Memorial Retrospective

Podbielniak, Inc., Gas Chromatography, and Seaton T. Preston

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Abstract

The early activities of Podbielniak, Inc. on low-temperature fractional distillation (LTFD) are outlined. The involvement of Seaton T. Preston, Jr. in the improvement of the LTFD apparatus and the development of the gas chromatographic instruments of the company is discussed in more detail. The possible reasons for the rapid decline of the company are investigated. Finally, Preston's role in launching a chromatography abstract service and the *Journal of Chromatographic Science* is emphasized.

Seaton T. Preston, Jr., the founder and long-term publisher of this journal, passed away on September 24, 1994. In his eulogy (1), John Q. Walker called him a "true gentleman's gentleman who had a complete vision of the explosion of chromatographic science in the early 1960's" and "one of the first U.S. chromatographers." Indeed, Seaton started in chromatography in the first part of the 1950's while associated with Podbielniak, Inc. and was primarily responsible for the development of the gas chromatographs (GCs) introduced by this company toward the end of 1955. Therefore, on the occasion of the 5-year anniversary of Seaton's death, we have felt that his memory could best be commemorated by summarizing the activities of Podbielniak, Inc. and indicating his role.

Podbielniak, Inc.

The evolution of commercial GCs developed and marketed by the presently existing major instrument companies has been discussed in a number of publications. However, very little is known about those companies that, after some early activities, ceased operation. Podbielniak, Inc. is one of those companies. Today, probably only a few chromatographers of significant age even remember the company's name, although for over 20 years, it had served as the supplier of a number of important laboratory and process equipment (among which GC was only one product line). Therefore, a brief explanation of the company's origin, activities, and the way it became involved in GC is needed.

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The company was founded by Walter Joseph Podbielniak. He was born on March 13, 1899, in Buffalo, NY, the son of Polish immigrants. He studied chemical engineering at the University of Michigan, receiving a Ph.D. in 1928. While still a student, he was recruited to join Phillips Petroleum Company's newly organized research group, which was investigating analytical methods for natural gas and gasoline. Within a couple of years, Dr. Podbielniak set out on his own, opened a testing laboratory in Tulsa, OK, in 1929, and started to develop fractional distillation equipment for such investigations. In 1934, he moved his operation to Chicago where he then formally established it as a company. In the 1930's, he also developed a number of other important process equipment, such as a centrifugal countercurrent solvent extractor that found application in the production of penicillin and other antibiotics. The importance of the latter can be seen in the fact that in the early 1940's, the full name of his company was the Podbielniak Centrifugal Super-Contractor Company, and only later was it simplified to Podbielniak, Inc.

It should be mentioned here that because his name was practically unpronounceable to Midwesterners, Dr. Podbielniak was mostly referred to as "Dr. Pod". This discussion shall also follow this usage and use the Podbielniak name only when speaking about the company.

Low-temperature fractional distillation

From the various equipment developed by Dr. Pod, we are interested here in his laboratory distillation apparatus. He developed systems for both low- (–180 to +100°C) and high-temperature (0 to +300°C) fractional distillation. Later models of the high-temperature unit were marketed under the name of Hyper-Ca13700 series, and such a system can be seen in Figure 1. However, these systems were not involved in the GC development at Podbielniak and, therefore, they are disregarded for our discussion.

The low-temperature fractionating distillation (LTFD) systems of Dr. Pod went through various stages of development. The first full description of a complete unit (he called it the Standard Precision Apparatus then) was included in a lecture presented before the Division of Petroleum Chemistry of the American Chemical Society (ACS) during the 1932 Fall

National Meeting in Denver, CO. This unit had both manual and automated versions, and according to the summary of the published paper (2), "the robot operator (of the equipment) controls reflux cooling, regulates distillation rate according to the difficulty of fractionation, summons the human operator with a buzzer and trouble light whenever necessary, takes all temperature and pressure readings, and turns out a complete printed fractional-distillation curve, including a distillation time rate curve."

The heart of the system was the specially constructed distillation column. By 1941, this was further improved, having sophisticated construction and a specially developed packing. According to a presentation by Dr. Pod at the 1941 Spring National Meeting of the ACS held in St. Louis, MO, these so-called Super-Cal columns containing the Heli-Grid packings (these were Podbielniak's trade names) could provide up to 100 theoretical plates (3). Present-day chromatographers would not be impressed by such an efficiency; however, this was a very high value for a laboratory distillation column.

