

Study on hydrosilylation of alkenes bearing an adamantyl pendant

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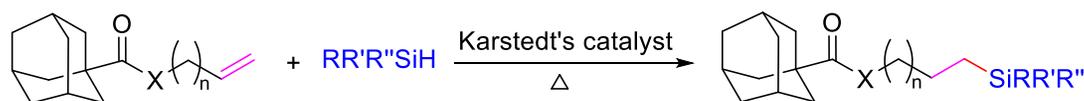
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Abstract

The adamantyl group is a distinctive structural motif that plays a pivotal role in polymeric materials. Polymers incorporating silanes have found extensive applications in material science. Hydrosilylation stands out as a robust methodology for constructing silane-based polymers. In this study, we investigate the hydrosilylation of alkenes containing ester or amide functionalities, featuring the bulky adamantyl group as a pendant moiety. Our primary objective is to elucidate the reactivity of hydrosilylation and to ascertain whether the presence of the adamantyl group influences the reaction kinetics. Employing the conventional Karstedt's catalyst, we have successfully synthesized a series of silanes bearing the adamantyl pendant group. This research presents a viable and efficient strategy for the preparation of silane polymers or monomer incorporating the adamantyl pendant group, thereby contributing to the advancement of functional polymer materials.



X = O, N

- Selective for hydrosilanes
- Applicable to dihydrosilylation
- Yields up to 98%

Keywords: Hydrosilylation, adamantyl group, hydrosilane, hydrosiloxane, Karstedt's catalyst

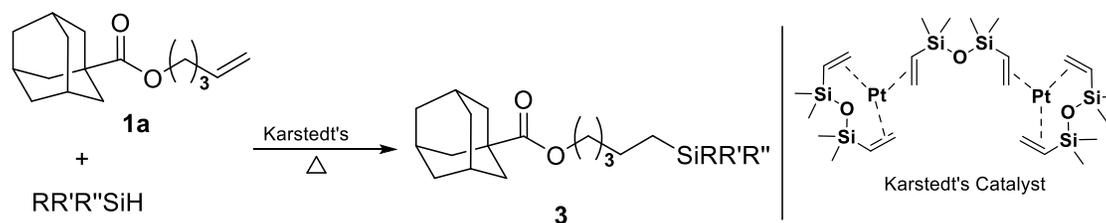
Introduction

Adamantane features a unique rigid yet strain-free ring system composing three fused chair-configured cyclohexane rings, analogous to the diamond lattice structure. This highly symmetric molecule exhibits exceptional thermal stability and demonstrates diamagnetic exaltation exceeding Pascal's atomic increments.¹ The adamantyl motif has found significant application in medicinal chemistry, notably in the antiviral drug amantadine (1-adamantylamine) for influenza A², Parkinson disease³ and COVID-19⁴ treatment. Furthermore, its reversible host-guest interactions with β -cyclodextrin enable applications in self-healing hydrogels with dual-state (wet/dry) healing capabilities.⁵ Recent studies reveal that adamantyl incorporation enhances material properties, including: (1) conductivity modulation in poly(methylthiophene)⁶, (2) elevated glass transition temperatures and thermal stability in polymethyl methacrylate (PMMA)⁷, and (3) superior optical/thermal performance in LED encapsulation materials.⁸ Current synthetic approaches for adamantyl-modified silicones employ esterification methods using toxic stannous octoate catalyst⁸ or hydrosilation on unreactive alkene using rhodium catalyst⁹, which face limitations in silane diversity, economic viability and toxicity.

Organosilicon materials are typically prepared via hydrosilylation¹⁰, catalyzed by Pt complexes^{11, 12} (e.g., Speier's¹³ or Karstedt's¹⁴ catalysts) and other metal complexes¹⁵⁻¹⁷. However, the hydrosilylation of adamantyl-functionalized alkenes remains largely unexplored, which motivates our investigation into the hydrosilylation of conventional adamantyl alkenes with diverse silanes/siloxanes, aiming to apply in the construction of high-performance organosilicons, such as silicon-based fouling-release coatings (FRCs)¹⁸.

Results and Discussion

Our investigation commenced with the evaluation of model substrate 4-penten-1-yl tricyclo[3.3.1.1^{3,7}]decane-1-carboxylate (**1a**), synthesized through carbodiimide-mediated esterification of 1-adamantanecarboxylic acid with pent-4-en-1-ol¹⁹.

