1 Introduction

1.1 EVOLUTION OF GAS CHROMATOGRAPHIC COLUMNS

The gas chromatographs and columns used today in gas chromatography have evolved gradually over five decades, similar to the evolution and advancements made in the cars we drive, the cameras we use, and the television sets that we view. In retrospect, the first gas chromatographs may be considered rather large compared to the modern versions of today, but these were manufactured for packed columns. Also, the prevailing thinking of the day was that “bigger was better,” in that multiple packed columns could be installed in a large column oven. This is not necessarily true in all cases today, as now we know that a large column compartment oven offers potential problems (e.g., thermal gradients, hot and cold spots) if a fused-silica capillary column is installed in a spacious oven. The columns used in the infancy of gas chromatography were prepared with metal tubing such as copper, aluminum, and stainless steel. Only stainless steel packed columns remain in use; columns fabricated from the more reactive metals copper and aluminum are no longer used, and the use of copper tubing in gas chromatography has basically been limited to carrier gas and detector gas lines and ancillary connections.

Packing of such columns proved to be an event, often involving two or more people and a stairwell, depending on the length to be packed. After uncoiling the metal tubing to the desired length and inserting a wad of glass wool into one end and attaching a funnel to the other end, packing material would be added gradually while another person climbed the stairs taping or vibrating the tubing to further settle the packing in the column. When no further packing could be added, the funnel was detached, a wad of glass wool inserted at that end, and the column coiled manually to the desired diameter. These tapping and vibration processes produced fines of packing materials and ultimately contributed to the overall inefficiency of the chromatographic process. Glass columns were soon recognized to provide an attractive alternative to metal columns, as glass offers a more inert surface texture, although these columns are more fragile, requiring careful handling; have to be configured in geometrical dimensions for the instrument in which they are to be installed; and the presence of silanol groups on the inner glass surface has to be addressed through silylation chemistries. Additional features of glass columns are that one can visualize how well a column is packed, the presence of any void regions, and the possible discoloration of the packing at the inlet end of the column due to the

Columns for Gas Chromatography: Performance and Selection, by Eugene F. Barry and Robert L. Grob

accumulation of high boilers and particulates, which indicates that it is time for fresh packing. Most laboratories today leave the preparation of packed columns to column vendors. A generation of typical packed columns fabricated from these materials are shown in Figure 1.1; packed columns are discussed in Chapter 2.

The evolution of the open-tubular or capillary column may be viewed as paralleling that of the packed column. The first capillary columns that demonstrated efficiency superior to that of their packed column counterparts were made primarily of stainless steel. Glass capillary columns gradually replaced stainless steel capillary columns and proved to offer more inertness and efficiency as well as less surface activity, but their fragility was a problem, requiring straightening of column ends followed by the addition of small aliquots of fresh coating solution. Perhaps the most significant advance in column technology occurred in 1979 with the introduction of fused silica by Hewlett-Packard (now Agilent Technologies) (1,2). Today, the fused-silica capillary column is in wide use and its features, such as superior inertness and flexibility, have contributed to concurrent improvements in inlet and detector modifications that have evolved with advances in stationary-phase technology. Because of the high impact of fused silica as a column material, resulting in excellent chromatography, numerous publications have focused on many aspects of this type of column. For example, the interested reader is referred to an informative review by Hinshaw, who describes how fused-silica capillary columns are made (3), and some guidance offered by Parmely, who has outlined how successful gas chromatography with fused-silica columns can be attained (4). A generation of capillary columns are shown in Figure 1.2; capillary columns are the subject of Chapter 3.

The first group of stationary phases were adsorbents, somewhat limited in number, for gas–solid chromatography with packed columns, and included silica gel, alumina, inorganic salts, molecular sieves, and later, porous polymers and graphitized carbons, to name a few. Today, porous-layer open tubular or PLOT columns employ these adsorbents as stationary phases where adsorbent particles adhere to the inner wall of fused-silica capillary tubing. However, more numerous were the number of liquids studied as liquid phases for gas–liquid chromatography. In 1975, Tolnai and co-workers indicated that more than 1000 liquids had been introduced as stationary liquid phases for packed columns up to that time (5); to state that almost every chemical in an organic stockroom has been used as a stationary liquid phase is probably not much of an exaggeration.

