The Evolution of Gas Chromatographic Instrumentation at PerkinElmer

Published on the occasion of the
50-year anniversary of
PerkinElmer's first gas chromatograph,
introduced in 1955.

PerkinElmer®
precisely.

achievements in gas chromatography

In 2005, PerkinElmer celebrates the 50th anniversary of the **Model 154 Vapor Fractometer**, the first gas chromatograph (GC) developed by The Perkin-Elmer Corporation. This instrument heralded the era of this versatile technique, which changed the way chemical analysis is carried out. It also represented the first truly automated, complex analytical instrument that did not need specially skilled scientists for its operation and could be used by practically every laboratory.

The Model 154 represented the first in a series of gas chromatographs, descendants of which are still manufactured by the Life & Analytical Sciences division of PerkinElmer. On the occasion of this anniversary, we would like to review the evolution of gas chromatographic instrumentation at PerkinElmer.

The saga of the Model 154

In 1953-54, Perkin-Elmer's representatives heard for the first time about the pioneering GC work done in England, by A.T. James and A.J.P. Martin, in the laboratories of the British Medical Council, London, and by C.S.G. Phillips at the University of Oxford. Visits to their laboratories helped in learning the principles of the new technique. Based on this information, an extensive development program was initiated in the company's headquarters, in Norwalk, Connecticut. The conclusion of this development work was the **Model 154 Vapor Fractometer**, introduced in May 1955. Its basic features, unique at that time, were: the use of an air thermostat (the "oven"), permitting to keep the temperature of the separation column adjustable between room temperature and 150 °C; a flash vaporizer, allowing syringe injection of liquid and gas samples through a rubber septum into the carrier gas stream; and a thermistor-type thermal-conductivity detector. Also, the company provided a number of standardized columns with a wide range of separation characteristics, permitting the use of the instrument for the successful analysis of diverse samples.

The instrument was an instant success. An editorial in *Analytical Chemistry* characterized it as "a splendid example of automatic analysis" and the chromatograms obtained "a delight to behold." Soon after the introduction of the instrument, Perkin-Elmer also published a simple 31-page booklet, explaining the principles of gas chromatography and how to select the operational parameters. *Analytical Chemistry* praised this text in a new editorial, calling it "a compact and very informative summary," helping "in disseminating scientific and technical information."

Naturally, development work did not stop with the introduction of the Model 154. By the beginning of 1956, an improved version was introduced; this was the **Model 154-B**. In this new version, the temperature range was extended to 225 °C, and an optional rotary-type valve, with variable sample loops, was added for the introduction of gas samples. It is noteworthy that all the rotary-type, multiport-sampling and switching valves in existence today, supplied by a large number of companies, can be traced back to the design of this valve.

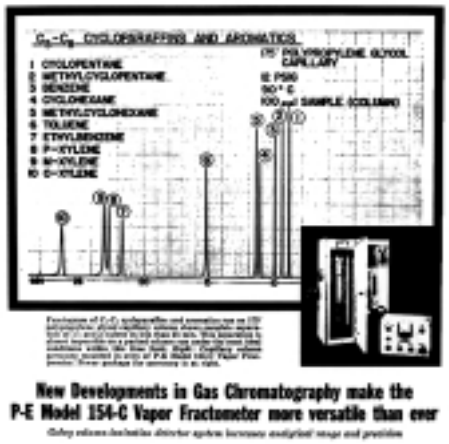
The **Model 154-B Gas Chromatograph**. The column oven is behind the door on the left-hand side of the unit. On the right-hand side, heating controls are on the top panel; pneumatic controls, as well as controls of the thermal-conductivity detector, are on the lower-right panel, while a flow meter is installed in the middle. The heated injection port for syringe injection is at the lower left. The potentiometric recorder was usually housed in a separate, equal-size cabinet. The Model 154-A had the same look as this instrument.

The year 1958 saw the invention of open-tubular (capillary) columns by Perkin-Elmer's Dr. M.J.E. Golay, one of the most respected scientists in the field of scientific instrumentation and, especially, gas chromatography. The next version of the instrument, the **Model 154-C**, introduced at the 1959 Pittsburgh Conference, provided the capability of using capillary columns and the newly developed flame-ionization detector.

In the Model 154-C, the flame-ionization detector amplifier was in a separate box. This was then consolidated into the **Model 154-D**, first shown at the 1960 Pittsburgh Conference. This instrument also provided a more sophisticated inlet system for capillary columns. Finally, in 1961, a simplified version of the original design, the **Model 154-L**, was introduced, specially developed for light gas analysis.

The Model 154 also served as the basis of other successful instruments at Perkin-Elmer's European subsidiaries in Germany (**Models 116** and **116E**) and England (**Models 451** and **452**).

The simplicity and ease of operation of the Model 154 series was a significant factor in the exponential growth of gas chromatography. Soon, every large analytical laboratory included GC among the techniques employed, and laboratories having a number of these instruments were not uncommon.



New Developments in Gas Chromatography make the P-E Model 154-C Vapor Fractometer more versatile than ever

The first advertisement of the **Model 154-C Gas Chromatograph** shows the instrument with the oven door open. The capillary column and the flame ionization detector are mounted in the oven. The amplifier of the FID is in a separate box. The chromatogram illustrates the full separation of ethylbenzene and the three xylene isomers.

