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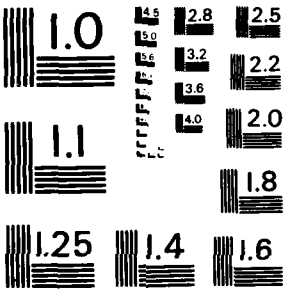

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Polysilahydrocarbon precursors for silicon carbide ceramic compositions have been prepared by potassium dechlorination reactions of mixtures of chlorosilanes and hydrocarbon olefins such as styrene or isoprene. The presence of branched silyl units in the polymer backbones is critical in providing significant ceramic yields.

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TECHNICAL REPORT 83-2

ORGANOSILANE POLYMERS, VI:

BRANCHED POLYSILAHYDROCARBON

PRECURSORS FOR SILICON CARBIDE

by

C. L. Schilling Jr. and T. C. Williams

Union Carbide Corporation
Tarrytown, New York 10591

September 1983

ORGANOSILANE POLYMERS, VI:

BRANCHED POLYSILAHYDROCARBON PRECURSORS

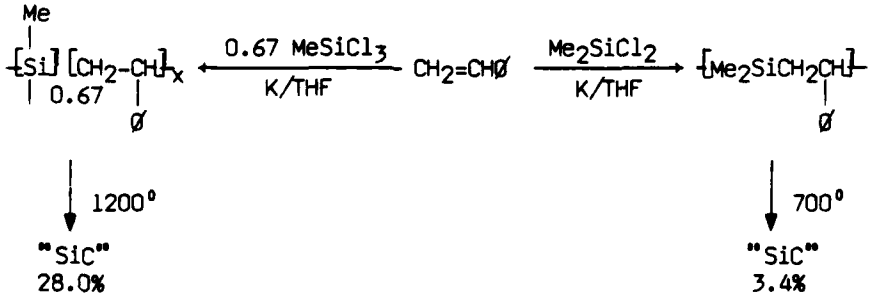
FOR SILICON CARBIDE

INTRODUCTION

In the course of developing highly branched polycarbosilane precursors for silicon carbide ceramic compositions,[1] it was recognized that tri- or tetrafunctional branching silyl units were critical in obtaining effective SiC yields. It was also felt that the same structural approach, i.e., branched silyl units, would provide improved ceramic yields if applied to polysilahydrocarbons (copolymers consisting of silyl monomer units and units derived from hydrocarbon olefins, such as styrene or isoprene). The concept has been validated, with further improvements in ceramic yield being obtained by incorporation of hydrosilyl (≡SiH) monomer units.

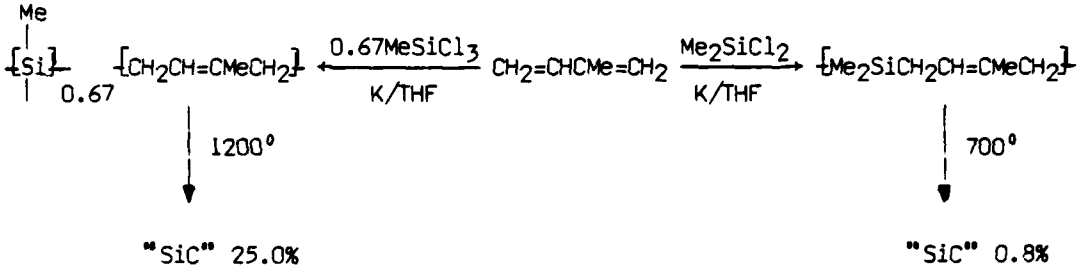
RESULTS AND DISCUSSION

Linear polysilahydrocarbons known in the open literature were prepared and compared with corresponding polymers which contained branching silyl units. For example, a linear copolymer derived from Me_2SiCl_2 and styrene[2,3] was prepared using potassium metal in tetrahydrofuran solvent (K/THF) as the dechlorinating medium. Pyrolysis of the linear polymer to 700° under an inert



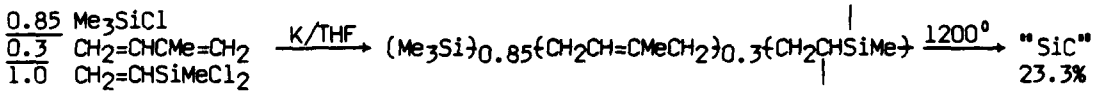
atmosphere provided 3.4% of SiC ceramic composition. An analogous branched copolymer, prepared with a lower molar ratio of $MeSiCl_3$, provided 28.0% of SiC ceramic composition when pyrolyzed to 1200°.