The new, improved LTFD system that incorporated such columns was called the Hyd-Robot. In it, the temperature of the distillate (its dew point) and the pressure changes in the fraction collection vessel were automatically recorded. The former indicated the identity of the particular fraction (its boiling point) and the "break" between the separated sample components, and the latter gave information on the cumulative quantity of the distillate, similar to the integral recording used in some early GC detectors.

The Podbielniak LTFD system was developed for the natural gas, petroleum, petrochemical, and synthetic rubber industries for the determination of the C_1–C_5 hydrocarbons (particularly the C_4 fraction) in the cracking products, which at that time became a very important analytical problem. Dr. Pod was highly praised for this development, and in 1951, the Natural Gasoline Association of America (NGAA) presented him with its coveted Hanlon Award. His company had a virtual monopoly of the field, and soon the system and measurement became the standard method in the industry (4,5). However, the operation of the equipment required much skill, attention, and patience (6–8), and fairly complicated calculations (involving a lot of corrections) were necessary to finally establish the composition of the sample (9). Because of the complicated procedure and the possibility of a number of error sources, the accuracy and precision of the measurements were continuously checked by round-robin tests, the results of which were reported (10–12).

Apparently, in spite of the automation, standardization, and testing, there were still problems with the routine use of laboratory LTFD. A later publication (13) characterized the situation circa 1950 by saying that "it was gradually realized that the accuracies which can be obtained in a model laboratory…with research-grade analysts completely familiar with the theory and technique of the method, were not necessarily duplicated on an industry-wide basis." Therefore, in order to improve the skill of the users, the NGAA conducted a Low Temperature Analysts' Training School at the University of Oklahoma in Norman, OK between September 1952 and June 1953. The instructor of this school was Seaton Preston.

Seaton Tinsley Preston, Jr.

Seaton Tinsley Preston, Jr. was born on August 29, 1921, in Perry County, KY, and graduated in 1943 as a chemical engineer from Virginia Polytechnic Institute, in Blacksburg, VA. Joining the U.S. Army, he served in various units in the European theater, and as a first lieutenant at the end of the War, he commanded signal repairs for the Port of Antwerp in Belgium. After his discharge, he continued his education, finally receiving an M.S. degree in chemical engineering from the University of Michigan in 1948. After graduation, Preston joined Standard Oil of California. Then, in 1951, he became associated with the University of Texas in Austin, and the following year with the University of Oklahoma in Norman, where (as already mentioned) he was in charge of the LTFD Training School. However, Preston not only taught the intricacies of the measurements but also proposed significant improvements to the Podbielniak system: for example, incorporating a thermal-conductivity detector (TCD) instead of dew point measurement for the identification of the distillate components. The NGAA school, and particularly his proposals for improvements, brought Preston in contact with Dr. Pod, and in August 1953, he joined Podbielniak, Inc. in Chicago.

Figure 1. A publicity photo showing four Podbielniak instruments: (from left to right) the Thermocon 8700 series LTFD apparatus, a Chromacon 9400 series single-column GC, the Hyper-Cal 3700 series high-temperature fractional distillation apparatus, and a Chromacon 9400-V series multicolumn GC (21).

In the next year, intensive development work was carried out at the company to redesign the existing LTFD apparatus with the aim of incorporating a TCD and automatic controls into the system, making it

more accurate and less dependent on the skill of the analyst. Study of the contemporary literature shows that the redesign of the system resulted in a completely new instrument, the so-called Thermocon 8700 series, introduced in late 1954 or early 1955 (13,14). In order to further improve the skill of the users, the company also planned to set up the so-called Podbielniak Institute; its opening was originally scheduled for the Fall of 1955, but it was delayed and became operational one year later. This Institute shall be briefly dealt with later in this paper.

In spite of the improvements, the Thermocon was too late to help in widening the use of LTFD. It is ironic that the paper describing this new instrument (14) was published practically simultaneously with the introduction of the first two American commercial GCs, the Burrell Kromo-Tog and Perkin-Elmer's model 154 Vapor Fractometer. GC made the LTFD method for the analysis of low-boiling hydrocarbon mixtures obsolete. In fact, Perkin-Elmer's first advertising in the September 1955 issue of *Analytical Chemistry* (15) directly hit the LTFD market, illustrating the analysis of C_1–C_5 hydrocarbons, including the separation of the five C_4 paraffin and olefin isomers (the main market of the LTFD instrument) in 23 min, also indicating the ease of quantitation.

