CHEMICAL HERITAGE FOUNDATION

HERBERT C. BROWN

Transcript of an Interview Conducted by

James J. Bohning

at

Purdue University West Lafayette, Indiana

on

11 November 1994

(With Subsequent Corrections and Additions)

ACKNOWLEDGMENT

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HERBERT C. BROWN

1912 Born in London, England, on 22 May

Education

1933	Crane Junior College, Chicago (school closed, 1933)
1935	Assoc. Sci., Wright Junior College, Chicago

- 1936B.S., University of Chicago
- 1938 Ph.D., inorganic chemistry, University of Chicago

Professional Experience

	University of Chicago
1938-1939	Eli Lilly Post-Doctoral Fellow
1939-1943	Instructor of Chemistry
1941-1943	Instructor and Research Investigator

Wayne State University

- 1943-1946Assistant Professor
- 1946-1947Associate Professor

	Purdue University
1947-1959	Professor of Chemistry
1959-1960	Wetherill Professor of Chemistry
1960-1978	Wetherill Research Professor of Chemistry

1978- Emeritus Wetherill Research Professor of Chemistry

<u>Honors</u>

1951	Sigma Xi Award, Purdue Section of Sigma Xi
1953	Harrison Howe Award, Rochester Section of the American Chemical Society
1955	Centenary Lectureship and Medal, The Chemical Society, London, England
1957	Elected to National Academy of Science
1959	William H. Nichols Medal, New York Section, American Chemical
	Society
1960	Award for Creative Research in Organic Chemistry, American
	Chemical Society

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1960	S.O.C.M.A. Medal, Society of Organic Chemistry Manufacturing Association
1961	Phi Lambda Upsilon Key for Honorary Membership, Phi Lambda
1962	Upsilon
1962	Honorary Member, Phi Lambda Upsilon
1962	Elected to American Academy of Arts and Sciences
1908	Linus Pauling Medal, Oregon and Puget Sound Sections, American Chemical Society
1968	Doctor of Science, University of Chicago
1969	National Medal of Science, Government of the United States
1971	Roger Adams Medal, Organic Division, American Chemical Society
1972	Phi Beta Kappa
1973	Charles Frederick Chandler Medal, Department of Chemistry, Columbia University
1974	Chemical Pioneer Award, American Institute of Chemists
1975	Madison Marshall Award
1976	City College Chemistry Alumni Award and Medal for Scientific
	Achievement, State University of New York, NYC
1977	Elected Fellow, Indian National Science Academy
1977	Honorary Fellow, Royal Society of Chemistry
1978	C. K. Ingold Lecture and Medal, The Chemical Society, London
1978	Elliot Cresson Medal, The Franklin Institute, Philadelphia, PA
1979	The Nobel Award and Medal, The Nobel Foundation, Stockholm, Sweden
1980	Medal of Culture, Ministry of Education, Taiwan, ROC
1980	Nobel Hall of Science, Museum of Science and Industry, Chicago, IL
1980	Honorary Member, Chinese Chemical Society
1980	Honorary Old Master, Purdue University
1980	Doctor of Science, Wayne State University
1980	Doctor of Science, Lebanon Valley College
1980	Doctor of Science, Honoris Causa, Long Island University
1980	Doctor Philosophiae Honoris Causa, The Hebrew University of Jerusalem
1980	Grado Academico Honorifico de Doctor Scientiae Honoris Causa, Pontificia Universidad Catolica de Chile
1981	Priestley Medal, American Chemical Society
1981	Doctor of Science, Purdue University
1981	Doctor in Scientiae Honoris Causa, University of Wales
1982	Perkin Medal, American Section, Society of Chemical Industry
1982	Elected Honorary Member, The Indiana Academy
1982	Honorary Member, Pharmaceutical Society of Japan
1982	Honorary Member, Chemical Society of Japan
1982	Corresponding Member, Academy of Arts and Sciences of Puerto Rico
1982	Doctor of Science, Butler University
1982	Docteur Honoris Causa, Universite de Paris-Sud

1983	A. J. Beckman Memorial Medal, Colorado School of Mines
1983	Honorary Member, Gold Key National Honor Society
1985	A.I.C. Gold Medal, American Institute of Chemists
1985	Doctor of Science, Ball State University
1986	Sixtieth Anniversary Commemorative Medal, Jewish Academy of Arts and Sciences
1986	Sesquicentennial Commemorative Medal, National Library of Medicine
1987	National Academy of Sciences Award and Medal in Chemical Sciences, National Academy of Sciences
1987	G. M. Kosolapoff Award and Medal, Auburn Section, American Chemical Society
1987	Dedication of the H. C. Brown Laboratories of Chemistry
1989	Medal of the Government of Japan: Order of the Rising Sun, Gold and Silver Star Emperor, Government of Japan
1990	Oesper Award, Cincinnati Section, American Chemical Society
1991	Honorary Member of the Academy of Natural Sciences of the Russian Federal Republic
1991	Corresponding Member, Academia Mexicana de la Investigation Científica
1994	Honorary Scholar, University of Wales, Swansea, Wales
1994	Honorary Professor, Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences
1995	University Medal of Highest Honor, Kyung Hee University, Seoul, Korea
1995	Distinguished Scholar Award, Indiana Academy of Science
1996	Visitante Distinguido, BUSA V-MEX, Mexico
1996	Visitante Distinguido, La Universidad de Guanajuato, Mexico

ABSTRACT

Herbert C. Brown begins this interview by describing his high school education and the death of his father, which forced him to follow a practical path in seeking work and eventually a college education. Brown next discusses his coursework and studies at Crane Junior College, where he became fascinated by chemistry and its history; when Crane closed down, Brown was among the students invited to work in Nicholas D. Cheronis' Synthetical Laboratories, where he earned enough to enroll in a University of Chicago correspondence course on qualitative analysis and supplemented his education by working with Fales's Quantitative Analysis. Brown continues the interview by recalling the details of his studies and lab work at Wright Junior College and the University of Chicago. He also describes his relationships with his fiancee, Sarah Baylen; his mentors, Juliuis Stieglitz and Morris Kharasch, who influenced the direction of his inorganic research and the path of this career; and Neil Gordon, who offered Brown a position at Wayne State University and supported his research efforts there. Brown recalls the course of his career decisions and research at Chicago, Wayne State, and later Purdue University, including details of studies on steric effects, boranes, and borohydride synthesis. In the second part of the interview, Brown discusses his WWII work for the National Defense Research Committee, which included research on the volatile compounds of uranium, uranium borohydride production and testing, sodium trimethoxyborohydride production, and sodium borohydride development. He concludes the interview with an overview of his post-war research at Purdue, which focused on reduction studies comparing sodium borohydride and lithium aluminum hydride, hydroboration discovery, explorations into organoborane chemistry, and the development of a general asymmetric synthesis program.

INTERVIEWER

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

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INTERVIEWER:	James J. Bohning
LOCATION:	Purdue University West Lafayette, Indiana
DATE:	11 November 1994

BOHNING: Dr. Brown, I know that you've written a lot about your early background. You were born in London on May 22, 1912, and came to Chicago with your family in 1914. Your high school education was interrupted by your father's death, but you did graduate from high school in 1930. In those early educational years in Chicago, did you have any teachers who inspired you or had any influence on you?

BROWN: No. I was not inspired to, let's say, try to achieve a certain career. I didn't have any hopes of going to college because of my father's death. Consequently, after I got out of high school, I spent three years trying to find a job to help support my family. But I couldn't. I finally decided that I'd better go back to college and see if, through further education, I could bring myself to a position where I could find a job.

BOHNING: Did you have any chemistry in your high school experience?

BROWN: I had a very good course in chemistry, but that's not what inspired me. There was actually a Ph.D. teaching at my school. I think it was a Dr. Smith and I enjoyed him; I enjoyed all of my courses. [laughter] I enjoyed high school. I enjoyed studying everything from Latin to German to physics, and chemistry I always enjoyed. I always did well in those subjects.

BOHNING: But I understand that when you went to Crane Junior College in 1932, you had intended to major in electrical engineering.

BROWN: Someone had told me that electrical engineers made very good money. [laughter] To me, that was the most important objective in my life, the reason for going to college, so I registered for a degree in electrical engineering.

BOHNING: But it was during that time at Crane that you switched to chemistry.

BROWN: I took chemistry the first year; all engineers had to go through chemistry in the first year. I did very well in it. I fell in love with the subject. I decided to forget about the money and concentrate on chemistry for my future career.

BOHNING: What was it about chemistry that fascinated you?

BROWN: I have a very good memory—or at least, I should say, I had a very good memory and I could remember all the things. I did very well in the course, because I could remember all the details, which most of the class couldn't. Consequently, I would do quite well in the course work and exams. I did a lot of reading, and I read a lot of history. I was <u>fascinated</u> by the subject, the way it had developed and had led us to the state of chemistry in 1932.

BOHNING: What were you reading? This was history of chemistry you were reading?

BROWN: Various books. As I remember, there was a general chemistry book by Lowry which presented chemistry from the historic viewpoint (1). I was fascinated by his treatment of the subject.

Actually, in the summer after I finished my first year at Crane, Crane was closed because of a lack of funds. I decided I would write my first textbook in chemistry. I wrote it that summer and paid my younger sister, four years younger than me, five cents an hour to type it. [laughter] I have it around somewhere, but I haven't looked at it for years.

BOHNING: I was going to ask if you still have the typescript of that; it would be interesting.

What kind of laboratory experience did you have at Crane?

BROWN: Just the usual course work.

BOHNING: Qualitative analysis?

BROWN: No. It was just general chemistry. One year. There were two courses there, and that's as far as I went there.

BOHNING: I understand that when Crane closed, Cheronis made his labs available.

BROWN: Nicholas D. Cheronis, yes. He operated a small commercial laboratory in the converted garage of his home, called Synthetical Laboratories. He made [W. M.] Clark and [H. A.] Lubs indicators and other high-cost materials, sold them, and augmented his income in that way.

He invited around ten of the students at Crane to come there and keep busy in the laboratory, <u>not</u> to do any work for him, but do whatever experiments we wanted to do, just to keep us off the streets. That's where I went, and that's where I managed to find the money to register for a correspondence course given by the University of Chicago, on qualitative analysis. That's where I did the laboratory work for which I later received credit at the University at Chicago. I also went through Fales's *Quantitative Analysis* (2), did all the experiments. I kept a

good notebook. Later on, when I went to the University of Chicago, I showed [W.] Conway Pierce, who was the professor there in quantitative analysis, that notebook, and he gave me credit for the course. [laughter]

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BOHNING: That's amazing. I have a couple of questions about that. Where was Cheronis's home located?

