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Carbonylative and Hydrosilylative Carbocyclization of 1,5-Diynes for the Synthesis of Cyclic Dienones

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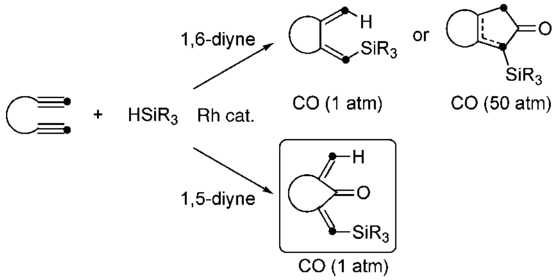
Summary: A rhodium complex, [Rh(acac)(CO)$_2$], has been identified to catalyze the cyclization of various 1,5-diynes with hydrosilanes under an atmospheric pressure of carbon monoxide at room temperature. The cyclization of 1,5-diynes proceeds along with hydrosilylation and carbonylation to give derivatives of 2,5-dialkylidenecyclopentanone.

Introduction

Transition metal-catalyzed silylcarbocyclization is an established synthetic protocol for the construction of various ring systems containing silyl groups, in which the carbometalation initiated by the silylation of unsaturated motifs, such as alkynes and alkenes, is a key step.[1] Silylcarbocyclization was first documented in Tamao's report, where a nickel complex catalyzed the reaction of 1,7-diynes with hydrosilanes to give 1,2-dialkylidenecyclohexanes.[2] Ojima discovered rhodium complex-catalyzed silylcarbocyclization.[3] Enynes,[3a,g,4] diynes,[3a–d,5] triynes,[3e] enediynes,[3f] and allenynes[6] have been submitted to the reaction and various types of functionalized cyclic compounds were obtained.[7]

Scheme 1. Different Patterns of Carbonylative Silylcarbocyclization of Diynes



For instance, the reaction of 1,6-diynes under an atmospheric pressure of carbon monoxide with [Rh(acac)(CO)$_2$] as a catalyst give silyl-substituted monocyclic 1,3-dienes; however, carbon monoxide is not incorporated into the products (Scheme 1).[3b] In contrast, when the reaction was examined under the pressurized conditions, carbon monoxide was inserted between the terminal acetylenic carbons (•) of diynes.[8] We here disclose a new pattern of carbonylation using 1,5-diynes, where carbon monoxide is inserted between the internal acetylenic carbons along with hydrosilylation. The cyclization proceeds under an atmospheric pressure of carbon monoxide at room temperature.

Results and Discussion

In the presence of a catalytic amount of [Rh(acac)(CO)$_2$], the reaction of 2-ethynyl-1-(hex-1-ynyl)benzene (**1a**) and dimethylphenylsilane (**2a**) was examined in toluene under an atmospheric pressure of carbon monoxide (Table 1, entry 1). 1,5-Diyne **1a** was completely consumed within 2 h even at room temperature, and a hydrosilylative/carbonylative cyclization proceeded to give 1,3-dialkylideneindan-2-one **3aa** in high yield.[9] Other hydrosilanes **2b** and **2c** also reacted with diynes **1a**, but the yield of corresponding dienones **3ab** and **3ac** was decreased (entries 2 and 3). In the case of trialkoxysilane **2d**, the reaction proceeded sluggishly; moreover, the product was unstable and could not be fully characterized (entry 4). We decided to use silane **2a** for further investigation.

We next examined the reaction of several aromatic 1,5-diynes with silane **2a** (Table 2). In addition to a butyl group, phenyl-

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(8) Carbonylative carbocyclization of enediynes proceeded under an atmospheric pressure of carbon monoxide, and CO was inserted between two carbons of enediyne termini. In this reaction, the silyl group was not incorporated into the product: (a) Ojima, I.; Lee, S.-Y. *J. Am. Chem. Soc.* **2000**, *122*, 2385. (b) Bennacer, B.; Fujiwara, M.; Ojima, I. *Org. Lett.* **2004**, *6*, 3589. (c) Bennacer, B.; Fujiwara, M.; Lee, S.-Y.; Ojima, I. *J. Am. Chem. Soc.* **2005**, *127*, 17756.

