ACCOUNTS OF CHEMICAL RESEARCH®

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VOLUME17

NUMBER 4

APRIL, 1984

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Research for Profit: The Chief Executive Officer Connection

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More than 40 years of continuous exciting experience as a chemical research scientist has brought one critical realization clearly into focus: the person who often holds the key to unlocking opportunities for "Research For Profit" is the Chief Executive Officer of a company whether the company is a giant or an adolescent. Why? Because the Chief Executive Officer (sometimes with strong input from his chief scientific advisor—usually the Vice President for Research and Development) holds the ultimate *decision*-making power. A few other pertinent conclusions are also worth mentioning:

(1) There is really no distinction between "Research For Profit" and "Research For Nonprofit". All research *profits* mankind because it expands the *reservoir* of knowledge.

However, the recognition of the utility of new knowledge does require the spark of creativity to build the bridge between new knowledge and its utility. In this sense, the terms basic, applied, pure, fundamental, academic, and industrial research (terms around which, alas, some cults of science have organized) are as archaic as the "buggywhip". In my personal view, they should be amalgamated into one uniform category, "Research For New Knowledge". New knowledge blankets its benefits—along with its attendant risks—upon all inhabitants of our planet just as the sun blankets our planet with its energy which has benefits that far outweigh its risks.

(2) It is the *profits* from research that make new jobs, pay salaries and pensions—a point often overlooked by academe.

(3) A shockingly high percentage of so-called research is based on "old hat" or "me too" concepts or ideas.

(4) Time often dims the memory of the initial creative events, so much so that years later those who were least receptive to a new idea promulgate recollections that involve them as key participants.

(5) The number of dollars invested in research is a completely misleading yardstick for ensuring or projecting profits that may emerge from any given research program.

(6) Research reviewed by committees can benefit from a variety of inputs by experts. But committees can be no better than those who are its members, and only rarely can they *plan* innovative research.

(7) The "bottom line" of research productivity diminishes with the cube of the required paperwork.

(8) Creativity in research diminishes with the square of the distance of the key man from the lab bench.

(9) The ultimate *commercial* success of a scientific or technological innovation depends on a combination of the newness of the knowledge, the soundness of the patents providing temporary exclusivity, and the competence of the marketing support.

(10) The ultimate commercial success of new creative scientific or technological innovation requires a sound academic training by the innovator or innovators. The original enthusiasm of the primary innovator loses much of its original *critical* importance by the time an invention is tested in the "fire" of the marketplace.

Let us embark on a real-life journey, visiting a few cases in my own career, and pinpoint the overriding importance Chief Executive Officers played as "Project Champions". These case histories are intended as documentable experimental data to support my thesis stated earlier: often, but not always, the critical person who holds the key to unlocking opportunities for "Research For Profit" is the Chief Executive Officer of an organization primarily because he holds the maximum decision-making authority. In stating this thesis, I nevertheless recognize that many products have reached high levels of commercial success when a vice president or research manager took the initiative to be "Project Champion".

As we expose the realities of my "experimental data", I will dwell briefly on some salient anecdotal illustrations of supporting attributes that are essential for the innovator—the conceptor—without whom there can be no "Research For Profit" at all!

Product Case History Number One: AVICEL Microcrystalline Cellulose

We will begin with the first of my commercially successful inventions, the conception of which occurred accidentally in April 1955: AVICEL Microcrystalline Cellulose.

Around 1955, nylon tire cord was introduced by Du Pont as a competitor of rayon tire cord in rubber tires. Rayon tire cord had replaced cotton tire cord about a decade earlier and had grown into a lucrative business of well over 200 million dollars a year in sales.

At American Viscose—where I was a research chemist in 1955—a plea went out from management to meet the growing competitive threat.

My preoccupation at that time was the study of the microcrystalline structure of cellulose fibers; hundreds of other cellulose chemists were equally preoccupied along the same lines. Cellulose was an ideal natural polymer for using X-ray diffraction and electron microscopy as tools to expose the internal molecular architecture of long-chain molecules.

