

REPORT DOCUMENTATION PAGE

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6c. ADDRESS (City, State, and ZIP Code) Department of Chemistry Los Angeles, CA 90089-1661		7b. ADDRESS (City, State, and ZIP Code) Building 410 Bolling AFB, DC 20332-6448			
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22a. NAME OF RESPONSIBLE INDIVIDUAL Dr. Anthony J. Matuszko			22b. TELEPHONE (Include Area Code) (202) 767-4963		22c. OFFICE SYMBOL NC

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19. Spirocyclosiloxanes have been prepared by insertion of $[O=Si=O]$ under flash vacuum pyrolysis conditions into silicon-oxygen single bonds of cyclic siloxanes. We have prepared several new types of silicon containing polymers. 1,3-Adamantyldimethylsiloxane copolymers have been prepared. These polymers are quite thermally stable. This may result from steric hindrance to the reversion reaction provided by the adamantane nucleus. Four novel types of unsaturated silyl substituted polymers have been prepared. Stereo- and regio-specific anionic polymerization of 2-trimethylsilyl-1,3-butadiene has been achieved. The reactive vinyl silane functional group of this polymer permits further chemical modification. Stereospecific anionic polymerizations of 2,3-bis(trimethylsilyl)-1,3-butadiene has also been carried out. Ziegler-Natta polymerization of 2-trimethylsilylmethyl-1,3-butadiene occurs in a cis-1,4 manner. Electrophilic substitution reactions on this poly allylsilane system permits further modification of the polymer system. Ring opening anionic polymerization of silacyclopent-3-enes have been achieved. This is a general reaction which permits the preparation of a family of new polymers.

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COMPLETED PROJECT SUMMARY

1. **TITLE:** Silicon Chemistry

2. **PRINCIPAL INVESTIGATOR:**

William P. Weber
Department of Chemistry
Loker Hydrocarbon Research Institute
University of Southern California
Los Angeles, CA 90089-1661



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3. **INCLUSIVE DATES:** November 1, 1985 to October 31, 1988

4. **GRANT NUMBER:** AFOSR 86-0042

5. **COSTS AND FY SOURCE:** \$128,350, FY86; \$130,833, FY87; \$133,094, FY88

6. **SENIOR RESEARCH PERSONNEL:**

Professor Kyung-Tae Kang
Professor Georges Manual
Dr. Antoine Baceiredo
Dr. Yi-Ming Pai
Dr. Clifford Juengst
Dr. Yi-Xiang Ding
Dr. Xuehai Zhang
Dr. Ron Shinomoto
Dr. Shui-Sheng Hu
Dr. Wan Jiang

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MATTHEW J. KERPER
Chief, Technical Information Division

7. **JUNIOR RESEARCH PERSONNEL:**

George Henry
Qingshan Zhou
Young Tae Park
Derek Stonich
Howard Lee
Hai Hong
Cheung-Xue Li
Chia-Piao Kuan

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8. PUBLICATIONS:

"Pyrolysis of 3,3-Dimethyl-6-oxa-3-germabicyclo[3,1,0]hexane. Novel Insertion Reactions of Dimethylgermanone $[(CH_3)_2Ge=O]$ into Silicon-Oxygen and Silicon-Chlorine Single Bonds", K.T. Kang, G. Manuel, and W.P. Weber, *Chem. Lett.*, **10**, 1685 (1986).

"Synthesis of Spirocyclosiloxanes by Flash Vacuum Pyrolysis of 2,7-Dimethyl-2,3:7,8-diepoxy-5-silaspiro[4.4]nonane and Cyclosiloxanes", C.D. Juengst, W.P. Weber, and G. Manuel, *J. Organometal. Chem.*, **308**, 187 (1986).

"Flash Vacuum Pyrolysis (FVP) of 2,7-Dimethyl-2,3:7,8-diepoxy-5-silaspiro[4.4]nonane (I) with cyclotrisiloxanes - Synthesis of Spirocyclosiloxanes. Characterization of Silicon Formed by FVP of I", G.K. Henry, R. Bau, G. Manuel, and W.P. Weber, *Organometallics*, **5**, 1818 (1986).