(9) When the reaction of diyne **1a** was examined under an atmospheric pressure of argon without CO gas, cyclization did not occur but, rather, hydrosilylation to unsubstituted alkyne moiety proceeded.

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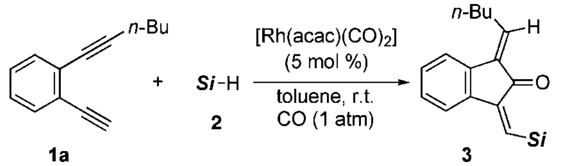
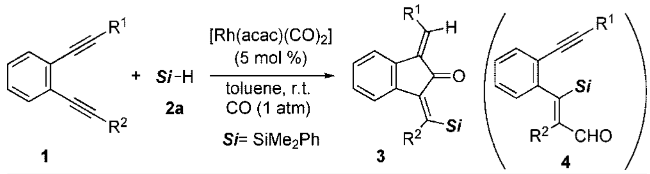
Table 1. Carbonylative Hydrosilylation with Several Silanes



entry[a]	*Si*−H	silane	time/h	yield/%
1	$Me_2PhSi-H$	**2a**	2	85 (**3aa**)
2	Ph_3Si-H	**2b**	4	73 (**3ab**)
3	Et_3Si-H	**2c**	10	73 (**3ac**)
4	$(EtO)_3Si-H$	**2d**	24	<30 (**3ad**)

[a] Diyne:silane = 1:1.

Table 2. Carbonylative Hydrosilylation of Aromatic 1,5-Diynes

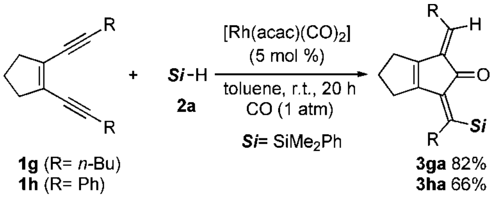


entry[a]	R^1	R^2	diyne	time/h	yield/%
1	Ph	H	**1b**	1	92 (**3ba**)
2	CH_2OH	H	**1c**	2	83 (**3ca**)
3	$CH_2N(Me)Ts$	H	**1d**	18	78 (**3da**)
4	*n*-Bu	*n*-Bu	**1e**	20	61 (**3ea**)
5[b]	Ph	Ph	**1f**	20	65 (**3fa**)

[a] Diyne:silane = 1:1. [b] The reaction was carried out at 40 °C.

and heteroatom-containing substituents, such as hydroxymethyl and (*N*-methyl-*N*-tosylamino)methyl groups, could also be the possible alkyne substituents of a viable substrate (entries 1–3). Diynes **1e** and **1f** with two substituted alkyne termini were also transformed into corresponding bicyclic compounds **3ea** and **3fa**, but the yield was lower than that of diynes with an unsubstituted alkyne terminus because the regioselectivity of the silylrhodation step to the alkyne moiety was not high and silylformylated compounds **4** were formed as byproduct (entries 4 and 5).

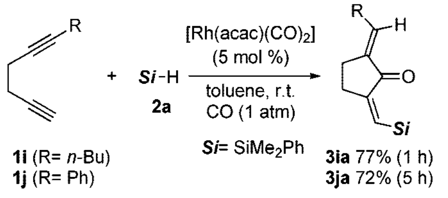
Not only aromatic 1,5-diynes, but olefin-tethered 1,5-diynes, namely conjugate enediynes **1g** and **1h**, were also appropriate substrates, and carbonylative hydrosilylation proceeded to give conjugate trienones **3ga** and **3ha** (eq 1). Even 1,5-diynes **1i** and **1j**, possessing a more flexible ethylene tether, were transformed into monocyclic dienones **3ia** and **3ja** in good yields (eq 2).



