THE BECKMAN CENTER FOR THE HISTORY OF CHEMISTRY

J. FRANKLIN HYDE

Transcript of an Interview Conducted by

James J. Bohning

at

Marco Island, Florida

on

30 April 1986

copy for your records

THE BECKMAN CENTER FOR THE HISTORY OF CHEMISTRY

Oral History Program

RELEASE FORM

This document contains my understanding and agreement with the Center for History of Chemistry with respect to my participation in a tape-recorded interview conducted by

Dr. James J. Bohning on <u>30 April 1986</u>. I have read the transcript supplied by the Center and returned it with my corrections and emendations.

- 1. The tapes and corrected transcript (collectively called the "Work") will be maintained by the Center and made available in accordance with general policies for research and other scholarly purposes.
- 2. I hereby grant, assign, and transfer to the Center all right, title, and interest in the Work, including the literary rights and the copyright, except that I shall retain the right to copy, use and publish the Work in part or in full until my death.
- 3. The manuscript may be read and the tape(s) heard by scholars approved by the Center subject to the restrictions listed below. The scholar pledges not to quote from, cite, or reproduce by any means this material except with the written permission of the Center.
- 4. I wish to place the following conditions that I have checked below upon the use of this interview. I understand that the Center will enforce my wishes until the time of my death, when any restrictions will be removed.
 - a. // No restrictions for access.
 - b. My permission required to quote, cite, or reproduce.
 - c. ____ My permission required for access to the entire document and all tapes.

This constitutes our entire and complete understanding.

(Signature) (trange (Date)

(Revised 24 February 1988)

J. FRANKLIN HYDE

Born in Solvay, New York on 11 March

Education

| 1923 | А.В., | chemistry, | Syracuse | University | | |
|------|-------|-------------|-----------|------------|----|----------|
| 1925 | M.A., | chemistry, | Syracuse | University | | |
| 1928 | Ph.D. | , organic c | hemistry, | University | of | Illinois |

Professional Experience

| TYRE TYRE TOBE ACCOUNT TETTOW, MALANTA CHIVELDIC | 1928-1930 | Post-doctoral | Fellow, | Harvard | Universi | _ty |
|--|-----------|---------------|---------|---------|----------|-----|
|--|-----------|---------------|---------|---------|----------|-----|

| | Corning Glass Works, Corning, New Yo | ork |
|--------------------|--------------------------------------|-----|
| 1930 - 1938 | Research Chemist | |
| 1938 - 1951 | Manager, Organic Laboratory | |

1951-1975 Dow Corning Corporation, Midland, Michigan

<u>Honors</u>

| 1963 | Distinguished Achievement Award, Dow Corning |
|------|--|
| | Corporation |
| 1963 | Hon. D.Sc., Syracuse University |
| 1963 | Outstanding Inventor Citation, Michigan Patent Law Association |
| 1971 | Whitehead Memorial Lecturer, Engineering Section, National Research Council |
| 1971 | Perkin Medal, Society of Chemical Industry |
| 1974 | Midgley Award, Detroit Section, American Chemical Society |
| 1975 | Hon. D.Sc., Michigan State University |
| 1979 | Elected to Plastics Hall of Fame, Society of the Plastics Industry |
| 1980 | Silicone Pioneer Award, Dow Corning Corporation |
| 1982 | Five of Genius Award, Saginaw Valley Patent Law Association |

ABSTRACT

In this interview Franklin Hyde briefly tells of his childhood and his schooling in Solvay, New York. At Syracuse University, Hyde majored in chemistry and continued on to a master's degree. It was during this period, under the influence of Reginald Boehner that Hyde became an organic chemist and he continued with that speciality with Roger Adams at Illinois and then with Conant at Harvard. He recalls both his colleagues and the faculty at Urbana and at Cambridge. Despite an offer to join Carothers at Du Pont, Hyde chose to accept the challenge of a position with Corning Glass works, where he was the lone organic chemist. At Corning, Hyde started his studies of organosilicon compounds and where he entered the growing field of polymer chemistry. During the interview Franklin Hyde summarizes several of his research endeavors that contributed to the present-day importance of silicones. Included in this section of the interview are instances of the critical role of newly introduced materials to the scientific contribution to World War II. Hyde also describes the chronology of the competition between Corning and General Electric that eventually led to a major patent interference suit. The interview ends with a survey of Hyde's later work with Dow Corning and his reflections on laboratory research and scientific management.

INTERVIEWER

James J. Bohning holds the B.S., M.S., and Ph.D. degrees in chemistry, and has been a member of the chemistry faculty at Wilkes College since 1959. He was chair of the Chemistry Department for sixteen years, and was appointed chair of the Department of Earth and Environmental Sciences in 1988. He has been associated with the development and management of the oral history program at the Beckman Center since 1985, and was elected Chair of the Division of the History of Chemistry of the American Chemical Society for 1987.

TABLE OF CONTENTS

1 Childhood

Family background. Summer work with horsedrawn transportation. Schooling, development of liking for chemistry.

3 University Studies at Syracuse and Illinois Concentration on math and chemistry at Syracuse, faculty and colleagues. Organic chemistry and Masters degree. Graduate studies at Illinois and recollections of faculty and fellow students. Experiences as teaching assistant. Research with Roger Adams, recollections of Marvel and Carothers. Marriage.

- 11 Postdoctoral Study at Harvard Research on chlorophyll. Reminiscences of Conant and other colleagues at Harvard. Effects of the Depression. Circumstances of acceptance of position as an organic chemist at Corning Glass Works.
- 17 Research at Corning Initial research projects at Corning. Polymers and organosilicon compounds. Dispute with Kipping. Competition in silicone research with General Electric Company. Wartime activities and expansion of the research group.
- 37 Transfer to Midland Resumption of research at Dow Corning, Midland. Continuous polymerization system. Study of equilibrium hydrolysis and bond rearrangement in siloxanes. Research and management.
- 48 Notes
- 51 Index

INTERVIEWEE: J. Franklin Hyde

INTERVIEWER: James J. Bohning

LOCATION: Marco Island, Florida

DATE: 30 April 1986

BOHNING: Dr. Hyde, you were born on 11 March 1903 in Solvay, NY. Could you tell me something about your parents?

HYDE: My father's name was Burton Deforest Hyde and was in the sand and gravel business. My mother was born Amelia Bennett. Solvay is a small village, a suburb of Syracuse, and it's the location of the Solvay Process Company. Although a small town, it was also home for a number of other industries, Frazier and Jones, Malleable Iron Castings, Pass and Seymour Insulator Works, Hammond Steel Company, and the Iroquois Pottery Company. My Dad had, along with the sand and gravel business, the equipment used to unload clay from the canal boats along the nearby Erie Canal. We used to unload coal cars, for instance, for the Hammond Steel Company. As soon as I was big enough to handle a shovel or drive a team of horses, I got plenty of outdoor exercise during the summers.

I was named after my two grandfathers, but I never knew them. One was James Bennett. I discovered, only recently, his discharge papers from the army. He was a sergeant in Sheridan's cavalry during the Civil War. I guess he was taken prisoner and spent some time in Libby Prison, which is I think, a suburb of Richmond. I don't know too much about my grandparents. My grandmother's name on my mother's side was Childs, Lucy Childs. I knew my grandmother on my father's side very well because she lived with us for many years. Her name was Martin, Lydia Ann Martin. She was born in Sandy Creek, Oswego County, between Syracuse and Watertown. I know some of the family history.

BOHNING: Did you have any brothers and sisters?

HYDE: Yes, I have an older brother, Solon W. Hyde. Back in those days, they used to name their kids after famous people. My brother was named after my father's uncle, who was Solon Webster Martin. My grandmother had several brothers, one of them was Henry Clay Martin, and another George Washington Martin. I had a sister, Helen Hyde Darby, who passed away many years ago.

BOHNING: You lived in Solvay, and went to school there?

1

HYDE: I went to the Solvay public schools. I managed to get a high school regents' diploma. Summers, of course, I got a pretty hardy outdoor life. I remember drawing brick sand with a team of horses and a dump wagon when they built one of the Arm and Hammer Baking Soda plants. They were part of the Solvay complex. And later, after the war, I did the same for one of the first atmospheric nitrogen, ammonia synthesis plants (the Haber process). It was confiscated after the war [WWI]. It was one of the first such plants in this country. They needed ammonia for the Solvay process.

BOHNING: Do you remember any teachers that had any influence on you in the public school system?

HYDE: When you look back, you realize that you had good teachers, and maybe some that weren't so good. For instance, I had a good German teacher. We learned a few German songs in the fifth grade. I took all my German in high school. Claude Duval was the principal of the high school. He taught geometry, and I was pretty good at it. When I look back I think it was partly because of that teacher who had the ability to interest and motivate youngsters. He was that kind of a teacher.

BOHNING: Did you have any chemistry courses in that high school?

HYDE: The chemistry laboratory was one bench with a few bottles sitting on it. The lady, I can't remember her name now, was a good teacher. She taught more than just chemistry, so I learned a little bit of chemistry in high school. There was no laboratory connected with it. She did a few experiments which were probably sufficient to get me interested a little bit. I don't know if living in that Solvay Process atmosphere had anything to do with my interest in chemistry. Maybe unconsciously. Of course, the Solvay population was only about three or four thousand people. Probably many of them worked for the Solvay Process Company.

BOHNING: You said you had the regents' scholarship. What was that?

HYDE: The regents' diploma, was given by a Commission of the Education system in New York State. The regents, put out an examination, that they called a regents' examination, in almost every course that was provided in high school. If you passed the state regents' examinations, you would get a state regents' diploma. You could graduate from high school, of course, without a regent's diploma. If you passed all these state regents' exams, you didn't need to take a college entrance examination. Or at that time you didn't. Solvay was not too far from Syracuse University. My folks didn't think they could afford to send me there. But I worked so hard that summer that they could hardly say no. I got a little help from a small scholarship, but I lived at home. That meant a street car ride, twice a day for maybe an hour or so. I don't know what motivated me. I guess it's just that I was interested in what made things tick, and I wanted to go to college.

BOHNING: What was the year when you started at Syracuse?

HYDE: I graduated from high school in June 1919. So I started as a freshman in the fall of 1919 at Syracuse.

BOHNING: Did you select any major when you first entered?

HYDE: No, I took a general liberal arts course. At that time, if you had a foreign language at high school you were expected to start a new foreign language, which I did. I started taking French. One of my weak subjects was Latin. The only course that I ever flunked in high school. At Syracuse, one thing I learned in the Latin class was the Greek alphabet. You really need that in science or in math, because many of those symbols are used. But I don't think I did very well in Latin. I think they just passed me to get rid of me.

BOHNING: When did you make your selection of a major, then?

HYDE: Well, I think that I went naturally towards chemistry. I took math and I had an interest in math. One of the bad things about it was that I missed advanced algebra in high school, where you learn something about infinite series. And that dogged me the rest of my life. It bothered me, and I missed it. But I did like math. I don't think I ever had to make the decision to go for chemistry, it was just my natural bent, I think.

Freshman chemistry was a very interesting subject for me and we had a professor, Ross Baker, who I think was one of the great teachers of that time. He was a very active person, and always had some kind of demonstration that would stir you up and get you interested. He was a very good teacher. That certainly led me towards chemistry. His hobby was glass-blowing, and he taught me that. He gave a small course in glass-blowing. If there were some little odd jobs around, like cleaning some of the demonstration glassware or something, I got a chance to do it. I made the chemistry building my home, or headquarters, when I was up at school. Professor McCroskey taught a course, a second year course, in inorganic preparations, that interested me. This was my first experience with silicon chemistry. I think the directions were probably those of Berzelius. I was impressed by the amount of energy that was involved in the reaction. What you did was mix sand and aluminum powder, and set it off with a piece of magnesium ribbon, and you got a little silicon regulus in the bottom of the crucible, which was then converted to silicon tetrachloride with chlorine. I don't know that that had anything particularly to do with things later.

Then I got into organic chemistry. I think that Dr. Reginald Boehner, was the greatest teacher that I've known. He was a graduate of McGill University, and got his doctorate degree at the University of Berlin under Emil Fischer. I think his thesis, I may not be right on this, was on the synthesis of asparagine. The way he used the blackboard, and his ability as a teacher is a little hard to describe. When he talked, he made you feel you were right there when something new was discovered. He not only gave you the formula, for instance, of uric acid, but he gave you the chemistry that proved the structure. And that was one of the keys, I think, to his ability to teach. He had a pleasant, easy-going way about him. You could just get everything down. He made an organic chemist out of me, for sure.

BOHNING: You had quite a combination between the organic and the inorganic, which played a vital part later on.

HYDE: Yes, some of these things occur unconsciously, you don't realize them. In the second year organic class we used the Perkin and Kipping text book(1). They had a new edition out. I never made anything out of it at the time, but the Grignard reagent was discovered about 1903 or 1904, and there was a page or two in Perkin and Kipping about its use in the synthesis of arsenic and silicon compounds. So I came to know that organosilicon compounds existed. I never figured I'd ever use it, it's just one of those things.

My advisor made a few mistakes, his arithmetic wasn't very good, and so I completed all the required courses and graduated in January instead of June of 1923. Then they gave me a job as a part-time laboratory assistant in the organic department. I was living at home, and I thought that I didn't know enough chemistry to go out and tell people I'm a chemist. I thought, "I'm going to get a master's degree." What this did was get me a chance to take all of the special courses that Dr. Boehner gave, which were in organic nitrogen compounds, and carbohydrates etc.; more concentrated, specialized courses. Call it "advanced chemistry" if you will. I took all of the extra courses he gave, and then I managed to get my master's degree in January of 1925.

BOHNING: Did you do any research at Syracuse?

HYDE: For a master's degree you were supposed to do some research. I think that Dr. Boehner being a Ph.D. himself, felt that their graduate school did not have what it takes to turn out Ph.D.s in organic chemistry. Although I'm sure that some people did get their doctoral degrees there. He was interested, and he suggested that I try to see if I could replace chlorine substituents on the aromatic ring. One of the ways I used was to put nitro groups, or other substituents, in the ring that would probably activate the chlorine. In fact, tri-nitrochlorobenzene has a fairly reactive chlorine. I never succeeded, but that was part of my thesis for my master's degree. Then I got to give a talk, at a Sigma Xi meeting. If there'd been an escape out, I could have jumped through the window. I just wasn't used to that sort of thing. I managed to stay alive, and after that report I became a member of Sigma Xi. Now I'm an Emeritus member of that scientific research society.

BOHNING: Do you recall any student colleagues from your Syracuse days, people that you knew there?

HYDE: Well, for instance, I did know Dr. [Floyd C.] Peterson, who later went to Midland. He was in one of those pictures. [Hyde refers to his collection of Dow memorabilia.] And I knew Dr. King, Shorty [Aden J.] King. He was a pretty close friend of mine. He got his doctor's degree at Syracuse. He worked with Professor McCroskey, who wasn't in organic but in inorganic chemistry. Most of the people that I knew there are gone now. You lose track of people after a while.

BOHNING: How were you supporting yourself through this time?

HYDE: Well, I was still living at home, and I had this part-time assistantship which paid for my tuition. The only disadvantage was the two or three hour ride on the street car for five years.

BOHNING: Did you do any reading on the street car all that time?

HYDE: It was pretty hard to do. I didn't really do much on that score, no.

BOHNING: What were you thinking about in terms of your career plans?

