standards, related technical information, and services having internationally recognized quality and applicability that promote public health and safety and the overall quality of life; contribute to the reliability of materials, products, systems and services; and facilitate national, regional and international commerce." Its first strategic objective is "to provide the optimum environment and support for technical committees to develop needed standards and related information."

Founded in 1898 to address problems with non-standardized steel railroad tracks, not-for-profit ASTM comprises an international technical framework within which committee members from various public and private concerns work to develop, maintain and disseminate standards. More than 30 000 members represent producers, users, ultimate consumers, those with a general interest and representatives of government and academia. Members from more than 100 countries participate in 140 or so technical committees concerned with approximately 11 000 standards in diverse areas, including stainless steel, skiing, food service, concrete pipe and, of course, chromatography.

Balanced participation: One of ASTM International's strengths is the requirement that committees maintain a balanced membership between producers, users and general interest members, which is reflected in the process of standards creation. This practice reinforces the intent of standards as tools to promote interchange between producers and users, as well as to protect the general welfare. No single interest can dominate a committee or unduly influence the standards.

ASTM committee structure — how it works: The primary organizational unit within ASTM International is the committee. ASTM committees are formed around a logical grouping of standards and activities. Table 1 lists the committees responsible for standards that use chromatography in some form. Currently, the ASTM web site lists 465 individual active standards that involve chromatography and 32 work items in

progress. These standards are written and revised constantly and discontinued occasionally as the needs for specific analytical tests arise and wane. Technology developments also provide the impetus for new standards creation. Standards can be revised to incorporate the latest technological advances as they become available.

Each committee consists of subcommittees responsible for a more focused topic area within the main committee's purview. Committee D02 on Petroleum Products and Lubricants, for example, includes among its 31 subcommittees several that involve chromatography — Hydrocarbon Analysis (D02.04), Gasoline and Oxygenated Fuels (D02.A0), Hydrocarbons for Chemical and Special Uses (D02.D0) and Liquefied Petroleum Gas (D02.H0). Each subcommittee is accountable for individual ASTM standards; subcommittee D02.D0 holds responsibility for eight chromatography standards.

Each of the subcommittees in turn forms task groups as needed to perform the detailed work of creating, modifying and preparing standards for approval; subcommittee D02.D0 includes individual task groups for ethylene, propylene, and C4 and C5 hydrocarbons. Task groups are formed when the subcommittee members identify and agree upon a specific standards development need; each task group can have responsibility for one or more standards. Figure 1 diagrams the relationships between the main committee, subcommittees and task groups.

Consensus standards: ASTM standards are classified into six categories: product specifications, test methods, practices, guides, classifications and terminologies. The consensus process enforces the usefulness and validity of ASTM standards by ensuring that standards are relevant and widely applicable from the beginning. After a standard is approved for use, periodic review and regular reapproval keep it current with changing requirements and technologies or bring about its eventual withdrawal or replacement.

With the consensus of a task group, a new or revised standard is returned to a subcommittee for voting. After being accepted at the subcommittee level, the entire main committee votes on the standard, and it is submitted for society review as well. Standards sometimes are voted concurrently by the sub- and main committees to streamline the approval process. The subcommittee responds to any negative votes, although a negative vote must be deemed persuasive and pass through a review process before a proposed standard is returned to the task group for further work.

Modern tools: Although it's now more than 100 years old, ASTM International is firmly ensconced in modern information technology and makes a number of electronic standards development tools available to its members. A series of detailed manuals, guidelines, templates and related documents are all available on-line at www.astm.org. ASTM maintains an on-line interactive standards forum facility for document development and discussions. A virtual meeting tool provides an on-line meeting place for groups to interact in real time. Electronic voting on standards facilitates timely balloting and greatly reduces mailing costs and the amount of paper used. A series of on-screen presentations for committee officer training also are available from the website. The *ASTM Annual Book of Standards* is always available in the print version, but the standards can be accessed more conveniently on annually updated CD-ROM disks. The standards also are indexed and searchable on the ASTM website.

Figure 1: ASTM committee, subcommittee and task group structure.