Similarly, a copolymer from Me_2SiCl_2 and isoprene[4] yielded 0.8% of ceramic composition, while the corresponding branched copolymer prepared with $MeSiCl_3$ provided 25.0% of SiC-containing ceramic.



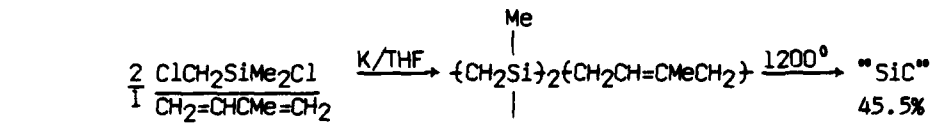
The lower molar ratio of $MeSiCl_3$ to olefin (0.67 relative to 1.0 for Me_2SiCl_2) was used to balance Si-reactive sites (≡SiCl groups) and C-reactive sites (two per molecule for styrene or isosprene), maximizing formation of ≡Si-C bonds.

Isoprene was also incorporated into a typical polycarbosilane (product from 0.85/0.3/1.0 molar mixture of $Me_3SiCl/Me_2SiCl_2/CH_2=CHSiMeCl_2$ and K/THF[1b]) as a substitute for Me_2SiCl_2. While the ceramic yield was lower (31.4% for the



Me_2SiCl_2 version), the product demonstrates the effectiveness of $CH_2=CHSiMeCl_2$ in providing backbone branching in polysilahydrocarbons.

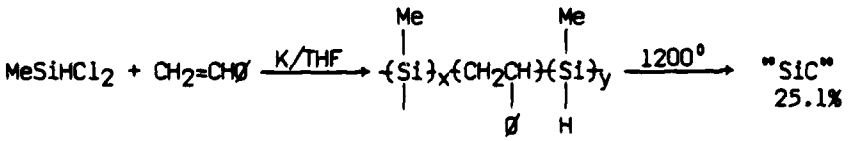
An effective SiC precursor was also obtained using $ClCH_2SiMeCl_2$ as the branching silyl monomer. The 2/1 molar ratio of $ClCH_2SiMeCl_2/CH_2=CHCMe=CH_2$ was



also used to balance C-reactive sites ($CH_2=$ and $ClCH_2-$ groups) with Si-reactive sites, (≡SiCl groups), and provided a corresponding increase in ceramic yield.

A similar precursor prepared from 2/1 $ClCH_2SiMeCl_2$/styrene also provided a high ceramic yield (60.4%), but was an intractable, insoluble solid.

$MeSiHCl_2$ provides both backbone branching units and difunctional hydrosilyl units in polycarbosilanes,[5] and is similarly incorporated into polysilahydrocarbon structures.[6] The product from $MeSiHCl_2/CH_2=CHCMe=CH_2$, although



$$MeSiHCl_2 + CH_2{=}CHCMe{=}CH_2 \xrightarrow{K/THF} \text{-(Si)}_x\text{-(}CH_2CH{=}CMeCH_2\text{)-(Si)}_y \xrightarrow{1200^\circ} \underset{49.5\%}{\text{"SiC"}}$$

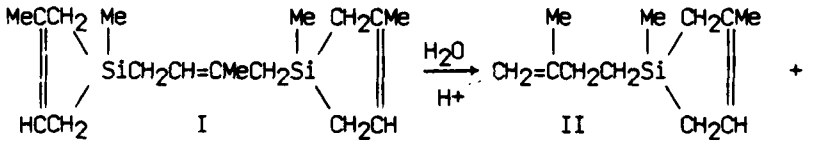
(Me on Si; H on Si)

$(x + y = 1,\ x \cong 0.8)$

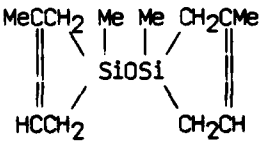
obtained in low yield (21.1% as nonvolatile polymer) was an excellent SiC precursor, providing bulk pieces with higher integrity and density than obtained from the other polysilahydrocarbons. This product, a low viscosity fluid, should perform well as a preceramic adhesive, binder, or coating.