BROWN: North side of Chicago, Ardmore Street, as I remember.

BOHNING: Where were you living?

BROWN: I was living on the west side of Chicago.

BOHNING: So you had a trip to make then.

BROWN: Ogden and Homan. It was a bus trip. Time was no factor; there was no TV to watch. [laughter]

BOHNING: Who paid for the chemicals and things that you were using for the experiments?

BROWN: No question was raised about the chemicals. The things I used were not very costly. [laughter] They were chemicals that were around the laboratories. After all, his was an operating laboratory, so no one ever raised the question there. But the correspondence course, I think, cost me about sixteen dollars for the course. [laughter]

BOHNING: I was curious how they would expect someone to do qualitative analysis by correspondence. You had an unusual situation in which you had a laboratory available, but how did Chicago expect people generally to do this?

BROWN: I don't know. All I'm saying is that one time I got an unknown that didn't behave right. The professor giving the course was listed as Julius Stieglitz, so I called him up. I explained all my difficulties, and he said, "Your unknown must have phosphates." Of course they weren't supposed to, so he said, "I will see that a new sample goes out to you." Later on, when I went to Chicago, I found out that Julius Stieglitz was the august professor [laughter] and actually at the time, emeritus professor of the University of Chicago. I took his organic course; I was very much impressed. He was officially in charge of the correspondence course, but the course was actually being run by one of his graduate students working for her Ph.D.

BOHNING: In September of 1934, Wright Junior College opened. I'm curious about these junior colleges in Chicago; I really don't know anything about them.

BROWN: At the time, Crane was the only one. There was only one junior college in the whole city of Chicago. There was no charge for tuition. I went there, and that's where I met the girl who was to become my wife. She was sixteen years old, studying chemistry. Her name was Sarah Baylen, and she was just a baby at the time, I thought, because I was nineteen. [laughter] She hated me originally because she had been the brightest student in the class until I joined, and she didn't like to be number two; later on, she forgave me. She was one of the students who went to Cheronis's laboratory.

So we went there and spent the year there. I did correspondence school work, and I went through quantitative analysis. I was reading Fales, answering every question in the book and then doing the experiments that were recommended; that's the way I got my quant.

Then the City of Chicago, with President Roosevelt in office, must have gotten federal money. Somehow they opened up three junior colleges—Wright Junior College on the north side, Herzl Junior College on the west side, and Woodrow Wilson Junior College on the south side. Nicholas Cheronis was appointed director of the Physical Science Division at Wright Junior College, and that's why I went there. Although I was living on the west side and Herzl was very close to me, I went there to be with Cheronis.

BOHNING: What type of chemistry did you do? Let's see, you graduated there the following year. Is that correct?

BROWN: In 1935, I was one out of nine students who graduated.

BOHNING: In all areas?

BROWN: Most of the other students who had done it at Crane had somehow managed to complete their education, some going to Illinois or other institutions, but I simply didn't have the funds to do that.

BOHNING: What type of chemistry did you do at Wright, then? Organic?

BROWN: I took organic chemistry. Nicholas Cheronis was writing a book on teaching organic chemistry with semimicro quantities (3) and he asked us to do many tests and many experiments. I also took physics, and they gave me a free hand. I was permitted to experiment with all the physical equipment that was around there. I used to publish a Physical Science Weekly, and I'd write it, mimeograph it, distribute it, and hold a monthly museum at which I'd demonstrate equipment.

I had a wonderful time there, and I could experiment there. One time I read about the Foucault pendulum, and I went to the engine house where the ceiling was accessible through three or four floors. I assembled several heavy weights and suspended them from the ceiling to just above the bottom floor. I caused the pendulum to swing and followed the plane of its

motion for a full day. The experiment worked as described.

So it was a wonderful education there. I did very well. One time, we had a Professor Quick who was head of physics; he gave a test to our class. The scores were abysmal! I think the average score was about twenty points, but I got a 96. [laughter] The class, and Sarah, hated me.

BOHNING: Interesting. Were you still, at this point, saying you were going to get out of college, get a job, and help your family?

BROWN: Yes. That was my ambition.

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Now, I had so impressed the faculty at Wright Junior College that they recommended I apply for a competitive scholarship to the University of Chicago. For this I had to take an examination. I went down there to take the examination, sat down with the exam, and to my amazement, it had almost <u>nothing</u> to do with chemistry, physics, or mathematics. You see, my education had been the old classical one of chemistry, physics, mathematics, German, English; I had nothing on the humanities, history, philosophy, art, and so on. [laughter] The whole damn exam was based on all of these subjects, and so on, because of President Robert Maynard Hutchins at the University of Chicago, who was then arguing for a liberal education based on the Great Books.

Well, it was hopeless, but I decided that as long as I was there, I might as well do my best. I sat down and did what I could, and to my amazement, I did win a competitive scholarship that permitted me to go to the University of Chicago.

Now, at that time, Hutchins was arguing that people should go through college as fast as they could. They shouldn't just go and set off on a four-year pace. It cost no more money to take ten courses per quarter—that's compared to the usual three—so I took the ten courses; I managed to fit them in. There was a famous professor there from Germany, Karl Ziegler, later a Nobel Laureate, who was teaching, and he gave courses based on his research. They were trying to entice him to come there as head of the department, so I included his graduate courses, although I was still an undergraduate.

I took the famous course of Julius Stieglitz, the advanced organic course which he gave there. He was such an excellent lecturer that they used to send people over from the English department to hear him lecture, as an example of how to give good lectures. I managed to finish both my junior and senior years in three quarters, nine months, and I was scheduled to graduate in June 1936.

BOHNING: What kind of laboratory experiences did you have or could you fit in with all of these courses that you were taking?

BROWN: Well, they gave me credit for the quantitative analysis. I took physical chemistry. I took a course in qualitative organic analysis and also the organic preparations laboratory. I had taken a full year of Julius Stieglitz's lecture course. Ziegler gave half-credit courses: his work on large rings, his work on lithium alkyls and also on free radicals. I took each of those courses. I was supposed to take a course in biology, but I didn't do that.

At the University of Chicago, when you reached the bachelor's degree, you had to take comprehensive examinations. I'd never had a course of biology, but I'd read *The Outline of Life*, by H. G. Wells (4). I sat down and spent one weekend re-reading that book. I went in to take the exam and passed it. [laughter]

I was left with a very low opinion of the people who take humanities and history. How could I compete with them, when <u>all</u> I had was a general education from reading newspapers and my general reading, but I'd been able to win a competitive scholarship, and I was able to pass these comprehensive examinations.

Now, I was planning to leave school with my bachelor's degree, find a job, and marry Sarah Baylen.

BOHNING: Did she go to Chicago with you?

BROWN: She went to Chicago with me. She was unusual. She was registered as an engineer at Crane Junior College. She was the <u>only</u> girl in the engineering school! They must have had a thousand students in engineering. No girls. Only Sarah. [laughter] When I saw her, I said, "Sarah, why in the world are you studying engineering?" She said, "I have a great hunger to see the world, and if I get a degree in chemical engineering, I figure I'll be going to Australia to put up a chemical plant, and I'll be going to Chile to put up a chemical plant; I'll be going to India to put up a chemical plant, and I'll have a chance to see the world."

Well, she's seen the world, but not through that mechanism. She had been promised a scholarship to Northwestern University. By that time, we were close and planning and hoping to get married. She decided to forego that scholarship and came to the University of Chicago and worked her way through school. She had also lost her father at an early age.

BOHNING: I know that you were persuaded to stay at Chicago by Stieglitz, but had you gone out for any job interviews? Had you started that process before you changed your mind?

BROWN: No, I hadn't reached that phase yet, because I was very busy with ten courses, [laughter] and having to take these comprehensive examinations. I was planning to get my bachelor's degree, and after I was certain I had passed all of these things, I would then go out and get a job. But I hadn't reached that stage when Stieglitz called me to his office and told me that he had seen me in his class, and he thought I would do very well with a Ph.D. degree. He was surprised I hadn't applied for a teaching assistantship which would allow me to go on for a doctorate.

I told him I had met this girl whom I wanted to marry, and in those days, you didn't get married without a job. [laughter] Parents were neither willing nor able to support young couples. He told me he would advise us to let our marriage wait while I went on for a Ph.D. degree.

I discussed it with Sarah, and to my amazement, she agreed that we should follow his advice. So I went and signed up for a teaching assistantship. It didn't make life very easy. As a teaching assistantship, they paid me four hundred dollars a year, but I had to pay the tuition, which at that time was one hundred dollars a quarter for three quarters; the summer quarter was free. It gave me one hundred dollars a year to live on. But I sold shoes on Saturdays, I did some tutoring, and so I was able to make it. [laughter]

BOHNING: We've reached a point here. I'd like to sort of put to the side the chemistry you were doing until later and look at some other aspects of the time from there to the time you got to Purdue. Then we'll come back and look at the chemistry.

BROWN: Well, one thing I will say, though, was that when I was getting my Ph.D., and I was getting it in two years, I had a job interview with the Sherwin-Williams Paint Company, but I didn't persuade them that they needed me. I had one in the Patent Department of Universal Oil Products Company; I didn't persuade them they needed me. When things looked bleakest, M. S. Kharasch offered me a post-doctorate fellowship. We have many people who've ended up in industrial work because they couldn't find an academic job. I'm a rare example of someone who ended up in academic work because he couldn't find an industrial job.

BOHNING: What kind of a person was Stieglitz? Could you tell me something about him? Did you interact with him a lot?

BROWN: Julius Stieglitz was the brother of Albert Stieglitz, who made quite a reputation for himself by marrying Georgia O'Keefe and was a photographer in New York. Albert Stieglitz was trained in medicine but never practiced as an M.D. Julius Stieglitz took a degree in chemistry and, to get a job, he had gone to Clark University, which was the first school in the United States to offer Ph.D. degrees.

Clark was a small college started in Massachusetts by the Clark who was head of the Clark Thread Company. Even today, we use Clark sewing thread. They developed a very good faculty there. I think they had Stieglitz, and I believe Robert A. Mullikan was there. When the University of Chicago started up, the first President, William Rainey Harper, managed to persuade many of them to come to the University of Chicago, so the University of Chicago acquired an outstanding faculty by raiding Clark.

I gather Stieglitz's parents were independently wealthy. His father was a wool merchant. He later became head of the department at the University of Chicago. He had done many interesting things. His was the first book on qualitative analysis that gave it a mathematical basis. I still have those books, *Qualitative Analysis* by Julius Stieglitz (5). He was the one then who taught qualitative analysis in the extension center, Home Study Centers, and whom I called that time to clarify my problems with one of the samples.