Some popular liquid phases in the early 1960s are listed in Table 1.1. The majority of these are no longer in routine use (exceptions being SE-30, Carbowax 20M, squalane, and several others) and have been replaced with more thermally stable liquids or gums. Also of interest in this list is the presence of Tide, a laundry detergent, and diisodecyl, dinonyl, and dioctyl phthalates; the phthalates can be chromatographed easily on a present-day column. From 1960 through the mid-1970s, a plethora of liquid phases were in use for packed column gas chromatography to provide the selectivity needed to compensate for the low efficiency of the packed column to yield a given degree of resolution. When classification schemes of liquid phases were introduced by McReynolds and Rohrschneider (see Chapter 2), the number of liquid phases for packed columns decreased gradually over time.

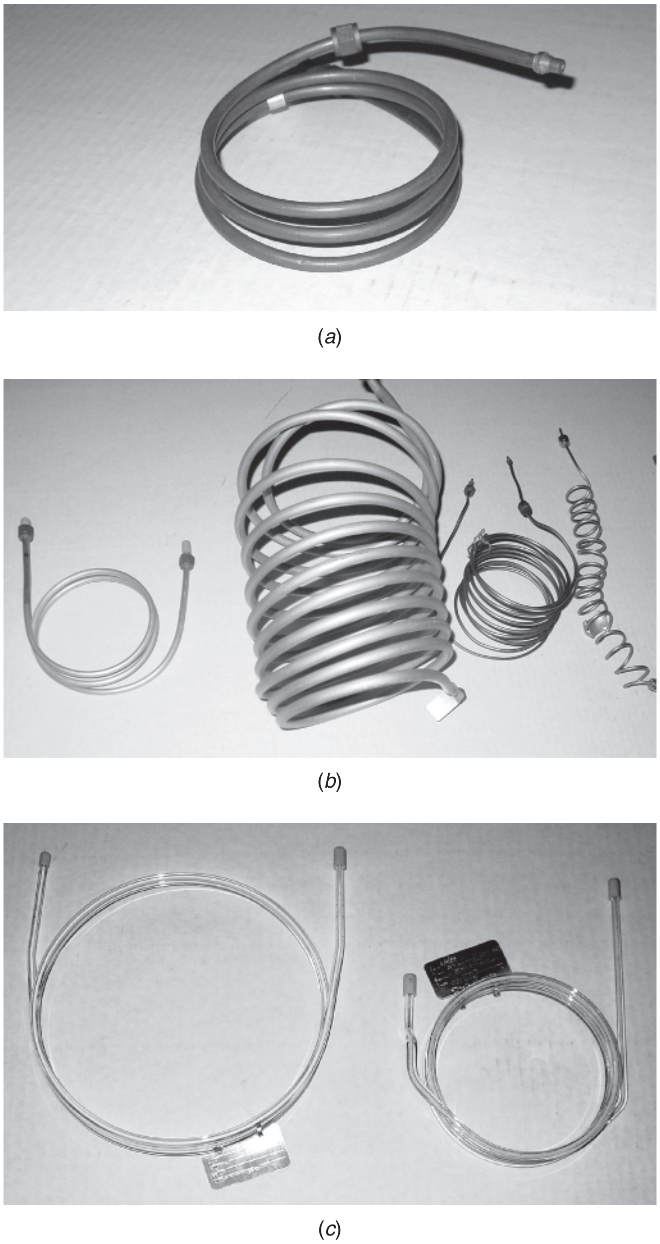
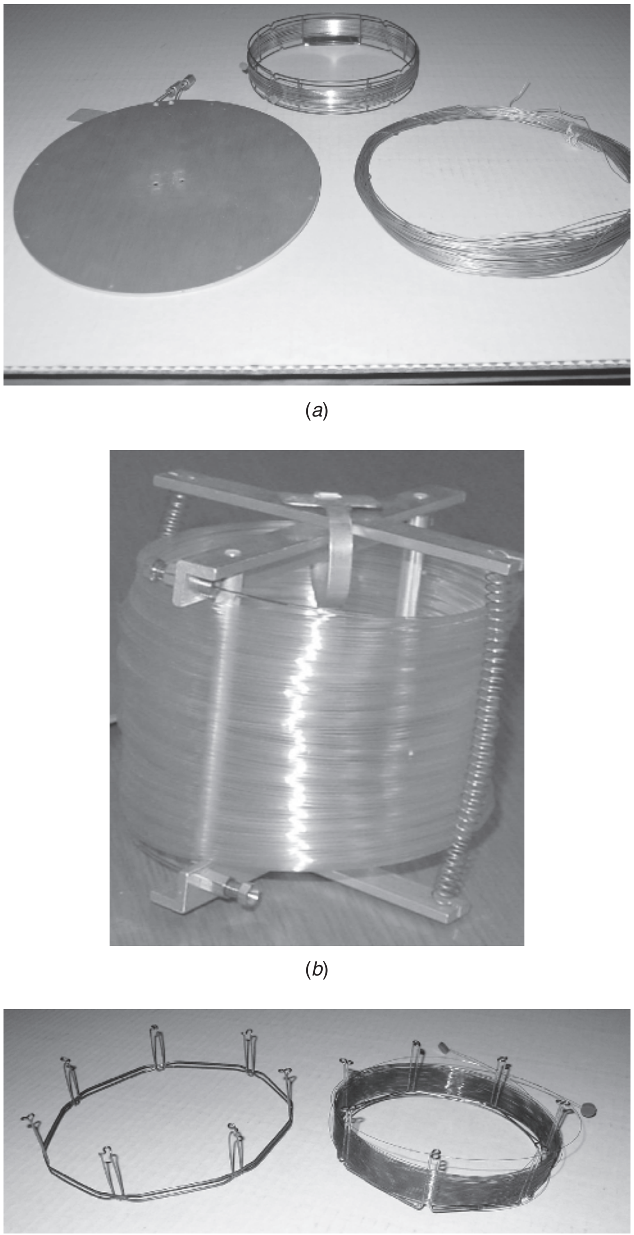