The reaction between $MeSiHCl_2$ and isoprene using magnesium metal in THF (Mg/THF) is reported to yield 1,3-dimethyl-1-silacyclopent-3-ene, plus a polymer.[7] While similar results were obtained by us, the Mg-derived polymer is not an effective SiC precursor (10.6% yield in pyrolysis to 700°) when compared to the K-derived polymer. Spectral and elemental analyses of the K-derived polymer show it to be higher in contained silicon than expected, probably due to formation of ≡SiSi≡ bonds and resultant lower incorporation of isoprenoid units.

Conversely, the major volatile products of the K reaction, before aqueous termination and neutralization, are isomers of 2-methyl-1,4-di[1-(1,3-dimethyl-1-silacyclopent-3-enyl)-2-butene (I), as identified by mass spectrometry and NMR analyses. Compound I is cleaved to a significant extent by



I



II

III

the acidic aqueous workup to 1-(3-methylbut-3-enyl)-1,3-dimethyl-1-silacyclopent-3-ene, II, and a disiloxane, III (see Figures 1, 2 and 3 for respective GC/MS patterns for Compounds I, II and III). Acidic aqueous cleavage of 1,4-disilyl isoprenoid groups has been reported,[8] and no doubt occurs as well with linear polymeric products in the present work.

Polymer Properties

The polysilahydrocarbons reported herein range from low viscosity fluids, such as the nonvolatile K-derived product from $MeSiHCl_2/CH_2{=}CHCMe{=}CH_2$, to insoluble solids, such as that derived from 2/1 $ClCH_2SiMeCl_2/CH_2{=}CH\emptyset$. With the latter exception, only soluble, nonvolatile products were screened in pyrolyses.

These polymers are not amenable to exact elemental analyses due to oxygen incorporation during hydrolytic termination/neutralization, loss of hydrogen from hydrosilyl groups during reactions, differing contents of silyl and hydrocarbon units in volatile and nonvolatile fractions, and problems in completely combusting preceramic polymers.[9] They are amenable to typical thermal analyses, and to the study of molecular weight distributions by gel permeation chromatography.

EXPERIMENTAL

All chlorosilanes were freshly distilled before use. THF was reagent grade, dried over Linde 4A molecular sieves. K Metal was purchased as practical grade ingots; all K metal transfers were made under nitrogen in a dry box. All reactions (preparations and pyrolyses) were run under argon or nitrogen. Routine NMR spectra were recorded on a Perkin-Elmer R24A spectrometer - VPC Analyses were run on a Hewlett-Packard 5840A gas chromatograph. Pyrolyses up to 700° were run in quartz reactors in a Lindberg 54242 tube furnace and those up to 1200° were run in an alumina reactor in a Lindberg 54233 tube furnace. Both furnaces have programmable controllers, which allow attendent-free operation from charging to removal of products. Conversion to SiC compositions were confirmed by x-ray diffraction. Pyrolysis programs are given in Reference 1b.

Reaction of 1/1 $Me_2SiCl_2/CH_2=CH\emptyset$ with K in THF

K metal (21.3g, 0.54 mol) and 179.3g of THF were combined in a 500 ml 3-necked round-bottomed flask, which was then fitted with mechanical stirrer (stainless steel blade), thermometer, addition funnel, Dewar condenser (filled with toluene cooled with ice water immersion coil), and valves for maintaining a nitrogen atmosphere. The addition funnel was charged with 33.5g (0.26 mol) of Me_2SiCl_2 and 27.0g (0.26 mol) of freshly distilled styrene, and contents of the flask heated to reflux, melting the K. Addition of the monomer mixture was completed in 31 min., and the reaction mixture then heated at reflux for 3 hours, followed by cooling, termination by the cautious dropwise addition of 6.63g H_2O in 17.24g THF, and neutralization with 2.5g conc. HCl. Solids were removed by filtration, and the THF solution dried over $MgSO_4$. Refiltration and vacuum stripping provided 41.2g (97.8%) of soluble solid product. Pyrolysis of this material to 700° left only 3.4% of ceramic.

Reaction of 1/1 $Me_2SiCl_2/CH_2=CHCMe=CH_2$ with K in THF

The above procedure was repeated using 23.5g (0.6 mol) of K metal, 421.7g of THF, and a mixture of 36.8g (0.29 mol) of Me_2SiCl_2 and 19.4g (0.29 mol) of isoprene. Reaction mixture was heated at reflux for five hours after completion of addition and was worked up as above. Workup yielded 10.6g (29.6%) of nonvolatile polymer, b.p. greater than 88°/0.5 mm, plus volatile products. Pyrolysis of the polymer to 700° left only 0.8% of ceramic residue.