Over five years after its introduction, the Model 154 and its improved versions were the most widely used gas chromatographs. From 1962 on, new and more sophisticated instruments were introduced, yet the Model 154 series and its European counterparts continued to be produced until the late 1960s. Many laboratories kept their old instruments in use for decades. The unparalleled history of this instrument is the best testimony of the foresight of its developers.

Dr. M.J.E. Golay (1902-1989), pioneering scientist in scientific instrumentation and the inventor of open-tubular (capillary) columns.

The Model 188

Very soon after the introduction of gas chromatography, chemists (mainly in the petroleum industry) wanted to analyze wide boiling-range samples. For this type of analysis, the operating range of a single, isothermal instrument was not enough. At that time, temperature programming of the column was not yet developed and therefore, another approach was suggested: to have multiple columns in series, each at a different temperature, with separate flow-through (thermal-conductivity) detectors at the end of each column. Such an instrument was Perkin-Elmer's **Model 188**, introduced in 1957, which, in essence, was three Model 154 oven-and-detector systems, combined in series. However, this instrument was short-lived. Within a few years, temperature programming of a single column proved to be a much simpler solution for such applications.

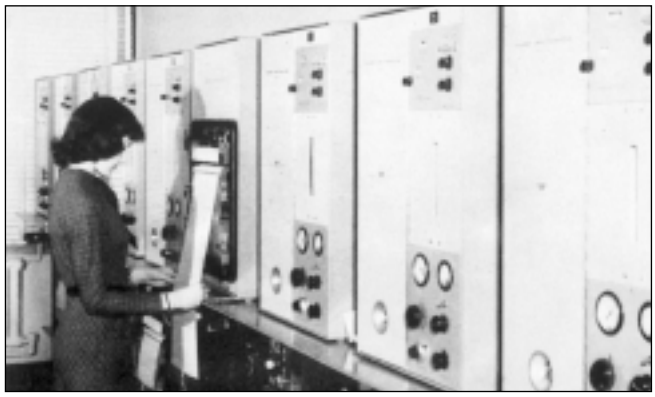
New instruments in the early 1960s

In 1959-61, various new innovations extended the range of gas chromatography. Perkin-Elmer pioneered the development of a number of new instruments, driving a higher level of results.

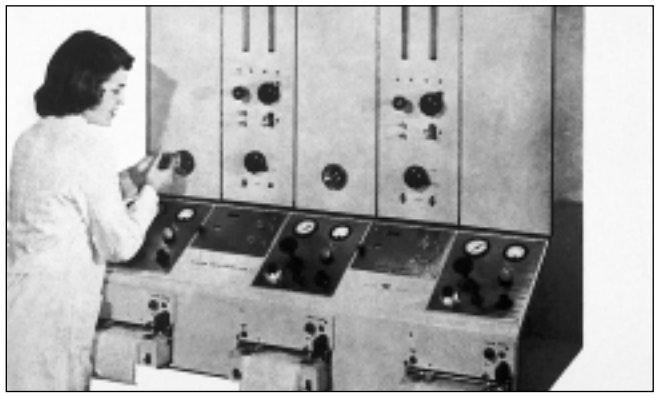
We have already mentioned that temperature programming started to be popular in this period. To adapt the Model 154 GCs (which only permitted isothermal analysis) for this need, a separate unit, the **Model 222**, was developed and introduced at the 1961 Pittsburgh Conference. This instrument, which housed the separation column and was connected to the Model 154 utilizing its detector, had two unique features:

The first was that the (packed) column was resistance heated, permitting 39 program rates from 0.5 to 52 °C/min. Recently, resistance heating was reintroduced and claimed as a new development. This is, however, not true – the technique was pioneered 43 years ago by Perkin-Elmer. One of the reasons why resistance heating was utilized in the Model 222, was a result of the poor design of early temperature-programmed ovens, where significant thermal lag was reported between set and actual column temperatures. This problem was eliminated by resistance heating. The column follows instantaneously with the set program.

The combination of the 222/154 system had a second feature which was important in those early stages of temperature programming, since relatively low-boiling



Routine gas chromatography laboratory at Esso R&D Co., in early 1958. This room contained at that time eight **Model 154 Gas Chromatographs**. Except for the unit attended by the operator, the potentiometric recorders were placed directly under each chromatograph. (From W.A. Dietz, *Instrument News*, Spring 1958 edition.)



The **Model 188 Triplestage Gas Chromatograph**. The sample was introduced through the injection port of the leftmost unit and was then conducted by the carrier gas through three columns in series, each in a separate oven at different temperatures. Each had a thermal conductivity detector at the column outlet, making separate records of the particular fraction analyzed in that column.

substances (truly “liquid” phases) were used as the stationary phase. With such columns, an excessive baseline drift was observed during programming, due to the exponential increase of vapor pressure with temperature, causing a similar “bleeding” from the column into the detector. In the 222/154 system, a short column coated with the same phase as the separation column was installed in the Model 154’s oven, in series with the temperature-programmed separation column. The temperature of this scrubber column was kept isothermally, at the maximum of the program. This way, the stationary phase concentration in the carrier gas, due to the bleeding, equilibrated and became constant toward the detector – thus, the baseline drift was eliminated.

The Model 222 was short lived for two reasons. The dual-column, baseline-compensation method, described in 1961 by E.M. Emery and W.E. Koerner of Monsanto Chemical Co. for the elimination of baseline drift during programming, proved to be simpler than the system used in the 222/154 combination. Perkin-Elmer immediately realized the importance of this technique and developed an instrument for its utilization. However, the company went one step further. While Emery and Koerner utilized a thermal-conductivity detector, with its standard two channels, Perkin-Elmer developed a differential flame-ionization detector for this use. The **Model 800**, introduced at the 1962 Pittsburgh Conference, was the first instrument permitting baseline compensation with an FID, also including capillary columns. This instrument also had a significantly improved oven design, assuring constant temperature along the column and a minimal thermal lag during programming.