The fine structure of nylon fibers used to make nylon tire cord was well known to me. It comprised ultrasmall ordered crystals strongly oriented in the direction of the length of the fibers.

In April 1955, an idea surfaced in my mind during a lunch hour in answer to the obvious question "How can I make the fine structure of rayon tire cord fibers more like that of nylon tire cord fibers?"

Specifically, the idea was: Recover the single microcrystals in rayon, chop them into smaller fragments, and then inject the smaller crystal fragments into the



Figure 1. AVICEL microcrystalline cellulose ($\sim 12\%$ aqueous gel).

viscose solution just before it emerged from the spinneret. Could we "seed crystal growth" in this way to produce rayon fibers having smaller, more perfect, highly oriented crystals?

The critical experiment that produced an unexpected result changed the course of my career even to this day. Yet this experiment—based on the idea—involved equipment no more complicated than a Waring Blendor. So impressed have I been by this inexpensive piece of equipment that it has continued constantly to do additional chores for me ever since. I am sure there are at least 10 Waring Blendors in various nooks and corners of my own Institute today!

I asked my laboratory assistant, Pat Smith, to perform an experiment for me. I asked her to put about 40 g of dry rayon microcrystals (I called them by the name "Level-off D. P. Cellulose" at the time) in 400 mL of distilled water and turn the Waring Blendor on at high speed. "Hopefully," I told Pat Smith, "the sharp blades of the Waring Blendor will disperse the rayon microcrystals and fracture them into smaller fragments. We will collect them from the supernatant water—the heavier particles will settle like sand because cellulose crystals are insoluble in water—and recover them as they remain in Brownian motion."

It was these tiny cellulose microcrystal fragments that I planned to inject into the viscose solution to "seed" the crystallization that occurs when the viscose solution is regenerated as it emerges from the spinneret holes and is transformed into cellulose fibers.

About one-half hour later, Pat Smith brought me the top part of the Waring Blendor and said to me, "Your idea didn't work. Look at the goo we produced. Shall I dump it down the sink and start all over?" What I looked at made me swell with excitement. "Pat," I replied, "that is the most beautiful sight I have ever seen! That gel doesn't have a calorie in a carload. We've got a zero-calorie, 'Crisco-like gel' for the food industry." A mound of the original AVICEL Microcrystalline Cellulose gel is shown in Figure 1.

So much for the genesis of a simple experiment that gave an unexpected result. By itself, this result may never have led to three large commercial plants around

Orlando A. Battista is a Canadian by origin, born in 1917 in Cornwall, Ontario, and educated at McGill University. In 1940, he moved to the United States and joined American Viscose Corp., where he was Assistant Director of Corporate Research, 1961-1963. After American Viscose was merged into FMC Corporation in 1964, he served as Assistant Director of the FMC Central Research Department uniti 1971, when he moved to Avicon, Inc., as Vice President, Science and Technology. In 1974, he retired early to organize Research Services Corporation and the O. A. Battista Research Institute, and he continues to be president of both. He was Adjunct Professor of Chemistry and Director of the Center for Microcrystal Polymer Science at the University of Texas, Arlington, in 1975-1976, and served as President of the American Institute of Chemists, 1977-1979. His current primary activity is that of Founder and President of World Olympiads of Knowledge. The present Account is adapted from his 1983 Creative Invention Award Lecture delivered at the 185th national American Chemical Society meeting in Seattle, WA. March 1983.

⁽¹⁾ Battista, Orlando, A.; Smith, Patricia A. U.S. Patent 2978446, 1961.

the world that have produced hundreds of millions of pounds of AVICEL, led to the employment of thousands of persons, and became a major adjunct of the cellulose industry by making cellulose available in a new, nonfibrous physical form. Hundreds of new uses for this colloidal form of pure cellulose have been found, and its future is brighter today than when it was first introduced commercially in 1962. Its major utility is in pharmaceutical tableting, as a superior binder and excipient, and in the food industry.

