Available online at www.sciencedirect.com
SCIENCE DIRECT®
Journal of Chromatography A, 1060 (2004) 1–7
JOURNAL OF CHROMATOGRAPHY A
www.elsevier.com/locate/chroma

Review

Scientific achievements of Jack Kirkland to the development of HPLC and in particular to HPLC silica packings—a personal perspective

Klaus K. Unger*

Institut fuer Anorganische Chemie und Analytische Chemie, Johannes Gutenberg-Universität, Duesbergweg 10–14, 55099 Mainz, Germany

Available online 13 September 2004

Abstract

Joseph (Jack) Kirkland is one of the outstanding protagonists of modern column liquid chromatography (HPLC). He started in 1953 as an analytical chemist at the Experimental Station of Du Pont de Nemours & Co, Wilmington, Delaware, analyzing pesticides by gas chromatography (GC). He early recognized the potential of HPLC as a powerful separation technique at the end of 1960. His major contributions to HPLC were in the field of silica based packings and stationary phases. At the beginning of the 1970's he manufactured Porous Layer Beads and later microparticulate porous silicas based at the Zorbax technology. He made outstanding contributions in the field of instrument design for HPLC and in the field of Sedimentation Field Flow Fractionation (SFFF), in HPLC method development and optimization strategies. In 1992 Jack resigned from Du Pont de Nemours & Co, Wilmington, Delaware, and joined Rockland Technologies, Newport, Delaware, as a Director of Research and Development. During this period he successfully developed a series of novel, designed reversed phase silicas. He resigned from Rockland technologies, now Agilent Technologies, Newport, Delaware, in 2001, but always remained dedicated to HPLC.

Keywords: Kirkland; Stationary phases, LC; Silica packings; Instrumentation, LC

Contents

1. Introduction

Joseph (Jack) Kirkland is one of the outstanding protagonists of modern column liquid chromatography. As many other fellow pioneers, he originated in the scientific field associated with gas chromatography (GC) and was attracted by HPLC as an alternative tool to GC to analyze pesticides for the Industrial and Biochemicals (later Agriculture) Department of E.I DuPont de Nemours & Co, Wilmington, Delaware. To expand his knowledge on this emerging technique, he visited the late Joseph Huber at the University of Amsterdam, John Knox at the University of Edinburgh, Istvan Halasz at the University of Saarbrücken and others. It is rather unsur-

* Tel.: +49 6131 392 5927; fax: +49 6131 392 2710.
E-mail address: k.k.unger@web.de.

0021-9673/$ – see front matter © 2004 Elsevier B.V. All rights reserved.
doi:10.1016/j.chroma.2004.07.076

prising that these close and intensive contacts resulted in an international, indeed globally reaching, scientific and technical network and discussion platform which progressively initiated the establishment of a number of successful symposium series such as the Zlatkis symposia and the International Symposia on HPLC. The latter were founded in May 1973 at Interlaken, Switzerland with the late W. Simon as chairman. The rapid development of HPLC, its transfer into an effective instrumental platform and its wide-spread application as a powerful analytical separation technique in the pharmaceutical and chemical industries was primarily a result of these intensive international links and collaboration activities in the period between 1970 and 1980.

After the completion of his Ph.D. degree in Analytical Chemistry from the University of Virginia in Professor John Yoe's laboratory in 1953, Jack entered the Experimental Station at E.I. DuPont de Nemours & Co, Wilmington. He worked at DuPont until his resignation in 1992 and it was at this unique research facility that most of his outstanding contributions to the exciting field of HPLC were developed. I had the extraordinary pleasure to meet Jack at the Experimental Station in 1973 while I was a Visiting Professor with Barry Karger at the Northeastern University, Boston, MA. It was through his intermediation that I was introduced to Lloyd Snyder at Tarrytown, NJ, R.P.W. Scott at Nutley, NJ, and the late Cal Giddings, Salt Lake City, UT. These congregations with leading experts in the field provided me a huge impetus and motivation for my personal research in this area.

After his retirement from DuPont, Jack entered Rockland Technologies at Newport, DE, a company he co-founded with DuPont colleagues and became Director for Research and Development. In 2001, he retired as a Senior Scientist from Agilent Technologies, Inc., the former Rockland Technologies, Inc.

