CHEMICAL HERITAGE FOUNDATION

JOHN H. SINFELT

Transcript of an Interview
Conducted by

James J. Bohning

in

Annandale, New Jersey

on

21 February 1995

(With Subsequent Corrections and Additions)

ACKNOWLEDGEMENT

This oral history is one in a series initiated by the Chemical Heritage Foundation on behalf of the Society of Chemical Industry (American Section). The series documents the personal perspectives of Perkin and the Chemical Industry Award recipients and records the human dimensions of the growth of the chemical sciences and chemical process industries during the twentieth century.

This project is made possible through the generosity of Society of Chemical Industry member companies.

THE CHEMICAL HERITAGE FOUNDATION
Oral History Program

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John H. Sinfelt

(Date) 10/25/95

(Revised 17 March 1993)

John H. Sinfelt, interview by James J. Bohning at Exxon Engineering and Research Company, Annandale, New Jersey, 21 February 1995 (Philadelphia: Chemical Heritage Foundation, Oral History Transcript # 0134).

Chemical Heritage Foundation
Oral History Program
315 Chestnut Street
Philadelphia, Pennsylvania 19106

JOHN H. SINFELT

1931	Born in Munson, Pennsylvania, on February 18

Education

1951	B.S., chemical engineering, Pennsylvania State University
1953	M.S., chemical engineering, University of Illinois
1954	Ph.D., chemical engineering, University of Illinois

Professional Experience

	Exxon Research and Engineering Company
1954-1957	Research Engineer
1957-1962	Group Leader
1962-1968	Research Associate
1968-1972	Senior Research Associate
1972-1979	Scientific Advisor
1979-1996	Senior Scientific Advisor
1996-	Senior Scientific Advisor Emeritus

Honors

1971	Alpha Chi Sigma Award in Chemical Engineering Research, American Institute of Chemical Engineers
1973	Paul H. Emmett Award in Fundamental Catalysis, Catalysis Society
1975	Elected to National Academy of Engineering
1975	Professional Progress Award for Outstanding Progress in Chemical Engineering, American Institute of Chemical Engineers
1976	American Chemical Society Award in Petroleum Chemistry
1977	Dickson Prize in Science and Engineering, Carnegie-Mellon University
1978	American Physical Society International Prize for New Materials
1979	Elected to National Academy of Sciences
1979	President’s National Medal of Science
1980	Elected a Fellow of the American Academy of Arts and Sciences
1981	Honorary D.Sc., University of Illinois
1981	Chemical Pioneer Award, American Institute of Chemists

1984	Perkin Medal in Chemistry, Society of Chemical Industry, American Section
1984	Gold Medal in Chemistry, American Institute of Chemists
1985	Distinguished Alumnus Award, Pennsylvania State University
1986	E.V. Murphree Award in Industrial and Engineering Chemistry, American Chemical Society
1991	Elected to New Jersey Inventors Hall of Fame
1994	Elected to American Philosophical Society

ABSTRACT

In this interview, Dr. Sinfelt recalls his childhood during the Depression, his early education, and his interest in mathematics. He then moves on to the awakening of his interest in science, first at Lycoming College, and then at Pennsylvania State University, where he decided to continue in graduate work at the University of Illinois. He describes the University's chemistry department under Roger Adams and his own studies under Harry Drickamer. Moving on to the Exxon Research and Engineering Company, Sinfelt recounts how post-World War II demand for increased production of high octane gasoline led to two developments—the choice of fixed-bed platinum hydroforming over fluid-bed hydroforming, and the choice of precious rather than non-precious metal catalysts. Sinfelt describes how his research on catalytic reaction kinetics meshed with Exxon's increased emphasis on basic research and how this led to his discovery of bimetallic clusters and the success of the platinum-iridium catalyst. He describes how Exxon's commercial use of this catalyst along with Chevron's platinum-rhenium catalyst led to the development of lead-free gasoline and decreased carbon monoxide emissions. Sinfelt next discusses current environmental concerns about this system. He then describes his research on the crystalline structure of bimetallic catalysts, which led to the characterization of small metallic particles and metallic adsorption. Finally, Sinfelt discusses Exxon's attitude toward research; his book *Bimetallic Catalysts*, in which he theorizes on the relationship between an element's catalytic activity and its place in the periodic table; and his views on innovation, teamwork, and the future of research in the chemical industry.

INTERVIEWER

James J. Bohning is Professor of Chemistry Emeritus at Wilkes University, where he was a faculty member from 1959 to 1990. He served there as chemistry department chair from 1970 to 1986 and environmental science department chair from 1987 to 1990. He was chair of the American Chemical Society's Division of the History of Chemistry in 1986, received the Division's outstanding paper award in 1989, and presented more than twenty-five papers before the Division at national meetings of the Society. He has been on the advisory committee of the Society's National Historic Chemical Landmarks committee since its inception in 1992. He developed the oral history program of the Chemical Heritage Foundation beginning in 1985, and was the Foundation's Director of Oral History from 1990 to 1995. He currently writes for the American Chemical Society News Service.

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INTERVIEWER: James J. Bohning

LOCATION: Exxon Engineering and Research Company
Annandale, New Jersey

DATE: 21 February 1995

BOHNING: Dr. Sinfelt, I know you were born on February 18, 1931, in Munson, Pennsylvania. Could you tell me something about your father and mother and your family background?

SINFELT: Yes. Munson is in central Pennsylvania. It's a small village in a very rural area about thirty miles from State College. Bellefonte, not very far away, is the county seat of Centre County.

My father worked in coal mines in the bituminous coal region of central Pennsylvania. He was a mine foreman and had worked in this area all his life, so I was very familiar with the coal mining tradition of Pennsylvania.

I went to school in a two-room schoolhouse in Munson. We had two ladies who taught all eight grades. One lady taught grades one through four in the front room, while the other lady taught grades five through eight in the rear room. In each room we had four rows of seats, one for each grade. This was a very interesting experience for me, and one of the things that's probably had a lasting effect on my life. It made a very strong impression on me.

The two women were extremely dedicated people. They really were interested in teaching. This was in the Depression years in the thirties, and the lady who taught the first four grades lived about three miles away from the schoolhouse and walked to school every morning through the woods. Even all through the winter, she was always there. I've always remembered her dedication. The other lady, who taught grades five through eight, lived much closer to the school. Both these teachers were extremely good, and I think they rank with the best teachers I ever had anywhere, and that goes for high school, college, and graduate school. I got a superb education in the first eight grades in a simple two-room schoolhouse—no fancy facilities whatsover, just pencil, paper, a blackboard and two ladies who really were interested in teaching us something.

BOHNING: What kinds of subjects were stressed there? Mathematics or reading?

SINFELT: The very basics. I remember, in particular, English. I've always been happy that I had these ladies. They taught us how to diagram sentences. I don't even know whether people worry about this anymore. We knew what a subject was, what a predicate was, a noun, a verb, a preposition, and so on. That was really strongly embedded in us. Apart from the basic reading, writing and arithmetic, we also got a strong sense of history. They embedded in us a strong idea of when things happened in our history. We had a sense of when the Revolutionary War took place, when the Civil War took place, and things of this sort. We had a pretty good knowledge of who our presidents had been over the years, and who some of the outstanding people had been in the U.S. Senate and Congress.

Even in things like poetry, we learned quite a bit. As a matter of fact, when I was in grade school I learned a lot of poems. We had to memorize and recite them. I still have some of them in my memory to this day. [laughter] So while they emphasized the basics, they also were looking out for cultural subjects that one might not ordinarily associate with education at the grade-school level.

BOHNING: You have one brother. You were the oldest of three children, and I was struck by the fact that both your parents were born in Munson as well. Your family goes back there a long way.

Your brother also had a Ph.D., but not in the sciences.

SINFELT: That's true. He went in a totally different direction from me. All of his life, he has been more interested in the arts. He wound up as a professor at Indiana University of Pennsylvania, which originated as a state teacher's college, Indiana State Teacher's College. Today it's quite a big state university with ten or fifteen thousand students. He started out by teaching high school when he finished college. He went to Penn State [Pennsylvania State University] and studied English literature and languages, which were a strong interest of his. When he served in the armed forces, he was overseas and had a lot of experience with languages. When he was discharged from the service, he accepted a position at what was then Indiana State Teacher's College, located in Indiana, Pennsylvania, which is known best as the birthplace of Jimmy Stewart, the great movie actor.

BOHNING: Just as an aside, that has meaning for our generation, but I'm sure the younger generation would not have any appreciation of that at all.

SINFELT: That's right. Well, Jimmy Stewart's father had a hardware store on the main street in Indiana, Pennsylvania, and I can remember that from the time when I was in my teens. I don't think it exists there anymore, but for years there was Stewart's Hardware. As I remember, not too long ago Jimmy Stewart had a celebration of some sort that took place there in Indiana, Pennsylvania.

BOHNING: Did the Depression have any effect on your family?

SINFELT: Yes. It affected just about everybody and there were periods when things were tough for us, but most of the time we were fortunate. My father generally was working for most of the time through the Depression.

You could see that everybody was pretty much in the same boat. Kids did not have lots of toys, but they somehow amused themselves in a variety of ways, and I don't remember it as being a particularly bad time, at least insofar as the kids were concerned. As a matter of fact, I think some of the happiest years of my life were as a child during the Depression. The adults might have looked at it a little bit differently, but from the standpoint of kids growing up, from what we knew, we had it pretty good. We had a lot of fun. We played baseball, frequently using old discarded balls which we covered with tape. We also played lots of other games, and generally had a good time.

BOHNING: So in spite of what was happening at the time economically, your childhood was a good time.

SINFELT: Yes, it was a very good time, a very happy time. But as I went from grade school into my teens, I was very aware, and my parents made me very much aware of the fact that I should be very serious about school, try to get as much out of it as possible, and be thinking at a relatively early age about what I was going to be doing when I got out of school. I started thinking quite early in terms of trying to prepare myself in a way so that I would get into some form of employment which would be very realistic and very practical.

BOHNING: Your family moved to Philipsburg in 1943.

SINFELT: That's right, and one of the reasons for doing this was that there was a high school located in Philipsburg, Pennsylvania. Munson was about ten miles away from Philipsburg, and while there were two other high schools in the area, we moved to Philipsburg so I would have the opportunity to be close to Philipsburg High School.

BOHNING: Did your father still work in the mines then?

SINFELT: Yes, and for about fifteen years after that until he retired. My father passed away about twenty years ago, but my mother still lives in Philipsburg to this day. I went to high school there and graduated from high school in 1947.

BOHNING: You had a sister who died at an early age, which was around this time.

SINFELT: In the spring of 1942, that's right. My sister Norma was killed by a truck. It was in the very early days of World War II, and it was a very difficult emotional experience for us and something that we didn't get over for a long time.

BOHNING: The war was on. You graduated from high school in 1947, so your high-school career was basically during World War II.

SINFELT: That's correct. I can remember Pearl Harbor. In 1941 we were still living in Munson, and I was in seventh grade. We lived in a house that didn't have central heating. That's another thing that was very typical of the Depression years. After we went to bed the fire went out during the evening and had to be started again in the morning. Well, we woke up on December 7, 1941, and my dad had turned the radio on and we heard this report about the attack on Pearl Harbor. I can remember that to this day. A year or a year and a half after that, we moved to Philipsburg and I began high school, so I was going to high school during World War II, and I was halfway through high school when World War II ended. I can remember in August 1945, when the Japanese part of World War II finally ended, the tremendous celebration that we had in Philipsburg. Everybody was out on the streets dancing. It was quite an event.

BOHNING: Your major interest in high school, as I understand it, was mathematics.

SINFELT: Yes. That probably goes all the way back to grade school. I always enjoyed a quantitative approach to things. From the time I was a young child, I was always interested in numbers. I was very much interested in quantifying things. My father had a great deal to do with this. Before I even started school, my dad used to sit down with me and, just for amusement, we would do arithmetical kinds of things. As a matter of fact, at a very early age, I was learning about algebra, way before I would ever be exposed to this in a normal school curriculum—I think because my dad saw that I enjoyed the arithmetical games we indulged in and thought, "Maybe I'll give it a crack and see how he might do with something like this." So I was studying algebra at a very early age with my dad just for fun.

That's something that I think is very important about science, although I didn't think much about it in terms of science at that time. It's very important to have fun. Today I sometimes think that people believe there is something wrong about having fun with what you're doing in making a living. I don't think that at all. As a matter of fact, unless you're having fun in science, it's not likely you're going to do very good science.

BOHNING: In an interview we have with Carl Marvel, he said, "I have talked people out of going into chemistry because it wasn't fun for them" (1). He said if you're going to do it, you should have fun—exactly what you're saying. He actually discouraged students because they didn't have fun at it.

You didn't get your first chemistry course, though, until your last year in high school.

SINFELT: That's right. My interest in chemistry didn't evolve until fairly late. I didn't have a chemistry set when I was in grade school, and I don't think anybody even knew what they were, where I came from. In high school our science program was set up so that we took general science as freshmen, biology as sophomores, physics as juniors, and chemistry as seniors. That was my first real exposure to chemistry, and it wasn't very extensive. In high school I still was very excited about mathematics. I always enjoyed algebra, geometry, trigonometry, the whole gamut of mathematics courses in high school.

By the time I finished high school, I was pretty sure I wanted to do something that had a good component of mathematics in it, in the sense that I was going to approach something quantitatively. I didn't have much knowledge about or awareness of chemistry until I was a senior, but by that time I had thought about things in a practical vein for quite some time and I thought about my choice of career. I thought, "I might enjoy being a mathematician." I think I would have really enjoyed that, but I said to myself, "What kind of a job will I get?" I didn't have an idea what mathematicians did. I could see that I might be able to teach mathematics, but that was about all I could see that one might do. I didn't have much perspective beyond that.

Then I thought, "Maybe it would be more practical to try to get into something where mathematics or quantitative thinking is an important component." I began to think that some kind of profession where I could get tied up with industry might be a worthwhile thing to pursue, where I was probably going to have a high likelihood of being employed when I was finished. I began to think about chemical companies, oil companies, et cetera. I could see that engineers and chemists were needed to design plants and so on. That's the way I thought about the whole thing, so I figured a good compromise might be something like chemical engineering. I didn't even know what chemical engineering was really, but I talked to some of the ex-GIs who were coming back from the war.

That was another important factor in helping me make up my mind. There was a whole flood of ex-GIs coming back around 1945 and 1946, and some of them were coming back to finish high school while I was still a high school student, so I began to talk to these fellows. They were already thinking in very practical terms; they were four or five or six years older than I was, and they were all talking about jobs, so it became something very crystal clear to me, that these people were talking about reality. That had a big effect on me. I heard people talking about going into engineering and working in places like Standard Oil, DuPont, or Monsanto, and I thought, "Chemical engineering might be a pretty good bet, because there's going to be chemistry in that and a quantitative approach to problems. Let's give that a crack." That's how I wound up making a decision to study chemical engineering, and I enrolled at Penn State when I graduated from high school.

