Bull Soc Chim Fr (1995) 132, 489-494
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# Poly(vinylmethylsilane) and poly(vinyldimethylsilane): synthesis and characterization. Reaction of the latter with phenylacetylene[†]

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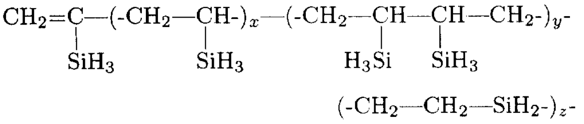
(received 18 October 1994, accepted 6 January 1995)

**Summary** – The $^{60}Co$ γ-radiation-induced polymerization of $CH_2{=}CHSiMeCl_2$ and $CH_2{=}CHSiMe_2Cl$ has been studied. The resulting poly(vinylmethyldichlorosilane) and poly(vinyldimethylchlorosilane) were reduced with $LiAlH_4$ to the respective polymeric silicon hydrides, $PVSiMeH_2$ and $PVSiMe_2H$, which were characterized by IR and $^1H$, $^{13}C$ and $^{29}Si$ NMR spectroscopy and by GPC molecular weight determinations. A structure containing $[CH_2CH(SiR_3)]$ and $[CH_2CH(SiR_3)\text{-}CH(SiR_3)CH_2]$ repeat units and $CH_2{=}C(SiR_3)$ end groups ($R_3Si{=}(CH_3)H_2Si$ and $(CH_3)_2HSi$) was deduced. The chloroplatinic acid-catalyzed addition of oligomeric $PVSiMe_2H$ to phenylacetylene gave an oligomer containing *trans*-$PhCH{=}CHSiMe_2$- and $CH_2{=}C(Ph)SiMe_2$- side chains.

**$^{60}Co$ γ-radiation / polymerization / polyvinylmethylchlorosilanes / reduction $LiAlH_4$**

## Introduction

In a previous paper [1] we reported the preparation of poly(vinylsilane), $PVSiH_3$, by $LiAlH_4$ reduction of poly(vinyltrichlorosilane). The latter had been prepared by $^{60}Co$ γ-radiation-induced polymerization of vinyltrichlorosilane. The $PVSiH_3$ thus prepared was not the simple $[CH_2CH(SiH_3)]_n$ that might have been expected. On the basis of $^1H$ and $^{29}Si$ NMR studies, its composition in terms of structural components was determined to be more complicated and formula **1** is a representation of the polymer that shows its constituent units but not how they are linked together.



**1**

$$CH_2{=}\underset{SiH_3}{C}\text{—}(\text{-}CH_2\text{—}\underset{SiH_3}{CH}\text{-})_x\text{—}(\text{-}CH_2\text{—}\underset{H_3Si}{CH}\text{—}\underset{SiH_3}{CH}\text{—}CH_2\text{-})_y\text{-}\quad(\text{-}CH_2\text{—}CH_2\text{—}SiH_2\text{-})_z\text{-}$$

It was of interest to extend this study to an investigation of the preparation of poly(vinylmethylsilane) and poly(vinyldimethylsilane), ideally $[CH_2CH(SiCH_3H_2)]_n$ and $[CH_2CH(Si(CH_3)_2H)]_n$. As in the case of poly(vinylsilane), our entry to these polymers was *via* the $^{60}Co$ γ-radiation-induced polymerization of the respective vinylchlorosilane. The polymerization of $CH_3Cl_2SiCH{=}CH_2$ had been studied previously by Japanese [2a,b] and Russian [2c] workers. An oligomeric product of relatively low molecular weight ($\overline{DP}$ (degree of polymerization) *ca* 2.5-3) was obtained.

## Results and discussion

### *Poly(vinylmethylsilane)*

To prepare the required precursor for this polymer, vinylmethyldichlorosilane was charged into a thick-walled Pyrex tube with **1** wt% of di-*tert*-butyl peroxide, which had been found to accelerate the γ-ray-induced polymerization of vinyltrichlorosilane [1]. The tube was sealed *in vacuo* and placed in the $^{60}Co$ γ-ray reaction chamber which provided a dose rate of 1.03 MRad/day, After a total dose of 43 MRad, the polymer was isolated by removal of all volatile components at reduced pressure with heating. Poly(vinylmethyldichlorosilane), $PVSiMeCl_2$, **2**, was obtained in 44% yield as a yellow-orange, transluscent solid. As expected, the product is hydrolytically unstable, forming an insoluble white solid on exposure to moisture.