GC at Podbielniak

It is interesting to follow Podbielniak's contact with GC in the first part of the 1950's. Dr. Pod heard about gas–liquid partition chromatography as early as in 1952 when visiting England (and also the laboratory of Martin and James) (16). Shortly after, Dr. S.F. Birch of British Petroleum Co. also visited Dr. Pod in Chicago; they already had contact for years due to the LTFD instruments that British Petroleum naturally had intensively used. During his visit, Dr. Birch mentioned the investigations that D.H. Desty was carrying out at British Petroleum on the adaptation of GC for the analysis of light hydrocarbon fractions and emphasized that this new method might seriously affect the LTFD market. However, Dr. Pod did not take this warning seriously. On the other hand, Seaton Preston kept himself up-to-date in the evolution of GC, and with his associate Peter J. Turkel, he actually carried out (albeit unofficially) some investigations and development work. So, when in the Spring of 1955 they were suddenly faced with the introduction of commercial GCs by other companies, they could initiate a crash program to develop their own instrument. The result, the Chromacon model 9400A (a large, floor standing unit) was introduced in December 1955. Figure 2 shows Preston with one of the Chromacon instruments.

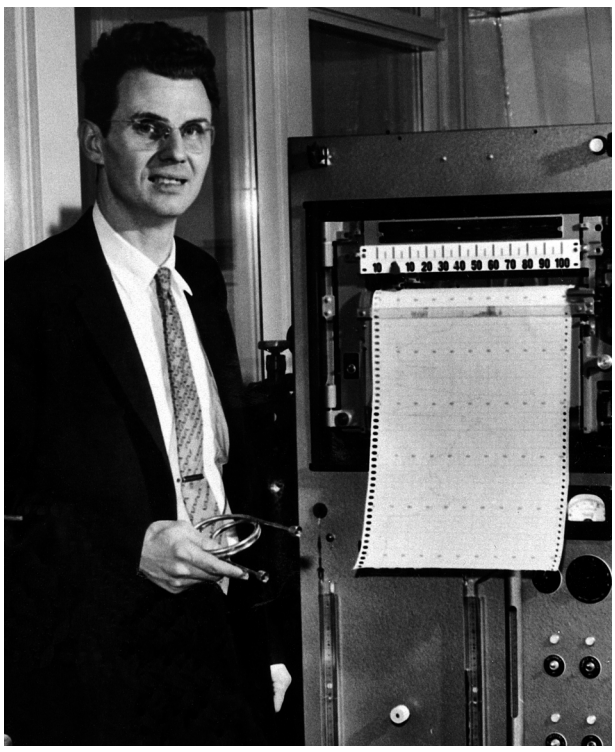


Figure 2. Seaton T. Preston, Jr. with one of the Chromacon GCs of Podbielniak, circa 1956–1958.

Simultaneously with the introduction of this instrument, Preston (co-authoring with Dr. Pod) also published a basic paper (17) that, with a follow-up paper published a few months later (18), has served as a very good introduction to GC and its possible applications. For some time these two papers, together with the brochure published in September 1955 by Perkin-Elmer's Harry H. Hausdorff (19), have served as the basic literature on GC. (I remember well that I have also used them extensively.)

The Chromacon series

Soon, Podbielniak expanded its GC instrument line, and by the end of 1956, the Chromacon series consisted of 12 laboratory instruments (20), corresponding to four basic versions: the 9400-A, 9400-V, 9475-A, and 9475-V series. These essentially differed in the upper temperature, which was 150–175°C for the 9400 series and 300°C for the 9475 series. In addition, the instruments indicated by the suffix V could also be operated under reduced pressure, and in each series, instruments with one, two, and three columns (A, 2A, and 3A and V, 2V, and 3V) existed. Podbielniak also offered two process GCs, the Chromacon 9485-1 and 9485-5 for the automated analysis of 1–5 streams. Figure 3 shows the two process GCs (21).