Reaction of 2/3 $MeSiCl_3/CH_2=CH\emptyset$ with K in THF

The experimental procedure was followed with 22.8g (0.58 mol) of K metal, 172.9g of THF, and a mixture of 27.5g (0.18 mol) of $MeSiCl_3$ and 28.8g (0.28 mol) of styrene. Reaction was heated for four hours after end of addition. Workup as above provided a 50.0% yield of soluble solid and 34.4% of insoluble solid (latter isolated by aqueous extraction of KCl filter cake, followed by water washing and vacuum drying of solid organic product). Pyrolysis of the soluble solid to 1200° in two steps yielded 28.0% of SiC ceramic composition. Its x-ray diffraction pattern was correct for microcrystalline β-SiC.[10]

Reaction of 2/3 $MeSiCl_3/CH_2=CHCMe=CH_2$ with K in THF

The above procedure was followed with 27.6g (0.71 mol) of K metal, 408.8g of THF, and a mixture of 33.5g (0.22 mol) of $MeSiCl_3$ and 22.8g (0.34 mol) of isoprene. Workup yielded 28.2g (85.2%) of soluble solid, b.p. greater than 28°/0.08 mm. Pyrolysis to 1200° provided 25.0% of SiC composition, having the correct x-diffraction pattern for microcrystalline β-SiC.

Reaction of 0.85/0.3/1.0 $Me_3SiCl/CH_2=CHCMe=CH_2/CH_2=CHSiMeCl_2$ with K in THF

The experimental procedure was repeated with 23.6g (0.60 mol) of K metal, 289.4g of THF, and a mixture of 18.6g (0.17 mol) of Me_3SiCl, 4.1g (0.06 mol) of isoprene, and 28.5g (0.2 mol) of $CH_2=CHSiMeCl_2$. Workup yielded 18.8g (61.1%) of soluble solid, which pyrolyzed to 23.3% of SiC ceramic composition (1200°, two steps).

Reaction of 1/1 $MeSiHCl_2$/isoprene with K/THF

In a 1l 3N RB flask were combined 373.5g of THF and 31.42g (0.804 mol) of K metal. Flask was fitted with mechanical stirrer (stainless steel blade), thermometer, addition funnel, condenser, and N_2 flow valves with bubbler. Heat was applied to reflux, melting the K, and addition of a solution of 43.7g (0.38 mol) of $MeSiHCL_2$ and 25.5g (0.38 mol) of isoprene begun. Addition continued intermittently (due to viscosity buildup) with continuous heating until competed (205 min). Reaction took on a rust color shortly after each incremental addition, with the color fading to clear during each addition. Additional THF (280 ml) was added to reduce viscosity and heating continued at reflux for 2 hr. Reaction was cooled on wet ice bath, terminated with a solution of 12.2g H_2O in 49.9g THF, and neutralized with 8g conc. HCl (weakly acidic by pH paper test). KCl was removed by filtration, triturated with THF, and the combined THF solution dried over $MgSO_4$. Dissolution of the KCl in water disclosed no insoluble organic products. The THF solution was filtered to remove $MgSO_4$, followed by stripping and vacuum distillation, yielding 8.93g (21.1%) of heavies, b.p. above 158°/0.24 mm, and 26.57g (63.0%) of volatile products, b.p. up to 158°/0.24 mm. Pyrolysis of the heavies to 1200° in two steps provided 49.5% of hard, black, dense, glossy ceramic chips. The x-ray diffraction pattern was correct for microcrystalline β-SiC.

A similar sample from a repeat preparation analyzed as follows: Cal'd: % C, 61.59, % H, 10.60, % Si, 27.81, % O (by difference) 0.00; Found: % C, 46.71, % H, 9.42, % Si,37.95, % O (by difference) 5.80, % Cl, 0.12. Analysis confirms that silyl units are more concentrated in the heavies than in the volatile products, contributing to the high SiC yield.

Reaction of 1/1 $MeSiHCl_2/CH_2=CH\emptyset$ with K in THF

The above procedure was repeated using 60.22g (1.54 mol) of K metal, 802.1g of THF, and a mixture of 84.46g (0.734 mol) of $MeSiHCl_2$ and 76.5g (0.734 mol) of styrene. Workup yielded 96.95g (89%) of nonvolatile polymer, which pyrolyzed (1200°, 2 steps) to 25.1% of SiC ceramic composition.