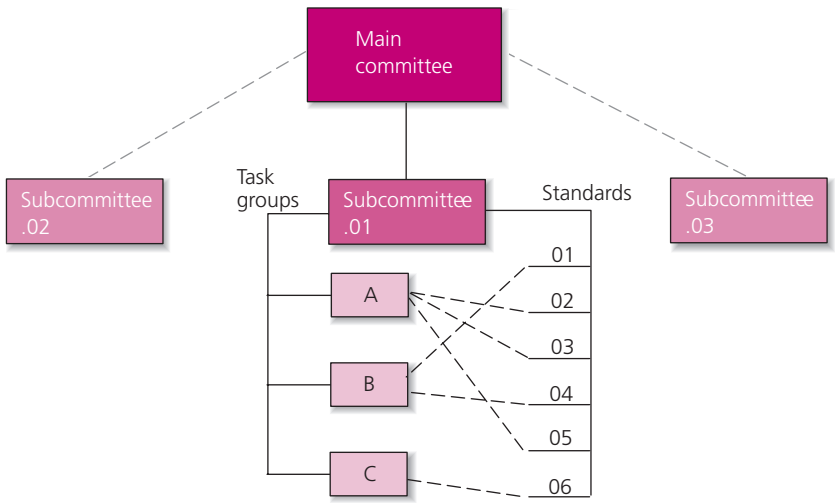

Table 1: ASTM technical committees responsible for standards that use chromatography

Committee Number	Committee Name
D01	Paint and Related Coatings, Materials, and Applications
D02	Petroleum Products and Lubricants
D03	Gaseous Fuels
D11	Rubber
D15	Engine Coolants
D16	Aromatic Hydrocarbons and Related Chemicals
D19	Water
D20	Plastics
D22	Sampling and Analysis of Atmospheres
D26	Halogenated Organic Solvents and Fire Extinguishing Agents
D27	Electrical Insulating Liquids and Gases
D34	Waste Management
E15	Industrial and Specialty Chemicals
E47	Biological Effects and Environmental Fate

ASTM Activities in Chromatography

ASTM International has been involved with gas chromatography (GC) almost from its beginnings in 1952. The first 10 years of GC saw a tremendous surge in research and applications in all areas. Petroleum chemists, in particular, immediately realized the potential of GC as a new and powerful means for characterizing crude, refined and synthetic petroleum materials. Simultaneously, GC columns and instrumentation underwent rapid development — new instruments and techniques appeared on the market almost every month.[2] However, the interchangeability of columns and GC methods that modern chromatographers take for granted did not exist in the 1950s. There was essentially no standardization between the different instrument companies and suppliers. Columns were made for a specific instrument–injector–detector combination, and stationary phases and supports varied considerably from one manufacturer to another.

Table 2: ASTM Standards from Subcommittee E13.19 on Chromatography.*

Designation	Year First Approved	Description
E260-96(2001)	1965	Standard practice for packed-column GC
E355-96(2001)	1968	Standard practice for GC terms and relationships
E516-95a(2000)	1974	Standard practice for testing thermal-conductivity detectors used in GC
E594-96(2001)	1977	Standard practice for testing flame ionization detectors used in GC or supercritical fluid chromatography (SFC)
E682-92(2000)	1979	Standard practice for LC terms and relationships
E685-93(2000)	1979	Standard practice for testing fixed-wavelength photometric detectors used in LC
E697-96(2001)	1979	Standard practice for use of electron-capture detectors in GC
E840-95(2000)	1981	Standard practice for using flame photometric detectors in GC
E1140-95(2000)	1986	Standard practice for testing nitrogen–phosphorus thermionic ionization detectors for use in GC
E1151-93	1993	Standard practice for ion chromatography terms and relationships
E1303-95(2000)	1989	Practice for refractive index detectors used in LC
E1449-92(2000)	1992	Standard guide for SFC terms and relationships
E1510-95(2000)	1993	Standard practice for installing fused-silica open-tubular capillary columns in GC
E1511-93(2000)	1993	Standard practice for testing conductivity detectors used in LC and ion chromatography
E1657-98(2001)	1994	Standard practice for testing variable-wavelength photometric detectors used in LC
E1698-95(2000)	1995	Standard practice for testing electrolytic conductivity detectors used in GC
E1747-95(2000)	1995	Standard guide for purity of carbon dioxide used in supercritical fluid applications

*The number after the hyphen indicates the year in which a standard was created or last revised; the year that it was most recently voted for approval is in parentheses. For example E355 was first approved in 1968, last revised in 1996 and reapproved in 2001. ASTM standards must be reapproved every five years, so all the E13.19 standards will be up for reapproval in the 2003–2004 period. If a standard is not reapproved after eight years, it can be discontinued.

