FEATURE

Development of Gas Chromatography Instruments at Shimadzu

第２２回キャピラリークロマトグラフィー国際シンポジウムが日本の岐阜で開かれるのに際して、本稿に招請された島津の代表者が日本の最も大きな科学機器製造会社の歴史とこの社のガスクロマトグラフィー製品の発展における主要な出来事をまとめている。１８７５年に普通の科学装置製造業者として出発した後、島津は１９５０年代中頃には最初のガスクロマトグラフを製造し製品化した。

在日本岐阜市召开的第２２届毛细管色谱国际会议即将到来之际，日本岛津公司的代表应邀总结了岛津公司这一日本科学仪器生产大厂的发展历史及其在气相色谱产品开发中的一些重大事情。岛津公司作为一综合科学仪器生产厂家创建于1875年，在1950年代中旬建立及安装了其第一个气相色谱实验室。

Guest authors Nagayanagi, Takimoto, and Saito review the development and manufacturing of gas chromatography instrumentation from Shimadzu Corp., Japan's leading scientific instrument company. They also provide a brief history of the company to explain the company's design and marketing philosophies.

In the past I have discussed the evolution of chromatographic instrumentation; however, these assessments always referred to the instruments developed and marketed by American companies. A major instrument industry also evolved in Japan in parallel to the American industry. Although some Japanese instruments are marketed in the United States, most consumers know very little about the background of the instruments and the companies that produce them. On the occasion of the forthcoming 22nd International Symposium on Capillary Chromatography to be held on 8–12 November 1999 in Gifu, Japan, I have asked members of Shimadzu's chromatography team to summarize the history of the company and the key events in the development of its chromatographic products.

— Leslie S. Ettre

The 10th annual meeting of the Japan Society for Analytical Chemistry was held at Tokyo University in the early spring of 1957 and accompanied by an exhibition. In the morning of the third day of the meeting, someone affixed a poster to a tree near the entrance hall with a handwritten message that stated "The first commercial gas chromatograph made in Japan will be exhibited this afternoon." This small poster worked quite well — a long queue in front of the booth formed soon after noon. The crowd included even most of the famous professors who were Japan's pioneers in gas chromatography (GC).

Their impressions and comments were warm and helpful. Most of the comments were considered in the subsequent improvements, and before the year's end the instrument's manufacturer — Shimadzu Corp. (Kyoto, Japan) — had produced 11 units of the model GC-1A gas chromatograph. Four were used within the company in the application laboratory and for demonstration purposes, the fifth went to Tokyo University, and the rest were sold to the following industrial companies: Tokuyama Soda, Maruzen Petroleum, Sumimoto Chemicals, Mitsubishi Rayon, Idemitsu Kosan Petroleum, and Kao Soap.