The IR and NMR spectra of $PVSiMeCl_2$ did not provide much information. A structure more complicated than that of the regular $[CH_2CH(SiMeCl_2)]_n$ was suggested by the observation of three resonances in the $^{29}Si$ NMR spectrum of the product. However, its elemental analysis was in good agreement with this simple formula. The $^1H$ NMR spectrum (resonances at δ 5.85 and 5.90) and the $^{13}C$ NMR spectrum (resonances at $\delta_C$ 132.2, 144.1 and 145.1) of $PVSiMeCl_2$ suggested that

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[†] Dedicated to professor Raymond Calas, a pioneer of modern organosilicon chemistry.

[*] Correspondence and reprints

$CH_2{=}$ end groups are present (as has been the case with $PVSiCl_3$), equivalent to a DP of *ca*, 23.

Further information was gained when $PVSiMeCl_2$ was reduced to $PVSiMeH_2$ with $LiAlH_4$. The Si-H-containing polymer obtained in this reaction was isolated as a mobile, pale yellow oil that is soluble in common organic solvents and is air- and moisture-stable. GPC molecular weight determinations gave $M_n = 1\,628$ and $M_w = 2\,742$ (polydispersity 1.7).

The $^1H$ (fig 1) and $^{29}Si$ (fig 2) NMR spectra of $PVSiMeH_2$, provided useful information concerning this polymer. The peaks in the $^1H$ NMR spectrum are rather broad and no spin-spin coupling was observed, The $CH_3Si$ signal is a single peak at 0.12 ppm, The $SiH_2$ proton signals appear as a singlet at $\delta$ 3.7. The rest of the peaks in the 0.5 to 2.3 ppm region look remarkably similar to the $^1H$ NMR pattern of $PVSiH_3$ [1]. There are three main resonances at $\delta$ 0.75, 1.15, and 1.50 ppm whose relative intensities also are very similar to the intensities of the corresponding resonances of $PVSiH_3$. Accordingly, the resonances in this region are assigned as follows : $\delta$ 0.75 ($MeH_2SiC\textit{H}C\textit{H}(SiMeH_2)$), 1.15 $CH_2C\textit{H}(SiMeH_2)$ and 1.50 $C\textit{H}_2CH(SiMeH_2)$. The two resonances at $\delta$ 5.5 and 5.7 are due to the protons of the vinyl end groups, $CH_2{=}$. Their presence is confirmed by the $^{13}C$ NMR spectrum which shows resonances in the vinyl region at $\delta_C$ 128.4, 145.1 and 145.8. The $^{29}Si$ NMR spectrum has a large resonance at $\delta_{Si}$ −29.3 and a smaller one at −32.7. Both resonances are inverted when DEPT sequencing is used, which indicates that both resonances correspond to $SiH_2$ groups. These chemical shift values are in good agreement with literature values : $(SiH_2CH_2CH_2CH_2)_n$ [3], −30.4 and $RCH_2CH_2SiH_2CH{=}CH_2$ [4] −31.8 ppm. Based on these data and on the relative intensities of these resonances in the $^{29}Si$ NMR spectrum of $PVSiMeH_2$, the smaller resonance at −32.7 ppm can be assigned to a silicon atom attached to a vinyl end group. The major resonance at −29.3 ppm is assigned to the pendant -$SiH_2CH_3$ groups on the polyethylene backbone of the polymer. No resonances corresponding to SiH groups were observed in the $^{29}Si$ NMR spectrum of $PVSiMeH_2$, which suggests that no chain transfer to polymer had occurred (as had been observed in the case of $PVSiH_3$). Based on all these data, the compositional formula (which implies no information on the connectivities of the component units) can be approximated as **2**.