1g (R= *n*-Bu)
1h (R= Ph)

3ga 82%
3ha 66%

(1)



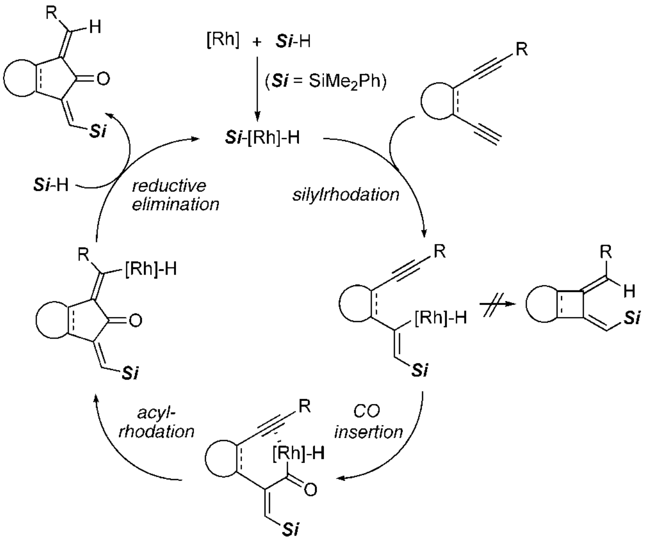
1i (R= *n*-Bu)
1j (R= Ph)

3ia 77% (1 h)
3ja 72% (5 h)

(2)

Scheme 2 shows a proposed mechanism of the present cyclization of 1,5-diynes. Silylrhodation to an unsubstituted alkyne moiety of diyne gives vinylrhodium intermediate.

Scheme 2. Proposed Mechanism of Carbonylative Silylcarbocyclization



Different from the reactivity of 1,6-diyne, the ring closure through carborhodation cannot proceed because this would result in the formation of a strained four-membered-ring system. Instead, a CO insertion provides an acylrhodium intermediate, and the subsequent intramolecular acylrhodation to another alkyne moiety makes a five-membered ring.

Conclusions

We have developed a rhodium-catalyzed hydrosilylative cyclization with carbonylation. Various 1,5-diynes, including aromatic-, olefin-, and ethylene-tethered 1,5-diynes, were transformed into 2,5-dialkylidenecyclopentanones in good to high yields. The present reaction can be recognized as a new member of the silylcarbocyclization and the synthetic utilities of these products are under investigation.

Experimental Section

General. Anhydrous toluene is commercially available. It was dried over molecular sieves 4A (MS 4A) and degassed by bubbling with CO before use. All reactions were examined under a CO atmosphere. IR spectra were recorded with a Horiba FT730 spectrophotometer. NMR spectra were measured with JEOL AL-400 and Lambda500 spectrometers, using tetramethylsilane as an internal standard and $CDCl_3$ as a solvent. Mass spectra were measured with a JEOL JMS-SX102A.

Typical Experimental Procedure. Under an atmosphere of carbon monoxide, $[Rh(acac)(CO)_2]$ (1.3 mg, 0.0050 mmol) was placed in a flask and a toluene solution (0.80 mL) of hydrosilane (0.10 mmol) was added. After the mixture was stirred for 5 min, a toluene solution (1.20 mL) of diyne (0.10 mmol) was added and the mixture was further stirred at room temperature. After completion of the diyne, the solvent was evaporated under reduced pressure, and the crude product was purified by thin-layer chromatography to give an analytically pure dienone.

(1*E*,3*Z*)-3-[(Dimethylphenylsilyl)methylidene]-1-pentylideneindan-2-one (3aa). Yellow oil. IR (CH_2Cl_2) 1718, 1641 cm^{-1}. ^{1}H NMR δ 0.55 (s, 6H), 0.95 (t, J = 7.3 Hz, 3H), 1.42–1.48 (m, 2H), 1.58–1.63 (m, 2H), 2.58–2.64 (m, 2H), 6.79 (t, J = 7.3 Hz, 1H), 6.85 (s, 1H), 7.30–7.37 (m, 5H), 7.59 (d, J = 7.3 Hz, 1H), 7.64–7.68 (m, 3H); ^{13}C NMR δ −2.2, 13.9, 22.6, 28.6, 30.9, 121.0, 124.4, 127.7, 128.3, 128.8, 129.6, 133.2, 133.3, 133.7, 133.8, 137.7, 138.3, 139.8, 147.5, 193.7. HRMS (FAB$^+$) for M found m/z 346.1772, calcd for $C_{23}H_{26}OSi$ 346.1753.