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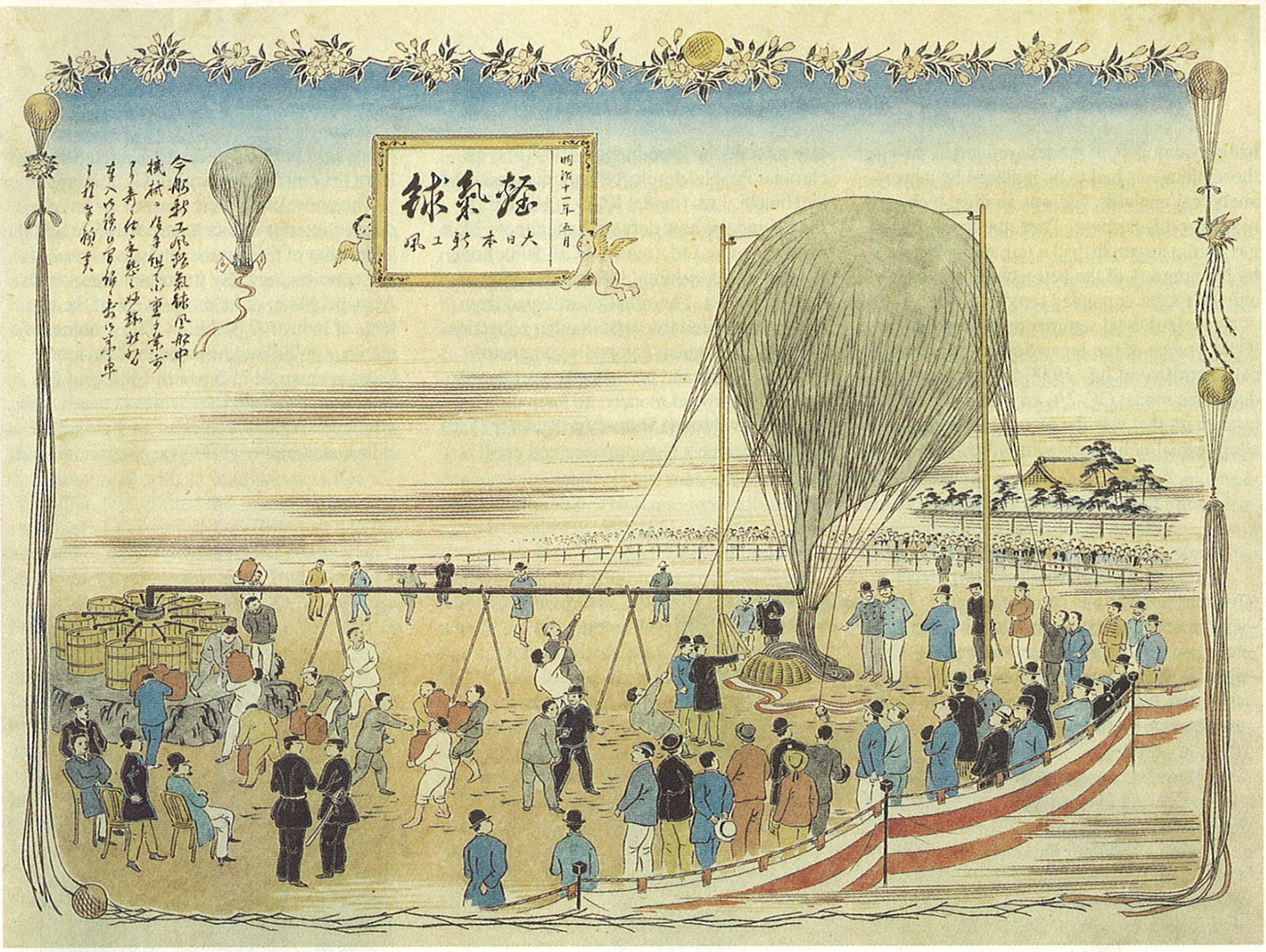


This instrument, the model GC-1A, actually was not the first commercial gas chromatograph in Japan. Earlier Shimadzu built an experimental model on special order for Maruzen Petroleum Co. Ltd. and installed it in February 1956. In the same year, Perkin-Elmer (Norwalk, Connecticut, USA) exhibited its model 154 gas chromatograph at an industrial fair in Japan and installed two units, one in an industry laboratory and one at a university laboratory.

THE EVOLUTION OF SHIMADZU

Shimadzu was founded in 1875 in Kyoto by Genzo Shimadzu (1839–1894), who was a traditional metal craftsman of Buddhist altar equipment. The Meiji Era, a period of rapid modernization, had just started. To facilitate the rapid introduction of science, the Japanese government invited several European and American scientists and technicians as teachers. One of them was Gottfried Wagener (1831–1892), a German chemist who had been active in Kyoto and had a key role in establishing chemistry as a scientific discipline in Japan. To fulfill Wagener's laboratory needs, Genzo Shimadzu began to produce scientific equipment, mainly for educational purposes, and in a short time became a pioneering manufacturer in Japan. He published his first illustrated catalogue in 1882.

Genzo Shimadzu also became famous for building a manned balloon and launching it on 6 December 1877 from the old imperial palace grounds in Kyoto before a large crowd. The intention of this flight was to promote the spread of scientific education and knowledge in Japan. Its success greatly contributed to these goals. The new company's symbol was the crest of the Shimadzu family — a circle with a cross — that was granted to one of Genzo's ancestors during the period of Toyotomi Hideyoshi (1537–1598), the famous Japanese general.

After the sudden death of Genzo Shimadzu in 1894, his son, also named Genzo Shimadzu (1868–1945), took over as the head of the company. He was talented in both science and business and is considered one of Japan's 10 greatest inventors. He is credited with a total of 178 inventions during his lifetime. In October 1896, less than a year after the discovery of X rays by Röntgen in Würtzburg, Germany, the younger Genzo constructed the first X-ray apparatus in Japan, and Japan's first commercial X-ray apparatus soon followed these first experiments. Medical X-ray equipment still represents one of Shimadzu's most important product lines.

Another business started by the younger Genzo was the production of storage batteries. He obtained an excellent patent for the production of a very powerful battery. In 1936 a dispute with American firms that infringed on this invention resulted in a judicial ruling that granted Shimadzu patent royalties in the sum of $80 million. Eventually the battery business became another company, called the Japan Storage Battery Co. Ltd.