He had been elected president of the American Chemical Society. He also became very active in introducing medicine to organic chemistry, and he wrote a book, *Chemistry in Medicine* (6), so he was very influential in a number of respects.

He did some rather interesting research. He was one of the first people in the U.S. to introduce the electronic theory to organic chemistry, and he discovered a rearrangement called the Stieglitz Rearrangement, which for many years was taught as part of organic. I don't know if it still is. He had a very large group of students, many of whom went around the United States and became faculty members of many universities. When I later went to Wayne University, Ed Remick was there, and he was a Julius Stieglitz product.

[END OF TAPE, SIDE 1]

BOHNING: What was Stieglitz like as a person? You worked with him; how was he?

BROWN: I didn't work with him, no. I worked with [H. I.] Schlesinger.

BOHNING: That's right. Okay.

BROWN: Actually, Stieglitz was also instrumental. I don't know if I should tell a story about how my wife gave me this book. But Stieglitz also encouraged me to go into inorganic. He thought that this was a field that was right for development and it would be a good field to get into.

BOHNING: I'm interested in that because the story of the Stock book (7) that Sarah gave to you is well documented (8), but I don't remember hearing about Stieglitz's comment about inorganic.

BROWN: No, I don't think I ever put it down. At that time, inorganic didn't attract the best students. I don't know whether Stieglitz wanted to encourage good students to work with Schlesinger; actually, Schlesinger had been one of <u>his</u> students. Even though he had gotten his degree in organic, he was invited back to Chicago to develop inorganic. John Bailar of the University of Illinois was also an organic major who became an inorganic chemist.

When I went to see him to discuss the fact that I had become interested in this field of inorganic, he thought it was a good idea to do my doctorate in this field. So he's one of the people who encouraged me to specialize in inorganic. You know, I didn't do it lightly, because at that time, people looked down upon inorganic as a field of research, and there were very few students getting Ph.D.s in it.

BOHNING: That actually continued for some time after that.

BROWN: Yes.

BOHNING: In a quote here, you said, "There was no interest in synthetic inorganic chemistry at this time, possibly because of the pervading influence of the Berkeley School"(9).

BROWN: Yes, I agree. I've forgotten I'd written that, but sure, because everyone there was emphasizing kinetics, and neglecting the exploration of new descriptive inorganic chemistry.

BOHNING: This was [Joel] Hildebrand and [Wendell M.] Latimer.

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BROWN: Yes. One of the books that I used at the time was *Inorganic Chemistry* (10). Cheronis thought that that was a wonderful book, Latimer and Hildebrand, so we used it as a second text. We used Deming (11) plus Latimer and Hildebrand.

BOHNING: Again, I would like to come back to the chemistry and treat it separately, but later. After your Ph.D. in 1938, you stayed on as a postdoc with Kharasch.

BROWN: Well, when I couldn't get a job, things looked very, very bleak. Morris Kharasch called me into his office and invited me to stay on as a postdoctorate. He offered me twelve hundred dollars a year as a postdoctorate stipend. I thought I was worth two thousand dollars [laughter] and we compromised at sixteen hundred dollars, but later on we had a development which caused him to spontaneously give me an additional four hundred dollars.

BOHNING: He wrote a personal check to you for that?

BROWN: Yes. Well, when we come to it, I will explain what happened. [laughter]

BOHNING: Okay. What kind of a person was Kharasch?

BROWN: Well, I liked him very much, personally. He was a very competent person, energetic, and he was imaginative. He was easy to get along with; I never had any trouble with him.

BOHNING: How would you compare him to Schlesinger?

BROWN: Schlesinger was more reserved. Again, I've never had any trouble with him either, but I'm saying he was not someone you got to know closely.

BOHNING: Kharasch was younger?

BROWN: Yes. Kharasch got his degree in 1918 from a Swiss chemist who later became famous as a balloonist going up in the stratosphere, [Auguste] Piccard. [laughter] At that time, he was a young professor at the University of Chicago, whereas Schlesinger had gotten his degree, I think, around 1905.

BOHNING: Now, you stayed at Chicago through the war years, but you at one point asked for tenure and were told that it would not be coming. Is that correct?

BROWN: Yes. Well, what happened was this. In 1939, after I'd finished one year with Kharasch, Anton Burg was told he would not get tenure. I should explain that at this time, the decision on tenure was usually made in the tenth year. A man would start as instructor. He'd be an instructor for nine years and then be promoted to tenure at assistant professor level in the tenth year or else be asked to leave.

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I'd seen several people treated this way. There was an R. W. Johnson who was an instructor in organic chemistry; there was a Kenneth Adams, who was half-time curator and half-time instructor in organic chemistry, and Anton Burg. Anton Burg got a job at the University of Southern California where he did very well, and where, at ninety, he's still active. [laughter] Anton Burg was Schlesinger's research assistant.

They hadn't been able to find a head of the department. They tried to bring in [Leopold] Ruzicka in 1935, and they weren't able to persuade him to come there. They then invited Karl Ziegler in 1936, and I took his courses, but they didn't persuade <u>him</u> to come. They tried to get Linus Pauling and didn't succeed. So, finally, they made Herman Schlesinger executive secretary of the department, and he handled the routine. He was given a research assistant, paid for by the university, in compensation for all of the work he was doing for the department.

So, after I finished one year with Kharasch, Herman Schlesinger invited me to take a position as his research assistant to replace Anton Burg. Anton Burg has always been very unhappy with me, because he figures that if I hadn't been at the University of Chicago at the time, he would have gotten tenure. It's nothing I could do anything about, but when I submitted papers to the journal, I used to ask that they shouldn't send them to Anton Burg, because he always was very caustic. [laughter]

Now, I had seen this, and I didn't want to wait. We got involved in war work. We had a group of twenty-five people that I was largely instrumental in directing. We opened up a major new field in research. I decided that if I was ever to find out whether I could get tenure, it would be then. I therefore asked Schlesinger in 1943 to find out from the department whether I had any future in the department, because I was in a special position. I was not an independent worker; I was his research assistant. When the answer came through that there was no future for me, I decided to leave.

I had confidence in my ability. I could see that whenever I had competed with others, I did very well, but it was during the war and it was very difficult to get a job. Schlesinger never did anything to help—I guess he just hoped that I would stay—but Morris Kharasch helped.

Morris Kharasch had gotten his first job at the University of Maryland. The chairman of the department at that time who had given him the job was Neil Gordon, the man who started the Gordon Research Conferences. Neil Gordon had gone from there to Johns Hopkins where he introduced a major program involving fellowships. There was to be one fellowship from each state, supported by industry in that state. He had come to Johns Hopkins because it was one of the pioneer schools in graduate education. The program was going very well.

The department didn't like him because he was always involved in various programs and not paying attention to his departmental duties. They finally made things difficult for him, so he went to a college in central Missouri called Central College. He became head of the department there. [Samuel C.] Hooker was the name of the man who owned a major library; he was a sugar chemist in New York City, and this was his private library.

BOHNING: Oh yes, yes.

BROWN: Neil Gordon wanted that library. He persuaded the college there to buy it and brought it there, but the college didn't have enough money to maintain it, so he opened up a translation service and other things the library could do. Various companies would become members and help support it, and it worked.

Then in 1942, he was invited to Wayne University as head of the department. Wayne University was a city school, operated by the Board of Education. It had gone from a four-year high school to a junior college, then to a four-year college. They were beginning to develop a graduate program, first offering master's degrees, and they wanted to start a Ph.D. program. He was brought in to improve the department, and he offered me a job to come there and help get research started at Wayne. At that time, the teaching load was eighteen contact hours. I told him I couldn't possibly get anything going there with a contact load of eighteen hours, so he agreed to give me a teaching load of twelve hours. Thirty-two hundred dollars, twelve-hour teaching load, and that was the only offer I got, so I went there.

BOHNING: Had you looked at any other places or made inquiries at any other places?

BROWN: I'd written a dozen letters or so to various schools, but during the war they weren't hiring anybody. I didn't care where I got started, as long as I could start.

I went to Wayne and things were a little difficult for me. First of all, my wife was pregnant, and she was having a difficult pregnancy. When I went to Wayne, the only housing I could find was to buy a new house they were putting up for the war. It cost eight hundred dollars down, sixty-six hundred dollars the total price, but we had only accumulated by this time about six hundred dollars. [laughter] I had to borrow two hundred dollars from my family; they were not well off, but we managed to do it.

Unfortunately, a problem developed at Wayne. The faculty learned that I was getting a teaching load of only twelve hours. I had arranged to teach a course in undergraduate organic chemistry, given in the evening—Monday, Wednesday, and Friday, four hours each, laboratory and lectures. That would take care of my twelve hours, so I had the days free to get my research program underway.

The faculty was very unhappy. They trooped in to see Neil Gordon and said, "We also want to do research. How can you give the new man twelve hours, and we're required to do eighteen?" So he called me in and asked what he could do. Well, I suggested a solution. I said, "The chances are that anybody who's been out of research for ten or twenty years will find it difficult to get back into research. What you could do is offer each of the staff, at a certain rotating basis, a sort of sabbatical in residence. They would get twelve hours during one year. If they managed to produce any research and had it published in a reputable journal, you would reduce their teaching load by two hours per publication, so that if they published three papers during the year, they'd get it reduced for next year to another twelve hours, but if they didn't, they'd go back to eighteen."

The faculty bought this, and I got busy and I wrote up some things I had done. I wrote a paper on doing sulfuryl chloride chlorinations as experiments in organic chemistry, published in the *Journal of Chemical Education* (12). I wrote up a review on sulfuryl chloride chemistry (13), and I managed to get out a total of six papers during the year, so my teaching load for the following year was six hours, and no one could object. [laughter]

BOHNING: How many of the others managed to reduce their load?

BROWN: None. [laughter] They tried, but didn't succeed.

BOHNING: Things were pretty spartan at Wayne when you started there, in terms of doing actual research.

BROWN: Yes. They gave me a laboratory on the corner of Cass and Warren, a room. It was a large bare room; it had been a classroom. In one corner, there was a sink with hot and cold running water and a couple of double outlets, but that was all.

It was filled with all kinds of packages of glassware. The faculty had to take all the stuff out and move it to the storeroom, and then I had to start planning for my vacuum line. What I did was make a T made for the water pipe. On the T we put on six faucets, so that in one bunch, we had six different outlets of water, and they could go by overhead glass lines to a vacuum line and back again. We put in multiple electrical outlets and so on, and later on, we bought the desks. After I'd gone, that laboratory was inherited by Carl Djerassi, [laughter] and he did much of his work there.