Figure 1.1 Various columns and materials used for packed column gas chromatography: (*a*) 6 ft × 0.25 in. o.d. copper tubing; (*b*) from left to right: 4 ft × 0.25 in. o.d. aluminum column, 20 ft × $\frac{3}{8}$ in. o.d. aluminum column for preparative GC, 10 ft × 1/8 in. o.d. stainless steel column, 3 ft × $\frac{1}{8}$ in. o.d. stainless steel column coiled in a "pigtail" configuration; (*c*) glass packed gas chromatographic columns, 2 m × 0.25 in. o.d. × 4 mm i.d. Note the differences in the length configuration of the ends, specific to two different chromatographs.



(*a*)

(*b*)

(*c*)

Figure 1.2 Various columns and materials employed for capillary gas chromatography: (*a*) left: 25 m × $\frac{1}{16}$ in. o.d. stainless steel capillary column in a "pancake" format, center: 30 m × 0.25 mm i.d. aluminum-clad fused-silica column, right: blank or uncoated stainless steel capillary tubing $\frac{1}{16}$ o.d.; (*b*) 60 m × 0.75 mm i.d. borosilicate glass capillary column for EPA method 502.2; (*c*) 30 m × 0.25 mm i.d. fused-silica capillary column; also pictured is a typical cage used to confine and mount a fused-silica column.

TABLE 1.1 Stationary Phases Used in Gas Chromatography Prior to 1962[a]

