A New Synthesis of Trimethylsilyl-Substituted Enyne and (*Z*)-Enediyne Compounds

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Trimethylsilyl (TMS)-substituted enynes 9—11, 13—17 and (*Z*)-enediynes 18 were prepared by dehydration of the TMS-substituted propargyl alcohols 1—8 with polyphosphoric acid trimethylsilyl ester.

Key words enyne; (*Z*)-enediyne; polyphosphoric acid trimethylsilyl ester; trimethylsilylenyne

Recently, the anticancer and antibiotic activities shown by conjugate enediyne compounds has attracted the attention of organic chemists and pharmaceutical scientists.[1] General synthetic methods involve the Pd-catalyzed cross-coupling reactions of *sp* and sp^2 carbons.[2] We have already reported a convenient synthesis of *Z*-enynes and *Z*-enediynes by the dehydration reaction of propargyl alcohols using polyphosphoric acid trimethylsilyl ester (PPSE).[3] This dehydration reaction has been found to be strongly affected by the substituent on the acetylenic carbon of the propargyl alcohols. A reaction of sulfur-substituted propargyl alcohols and PPSE gave the alkenoate thioesters *via* the Meier–Schuster rearrangement in good yields.[4] We were interested in the substituent effects on the acetylenic carbon and examined the dehydration of silyl-substituted propargyl alcohols using PPSE. The acid-catalyzed dehydration of silyl-substituted propargyl alcohols has been reported to give the ynones *via* the Meier–Schuster rearrangement.[5] However, it is difficult to obtain the enyne silanes by the dehydration of silyl-substituted propargyl alcohols, as concomitant desilylation reactions occur. However, silyl acetylenes are good tools for the synthesis of terminal alkynes,[6] ynones[7] and the acetylenic sulfones.[8] If this method is applicable to the synthesis of the silyl-substituted enynes and (*Z*)-enediynes, it would provide convenient intermediates for the synthesis of enediyne analogs.

Trimethylsilyl (TMS)-substituted propargyl alcohols are prepared from the reactions of 1-(trimethylsilyl)acetylene/EtMgBr and aldehydes or ketones. First, we performed the reaction of cyclododecanol **1** and PPSE at 83 °C to give 1-(trimethylsilylethynyl)-1-cyclododecene (**9**) in 91% yield. Structural assignment of **9** was performed based on its IR, ^{1}H- and ^{13}C-NMR spectroscopies. The IR spectrum showed the disappearance of the hydroxy group and the ^{1}H-NMR spectrum showed the olefinic H at δ 5.36 (t, J=8 Hz). The cyclohexanol derivative **2** also gave the enyne silane **10** in good yield. The bulky alkynyl alcohol **3** afforded the enyne silane **11** accompanied by the ether **12** in 40% yield. The reaction of **3** and PPSE under diluted conditions gave the ether **12** in low yield and the enyne **11** was obtained in 63% yield. Methyl-substituted propargyl alcohols **4** and **6** gave the enynes **13** and **16**, respectively, accompanied by the *exo*-methylene derivatives **14** (41%) and **17** (17%). The stereochemistry of the products **13** and **16** was determined by means of difference Nuclear Overhauser effect (DNOE) experiments. Irradiation of the methyl protons substituted at the olefinic carbon of **13** and **16** increased the intensities of the olefinic proton signals. 3*H*,4*H*-Dihydronaphthalene **15** was obtained in good yield. The synthesis of a *Z*-enediyne compound also produced **18** *Z*-selectively; however, the propargyl alcohol **8** gave a complex mixture.

Plausible mechanisms are shown in Chart 2. The oxygen atom of the alcohol **19** attacks the phosphorus atom of PPSE to give the intermediate **21A**. The intermediate **21A** formed from the secondary alcohol (R^1=H) does not undergo dehydration of the alcohol **19** (R^1=H) because of the γ-substituent effect of the propargyl alcohols. γ-Silyl-substituted propargyl alcohols have been found to be very slow to undergo dehydration of the alcohols compared to γ-sulfur-substituted alcohols.[4] 1-(Phenylthioethynyl)cycloalkanols readily underwent dehydration by PPSE at room temperature to give the enyne sulfides and the alkenethioates; however, the dehydration of the silyl-substituted propargyl alcohol **2** at room temperature did not proceed and the alcohol was recovered. These results show that electron-donating substituents at the acetylenic carbon strongly affected the dehydration reactions of the alcohols. In other words, the carbon–oxygen bond of the γ-silyl-substituted intermediate **21A** would be more difficult to cleave than that of the γ-sulfur-substituted alcohols and the alkenyl silyl ketones **24** could not be obtained *via* the Meier–Schuster rearrangement. On the contrary, the phosphorus pentavalent intermediate **21A** ($R^1 \neq$H), formed from the tertiary alcohol, is cleaved more readily than intermediate **21A** (R^1=H) and gives the products in good yields. The dehydration of the alcohol would proceed *via* the 6-membered transition state **25A** and **25B**, which gives the (*Z*)- and (*E*)-enyne silanes, respectively. The (*Z*)-stereoselectivity of the products can be explained as follows: the dehydration of **21A**, in which the alkynyl groups of the alcohol lie on the side opposite to the bulky phosphorus moiety **25A**, would proceed and