It was the realization of the *commercial potential* of that beautiful, unexpected gel in the Waring Blendor jar that was a most important part of the conception process that initiated the germination of a new industry. Without this extrapolation of the "Research For Profit" side of the *unexpected* result, there would have been no germination, and the value of the idea and the experiment would have been completely aborted. Nevertheless, I insisted that Pat Smith join me as the coinventor of the composition of matter patent for colloidal microcrystalline celluloses.¹

Cellulose chemists—including myself—had been studying cellulose microcrystals for years. It was the realization of their commercial value as high solid suspensions that catapulted them into profits. But this product, as you will see, still would never have sustained the fetal gestation period, let alone birth, without the *direct* support of the Chief Executive Officer of American Viscose Corporation. His critical action as "Project Champion" is described a little later as this story evolves.

Actually, the Vice President of Research and Development at American Viscose came to my rescue in 1961. At that time, my immediate supervisor wanted to fire me for "playing around with a useless form of powdered cellulose". I cringe when I reflect how my career could have come to a standstill if this supervisor had prevailed. This Research and Development Vice President transferred me to a new Special Products Section and gave me a laboratory of my own and two assistants-my first "invention development laboratory". In fairness to the supervisor, I should mention, for the record, that, at the time I had been given my first managerial training assignment as Head of American Viscose's Analytical Laboratories; cooking low-calorie cheese cakes and whipping up zero-calorie imitation butter spreads (during lunch hours and weekends) was not quite within the scope of my official title.

The original emphases for AVICEL were directed to controlling the calorie contents of high-fat foods. Food companies were hesitant to produce such "dietary products" because of legal "standards of identity" of basic foods *and* the fact that dietary foods in the early 1960s had a limited market because they were generally not appetizing.

In retrospect, I remember meeting with Dr. Frederick Stare, a famous nutritionist at Harvard. He agreed enthusiastically with me that the concept of reducing fats in foods by adding a safe noncaloric, nonfood ingredient had important health value. It would permit overweight persons to eat ice cream, desserts, etc., with much less restraint—at half the calorie content. He wrote a syndicated column to this effect, but the idea died on the vine temporarily. Over 20 years later, we are getting close to reopening this potentially valuable route to controlling obesity.

It was, however, the Chief Executive Officer of American Viscose who came through with a decision that planted the roots for AVICEL's success. He convinced the Board of Directors to approve building a commercial facility to manufacture AVICEL at Newark, DE even before orders were in hand and before a market had been demonstrated. However, in the interim, numerous patents were filed.

For the record, however, let me assure you that the approval to invest several million dollars in a commercial plant to manufacture AVICEL was not made casually. I remember the occasion well! Out of the blue one day, months before the decision to go ahead with the plant, the Chief Executive Officer (Chairman) and Chief Operation Officer (President) of American Viscose Corporation showed up in my little office and laboratory. They told me they were considering a major decision with respect to AVICEL. Before doing so, they wanted *me* in front of them to repeat that Pat Smith "Waring Blendor" experiment I had been repeating at every appropriate occasion thereafter. They wanted to see for themselves why I had been (almost outrageously) enthusiastic about AVICEL.

As they stood by, I weighed out the "unhinged" cellulose microcrystals in the form of a dry white powder. I added about 40 g of it to 400 mL of distilled water in a Waring Blendor. I turned on the switch and explained I would run the blender in 5-min spurts, letting the motor cool in between, to avoid ruining the motor. Both of them nodded their heads as they sat close by on lab stools.

The Waring Blendor hummed away. I had done this experiment dozens of times just to make supplies of AVICEL gels to experiment with. If there is one time—perhaps the only time—one can develop a feeling of overconfidence it is when repeating an experiment that has been repeatedly reproduced by you—and especially others as well—time and time again.

On schedule, about 45 min later, I turned the jar of the Waring Blendor over to them. Pointing to the smooth "Crisco-like gel" it contained, I said simply what I said originally to my assistant, Pat Smith: "When I first saw that gel, I exclaimed that it was the most beautiful sight I had ever seen. It has many of the properties of a fat, but there isn't a calorie in a carload. It consists of nothing but pure, underivatized cellulose microcrystals suspended in water."