Table 1. Hydrosilylation of 4-penten-1-yl tricyclo[3.3.1.1^{3,7}]decane-1-carboxylate (**1a**) with hydrosilanes^a

Entry	Hydrosilane or hydrosiloxane	Product	Yield (%)
1	PhMe ₂ SiH		98
2	Ph ₂ MeSiH		37
3	Ph ₃ SiH		0 ^b
4	Ph ₂ SiH ₂		60 ^c
5	Et ₃ SiH		33 ^d
6	(EtO) ₃ SiH		71

^aAlkene (**1a**), silane (or siloxane) (**2**, 1.1 equiv.) and Karstedt's catalyst (0.01% equiv.) were added subsequently into a Schlenk tube, the mixture was purged with N₂ before heating. The reaction was heated to 130°C for 4 hr. All yields are recorded as isolated yields, otherwise they are indicated separately. ^bXylene was added (100 μ L for 100 mg mixture of **1a** and Ph₃SiH). ^c3.0 Equiv. of Ph₂SiH₂ was used. ^d3.0 Equiv. of silane was used and the reaction was heated to 80 °C.

As shown in Table 1, the hydrosilane structure significantly influenced the reaction outcome. PhMe₂SiH proved most effective, affording adduct **3a** in nearly quantitative yield (98%). However, replacing one methyl group with phenyl (Ph₂MeSiH) substantially reduced the yield of **3b** (37%). Complete substitution with phenyl groups (Ph₃SiH) completely inhibited productive hydrosilylation, with crude NMR analysis revealing only unreacted alkene **1a** and rearrangement products in ~1:5 ratio. For dihydrosilane Ph₂SiH₂, despite using excess

reagent to minimize dihydrosilylation, the reaction yielded a complete mixture of monoadduct **3d**, unreacted **1a** and rearrangement products. The yield of **3d** (60%) is slightly higher than that of **3b**. Triethylsilane (Et_3SiH) required modified conditions (3 equiv., reduced temperature) due to its volatility, but delivered **3e** in only 33% yield. Notably, triethoxysilane exhibited excellent reactivity, producing **3f** in 71% yield.

This represents the first systematic demonstration that while the bulky adamantyl group does not impede Pt-catalyzed hydrosilylation, electronic property and steric hindrance of silanes affect reactivity. PhMe_2SiH and alkoxy silanes emerge as optimal reagents^{11, 20, 21}.

We subsequently investigated the hydrosilylation reactivity of *N*-(2-propen-1-yl)tricyclo[3.3.1.1^{3,7}]decane-1-carboxamide (**1b**), which was prepared through DIC/HOBt-mediated coupling of 1-adamantanecarboxylic acid with allylamine²².

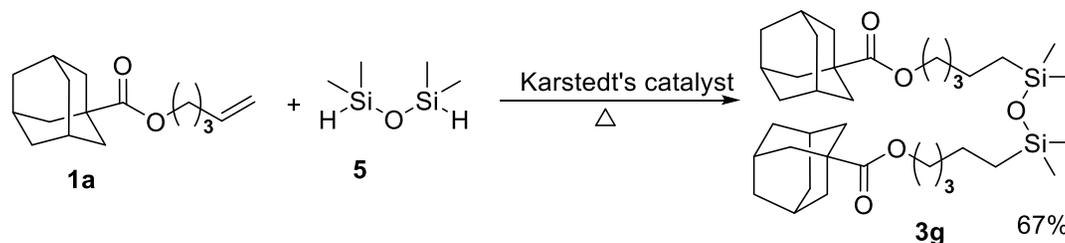
Table 2. Hydrosilylation of *N*-(2-propen-1-yl)tricyclo[3.3.1.1^{3,7}]decane-1-carboxamide (**1a**) with hydrosilanes^a

Entry	Hydrosilane	Product	Yield (%)
1	PhMe_2SiH		82
2	Ph_2MeSiH		45

^aAlkene (**1b**), silane (**2**, 1.1 equiv.) and Karstedt's catalyst (0.01% equiv.) were added subsequently into a Schlenk tube, the mixture was purged with N_2 before heating. The reaction was heated at 130 °C for 4 hr. Both yields are recorded as isolated yields.

Consistent with our previous observations, the adamantyl moiety did not affect the hydrosilylation reaction. PhMe_2SiH demonstrates superior performance compared to Ph_2MeSiH . Notably, the yield of adduct **4b** (45%) exceeded that of its ester counterpart **3b** (37%), which we attribute to reduced competing rearrangement pathways in the amide substrate system.