The Janák instrument

In the Fall of 1956, Podbielniak also introduced a unique instrument, the Chromanette 9495 series (21) (Figure 4). It was a small, portable GC weighing only 15 lbs and having dimensions of 24 × 26 × 5.5 inches (61 × 66 × 14 cm). It was based on the principles developed by J. Janák in Czechoslovakia. Because this development is practically unknown today, and because of its interesting connection with the political events toward the end of the 1940's, a brief summary is given here.

Jaroslav Janák (born 1924) is a Czech scientist who started his professional career in 1947 at the West Bohemian Chemical Works in Most, Czechoslovakia, and in 1956 became the head of the Laboratory for Gas Analysis in Brno, which he later expanded into the Institute of Instrumental Analytical Chemistry of the Czechoslovak Academy of Sciences. (The Institute still exists as the Institute of Analytical Chemistry of the Czech Academy of Sciences, and Professor Janák is Director Emeritus of the Institute.) In Most, Dr. Janák had been involved in investigations of

low-boiling hydrocarbons, and because LTFD seemed to be the best method for his purpose, he ordered the Podbielniak LTFD apparatus in 1948. Meanwhile, however, the political situation in Czechoslovakia (and in the other eastern European countries) changed: the Cold War started. Due to these events, the U.S. government imposed an embargo on exports to Communist block countries; thus, the ordered instrument could not be delivered anymore. Therefore, another way had to be found to carry out Dr. Janák's planned investigations. As a conclusion, he eventually developed (independently of James and Martin) a GC technique using CO_2 as the carrier gas and a nitrometer as the detector. The CO_2 content of the column effluent was absorbed in the sodium hydroxide solution of the nitrometer, and the eluted fractions were collected in it; in this way, the volume of the individual sample components could be directly established (22). Janák's work had been published in a series of publications starting in 1953 in a Czechoslovak chemical journal (23–26) and also in German translation (27–30), and his method had a considerable influence in Europe over the acceptance of GC as an industrial analytical tool. In fact, for a brief period, it was the tentative method for the analysis of natural gas issued by the British Institute of Petroleum (31). (When I started to work in gas chromatography in Germany in 1957, I also built a Janák apparatus and used it extensively beside two commercial GCs for the analysis of inorganic gases and light hydrocarbons.)

It is extremely interesting that Podbielniak developed this GC and even more interesting that they made it a portable instrument. Apparently, they also automated the recording of the volume increase in the nitrometer, resulting in an integral chromatogram which they called the "graphette gas analysis curve" (Figure 5). This was definitely an original development carried out at Podbielniak, because their description of the system (21) was presented months before Janák's publications on the automatic reading of the collected fractions' volumes (32,33). All of these examples show how far-sighted Preston was. According to the report presented in 1956 at the meeting of the California Natural Gasoline Association (CNGA) (21), the Podbielniak Institute had donated Chromanette instruments to "each of the natural gasoline associations". (I know of two such organizations: the NGAA and the CNGA. Apparently, it was a fine distinction that the instruments were donated by the not-for-profit institute and not by the company.) However, I could not find any report on its actual use by American industrial laboratories.

Preparative GC

Podbielniak not only developed and marketed laboratory and process GCs, they also investigated the possibility of using GC for preparative purposes and announced the availability of the Chromaneer "chromatographic plants". These used columns of 2–4 inches (50–100 mm) in diameter and lengths up to 20 feet (610 cm) in the temperature range of 20–200°C with liquid sample sizes up to 25 mL. A Podbielniak advertisement (20) mentions the production of $SiCl_4$, flavoring oils and perfumes, and fine chemicals as possible application fields. (I have no knowledge of whether any of such "plant" was actually installed in the U.S. However, I remember that Seaton told me many years ago that at least one was supplied to an Italian company.) Figure 6 shows a picture of the Chromaneer GC plant from Podbielniak's advertisements (20).

Figure 3. The Chromacon 9485 series process GCs. Left, model 9485-5, instrument for sampling five process streams; right, model 9485-1, instrument for sampling a single process stream (21).

Figure 4. The Chromanette 9495 series portable GC of Podbielniak, based on the principle of Janák (21). On the right there is a lecture bottle of CO_2 used as the carrier gas. The chromatographic column is in the middle in a Dewar flask for thermostatting. The nitrometer collecting the separated fractions is on the left.