The major activities and achievements of Jack's 50-year lasting period of scientific life are highlighted in Table 1. Based on Table 1, I have picked out the most pronounced contributions which I will briefly discuss in the following six paragraphs.

2. The early days: reorientation from GC to LC

As already pointed out, most of the pioneers in HPLC were the leading experts in GC in which packed columns with non-volatile liquid stationary phases were applied. The use of Capillary GC began a decade later when coated fused silica capillaries were invented in 1979. Jack established collaboration with Steve Dal Nogare at DuPont and worked primarily in bonded phases for GC and in Preparative-Scale GC. Early modern LC was particularly coined by two significant contributions:

(i) the discovery of the chemical bonding of stationary phases to overcome the bleeding of liquid stationary phases, particularly at high mobile phase flow rates, and

(ii) the introduction of pellicular or porous layer bead types of support as intermediate supports to microparticulate totally porous silica beads.

The porous layer beads (PLB) were composed of a solid inert core of approximately 35–50 μm average particle diameter exhibiting a porous layer of approximately 1 μm in thickness. Table 2 provides the pore structural data of PLBs commercially available at the beginning of 1970 [1]. Among them is Zipax, a product which was invented by Jack in 1970 [2]. In comparison to the other PLBs, Zipax showed the largest pore diameter and a low surface area being particularly suited as a support for binding a stationary liquid. Zipax was manufactured by depositing and binding consecutive layers of colloidal silica particles with intermediate polymer layers as binders. The basic idea of using PLBs was to accelerate the mass transfer kinetics of solutes in the stagnant mobile phase as compared to large totally porous particles. As shown in Table 2, the specific surface areas of such PLBs were by more than one order of magnitude smaller than those of totally porous silica particles with the consequence that the mass loadability of such columns were considerable smaller. Also, the mass transfer kinetics of retained solutes increased quite substantially with increasing linear velocities as expressed by the magnitude of the C-term in the plate height versus linear velocity dependencies. Jack made significant contributions in both fields which are documented by a number of publications [3–7].

The PLBs can be considered as an intermediate step to the development of totally porous microparticulate spherical packings. The PLBs disappeared from the market and came back recently as Poroshell particles for the rapid resolution of proteins marketed by Agilent Technologies (see Table 3).

3. The pioneering work in silica packings for LC: the Zorbax technology

Before I discuss the merits of Jack in this important development of HPLC, I would like to highlight the scientific and technical situation of this period as a whole from a personal point of view. It was exactly the period when the author entered the field as a young scientist experienced in the field of silica synthesis.

The thrust to use small totally porous particles of approximately 10 μm average particle diameter and smaller was an obvious conclusion derived from the results of theoretical predictions on the column performance of HPLC columns as the function of the linear velocity with the particle diameter of support representing the decisive parameter. It was clearly shown by the late J.F.K. Huber [8] and others that the dominant contribution to the total plate height at increasing velocity stems from the mass transfer kinetics of an analyte in the stagnant mobile phase within the particles (H_{Eb} in Fig. 1). The term increases with the particle diameter squared

Table 1
Time table of major scientific activities and achievements of Jack

Period	Activities and achievements
1955–1969	Synthesis of permanently bonded phases for gas chromatography (GC), preparative GC.
1965–1973	Synthesis of porous layer beads (Zipax), manufacture of microparticulate silica beads in collaboration with R.K. Iler (Zorbax technology) development of monolayer bonded phases based on Zorbax (Collaboration with Joe de Stefano).
1984–1992	Development of improved silica (Zorbax Rx, Type B silica) with improved chemical properties for chromatography of basic compounds (Collaboration with Juergen Koehler). Development of steric-protected and bidentate silanes for improved bonded-phase stability at low and high pH (Collaboration with Joe Glajch).
1966–1991	Development of novel LC instrumentation and of Field Flow Fractionation (FFF) equipment (collaboration with C. Giddings) at the Experimental Station of E.I. DuPont de Nemours.
1975–1992	Advanced polymer analysis: manufacture of silica based SEC packings, fractionation of colloidal particles by Sedimentation Field Flow Fractionation (SFFF) (collaboration with C. Giddings).
1979–1983	Development of peak detection, data handling algorithms and software for LC (collaboration with W. Yau).
1980–1987	HPLC method development and optimization (collaboration with J. Glajch, Jim Minor, and L.R. Snyder).
1991–2001	Commercial introduction and development of a series of novel, specially designed bonded silicas at Agilent Technologies, formerly Rockland Technologies (collaboration with Joe De Stefano, John Adams, and Brian Bidlingmeyer). Development of a small-particle, superficially porous silica support (Poroshell) for high-speed separation of macromolecules. (collaboration with Tim Langlois).