Reaction of 2/1 $ClCH_2SiMeCl_2/CH_2=CHCMe=CH_2$ with K in THF

The procedure was followed with 22.3g (0.57 mol) of K metal, 413.0g of THF, and a mixture of 29.53g (0.18 mol) of $ClCH_2SiMeCl_2$ and 6.14g (0.09 mol) of isoprene. An additional 120 ml of THF was added during the reaction to reduce viscosity. Workup yielded 37.7% of volatile products, 40.6% of nonvolatile polymer, and 4.5% of insoluble solid product. Pyrolysis of the nonvolatile polymer to 1200° provided 45.5% of SiC composition.

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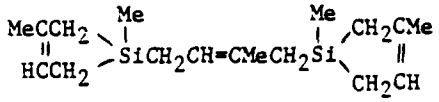
FIGURE 1

Mass Spectrum of 

Spectrum # 217.028 ** Sample # 1256 Retention Time = 14.4 minutes
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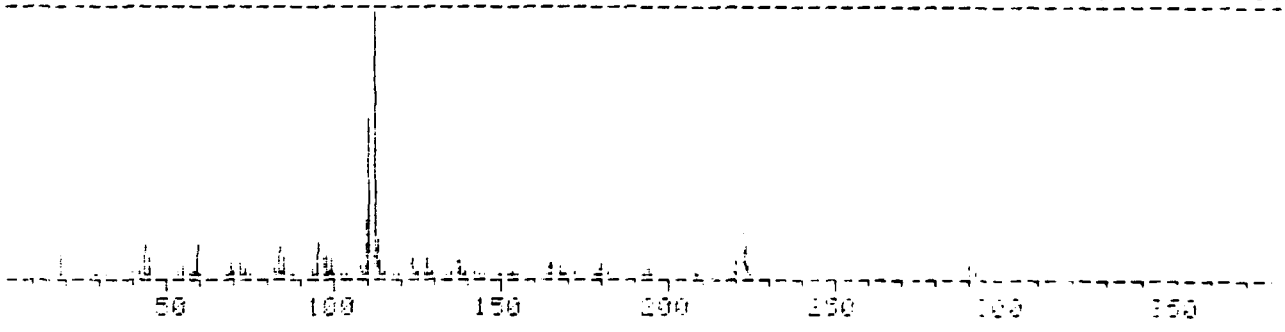
File type = linear
Base Peak = 111.00 Base Peak Abundance = 459 Total Abundance = 1454



Lower Abundance Cutoff Level = 5.0%

MASS	ABUNDANCE (%)	MASS	ABUNDANCE (%)	MASS	ABUNDANCE
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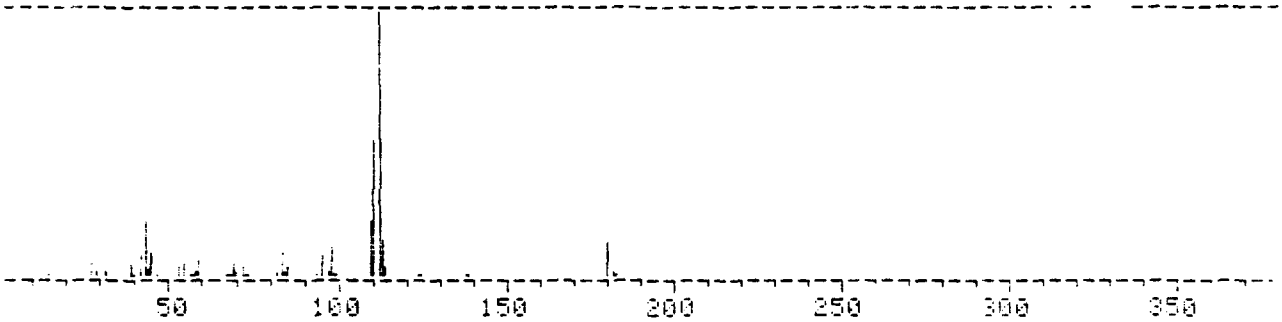
26.99	5.9	97.05	7.1	122.05	5.9
43.95	12.2	99.05	8.1	123.10	9.1
45.05	6.8	109.00	11.2	124.00	6.2
58.95	11.5	110.00	13.5	125.00	5.2
71.00	5.0	111.00	100.0	126.00	7.1
82.90	10.9	112.00	11.5	127.10	10.7
84.95	7.2	113.00	8.1	132.15	12.9
94.95	13.3	123.00	6.9	350.15	5.2

