The gas chromatographs scientists are familiar with today have a more-or-less standard design. The column is contained in a thermostat, and helium, hydrogen or nitrogen serves as the carrier gas. Samples are introduced instantaneously upstream of the column, and the column effluent is monitored continuously, with some characteristic change, such as its thermal conductivity, measured when a separated sample component exits the column. This change is usually recorded in the form of a peak, the area (height) of which is proportional to the amount of analyte present. Naturally, present-day instruments are more sophisticated than those used 40–50 years ago. They have more-precise, automated control of both pressures and temperatures; permit the programming of the column temperature during analysis; and include more-sensitive detectors. However, these basic operational principles have not changed since the introduction of the first commercial instruments 47 years ago.

Some early systems, however, did differ from this basic design in two main aspects: the manner of detection and, in conjunction with this, the choice of the carrier gas. One type of instrument was quite popular in the 1950s in laboratories in which the main analytical problem was the determination of inorganic gases and volatile C1–C4 hydrocarbons. They did not use a detector; instead, they collected the separated sample components and measured their volume. These instruments had very simple construction and could be built easily using standard laboratory hardware.

A principal advantage of these instruments was that users did not have to consider the problem of detector response factors, which, in a thermal-conductivity detector, can be quite different for the components of a sample with both inorganic and organic substances. Workers simply measured the volume of the collected fractions, which permitted sample concentration to be established directly in volume per cent. These systems were based upon the work of Jaroslav Janák of the former Czechoslovakia in 1951–1952. Because many of today’s readers are unaware of the principles of the Janák-type gas chromatographs, their 50-year anniversary is a fitting occasion to resurrect this work from oblivion. In addition, the circumstances that led to this development 50 years ago also provide a good illustration of the events happening in Central Europe during and after World War II.

A Technique Is Born
Our story starts in early 1939, a few months after the Munich agreement giving the Sudetenland — the German-speaking mountainous area on the western and northern part of Czechoslovakia — to Germany. At that time, Germany was already preparing for war, and an important part of this preparation was the building of factories that produced synthetic gasoline from brown coal. Large coal deposits were located in the northwestern part of the area annexed by Germany, and, thus, within a few months, the construction of a large plant started in the town of Most (its German name was Brüx). The plant used a modified Bergius process (named after Friedrich Bergius, who, together with Carl Bosch, received the 1931 Nobel Prize in chemistry for its invention), producing gasoline-type fuel through high-temperature and pressure hydrogenation of brown coal. Production started toward the end of 1942, and by the end of World War II the plant provided a significant part of the German armed forces’ needs. After the war, when the territory was again incorporated into Czechoslovakia, the profile of the plant shifted from gasoline production to brown coal carbonization and other chemical utilization of brown coal and coal tar, to the recovery of phenols from wastewater and the synthesis of some chemicals. At that time, the plant in Most was one of the most modern facilities in East Central Europe, and I remember that several of my colleagues in Hungary visited it in 1950–1952 to learn about the new techniques. (In fact, I also was scheduled for such a visit, but I changed jobs in the meantime.)

In 1947, Jaroslav Janák (born in 1924) was a recent graduate of the Technical University in Prague who had joined the Most plant and was put in charge of the microchemical and the gas and water analysis control laboratories. At that time, gas analysis was carried out using the classic Orsat method, which did not determine the individual volatile hydrocarbons. Therefore, a more advanced analytical method was needed.
The plant had an extensive library containing not only German journals but also the newest American publications; even though Germany was at war with the United States, these journals (and even some special chemicals) were obtained from the US through neutral Switzerland until the end of 1944. Janák felt that low-temperature fractional distillation would be the best method for his needs, and an order was placed for a unit to the Podbielniak Co. (Chicago, Illinois, USA), which had a virtual monopoly of such systems. However, due to the Communist takeover in Czechoslovakia in the spring of 1948, the US government imposed trade restrictions and denied export of this instrument. Therefore, other ways had to be found to perform the necessary analytical work.

Janák was very familiar with the liquid column and paper chromatographic analysis of mono- and diphenols and felt that the chromatographic principle could also be applied to gaseous samples. He studied the available literature, including the papers of Turner2–3 and Claesson,4 which described systems for gas-adsorption chromatography and illustrated the analysis of the type of samples he encountered. However, Janák had three objections: First, he felt that the elution technique (used in liquid chromatography) would be preferable to the displacement chromatography used by Turner and Claesson. Second, he considered their systems much too complicated for routine work. Third, he realized the shortcomings of the thermal-conductivity detector (used by both Turner and Claesson) for quantitative analysis in that substance-specific response factors had to be applied to establish actual sample concentration, and this process required proper calibration and an elaborate calculation of each result. One of the methods used in the microanalytical laboratory was the Dumas method for nitrogen determination in which a nitrometer was used to measure the volume of the product gas, and this method also seemed to be a good way to directly measure the volume of the separated fractions in gas chromatography (GC). Carbon dioxide was selected as the carrier gas that was absorbed by the potassium hydroxide solution used in the nitrometer. A particular advantage of using carbon dioxide was that laboratories did not have to rely on suppliers of pure gases because high-purity carbon dioxide could be produced directly by adding hydrochloric acid to crushed marble.