BOHNING: Had you thought about anywhere else but Penn State? It was virtually in your backyard.

SINFELT: That was pretty much it. Maybe I thought about some other schools close by, I don't remember. I think my whole family had the idea that I would go to Penn State.

BOHNING: But as you told me earlier, you ended up your first year at Lycoming College.

SINFELT: That's correct. When I graduated from high school just shortly after World War II, there were many GIs coming back, some to finish high school, some to enroll in college. There was a flood of these people on the campus. There just wasn't enough room to handle all the freshmen who wanted to come on campus, so Penn State farmed a lot of their freshmen out to smaller schools throughout Pennsylvania. I wound up at one of these places, Lycoming College in Williamsport, Pennsylvania. For the most part it was a small liberal arts school, but it was a very good experience for me. I had come from a rural area, and this was not such a large place to begin college. It was the kind of place where I could know just about everybody; almost everybody there was in the same boat. The classes were relatively small, so it was a good way to start. I found my year at Lycoming to be very satisfying for me.

BOHNING: What courses did you take in that year?

SINFELT: There was the traditional freshman chemistry course, a course in college algebra and analytic geometry, and an English composition course which was pretty standard for students at the time. I took a language, German, and I have never been sorry for that. I had an excellent German teacher, and I remember her to this day, a lady named Charlotte Finkenthal. She was an outstanding teacher. She used to have a German club, and she arranged for some of us to go down and appear on a radio program in Williamsport. I don't know whether we were singing some German songs or what; I don't remember the details, but I remember that course quite well.

Lycoming College had formerly been Dickinson Seminary. There was a strong church affiliation, and one course which was strongly required of everybody was a freshman course in religion. I took that and enjoyed it because I was exposed to some things that I might never have been exposed to, and probably read more of the Bible than I might ever have done if I hadn't taken that course.

BOHNING: What was your chemistry experience like?

SINFELT: Well, we didn't have much in the way of facilities. The laboratory was small, but we got a good grounding in freshman chemistry. We used a textbook that was written by a man named Herman [T.] Briscoe, who taught at the University of Indiana, and it's an excellent textbook (2). As a matter of fact, I still have it. The book must be fifty years old now, and I still often refer to it. I find it useful today, and very frequently I open it up and look at some things, because it's a very different kind of textbook from what I imagine freshman chemistry texts are today. There is a lot of descriptive chemistry, and a lot of discussion of chemical processes conducted in chemical plants as well as the laboratory, and also discussion of some of the modern aspects of chemistry as of the time the book was written. There are good discussions of modern chemistry prior to World War II and of some of the developments in modern physics, things like radioactivity and isotopes. It's a very excellent text. As a matter of fact, if anybody has the opportunity to open up Briscoe's college chemistry book, I think that it might be a good experience.

BOHNING: Did you have qualitative analysis in the laboratory?

SINFELT: We started that in the second half of my freshman year, and that was the first sense of something exciting in chemistry—where we were going into the laboratory and essentially becoming acquainted with the spirit of the research process. The idea of going in and getting an unknown substance and trying to find out what was in it was a new and exciting experience.

BOHNING: Did you know that you would only be one year at Lycoming and then you would head back to Penn State?

SINFELT: That's what they told us, and they came through with their promise. [laughter]

[END OF TAPE, SIDE 1]

BOHNING: In 1948, you were back at Penn State as a chemical engineering major.

SINFELT: That's right. I started on the main campus in State College, Pennsylvania, in the fall of 1948. My first chemistry course there was quantitative analysis. I don't even know how much this type of thing is taught anymore, but that is a very good experience for a person. It's very good discipline for the chemist, because that's the way a lot of chemistry really evolved, by the kind of laboratory work conducted on problems in quantitative analysis. All of it was wet chemistry in those days. We didn't use any fancy instruments. It was all either gravimetric or volumetric analysis, lots of titrations, a lot of use of the analytical balance, et cetera. We became very familiar there with the quantitative aspects of chemistry.

Since I was interested in quantitative things from my earliest memories as a grade school student or even before grade school, quantitative analysis appealed to me. It was a lot of work. It was hard work. In the laboratory part we had two full afternoon sessions a week, and that wasn't enough to do all the work we had to do in this course. We had an enormous number of unknowns to work through, and the course consumed much of our time. There was that course, and then I had a course in physics and my first course in differential calculus that semester. I also had a course in history; it wasn't an elective course, but was one of the courses that was required to round out a student. I had a course in analytical mechanics. Since chemical engineers were part of an engineering program as well as a chemistry program, we had a fair component of strictly engineering courses.

That was also the year I was first exposed to a chemical engineering course, industrial stoichiometry, which is concerned with mass and energy balances, the bread and butter of chemical engineers in their everyday lives. One of the things I liked very much about being a chemical engineering student at Penn State was that chemical engineering was part of the School of Chemistry and Physics. It was not part of the College of Engineering. This was to my liking, because by the time I was on the campus at Penn State, I knew I liked chemistry a great deal, and as a matter of fact, I already was thinking, "Should I be a chemist rather than a chemical engineer?" As it has turned out in later years of my career, I've been more of a chemist than an engineer for a long time. I was already thinking about it at the time I was a sophomore.

We took all the basic core chemistry courses that the chemistry majors took, in addition to the engineering courses that were required. For example, in organic chemistry I took the full component of courses, and I took the full component of physical chemistry courses. Physical chemistry, particularly, was a real pleasure for me, because it emphasized quantitative aspects of chemical phenomena throughout. We had four semesters of physical chemistry at Penn State, so the program was as heavily loaded with physical chemistry as any chemistry program in the country at the undergraduate level. We used an old book that the students all grumbled about, but it was an excellent book for learning by doing. It was a book by [Arthur A.] Noyes and [Miles S.] Sherrill called *A Course of Study in Chemical Principles*, and the first edition went all the way back to around 1920 (3). It's so old that Linus [C.] Pauling used it when he was a graduate student at Cal Tech. [laughter] Penn State was one of the few places in the country where the book was still used. It was not a descriptive book, it was problem-oriented. The whole book was a set of problems, and we learned basic chemical principles by working our way through problems. This is a sound way to learn something about the various principles of chemistry, by actually working a problem, and the authors were very skillful in the way they set up the book. Although some people thought Noyes and Sherrill was very difficult, I liked it, so I was very pleased.

By enrolling in the chemical engineering curriculum at Penn State, I got just about everything I wanted. I got the mathematical component; I was also introduced to physical chemistry, and I could see by the time I completed four semesters of physical chemistry that this was what I liked. As a matter of fact, I thought seriously at the time about the possibility of going over into physical chemistry as a graduate student. But as it worked out, I didn't do that. I went to another school where chemical engineering was part of the chemistry department and where I was able to develop a thesis which was heavily physical chemistry oriented, so I got just about everything I wanted.

BOHNING: Was Frank C. Whitmore at Penn State then?

SINFELT: He died the first year that I was in college. He died in 1947, the year that I went to Lycoming, so by the time I got on campus, he had already passed away. That was unfortunate, because he was a very outstanding figure in organic chemistry and a very prominent figure on the campus of what was then Pennsylvania State College, known today as Pennsylvania State University.

BOHNING: What about Gilbert Pond? Was he there then?

SINFELT: No. I remember Pond Laboratory because that's where I took my physical chemistry laboratory courses, but I think Pond was gone long before the time I was there.

Talking about the setup at Penn State, chemical engineering and chemistry were part of the School of Chemistry and Physics, so there was a very close tie-in between all of these physical sciences and chemical engineering. That was just exactly what appealed to me and what I really wanted. Although I didn't have a real good idea when I was a senior in high school about what I wanted to do, I had some feel for the kind of activity I would like to be involved in. I made a very fortunate choice of chemical engineering without having a very good appreciation of what it entailed, since I got just about everything that I had hoped to get.

BOHNING: Who were some of the professors then in both chemistry and chemical engineering whom you had?

SINFELT: In chemical engineering, the department head was Donald S. Cryder. He had been a graduate student at MIT and had taken his doctorate there back in the twenties, so he was part of that very famous group of people from MIT. There was another man named Arthur Rose, who later became involved in an entrepreneurial activity of his own that was known as the Applied Science Laboratories in State College. He was a professor of chemical engineering and involved in this industrial activity. Several of Rose's students later became involved in that activity and then went out on their own in another venture in the State College area. Another well known chemical engineer was Michael [R.] Cannon. He was the man who was involved with the development of the so-called Cannon packing for distillation columns. He, too, had a venture of his own going in State College, in addition to being professor of chemical engineering. His company manufactured the Cannon-Fenske viscometers and the Cannon packing for distillation columns. There was also Floyd [L.] Carnahan, who was very well known as a teacher of chemical engineering. Just about everybody who took the basic chemical engineering courses in fluid mechanics, heat transfer, and unit operations had Floyd Carnahan for their teacher.

In chemistry I remember some people very well. In physical chemistry there were A. Witt Hutchison and Ralph [P.] Seward, two people I remember very well. I learned a great deal from both these people. They both stimulated a strong interest in physical chemistry in me. In organic chemistry there was a man named J. Harris Olewine, who was sort of a legend in Penn State. Not that everybody liked him all that much, [laughter] but he was a legend from the standpoint of giving a really tough organic chemistry course to undergraduates. I guess he got great joy out of making it as difficult as he possibly could, [laughter] but he was an outstanding lecturer. His lectures were crystal clear, and we learned classical organic chemistry very well.

BOHNING: Did you do any independent research as an undergraduate?

SINFELT: No. The main problem with the chemical engineering curriculum in those days was the number of courses. The program was so loaded up there was just no time for anything else. There was so much laboratory work in the curriculum. Typically the organic chemistry courses would have a couple of complete afternoons taken up with lab work. I already mentioned the quantitative analysis course in that respect. Physical chemistry included a laboratory period for at least a whole afternoon once a week. In chemical engineering, there were unit operations laboratories two times a week for half a day each. There were also laboratories in the electrical engineering courses we took. We had laboratories in a metallurgy course that I had to take. There were laboratory sections in all the physics courses. The students were very busy. We packed in all the chemistry that was part of the chemistry core, and in addition many engineering courses. Then on top of that there were courses we were asked to take in history, economics, business law, report writing, and specialized German for scientists. There just wasn't much time. Our week was taken up almost entirely in classes or laboratory sessions. The only time we had for study was in the evenings, and there wasn't much opportunity for independent research.

BOHNING: As you were approaching the end of your Penn State career, what were you thinking about for the future?

SINFELT: When I started at Penn State, I didn't have any idea about going to graduate school. I didn't even have an appreciation of what was involved in graduate study or why anybody would go beyond a four-year college course. But as I came close to the end of my college career, I began thinking about possibly going to work in an industrial job, so I took some summer jobs. I did summer work with the Koppers Company in Pennsylvania. They had a facility in Kobuta, Pennsylvania, which is in the Pittsburgh area. One summer I wound up working in a chemical plant in the little town of Petrolia, Pennsylvania, out near Butler and Kittanning. It was an old coal tar products plant, and I was doing pilot plant work there. Koppers had some space there where they set up a pilot plant, and I spent a summer there doing a lot of shift work and got a feel for what working in the chemical industry was like.

It was my first exposure to process development work, and it was a very good experience for me because of the aspect of responsibility for that type of an operation. It was an operation where we had to be aware of what we were doing at all times, otherwise we might have run into some serious problems. We were handling such nasty things as sulfuric acid, sulfur trioxide, benzene—just lots of things that would make anybody shudder today. [laughter] The pilot plant was fairly complex. I'm not going to get into too much detail here. I don't know what Koppers ever did with the results of the work, but it involved a number of types of operations. There was a reactor, a crystallizer, and a centrifuge, so I became familiar in a very real way with some of the kinds of things that go on in the chemical industry. It was a very valuable experience for me. It taught me a lot. I learned through a few mistakes, as I'm sure everybody does, but it was a hazardous assignment and sometimes I wondered, "Why did they ever trust this sort of thing to a student?" [laughter]

BOHNING: Did that experience reinforce your determination to stay in the chemical industry?

SINFELT: Yes. It was hard work, but it was a very valuable and positive experience for me. It was my first experience with the development part of R&D, and I became very conscious about the way a chemical engineer has to think about problems in the chemical industry. I began to appreciate the importance of a material balance, in a very real way. Because we're making product, we're very much interested in how much we're recovering; we become very conscious about how precisely we want to determine yields, because yields translate into dollars, and so on. Then after I completed that summer, I went back to my senior year, so it gave me a much better appreciation of at least the chemical engineering part of my senior year at Penn State. That was a very valuable experience.

By that time, I knew that I liked physical chemistry quite strongly, and I thought I might like to go to graduate school. I finally became aware of what graduate school was all about, that I would have an opportunity to do a research problem and to delve in some depth into it. I probably could have gone into a physical chemistry program in graduate school almost anywhere, because I had completed the basic courses that would be required, but I decided to enter the graduate program in chemical engineering at the University of Illinois. I decided to do that mainly because chemical engineering was in the Chemistry Department at Illinois. Also, they offered me a very handsome assistantship. With the assistantship, I could go all the way through on my own. I wouldn't have to depend on my parents for any financial support.

One thing I like to tell people about my experience at Penn State is the total cost of my four years there shortly after World War II. My entire college education cost me about three thousand dollars, for <u>everything</u>—tuition, board, room, books, fees, you name it. That was it. That was a real bargain. The assistantship at Illinois, which covered everything I would need in graduate school, was another bargain.

There was also the fact that Illinois had a very strong chemistry department. In those days it was chaired by Roger Adams, probably as well known a figure as there was in the whole world in organic chemistry. Chemical engineering was part of this department. It was one division, just like physical chemistry, organic, analytical, or inorganic. When students started graduate work at Illinois, the first thing they did was take exams. Before they could take any courses, they had to pass qualifying exams in inorganic, organic, and physical chemistry, and a couple of chemical engineering exams as well. This was to determine whether students needed to have any makeup work before they got into the full program. The closeness of chemistry and chemical engineering there was very important to me, and it gave me the opportunity to take courses in physical chemistry and mathematics. In fact, I took more courses in physical chemistry and mathematics together than I did in chemical engineering.

Plus, I was able to do my thesis with an individual who was very much oriented toward physical chemistry and chemical physics. That was Harry Drickamer, and he probably has had as important an effect on me as anybody I've been involved with in my whole career.

BOHNING: In what way?

SINFELT: First of all, he was the one who really introduced me to research. I did my Ph.D. thesis with him. We had very similar interests. Harry Drickamer had been trained as a chemical engineer and worked for a couple of years in the early forties with the Pan-American Refining Corporation in Texas City, Texas. He was a superb engineer, and he made some very notable contributions in chemical engineering, writing some classical papers in the subject, but he decided that was not what he wanted to do. He decided that he would like to be more deeply involved with chemistry or physics. He took a teaching position in chemical engineering at Illinois for the same reason that I took an assistantship there—namely that it was part of the Chemistry Department, and he already knew he wanted to work in the area of physical chemistry.