Now, surprisingly, I was not really discouraged about the possibility of getting some good research done there. At the University of Chicago I had heard from a physical chemist, Simon Fried, a student of G. N. Lewis at Berkeley, a story about G. N. Lewis. In 1905, G. N. Lewis, who had a new Ph.D. in chemistry, was employed by the Bureau of Weights and Measures, and he was located in the Philippines. His official duties didn't take up much of his time, and he wanted to get some research going. Even then he was interested in thermodynamics.

He wanted to study the dissociation of silver oxide, to measure the dissociation as a function of temperature so he could calculate delta H, delta F, and entropy. This would require measuring high pressures at very great accuracy, normally requiring very expensive equipment. But there was actually no equipment available in the Philippines at that time, so what he did was take thick-wall capillary glass tubing. He sat down with a glass-blowing torch and constructed an apparatus which could measure the high pressures at different temperatures. In this way he obtained the data he wanted and published the study in a paper which appeared in JACS for 1906 (14).

Sixteen years later, in 1922, Professor F. G. Keyes at MIT decided this experiment ought to be done right. He invested many thousands of dollars for an autoclave that would measure

pressures with high precision and repeated the experiment. The results were exactly the same. This also appeared in a publication in JACS, in 1922 (15).

This story had been told to me, so I said, "Well, I can't do the experiments I was doing in Chicago, but I can do other things." I decided to study the chemical effects of steric strain. I could have my students in the organic course run Grignard reactions and make highly-hindered alcohols for me. I could take these alcohols, treat them with concentrated HCl, convert them to the chlorides, take the chlorides and study the rate of hydrolysis in eighty percent alcohol-water, titrate them with a burette using standard sodium hydroxide and phenolphthalein, and determine the rates of hydrolyses. That's how my program on steric effects began, and that was the work that got me invited to Purdue University, four years later.

BOHNING: You've commented in writing about this aspect, too, of being able to accomplish good results without the expenditure of huge amounts of money and fancy equipment (9).

BROWN: All you need is imagination. And I think more students should be taught this, because many of them go on to a small college and then can only think of all the equipment they had where they had been, instead of thinking of what they could do with what they have at hand.

BOHNING: You were at Wayne four years. Had you thought of leaving when you got the offer from Purdue?

BROWN: Well, in 1946, I was invited to Ohio State to give a lecture. Mel Newman decided that he wanted me to join him at Ohio State. There was a professor, Ed Mack, who was head of the department. I was down there to give a lecture, and I told them I was interested in inorganic, organic, and physical chemistry. I described some of our results in these areas. The Head was not impressed. He told Mel Newman, "He'll never amount to anything; he's interested in too many things," so he didn't give me an offer.

Then, at a Gordon Research Conference, I met Henry Hass. He invited me down in the summer of 1946 to give a series of lectures here at Purdue. I gave those lectures, and he was impressed. Then he decided the next year, when there was an opening in the inorganic division, to invite me as Professor of Inorganic.

I was told that Henry Hass told one of his students, with whom I later became very good friends, that when he presented this to the department, every member of the department voted against it—except one. There was a physical chemist there who was a student of G. N. Lewis—Roy Newton—and he was in favor, and Henry Hass was in favor. The others were not, but at that time, we operated as a headship, and he didn't have to pay attention to what the staff wanted. He hired me, and I came down here. Now, he gave me, as I recall, fifty-five hundred dollars annual salary. Wayne wanted to keep me; they offered me six thousand dollars. See, I was then associate professor at Wayne. They were going to make me a full professor—and six thousand dollars.

But I came here. At that time, my wife was very unhappy in Detroit. She is an exceptionally intellectual person. At the University of Chicago, she had participated in the life of the university, going to lectures and so on. Wayne was situated in the central part of Detroit, so it was not possible to live anywhere close. Practically all the faculty lived in the outskirts of

the city of Detroit. They were all scattered, so there was no university life at all. She had a young son and was getting very frustrated because there was no way she could attend lectures.

We have a nice little town here. When I came here and looked up at the sky and saw the Milky Way, I said, "This is the place to bring up a young child and make my wife happy," so we decided to come here.

[END OF TAPE, SIDE 2]

BOHNING: What kind of reception did you get? Were you aware of this feeling, on the part of the other faculty, that they had?

BROWN: No, [laughter] that was told to me, later on, by a friend. My wife, in junior college, had a very good friend, a Chinese girl, Florence Lee. She married a student who had gotten a Ph.D. degree with Hass, named [T. H.] Chao, and he was not able to find a job. At that time, I had become a member of the staff of the University of Chicago. I went to Morris Kharasch, and I told him that I thought Chao was a very competent person, and would he please offer him a position; he did that. He came and worked with me and Morris Kharasch; we did a lot of work. Then he got a job at American Cyanamid. They were always very grateful; we've been friends all our lives. He was the one who told me that Henry Hass had told him in confidence about the reaction to my joining the faculty. [laughter]

BOHNING: I'd like to go back now and look at some of the chemistry that was going on. You've documented a lot of this, but I want to quote from your introduction to your book, *Boranes in Organic Chemistry* (9). I was very intrigued by this statement, and I quote: "Perhaps in the effort to bring our students to a level of competence in the leading edge of chemical progress, we have been denying them significant contact with the past. Perhaps if they had more experience with the difficulties under which we operated, they would feel more optimistic about the future possibilities for chemistry."

I was very intrigued with that. I wonder if you could elaborate a little more on that comment.

BROWN: Well, I agree. You see, I knew what G. N. Lewis had done in the Philippines. That encouraged me not to give up, but to find a way of doing what I wanted to do with the equipment that was at hand at Wayne University. Today, I'm afraid that we're so intent on teaching the latest ideas to them that we don't tell them where they came from. In general, my education in chemistry was much better because I had read things on the history of chemistry; I had traced it back. Actually, my wife and I, when I was just starting at the University of Chicago as an instructor, decided to write a book, an anthology called *Our Chemical Heritage*. We actually got it about ninety percent finished. Then the World War II research came along and we had to drop it. I never got back to it.

BOHNING: Do you have the typescript for that?

BROWN: Somewhere! [laughter]

BOHNING: That would be interesting to see. Of course, given the name of our organization now, Chemical Heritage Foundation, it would be very interesting to see.

BROWN: Well, I'd have to look through all my papers, piled up in various places here and there, and see.

BOHNING: Keep that in mind, sometime, to look for that typescript; that would be interesting to look at. So you were working on this during what years?

BROWN: From 1939 to 1940. We got involved in the war work in 1940, 1941.

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BOHNING: The other aspect of the *Boranes* book is that you tried to show, not just the equations of what occur, but the rationale behind that—the things that don't appear in all those publications—which is also something that I think is important for students to see, and which doesn't see the light of day very often. That *Boranes* book, the Baker Lectures series, is really quite lively because of your interjection of the personal aspects of it. Do you see anyone else really doing that kind of thing, or do you think that you were more of an exception to it, to the personal accounts of what happened?

BROWN: Well, remember, in the book, I called it an experiment in scientific autobiography. I said I was going to be making an effort, and I was giving the Baker Lectures to explain to the students what the background was, what we did, why we did it, what conclusions we came to, and where it led.

I've had an unusual career. It began in 1936; that would be sixty years of research in the boron area. I have, in effect, uncovered a brand new continent of chemistry, and I'd like to show how this was achieved. I just got started; I got interested and made discoveries. Each discovery led to more discoveries, and the program just broadened out.

For example, I didn't intend to get into asymmetric synthesis, but since the Nobel, I've developed the only general way doing asymmetric synthesis—not because I sat in the easy chair and had a bright idea, but we did certain things; we made certain discoveries, and then it was clear what we could achieve if we could manage to overcome certain hurdles.

BOHNING: This might be a point to follow up on that, because I wanted to eventually get into industrial research as opposed to academic research. You've commented in print about your attitudes towards what's happening in industry today, as opposed to what happened in industry a while back—the <u>encouragement</u> of research without a specific purpose. In the twenties and thirties, industry was doing a lot of that, but it has changed considerably right now. Do you have any comments about that general area? Your experiences?

BROWN: I would merely say that, first of all, in the thirties and forties, there were lots of people who were very competent, but who couldn't find academic jobs, people like Max Tishler and Karl Folkers; they took to industry, and they could do things. They had strong enough minds so that when they developed a process, they would push it through against opposition.

I think I heard once that the Harvard Business School had made a study of the origin of most of the major discoveries in the chemical industry, and they have found that in back of each discovery there was an S.O.B. who wouldn't take no for an answer. In any large company, you've got ten committees it's got to go through. Any one of the committees can stop it, and usually does, but if there is somebody there with the drive and desire who would push it through, they suddenly discover they have a success on their hands. Then, everybody stands back and takes credit for it. [laughter]

I'm afraid now there are not enough bright students going into industrial chemistry. All of those students can get jobs in academic work, and the people left over for industry, in general, with few exceptions, don't have the same drive and energy and desire to accomplish things, so what has happened is that industry has become routine and unimaginative. That's my analysis there.

Now, you see, we in the universities still look for the man who can originate things. I'm not sure that he is appreciated in industry, and that is why industry has fallen on such hard times. When George Olah escaped Communist Hungary around 1957, he obtained an industrial job with Dow. He could do original things, fight, and get the right to do more original things. He was invited to academia. Now he has been awarded the Nobel. Had he been in industry, he would surely have gotten some major industrial process started.

BOHNING: Irving Langmuir and Charles Pederson were probably the only two industrial people who won Nobels, and Langmuir probably fits into the mold of the person you were talking about earlier.

Well, let's look at some of the work that I have a few questions about, some of the chemistry that started back with your Ph.D. work with Schlesinger, in reacting diborane with aldehydes and ketones. What was the impetus behind starting? You had this interest. Did you go to Schlesinger and say, "I want to do this?"

BROWN: Well. Should I tell the story of how my wife gave me this book?

BOHNING: Okay.

BROWN: Well, my interest in the hydrogen compounds of boron began with this graduation present that my girlfriend, Sarah Baylen, gave me in 1936 when I graduated. I had gone faster than her, and I'd completed my two years in one year, so she was still a junior, getting ready toward her senior year, when I graduated, and she gave me this book, *The Hydrides of Boron and Silicon*, by Alfred Stock (7). That was one of the Baker Lectures. I read this book and I became interested in the subject.