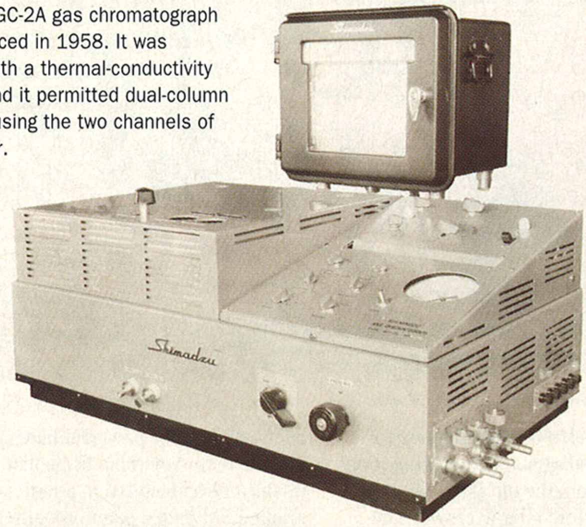
In the recovery period after World War II, Shimadzu concentrated on scientific and medical instrumentation, first absorbing foreign technologies, but also developing new and original technologies and designs, particularly since the 1960s. More recently Shimadzu expanded and became a truly global corporation, based on four cornerstones: Japan, the United States, Europe, and Asia–Oceania. Today Shimadzu has approximately 4500 employees in Japan.

GC AT SHIMADZU IN THE '50S AND '60S

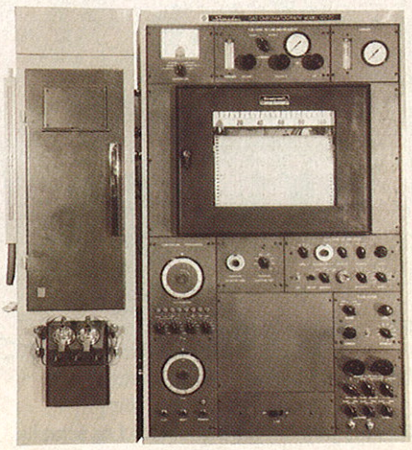
The shortage of foreign currency in Japan after World War II forced the country to rely on domestic coal supplies for the production of energy. However, the efficiency of oil and the demand for petrochemical products changed the situation within a decade. By the mid-1950s the country had a construction boom of petroleum refineries and petrochemical complexes. The old distillation and other testing methods were inadequate for the quality control requirements of modern refineries, so those processes had to be replaced by newer analytical methods. GC was an ideal technique for this purpose. Thus, the first Japanese gas chromatographs had a tailwind generated by the progress of the petrochemical industry, and their sales expanded rapidly.

At an analytical seminar organized by the Kinki branch of the Japan Society for Analytical Chemistry in July 1958, Shimadzu introduced the model GC-2A, an improved instrument that was the first in the world to adopt a dual-column flow line. The year 1958 also marked the introduction of the first gas chromatographs developed and manufactured by Hitachi, Ltd. (model KGL-1, Hitachi, Japan) and the now defunct Yanagimoto Manufacturing Co. Ltd. (model GCG-500). Soon JEOL, Ltd. (Akishima, Japan), also entered the GC market. The situation in Japan from then on was similar to that in other countries — major instrument companies competed with each other and periodically introduced new and improved models. To keep up with the rapid evolution, Shimadzu regularly sent several engineers to congresses and conferences held in Europe and the United States to learn the newest advances in GC.

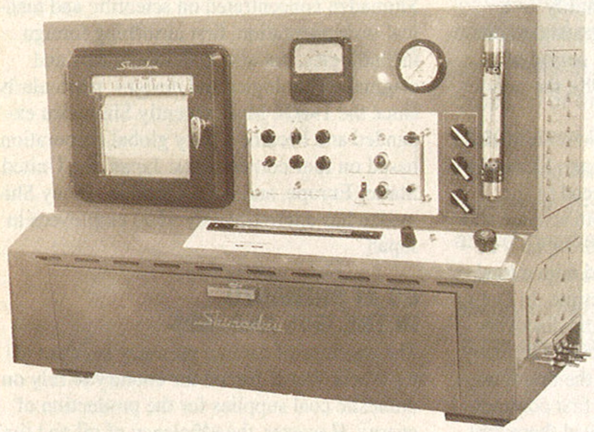
The early 1960s were marked by an increased interest in biochemical studies and the adaptation of GC for the analysis of steroids and other biologically important compounds. Also, people became more aware of the effects of industrial pollution. The problem of mercury pollution known as the Minamata Disease emerged in Japan in 1964, and the detection of residual pesticides in cow's milk astounded Japanese citizens. In 1971 polychlorinated biphenyl (PCB) contamination in



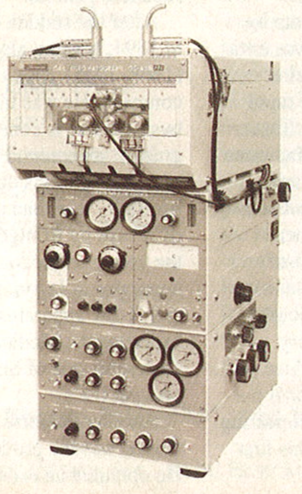
The model GC-2A gas chromatograph was introduced in 1958. It was equipped with a thermal-conductivity detector, and it permitted dual-column operation, using the two channels of the detector.