at increasing linear velocity. Microparticulate particles reduce this contribution considerably. As a consequence, the average diameter of the particles had to be substantially reduced as compared to 35–50 μm of the PLBs.

At this point of time, however, microparticulate supports were manufactured by milling and consecutive sizing of larger particle's batch production. The products were essentially irregular particles. The foremost problems in view of their use was the large proportion of fines adhering at the surface of such particles and the achievement of a stable and regular column bed. Thus, the major activity was directed to the synthesis of spherical particles with controlled pore size and porosity. The spherical particles were formed during the synthesis and only a single sizing step was required. One of the major routes of making spherical particles is to agglomerate colloidal or non-porous nanoparticles to assemblies or stable aggregates. DuPont de Nemours at Wilmington, DE, was a manufacturer of a large variety of silica sols called LUDOX which served as lubricants, fillers and thickeners in various products. Moreover, one of the world's most renowned experts in the field of colloidal silica chemistry of the time was working at DuPont: R.K. Iler. He was the author of the famous book on "The Chemistry of Silica" published by Wiley Interscience, New York, 1979, which became the most reputed book in this field [9].

Based on the phenomenon of flocculation Iler patented a process by which colloidal silica particles were bridged in the presence of a urea–formaldehyde polymer forming spherical beads [10]. The polymer located in the interstices of the agglomerated particles were removed by burning the polymer at elevated temperatures from the agglomerates maintaining the shape and size and simultaneously mechanically strengthening the assembly. The resulting product was called Zorbax which became one of the most popular silica packings in HPLC. The pore structural parameters of Zorbax were as follows: specific surface area 275 m^2/g, specific pore volume 0.4 cc/g and average pore diameter 7.5 nm, being a typical mesoporous adsorbent. In contrast to other comparable silica products, Zorbax possesses a relatively low specific pore volume which leads to a high packing density of approximately 0.8 g/cc. The specific surface area (a_s), the specific pore volume (v_p), and the average pore diameter of agglomerated products (p_d) are determined by the size of the colloidal silica particles and the contact number in the particle as follows:

$$a_s = \frac{6}{d_p \times d_{app(He)}} \quad (1)$$

where d_p is the average particle diameter of the colloidal sil-

Table 2
Pore structural data of commercial pellicular types of silicas and comparable data of Zorbax (extracted from Ref. [1] with permission of the publisher)

Type	Particle diameter range (μm)	Specific surface area by BET (m^2/g)	Specific pore volume v_p (μl/g)	Mean pore diameter p_d (nm)
Zipax (DuPont de Nemours)	35–37	0.93	23	38
Corasil I (Waters)	37–50	14.7	40	3
Corasil II (Waters)	37–50	24.4	49	5
Perisorb (Merck)	35–45	14.2	24	3
Zorbax-Sil (DuPont de Nemours)	3–10	300	400	6

Table 3
Advanced native and bonded silica columns developed by Jack and his collaborators at Agilent Technologies