HYDE: Well, I began to realize that there was so much still to learn, and if I wanted to go further I would have to go somewhere else. I had heard that Professor [Roger] Adams was one of the leading organic chemists in the country, and that he was at the University of Illinois. So I started sending applications for half-time assistantships. I wasn't good in terms of research, or research papers. We had an Alpha Chi Sigma meeting at Colgate where they were initiating a new Alpha Chi Sigma chapter. This

5

is how little incidents do influence your career sometimes. I sat next to one of the professors whom they called "Cookie-Cutter" I do not know Professor Smith's given name but I know his Smith. students liked him. The reason they called him "Cookie Cutter" was that one of his legs was shorter than the other, and he had a metal frame on the bottom of his shoe. He told me that Professor Adams was one of the leading organic chemists in this country. So I wrote to the University of Michigan, the University of Wisconsin, and the University of Illinois. I heard first from the University of Michigan, and I would have worked with Moses Gomberg, but that was right in the middle of the year, and I'd agreed to teach part-time at Syracuse for the semester, and I didn't want to break my promise. The next university I heard from was the University of Illinois. Exactly the one that I wanted to hear from. Professor William A. Noyes Sr. was the head of the chemistry department at that time. I got a job as a part-time assistant so in the fall of 1925 I went to Urbana. The part-time assistantship involved working for Professor B. Smith Hopkins who taught freshman chemistry. I knew some of the people that worked with him, for instance, Lawrence Quill. Th job he had under Professor Hopkins was to look for element 61, The which is now promethium, that was then missing. Dr. Quill later became Professor of Inorganic Chemistry at Michigan State University.

BOHNING: They were looking for illinium?

HYDE: They thought they found X-ray lines that showed that they had isolated illinium, which is what they were going to name it. It turned up quite a bit of controversy because some Italian chemists came along, and argued that they were the first to detect it and they were going to call it italium. It is not clear whether they really had detected lines or not. Promethium was made later synthetically. It's half life is pretty short. I don't think it has been settled whether they actually had isolated any stable promethium, but they did have an argument with the Italians over the name. As it turned out it wasn't named either illinium or italium.

BOHNING: Where did you stay when you got to Illinois?

HYDE: Temporarily at the Alpha Chi Sigma fraternity house, which gave me a chance to get to Illinois and find a place to live. I went then into a private home where they were renting out rooms for students. Then I moved when I got acquainted with some of the fellows who were also in chemistry. Robert Yohe was one of them, and Lawrence Quill the other one, the one who worked with Hopkins. I've forgotten now, Yohe went into organic chemistry, I think. I think he's retired now, but I think he went into a survey lab at Illinois, which had something to do with coal and natural resources.

6 .

BOHNING: When did you first meet Roger Adams?

HYDE: As soon as school opened there in the fall of 1925. He took me on, and suggested a research program. Then I worked part-time in freshman chemistry. One of the things that I wasn't used to was having to do a recitation section. That nearly floored me, because I wasn't used to that sort of thing. On top of that, Professor [Harry C.] Kremers, who was also one of the inorganic professors there, used to walk in and he embarrassed me to the extent that the only thing that kept me from jumping out the window was that it was too high.

[END OF TAPE, SIDE 1]

BOHNING: What courses were you taking when you first went there?

Well, I had the required courses to take departmental HYDE: exams of the graduate school in analytical, physical, inorganic and organic chemistry. There were these four, and I had to take them again to get into the graduate school, which I did. There was a required course at Syracuse in physical chemistry based on the textbook Thermodynamics by Lewis and Randall (2), which I had to take over again. [Worth H.] Rodebush, who was professor of physical chemistry at Illinois, had been a student of G. N. Lewis. That was a required course. Adams gave an advanced organic chemistry course. I was in a peculiar situation there. I had to take the course, but also I got the job of grading exam papers and to design little problems for the class to solve. For example, "A material does this and does that. This is some of its chemistry. What is it?" I used to make out those things for people who were taking the course along with me. My colleagues didn't mind barking at me, or telling me I was wrong here and there. Once in a while I used to make mistakes when grading their papers, and when they found out about it they'd go down to Adams and complain. Adams would say, "Well, if that's the way he has marked it, that's the way it's going to be." He was that kind of a person, he didn't let you down, not matter what.

Actually, one of the students who objected to my grading was Wendell Stanley, who later got the Nobel Prize [1946] for crystallizing the tobacco mosaic virus. Well, his writing was so bad that sometimes I couldn't read it. I would get a little irritated about that although he was a pretty good student. Adams' writing wasn't anything to brag about, either. He'd leave a list of things he thought I should be looking at, and I had to go to his secretary to get them read.

BOHNING: When did you start your research with Adams?

7

HYDE: Almost immediately. One of the first things he wanted me to make were some mixed esters of phthalic acid. He was interested in looking at useful industrial things which could be patented. Now I don't know whether he got a patent on them but it is highly probable.

BOHNING: There were two early patents (3).

HYDE: That might be them. Anyway, he was interested in looking at the useful side of things. After that, I tried to make some derivatives of glucose, which wasn't very successful. Glucose is sensitive to some reagents, and I wanted to start with acetobromoglucose, and make some derivatives, but it never got off the ground. You remember the Kaufler formula for diphenyl derivatives, which was called "The Sandwich?"

BOHNING: Oh, yes.

HYDE: When folded back, it gave optical activity. About this time, [Frank] Bell and [J.] Kenyon in England, came along with an optically active species which couldn't be a sandwich and disproved the Kaufler formula (4). These were very difficult compounds to synthesize but they did show that restricted rotation was the cause of optical activity. I got the idea that we could get the interference with the same groups in the ortho positions if we got the asymmetry by substituting further back. That was part of my thesis.

BOHNING: I think I have that publication. The one from JACS [Journal of the American Chemical Society] (5).

HYDE: I didn't succeed in isolating a pure compound, but Wendell Moyer, who came after me, took on that problem, and in a month's time he isolated one, by reacting the bromo compounds, in the presence of cuprous bromide, I think, so joining the two phenyl groups together. Then there was another fellow working on ephedrine. One of Adams' interests at that time was natural products. In fact, one of our fellows in the lab worked with chaulmoogra oil, used in India against leprosy. Jim [James A.] Arvin was working on this problem, and trying to substitute different groups on the end to compare their behavior with that of chaulmoogra oil. There was a fellow, Browning was his name, working on the ephedrine problem already, but he wasn't moving very fast. He was trying to purify some compounds by distillation, and they were not that stable. All he had to do was what I did, make the hydrochloride and recrystallize it.

BOHNING: Adams suggested that you move onto that?

I've forgotten the details of whether he wanted me to HYDE: It is possible that Browning had been working towards a help. Masters degree, and had already left the University, when Professor Adams asked me to take on the ephedrine problem. When things used to go bad, things go haywire in the afternoon, I'd go down to the gym and spend a couple of hours down there trying everything [all the facilities]. One of the things I tried to use in the ephedrine problem was hexahydrobenzoyl chloride. I wanted to make the ketone and convert it to an ephedrine analogue. The way I tried to do it was to take hexahydrobenzoyl chloride and react it with diazomethane to put a methyl group in the place of chlorine. I built a fancy all-glass apparatus, carried out the reaction, and then tried to start distillation. I didn't realize that you had to be careful with diazo compounds. At a certain temperature, the top of the glass apparatus and the thermometer hit the ceiling. Hexahydrobenzoyl chloride is a nasty lachrymator so I had to go out and sit on the fence in the fresh air for a little while. And then I just tried it again. was pretty determined, and when the temperature got to the same point, away it went again. I hadn't completely decomposed the diazo compound. Then I decided to let that one go. Those were the two things that were part of my doctor's thesis.

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

HYDE: Wonderful. In 1928, there were the four of us who got doctor's degrees in organic chemistry: myself, Jim Arvin, Letha Davies, and Paul Salzberg. Paul Salzberg was one of Marvel's students. He retired about five or ten years ago. He was director of research at Du Pont.

This idea of producing restricted rotation in the di-phenyl synthesis was the first of a whole series. Before Adams got through more than two hundred papers and many graduate student theses were produced. All kinds of pretty fancy molecules, with optical activity due to restricted rotation. This attempted synthesis was part of my thesis.

BOHNING: You mentioned earlier that you had some interaction with both [Carl S.] Marvel and [Wallace H.] Carothers.

HYDE: Oh, yes. I had Professor Carothers in his qualitative analysis course. He was a good teacher, if you could stay awake. But there wasn't any question that he was a great scientist. Marvel, Carothers and Jack [John R.] Johnson were always together talking about chemistry. Because it was against the law to smoke cigarettes on campus they went over to Farwell's restaurant across the road and talked about chemistry while smoking. That was almost a daily routine. I remember one time, Letha Davies, who came from Pleasantville, New York, where the Reader's Digest is located, was selling subscriptions and she was trying hard to sell me one. I didn't have the money, but Adams happened to walk in and heard some of the conversation. He said, "I'll take a dollar bill out of my pocket, and you guess whether the last digit is odd or even. If you lose, you buy me a subscription; and if I lose I'll buy you one." Well, I guessed right, and he bought me my first subscription to the Reader's Digest. He was that kind of a person, very personable. And of course, very knowledgeable and a good teacher. In the classroom he immediately attracted your interest.

I don't think I had a course from Professor Marvel. He was a very nice person, always ready to help. I remember in my last year, he came one day towards the end of the term and said, "Did you send in an application for a scholarship?" I replied , "No, I'm satisfied with what I'm doing. I like it." He said, "Here's a form, you better send it in." The next morning he came around and said, "Well, you have a scholarship for your last year." I've forgotten just what it amounted to. But I got my tuition and fees with it and it gave me a chance to work full time on my thesis. So that was a very nice thing. As a boy, I had saved up about a hundred and twenty dollars peddling papers, and I used that up too. I wound up graduating without owing anybody anything. Well, you can see, I had some pretty good breaks. I was lucky, if you want to put it that way.

BOHNING: Did Adams give you freedom when working in the lab? Did he come in every day and see you?

HYDE: Oh, yes, he'd stop by almost every day and say, "What's new?" If you had something you wanted to tell him, he'd stop and come in and talk with you. If you didn't, he'd just keep on going. There was no pressure or anything like that. He was a research man himself, all the way through.

BOHNING: Did the Adams and Marvel groups work close to each other?

HYDE: I don't know whether they got together, or whether they compared notes or whatnot. Their projects were pretty much separate. One of the students at that time was Bill [William H.] Lycan. I remember Adams had a faculty meeting and invited all of us there. I was talking to Lycan, when Professor Adams came up and said, "Where's that stilbene you were making for me?" Lycan was supposed to have been making something. And right off his hat, he said, "Well, it's "still-being" [stilbene] prepared." This was typical of Lycan. He was a nice fellow, I knew him quite well. He lives up here in Naples. When he retired I think he was director of research for Johnson & Johnson. He first went to Du Pont, and then left Du Pont and went to Johnson & Johnson. Back in those days, almost every Ph.D. that came out of Illinois, wound up in Wilmington, Delaware. BOHNING: What were you thinking about career plans, as you were completing your Ph.D. work?

HYDE: Well, I really hadn't given it much thought. It was becoming clear that my greatest interest was in exploratory research and synthetic chemistry. I guess I just took it for granted that I would probably work for Du Pont.

About the time of my graduation, Professor Adams came in one day, and said, "I've just learned from Professor James B. Conant that he has some funds for post-doctoral work. If you are interested, you should write him." I thought that sounded good. I sat down and wrote to him, and he offered me a post-doctoral scholarship. And so I received my Ph.D in June with an opportunity to continue my education, and just about enough money for the fare home.

At this point I have to go back to the summer of 1923 to explain the next big event in my life. A sand bed leased by my father was located on a 200 acre farm. The farm was owned by a man named Westphal. He was a gentleman farmer who spent his summers at Thousand Islands. The people working for him quit that summer, and he wanted my father to take over the farming on shares. I had never lived on a farm, but I got the job.

I cut a hundred and fifty acres of alfalfa three times, ten acres of barley and fifteen acres of wheat. I had only one person to help me get it into the barn. I started June 10 and finished by September 3. Then I figured that I had earned a vacation.

A friend of mine, a chemical engineer, named Edward Ray, was involved in a training course for new young engineers at the Bethlehem Steel Corporation in Bethlehem, PA. When I finished the work in September, he invited me down to pay him a visit, and have a tour of the plant. So I went down to Bethlehem. That was in the Fall of 1923. And that's where I met Mrs. Hyde, a very attractive and intelligent 17 year old girl named Hildegard Lesche, who didn't know much English. From then on we corresponded regularly. She took an excellent citizenship program sponsored by Bethlehem Steel, and became a citizen in 1927.

On my way home after graduating in June 1928, I planned to stop to visit her in Bethlehem. I didn't have a cent left, but she had saved up about \$75, which was enough to buy a wedding ring. We decided we would get married. So we went to the county seat in Easton, got a license, and went to the home of a Lutheran minister, who performed the ceremony in his garden.

I went home for the summer to work with my father. I went to Cambridge in early September, found a place to live, brought Hilde from Bethlehem, and so we started life together in Cambridge.

BOHNING: Do you remember how much Conant was paying you when you got there?

HYDE: Yes. The Mallinckrodt Chemical Company or a related Foundation had furnished the funds for the new fancy chemistry building just completed, and Converse Hall was the big organic laboratory part. Conant had been on sabbatical leave at the University of California. This is the story that I heard, I don't know if it's true. They had offered him a pretty good salary at Berkeley I guess. The Mallinckrodt people didn't like the idea of losing Conant so, to get Conant back, they gave him a grant of twenty-five thousand, or something like that, to hire post-doctoral people. This is the story I heard. Anyway, I got one of the fellowships. I think it was for twenty-five hundred dollars per annum.

BOHNING: How does that compare with what they were paying you at Illinois?

HYDE: I think around thirty dollars a month. It just about paid your room and board. I used up that little bit of money that I had saved earlier. But Harvard was good for me. It really helped me a lot. Conant, at this time, was interested in blood pigments and chlorophyll. The first thing he wanted me to do was work with chlorophyll. I didn't know beans about chlorophyll at the time.

BOHNING: Did you meet him as soon as you got to Harvard?

HYDE: Yes, he was there. Boylston Hall was starting to fall apart, and that's where I started working. Actually, Conant and many of the other people had already gathered their stuff to move into Converse. I started work in Boylston Hall, where you weren't sure that the ceiling wasn't going to fall down on your bench. So I started working on chlorophyll and learned pretty rapidly many of the details. Chlorophyll E was one of the products that we finally isolated. Hans Fischer, in Germany, was one of the big porphyrin chemists. He got the Nobel Prize [1930] for his synthesis of porphyrins. [Richard] Willstätter and [Arthur] Stoll had done quite a bit of work to isolate chlorophyll and chlorophyll compounds (6). So there was a background of work to start on. When I started to isolate some of the compounds many of them were so insoluble, that I remember using five gallons of ether to extract a fraction of a gram of material. I learned pretty rapidly what chlorophyll was all about.

[END OF TAPE, SIDE 2]

BOHNING: Yes, I think I remember you had three papers with Conant (7), and also a paper on microhydrogenation (8).