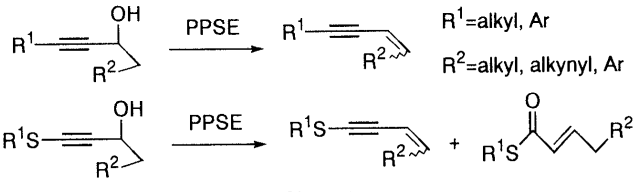

Chart 1

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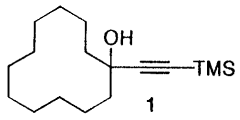
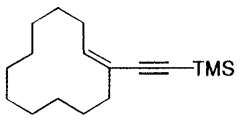
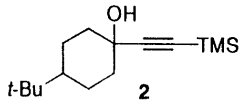
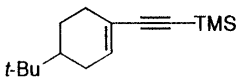
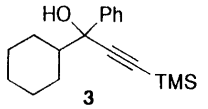
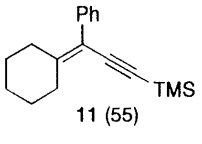
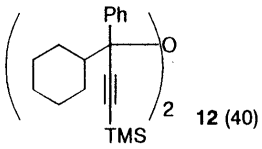
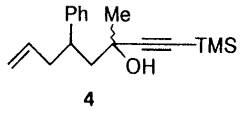
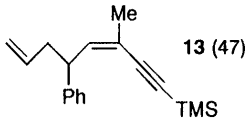
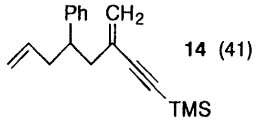
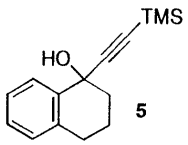
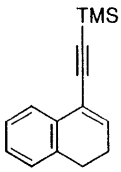
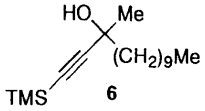
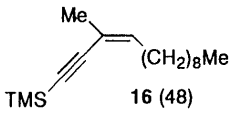
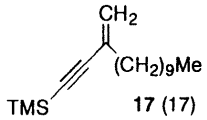
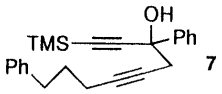
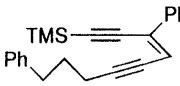
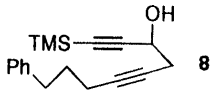
Table 1. Reaction of Alkynyl Alcohols with PPSE

Entry	Alkynyl alcohol	Products (% yields)
1	 **1**	 **9** (91)
2	 **2**	 **10** (60)
3	 **3**	 **11** (55)  **12** (40)
4	**3**	**11** (63) **12** (18)
5	 **4**	 **13** (47)  **14** (41)
6	 **5**	 **15** (92)
7	 **6**	 **16** (48)  **17** (17)
8	 **7**	 **18** (51)[a]
9	 **8**	—

a) $E:Z=1:4$.

