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DAVID M. HERCULES

Transcript of an Interview
Conducted by

David C. Brock and Arthur Daemmrich

at

Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy
New Orleans, Louisiana

on

20 March 2002

(With Subsequent Corrections and Additions)

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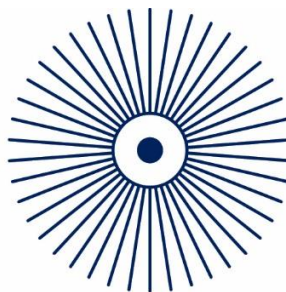
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DAVID M. HERCULES

1932 Born in Somerset, Pennsylvania, on 10 August

Education

1954 B.S., chemistry, Juniata College
1957 Ph.D., analytical chemistry, Massachusetts Institute of Technology

Professional Experience

1954-1957	Massachusetts Institute of Technology Assistant, Department of Chemistry
1963-1968	Assistant Professor, Department of Chemistry
1968-1969	Associate Professor, Department of Chemistry
1957-1960	Lehigh University Assistant Professor, Department of Chemistry
1960-1963	Juniata College Associate Professor, Department of Chemistry
1969-1974	University of Georgia Associate Professor, Department of Chemistry
1974-1976	Professor, Department of Chemistry
1976-1995	University of Pittsburgh Professor, Department of Chemistry
1980-1989	Chairman, Department of Chemistry
1995-present	Vanderbilt University Centennial Professor of Chemistry, Department of Chemistry
1995-present	Chairman, Department of Chemistry
1998-1999	Assistant to the Provost

Honors

1974 Fellow, Guggenheim Foundation
1981 Lester W. Strock Medal, Society for Applied Spectroscopy
1983 Alexander von Humboldt-Stiftung Prize

1986 Fisher Award in Analytical Chemistry, American Chemical Society
1987 Benedetti-Pichler Award, American Microchemical Society
1989 Eastern Analytical Symposium Award
1993 Adamson Award in Surface Chemistry, American Chemical Society
1996 Pittsburgh Spectroscopy Award, Pittsburgh Conference on Analytical
Chemistry and Applied Spectroscopy
1997 Award, American Chemical Society, Pittsburgh Section

ABSTRACT

David M. Hercules begins his interview by discussing his childhood and education. He describes his youth in Somerset, Pennsylvania, and his childhood curiosity with science. When he was in high school, he continued to develop an interest in chemistry. Harold B. Brumbaugh and his chemistry teacher, William B. Howe, convinced him to attend Juniata College, a liberal arts school in central Pennsylvania which had a well renowned chemistry department. While at Juniata, he honed his interest in analytical chemistry. He was exposed to a few different types of spectroscopy at Juniata, and was able to attend tours of major academic and industrial labs in Pennsylvania and Delaware. He chose to attend Massachusetts Institute of Technology [MIT] for graduate school, and selected Lockhart B. Rodgers as his graduate advisor. He did his thesis work about the emission spectra of naphthalene compounds. While at MIT, Hercules worked as a teaching assistant for Stephen G. Simpson. After graduation, Hercules decided to pursue an academic career.

Hercules began his professional career at Lehigh University as an assistant professor. He describes how he built a spectrofluorometer at Lehigh and did research on photo-induced luminescence. When he worked at Lehigh, he had summer positions at United States Steel Corporation and Sun Oil Company. After three years at Lehigh, he returned to Juniata and conducted undergraduate research. He became an assistant professor at MIT after three years at Juniata. While at MIT, Hercules used a wide array of instrumentation, including one of the first ESCA [electron spectroscopy for chemical analysis] instruments. He corresponded and collaborated with Kai Siegbahn from Uppsala University in Sweden. Hercules used ESCA and XPS [x-ray photoelectron spectroscopy] to investigate a variety of phenomena, including heterogeneous catalysis. He also consulted for the Central Intelligence Agency, Instrumentation Laboratories, W.S. Merrill and Company, and Exxon Mobil Corporation.

Hercules moved to the University of Georgia after six years at MIT. He then describes the position of analytical chemistry within the chemistry department and the variety of instrumentation that he was able to work with in Georgia. He continued to be interested in catalysis and attended a International Catalysis Society Meeting in Florida. After receiving a Guggenheim Foundation fellowship, he was able to study at Northwestern University with Robert L. Burwell Jr. To continue his work on catalysis, Hercules moved to the University of Pittsburgh [Pitt] after seven years in Georgia. He got to work with an impressive variety of instrumentation at Pitt, and consulted for W.S. Merrill and Exxon. He helped develop and establish the surface science center at Pitt, and helped recruit John T. Yates Jr. to be the head of it. He used many different types of instrumentation, including SIMS [secondary ion mass spectrometry], ion scattering spectroscopy, and Auger electron spectroscopy. At Pitt, he gained interest in mass spectroscopy and began to consult for Leybold-Heraeus. After that, Hercules worked with a LAMMA [laser microprobe mass analyzer] and the MALDI [matrix assisted laser desorption/ionization] process. He served as chair of the chemistry department for nine years and won the Alexander von Humboldt Stiftung Prize. After nineteen years at Pitt, Hercules transitioned to working at Vanderbilt University. He describes the state of the Vanderbilt chemistry department and his place within it.

To conclude, Hercules recounts his role in various conferences, including different Gordon Research Conferences [GRC], as well as the Asilomar Conference on Electron Spectroscopy and the Namur conference. He also recalls the funding of the GRC on electron spectroscopy. He ends the interview by reflecting on his current research on polymers using SIMS and MALDI and on the state of analytical chemistry today.

INTERVIEWERS

David C. Brock is Program Manager for Educational and Historical Services at the Chemical Heritage Foundation in Philadelphia. He is currently a Ph.D. candidate in the History Department, Program in the History of Science at Princeton University. In 1995, Mr. Brock received his M.A. in the History of Science from Princeton University and in 1992, he earned a M.Sc. in the Sociology of Scientific Knowledge from the University of Edinburgh.

Arthur Daemmrich is a policy analyst at the Chemical Heritage Foundation in Philadelphia. He holds a Ph.D. in Science and Technology Studies from Cornell University and has published on biotechnology policy and politics, the sociology of medicine, and pharmaceutical drug regulation. In his research, he brings long-range perspectives to bear on the analysis of globalization, risk, health, and environmental policy. Daemmrich has held fellowships from the Social Science Research Council/Berlin Program for Advanced German and European Studies, and the Kennedy School of Government at Harvard University.

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INTERVIEWEE: David M. Hercules

INTERVIEWERS: David C. Brock and Arthur Daemmrich

LOCATION: Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy
New Orleans, Louisiana

DATE: 20 March 2002

BROCK: I'd like to begin at the beginning. You were born in Somerset, Pennsylvania. Could you tell us a little bit about your family background and your childhood?

HERCULES: I have one sister. My father was a salesman and then a manager of a furniture store. I lived in Somerset until I was fifteen, at which point Dad was transferred to be the store manager in Lewistown, Pennsylvania. That was nice because later on I got summer jobs delivering furniture. [laughter]

BROCK: Nice light work in the summer heat. [laughter]

HERCULES: Yes. We found a correlation between the size of refrigerators and how high people lived in buildings without elevators! [laughter]

I graduated from high school in Lewistown. I went to Juniata College as an undergraduate, which is not far from Lewistown. My father was then transferred to Altoona, Pennsylvania, as store manager and stayed there for a considerable amount of time. I basically roamed around central and western Pennsylvania.

In 1954, the fall after I graduated from Juniata, I went to MIT [Massachusetts Institute of Technology] as a graduate student. During that summer, I worked for U.S. Steel's [Corporation] research labs in Pittsburgh. When it came time to pick a thesis advisor at MIT, I picked Lockhart B. [Buck] Rogers as my thesis advisor. Much to Buck's chagrin, I spent my first summer at MIT working for Sun Oil [Company, Inc. (Sunoco)]. I got my Ph.D. from MIT in 1957. I started my academic career immediately, although I worked for two summers. When I was at Lehigh [University], I worked at the Experimental Station at DuPont [E.I. DuPont de Nemours, and Company] during the summers. I always liked the idea of getting some experience in industry, but I was never tempted to permanently remain there.

DAEMMRICH: You worked at a steel plant in Pittsburgh in the early 1950s. What was that like?

HERCULES: It was not a steel plant. It was their research lab. That was between my senior year at Juniata and my first year at MIT, and I was doing electrochemistry. I was in an analytical group, which was sort of a non-routine analysis group. They had people who did sulfur and steel, and they had groups who did lots of analyses. If nothing fitted into the conventional scheme of things, my group got it. In order to keep busy when we didn't have too many weird things to do, we also ran stripping curves on tin plates to measure their quality.

I remember one morning, we came in and there were several bottles of scotch and cans of soda sitting there. In the president of U.S. Steel's plane, they had been having scotch and soda, and when they mixed the scotch and the soda, it turned green. They immediately wanted to know why, and it turned out it was because of a defective U.S. Steel tin plate in the soda cans. [laughter] They had extracted some iron which, when combined with the stuff in scotch, formed an iron 3-chelate that is green.

BROCK: I'd like to go back a little bit and talk about the development of your interest in chemistry in high school and then the course of your chemical education at Juniata.

HERCULES: For some reason, I always had an interest in chemistry. I was one of those kids who experimented with building radios. One very dear aunt of mine, over the objection of my parents, brought me a chemistry set when I was in fourth grade. Somerset had a population of five thousand people, and everybody knew everybody else. My mother knew all the pharmacists, so I went to the drug store and got a nickel's worth of this, a dime's worth of that, and a quarter's worth of something else. That worked until I decided to build an atomic bomb and asked for a dollar's worth of uranium. They didn't have it. [laughter]

I was always involved with things like that with various friends. My friend and I lived diagonally opposite from each other on our block. We went to the local telephone company and got some old wire, which had some breaks in it, and used a circuit tester to patch it up. Then we strung our own private telephone line between our houses. We used to build our own fireworks displays for the 4th of July which were totally illegal, but because my buddy's brother was a deputy sheriff, we were okay.

In the spring of my junior year of high school, when colleges started looking for potential students, a man named Harold [B.] Brumbaugh came from Juniata. He got me interested in Juniata and turned out to be a long-term friend. I was interested in physics and chemistry, but the physics teacher I had was a flunked-out football coach, whereas my chemistry teacher was a graduate of Juniata College and had worked during the War with the chemical corps. He was very knowledgeable. His name was William [B.] Howe. He and Harold Brumbaugh convinced me to go to Juniata, which had, even at that time, a reputation of producing excellent undergraduate chemists.

BROCK: At Juniata you studied chemistry. Did you develop a strong interest in analytical chemistry at that time?

HERCULES: I was developing an interest in it. At Juniata, I basically assumed that I was going to go to graduate school. I had three real interests in chemistry: analytical chemistry, biochemistry, and spectroscopy. I had a really outstanding teacher, Eva Hartzler, and she taught both analytical chemistry and biochemistry at Juniata. I finally decided that I would opt for analytical because I could do analytical spectroscopy. I was really not interested in quantum mechanics and a lot of the really serious computational stuff that went with being a hardcore spectroscopist. It seemed to me at the time that biochemistry was still developing. It was still what I refer to as gizzard squeezing, where you would basically get samples and try to figure out what they were. I couldn't get involved in that. Analytical chemistry served as a nice compromise between the two.

BROCK: Could you talk about spectroscopy during the period from 1950 to 1954? What instruments were you using? What was your vision of the big directions?

HERCULES: We really did very little spectroscopy. I mean, we did some spectrophotometry, but it was point-by-point. Most of the analytical stuff I did was either classical or electrochemistry. We used potentiometry and gravimetry by plating things out or polarography. We had a dropping mercury electrode polarograph.