"1,3-Adamantyl Dimethylsiloxane Copolymers Preparation and Properties", Y.M. Pai and W.P. Weber, *Applied Organometallic Chemistry*, **1**, 171 (1987).

"1,3-Adamantyl Dimethylsiloxane Copolymers Preparation and Properties", Y.M. Pai and W.P. Weber, *Polymer Preprints*, **28**, 116 (1987).

"Preparation and Properties of Dimethylphosphonomethylmethyl Siloxane Dimethylsiloxane Copolymers", C.D. Juengst and W.P. Weber, *J. Poly. Sci. A, Polymer Chemistry*, **25**, 1967 (1987).

"Dimethyl Phosphonomethylmethylsiloxane Dimethylsiloxane Copolymers", C.D. Juengst and W.P. Weber, *Polymer Preprints*, **28**, 114 (1987).

"Reaction of 6-Oxa-3-silabicyclo[3.1.0]hexanes with Phosphinimines. Synthesis of 6-Vinyl-1,3-dioxa-2,4-disilacyclohexanes", A. Baceiredo, C.D. Juengst, G. Manuel, and W.P. Weber, *Chem. Lett.*, **2**, 237 (1987).

"Gas Phase Negative Ion Chemistry of 6-Oxa-3-Sila- and Germabicyclo[3.1.0]-hexanes", R. Damrauer, W.P. Weber, and G. Manuel, *Chem. Lett.*, **2**, 235 (1987).

"Preparation of 1-Aryl-5(N-aryl)-N-benzoylamino)tetrazoles", Y.X. Ding and W.P. Weber, *Syntheses*, 823 (1987).

"Synthesis of Symmetrical Sulfur Bis(aryl) Sulfur Diimides", Y.X. Ding and W.P. Weber, *J. Org. Chem.*, **52**, 4625 (1987).

"1,1-Dimethyl-1-Silacyclopent-3-ene", G. Manuel and W.P. Weber, in *Organometallic Synthesis*, R.B. King and J.J. Eisch, eds., Elsevier Publishers, Amsterdam, 1988, Vol. 4, p. 477.

"Relative Rate Constants for the Reactions of Methylphenylsilylene", J.A. Hawari, M. Lesage, D. Griller, and W.P. Weber, *Organometallics*, **6**, 880 (1987).

"Self-Reaction of Pentamethyldisilyl Radicals: Is Dimerization a Product?" J.A. Hawari, D. Griller, W.P. Weber, and P.P. Gaspar, *J. Organometal. Chem.*, **326**, 335 (1987).

"Exchange Reactions Between Trimethylsilyl Azide and Hexamethyldigermoxane", Q. Zhou and W.P. Weber, *J. Organometal. Chem.*, **342**, 291 (1988).

"Anionic Ring Opening Polymerization of Sila- and Germacyclopent-3-enes", X. Zhang, Q. Zhou, W.P. Weber, R.F. Horvath, T.H. Chan, and G. Manuel, *Macromolecules*, **21**, 1563 (1988).

"Stereospecific 1,4-Polymerization of 2,3-bis(trimethylsilyl)-1,3-butadiene", Y.X. Ding and W.P. Weber, *J. Organometal. Chem.*, **341**, 267 (1988).

"Stereoregularity in Ziegler-Natta and Anionic Polymerization of 2-Trimethylsilylmethyl-1,3-butadiene. Protodesilation of *cis*-1,4-poly(2-trimethylsilylmethyl)-1,3-butadiene", Y.X. Ding and W.P. Weber, *Macromolecules*, **21**, 2672 (1988).

"Regio- and Stereospecific 1,4-Polymerization of 2-(Triethylsilyl)-1,3-butadiene", Y.X. Ding and W.P. Weber, *Macromolecules*, **21**, 530 (1988).