Type and assignment	Specific structure and surface chemistry	Recommended use in LC
Normal phase columns		
Zorbax Rx-SIL	Highly pure silica, low surface acidity	Basic analytes
Reversed phase columns		
Stable bond (SB) columns (8 nm)	Monofunctional silanes with diisopropyl side groups and C8, C3, Phenyl, cyano terminating groups; no endcapping monofunctional silanes with diisobutyl side and terminating C18 groups; no endcapping	Low pH separations with acidic mobile phases superior stability and reproducibility; wide window of selectivity; choice of high temperature operation (SB C18 up to 90 °C)
StableBond (SB) columns (8 nm, 30 nm)	Ultrapure Zorbax Rx silica as supports with 8 and 30 nm pore size, respectively, diisopropyl side groups and C8, C3, cyano terminating groups; no endcapping monofunctional silanes with diisobutyl side and terminating C18 groups; no endcapping	Separation of biologically active analytes, e.g., peptides and proteins at low pH high temperature operation (SB C18 up to 90 °C)
Zorbax Eclipse XDB columns (extra densely bonded)	Zorbax Rx Sil as a support (8 nm pore size), dimethylalkylsilyl spacer with terminating phenyl, C 8, and C18 groups, respectively	Basic compounds, stable at low to mid pH mobile phases, rapid separations pH working range between 3 and 8
Zorbax Extend-C18 (bidentate silane)	Bidentate silane with C18 groups	Increases utility of silica-based packings to high pH limits (useful in pH range of 2.0–11.5).
Poroshell 300SB-C18 column	Solid core with a porous shell of 30 nm pore size stable bond C18 functionality	Very fast protein separations at low pH, high column efficiency

icas and $d_{app(He)}$ is the apparent density of the silica skeleton due to helium (2.2 g/cc), and

$$p_d = \gamma \left(\frac{v_p}{a_s} \right) \times 10^3 \quad (2)$$

where γ is the contact number between the particles (average value 6), v_p represents the specific pore volume, and a_s denotes the specific surface area of the particles [11].

The process was optimized with respect to high yield and to generate microparticle of approximately 5 μm average particle size. Jack immediately recognized the value of Zorbax in HPLC and investigated the column performance of these particles in normal phase chromatography and liquid–liquid partition chromatography [11–14].

One should keep in mind that there are a number of technical obstacles to overcome to successfully use such microparticulate packings:

(i) the appropriate technology to cut narrow size fractions from the material as synthesized material,
(ii) the choice of a pressure stable column hardware as stainless steel columns with a mirror finish inside,
(iii) the development of a porous frit system which holds the particles and exhibits a low extra column dead volume, and
(iv) the development of an effective column packing procedure which generates stable columns such as the slurry packing technique.

It took the column manufacturers approximately a period of 10 years to produce stable high performance columns with a high column-to-column reproducibility.

By varying the size of colloidal silicas, the pore size of the Zorbax particles can be adjusted in a wide range of mesopores, namely between 3 and 50 nm. Columns packed with such microparticles were introduced as Size Exclusion columns by Jack to fractionate synthetic polymers [15,16] and colloids [17].

High Performance Size Exclusion Chromatography (SEC) of synthetic polymers became a major field of activity of Jack during the mid seventies as a tool to assess the molecular weight distribution of technical polymers at DuPont de Nemours. The collective research in SEC finally lead to the famous monograph on "Modern Size Exclusion Chromatography" by W.W. Yau, Jack, and D.D. Bly, John Wiley & Sons, New York, 1979.

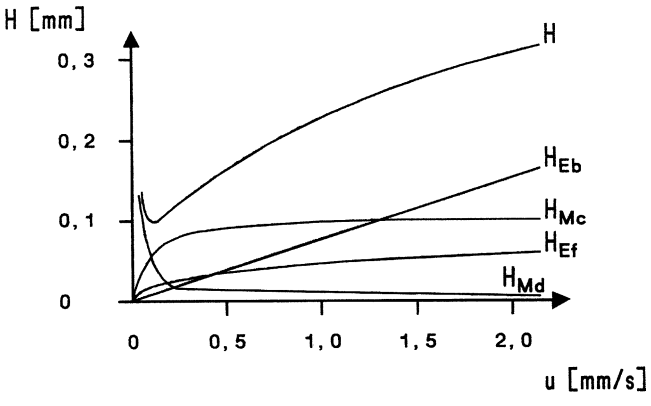

Fig. 1. Dependency of the total theoretical plate height and their terms from the linear velocity of the mobile phase in liquid chromatography (reprinted from [8] by permission of the publisher). H_{MD} contribution due longitudinal diffusion, H_{MC} contribution due to convective mixing, H_{Ef} contribution due mass transfer in the moving phase, and H_{Eb} contribution due to mass transfer in the stagnant phase.