We went on a lot of tours. We had a good program at Juniata for juniors and seniors called the industrial chemistry seminar. Since we were located approximately in the middle of the state, one year we would go to Pittsburgh, and the next year we would go to Philadelphia/Wilmington. We would actually spend three days or so touring various laboratories. We went through the Mellon Institute in Pittsburgh, which was probably one of the premier spectroscopy institutes at that time. That was the first time I met Foil [A.] Miller. Alcoa [Incorporated] had a lot of spectroscopy instruments including automated quantometers for aluminum analysis. The guy who was the director of research at U.S. Steel was a Juniata alum, so we visited them. They did a lot of spectroscopy, of course. On the other end of the state, we visited a number of the DuPont plants because Juniata had several alumni in the DuPont structure. We went to a [U.S.] Department of Agriculture laboratory in Philadelphia, so I knew what was going on there. I forget where else we went. Those tours helped generate my interest in spectroscopy.

BROCK: Building on what you did as a kid, were you tinkering with instruments or building new instruments when you were in college?

HERCULES: No, I didn't build instruments. I did undergraduate research, but my undergraduate research was done with a man by the name of Raymond [J.] Davis, who's still alive, actually. He was a student of Paul [M.] Emmett, and I did research in heterogeneous catalysis. I didn't do much instrument building. We built more apparatus related stuff than instruments. I really didn't get that heavily involved, one-on-one, with analytical instruments until I got to MIT.

BROCK: Could you talk about your graduate school decision? What places were you looking at, what attracted you to MIT, and what clinched the deal there?

HERCULES: I applied to four or five places. I forget which. Purdue [University] had a very good analytical chemistry program, so I applied there. I applied to Princeton [University], which had a very good program. I also applied to MIT, Cornell [University], and [University of] Minnesota, who had [Izaak M.] Kolthoff at the time. I thought of applying to Indiana, but I don't remember whether I did or didn't. Basically, the reason that I went to MIT was that, as a kid, I'd always wanted to go to MIT.

BROCK: [laughter] That's a good reason.

HERCULES: That's what really clinched the deal. My parents were smart enough to realize that I was not mature enough when I started college. When I got to MIT, I learned that I wouldn't have ever survived the first semester. I just wasn't mature enough to do that.

BROCK: In terms of the discipline that it would take to handle the weeding out system?

HERCULES: That's right.

BROCK: One of the first things that you did when you got to MIT was figure out who you were going to work with. Could you say more about your decision to work with Rogers?

HERCULES: I also got to know Dick [Richard C.] Lord early on. At that time they had a standard set of courses that you had to take. Within these, we had some selection. I opted to take Dick Lord's spectroscopy course. He and Rogers were very good friends. I was trying to figure out what would be the best thing to do because I didn't want to work specifically for Lord. He worked with vibrations and rotations, calculated the vibrational modes, and interpreted spectra, and I just didn't want to do that.

I spoke with Buck and Lord about fluorescence, about which I knew very little. Lord said that he felt that vibrational spectroscopy had developed into a plateau—those are my words, not his—and that electronic spectroscopy was really the area of spectroscopy where there was a lot of insight being gained.

That was when Mike [Michael] Kasha was starting to help to define which electronic transitions gave rise to UV absorption bands, which a lot of people didn't really know in those days. Lord thought that electronic spectroscopy would be a good choice, and in fact, it was. That was how I got involved in fluorescence with Buck. They had put together instrumentation which worked. In order for us to be able take spectra at low temperatures, I built a low-temperature cell. We had a UV-visible spectrophotometer, but that was essentially a commercial instrument. However, when I first started, I was still taking point-by-point spectra on the DU.

BROCK: Was that a recording instrument?

HERCULES: Beckman [Instruments, Inc.] put out a recording UV-visible instrument. That would have been 1954 or 1955, somewhere in there. I'm pretty sure it was a Beckman instrument. It was a great innovation in the laboratory. We made a spectrofluorometer by adapting a DU to be used as the monochromator.

BROCK: What was the basis of your thesis work?

HERCULES: The basis of my thesis was the interpretation of emission spectra from naphthalene compounds. I had learned a lot from Dick Lord about symmetry, and I used that information to interpret the spectra of the compounds. We came across a phenomenon which had been discovered a couple years earlier, it turns out. That was the idea that the acidity of molecules changes in the excited state. Theodor Förster and Albert Weller had published that in 1952 or 1953, somewhere around there, and I had not picked that up in the literature (1).

I was looking at the fluorescence behavior of either alpha or beta naphthol. They both showed excited state acidity. I wanted to get spectra in the acidic form because it was very different than the spectra you got in the ionized form because the electronic structure was different. I remember that I always ran the acidic form basically in pure ethanol. Then I took the basic form and I put in a basic ethanol-water mixture. One day, I forgot to put in the sodium hydroxide and was probably operating around a pH of 5 or so. I got a weird looking spectrum which had two molecular spectra in it—the molecular form and the ionized form.

I thought that was very strange. I thought that couldn't happen. It had to be one or the other. I had run a plot of UV absorption as a function of pH, so I knew exactly where the anion formed. I ran the fluorescence as a function of pH. It started out at a high pH and then came

down. All of a sudden, the molecular form showed up at about five or six pH units away from where it should have been and then eventually came down around pH 2. We puzzled about that for a while and finally interpreted it correctly. When I did a literature check, I found out that Förster and Weller had discovered that before. I was very interested in how the pH behavior of different kinds of aromatic hydroxy compounds and followed those findings. My thesis was full of all sorts of pH fluorescence curves and so forth.

BROCK: Were you using the hydrogen lamp and the DU?

HERCULES: No. We used a high-pressure mercury arc. GE [General Electric Company] put out a really intense high-pressure mercury arc. It was one that was so intense that there was an inversion at the 257 nm line, so we used the 313 nm line. Naphthalene compounds were great for us to use because they all absorbed there. We used a monochromator to isolate it and to keep the heat down, because we didn't want to just use a plain filter. It was a pretty decent instrument except for the prism instrument's poor resolution at longer wavelengths. Therefore, the first diffraction grating system I had was one that I built myself. We started at Lehigh.

BROCK: As you were heading towards the completion of your thesis research, were you thinking about your next steps? Can you tell us a little bit about your vision for your future career at that time and the sorts of decisions that you were making?

HERCULES: I wasn't really sure what I wanted to do with my career at that time. I had made a fairly clear decision to take an academic career. I had been the DuPont teaching fellow at MIT and got all sorts of offers to go everywhere throughout DuPont. That's how I ultimately ended up at the Experimental Station during those couple of summers. But I decided that I just didn't want to do that.

What I did know was that I wanted to be at an academic institution and I had very strong ties to Juniata College. I thought it would be nice to be at a liberal arts college, but I wasn't sure. I was not really sure I wanted to get involved in the big research game.

Jim [James J.] Lingane at Harvard [University] asked Buck to send him one of his people for an instructorship that Harvard always offered. I was offered that position but finally decided not to take it. I forget exactly how I learned about the position at Lehigh. It might have been through Buck or through an ad in *C&E News* [*Chemical and Engineering News*]. At any rate, I applied, interviewed, and was offered the job. That was how I ended up at Lehigh.

DAEMMRICH: You mentioned the summer job in the steel research lab. Did you say that you had at least one or two other summer positions?

HERCULES: Yes. Those were after I was at Lehigh. My first summer position was at U.S. Steel. My second summer position was with Sun Oil in Philadelphia after my first year of graduate school. I spent the summer building an automated distillation system for them. That was fun.

BROCK: What was it about the big research game that wasn't attractive to you? Did you know what sort of research you wanted to do?

HERCULES: Yes. I'd only been exposed to the big research game while I was at MIT, which was a pretty intense place. There wasn't anything that I intrinsically disliked about the idea of big research, since I ended up doing that. At that point in my life, I was not really ready to make that kind of commitment. I wasn't sure if I wanted to be in a large institution or a small institution, so I picked one in the middle. [laughter]

DAEMMRICH: How was teaching at MIT? Did you do some teaching fellowships?

HERCULES: Yes, I was a teaching fellow. It was pretty much like it is at most places; in the beginning you do your T.A. [teaching assistant] job and you run a lab. I had the good fortune of working for Professor [Stephen G.] Simpson who was one of the co-authors of one of the classic analytical books, *Calculations of Analytical Chemistry* (2). Professor Simpson was very good to work with because he was a traditional, classical, analytical chemist. To him, instrumental analysis was using a pH meter, but he really knew his basic chemistry. They taught qualitative and quantitative in separate courses, and boy, Steve put his students through their paces in that. I taught that and then, in my third and final year at MIT, I actually lectured in quantitative analysis, so I actually got to teach a section of the course. I remember they wanted me to teach freshman chemistry, and I said, "I don't want to teach freshman chemistry, I want to teach analytical." I got to do that and really enjoyed it.

BROCK: Can you tell us about the chemistry department at Lehigh when you joined it and the place of analytical chemistry within it?

HERCULES: The place of analytical chemistry within the department was pretty good because the head of the department was an analytical chemist named Earl J. Serfass. There were three analytical chemists: myself, a microchemist by the name of Velmer Fish, and Earl. Analytical chemistry was very well entrenched at Lehigh. I was given a laboratory, and I came with an NSF [National Science Foundation] grant. I either came with it funded, or it was funded within the first month I was there. I wrote the proposal for the grant when I was at MIT. I guess it was

funded right after I got to Lehigh. I came with money for instrumentation, and we built a spectrofluorometer at Lehigh.

I came with a whole list of projects I was interested in doing; some were more classically oriented and some continued spectroscopy. I happened to have a guy, Dick King, who had a background in electrical engineering as well as chemistry, and he helped me put the instrument together. We built an automatic recording spectrofluorometer completely from scratch. I had met Mike Kasha by that time too, and he said, "Don't build an instrument, buy one." But I didn't pay any attention to him. I built one, and in retrospect, it probably would have been smarter to buy one.

DAEMMRICH: Did it take a lot of adjusting to get it to work?

HERCULES: No, it took a lot of work to get it done because we had to build parts and cells. We had to worry about light reflections. We got a recording, grating monochromator which was made for that, but it was designed to be hand powered, not driven by a motor. Building it took an enormous amount of time and probably cost me a year in terms of productivity. In this day and age, that would probably be fatal, but in that day and age it wasn't.

BROCK: What sort of chemical questions did you start asking with the instrument?

HERCULES: I wanted to continue on studying excited state emissions because there were lots of interesting systems to study, and we were one of the few groups in the world doing it besides the people in Europe. The number of possibilities was huge; it was the whole Aldrich [*Aldrich Catalog Handbook of Fine Chemicals*] catalog. I wanted to look at the interactions that occur when you put different groups next to each other on the same ring system. I never was really interested in being an organic chemist, but I always liked working with organic molecules. For instance, one of the first things we did was make three hydroxylated naphthoic acids. We took a naphthalene ring where we had the OH in the 1-position and the carboxyl in the 2-position. Then we made it with the OH in the 2-position and the carboxyl in the 1-position, and then with them in the 2- and 3-positions. The 2, 3 compound is yellow, whereas the other two are white. The extra extension of the ring through the hydrogen bonding enhances the electronic transition.

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HERCULES: I was also very interested in a phenomenon I had read about called photo-potentials. There was a huge amount of literature—primarily physics literature—available that described it. People would take an electrode, put it in a solution containing an organic

compound, and shine a light on it. It would change potential. There were all sorts of very interesting theories as to what was causing that.