The beginnings of ASTM E19: ASTM Committee D02 on Petroleum Products and Lubricants, founded in 1904, is arguably the first ASTM committee that became involved with GC early in its development. By the end of the 1950s, ASTM members recognized the need for a separate committee to coordinate standards concerning the practice of GC, so they formed the ASTM Committee E19 on Chromatography in 1961. The purpose of the new committee was — and still is — to advance the field of chromatography by promoting exchange of information, by sponsoring meetings and symposia, by standardizing terminology, and by coordinating and formulating scientific practices and methods of analysis applicable to the entire range of chromatography practice. The work of this committee is coordinated with other ASTM committees and organizations that have mutual interests. In 1968, the scope of the committee was generalized from GC to chromatography to accommodate liquid chromatography (LC). Today, the committee is involved with all forms of chromatography, including ion chromatography, gel-permeation chromatography, thin-layer chromatography and supercritical fluid chromatography.

The first products of E19 activities included the "Recommended Practice for Gas Chromatography Terms and Relationships (E355)," which was approved in 1968, and a series of standards on flame ionization, thermal-conductivity and electron-capture detectors followed soon after (see Table 2). During its first decade, E19 members were active in all areas of chromatography. E19 committee reports from 1968–1970 describe, among other things, task groups for standardization of methods, general GC procedures, cooperative sample testing, LC data, and standard materials and reagents.[3,4]

During the late 1960s, members were interested in assembling standardized GC retention data and related standard methodology that would permit chromatographers to identify unknown substances by their retention behaviour. The subcommittee on Storage and Retrieval of GC Data reported at the E19 Ninth Annual Meeting in 1970 that 1817 copies of Gas Chromatographic Data Compilation[5] had been sold, and that the "…first supplement

will soon be published…[and] a second supplement…will appear in 1972".[4] The compilation included more than 23 000 individual pieces of retention data. Several related task groups and committees described their efforts to abstract data from published sources and to correct errors in the compilation. This Herculean effort might seem unwieldy and unnecessary to modern chromatographers, but the routine use of mass spectrometry (MS) detectors with gas chromatographs was completely unknown at that time.

The 1960s–1970s: The E19 meetings of the 1960s and 1970s were well attended; for example, 235 registrants attended in 1968 and 151 in 1970. The committee membership grew from 208 to 246 between 1968 and 1970 and remained more or less at that level until the 1980s. The annual meetings included topical sessions and subcommittee and task-force activities. Topics for the 1970 and 1971 meetings included GC quantification and standardization, high-resolution capillary column analysis of hydrocarbons, LC of high molecular weight hydrocarbons, applications of gel-permeation chromatography, life science applications of LC, new technology in LC, practical aspects of performing LC separations, environmental analysis by chromatography, chromatography laboratory automation and troubleshooting in GC.

The 1980s–1990s: The high interest level and broad-ranging treatment of chromatography topics continued at E19 annual meetings for almost two decades. I attended my first ASTM meeting in 1980 and joined soon afterward. I remember attending meetings in San Diego, California, USA; Atlanta, Georgia, USA; Norfolk Beach, Virginia, USA; and Kansas City, Missouri, USA. Registration often exceeded 100 and various seminars and short courses on current chromatography topics were held. Standards development activities continued at a high pace during this period. The committee produced new standards in areas such as LC refractive index, fixed-wavelength and conductivity detectors; GC nitrogen–phosphorus thermionic, electrolytic conductivity and flame photometric detectors; capillary column installation; supercritical fluid chromatography; and ion chromatography as listed in Table 2.