At that time, the hydrides of boron were very rare substances. They could be made in only two laboratories in the entire world—the laboratory of H. I. Schlesinger at the University of Chicago and the laboratory of Alfred Stock in Karlsruhe, Germany. Alfred Stock had done most of the work in the field. I had merely heard of Schlesinger's work, but when I read this book, I became impressed with the unusual chemistry, and I decided it would be a good field in which to do research.

Now, why did my wife pick out this particular book to give me? It had no market; it was the cheapest chemistry book in the University of Chicago bookstore.

You should recall that at that time diborane was very rare. The chief interest in it was that it provided a severe test for the Lewis theory of electronic structure. Boron has three valence electrons; the three hydrogens have three. The simplest molecule of boron hydride should be BH₃, and no one had been successful in isolating BH₃. The simplest hydrogen compound of boron was B_2H_6 . It was thought that since the empirical formula B_2H_6 is the same as that of the carbon compound ethane, C_2H_6 , they must have similar structures. The trouble is that ethane has seven bonds, requires fourteen electrons to provide a pair for each bond, and there are fourteen electrons—four each from two carbons, eight, plus six from six hydrogens, fourteen, so you can provide a pair for every bond. But you have only twelve electrons in diborane. How can you provide a pair for each bond? Obviously, you can't.

One chemist wrote a formula for diborane, having $2BH_3$ held together by a question mark. Linus Pauling published a structure with both one-electron bonds and two-electron bonds, and he had supposed that they could resonate among all the bonds. So, in effect, each bond would be a 6/7th bond. This turned out to be wrong.

An undergraduate at Oxford, [E. A. Moelwyn-Hughes], and his tutor, R. P. Bell, examined these things and found that all the physical evidence could be better explained, not by an ethane-like structure, but by a bridge structure. Now, I had reached the same conclusion as a result of some research I was doing with Norman Davidson on trimethylaluminum.

I had a classmate in Chicago named Norman Davidson. He had won a Rhodes Scholarship in 1937 or 1938, and had gone to Oxford. When the war broke out, they sent all these Rhodes scholars back to the United States, and he came back to the University of Chicago. He had started to work on aluminum alkyls at Oxford, and he wanted to continue in this area. By that time I had become Schlesinger's research assistant. Schlesinger was too busy to be involved getting into a new field, so he turned him over to me. We began working together. When we examined the molecular weight of gaseous trimethylaluminum, it was a dimer. We examined aluminum bromide; it was a dimer. We took dimethylaluminum chloride; it was a dimer. And so on. So I came to the conclusion that both methyl groups and halogen atoms can bridge, forming dimers. Perhaps hydrogen could also bridge, accounting for the dimeric structure of B_2H_6 .

I went to Schlesinger with this theory, and he said, "This is not our field. We should be very careful. I will correspond with Mullikan and with Pauling, tell them your ideas and see what they think of it." I've never seen the correspondence, but he told me the answer came back and they said the one-electron interpretation of Pauling was valid, even for these compounds.

Well, I couldn't fight that. After all, I was his research assistant. A year or two later, Moelwyn Hughes and R. P. Bell solved the problem. Well, since that time, I decided I will speak out irrespective. So, when the non-classical ion problem came along, I spoke out, even though a great majority of organic chemists have been convinced there were such things. [laughter]

BOHNING: Yes, that's an interesting area, all in itself. [laughter]

Even though you had used diborane to reduce aldehydes to alcohols, still no one ever picked up on it at that point, because of the scarcity of making diborane itself. You had the only place to make diborane, outside of Stock.

BROWN: The point was, even then, we made it only in essentially milligram quantities; Stock, also. His people would work three to four weeks to make about five grams, so how could you use it to reduce aldehydes or ketones?

BOHNING: I understand at one point you also tried to get Warren Johnson interested in a new technique to make B_2H_6 , and he didn't show any interest either.

BROWN: He was working on lithium and calcium hydrides, but he wasn't interested. Well, he was also involved in war work at the time. Where in the world did you run into that?

BROWN: It must be in the Baker lecture. I'll have to go back and reread it. [laughter]

BOHNING: I think that's where it came from. I think it was out of the Baker Lecture.

Then with Schlesinger, in 1939 and 1940, you started synthesizing the aluminum, beryllium and lithium borohydrides. What was the origin of getting into that combination?

BROWN: Oh, this was when my lab mate, Tom Sanderson, who died not too long ago, was working in my laboratory. Since trimethylaluminum was dimeric, and diborane was dimeric, he wanted to put the two of them together to make a new molecule, trimethylaluminum-borane. So he put them together, but they didn't get him the compound he wanted. Instead, he got trimethylborane and a new compound, aluminum borohydride, $Al(BH_{4)3}$. That was a volatile liquid, boiling point about 45 degrees, and it's the most volatile compound of aluminum known. Anton Burg followed up on that and treated trimethylberyllium with diborane, and he obtained beryllium borohydride. It was a volatile solid, with a sublimation point of about 93 degrees.

Then the first thing I did with Schlesinger, when I became his research assistant, was to make lithium borohydride, by taking methyllithium and diborane, making lithium borohydride. But this was a typical salt-like compound, not volatile.

We didn't mention the fact that before we were involved in the war, there was a National Defense Research Committee set up in Washington. They could see that there was a good chance that we might get involved in the war, and we'd better start preparing because we had done no research on military things. We had a tiny army. Our planes couldn't fight anyone in

the world, and our tanks were poor, and so on, so they started to work on various problems. Roosevelt was persuaded that we should try to make the atom bomb, and they wrote to Henry Gilman and to H. I. Schlesinger and said that they were interested in finding new volatile compounds of uranium. They couldn't tell us what they wanted it for, but we knew. [laughter] I'd given a seminar at the University of Chicago on the topic of the original experiments in splitting the uranium atom.

So they said they knew of UF_6 . It was volatile enough and it could be useful, but it was highly corrosive, and they were not certain they were going to be able to use it. Actually, in order to use it, they had to develop new oils, made of fluorinated carbons, but they said it would be nice if you could find a new volatile compound of uranium which was stable at a temperature where it had a vapor pressure of at least 0.1 millimeter.

Well, we started to make the acetylacetonate. It was not volatile enough or stable enough. Ultimately we ended up with a perfluorinated derivative that was volatile enough and stable enough. We thought we had solved the problem. The material had a molecular weight of 1066. When we reported this to Washington, they said they were sorry; there was a little thing they had forgotten to tell us. It was important the material have a low molecular weight, if possible, not higher than 238! [laughter] So, clearly, we had to find some other tack.

As I mentioned earlier, in the course of our academic research we had made volatile compounds of aluminum and beryllium metal borohydride. So we made an extrapolation all the way to the end of the periodic table as we then knew it and said, "Maybe we can make a uranium borohydride which would be volatile enough." I went in the laboratory myself. I put into a sealed glass tube, which I took off the vacuum line, aluminum borohydride and uranium tetrafluoride. When they were allowed to come to room temperature, there was a reaction; a grey mass formed. When I warmed the tube in my hand, green crystals sublimed to the top end, the cool end of the tube. I isolated those crystals and analyzed them. The crystals were uranium borohydride, $U(BH_4)_4$, and the material was volatile.

It created great excitement in Washington, because its molecular weight was even less than that of UF₆. Actually, it is 298 for 238U. They said, "This could be very important. Gather a group together; make it in quantity. We need about one hundred pounds for large-scale testing."

BOHNING: Wow!

BROWN: Up to this time, we'd only make these things in milligram quantities, so we had to start thinking of how to make it. Originally, we took the method that we knew. We had six young men operating six diborane generators. When all went well, using a modified Schlesinger-Burg procedure, they could each make half a gram in an eight-hour day. That meant that our production was three grams a day or a kilogram a year.

Clearly, the war would be over, one way or the other, before we could get a hundred pounds for testing; so they then asked us to go to work, find a better method. Schlesinger said, "Take three or four people, go into a laboratory, put in cots, and work on this day and night. Don't go home to your wives until this problem is solved."

It turned out that the first solution we tried worked like a charm. We took lithium hydride and BF₃—that was the method that I had tried to persuade Warren Johnson to test—and

the mixture reacted rapidly and diborane was rapidly evolved. We could easily make a kilogram a day, just in plain glass apparatus. When we reported this to NDRC, National Defense Research Committee, they said "Sorry; you can't use lithium hydride."

Prior to the war, all the lithium hydride had been made in Germany. We had only limited supplies of it. There was an urgent need for it. Every airplane which crossed the water carried two one-pound canisters of lithium hydride. If the plane was forced down and on to the ocean by motor trouble or enemy action, the pilot and crew could inflate a rubber raft, get on that rubber raft, take one of these pound canisters of lithium hydride, attach it at the handle, and dip it in sea water; hydrogen would be generated to go aloft to a balloon and carry an antenna so that they could signal for help. There was not enough lithium hydride for this important application.

[END OF TAPE, SIDE 3]

BOHNING: Who was making lithium hydride at that time?

BROWN: No one in the United States. [laughter]

BOHNING: So was this just a stockpile reserve that they had?

BROWN: Yes.

So then they said, "Why don't you use sodium hydride?" There was lots of sodium around. The chief use of sodium had been to make tetraethyllead. We had gasoline rationing, so the sodium plants were operating at only fifty percent capacity. They said, "Sodium can easily be converted to the hydride, so why don't you use that?"

At first, it wouldn't work, but then we solved that problem. We found that if we refluxed methyl borate over sodium hydride for several hours, it would add to give us a new compound, sodium trimethoxyborohydride, and that would do everything that lithium hydride would do. So we were now in position to make the material they wanted. By this time we were actively involved in the war. Well, they said, "Sorry, we now have solved the problems of handling UF₆. Now, we no longer have any need for uranium borohydrides."

So we were at the point of disbanding our research group when we got a call from the Army Signal Corps. They said they had a problem generating hydrogen in the field. They heard we had a new compound that might solve their problem. Could they come and discuss it with us? So we invited them to come. Four colonels showed up, with four senior civilian advisors, and told us about their problem.

They were generating hydrogen by a method developed in World War I—the reaction of caustic soda with ferrosilicon. They had large steel cylinders about twice the diameter of the large gas cylinders we now have in our laboratories. The top would unscrew. Into the cylinder they would put a weighed amount of caustic soda, a measured an amount of ferrosilicon, and a controlled amount of water. The top would be screwed on as rapidly as possible. The caustic

soda, as it dissolved in the water, would bring the temperature to about a hundred degrees. At that temperature, the caustic soda began acting on the ferrosilicon; the temperature going up to about two hundred degrees. The reaction produced sodium silicate and hydrogen. The pressure went up to around three thousand pounds—a thousand pounds of steam and two thousand pounds of hydrogen.