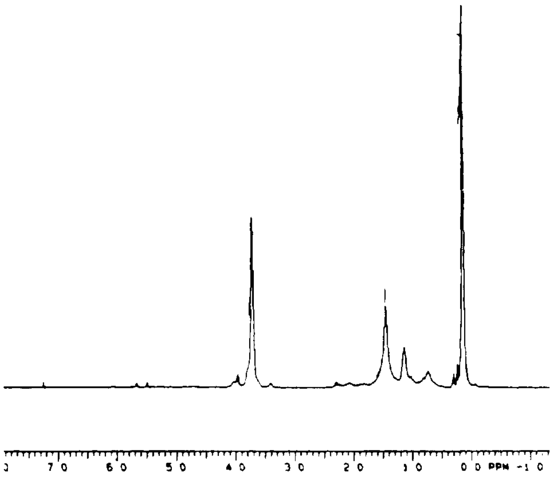


**Fig 1.** $^1H$ NMR spectrum of poly(vinylmethylsilane).

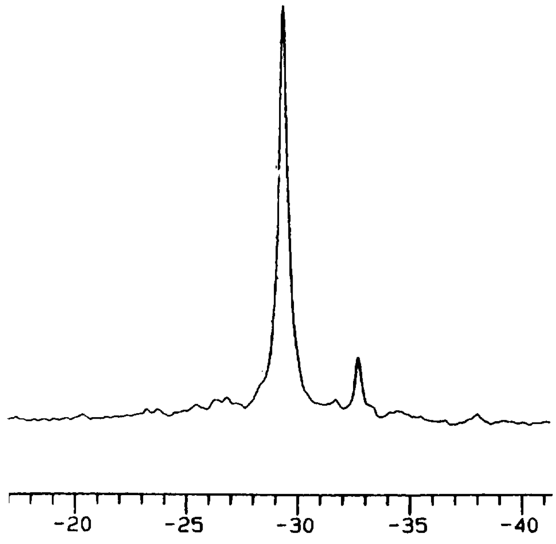
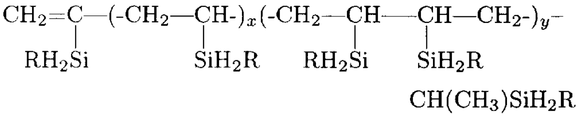


**Fig 2.** $^{29}Si$ NMR spectrum of poly(vinylmethylsilane).



$$CH_2{=}\underset{RH_2Si}{C}{-}(\text{-}CH_2{-}\underset{SiH_2R}{CH}\text{-})_x(\text{-}CH_2{-}\underset{RH_2Si}{CH}{-}\underset{\substack{SiH_2R\\ CH(CH_3)SiH_2R}}{CH}{-}CH_2\text{-})_y\text{-}$$

**2**, $R = CH_3$

*Poly(vinyldimethylsilane)*

The polymerization of the precursor, vinyldimethylchlorosilane, was carried out in a manner similar to that used for vinyltrichlorosilane [1] and vinylmethyldichlorosilane. The tube containing the monomer was irradiated with a 1.03 MRad/day $^{60}Co$ $\gamma$-source in the presence of 1 wt% of di-*tert*-butyl peroxide with a total dose of 43 MRad. Removal of volatile components under reduced pressure left poly(vinyldimethylchlorosilane), $PVSiMe_2Cl$, as a viscous, yellow material in 28-32% yield in various experiments. The product forms an insoluble white solid on exposure to moisture. Its elemental analysis was in good agreement with the simple formula $[CH_2CH(SiMe_2Cl)]_n$. Here also, the $^1H$ and $^{13}C$ NMR spectra indicated the presence of vinylic end groups.

$LiAlH_4$ reduction of $PVSiMe_2Cl$ gave $PVSiMe_2H$ as a clear, pale yellow oil which was soluble in all common organic solvents and stable toward air and moisture. $PVSiMe_2H$ is oligomeric rather than polymeric. GPC-derived molecular weight data, $M_n = 447$, $M_w = 483$ (polydispersity 1.1, indicated that there are only 5-6 units per chain.