The Podbielniak Institute

Starting in the Fall of 1956, Podbielniak offered customers training at the Podbielniak Institute set up in Chicago at 632 North Dearborn Street in the former building of the Illinois Institute of Design as a self-sustaining, essentially nonprofit institution. Apparently, the original intention of this institute was to continue the training offered in 1952–53 by the NGAA; however, when the institute was finally opened after some delay, the emphasis had already become GC. Aside from courses on LTFD, extensive training was also offered in GC, covering instrument operation, the selection of optimum conditions, and selected application problems. The institute also prepared and distributed a number of notes (written mostly by Preston) dealing with various aspects of theory and practice; a few titles (34) included *Notes on the Van Deemter Equation*, *Summary of the Correction of Retention Volumes*, *Guide to Column Selection*, and *Notes on the Calculation of Column Efficiency in GC*. (I found one announcement of the courses offered for 1958–59 (34). According to this, between September 1958 and the end of May 1959, four 2-week courses were offered in GC, and two 1-week courses were offered in distillation.)

Decline

In spite of their knowledge and wide range of GCs, Podbielniak could never gain a significant niche in the GC market. It is very difficult to evaluate the reasons for this now (40 years later) when all the participants are gone and one can only rely on personal assumptions. There are three reasons which may explain the rapid decline of Podbielniak by the end of the 1950's.

First, I believe that they over-extended themselves, trying to have just too many different instrument types. I already mentioned that by the end of 1956 they had 13 laboratory and 2 process instruments and also offered preparative "GC plants". In contrast, at the end of 1958 (when I joined the company), Perkin-Elmer had two laboratory instruments, the model 154-C Vapor Fractometer with the model 188 triple-stage GC (which, however, was soon discontinued) and the model 184 process GC. To manufacture and market 15 instruments, even if they differ relatively little from each other, is a major task; a large staff would be needed for it (something Podbielniak never had). To this, one must also add the very complicated situation with large-scale GC where each unit must be custom designed.

Second, for reasons I never fully understood, while offering a very broad range of instruments, Podbielniak's marketing was restricted to the petroleum and petrochemical industries and advertisements in a few periodicals specializing in this field. It is true that in the first years of the GC's evolution, petroleum and petrochemical laboratories represented the most important field of application; however, the analysts working in these laboratories regularly participated in the general analytical meetings and exhibitions and were mostly making their instrument acquisition decisions based on what they saw there. For example, Podbielniak did not exhibit at the Pittsburgh Conferences of 1956–59 (the four years I checked), and in the same period I could find only two advertisements in *Analytical Chemistry* (20,34). At the same time, all the other companies offering GCs had strong participation at the Pittsburgh Conferences and other general chemical meetings and frequent advertisement in *Analytical Chemistry*. It is even more surprising that Podbielniak's people did not participate at the first American international GC symposium organized by the Instrument Society of America (held on the campus of Michigan State University, East Lansing, MI, August 28–30, 1957), where representatives of all the other instrument companies making GCs were present and a number of new instruments, accessories, and applications were described (e.g., process GCs used in the petroleum industry) (35).

The third reason for the company's reduced role in the GC market was that even after it became obvious that GC made

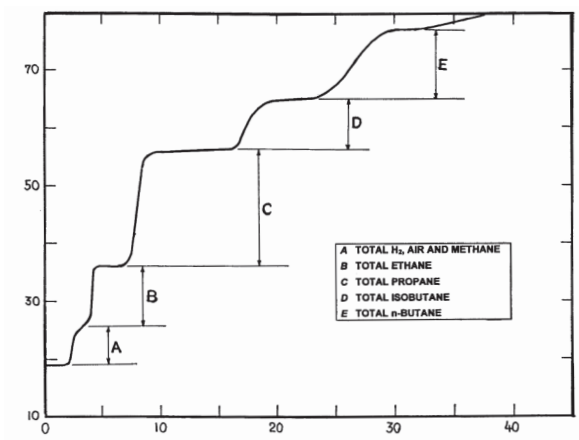


Figure 5. The integral recording of the Chromanette 9485 series Janák-type portable GC (21).

Figure 6. The Podbielniak Chromaneer "chromatographic plant" (20).