My first graduate student was named John Surash. We had one of these very high intensity mercury arcs, so we could build a system that could photolyze the electrode and record the fluorescence spectrum at the same time. We got very large potential changes. Luckily, the system we started out with, anthraquinone, was a really good one. We chose anthraquinone because we knew that somebody reported that it gave a big result. When we turned the light on, we got huge changes in potential, something like 300 millivolts. We soon discovered that when we turned off the light, the potential would drop. It would rise very fast, but it would decay slowly. Whatever was causing it to rise was not what was causing it to decay. It turned out what we were simply doing photolysis of the anthraquinone in the solution, and that was changing the redox potential at the electrode surface. That's all it was. [laughter] It wasn't anything spectacular, but it was a neat thing to be able to do.

We did that, and I spent a lot of time on fluorescence, but the experiment with the photo-potential got me interested in photochemistry. I was particularly interested in what others referred to as photo-induced luminescence. Photo-induced luminescence was used for nothing more than photolyzing a compound that wasn't fluorescent. As it was photolyzed, a fluorescence species was produced and light was emitted from the solution. We did some of those experiments for quite some time.

BROCK: When you arrived, you spent about a year building the instrument, and then you spent two years working with it at Lehigh.

HERCULES: Yes. I had four students work for me at Lehigh. John Surash was the first student I actually recruited. Roy Maron was the first student who finished under my nominal direction. He had been working for Earl Serfass. Roy had become very interested in non-aqueous titrations, and Earl said to me, "I don't know anything about non-aqueous titrations, but you do." I had done some work at MIT where I learned a fair amount about them. Roy was actually working with a guy by the name of Streuli at Cyanamid [American Cyanamid Company]. I took on Roy and oversaw his work. My first student did a thesis on non-aqueous titrations, but basically it was Streuli and Roy doing it. I was sort of helping out.

The other two students were Ed [Edward] Gregorek and Dick King. I put Ed on a project of looking at donor acceptor complexes because that was another big thing at that particular time. They were organic complexes which formed when nitrobenzene and aniline were poured together. The solution got dark in color because they formed a complex. He used UV-visible spectrophotometry because we had a very good UV-visible spectrophotometer.

BROCK: Can you talk about your return to Juniata?

HERCULES: That was purely an emotional decision. Let's back up a little bit to when I was a student at Juniata. I dated a woman whom I eventually married. She put herself through school, and when she was a student she lived with Calvert [N.] and Elizabeth [W.] Ellis. Calvert Ellis was the president of Juniata College. Because I was dating that woman, I learned to know the Ellises very well, and David [W.] Ellis still is a very close friend of mine. He also got a Ph.D. in analytical chemistry at MIT with Buck. [laughter] David's sister, Betty Ann, married Ron [Ronald L.] Cherry, a high school friend of mine and a cohort of mine in Juniata.

I got to know Calvert very well. He was very pleased to see a Juniata graduate go to MIT and become a star student. He asked me if I would consider coming back to Juniata to teach, and the answer was yes, although it took me a while to decide. I had always wondered—even from the time when I was a graduate student because I remember talking about that with several of my cohorts—whether a small college with only undergraduates to work with could be the site of a meaningful research program.

Returning to Juniata was an emotional thing, but it also gave me a chance to really see if that could be done. I was there for three years and it was a very good experience. My answer was yes, a successful research program could be developed. That being said, it took so much effort to do things that I slowly came to the conclusion that it took too much effort to do research at Juniata. If you came across something that was really neat, there were five or six people out there who had big groups and they would jump on it and take it away from you. That didn't happen, but I could see the possibility that it would.

We were fortunate at Juniata because we were able to get lots of money to support the undergraduate research program. The early 1960s, post-Sputnik, was the day of easy research money. We had research funding from NIH [National Institutes of Health], NSF, AEC [Atomic Energy Commission], and the Research Corporation. We had piles of money. We would support summer research for about twenty of our undergraduates. I learned to accomplish research in the summer, because seniors were the only ones who had time for doing research in the academic year. By the end of three years, when I got the opportunity to go back to MIT, I decided that I should go. Again, that was a difficult decision, but it was the correct one.

DAEMMRICH: Was there a problem of continuity with the students, since undergraduates have a much more rapid turnover rate?

HERCULES: No, we'd start them as freshmen and then carry them through until they were seniors. Continuity was not the problem. The real problem was the amount of effort that they could give to the projects.

BROCK: Did you bring your instrument from Lehigh to Juniata?

HERCULES: No, but NIH and AEC were very generous. We got very nice instrumentation. I had very good instrumentation at Juniata.

BROCK: What sorts of equipment were you using in your research there?

HERCULES: I decided, for a variety of reasons that I was not going to build a recording spectrophotometer because I wanted to do some work with low-level light emission. Photomultipliers were not good enough because the circuitry was not good enough to get the signal-to-noise ratio you needed. I still wanted a photographic instrument for doing emission readings. We actually put together a really good system for reading photographic emissions. We did all of our emission work by photograph and then by scanning on the densitometer. It was slow, but it didn't matter.

BROCK: Can you tell us about your offer from MIT and your return there?

HERCULES: That was after Rogers had left MIT to go to Purdue. They were looking for someone to take over the position that Buck had held. Dave [David N.] Hume, the other senior analytical guy at MIT, called and asked me if I would be interested in considering it. At that point in my career, I was ready to say yes, so I had an interview, they made me the offer, and there I was. I learned from Phil [Philip J.] Elving later that he was going to make me an offer the next year to go to Michigan. [laughter] I guess people thought I should be at a big-time institution.

DAEMMRICH: Do you think they were basing that on your talks at conferences, your publications, or just on their knowledge of what you were doing with instruments?

HERCULES: Probably all of the above. I was very active in giving conferences, like the Pittsburgh Conference [on Analytical Chemistry and Applied Spectroscopy, Pittcon]. To this day I do it, and I always had students go to conferences and give papers, which was not as common then as it is now. The Pittsburgh Conference was in Pittsburgh, so that made it easier to send people.

I was around the all of the analytical people. I knew most of them through my connection with Rogers. That's where you gain by working for a very high profile scientist, because you meet all the other high profile community members. Most of the analytical people at that time were really electro-analytical chemists, but I had met most of them and was pretty well known in the community. I kept up a continuous publication rate. My first publication was in 1957, and I've kept up a continuous flow of one or two papers a year.

DAEMMRICH: When did you go to your first Pittsburgh Conference?

HERCULES: My first Pittsburgh Conference was while I was at Lehigh. I came to Lehigh in 1957 and left in 1960, so it was probably 1959. My parents were living in Altoona then, and I remember driving to Altoona and then taking the train into Pittsburgh. That was when they still had decent train service across Pennsylvania. It was useful because there was a heavy snowfall, and everybody else was having trouble driving. Penn Station in Pittsburgh is very close to the William Penn Hotel, which is where they had the meeting, so therefore all I had to do was walk down the street. It was definitely before I went back to Tech [MIT], probably 1958 or 1959.

DAEMMRICH: At those early Pittsburgh Conferences, were you mostly going to hear papers or to walk the exhibit floor and see what kind of new machines were out there?

HERCULES: All of the above, because at that time, the exhibit was at the top. They had a top floor in the William Penn and then they had two club floors. At the time I started going, they were beginning to feel the squeeze in having it at the William Penn, but you could walk the exhibit in less than a day, so you could actually stop and talk to everybody along the way. People were developing new instrumentation, and as a smart-ass, young assistant professor, I told people what was wrong with the design of their instruments. It was fun.

I remember going in with the students from Juniata, and there was somebody—I forget who it was—who designed a UV-visible spectrophotometer wrong. Instead of putting the light source, monochromator, and then the sample, they put the light source, sample, and then the monochromator. [laughter] Optically it doesn't make any difference, but it means that you have the UV light source sitting right next to your sample. I told them, "I want to see you take a spectrum of anthraquinone." By the time they were done taking the spectrum of anthraquinone, it was glowing because it had undergone photo-reduction. For some reason they chose to use a really intense light source, I think to gain signal and a chance to keep their signal-to-noise ratio high. [laughter] I forget who built that one, but I remember that specific incident. That was the first big conference; it was probably 1959 or something like that.

BROCK: Have you continued to go there?

HERCULES: When I was at MIT I went a lot because I worked as a consultant. To jump ahead a little bit, I was a consultant for Instrumentation Laboratories [IL]. Tom Ross, who was the president, had his own plane, and we would fly out to every Pittsburgh Conference. After MIT, when I worked at [University of] Georgia, I don't think I went to all of them. When I got back

to Pittsburgh, I became a member of the Conference Committee and missed a few, but not too many.

BROCK: I was wondering if you could speak about 1963 and your return to MIT. Could we get a snapshot of the state of analytical chemistry then and where you thought the really exciting areas and opportunities were?

HERCULES: In 1963, the state of analytical chemistry at MIT was actually pretty good. The politics were in favor of it because Arthur C. Cope was a strong supporter of analytical chemistry. Art Cope was one of the most amazing people I've ever met. I knew Art reasonably well, actually. One time I remember him sitting and saying to me, "Dave, there are only two kinds of chemists that are important: synthetic chemists and analytical chemists. Synthetic chemists make things and analytical chemists keep them honest." [laughter]

In reality, what he was saying was that synthetic and analytical chemistry were really important in terms of what the chemical industry does. Dave Hume was the head of analytical, and, in addition, there were David [K.] Roe, Klaus Biemann, and myself. That was a good operation. Dick Lord was still the head of the spectroscopy lab at that time. Dick was a very strong supporter of analytical chemistry, so we had all sorts of things going on with the spectroscopy lab as well.

Instrumentation was one of the exciting things in analytical chemistry at that time. About the time I got back to MIT, instrumentation was clearly there. There were a lot of very intellectually exciting and pragmatically important problems to address with that instrumentation. Unfortunately, there was often a disconnect between the two. I think the academic, analytical chemists, myself included, became enamored of looking at the more intellectually challenging things, which were really not related necessarily directly to the practical, analytical uses of these things. I think that is what got the analytical chemists into hot water at that particular time. Al [Allen J.] Bard's talk showed the progression very well. *The Fall and Rise of Analytical Chemistry*—which I thought was a neat title that Al chose—was really true, and that certainly happened at MIT (3).

My group was still looking at electronic transitions because there were some important things about luminescence. By that time energy transfer had become very important, so we studied that. I had become very interested in chemiluminescence. I was the first person to publish anything about electro-generated chemiluminescence. That was during the big heyday of organic photochemistry, because the spectroscopy and apparatus had been developed to the point that they could actually understand why exposing a sample to sunlight yields a product.

After that, we built a flash photolysis apparatus. It was actually commercially marketed, although it was not commercially successful. We probably built one of the best systems for conventional flash photolysis. That was where my interest was. I was not really spending a lot of time looking at what the important analytical implications of all of that was.

Interestingly enough, the electro-analytical chemists were doing exactly the same thing. They were looking at the solutions of boundary-value problems and the ways of using different pulse techniques to understand electrochemistry instead of looking at whether it would be useful analytically. I think we were all pretty much in the same boat. The intellectual projects were going well.

I had a good size group. We were well supported and we turned out a lot of interesting work. But little of it had to do with analytical chemistry directly. We were looking at the fundamentals of the measuring methods, but not paying too much attention to whether or not they were useful for anything. And that was, I think, the mistake. To go in to do these things was not a mistake, but to do them exclusively, I think, was.

BROCK: I have two questions. You were at MIT from 1963 to 1969. I'd like to hear you talk a little bit more about the instrumentation you and your group were using and developing. Also, computing resources were becoming more available. Could you talk about the time period when you were shifting from chart recorders to computer systems?

HERCULES: Yes, the electrochemists were ahead of us and had started to use operational amplifiers to do what I call small computational stuff. That was still the heyday of big mainframe computers, and those things were about as handy to use as a Mack truck. We used a lot of them in my group because my very first graduate student at MIT was Paul [D.] Anderson. He came from [University of California] Berkeley and had a strong background in computers.