The Model 800 could be used with both packed and capillary columns. A few months after its introduction, a modified version, the **Model 801** became available. This instrument was specially designed for the growing biochemical-clinical market, providing all-glass systems including glass on-column injectors and glass packed columns.

In the next few years, further versions of the Model 800 were available, with both the differential FID (the **Models 810** and **880**) and thermal-conductivity detectors (the **Model 820**). These instruments also had all-glass versions: the **Models 811** and **881**.

The **Model 213 Hydrocarbon Analyzer**. It was a portable unit, permitting the analysis of atmospheric samples for the total organics content, with help of a flame-ionization detector.

The **Model 154-D.** In this unit, the amplifier of the flame-ionization detector was incorporated in the upper part of the recorder cabinet (right). The rotary-type gas sampling valve, originally introduced with the Model 154-B, is mounted on the left side of the instrument cabinet.

Parallel to the Model 800, Perkin-Elmer's German affiliate introduced the **Model F-6** gas chromatograph. This was the first truly modular GC based on a building-block system, with multi-detector possibilities, permitting both isothermal and programmed-temperature operation. Two years later, in 1964, a dual-column version, the **Model F-7**, also became available.

Both the Models 800 and F-6/7 series were general-purpose instruments, although they permitted the use of both packed and capillary columns. The application of capillary columns, however, expanded in a number of fields; an instrument specially designed for capillary-column work became necessary. This instrument, the **Model 226**, was also introduced at the 1962 Pittsburgh Conference. It had a unique design, eliminating the need for a column oven: the thin-walled capillary columns were embedded in a flat metal disk and heated by direct contact with a similarly shaped flat heater. This system provided extremely accurate temperature control and an almost zero lag between set and actual temperatures in both isothermal and programmed-temperature operations.

In 1964, another highly successful gas chromatograph was introduced by Perkin-Elmer, the **Model F-11**, designed by the company's British affiliate. This compact, low-cost, modular instrument could be expanded from a simple single-column, single-detector, isothermal instrument to a temperature-programmed, multi-detector version. This instrument and its later version, the **Model F-33**, were sold worldwide for over a decade. Another low-cost GC, the **Model F-20**, was introduced in 1965 for the German market. This instrument and its improved version, the **Model F-22**, were also produced for over a decade.

Instruments of the second decade

In the first decade of commercial gas chromatography, almost every year saw the introduction of a new, improved model of the popular series of instruments. However, by the mid-1960s, the field started to settle down. The instrument-design principles were consolidated. Improvements then occurred in the areas of electronics and engineering components, such as pressure controls and flow regulators. These advances also permitted optimization of instrument design for

The Perkin-Elmer-Shell **Model 212 Sorptometer** was developed for the determination of the surface area of solids according to the BET method, using a GC-type measurement. The sample holder was placed in the Dewar flask on the right where adsorption took place at liquid-nitrogen temperature.

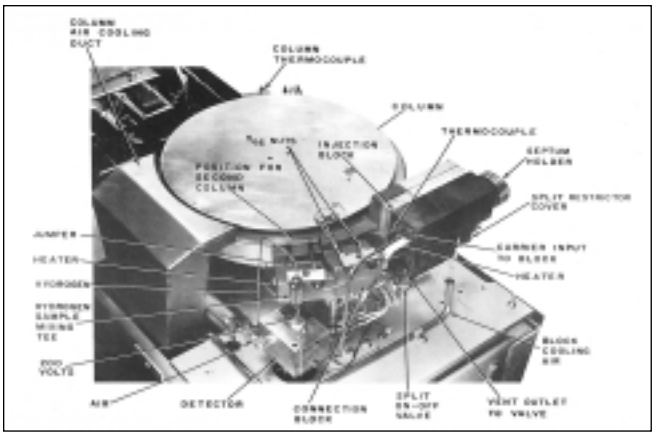
The **Model 222 Temperature Programming Accessory** for the Model 154 Gas Chromatographs. The resistance-heated column is behind the vertical "chimney." The injection port (for syringe injection) is just below the chimney.

both packed and capillary columns. A certain design could last for a number of years with only minor enhancements that did not change the overall features of the instrument. The Models F-11 and F-20 already represented such long-lasting designs, successfully marketed for over a decade. However, both were, by definition, relatively simple, low-cost instruments, aiming for the routine analysis market.

By the mid-1960s, it was clear that a new major, top-of-the-line gas chromatograph was needed, which would provide high sophistication and flexibility and could be used in both research and routine analysis. A special requirement, raised at that time by users, was the possibility of using two columns and multiple detectors. The **Model 900**, introduced at the 1967 Pittsburgh Conference, fulfilled this need. This instrument, which we consider one of the finest GCs ever built, set the standard for high-performance gas chromatography. With the enhanced **Model 990** introduced three years later, the 900 series was one of the dominating gas chromatographs of the decade. Even 38 years after its introduction, many can still be found operational in laboratories around the world.

The 900/990 series was phased out in 1974 by the **Model 3920**, one of the most successful instruments in the medium-price range. In addition to updated electronics, a new feature was the N/P detector, selective to nitrogen- and phosphorus-containing organic compounds. This detector became very important in drug screening, particularly at the Olympic Games. The debut of its application was at the 1976 Winter Games in Innsbruck, Austria, where Perkin-Elmer provided the instrumentation needed for routine testing of the athletes.