"Diels-Alder Reactions of 1,1-Dimethyl-2,3,4,5-tetraphenyl-1-silacyclopentadiene", G.K. Henry, R. Shinomoto, and W.P. Weber, *J. Organometal. Chem.*, **350**, 3 (1988).

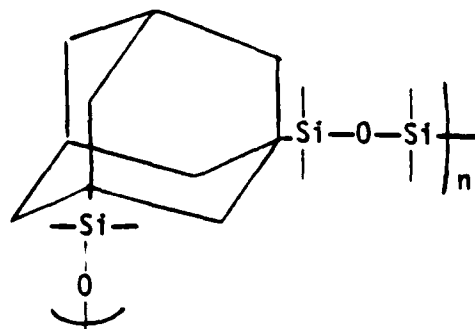
"Reaction of 1,4-poly(2-trimethylsilylmethyl-1,3-butadiene) with phenylsulfonyl chloride - Synthesis of 1,4-poly(3-methylene-2-phenylthiobutane)", Y.X. Ding and W.P. Weber, *Polymer Bulletin*, **20**, 7 (1988).

"Reaction of *E*-1,4-poly(2-trimethylsilyl-1,3-butadiene) with bromine. Stereoselective Synthesis of *Z*-1,4-poly(2-bromo-1,3-butadiene)", W. Jiang and W.P. Weber, *Polymer Bulletin*, **20**, 15 (1988).

"Epoxidation of *E*-1,4-poly(2-trimethylsilyl-1,3-butadiene) and *E*-1,4-poly[2,3-bis(trimethylsilyl)-1,3-butadiene]. Stereochemical Analysis of *E*-1,4-poly(2,3-epoxy-2-triethylsilyl-1,3-butadiene) and *E*-1,3-poly[2,3-bis(trimethylsilyl)-1,3-butadiene] by ^{13}C and ^{29}Si NMR", W. Jiang and W.P. Weber, *Polymer Bulletin*, **20**, 249 (1988).

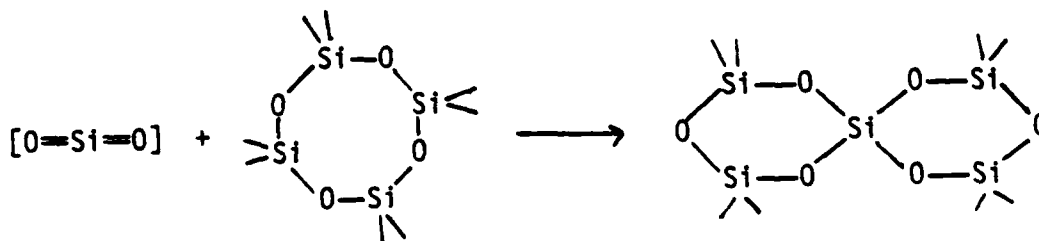
9. ABSTRACT OF OBJECTIVES AND ACCOMPLISHMENTS:

Spirocyclosiloxanes have been prepared by insertion of $[O=Si=O]$ under flash vacuum pyrolysis conditions into silicon-oxygen single bonds of cyclic siloxanes. See references 1 and 3.



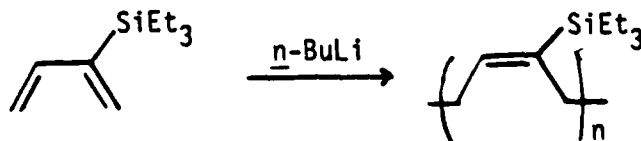
We have prepared several new types of silicon containing polymers.

1,3-Adamantyldimethylsiloxane copolymers have been prepared. See publications 4 and 5. These polymers are quite thermally stable. This may result from steric hindrance to the reversion reaction provided by the adamantane nucleus.