During the first period of introducing microparticle silica packings, the major focus was directed towards the gain of high column efficiency in terms of theoretical plates per column length or number of effective plates per unit time. It was nearly forgotten that the chromatographic resolution is governed by the selectivity of the phase system (stationary/mobile phase). At this period, silica columns were either run under normal phase conditions with organic solvents such as *n*-hexane with controlled water content or run under liquid–liquid partitioning conditions with non-volatile polar or non-polar liquid stationary phases.

The evolution of chemically bonded hydrophobic stationary phases which were collectively named as reversed (reverse) phase packings in HPLC began in 1970. Kirkland and DeStefano synthesized a hydrophobic bonded phase by coupling a poly-*n*-octadecylsiloxane to the surface of a pellicular support [5,18]. Later on, Majors [19] prepared bonded phases by the reaction of organotrichlorosilanes with microparticulate silicas.

During the mid seventies the vast majority of the commercial reversed phase (RP) silicas were synthesized and manufactured. Jack and his co-workers developed a series of such columns based on Zorbax SIL: Zorbax C18, Zorbax C8, Zorbax Phenyl, Zorbax CN, and Zorbax TMS with an average pore diameter of 7 nm and a specific surface area of 300 m^2/g. The surface modification was achieved by employing monofunctional silanes.

It was recognized early that the reversed silicas of the mid-seventies were not suited for the resolution of basic analytes; peak tailing occurred and the retention coefficients of bases varied. The key of minimizing these undesired interactions between the surface hydroxyl groups of the silicas and the basic groups of the analytes was to control and to adjust the acidity of the silica. A chief contribution to the understanding of these effects were made by Jack and his co-worker J. Koehler (Fig. 2) who spent 2 years as a post doc at the Experimental Station of DuPont de Nemours [20,21].

4. Instrument design and development at the Experimental Station at DuPont de Nemours, Wilmington, DE

Although columns are often referred to as the heart of any chromatographic separation system, it cannot function without a suitable detection system and other adjusted modules. Thus, some of Jack's activity was directed towards the development of chromatographic systems in collaboration with the Instrument Products Division of DuPont de Nemours.

A particularly challenging task was the design of a commercial Field Flow Fractionation (FFF) instrument based on sedimentation of the sample in a centrifuge (Fig. 3). Sedimentation Field Flow Fractionation (SFFF) is a method of sorting macromolecules and particles according to their size and does not need any calibration. This development was based on the theoretical work of the late C. Giddings and conducted in cooperation with DuPont's Instrument Products Division, Wilmington, which was already marketing high speed centrifuges. After an intensive period of construction and testing between 1980 and 1990, it became a commercial instrument. Eastman Kodak, Rochester, NJ, was one of the first customers. Today FFF is a common technique in characterizing nanoparticles, agglomerates, and polymers.

Jack and his co-workers W.W. Yau, F.C. Szoka, A. Fox, and L.E. Schalinger have advocated the method and their application in a number of internationally recognized and leading scientific journals [22–29].

5. Method development and optimization strategies for LC: the successful network between Jack, Joe Glajch, and Lloyd Snyder

Due to the large variety of application of HPLC, in virtually all fields of the pharmaceutical and chemical industries the common user frequently seeks for guidelines to develop an appropriate methodology and to solve the analytical problem. Jack recognized early the needs in method development to optimize column selectivity and developed retention maps and experimental design techniques [30–34] with his co-worker J.L. Glajch. These optimization strategies were implemented as software into the DuPont HPLC instruments.

A comprehensive review on "Practical HPLC development" was published as a monograph by L.R. Snyder, J.L. Glajch, and Jack, John Wiley and Sons, New York, 1988 (first edition) and 1997 (second edition).

Fig. 2. Juergen Koehler (left), Jack (middle), and K.K. Unger (right), 1985 at the University of Mainz celebrating the Ph.D. thesis of Bernd Straube.