Meanwhile, Perkin-Elmer Ltd., the company's British affiliate, was also engaged in GC development, with the aim of replacing the Model F-11 with a more up-to-date design and, at the same time, designing a top-of-the-line instrument. The Model F-11 was eventually replaced in 1974 by the Model F-33, while in 1971 the **Model F-30**, a superb, high-performance gas chromatograph was introduced. This instrument was the basis of the development of the **Model F-17**, a highly versatile, medium-priced GC, introduced in 1973, that enjoyed considerable success in Europe.



Configuration of the capillary column and heater of the **Model 226 Gas Chromatograph**. The heater is the disk at the bottom, while the capillary column is embedded in the disk above it.

The **Model 810/820 Gas Chromatographs**. These represented advanced versions of the Model 800 Gas Chromatograph, originally introduced in 1962, offering, for the first time, dual-column baseline compensation in programmed-temperature operation, with differential flame-ionization detectors or with a dual thermal-conductivity detector. The Model 810 (on the left) is the FID and the Model 820 (on the right) is the TCD version.

Instruments of the electronic age

By the mid-1970s, electronic technology had advanced to such an extent that it permitted the development of an entirely new line of microprocessor-controlled instruments, replacing all existing GCs. This rapid advance has continued ever since. As a result, instrument lines were replaced periodically by newer designs, incorporating the latest achievements in computer technology.

In the last 30 years, PerkinElmer has developed four distinct GC instrument lines, always corresponding to the newest advances.

The Sigma series

The first new line of gas chromatographs was represented by the highly successful **Sigma series** of instruments, developed in 1975-77. During development, some of the new features had already been incorporated into the **Model 910**, an upgraded version of the Model 3920, introduced in 1976. The whole line of new instruments was introduced at the 1977 Pittsburgh Conference.

Four versions were offered with interchangeable components and accessories, from a simple, isothermal GC, the **Sigma 4**, to the very sophisticated, complex, automated instrument, the **Sigma 1**. Further improvements in 1980 led to the **Sigma B series**. The **Sigma 1B** was also enlarged to include full data-handling capabilities; the result was the **Sigma 115**, introduced in 1981. Based on the Sigma 2B, the **Sigma 2000**, an advanced, flexible, high-performance instrument of modular design, was developed. Introduced in 1982, this instrument remained highly popular for almost a decade. The moderate-cost Sigma 3B was also further improved in 1983, resulting in the **Sigma 300**.

The 8000 series

The next phase of GC innovation was the **8000 series**, incorporating real-time screen display of the chromatogram with built-in method development and data-handling capability. The instruments were developed at Perkin-Elmer's British affiliate. The first of this series was the **Model 8300**, a simple, single-channel gas

The **Model 900 Gas Chromatograph**. The column oven opens on the top left, with two FIDs in the wire cage to its right. Behind this are the controls for three gases, the carrier gas (helium), as well as hydrogen and air for the FIDs. The dual injection port is left of the name plate. The various knobs represent temperature controls and controls for the detector electronics.

The **Model 3920 Gas Chromatograph**. Its design represented an advanced version of the system originally incorporated in the Model 900.

chromatograph, introduced in 1983. This was then followed by three models with increasing capabilities: the **Models 8400** and **8500** (1986) and the **Model 8700** (1987). Unique features of these instruments were the sliding oven door, optional installation of additional injectors and detectors, and automated bleed compensation.

The AutoSystem instruments

In 1990, Perkin-Elmer introduced the **AutoSystem™ GC**, a high-performance instrument, incorporating the newest advances in chromatography and electronic control, with a fully integrated autosampler, capable of handling up to 83 samples and injections of different volumes. In October 1995, an advanced model, the **AutoSystem XL™ GC**, was introduced with an automated programmable pneumatic control for temperature-programmable split/splitless or on-column sample introduction, as well as large-volume injection and a number of universal and selective detectors. This instrument provided the highest-quality gas chromatographic instrumentation available at that time.

The Clarus 500 GC

Following a change in ownership, the company – renamed PerkinElmer, Inc. – introduced a new GC platform in the form of the **Clarus® 500 GC** and **Clarus 500 GC/MS** in July 2002. Both feature a revolutionary platform design, incorporating the easy-to-learn, touch-screen user interface. The Clarus 500 GC offers a whole new approach to the way users interact with the instrument – the intuitive graphical user interface features real-time signal display and eight-language support. The Clarus 500 GC retains the analytical performance of the AutoSystem GC, and the Clarus 500 MS incorporates all the high-performance technology of the TurboMass™ Gold mass spectrometer.

The microprocessor-controlled **Sigma 1 Gas Chromatograph** is shown with its data system on the right.