The model GC-1C gas chromatograph was introduced in 1963. It was a programmed-temperature gas chromatograph that permitted the simultaneous installation of as many as four detectors and the addition of a number of accessories. The vertical box on the left-hand side contained the column oven and the detectors.



The model GC-1A gas chromatograph was introduced in 1957. This large instrument measured 108 cm × 55 cm × 73 cm and weighed 120 kg. It could be heated to temperatures as high as 280 °C and was equipped with a thermal-conductivity detector.



The model GC-4BM gas chromatograph. The injector, column, and detector were incorporated in the top module, which could be replaced easily by another module containing different components. The other modules in this photograph contained the detector, carrier gas, and temperature controls; in this particular case, the detectors were a flame-ionization detector and an electron-capture detector.

humans generated societal concern and anxiety regarding environmental pollution. The Air Pollution Control Law was enacted in 1972. GC had been applied in the analysis of contaminants and pollutants responsible for all these problems, and being accelerated by the social trend, popularization of GC further advanced.

Of the instruments developed by Shimadzu in this period the model GC-1C gas chromatograph, introduced in 1963, should be mentioned. This gas chromatograph was a multistage, universal, programmed-temperature gas chromatograph with an optional mass flow controller. It permitted the simultaneous installation of as many as four detectors and the addition of a pyrolyzer, a precut column, and an automatic preparative unit. This instrument was exhibited in the United States at the 15th Pittsburgh Conference in March 1964. Tatsuro Haruki, Teiji Mori, and Katsuya Sato of Shimadzu presented a paper titled "The Effect of Injection Port Temperature on Peak Broadening in Gas Chromatography" at this conference.

In the evolution of Japanese gas chromatographs, Shimadzu's models GC-4A, GC-5A, and GC-4BM — introduced in 1965, 1969, and 1971, respectively — were particularly important. The GC-4A gas chromatograph was a popular, compact instrument with a modular design that permitted the simultaneous installation of two detectors. The GC-5A gas chromatograph had an all-glass flow path with greatly upgraded performance. The GC-4BM gas chromatograph had a unique design integrating the injector, column, and detector into one easily interchangeable unit.

By the second part of the 1960s, Shimadzu's gas chromatographs represented more than 80% of the market in Japan. Many factors contributed to this success, particularly the company's continuous contact with key scientists at universities and in the industry from whom they learned about the newest developments and needs.

DETECTORS AND COLUMNS

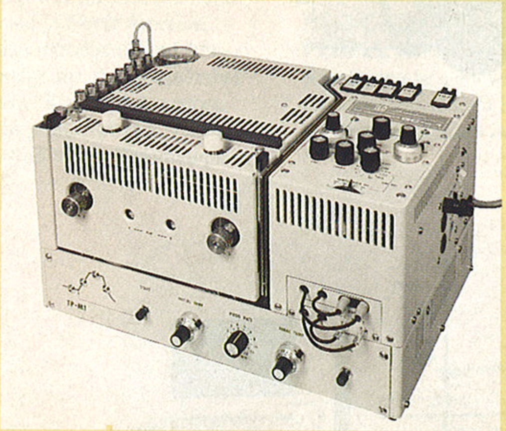
The first gas chromatographs used thermal-conductivity detectors but soon other detectors became available. The flame-ionization detector was offered by Shimadzu in 1959, and the company's lead soon was followed by Hitachi, Yanagimoto, and JEOL. The electron-capture and flame thermionic detectors became available in the first part of the 1960s, and the flame photometric detector was introduced in 1971. The electron-capture and flame photometric detectors in particular played major roles beginning in the 1970s in GC applications for the control of pesticide and PCB residues and atmospheric pollution.

In the early evolution of GC, the instrument manufacturers provided their own columns to users, and Shimadzu followed this example. Later dedicated column supply companies started to take over this task.