He started as an assistant professor there in 1946, just after the war, very close to the same time I was starting at Penn State. He developed a program which was very heavily physical chemistry oriented, and he shortly became a professor of physical chemistry as well as of chemical engineering. The appealing thing about Harry Drickamer's program at Illinois was that he was a professor in both physical chemistry and chemical engineering, so he had a mixture of graduate students coming from chemical engineering and chemistry.

It wasn't very long after that that he started to have graduate students trickling over from the physics department. It was a very early example of an interdisciplinary research program in a university, where there were chemists, physicists and engineers all involved in research problems, all doing similar kinds of research. It had a very useful purpose for somebody like myself who later wound up in industry, because it tended to emphasize research problems rather than disciplines. It tended to develop a philosophy, on the part of the student, of concentrating on solving a research problem rather than worrying about whether it was strictly physical or organic chemistry, physics, engineering, or what have you. We learned what we had to learn to work the problem, which is about the best kind of preparation I can think of for an industrial research career.

[END OF TAPE, SIDE 2]

BOHNING: Did you have any help in selecting Illinois?

SINFELT: Yes.

BOHNING: Well, first of all, were there other schools you applied to? Did you have any guidance from your faculty at Penn State?

SINFELT: Yes, I did have guidance from my faculty at Penn State. For some years there had been an exchange program going on between Illinois and Penn State, for chemical engineering students. [H.] Fraser Johnstone was the head of the Department of Chemical Engineering at Illinois. Cryder would send some students out to Illinois, and Johnstone would send students from Illinois to Penn State. A tradition had been built up over a number of years, so there was sort of a trade between the two places, and it had worked out quite well. I did know from Professor Cryder about the very successful interchange of students between Illinois and Penn State.

Cryder had a pretty good feel for how the students did at Illinois, and he knew something about their experiences finding positions after they left Illinois. That was all positive. There was a totally positive picture about Illinois from what I could learn from the Penn State faculty. I had no concerns at all about that, and I could then act upon the things that were important to me—chemical engineering being part of chemistry, and the opportunity to develop my interests in physical chemistry and mathematics.

I had considered some other schools, yes. As I said, I had even been thinking about the possibility of going into a physical chemistry progam somewhere. But I chose Illinois for the two reasons I mentioned—the very good assistantship that eased the financial problem as well as it would have been eased anywhere else, and the fact that chemical engineering was part of the Chemistry Department. I did not know Harry Drickamer at the time I made the decision to go there, but his being there made it very clear to me shortly after I got there that I'd made the right decision.

BOHNING: What kind of a person was Drickamer to work for?

SINFELT: Tough. [laughter] He put the fear of God into you. He was the type of person who expected a lot. He wanted us in the laboratory, working hard, but he was also the type of person who supported you strongly. He would always go to bat for us if we showed that we were really interested and really wanted to get something done. He always tried to encourage students to focus on a research problem, rather than thinking of disciplines, and to get background in other fields. "Don't think of yourself as a very narrow entity. If you're a chemical engineer, try to learn something about allied fields. Try to find a niche for yourself. Try to develop expertise in some aspects of problems that might involve other disciplines." I remember that very strongly. He was always telling us that we should learn something about solid-state physics. If we were interested in physical chemistry, we shouldn't be thinking only in terms of gases and liquids. In those days, physical chemists tended not to be identified with the solid state. He said, "Why not learn some solid-state physics? There are lots of things in solid-state physics that relate to physical chemistry, and vice versa." These views told us a lot about Drickamer's way of thinking about science and had a very strong influence on me.

My Ph.D. problem involved diffusion in liquids, and I was measuring diffusion coefficients with radioactive tracers. This was something very new. This was not long after the end of World War II and some radioactive tracers were available, things like sulfur-35 [^{35}S] in particular, and I actually used sulfur-35. I was doing a thesis where I was measuring diffusion coefficients of sulfur-35 in organic liquids. In particular, I was interested in measuring the diffusion across an interface between two liquid phases, and I wanted to find out whether there was any resistance to diffusion right at the interface itself. In other words, in addition to having the resistance to diffusion in the individual phases, is there a barrier at the interface to the diffusion process? This was a very important fundamental problem in the design of all kinds of separations equipment, whether we talk about an absorption tower or an extraction tower. In the design of equipment like that, for many years people made the assumption that equilibrium was established at the interface and that there was no resistance to the transport at the interface itself. But for all of the time that this assumption had been used in the design of equipment in the chemical industry, nobody had ever investigated how molecular properties might influence the existence of a resistance in the interface. This is what I was involved with in my Ph.D. thesis.

BOHNING: Had you selected that, or did he assign it to you?

SINFELT: I selected it from a number of problems that he had. Now, when I started working with Drickamer, his main interest was in high pressure. He had already developed a program there where he was measuring diffusion properties of gases under very high pressures; then he went to liquids, measuring diffusion in liquids under high pressures, and eventually he went to solids. Later he got involved in the effect of high pressures on electronic properties of solids.

I made the choice. He had this one problem off to the side on transport through an interface, which was not utilizing high pressure. I was one of the only people in Drickamer's laboratory who did all of his work at atmospheric pressure. I was sort of an outcast from the rest of the group. [laughter] But that didn't bother me, because I could see that it was a very important problem in the design of all equipment for separations processes, plus the fact that it was a very important fundamental problem. I picked that problem and I've never been sorry. That's where I got involved in surface chemistry, because it was the physical chemistry of an interface. Heterogeneous catalysis, which I later became involved with in industry, is really a surface problem, but I can say that I began my surface science work as a graduate student.

BOHNING: How many people were there in Drickamer's group?

SINFELT: I don't remember exactly, but maybe eight to ten students at various stages in the course of obtaining a Ph.D., and then he might have had a postdoc or two. You didn't see as many postdocs at that time as you do today.

BOHNING: Did you have much coursework to take at this point?

SINFELT: My courses were completed during the first two years. My third and final year was devoted exclusively to research. That was another interesting philosophy that Drickamer had. He did not believe in lots of courses for graduate students. He thought that by the time we completed undergraduate studies, we probably should have had most of our coursework, and graduate school should be mainly a research experience. That was his personal philosophy. If he would have had his way, we would have taken only a couple of courses the whole time we were graduate students. He would have been much more in favor of emphasizing research in place of a number of the courses in a graduate curriculum. I wound up taking typically three courses a semester for the first couple of years, in addition to doing research. One thing Drickamer insisted on was that his students begin research right away, as soon as they started working with him. He wanted us to begin doing research while we were still doing our coursework and studying for our doctoral candidacy examinations, so I was doing research essentially the whole time I was a graduate student.

BOHNING: You had no teaching?

SINFELT: I did some teaching, yes. The first year there my assistantship was actually listed as a teaching assistantship, and I did that the first year. Then the second year I went into a research assistantship. The kind of support I had was comparable for either type of assistantship. The teaching was primarily in recitation sections or laboratory sections. Generally, a professor would handle the lectures in the courses, and teaching assistants would have the quiz or recitation sections.

BOHNING: In 1954, you were looking at finishing up your work and getting your degree. Did you have any thought of an academic position, or were your interests strictly industrial?

SINFELT: I was pretty much oriented toward industry, probably largely because I hadn't really thought too much about an academic position. That's a very interesting question. I would have to say that I had pretty much made up my mind at an earlier stage, maybe in high school. I was strongly oriented toward an industrial position with an oil or chemical company. That was very real to me, and I don't think I ever got over that. I think that was it. I had pretty much made up my mind. I didn’t even go in and talk to Professor Drickamer about arranging some interviews with schools. I just didn’t think along those lines. I was so conscious of getting a job with a strong company in an important industry. I thought that this was the best place to become firmly established in a lifelong career.

My interviewing was restricted entirely to industrial companies. I didn’t do a lot of interviewing. I only made a couple of trips. In those days, there were many jobs available, so different from today. I got out of school at exactly the right time. There were fellows who went all around the country on interview trips. In other words, it wasn’t a question of whether we could get jobs, but it was deciding where we wanted to live and picking the company where we wanted to work. [laughter] I mean that in all seriousness.

I remember interviews with only four or five companies. I decided I’d like to come back to the Northeast area somewhere, so I interviewed the big companies there—DuPont; Socony Vacuum, which today is Mobil Corporation; the Standard Oil Development Company, which is now Exxon Research and Engineering [ER&E]; Union Carbide [Corporation]; and maybe Sun Oil Company. I made maybe two trips, hitting three companies or so on each trip. I got offers from all of them and multiple offers at different locations from several of them, and I finally decided on going to the Standard Oil Development Company. It was located in New Jersey, not terribly far from where I grew up in central Pennsylvania. If I wanted to travel back home, it wasn’t such a major operation. I liked what I saw here. The Standard Oil Development Company was doing mainly process development work, and that was something I thought I was pretty familiar with as a result of the experience I’d had at Koppers. I thought I’d like to take a crack at it.

To go back for a few moments to Illinois, I’d like to re-emphasize the importance of that experience for me, probably as important as anything in my ultimate development. The experience was valuable largely because of Harry Drickamer. I owe him an enormous amount. He was not an easy man to work for. In fact, I had plenty of arguments with him, [laughter] as everybody in the place did. But I could see he really liked what he was doing. He didn’t fool around; he was highly dedicated to research. I figured, “Anybody who likes his work that much has got something.” That impressed me, probably more after I left than when I was a student there. I didn’t appreciate it then. I began to appreciate it much more after I left Illinois.

I fondly remember my time at Illinois. I have never regretted it. That was very important for me—the whole university, all the people there. I didn’t know Roger Adams very well, but I sure knew who Roger was, and when I used to go in and register and talk to Roger, he made his presence felt very strongly with regard to what he thought graduate students ought to be doing. I remember professors like Worth [H.] Rodebush in physical chemistry. He was pretty disorganized, a real character, but I liked Rodie. He had made some very important contributions to physical chemistry in his early days. I remember Fred [Frederick T.] Wall in physical chemistry quite well, John [C.] Bailar in inorganic chemistry, and in organic chemistry Reynold [C.] Fuson and Speed [Carl S.] Marvel. Illinois was loaded with people like that. Their organic chemistry at that time was probably as good as there was anyplace in the world. But then again, they had a fabulous inorganic chemistry division with people like Bailar, Lou [Ludwig F.] Audrieth, and [Therald] Moeller. In physical chemistry they were very strong with Rodebush and Wall, and then Herb [Herbert] Gutowsky, who was among the very first if not the first chemist to really do anything with NMR [nuclear magnetic resonance]. In chemical engineering, apart from Drickamer, the professors I knew best were H. Fraser Johnstone, the Head of Chemical Engineering at that time, and James W. Westwater, a very distinguished researcher in heat transfer. Thomas Hanratty, who would later become very well known for his work in fluid mechanics, was just beginning his career. There was a tremendous group of professors in the department.

BOHNING: You don't find those kinds of collections of faculty much anymore—not to that extent. Whom did you interview with at Exxon?

SINFELT: I interviewed at least two, maybe three, different divisions. I interviewed what was then the Process Research Division, located in the Bayway Refinery. That division just happened to be located there, even though ER&E was independent of the Bayway Refinery operation. Then there was a research division located in Linden across Route One from the Bayway Refinery. The interest there was product quality—lubricating oil additives, gasoline composition and issues of that sort. Then I think I may have had an interview with the Chemicals Research Division. I chose to work in the Process Research Division, and two things might have had a big bearing on that. Because of the experience I'd had with Koppers in process development work, I thought I might like it. The Process Research Division was concerned with developing processes like catalytic cracking and catalytic reforming. Also, since I had worked at Illinois on diffusion through liquid-liquid interfaces, I thought it might be a way of getting involved in some research on a separations problem that might make use of that background. I thought I might get involved in something like an extraction or absorption type of process, which never happened. [laughter]

BOHNING: What was your first salary?

SINFELT: It was sixty-nine hundred dollars a year for a Ph.D. A person with a bachelor's degree might have gotten around forty-five hundred dollars a year. Koppers had offered me a job when I finished my undergraduate work for a salary of about thirty-six hundred dollars a year.

BOHNING: In 1954, then, you arrived at Bayway.

SINFELT: Yes. Actually, I reported at the research center in Linden for a medical exam and some processing my first day. It was October 18, 1954. I spent half the morning in Linden and then drove across the highway to the Bayway refinery and met the people whom I was going to work with.

Before I reported for work, the people in the Process Division already had in mind an assignment for me. In later years, many young Ph.D.s began their careers with a number of choices of problems they could work on, particularly if they were reporting to a corporate research laboratory. At the time I started, people pretty much went into a specific assignment.

In 1954 there was a need for gasoline components with improved anti-knock ratings, since automobile compression ratios were going up. Everybody wanted larger, more powerful automobile engines in their cars and needed higher quality fuels to satisfy these high-powered engines. At the time of World War II, the kinds of processes that were available in a refinery for making high octane gasoline were catalytic cracking, which came on largely as a result of World War II, and alkylation. We didn’t have the quantity of high-octane components that would be needed. How could we get these? We had to go to aromatic hydrocarbons—benzene, toluene, xylene, and so on. They are superb anti-knock components. The process to do this is known as catalytic reforming. In those days it was called hydroforming.

During World War II, the Standard Oil Development Company, with Standard Oil of Indiana and M.W. Kellogg, introduced a hydroforming process which first went on-stream around 1940 in Texas City, Texas. This supplied much of the toluene that was needed by the United States and its allies during World War II for explosives like TNT. This hydroforming process converted saturated hydrocarbons like paraffins or cycloparaffins to aromatic hydrocarbons. A typical reaction was cyclohexane to benzene, methylcyclohexane to toluene, or normal heptane to toluene. A transition metal oxide was used as a catalyst, molybdena supported on alumina.

Shortly after World War II, it became clear to petroleum companies that they were going to need much more capacity for the production of aromatic hydrocarbons. It wasn’t too long until people began to get interested in precious metal catalysts like platinum for reforming, because they thought these might be better catalysts than the transition metal oxide catalysts. There were a number of places in industry where people were interested in this, but one company in particular really pushed this—Universal Oil Products. They actually commercialized a platinum catalyst for reforming in 1949, and one person who was closely involved with this was another Perkin Medal winner, Vladimir Haensel.

BOHNING: I talked with him (4).

SINFELT: I know Val quite well. Val's major contribution in my mind was having the faith that a platinum catalyst could be commercially practical in a petroleum refinery, with all the possibilities for catalyst "poisoning" which existed. That was the big thing. Other people were interested in platinum catalysts too, but UOP <u>pushed</u> this and actually <u>did it</u>.