(1*E*,3*Z*)-1-Pentylidene-3-[(triphenylsilyl)methylidene]indan-2-one (3ab). Yellow solid. Mp 98 °C. IR (CH_2Cl_2) 1718, 1637, 1427 cm^{-1}. ^{1}H NMR δ 0.92 (t, $J = 7.4$ Hz, 3H), 1.38–1.43 (m, 2H), 1.53–1.57 (m, 2H), 2.55–2.57 (m, 2H), 6.65 (t, $J = 7.4$ Hz, 1H), 7.18 (s, 1H), 7.31–7.42 (m, 11H), 7.61–7.75 (m, 8H); ^{13}C NMR δ 13.8, 22.5, 28.6, 30.8, 121.5, 124.4, 127.7, 128.0, 128.3, 129.2, 130.1, 132.8, 135.3, 135.7, 138.1, 138.2, 138.3, 149.5, 192.3. HRMS (FAB$^+$) for M found *m/z* 470.2036, calcd for $C_{33}H_{30}OSi$ 470.2066.

(1*E*,3*Z*)-1-Pentylidene-3-[(triethylsilyl)methylidene]indan-2-one (3ac). Yellow oil. IR (CH_2Cl_2) 1720, 1641 cm^{-1}. ^{1}H NMR δ 0.83–0.99 (m, 18H), 1.44–1.49 (m, 2H), 1.61–1.64 (m, 2H), 2.60–2.66 (m, 2H), 6.72 (s, 1H), 6.80 (t, $J = 7.3$ Hz, 1H), 7.33–7.36 (m, 2H), 7.61–7.63 (m, 1H), 7.68–7.69 (m, 1H); ^{13}C NMR δ 3.3, 7.8, 13.9, 22.6, 28.6, 30.9, 120.8, 124.4, 128.2, 129.4, 133.2, 133.4, 137.4, 137.5, 138.6, 147.7, 194.0. HRMS (FAB$^+$) for M − 1 found *m/z* 325.1988, calcd for $C_{21}H_{29}OSi$ 325.1988.

(1*E*,3*Z*)-1-Benzylidene-3-[(dimethylphenylsilyl)methylidene]-indan-2-one (3ba). Yellow solid. Mp 80 °C. IR (CH_2Cl_2) 1716, 1628 cm^{-1}. ^{1}H NMR δ 0.60 (s, 6H), 6.90 (s, 1H), 7.13–7.17 (m, 1H), 7.27–7.31 (m, 1H), 7.38–7.44 (m, 6H), 7.55–7.62 (m, 4H), 7.64–7.70 (m, 3H); ^{13}C NMR δ −2.2, 121.1, 123.6, 127.8, 128.6, 128.8, 128.9, 129.1, 129.2, 129.3, 132.8, 133.0, 133.7, 133.8, 135.4, 136.5, 139.2, 139.6, 147.1, 194.3. HRMS (FAB$^+$) for M found *m/z* 366.1467, calcd for $C_{25}H_{22}OSi$ 366.1440.

(1*E*,3*Z*)-1-(2-Hydroxyethylidene)-3-[(dimethylphenylsilyl)methylidene]indan-2-one (3ca). Pale yellow solid. Mp 91 °C. IR (CH_2Cl_2) 3419, 1718 cm^{-1}. ^{1}H NMR δ 0.57 (s, 6H), 2.10 (m, 1H), 4.75 (d, $J = 5.6$ Hz, 2H), 6.83 (t, $J = 5.6$ Hz, 1H), 6.88 (s, 1H), 7.32–7.43 (m, 6H), 7.59–7.66 (m, 3H); ^{13}C NMR δ −2.3, 60.2, 121.1, 125.0, 127.8, 128.9, 129.1, 129.9, 132.9, 133.8, 134.2, 135.0, 136.6, 138.8, 139.4, 147.1, 193.4; HRMS (FAB$^+$) for M found *m/z* 320.1250, calcd for $C_{20}H_{20}O_2Si$ 320.1233.