LTFD an obsolete technique, Dr. Pod still believed that GC was only a supplemental method to fractional distillation. In this respect, it is indicative to quote from his first paper on "vapor-phase chromatography" (17). At the end of the paper, when discussing the correlation of GC with other analytical methods, the following statement was made: "In relation to analytical distillation the latter has always required supplementary analyses on fractions inseparable by distillation... Here, vapor-phase chromatography fills the bill uniquely, perhaps better than any other supplementary method. Also, for the simpler fractions, and for determination of specific components in complex mixtures, etc., vapor-phase chromatography is beginning to compete with analytical distillation... However, analytical distillation is still the only method for handling sizable samples... In low-temperature analytical distillation of natural and cracked refinery gases, the natural combination seems to be that of the highly developed low-temperature fractional distillation apparatus of proven accuracy…with the vapor-phase chromatographic apparatus to simultaneously and expeditiously resolve the cracked C-4 and perhaps other ordinarily difficult and time-consuming fractions."

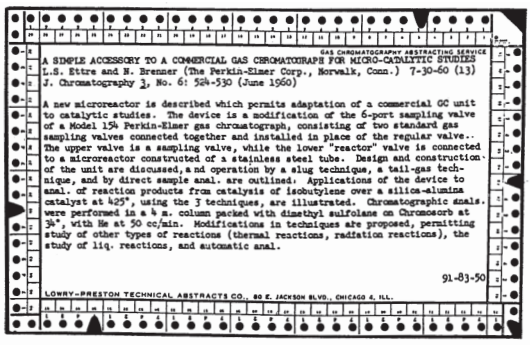
This was almost certainly written by Dr. Pod himself. In this respect, it is also indicative to quote Dr. Janák's personal recollections (31). He mentioned that when he personally met Dr. Pod in 1958, Pod was still optimistic concerning the future of low-temperature distillation analysis of gases and brushed aside Janák's warning. As Janák added, this happened at the time when "Podbielniak's world-famous company was actually rapidly losing ground".

It is also interesting to note in the previous quotation the emphasis on the advantages of using large sample sizes with analytical distillation. For some people, it was difficult to realize that the small sample sizes needed in chromatography could actually give more information than the large samples needed with classical methods; this controversy could also be seen during the evolution of chromatography, starting with Tswett's time.

Dr. Pod's belief that GC was only a supplemental method to fractional distillation is best illustrated in the publicity photo included in the reprint of the 1956 presentation at the meeting of the CNGA (21) shown in Figure 1. In this picture, the two Podbielniak fractional distillation apparatuses are shown side-by-side with two Chromacon GCs, implying that they are used together.

To these three possible reasons, one may add a personal issue: at that time, Dr. Pod had a major family controversy, and this certainly averted his attention for a couple of years.

I do not know the exact time when Podbielniak discontinued its GC product line, but it was around 1959–60. The Podbielniak Institute also ceased operation in 1959. In 1961–62, the company was not listed anymore among the chromatography suppliers (36). Podbielniak, Inc. continued to exist until 1967 when it was sold to Dresser Industries. For a few years, Dr. Pod acted as a consultant to Dresser Industries, but then, in the early 1970's, he moved to Rancho Santa Fe (San Diego, CA) where he died on December 13, 1978.



GAS CHROMATOGRAPHY ABSTRACTING SERVICE

A SIMPLE ACCESSORY TO A COMMERCIAL GAS CHROMATOGRAPH FOR MICRO-CATALYTIC STUDIES
L.S. Ettre and N. Brenner (The Perkin-Elmer Corp., Norwalk, Conn.) 7-30-60 (13)
J. Chromatography 3, No. 6: 524-530 (June 1960)

A new microreactor is described which permits adaptation of a commercial GC unit to catalytic studies. The device is a modification of the 6-port sampling valve of a Model 154 Perkin-Elmer gas chromatograph, consisting of two standard gas sampling valves connected together and installed in place of the regular valve.. The upper valve is a sampling valve, while the lower "reactor" valve is connected to a microreactor constructed of a stainless steel tube. Design and construction of the unit are discussed, and operation by a slug technique, a tail-gas technique, and by direct sample anal. are outlined. Applications of the device to anal. of reaction products from catalysis of isobutylene over a silica-alumina catalyst at 425°, using the 3 techniques, are illustrated. Chromatographic anals. were performed in a ¼ in. column packed with dimethyl sulfolane on Chromosorb at 3½°, with He at 50 cc/min. Modifications in techniques are proposed, permitting study of other types of reactions (thermal reactions, radiation reactions), the study of liq. reactions, and automatic anal.