I was part of the Laboratory for Nuclear Science at MIT, which had its own computer, a forerunner of the IBM [International Business Machines] system 360 called the 7044 or something that took up a room two-thirds this size. Paul and I could actually go over and use it ourselves at night. I remember mounting tapes and doing all those other kinds of things. We could actually do computations with it. But other than some electronic calculators, the instrumentation for computations wasn't really that far advanced at that point. That would have to come later.

BROCK: What instrumentation did you use in your group?

HERCULES: As I said, we had built the flash photolysis apparatus. We had a stop-flow apparatus which was used for fast reactions. We had a good, commercial spectrofluorometer by that time.

BROCK: Who made that?

HERCULES: It was a Turner [Designs, Inc.] instrument. Turner built a small filter fluorometer first and then built a spectrofluorometer. The spectrofluorometer was compensated for the response function of the detector and everything else. It was a really good instrument. Of course, we had a UV-visible Cary-14. I was still interested in looking at very low-level light emission. We wanted to start looking at chemiluminescence from some reactions where there were very weak emissions, not just at reactions like Luminol, which could light up a room. We made an image-intensifier system. We built a spectrograph, but put an image intensifier instead of a photographic plate on it. It was like the ones they used for night vision, except that it was for UV-visible.

I had been doing some work with the army and had access to an image intensifier tube. That thing was really sensitive. I'll tell you a funny story about it that gives you an idea of how sensitive it was. Fred [D.] Green was an organic chemist at MIT and a good friend of mine. One day, Fred came down to me and said, "Dave, do you think you could get a spectrum of a compound?" He continued, "We have an organic compound and when it melts, it gives off light. But we can't get a spectrum." He said that he'd taken it down to the spec [spectroscopy] lab to Dick Lord, and they kept melting tube after tube. It was a weak source and they couldn't get any light from it. It was an anthracene compound, which was what Fred worked on a lot. I said to him, "How did you know that it thing gives off light?" He said, "We were running a melting point and we saw it." I said, "Do you always run your melting points in the dark?" He replied, "No, but we thought it might give off light, and so we tested it."

We put it in front of the image intensification spectrometer, melted it, and burned the plate. [laughter] When we started, I said that we had to open the slits real wide, but we ended up cutting down the slits because it was so sensitive. We looked at the light emission that you get when you oxidize Grignard reagents. It turns out that most of the light you get when you oxidize Grignard reagents is from singlet oxygen, and it's the emission of singlet oxygen that you see. It has nothing to do with the Grignard reagent, except for its being oxidized.

Then, in 1964, I came across Kai Siegbahn's paper on photoelectron spectroscopy, and so I decided that it would be a great new area of research. I had extensive correspondence with Kai Siegbahn who told me that Stig [B.] Hagstrom was at MIT. Stig and I spent a lot of time together planning what the instrument would be like after Kai and Carl Nordling said they would build me one. The AEC decided to fund it, so we got the instrument. That was a totally different tack for me in the area of research. All of my other decisions were logical progressions of one thing to the next, whereas that was a ninety-degree turn for me. I went to the department and they found a room in the basement of a building at MIT where we could build the instrument. It had to be a big room because it was a magnetic instrument. With a magnetic instrument, one has to zero out the vertical component of the earth's magnetic field.

[END OF TAPE, SIDE 2]

HERCULES: It needed to be done because the vertical component of the earth's field is about three-quarters of a gauss and would have screwed the whole thing up. We had to zero out the vertical field primarily. However, because the instrument Kai built was a double focusing instrument, we also had to zero out the horizontal components of the earth's field. We built a Helmholtz coil system and got that set up. We did a magnetic field survey in the room.

It was a good-sized room, so we had some options about where we could place the instrument. We surveyed the earth's magnetic line so we could find the most homogeneous place, and that's where we put the instrument. We set up the Helmholtz coil system to balance out the field. We had a magnetometer with a probe on it and we had three probes mounted vertically so we could make sure there was no gradient.

I remember when we first got the instrument; we were still getting the whole Helmholtz coil system set up. We balanced out the field, but kept seeing variations. The field kept shifting and we would see magnetic noise. To make a long story short, we learned that since MIT sits at the junction of Massachusetts Avenue and Memorial Drive, there are big pieces of iron (automobiles) going up and down close to the lab. Right around rush hour, it's like a big string of iron of varying size. We were getting magnetic field perturbation from the traffic. We decided that we had to get rid of that. That was easy to solve, actually, because if you did your experiments between the hours of about 11:00 pm and 6:00 am, there was very little iron that went past.

Then we noticed that there was another phenomenon, and we found out that it had to do with the air-conditioner. A resistor which we were using with one of our circuits was not temperature-compensated. In other words, the air-conditioner would come on and it would change the temperature of the resistor a little bit, which would change its resistance a little bit, which changed the current in our Helmholtz coil system. We put that into a heated oil bath and solved that problem.

Then we saw another perturbation. The perturbation would come and we'd see two 10 milligauss blips in a matter of maybe fifteen seconds. We put our probe on a twenty-four hour a day monitor, and we kept seeing those damn blips. If we were running an experiment, they would destroy the experiment.

They were slow enough that we could easily build a feedback system to compensate for them, but we were wondering where they were coming from and it was just bugging the hell out of us. It was in the middle of the summer and I had them shutting down the air conditioning fans. We had elevators. We had fans everywhere. Tony [Anthony] Waraksa, who was the electrical engineer who worked with me, [John J.] Jack, Bill [William E.] Swartz [Jr.], and I were all baffled.

Tony came to me one day and said, "Dave, do you think it could possibly be the Kendall Square subway?" I said, "Tony, that's 600 meters away." He said, "Whenever you start up a train like that, you pull a hell of a current. It's over a big dipole, because it's from up here to down the tracks." He did a rough calculation and said, "I calculate that over that distance it

should be on the order of 5 milligauss or something like that.” I said, “Biemann’s lab is closer to the subway than we are. What should be the field at Biemann’s lab?” Biemann had been having a terrible magnetic interference. In fact, he had lined the walls of the mass spec labs with iron screening. He said, “It should be about 50 milligauss over there.”

So I went over and saw Klaus and I said, “Hey Klaus. If you tell me what the stray field in your lab is, maybe I can tell you what it’s coming from.” He said, “It’s about 40 to 50 milligauss.” [laughter] Then I told him what it was. We actually sent people over to clock the times that the trains came and went, and then went back and looked at the recording. Our feedback system took care of it, but that’s what it was. It was the Kendall Square subway system. That’s chronicled in a book called *The History of Analytical Chemistry* by Herb [Herbert A.] Laitinen and Galen [W.] Ewing. Bill Swartz wrote the chapter on the history of electron spectroscopy and that story is in there (4). It’s true. [laughter]

BROCK: Before we talk about the ESCA [Electron Spectroscopy for Chemical Analysis], I wanted to ask you about the consulting that you were doing. Was your work for the army on a consulting basis? Also, I’m interested in hearing more about instrumentation laboratories. I don’t know much about them.

HERCULES: Actually, “the army” was a euphemism. I was working for the CIA [Central Intelligence Agency]. I started doing that right after I got to MIT, because they were interested in secret writing. Somebody told them that I knew something about spot-testing. I spent some time with the CIA in some of their labs. For example, I went out to the Midwest Research Institute, where they were trying to develop ways to do secret writing so that people behind the Iron Curtain could send things that the Soviets couldn’t detect but we could. It was one of the most frustrating moments in my life because I figured out that they really should use an image intensifier system that could read something at a low level that would be undetectable by standard screening techniques. These were guys who had been spies, so they wanted something where you did something chemical to it and it came out on the paper. [laughter] The other thing they said is it has to be something that is readily available. I said, “All right, what have you looked at in the components at your end? Saliva?” They hadn’t looked at anything like that. They wanted something they could send over. It was a neat group and I had some funny experiences with them, but it was very clear that my work wasn’t doing anything.

IL was a different issue. IL was an instrument company from outside of Boston which probably started a year or two before I went to MIT. They grew to be a very large instrument company. They were big exhibitors at Pittcon. They were taken over by Fisher [Scientific International, Inc.], probably in the mid-1980s. I think they were eventually taken over by somebody else first. They had developed one of the best atomic absorption spectrometers around. They started by making pH meters. There were two pH meter manufacturers in the United States in the early 1960s—Beckman and Leeds and Northrup [Company]. Beckman clearly had the lion’s share of the market. Beckman sold their pH meters through distributors like Fisher Scientific. They were good pH meters, and somebody at Beckman got the brilliant

idea to go to direct sales for all of their equipment. They always did direct sales on their big instruments.

BROCK: They did that in 1963.

HERCULES: That was one of the dumbest decisions anybody ever made because it basically opened up the pH meter market for entrepreneurs. In Boston, there was a guy named Tom Ross. I don't know for sure, but he was a salesman for Fisher. He got together with an electrical engineer by the name of Dave Blackmeer, and Tom and Dave put together a pH meter in Tom's garage and started selling it.

That was the beginning of IL. Then they quickly put together a very good flame photometer that could detect sodium and potassium in blood. It was small, compact, and reasonably priced. Tom would stand and hold the thing up and drop it on a table from two feet, and it would still function. They built excellent instrumentation.

My good friend, Dave Roe, had come to MIT, a year or two before I did. Dave met one of the two, and they hired him as a consultant. Dave consulted for IL for about a year and then asked me to consult because they were thinking of going into optical instrumentation. Dave was an electrochemist. They wanted to start talking about AA [atomic absorption], so I went out and talked with Tom.

I always joked with Tom that while I consulted for IL, my position kept declining. Initially I consulted for the president, but by the time I left MIT, they had grown so rapidly that I was consulting for the bench chemists. [laughter] They were very successful, and it was a wonderful company to work with. I worked with them on their design of AA equipment and on the design of chemiluminescence detectors which they never marketed. They had an oxygen hemoglobin system that I worked on a lot. We spent a lot of time working on the photoelectron spectrometers. They were going to sell them until Varian [Inc.] beat them to it.

BROCK: Did you do other consulting work? Was IL one of your main clients?

HERCULES: Yes. IL was my main client. I consulted a fairly diverse set of companies because of the assorted set of topics I dealt with. I consulted for W.S. Merrill [and Company], which was a pharmaceutical company in Cincinnati. They were taken over by Dow [Chemical Company]. I think I must have consulted for Merrill's analytical group for fifteen years. I consulted for Exxon [Mobil Corporation] for a long time in their lab in Baton Rouge. Those were two really long-term ones. I also consulted for publishers.

DAEMMRICH: Siegbahn's book came out shortly before you moved to the University of Georgia (5). What kind of impact did that have on you? Did you know about it ahead of time?

HERCULES: I knew about it ahead of time. In fact, I had a copy of it. It had come out earlier as part of a report to the Air Force, as I recall. I knew almost everything that was in it. [laughter] There were no surprises.

BROCK: Could you tell us about when you first read his paper in 1964 and decided to make a u-turn?

HERCULES: Yes. This is a true story. I always told my graduate students that to pick out the next major analytical or spectroscopic technique, look at the physics literature because almost all of the major chemical analytical techniques have come from physics. Cast your line where you know there are fish. Look at the physics literature to see what you can come up with.

Sometimes I even follow my own advice. I remember very clearly that I had gone over to Hayden Library. I think it was on a Saturday. Everything at MIT is interconnected, and my office wasn't far from the library. I had a list of journals that I always looked through. I'd look at maybe three or four journals and then three or four issues of each. I was looking down the pages, and I was just glancing at titles. I saw one that read, "Electron Spectroscopy for Chemical Analysis." I thought that was a very strange title. I first read it as *electronic* spectroscopy, like UV-visible, for example (6). I thought, "This is a legitimate physics journal, and these people can't be dumb enough to think that they've discovered that you can do chemical analysis with UV-vis." That's what it seemed like to me. I thought, "I better look at this."