To demonstrate the potential for polymer synthesis, we investigated a dihydrosilylation approach using bifunctional siloxane reagent (Scheme 1). The hydrosilylation of 4-penten-1-yl tricyclo[3.3.1.1^{3,7}]decane-1-carboxylate (**1a**) with 1,1,3,3-tetramethyldisiloxane (**5**) was investigated using Karstedt's catalyst (0.01% loading). The reaction proceeded efficiently at 130°C in xylene, affording the corresponding dihydrosilylation product **3g** in 67% isolated yield.



Scheme 1. Dihydrosilylation of 4-penten-1-yl tricyclo[3.3.1.1^{3,7}]decane-1-carboxylate (**1a**).

Conclusions

A systematic investigation of Karstedt's catalyst-catalyzed hydrosilylation reactions involving alkenes bearing adamantyl pendants with diverse hydrosilanes and hydrosiloxanes has been conducted. Contrary to expectations, the bulky adamantyl moiety exhibited negligible steric hindrance to the transformation. We have demonstrated that the hydrosilane structure profoundly influences the product distribution, markedly affecting the ratios between hydrosilylation adducts, rearranged alkenes, and unreacted starting materials. Furthermore, the methodology proved equally effective for dihydrosilylation reactions. This approach has potential to be applied in constructing high-performance silicone-based fouling-release coating with high thermal stability.

Experimental Section

General. All solvents were analytical grade reagent without purification after purchase. Starting materials were synthesized in-house. All reactions were conducted in conventional glassware. Melting points were recorded on hot-plate melting point apparatus (uncorrected). Infrared spectroscopy was recorded on a Nicolet Avatar 370 FT-IR spectrometer. ¹H and ¹³C NMR spectra were acquired on a Varian 400MR equipment (400 MHz for ¹H, 101 MHz for ¹³C) and chemical shifts were reported in ppm relative to TMS or residual solvent peaks. High-resolution mass spectrometry (HRMS) was performed on an AB SCIEX X500R Q-TOF mass spectrometer.

General procedure for hydrosilylation reaction. All hydrosilylation reactions were performed under a nitrogen atmosphere. To a Schlenk tube, the adamantyl-functionalized alkene substrate (**1**, 1.0 equiv.) and the respective hydrosilane (**2**, 1.1 equiv.) and Karstedt's catalyst (0.01% equiv.) were subsequently added. The reaction vessel was purged with nitrogen (three vacuum/N₂ cycles) and heated to 130 °C for 4 hours. For dihydrosilane (Ph₂SiH₂) or dihydrosiloxane (1,1,3,3-tetramethyldisiloxane), the silane or siloxane loading was reduced to 0.51 equiv. to minimize dihydrosilylation side products. After cooling to room temperature, the crude products were purified by flash chromatography on silica gel to afford the desired hydrosilylation adducts (**3** or **4**). (note: for preparation of **3g**, xylene was added, and concentration is 100 μ L for 100 mg mixture of alkene **1a** and 1,1,3,3-tetramethyldisiloxane)

5-(Dimethylphenylsilyl)pentyl adamantyl-1-carboxylate (3a). Colorless oil, 98% (168 mg, 0.44 mmol). *R*_f = 0.20 (PE-EtOAc, 100:1). ¹H NMR (400 MHz, CDCl₃) δ 7.51-7.49 (m, 2H), 7.36-7.34 (m, 3H), 4.00 (t, *J* = 6.6 Hz, 2H), 2.00 (br s, 3H), 1.87 (d, *J* = 3.0 Hz, 6H), 1.74-1.67 (m, 6H), 1.61-1.55 (m, 2H), 1.36-1.33 (m, 4H), 0.77-0.73 (m, 2H), 0.25 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 177.9, 139.6, 133.7, 128.9, 127.9, 64.3, 40.8, 39.0, 36.7, 30.0, 28.5, 28.1,

23.8, 15.9, -2.9. IR (KBr pellet) ν_{\max} 1725, 1453, 1233, 1077, 834 cm^{-1} . HRMS-ESI calcd. for $\text{C}_{24}\text{H}_{36}\text{NaSiO}_2$ ($[\text{M}+\text{Na}]^+$) 407.2377, found: 407.2359.