HYDE: Well, after working to separate one of the compounds which was very insoluble, I came out with about fifteen milligrams of it and I had to have carbon, hydrogen, and nitrogen analyses run so I had to learn something about microtechniques. One of the interesting things about these chlorophyll compounds is that they are conjugated, they form a large conjugated ring with those four pyrrole rings tied together. You could reduce them to a colorless form. And soon as the air got in, they'd go back to maybe the same compound, maybe some other compound. In some of the work, we hydrogenated some of these compounds. When you had only a small quantity of the material, you had to get small gadgets to do it. That was the basis for my paper on microhydrogenation with Howard Scherp, who was also one of Conant's students. I've forgotten just what he was working on, but he had something to do with this work. I think later on he went to the University of Rochester. I'm not too sure about it now. I lost track of many of these people, of course. Dr. [Wendell W.] Moyer, who came to Illinois and worked on this restricted rotation problem with Professor Adams followed me to Cambridge. There was another chapter in that.

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

He was rather brusque, and he didn't mince his words. But HYDE: he was a nice person, there is no doubt about it. One fellow I knew was working on a problem, and he obviously was not going to He should have gone to Conant and told him that he solve it. couldn't do anything with it. Instead of that, he kept trying, which is probably good, but it didn't lead him to any results. So Conant told him that he would have to go somewhere else for He was a fairly strict person. He also wanted his Ph.D. There was no question about him being a great chemist. results. I learned from Professor Conant how to perform critical tests in a test tube which would give you a simple way of finding whether you're right or wrong. He was very good at this sort of thing. I learned from Professor Conant a lot of things.

BOHNING: Did he come into the lab, like Adams did? Was he there regularly?

HYDE: Not as frequently. He would rather call you into his office with your results. Another person I got acquainted with at Cambridge was Professor Kohler, Elmer Peter Kohler. He was the grand old man of organic chemistry there at this time. He was a very nice person. I'd go over to the lab sometimes after supper and I'd run into him. He used to take me up to his laboratory and show me some of the experiments he was doing. It isn't everybody that would do a thing like that. But he was that kind of a person. A real nice person. He was research minded, and he wouldn't publish a paper until every little sentence was dotted.

BOHNING: Was there anyone else there that you made acquaintance with, whether post-docs or professors?

HYDE: There were a few other people there. I guess I've forgotten them pretty much now. Oh, there was another fellow from Wisconsin, [Wesley R.] Peterson, who worked for Professor Conant. He was kind of a happy-go-lucky person. He and another fellow got interested in the stock market. When he came in the morning, he looked over the stock reports and he would say, "Well, I came down in an old flivver [beat up car], and I'm going back in a Cadillac." It was the time just before the stock market crashed. He went into it with another fellow and had invested whatever he had. And you could buy on a pretty big margin then. When his friend got a little worried, Peterson bought him off. A week or two before the stock market went to pieces. So he didn't go home in a Cadillac but in his old flivver.

There were two Petersons. My recollection is that "Iowa State" [Willard D.] Peterson had come to Harvard to get his doctorate, and "Wisconsin" Peterson was there working with Prof. Conant as a postdoctoral fellow. At this time Prof. Conant had a new extreme pressure machine based on the design used by P. W. Bridgman of the Physics Department in his extreme pressure studies. I can remember seeing a polypropionaldehyde gel sample so formed, slowly going back to it's normal liquid state.

BOHNING: What kind of an effect did the stock market crash have? You were there until 1930?

HYDE: I don't recall it had any influence as far as I was concerned. It was just something that happened. I don't know whether it affected the prices of things. It certainly had no impact on me.

BOHNING: And how did you find living in Cambridge at that time?

HYDE: We had a one-room apartment. It was quite expensive at that time. Fifty dollars a month, something like that. But we managed. The first month was however a tough one. We didn't have anything because I hadn't been paid yet. And we were down to our last dime. We were getting milk, because you paid that by the month. We were once walking down the street and my wife found a dime, and she went into one of the delicatessen stores there to buy a loaf of bread. But a dime was not sufficient for a loaf of bread. Finally the clerk gave her one. I could have borrowed some money but it wasn't me. We made a go of it. BOHNING: Did Conant tell you how long you would be there when you arrived? Or was that open?

HYDE: Well, I think we just took it for granted that it was for two years. Whether he told me, or whether that was the understanding, [I assumed that] two years was the limit.

Prior to going to Wilmington, Carothers taught a year at the girls' school [Radcliffe] across the road, it's part of Harvard now. Conant was a consultant for Du Pont, like Professors Adams and Marvel. Early in 1930, the recruiter from Du Pont was making the rounds, and he invited me down to their research labs at Wilmington. I went and I talked with Dr. Carothers. I would have undoubtedly worked in his polymer group, but when I returned, I met Professor Conant in the hall, and he said, "How'd you make out?" "Well," I said, "they offered me a job." I think the offer was thirty-two hundred. That was the going rate for Ph.D.'s at that time. He said, "They ought to be able to do better than that. While you were away there was a man here from Corning Glass Works looking for an organic chemist." And I said, "What in the world do they want from an organic chemist in a glass factory?" "I don't know, why don't you go down and find out?" That was his way, very abrupt and to the point.

out?" That was his way, very abrupt and to the point. So I called W. C. Taylor. Later he got an honorary degree from Alfred University for his work. It was [William C.] Taylor and [Eugene C.] Sullivan who developed the Pyrex formula. trademark, they don't like to have you call it Pyrex. They It's a almost lost Pyrex because it became such a common name. But it was Taylor that had been up to Cambridge. He had graduated from MIT, and he was the most appropriate person to visit. I wrote to W. C. Taylor and he invited me down to visit them. They were very nice people. I got to meet Dr. Sullivan. There was nobody there with an organic background, so it looked to me like a place where one could get a fresh start. Before I went down I wondered if they might be interested in organosilicon chemistry. I went to the library up at Cambridge and looked up a few of [Frederick S.] Kipping's papers, so I wouldn't be at a complete loss if that was what they were interested in. When I got there, I understood that they had seen some of the new organic transparent materials which were coming out like Pollopas, a urea-formaldehyde polymer that was just coming out named after Fritz Pollak, the Austrian inventor (9). It was a beautiful, transparent material. Also another perfectly clear material, polyvinylacetate was just coming out. I had the idea that they were considering these materials as possible competitors for glass. When talking with Dr. Sullivan, he intimated or suggested that there might be some field in between Some people had wondered if organic polymers field in between. Some people had wondered if organic polymers could be mixed with glass. "It's not a very good idea, but I wouldn't want to say it's impossible," Dr. Sullivan thought.

Then I suggested that organic silicon chemistry might have something to offer. What they were doing was, in effect, hiring me to tell them what to do. They offered me a salary something like two hundred dollars more than Du Pont. I thought, "Well, here's a place for a fresh start. I'm fairly confident of what I know," and I decided to take their job. They were just building some new laboratories in one of the older buildings, and I came up with ideas of what an organic lab needed. I had them order silicon tetrachloride and some of the reagents and other things that I thought I needed and I took the job.

BOHNING: When did you go for the first time to Corning?

HYDE: That was probably in April or May of 1930.

BOHNING: And you gave them this information about supplies and the laboratory before you went?

HYDE: Yes, before returning in the Fall, because they were in the process of building some of these laboratories. Then I thought of a couple of books that might be useful, that might be needed in the laboratory. Their information on organic chemistry was pretty meager.

BOHNING: Do you remember which books you recommended?

HYDE: I don't recall.

BOHNING: I had one other question I wanted to ask you on your trip to Du Pont. You talked to Carothers when you went down to interview. Could you tell me something about that interview? Did he talk to you about polymers?

HYDE: He did, and he talked about some of the chemistry. I wouldn't be able to say just what, now. One of them involved making some mixed esters. I made a few suggestions, that probably weren't much good. I did meet some of the other people that worked with him in the research laboratory there. As far as the conversation is concerned, I don't have a good recollection of it.

BOHNING: Then you came back, you turned Du Pont down and accepted Corning. You hadn't turned Du Pont down before you went to Corning?

HYDE: No, I hadn't. As soon as I got back, I wrote to the director of research and told him. And I received a long telegram wondering why I was turning them down, and they were sure they could adjust the salary. A lot of the fellows I knew had already gone to Du Pont.

BOHNING: When did you make your move to Corning?

HYDE: That would be in the latter part of August, 1930. I think by the first week of September I was already beginning to prepare some organosilicon compounds.

BOHNING: That was my next question. You did have some patent applications that came out in November of 1930. Electrically conducting coatings, for example (10).

That's a funny one. This is another little anecdote about HYDE: getting started there. The silicon tetrachloride that you could buy at that time had some titanium and a few other impurities in small amounts. It wasn't really pure. At this time there were people in the midst of developing Pyrex glass high-voltage insulators. If you tried to use such insulators for high voltage applications, you'd get a corona discharge that raised havoc with radios etc. They were iridizing surfaces and checking with small volt meters. (If the film on the glass is transparent and of a thickness in the range of the wave-length of light, a colored pattern results by interference, as in the case of a grating spectroscope. They wanted thin semi-conducting coatings to spread out the voltage drop and reduce the possibility of I was too timid at that time to ask them what they were They were part of the research lab there, and they were arcing.) doing. trying to get thin iridescent films on glass. One day I asked if one of the things I had noticed when redistilling silicon tetrachloride, namely the formation of an iridescent film if there was moisture on the glass, could be of any use. It seemed to be just like some of the things these guys were making. Finally, I got up courage and asked them if they'd ever tried it. "No, it's no use trying that, we're after something that's conducting." That was the first time I knew that. I kept thinking, "Why didn't they at least try it?" So one day, I thought, "If they're not going to try it, I will." I blew some silicon tet on a glass test sample one while I was heating it in a flame. An iridescent film formed. They tested it, and sure enough it was a conductor. In other words, you got enough reduction of some component in there, at least. That was the subject of this patent. I don't call it serendipity, I don't like that word. You have to have some insight to recognize something that's useful. Those are two expressions that I dislike: serendipity and anomalous behavior. Molecules will always do what they`re supposed to. It's our conceptions that are anomalous, or misconceptions.

BOHNING: I think it was Pasteur who said, "Chance favors the prepared mind."

HYDE: I guess there's something to that.

BOHNING: If you were purifying silicon tetrachloride, what did you first try to synthesize there?

I looked at some of Kipping's work. Of the Kipping HYDE: papers, the ones involving the phenyl and the diphenyl compounds These are the best of his papers. are the most significant. He wrote many papers, but those involving the preparation of crystalline compounds were the easiest to work with and to examine them by the traditional ways, e.g. melting points, and I decided to work with the phenyl compounds molecular weights. to start with. I hydrolyzed some of the crude reaction mixture and I got a resinous, low-melting, low molecular weight material. When it melted, it became quite fluid, and I stuck some of this in an ignition tube, a thick-walled Pyrex tube. I kept heating in the burner. Pretty soon, the tube started sagging and yet I had a nice clear liquid inside. This was a striking thing, of course. I was looking for thermal stability, so this was one of the materials that I thought was important. It was not a good glass, put it that way.

Along with some of these syntheses I also tried some of the alkyl materials, and I found out that they were water repellent. I also made some urea-formaldehyde polymers, as well as some polyvinyl acetate, and some phenol-formaldehyde compositions; that is, some of the resins that were common at the time, in order to compare their properties. None of them, of course, looked like glass. And in fact, the materials I got from the phenyl silicon compounds were a long way from glass. These experiments were part of trying to learn something about what I could get out of the organosilicon compounds. When I saw this water repellency, I thought, that it might be used in the frosting process of light bulbs. They used to leave some crystalline material on the surface which gave this frosted appearance. But it also weakened the glass so that they were subject to weathering, and when these light bulbs were stored in a warehouse where the humidity was high, many of them lost their strength and cracked. I thought, that as a water repellent, maybe it'll protect the glass. Which was not the case. What was funny about it was that you could treat that surface, and you'd get a perfectly water-repellent film [with water drops] running around it like mercury, but actually if you put a little phenolphthalein in the water, you would see it extracting alkali out of the glass surface. These were very crude exploratory experiments, of course. One of the disadvantages of ureaformaldehyde, and many other organic polymers was the lack of dimensional stability. Glass in contrast, has got dimensional stability. I went to an ASTM [American Society for Testing and Materials] meeting up in Rochester, and a speaker described the nine properties an ideal plastic material should have as an engineering material. The only one that glass lacked was that

you couldn't hit it with a hammer. In other words, glass was not considered a polymer but rather as a supercooled liquid. It didn't take me long to realize that silica is really a high polymer, and that's what glasses are. I couldn't prove it then, but with hindsight these siloxane compositions, and other organosilicon compounds, couldn't be anything else but high polymers.

BOHNING: For the first few years at Corning you were just making these materials?

HYDE: Taking a look at them, to see what kinds of things we could get out of them.

BOHNING: Did you have a specific goal in mind, in terms of their usage?

HYDE: Not at that time. One of the things that came along was this fifteen by forty foot architectural window over the bank of swinging doors at the RCA Building, over the Sunken Garden.

BOHNING: In New York?

HYDE: Yes, in New York. They were making these panels. It is a relief map, really, that fits into the stonework. There's a man with a calipers measuring off the universe, or something. Here they were making these things about that size [Hyde demonstrates], which had to be cast. Almost every one of them was different. If you cast glass panels of a size around 18x26 inches and can get it to the same dimensions, within an eighth of an inch, you're doing pretty well. They were making this large glass window already, and didn't know how they were going to put them together. Polyvinylacetate [PVAc] has the same refractive index as Pyrex glass within the third decimal place. I did some experiments (loading tests) that showed that it would hold the weight without too much plastic flow.

[END OF TAPE, SIDE 3]

HYDE: Union Carbide made big slabs of PVAc and cut them into slices. We put these against the glass, and brought hot platens in to melt it on to the glass. That brought the glass out to accurate dimensions. Still something had to be put in between them while in place. But what? I came up with a very viscous, a little bit sticky solution of PVAc in its monomer. I went over to Carbide's laboratory at Long Island City, where they made this material up for me. So when they put this window in, they had metal clips at each corner, of course, that held the glass. I went down in New York for a month, squeezing out this stuff that was going in between. I had permission from the Glaziers' Union in New York City to be up there on the platform. So this is the way that we put that window together. With this "glue" which was easy to work and it fitted perfectly between the other slabs. Then the monomer would slowly polymerize and make a solid seal (11). That's how that window was put together and how I got involved with it.

BOHNING: Next time I'm in New York I'll make sure I take a good look at that.

HYDE: It's interesting. It's still there. I go over and look at it frequently. This was one of the opportunities that came along. Then there was talk about building a large telescope mirror for Carnegie, I think the Carnegie Institute was backing and funding it, and this resulted in the large Pyrex mirror for Mt. Palomar. When I heard and thought about it, I knew it would be difficult trying to fuse powdered quartz, as it melts way up around 1,720°C, and to get rid of crystal structure residues (ghosts), which cause distortion of transmitted light, would be almost impossible.

I thought, "Here I've got silicon trapped in a monomeric form. Since vitreous silica, is a high polymer, I can build on it by putting a volatile compound such as SiCl4 through a flame to hydrolyze and form small silica units, with the flame directed at base target. That was the basis for this other patent (12).

Because of it's low expansion and its beautiful optical clarity, the vitreous silica formed this way, the method has become an important industrial process for making disks that are component in radar relay systems, large telescope disks and optical fibers.