The **Sigma 2000** was an electronically controlled gas chromatograph with a built-in display of the method and the actual conditions. Shown in the middle are the four injection ports that permitted the installation of four columns simultaneously.

successes in GC mass spectrometry

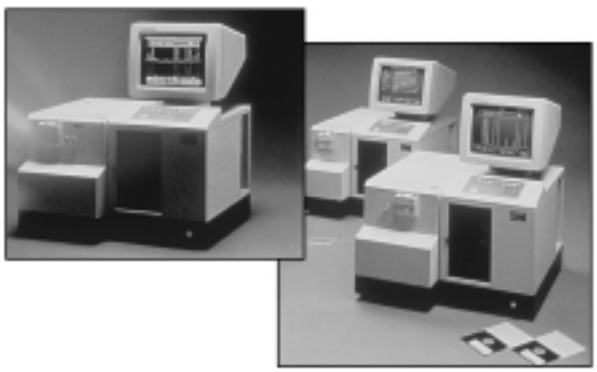
Mass spectrometry (MS) had always been considered the ultimate identification technique for organic compounds. However, early mass spectrometers were large, complex and expensive. Also, for some time, direct coupling of a GC with an MS was practically impossible, due to the large carrier gas flow and the small concentrations of the sample components to be investigated.

In 1963, Perkin-Elmer started to be involved in GC/MS, first by taking over the marketing of the mass spectrometers produced by Hitachi, Japan. These were stand-alone instruments that could be successfully coupled with Perkin-Elmer gas chromatographs, using the Watson-Biemann type separator, consisting of a porous quartz tube through which helium molecules diffused. The heavier organic molecules remained in the gas stream in the tube and were conducted into the MS. Then, in 1967, Perkin-Elmer introduced the **Model 270**, a double-focusing, magnetic-sector mass spectrometer developed at Perkin-Elmer, which was directly combined with a gas chromatograph. This instrument was discontinued in the first part of the 1970s.

The next major involvement of Perkin-Elmer in mass spectrometry was the introduction of the **Ion Trap Detector**™ in 1986. This was a benchtop unit, directly interfaced to the 8000 series gas chromatographs via an open split interface, permitting the identification of the sample components eluting from a capillary column. This was then replaced in 1990 by the **Q-Mass 910**™ **Quadrupole Mass Spectrometer**, another benchtop unit which was interfaced with the AutoSystem GC.

In the 1990s, Perkin-Elmer started the development of a mass spectrometric detector that could serve as an integral part of the gas chromatography system. The new instrument, the **TurboMass Mass Spectrometer**, was introduced in the fall of 1997 and could be directly coupled to the AutoSystem XL GC.

As a further improvement in mass spectrometric detection, PerkinElmer introduced the **TurboMass Gold**™ **Mass Spectrometer** in March 2001. By combining the technology with the Clarus 500 GC to form the Clarus 500 GC/MS, it represents the highest performance available today.



The **8000 series instruments** permitted realtime screen graphics, data handling and method development through the control of a single keyboard.

The **Q-Mass 910 Quadrupole Mass Spectrometer** coupled with the **AutoSystem GC**. The Q-Mass 910 offered the fastest pumpdown in the industry, providing the analytical laboratory high uptime and efficiency of operation.

accomplishments in GC sample handling

PerkinElmer is a pioneer in the development of two major sample handling systems, permitting headspace analysis and the use of automatic thermal desorption sampling.

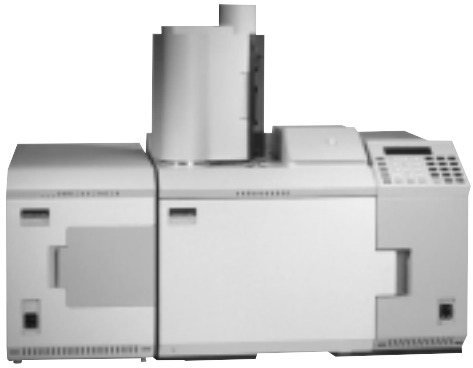
Headspace analysis

The analysis of volatile components present in an essentially non-volatile matrix is fairly difficult. Such a sample cannot be directly introduced into a gas chromatograph and, therefore, sample pretreatment (e.g., extraction) is necessary. This problem can be overcome by the direct sampling of the gas phase in equilibrium with the sample (static headspace). The technique of headspace-gas chromatography (HS-GC) was originally invented by Professor G. Machata of the University of Vienna in Austria. The proper instrumentation for the practical realization of the technique was developed by Perkin-Elmer's German affiliate, using a unique pressure-balanced, time-based sampling of the headspace of the thermostatted sample vials. The first instrument, the **Model F-40** was introduced in the fall of 1967; it was then followed by the **Model F-42** in 1975 and the **Model F-45** in 1978. The individual units mainly differed in the upper temperature of the thermostat which, in the Model F-45, was extended to 150 °C.

These and the successive instruments were capable of handling a large number of samples and of carrying out the sampling and analysis according to a preprogrammed schedule.

The previous three models were complete instruments, including not only the sample thermostat and the sampling unit, but also the gas chromatograph. The Model F-20 and F-22 GCs were used for this purpose. After the introduction of the Sigma series of gas chromatographs, the design concept was changed by having the headspace sampler as an attachment to the prevailing gas chromatographs. The first such unit was a small, manually operated system, the **Model HS-6** introduced in 1978, capable of handling six samples. Subsequently, a series of fully automated HS systems have been developed, capable of handling a large number of samples and combined with the PerkinElmer® gas chromatographs. These were the **Model HS-100** (1983) to the Sigma Series, the **HS-101** (1986) to the 8000 series, the **HS 40** (1991) to the AutoSystem and the **HS 40XL** (1996) to the AutoSystem XL gas chromatographs. In addition to the gas chromatograph for which they were optimized, these headspace samplers differed from each other by the increasing degree of sophistication of the programmer

The **HS 40XL Automatic Headspace Sampler** was the seventh generation of headspace samplers. On the left is the carousel where the samples are placed. The tower on the right side is where the vials are heated and the volatiles transferred to the gas chromatograph.