5-(Methyldiphenylsilyl)pentyl adamantyl-1-carboxylate (3b). Colorless oil, 37% (72 mg, 0.16 mmol). $R_f = 0.36$ (PE-EtOAc, 24:1). ^1H NMR (400 MHz, CDCl_3) δ 7.54-7.51 (m, 4H), 7.39-7.34 (m, 6H), 4.01 (t, $J = 6.6$ Hz, 2H), 2.01 (sept, $J = 3.1$ Hz, 3H), 1.88 (d, $J = 3.0$ Hz, 6H), 1.76-1.68 (m, 6H), 1.64-1.57 (m, 2H), 1.44-1.40 (m, 4H), 1.11-1.07 (m, 2H), 0.56 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 177.9, 137.4, 134.6, 129.2, 127.9, 64.3, 40.8, 39.0, 36.7, 30.1, 28.4, 28.1, 23.7, 14.3, -4.3. IR (KBr pellet) ν_{\max} 2904, 2850, 1723, 1487, 1453, 1232, 1106, 1076, 698 cm^{-1} . HRMS-ESI calcd. for $\text{C}_{29}\text{H}_{38}\text{NaO}_2\text{Si}$ ($[\text{M}+\text{Na}]^+$) 469.2533, found: 469.2527.

5-(Diphenylsilyl)pentyl adamantyl-1-carboxylate (3d). Colorless oil, 60% (361 mg, 0.83 mmol). $R_f = 0.53$ (PE-EA, 30:1). ^1H NMR (400 MHz, CDCl_3) δ 7.57-7.54 (m, 4H), 7.42-7.34 (m, 6H), 4.85 (t, $J = 3.7$ Hz, 1H), 4.00 (t, $J = 6.5$ Hz, 2H), 2.00 (quint, $J = 3.2$ Hz, 3H), 1.87 (d, $J = 3.0$ Hz, 6H), 1.75-1.66 (m, 6H), 1.60 (quint, $J = 7.0$ Hz, 2H), 1.54-1.39 (m, 4H), 1.16 (td, $J = 7.9, 3.8$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 177.9, 135.2, 134.6, 129.7, 128.1, 64.2, 40.8, 39.0, 36.7, 29.6, 28.4, 28.1, 24.3, 12.3. IR (KBr pellet) ν_{\max} 3068(w), 2905, 2851, 2114, 1724, 1428, 1408, 1233, 1077, 805, 731 cm^{-1} . HRMS-ESI calcd. for $\text{C}_{28}\text{H}_{40}\text{NO}_2\text{Si}$ ($[\text{M}+\text{NH}_4]^+$) 450.2823, found: 450.2829.

5-(Triethylsilyl)pentyl adamantyl-1-carboxylate (3e). Colorless liquid, 33% (111 mg, 0.30 mmol). $R_f = 0.37$ (PE-EA, 4:1). ^1H NMR (400 MHz, CDCl_3) δ 4.03 (t, $J = 6.6$ Hz, 2H), 2.01 (quint, $J = 3.1$ Hz, 3H), 1.88 (d, $J = 3.0$ Hz, 6H), 1.74-1.67 (m, 6H), 1.61 (quint, $J = 7.0$ Hz, 2H), 1.40-1.27 (m, 4H), 0.92 (t, $J = 7.9$ Hz, 9H), 0.49 (q, $J = 8.0$, 6H), 0.52-0.46 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 178.0, 64.4, 40.8, 39.0, 36.7, 30.3, 28.5, 28.1, 23.8, 11.4, 7.6, 3.4. IR (KBr pellet) ν_{\max} 2906, 2874, 2852, 1727, 1454, 1267, 1103, 722 cm^{-1} . HRMS-ESI calcd. for $\text{C}_{22}\text{H}_{41}\text{O}_2\text{Si}$ ($[\text{M}+\text{H}]^+$) 365.2870, found: 365.2855.

5-(Triethoxysilyl)pentyl adamantyl-1-carboxylate (3f). Colorless oil, 71% (131 mg, 0.32 mmol). $R_f = 0.34$ (PE-EtOAc, 15:1). ^1H NMR (400 MHz, CDCl_3) δ 4.03 (t, $J = 6.6$ Hz, 2H), 3.81 (q, $J = 7.0$ Hz, 6H), 2.00 (br s, 3H), 1.88 (d, $J = 3.0$ Hz, 6H), 1.74-1.67 (m, 6H), 1.61 (quint, $J = 7.1$ Hz, 2H), 1.45-1.38 (m, 4H), 1.22 (t, $J = 7.0$ Hz, 9H), 0.66-0.62 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 177.9, 64.2, 58.4, 40.8, 39.0, 36.6, 29.5, 28.4, 28.1, 22.7, 18.4, 10.5. IR (KBr) ν_{\max} 2906, 1726, 1233, 1075, 956, 780 cm^{-1} . HRMS-ESI calcd. for $\text{C}_{22}\text{H}_{40}\text{NaO}_5\text{Si}$ ($[\text{M}+\text{Na}]^+$) 435.2537, found: 435.2526.