Beginning in 1995 or so, active interest in E19 began to wane. Probably as a reflection of the beginnings of a consolidation in chromatography industries, fewer committee members had the time and corporate endorsements for active pursuit of E19 activities. As a consequence, the annual E19 symposia were discontinued, although the biannual committee business meetings in conjunction with the Pittsburgh Conference and other conferences continue. In 2000, Committee E19 was consolidated into Committee E13 (Molecular Spectroscopy) as the E13.19 Subcommittee on Chromatography. At that time, all existing E19 standards were reapproved, which has ensured their continuity for the time being. Today, E13.19 has 39 active members from a cross-section of the chromatographic community. (See sidebar for more information about upcoming events.)

The consolidation of the late 1990s heralded a new phase for traditional GC and LC as once-novel techniques found their way onto the palettes of routine chemical analysts. Ironically, the widespread acceptance of fused-silica capillary GC columns and diode-array LC detectors — to name only two of the many new chromatography instruments whose use became routine — meant that the need for high-quality consensus standards became greater than ever. The downsizing trend in industrial laboratory staffing challenged less-experienced personnel with unfamiliar techniques, which in turn caused an upswing in demand for chromatography standards as guidelines for laboratory operations; instrument purchase, installation and maintenance; data storage and retrieval; interpretation of results; and other aspects of routine chromatographic analysis.

Both of these trends — the adoption of new chromatography techniques into the array of routine methodologies and the development of new as-yet unproven technologies — will determine the course of chromatography standards from this time forward.

The Future of Chromatography Standards

What is the future of chromatography standards? It is the same as the future for chromatography. No one will argue that chromatography is not a viable and growing field, nor will they say that the routine chromatographic techniques have no place in present or future laboratories. Technologies that use chromatography continue to develop rapidly, but they don't necessarily look like the more mature methodologies. Chromatography meetings, publications, research projects and other activities abound in forward-looking areas such as LC–MS and proteomics, micro–total analysis systems (μTAS) and comprehensive two-dimensional GC. At the same time, further-developed techniques — such as capillary electrophoresis (CE) and related electrodriven liquid separations, high-speed GC, high-temperature LC, chiral and molecularly imprinted separations, solid-phase microextraction and high-speed MS, to name a few — are at various stages of integration into laboratory environments.

Both of these trends — the adoption of new chromatography techniques into the array of routine methodologies and the development of new as-yet unproven technologies — will determine the course of chromatography standards from this time forward. In turn, the creation of appropriate consensus standards with the participation of all interested parties will help the transition from academic discovery to practical implementation.

ASTM Subcommittee E13.19 is ideally suited as a forum and framework for developing standards and maintaining existing chromatography standards as appropriate. Successful engagement in these activities, however, requires strategic planning, research, standards proposals, task group formation and standards creation. The current situation in E13.19 represents a great opportunity for interested parties to become involved and have a chance to contribute in these activities. The committee invites participation at any level

Future ASTM E13.19 Meetings

18 November 2003:

Autumn 2003 meeting of ASTM E13.19, Somerset, New Jersey, USA; held in conjunction with Eastern Analytical Symposium.

7–12 March 2004:

Spring 2004 meetings of ASTM E13.19, Chicago, Illinois, USA; held in conjunction with Pittcon 2004; exact time and location to be determined.

by academic, manufacturing, end user and other interested parties. For additional information, please go to the ASTM International website at http://www.astm.org. I have created a special e-mail address on my personal website at e13.19@chromsource.com and I will try to answer any questions that the readers of this "GC Connections" column might have about reenergizing chromatography standards activities within ASTM International.

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"GC Connections" editor **John V. Hinshaw** is senior staff engineer at Serveron Corp., Hillsboro, Oregon, USA, and a member of the Editorial Advisory Board of *LC•GC Europe*.

Direct correspondence about this column to "GC Connections," *LC•GC Europe,* Advanstar House, Park West, Sealand Road, Chester CH1 4RN, UK, e-mail: dhills@advanstar.com

For an on-going discussion of GC issues with John Hinshaw and other chromatographers, visit the Chromatography Forum discussion group at http://www.chromforum.com