Liquid Phase	Maximum Temp. (°C)
Inorganic eutectic mixtures	>350
Silicone elastomer E301	300
Silicone rubber gum SE-30	250–350
DC high-vacuum grease	250–350
Apiezon M	275–300
Polyethylene	275–300
Apiezon L	240–300
Ethylene glycol–isophthalic acid polyester	280
Embaphase silicone oil	250–260
Neopentyl glycol succinate	230–250
Carbowax 20M	220–250
Polyphenyl ether	250
Tide detergent	225–250
Resoflex R446 and R447	240
Polyester	
Diethylene glycol succinate (LAC-3-R728)	200–225
Cross-linked diethylene glycol adipate (LAC-2-R446)	200–225
Ucon polyglycol LB-550-X	200–225
Ucon 50 HB 2000	200–240
Carbowax 6000	200–225
Carbowax 4000	200–225
Carbowax 4000 monostearate	200–220
Celanese ester No. 9	200
Convoil 20	200
Nonylphenoxypoly(ethyleneoxy)ethanol	200
Convachlor-12	200
Triton X-305	200
Reoplex 400	190–270
DC silicone oil 550	190–225
DC silicone oil 200	175–225
Carbowax 1500	175–200
Diisodecyl phthalate	175–180
Dioctyl phthalate	160
Zinc stearate	160
Nujol paraffin oil	150–200
Sucrose acetate isobutyrate	150–200
Apiezon N	150
Sorbitol-silicone oil X525	150 150
Bis(2-ethylhexyl) tetrachlorophthalate	150
Polypropylene glycol	140–150
7,8-Benzoquinoline	115–150
Dinonyl phthalate	130–150
Carbowax 1000	125–190
Bis(2-ethylhexy1) sebacate	125–175
Tricresyl phosphate	125–160
Carbowax 600	125–150
Benzyldiphenyl	120–140
Fluorene picrate	120
Diglycerol	100–150
THEED (tetrahydroxyethylethylenediamine)	100–135
Carbowax 400	100–130
Squalane	80–140
*Glycero*l	70–120
β, β'-Oxydipropionitrile	50–100
Dibenzyl ether	60–80
Hexadecane	40–60
Tetraethylene glycol dimethyl ether	40–80
Propylene glycol–$AgNO_3$	40–50
Di-*n*-butyl maleate	40–50
Dimethylsulfolane	35–40
Quinoline–brucine	25
Dimethylformamide	0

Source: Data from ref. 6.

[a] Phases in italic type may be viewed as obsolete.

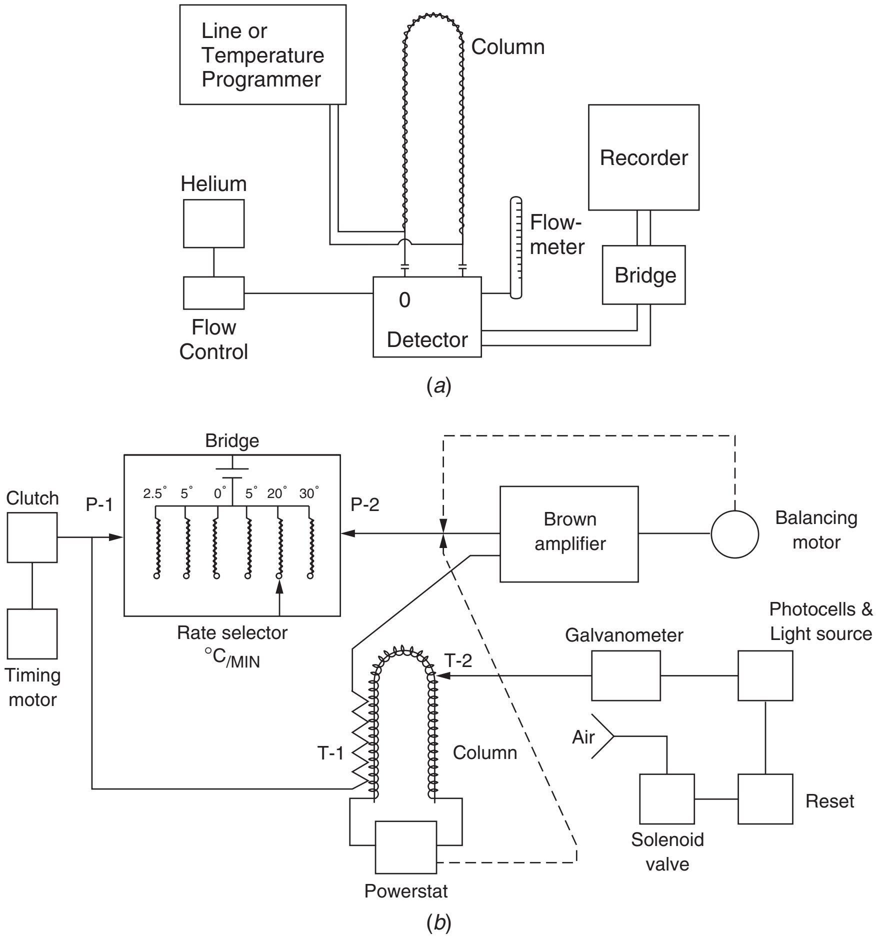
In this reduced number of phases, only a small fraction proved useful in capillary gas chromatography (GC), where thermal stability of thin films of stationary liquids at elevated temperatures and wettability of fused silica, for example, become key chromatographic issues. On the other hand, only a few stationary phases of relatively lower selectivity are needed in capillary GC because of the much higher efficiency of a capillary column. Industrial-grade lubricants such as the Apiezon greases and Ucon oils suitable for the packed column needs of the day were replaced by more refined synthetic or highly purified versions of polysiloxanes or polyethylene glycols. Polysiloxanes, for example, are one of the most studied classes of polymers and may be found as the active ingredients in caulks, window gaskets, contact lenses, and car waxes; the first footprints on the moon were made by polysiloxane footwear (7). Another well-studied class of polymers are the more polar polyethylene glycols (PEGs), which also have use in a variety of applications (e.g., one active component in solutions used in preparation for colonoscopy procedures is PEG 3550). However, as efficacious and effective as polysiloxanes and polyethylene glycols may be in these applications, many studies have shown that only those polysiloxanes and polyethylene glycols that have well-defined chemical and physical properties satisfy the requirements of a stationary phase for capillary GC, as discussed in Chapter 3.