The two top executives looked at the gel. I handed them each a spatula, and they convinced themselves that it did indeed "spread like butter". They looked at each other, smiled, and nodded their heads approvingly. I smiled, too, thinking I had made my point and all was well.

Then, catching me completely by surprise, the Chief Operating Officer said to me: "We're very impressed indeed. But now we want you to do one more experiment for us. We want you to repeat this experiment exactly as you just did. This time, however, we want you to run it using finely ground α -cellulose wood pulp in which you have not yet unhinged or disconnected the microcrystals."

Again, this request caused me no alarm. I had done this experiment many times before also. So I loaded the Waring Blendor with pure fine ground wood pulp and began letting it whirl away. About 45 min later when I stopped it after the intermittent spurts, I turned the jar of the Waring Blendor over to them. The finely ground α -cellulose was settling out of the water almost like sand. The water at the top of the Waring Blendor was getting clearer all the time. When they removed some of the material that had settled and placed it between their fingers they found that it was still rough and fibrous to the touch; that there was no hint of a "Crisco-like gel" forming.

They again smiled, nodded their heads approvingly, thanked me, shook my hand, and left. I was never to visit either of these gentlemen in person again! But the approval to build the Newark, DE, plant came shortly thereafter and American Viscose stock increased \$60 000 000 in 10 days when LIFE Magazine ran a feature article about AVICEL. About two years later FMC bought out American Viscose.

The foregoing experiments had to be repeated once again, but this time before the Examiner in the Patent Office who was handling the composition of matter case. He—like the Chief Executive Officer and Chief Operating Officer at American Viscose-had to "put his hand into the wound" before he would believe. The patent attorney in charge of the application accompanied me to the Patent Office-Waring Blendor and all. Again the laborious, noisy experiments were repeated. This time the patent attorney said to the examiner: "I want you to see first hand the significance of this invention. Spread some of the "Crisco-like" suspensoid on your open window. Beside it, spread some of the material in the Waring Blendor from the wood-pulp run. Let them dry and then scrape each of them with your penknife."

The patent examiner did just that. When he scraped the wood-pulp "film", it came off the glass instantly as loose fiber particles. When he tried to scrape the AV-ICEL gel that had dried on the glass, he got nowhere. It had dried on to the glass as a continuous coating that stuck to the glass and resisted scraping by the penknife.

Soon thereafter the composition of matter patent was allowed and later issued. Hundreds of millions of pounds of AVICEL in bags and drums have since been sold, and each and every one of these containers was labeled with "U.S. Patent 2978446". Furthermore, the original AVICEL patent case was cited in a U.S. Patent Court Case as an exemplary example and reference. Throughout the life of this original U.S. composition of matter patent—and numbers of foreign equivalent patents—I have never heard of a single valid serious challenge by competitors.

The plant at Newark, DE, was operating at 10% of capacity for months (trying days for me!) before our first truckload order was received from a pharmaceutical company. I had given my standard lecture about AVICEL to this company's research staff months before and had demonstrated samples of hard tablets made by compacted AVICEL powders. I understand one of the chemists in my audience took this cue and explored AVICEL in tableting, found that it was a superb binder and excipient, that it reduced tablet breakage in high speed tableting machines, etc. Ergo, that first truckload order. Fortunately, we had filed early patent applications on the composition of matter and dozens of uses of AVICEL—including its use in tableting.

The pharmaceutical uses of AVICEL mushroomed throughout the world. Numerous less important uses

were found along the way, and I believe it now is on the threshold of growing into the food and industrial markets, many of which I had prematurely promoted over 25 years ago.

The bottom line is that AVICEL has been the mainstay and a major profitmaker of FMC's Food and Pharmaceutical Division. Three plants: the original one in Newark—expanded several times, a plant in Japan, and an ultramodern plant in Ireland now produce the world's supply of colloidal microcrystalline celluloses.