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Figure 7. Example of the chromatography abstract cards supplied by the Lowry–Preston Technical Abstracts Co. (later Preston Technical Abstracts Co.).

Seaton Preston, the Publisher

Preston left Podbielniak in 1959 amid the controversies, becoming associated with the Sunbeam Corporation as Director in the Advanced Research Department. Meanwhile, however, he had already started (while still employed by Podbielniak) in 1957 the Gas Chromatography Abstracting Service, initially together with Doak Lowry of Universal Oil Products Co. In a few years, he took it completely over. This service regularly abstracted over 400 of the most important journals throughout the world; the abstracts were printed on Unisort punch cards, permitting their coding according to subject, author, etc. Figure 7 shows such an abstract card. By 1967, over 10,000 abstract cards had been issued. This service was extremely useful, particularly in the time of the exponential growth of GC when rapid information on new results was of vital importance.

By the early 1960's, chromatographers had realized a serious

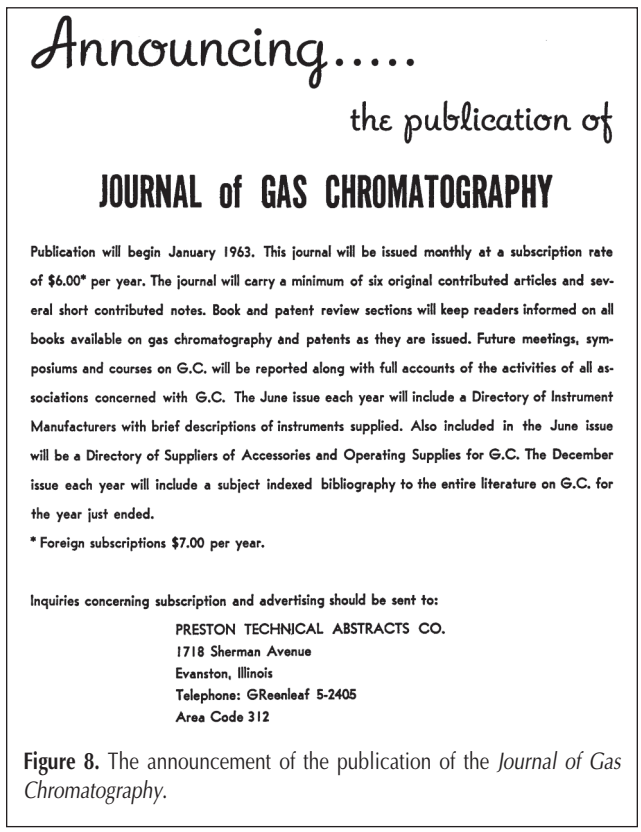

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Figure 8. The announcement of the publication of the *Journal of Gas Chromatography*.

problem that restricted the rapid dissemination of information. Papers could be published in *Analytical Chemistry* or the *Journal of Chromatography*; however, the former was a general-purpose scholarly journal and required full-rounded research-type publications, whereas the latter was edited in Europe and at the time was considered in the U.S. as mainly a theoretical publication with limited circulation. There was definitely a need for a journal that could provide a forum for the rapid publication of short, practical information: not only papers describing new results, but also reports on meetings and brief notes on some interesting happenings.

Seaton decided to fill this gap and launch a new journal, the *Journal of Gas Chromatography*. The idea was crystallized by the middle of 1962. I remember that he first discussed his plans with me at the 142nd National Meeting of the American Chemical Society held in Atlantic City, NJ, September 9–14, 1962 (37). Around that time, Seaton left Sunbeam to devote his full time to publishing, and the first issue of the new journal was published in January 1963. Figure 8 shows the original announcement. In 1969, the journal's scope was enlarged to encompass not only gas but also liquid chromatography, and its title was changed to the *Journal of Chromatographic Science*. This journal still flourishes and serves as a lasting memorial to Seaton T. Preston, Jr., one of the true pioneers of American chromatography.

Acknowledgments

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