As of 1949, there were two processes out there—a hydroforming process using a non-precious metal catalyst, and another using a platinum on alumina catalyst. In the meantime, Standard Oil Development Company had gone one step further with the transition metal oxide catalyst, introducing fluidized bed technology in hydroforming. As in the case of catalytic cracking, catalyst was continuously circulated between a reactor and a regenerator.

The first hydroformers during World War II were fixed bed units. They were the units that were making the toluene. Standard Oil Development Company then successfully applied its fluidized bed technology in hydroforming. There were a number of commercial units of that type being designed after World War II. At the same time, there was another group of people in Standard Oil Development Company interested in platinum catalysts, just like UOP was, so there was a battle within the organization, "Which way should the company go?"

[END OF TAPE, SIDE 3]

SINFELT: "Should the company go with fluid hydroforming or with fixed bed platinum hydroforming?" It wasn't a situation like that at UOP where they were concentrating completely on platinum catalysts. Here there was a spirited battle going on inside the company. There was enough interest in fluid hydroforming so that a number of units were actually built. One of these units achieved quite a bit of notoriety because of a major accident. A fluid hydroformer located in Whiting, Indiana, blew up in 1955 and several people were killed. It was a major disaster.

BOHNING: One of my roommates at Valparaiso [University] lived down the street, so I saw it a week or so later.

SINFELT: Well, that didn't do fluid hydroforming any good, but it was something that could have happened with other processes. It was not unique to fluid hydroforming. However, for refiners trying to make a decision between fluid hydroforming and fixed bed platinum reforming, the accident didn't help the case for a fluid hydroformer, human nature being what it is. Also, it became evident over a period of time that the gasoline yields with a platinum catalyst were somewhat higher than they were with a molybdena or chromia catalyst.

When I reported for work in 1954 at the Standard Oil Development Company, I was asked to work in the area of hydroforming. There was a small group in the Process Division working with platinum catalysts. The president of the company was E.V. Murphree, a very highly respected engineer. He had been the head of a planning board for engineering for the Manhattan Project during World War II and was one of the key people involved in the development of fluid catalytic cracking. He was also one of the early people to be very interested in fluid hydroforming.

While the small group interested in the platinum catalyst was able to keep working and to really evaluate this approach, the competition with fluid hydroforming meant that the company did not get into platinum reforming until somewhat after UOP did. The first UOP application was in 1949. At the time I reported for work in 1954, Exxon did not yet have a commercial platinum reformer. UOP's first couple of years of commercial experience helped the case for the fixed bed platinum people inside companies like ours, and in other companies as well.

When I reported for work, I became involved in some development work with the purpose of getting information for a catalytic reformer planned for the Baltimore Refinery, which, incidentally, is no longer in operation. This was to be the first commercial application of catalytic reforming with a platinum catalyst in Exxon. That unit went on-stream in mid or late 1955, so I had been working for about a year before the platinum catalyst was applied in the Baltimore Refinery.

BOHNING: Whom were you working with?

SINFELT: Fortunately, the director in charge of our work was a man named A. [Albert] B. Welty. He had been one of the early people in fixed bed hydroforming during World War II. He was a Purdue [University] graduate who came to work at Standard Oil Development Company during the Depression in 1935.

Al Welty had extensive experience in hydroforming. He had all this background with the non-precious metal catalysts in hydroforming, and he was now the director in charge of the platinum reforming program. He was not somebody who was strictly an administrator. He was an extremely good technical man. I was very fortunate in coming to work under somebody like him. Welty wasn't my immediate boss; I reported to a section head at that time, but Welty was responsible for the overall research on that project.

After the Baltimore unit went on-stream, it performed quite well. The operation in the refinery agreed very nicely with our pilot plant results. We could take the data for the pilot plant and practically superimpose them on the data obtained at the refinery for a given feedstock. Then it became clear that we were going to have to build many more reformers, because the initial application turned out to be quite a success, and there was a demand for reformers in our refineries all over the world. We could see the tasks that lay ahead for us. We had to do an awful lot of process development work, and we had to start looking at different feedstocks. The feedstock for Baltimore was a West Texas feedstock. Some of the units in other refineries in Europe were going to use Middle East stocks which were very different, highly paraffinic type stocks, much harder to reform. We had to get more and more data and were doing process development work with different feedstocks, trying to get information for a number of different designs.

A couple of years after we were in this, some of us began to wonder if we were ever going to be able to satisfy the needs with this kind of an approach to the problem, without building an enormous development effort with pilot plants all over the place and feeds coming in from all over the world. Also, by that time people wanted to license the process from us. Even though UOP had a process, we had a competing platinum process, as did a couple of other companies, so there were a number of platinum reforming processes out there. A number of us began to wonder if we should be taking a longer-term, more fundamental approach to the problem. Maybe we ought to start trying to look at the process from a kinetic point of view, investigating the kinetics of the individual reactions occurring in this complicated process.

When I joined the Standard Oil Development Company, it was a superb development company, but there was very little basic research going on in the company. There was little interest in this kind of research, and there was a lot of resistance to it. People would say, “You mean you want to start with pure hydrocarbons and study the kinetics of reactions such as the conversion of cyclohexane to benzene and of normal heptane to toluene? Do you think you’re going to be able to put all this stuff together and predict what happens with a naphtha feed, which has anything from C_5 to C_{10} hydrocarbons in it? You’ve got paraffins, cycloparaffins and aromatics in the feed; you’ve also got C_5 as well as C_6 ring cycloparaffins. You’ve got all kinds of isomers in the feed, and you think you’re going to predict reforming performance from studies of pure hydrocarbons?” People didn’t think that was very smart.

Fortunately, I had a section head at the time who was strongly behind us. We argued our case something like this. “We’ll study the reactions of some of these selected hydrocarbons; we’ll try to understand the kinetics from some sort of a mechanistic point of view, and then we’ll do some experiments with simple blends to see whether there are interaction effects. For example, if we’re studying the dehydrogenation of cyclohexane to benzene, we will determine if the reaction is affected by the presence of other aromatics in the system. Other aromatics could compete for adsorption on the surface. We will do some experiments of that type, and from the data we will attempt to obtain some information on relative adsorption constants of different components on the surface of the catalyst, and so on.” We finally received support from our management. They said, “Well, okay, go ahead,” but there were many people who didn’t feel that we were going to succeed.

BOHNING: Who was the section head?

SINFELT: A fellow named Bill Priestley. I don't even know whether he's still living, but he's been long retired from the company. He wasn't known for this area. He had made his mark mainly in the analytical part of the company, bringing mass spectrometry into the laboratories relatively early in the petroleum industry. He then became a manager and a section leader in hydroforming without having much background, but he tended to see things the way a research person saw them.

The reason the kinetic studies came about was because reforming was such a huge undertaking. We could see that there was going to be an enormous number of applications. We were already thinking that this process, in its ultimate usage, was going to rival catalytic cracking, so we thought, "Maybe we should do this. In addition to trying to develop some kind of a kinetic basis for making predictions about the process to give us some guidance, maybe we'll learn something fundamental that could be useful in research." Therefore, we embarked on a kinetic program within the section. I was given the opportunity to lead this group, and that turned out to be very important for me.

We spent a couple of years studying the kinetics. It's hard and demanding work. Computers were just coming into their own in those days. We had another section in the division known as the Correlations Section, where there were people who specialized in developing correlations of all kinds for refinery processes, and they had expertise in the computers which were then available. We studied the kinetics, developed rate equations for all the reactions, and studied the interaction effects. We even studied the kinetics of coke deposition on the catalyst, because one of the problems in the process is that side reactions produce carbonaceous residues on the catalyst surface. These residues deactivate the catalyst; eventually, the catalyst must be regenerated by burning the coke off. We studied all of this, and after about three years and some work with people in the Correlations Section, the information from these kinetic studies was incorporated in a kinetic model of the process.

We didn't get all the parameters that were needed in a kinetic model of the overall process from studies with pure hydrocarbons. We used the pure hydrocarbon work to get an idea of what the rate equations should look like, what the partial pressure dependencies should be, what the temperature dependencies would be, and what adsorption constants might be. Some of the parameters were obtained from actual experiments with naphtha feeds. Values of parameters were not obtained for the hundreds of components present in a feed stream. Many were lumped together. For example, normal paraffins above a certain carbon number were treated as a single component. We had to build some simplification in, but we had a kinetic model of the process.

All this was programmed on a computer by engineers in the Correlations Section to see how well results could be predicted for a real feedstock. At that time gas chromatography was just coming in, so we were able to analyze many of the components in a naphtha. After a couple of years of work, the kinetic model was developed to a point where it could be used to predict reforming results for a variety of feedstocks. The model has been used extensively for planning new applications of reforming for more than three decades. The successful development and application of this model really plowed some new ground in the petroleum industry.

One particular individual who played an important role in the development and use of the model was Walter Kmak, a very good friend of mine who had also been a graduate student at Illinois. He died of cancer about four years ago, in 1991. He spent a good deal of his career using this kinetic model in the engineering division of our company to plan new applications of reforming.

BOHNING: How long did it take you to convince people? Although you had support for this project, you still had to sell people on the value of the work.

SINFELT: That's right, and it's not something which you ever completely sell. As a matter of fact, there were always skeptics who questioned the approach we took.

With the kinetic model, we can't get information as good as that from an actual pilot plant demonstration, but we can get a pretty good idea of what the performance of a reformer will be. Also, the amount of time spent on demonstration runs in pilot plants can be decreased, because the kinetic model will already scope out <u>most</u> of what we want to know about the effect of operating conditions on product yields, catalyst deactivation, et cetera. The kinetic model proved to be very valuable for planning and for making key decisions; for example, how should operating conditions be altered to make more benzene than is normally made in a reformer because of a need of benzene as a chemical rather than a minor component of gasoline. The model helped enormously with these kinds of questions. An indication of the success of the model is the fact that it became increasingly important and increasingly accepted over a period of time.

Our research on the kinetics of reforming reactions also had an intangible value. Apart from the fact that it had a direct effect on approaches to the design of reformers, what about the effect it had on us research people? It taught us an enormous amount about what was going on in this process that we would never have learned otherwise. What did that mean in the case of my career? It meant that I had learned a lot about reaction kinetics and the fundamental aspects of heterogeneous catalysis, and it set me up for my next job.

After this work was done, I was asked to join the Exploratory Section of the Process Research Division to set up a longer-term group doing fundamental work in heterogeneous catalysis, with longer range and more general objectives than what I had in the area of catalytic reforming. My work in catalytic reforming introduced me to the area of reaction kinetics and provided me with excellent background for the new assignment. This background probably influenced everything I've ever done in forty-plus years in the company. It had a strong influence on my future research and had a lot to do with what I ultimately got involved in—bimetallic catalysts.

It's interesting from a historical point of view to reflect on some changing attitudes about research at that time. As I said, when I came to work, my employer was known as the Standard Oil Development Company, a superb place for developing processes. Not very much long-range fundamental research was done. E.V. Murphree was the president of the company at that time. He had been involved in the Manhattan Project during World War II and was widely respected. I think he had been developing a sense that we needed to do some basic research in the company.

I remember one of our technical committee meetings during my early years with the company. We used to have these meetings in the Poconos at a place called Bushkill Falls. It was a nice place to have a meeting. The meeting lasted for one week and was held annually. It involved people from all over the company, and Murphree would be there. He was a superb technical person, and his interest in technical issues was unusual for a person at his level. He would get up at the end of these meetings and give a summary of what went on at the meeting and the way he saw things.

One year he devoted his concluding remarks to the thesis that we ought to be doing more basic research in the company. He was saying, "Our scientists don't devote enough attention to publishing and things of this sort. We ought to be trying to look at all of our research programs as fundamentally as possible, and we ought to publish some of the work. We ought to get out and get more involved with the scientific community and the engineering community." That led to a lot of managers taking some action, and I was pleased about this, because I was interested primarily in a scientific career. I was in the right place at the right time. An exploratory section was organized in the Process Division. Later this led to a separate division, the Central Basic Research Division, and ultimately that led to the Corporate Research laboratories [Corporate Research Labs].

So, for most of the time I've been with the company, there has been a growth of basic research. Right up until the early 1980s it had been going up at a good pace, and then things turned around. For the past decade, the interest in basic research has declined sharply. I was very fortunate to have a career which for the most part spanned a period of growth and interest in research in line with my particular interests. It was just right for me.

BOHNING: Before we move on to the next stage, I have two questions from this period yet. One is, you published some papers very early, in 1960, on kinetics of isomerization. You've just mentioned Murphree saying, "We've got to publish more," but I'm wondering what the publication situation was like in 1960?

SINFELT: Well, it was more difficult to get things published in those days because it was new to the company. There weren't too many examples. Certainly there had been papers published—I don't mean to indicate that there hadn't been—but not on a widespread basis across the company. It was more difficult. But when Murphree made his speech at the technical committee meeting, the attitude toward publication changed. There had to be acceptance of it because Mr. Murphree had made his views very clear. He thought it was important for the company. He wasn't asking us to forget about proprietary aspects of our work. He was simply saying that he expected all the people engaged in scientific efforts in the company to be thinking about publication as an important part of their work.

[END OF TAPE, SIDE 4]

SINFELT: So that was something new.

BOHNING: The other thing was that in your Perkin Medal address, you made an interesting comment, which I'd like to bring in here and keep in mind as we go on. You said that many researchers trying to find new catalysts have an attitude that catalysis is too complex for a fundamental understanding, that other workers trying to find a fundamental understanding of catalysis are not necessarily driven by a desire to find new catalysts, and that much is to be gained when you do both, which is what you were doing. Would you say you were unique at that time, that your counterparts in other companies were not doing that?

SINFELT: Oh, I can't say I was unique. What I was getting at in the Perkin Medal address (5) was that researchers were motivated in two different ways. It was a very general statement. I was not implying that workers in other industrial organizations were less interested in fundamental work. I simply meant that researchers tended to separate into two camps, as I said. There would be people developing catalysts who were not primarily concerned with the mechanism or kinetics of reactions, and many of them would even go to the extreme of saying, "It's a waste of time. Don't bother me with this stuff."

BOHNING: Would you say they had an Edisonian approach, taking something off the shelf and trying it?

SINFELT: I hate to use those words, too. One thing I'm sensitive about are the words Edisonian and empirical. Edison needs no defense of his approach. There are a lot of people who would love to to have the accomplishments of Thomas A. Edison [laughter]. The word empirical is another one that I think is misused, because all science is empirical, in my mind. That's the way chemistry developed. That's the way most things develop. The word empirical has the connotation of building. I think that's why the history of science is so important, because it gives us perspective about the way things actually happen. We first build up a collection of empirical facts; observation is a very important part of the process.

When I say there are people out there who are identified with developing catalysts and not being interested in mechanism or kinetics, I'm not downgrading those people. Some of the most successful people out there were in this camp, and they've got the facts to prove it. Whatever is responsible for their success, their motivation is different from that of another group of people oriented toward understanding mechanism and kinetics. A while back the only people interested in the fundamental aspects of heterogeneous catalysis might have been people in academia, and maybe a very few people in industrial organizations.