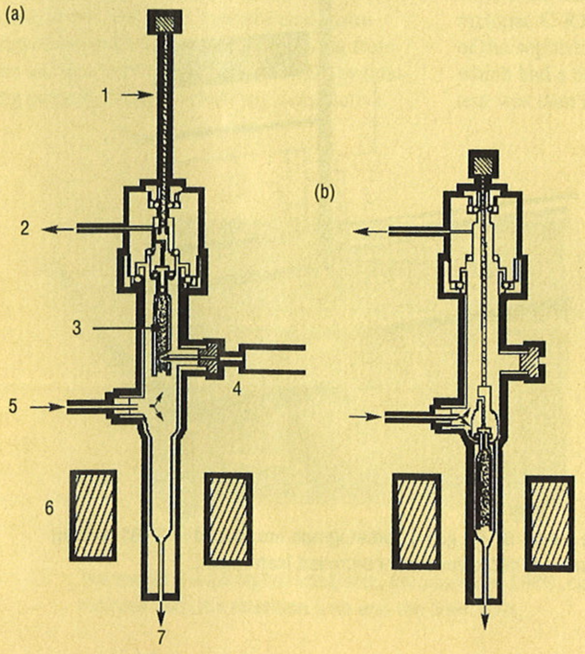
With respect to the capillary columns invented in 1958 by M.J.E. Golay (1), development in Japan was initially relatively slow, due mainly to the patent situation and the fact that columns made of metal tubing were not inert; thus, their use was restricted, except in petrochemical laboratories. The situation started to change with the introduction of glass tubing, and Shimadzu introduced the model GDM-1 glass capillary drawing machine in 1971, following the success of D.H. Desty in England approximately 10 years earlier (2). Shimadzu also developed the model MCT-1 system for the coating of the capillary tubes with the stationary phase, and introduced it in 1972. Both instruments were used extensively worldwide until circa 1980, when glass was replaced by flexible fused-silica tubing, following the development of Dandeneau and Zerenner in 1979 (3). In 1980 Shimadzu also introduced fused-silica capillary columns coated with a variety of liquid phases to Japan and soon followed with bonded-phase columns.

A crucial question with the use of capillary columns is the inlet system, and Shimadzu devoted considerable time and energy to the improvement of this part of the instrument. Two particularly noteworthy systems were the model SPL-1A injector (introduced in 1974), which had a fixed precolumn and could separate the solvent and solute in the injector, and the model SPL-7 split–splitless injector (introduced in 1983), which included a movable precolumn to eliminate the solvent at room temperature in the injector before the solutes were evaporated and conducted into the column.

In the mid-1970s Shimadzu developed a gas chromatograph that was specifically optimized for the use of glass capillary columns.



The GC-mini-1 gas chromatograph, designed primarily for glass capillary columns, was introduced in 1975. It had two injectors and two flame-ionization detectors, and it permitted the installation of one capillary column (with a make-up gas line to the detector) or two packed columns.



Schematics of the model SPL-7 split–splitless injector for capillary columns. It contained a movable precolumn into which the sample was injected. (a) Sample is injected into the precolumn which is at or close to room temperature. The solvent is eliminated through backflushing with the carrier gas. (b) By lowering the precolumn into the heated zone, the sample is evaporated and its vapor transferred with the carrier gas flow into the capillary column. 1 = plunger, 2 = carrier gas outlet, 3 = precolumn, 4 = syringe (sample injection), 5 = carrier gas inlet, 6 = heaters, 7 = capillary column connection.

Introduced in 1975, this system was the model GC-mini-1 gas chromatograph. With the increased use of capillary columns following the introduction of fused-silica columns, the newer gas chromatographs were designed primarily for capillary column operation.

OTHER SYSTEMS AND ACCESSORIES

In the early stage of GC development, preparative capability was much more in demand than it is today. Shimadzu developed the model GC-10A dedicated preparative gas chromatograph, introduced in 1963. However, customers preferred to have accessories for the laboratory instruments that provided the capability of injecting larger sample volumes and fraction collection. The model GC-1C gas chromatograph introduced in 1963 was available with this type of accessory.

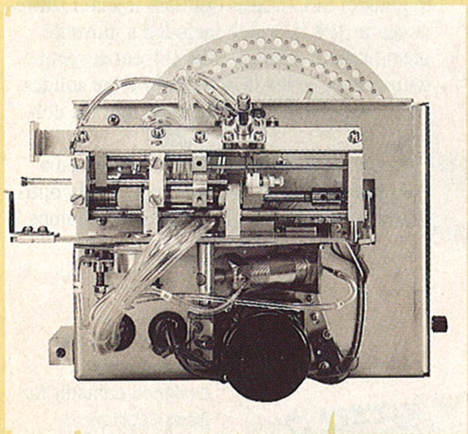
Another accessory sought by users of gas chromatographs, particularly in the first decade of its evolution, was a pyrolysis attachment. In 1964 Shimadzu introduced the model PYR-1A pyrolysis attachment, the first accessory of this type, and later the PYR-2A accessory.