The reader will find equations for the calculation of column efficiency, selectivity, resolution, and so on, in Chapter 2. Included among these equations is an expression for time of analysis, an important parameter for a laboratory that has a high sample throughput. Temperature programming of a column oven, operation of a gas chromatographic column at a high flow rate or linear velocity, selection of favorable column dimensions, and optimization of separations with computer assistance can all reduce analysis time. In the last decade, fast or high-speed GC has emerged as a powerful mode in gas chromatography and is treated in Chapter 4. As gas chromatography comes closer to becoming a mature analytical technique, one tends to focus on the present and may forget early meritorious pioneering efforts, particularly the role of temperature programming for fast gas chromatography. Such is the case with temperature programming in GC, introduced by Dal Nogare and his colleagues, the first proponents of its role in reducing the time of analysis (8,9). The first reported separation in fast GC and schematic diagrams of circuitry of the column oven are shown in Figure 1.3.

1.2 CENTRAL ROLE PLAYED BY THE COLUMN

The gas chromatographic column may be considered to be the central item in a gas chromatograph. Over the last three decades, the nature and design of the column has changed considerably from one containing either a solid adsorbent or a liquid deposited on an inert solid support packed into a length of tubing to one containing an immobilized or cross-linked stationary phase bound to the inner surface of a much longer length of fused-silica tubing. With respect to packing materials, as noted earlier, solid adsorbents such as silica gel and alumina have been replaced by porous polymeric adsorbents, and the vast array of stationary liquid phases in the 1960s was by the next decade reduced to a much smaller number of phases of greater thermal stability. These stationary phases became the precursors of the chemically bonded or cross-linked phases of today. Column tubing fabricated from copper, aluminum, glass, and stainless steel served the early analytical needs of gas chromatographers. Presently, fused-silica capillary columns 10 to 60 m in length and 0.20 to 0.53 mm in inner diameter are in widespread use.

Although gas chromatography may be viewed in general as a mature analytical technique, improvements in column technology, injection, and detector design appear steadily nonetheless. During the last decade, innovations and advancements in gas chromatography have been made with the merits of the fused-silica column as the focal point and have been driven primarily by the environmental, petrochemical,



(*a*)

(*b*)

Figure 1.3 (*a*) Programmed-temperature apparatus constructed by Dal Nogare and Harden; (*b*) closed-loop proportional temperature controller; (*c*) programmed-temperature separation of a C5–C10 paraffin mixture: (A) heating rate 30°C/min, flow rate 100 mL/min, starting temperature 40°C, (B) same conditions except heating rate 5°C /min, (C) isothermal separation at 75°C, flow rate 100 mL/min. [From ref. 8. Reprinted with permission from *Anal. Chem.*, **31** 1829–1832 (1959). Copyright © 1959 American Chemical Society.] (*Continued*)