I looked at it and immediately saw that was not the case. It was only a few pages long, and I read it. I thought, "Wow, they can tell the difference between the oxidation states of sulfur in thiosulphate." I thought it was really interesting because it was like NMR [nuclear magnetic resonance], but could be done with other elements. The authors were Siegbahn, Nordling, and [Ulrik] Gelius. I didn't know who was who, except that Siegbahn's name did ring a bell with me because of his father [Karl Manne Georg Siegbahn].

I remember going out and getting the five-year indices of *Chemical Abstracts* and seeing all of Siegbahn's papers on building magnetic spectrometers. I had no idea how to measure an electron spectrum. I thought, "Wow, this is a whole new field." I remember I had something else to do that day, so I came back a few days later and started doing what I normally did when I hit something new: I backtracked on all the references.

I read *Chemical Abstracts*, backtracked the references, and went back to the original Siegbahn papers. I think his first paper on the double-focusing magnetic spectrometer was published probably between 1948 and 1952, somewhere in that ballpark (7). I started reading

about building magnetic spectrometers, and that was about as much fun as watching grass grow. It was really something that I had no background in, but I waded through the papers until I understood what he had done.

I read their work about measuring electron binding energies in the elements, and they referenced one paper that a Polish woman had worked on. It was the first place where a chemical shift was noted, but they didn't realize what it was at that time. I picked up the other papers that had been published at that time and did a search for Siegbahn's current stuff.

I remember talking to Dave Hume about it, and he said, "You know, it sounds interesting. Are you sure you want to do this? Starting a new program in an area is a real major task." I replied, "I know that, but I think I will. Let me write him a letter. Do you think I can get funding from the AEC?" He said that I probably could.

I wrote Kai a letter and he wrote back fairly promptly, apologizing for not getting back sooner because he'd been on a trip or something. I wrote and asked him if he had schematics for the instrument and if he could share the plans with me so I could build it. He said that they didn't have plans because they were continuously modifying the instruments. He said that building the analyzer was very difficult and it took very high-precision machining. We exchanged various letters back and forth.

He said, "If you really want to learn about electronic spectroscopy, one of my people, Stig Hagstrom, is at MIT right now." Stig was housed over in an old, temporary building at MIT which was left over from World War II. It was called Building 20. He was supposed to work on a magnetic spectrometer that somebody had at MIT, which was just not what he was interested in doing. At that time, he had already made commitments to work at Berkeley after he finished up at MIT. He was only at MIT for maybe six months, but I worked with him. He worked with me out of the kindness of his heart.

I asked him a lot of questions about it, and he said, "Yes, you have to have the magnetic spectrometer coil. The machining tolerances are tight on it, and it has to be circular because little perturbations mean perturbations in the field." I remember that Stig sat down and drew the layout and described the electronics. Between talking with Stig and my correspondence with Kai and Carl, the thing started to move along. They were coming to the United States and visited me in Boston probably in 1965. We talked there and then got the thing ordered.

It looked like we were going to get funding, so in the spring of 1966, I went over to Uppsala [University] for a while with Tony Waraksa, and we went through the whole thing. I have a whole set of photographs, like the one I have of Carl and Kai on the instrument. We took a whole series of Polaroid photographs of the instrumentation and how things were setup so we knew exactly what things would look like whenever we got the instrument. The instrument probably was delivered in 1968.

BROCK: Who built it?

HERCULES: Siegbahn did.

BROCK: He did. Wow.

HERCULES: Yes. He built one for me. In 1969, when the first commercial instrument came out, there were four functioning electron spectrometers in the United States, three of which had been built by Siegbahn. The one at Berkeley that Dave [David A.] Shirley, Bill [William L.] Jolly, and various other people used, was an older version built by Siegbahn, because Siegbahn had spent time there. Berkeley had a brass instrument, and there were two split coil instruments which were newer ones that were made out of aluminum instead of brass. Siegbahn built one for Roy [Royal G.] Albridge at Vanderbilt [University] and built one for me. The fourth instrument, an electrostatic instrument based on the Siegbahn design, was built by Tom [Thomas A.] Carlson at Oak Ridge [National Laboratory]. Those were the four instruments.

DAEMMRICH: What did he charge you for it?

HERCULES: I'd have to look back in the records. I think it was about forty thousand dollars. It was not a lot.

DAEMMRICH: He made it as a colleague.

HERCULES: I don't think he made much money on it. [laughter] We got it, put it together, and got it running.

BROCK: Was it running by 1968?

HERCULES: Yes. I think that it was late 1968. I'd have to check to be sure when we got our first spectrum.

BROCK: What did you do with the spectrometer?

HERCULES: The thing which had gotten me and a lot of other people interested in it was the whether it could be used as a structural tool for things other than hydrogen, for which NMR

works perfectly well. At that time, we only had proton NMR. People knew that C-13 [carbon-13 NMR] worked, but with the sensitivities it had, anytime we wanted to do something with C-13, we had to enrich it isotopically.

There were basically just proton NMR and unit resolution mass spectroscopy. There were double-focusing instruments out there, but they were basically research tools at that time and not commonly used. Everybody knew which nuclei gave NMR signals, but measuring them was another thing. People measured phosphorus and fluorine because you could measure them with a 60 megahertz instrument.

That's why there was an interest in using ESCA as a structural tool. It was sensitive and potentially you could get chemical shifts for any element. We were interested in seeing what kind of structural correlations one found between electron binding energies and photoelectron spectra. The first two Siegbahn books make the case very clearly for carbon, sulfur, and nitrogen (8).

The first two students I had were Swartz and Jack. Our work was not like the big brush stroke work that Siegbahn's group had done. We took a given kind of functionality where there were variations on it and saw how much we could tell in the second order effects. Jack worked on quaternary nitrogen compounds. We basically had nitrogen with a charge, and we wanted to see how the binding energy shifted if you varied the functional groups around the nitrogen. Swartz did the same thing and he worked on phosphonium compounds.

BROCK: In that case then, you knew the structure of these compounds?

HERCULES: Yes.

BROCK: Right, because you knew where you were putting things.

HERCULES: That's right. We wanted to see what effects there were.

The first paper I wrote on electron spectroscopy was an A-pages paper for *Analytical Chemistry* and was published in 1970 (9). In it, there's a chart, and I had the idea of doing it like a Colthrup chart for infrared frequencies. I had pulled a lot of data out from the literature, mainly from Siegbahn's measurements, but I had some of my own measurements. We could see that a functional group had binding energies in a well defined and reasonably narrow range. But then, as we got more measurements, the ranges kept getting broader and broader, and we began to see that ESCA was not going to work. This was at the same time when Siegbahn was publishing a lot of correlations between binding energy shifts and charge calculations. It became very clear that it was not going to be a structural technique *per se*, but a technique which showed the charge on the atom.

What can you learn about structure from the charge on the atom? There were several problems. One of the problems was that CNDO [complete neglect of differential overlap] calculations were not easily done at that time. To do CNDO calculations, you had to punch all the data into punch cards, carry three stacks of punch cards over to the computer, and two days later you'd get the output back. That wasn't exactly something you could do in the laboratory. If you were running a spectrum and came to me with a question, I would say, "I'll give you the answer in two days if we do a calculation."

Various people started looking at alternative, simple ways of looking at charge. Pauling electronegativities didn't cut it. Bill Jolly at Berkeley tried what he called electronegativity equalization method. He tried doing it by thermodynamic calculations. He tried a number of very creative ideas. We adapted [Robert T.] Sanderson electronegativities and the group shift idea that the Siegbahn group came up with and we did Sanderson group shifts. We decided to work on organic silicon compounds because I didn't want to overlap with Kai, who was working with carbon. Our final conclusion was that all the ways you calculated charge were bad. That was about the time when Jim Carver joined the group. We started saying, "What can we do with this technique that is relevant to analytical chemistry?"

[END OF TAPE, SIDE 3]

HERCULES: First, we tried to use XPS [x-ray photoelectron spectroscopy] for trace analysis, which was a very fortunate choice. The chemical physics community said, "You'll never be able to do quantitative analysis because there are too many variables in the ways you do the experiment. They had forgotten about using an internal standard, and therefore, XPS suddenly became very quantitative. We were probably the first group to publish quantitative analysis by XPS. Then we started looking at other kinds of problems.

We did some work on the fluoridation of dental enamel, especially with stannous fluoride. We could show that the tin from the stannous fluoride stayed on the surface of the tooth, and the fluorine went down in. This must have been about 1974. I remember one time getting the whole group together and saying, "Troops, we can get a periodic table, sit here and throw darts at it, and do second order correlation effects for any element we hit, but that's not the way we do science. We've looked at various kinds of things. We've tried a bunch of different things, and trace analysis is okay, but that's never going to really sell as a trace analytical tool. What should we do?" We sat there and talked about it and suddenly it dawned on me. I said, "I just thought of a field that could really use us. Heterogeneous catalysis."

There was almost no work published on using ESCA for studying heterogeneous catalysis. There had been about three or four papers, and they had all done short investigations. They'd run catalyst and look at it, and that was it. Because of my background as an undergraduate, I knew something about catalysis work. I applied to the Guggenheim

Foundation and proposed to take some time off and study heterogeneous catalysis. They said, “Okay, go ahead.”

There were two places I planned to go to study catalysis. One was with Dick [Richard J.] Kokes at Johns Hopkins [University], and the other was with Bob [Robert L.] Burwell [Jr.] at Northwestern [University]. Unfortunately, Dick Kokes died between the time I proposed it and the time it was funded, so I spent some time with Bob Burwell at Northwestern, and he was very helpful. I came back and said, “We have to figure out how to carry out a catalytic reaction so that we can look at the catalyst. We can’t do it as it’s undergoing the reaction because we have to worry about the pressure difference—something like six orders of magnitude, or greater. On the other hand, we should be able to move the probe in and out of the instrument.”

They were starting to put attachments on the instrument, so the instrument manufacturers suggested we install a reaction chamber on the instrument. I disagreed because you want to study catalytic reactions for a long period of time, and if you have the reaction chamber on the instrument, nobody else can use that instrument while you’re carrying out your reaction. We have to build a probe such that we can do a reaction in a tube furnace and then carry it, without exposure to the air, and put it into the instrument, which we did.

That was what really got our catalysis program off and going. We published two papers. I had a Chinese student, [Kung-Tat] Ng, who was anxious to work with me. My colleague at Georgia, Don [Donald E.] Leyden, had a student, Tom Patterson, who had done x-ray work with him. Tom was interested in trying this new technique. Don agreed that for half his thesis, Tom could work jointly with me. I gave Tom a cobalt molybdenum alumina catalyst and I gave Ng a nickel tungsten alumina catalyst. They both went off and did experiments with the probe that we had built. We published our first two papers on catalysis in 1976 and then we spent a long time doing catalytic research after that (10).

BROCK: Please describe your move from MIT to Georgia in 1969, and the state of the instrument that you had bought from Siegbahn.

HERCULES: I took the instrument with me when I moved to Georgia. I decided to move there because a major change occurred in MIT’s chemistry department after Art [Arthur C.] Cope died in 1966. The department became strongly anti-analytical, and since I was not tenured, I decided to leave. I forget whether it was 1966 or 1965.

Dave Roe was a year ahead of me, so he came up for tenure and they decided not to tenure him. I was coming up the next year and asked Dave Hume what to do. He said there wasn’t much that I could do except give it a try. My tenure process apparently caused a considerable problem for MIT’s tenured faculty because they were about evenly split as to my tenure, from what I’ve gathered—of course, this is all hearsay because I was not privy to the communication.