The elemental analysis for C and H was in agreement with the simple formula $[CH_2CH(SiMe_2H)]_n$, but as with PVSiMeH$_2$, the $^1$H and $^{29}$Si NMR spectra indicated a less regular structure. In the $^1$H NMR spectrum (fig 3) the main $(CH_3)_2Si$ proton signal is observed as a broadened singlet at $\delta$ 0.0 and an adjacent downfield resonance of low intensity can be assigned to the $(CH_3)_2Si$ protons of the $CH_2{=}C(SiMe_2H)$-end group. The broad signals in the $\delta$ 0.4-1.8 ppm region are similar to those found in the $^1$H NMR spectra of PVSiH$_3$ and PVSiMeH$_2$ and may be assigned to similar structural units. Also present in the $^1$H NMR spectrum of PVSiMe$_2$H are very low intensity multiplets in the 2.0-2.4 ppm region, These are in the allyl proton region $(C{=}C\text{-}CH_2)$ [5] and can be assigned to the $CH_2{=}C(SiMe_2H)CH_2$ protons at the end of the oligomer. The SiH proton signal is a broad resonance at $\delta$ 3.9. A small multiplet at $\delta$ 4.1 can be assigned to the $CH_2{=}C(SiMe_2H)$-proton. Vinylic proton signals are observed at $\delta$ 5.4 and 5.6. If the signals due to end group CH and SiH protons are excluded, the $^1$H NMR spectrum of PVSiMe$_2$H is almost identical to that of the polyethylene with irregularly spaced SiMe$_2$H substituents obtained as shown in scheme 1 in previous studies in these Laboratories [6].

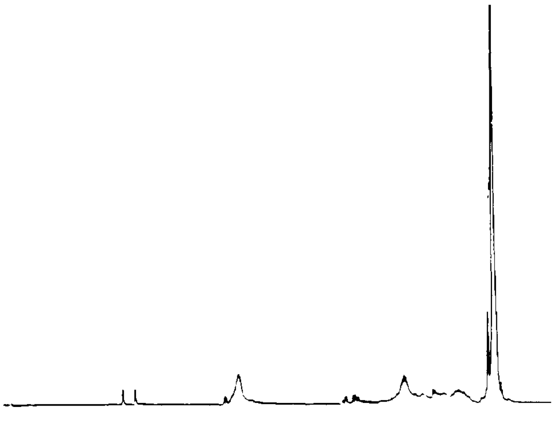
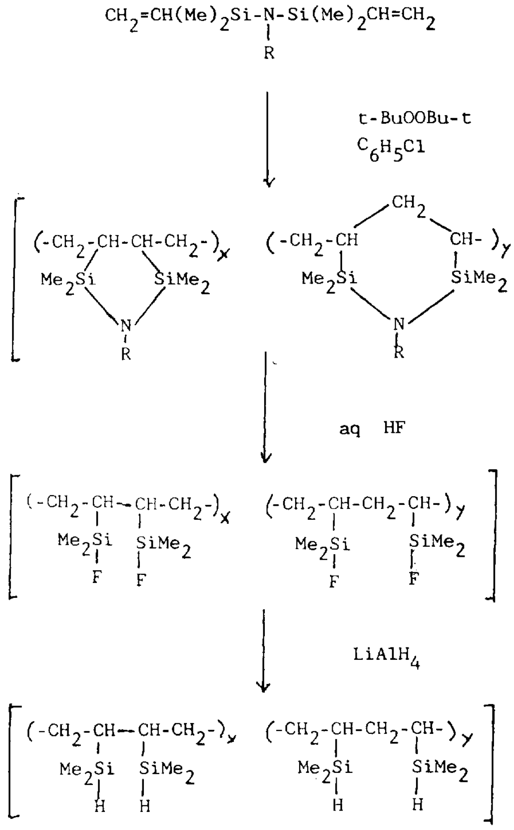


**Fig 3.** $^1$H NMR spectrum of poly(vinyldimethylsilane).

The $^{29}$Si NMR spectrum of PVSiMe$_2$H (fig 4) is more complex than that of PVSiMeH$_2$, Three principal resonances are seen at $\delta$ Si −12.6, −11.1 and −9.8. These are in the general region observed for R$_3$SiH compounds [7], including the polymer of [ref 6]. A low intensity signal at $\delta$ Si −19.3 is assignable to the $CH_2{=}C(SiMe_2H)$- end group silicon atom. The other minor resonances downfield from the principal resonances have not been assigned.

If, as these data suggest, the polymerization of $CH_2{=}CHSiMe_2Cl$ proceeded similarly to that of $CH_2{=}CHSiMeCl_2$, then the compositional formula of PVSiMe$_2$H may be approximated by **3**.