I do remember one person at Shell, for example, who went back to the World War II days, and prior to that, who was interested in a more fundamental approach. That was a man named Otto Beeck, who died in the early fifties. He was interested in fundamental studies of catalysts and had a program going even before World War II out in the Emeryville labs at Shell. There were a few people in Universal Oil Products who were very much oriented that way, too. Louis Schmerling is one I remember who was very much interested in the mechanisms of acid-catalyzed reactions, carbo-cation chemistry in general.

So we can't say that all the people in the fundamental camp have been from academia. In the search for new catalysts, there is clearly nothing wrong with having an interest in fundamental aspects of catalysis. I think there can be real advantages, but not necessarily for the reason that a fundamental attitude about things is going to lead to a new catalyst or a new process. That might happen; that could happen, but it might not. Science, in the way I see it, is frequently most useful when we get to the development stages of a project. It has been my experience that the important role of science is frequently not in the initial discovery; it's in doing something with a discovery once you've made it. I have seen situations where discoveries have been made and where things have fallen by the wayside because the science wasn't followed up. People didn't try to understand in sufficient detail what they were doing; something was transferred from an initial discovery stage to a development stage without a proper understanding of some of the things that were important in the process.

If a researcher interested in fundamentals does make a discovery, he or she is likely to have the attitude, "Gee, I'd like to understand this better." For example, the researcher might study the structure of a new catalyst and determine whether that structure is going to be stable at the conditions at which a commercial operation is conducted. Information of this kind can be critical for the successful exploitation of a new catalyst in a process. There is no substitute for good fundamental understanding in the development stage of a program.

BOHNING: That's a very good point. Before we leave this early six-year period ending around 1960, you indicated on the questionnaire you filled out for us that a person of influence during this time was a colleague in catalysis (6).

SINFELT: Michel Boudart?

BOHNING: Yes.

SINFELT: It's time to bring him up, definitely. When I reported for work in 1954, at the process research division, Michel Boudart, who was born in Belgium in 1924, was on the faculty at Princeton University. He had been a student of Hugh Stott Taylor. Michel is six or seven years older than I am, and I met him within a few months after I joined the company. How did I happen to meet him? He just happened to be visiting our laboratory. He had made a contact with somebody in the company, maybe Jack [John P.] Longwell or Don Baeder. Michel was just beginning his first term as an assistant professor at Princeton at the same time I joined the Standard Oil Development Company. I met Michel, and somehow Michel happened to see a copy of my Ph.D. thesis—probably via Don Baeder. Don Baeder was in another part of the process research division, and when I first came to work we got to talking about my doctoral thesis. Don was interested in it, so I brought it along to work. After seeing my thesis, Michel asked me if I would come to Princeton to give a seminar on the work. I presented the seminar in April 1955. I remember the occasion very well, since it occurred within a few days of the date of the death of Albert Einstein, a long-time resident of Princeton.

I was just starting in catalysis, whereas Michel already had five or six years of experience with Hugh Stott Taylor in this field. One thing led to another. Michel organized a seminar on catalysis at Princeton, which he was kind enough to schedule for Saturday mornings so that some industrial people could attend. Michel gave a number of the seminars. I regularly went to these seminars, so I got a lot of background information on catalysis. Sir Hugh Taylor attended all of them, and that's where I met him.

Michel is a superb kineticist. He's one of the world's great kineticists, I think, certainly in the area of heterogeneous catalysis. When I started working on kinetics, we began talking about some of the things I found, and Michel was getting interested in some of our results. He was also involved with setting up several outside meetings, and he invited me to present papers at some of them, around 1960. By that time, Michel had developed a continuing relationship with our company where he spent a couple of summers actually working in our laboratories as a summer employee. He did some work on reforming catalysts and their characterization with adsorption measurements; the work produced very interesting results.

Michel has had a consulting relationship with Exxon for close to forty years. We've become very good friends over the years. We discuss all kinds of problems in catalysis and we're very much aware of each other's work. Michel left Princeton in about 1961 to go to the University of California at Berkeley. He was there three years, and then he moved to Stanford for the rest of his career. He retired in June 1994 to become Professor Emeritus. I presented a talk at the retirement symposium in his honor. This term, he is completing a sabbatical at Cal Tech. He no longer maintains an active laboratory research program. He will undoubtedly spend a fair amount of time writing and travelling in coming years.

My long friendships with both Michel Boudart and Harry Drickamer have been very important to me. In my student days, Harry Drickamer stimulated my interest in pursuing research as a career. Then in my early work days, Michel Boudart came along and provided a second stimulus. He was an ideal person for me to talk to, and he did much to encourage my interest in the kinetics of catalytic reactions. My many discussions with Michel in the early stages of my career in catalysis had a very important influence on my work.

BOHNING: Well, here you are in the early 1960s; you've got a mandate from the president, Murphree, saying, "We need more fundamental research," and you created this exploratory group.

SINFELT: I was asked to head a catalysis group within a larger section doing exploratory research.

BOHNING: How many people were put into that originally?

SINFELT: About four or five professional researchers were in my catalysis group, out of a total of about a dozen in the Exploratory Section overall.

I was in that Exploratory Section in Process Research for about six years. The Process Division in Bayway was eventually broken up. You may ask why was this done, because it had been a very successful division. There were a couple of factors. We had another laboratory doing process development work in Baton Rouge, so there were two laboratories, not working on the same problems, but engaged in the same general kind of activity. Also, we had a bad strike at the Bayway Refinery in 1964. That was the year of the [Barry] Goldwater-[Lyndon B.] Johnson presidential election. I don't know whether the strike had anything to do with the demise of the Process Research Division; I'm just mentioning it. Since the research people were actually working within the refinery—our laboratories were inside the refinery—we had trouble with access to the refinery for about three or four months. There were some problems with picket lines during the strike.

After a couple of very uncomfortable weeks gaining access to the refinery in situations which could have been pretty hostile, we were instructed to report for work across the highway at Building One, where the main research center was located. We did that for several months until the strike ended. Within another year or two, it was decided to do away with the Process Research Division altogether. I don't know the main reason for the decision, but the strike certainly didn't help. It might have been that the company wanted to consolidate process development work in the Baton Rouge laboratories and transfer activities like mine to the Central Basic Research Lab [CBRL] already set up for chemicals research. I brought into CBRL a catalysis group directed toward fuels processes. Gradually, however, some research related to chemicals was initiated.

It might be well for me to make another point now. I was transferred to CBRL in 1966 and worked in that laboratory for a couple of years. Then at some point it was decided, "We have to increase the scope of research in Exxon by a step-change, and go to something new, larger than CBRL." In 1968 the Central Basic Research Lab was replaced by the Corporate Research laboratory.

The new feature of this was the mode of funding. The Corporate Research lab was to be funded centrally by the corporation itself, the Exxon Corporation. Prior to that, all the research had been funded by the affiliates. You see, the Exxon Corporation is basically a holding company, and there are many affiliates worldwide. For example, Exxon Company USA is one affiliate. Imperial Oil in Canada is another affiliate, and there are affiliates in Europe, in the Far East, and in other parts of the world as well.

As of 1968, the corporation itself—rather than the affiliates—funded the long-range basic research in the company. The extent of basic research then increased sharply. The number of scientists increased to about one hundred in the seventies. In the latter part of the seventies, Ed David became president of Exxon Research and Engineeering. It was decided that Corporate Research should expand again, and that the facility should be moved to Annandale, New Jersey.

BOHNING: By the early sixties, you had already been successful with your kinetic studies, and that was a great asset to the company. What did you do for an encore? Where did you go from there?

SINFELT: In the early sixties, when I was first transferred to the Exploratory Section of Process Research, I embarked on a personal research program with bimetallic catalysts, i.e., alloys and related systems. Why did I get interested in this? Well, I wasn't the first one to ever get interested in an alloy catalyst. I want to make that clear immediately. There was a widely used industrial catalyst known as Raney nickel, a nickel-aluminum alloy, which Merritt Raney had patented. It was used extensively for hydrogenation. Some people researching the fundamental aspects of catalysis had been interested in alloys to study the so-called electronic factor in catalysis by metals. There were actually a few investigations on this before World War II; that's how far back that went. It became a very popular activity just shortly after World War II. Some of the ideas of solid-state physics were coming into the catalysis community. The electron band theory of solids had been developed and attracted the attention of catalytic chemists.

Catalytic chemists had this idea about the electronic factor: Group VIII metals like nickel and platinum have incompletely filled d-bands, whereas Group IB metals such as copper and silver have d-bands which are filled completely. According to an early theory of [N.F.] Mott applied to a nickel-copper alloy, the copper would serve as an electron donor to the nickel, and when the alloy contained about 60 percent copper, the d-band would be completely filled. Experimental data showing the disappearance of ferromagnetism at this composition supported Mott's theory, which was known as the "rigid band" theory.

Well, some people had the idea that maybe this is important in catalysis, because the good catalysts were usually metals like nickel, platinum, palladium—all of which had incompletely filled d-bands. These metals were markedly better catalysts than a metal like copper, silver or gold for the hydrogenation of an olefin or the dehydrogenation of a hydrocarbon like cyclohexane. For nickel-copper alloys, the postulated variation of the extent of d-band filling with composition was viewed as a way to interpret observed effects of composition on catalytic activity.

That was a theme that researchers were interested in, and I can't tell you how many investigations were done on that in the late 1940s and throughout the 1950s. But they were all done with the same thing in mind—with either the nickel-copper system or the palladium-----gold system, which are very similar systems. The idea was to study a reaction—like the hydrogenation of ethylene to ethane, or H_2-D_2 exchange, or ortho-para hydrogen conversion—as a function of alloy composition to determine the relationship between catalytic activity and electronic structure.

There were many reported investigations out there, with widely varying results. It was very hard to draw firm conclusions after all this work was done. By 1960, an awful lot of work had been done and people were becoming skeptical about the whole idea. That didn't mean there was doubt about the existence of an electronic factor in catalysis. There certainly had to be; all chemistry is electronic. What it meant was that alloys of this type apparently weren't going to be a simple way of getting at this problem.

Now I come along, and I'm interested in alloys. The first question you might have asked is, "Are you crazy? What do you want to get involved in this mess for?" Well, the first thing I said to myself was, "I don't want to work on the electronic factor with alloys." Why? I don't know how to do it. [laughter] There was good reason for that. There were lots of other workers out there showing that they didn't know either. So, what's left?

This shows something about research, and why being in industry can sometimes be an advantage in research. All the effort with the alloys was directed toward one single issue—can we take a chemical reaction and correlate the catalytic activity of the alloy in some way to an electronic factor? Investigators seemed to view this as the only useful thing we could do with an alloy. Nobody asked an essential question: “If I have a molecule which can undergo a couple of different reactions, like dehydrogenation or scission of carbon-carbon bonds, will alloying affect them differently?”

I thought, “That’s simple. I know how to do that. I pass reactant molecules through a reactor and see what happens. The reactant will be a hydrocarbon which can exhibit different reactions.” I don’t mean that ethylene doesn’t know any chemistry, but there’s not much it can do in reacting with hydrogen. It simply hydrogenates to ethane. If I pass normal heptane over a metal catalyst, it can go to toluene or to methylheptanes; it can crack into C_3 and C_4 or into C_5 and C_2. It can do a lot of things. In one of the early experiments I made, a hydrocarbon like heptane was passed over nickel and nickel-copper catalysts, and I found that copper suppressed the catalytic activity of nickel for reactions involving carbon-carbon scission very selectively.

[END OF TAPE, SIDE 5]

SINFELT: Reactions that break carbon-carbon bonds are generally the reactions we want to eliminate in reforming, because we want to make aromatic hydrocarbons rather than crack hydrocarbons to little molecules. It turned out that reactions involving carbon-carbon scission were strongly inhibited when I alloyed copper with nickel, or gold with palladium, or essentially any Group IB metal with a Group VIII metal.

It turned out to be a general phenomenon. We make these Group VIII-Group IB alloys, and we find that that cracking of C-C bonds is very selectively inhibited. The Group VIII metal becomes progressively more selective for the reaction we want—namely, making an aromatic hydrocarbon or making an isomer. Certainly we don’t want to make methane and ethane. We were very fortunate that our very first experiments produced such exciting results. Then I knew selectivity was a very important aspect of catalysis by alloys. That was a new scientific finding. That was a very important scientific development.

A second question then followed. I knew if we found out anything interesting about an alloy, we were going to have to face up to how we could make a practical alloy catalyst. The alloys that had been studied in the fundamental investigations had all been low surface area materials, such as nickel-copper films or powders. As a matter of fact, some of my first experiments were with nickel-copper powders, because I wanted to get a good characterization of the alloys with various physical measurements. We obtained valuable x-ray diffraction data and magnetic data on the powders, but the surface areas of those kinds of materials are often several orders of magnitude too low for a commercial catalyst. We want a commercial metal catalyst to be in a state where it’s almost all surface; in other words, where the metal crystallites we have a high surface-to-volume ratio. If I’m talking about a nickel crystallite, I’d like it to be maybe ten Ångströms in size, if I could achieve something like that—because if it’s ten Ångströms in size, almost all the atoms in that nickel crystal are on the surface, so they’re accessible to the reactant.

Well, we knew we could make monometallic clusters or crystallites with such a size, because that's what made platinum reforming a success. The only way this expensive metal can be practical is to have crystallites small enough that virtually all the platinum atoms are surface atoms. The way to do that is to disperse the platinum on a high surface area oxide, like aluminum oxide. Aluminum oxide is a porous material. It has perhaps two hundred square meters per gram internal surface area in the pores. We simply contact chloroplatinic acid solution with the alumina and then dry it and heat it in air at about 500˚ C. When the material is subsequently exposed to hydrogen at about 400 to 500˚ C, very small platinum crystallites or clusters are formed. They are typically about ten Ångströms in size, as demonstrated by adsorption and high resolution electron microscopy.

Now, in the case of a nickel-copper alloy, I know how to make it in the form of a powder with a low surface area. How am I going to make a highly dispersed nickel-copper alloy? The approach taken was to disperse the alloy on a support.

Supported metal catalysts frequently contain a very small amount of metal relative to the support. In the case of a platinum reforming catalyst, the metal content is typically about one-half percent. If nickel and copper are dispersed on a support in such small amounts, how do I know they're going to form an alloy? Suppose I make a solution of nickel and copper nitrates and wet a support with it. After the material is dried and the metal salts are reduced with hydrogen, how do I know I don't have separate crystallites of nickel and crystallites of copper on the support? In those days I didn't have any physical tool to address that problem, because the crystallites of the metals were too small to get a diffraction pattern. If we tried to get a diffraction pattern, we would see essentially nothing. We would just see a background, with no sharp lines.