By that time, John Ross had become head of the department. I went to John, and he said that it was a very difficult decision and they were split. I said, "Whether I have tenure in this place or not is not important to me. Why don't you guys promote me to associate professor? I must be the oldest assistant professor in the country. Why don't you promote me to associate professor without tenure and worry about the tenure decision some other time?" They agreed, and I was promoted to associate professor.

After that, they still wanted to deal with the tenure issue and had a meeting. John said that the outcome of the meeting was very close. They had decided they would not offer me tenure, but I could stay there as an untenured associate professor as long as I wanted to. I said to him, "It sounds like Harvard." [laughter] He responded, "You can stay. Your teaching is fine, and we'd be glad to have you stick around. You're funded under this big umbrella thing."

This was about the time that the student radical stuff was going on in Boston, and it seemed to me that things on the horizon for research funding were not what they had been earlier. This was when they were beginning to worry about the industrial military complex in the association of universities and that affected funding for the sciences. I thought that the AEC was potentially vulnerable. Charles [D.] Coryell, who had been the head of nuclear chemistry at MIT, died, and they basically shut down the nuclear chemistry program.

I decided that I would accept that they had decided not to give me tenure as a decision that they were not going to give me tenure ever. Therefore, I had to depart. I ended up going to the University of Georgia. I took the instrument with me, and MIT was very good about it. In fact, I took everything in the lab except for the common equipment.

DAEMMRICH: There wasn't anyone at MIT who wanted the instrument to stay?

HERCULES: No. There was nobody there to use it. [laughter]

DAEMMRICH: You arrived at Georgia. Did that cause a break in your consultant relationship with the companies that you were working with?

HERCULES: Yes. It terminated my relationship with IL because when I worked with IL, I would spend about half a day a week out at IL in Lexington, Massachusetts. My consulting with IL was really on a line-consulting basis. I was dealing with specific problems. They offered me a job, which I really didn't want to take. So, that ended it.

BROCK: Can you tell us about the chemistry department at Georgia when you joined and the status of analytical chemistry?

HERCULES: By the time I arrived, the chemistry department at Georgia was well on its way to becoming a very good chemistry department. It had been built up by Bill [S. William] Pelletier. He had come to Georgia in the late 1950s, or maybe early 1960s, from Rockefeller [University]. He was a natural products chemist and a very dedicated guy who really put the department on the map. The best characterization I ever heard of the Georgia chemistry department was from the late Fred [Arthur Frederick] Findeis [Jr.] at NSF. One time Fred said to me, "Before 1960, if anyone had asked me if there was a chemistry department at the University of Georgia, my reply would have been, there must be." [laughter] It was not a distinguished department.

Bill brought in a variety of people. He brought Lou [Norman Louis] Allinger and me at the same time. [Robert] Bruce King was there and that was when he was the darling of the synthetic and organic community. He was making more compounds in a week than most people made in a career. Bill had brought in a lot of people, most of whom were really quite good, and he was successful with them. He brought [G.] Paul Storey, for instance. The department was really dynamic. Analytical was happily ensconced because when I came in, I was the nominal head of the analytical program. There were four of us: myself, Don Leyden, Leon [N.] Klatt, and Pete [Peter W.] Carr. We later hired a fifth, Rudi [W. Rudolf] Seitz, who's now at [the University of] New Hampshire. Pete's at [the University of] Minnesota. Leon just retired from Oak Ridge and Don went through a circuitous route to Philip Morris [USA], retired early, and is living happily on the Virginia coast.

We were cohesive because the whole climate at Georgia was that we were going to build the best department that we could. The climate within the analytical department was similar. The people at Georgia were very nice. I told them that I didn't want to repeat the trauma that we had at MIT with the magnetic spectrometer, and they gave me an old storage building. It had been an old warehouse kind of building, and we finally were able to put the magnetic spectrometer in a building that had no iron material in it. I had been dealing with the fields in Cambridge, Massachusetts, and then, all of a sudden, I was going to the cotton fields of Georgia. It was a wooden structure and there were no subways. The only big problem we had was that the students would pull their cars back up against the building. When classes would change they would shuffle the cars and it would cause a perturbation, so we had the campus police block those parking spots off. It worked very well.

We bought our first commercial instrument in about 1971. The instrument was one made by AEI [American Electrical Industries], which ultimately become Kratos [Analytical, a Shimadzu Group Company]. It became our workhorse instrument because it had a much better analyzer. It was a higher vacuum instrument. It was much better at scanning and very easy to use with the kind of probe system that we ended up building.

BROCK: Did you and your colleagues stop using ESCA as a structural tool at the time of this move or slightly after?

HERCULES: After the time of the move. We had not published any papers at MIT. Bill and John's first papers were published at Georgia, even though they both got their degrees from MIT. We had done measurements, but when you start doing measurements you want to start with known quantities, so that you're sure the instrument is really telling you the truth.

The time that my realization about ESCA came about was after the AEI instrument had arrived, so it was in the early 1970s. That was about the time everybody else was coming to the same conclusion. I mean, it wasn't like we had figured it out and everybody else was still in the dark. I think everybody came to the same conclusion at about the same time.

BROCK: You were at Georgia from 1969 to 1976. Could you talk about the funding for your ESCA work during those years?

HERCULES: When I first moved to Georgia, I talked to the people at the Atomic Energy Commission. They continued my funding until they were hit by the anti-military industrial complex attitude in the early 1970s. At that point, they said that they could not continue my funding. This was before they became the [U.S.] Department of Energy, which was around 1973. When they were still the Atomic Energy Commission, they were forced to only deal with things related to atomic energy. My project didn't happen to be one. At the same time, they basically shut down the operation at MIT as well. We were funded under a program called "The Analytical Chemistry and Fission Elements" which dated back to World War II. They transferred money to NSF for a year, so NSF could fund me. I had smooth transition of support from the AEC to NSF, and I had NSF support as long as I was doing any photoelectron spectroscopy.

BROCK: How long did you go to Northwestern to study and when was that?

HERCULES: That was in about 1974, and I was at Georgia at the time. The amount of time I physically spent at Northwestern was only a couple of weeks. The other thing I did when I was on the Guggenheim grant was put together this tape course I did for the ACS [American Chemical Society]. I started that. I was really going over a whole bunch of background stuff on electron spectroscopy, aimed particularly at its chemical applications. I think the tape course was published right about the time I moved to Pittsburgh, I think, about 1976 (11).

BROCK: Did you just work on catalysis until 1976, or did you add more topics?

HERCULES: We got started on the catalysis project in about 1975. One of the reasons that I moved to Pittsburgh was that I wanted to do catalysis work. I had gone to an International Catalysis Society meeting down in Florida in the late 1960s. When was the first [Richard M.]

Nixon election? We were supposed to have the Catalysis Society meeting in Miami Beach, but the Republican [National] Convention was also happening there and needed the hotel that the Catalysis Society had. The Republicans said, "If you give us your hotel, we'll put you up at the Breakers in Palm Beach, which is a millionaire's resort." [laughter] I had never been in such a nice place in my life.

That was the first place that I went to learn about catalysis. We tried some other techniques before we hit on cobalt molybdenum aluminum catalysis. I think 1968 might be right. During that meeting, I came to the conclusion that more work on electron spectroscopy was needed. It was an International Catalysis Society meeting, and there was not one paper on electron spectroscopy.

BROCK: Could you talk about [the University of] Pittsburgh and catalysis?

HERCULES: I knew the scene in Pittsburgh pretty well because I was a native of that area. Gulf [Oil Corporation] Research Center was there. I actually got the second AEI instrument in the United States. The first one went to Joe Lester who was then at Gulf. There also was a guy at Gulf named Leon Petrakis who had published ESR [electron spin resonance] work on the kind of catalysis we had worked on. Our XPS data showed exactly what he had shown except that we could do it better. I corresponded with him.

The Department of Energy laboratory in Pittsburgh was very actively involved in the same kind of catalysts we were. Other major companies like Koppers [Inc.] and Alcoa [Inc.] were also in Pittsburgh. The big SOHIO [Standard Oil Company, Ohio] lab, which ultimately became BP [British Petroleum Company plc], was in Cleveland. My conclusion was to try to develop a program in surface analytical chemistry in a place like Pittsburgh where there was a lot of work going on in the real world rather than in a place like Georgia where there was nothing. That wasn't the only driving force behind my move. I did not approach them. Pitt approached me.

In fact, Foil Miller was the guy who approached me because I knew him from when I was at Juniata College. Foil was a student of Dick Lord's. Pitt had had [J.] Wayne Rabalais, who's at [the University of] Houston now. I think he left Pitt for personal reasons. They had a photoelectron spectrometer at Pitt, and they said they wanted to get somebody else in analytical. Johann Coetzee was there. They thought that since they had an electron spectroscopy program, my program was dealing with catalysis, and they got very positive vibes from the community about this, Foil would at least make a call and see if I was interested.

I remember the call very clearly. I was used to getting offers from various places. I would come home and say to my wife, "How would you like to live in—wherever it was?" [laughter] That would usually be the end of the conversation. I came home that evening, poured her a drink, and said, "I had an interesting phone call today." She asked, "Where now?" I inquired, "How would you like to live in Pittsburgh?" Shirley [H. Hercules] stopped and

asked, “Did you say Pittsburgh?” Our daughter was born six months before we moved. She was born in December and we moved in June.

Shirley’s parents and my parents both lived within sixty miles of Pittsburgh, but it was still very difficult for us to leave Athens, Georgia. The university had been very good to me. They had really done everything that they could possibly do to help my program along, and we liked living in Athens. We enjoyed it very much and we were very happy there. It took a lot of thinking, but we finally decided that the decision would be made for professional, not personal, reasons. Professionally, I thought it was a good move, and I think historically it turned out that it was.

BROCK: You were at Pittsburgh from 1976 until you went to Vanderbilt. When was that?

HERCULES: I started at Vanderbilt on 31 December 1994, or in other words, January 1995. During the fall term of 1994, I spent 50 percent of my time at Pitt and the other 50 percent at Vanderbilt. I made the decision to go to Vanderbilt in August, so I split my time and Vanderbilt paid half my salary for the term.

BROCK: Can you tell us about how the research program and the catalysis program started at Pitt?

HERCULES: It got off to a good start because when we moved to Pittsburgh from Georgia, we also took along the old magnetic spectrometer from MIT. It made it all the way to Pittsburgh even though we never set it up. We also brought the AEI instrument. In the meantime, we also bought an Auger [electron spectroscopy] instrument. These were all paid for by NSF, so they moved with me. On top of that, Pitt bought me an ion scattering and SIMS [secondary ion mass spectrometry] instrument. Plus, I got the Hewlett-Packard [Development Company, L.P.] ESCA which they already had. I had artillery like you couldn’t imagine.

DAEMMRICH: Wow. We’re talking two rooms this size.

HERCULES: I had two 900 square foot laboratories, plus a chemical lab. There was an interesting glitch with the equipment. The Auger was not difficult to buy because it came right about the time that I was moving and so I was able to have Pitt pay for it. Georgia had a law that you cannot sell state property; therefore, Pitt could not buy the other XPS. Georgia had to let me take the AEI instrument on loan to the University of Pittsburgh, but they had put some money into it for an upgrade and they felt they should get some of that money back. Georgia and Pitt agreed to do it by prorate. Pitt couldn’t pay Georgia for anything unless they had something to show for it. It was about thirty thousand dollars; you couldn’t just give it to them.