The **TurboMass Mass Spectrometer** is a quadrupole benchtop mass spectrometer with a standard mass range of 2-1200 daltons. It interfaces through the left side of the **AutoSystem XL Gas Chromatograph**, as shown in this photo.

controlling the automatic analytical sequence and the availability of additional techniques to further extend the range of HS-GC.

In March 2000, PerkinElmer introduced the **TurboMatrix™ series**, a new generation of headspace samplers. The intuitive touch-screen graphical user interface provides easy control of the sampler in eight languages. Building on more than 40 years of experience in GC sample handling and introduction, TurboMatrix Headspace Samplers incorporate proven pressure-balanced sampling for better performance. The TurboMatrix series includes three models:

- **TurboMatrix HS 16**, providing 16 positions
- **TurboMatrix HS 40**, providing 40 positions
- **TurboMatrix HS 110**, providing 110 positions

These instruments provide integrated, programmable pneumatics control, platform technology and fully automated operation. The three models have complete upgradeability, expanding with the user's needs.

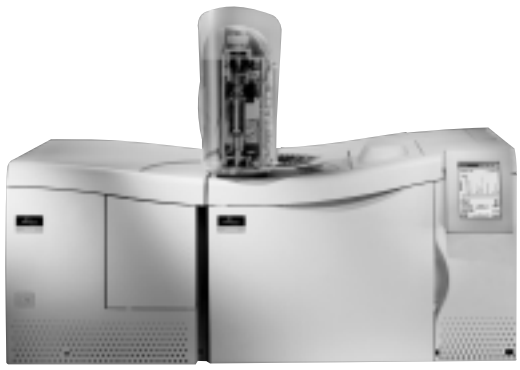
In 2003, PerkinElmer further improved the **TurboMatrix HS 40** and **HS 110** headspace samplers with patented **built-in-trap technology**, a key differentiator in headspace sampling. This system provides the ability for complete headspace extraction with built-in preconcentration, allowing up to 100 times lower detection limits than standard headspace samplers.

Automated thermal desorption sampling

The aim of the Automated Thermal Desorber (ATD) is to permit the automatic handling of sorbent-filled air-sampling tubes for the determination of the adsorbed volatile compounds (e.g., VOCs in air) in concentrations from ppt to percent values. However, it can also be used for the determination of volatiles present in a solid sample, which can be vaporized upon heating. These instruments were developed by the company's British affiliate. The first unit, the **Model ATD-50**, was introduced in 1981. It was followed in 1990 by the **Model ATD-400**. Each can automatically handle 50 sampling tubes. The difference is in the upper desorption temperature, which was 250 °C in the Model ATD-50 and 400 °C in the Model ATD-400.

In 2000, PerkinElmer introduced a new generation of instruments, the **TurboMatrix TD** thermal desorbers. These allow the choice of single-shot analysis of one sampling tube, or the use of an autosampler capable of handling 50 tubes and, similarly to the TurboMatrix headspace samplers, provide complete upgradeability to grow with user requirements.

The **TurboMatrix HS 110** (left) and **TurboMatrix Automated Thermal Desorber** (right). An intuitive, color touch-screen user interface provides instant access to system functionality. They can be interfaced to almost any GC system, giving access to PerkinElmer's proven technology, regardless of the GC brand or model.



The **Clarus 500 GC/MS** system includes a quadrupole MS with the mass range of 1-1200 daltons, providing very fast scan speed, up to 60 scans/second, and permits Selected Ion Full Ion (SIFI™) monitoring in the same run, simultaneously acquiring data in full scan together with Selected Ion Monitoring (SIM).

contributions to special applications

The principles of gas chromatography have also been applied to instruments aiming at some special applications. GC has also been combined either with special sampling techniques or with other major instrumental analytical techniques used for the identification of sample components separated in the gas chromatograph. PerkinElmer has been active in the development and marketing of a number of such dedicated systems. Below is a brief summary of the most important units.

Preparative GC

The potential of using gas chromatography to obtain sample components in pure form had been realized practically since the beginnings of GC. The primary aim was to have pure fractions available for identification, using ancillary analytical techniques such as infrared, ultraviolet, mass spectroscopy or chemical reactions and not to "produce" substances in pure form. Thus, when speaking about "preparative" GC, we essentially consider analytical gas chromatographs or instruments with somewhat increased throughput and fraction-collection capability.

Perkin-Elmer had already provided an accessory in 1956 to the Model 154-B Gas Chromatograph to collect eluted, pure sample components as they emerge from the instrument's outlet, and heated collection systems were also available for successive gas chromatographs.

In the 1960s, Perkin-Elmer also introduced two instruments specially developed for preparative GC. The first was the **Model 222P**, a scaled-up version of the Model 222, utilizing resistance heating of the column. The instrument, introduced at the 1963 Pittsburgh Conference, also included a thermal-conductivity detector and a sample-collection system with two heated exit ports to which metal or glass collection traps could be attached. Columns of 1-inch OD, up to 10 feet long, could be used in the system, even with temperature programming. The reason for selecting resistance heating, was that the rapid heat-up, or particularly, temperature programming of such a big mass represented by these columns in an air thermostat, would have been almost impossible. However, this instrument was not popular – it represented an about 16-fold scale-up of sample sizes which, as already mentioned, was more than most laboratories required.

The **TurboMatrix HS 110 Trap** provides built-in analyte-trapping capabilities that maximize the extraction and transfer of headspace vapor into the GC column, thereby lowering the detection limits by up to 100 times.