When first introduced, the price of AVICEL was 53 cents a pound. This was considered a premium product inasmuch as American Viscose was doing very well selling rayon at about 32 cents a pound. I pleaded without success—that the introductory price be much higher. In retrospect, it could have easily commanded an introductory price of at least a dollar a pound. Today, millions of pounds are sold each year at prices of about two dollars and up a pound, depending on the grade. The "Research For Profit" bottom line is in heavy black ink, and future uses for this product which got its start by an accidental observation in a Waring Blendor in 1955 is such that the three major plants producing it should have pleasant "back-order" pressures for many years to come.

Whatever happened to that idea I had to make a better rayon tire cord by seeding viscose solutions? The idea still appeals to me as being technically sound; but, to date, neither I nor anybody else I know of has tried it out!

Product Case History Number Two: AVITENE Microcrystalline Collagen²

After FMC bought out American Viscose in 1963, it closed out their central research department and moved me along with a few others to its Princeton, NJ, research center. I was given encouragement to pursue my enthusiasm for microcrystal polymer science and decided to explore collagen from beef hides in microcrystalline form. My first thought was to produce an ultrapure, very high viscosity gelatin for the photographic industry from this abundant raw material. We succeeded in short order and found we could make photographic films from AVITENE emulsions onetwentieth the thickness of conventional photographic gelatin emulsions. We received a U.S. Patent on this use (composition of matter patents had already been filed), and I still have some of the first photographs of our small Princeton Pilot Plant made by us from crude AVITENE-coated photograph papers.

One morning, I arrived at my Princeton office with a rather deep facial cut from trying to shave too fast because I was—as always—so anxious to get to work, especially on *monday* mornings. When I entered my office, I felt the cut—on which I had placed some tissue paper—and it had not stopped bleeding. My eyes caught a jar of the white collagen flour I had been using to make photographic gelatins. The thought occurred to me as I looked at the jar: "My face is largely collagen. What will happen if I put this form of collagen on the bleeding surface of the cut on my face?"

I applied the white powder, held it against the cut for

⁽²⁾ Battista, Orlando A. U.S. Patent 3628974, 1971.

a little while, pressing it with a finger. I went to the washroom, washed the surface of the cut, and, to my surprise, the cut did not start to rebleed. "Wonderful," I mused. "AVITENE is a hemostat, it doesn't sting, and here's a painless replacement for barbershop styptic pencils or powders."

Once again, my research took a tangential course. I forgot about the photographic gelatin use and put almost all my energy—along with that of several associates who reported to me—into pursuing medical uses of AVITENE microcrystalline collagen. Within weeks, we had AVITENE prostheses (arteries, cartilage, bone, etc.), membranes, sutures, etc. The excitement of making these products with a high potential of humanitarian benefit was nothing short of exhilarating.

But, as was the case with AVICEL and American Viscose (American Viscose had absolutely no involvement in foods and pharmaceuticals per se), FMC had no in-house pharmaceutical or medical expertise at that time. Once again, I had to buck the tide of a minimal fit with management's "five-year plans" as well as carry the bulk of the initiative to keep my budget alive! Fortunately, my boss at the time was FMC's Director of Central Research, one of the finest persons to whom I have ever reported. He did see the potential of the products I was now trying to introduce and gave me much continuous moral and budgetary support from 1964 until I moved to Texas in the beginning of 1971.

But the problem was how to get a pharmaceutical company interested in medical products originated by a chemist in a chemical company that had essentially no expertise with medical products much less one willing to undergo the slow, costly travail involved in even getting the FDA to allow animal and human testing—let alone marketing it.

As was the case with AVICEL, I prepared a formal lecture on AVITENE microcrystalline collagen. One by one, I invited myself to give this lecture before scientists at almost every major pharmaceutical company in the United States. One by one, the decisions proved negative-usually based on a skepticism so rampant, especially among medical directors at the time: Battista is a chemist with no pharmaceutical or medical background and is hardly qualified to champion medical products that will require millions of dollars just to get FDA approval to market the product. (Early on in mid-1964, patent applications were filed covering numerous uses for microcrystalline collagens, including its use as a hemostat. This was long before any animal testing whatsoever had been done by anyone).