It got me a little off the track of organosilicon compounds again. On top of that, it occurred to me that you can get a very finely divided material if you just blow it out in the air. In this form I used to call it silica soot, because of its extreme fineness. I thought that it ought to be a good filler for rubber. So I spent quite a bit of time filling up a burlap bag with silica, that we sent to some rubber company, I don't remember now which one. This was probably in 1933 or 1934. Never heard back from them. Now, of course, it's a widely used filler. That was one of these things born forty years too soon, I guess. I tried to work with it, and tried to get other people interested in it. I did make some good sized quartz tubes, vitreous silica tubes. I made the gadget here [Hyde demonstrates object] that worked out pretty well. Also I could make preforms. You could blow this stuff without melting it to make a porous mass, and then sinter it.

porous mass, and then sinter it. I don't know just when it was decided that Corning would go into the fiber products business. It must have been during 1934, because before they built the plant, I dealt with some of the bonding, lubricating and sizing problems. The plan was that they would build a fiber products division in Corning. They got together with Owens-Illinois, and I think that there was a cross licensing agreement which came about because Corning had worked with Fiberglass materials, and had some pretty good patent coverage in this area. Then I think it was John Thomas, at Owens-Illinois who had come up with the idea of melting glass in an electrically heated platinum boat with a whole bunch of little holes in it. Or Owens-Illinois may have already set up their plant in Newark, I'm not sure about this. It was later in 1937 that it was decided to form Owens-Corning Fiberglass Corporation. In the meantime, Corning set up a fiber products division amongst their various plants in Corning. The research problems associated with that were directed to me and I went down to the fiber products division, in the early part of 1936, I think. We tried all kinds of things for binders and sizes and things like that. Corning's intention was to go into the high technology aspect. Electrical insulating tape and woven fabrics, rather than straight insulation. But this insulation gave them a problem, too. I did look at some of the patents which covered fiber products.

Bakelite had come out with low molecular weight materials that were water dilutable. At that time, phenol-formaldehyde resins were about the most stable type of material. They had another plus in the anti-fungal activity. It has some phenol in there that keeps bugs away. When you have molten glass and some flames and hot gases coming from the melt spinner, you don't want to have flammable solvents around. It occurred to me that it was just the right stuff to bond fiberglass (13). This is the pink stuff that you see around. I think we did the first work on that, back at Corning in the fiber products area. They're still using it. Then we tried to use glass as an ion-exchange material, to study the chemistry of glass surfaces, because when you've got a lot of fibers, you've got a lot of very easily determined and accurate surface area to work with. This was one of the things that I was interested in at the time. When our salesmen went out and tried to sell glass tape as an insulating material, they got turned down by GE and Westinghouse. It was thought a good material, but more expensive and because there was nothing in the interstices the insulating value was no better than the air in there. After I heard that story a couple of times, I thought, "If that's what it's going to take to sell glass tape, I'll make a thermally stable insulating varnish." Which is the first time that this thing had come along.

BOHNING: What were you reading at the time? Were you keeping up with what other people were doing in the 1930s? How were you doing that?

HYDE: At this point I should mention that for a number of years before WWII I abstracted the <u>Glastechnische Berichte</u> for <u>Chemical</u> <u>Abstracts</u> and <u>Ceramics</u> <u>Abstracts</u> during which time I learned something about glass chemistry. You mean in the area of organosilicon compounds? There was really little of it going on.

21

BOHNING: Was Kipping still publishing?

HYDE: Kipping had published a couple of papers, but they were more general. One paper that I never liked, quotes Kipping as saying that everything had been done in the organosilicon field (14). I think that wasn't quite the way Kipping said it. But that's quoted many times.

BOHNING: I think I have a quote here, I guess in 1937, that Kipping had said there was little hope of any practical application of the material.

Well, it amounted to about that. I can't give you his HYDE: exact words. I always thought he overdid it a little bit. He didn't publish any more papers that were significant, let's put it that way. But the Russians had done some more. [Kusma A.] Andrianov had used the ester Grignard reaction (15), that Mac [Rob R. McGregor] and Earl [Earl L. Warrick] came along with later. I don't remember exactly when Andrianov's paper was published. Friedrich Wöhler is the one that coined the "silicone" terminology. I have his original paper (16). Frederick Wöhler had worked with metal silicides and had isolated a complex hydrosilane composition, probably a mixture and partially oxidized, which he called a "silicon", presumably because it resembled a ketone in composition. Kipping used the term more directly later on, in some of his work with the diphenyl, but I'll give Wöhler the credit. In fact, [Albert] Ladenberg and [Charles] Friedel made some organosilicon They used zinc alkyls, for instance (17). [Walter] compounds. Dilthey had made the cyclic diphenyl compound, I think (18). Ι gave a talk one time on this. Kipping's primary interest was to show that silicon had a tetrahedral structure, the same as carbon. He was itching to get an asymmetric silicon atom. When the Grignard reagent came along, that gave him the opportunity he needed. I would still question a little bit his resolution of a compound. I think he probably did show that he did get some optical activity. The big advance in that area was later on, by Leo Sommer and his students (19). He went into it in a big way, and studied the mechanism of some of these reactions and made a real contribution to organosilicon chemistry. But I think that stereochemistry was Kipping's primary objective. He was certainly an academic researcher.

That paper we published (20) got up his ire (21). He also wrote a nasty letter to Arthur B. Lamb who passed it on to me. Kipping was right on the identification of one of those compounds as the cyclic tetramer. He was off base on the diphenyl silane diol. When [Richard C.] DeLong and I published this paper, we were forced a little bit into it, because Rochow had published before us (22). I hadn't thought about publishing at the time, but when we got through with the diphenyl compounds, I considered that our molecular weights, which were the traditional way of pinning down something like that, were closer than his so I didn't hesitate to say what I did in the paper. That upset him very much. I still haven't been able to find that letter that I got from Kipping directly.

BOHNING: Oh, you heard from him?

HYDE: Yes, I got a letter directly from him. He was pretty vitriolic, if you want to put it that way. He accused me of being misled by some of my co-workers. Then we had to get busy and really pin the thing down after that, I went out to work with Ludo Frevel at Dow, who was an expert in X-ray analysis. We found that the diphenyl cyclic tetramer was a case of polymorphism (23). There were three forms of the cyclic tetramer, if I remember well. One of them was a crystal containing benzene of crystallization. One form of the cyclic trimer melted right at 188°C, and the other melted at 201°C. So we were both wrong, if you want to put it that way.

It was much more complicated than it showed. In the case of the diphenyl diol monomer, there is just another little anecdote. He found fault with our attribution. He had a melting point of 132°C, but it wasn't very definite or very clean cut. So we got busy, and kept recrystallizing the diphenyl diol monomer; the more we recrystallized, the higher the melting point. Finally, it was up around 170°C. What had happened was that Kipping was using sodalime melting point tubes which catalyzed the condensation of the compound, so he wasn't getting the melting point of the diol. It really was up sky-high. Later on I sent them a sample of that diphenyl monomer diol. I never heard back. But we sent a little note to <u>Chemistry and Industry</u> correcting it (24). I wasn't about to take all of that sitting down. I respected Kipping. He was right on the one compound that he got angry over, there's no doubt about that. But he wasn't right about the polymorphic character of those darn things. And to have a tetramer and a trimer that melt at the same point, and you mix them you get a melting point depressed to around 160 or 170°C, something like that. So it was not a simple thing. It turned out to be useful. Disagreements sometimes do.

We had one of these Kipping Award Dinners at an ACS meeting in Philadelphia. His son was over but he was really nonchalant. He didn't realize what it was all about, didn't act as though it was any thing of a big deal as far as he was concerned. It seemed that way from his attitude. I don't think he followed in his father's footsteps very far.

BOHNING: Maybe we should go to that first meeting between Corning and General Electric.

HYDE: It was towards the end of 1937, I think. After our salesmen came back from GE and Westinghouse with the story that they didn't want our tape, I told the company that I'd make some thermally stable resins. This stirred up some interest in our

sales people. Leslie Morrow was our manager at the fiber products division. He was an electrical engineer, previously the editor of <u>The Electrical World</u>. He invited these people from GE to come down and see what I had in mind, and to see what I was doing. GE already was in the resin business, making their own insulating varnishes. As far as I knew, I can't speak for other people, but I thought that this meeting might result in our getting together to develop something. [Winton I.] Patnode, a research man, and I think A. L. Marshall, the chief chemist of General Electric at the time came and I talked with them about what I had done. These were crude phenyl-containing materials and I thought by changing the groups, and doing various things it would lead to a quite versatile material.

BOHNING: You described the chemistry pretty carefully to them.

HYDE: Well, what little I knew, which wasn't very much, at the time. I knew that we could put a variety of groups on there, and that probably we could modify the properties over quite a range, by this way. When they left, I detected some sarcasm when they said, "See you in a year or two, and we hope you'll have something developed or ready by then." This always bothered me a little. I didn't like the way they said it. Maybe I misinterpreted their meaning. Almost about this time, Dr. Sullivan came down to the fiber products place, and he let me know confidentially, it hadn't been let out yet, that they were planning to put their fiber products division and Owens-Illinois fiber products division together to make the Owens-Corning Fiberglass Corporation at Newark, Ohio. He wondered what I thought about it, and what I'd like about it, or what I'd like to do. I told him, "There are two important things, as far as I'm concerned. One of them is chemistry of glass surfaces, and the other one is this organosilicon chemistry, which I think has quite a lot to offer."

I rescued some of my equipment that I liked, such as a nice analytical balance, and I got them back up to the main laboratory before these Owens-Illinois guys descended on the place. They put a tag on everything. This gave me a chance to get back fulltime on organosilicon chemistry. I decided to put an alkyl group in that brittle resin to modify it. We still didn't know how to make a polymer, really. I started making up phenylethyl siloxanes, and looking for ways to polymerize them. The only way we knew was to take a crude mixture that had a lot of polyfunctionality in it, heat it, and take what came out. This happened along about April 1938, about three months after the GE fellows had been down. About three or four months later, I learned from a GE salesman that was in Corning that they'd started a research project and put a man by the name of [Eugene G.] Rochow on it. That was the first time that I heard his name. That alerted me: Morrow had invited these people down. They hadn't barged in, we had invited them.

We started making some of these materials. Westinghouse was getting a little bit interested. They wanted some preliminary materials to work with. Still, making these things at that time in the lab was quite a job. Making the Grignard reagent, separating the solids, and then distillation wasn't easy. We were able to cause gelation, or polymerization by acid treatment. I wasn't sure just what was going on when we did it, but at least I could get a polymeric resinous material out of the hydrolysis. We made up pint samples, a reasonable amount to make in the laboratory. We sent samples to Westinghouse. I don't know that anything ever came of it. But we kept looking for ways of making polymers. It wasn't long after I moved back to the main laboratory that, I don't know whether you'd call it a merger or a buy-out, but in 1937 Corning took over the Macbeth-Evan Company of Charleroi [PA]. Macbeth moved to Corning. Apparently it involved some Corning stock in a stock

Apparently it involved some Corning stock in a stock exchange. In April I learned that Corning had acquired, as part of the deal, a fellowship at the Mellon Institute.

[END OF TAPE, SIDE 4]

HYDE: The first I knew about it, Rob Roy McGregor, the senior Fellow, came up to talk with Mr. Macbeth. This is how I learned that they had begun work on Corning projects.

The first job that Corning had given the fellowship was to develop a coating for their glass building blocks, which were just emerging as a new building material. Corning did not want to go into the building materials business, so what they had done was to form a joint company with Pittsburgh Plate, the Pittsburgh Corning Corporation located in Port Allegany, Pennsylvania to manufacture and sell the product. A major problem was that the cement used to hold the blocks together adhered so well to the glass that, on curing, they caused spalling. The first coating they tried was polyvinyl acetate. Apparently the moisture and alkalinity of the cement adversely affected the adhesion, and this led to the addition of ethyl polysilicate to modify the polyvinyl acetate coating. Union Carbide had a large fellowship at Mellon Institute so their

The first coating they tried was polyvinyl acetate. Apparently the moisture and alkalinity of the cement adversely affected the adhesion, and this led to the addition of ethyl polysilicate to modify the polyvinyl acetate coating. Union Carbide had a large fellowship at Mellon Institute so their products were readily available to Mac and Earl [Warrick]. I think this probably got them thinking about modifying the silicate ester by attaching methyl groups to the silicon. They called it internal plasticizing. I never did like the terminology nor the idea that dimethylsiloxane is high in silica. It's a different chemical, which yields a 81% silica residue on combustion. This is some of the slang that gets into the picture as you go along. When we found out that they were interested in organosilicons, we got together, planned programs and worked together. The idea was, whatever they're doing, keep on doing it, and whatever we're doing, we'll keep on doing. That was a good start. We did start talking about some of the fancy things we might be able to do with organosilicon chemistry.

BOHNING: How frequently were you getting together?

HYDE: Every few months, maybe three or six months. It wasn't on

a particularly regular basis. Later on, it was on a more regular basis. I started getting their weekly reports, so I could keep abreast of what they were doing. We were getting towards the War. Things were getting pretty complicated. The pressure started building to get more materials to work with. Owens-Corning and Corning were anxious to sell Admiral Hyman [then Capt.] Rickover something. They wanted to sell electrical tape, and Rickover wanted more power out of smaller motors. This thing dragged along a little bit.

By this time I had found out a little more about polymerization. Acid was not a good way to polymerize, for instance, a phenylmethyl or a phenylethyl compound. We found out later that it was clipping off some of the phenyl groups, and these gave body, increased the viscosity. We were probably doing some rearrangement. We didn't know at this time how reversible the chlorosilane hydrolysis was, and how important to the equilibrium was the nature of the attached group. We learned quite a lot about it later on.

I've forgotten now just when DeLong came. We began to have the opportunity to build up our group a little bit. After we realized what was going on, we heard that Rochow had given a paper. I think it was at a Detroit ACS meeting, and somebody brought a copy back to Dr. Sullivan. This disturbed Dr. Sullivan a bit, because he felt that there was a matter, from an academic point of view at least, of priority involved in the case. This didn't suit him very well. Then we heard that Rochow was going to publish a paper. Dr. Coolidge, at this time, was the director of research at GE. He was the great inventor of the Coolidge X-ray tube and things like that. I'm sure that he and Sullivan were good friends. I think it was by by this route Rochow finally came to Corning; he agreed to hold up his paper while we got one written, which wasn't for me a very good thing. But that's where this DeLong and Hyde paper came from (20). We had decided already that, although Mac was still using the ester synthesis, in the long run we were going to have to go through chlorides, the chlorosilanes. So that's why, when I got DeLong into our group, I had him synthesize some of the chlorosilanes. He liked distillation although this was aggravating for me, because it was taking him time to do a good job. The compounds boiled awfully close together. He did separate them however, so that we were able to characterize the methylchlorosilanes, the monomethyl and the dimethyl. And I've forgotten about the trimethyl chlorosilane, I guess that was in there, too. But they're all close boiling. And close to silicon tetrachloride as well. They're mean materials to handle. That's the background on how we got a patent on the methylchlorosilanes (25).

BOHNING: That was before the War, was it later?

HYDE: Rochow's patent came out before the War (26), then secrecy orders were placed on all patents and publications, including our patent on the phenylethyl silicone (27) and the use of silicones for electrical insulation, and so they were not available until

the war ended in 1945. Our first patent was on the phenylethyl [silicone] (27), it was only later that we got a patent on methylchlorosilane (25). That caused an uproar. This is a little anecdote. When the patent came out on the methylchlorosilanes and hit Schenectady, their research people were up in arms. They thought Rochow had been robbed, because he'd made chlorosilanes. We got the story because Dr. Lee Fowler of our patent department was acquainted with some of the patent people down at GE, and he knew what had happened. Their patent department called the research people together to tell them that Hyde got the patent because he'd made them first. That didn't sit very well with them, I suppose. This is the story, as I understand it. Of course, it's second hand.