That's where the chemical probes come in. Do you remember what I said about nickel-copper powders, which I knew were alloys from x-ray diffraction and magnetic data? They were low surface area materials and therefore not practical catalysts, but they were useful for showing how a reaction such as the hydrogenolysis of ethane would be affected when nickel was known to alloy with copper.

Now, if I disperse nickel and copper on the support to form very small crystallites, I can't characterize the crystallites with physical probes, but I know how the ethane hydrogenolysis reaction should be affected if the nickel forms an alloy with the copper. The activity of nickel is inhibited markedly. When I pass ethane over the small crystallites in the supported nickel-copper catalyst, therefore, I can tell from the extent of hydrogenolysis observed whether or not the nickel is interacting with the copper. I actually found that the hydrogenolysis reaction was inhibited markedly, just as it was on a well characterized nickel-copper alloy. That told me right away that the nickel and copper had to be interacting with each other in the small crystallites, and therefore that highly dispersed nickel-copper alloys were present.

I used that approach to look at lots of catalysts, and I developed a whole body of knowledge of bimetallic catalysts based purely on chemical probes. At that time I didn't know anything about the interatomic distance in a small crystallite and whether it was going to be any different from what it was in a big nickel-copper crystal. I didn't have any idea about the extent of coordination of nickel to copper, but I did know from the chemical probe that I had nickel-copper interaction. I used chemical probes to look at all kinds of supported systems: platinum-iridium, platinum-copper, ruthenium-copper, palladium-gold, and many others. I found that small alloy crystallites could be made on supports. That was the second very important scientific finding—that a highly dispersed alloy could be prepared on a support. This was accomplished strictly with the use of chemical probes.

I found another interesting thing, a third scientific finding, if you will. In the course of exploring lots of these bimetallic systems, I found interesting catalytic effects even for combinations of metals which ordinarily would not form bulk alloys. I could take two metals which were completely immiscible in the bulk. A good example is ruthenium and copper. Ruthenium is a hexagonal, closely-packed metal; copper is a face-centered, cubic metal. In the bulk they are effectively completely immiscible. The solubility of one in the other is very low. Nevertheless, when I made catalysts with ruthenium and copper, I found the same kind of inhibiting effect of copper on the hydrogenolysis activity of ruthenium that I'd found when copper was added to nickel. What the heck was going on? If they didn't alloy, how could they be interacting? How could that be happening?

Well, I thought, "Is it possible that one component could be covering the surface of the other component? Even though they don't mix to form a homogeneous solid solution, could one of them be forming a layer on top of the other?" In other words, could copper be coating the surface of ruthenium? Could this occur like the adsorption of a gas? Hydrogen adsorbs on platinum or on ruthenium, but now, instead of hydrogen adsorbing on ruthenium, I'd be talking about copper adsorbing on ruthenium. If that is what's happening, we ought to be able to get at this pretty nicely by measuring adsorption. We pick a gas like hydrogen which adsorbs on ruthenium but doesn't on copper, and if indeed the copper is covering the surface of ruthenium, we ought to be able to see inhibition of hydrogen adsorption. That's exactly what happened. Then, later on, surface sensitive electron spectroscopy came along, and we could confirm that indeed the copper was covering the surface of the ruthenium.

If I had a large ruthenium crystal, with a monatomic layer of copper on the outside, I would have an entity with a very low atomic ratio of copper to ruthenium, since the big ruthenium core would constitute most of the metal. But suppose I make this crystal extremely small, which is what I'm doing when I use a support. Eventually I get to a point where the ruthenium core is so small that the amount of copper on the outside is comparable to the amount of ruthenium on the inside.

We actually did adsorption and catalytic experiments to show that this picture was applicable. The results of the experiments established the very important point that a highly dispersed bimetallic entity can have a stoichiometry that cannot be achieved in the bulk. The stoichiometry exists simply because the ruthenium-copper crystallites are so small. If they are small enough, the amount of copper that it takes to cover the ruthenium core is comparable to the amount of ruthenium in the core. The crystallites then resemble molecular species.

I said, "I need a new terminology now, other than alloys, because obviously ruthenium and copper are two elements that don't form an alloy in the bulk. If I'm going to try to get a patent, I'm not going to talk about a supported alloy catalyst when the two components don't form alloys." Therefore, I introduced the term bimetallic clusters in referring to highly dispersed bimetallic entities in general. They include combinations of metals which are miscible in the bulk, but are not limited to them. In ruthenium-copper clusters, there is a copper-ruthenium bond at the interface between the two elements, despite the fact that one does not dissolve in the other. Today, some of the inorganic chemists are making mixed transition metal cluster compounds which have bonds involving such elements. The work on ruthenium-copper and related systems established a third important scientific point, namely, that bimetallic catalysts need not be restricted to components which are totally miscible in the bulk.

BOHNING: In 1972, you wrote a paper which became a citation classic (7).

SINFELT: That's the nickel-copper paper. That was some of the work that I mentioned earlier. We didn't report that work immediately after it was done. As you know, in industry there's usually a lag between the time that we first do the experimental work and the time when we publish it. The work was done in the sixties, but we published it in 1972. It was a basic paper which revealed this strong selectivity factor in catalysis by alloys, where reactions that involve rupture of carbon-carbon bonds behave very differently from hydrogenation or dehydrogenation reactions. It also tended to reinforce the idea that we could have surface segregation of one of the components in a metal alloy, but the most important feature of that paper was the selectivity. We have some very basic chemistry there. You're talking about a basic difference in the way the alloy treats carbon-carbon versus carbon-hydrogen bonds, and that's very important for catalysis.

BOHNING: How readily was the term bimetallic clusters—which you coined—accepted?

SINFELT: I didn't have too much trouble, but we can see a reason why there might be a problem there. At about the time that I coined the term bimetallic clusters, inorganic chemists were also talking about clusters, but they were talking about clusters of metal atoms within a molecule—in other words, a transition metal cluster compound, like a carbonyl. I'm talking here about an essentially naked cluster, in the sense that there are no ligands around it, at least until we start doing catalysis on it or start adsorbing molecules on it. Once we start adsorbing molecules on it, we may have something very much like their transition metal cluster compound.

The word cluster had been in chemistry for a while before I started using it. The people interested in statistical mechanics and in phenomena of condensation of vapors to liquids had also talked about clusters. We can see this throughout their literature. They talked about clusters being anything from a few molecules to something the size of a liquid drop, which is going to have 10^{22} molecules or something like that.

There was some feeling that the word cluster should be reserved for very small collections of atoms. They said, “Clusters have to be pretty small,” because they were thinking in terms of the cluster of the inorganic chemists. That certainly applied to lots of our catalysts, too—the clusters are small—but I did not want to go along with the idea that I should limit their sizes. I thought that was pretty arbitrary, and I used arguments like the statistical mechanics usage of the word cluster. I’m very happy I didn’t get too specific about the size. It might be anywhere from a few atoms up to hundreds and maybe thousands of atoms. It now turns out that the inorganic chemists themselves are synthesizing compounds containing very large clusters of metal atoms. One of the last ones I heard about had 512 gold atoms in it, in a compound with a bunch of ligands around the cluster. Now you’re not talking about a few atoms. [laughter]

For a little while, some people were concerned with my reference to crystallites as large as fifty Ångströms in size as clusters. However, this doesn’t seem to be much of an issue today. People are synthesizing these giant molecules with huge clusters in them, so I am glad that I used the cluster terminology in a broader sense. The term bimetallic cluster has served its purpose very well. It is not misleading, but is sufficiently broad to include a variety of kinds of bimetallic systems of interest as catalysts.

There was also the practical issue about dealing with patent matters. In the case of nickel-copper, there’s no problem. The patent office might say, “Okay, so you’re claiming a nickel-copper alloy on a support. Fine.” But if I try to patent a ruthenium-copper alloy, a patent examiner with a phase diagram in front of him for the bulk solid system will say, “These don’t alloy. You’re out of your head.” I can go into all kinds of explanations, but it’s sort of like trying to reverse a bad initial impression. It would be better to have terminology which would eliminate this potential difficulty. I thought it was better to talk about bimetallic clusters, which would then include highly dispersed alloys as well as bimetallic combinations which didn’t ordinarily form alloys.

BOHNING: You worked with different metal combinations, but it was the platinum-iridium one which was key.

SINFELT: That was the one that was applied extensively as a commercial catalyst, yes. I mainly emphasized two types of systems. One, as I've already mentioned, was a combination of a Group VIII metal with an IB metal—nickel-copper, palladium-gold, ruthenium-copper, et cetera. Another type of system that I worked on very broadly consisted of a combination of two Group VIII metals—platinum-palladium, platinum-iridium, platinum-ruthenium, et cetera. I also did some work on combinations of two Group IB metals, like gold-copper and gold-silver, in the area of oxidation.

Why did I restrict myself to those combinations of metals? The main reason for sticking to those metals is that they are metals which are likely to remain in the metallic state under the conditions of catalysis. Usually, in a practical catalyst, to get high surface areas, we disperse the metal on a support. When we do this, the Group VIII or Group IB metals are about the only metals that can be reduced readily to the metallic form on the support. If we supported molybdenum, we would wind up with an oxide of molybdenum. We would have a hard time reducing molybdena to metallic molybdenum on alumina and keeping it that way. The same would be true for tungsten, chromium, or manganese. For all practical purposes, catalysis by metals is pretty much catalysis by the Group VIII and Group IB metals.

One metal that's not in Group VIII that I think can be reduced on a support is rhenium. The reducibility of supported rhenium was an issue which was hotly debated at one time, but now it's pretty clear that we can reduce rhenium down to the metallic form on a support.

Looking at metals within Group VIII, we already knew platinum was a good catalyst in reforming from the early work of Vladimir Haensel and others, and we knew that iridium had properties very much like platinum. It's right next door to platinum in the periodic table. However, iridium tends to have a very much higher activity to break carbon-carbon bonds than does platinum. Even though iridium will catalyze the aromatization of a molecule like normal heptane to toluene, it has a pretty high ability to crack carbon-carbon bonds also.

Then you might ask, "Why do you want a metal like this in a reforming catalyst? You're going to just butcher your feed down to low molecular weight hydrocarbons." Well, that's not quite the way it works out. It turns out that when the platinum and iridium are present in the form of a bimetallic cluster, the platinum tends to moderate the hydrogenolysis activity of the iridium. It turns out that platinum-iridium bimetallic clusters will give a product distribution not very different from that obtained with platinum, but will give it at a much higher rate. If I were building a reactor, I could build the reactor a lot smaller with a platinum-iridium catalyst—in other words, use much less catalyst because it's a good deal more active.

In addition to possessing higher activity, the platinum-iridium catalyst forms coke less readily than a platinum catalyst. Therefore, it deactivates less rapidly. We can run it for a longer period of time before we have to regenerate the catalyst.

These advantages were very important at the time the catalyst was introduced commercially. We first applied it commercially in 1971, in the early days when people were becoming interested in lead-free gasoline. When we take lead out of gasoline, if we want to maintain the quality of the gasoline, we need a higher concentration of hydrocarbons of high anti-knock quality. This need can be met with a greater rate of production of aromatic hydrocarbons. So higher activity reforming catalysts were very exciting at the time when we were first interested in lead-free gasoline. As I said, it turned out that the platinum-iridium catalyst had the high activity and the high activity maintenance, so we commercialized that catalyst.

At about the same time, one of our competitors was interested in a different type of bimetallic catalyst. This was the Chevron Company.

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SINFELT: The Chevron catalyst was platinum-rhenium, which had some features like platinum-iridium but also some which were different. Like platinum-iridium, it definitely had advantages over platinum catalysts. The virtue of the platinum-rhenium catalyst wasn't higher activity than platinum, but higher activity maintenance. Both platinum-rhenium and platinum-iridium showed better activity maintenance than the platinum catalyst, so they could run for longer periods or at more severe conditions for the same length of time, but the platinum-iridium catalyst also had higher activity. Chevron commercialized the platinum-rhenium catalyst in 1969, two years before Exxon commercialized the platinum-iridium catalyst commercially in the fall of 1971.

Now, we use both platinum-iridium and platinum-rhenium catalysts in Exxon, and in some applications we use both catalysts in the same reforming unit, because they have different features. A catalytic reformer is a multi-reactor system. There are usually three or four reactors in series, and as I mentioned, there are many different reactions going on in catalytic reforming. The way it turns out, we can almost isolate certain reactions in a particular reactor. The conversion of cyclohexanes to aromatics, for example, occurs very much faster than any other reaction in the system, so the first reactor will largely exhibit conversion of cyclohexanes to aromatics. Some conversion of alkylcyclopentanes to aromatics is also observed in the first reactor. Platinum-rhenium catalysts are particularly effective in catalyzing these reactions very selectively, whereas platinum-iridium catalysts are highly active for the conversion of noncyclic alkanes to aromatics.

The latter type of reaction is much slower than the conversion of alkylcyclohexanes and alkylcyclopentanes to aromatics and, therefore, is relatively more important further along in the train of reforming reactors. Consequently, we have units where we have platinum-rhenium in the front end of the system and platinum-iridium in the tail end. Both of these catalysts have major advantages over the original platinum reforming catalysts. In units where only one of these bimetallic catalysts is to be employed, the choice of catalyst depends on the particular application.

After the platinum-rhenium and platinum-iridium catalysts had been applied extensively in reforming, the Universal Oil Products Company came up with a different type of process for reforming. They developed a process called continuous catalytic reforming, which is somewhat reminiscent of fluid hydroforming, whereby spent catalyst is continuously withdrawn from the system for regeneration and freshly regenerated catalyst is added back. Although there is continuous replacement of catalyst in the system, the process does not utilize a fluidized bed. A platinum-tin catalyst is used in the process.

Now, there are advantages to operating at low pressures, because low hydrogen pressures favor the formation of aromatics, but they are also conducive to high rates of catalyst deactivation. The lower in hydrogen pressure you go in a reformer, the more you're susceptible to coke formation. That's why they had to have continuous catalyst replacement, because they experience enormously high rates of coke formation. That process is being used quite widely now. Although the capital investment is much higher for this type of process, the gasoline yields are appreciably higher.

These developments in reforming show the changing nature of things in the industry and what can happen to the researcher as a result. I mentioned earlier that when the bimetallic catalysts were first introduced commercially by Chevron and Exxon around 1970, there was a great interest in low-lead and lead-free gasoline. Something was needed to replace the lead in gasoline, and that was going to be more aromatic hydrocarbons. Bimetallic catalysts, then, played a very important role in influencing the economic feasibility of lead-free gasoline and, thereby, contributed enormously to the improvement of our environment. In addition to eliminating the hazard of lead itself, removal of lead from gasoline had the additional impact of making it possible to use catalysts for removal of carbon monoxide, unburned hydrocarbons, and nitrogen oxides from automobile exhaust gases. When you put a catalyst in the exhaust system of an automobile, if you have lead in the gasoline, the lead going through the exhaust system will deposit on the catalyst and poison it, and then the catalyst will not be effective. The use of catalysts for exhaust gas purification is therefore critically dependent on the availability of lead-free gasoline.