Circa 1970, chromatographers began to look for automated sample introduction and labor-saving devices for analyzing many samples. Beginning in 1969 Shimadzu temporarily imported the model HP 7670 autosampler (Hewlett-Packard Co., Avondale, Pennsylvania, USA) for liquid samples and adapted it for the company's instruments. Meanwhile Shimadzu developed the model AOC-5A autosampler and introduced it in 1970. This unit permitted the automatic introduction of solid and liquid samples stored in capsules. However, the finest Shimadzu autosampler was the model AOC-6 introduced in 1973. It was a fully automated injector that could accommodate as many as 100 samples, operating with more than 50 relays and pneumatic valves. The operation of the autosampler's micro-syringe was very similar to manual operation — gentle pulling and quick pushing of the plunger was the key.

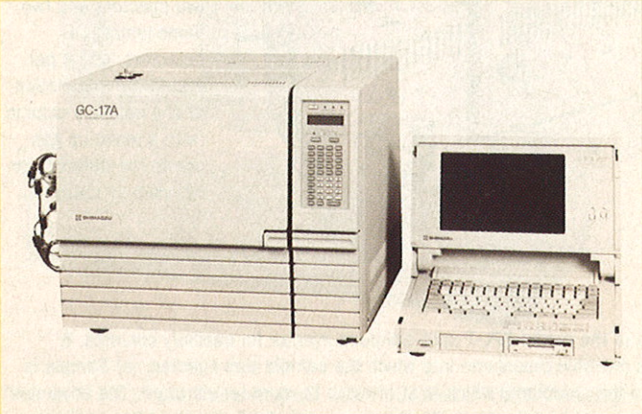
INSTRUMENT DEVELOPMENTS IN THE PAST 25 YEARS

Computerized gas chromatographs were introduced in the second part of the 1970s. The first of these instruments from Shimadzu was the microprocessor-controlled model GC-R1A gas chromatograph, which was introduced in 1978. After presetting the analytical conditions, the operation of the instrument, including data processing, could be performed automatically. This instrument was followed three years later by the microprocessor-controlled model GC-9A gas chromatograph, which had greatly improved control functions. This gas chromatograph also was marketed in the United States where it was received very favorably.

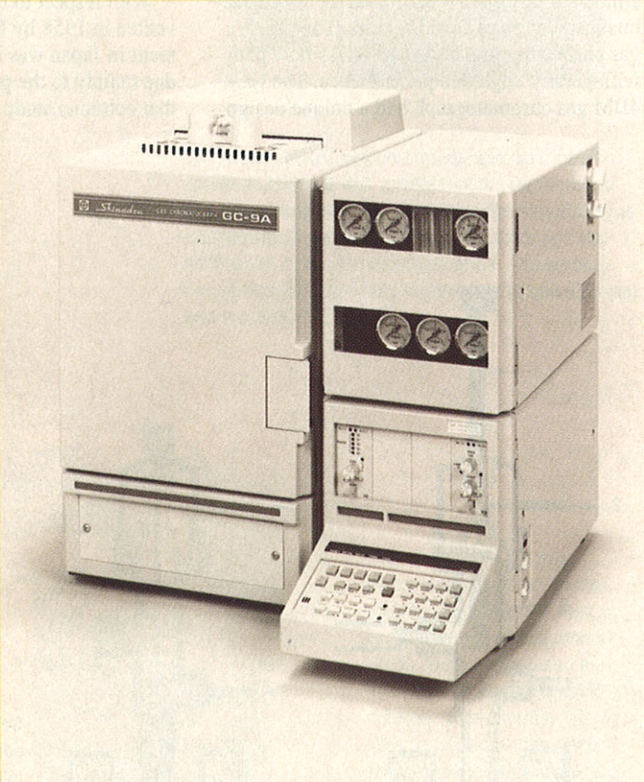
As mentioned earlier, the introduction of fused-silica tubing boosted the use of capillary columns, and this greatly influenced the future course of GC. Today most gas chromatographs are designed to use these columns. GC instruments also incorporate several new, sophisticated functions that broaden their use



The model AOC-6 autosampler. The photograph shows the top view of the unit.



The model GC-17A microprocessor-controlled gas chromatograph, introduced in 1992, had electronic flow controllers that permitted the digital setting of the carrier gas flow rate, pressure and velocity, and split ratio.



The model GC-9A gas chromatograph introduced in 1981 was an improved, microprocessor-controlled instrument.

and improve their reproducible operation. Of these new functions, electronic carrier-gas flow rate controllers are particularly noteworthy. In 1992 Shimadzu introduced the model GC-17A gas chromatograph, which had electronic flow control. This instrument included another pioneering feature: it enabled not only the digital setting of the carrier gas flow rate, pressure, and velocity but also of the split ratio. In the last seven years, Shimadzu made additional upgrades to the model GC-17A gas chromatograph, including digital setting capability of the detector gas flow rates.