**Scheme 1**

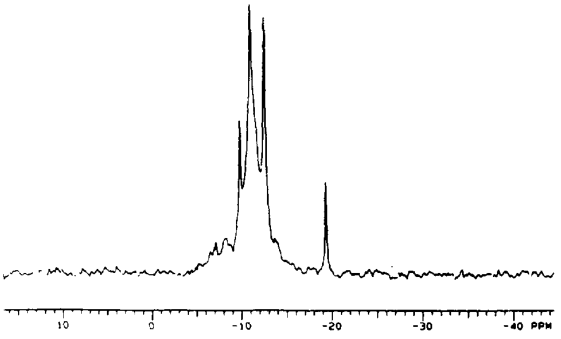
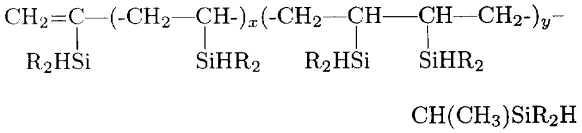


**Fig 4.** $^{29}$Si NMR spectrum of poly(vinyldimethylsilane).



$$\underset{R_2HSi}{CH_2{=}C}\text{—}(\text{-}CH_2\text{—}\underset{SiHR_2}{CH}\text{-})_x(\text{-}CH_2\text{—}\underset{R_2HSi}{CH}\text{——}\underset{SiHR_2}{CH}\text{—}CH_2\text{-})_y\text{-}CH(CH_3)SiR_2H$$

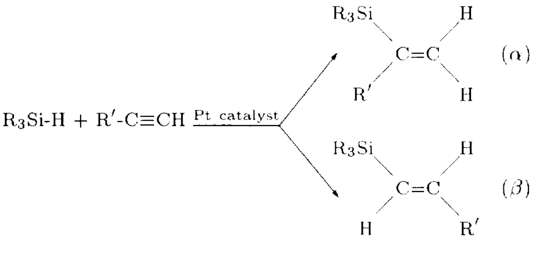
**3**, R = CH$_3$

## *Hydrosilylation of Phenylacetylene with PVSiMe$_2$H*

The hydrosilylation reaction is an efficient procedure for introducing diverse functionality into organosilicon compounds [8]. Several reports of organosilicon polymer functionalization by this means have been published. In one recent example, Polish workers [9] used the Pt-catalyzed addition of the Si-H bonds of a polycarbosilane, $((CH_3)HSiCH_2)_n$, to an appropriate terminal olefin to introduce mesogenic side chains into the polycarbosilane, thus forming a liquid crystalline organosilicon polymer.

The Si-H bonds of the pendant $SiMe_2H$ groups of $PVSiMe_2H$ can be used in a similar manner to introduce functionality. Since the C≡C bond in general is more reactive than the C=C bond in Pt-catalyzed hydrosilylation [10], phenylacetylene was chosen as the substrate for a brief study of a hydrosilylation reaction of $PVSiMe_2H$.

The addition of an Si-H bond to the triple bond of a terminal acetylene can take place in either the $\alpha$ or the $\beta$ sense (eq 1).

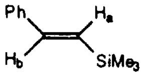


(1)

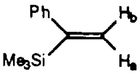
The vinyl protons in each case are distinguishable in the ¹H NMR spectrum, Usually, a mixture of both isomers is formed, with their ratio dependent on the alkyne and on the silane. The formation of the $\beta$ isomer of only *trans* configuration is a characteristic feature of the platinum complex-catalyzed hydrosilylation [10]. Thus, Pt-catalyzed hydrosilylation of phenylacetylene with $PVSiMe_2H$ would be expected to give $SiMe_2C(Ph)=CH_2$ and $SiMe_2CH=CHPh$-*trans* substituents on the carbon atom backbone.

Reactions of phenylacetylene with $PVSiMe_2H$ in the presence of chloroplatinic acid (CPA) at room temperature or 50°C did not result in complete reaction of all Si-H bonds, giving about 30 and 69% conversion, respectively. Good results were obtained when the reaction mixture was stirred for 24 h at 75°C. The product was a brown, plastic-like material, obtained in 93% yield after removal of volatiles. Similar results were obtained when the reaction was carried out for 24 h at 75°C in toluene solution.