My budget had only a few months to go. Because I had as yet been unsuccessful in finding a licensee, I was advised that the chances of additional funds to continue research on microcrystalline collagen were remote.

One day in 1968, a critical, unsolicited telephone call came to me from a friend who had recently joined Alcon Laboratories in Fort Worth, TX.

"We're looking for a swab to soak up blood in cataract surgery made of a bioassimilable material. Cellulose swabs are dangerous because even a microscopic fiber of cellulose cannot be absorbed by the body. Do you have anything to fill this bill?"

I described AVITENE microcrystalline collagen to him. It was a hemostat. It was fully absorbable.

This resulted in an immediate invitation to come to



Figure 2. AVITENE microcrystalline collagen flour.

Fort Worth and talk with the cofounder and Chief Executive Officer of the Company.

During my talk, I described the accidental discovery on that September 1964 morning of the remarkable effectiveness of AVITENE as a hemostat. After I had finished, this Chief Executive Officer approached me and said: "I am much more interested in your AVIT-ENE as an antibleeding agent than in its use as a swab." Within a week, he contacted FMC Corporation, my employer. They were so pleased that I had found a licensee that they licensed all of my patents for medical and cosmetic uses of microcrystalline collagen. If this Chief Executive Officer had not licensed my AVITENE patents, this product would have become buried-like more than 60 of my FMC patents-in records stored away on microfilm. Indeed, it took an additional 8 years and the expenditure of about \$10,000,000 before the first gram of AVITENE was sold. (The NDA was approved in 1976.) But today, used largely in life and death emergencies when all other efforts to stop bleeding fail, AVITENE hemostat collagen has saved and is saving countless lives. It sells for over \$13000 a pound (\$26000000 a ton!) but only a gram or two is needed in many cases (\$30-\$60). Patients who are alive today because of AVITENE have told me how vitally inexpensive this remarkable form of collagen is! It may be found in the operating rooms of every major U.S. and Canadian hospital. A large commercial plant is on stream in Puerto Rico so that AVITENE will serve the world in the immediate years ahead.

AVITENE hemostat is natural beef collagen in a new physical form. It comprises 30- μ m fibrils in a network matrix which serves as a substrate for the *immediate* deposition of antibleeding blood factors when placed on a bleeding surface. It "mimics" naturally synthesized fibrin to effect instant hemostasis until the body is able to synthesize fibrin at the site of the injury and take command of the bleeding. It is assimilated by the body in a matter of 5–6 weeks. Used in life-and-death emergencies, AVITENE hemostat is remarkably effective in stopping bleeding during surgery on the spleen, liver, and pancreas. One of its major medical contributions is that it has reduced, if not eliminated, the removal of injured spleens. Figure 2 is a photograph of AVITENE hemostat in a white fluffy physical form.

In 1974, after the commercial successes of both AV-ICEL microcrystalline cellulose and AVITENE microcrystalline collagen were well assured, I retired early. FMC/Alcon welcomed me to remain on the payroll in a state of "passive animation". They told me they did not expect to commercialize other inventions of mine until the high investment in AVITENE was substantially recovered and large profits began to build up.

Ergo, I started my own research institute and launched an extremely rewarding invention development and licensing career. The results have been uniquely rewarding. I have developed an environment of freedom, security, and mounting creativity never before experienced by me. My formula is consistent with the experimental facts of the two major case histories I have already described:

(1) Each idea I conceive is reduced to practice in my own research institute.

(2) U.S. Patents are filed just as soon as sufficient data to support a patent application are obtained. Unlike the situation that existed while I was employed by AVISCO/FMC for 33 years—when all of my patents were owned 100% by them—all of my patents received or pending since I retired are mine, owned wholly by me until I license them to appropriate licensees.