Another part of the methylchlorosilane story is that the patent wasn't issued for very long before Professor Henry Gilman, our first Kipping Award recipient [1962], got interested in organosilicon chemistry. He published quite a few papers and being a very thorough person dug up a Berichte reference that mentioned methylchlorosilane although I am quite sure the author never made one. There wasn't much for us to do but disclaim the It's like giving it to the world. This was the patent. background when we had this big patent interference with General Electric Company. The way they settled it was to cross-license the patents up to that time. One of the important GE patents was the direct process of reacting silicon with methyl chloride. We had many good patents, too, as far as that goes. I wouldn't want to go through an ordeal like that again. GE came to Corning with their attorneys, and they started taking testimony Monday This involved Dr. [O. Kenneth] Johannson and Dr. morning. [William H.] Daudt and some others for a short time. But I got the worst of it. They started taking testimony Monday morning until five. Then on Tuesday they decided that they couldn't get it all in, so they came back after supper from seven to nine. They finished around Friday noon. I thought I'd been put through the wringer. I wouldn't want to go through that again. Every time you made a statement, you're a liar unless you've got some evidence there to prove otherwise. It just gets you after a while.

BOHNING: What year was that?

HYDE: This could have been in September of 1945. Because later the patent lawyers and myself went down to GE to take testimony and put Rochow on the spot. Which they did. It was winter, I remember that, so it could have been in early December. They gave him a pretty good workout, I think. The man who was the main lawyer in this interference for Corning was Fulton B. Flick, I think from Pittsburgh. I understand that he had a degree in chemical engineering before he went into law. He worked for the Aluminum Company of America and had the patent for this heavy anodizing of aluminum, so that it would take dyes (28). I don't know whether you ran into Harry Dingman. He was a chemist out at Midland who went to Pittsburgh, worked in Flick's office, and got his law degree, I think from the University of Pittsburgh. He was head of the Dow Corning patent department until his retirement last year. Fulton B. Flick died quite a long time ago.

BOHNING: When was the joint licensing agreement reached? They took testimony in late 1945. When did the settlement occur?

HYDE: It would have to be in 1946. Then papers and patents started coming out.

BOHNING: I wanted to ask you about Rochow. You said he came to Corning.

HYDE: That's the first time that I met him.

BOHNING: Do you remember anything about that first meeting with him?

HYDE: It was in Dr. Sullivan's office. I think he had talked with Dr. Sullivan first. I think Dr. Sullivan invited me to come over and meet him. I don't recall any particular thing that was talked about. But I'm pretty sure that he had agreed to hold up his paper until we had one ready to publish. Which didn't quite happen; I think his paper came out a month or two before our paper (20,22). I got a little better acquainted with him when we had this interference, although not really well. I figured that Rochow probably never realized, or may not have ever been told, that Patnode and Marshall came down to Corning. There's another interesting sideline that I learned: that Rochow got his doctorate at Cornell. Cornell had this Baker Lecture Series where they brought in people for a year. In fact, Pauling spent a year in Cornell. I listened to one or two of Pauling's lectures when I was at Corning. But also, Alfred Stock was a Baker lecturer. Stock did some early work with organosilicon compounds. I don't want to belittle Rochow or anything, but Rochow didn't seem to remember that he worked as a graduate student with Stock. Stock had made dimethylsilane, with two hydrogens on the silicon (29). He never did anything with it, probably never hydrolyzed it.

BOHNING: This was in the late 1930s. Then the War started, and there was demand for more of these materials. Somewhere along the line a fire occurred, as I understand it.

HYDE: Oh, I used to call that "The Chicago Fire." What happened was that we got pretty careless with ether. You use a lot of

ether in the Grignard reaction. I had been trying to make a Grignard reagent with the phenyl compound, and I had a stirring motor that wasn't exactly explosion proof. DeLong had set up a distillation apparatus. We used hot water to boil off the ether. What he and I had forgotten to do was to take out one of the corks we had put at the ends of the condensers to keep everything dry. He was heating a pretty good sized pot of ether with the condenser blocked off. I had a little glass-working torch on the side bench and there was a little pilot flame on it. We were sitting there talking. All of a sudden he remembered what he had done. I wondered why it wasn't boiling, why we weren't getting a distillate. All of a sudden it hit him. He shot out of the door and down the stairs. I think he was on Market Street by the time it happened. He knew what he had done. As soon as I saw the thing pop like this, I quickly ran over to douse it, I put the pilot flame out and started out the door about the time it caught fire. Here was a great big billow of fire and smoke over my All I got were a few little spatters of ether on the back head. of my ears. We learned a lesson from that. You can't get careless with ether. The sprinklers were set off in the whole floor. We had people sweeping water down the hall, and down the stairs. Fortunately, the building was made of concrete, so it didn't soak in, and cause water damage.

BOHNING: I guess I'm looking at when the connection with Dow was made. Was this accident the beginning of that?

HYDE: This was before that, it didn't have anything to do with it. It was Admiral Rickover and Owens-Corning people that were putting pressure on Dr. Sullivan. I was trying to make the polymers in larger quantities. Then I began to realize that when it came to the engineering and the pilot plant work, that this was a long term effort. I don't think at this time we had contacted anybody at Dow. But we weren't going to be able to do anything in a reasonable length of time. I knew that Dow had the raw materials and the engineering skill and equipment so I suggested to Dr. Sullivan that we contact them and see if they might manufacture it for us.

At that time, the only way we had of resinifying was to use acid. Phenylethyl looked like it would be the monomer to go with as the methyl Grignard was a miserable material to handle because the solubility was very low. Dr. Sullivan talked to Dr. Britton and invited him down to see what we had in mind, and later on they agreed to try some pilot plant runs in a larger kettle, out in Dr. Britton's Building 20 laboratory in Midland. As is usual with pilot plant efforts, the first few are disasters. This was no exception. Dr. Clarence Moyle was a new man coming into Dr. Britton's laboratory and he got the job of trying out these first experimental pilot plant attempts at making a resin. He finally got some that was reasonably good. This was during 1940 and 1941. The result was that Britton saw some broad possibilities, and he got interested in it, too. It began to look like it was a pretty complex thing. I don't know all of the details, back and forth, between the two managements. I think the War had already started, hadn't it.

BOHNING: The War started in 1941.

HYDE: December 7, 1941, wasn't it? So it was during 1941 that Moyle worked on the pilot plant. Then, I think during 1942, the decision was taken to join efforts. It was a complex project and Rickover [Head, Bureau of Ship's Electrical Section, Department of the Navy] was on their heels. Some of that is described in this book on Collings (30). It was decided, over a handshake, that the best way to handle the development was to form the Dow Corning Corporation and this happened in early 1943.

At about this time, probably 1941, Mac and Earl were making dimethyl siloxane fluids using ethyl silicate and Grignard reagents, requiring no solvent, and we needed larger quantities of phenylethyldichlorosilane. Sullivan thought, "We've bought them a nice kettle down there, why can't they make some of this chlorosilane? He kept insisting, and they tried it, and it was a kind of a disaster. They just drove everybody out of the Mellon Institute basement, I guess.

Rickover was on our tail. He was trying to push the thing. The only time I met Rickover, was when they had started to do something over in the Dow plant. This was after [William R.] Collings and [Shailer L.] Bass came down to Corning to help get things all together. I think that was the start of the motor program over in Dow, since Dr. Clarence Moyle had now made some reasonably good phenylethyl silicone impregnating varnish. I can't say just when this meeting was in Corning, probably in the late Spring of 1943. I think Dr. Bass, W. R. Collings and Dr. Britton came down. And Mac and Earl came up from Mellon. The idea was to put every bit of experience and understanding and research together to get things rolling. I know that we still didn't have a decent polymerization technology. We were getting into the alkali monkey business before this time. We already had learned how to make some high polymers, and that we could depolymerize and purify the intermediates. We met in Dr. [Jesse T.] Littleton

called a rubber, but it was made from a gel, and we'd call it a mush, I guess, now. But they had attempted to make a piece of rubber out of it. Again, this was going to depend on making some decent polymer. They brought that along, and they brought some of the oils. We already had some of Mac's oils in the laboratory up at Corning. Dr. Bass was taking notes at a terrific rate. I didn't know of anyone that could get down so much in a short space, and have it all organized, and readable.

space, and have it all organized, and readable. I don't know whether Mac had already measured some viscosity/temperature slopes, but when they saw some properties of the oils they thought of making a lubricant for the Mine Safety Appliance Company. We still didn't have a decent polymer process. I think Mac must have had this flat temperature/ viscosity slope property, because when Shailer saw it he recognized it right away. He knew about this ignition junction failure on planes that were trying to fly across the Atlantic. Right off the pad, it dawned on him that if you put a filler in there with the polymer showing this flat temperature/viscosity slope, you'd have a grease to fill these ignition harness junctions that would prevent flash-over, and that wouldn't run out. That was our first product. They used the big alkylation reactors for cellulose in which they made ethyl and methyl cellulose for making the dimethyl silicone by the etherless Grignard.

[END OF TAPE, SIDE 5]

BOHNING: In 1938, you became manager of the organic lab. How many people did you bring in, and what was your staff like, in that very busy period just before the War?

HYDE: Johannson had been working with Dr. [Charles H.] Greene. You may not realize it, but a glass surface completely free of flaws is very strong. I've seen a pencil-sized glass rod bent into a U. If you've got a flaw in the surface, that concentrates the strain in one place which goes beyond the strength limit of the material. If a fly would light on one of those things it would explode, practically. They were trying to turn out some fortified glassware. And they needed something to protect the surface to make the glassware stronger. surface to make the glassware stronger. Johannson was working in our lab there and he gradually moved into the silicone project. He saw some of the wetting properties, and he saw how the monophenylchlorosilane could react with adsorbed water on the glass surface to form an oriented film. He thought he'd try it as a primer for ethylcellulose and some of the other coatings that they wanted to put on glass. So that was really the start of some of this priming business. That was Kenneth Johannson. It wasn't long before he was working full-time with me. Then, in 1940 there was a little pressure, and I kept telling Dr. Littleton, that we needed more people. So in 1940 while out on a recruiting trip in Cornell, he contacted Bob Fleming who was getting his master's degree in organic chemistry at Cornell and invited him for an interview. He came to work with us in that period.

I don't know whether we'd already had the fire and DeLong had gone, but we needed some people to do analytical work and things like that. Because the analytical needs of the glass works were somewhat different from organic chemistry, I kept asking for more help, and Dr. Littleton said, "You know people up at Harvard. Why don't you call Harvard and see if they've got anyone there who is interested." I knew Professor [Louis F.] Fieserat Harvard. One of his students who was just graduating was William H. Daudt. Daudt came over, and they offered him a job, and he stayed. Julius Torok was an electrical engineer who had been working in the high tension area; he came to Corning quite early. One of the things that he did to test some of the resins we were making, was to make up some solenoid coils, and then overload them to take the temperature up so that he could get a reasonable thermal stability test. During the War, we had
about fifteen people working at Corning doing analytical work, and others doing testing, bench work, and the synthesis of intermediates.

About this time, 1939-1940, we had learned how to make a reasonably good impregnating varnish that could be cured by heating. I wanted to make a batch of phenylethyl resin for Westinghouse. It took quite a while, I struggled to get about a pint of it together. I was bodying it by using HCl. You could also body these things by air blowing. That's another bad way to do it. Because what it was doing was chewing up some of the groups that you went to a lot of trouble to put on there. What happened was that I had one experiment underway, and I went out to dinner to the Kiwanis Club. When I came back the confounded thing had gelled. I kept scratching my head over it. I was not about to throw it in the scrap jar when it came to me that it had to be the last formed crosslinking siloxane bridges that must have caused the gelation. If it would react with alkali, I could un-gel it. That worked like a charm, a little bit of alcoholic alkali. That was a clue to some of the behavior we got into We felt that we had to make polymers in a after that. controllable way. I thought, "Here's one thing we can do if it works that way, we can use stoichiometric amounts of alkali on the dimethylsiloxane hydrolyzates for example, to make the endgroups, and make any size polymer we want." It didn't turn If you tried to polymerize some of the material, you would out. get a gel. If you wanted to make a high polymer, the monomethylsiloxane content had to be below ordinary analytical detection. What I found was that if I put small amounts of alkali in there with the dimethyl hydrolyzate, and heated it up to around 200 or 250C, then a mobile system formed causing depolymerization, and the cyclics distilled out while the alkali ties up the mono and substituted compounds which are nonvolatile, and you've got a beautiful purification system. That

worked, and turned out to be a pretty important thing. After we did that, we found that we could use a small amount of alkali on these purified cyclics, mostly cyclics. I think they were all cyclics, probably, under these conditions. You could take a polymer up to a pretty good viscosity. Then Johannson, a physical chemist, came into the picture again. He got into the business of studying the mechanism of the reaction. By this time we'd also hired another physical chemist, who worked with Johannson on this polymerization. It turned out that this was a very mobile system, and it was entropy that led to a polymerization. It's such a bond-rearranging system that all bonds are possible to form and what you do is wind up with a probability distribution of chain lengths, polymer sizes, etc. We could go up to where it was a perfectly dry plastic solid, which meant a high degree of polymerization. In studying this, I found out that qualitatively, it was an ionic reaction, because you could speed it up with higher concentrations of alkali. The The active polymerization catalyst wasn't really the sodium hydroxide but the silanolate salt that was formed <u>in</u> <u>situ</u>. A little bit of a polar solvent, like acetonitrile, would speed the reaction up about a thousand fold, another evidence that it was an ionic It may have increased the concentration of silanolate reaction.

ion in there, too. Johannson got pretty interested, and started working on the kinetics of the reaction (31). It turned out that potassium silanolate was about five hundred times faster than the sodium. It ionized more and gave a higher concentration of the actual catalyst doing the job. This got us into making polymers that were half-way decent for making rubbers with melt viscosities of 10⁶ centistokes or more.

BOHNING: This was occurring about the time that Dow Corning was formed?

HYDE: Yes, this was about that time. The patent question was also getting to be important. Corning hired a fellow by the name of William Blackburn, whose main job was to get an insight in to siloxane chemistry and the things we were doing, and be a part of the patent department out at Dow Corning after it got started.

BOHNING: You stayed in Corning during the War. Dow Corning started in Midland about 1943?

HYDE: During 1943 they were already making dimethyl for this oil for the ignition sealing compound at Dow before the Dow Corning facilities were ready. They started a motor program, too, I think before Dow Corning facilities were actually ready. George Grant and later, John Dexter, were involved in that. I think this was the upshot of Rickover's visit to Midland because he was putting pressure on them. This would have to be after the War started. Making this ignition sealing compound would have to be after we had this meeting in Littleton's office. I didn't finish that story. We had learned how to depolymerize, and were just getting into the business of making half-way decent polymers. Dr. Britton, didn't believe this depolymerization story. I said, "All right, I'll show you." During a break at noon, I took him over to our lab, and I put some stuff in the pot, some alkali, and heated it up. And he just stood there with his mouth open. It really struck him right between the eyes. He didn't realize it was possible. Here was a nice batch of cyclics coming over, and all of the stuff you didn't want staying back in the pot. This was at the time when Shailer realized that he could make this ignition sealing compound. That would have to be in 1943. Anyway, that was part of the story. I've forgotten just how soon the buildings were up. Would that be in 1943?