You had to buy something with it. Georgia didn't have anything to sell them. It turned out that when we took the old Siegbahn magnetic instrument to Georgia from MIT, for some curious reason, it never got on the Georgia property books. Georgia would let me take it, and Pitt would buy it, but Georgia couldn't officially sell it! [laughter] Finally, a bill of sale was given to Pitt—

[END OF TAPE, SIDE 4]

HERCULES: —for the magnetic spectrometer which had never gone on the books. Every so often I got a call from the property office at Georgia saying, “Do we still have this instrument?” “Yes. We still have this instrument.” [laughter]

BROCK: What about the SIMS instrument?

HERCULES: In the mid-1970s, 3M Corporation marketed a combined SIMS low-energy ion scattering instrument. It was an instrument that had an energy analyzer for the low-energy ion scattering and a little quadrupole mass spectrometer with an ion source. That was our first SIMS instrument.

BROCK: Was that used in surface science?

HERCULES: Yes. I got a Fourier transform infrared instrument as well, so I ended up with a fair chunk of hardware.

DAEMMRICH: Did you any consulting work in Pittsburgh?

HERCULES: Yes, I did a whole bunch of consulting with various companies.

DAEMMRICH: Did you help them design instruments?

HERCULES: No, my consulting was mostly about applications of surface analysis. Exxon was my major client along with W.S. Merrill.

DAEMMRICH: Merrill was a pharmaceutical firm, so why did they need surface chemistry consulting?

HERCULES: That was from way back when I was doing luminescence, and the guy who was the head of analytical chemistry at Merrill was a friend of mine from graduate school. He needed an analytical chemist who had fairly broad knowledge and could come in and talk with them about techniques and problems. Hubert Keily had me consult for him.

BROCK: How many grad students did you have working in your labs at Pitt?

HERCULES: When I started, there were eight or nine graduate students, but the group grew. Probably the largest that the group ever was, was a total of twenty people.

DAEMMRICH: Did that include post-docs?

HERCULES: That included post-docs, and that group was too big. Typically, I was comfortable with groups with between twelve and fifteen people. Those were the numbers we usually had.

I became chairman of the department at Pitt in 1980. At that point, I had been pushing the university to build a surface science center. The reason for that was that Pitt had built a new chemistry building in 1975. I moved to a nice, new fifteen-story building. The building behind that, Alumni Hall, was the old chemistry building, and the first floor had been kept vacant for the chemistry department. By the time I became chairman, the administration was being pressured to use that space for something other than the chemistry department. They asked me what I was going to put there. I said we should make it into an interdisciplinary surface science center. They asked me if they needed to hire anyone else to work there. I told them that I wanted somebody who did more fundamental surface science since I did mainly analytical.

Two interesting events occurred. One was Texas A&M [University] was trying to recruit me. We came very close to going, actually. The second thing was that John [T.] Yates [Jr.] surfaced as a potential candidate for the surface center. John Yates and I had been good friends since we were in college. Interestingly enough, John, his wife, my wife, and I were all chemistry majors in Juniata College—an incestuous group. [laughter]

I was trying to recruit John, and A&M was trying to recruit me. One time John was visiting, we were having a drink, and I said to John, “John, are you going to come to Pitt or aren’t you?” He replied, “Dave, are you going to stay at Pitt, or aren’t you?” [laughter] I answered, “I’ll tell you what, John. If you come, I’ll stay.” It sounded like a good deal to me. I went over to Wes [Wesley W.] Posvar, who was the Pitt chancellor, and said, “Wes, I have a

problem for you. If you are willing to create a surface science center, then John Yates will come and I will stay. If you don't, John Yates won't come and I probably won't stay." Wes responded, "Surface science sounds very nice." [laughter]

We started a surface science center. It still exists, and John Yates is still there. John and I set the center up together. We decided that since I was chair of the department, I shouldn't be head of the surface science center. I viewed that as a conflict of interest. John was head of the surface science center and I was head of the department.

DAEMMRICH: What other sources of funding were there in addition to the university itself?

HERCULES: John and I were both well-funded. The university also spent nearly one million dollars in renovations. They bought some equipment for John to entice him to move there from the [National] Bureau of Standards. He could not take anything from the Bureau of Standards, so they gave him startup equipment. On the other hand, John came along with equipment money, so they bought me some equipment, like a new XPS system. The university probably came close to putting two million dollars into the surface science center. It was one floor and you sort of walked down the middle. John's area was on the left side and mine was on the right side. We had neighborhood kids wandering through from one high school, so we had to keep the doors locked to keep them from going through one end, picking up stuff, and going out the other side.

BROCK: When was it established?

HERCULES: It was in the early 1980s, when the catalysis program was still growing because we had gotten really nice facilities for the chemical work. By that time, we had surface area measuring stuff and were very well equipped to study catalytic reactions. We actually correlated the measurements from the ESCA on the catalyst with their activity, so we had activity measuring equipment. We set up a really nice working relationship with two organizations in Pittsburgh. Right after I first got there, we connected with the Department of Energy. When we were working on cobalt molybdenum catalysts, they were too, and they had an infrared and Raman group. We actually teamed up and published a number of papers together, but more importantly, we had an awful lot of dialogue going on (12).

Milt [Milton L.] Lee, from Brigham Young [University], was working there for one summer, so I got to know him. He was looking at pretreatment of capillaries, and we teamed up to use the Auger microprobe and look at what was forming inside capillaries. We split the capillary and looked down inside it, and so forth.

The second major collaboration I had was with Gulf, primarily through Leon Petrakis. Leon was the head of physical science for one sub-group at Gulf, and they were interested in the

same kinds of catalysts we were interested in. We set up a collaborative program with Gulf, and then Leon and I applied for one of the NSF academic-industry programs, and that was a great success. It was the one they used as their example of a successful collaboration, and it got renewed. Then Chevron [Corporation] bought out Gulf and closed the lab, ending our partnership. However, in that period of time, we had strong interactions with the government lab, strong interactions with Gulf, and a lot of catalyst issues to work on. That was very positive.

BROCK: By the mid-1980s, your group was going strong in catalysis. How was ESCA being used by 1985?

HERCULES: By 1985, people weren't doing ESCA by itself. This happened because around 1975 to 1977, everybody realized that if you wanted to get a wrong answer, you should use just one technique. People began to use multiple techniques on surface problems. By 1985, everything was mostly being done using a multi-technique mode. It was more about focusing on solving problems than doing ESCA experiments. A lot of the experiments that were purely ESCA experiments were being done more by people who were interested in studying certain kinds of interactions on very clean surfaces.

There was a lot of very good work being done in the clean surface chemistry community—John Yates included. The applications were really oriented for industry. Dick [Christopher Richard] Brundle at IBM did an enormous amount of work, which, unfortunately, was mostly unpublished. He did work on surface stuff because that's where they have all their problems. These weren't problems with silicon, but problems with what they called packaging. Circuit boards are made with many layers. Putting stuff on the layers was where your circuits failed, not at the silicon devices. I was also working extensively with people at Exxon in Baton Rouge to establish a surface science laboratory adjacent to the refinery so they could use that to look at catalyst problems in the refinery. We had really begun to look at specific problems.

Around that time, [W.] Keith Hall, who was the editor of *The Journal of Catalysis*, decided to retire from the University of Wisconsin and come back to Pittsburgh where he was from. I welcomed Keith into our department with open arms. We appointed him visiting distinguished professor in the department, and he brought his catalyst program and *The Journal of Catalysis* to the University of Pittsburgh. That immediately tied all of our work into the rest of the catalyst community, so we began to approach other kinds of problems that we hadn't thought of. Keith was a guy who probably forgot more about catalysis than I ever knew. He'd been in it his entire career and was really a fine scientist and a wonderful guy.

We began to work on other kinds of things, like the following problem. You have a molybdenum catalyst. I'll use molybdenum because it's one we worked on, and I know the system well. If you take a molybdenum catalyst, as you put it in the bottle off the shelf, it's dead. It doesn't do anything. You have to activate it by reducing it in hydrogen. We had shown that you get a progression of reduction of molybdenum from plus 6, in conjunction with

the atmosphere, to molybdenum 0 at 800°C. At any other temperature, you will get mixture of different oxidation states.

One of the things we did was show what the mix looks like and then say, “We can use the distribution of oxidation states to determine what oxidation state of molybdenum catalyzes a particular kind of reaction like hydrogenation of ethylene.” We then said, “How about if we take a molybdenum compound and put it on the surface? That molybdenum compound is in a known oxidation state. What’s it going to do?” The question was whether carrying out reactions changes its oxidation state. We began asking more detailed catalytic questions. We were still developing our SIMS program at the same time, so I was actually beginning to look at polymers and stuff like that. We were really beginning to ask more and more catalytic questions, particularly in conjunction with Keith Hall.

BROCK: You said that if you want to get the wrong answer, just use one technique. Was SIMS your other technique to use?

HERCULES: Ion scattering and Auger are very valuable.

We did little research problems. One of the things I always liked about Pittsburgh was that it had so many little companies running around it. I’ll give you one example, which is one of my favorite stories. This guy came into my lab one day. I would say this was around 1980. He said, “I have a problem. I’m a steel maker and I sell rolls of galvanized steel this high and this wide to a guy who makes bread pans. Recently, they’ve been having a problem. Their problem is that the bread pans turn purple. They’re not gray like bread pans are supposed to be. They’re purple.” [laughter] I said, “What’s wrong with purple bread pans?” He said, “They don’t sell.” The guy who makes the steel continued, “The fellow who’s making the bread pans is doing something wrong.” The guy who makes the bread pans said, “You sold me a lousy batch of steel.” The usual. He asked me if there was anything I could do. I said, “I haven’t the foggiest idea, but why don’t you bring me a piece of your steel that’s good, a gray bread pan, and a purple bread pan. We’ll put them in the instrument and see.”

We did, and we ran a depth profile on them where we looked at the composition as a function of depth. In the steel that he was shipping to the bread pan manufacturer, I saw this absolutely gorgeous profile where there was nothing but zinc and then it dropped off and I could see the iron from underneath. There was a nice zinc layer. I looked at a bread pan which was gray and there was a nice zinc layer and it dropped off to iron as well. I took a purple bread pan, and the first thing I saw was that there was iron at the surface. The profile of the zinc went down, sort of linearly, and the iron came up, but the iron started out as a finite composition.

I said to him, “How do they make the bread pans?” He replied, “They go through a process. They clean them. They heat or dry them, and then they put them in a cleaning solution which is about the same sort of stuff you would use to blue a gun barrel.” I said, “They have

over-heated the bread pans. Then you have the iron on the surface. They put it through this process and it turns it blue, which looks purple. There's nothing wrong with your steel."

They went and checked it, and guess what—they were overheating their bread pans. They cut the temperature back and got gray bread pans. I told them afterwards, "You know, you guys were missing something there." They asked, "What's that?" I responded, "Why don't you make the purple bread pans and say they are the newest thing from Paris? You can probably sell them for five times the price." [laughter]

BROCK: Let's jump forward a little bit to 1995. Could you talk about the catalysis program in 1995? We should also talk about mass spec.

HERCULES: There's another part of my mass spec work that we haven't talked about yet, and that was when I became a consultant for Leybold-Heraeus [L-H] in Germany in the 1980s. L-H was a great big vacuum company. They built vacuum chambers that were ten times the size of this room. They also decided to have a scientific instrument division. This was located in Cologne, and, in fact, we bought a lot of Leybold equipment. They put out an instrument called the LAMMA—the laser microprobe mass analyzer. It was the first commercial laser-induced mass spectrometer marketed. I had a former student who was working for Leybold in the United States, and he kept telling me about that thing and saying, "You know, Dave, you really ought to get one of those."