(3) The exclusive world rights to the invention are

then offered by me-whenever at all possible-directly to the Chief Executive Officer of one or two companies who should have the highest motivation to champion the invention and bring it to the marketplace.

(4) My offers are overgenerous and hard to turn down: In return for *exclusive* and full ownerhsip of the invention, all I ask is that I be retained as their consultant in support of the invention for a period of 2 years. This provision is to let me help them during the most difficult gestation period of a new invention.

(5) At the end of 2 years, they need not renew my consultancy agreement unless they wish to do so.

(6) A modest royalty must be paid only if and when the invention reaches commercial success—a term no licensee objects to because there is no obligation to pay royalties before and until profits are in hand.

Since my retirement, I have concluded many successful consulting-licensing agreements. These include agreements with Essilor (Paris); Capsugel (Basel); L'-Oréal (Paris); Hormel; Smith, Kline, Beckman; Tandycrafts; and several other equally prestigious clients.

McGraw-Hill Book Company invited me to write a book about my career in pioneering the utility of polymer microcrystals.³ This treatise tells it all for the first 20 years!

As a retired scientist, I have tasted the rewards of "Research For Profit" and the bottom line for The O. A. Battista Research Institute is in heavy black ink. Not surprisingly.

(3) Battista, O. A. "Microcrystal Polymer Science"; McGraw-Hill: New York, 1975.

Kinetics and Mechanism of Metal Chelation Processes via **Solvent Extraction Techniques**

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Received May 3, 1983 (Revised Manuscript Received September 22, 1983)

Solvent extraction separation techniques provide simple and effective means for improvement of analytical methods by enhancement of sensitivity (by preconcentration) and selectivity (by interference removal).¹ Extraction techniques have proven of great service in metals analysis, particularly because a wide variety of extractants are available. In most instances the extent of extraction is independent of the total concentration of the metal, making it possible to describe optimal experimental conditions that are applicable both to extremes of "weightless" trace levels as well as macro levels that can be encountered in hydrometallurgical process technology.

Until about 40 years ago, kinetic aspects of solvent extraction were largely neglected. In most analytical extraction procedures, conditions favoring extraction

Henry Freiser was born in New York City in 1920. He received his Ph.D. from Duke University and, after a year each at North Dakota State College and Mellon Institute of Industrial Research, became Associate Professor at the University of Pittsburgh. He moved to the University of Arizona in 1958 where he was for 10 years Head of the Chemistry Department and remains as Professor of Chemistry and Chairman of the Strategic Metals Recovery Research Facility today. Professor Freiser received from the American Chemical Society in 1978 the Fisher Award in Analytical Chemistry.

are usually sufficiently far from equilibrium so that vigorous shaking of the two phases serves to give essentially complete (>95%) extraction within several minutes. Some early work revealed qualitative dependence of extraction rates on such chemical variables as the nature of the organic solvent employed, pH, and extractant concentration.²⁻⁴

In 1962, Carl Honaker and I, intending originally to determine the equilibrium formation constant of the zinc chelate of diphenylthiocarbazone (dithizone) via solvent extraction, observed an unexpectedly slow attainment of extraction equilibrium.⁵ The pink color in the aqueous phase, which indicated the formation of the positively charged 1:1 zinc chelate, persisted despite vigorous shaking for as long as 20 min. This observation launched a series of detailed systematic kinetic studies of extraction processes involving metal chelate forma-

(1) Morrison, G. H.; Freiser, H. "Solvent Extraction in Analytical (1) Moliney, Wiley: New York, 1957. (2) Walkley, A. Proc. Aust. Chem. Inst. 1942, 9, 29.

- (2) Irving, H.; Williams, R. J. P. J. Chem. Soc. 1949, 1841.
 (4) Irving, H.; Andrew, G.; Risdon, E. J. J. Chem. Soc. 1949, 541.
- (5) Honaker, C. B., Freiser, H. J. Phys. Chem. 1962, 66, 127.

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