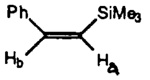
The IR spectrum of the hydrosilylation product showed only a very weak peak at 2 157 cm$^{-1}$, showing that the reaction went essentially to completion. The near absence of Si-H bonds was confirmed by the ¹H NMR spectrum of the hydrosilylation product : the SiH proton signal in the spectrum of $PVSiMe_2H$ at $\delta$ 3.6-4.0 had virtually disappeared. Two pairs of broad multiplets were observed in the ¹H NMR spectrum of the hydrosilylation product; for one pair, resonances centered at $\delta$ 5.80 and 6.05; for the other, resonances centered at $\delta$ 6.70 and 7.10. In the assignment of these pairs of multiplets the ¹H NMR spectra of model compounds **4**, **5** and **6** were useful. Based on these data, the resonances in the ¹H NMR spectrum of the hydrosilylation product may be assigned. The resonances at $\delta$ 5.80 and 6.05 can be assigned to the $\alpha$-addition product and those at $\delta$ 6.70 and 7.10 to the $\beta$-addition product. The hydrosilylation product does not contain an adduct similar to model compound **6**; if such were present, a single broad resonance around $\delta$ 5.8 would have been observed for $H_a$ and the signal for $H_b$ would have been buried under the phenyl proton signal. The ratio of $\alpha$-addition to $\beta$-addition was 0.91 based on integration of these resonances.



11
4 $\delta(H_a)$, 6.49
$\delta(H_b)$, 6.88



12
5 $\delta(H_a)$, 5.61
$\delta(H_b)$, 5.83



11
6 $\delta(H_a)$, 5.83
$\delta(H_b)$, 7.36

The GPC molecular weight data obtained for the hydrosilylation product are of some interest : $M_n$ = 939; $M_w$ = 2 147, polydispersity = 2.3. Thus the number average molecular weight is roughly double that of $PVSiMe_2H$. This is close to the expected ratio (2.1 experimental; 2.2 theoretical). However, the polydispersity is much greater (2.3 vs 1.1). It is apparent from the GPC trace that small amounts of higher molecular weight materials are present in the hydrosilylation product (fig 5). This most likely is due to the addition of some of the $SiMe_2H$ groups in the polymer to $CH_2=C(SiMe_2H)$- end groups, which would lead to effective doubling of the molecular weight of the oligomer molecule involved. Support for this interpretation is provided by the fact that the vinylic end groups of $PVSiMe_2H$ no longer are present in the hydrosilylation product, as evidenced by the absence of the 5.7 ppm vinyl proton signal of $PVSiMe_2H$.

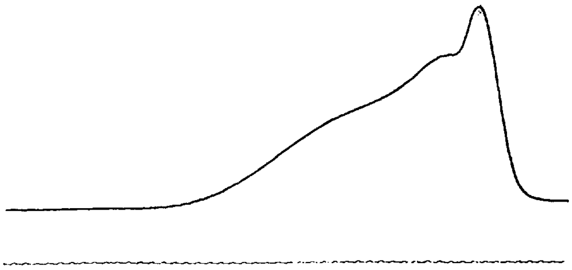


**Fig 5.** GPC trace of the product of the phenylacetylene/poly(vinyldimethylsilane) reaction.

## Experimental section

All reactions were performed under an argon atmosphere. Solvents were dried by established procedures. Infrared spectra were recorded on a Perkin Elmer 1600 FTIR spectrophotometer. All NMR spectra were recorded on a Bruker 250 or a Varian 300 instrument. GPC molecular weight determinations were made using a Waters Millipore 150-C ALC/GPC chromatograph equipped with a Waters Millipore Ultrastyrogel $10^3$ column with toluene as solvent. Elemental analyses were performed by Scandinavian Microanalytical Laboratory, Herlev, Denmark.

Polymerizations were carried out at 25°C with exposure to a $^{60}$Co $\gamma$-source, dose rate 1.03 MRad/day. The samples were sealed *in vacuo* in thick-walled Pyrex tubes (2.5 cm od, 14 cm length, with a 10 cm, 8 mm od neck).

Vinylmethyldichlorosilane and vinyldimethylchlorosilane were purchased from Hüls America and distilled from magnesium turnings and degassed before use.