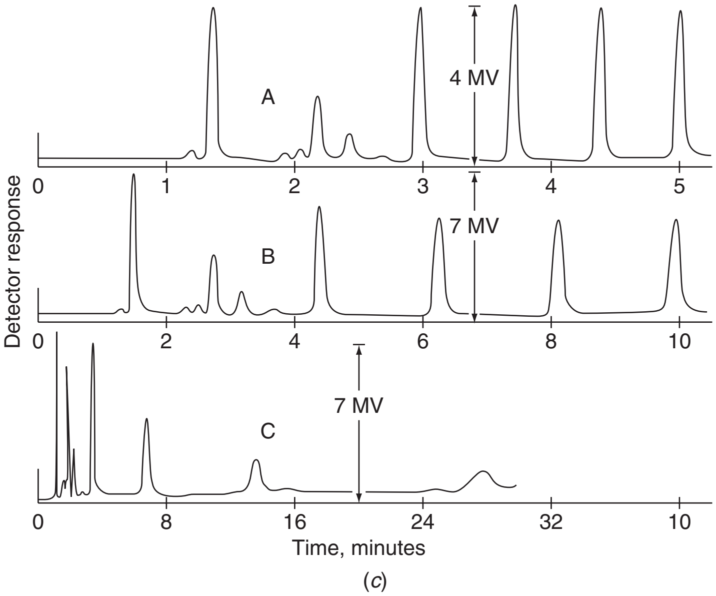


Figure 1.3 (*continued*)

and toxicological fields as well as by advances in sample preparations and mass spectrometry. Despite being a mature technology, there are three parallel tracks on which advancements in gas chromatography steadily appear. First, continued improvements in column technology have resulted in more efficient and thermally stable columns; second, advances in both electronics and pneumatics have provided impetus to fast GC, and third, sample preparation, mass spectrometry, and multidimensional GC represents three additional areas where great strides continue to be made. The interested reader may refer to the fourth edition of *Modern Practice of Gas Chromatography* for detailed coverage of all aspects of GC (10).

1.3 JUSTIFICATION FOR COLUMN SELECTION AND CARE

The cost of a gas chromatograph can range from $6000 to over $100,000, depending on the type and number of detectors, injection systems, and peripheral devices, such as a data system, headspace and thermal desorption units, pyrolyzers, and autosamplers. When one factors in purchase of the high-purity gases required for operation of the chromatograph, it quickly becomes apparent that a sizable investment has been made in capital equipment. For example, cost-effectiveness and good chromatographic practice dictate that users of capillary columns should give careful consideration to column selection. The dimensions and type of capillary column should be chosen with the injection system and detectors in mind, considerations that are virtually nonissues with packed columns. Careful attention should also be paid to properly implemented connections of the column to the injector and detector and the presence of high boilers, particulate matter in samples, and so on.

The price of a column ($200 to $800) may be viewed as relatively small compared to the initial, routine, and preventive maintenance costs of the instrument. In fact, a laboratory may find that the cost of a set of air and hydrogen gas cylinders of research-grade purity for FID (flame ionization detector) operation is far greater than the price of a single conventional capillary column! Consequently, to derive maximum performance from a gas chromatographic system, the column should be carefully selected for an application, handled with care following the suggestions of its manufacturer, and installed as recommended in the user's instrument manual.

The introduction of inert fused-silica capillary columns in 1979 markedly changed the practice of gas chromatography, enabling high-resolution separations to be performed in most laboratories (1,2). Previously, such separations were achieved with reactive stainless steel columns and with glass columns. After 1979, the use of packed columns began to decline. A further decrease in the use of packed columns occurred in 1983 with the arrival of the megabore capillary column of 0.53-mm inner diameter (i.d.), which serves as a direct replacement for a packed column. These developments, in conjunction with the emergence of immobilized or cross-linked stationary phases tailored specifically for fused-silica capillary columns and the overall improvements in column technology and affordability of mass spectrometry (MS), have been responsible for the wider acceptance of capillary GC.

Trends. The results of a survey of 12 leading experts in gas chromatography appeared in 1989 and outlined their thoughts on projected trends in gas chromatographic column technology, including the future of packed columns versus capillary columns (11). Some responses of that panel are:

1. Packed columns are used for approximately 20% of gas chromatographic analyses.
2. Packed columns are employed primarily for preparative applications, for fixed gas analysis, for simple separations, and for separations for which high resolution is not required or not always desirable [e.g., polychlorinated biphenyls (PCBs)].
3. Packed columns will continue to be used for gas chromatographic methods that were validated on packed columns, where time and cost of revalidation on capillary columns would be prohibitive.
4. Capillary columns will not replace packed columns in the near future, although few applications require packed columns.

Shortly thereafter, in 1990, Majors summarized the results of a more detailed survey on column use in gas chromatography, this one, however, soliciting response from *LC/GC* readership (12). Some conclusions drawn from this survey include:

1. Nearly 80% of the respondents used capillary columns.
2. Capillary columns of 0.25- and 0.53-mm i.d. were the most popular, as were column lengths of 10 to 30 m.