BOHNING: I think by the end of 1943. Because didn't Rickover assist in getting the materials and everything for that building?

HYDE: One of the things that Rickover did: they needed a water tower, a water tank for the power plant. I don't know whether he tore down something else to get it or what but he got it for them. He was pushing the thing, there was no question about it. That motor program, though, was a long, drawn-out thing. Back in the forties, when we were making this stuff, we figured that the people who knew how to make motors would be the ones that would be interested in making a motor varnish. But they kind of shied away from it for some reason or other. When they got going out at Midland, then I began to realize that it isn't just a varnish. It's everything that goes into a motor if you're going to run it at 250°C. And they got life tests going, and they had to develop a lubricant, and they had to develop some design changes. And they had to develop slot sticks, and all of the components that go into a motor. They did this and then they had a long life test. It didn't get all done until after the War, of course. So it didn't help Rickover much. Now, when you see a locomotive go by, one of these diesels, undoubtedly it's got a siliconeinsulated motor and generator in it. So, it paid off, in the long run. It was a long, hard haul, though.

BOHNING: When the group started up at Dow, in Midland, you still stayed at Corning for many years. What were you doing back in Corning when they were working on pilot plants?

HYDE: We were still making polymers, and looking for other ways of polymerization. There's an interesting thing, although it was never used, but phosphoric acid has some interesting possibilities that none of the other acids have. I don't know, I guess we have a patent on it (32). Even in the case of sulfuric acid, if you are not careful it'll clip off methyl groups by I don't know, I hydrolysis. It's the silyl sulfate that is the bond-rearranging catalyst, and because it's bifunctional. It gets tied into the structure, it's going to be in the middle of something. The phosphoric acid is the same way. It's trifunctional. It's interesting that it will not oxidize. Sulfuric is capable of oxidizing at higher temperatures. But the phosphate is three-It does form a gel, when you take it all the way. dimensional. But the interesting thing is that the doggone gel is soluble in toluene. I don't think it was ever used. But the idea was that you could use a polyfunctional material which would replace the phosphate group, and get a crosslinked polymer that would be good for something. One of the things we tried was to put ethyl silicate in there. It's soluble, but at higher temperatures it would tend to interact and would crosslink. There were ways of putting fillers in that would neutralize the phosphoric acid. Calcium phosphate and some of these things probably would be pretty good fillers, as far as inertness is concerned. One of the other things about it, was that you could hydrolyze it, wash out the phosphoric and put silanol groups in place of it.

I think we made some of the first RTV [room temperature vulcanization] products. When the hydroxyl groups diminish in concentration, an engineer might ignore it. But if you've got something that's five thousand units long you probably can't detect the hydroxyl group, but it's still there as a functional group. This was the basis of making some of our RTV systems. The recognition of the fact that the silanol group is there, and you can still do reactions on it. If you have a longer chain polymer, and you react it with monomethyltriacetoxysilane, one acetoxy group will react with the silanol group, and put a diacetoxymethyl end group in there. But those other acetoxy groups are still moisture sensitive, so they will hydrolyze and, as they're polyfunctional they'll give a cross-link. This is the basis for the different kinds of compositions we have now. That was the basis of trying to use the hydroxyl endgroups. Always, I was trying to avoid the probability distribution of these polymer structures where you have an active bond-rearranging system. It bothered me that you couldn't build a more accurate structure. Quite a bit of the work that I continued at Corning was to be able to do this sort of thing. Of course, we did a lot of back and forth. I used to go out almost once a month, and we'd have fairly frequent contacts, talk about problems, discuss what we'd done, and where it might fit into the whole pattern.

BOHNING: Did the three groups meet together?

HYDE: One of the first things that happened was that we gradually reduced the effort at Corning. After the war Dow Corning was getting to the point where they didn't have any That created a problem for them. But as things went customers. on and they began to get good business, Corning thought they ought to be paying their own way, and paying for what was being done at Corning, so we gradually moved things out there. Johannson went out first. He was the one that had some good know-how on the potassium catalysed polymerization. They we They were making it in batch quantities, but they were making also a kind of rubber now, and higher polymers that were more suitable. You've got to have pretty high polymers if you want good working properties on a rubber mill. That's one of the things that we got out of the alkali polymerization. Later on, with Jack Wehrly, one of the first things that we did together was make a continuous polymerization gadget, where we'd feed the catalyst and the raw materials in one end, polymerize at around 150C fast enough so that polymer could be taken out at the other end. When Collings saw this, he started building bigger equipment. He never said anything to me, but I'm sure, because they were using these big Baker-Perkins mixers, large dough mixers. It wasn't long before the engineers were building continuous systems. Potassium was five hundred times faster than sodium, and gave them the speed they needed. It can be done with the sodium catalyst by adding acetonitrile, but that complicates things. Another interesting thing that occurs in this polymerization, because of the mobility of the system, even as it stands, the least soluble component, a short chain such as the dimer salt, starts crystallizing out on standing, and that means that as the salt comes out and two other big molecules are being tied together. That has something to do with polymerization systems. There's no doubt about it.

I've forgotten just the last things that we did at Corning when I realized that we were going to have competition. Way back

at the start in 1940 and before I realized that we could use organic groups to control structure and then I realized that GE was going to be in the business too. By this time I got Johannson, Daudt, Bob [Robert F.] Fleming and Joe Domicone together and we made all kinds of di-, tri- and monosubstituted intermediates. Then we used them with predetermined amounts of mono-substituted, di-, and tri- substituted [monomers] to make specific structures by tying them together by co-hydrolysis. By the time we were through, we had 125 examples. I was making sure that Rochow and his group couldn't walk off with something. That was a good thing because later on that was the way that we made some of our resins and still make some of our resins. I think we would have probably lost much to competition if we hadn't done that. The result of this was a patent application with broad coverage that had to be broken down to a number of patents later on.

[END OF TAPE, SIDE 6]

So the amphoteric character of these intermediates got into the picture before I got the story complete. I was not a very good organizer in saving some of these samples. Now I regret that I didn't pay a little more attention to placing them together in a half-way systematic manner. But then I didn't think that way about it. I was more interested in what's ahead than what's back, I guess.

BOHNING: I wanted to ask you one more thing about the War. What effect did the War have on your group in terms of personnel? Did you have any change-overs, and did that create any problems?

HYDE: One of the things that Johannson and Torok got involved with: when the troops started moving through the jungles, a lot of their radios and communication gear started failing, due to moisture. Somebody wanted water-repellent insulators on submarines antennas. Salt shorts out their aerials. They wanted water-repellent materials, so we started a research project that involved Dr. Johannson and Mr. Torok, which involved treating pyrex rods to see what humidity and salt exposure did to them, and in the process to try to develop a water repelling material for the components in some of these communications instruments. That was another one of the projects that got started in the War. We had about fifteen people working at this time, one of whom worked on lubrication. I wanted to see if we couldn't relate the coefficient of friction to the structure of the molecules of the fluid under study. And I believe you could. We built an inclined plane that you could put a glass or metal plate on, and we had a hemispherical glass or metal riders and we looked at all the disiloxanes that had a phenyl and a methyl group. You could see changes in the coefficient of friction with these structures. I don't know that we ever did anything with it, or if it amounted to anything. But that tilt took you directly into a sliding friction, without overcoming this initial resistance to sliding.

It was a good method, I think, but we never really made good use of it. But we did have people working on this aspect of the problem, hoping that we'd find some unusual lubrication behavior, related to structure.

BOHNING: When the War ended, did that group of fifteen stay that way, or did it change?

HYDE: No, we lost them by various means. Bob Fleming was a lieutenant in the ROTC. I think he used to teach, probably in Laurens, South Carolina, his home town. Whether he taught high school chemistry, I'm not sure. He came up to Cornell to get an advanced degree from the university. He was with us about a year. He wore glasses, and he was color blind. When the War broke out, it wasn't but a week later, he got a notification from the New York District to report. That was on a Saturday, or a Friday, I guess. "I'll be back Monday, they won't take me, with my glasses, and my color blindness." Monday morning I got a call from him. He said, "I'm in the Amphibian Command at Fort Edwards." (That's up in Massachusetts somewhere.) We lost him for the duration of the War. Joe Domicone got drafted. He had a bachelor's degree, with a major in chemistry. I don't know what route he followed, but he came out a captain. He was involved in the Battle of the Bulge. He managed to get through. He stayed the Battle of the Bulge. He managed to get through. He stayed at Corning and he didn't go out to Midland with us. So he was their source of silicone information. When somebody around Corning needed some information, they went to Joe. So we didn't see him again until after the War. Johannson moved out to Midland because they were beginning to produce some rubber compositions. Johannson knew a lot about this polymerization business. He went out to Midland first. I've forgotten what happened with Torok. These people had other interests at Corning. In the patent department, we had both Blackburn and Dr. Lee Fowler. Dr. Fowler had his Ph.D. in organic chemistry, too, before he went to law school.

BOHNING: When was the decision made to move your group to Midland?

HYDE: It was really piecemeal. I think that Dow Corning was paying my salary for quite a while before I moved out there. Johannson went out. Then, about a year or two later, Dr. Daudt went out. Bob Fleming came back, after the War, he wasn't with me very long before he left. Bob was a very meticulous person. He had a very good mind. These guys out at Midland grabbed him for the patent department. Bob spent most of his time, since he got back from the War, in the patent department out there. Actually, he was in the Pacific area. He was one of the engineers that went in and made a good landing-place for the rest of the Army. BOHNING: Did your group then, get smaller, as these people went?

HYDE: Oh, yes. I've forgotten now what I was left with. Maybe three to four people. As soon as the War was over they started moving things to Midland. I didn't have to go to Midland, but I could see so many interesting things that still needed to be done, that I couldn't resist the temptation. All of the raw materials, and the things you need for research, were there.

I get back to Corning once in a while. They like to have me come in. I go down and they pay a consulting fee. It keeps me from going completely stale. I get down there and try to help them with problems. I don't know whether I do or not. With optical fibers, I think Dow Corning came up with a good coating for it, but it may already be be obsolete because of rapid developments in this areas. You need a coating, and it has to be fast curing. The number of patents and publications resulting from the initiation and growth of the silicone industry must be up in the thousands. Fiber optics is one of their big items at Corning.

BOHNING: It's an excellent idea.

HYDE: This is another one of these Gaffers [monthly in-house publication of Corning Glass Works]. [Hyde refers to a copy of <u>The Gaffer</u>] These are some of the spools. How they can transmit a laser beam a hundred miles and get anything off the other end, I don't know.

BOHNING: We were talking about your move to Midland. I was curious, you sort of indicated that you didn't have to go to Midland, that you could have stayed at Corning.

HYDE: That was a decision that I had to make. I think that Dr. Sullivan liked to have me stay, but I don't know. I saw so much that was interesting to me that I just about had to go.

BOHNING: I guess Warrick had already gone?

HYDE: Yes, just before. I'm not sure just what Mac and Earl did, I don't think they got involved in research. I knew Mac before, I knew Mac at Illinois.

BOHNING: Oh, you did!

HYDE: I didn't know him real well, but he got his degree working

for Professor [George D.] Beal. Beal and [Silas A.] Braley of the analytical department in Illinois both went to the Mellon Institute, after Mellon built the new building, and McGregor went with them. I'm sure he was on some other fellowship, at the time. Beal's main interest was in toxicology, I think, but whether or not that was Mac's area, I'm not sure. I don't know that much about it. Mac got his doctorate, I think two years before I did in 1926. I knew him personally, but I didn't really know too much about what he was working on.

BOHNING: When you got to Midland in 1951, what did you do then? Were you in charge of the group?

HYDE: I was a little disappointed that they hadn't really gotten the building finished, where they were going to have a research lab that I could take over. I had a little trouble getting organized and started again. Then I got acquainted with Dr. [Arthur] Barry, Dr. [Melvin] Hunter, and the other people at Midland. It was good. I think I probably spent quite a bit of time trying to plan what I wanted to do. It was a kind of a disruption. And they didn't have the place ready. Trying to work with other people's tools, and in other peoples' areas, is not an easy thing to do. So I probably lost a little time. I sometimes think back now, and think of a lot of interesting things that I could have done, and didn't. Research is that way, of course. They started building up the group there, as soon as they had the building where we could start some work. The dates confuse me a little bit.

Paul Brown came to work with me pretty early in the game, and we had quite a few patents together. One of the things that we were looking at was hydrolysis behavior. He actually had started in the analytical laboratory there. Then he came over to work with me. I had this idea of continuous polymerization. Ι had a chance to hire an engineer named Jack Wehrly. He came to work with me, and we built this continuous polymerizer. We made pilot plant quantities of polymer. Then, we let it go and pilot plant people took over the project. It seemed to me that a lot of our products were going into emulsions and that we ought to make a polymer emulsion. We used a distearyldimethyl ammonium chloride. It's a pretty good emulsifier for the starting material and for the siloxane intermediates. The quaternary bases are very strong bases, like sodium and potassium hydroxide. So what we did, and Jack Wehrly worked with me on this, we made an ammoniacal solution which generated enough of the free quaternary base to form the siloxane salt in the siloxane phase, and it worked beautifully. They still make many emulsions that way.

Dr. Donald Weyenberg and his group were coming along. I can't tell you all about the work that they did, but they were working in the direction of making room temperature curing systems. His group came along with an anionic emulsion. We were using a cationic emulsifier. They used laurylbenzenesulfonic acid, or one of its salts, as an anionic emulsifier and, on the alkaline or acid side, it would give a nice emulsion polymer. So we had it on both sides. Usually, with an anionic emulsifier, if you tried to use it in a cationic system, they'll clobber each other, and vice versa. So there's good use for having both of those materials. When the War ended, the only product we had was DC-4 sealing compound and Uncle Sam was the only customer. Charlie [Charles E.] Sanford, one of the sales people, knew that the rubber industry produced quite a lot of rejects. It only takes one or two percent of rejects, and because of the size of that business, you've got a lot of wasted tires. He knew about this problem, and we already knew that silicones were lubricants that did not swell rubber. Compatibility is another one of the unique and important properties of some of these things. Anyhow he sold them the idea of treating the molds with an emulsion, and that turned into something good. I think Charlie Sanford is the one that got it underway. He and Olin Blessing were the two mainstays of our sales department.

BOHNING: Were you talking to anybody outside of the company?

HYDE: No.

BOHNING: You didn't have any external consultants?

HYDE: We supported a fellowship at Penn State University under Dr. Leo Sommer, but I don't consider this a consulting basis. No, I felt that I'm part of the company, you know. There's nobody else that has anything to do with it.

Another thing that we did at Midland, was study this equilibrium hydrolysis. That turned out very well. It emphasized the equilibrium nature and the amphoteric character [of silicon] and the change with electronic character as you change the groups. You could take the hexamethyldisiloxane, and the hexaethyldisiloxane, and the equilibrium constant is different for the two. Just because you change the groups. Tn the presence of a drying agent, gaseous HCl will convert them quantitatively back to the chlorosilane. Then Paul Brown and I studied the equilibrium hydrolysis of the dimethyldichlorosilane. In the case of the dimethyl hydrolyzates, there was always trouble with endgroups, trying to get rid of the last traces of chlorine. We didn't understand why. Even with extensive washing, their presence in some of the products of intermediate viscosity adversely effected their electrical properties, for example. We showed that if you put aqueous concentrated HCl in with dimethylsiloxane, it wouldn't matter what polymer you started with. If you put the system under high pressure, and put the HCl pressure up to a couple hundred pounds, you can reverse the hydrolysis and take it all the way back to practically all dimethyldichlorosilane and dimer.