### *Polymerization of vinylmethyldichlorosilane*

A mixture of 57.6 g $CH_2{=}CHSiMeCl_2$ and 0.72 mL di-*tert*-butyl peroxide was irradiated for 42 days in the $^{60}$Co $\gamma$-ray chamber (43 MRad total). The sealed tube was opened and the volatiles were distilled *in vacuo*, heating at 130°C for 3 h. The residue, $PVSiMeCl_2$, a yellow-orange, transluscent solid, was obtained in 44% yield.

IR (thin film, NaCl, $cm^{-1}$) : 3 068w, 1 456m, 1 263s ($\nu$ $SiCH_3$), 1 059w, 947w, 788s, 746s.

$^1$H NMR (250 MHz, $CDCl_3$) : $\delta$ 0.7-0.9 (broad), 1.0-2.8 (broad, unresolved), 5.85 and 5.90 (s).

$^{13}$C NMR (75.5 MHz, $CDCl_3$) : $\delta_C$ 5.0, 19.9, 20.6, 27.4, 30.7, 32.9, 132.2, 144.1, 145.1.

$^{29}$Si NMR (59.59 MHz, $CDCl_3$) : $\delta_{Si}$ 17.7, 32.1, 33.5.

Anal calc for $(C_3H_6Cl_2Si)_n$ : C, 25.55; H, 4.25; Cl, 50.28. Found : C, 25.43; H, 4.22; Cl, 49.96.

### *Reduction of poly(vinylmethyldichlorosilane)*

A suspension of 6.21 g (0.164 mol) of $LiAlH_4$ in 500 mL of diethyl ether was prepared in a 2 L, three-necked, round-bottomed flask equipped with a reflux condenser topped with an inert gas inlet/outlet tube, a mechanical stirrer and a pressure equalizing addition funnel and cooled in an ice bath. To this mixture was added 23.09 g (0.164 mol, based on monomer) of $PVSiMeCl_2$ in 150 mL of diethyl ether. The reaction mixture was allowed to warm to room temperature and stirred for 17 h. The reaction mixture was filtered through a pad of Celite and the filtrate was added cautiously ( !) to 600 mL of ice-cooled 2 N HCl. The layers were separated and the aqueous layer was extracted twice with $Et_2O$. The organic layers were combined, washed twice with distilled water and dried over anhydrous $MgSO_4$. Volatile components were distilled *in vacuo* with heating to 120°C, leaving $PVSiMeH_2$ (10.05 g, 85%) as a pale yellow, mobile oil.

IR (thin film, NaCl, $cm^{-1}$) : 3 047w, 2 962m, 2 892s, 2 128s, 1 448m, 1 419m, 1 252s ($\nu SiCH_3$), 945s, 892s, 735m.

$^1$H NMR (250 MHz, $CDCl_3$) : $\delta$ 0.0-0.2 (broad), 0.5-2.3 (broad), 3.3-3.4 (broad), 3.5-3.8 (broad), 4.0 (s), 5.5 (s), 5.7 (s); $T_l$ (3.69) = 2.0 sec).

$^{13}$C NMR (75.5 MHz, $CDCl_3$) : $\delta_C$ −10.3, −8.6, 9.4, 20.1, 33.9, 128.4, 145.1, 145.8.

$^{29}$Si NMR (59.59 MHz, $CDCl_3$) : $\delta_{Si}$ −32.7, −29.3 ($J_{SiH}$ = 189 Hz).

Mol wt (GPC) : $M_n$ = 1 628, $M_w$ = 2 742, polydispersity 1.7.

Anal calc for $(C_3H_8Si)_n$ : C, 49.96; H, 11.09. Found : C, 49.83; H, 10.91.

### *Polymerization of vinyldimethylchlorosilane*

The same procedure was used in the polymerization of 46.9 g of $CH_2{=}CHSiMe_2Cl$ in the presence of 0.59 mL of di-*tert*-butyl peroxide using a total dose of 43 MRad of $^{60}$Co $\gamma$-radiation. The product (13.5 g, 32%) was a viscous, yellow material.

IR (thin film, NaCl, $cm^{-1}$) : 3 053w, 2 963s, 2 905s, 1 601w ($\nu$ C=C), 1 450m, 1 407s, 1 250s ($\nu$ Si-$CH_3$), 1 052m, 804 (broad)s.