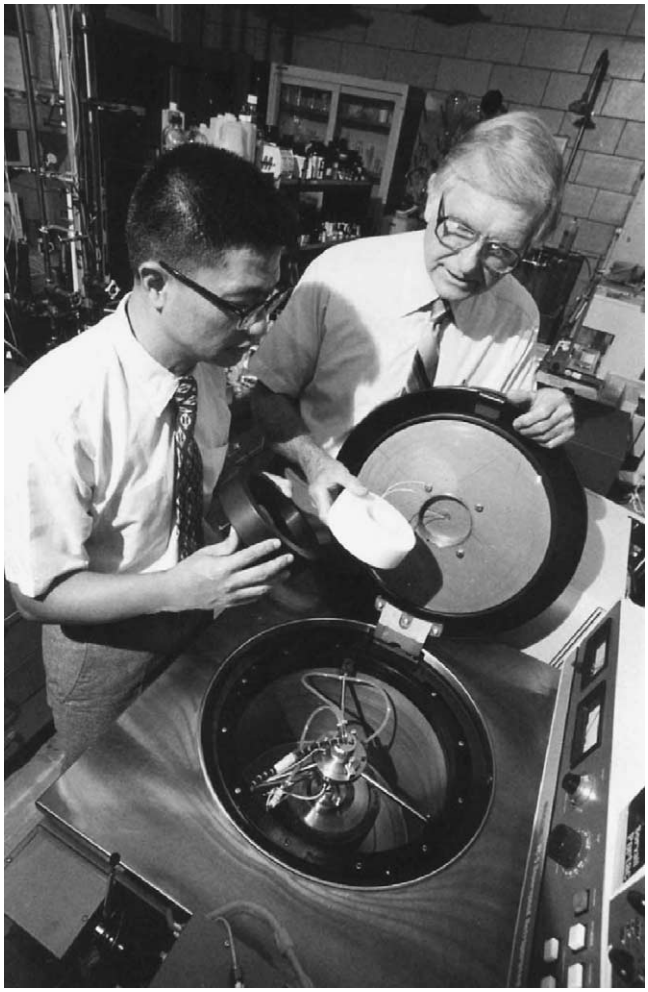

Fig. 3. W.W. Yau (left) and Jack (right), in front of an SFFF instrument 1985 at DuPont de Nemours, Wilmington (kindly supplied by Joe DeStefano, Agilent Technologies, Inc.).

6. Learning by teaching

Teaching is often considered to be of minor importance in the career of a scientist. In developing novel analytical methodologies, the teaching of students becomes an extremely valuable exercise in a twofold way: one is directly confronted with the daily problems of the user and one learns to know the major application areas of the emerging technique. Jack conducted short introductory courses on HPLC and on practical HPLC method development over a period of 25 years from 1971 to 1996 in collaboration with L.R. Snyder and organized by the American Chemical Society (ACS). In addition to this successful undertaking, he was the co-author of two taped ACS Audio Short Courses on HPLC. This collective teaching exercise was probably the initiator for writing his most popular monographs: Modern Practice of Liquid Chromatography 1971 (co-author: L.R. Snyder), Introduction to Modern Liquid Chromatography 1974 (co-author: L.R. Snyder), and Practical HPLC Method Development 1988, second edition 1997 (co-authors L.R. Snyder and J.L. Glajch).

7. Designed reversed phase columns: the period at Agilent Technologies, formerly Rockland Technologies

It was recognized early by column manufacturers that reversed phase columns should possess different properties in terms of surface chemistry to fulfill the demands on selectivity in a wide range of application areas. The major need was to synthesize RP silicas to separate basic analytes with sufficiently symmetrical peak shape and reproducible retention coefficients. The problem was solved by manufacturing silicas with a reduced acidity and a high purity and appropriate *n*-alkyl functionality. Furthermore, many separations were performed using weakly acidic mobile phases or acidic/organic mobile phases at pH 2–3 (e.g., peptides). This requires RP packings with a high stability at the acidic pH range. The same requirement was needed at higher pH values between 8 and 10. Jack and his team at Rockland Technologies successfully developed a number of surface chemistries, including the introduction of steric protection of the siloxane bond that holds the bonded phase to the silica surface (StableBond) and bidentate bonded phases with two anchoring siloxane bonds (Extend) to tackle these problems [35–41].

A survey of HPLC columns which were developed under the Jack's guidance is given in Table 3. It demonstrates a wide spectrum of packing types, including a number of customized columns for specific applications.