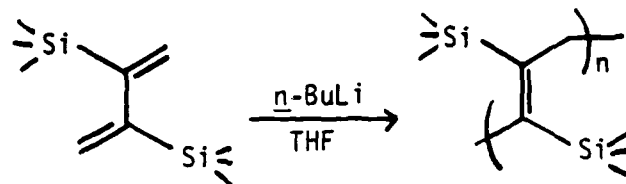
Four novel types of unsaturated silyl substituted polymers have been prepared.

Stereo- and regio-specific anionic polymerization of 2-trimethylsilyl-1,3-butadiene has been achieved.

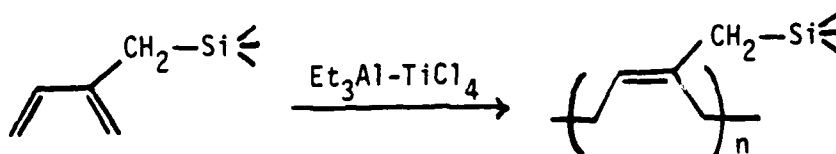


The reactive vinyl silane functional group of this polymer permits further chemical modification. See references 19, 22, and 23.

Stereospecific anionic polymerizations of 2,3-bis(trimethylsilyl)-1,3-butadiene has also been carried out. See reference 17.

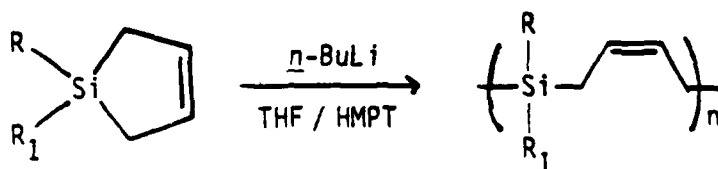


Ziegler-Natta polymerization of 2-trimethylsilylmethyl-1,3-butadiene occurs in a *cis*-1,4 manner.



Electrophilic substitution reactions on this poly allylsilane system permits further modification of the polymer system. See reference 21.

Ring opening anionic polymerization of silacyclopent-3-enes have been achieved. See referenced 16.



This is a general reaction which permits the preparation of a family of new polymers.

FINAL REPORT

Period Covered: November 1, 1985 to October 31, 1988

Grant Number: AFOSR 86-0042

Title: Silicon Chemistry

Principal Investigator: William P. Weber

Department of Chemistry

Loker Hydrocarbon Research Institute

University of Southern California

Los Angeles, CA 90089-1661

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PERSONNEL WORKING ON GRANT

Senior Investigators:

Professor W.P. Weber, principal investigator worked on this contract for one month during the summer 1986, seven weeks during the summer 1987, four weeks during the summer 1988, and 15 percent of time during academic year 1985/86, 1986/87, 1987/88, and 1988/89.

Professor Kyung-Tae Kang, on sabbatical leave from the Department of Chemistry, Pusan University, Pusan, South Korea, worked on this contract 11-1-85 to 1-6-86. He then returned to his position in Korea.

Professor Georges Manual, a visiting professor from the Universite Paul Sabatier, Toulouse, France, spent the period from 7-15-86 to 9-30-86 working on this contract. He also spent the period 7-15-87 to 9-1-87 and 7-15-88 to 9-1-88 in our laboratory. While he was not supported by this contract in either 1987 or 1988, his work was related to this effort. Professor Manual has since returned to his position in France.

Postdoctoral Research Associates:

Dr. A. Baceiredo, 11-18-85 to 1-31-85. Dr. Baceiredo returned to his position at the CNRS Laboratory, Universite Paul Sabatier, Toulouse, France.

Dr. Y.M. Pai, 11-1-85 to 7-18-86. Dr. Pai is now employed by Ciba-Geigy in White Plains, New York in the polymer synthesis area.

Dr. C. Juengst, was supported as a research assistant from 11-1-85 to 6-30-86. He was then a postdoctoral research associate in my group from 8-1-86 to 9-30-86. He is now working at the Swedlow Company on composite materials for aircraft canopies.