So the cylinders had to be strong and very heavy, and the army was facing the problem of having to ship these things to all the battlefields around the world.

BOHNING: What were they using the hydrogen for?

BROWN: Well, they'd use it for inflating balloons carrying antenna loft to send out radio signals. They also sent up balloons to predict the weather. There were a number of problems with this method. One was that after the cylinder cooled, the water would condense out and sodium silicate would deposit. When all the hydrogen had been used up, they had to recharge it to repeat the operation.

The sodium silicate used to coat the walls of the cylinder of the tank with a glassy-like film. To get it out, a soldier had to go and chip it out, bit by bit. It took the average GI a full day to do this. At the end of the day, he was dog-tired. He had strict orders to dig a hole and bury this sodium silicate, but they'd usually be too tired, and they'd throw it behind the nearest bush. For some reason, cattle seemed to love this stuff, and the Army was getting a lot of suits for damaged cattle.

Then the last thing they said was, "Caustic soda is too dangerous a material to have soldiers handle." They were developing caustic burns, having to be hospitalized. "Could we solve their problem for them?" We said we thought we could.

We had developed a new compound, sodium borohydride, which would hydrolyze to give off hydrogen, just like diborane does, on contact with water. The amount of hydrogen that would come would be five times greater than the amount of hydrogen produced per weight of material than the caustic soda process. Moreover, the reaction should take place at room temperature, so that you should be able to use a small light-weight galvanized steel generator, which would generate the gas as needed, not a heavy steel cylinder which must store the gas under high pressure.

They asked us for a demonstration. I went into the laboratory and I set up a roundbottom flask with a magnetic stirrer, put sodium borohydride in it, and put above it a dropping funnel containing water. The whole thing then led through a condenser to a gas meter to measure the hydrogen as it was produced. With all, Professor Schlesinger and the four colonels and the four civilian advisors watching me from a distance, I put my hand into a padded glove. I had a screen in front of the apparatus. Because we had never hydrolyzed sodium borohydride, I didn't know how violent that reaction would be. I cautiously added the water to it. The sodium borohydride dissolved in the water, and the solution stood there looking at me. [laughter] No hydrogen! That's the way we discovered that sodium borohydride is stable in water.

I quickly recovered. I reached for a bottle of hydrochloric acid and added it to the dropping funnel. As soon as it hit the sodium borohydride, hydrogen was generated. They were impressed; they gave us the contract to find a cheap way of making sodium borohydride and to

find a catalyst that would catalyze its hydrolysis by water.

So we continued our research with some of our group; the others went and joined the Manhattan Project, which was looking for people at that time. We took sodium hydride and heated it to two hundred fifty degrees in a glass apparatus with a Glascol heater and a mechanical stirrer, stirring the stuff up. We would add the methyl borate to it, drop by drop, at two hundred fifty degrees. As soon as it hit, it reacted. We ended up with a quantitative mixture of three moles of sodium methoxide and one mole of sodium borohydride. The problem was how to separate these two.

Now, at that time, we had very few solvents, and we didn't know of any solvent that would dissolve sodium borohydride. We tried benzene; neither one dissolved. We tried ether; neither one dissolved. Today, we could use diglyme or certain other solvents. But we tried methanol. At first, both dissolved. Moreover, we got hydrogen off, so we were destroying the sodium borohydride. We tried acetone. At first, everything looked perfect. The sodium borohydride dissolved; the sodium methoxide did not. But when we tried to recover the sodium borohydride from the solution, we couldn't get it. We got back a new compound, sodium tetraisopropoxyborohydride. That's the way we discovered that sodium borohydride is a wonderful new reducing agent for organic compounds, which is one of the large uses for it today.

We went on to discover that liquid ammonia would dissolve sodium borohydride—but it has a boiling point of -33 degrees and is not very convenient to use on a small scale, so we went to a higher homologue, isopropylamine. It would dissolve the sodium borohydride. We could distill it off and get back pure sodium borohydride. That is the basis of the present commercial method of making sodium borohydride.

Then we discovered that putting in three percent of cobalt chloride into the pellets gave us a catalyst. When the pellets hit the water, you get a reduction of the cobalt to a finely-divided active metal, which catalyzes the hydrolysis of the sodium borohydride.

The Signal Corps was delighted with the material. It was subjected to a field test, and they entered into an agreement with the Ethyl Corporation to put up a twenty-five million dollar plant to make it for them. This was just before the Battle of the Bulge, at the end of 1944. Orders came through from Washington that the end of the war was in sight and no new war plants should be built, so this wasn't used either.

BOHNING: What was the attitude towards getting patents? You and Schlesinger had the patent on sodium borohydride (17)?

BROWN: Yes.

BOHNING: How quickly did you apply for that?

BROWN: Actually, we didn't have the sense to get the patent. The Army urged us to patent this development, giving the Army a royalty fee license to make and use sodium borohydride, so we got the patents and gave them their license. Later on, Schlesinger and Finholt developed lithium aluminum hydride, following similar procedures, and they also got patents.

There was a company in Beverly, Massachusetts, named Metal Hydrides. During the war, they made calcium hydride and used it to reduce oxides to the metals. They're the ones who supplied the uranium for the first atomic pile at the University of Chicago. Well, at the end of the war, they became desperate. Their sales then went from several million dollars a year down to about three hundred thousand dollars a year, so they were anxiously looking for new business. They got a license to make lithium aluminum hydride. They used to charge about a dollar a gram initially, which covered their manufacturing cost on a pilot plant scale. They did the same thing with sodium borohydride. Both chemicals began to be used widely, and that put them into a large-scale business.

BOHNING: Somewhere you also commented that Hass, here at Purdue, had done the same thing.

BROWN: Yes, with the nitration of gaseous paraffins. He could make nitromethane, and nitroethane, and nitropropane, and so on, and this development was commercialized by Commercial Solvents, located in Terre Haute, Indiana.

BOHNING: Was it common at that time that when an academic individual had a process of this kind he first proceeded to get a patent and then moved to an industrial application of that process?

BROWN: Well, it was in the process of changing. At the time I left Chicago, which was 1943, the President, Hutchins, had announced a new policy that the Universities would own the patent. When I was invited here to Purdue in 1947, I wrote the President a letter in which I asked him what was the university policy with regard to patents. He said, "Well if you do it on a contract through the Purdue Research Foundation, then the patents would belong to the Purdue Research Foundation, but if you did it with students, it wouldn't be." Therefore, I came here on that basis. It's one of the factors I considered. Whereas the University of Chicago had already changed its policy, Purdue changed it somewhat later, after I got here, about 1950. So Henry Hass's patents, I think, were owned by the Purdue Research Foundation. I was very unhappy, but I don't like to get into fights. I've never sued anybody.

But in 1960 I was invited to go to the University of Wisconsin. I was told that I <u>could</u> assign my patents to the Wisconsin Research Foundation, but I didn't need to—it wasn't a requirement—and that was very tempting. When it was learned here that I was thinking about going to Wisconsin, the president, Frederick R. Hovde, said to me, "Why are you unhappy here?" and I explained. He said, "Well, I was obviously unfair," and within twenty-four hours, he drew up an agreement and went to the chairman of the Board of Trustees to give me back my patents [laughter] and future rights. So since that time, I've been operating here under a special agreement which gives me the right to any patents that I develop.

BOHNING: The sodium borohydride was your first patent?

BROWN: Yes.

BOHNING: You said that happened because the Signal Corps of the Army said that you should do that. Did that open you up into saying, "In the future I should look at applying for patents"?

BROWN: Of course. We also developed a patent on using sodium borohydrides as a reducing agent, and we licensed Metal Hydrides (18). At first they wanted an exclusive license for only five years, and we gave it to them. Then when it became open again, Callory wanted to get into the field, and they came to us and said that they would like to get a license. They were willing to offer rather large sums of money and annual guarantees; we gave them a license exclusive for five years, subject only to Metal Hydrides' non-exclusive license, provided they paid certain minimum payments. They were then involved in the high-energy fuel program, and they really didn't have the right people to put to work on this thing; they never got it to work in their laboratories, although it was being done at Metal Hydrides. So after five years, they finally gave up and gave it back to us, and then we gave an exclusive license to Metal Hydrides, for the life of the patents.

BOHNING: When you came here to Purdue, you continued some of the work you had started at Wayne, still on steric strains. Then you got into aromatic substitution, and it wasn't until the early fifties—1951, I'm going to say—that you started doing the reduction studies comparing sodium borohydride and lithium aluminum hydride. No one had been using sodium borohydride as a reducing agent. Everybody went to lithium aluminum hydride. Even though it was too strong for certain things, they were still using it for all the reductions.

BROWN: It happened that lithium aluminum hydride was discovered <u>after</u> sodium borohydride, using similar methods. It was discovered <u>after</u> the war and, consequently, was not under secrecy order, whereas the sodium borohydride was under secrecy order, and it wasn't until about 1948 that that was lifted and we could begin publishing the work. Sodium borohydride is a very gentle reducing agent. It reduces aldehydes, ketones, and acid chlorides, not any of the other major groups. Lithium aluminum hydride reduces practically everything.

Many people were using lithium aluminum hydride to reduce one group in the presence of another group; but that was a difficult job, because it could reduce both groups. So I decided when I came here that it would be a good idea if we could just make a series of reducing agents with variable reducing capability, so each person could then select that reagent which would reduce the group that he wanted to reduce and avoid the group he didn't want to reduce. So we set out on that program. Then we discovered easy ways of doing that. All we had to do was replace the metal in the borohydride. If we went to lithium borohydride, it would reduce esters, in addition to the other three groups. If we went to the corresponding magnesium compound, it was even stronger. Aluminum borohydride was almost as good as lithium aluminum hydride. If we added aluminum chloride to sodium borohydride, the system was almost as powerful as lithium aluminum hydride. The introduction of alkoxy groups into lithium aluminum hydride decreased the reducing power. Indeed, lithium tri-t-butoxyaluminum hydride was almost like sodium borohydride. So we were able to go from the two extremes, providing a series of reagents which could reduce group A in the presence of B, and others that could reduce group B in the presence of A.

We also discovered that diborane is an <u>acidic</u> reducing agent. It had different characteristics than the sodium borohydride, which was a <u>basic</u> reducing agent. For example, if we had a dicarboxylic acid, one end of which is hindered, and converted it to a half ester, diborane would reduce the free carboxyl group without touching the ester grouping. Lithium borohydride would reduce the ethyl grouping without touching the carboxyl group. So it became commonplace to reduce group A in the presence of B, and group B in the presence of A.