$^1$H NMR (250 MHz, $CDCl_3$) : $\delta$ 0.3-0.7 (broad), 0.7-2.6 (broad), 5.6 (s), 5.7 (s).

$^{13}$C NMR (75.5 MHz, $CDCl_3$) : $\delta_C$ 1.5, 1.9, 17.5, 21.3, 28.5, 34.0, 128.7, 147.3.

$^{29}$Si NMR (59.59 MHz, $CDCl_3$) : $\delta_{Si}$ 19.8, 31.7, 33.8.

Anal calc for $(C_4H_9ClSi)_n$ : C, 39.84; H, 7.46. Cl, 29.40. Found : C, 40.08; H, 7.67; Cl, 29.04.

### *Reduction of poly(vinyldimethylchlorosilane)*

The same procedure as above was used in the reaction of 2.1 g (0.056 mol) of $LiAlH_4$ in 200 mL of $Et_2O$ with 13.5 g (0.11 mol as monomer) of $PVSiMe_2Cl$ in 100 mL of $Et_2O$. Distillation of the final organic layers at reduced pressure left 7.56 g (78%) of $PVSiMe_2H$ as a mobile, pale yellow oil.

IR (thin film, NaCl, $cm^{-1}$) : 3 046w, 2 957s, 2 902m, 2 110s, 1 418w, 1 249s ($\nu$ Si-$CH_3$), 1 051w, 876s, 833s, 757m.

$^1$H NMR (250 MHz, $CDCl_3$) : $\delta$ 0.0-0.3 (broad), 0.4-2.4 (broad), 3.6-4.0 (broad), 5.4 (s), 5.6 (s); $T_l$ (3.8) = 2.7 sec.

$^{13}$C NMR (75.5 MHz, $CDCl_3$) : $\delta_C$ −5 5, −4.5, 12.8, 21.7, 24.2, 25.7, 37.5, 126.2, 149.4.

$^{29}$Si NMR (59.59 MHz, $CDCl_3$) : $\delta_{Si}$ −19.3, −12.6, −11.0, −9.8 ($J_{SiH}$ = 182 Hz).

Mol wt (GPC) : $M_n$ = 447, $M_w$ = 483, poly-dispersity 1.1.

Anal calc for $(C_4H_{10}Si)_n$ : C, 55.78; H, 11.61. Found : C, 54.74; H, 11.25.

### *Hydrosilylation of phenylacetylene with $PVSiMe_2H$*

A 25 mL two-necked, round-bottomed flask equipped with a reflux condenser topped with an inert gas inlet/outlet tube, a magnetic stirbar and a rubber septum was charged with 1.0 g (11.6 mmol as the monomer) of $PVSiMe_2H$ and 1.4 mL (12.8 mmol) of phenylacetylene. Three drops of CPA solution (0.1 M in isopropyl alcohol) was added and the reaction mixture was stirred-and heated at 75°C for 24 h. A brownish, viscous mixture resulted. Volatile components were removed *in vacuo* with heating at 100°C for 1 h, leaving 2.04 g (93%) of a brown, plastic-like residue.

IR (thin film, NaCl, $cm^{-1}$) : 3 057m, 2 950s, 2 248w, 2 157w, 1 942w, 1 874w, 1 801w, 1 603m, 1 574m, 1 493s, 1 446m, 1 408m, 1 248s, 1 028m.

$^1$H NMR (300 MHz, $CDCl_3$) : $\delta$ 0.38 (broad s), 0.6-2.7 (broad), 5.6-5.9 (m), 5.9-6.2 (m), 6.5-6.9 (m), 6.9-7.2 (m), 7.2-7.8 (broad).

$^{13}$C NMR (75.5 MHz, $CDCl_3$) : $\delta_C$ −3.1 (broad), 14.0, 23.0, 36.0, 126.6, 127.9, 128.1, 128.5, 138.4, 144.2, 144.6, 152.5.

Mol wt (GPC) : $M_n$ = 939, $M_w$ = 2 147, polydispersity 2.3.

Anal calc for $(C_{12}H_{16}Si)_n$ : C, 76.52; H, 8.56. Found : C, 73.59; H, 8.50.

The analysis indicates that the hydrosilylation reaction was not quite complete.

## Acknowledgment

The authors are grateful to the National Science Foundation for support of this work.

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