Dr. Y-X. Ding, 1-1-86 to 3-31-88. Dr. Ding received his Ph.D. from the Institute of Organic Chemistry, Shanghai, China. He has returned to his position as Associate Professor in Shanghai.

Dr. X. Zhang, 7-28-86 to 7-31-87. Dr. Zhang received his Ph.D. from the Institute of Organic Chemistry, Shanghai, China. Dr. Zhang is now a postdoctoral research associate at the University of South Carolina.

Dr. R. Shinomoto, 12-1-86 to 10-31-87. Dr. Shinomoto received his Ph.D. from the University of California at Berkeley. He is now employed by the Mobil Chemical Company in New Jersey.

Dr. Shui-Sheng Hu, 8-6-87 to 10-31-88. Dr. Hu received his Ph.D. from the Institute of Organic Chemistry, Shanghai, China and did a year postdoctoral work at Iowa State University with Professor Glen Russel before joining our group.

Dr. Wan Jiang, 8-6-87 to 10-31-88. Dr. Jiang received her Ph.D. from Iowa State University, Ames, Iowa.

Graduate Research Assistants:

G. Henry, 11-1-85 to 7-31-86. G. Henry completed his Ph.D. and is now employed by the DuPont Company in Wilmington, Delaware.

Q. Zhou, 6-1-86 to 8-31-86, 6-1-87 to 8-31-87, and 6-1-88 to 10-31-88. Q. Zhou passed his Ph.D. Qualifying Exam in June 1987.

Y.T. Park, 6-1-87 to 8-31-87, and 6-1-88 to 10-31-88. Mr. Park is a graduate of Yonsei University, Seoul, Korea. He passed his Ph.D. Qualifying Exam in Spring 1988.

D. Stonich, 6-1-87 to 8-31-87, and 6-1-88 to 10-31-88. Mr. Stonich is a graduate of San Jose State University. He passed his Ph.D. Qualifying Exam in Spring 1988.

H. Lee, 6-1-87 to 8-31-87 and 6-1-88 to 8-31-88. Mr. Lee is a graduate of Fu Jen Catholic University in Taiwan.

H. Hong, a graduate of Oregon State University and the Shanghai Institute of Science is a second year student and joined our research group in January 1988.

C-X. Li, 6-1-86 to 8-31-86 and C. Kuan, 6-1-86 to 7-31-86. After evaluation of their contribution and progress, it was mutually agreed to end their relationship with our group.

EQUIPMENT PURCHASED

A controlled low temperature water bath was purchased. This is useful for low temperature polymerization reactions.

PUBLICATIONS

1. Pyrolysis of 3,3-Dimethyl-6-oxa-3-germabicyclo[3,1,0]hexane. Novel Insertion Reactions of Dimethylgermanone $[(CH_3)_2Ge=O]$ into Silicon-Oxygen and Silicon-Chlorine Single Bonds, K.T. Kang, G. Manuel, and W.P. Weber, **Chem. Lett.**, **10**, 1685 (1986).
2. Synthesis of Spirocyclosiloxanes by Flash Vacuum Pyrolysis of 2,7-Dimethyl-2,3:7,8-diepoxy-5-silaspiro[4.4]nonane and Cyclosiloxanes, C.D. Juengst, W.P. Weber, and G. Manuel, **J. Organometal. Chem.**, **308**, 187 (1986).
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14. Self-Reaction of Pentamethyldisilyl Radicals: Is Dimerization a Product? J.A. Hawari, D. Griller, W.P. Weber, and P.P. Gaspar, **J. Organometal. Chem.**, **326**, 335 (1987).
15. Exchange Reactions Between Trimethylsilyl Azide and Hexamethyldigermoxane, Q. Zhou and W.P. Weber, **J. Organometal. Chem.**, **342**, 291 (1988).
16. Anionic Ring Opening Polymerization of Sila- and Germacyclopent-3-enes, X. Zhang, Q. Zhou, W.P. Weber, R.F. Horvath, T.H. Chan, and G. Manuel, **Macromolecules**, **21**, 1563 (1988).
17. Stereospecific 1,4-Polymerization of 2,3-bis(trimethylsilyl)-1,3-butadiene, Y.X. Ding and W.P. Weber, **J. Organometal. Chem.**, **341**, 267 (1988).
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23. Epoxidation of *E*-1,4-poly(2-trimethylsilyl-1,3-butadiene) and *E*-1,4-poly[2,3-bis(trimethylsilyl)-1,3-butadiene]. Stereochemical Analysis of *E*-1,4-poly(2,3-epoxy-2-triethylsilyl-1,3-butadiene) and *E*-1,3-poly[2,3-bis(trimethylsilyl)-1,3-butadiene] by ^{13}C and ^{29}Si NMR, W. Jiang and W.P. Weber, **Polymer Bulletin**, **20**, 249 (1988).