Joe [Joseph A.] Gardella [Jr.], who's now on the faculty at SUNY [State University of New York, University at] Buffalo, was my first student on polymers. We started doing SIMS of polymer surfaces. We learned a fair amount with a small quadrupole mass spectrometer. Larry said, "You ought to really look at this Leybold instrument because I think it's better to do the kind of stuff you're doing than that SIMS." Since I was talking with the Leybold people, I went over and looked at it. In fact, I spent a week in Cologne measuring things. It's something you would never be able to do now, but I took a suitcase full of samples with me, through airport security. [laughter] We bought a LAMMA when I came back.

We worked with the LAMMA. We then decided that the SIMS we had was not a good enough unit. We needed a better one because it didn't have the resolution or mass range that we needed. We bought a new SIMS unit and put it together. After that, we had another program going with the LAMMA and the SIMS looking at laser and ion-induced mass spectrometry of polymers and other things. It didn't really give us much new information about catalysts, unfortunately. We put all kinds of catalysts in, but it never really panned out very well. We kept doing that and had that program running along with our XPS program in catalysis.

It must have been about 1988 or 1989 when [Michael] Karas and [Franz] Hillenkamp discovered the MALDI [matrix assisted laser desorption/ionization] process. We had been doing similar kinds of things, but we were never smart enough to think of that. We were immediately ready to get involved with that kind of research. I had gotten an Alexander von

Humboldt-Stiftung Prize from the German government to spend time in Muenster with Alfred Benninghoven. That started in 1984. We bought a time of flight SIMS after he developed it. We still have it and it's working very well.

I was very involved with mass spec, and the time of flight SIMS turned out to be absolutely marvelous for doing polymers. We could see much bigger pieces than we could see with the older quadrupole instruments. I started a whole program because we had a piece of hardware that could produce mass spectra from polymers like other people would die to get. With Benninghoven's background in instrumentation, it was a wonderful collaboration. We were running the ESCA program at the same time.

We were always driven by was our students' interest. Their interest may have reflected their professor's enthusiasm for things, but I began to get more students who were interested in the mass spec stuff than in the catalysis. The program began changing slowly from a major emphasis on catalysis to equal emphasis to even greater emphasis on mass spec. By the time I moved to Vanderbilt, I only had about four people doing catalysis stuff, and all the rest of the group was doing mass spec stuff. When I moved, I had a couple students finish up at Pitt on catalysis, but I only had one student at Vanderbilt who actually worked on catalysis. At that point, the group made a total transition. Now, we basically don't do any catalysis work. We do all mass spectrometry. If it's on surfaces, we work primarily with SIMS, and we use electropray and MALDI for looking at other things.

BROCK: Do you have an XPS?

HERCULES: We have two of them, but they are used as analytical tools. The major technique is mass spec.

DAEMMRICH: What persuaded you go to Vanderbilt? It sounds like you had a very nice setup in Pittsburgh.

HERCULES: Yes, I did. It was a fantasy, really.

I had been chairman of the department at Pitt for nine years. In 1989, I stepped down and [N.] John Cooper took over. I stepped down because I was tired of being department chair. I was not driven out. In fact, I tried to step down after seven years, and it took two years to get out of the job. I had always thought that it would be very interesting to go to a university that was a really fine institution, but had a chemistry department that really wasn't up to the rest of the university. There had been a number of institutions interested in me after I stepped down at Pitt, because I had gotten a reputation of being fairly successful in building up that department. It was probably more credit than I deserved because I just took over a department that was on the rise and made sure that it stayed on the rise. Nevertheless, I've got to take some credit for it.

I had a couple of other institutions approach me and ask me if I would like to be chair. I actually went and looked at a couple of them, but I couldn't generate any enthusiasm for them. Then Vanderbilt appeared. I had heard of Vanderbilt because Roy Albridge was there, and he'd helped me early on in the ESCA program.

When my daughter was going to go to college, I wanted to take her to see large, medium, and small institutions. Vanderbilt was one of the institutions I was going to take her to, but she said, "I don't want to go to a college that has more than two thousand students, Daddy." She went to Juniata College.

I knew something about Vandy [Vanderbilt], and it certainly qualified as an outstanding institution. They had had a decent chemistry department, but there had been lots of retirements. Some of their people who had been doing research had sort of fallen into inactivity. The department had stagnated for some other internal reasons I won't go into—personnel problems, and so forth—and their administration figured they needed somebody to come in from the outside and lead them in a new direction. I said to Shirley, "How would you like to live in Nashville?" Our daughter was at college and our son was in boarding school; both kids were gone. We were very happy in Pittsburgh, but Shirley visited Vanderbilt with me. We liked it and talked to the people. They made me a very nice offer, and we decided to go. I think, overall, it was a good move.

BROCK: How is it going with the department?

HERCULES: I think the department is moving along very nicely. Maybe not as rapidly as I had hoped, but I'm always very impatient. On the other hand, I think we've made a lot of progress. The people at Vanderbilt and the outside community say the same thing. If they think so, then I think so, and so I'm certainly happy with it. I'll be chair for another year or two.

[END OF TAPE, SIDE 5]

DAEMMRICH: Did you chair any Gordon Research Conferences in the early 1960s or 1970s?

HERCULES: I chaired the analytical Gordon Conference. That would have been in the 1960s, I think. I'd have to go back and check.

DAEMMRICH: In the early 1970s, did you do the electron spectroscopy conference?

HERCULES: Yes. I actually initiated that conference.

DAEMMRICH: Tell us more about that.

HERCULES: We had the first international conference, the Asilomar Conference on Electron Spectroscopy, which Dave Shirley initiated. We also had the Namur conference which happened somewhat later. I felt that we should have another conference on electron spectroscopy. I called Dave Shirley and said, “Why don’t we do it in the framework of a Gordon Conference because that way it can be a recurring thing on a periodic basis? As the field changes, you can change the nature of the conference.” He thought that sounded like a pretty decent idea, so I approached the Gordon Conference people, they liked the idea, and we ended up with the Gordon Conference.

DAEMMRICH: How was the conference funded?

HERCULES: At that time, they gave a certain amount of funding. We had some outside funding too, because it was easy to get. Electron spectroscopy was a new area and was of interest to everybody. We got money from some external agencies—I don’t even remember who they were. We had the usual support from the conference for some number of speakers or whatever, but we also had money to help students to attend. A number of graduate students and post-docs came.

DAEMMRICH: Part of the original thought of the Gordon Research Conferences was to have people from the academic setting speaking with people who were in industrial research labs. Did that happen?

HERCULES: It was mostly academic at that point. There were some people from industry there. What I tried to do was to get the people who were the leaders of the field to come in and talk about the whole thing. We had some people from government labs and industry, but it was mostly an academic group.

DAEMMRICH: It must be difficult for people to talk about the research that they did without being in the lab, near the equipment. How do you go about doing that when you’re suddenly on a campus in New Hampshire?

HERCULES: It’s just like giving any other kind of talk. You can talk about results. The thing about the Gordon Conference that is so important and unique is that the talks themselves are

generally unimportant. They just stimulate discussion, and you have a lot of discussions informally. That certainly occurred in those particular meetings. I remember going over to the social place and at 10:00 pm or at 11:00 pm there were still groups sitting around talking. That said to me that it was a successful conference.

It continued for a while. It moved more toward the physics community, away from the ESCA kind of spectroscopy to the many other forms of electron spectroscopy. It does not exist at the moment, as far as I know. It went for a while, and that's the way Gordon Conferences should be. When there aren't new things going on in a field, then you should bring in another conference.

BROCK: I would like to ask you about the period from 1995 to the present, when SIMS and MALDI were your major tools. What did you do with polymers and where are you headed next?

HERCULES: With the SIMS, we could get on the average peaks up to mass 2,000 or 3,000 for virtually any polymer. We learned a lot from that. We went back to the idea of structural interpretation, because you get fragment ions with the SIMS.

The question is: can you learn something about the structure of the polymer from the fragment ions? That's what we spent a lot of time on and published a whole pile of papers about. I think that the answer is yes, so it was certainly worthwhile. MALDI could measure polymer distributions up to mass 100,000 or more. That tells you one thing about the polymer, but the SIMS still has the advantage in that it gives you fragment ions and you can learn something about the structure.

The second, and even more important, thing about polymers is that when you start studying copolymers—especially copolymers where there are blocks of monomer units—you see surface segregation. Let's take polyurethane, for example. It has what they call hard blocks and soft blocks. The soft block is typically something like a polyester. The hard block is diisocyanate and whatever goes with that. In general, the thing with the lowest surface free energy will segregate on the surface when you make a polymer film. A polymer surface, like any other surface, is different from the bulk. The question is, which polymer component segregates to the surface—the soft or the hard?

SIMS is a great technique to tell you that because it really only samples about the first one or two molecular layers. Therefore, you can see changes in the surface structure with very small changes in composition. If you take a polymer that's 90 percent polystyrene and 10 percent polydimethylsiloxane, the spectrum is completely that of polydimethylsiloxane. You don't even see the polystyrene. Everybody says that's because the ion yield of polydimethylsiloxane is high and the ion yield of polystyrene is low. We measured them independently, and the ion yield of polystyrene is higher than the ion yield of

polydimethylsiloxane, yet you still only see polydimethylsiloxane if you have 10 percent of it in the polymer.

We did a lot of work on answering those kinds of questions. We published a paper not too long ago that actually looked at relative ion yields and showed that those surface segregation things are not an artifact (13).

When we started, we did some early work with polyurethanes. We said that it would be nice if you could somehow chemically break the polymer apart rather than rely on an ion beam. If you do it chemically, you can control it better. We did some chemical cleavage reactions which actually worked, but we still got fragmentation, so it wasn't that good.

With MALDI, that turns out to be something that's quite important. You might want to measure the molecular weight distribution of the polyester part of a polyurethane because they make the polyurethane by taking a small polyester and hooking them together. That's very difficult to do, and the wet methods for doing it are very long and very involved. It turns out that if you do a simple chemical cleavage reaction that cleaves the urethane bond, but doesn't cleave the ester bond, then you can just cut out the ester and measure it by MALDI. Then you have the molecular weight distribution. We looked at those kinds of problems with polymers as well.

BROCK: I'd like to ask you to reflect on your experience with analytical chemistry over the course of your career—the changes in it, its impact, and the big picture story as you see it.

HERCULES: I think I would go back to Art Cope's original comment: analytical chemistry is very important because it's aimed at finding out what you've done. It solves problems. We went through a stage where we sort of lost track of that, but now we have clearly returned to it. This has strengthened the discipline and the subdiscipline very much.

If you reflect on what an analysis is, you have to take a sample. You have to somehow isolate the thing you want to measure. You have to make the measurement. You have to interpret the result. Most academic analytical chemists spend most of their time making the measurement. We are a measuring tool oriented society, and I'm certainly as guilty of that as anybody. However, we seem to still remember those other things, and it's much more intellectually challenging and fun to teach about using those tools and how they work.

Sampling is difficult to teach about because you either do a mathematical analysis of taking basically random statistics or specific case studies, but you can't do it much in between. It's not something people spend a lot of time on. The awareness of that in the academic community has been changing, and I think the new stuff that's coming out in analytical chemistry is going to focus on that even more. Even in the basic research part, the problem is that the intellectual drivers of these problems are still practical things.

Take proteomics, for example. You have to worry about all these other kinds of issues. I would say that as a subdiscipline of chemistry, analytical chemistry is alive and doing pretty well. I think that it's an exciting field because we always have some new toy coming out that we can play with. What we have to do is to be creative enough to think about how that might be used to some advantage for something. Those are the questions that NSF is asking these days. If you can do that, then you can get a program funded and you can do it. My overall reflection is that in terms of hardware and measurement capabilities, we've come a long way from when I did my first instrumental experiments as a junior and senior in college, but the fundamental issues are still pretty much the same.

BROCK: Thank you very much.

HERCULES: My pleasure.

[END OF TAPE, SIDE 6]

[END OF INTERVIEW]

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