Incidentally, on March 15th, 1994, the British Society of the Chemical Industries had a symposium held by the Fine Chemicals Division of the Society of the Chemical Industries, on "Reductions in Organic Chemistry." I was one of the speakers at this symposium, and I spoke on "Over Half a Century of Research of Hydride Reductions."

BOHNING: Do you have a copy of that talk?

BROWN: I've got all the slides, but we published one in 1979, called, "Forty Years of Hydride Reductions" (19).

BOHNING: I have that one.

BROWN: That's essentially the same thing, brought up to date with a few more examples, that's all.

BOHNING: Now it was in the course of these studies on selective reduction that you discovered hydroboration.

BROWN: Yes. We were studying the reducing action of sodium borohydride in the presence of added aluminum chloride. The idea was that perhaps aluminum chloride would react with the sodium borohydride to give sodium chloride and aluminum borohydride. Anyhow, we got no precipitate of sodium chloride, so probably we were getting some kind of equilibrium mixture. But this was a rather powerful reducing agent, and it will reduce esters cleanly. Ethyl acetate would take up two hydrides per mole, with the corresponding reduction to the alcohol. Ethyl stearate did the same thing. One of the compounds we tried was ethyl oleate. It took up 2.37.

I remember, Dr. B. C. Subba Rao was the one doing the research. He was a very productive coworker. He came to me with masses of data and tables. I looked them over; everything looked fine. I should explain that we had a standard list of fifty-six compounds we used to try each new reducing agent against. Each of these had been carefully purified, kept separate from other chemicals and used only for this purpose. Aldehydes and betones took up one hydride per mole. Esters took up two hydrides per mole. Both corresponded to reduction to the alcohols. But ethyl oleate showed an uptake of 2.37 hydrides per mole. I asked him, "Why does this show a discrepancy?" He said, "You want to remember that ethyl oleate was not one of our standard compounds. When I told you that the reagent was reducing ethyl acetate and ethyl sterate, you said, 'See what happens with an unsaturated ester"—which was readily available in the stockroom;—"take it and try it." He did. He went to the stockroom; he found ethyl oleate there. He said, "It was slightly discolored, possibly containing some peroxides, but I used it directly, and possibly, the 0.37 represents additional hydrides used by the peroxides present." He said, "What do we want to bother with this for? It's not one of our fifty-six standard compounds. Why don't we publish the rest of the data and go on to the next problem?"

The research director is in a wonderful position to insist upon high standards; he doesn't have to do the work himself. So I thought for a few seconds. Then I said, "No, I think you should go back. Redistill the ethyl oleate, get a pure sample, and repeat the experiment." He did; it came to 2.37. We let it go for three hours. These experiments were all done at room temperature: 1 hour, 2.37; 3 hours, 2.9; 6 hours, 3.0; 12 hours, 3.0; 24 hours, 3.0. Clearly, the carbon-carbon double bond was using up one equivalent of a boron-hydrogen bond, and the only thing we could postulate was that it was adding to the double bond, giving us an organoborane, and that is how we discovered hydroboration.

[END OF TAPE, SIDE 4]

BOHNING: You continued to pursue this for ten years or so. In another quote, you said, "Many individuals expressed skepticism to me as to the wisdom of devoting so much research effort to this reaction. After all, hydroboration produced organoboranes. They took the position that the lack of published material in this area meant that there was little of value there" (8).

BROWN: First of all, let me say, when we discovered this hydroboration reaction, we were excited about it. We sent in a Communication to the *Journal of The American Chemical Society*. It was not that many people said this to me, but the referees said, "Don't publish this." You see, organoboranes had been discovered by E. C. Franklin in 1862, published in the *Journal of the Chemical Society*. They said, "Nobody has found anything useful to do with it in organic chemistry since that time. Therefore, what hydroboration does is largely make organoboranes readily available. What's the point of publishing a Communication when there is no use for organoboranes in organic chemistry?"

Well, I pointed out to the editor that it's true that nobody had done anything significant with organoboranes since 1862. Franklin had found that they were oxidized by air; they were stable in water; they formed addition compounds with amines; but nothing else. I pointed out to him that it was not the fact that people had tried to find some uses for organoboranes and had failed; nobody had ever tried, so how could we say that they weren't going to be useful until we tried. Now that we had an easy way of making them, we proposed to try. He was persuaded by my arguments and the communication was published (20).

So we spent ten years studying hydroboration, until we felt we understood it thoroughly. Then we turned our attention to exploring the chemistry of organoboranes, and we discovered that we had uncovered a new gold mine. They would do <u>everything</u> that we wanted them to do. We had a chart showing twenty-four major reactions. We could transpose the R-B bond to form an alcohol, an aldehyde, a carboxylic acid, a primary or secondary amine, ketone, secondary alcohol, tertiary alcohol, terminal acetylene, internal acetylene, cis-olefins, trans-olefins, et cetera, et cetera.

That, of course, was the work that led to the Nobel. But it had one thing that we hadn't anticipated. Substitution in the borane compound generally occurs with complete retention of configuration. Substitution in carbon compounds goes with inversion or racemization. With borane compounds, generally the R₃B, a Lewis acid, reacts with the anion reagent, forming an internal complex, and then substitution involves an internal rearrangement, so that the group R

transfers with its pair of electrons from boron to oxygen or nitrogen, or metals, and there is no rearrangement or inversion.

This in effect told me that if I could only learn an easy way of putting asymmetric R^{*} groups, non-reactive groups on boron, I would have a general way to do asymmetric synthesis. That's the program to which I've been devoted ever since the Nobel. We have the only general asymmetric synthesis that's available today.

BOHNING: During that ten-year period when you were simply exploring hydroboration and production of organoboranes, didn't anyone think of looking at the chemistry of the organoboranes that you were making? It seems like a long period of time to put all that out there for somebody else to start doing something with.

BROWN: I'm trying to think. Of this chart that I have, as far as I know, we are the only ones that contributed to that chart.

BOHNING: Okay.

BROWN: Later on, some things happened. Don Matteson at Washington State began doing some things, and DuPont did some work with carbon monoxide. They found that R₄B reacts to give transfer groups from boron to carbon so you can make R₃BCO as the product. The Ethyl Corporation showed they could make tetraethyllead from lead oxide plus triethylboron.

But, otherwise, as reagents in organic chemistry, we were the only ones who really pursued this. Maybe we moved so fast that we discouraged other people from getting in.

BOHNING: Well, the papers that you put out were certainly prolific and very thorough. Were you, again, obtaining patents along the way, as this developed?

BROWN: We got some, yes, and we entered into an agreement. The Ethyl Corporation was interested. One time, we found that if we hydroborate a long-chain internal olefin, the boron goes to the middle of the chain. If we heat the compound to about two hundred degrees, the boron migrates to the end of the chain, so we had a way of making either primary alcohols from straight-chain paraffins or olefins, or we could make alpha-olephines from it. Because if we heat it with another olefin, the boron will transfer. So we had some promising transformations, and we got certain patents on these developments.

The Ethyl Corporation entered an agreement with the university to pursue this, and they explored this, but they had a competing method going through aluminum and decided to stick with the aluminum route. Shell also came along and got a license there. They also were interested there, but it was never really commercialized.

BOHNING: I also understand that you tried to interest large chemical companies in making
boranes available and that you weren't very successful until you got to Al Bader.

BROWN: Well, I tried a number of companies. I tried the Ethyl Corporation. I tried Union Carbide, Eastman Kodak. Often these companies would spend twenty-five or fifty thousand dollars making a market survey and make the astounding discovery that no one was doing work with hydroboration commercially, and therefore, there was no possibility of getting it to commercial production. Finally, I discussed this with Alfred Bader. He was a Ph.D. in organic chemistry and had the imagination which many industrial managers no longer seem to have. He decided that he would take a chance and do it, and the thing has grown from there. We now have a very big plant and millions of dollars of sales a year.

BOHNING: What year was that when Al Bader decided there was something in this?

BROWN: In 1972. They started out with four people. First-year sales were about ten thousand dollars. Now, as I say, they've got a large group and a big plant in Sheboygen Falls, Wisconson.

BOHNING: Is that the picture you showed me earlier?

BROWN: Well, that was the original plant; it's much bigger now.

BOHNING: This may be the time to get to some of the questions on this sheet, but also you've made some comments, which we touched briefly on earlier, about industry as compared to academic research. I'm quoting from the *C&E News* article when you won the Priestley Medal. "Once, research directors were primarily like chemists, and they had a lot of influence." This is the point you were making earlier. "Today, industries have gotten out of the hands of scientists and into the hands of accountants" (16). Do you want to comment any more on that? [laughter]

BROWN: Well, I think that's clear. I'll also say this. It's my experience that the narrower you define your objective, the less chance you have of achieving it and doing productive research. If you map out a field and explore it systematically, you're bound to make major discoveries. I've made one after another by following this philosophy. How do you sell an accountant the idea that you're going to explore a field? [laughter]

BOHNING: I just talked to Vladimir Haensel at UMass two weeks ago (21). He had worked with Ipatieff at UOP.

BROWN: I know him, yes.

BOHNING: He said Ipatieff worked on instinct, on gut feeling, and I said, "But you can't sell a company today that your <u>instinct</u> is right, that this is going to work."

BROWN: No, but it's a different philosophy. Now, I think I discussed it somewhere; I don't know if you've seen it. Do you have that paper that was published in the book by Atta-ur-Rahman?

BOHNING: I think I do, yes (22).

BROWN: I'll find the paragraph for you. Yes. "Research over a short time is a relatively slow, often agonizing process. Day by day, week by week, it is often difficult to know if there is any real progress. However, the present review of a half century of borane research reveals the cumulative nature of research: over the long run, amazing progress is possible."

Then, research strategies: "There are many different ways to do productive research. Some people enjoy seeking or tackling problems of major importance, with appalling complexity. I've never had the courage to tackle such Mount Everests. Instead, I have found it productive to adopt an alternate research approach, one that I believe is satisfying and productive, though without the serious gambles involved in the Mount Everest approach. I select an area that is relatively unexplored, and then investigate it systematically until I feel I understand the chemistry completely. Invariably, certain observations and phenomena are noted that apparently fail to fit the general pattern behaviors that our investigation has indicated. Investigation of these anomalies often opened up major new areas. This approach is nicely illustrated by the present review."

BOHNING: It states it very well.

BROWN: That has been my research philosophy.

BOHNING: One of the things that we're looking at, in this project, is exactly that. The differences in approaches that people use that are successful. I have a question here that's sort of general, but let me ask you at this point. The concept of scientific innovation—how would you define scientific innovation or chemical innovation, from your experience?