13-(Adamantyl-1-carboxyloxy)-6,6,8,8-tetramethyl-7-oxa-6,8-disilatridecyl adamantyl-1-carboxylate (3g). Colorless oil, 67% (63 mg, 0.122 mmol). $R_f = 0.30$ (PE-EtOAc, 30:1). ^1H NMR (400 MHz, CDCl_3) δ 4.03 (t, $J = 6.1$ Hz, 4H), 2.00 (sept, $J = 3.1$ Hz, 6H), 1.88 (d, $J = 3.0$ Hz, 12H), 1.75-1.66 (m, 12H), 1.61 (quint, $J = 6.9$ Hz, 4H), 1.40-1.30 (m, 8H), 0.53-0.49 (m, 4H), 0.03 (s, 12H). ^{13}C NMR (101 MHz, CDCl_3) δ 177.9, 64.3, 40.8, 39.0, 36.6, 29.8, 28.5, 28.1, 23.11, 18.41, 0.5. IR (KBr pellet) ν_{\max} 2905, 2851, 1726, 1453, 1231, 1103, 837, 782 cm^{-1} . HRMS-ESI calcd. for $\text{C}_{36}\text{H}_{62}\text{NaO}_5\text{Si}_2$ ($[\text{M}+\text{Na}]^+$) 653.4028, found: 653.4023.

N-[3-(Dimethylphenylsilyl)propyl]adamantyl-1-carboxamide (4a). White solid, m.p. 50.5-51.5 $^\circ\text{C}$. 82% (60 mg, 0.17 mmol). $R_f = 0.35$ (PE-EtOAc, 3:1). ^1H NMR (400 MHz, CDCl_3) δ 7.51-7.48 (m, 2H), 7.36-7.34 (m, 3H), 5.56 (s, 1H), 3.20 (td, $J = 7.2, 5.7$ Hz, 2H), 2.03 (sept, $J = 3.3$ Hz, 3H), 1.82 (d, $J = 3.0$ Hz, 6H), 1.75-1.64 (m, 6H), 1.48 (tt, $J = 8.2, 7.6$ Hz, 2H), 0.75-0.70 (m, 2H), 0.27 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 177.9, 139.1, 133.6, 129.1, 127.9, 42.3, 40.6, 39.4, 36.6, 28.2, 24.3, 13.0, -3.0. IR (KBr pellet) ν_{\max} 3341, 2904, 1632, 1529, 1248, 1113, 834, 700 cm^{-1} . HRMS-ESI calcd. for $\text{C}_{22}\text{H}_{33}\text{NNaO}_6\text{Si}$ ($[\text{M}+\text{Na}]^+$) 378.2224, found: 378.2223.

N-[3-(Methyldiphenylsilyl)propyl]adamantyl-1-carboxamide (4b). Colorless oil, 45% (93 mg, 0.22 mmol). $R_f = 0.45$ (PE-EtOAc, 3:1). ^1H NMR (400 MHz, CDCl_3) δ 7.52-7.49 (m, 4H), 7.39-7.33 (m, 6H), 5.55 (brs, 1H), 3.23 (td, $J = 7.2, 5.7$ Hz, 2H), 2.03 (brs, 3H), 1.81 (d, $J = 3.4$ Hz, 6H), 1.75-1.65 (m, 6H), 1.54 (tt, $J = 8.1, 7.6$ Hz, 2H), 1.08-1.03 (m, 2H), 0.55 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 177.8, 136.8, 134.4, 129.3, 127.9, 42.2, 40.5, 39.3, 36.5, 28.2, 24.22, 11.42, -4.5. IR (KBr) ν_{\max} 3350, 2903, 2850, 2235, 1634, 1516, 1450, 1427 cm^{-1} . HRMS-ESI calcd. for $\text{C}_{27}\text{H}_{36}\text{NOSi}$ ($[\text{M}+\text{H}]^+$) 418.2561, found: 418.2573.

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Supplementary Material

¹H NMR and ¹³C NMR data for this article can be accessed on the publisher's website.

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