MEETINGS ATTENDED AND PAPERS PRESENTED

Attended the 20th Organosilicon Symposium in Tarrytown, N.Y. from April 18-19, 1986 and presented a plenary lecture entitled "Flash Vacuum Pyrolysis of 2,3:7,8-Diepoxy-5-silaspiro[4.4]nonane".

Attended the National American Chemical Society Meeting in Anaheim, CA, September 7-12, 1986.

Attended the Ultrastructure Processing of New Materials Meeting sponsored by the AFOSR, San Diego, CA, February 24-26, 1987.

Attended the 193rd National American Chemical Society Meeting in Denver, CO from April 5-7, 1987 and presented two posters entitled, "1,3-Adamantyl Dimethylsiloxane Copolymers Preparation and Properties" and "Dimethyl Phosphonemethylmethyilsiloxane Dimethylsiloxane Copolymers".

Attended the 8th International Symposium in Organosilicon Chemistry in St. Louis, MO from June 7-12, 1987 and presented a paper entitled "Reaction of 6-Oxa-3-silabicyclo[3.1.0]hexanes with Phosphinimines. Synthesis of 6-Vinyl-1,3-dioxo-2,4-disilacyclohexanes".

Attended the International Topical Workshop "Advances in Silicon-Based Polymer Science", Makaha, Oahu, Hawaii, November 23-25, 1987. Presented two posters entitled "Stereoregular 1,4-Polymerization of 2-Triethylsilyl-1,3-butadiene" and "Stereospecific Anionic Ring Opening Polymerization of Silacyclopent-3-enes".

Attended the AFOSR and AF Wright Aeronautical Laboratory Meeting on Non-Linear Optical Polymers in Washington, D.C. from April 20-21, 1988.

Attended the National American Chemical Society Meeting in Los Angeles, CA from September 25-30, 1988.

SEMINARS PRESENTED

"Unsaturated Silyl Substituted Polymers", Howard University, August 18, 1987.

"Unsaturated Silyl Substituted Polymers", Naval Weapons Station, China Lake, CA, September 15, 1987.

"Unsaturated Silyl Substituted Polymers", Johns Hopkins University, October 5, 1987.

"Unsaturated Silyl Substituted Polymers", DuPont Central Research, Wilmington, DE, October 6, 1987.

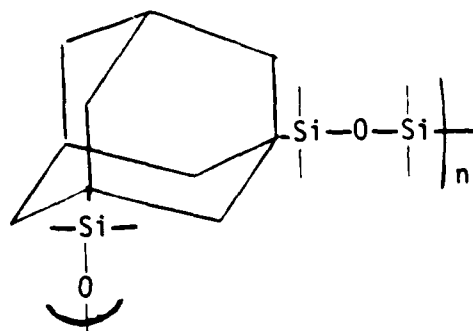
"Unsaturated Silyl Substituted Polymers", Dow Corning, February 22, 1988.