In 1951, while the development of the new method was under way, Janák was transferred to the newly organized Institute of Petroleum Research in Brno. There he studied the most recent publications of Zhukovetskii5 and Cremer6–8 on gas (adsorption) chromatography, and these studies helped him in his final work. (It is interesting to note that the seminal papers of Martin and Synge9, and James and Martin10 became known to Janák only years later. Obviously, the Biochemical Journal in which these papers were published was outside the field of the Most plant and the Brno institute and unavailable in their libraries.)

Janák concluded the development of the new technique in 1952. He first reported on it in September 1952 at the First Analytical Chemistry Conference, organized in Prague by the Czechoslovak Chemical Society. This lecture is mentioned specifically by Smolková-Keulemansová as the highlight of the meeting.11 At that time, Janák also applied for a patent on the system.12 Figure 1 is a photo of his early breadboard (shown with Janák’s collaborator, M. Rusek), and Figure 2 shows the detailed schematic of the system.13

As mentioned earlier, carbon dioxide was generated in the system by adding hydrochloric acid to crushed marble; the carbon dioxide formed in this process was purified by conducting it through two scrubbers and then drying it in a scrubber that contained calcium chloride. The carbon dioxide flow-rate was controlled by a mercury pressure regulator. At that time, proper gas-sampling valves did not exist; therefore, the sample volume was injected into the carbon dioxide stream through a calibrated microburette. The column was thermostated in a Dewar flask, and the effluent was conducted into the nitrometer, which contained concentrated potassium hydroxide solution that absorbed carbon dioxide. The volume of the collected analyte fractions could be read directly: one fraction ended when the bubbles temporarily ceased to rise in the nitrometer. However, analysts also could prepare a plot of collected gas volume versus time and read the heights of the individual plateaus. Figure 3 shows an example.

The Technique Matures

Soon after the development of the new technique, Janák started to publish detailed reports about it and its variations and applications. The first paper was published in 1953, and he authored or co-authored 18 papers on this subject during the next six years. All these papers were published first in Czech in Chemické Listy, the journal of the Czechoslovak Chemical Society. Their German (and in two cases, Russian) translations were subsequently published in the Collection of Czechoslovak Chemical Communications, an international scientific journal founded in 1929. The sidebar, “A Collection of Janák’s Publications,” lists the titles of these papers and indicates the wide variety of samples analysed.

In his original work, Janák used activated charcoal and silica gel as the column packings for separating inorganic gases and

![Figure 1: The breadboard of Janák’s GC system, with M. Rusek, his collaborator (1952).](image-url)
gaseous hydrocarbons. Later, zeolites (molecular sieves) and partition-type columns prepared with various liquid phases were also used.

Janák’s gas chromatograph almost immediately aroused a lot of interest in Europe, and a number of laboratories built their own systems for the routine analysis of gaseous samples. (Remember that commercial gas chromatographs were not yet available at that time or were just beginning to be introduced.) Companies that used self-built Janák-type gas chromatographs included BASF, Farbwerke Hoechst, and Lurgi in Germany; British Petroleum in the United Kingdom; Shell Laboratories in the Netherlands; and the Société Française du Pétrole in France. In fact, these instruments were so popular that chemists at Hoechst (Frankfurt, Germany) even created a new verb to describe analysis with this system, janákieren (“to Janák”). A few small instrument companies such as Hereus in Germany, Baird and Tatlock in the United Kingdom, and Kavalier in Czechoslovakia also developed commercial versions. However, because it was very simple to construct such a unit using standard laboratory hardware, most laboratories built their own. (At that time, laboratory chemists were accustomed to building their own systems.) The most advanced units were constructed by British Petroleum in the United Kingdom and Podbielniak Co. in the United States, which even built the prototype for a portable commercial unit named the Chromanette 9495 series (Figure 4). It was described at a meeting of the California Natural Gasoline Association in 1956; however, I have no information that it was actually sold commercially.

The self-built units deviated somewhat from Janák’s original design and improved it in certain aspects. In the system I built in 1957 at Lurgi (and used extensively for two years, along with two commercial gas chromatographs with thermal-conductivity detectors), the carbon dioxide was not generated from marble. A small autoclave was filled with solid carbon dioxide (dry ice), and this source provided the needed continuous gas flow. When filling the autoclave, some air was also trapped inside; however, this remained in a gaseous state and could be blown out easily by opening the autoclave briefly. This setup also was essentially the technique of Rouit, who, besides Janák, presented the most detailed description of this type of gas chromatograph and its operation. Another modification of my system was the way the gaseous samples were introduced. Janák used gas microburettes, which pressed the desired sample volume into the carrier-gas flow with mercury; in my system, I used a standard rotary-type gas sampling valve with calibrated sample loops, available at that time from the Perkin-Elmer Corp. Also, I used a standard rotameter for the measurement of the carrier gas flow that could be controlled by the exit valve of the autoclave.