(1*E*,3*Z*)-3-[(Dimethylphenylsilyl)methylidene]-1-[2-(*N*-methyl-*N*-tosylamino)ethylidene]indan-2-one (3da). Yellow solid. Mp 117 °C. IR CH_2Cl_2) 2360, 1722, 1644 cm^{-1}. ^{1}H NMR δ 0.55 (s, 6H), 2.44 (s, 3H), 2.83 (s, 3H), 4.30 (d, $J = 6.3$ Hz, 2H), 6.49 (t, $J = 6.3$ Hz, 1H), 6.89 (s, 1H), 7.30–7.40 (m, 7H), 7.52–7.55 (m, 1H), 7.61–7.70 (m, 5H); ^{13}C NMR δ −2.3, 21.5, 35.1, 48.3, 121.3, 125.0, 127.6, 127.8, 128.9, 129.1, 129.4, 129.8, 130.0, 133.7, 134.4, 134.5, 135.0, 136.1, 139.1, 139.3, 143.8, 146.8, 192.7. HRMS (FAB$^+$) for M − 1 found *m/z* 486.1554, calcd for $C_{28}H_{28}NO_3SSi$ 486.1559.

(1*Z*,3*E*)-1-[1-(Dimethylphenylsilyl)pentylidene]-3-pentylideneindan-2-one (3ea). Yellow oil. IR (CH_2Cl_2) 1734, 1712, 1641 cm^{-1}. ^{1}H NMR δ 0.54 (s, 6H), 0.87–0.95 (m, 6H), 1.35–1.60 (m, 8H), 2.56–2.61 (m, 2H), 2.72–2.76 (m, 2H), 6.70 (t, $J = 7.6$ Hz, 1H), 7.30–7.35 (m, 5H), 7.56–7.57 (m, 2H), 7.70–7.78 (m, 2H); ^{13}C NMR δ 0.0, 14.7, 14.9, 23.6, 24.1, 29.6, 30.5, 31.9, 34.3, 125.3, 126.1, 128.4, 129.1, 129.2, 129.3, 133.7, 134.6, 138.4, 139.3, 139.4, 141.4, 142.0, 158.3, 194.0. HRMS (FAB$^+$) for M + Na found *m/z* 425.2280, calcd for $C_{27}H_{34}ONaSi$ 425.2277.

(1*Z*,3*E*)-3-Benzylidene-1-[(dimethylphenylsilyl)phenylethylidene]indan-2-one (3fa). Yellow solid. Mp 109 °C. IR (CH_2Cl_2) 1714, 1624 cm^{-1}. ^{1}H NMR δ 0.35 (s, 6H), 6.21 (d, $J = 8.1$ Hz, 1H), 6.85–6.89 (m, 1H), 6.96–7.00 (m, 1H), 7.09 (d, $J = 7.3$ Hz, 2H), 7.30–7.43 (m, 9H), 7.53–7.55 (m, 3H), 7.62–7.66 (m, 3H); ^{13}C NMR δ 0.0, 124.0, 126.1, 126.2, 127.2, 128.2, 129.1, 129.4, 129.5, 129.6, 129.8, 130.0, 131.9, 133.3, 133.3, 134.5, 136.2, 138.2, 139.4, 139.7, 141.7, 144.6, 155.3, 194.8. HRMS (FAB$^+$) for M − 1 found *m/z* 441.1665, calcd for $C_{31}H_{25}OSi$ 441.1675.