"Synthesis and Reactivity of Silyl Substituted Unsaturated Polymers", California State University at Los Angeles, October 25, 1988

"Synthesis and Reactivity of Silyl Substituted Unsaturated Polymers", Pierce College, Woodland Hills, CA, October 28, 1988

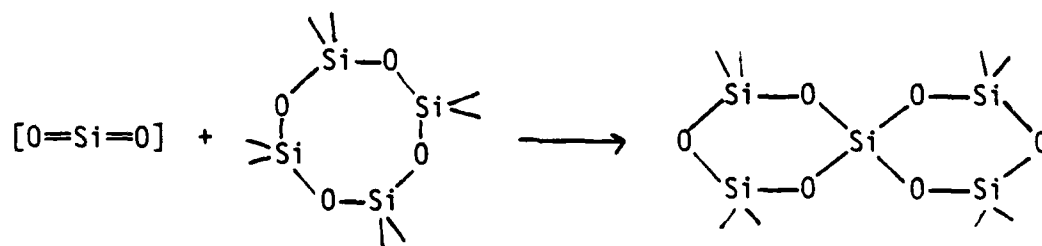
SCIENTIFIC ACHIEVEMENTS

Spirocyclosiloxanes have been prepared by insertion of $[O=Si=O]$ under flash vacuum pyrolysis conditions into silicon-oxygen single bonds of cyclic siloxanes. See references 1 and 3.



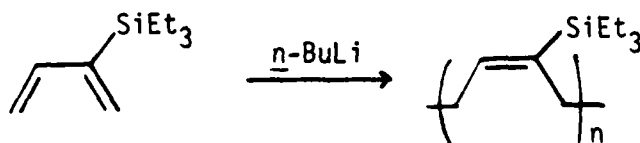
We have prepared several new types of silicon containing polymers.

1,3-Adamantyldimethylsiloxane copolymers have been prepared. See publications 4 and 5. These polymers are quite thermally stable. This may result from steric hindrance to the reversion reaction provided by the adamantane nucleus.



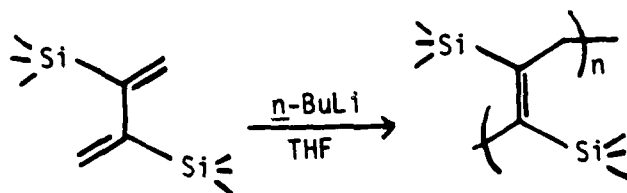
Four novel types of unsaturated silyl substituted polymers have been prepared.

Stereo- and regio-specific anionic polymerization of 2-trimethylsilyl-1,3-butadiene has been achieved.

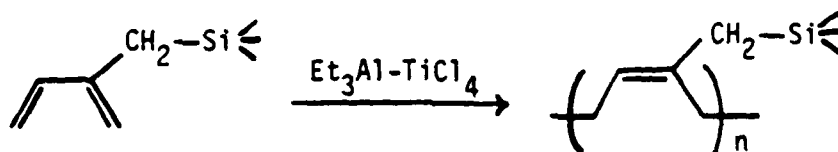


The reactive vinyl silane functional group of this polymer permits further chemical modification. See references 19, 22, and 23.

Stereospecific anionic polymerizations of 2,3-bis(trimethylsilyl)-1,3-butadiene has also been carried out. See reference 17.

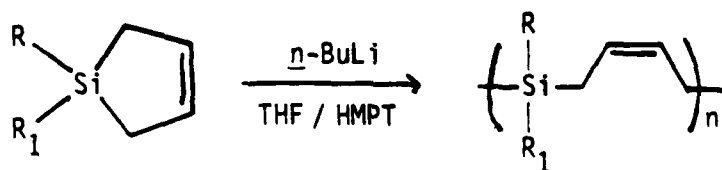


Ziegler-Natta polymerization of 2-trimethylsilylmethyl-1,3-butadiene occurs in a cis-1,4 manner.



Electrophilic substitution reactions on this poly allylsilane system permits further modification of the polymer system. See reference 21.

Ring opening anionic polymerization of silacyclopent-3-enes have been achieved. See referencd 16.



This is a general reaction which permits the preparation of a family of new polymers.