The success leading to produce such column types may be sought in joint activities in three fields: (i) systematic studies on the bonding chemistry, (ii) the comprehensive physicochemical and chromatographic characterization, and (iii) the column life-time tests in different mobile phases. The latter subject was intensively studied with the group of H. Claessens at the Technical University of Eindhoven, The Netherlands [42–48].

Jack's latest developments were superficially porous silica microspheres for the fast HPLC of macromolecules [49,50]. The product was commercialized as Poroshell 300SB-C18 (see Table 3)

The keys to the success were the following requirements: both the basic silica supports as well as the silanes were in-house products of Rockland Technology: in other words, all production steps were at one place with minimum loss in information and maximum competence.

In contrast to the market strategies of some column manufacturers to throw low price columns on the market, the columns based on the Zorbax technology were always high price products. Quality manipulates its price, if one takes into account the enormous efforts of extended studies and the in-depth experiences which are required.

8. Conclusion—a personal remark

The author had the pleasure to follow the scientific activities of Jack closely over a period of 30 years. To merit

his credits for the scientific community in separation science Jack deserves the following attributes.

He is an all-around scientist who dedicates meticulous attention to the principles and basic phenomena before he starts an experiment. He deals thoroughly with the basic underlying chemistry and the system technology. He has golden hands in chemical experiments and an open mind to find and to motivate his collaborators. Jack always gives credit to the people who performed the hands-on work for him. He particularly appreciated the diligent work of Glenn Wallace, Charlie Dilks, Jack Henderson, and Frank Truszkowski.

He applies his knowledge and expertise to develop practical solutions. In these aspects he is a very unique person.

The environment of the Experimental Station at DuPont de Nemours and later at Rockland Technologies, now Agilent Technologies, gave him a strong motivation, stimulation and support.

Jack has a warm and open minded character and personality. He was and is an immense supporter to me over a long period of time and we are good friends. It was a pleasure and honor to write this article.

Acknowledgements

I thank Dr. Joe DeStefano, Agilent Technologies, Inc., Newport and Andre Dams, Agilent Technologies BV, Amsterdam for their helpful information and providing Fig. 3. My particular thanks are to Dr. Tom Hennessy of my group for linguistic improvements of the manuscript.