(2*Z*,4*E*)-2-[1-(Dimethylphenylsilyl)pentylidene]-4-pentylidenebicyclo[3.3.0]-oct-1(5)-en-3-one (3ga). Yellow oil. IR (CH_2Cl_2) 2956, 1689 cm^{-1}. ^{1}H NMR δ 0.48 (s, 6H), 0.81 (t, $J = 7.0$ Hz, 3H), 0.89 (t, $J = 7.3$ Hz, 3H), 1.19–1.42 (m, 8H), 2.22–2.28 (m, 2H), 2.29–2.34 (m, 2H), 2.40–2.43 (m, 2H), 2.77 (t, $J = 7.5$ Hz, 2H), 2.86 (t, $J = 7.3$ Hz, 2H), 6.14 (t, $J = 7.7$ Hz, 1H), 7.28–7.31 (m, 3H), 7.54–7.56 (m, 2H); ^{13}C NMR δ 0.0, 15.1, 15.2, 23.7, 24.1, 27.3, 29.6, 31.1, 32.6, 32.9, 34.6, 35.1, 128.6, 129.4, 130.6, 134.8, 135.1, 141.7, 143.5, 147.2, 151.2, 151.5, 197.4. HRMS (FAB$^+$) for M − 1 found *m/z* 391.2441, calcd for $C_{26}H_{35}OSi$ 391.2457.

(2*Z*,4*E*)-2-Benzylidene-4-[(dimethylphenylsilyl)benzylidene]-bicyclo[3.3.0]-oct-1(5)-en-3-one (3ha). Yellow solid. Mp 102 °C. IR (CH_2Cl_2) 1716 cm^{-1}. ^{1}H NMR δ 0.34 (s, 6H), 1.54–1.78 (m, 2H), 1.84–1.91 (m, 2H), 2.54–2.58 (m, 2H), 7.00–7.02 (m, 2H), 7.10 (s, 1H), 7.21–7.40 (m, 11H), 7.63–7.64 (m, 2H); ^{13}C NMR δ 0.0, 26.7, 31.0, 32.5, 126.7, 126.8, 128.2, 128.3, 128.4, 129.0, 129.2, 129.3, 130.5, 133.6, 134.8, 136.7, 140.1, 142.8, 144.5, 146.5, 152.2, 156.3, 197.5. HRMS (FAB$^+$) for M found *m/z* 432.1909, calcd for $C_{30}H_{28}OSi$ 432.1909.

(2*E*,5*Z*)-5-[(Dimethylphenylsilyl)methylidene]-2-pentylidenecyclopentanone (3ia). Yellow oil. IR (CH_2Cl_2) 1702, 1643 cm^{-1}. ^{1}H NMR δ 0.47 (s, 6H), 0.91 (t, $J = 7.2$ Hz, 3H), 1.30–1.49 (m, 4H), 2.14–2.19 (m, 2H), 2.54–2.61 (m, 2H), 2.70–2.74 (m, 2H), 6.31 (s, 1H), 6.64 (t, $J = 7.6$ Hz, 1H), 7.33–7.36 (m, 3H), 7.59–7.61 (m, 2H); ^{13}C NMR δ −2.2, 13.8, 22.5, 23.5, 29.1, 29.7, 30.4, 127.6, 128.6, 133.7, 138.6, 138.7, 138.9, 140.3, 154.8, 194.9. HRMS (FAB$^+$) for M-1 found *m/z* 297.1693, calcd for $C_{19}H_{25}OSi$ 297.1675.

(2*E*,5*Z*)-2-Benzylidene-5-[(dimethylphenylsilyl)methylidene]-cyclopentanone (3ja). Yellow solid. Mp 91 °C. IR (CH_2Cl_2) 1691, 1616, 1599 cm^{-1}. ^{1}H NMR δ 0.52 (s, 6H), 2.82–2.86 (m, 2H), 3.01–3.05 (m, 2H), 6.42 (s, 1H), 7.27–7.47 (m, 7H), 7.54–7.56 (m, 2H), 7.62–7.64 (m, 2H); ^{13}C NMR δ −2.2, 26.3, 29.9, 127.6, 128.6, 128.7, 129.4, 130.6, 133.7, 134.3, 135.7, 137.9, 139.3, 140.2, 153.7, 195.8. HRMS (FAB$^+$) for M − 1 found *m/z* 317.1359, calcd for $C_{21}H_{21}OSi$ 317.1362.

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Supporting Information Available: ^{1}H and ^{13}C NMR spectra for compounds in the text. This material is available free of charge via the Internet at http://pubs.acs.org.

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