3. The methyl silicones and polyethylene glycol stationary phases were preferred for capillary separations.
4. Packed columns were used primarily for gas–solid chromatographic separations such as gas analyses.
5. The majority of respondents indicated the need for stationary phases of higher thermal stability.

Majors conducted helpful GC user surveys again in 1995 (13) and 2003 (14). In the 2003 survey, the use of packed columns continued to decline because many packed column gas chromatographic methods have been replaced by equivalent capillary methods. There are now capillary column procedures for the U.S. Environmental Protection Agency (EPA), American Association of Official Analytical Chemists (AOAC), and *U.S. Pharmacopeia* (USP) methods. Despite the increase in capillary column users (91% in 2003 compared to 79% in 1990), there is still a significant number of packed column users, for several reasons: (1) packed columns and related supplies and accessories have a substantial presence in catalogs and Web sites of the major column vendors, and (2) the use of packed columns become apparent to the authors of this text after discussions with attendees in short courses on GC offered at professional meetings.

Other interesting findings in this 2003 survey included:

1. A pronounced increase in the use of columns of 0.10 to 0.18-mm i.d. Their smaller inner diameter permits faster analysis times and sensitivity, and their lower capacity is offset by the sensitive detectors available.
2. Columns of 0.2 to 0.25- and 0.32-mm i.d. in 20 to 30-m lengths continue to be the most popular.
3. 100% Methyl silicone, 5% phenylmethyl silicone, polyethylene glycol (WAX), and 50% phenylmethyl silicone continue to be the most popular stationary phases.
4. There appears to be a shift from gas–solid packed columns for the analysis of gases and volatiles to PLOT columns.

Column manufacturers rely on the current literature, the results of marketing surveys, the number of clicks on their Web sites, and so on, to keep abreast of the needs of practicing gas chromatographers. The fused-silica capillary column has clearly emerged as the column of choice for most gas chromatographic applications. A market research report covering 1993 (15) showed that $100 million was spent on capillary columns worldwide, and at an estimated average cost of $400 for a column, this figure represented about 250,000 columns. The number of columns and users has increased considerably since then, along with the cost of columns. Despite the maturity of capillary GC, instrument manufacturers continue to improve the performance of gas chromatographs, which has diversely extended the applications of gas chromatography.

Chromatographers can expect to see continued splendid efforts by capillary column manufacturers to produce columns that have lower residual activity and are capable of withstanding higher-column-temperature operation with reduced column bleeding. With the increasing popularity of high-speed or fast GC (Chapter 4) and the increasing presence of GC-MS in the analytical laboratory, especially for environmental, food, flavor, and toxicological analyses, improvements in column performance that affect the MS detector have steadily evolved, such as columns with reduced column bleed. There is also an increased availability of capillary columns exhibiting stationary-phase selectivity tuned for specific applications obtained by synthesis of new phases (16). For example, enhanced separation of the congeners of polychlorinated dibenzodioxin (PCDD), furan (PCDF), and PCBs can be achieved with the selectively tuned columns commercially available (17–21), discussed in more detail in Chapter 4. Interesting studies on blending stationary phases and phase preparation with guidance from computer modeling (22) and molecular simulation studies in gas–liquid chromatography have appeared in the literature (23,24).

1.4 LITERATURE ON GAS CHROMATOGRAPHIC COLUMNS

The primary journals in which developments in column technology and applications are published in hard-copy format and online versions include *Analytical Chemistry, Journal of Chromatography* (Part A), *Journal of Chromatographic Science, Journal of Separation Science* (formerly the *Journal of High Resolution Chromatography*, including the *Journal of Microcolumn Separations*), *and LC/GC* magazine. The biennial review issue of *Analytical Chemistry*, Fundamental Reviews (published in even-numbered years), contains concise summaries of developments in gas chromatography. An abundance of gas chromatographic applications may be found in the companion issue, Application Reviews (published in odd-numbered years), covering the areas of polymers, geological materials, petroleum and coal, coatings, pesticides, forensic science, clinical chemistry, environmental analysis, air pollution, and water analysis.