FIGURE 2

Mass Spectrum of $CH_2=CMeCH_2CH_2Si(Me)(CH_2CMe=CH_2)(CH_2CH)$

Spectrum # 191.038 ** Sample # 1256 Retention Time = 1 minutes
Scanned from 10 to 600 amu Number of Peaks Detected = 77
File Type = linear
Base Peak = 111.00 Base Peak Abundance = 3115 Total Abund.. = 10515



Lower Abundance Cutoff Level = 5.0%

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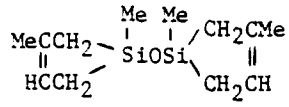
41.00	7.7	83.00	10.9	110.00	50.7
42.95	20.7	95.05	8.0	111.00	100.0
45.05	8.6	97.05	11.2	112.00	13.7
54.95	5.2	109.00	20.9	180.05	13.2
59.95	5.6				

FIGURE 3

Mass Spectrum of 

Spectrum # 199.008 ** Sample # 1256 Retention Time = 9.3 minutes
Scanned from 10 to 600 amu Number of Peaks Detected = 136
File type = linear
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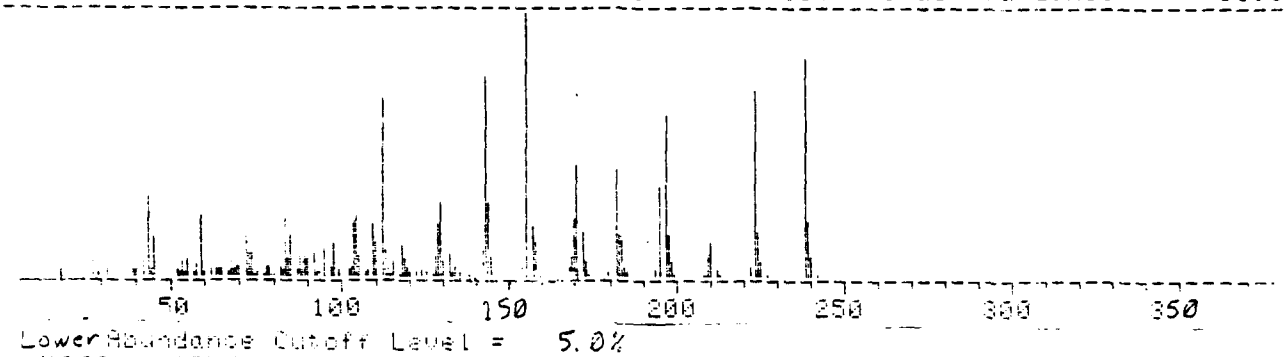

Lower Abundance Cutoff Level = 5.0%

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[illegible]	31.0	105.00	8.1	170.05	42.[illegible]
[illegible]	15.1	109.00	20.9	171.05	14.[illegible]
[illegible]	6.4	110.00	13.1	171.[illegible]	[illegible]
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UNCLASSIFIED TITLE

ORGANOSILANE POLYMERS. VI. BRANCHED POLYSILAHYDROCARBON PRECURSORS FOR SILICON CARBIDE.

ABSTRACT

(U) POLYSILAHYDROCARBON PRECURSORS FOR SILICON CARBIDE CERAMIC COMPOSITIONS HAVE BEEN PREPARED BY POTASSIUM DECHLORINA TION REACTIONS OF MIXTURES OF CHLOROSILANES AND HYDROCARBON OLEFINS SUCH AS STYREENE OR ISOPRENE. THE PRESENCE OF BRANC HED SILYL UNITS IN THE POLYMER BACKBONES IS CRITICAL IN PROVIDING SIGNIFICANT CERAMIC YIELDS. (AUTHOR)

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CERAMIC YIELDS
USE CERAMIC MATERIALS
YIELD

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USE HYDROCARBONS
~~OLEFIN POLYMERS~~ unsaturated hydrocarbons

ORGANOSILANE POLYMERS
USE ORGANIC COMPOUNDS
POLYMERS
SILANES

SILICON CARBIDE CERAMIC COMPOSITIONS
USE CERAMIC MATERIALS
CONCENTRATION(COMPOSITION)
SILICON CARBIDES

CHLOROSILANES
USE CHLOROSILANES

ISOPRENE
USE ISOPRENE

SILICON CARBIDE
USE SILICON CARBIDES

PHRASES NOT FOUND DURING LEXICAL DICTIONARY MATCH PROCESS

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