The PerkinElmer-Arnel **Model 4088 Beer Analyzer.** This system includes a PerkinElmer-Arnel Clarus GC with FID detector, coupled with a TurboMatrix HS Trap for off-flavor testing.

The second preparative gas chromatograph was developed by Perkin-Elmer's German affiliate. This was the **Model 21** introduced in 1966. This instrument had an advanced design, where higher throughput was achieved by automatic, repetitive sample introduction and fraction collection into a standard or somewhat larger (e.g., about 1/3 inch instead of 1/4 inch) analytical column, rather than by injecting a single, large sample volume into a significantly scaled-up column. A few years after the introduction of the Model 900, a special accessory permitting the same technique (automatic repetitive sample introduction and fraction collection) also became available for this instrument.

By the beginning of the 1970s, interest in "preparative" gas chromatography (i.e., fraction collection for subsequent analysis) diminished. It was slowly replaced by the direct connection of a gas chromatograph (the GC column) to another instrumental analytical technique (mainly mass spectrometry) for component identification.

Sorptometer

The Sorptometer was a special instrument, permitting the determination of the specific surface area of powdered or granulated solids according to the BET theory, utilizing a GC-like technique instead of a vacuum system for adsorption-desorption measurements. It was originally developed at Shell Development Co., in Emeryville, California, and Perkin-Elmer was licensed to design an instrument and further develop the technique. The original instrument, the **Model 212**, was introduced at the 1960 Pittsburgh Conference. In subsequent years, improved models such as the **Models 212-B**, **212-C** and **212-D** were introduced. The Sorptometer enjoyed considerable success as the only instrument of its kind. However, its field of application was very limited. Toward the end of the decade, its marketing was discontinued.

FID for total organics analysis

The introduction of the flame-ionization detector (FID) in gas chromatography – 40 years ago – coincided with the start of air-pollution research. At that time, the importance of determining the total organic content of the atmosphere or other gases (e.g., automobile exhaust) was identified by a few far-seeing researchers. It was also evident that the FID, which senses all organic compounds (and the response of which is essentially proportional to the total concentration of the organics present in the sample gas), could be used for this purpose.

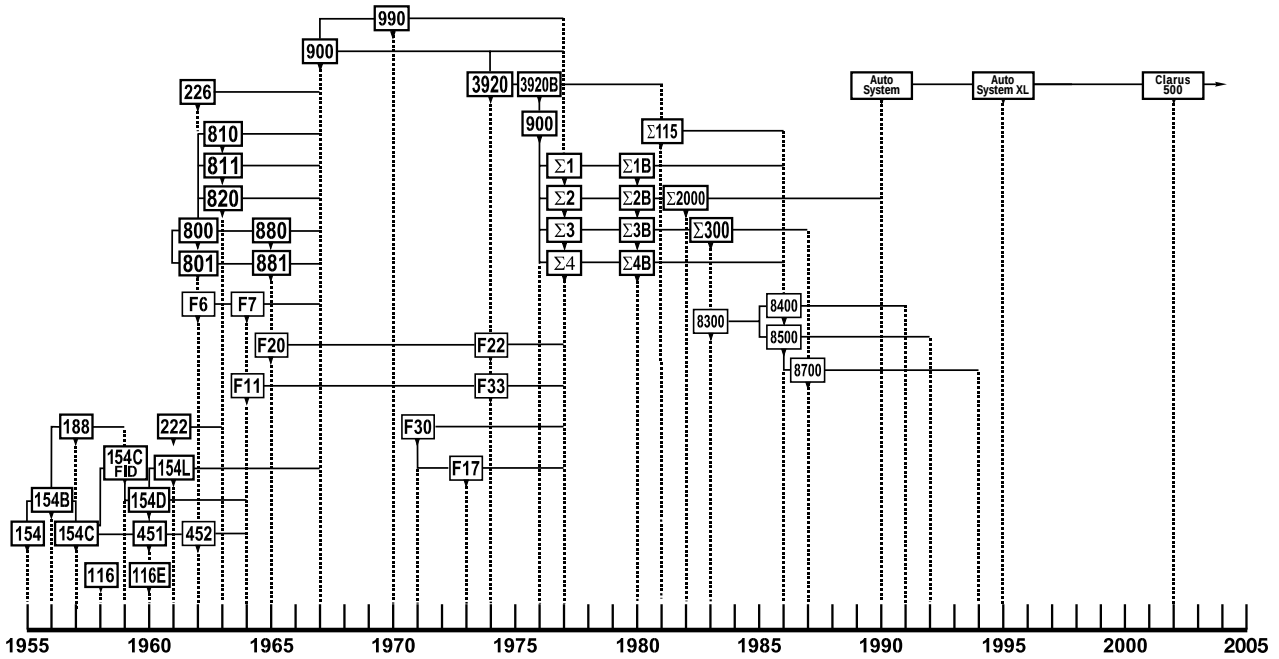
To facilitate such investigations, Perkin-Elmer developed a portable instrument, the **Model 213 Hydrocarbon Analyzer**, which was introduced in late 1959. In 1961, a benchtop version, the **Model 223**, also became available. Both instruments were based on the FID design of the Model 154-C, but no column was used and the sample gas (e.g., atmospheric air) was pumped through the detector at a constant flow rate in lieu of the carrier gas. The detector response, as compared to having a pure inert gas (nitrogen or helium) flow, was proportional to the total concentration of the organic compounds present in the sample gas.