BROWN: I've never thought about it. As a way of working, I've explained my philosophy. I believe, in industry, they try to define the objective very narrowly, and they rarely get anywhere. Let's take an example. Suppose after Columbus discovers America, he goes to a group of businessmen back in Spain. He says, "I've discovered this brand new continent. Surely it's good for something." "Well," they say, "We can't figure that out. We can't calculate that. Define an objective." He says, "Well, in America there doubtless are rich diamond mines. If I find the diamond mines and produce so many diamonds per year, you can calculate what the market is worth, and you would have a huge return on your investment."

So they give him money to go out and explore the continent for diamond mines. Well, he can explore all over America and probably never find any diamond mines, but if he'd gone out there just to explore it, he would have found coal, rich farm land, lakes, gold deposits, et cetera. [laughter] That's the difference in the two approaches.

BOHNING: After the Priestley, you commented about how things were declining in American industry (16). What do you think is important for the future of R&D in an industrial setting?

BROWN: Well, there is a real worry, in that the brighter students are no longer being encouraged to go into chemistry. In 1954, I was invited to the University of Mexico to spend the summer lecturing there. It was a beautiful setting; it was a new university in the lava fields, south of the city. The students were enthusiastic, but they were not up to the quality of our students. I then inquired and found that in Mexico, the brightest students go out to become lawyers or doctors, and only the second-raters go on for science. I said how fortunate we were at that time; in the United States we were getting a certain percentage of the bright students who were going on to chemistry and becoming the leaders in the field.

But now, I'm afraid, we are not getting them. Lots of students have come from abroad, and some of them are taking their place. But, still, that's an unfortunate aspect, because the new approach to running large companies is that you've got to define exactly what the objective is. It makes it very difficult to achieve that objective, and you stop all creativity among the staff, so nobody wants to work in industry; no one <u>competent</u> wants to work in industry.

BOHNING: You consulted with companies. In fact, I guess, you were on a consulting trip to Exxon when you heard you had won the Nobel.

BROWN: Yes.

BOHNING: What have your experiences been in that? Do the companies listen to what you say to them?

BROWN: Well, the people you deal with do. You don't advise the research directors. [laughter] But certainly the people in the laboratories do, and they would all come and we would discuss their problems. We'd find solutions for them and so on, but initially, the objective was broad, you see. I think it was the first time that I visited Amoco, Standard Oil of Indiana; in 1944, I think it was, they invited me to come in as a consultant. They told me that they had a process of removing sulfur compounds from gasoline, called the sweetening process. It gave them dimethyl disulfide, methylethyl disulfide and so on, and these they left in the gasoline to be burned. They said in the future they were probably going to be restricted from doing this, so they would have a large amount of these sulfur compounds. Could I suggest something that they could do with them.

So I suggested that they consider oxidizing them to sulfonic acids. I said that the methanesulfonic acid and ethanesulfonic acid would be strong mineral acids and have quite different properties from sulfuric, which has the disadvantage that it is easily reduced, oxidizing the reactants. These cheap, stable acids should find a market. The study took only about two months. They took the disulfides, and they found that if they heated them up and put in a little bit of nitric oxide as a catalyst, air rapidly oxidized them to the sulfonic acids.

They went through their engineering group and asked them to figure out how much it

would cost to make a million pounds a year, in order to test the market. They came back with an estimate that it would cost several million dollars, and the price of the sulfonic acids would have to be about ten to twenty dollars per pound. We had been talking about making a cheap chemical. They then went to Edwall Laboratories. Edwall could provide the acids for five to ten cents a pound, with no significant investment necessary.

Well, this was a big difference, so we looked at it. Their engineering department had calculated that to get the nitric acid for a catalyst, they were going to have to build a railroad siding so that a rail tank car could reach their oxidizing unit to provide the nitric acid. This was going to be pumped into the reactor and so on. The Edwall chemical engineers were going to have a truck come there with carboys of nitric acids. You see, that's the difference between the two. [laughter] So you have got to apply some common sense to these things too.

Well, they got it from Edwall and went to market it, but after a while, it was too small for them to worry about, so they sold it to somebody else.

BOHNING: I think we've pretty well covered the agenda that I had sent you. The last question is one which I want to expand a little bit. You've won a number of medals and awards in your lifetime. What effect did winning the Perkin Medal have?

BROWN: [laughter] Let me say this. Watson wrote a book on a double helix, and I think he did science a great disservice. He seemed to give the idea that you do research to win medals. He was single-mindedly pursuing the Nobel Medal, and that's why he was doing his research. I've never believed in this. I believe that the primary reason to do research is to influence the course of your science. If the medals come along, fine, you enjoy them, but you don't do research to win any medals.

People have asked me, "What did the Nobel do for me?" Nothing. I still continue working. My wife has the idea that men last longer if they keep on working. So, here I am, sixteen years past my retirement, and I'm still active in the laboratory. [laughter] It is nice to know that she still wants me around.

BOHNING: Marvelous. How many postdocs do you have working with you?

BROWN: About twelve.

BOHNING: Twelve. That's a big group yet.

A large number of your publications have come since you retired.

BROWN: About half. [laughter] And most of my major awards have come since I retired. [laughter] The Nobel came a year after, then the Priestley, the Perkin, and then the Gold Medal of the American Institute of Chemists. Then I got a medal from the National Academy of Sciences, a special award supported by Occidental Petroleum, I guess, for pioneering work in the chemical sciences. Then I got, from Japan, a Decoration: The Order of the Rising Sun, Gold and Silver Star. That's their highest decoration for foreign scientists.

BOHNING: Hoyt Hottel was a chemical engineer at MIT. He was the first person I ever interviewed, almost ten years ago now, and he was in a similar position (23). At that time, <u>one-third</u> of his papers had been published <u>after</u> he retired, and he's still active at ninety-five.

BROWN: Heavens! [laughter] I would like to see the year 2000 come around, but that's about all. [laughter]

BOHNING: Well, on that note, is there anything else you would like to add?

BROWN: I didn't explain what we've done on the asymmetric synthesis. I explained that when we did the work on studying the chemical properties of organoboranes, we discovered that they are very versatile compounds, and we also discovered that they undergo substitution with retention. This suggested that if we could only learn an easy way to put R^{*} groups, stereoisomeric groups, on the boron, we could transmit them unchanged in all of these different reactions.

As it happened, we had a hint. George Zweifel here, in 1960, had examined the hydroboration of alpha-pinene, purely as part of our systematic way of going at things. We selected alpha-pinene as an olefin which undergoes easy rearrangement. We wanted to see whether hydroboration would cause rearrangement. We discovered it did not rearrange, but it was unusual in that only two of the three hydrogens on the boron were utilized. Two moles of alpha-pinene reacted; the third mole did not react. We got a new compound, diisopinocampheylborane.

I said to him, "This is an asymmetric hydroborating agent. The first one we've had. It won't hydroborate alpha-pinene, but it might hydroborate less-hindered olefins such as cis-2butene. Why don't you try it and see what you get?" We were thinking we might get the usual ten or twenty percent ee, so he went and did it. The next day he came running into my office, saying the compound had undergone hydroboration. When he oxidized it, he isolated 2-butanol. He said the 2-butanol had an ee of 87 percent. The alpha-pinene used had had a purity of only 93 percent ee. This is the first non-enzymatic asymmetric synthesis in high ee in the world! In 1960.

Since that time, we've improved it. We've learned how to make diisopinocampheylborane, Ipc₂BH, from alpha-pinene and BH₃, in one hundred percent ee. When we use it, the alpha-pinene recycles in 100 percent ee. Some people are now developing some catalytic methods, but when I do the calculations, as far as I can see, our method is actually cheaper than some of the catalytic methods for doing asymmetric reduction, because we recover the alphapinene without losing any activity. You may have to add a small quantity of makeup alphapinene, but this should be a negligible cost. We have now figured out that by using just the chemistry we know, we have done around thirty-four different olefins and converted them to the boron derivatives in one hundred percent ee. If we use the stereoisomer, l-alpha-pinene, it's twice that, sixty-eight.

Don S. Matteson of Washington State University has come up with a new method of

making these active R-groups through homologation, and this will double the number. Then we can take any $R^*-B=$ and homologate it with CH_2 , and that doubles the number. Put in a second CH_2 , and it doubles the number again. Put in another one, again. So we come out with five hundred and twenty-eight different systems you could make. We now have twenty-four major reactions. If you multiply this five hundred and twenty eight by twenty-four, you come out with thirteen thousand.

[END OF TAPE, SIDE 5]

BROWN: Now if you take each of these reactions, it will produce many different compounds. If you make a ketone, R COR, R could be methyl, ethyl, isopropyl, tertiary butyl, phenyl, cyclopentyl, cyclohexyl, and so on. Let's make a conservative estimate of ten, so we're now in a position to make one hundred thirty thousand pure enantiomers—one compound only, one hundred percent ee—by this chemistry. I don't think the chemical world yet appreciates what this can do.

BOHNING: That's amazing. Incredible! There's no other technique that can come close to anything like that.

BROWN: And that was all done since the Nobel. [laughter]

BOHNING: You were talking about students earlier. What about the students who come to Purdue? Although you're only dealing with postdocs now, I'm sure you must have a feel for the students Purdue is attracting as graduate students, and then also the postdocs who you see coming in.

BROWN: Well, I won't talk about Purdue, because I have no contact with them. Since my retirement I have worked only with post-doctorates. But I'm just saying, country-wise, it's obvious! We are not getting the same quality students we were. People find there are other fields where they could make more money—law, medicine, advertising, business. So they're getting the bright people, and we're going to suffer in science. Right now, bright students from China and India are coming and staying, and they will solve some of the problem.

But I told you of my experience in Mexico. At that time I thought how fortunate we were, we were getting top-notch students then, but the way industry has treated them has made industrial research even less attractive, and I'm afraid that while there are still lots of students who would like to get into academic work, still I don't see what the future is if industry can no longer afford to take the run-of-the-mill people. They've got to have outstanding people if they want to achieve something in research. And we're not producing the number we need.

I'm normally very optimistic, but I think industry has worked its way into a very sad state.

BOHNING: What about your postdocs? How do you see your postdocs?

BROWN: I get students from all over the world. Many of them are bright, many of them are hungry. To them, it's a step up. If they go back to their country, they don't achieve a nice, easy life.

BOHNING: Well, I think on that note then, I'll bring this to a close. Thank you very much for spending the morning with me. I appreciate your time.

BROWN: I've enjoyed the experience of reviewing with you my sixty years of discovering and exploring the New Borane Continent.

[END OF TAPE, SIDE 6]

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