GC–MS SYSTEMS

No report about GC development can be complete without discussing the combined GC–mass spectrometry (MS) systems. Shimadzu started MS work in 1967 and produced its first quadrupole mass spectrometer in 1968. Beginning in 1970 Shimadzu produced the LKB-9000 and the LKB-7000 GC–MS systems under license from the Swedish company LKB for a few years. Both of these systems used single-focusing, magnetic-sector mass spectrometers.

Meanwhile the company also developed the model QP-1000 GC–MS system, which contained a quadrupole-type mass spectrometric detector, and introduced it in 1972. This instrument underwent a number of modifications and improvements. The model QP-5000, introduced in 1992 at the Japanese Analytical Instruments Manufacturers' Association conference, was the next major GC–MS system. Updated versions of this system are still marketed today.

INTEGRATORS AND DATA SYSTEMS

The advances in data handling also are important to any report about the evolution of GC instrumentation. Shimadzu developed successively more and more sophisticated instruments that relieved analytical chemists from the cumbersome process of manually evaluating peak areas and performing quantitative

In the early stage of GC development, preparative capability was much more in demand than it is today.

calculations. Shimadzu's first digital integrator was the model ITG-1A integrator, introduced in 1969; its price at that time was ¥200 million (corresponding to $5600 at ¥360/$1). In the next five years Shimadzu developed further improved models of this line.

One weak point of digital integrators was that peak detection was easily affected by noise and baseline drift. In addition, demands for other calculations that use peak area increased and surpassed the computing power available in those early integrators. Based on a cooperative development agreement with Intel (Santa Clara, California, USA), Shimadzu started to develop a completely new concept of instruments that used Intel's 8080 microprocessor. The result of this activity was the Chromatopac C-1A digital integrator introduced in 1974. The integrator comprised a central processing unit, memory, and an input–output system, very much as in today's personal computers. The integrator used a sophisticated peak detection algorithm, and various calculation functions could be included. The Chromatopac C-E1A integrator was introduced in 1977, and it was the first Shimadzu data system marketed in North America.

In 1978 Shimadzu introduced the Chromatopac C-R1A integrator, the first member of the sophisticated C-R series integrators, which had a built-in printer–plotter. This system was then followed by the Chromatopac C-R2A and C-R3A integrators in 1981 and 1984, respectively. These instruments included two important new features: the ability to memorize chromatograms for reprocessing and the capability to run user-defined BASIC programs during data acquisition and processing. These two features facilitated the development of truly automated chromatographic functions, including complete system control, logical decision making, and detailed report generation. Even today, the software and the hardware of the C-RE3A remain essentially unchanged; only its appearance was modified periodically. The C-R3A series has been the world's best-selling integrator with a total production of more than 70,000 units.

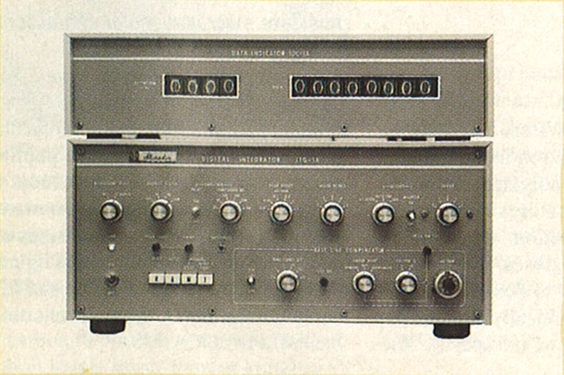
In the past decade Shimadzu developed a PC system named CLASS (for chemical laboratory analytical system and software), and the company continues to update it.

ACTIVITIES IN THE U.S. MARKET

When Shimadzu gas chromatographs were first marketed in the United States, they were distributed through the now defunct American Instruments Co. (Aminco). In 1975 Shimadzu established a fully owned subsidiary called Shimadzu Scientific Instruments Inc. in Columbia, Maryland. Since then the company's instruments have been marketed from this center, which continuously grew in size and importance. The facilities currently occupy more than 100,000 ft^2 and include a customer training and education center. From their modest start, the use of Shimadzu gas chromatographs grew rapidly and, according to a recent report about the 1999 Pittsburgh Conference, it reached a major place in the American market (4).

RECOGNIZING THE PEOPLE AT SHIMADZU

Results can be achieved only if the right persons are involved. At Shimadzu we were fortunate to have many dedicated scientists,


The model ITG-1A digital integrator, introduced in 1969, could indicate both the retention time and the peak area.