Specialized gas chromatographs

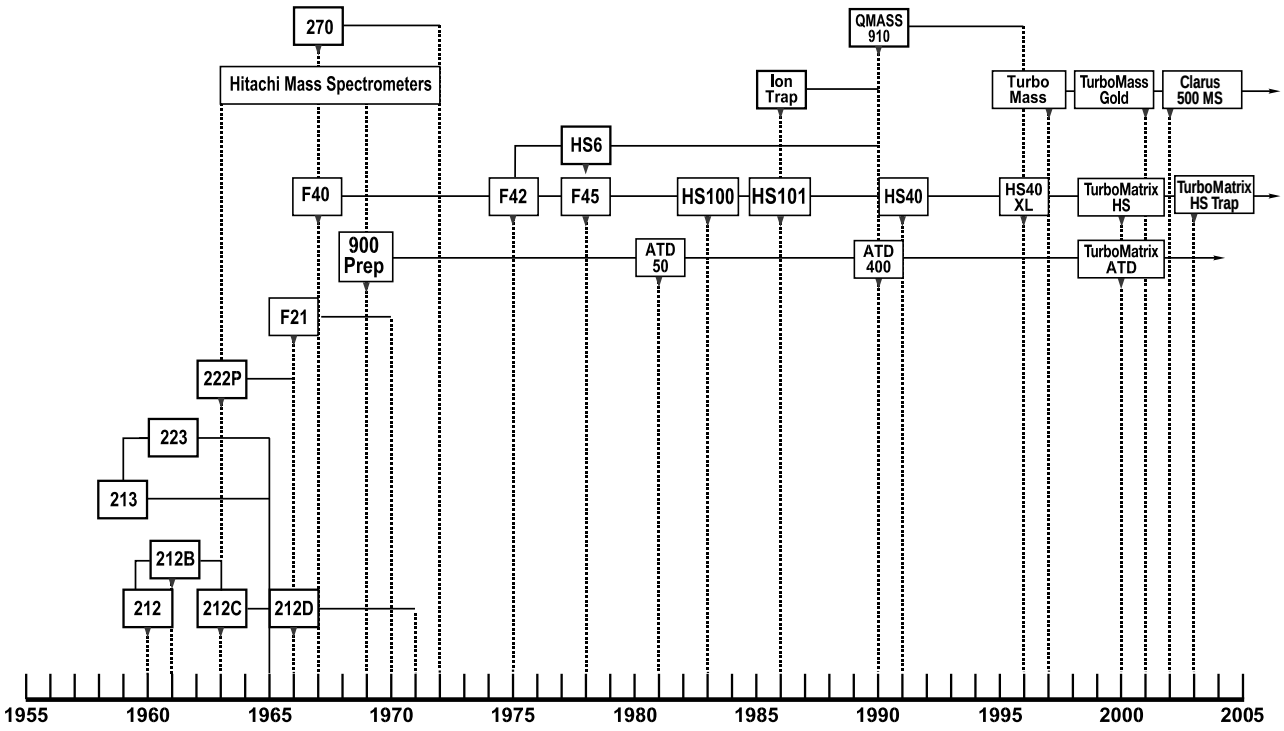
In some cases, a general-purpose instrument is so extensively used for one special application, that it justifies customizing it. PerkinElmer has a long-standing relationship with Arnel™, a market leader in customized chromatography solutions, to provide, install and support proven dedicated systems for a wide range of the applications and standard methods, including light-gas analysis through transformer-oil gas analysis, simulated distillation and detailed hydrocarbon analyses. PerkinElmer also provides customized solutions to industries downstream from petrochemical processes, for example, lubricants, polymers, fine chemicals and beverages.

One of the most recent additions to the PerkinElmer-Arnel customized GC offering is the **Model 4088 Beer Analyzer** (introduced in 2005), specialized for off-flavor testing.

PerkinElmer Gas Chromatographs 1955 - 2005



PerkinElmer GC Mass Spectrometers, Sampling Systems and Instruments for Special Applications 1955 - 2005



PerkinElmer, Inc.

The future

Evolution of scientific instruments never stops. Advances in gas chromatography, in the field of electronics, computer and microprocessor technology, as well as in production techniques, provide both the possibility and the need for continuous improvements in design and performance.

Fifty years have passed since the introduction of the first PerkinElmer gas chromatograph. During this period, many instruments have evolved, representing a continuous, unbroken line. Each instrument logically followed its predecessor, retaining its good features, while incorporating the newest advances. This continuity in instrument evolution and the accumulated knowledge of PerkinElmer's chemists, engineers and physicists is the best guarantee for excellence in scientific instrumentation in the 21st century.

About the author

Dr. Leslie S. Ettre, a graduate of the Technical University of Budapest, Hungary, has been active in the field of gas chromatography for almost 50 years, in both research and instrument development. His main field had been in the theory and practice of open-tubular (capillary) columns. Dr. Ettre had been associated with The Perkin-Elmer Corporation for over 40 years, retiring at the end of 1990, as a senior scientist. From 1988 until last year, he has been associated with the Department of Chemical Engineering of Yale University, first as an adjunct professor and, since 1995, as a research affiliate. In 1994, he was a guest professor at Johannes Kepler University, in Linz, Austria. Dr. Ettre has lectured throughout the world on chromatography and received numerous national and international awards. He is best known as the co-author of the basic textbook *Static Headspace Gas Chromatography* and the contributor of the series on Milestones in Chromatography published regularly in *LC•GC Magazine*.

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