References

[1] K.K. Unger, P. Ringe, J. Schick-Kalb, B. Straube, Anal. Chem. 264 (1973) 267.
[2] J.J. Kirkland, Superficially Porous Supports for Chromatography, US Patent 3,505,785, April 14, 1970.
[3] J.J. Kirkland, Anal. Chem. 41 (1969) 218.
[4] J.J. Kirkland, J. Chromatogr. Sci. 7 (1969) 7.
[5] J.J. Kirkland, J.J. de Stefano, J. Chromatogr. Sci. 8 (1970) 309.
[6] J.J. Kirkland, J. Chromatogr. Sci. 9 (1971) 201.
[7] J.J. Kirkland, J. Chromatogr. Sci. 10 (1972) 129.
[8] J.F.K. Huber, Ber. Bunsenges. Physikal. Chem. 3 (1973) 179.
[9] R.K. Iler, The Chemistry of Silica, Wiley Interscience, New York, NY, 1979.
[10] R.K. Iler, H.J. McQueston, Uniform Oxide Microspherea and a Process for Their Manufacture, US Patent 3,855,172, December 17, 1974.
[11] K.K. Unger, in: K.K. Unger (Ed.), Packings and Stationary Phases for Chromatographic Techniques, M. Dekker, New York, NY, 1990, p. 338.
[12] J.J. Kirkland, J. Chromatogr. Sci. 10 (1972) 593.
[13] J.J. Kirkland, J. Chromatogr. 83 (1973) 149.
[14] J.J. Kirkland, in: S.B. Perry (Ed.), Gas Chromatography 1972, Applied Science Publishers, Essex, UK, 1973, p. 39.
[15] J.J. Kirkland, J. Chromatogr. 125 (1976) 219.
[16] J.J. Kirkland, P.E. Antle, J. Chromatogr. Sci. 15 (1977) 303.
[17] J.J. Kirkland, J. Chromatogr. 185 (1979) 273.
[18] J.J. Kirkland, Chromatographia 8 (1975) 661.
[19] R.E. Majors, Anal. Chem. 44 (1971) 1722.
[20] J. Koehler, D.B. Chase, R.D. Farlee, A.J. Vega, J.J. Kirkland, J. Chromatogr. 352 (1986) 275.
[21] J. Koehler, J.J. Kirkland, Porous Silica Microspheres Having Silanol Enriched and Silanized Surfaces, US Patent Nr. 5,032, 266, July 16, 1991.
[22] J.J. Kirkland, W.W. Yau, F.C. Szoka, Science 215 (1982) 296.
[23] J.J. Kirkland, W.W. Yau, Science 218 (1982) 121.
[24] J.J. Kirkland, W.W. Yau, Anal. Chem. 55 (1983) 2165.
[25] J.J. Kirkland, L.E. Schalinger, W.W. Yau, Anal. Chem. 57 (1985) 2271.
[26] J.J. Kirkland, W.W. Yau, Macromolecules 18 (1985) 2305.
[27] A. Fox, L.E. Schalinger, J.J. Kirkland, J. Microbiol. Methods 5–6 (1985) 273.
[28] J.J. Kirkland, W.W. Yau, J. Chromatogr. 353 (1986) 95.
[29] J.J. Kirkland, C.H. Dilks, Anal. Chem. 64 (1992) 2836.
[30] J.L. Glajch, J.J. Kirkland, Anal. Chem. 55 (1983) 319A.
[31] J.L. Glajch, J.J. Kirkland, J.M. Minor, J. Liquid Chromatogr. 10 (1987) 1727.
[32] J.L. Glajch, J.J. Kirkland, J. Chromatogr. 485 (1989) 551.
[33] J.J. Kirkland, J. Chromatogr. Sci. 31 (1993) 493.
[34] J.J. Kirkland, LC–GC Int. 14 (1996) 486.
[35] J.L. Glajch, J.J. Kirkland, Stable Support Structure for Chromatographic Columns—Comprising a Substrate Coated with a Monofunctional Silane Containing Two Sterically Protected Groups, US Patent 4,705, 725, November 10, 1987.
[36] J.L. Glajch, J.J. Kirkland, Structures Surface-Modified with Bidendate Silanes, US Patent 4,746,572, May 24, 1988.
[37] J.L. Glajch, J.J. Kirkland, Substrates Coated with Organosilanes that are Sterically Protected, US Patent 4,847,159, July 11, 1989.
[38] J.J. Kirkland, J.B. Adams Jr., Asymmetric Bidendate Silanes, US Patent 5,869,724, February 9, 1999.
[39] J.J. Kirkland, J.B. Adams Jr., Propylene-bridged Bidendate Silanes, US Patent 5,948,531, September 7, 1999.
[40] J.J. Kirkland, J.B. Adams Jr., Propylene-bridged Bidendate Silanes, US Patent 6,057,468, May 2, 2000.
[41] J.J. Kirkland, J. Chromatogr. Sci. 34 (1996) 309.
[42] J.J. Kirkland, J.W. Henderson, J.J. de Stefano, M.A. van Straten, H.A. Claessens, J. Chromatogr. A 762 (1997) 97.
[43] J.J. Kirkland, M.A. van Straten, H.A. Claessens, J. Chromatogr. A 797 (1998) 111.
[44] J.J. Kirkland, J.B. Adams Jr., M.A. van Straten, H.A. Claessens, Anal. Chem. 70 (1998) 4344.
[45] J.J. Kirkland, J.D. Martosella, J.W. Henderson, C.H. Dilks, J.B. Adams Jr., Am. Lab. 31 (1999) 22.
[46] J.J. Kirkland, J.W. Henderson, J.D. Martosella, B.A. Bidlingmeyer, J. Vasta-Russell, J.B. Adams Jr., LC–GC Int. 17 (1999) 634.
[47] J.J. Kirkland, M.A. van Straten, H.A. Claessens, J. Chromatogr. A 691 (1995) 3.
[48] H.A. Claessens, M.A. van Straten, J.J. Kirkland, J. Chromatogr. A 728 (1996) 259.
[49] J.J. Kirkland, F.A. Truszkowski, C.H. Dilks Jr., G.S. Engel, J. Chromatogr. A 890 (2000) 3.
[50] J.J. Kirkland, J. Chromatogr. Sci. 38 (2000) 535.