The Chromatopac C-R1A electronic data system had a built-in printer–plotter and was introduced in 1978.

chemists, and engineers participating in the development of gas chromatographs during the past 40 years. In this respect, we particularly must recognize the leading role of Tatsuro Haruki. He organized the GC research and development and production groups and the service system. His original training was that of a mechanical engineer, and he contributed the instrumentation chapter to a Japanese book on modern GC published in 1965.

The Shimadzu gas chromatography R&D group has been characterized by the participation of both chemists and engineers. The role of the chemists was particularly important: the engineers always needed to consult them before any decision on design was made. Sales documents also were written by chemists, because the main users were chemists. The evaluation, customer training, and collection of application data have always been the responsibility of the application laboratory, which is staffed by chemists. Without the dedicated members of these groups, Shimadzu could not succeed.

ACKNOWLEDGMENTS

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(3) R.D. Dandeneau and E.H. Zerenner, *HRC&CC* **2**, 351–356 (1979).
(4) R. Stevenson, *Am. Lab.* **31**(10), 36–59 (1999).

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***Illustration p. 41:** 1877 woodblock print by Fumio Satomi. Originally made for a Japanese newspaper to promote science, the print shows the launch of a hydrogen balloon by Shimadzu Corp. founder Genzo Shimadzu.*

APPLICATION NOTES

Capillary gel electrophoresis of polymerase chain reaction products

An application note from J&W Scientific describes a capillary electrophoresis analysis to determine the molecular size of DNA restriction fragments, polymerase chain reaction products, and synthetic oligonucleotides. The publication discusses a polymerase chain reaction sample dialysis procedure. The note includes sample chromatograms and lists analysis conditions. J&W Scientific Inc., Folsom, CA, USA.

Circle 100

Analysis of residual ethylene oxide in sterilized medical devices

Hewlett-Packard's application note describes the analysis of residual ethylene oxide in sterilized medical devices using headspace sampling and GC. The publication includes tables of system configuration and conditions, standard curve preparation, a method of standard addition, external calibration results for both systems, and ethylene oxide results from spiked polyvinyl chloride and high-density polyethylene resin beads. The note includes a chromatogram and standard addition plot. Hewlett-Packard Far East Pte. Ltd., Chemical Analysis Group, Singapore.

Circle 101

GC analysis

Restek's application note describes how a headspace autosampler and two capillary columns can be used for confirmational analyses of blood alcohols. The note discusses separations achieved by Rtx-BAC1 and -BAC2 capillary GC columns. The publication lists chromatographic conditions and instrumentation. Restek Corp., Bellefonte, PA, USA.

Circle 102

GPC–MS analysis of an internal suture polymer

An application note from Waters describes the coupling of GPC with electron ionization MS to analyze a lactide-based polymer used in the manufacture of internal sutures for surgical procedures. The publication discusses a method for comparing analyses of irradiated and nonirradiated material to determine if the irradiated material was chemically altered. The note includes chromatograms and spectra. Waters Asia Ltd., Singapore.

Circle 103

High-temperature GPC–viscometry

An application note from Viscotek describes the company's four-capillary differential viscometers for high-temperature GPC–SEC separations. The publication discusses the four-capillary design and its benefits. The note includes chromatograms, a Mark–Houwink plot, and a schematic of the viscometer's fluid pathways. Viscotek Corp., Houston, TX, USA.

Circle 104

IC analysis of fermentation broths

Dionex's application note describes methods for determining organic and inorganic acids in bacterial fermentation broths using IC and suppressed conductivity detection. The publication discusses the use of the company's IonPac AS11 and IonPac AS11-HC columns for resolving organic acids and inorganic anions. The note includes chromatograms and lists analysis conditions. Dionex Corp., Sunnyvale, CA, USA.

Circle 105

Purity analysis of vinyl chloride

An application note from Unicam Chromatography describes the purity analysis of vinyl chloride monomer used for manufacturing polyvinyl chloride. The publication discusses the use of multidimensional capillary GC to measure the concentration of a range of hydrocarbon and chlorohydrocarbon impurities in polymer-grade vinyl chloride monomer. The note describes the use of sample vaporizer unit for repeatedly vaporizing vinyl chloride monomer and similar pressurized liquid samples. Unicam Chromatography, Cambridge, United Kingdom.

Circle 106

Absolute macromolecular characterization of lens protein

An application note from Wyatt Technology describes the use of multiangle light-scattering detectors for absolute macromolecular characterization of lens protein. The publication discusses the GPC–SEC separation of a lens protein sample and the determination of absolute molecular weights and sizes of the eluting fractions. The note includes figures plotting the molar mass versus volume and RMS radius versus molar mass. Wyatt Technology Corp., Santa Barbara, CA, USA.

Circle 107