The improvements in catalytic reforming from about 1970 onward played a crucial role in some of the most important environmental progress that society has witnessed. In the last few years, however, there has been increasing concern about the possible environmental effects of aromatic hydrocarbons themselves. Could <u>they</u> be bad actors? Benzene has been a concern, so for some time now there have been restrictions on the amount of benzene that you can put in gasoline. People are becoming more and more concerned as we dig deeper into environmental questions, and they wonder if aromatics in general could be a problem. Even though reforming has played a huge, positive environmental role for more than twenty years, environmental issues are now being scrutinized to the point where the enormous positive benefits of advances in reforming are being challenged to some extent by the perceived negative environmental effects of aromatic hydrocarbons. You know about the pressures in the last couple of years to try to cut back further on aromatics in gasoline and partially replace them with things like ethers. Apart from replacing some of the aromatics in gasoline, the ethers are supposed to decrease automobile exhaust emissions.

Reforming grew over a period of more than half a century from the World War II days with non-precious metal catalysts to the introduction of platinum catalysts shortly after the war and subsequently to the replacement of the early platinum catalysts with bimetallic catalysts. The application of the process has been on the same scale as catalytic cracking. The process has been one of the top couple of developments in the history of petroleum refining. We're probably not going to see much expansion in reforming in the near future because of the increasing efforts to limit the amount of aromatics in gasoline. The question is, "What does the future hold?" I don't think that we will abandon reforming altogether in the foreseeable future. It's so important in fuels that for a long time it is likely to be an important contributor to the gasoline pool. The reason I go into all this is to show how the landscape looks over a period of time—and, for the industrial researcher, how it influences the course of his work. You could have been an environmental hero twenty years ago, but today they're giving you a hard time. [laughter] There were lots of people who were important in the evolution of lead-free gasoline and who have been affected by subsequent developments. We can see the changing nature of things over a period of time and how we have to adapt.

BOHNING: You really spent the rest of your career, or a good part of your career, continuing to detail these bimetallic catalysts.

SINFELT: Maybe I ought to talk a few minutes about that. After I had worked on the bimetallic catalysts with chemical probes and we had developed a commercial catalyst, I asked myself in the early 1970s, "What do I do now?" One thing I could have done would have been to explore bimetallic catalysts for all kinds of different applications. That would have been a perfectly valid thing to do, and maybe some people think that was the thing I should have done. I did look at some other applications, but I was interested in something else, and I think that points to the fact that I'm primarily science-oriented. I wanted to dig deeper into the bimetallic catalysts with some other types of studies. It was becoming possible to do this in the early seventies, whereas it hadn't been a decade earlier when I'd had to depend solely on chemical probes to characterize the catalysts.

One of the things that made it possible was a development going on in the Boeing laboratories in Seattle, Washington. Farrell [W.] Lytle out there had been playing around with x-ray absorption spectroscopy for about a decade before I ever met him. He was pursuing that in the sixties when I was investigating bimetallic catalysts. Lytle made some very important contributions in x-ray absorption spectroscopy. He was particularly interested in the fine structure that you see in an x-ray absorption spectrum beyond an absorption edge. What you see after an absorption edge are some oscillations in the absorption coefficient, a periodic type of behavior, which extends maybe a thousand electron volts beyond the edge. If you are looking at the copper K-absorption edge, which is seen at an x-ray energy of about 9KeV—nine thousand electron volts—you are observing absorption of radiation associated with the ejection of a K shell electron from a copper atom. For about a thousand volts beyond the edge you'll see oscillations in the absorption coefficient.

This phenomenon is very old. It goes back to the twenties, but for a long time nobody did anything with it. People wondered about what caused it and some theories were advanced, mainly by physicists in the early thirties. There was always the question, "Are the oscillations due to some sort of ordering in the material? Does the ordering have to be long-range, or is short-range sufficient?" It was felt that the oscillations had to be tied into structure somehow, and people developed long- and short-range order theories. Long-range order is the kind of thing that you have in crystalline materials which you would ordinarily investigate with x-ray diffraction. By contrast, the ordering in a non-crystalline—amorphous—material, or in very small crystallites, is of the short-range kind.

Lytle cast some light on this. He started working with x-ray absorption in the early sixties, doing experiments at very low temperature. A lot of the work which had appeared many years ago had been done around room temperature. When Lytle started doing experiments down around liquid nitrogen temperature, he found that the oscillations were more pronounced at lower temperatures. This made the experimental determination of the oscillations easier, because the oscillations were small and low temperature experiments greatly magnified them. Another thing he noticed was that he could observe these oscillations for amorphous materials as well as crystalline materials, so he had good experimental evidence that this was a short-range order phenomenon. He didn't need long-range order to observe oscillations. Those were very important findings. Then Lytle came up with a sort of zeroth theory to explain some of this, and made a rough attempt to derive information like interatomic distances, coordination numbers, et cetera, out of analyses of these oscillations. These were enormous contributions in my mind, and Lytle appreciated that he had something here that might be important for determining the structure of materials. He got together with a physicist from the University of Washington named [Edward A.] Stern and a graduate student named [Dale E.] Sayers. The three of them collaborated and developed a theory of electron scattering to account for the oscillations in absorption coefficient, and they developed some improved techniques for getting structural information from them.

Well, it wasn't long after that that they published a paper in *Physical Review Letters*, which I was not aware of at the time (8). The paper was published in 1971, the same year that our platinum-iridium catalyst was commercialized. I met Lytle shortly after, simply by chance. He started talking to me about this phenomenon of extended x-ray absorption fine structure, which was known by the acronym, EXAFS. When he started talking to me about it, he got me very excited, because I was ripe for it. I was trying to decide, "What do I want to do now?" and one thing that appealed to me was doing something to try to dig deeper into the structure of bimetallic clusters. He started telling me that he could get structural information on amorphous materials, that he didn't need long-range ordering in a material. He got through to me despite the fact that I knew nothing about x-ray absorption spectroscopy. He had the ability to explain it in the most elementary way. He explained that the crucial thing that he could do was learn something about the local environment about an atom.

He said, "If I have an atom A and I have neighboring atoms of A and of B around it, I can tell you how many A atoms and how many B atoms I have coordinated to it." I said, "That's what I need. Suppose I have a nickel-copper bimetallic cluster. I'd like to know how many copper atoms are coordinated to nickel, and how many nickel atoms are coordinated to nickel. I'd like to know the interatomic distances as well." He said, "I can also tell you something about the disorder." In a crystal, atoms are vibrating back and forth and the vibrations are termperature dependent. That's why the EXAFS oscillations depend on temperature. He said, "We can learn something about that." I thought, "Oh, my gosh, we can get a huge amount of information out of this."

I said, "I've got to learn something about x-ray absorption spectroscopy. How do I go about this? I don't know anything about it." I figured the only sensible thing to do was to somehow get a collaboration going with Lytle because he knew what he was doing, and maybe I could learn from him and we could develop techniques that could tackle catalyst problems. I talked to Lytle about that, and he was very much interested in it, because he had a technique and he wanted to address an important problem with the technique. I had the problem, so we were natural collaborators.

There was a researcher at Exxon named Grayson Via whom I had worked with doing x-ray diffraction on lots of problems, and I thought it would be good to collaborate with him as well. Then I had to sell the people in the company on allowing me to collaborate with a scientist at Boeing. There wasn't much of this type of collaboration between scientists in different companies. People tended to keep things to themselves. I approached my management: "Let's get Lytle in here on a consulting basis that will allow us to have a collaboration. Boeing's not interested in catalysts. We don't have to worry about proprietary matters." I had to convince my management, and finally they let me go ahead. We got this collaboration going, and Lytle came here and showed Grayson and me how to set up some equipment of the type that he had used at Boeing to do this. We modeled an apparatus after his apparatus, and we started doing some experiments.

We were using ordinary x-ray tubes and the flux of the x-rays was not great enough to do the experiment in an ideal way. With the fluxes we could get, it would take us three or four weeks to do an experiment on a single sample. We were measuring small oscillations in absorption coefficients, and to obtain statistically significant results, it was necessary to collect data over long periods of time. With the ruthenium-copper system, it took us about a year, and we still weren't completely done with the experiments we wanted to do, but we did get some convincing data that showed we were on the right track. We could definitely show that we could get information on the structure of these bimetallic clusters.

That was an important development, because it said that we could take these dispersed catalysts and learn something about their structure with a physical probe like this. In other words, we were no longer limited to information obtainable from experiments on adsorption and on catalytic reactions. We could now get a different kind of information and see how consistent it was with what we learned from chemical probes. I was very excited about it, but it was very slow going. We finally said, "Okay, we'll look at a couple of systems, but we're never going to be able to look at all the things of interest to us."

Then we were lucky again. There is a high-energy physics facility at Stanford, a synchrotron, and a by-product of the operation of the synchrotron is x-ray radiation with a range of energies. You have a whole spectrum of x-ray energies, and the flux is about a million times greater than it is from an x-ray tube. What does that mean? If you have access to the Stanford facility some way, you can do experiments there in a few minutes that would otherwise take three or four weeks to complete. It turned out that at Stanford they were interested in making use of this by-product. This by-product radiation was probably a pain in the neck to some of the physicists, but to us it was great, and they made it possible for us to go out there and do experiments using this synchrotron radiation. In 1975, about Christmas of that year, we went out there and did our first experiments with the Stanford synchrotron source. Over the next ten or fifteen years we did a lot of work there. At Brookhaven on Long Island a similar facility opened up later on, and we were able to do experiments there as well. In all of our experiments we can look at samples in an environment similar to that encountered in a catalytic reaction. Over a period of time we looked at lots of bimetallic cluster catalysts, we got information on coordination numbers and interatomic distances, and we could see from the EXAFS data when one of the components was tending to segregate at the surface of the cluster. This was a very productive and important effort, and we devoted a lot of time to it.

As I said, I could have taken another course in my research and explored many different kinds of chemistry over bimetallic cluster catalysts, but I preferred to pursue the application of x-ray absorption spectroscopy to the study of the structures of bimetallic clusters. Somebody else might have taken a different course, and he might have had an equally productive or maybe more productive experience. Who can say? I'm still interested in x-ray absorption. In addition to the information you get from the fine structure beyond the edge, which tells us about crystal structure or cluster structure, there are structural features in the absorption edge itself which tell us something about the electronic properties of a material. At an absorption edge, there is an abrupt discontinuity in the absorption coefficient. The energy of the incident x-rays is high enough to eject a particular electron from the atom. In the case of copper, it takes 9KeV to eject an electron from the K shell of a copper atom. The absorption of this energy is responsible for the edge. The edge itself has features which relate to electronic properties of the system. We can learn about things like oxidation states in a system. We can learn something about the d-band filling that I was talking about. In fact, we can correlate certain structural features in the edge with the degree of filling of the d-band in the pure metals. I've gotten involved in a lot of work on this topic, which has been quite interesting.

In the course of the last twenty years I've gotten involved in another area that was also new for me, through a collaboration with a physicist in a university. I got involved in nuclear magnetic resonance studies of catalysts. In the mid-1970s, I was giving a lecture at the University of Illinois. There were chemists, physicists, and materials science people in the audience. Professor Charlie [Charles S.] Slichter, who has done a lot of work with nuclear magnetic resonance during his whole career, was in the audience. I was talking about dispersed metal catalysts, and after the talk he told me that he had always wanted to study the nuclear magnetic resonance of small (10-100 Ångström) metallic particles, but he didn't have the particles.

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SINFELT: He was intrigued by the fact that the catalysts contained metallic clusters with sizes in the region of interest. He asked me whether I would be interested in working with him on this problem, applying nuclear magnetic resonance for the characterization of small metal crystallites. I knew nothing about NMR at the time, but I knew Charlie Slichter was darn good at doing NMR, and I figured, "If I'm going to learn anything about NMR, why not learn it from the best?" [laughter]

The first thing we wanted to do was study the effect of the size of a platinum crystallite or cluster on its NMR properties and see whether we could distinguish the platinum at the surface from the platinum inside a metal crystallite. For a whole series of platinum catalysts prepared in our laboratory, we estimated crystallite sizes by gas adsorption. We had a series where the average crystallite size varied by a factor of ten or thereabouts, ranging approximately from ten to one hundred Ångströms.

Slichter got some students working on this problem. It wasn't like doing NMR on liquids where the resolution is high. It's solid state NMR and one observes broad NMR lines. Platinum NMR is very difficult to do, but with extensive signal averaging to obtain statistically significant data, Charlie's students were successful in obtaining NMR spectra for the platinum.

The shape of the NMR line for platinum varies markedly with the size of the platinum crystallites. For the catalysts with the largest platinum crystallites, the NMR spectrum exhibits a pronounced peak at the frequency where such a feature is observed for crystals of bulk platinum. As the average platinum crystallite size decreases, the intensity of this feature in the NMR spectrum decreases, and a peak at another frequency becomes increasingly evident.

We associated this latter peak with the surface platinum. For the samples with the smallest platinum crystallites, this peak was the only one observed. There was no peak at the frequency characteristic of bulk platinum. The extent of hydrogen chemisorption on the platinum in a catalyst correlated nicely with the spectral area of the "surface" peak normalized to the spectral area of the entire NMR line. When I saw this correlation, I said to myself, "I may not have been totally convinced before this, but now I believe that NMR can contribute important information in the characterization of catalysts."

We've been working together on NMR now for more than fifteen years. It's turned out to be a very good collaboration. In addition to characterizing the catalysts, we've been studying the structures of the species formed on the surface in the adsorption of gases such as acetylene, ethylene, and carbon monoxide on the metal clusters or crystallites present in the catalysts. We've also been studying the reactivity of adsorbed molecules, to obtain insight into catalytic reaction mechanisms. This work has been an industry-university collaboration which evolved purely from the scientific interests of the individual researchers. It doesn't depend on grants from Exxon. Collaborations initiated by the scientists themselves have the desirable feature of strong commitment on the part of the participants. This commitment is essential for a successful collaboration.

A particularly important feature of the work is its emphasis on <u>real</u> <u>catalysts</u>. We don't have to look at idealized materials like single crystals. The collaboration with Charlie is good for me because it keeps me in contact with the academic world. I talk with Charlie's students on a continuing basis. They send me their papers and theses. It's been a very good experience for me. It keeps me alive, if you will. I think this says something about the value of the collaboration.

I'm grateful that Exxon has permitted me to be a party to the collaborations with Farrell Lytle and Charlie Slichter. I have been greatly enriched by the experiences. There are many intangible benefits. I have been able to stay at the forefront of important areas, because I have been able to participate in their development. I think this type of collaboration provides an ideal way for a scientist to advance his own research interests through the application of new developments in scientific fields other than his own. It is important to become deeply involved. Maybe that's hard; it may mean that one has to do a lot of studying in an area which is totally new. But I think it's necessary to make collaborations really fly.