As mentioned above, the main advantage of the Janák system was the possibility of directly reading the volumes of the separated fractions. Remember that at that time — even after the introduction of the first commercial gas chromatographs — peak area had to be established by measuring its height and width, or, for more accurate results, utilizing a hand-held planimeter to obtain the peak area. This process was tedious and time consuming. Next, scientists had to normalize the peak area with the help of the predetermined detector response factors to calculate sample concentration. Thus, the Janák system had many advantages for the routine analysis of gas samples. I remember that I had to analyze a fair number of such samples each day for quantitative composition. I developed the method in a standard, commercial gas chromatograph (a

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**Figure 2:** The basic Janák GC system. 1 = hydrochloric acid container; 2 = marble for the production of carbon dioxide; 3 = sodium bicarbonate scrubber; 4 = concentrated sulfuric acid scrubber; 5 = mercury pressure regulator; 6 = manometer; 7 = calcium chloride scrubber; 8 = three-way valve; 9 = calibrated volume tube for sample; 10 = mercury manometer; 11 = thermostat (Dewar flask); 12 = chromatographic column; 13 = nitrometer.
Perkin-Elmer model 154), with a thermal-conductivity detector and a potentiometric recorder providing the usual chromatogram; then, I transferred the method (and the column) to my Janák-type instrument on which I carried out the routine analyses and read the volumes of the separated fractions directly.

Despite its ease of operation, the Janák system also had a number of limitations. It was essentially restricted to samples that were gaseous at ambient temperature. Because the column was in a liquid thermostat (a Dewar flask), its temperature could be varied from subambient (needed for the separation of rare gases) to approximately 50–60 °C; however, the nitrometer and the connecting tubes were at ambient temperature, and their heating would have made the system much too complicated. Obviously carbon dioxide, if present in the sample, could not be detected because it was absorbed by the caustic solution. Similarly, sample components with an acidic character such as hydrogen sulfide were absorbed in the nitrometer. Finally, compounds that are partially soluble in potassium hydroxide solution gave incorrect results; a typical case was acetylene. These problems were discussed in detail during the First German Gas Chromatography Symposium held 14–16 November 1956 in Hamburg, where Janák served as the key-note speaker.

Towards the end of the 1950s Janák tried to automate the readout of the nitrometer (see number XV of his publication series), and the Podbielniak instrument also was supposed to provide some automation of the readout. However, these features made the system too complicated. Furthermore, by that time general-purpose laboratory gas chromatographs were being marketed by several companies, and these instruments were much more versatile than the Janák-type instruments. Thus, by the end of the 1950s, these units were being replaced gradually by the more modern, commercial, general-purpose instruments. Today, the Janák-type gas chromatograph exists only in our memories, but, nevertheless, it played an important role in the history of GC.

Epilogue
I’ll finish this discussion with a few words about the later activities of Jaroslav Janák. After five years at the Institute of Petroleum Research, he was appointed by the Czechoslovak Academy of Sciences to organize a separate Laboratory of Gas Analysis in 1956, with the goal of researching the applications of GC on a broader basis. This laboratory, also located in Brno, expanded in 1964 into the Institute of Instrumental Analytical Chemistry and in 1974 into the Institute of Analytical Chemistry. Janák served as the director of these institutions from 1964 to 1980 and then again from 1990 until he retired in 1993. He maintains regular contact with the institute and is also cooperating in the editorial work of the Journal of Chromatography. Janák has also been active in the construction of general-purpose gas chromatographs. Today, the Janák-type gas chromatograph exists only in our memories, but, nevertheless, it played an important role in the history of GC.
of gas chromatographs manufactured in Czechoslovakia and in teaching various chromatography courses. The photograph shown in Figure 5 was taken in 1960 during a course.

Acknowledgements
Jaroslav Janák twice described his recollections of his involvement in the evolution of GC.18,19 This article is partly based upon these publications. In addition, he was kind enough to explain to me some further details about his work and provide the photographs shown in Figures 1 and 5. Figure 4 was given to me by S. Tinsley Preston III, the son of S.T. Preston, who had been involved in the development of the Podbielniak instrument. Their cooperation and help is most appreciated.

References

Leslie Ettre is a research affiliate of the Chemical Engineering Department of Yale University and a member of the editorial advisory board of LC•GC Europe.

A collection of Janák’s publications
Jaroslav Janák’s articles about the chromatographic semimicroanalysis of gases were published in Chemické Listy and in the Collection of Czechoslovak Chemical Communications from 1953 to 1959. Parts I–III and IV–V were published as separate publications but in the same issue of Chemické Listy and then republished as combined papers with somewhat modified titles in the Collection of Czechoslovak Chemical Communications. From Part VI on, each paper was published individually in both Czech and in a foreign language. The language of the publications in the Collection of Czechoslovak Chemical Communications were in German, except the two that are noted as Russian with an English summary.