[END OF TAPE, SIDE 7]

HYDE: Paul Brown and I accomplished that at Midland.

BOHNING: You stayed in research your entire career at Midland. Did you ever have the opportunity to move away from research?

HYDE: I never looked for it. And I don't know whether they were looking for it either. I was considered a senior scientist out This was satisfactory to me. There is this problem that there. people in research take longer to develop things, and to see their accomplishments. Many people that get into research see their colleagues probably making more money, and all of this gets into the picture. Maybe I could have gone into management; I've talked with some of the psychologists that they bring in to analyze you. I've never really thought too much of this business. I think it was really my preference to stick to It looked like something too far away, I mean research. something that wasn't definite. The guts of the thing, and the thing that really counts, are the things that are being done in the laboratory. I think that was part of my attitude. It never Many people that came in to work with me, wanted to bothered me. get out of research as soon as possible and get into production or sales because they figured that's where the money was. But I think it's different now. Companies gradually built a scientific, a more academically-oriented, career pathway. Many times you would think about "senior scientists" that's just a kind of a nicety, to give people titles that they like. But it's more than that, it is a recognition of some of the skills and abilities that go into the technical aspects of the industry abilities that go into the technical aspects of the industry. People are just as dedicated, whether they're selling, or whether they're in the accounting department. Many of the research people are just as dedicated and essential to the progress of a company and its growth, as anybody else. If I got interested in something, I wanted to do it myself. I wanted to find out first hand what was cooking. So I always had a little trouble that You can't compete with people that are working for, or way. with, you. It takes a lot of people in all these areas to make a success out of a company like Dow Corning.

BOHNING: How would you contrast the two companies, Corning and Dow Corning, in terms of companies to work for?

HYDE: In a way, they have a similar background, as far as company organization and build-up is concerned. Dow was a privately-owned company for a while, and so was Corning. Corning was private up to about 1938 or so. Then they went to the stock market because they needed more money for expansion. In many ways, it was the Dow family, and here it was the Houghton family in the glass business. But they also had outside managers coming in, like Dr. Sullivan, trained scientists. Dr. Sullivan, was a physicist, or a physical chemist. For guite a while he was the

editor-in-chief of the Chemical & Engineering News. Incidentally, his father was feature news editor for the Chicago Tribune. Sullivan got the Perkin Medal in 1929, I think, for his work in developing Pyrex glass. He was a gentleman, through and through. He appreciated research work, and he knew that everything wasn't going to be a ringer. He had patience. There were times when I thought, "I'm not doing myself, or Corning, either, much good." You know, some things go backwards a little bit, and you begin to feel, "Am I doing something useful?" But Dr. Sullivan was always an encouragement. You could talk with him, and you'd go over to his office, and talk a little while with him, and you'd come out feeling pretty good. He was that kind of a person. He never talked very much, but what he said was usually something worth listening to.

BOHNING: Sounds like an ideal person to work for.

HYDE: I never knew too much about him, but I think Dr. Sullivan went to the University of Michigan, before he went to Europe. He went to the University of Leipzig to receive his doctorate.

BOHNING: When you were at Dow Corning, did you know Britton at all? He was over at Dow.

HYDE: He was over at Dow. Of course, I had met him when he came down to Corning. Then I'd visited his laboratory on several occasions, met some of the other people that got involved in some of the organosilicon work out there. But I knew of him earlier than that because in 1932, at an ACS meeting that was held at Cornell University, he gave a paper involving a preparation of phenol from chlorobenzene. That's one of the things that you used to get marked off your paper. That was too simple.

BOHNING: I think we've covered an enormous amount of material. Is there anything else in your notes that you might like to share?

HYDE: Here's one of the other aspects of the research that we were doing. Paul Brown was involved in this. The question was to try and synthesize structures with greater precision. This inherent flexibility of siloxane bond-rearrangment with strongly basic or acid catalysts that shuffles everything was not the way to make some kinds of materials. This is another very interesting thing that they've never capitalized on. The cyclic tetramer of dimethyl is one of the most stable siloxanes when compared to a linear chain molecule. The cyclic trimer is relatively very reactive, and you can treat the cyclic trimer with a much milder catalyst, that is relatively non bondrearranging, and polymerize to form mono-disperse siloxane polymer chains. Another thing that we learned to do was to react the cyclotrisiloxane with another chlorosilane, such as methyl trichlorosilane under relatively non-bond rearranging conditions, and make special chlorosiloxane structures. Because of decreasing reactivity, the chlorine groups can be reacted consecutively to form new species.

We still hope that we'll make better materials. I think that already some of this is getting into the works. **Research** is slow; getting into a useful area is a slow process. Once the process is set up, and the production people become accustomed to running it, they begin to turn out a lot of results but they don't want anybody messing it up for them. So it's not easy for them to accept process and product improvements. These are longrange things, that happen gradually. When they get into trouble, you can help them. This is one of the jobs of research, I think. This bond-rearranging is an interesting aspect of making polymers from siloxane systems. Every one; the dimethyl polymer, if you take it to an equilibrium condition where all the bond rearranging is complete, you have about 15% of cyclics in there. If it's diethyl, you'll have more like 30%. If it's the phenylmethyl, a different percent of cyclic. This is one of the interesting things that Dr. Johannson straightened out. With the trifluoropropylmethyl, it polymerizes so fast, and de-polymerizes again so fast, that they never knew they had a polymer in Now they make good polymers with what they call their between. LS intermediates. I don't know whether they still make use of I would guess that they do. So this difference in the that. amount of cyclics tells us something about the effect that the various groups have on the silicon. If you want to make a diphenyl polymer, I'm not sure they still know how to do it. Probably, at equilibrium, if you didn't use some dodge or some hydroxyl condensation system, you would probably have 95% cyclics and 5% polymer. That's one of the things that we're after with this non-rearranging system: to develop mild condensation catalysts, so that they'd tie them together. Not only that, but then you could put different siloxanes in there and make block polymers, with a non bond-rearranging system. This was one of things that we had in mind with the research that we were doing towards the end of my career, if you want to call it that. Ι worked until I was seventy two. I don't know whether they were wanting to get rid of me or not.

BOHNING: It doesn't sound like it, if they kept you on that long.

HYDE: Well, it was 1972, and I was sixty-nine when we moved to Florida. I was really working part-time to age seventy-two, if you want to put it that way. Now, it's a little harder to find people doing exploratory or basic research. A lot of these very competent people are tied up in different product areas. Of course, there's nothing wrong with that. These guys have the insight and know-how. But it doesn't help over the horizon. There needs to be more. There's nothing wrong with that, in a

way, either, because sometimes a customer finds out what's wrong with your product, and that's a good source of learning something new about it. I'm not criticizing, from the point of view of doing that, but there needs to be more people that are not tied down. Nobody tried to say, "You're off in the wrong direction, let's go this way." And usually, I think, we were turning out in our lab useful things, which was one of the things in the uppermost in my mind. I was curious about things, so when I didn't understand them, that was a good start for research, too. That was part of the game. I don't know whether I could jot them down, but I think this place was still open for new, interesting things, and new discoveries, and possibly new uses for new materials. The silicones are already in uses that we never dreamed of back in those early days. I think that every one of these Xerox machines has got some silicone insulation in it. Ι don't know just how it's used. It's gotten into so many places, now one of the big applications may turn out to be the replacement of the chlorinated biphenyls that are hard to get rid of. Silicones are getting in there and replacing immersion fluids for transformers, and things like that. It looks like there's no end to it. I'd like to think that we've added another dimension to silica's usefulness as well as silicon itself, in a It's the basis for all the mineral silicates. It's the way. basis for all the glasses, not all of them, but the majority of And the element itself is the basis of semiconductors. them. And here we've added another dimension to the usefulness of silicon in the form of silicones. That, more than ever, puts it in the middle of the periodic chart, as far as I'm concerned. I drew these lines; a lot of people objected to those lines that I drew through the center (33). [Hyde points to his version of the periodic table]. They are there to help emphasize that there's the two related sub-groups, and also color, that helps relate the two sub-groups A and B, as shown by Mendeleev's original table. I thought we ought to leave them in there. And so they did. They've sold something like over ten thousand of them. They are sold for three dollars a piece, I think that's about what it costs to get them printed.

BOHNING: Are they sold through Dow Corning?

HYDE: Yes, as far as I know, Dow Corning is the only one that sells them.

[Hyde quotes from document (34)]

"The continuing research program was directed toward developing non bond-rearranging synthetic methods for more precise structure building, offering the possibility of new products not otherwise available." This work involved Terry Swihart, Dr. James Hampton, as well as others. "In such an enterprise, the individual contributions are so intertwined that separation of individual contributions are impossible." That's true, you're working closely together with people. "Along with recognition by Corning and Dow of the importance of research, the dedicated effort of

many people along the way has brought success and growth to Dow Corning. This includes all phases essential to the successful operation of a chemical company. The computer and many new analytical tools are marvels to behold, and have advanced not only our research capabilities, but our technology and manufacturing capabilities enormously, as well. Without them, Dow Corning would have difficulty operating at all." This is true, I think. "The shortest path to new and better products is an insight into the basic principles in the background of a project. Well-directed research is the life insurance of an industry. Scientific facts never get obsolete. Only our misconceptions do, in the light of new knowledge." Molecules always do what they're supposed to, under a given set of conditions. "To gain new knowledge and to fill the blank spots in the understanding of ourselves and our environment, and to make it useful, are worthy goals. For the person who wants to explore and discover, the field of chemistry is wide open. Along with minerals, glass, and semi-conductors, the silicones have added another dimension to the usefulness of the silicon atom. At the present time, patents and publications number in the thousands, and there are many interesting and useful things still to be done." They had one international silicone symposium at Midland, I've forgotten the date of it now. But I think the last one was at GE. And there was about two hundred people or more there, for that symposium, from all over the world. It's really gotten pretty big.

BOHNING: It's come a long way.

HYDE: Never would have dreamed that some of those things would happen.

BOHNING: When you first read Kipping's papers.

HYDE: That's right. I still think, "Gosh, there's a lot of things that I should have gotten done that I never did."

BOHNING: But you have an enormous amount of work to your credit, with your papers and your patents.

HYDE: Well, it was a lot of fun, but there were a lot people involved, and a lot of hard work by a lot of people, too. That motor program was a tough one. Grant and Dexter wound up with traction motors that were running at 250°C for I don't know how long. There isn't any question that in the long run it added something very useful. So it wasn't in vain.

BOHNING: No, absolutely not. Is there anything else that you think we should cover at this point?

HYDE: Well, you know McGregor, down at Hemlock, wrote a book on biomedical and medical applications (35). That was pretty much up McGregor's alley. He spent quite a bit of time in that area. They used to test materials. Dow was big at this kind of thing. They don't want to hurt people, they don't want to put products out that are going to cause trouble. Dow spends, what, a hundred fifty million dollars a year checking and testing products? Dow Corning got into some of this, and they found that a little bit of the crude phenylmethyl, in the bottom of a spray can, one of these pressure spray cans, would keep them from rusting. But they got into a little bit of a jam. It turned out when they did rabbit tests, that there was some biological activity to this stuff. What it did was have a similar effect to estrogen. I quess it was a mild thing. But they started digging into it, and there was everything but the kitchen sink in this residue. So they started trying to separate it. Without GLC [gas-liquid chromatography], which spreads these things out, it would have been an impossible thing. They had some crystalline residues, but they were still pretty complex mixtures. We started looking through some of them. Some of the fractions began to show some activity, but the other ones didn't. It began to limit things a little bit. Finally, one of them crystallized. There were a lot of people involved in this, it wasn't just

There were a lot of people involved in this, it wasn't just us in our laboratory. Apparently these crystal materials were very pure, and it looked like it would be a mixed cyclic with two phenyl-methyls and two di-methyl groups in it, in the para position. And it turned out that the one that had shown some biological activity was the cis [isomer]. The trans didn't show it. Finally we got these things out, and then we synthesized some of them in our laboratory. They'd set up this biolaboratory in Midland. Dr. Bennet was the first manager of that place. Then they set up animal tests. This one particular compound was a 2,4-cis diphenyl cyclic siloxane compound. The rest were methyl groups. Finally, they got around to the point of testing it in Sweden, on terminally ill people with prostate cancer. And what it did, apparently, was it didn't cure anything, but it relieved some of their pain and suffering. So these people could go home. Somehow or other, the synthesis was too complex, and they tried to get something approved by FDA. I think it got too expensive to try to synthesize it, for the benefit that could be derived from it. So they quit doing it. I wasn't involved, except for that part of our group which managed to isolate and synthesize some of the material. It was not a good commercial synthesis, let's put it that way.

[END OF TAPE, SIDE 8]

BOHNING: I really don't have any more questions; we've covered all of my notes.

HYDE: Off-hand, I can't think of other things. You've covered it pretty well, I think. There may be some spots in here. Oh, this business of establishing the melt viscosity curve there. [Hyde refers to fig. 4 of the Whitehead Memorial Lecture (36)] Dr. Leonard Bruner came to work in our group. I don't know whether he was making polymers, but what he did was to convert the hydroxy end groups to acetoxy groups. That meant you could titrate the end groups. Dr. Brunner is the one that first came up with this curve here, this bottom curve. That was the basis, looking at these hydroxy groups and the acetoxy groups, that was part of the development of the improved RTV's, room temperature curing systems. A lot of other polyfunctional cross-linking components are used now in a more precise synthesis. These monodispersed polymers would give us, maybe, some new properties. I don't think it's ever been done.

BOHNING: You have all these ideas of things to be done, and you said you do go back to Corning. Do you still go back to Midland at all? Do you talk with people there?

HYDE: Once in a while. And I'll go around through the labs to see what's cooking, and if I happen to see something, if I have a good suggestion, then I make it. They'll pay my way if I come back, which is alright. Of course, since Mrs. Hyde had this trouble, it's a little too hard to make the trip. We do go up to Keuka Lake, near Corning, where we have a small cottage. [Mrs. Hyde died in September 1989: ed.] That gives me a chance to get down to the Corning research laboratories. So I try to help them out wherever I can. I'd like to get back to Midland and Dow Corning more often, that's where my major interests are.

BOHNING: I'd like to thank you very, very much for your detailed responses.

HYDE: Talking about these things, it just brings back memories. You just like to talk about them, that's all. There's probably other things that I'll remember next week, details that might be of interest, or that might help you.

BOHNING: I appreciate all of the responses that we have, and thank you again.