Most industrial and corporate laboratories as well as college and universities have access to literature searching through one of a number of online computerized database services (e.g., *SciFinder Scholar*). Although articles on gas chromatography in primary journals are relatively easy to locate, finding publications of interest in lesser known periodicals can be a challenge and is often tedious. *CA Selects, SciFinder Scholar*, and *Current Contents* are convenient alternatives. The biweekly *CA Selects*, Gas Chromatography topical edition available from Chemical Abstracts Service is a condensation of information reported throughout the world. *SciFinder Scholar* is a powerful searching capability, as it is connected to Chemical Abstracts Service but can retrieve information rapidly by either topic or author. *Current Contents*, in media storage format, provides weekly coverage of current research in the life sciences; clinical medicine; the physical, chemical, and earth sciences; and agricultural, biology, and environmental sciences.

With each passing year, the periodic commercial literature and annual catalogs of column manufacturers (in compact disk format from many column vendors) describing applications for their columns contain more and more useful technical information of a generic nature. In addition, we strongly recommend *LC/GC* as a valuable resource not only for timely technical articles but also “Column Watch” and a troubleshooting section for GC. However, the Internet has emerged as the most extensive source of chromatographic information in recent years, particularly the Web sites of column manufacturers.

1.5 GAS CHROMATOGRAPHIC RESOURCES ON THE INTERNET

The World Wide Web (WWW) has provided us with copious amounts of information through retrieval with search engines offered by an Internet service provider (ISP) (25). The Internet has affected our everyday activities with the convenience of communication by e-mail, online placement of orders for all types of items, and many other functions. There are numerous Web sites on gas chromatography in general, gas chromatographic columns, gas chromatographic detectors, and so on; all one has to do is locate them by “surfing the net.” All manufacturers of gas chromatographic instrumentation, columns, and chromatographic accessories and supplies maintain Web sites and keep them updated. We strongly suggest that you identify and visit regularly the Internet addresses of column manufacturers, for example, and “bookmark” the corresponding Web sites. Internet addresses may change over time, as in the case of expansion or consolidation. For example, there has been some consolidation in the column industry for GC: J&W was purchased by Agilent Technologies, Chrompack by Varian, and Supelco by Sigma-Aldrich, and new column manufacturers (Phenomenex and VICI Gig Harbor Group) have entered the marketplace. It is thus impractical here to list the Web addresses of vendors, but “home pages” are easily searchable and updated continually, thus serving as an outstanding source of reference material for the practicing chromatographer. Lists of Web sites and addresses of vendors may be found in the annual *ACS Buyers’ Guide* as well as in its counterparts in *LC/GC* and *American Laboratory*.

Listing all the gas chromatographic resources available on the Internet is not practical, but resourceful guides and information (often, in streaming video) that are available at gas chromatographic sites include:

- Information describing e-notes, e-newsletters, and e-seminars offered by a vendor
- Free downloads of software (e.g., retention time locking, method translation)
- Technical libraries of chromatograms searchable by solute or class of solute
- Column cross-reference charts
- Application notes
- Guides to column and stationary-phase selection
- Guides to selection of inlet liners
- Guides to column installation
- Guides to derivatization

- Troubleshooting guides
- Guides for syringe, septa, and ferrule selection
- Guides for setting up a gas chromatograph
- Past presentations at professional meetings such as Pittcon and EAS

Of the plethora of informative and significant ".com" and ".org." sites that are available, one deserves special mention because it serves as a path both for immediate assistance and for the continuing education of users of GC and HPLC: *the Chromatography Forum*, maintained by LC Resources (www.lcresources.com). There are several message boards (i.e., a GC message board, an LC message board, and several others) where one can post anonymously a chromatographic problem or question and others can post a response, initiating a dialogue on the topic. This site offers broadening of one's knowledge of the technique, even for the experienced user, and is a particularly valuable asset for an analyst working in an environment in which he or she is the sole chromatography user or does not have access to other resources or assistance with technical problems. Sometimes clickable links are overlooked as possibly noteworthy chromatographic sites; such sites may be embedded in a primary site selected by a search engine such as *Yahoo* or *Google*. An illustration of this is the site maintained by John Wiley, www.separationsnow.com, where there are several links, including one for Discussion Forums on GC, hyphenated techniques, HPLC, and others. Again, visit pertinent Web sites as part of the ongoing professional growth process of a chromatographer; also remember that a Web address may change sooner or later.

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