BOHNING: You stayed in the research area your entire career here. The latest date I have is 1979, when you got the title of senior scientific advisor.

SINFELT: I still am senior scientific advisor. That's the highest position on the scientific ladder in the company.

BOHNING: Did this scientific ladder exist when you came?

SINFELT: No. I commented earlier that Murphree had given a talk at the technical committee meeting about the importance of being more involved in basic science and being a part of the scientific community through publication. The idea of advancement along technical as well as administrative lines evolved about that same time. It was not long after I came into the company that a formal technical ladder was instituted. There may have been an informal program before which had never really been spelled out. A formal technical ladder was introduced sometime after I joined the company, but I don't remember exactly when.

BOHNING: What is Exxon's attitude toward patents? What kind of reward system do they have for patents?

SINFELT: When a patent is applied for, an inventor gets a certain cash award. Then when a patent is issued by the U.S. Patent Office, the inventor gets an additional award. Patents are regarded quite highly. There has always been a question of how well you should reward patents relative to publications, and I think this has changed from time to time in the organization. When I first joined the company, patents were valued much more than publications. Then we had a period where publications were also valued highly. I would say that we are now seeing a return to the attitude that patents are more valuable than publications. These things tend to change, and I suspect this is probably going on all over the place—at Bell Labs, IBM and other places as well.

BOHNING: As I said at lunch, you had the ideal situation of really having an academic position in an industrial setting.

SINFELT: Well, I've had the best of both worlds in lots of respects, I agree. I hate to distinguish fundamental from applied research—I don't like to do that—but I have had the opportunity to do fundamental research and at the same time be involved in significant commercial developments, which for me has always been very exciting. There's something nice about being able to develop something that has practical use. There's a special feeling of satisfaction for the scientist who is involved.

This year marked the seventy-fifth anniversary of the founding of ER&E. A videotape was prepared for the occasion, and various people in the company were asked to participate in short segments to express some philosophy. I said something like this, "There's an aura of excitement about science in Corporate Research, and I think this is very vital for basic research. When the science eventually results in a significant commercial application, there's a special feeling of satisfaction which accompanies the excitement. For the industrial scientist or engineer, this is the ultimate reward." As far as I'm concerned, that's really what it's all about for people with the kind of interests that I have.

BOHNING: I'd like to look at the agenda list that I sent you. Although you've covered almost all of it, there are a few things that I would like to touch on. Let me ask you a general question about scientific innovation. What does scientific innovation mean to you, as it was derived from your experience?

SINFELT: When I think of scientific innovation, I think of new scientific concepts. To me, it has to be some new way of looking at or thinking about something. If I had to pick something of mine that I thought was a scientific innovation, I would say the idea of a bimetallic cluster. It was a new idea with important implications for catalysis and solid state science.

BOHNING: What has your experience been with teamwork in the industrial and scientific settings?

SINFELT: Teamwork is best when it originates with the people doing the work.

BOHNING: Rather than forced by management?

SINFELT: Yes. I've had lots of collaborations, and the ones that really work and achieve something are those where we simply have a couple of scientists getting together and working on it because they're interested in it. They aren't being manipulated into working on something. I don't think it's easy to put together teams or manipulate things and achieve the best scientific results. The natural collaborations are the ones that tend to work. My experience has been that way with the outside collaborations, too. The outside collaborations I've described were successful because the scientists were comfortable with each other, and both had something to bring to the problem. One participant in the collaboration did not feel threatened by the other, because each felt he had an important role to play; both participants made an important contribution.

I don't think collaborations are very good unless both participants contribute significantly to the work. In science, a meaningful collaboration is one in which both people are doing science.

BOHNING: What changes have you seen in R&D support by the company during your career? You've talked about part of that, starting with Murphree.

SINFELT: From a broad perspective of R&D that includes development with research, I would have to say that Exxon has been a leader in such activities for an awfully long time. We recently celebrated the seventy-fifth anniversary of Exxon Research and Engineering Company, so there has to have been a sense, starting back around 1919, that it was important to be doing research and development for the good of the corporation. The company has been doing it continuously for a long time and has had a very impressive record from the standpoint of the development of processes. The company has always tried to develop its own processes, and processes like fluid catalytic cracking and hydroforming have been major innovations in the industry.

For most of the time that I've been with the company, there has been an increasing interest in longer-term, fundamental research. It's just been in recent years that we've seen a decline of interest, but I'd say overall that the company is still interested in research. Today people are much more sensitive to, as they say, the needs of the customer out there, trying to get the potential customer involved as early as possible in a research program.

There's much more of this today than there was ten or fifteen years ago when fundamental research was at its peak in an organization like ours. There's probably a happy medium you have to come to, and the issue we have to be concerned about is the strong possibility that we are going too far in the direction of downgrading basic science.

BOHNING: That leads to the next question, which is what is important for the future of R&D in industrial chemistry settings?

SINFELT: I would think right now, the way things are going, that we don't lose our appreciation of the value of basic science over the long term in these companies. A few years back, in corporate laboratories in particular, we tended to elevate science for science's sake to a pretty great height. Now we have to be careful that we don't lose that entirely. I feel that we are now being overwhelmed by the perceived needs of the customer. Although we have to be interested in the business aspects when we are doing research in industry, there is a real danger of smothering scientists with this sort of thing. The quality of the science can suffer accordingly.

We have to be careful that we don't lose our regard for the intellectual component of science itself, because, in order for scientists to do a good job, they have to be interested in it for the intellectual content. I'm absolutely convinced of that. It's not enough just to say, "I'm going to make a superior product for Exxon or Chevron or American Oil," or what have you. That's fine, but we can't sacrifice being interested in the intellectual component. Otherwise, the science ultimately will not be first-rate. Why does a scientist become a scientist in the first place? If he's going to be a good scientist, he has to be primarily interested in the intellectual content of science, rather than business concerns. When we do industrial research, we do have a responsibility to do something that's going to be in the employer's interest. I absolutely believe that, otherwise I wouldn't be here. But I also believe that if science has any role in this business, we have to be careful to foster an environment which is conducive to good science. Some companies might be able to get by without any basic science at all. Maybe they can. But if they say, "I want to have science be a part of it," they're probably kidding themselves unless they have the right kind of environment conducive to good science.

BOHNING: What did it mean to you to win the Perkin Medal?

SINFELT: The Perkin Medal has been conferred for a long time, since 1906 or so. For somebody who's worked in an industrial organization, this has to be close to the ultimate recognition for an achievement, so it's meant a great deal to me. If we look at the Perkin Medal winners, there's some very good company. It's interesting that it's been a very varied type of group. They include some outstanding academic scientists as well as industrial scientists, so we have people who are extremely good at the most fundamental aspects of science, as well as the most applied.

BOHNING: That brings me to the end of my list of questions. Is there anything you'd like to add that I haven't covered?

SINFELT: I think the most important thing about science is that to do good science, people should really have fun doing science. I think that's the thing that ultimately keeps us going in this kind of career. People sometimes develop the attitude that it's a little frivolous to talk about fun, but it's very important. I've found that most people whom I have a very high regard for in science have fun doing science, and they will be right up front about the fact that it's important to them. I don't see anything wrong with that. If you're having fun doing science, that tells you something about the fact that it has a powerful attraction. It's something that makes it real and believable.

After all, I don't think the average scientist is in it for the money. I've been fortunate to have worked in a time when scientists became increasingly rewarded for their efforts. We've been fortunate, but I don't think most people who go into science do so for that reason. If money is their only interest, there are other opportunities that might serve better. They have to have a genuine interest and really derive fun from it. That's quite important.

I genuinely like science. I don't have many hobbies outside of it. When I go home, I often read about science. I'm interested in it. I would rather read about science than a lot of other things, even at home, because I can't think of anything that interests me more.

[END OF TAPE, SIDE 8]

SINFELT: You asked the question about the way chemical R&D is going. I just hope we don't lose sight of the excitement that an individual has about the intellectual content of science. That's very important to doing good science.

I look at it from the point of view of science. The scientist has an obligation to his employer, whether he's working for an academic institution or an industrial organization, to do research with the best interests of the institution at heart. I've tried to do that my entire career. I haven't done science here to the exclusion of thinking about company problems. As a matter of fact, I've tried very hard to integrate science with company problems, because I've found it's not all that hard to do. Science questions abound in almost anything. If we set our minds to it, we can find some way of taking a scientific approach to all kinds of problems.

BOHNING: You mentioned the fact that even when you go home and read, you like to read about science. I was struck by the fact that you wrote an article for *Scientific American* (9), and I was wondering what your experiences were with writing something for the educated lay man, as it were, not the specialist.

SINFELT: I liked doing that. I'll tell you, it's not always easy, but it makes you see it in a different perspective, and sometimes doing that helps you understand your own science, your own problem, better. I don't find it easy to write. I find it difficult to write, because I try to think about and communicate with my audience rather than the specialist.

That's one of the criticisms I have about a lot of what is published in science. Some scientists tend not to have the general readership at heart, and it can hurt science. We develop so much specific jargon in specific areas, and we fall back on that when we write papers. We don't try to explain enough. Even for the reader who is a well-accomplished scientist, we don't explain well enough, in my view, in many cases. When I write a paper, I try to do that. I hope I've been successful, but it's hard work. Science can get too specialized. Relying on the jargon is not in the interests of a specialty, either, because pretty soon it shuts out a lot of other people in science and reduces the impact of the work.

BOHNING: One of the things that I was struck by in the *Scientific American* article was the chart which illustrated the relationship between catalytic activity and periodicity. The way that particular graph was set up was so striking; the relationship just leaped out at you. There's an incredible relationship that I don't think I've seen quite in that way before.

SINFELT: I've done a lot of that kind of work, and that is the kind of thing that reveals the scientific underpinnings of catalysis. It's not voodoo. [laughter] The periodic table is the guiding light in catalysis as it is in chemistry in general. We can't throw away the periodic table when we're doing catalysis. [laughter]

BOHNING: I was a professor for a long time, and we would go through introductory chemistry, [Dmitri] Mendeleev, and the periodic table, and we would point out how new elements were discovered based on his predictions. Then I'd like to say, "Now, here are all these other things that have happened because of the periodic table," but it's not easy to come up with examples. They're out there, but the textbooks don't give them to us; we try looking for them ourselves, and it's not easy to come up with them. In these interviews I've uncovered several cases in which the periodic table was a crucial factor in the thinking of the individual.

SINFELT: Definitely. That's something that I'm doing all the time.

BOHNING: Could you tell me a little bit more about the origin of your book on bimetallic catalysts (10)?

SINFELT: The book on bimetallic catalysts that I wrote was published in 1983. This book was the first in a series of Exxon monographs. The program was brought about through the efforts of Ed David, who was then the president of ER&E. He thought it would be a good idea for industrial researchers to communicate with the larger scientific community about industrial research that had been conducted over a long period of time. The accounts were written from a personal point of view.

The purpose was to discuss the research that was done, the approaches that were taken, what the impact of the research was, and what might have been learned about the science along the way. You'll note the subtitle, "Discoveries, Concepts and Applications," which are indeed the things I wanted to emphasize—some of the aspects of research—what's the nature of discovery in an industrial environment, what do I mean by the concepts, and how are the concepts evaluated and ultimately applied. Ed David thought this would be very useful for communicating with the scientific community, and he hoped that this would have an effect on education.

One of the most satisfying things that happened to me in relation to this—it's happened on at least one occasion, and maybe on a couple of occasions—I got a telephone call from a graduate student in a university, whom I had never heard of, who had seen my book and had read it. He asked me a specific question about it, and had devised a research program for a thesis based on something that he had read in the book. If that has happened even once, if it has had any effect to interest a student and spur him to follow up, then it has made the writing of that book worthwhile. Actually, I've found several instances where students have been interested in the book, and in a couple of cases have pursued certain issues in the book in considerable depth. I was amazed, because the book is not light reading. [laughter]

BOHNING: How many other monographs were published in this series?

SINFELT: I don’t know exactly, but there were a few. I’m guessing, but maybe three to six. That project is not being pursued anymore, and it’s too bad. I spent a lot of my own time writing my monograph, because I wanted to do it. Ed David asked me whether I would do it, and I thought it was a worthwhile activity. It helped my own thinking about a lot of things, particularly broader issues of scientific research—how do you think about scientific research, how do you approach it, and how do you communicate it? You try to analyze what you’ve done, to communicate it in the best way you can, and to reflect on ways you might proceed with the research program again to improve it—that’s a good disciplinary exercise.

The book encompasses about a twenty-year period. Over that period we see the evolution of approaches to problems. As I mentioned, I had to begin my studies on bimetallic clusters with chemical probes, and then things advanced enough during that period so that we could see physical probes evolving to work on the problems. There are good examples showing how science progresses in the course of research on a problem.

BOHNING: Well, I want to thank you for taking the time this afternoon, and thank you for lunch. I’ve enjoyed talking to you, and I appreciate your time.

SINFELT: I’m delighted that you came. I have enjoyed talking with you very much.

[END OF TAPE, SIDE 9]

NOTES

1. Carl S. Marvel, interview by Leon Gortler and Charles Price in Wilmington, Delaware, 13 July 1983 (Philadelphia: Chemical Heritage Foundation, Oral History Transcript #0003).

2. Herman T. Briscoe, *General Chemistry for Colleges,* 3d ed. (Boston, New York: Houghton Mifflin Company, 1943).

3. Arthur A. Noyes and Miles S. Sherrill, *A Course of Study in Chemical Principles* (New York: Macmillan Co., 1923).

4. Vladimir Haensel, interview by James J. Bohning in Amherst, Massachusetts, 2 November 1994 (Philadelphia: Chemical Heritage Foundation, Oral History Transcript #0115).

5. John H. Sinfelt, "Some Reflections on Catalysis" (Perkin Medal Address), *Chemistry and Industry*, 11 (June 4, 1984): 403-6.

6. *Chemical Heritage Foundation Oral History Project, The Society of Chemical Industry Project: Questionnaire for SCI Interviewees*. See Chemical Heritage Foundation Oral History Research File # 134.

7. J. H. Sinfelt, J. L. Carter and D. J. C. Yates, "Catalytic Hydrogenolysis and Dehydrogenation over Copper-Nickel Alloys," *Journal of Catalysis*, 24 (1972): 283-296.

8. D. E. Sayers, E. A. Stern and F. W. Lytle, "New Technique for Investigating Non-Crystalline Structures: Fourier Analysis of the Extended X-Ray-Absorption Fine Structure," *Physical Review Letters*, 24 (1971): 1204-1207.

9. John H. Sinfelt, "Bimetallic Catalysts," *Scientific American*, 253 (September, 1985): 90-98.

10. John H. Sinfelt, *Bimetallic Catalysts: Discoveries, Concepts, and Applications* (New York: John Wiley & Sons, 1983).

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