HYDE: Well, see, it's my pleasure. I've enjoyed this very much. [END OF TAPE, SIDE 9]

NOTES

- 1. W. H. Perkin and F. S. Kipping, <u>Organic Chemistry</u>, revised edition (London, W. & R. Charles, 1923).
- 2. G. N. Lewis and M. Randall, <u>Thermodynamics</u> <u>and</u> <u>the</u> <u>Free</u> <u>Energy of Chemical Substances</u> (New York: McGraw-Hill, 1923).
- 3. Roger Adams and J. Franklin Hyde, "Cyclohexyl-Alkyl-Phthalic Esters," U.S. Patent 1,689,761, issued 30 October 1928 (application filed 11 June 1926). <u>idem.</u>, "Organic Solvent," U.S. Patent 1,689,762, issued 30 October 1928 (application filed 8 December 1926).
- 4. F. Bell and J. Kenyon, "The Space Formula of Diphenyl," <u>Chemistry and Industry</u>, 45 (1926): 864-865.
- 5. J. F. Hyde and R. Adams, "Possible Isomers of Certain Analogs of Resolvable Diphenyl Compounds," <u>Journal of the</u> <u>American Chemical Society</u>, 50 (1928): 2499-2506.
- 6. R. Willstätter and A. Stoll, <u>Investigations</u> on <u>Chlorophyll</u>. <u>Methods</u> and <u>Results</u>, English translation by F. M. Schertz and A. R. Merz. (Washington DC: Schertz, 1928).
- 7. J. B. Conant and J. F. Hyde, "Relationship of Chlorophyll to the Porphyrins," <u>Science</u>, 70 (1929): 149. <u>idem.</u>, "Chlorophyll Series. I. Thermal Decomposition of the Magnesium-Free Compounds," <u>Journal of the American</u> <u>Chemical Society</u>, 51 (1929): 3668-3674. Conant, Hyde, W. W. Moyer and E. M. Dietz, "Chlorophyll Series. II. The Degradation of Chlorophyll and Allomerized Chlorophyll to Simple Chlorines," <u>ibid.</u> 53 (1931): 359-373.
- 8. J. F. Hyde and H. W. Scherp, "Apparatus for Micro-Catalytic Hydrogenation," <u>Journal of the American Chemical Society</u>, 52 (1930): 3359-3363.
- 9. Fritz Pollak, "Urea-Aldehyde Condensation Products," British Patent 248,729, issued 3 March 1925.
- 10. J. Franklin Hyde, "Electrically Conducting Coating on Vitreous Substances and Method of Producing It," U.S. Patent 1,964,322, issued 26 June 1934 (application filed 7 November 1930).
- 11. J. Franklin Hyde, "Joining or Cementing Glass Building Units," U. S. Patent 2,052,229, issued 25 August 1936 (application filed 21 May 1934). <u>idem.</u>, "Joining Glass Blocks," U. S. Patent 2,112,241, issued 29 March 1938 (application filed 22 April 1937).
- 12. J. Franklin Hyde, "Method of Making a Transparent Article

of Silica," U. S. Patent 2,272,342, issued 10 February 1942 (application filed 27 August 1934).

- 13. J. Franklin Hyde, "Method of Bonding Fiber Glass with Carbon," U.S. Patent 2,225,009, issued 17 December 1940 (application filed 25 August 1937).
- 14. F. S. Kipping, "Organic Derivatives of Silicon," <u>Proceedings of the Royal Society</u>, 159 (1937): 139-148. Kipping concludes his Bakerian Lecture with the following: "We have considered all the known types of organic derivatives of silicon and we see how few is their number in comparison with the purely organic compounds. Since the few which are known are very limited in their reactions, the prospect of any immediate and important advance in this section of chemistry does not seem hopeful."
- 15. K. A. Andrianov, "Synthesis of Alkyl-Substituted Ortho-Esters of Silicic Acid," <u>Journal of General Chemistry</u>, <u>USSR</u>, 8 (1938): 552-556.
- 16. F. Wöhler, Leibig's Annalen, 127 [51] (1863): 263.
- 17. A. Ladenberg, <u>Leibig's Annalen</u>, 164 (1872): 300.
 C. Friedel and J. M. Crafts, <u>Annales de Chimie et de Physique</u>, [iv.] 9 (1866): 5.
- 18. W. Dilthey, <u>Berichte</u>, 38 (1905): 4132.
- 19. F. C. Whitmore, L. H. Sommer, P. A. DiGiorgio, W. A. Strong, R. E. Van Strien, D. L. Bailey, H. K. Hall, E. W. Pietrusza and G. T. Kerr, "Organosilicon Compounds. I. Synthesis and Properties of Alkyltrimethyl- and Alkyltriethylsilanes," <u>Journal of the American Chemical Society</u>, 68 (1946): 475-481.
- 20. J. F. Hyde and R. C. DeLong, "Condensation Products of the Organo-Silane Diols," <u>Journal of the American Chemical</u> <u>Society</u>, 63 (1941): 1194-1196.
- 21. F. S. Kipping, "Condensation Products of the Organo-Silane Diols," <u>Chemistry and Industry</u>, (1945): 168.
- 22. E. G. Rochow and W. F. Gilliam, "Polymeric Methylsilicon Oxides," <u>Journal of the American Chemical Society</u>, 63 (1941): 798-800.
- 23. J. F. Hyde, L. K. Frevel, H. S. Nutting, P. S. Petrie and M. A. Purcell, "Cyclo-Diphenylsiloxanes," <u>Journal</u> of the <u>American Chemical</u> <u>Society</u>, 69 (1947): 488-492.
- 24. J. F. Hyde, "The Condensation Behavior of Diphenyl Silanediol," <u>Chemistry</u> and <u>Industry</u>, (1945): 270.
- 25. J. Franklin Hyde, "Methyl Silicon Halides and their

Production," U.S. Patent 2,413,049, issued 24 December 1946 (application filed 3 July 1942).

- 26. Eugene G. Rochow, "Polymeric Methyl Silicone," U.S. Patents 2,258,218-2,258,222, issued 7 October 1941.
- 27. J. Franklin Hyde, "Organo-Silicon Polymers and Method of Making Them," U.S. Patent 2,371,050, issued 30 March 1943 (application filed 19 August 1940).
- 28. Fulton B. Flick, "Oxide Coating on Aluminum," U.S. Patent 1,526,127, issued 10 February 1925.
- 29. A. Stock, "Silicon Hydrides," <u>Zeitschrift für Elektrochemie</u>, 32 (1926): 341-349. In this paper Stock reviews his earlier synthetic work and compares the physical constants of fifteen organo-silicon compounds with the corresponding carbon compounds.
- 30. D. L. Yates, <u>William R. Collings.</u> <u>Dow</u> <u>Corning's</u> <u>Pioneer</u> <u>Leader</u> (Midland, Michigan: Dow Corning, 1985).
- 31. J. F. Hyde, O. K. Johannson, W. H. Daudt, R. F. Fleming, H. B. Laudenslager and M. P. Roche, "Sodium and Potassium Salts of Triorganosilanols," <u>Journal of the American</u> <u>Chemical Society</u>, 75 (1953): 5615-5618.
- 32. J. Franklin Hyde, "Siloxane Elastomers Comprising a Modified Acid Polymer and Filler," U.S. Patent 2,571,039, issued 9 October 1951 (application filed 12 April 1950).
- 33. J. F. Hyde, "A Newly Arranged Periodic Chart," <u>Chemistry</u> 49 (7) (1976): 15-18.
- 34. J. F. Hyde, Personal Memoir. BCHOC Research File 0026
- 35. R. R. McGregor, <u>Silicones</u> in <u>Medicine</u> and <u>Surgery</u> (Midland, Michigan: Dow Corning, 1957).
- 36. J. F. Hyde, "Engineering Tools from Organosilicon Chemistry," Conference on Electrical Insulation and Dielectric Phenomena, Washington, D.C. (1971): 273-280.

INDEX

Ά Adams, Roger, 5-11, 13, 15, 48 Alfred University, 15 Alpha Chi Sigma fraternity, 5, 6 Aluminum Company of America, 27 Andrianov, Kusma A., 22, 49 Arvin, James A., 8, 9 Asparagine, 4 ASTM [American Society for Testing and Materials], 18 В Bakelite, 21 Baker, Ross A., 3 Baker Lectures [Cornell University], 28 Barry, Arthur, 39 Bass, Shailer L., 30 Beal, George D., 39 Bell, Frank, 8, 48 Bennet -- 46 Bennet, --, 46 Bennett, James [grandfather], 1 Berzelius, Jöns, 3 Bethlehem Steel Corporation, 11 Blackburn, William C., 33, 37 Blessing, Olin D., 40 Boehner, Reginald, 4 Bond-rearrangment, 35, 42 Boylston Hall [Harvard University], 12 Braley, Silas A., 39 Bridgman, Percy W., 14 Britton, Edgar C., 29, 30, 33, 42 Brown, Paul L., 39-42 Browning, E., 8, 9 Bruner, Leonard B., 47 Cambridge, Massachusetts, 11, 14 Carothers, Wallace H., 9, 15, 16 Chaulmoogra oil, 8 Childs, Lucy [grandmother], 1 Chlorophyll, 12, 13, 48 Chlorosilanes, 26, 27, 30, 31, 40, 43 Coefficient of friction, 36 Colgate University, 5 Collings, William R., 30, 35, 50 Conant, James B., 11, 12, 14, 15, 48 Continuous polymerization, 35, 39 Coolidge, William D., 26 Cornell University, 28, 31, 42 Corning, New York, 16, 17, 28, 34, 38 Corning Glass Works, 15-17, 19-21, 23-28, 34, 35, 37, 38, 41, 44 Daudt, William H., 27, 31, 36, 37, 50

Davies, Letha A., 9 DeLong, Richard C., 22, 26, 29, 31, 49 Depolymerization, 33, 43 Dexter, Jihn, 33, 45 Diazomethane, 9 Dilthey, Walter, 22, 49 Dimethyl siloxane, 30 Dingman, Harry, 27 Domicone, Joseoh, 36, 37 Dow Corning Corporation, 5, 23, 28-30, 33-35, 37, 38, 41, 42, 44-46 du Pont de Nemours & Co., E. I., Inc., 9-11, 15, 16 Duval, Claude, 2 Е Emulsifier, 39, 40 Ephedrine, 8, 9 Equilibrium hydrolysis, of siloxanes, 40 Ether, 28, 29 F Family, brother [Solon W. Hyde], 1 [Burton D. Hyde], 1 father grandfather [James Bennett], 1 grandmother [Lucy Childs], 1 grandmother [Lydia A. Martin], 1 mother [Amelia Bennett Hyde], 1 sister [Helen Hyde Darby], 1 wife [Hildegard Lesche Hyde], 11, 14, 47 Fiber optics, 38 Fiberglass products, 20, 21, 24 Fieser, Louis F., 31 Fischer, Emil, 4 Fischer, Hans, 12 Fleming, Robert F., 31, 36, 37, 50 Flick, Fulton B., 27, 50 Fowler, Lee, 27, 37 Fowler, Lee, 27, Frevel, Ludo K., 23 Friedel, Charles, 22, 49 The Gaffer, 38 Gelation, 25, 32 General Electric Company [GE], 21, 23, 24, 26, 27, 36 Gilman, Henry, 27 Gomberg, Moses, 6 Grant, George, 33, 45 Greene, Charles H., 31 Grignard reagent, 4, 22, 25, 29-31 H Hampton, James, 44 Harvard University, 12, 15, 31 Hopkins, B. Smith, 6

Illinium, 6 Illinois, University of, 5, 6, 10, 12, 38, 39 Italium, 6

J

Johannson, O. Kenneth, 27, 31-33, 35-37, 43, 50 Johnson, John R., 9 Johnson & Johnson, 10

K

Kaufler formula, diphenyl derivatives, 8 Kenyon, J., 8, 48 Kinetics, of polymerization, 33 King, Aden J., 5 Kipping, Frederick S., 4, 15, 18, 22, 23, 45, 48, 49 Kohler, Elmer P., 13 Kremers, Harry C., 7

L

Ladenberg, Albert, 22, 49 Lamb, Arthur B., 22 Leipzig, University of, 42 Lesche, Hildegard [wife], 11, 47 Lewis, Gilbert N., 7, 48 Littleton, Jesse T., 30, 31, 33 Lubrication, 20, 30, 36, 37, 40 Lycan, William H., 10

Houghton , Amory and family, 41

Hunter, Melvin, 39 Hydrogenation, 48

Μ

Macbeth-Evan Company, 25 Mallinckrodt Chemical Company, 12 Marshall, Abraham L., 24, 28 Marvel, Carl S., 9, 10, 15 Marver, Carr S., 9, 10, 15 McCroskey, --, 3, 5 McGregor, Rob Roy, 22, 25, 26, 30, 38, 39, 46, 50 Mellon Institute, 25, 30, 39 Melt viscosity, 47 Mendeleev, Dmitri, 44 Michigan, University of, 6, 42 Michigan State University, 6 Microbydrogenation 12 Microhydrogenation, 12 Midland, Michigan, 38 Mine Safety Appliance Company, 30 Monophenylchlorosilane, 31 Morrow, Leslie, 24 Moyer, Wendell W., 8, 13 Moyle, Clarence B., 29, 30

N

Noyes, William A., 6

Optical fibers, 38 Organosilicon chemistry, 15, 17, 18, 20-22, 24, 25, 27, 28, 42, 49 Owens-Corning Fiberglass Corporation, 21, 24, 26, 29 Owens-Illinois, Glass Company, 20, 21, 24 Ρ Patents, 8, 26, 27, 36, 45, 48 Patnode, Winton I., 24, 28 Pauling, Linus C., 28 Periodic table, 44, 50 Porkin William W. 4 Perkin, William H., 4, 48 Peterson, Floyd C., 5 Peterson, Wesley R., 14 Peterson, Willard D., 14 Phenol-formaldehyde resins, 18, 21 Pittsburgh Corning Corporation, 25 Pittsburgh Plate Glass Company, 25 Pollak, Fritz, 15, 48 Pollopas, 15 Polymerization, 26, 30, 32, 34, 35, 42 continuous, 35, 39 Polymorphism, 23 Polysiloxanes, 32, 42, 43 Polyvinylacetate [PVAc], 18, 19, 25 Porphyrins, 12, 48 Promethium, 6 Pyrex glass, 15, 17, 19, 42 Quill, Lawrence L., 6 R Radcliffe College, 15 Randall, Merle, 7, 48 Ray, Edward, 11 RCA Building [New York city], 19 Resins, thermally stable, 23 Rickover, Admiral Hyman G., 26, 29, 30, 33, 34 Rochester, New York, 18 Rochester, University of, 13 Rochow, Eugene G., 22, 24, 26-28, 36, 49, 50 Rodebush, Worth H., 7 RTV [room temperature vulcanization], 34, 47 Rubber, natural, 40 Rubbers, silicone, 33 S Salzberg, Paul L., 9 Sanford, Charles E., 40 Scherp, Howard W., 13, 48 Sigma Xi, 5 Silica soot, 20

Silicon, chemistry of, 3 Silicon tetrachloride, 17, 18, 20, 26 Silicones, 22, 26, 31, 38, 44, 45 Siloxane, 24, 25, 30, 32, 39, 40, 42, 43 Smith, 'Cookie-Cutter', 6 Solvay, New York, 1, 2 Solvay Process Company, 1, 2 Sommer, Leo H., 22, 40, 49 Stanley, Wendell M., 7 Stereochemistry, 22 Stilbene, 10 Stock, Alfred, 28, 50 Stoll, Arthur, 12, 48 Sullivan, Eugene C., 15, 24, 26, 28-30, 38, 41, 42 Swihart, Terrence, 44 Syracuse, University of, 3-7 Taylor, William C., 15 Torok, Julius, 31, 36, 37 TT Union Carbide Company, 19 Urea-formaldehyde resins, 18, 48 V Varnish, 21, 32 Viscosity/temperature relationship, 30 W Warrick, Earl L., 22, 25, 30, 38 Wehrly, Jack, 35, 39 Westinghouse Electrical Corporation, 21, 23-25, 32 Wetting properties, 31 Weyenberg, Donald, 39 Willstätter, Richard, 12, 48 Wilmington, Delaware, 10 Wisconsin, University of, 6 Wöhler, Friedrich, 22, 49

Y

Yohe, G. Robert, 6