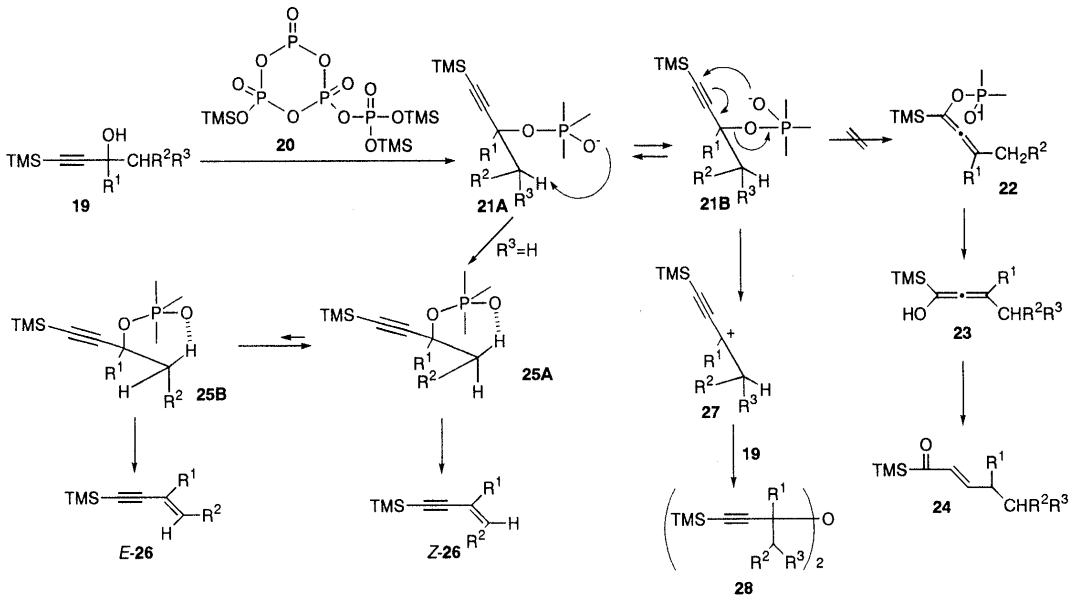


Chart 2

(*Z*)-selectively give the enediyne. The dehydration of the bulky cyclohexyl derivative (**21**A: R^2, $R^3=(CH_2)_5$) is difficult and the nucleophilic attack of another alcohol **19** gives the ether **28**. We are now examining the dehydration reactions of γ-alkoxy-substituted propargyl alcohols. These results will be reported elsewhere.

Experimental

Melting points were determined on a Yanagimoto micro melting point apparatus and are uncorrected. IR spectra of solids (KBr) and liquids (film) were recorded on a JASCO IRA-100 spectrophotometer. ^{1}H-NMR spectra were obtained for solutions in $CDCl_3$ on JEOL GX-270 and Varian Gemini-2000 spectrometers at the instrumentation center of Gifu University with tetramethylsilane as an internal standard, unless otherwise indicated. The ^{13}C spectra were run on JEOL GX-270 and Varian Gemini-2000 spectrometers. Mass spectra were recorded on a JEOL JMS-D300 spectrometer with a direct-insertion probe at 70 eV. Exact mass determination was done with a JMA 2000 on-line system.

1-(Trimethylsilylethynyl)cyclododecan-1-ol (1). Typical Procedure for Syntheses of Propargyl Alcohols An Et_2O (5 ml) solution of 1-(trimethylsilyl)acetylene (1.96 g, 20 mmol) was added to an EtMgBr solution (prepared from Mg (0.32 g, 13.0 mmol) and EtBr (1.42 g, 13.0 mmol) in 15 ml of Et_2O) at room temperature. The reaction mixture was refluxed for 0.5 h. An Et_2O (20 ml) solution of cyclododecanone (1.82 g, 10.0 mmol) was added dropwise to the mixture at 0 °C. The whole was added to water (100 ml) and extracted with ether. The extracts were combined, dried over $MgSO_4$ and evaporated under reduced pressure. The residue was purified by column chromatography on silica-gel with AcOEt : *n*-hexane (1 : 10). **1** (mp 114—116 °C) (2.38 g, 85%) was obtained as white needles.

1-(Trimethylsilylethynyl)cyclododecan-1-ol (**1**): IR (KBr) cm^{-1}: 3460 (OH), 2150 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.16 (9H, s, TMS), 1.35 (15H, br s, alkyl H), 1.61—1.70 (4H, m, alkyl H and OH), 1.78—1.88 (4H, m, alkyl H). *Anal.* Calcd for $C_{17}H_{32}OSi$: C, 72.79; H, 11.50. Found: C, 72.72; H, 11.68.

4-*tert*-Butyl-1-(trimethylsilylethynyl)cyclohexan-1-ol (**2**): mp 145—148 °C. IR (KBr) cm^{-1}: 3260 (OH), 2960, 2170 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.17 (9H, s, TMS), 0.87 (9H, s, *tert*-Bu), 1.34—1.53 (6H, m, alkyl H), 1.71—1.77 (2H, m, alkyl H and OH), 1.96—2.01 (2H, m, alkyl H). *Anal.* Calcd for $C_{15}H_{28}OSi$: C, 71.36; H, 11.18. Found: C, 71.25; H, 11.30.

1-Cyclohexyl-1-phenyl-3-(trimethylsilyl)prop-2-yn-1-ol (**3**): mp 71—78 °C. IR (KBr) cm^{-1}: 3330 (OH), 2160 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.23 (9H, s, TMS), 1.08—1.24 (4H, m, alkyl H), 1.40—1.48 (2H, m, alkyl H), 1.57—1.82 (3H, m, alkyl H), 1.93—1.98 (1H, m, alkyl H), 2.34 (1H, br s, OH), 7.25—7.36 (3H, m, ArH), 7.57—7.60 (2H, m, ArH). *Anal.* Calcd for $C_{18}H_{26}OSi$: C, 75.46; H, 9.15. Found: C, 75.42; H, 9.20.

3-Methyl-5-phenyl-1-(trimethylsilyl)-7-octen-1-yn-3-ol (**4**): IR (film) cm^{-1}: 3590, 3460 (OH), 2160 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.14 (s, TMS), 0.23 (s, TMS), 1.27 (s, Me), 1.43 (s, Me), 2.04—2.08 (m, alkyl H), 2.42—2.43 (m, alkyl H), 3.16—3.26 (m, alkyl H), 4.97—5.05 (m, olefinic H), 5.60—5.75 (m, olefinic H), 7.23—7.35 (5H, m, ArH). *Anal.* Calcd for $C_{18}H_{26}OSi$: C, 75.46; H, 9.15. Found: C, 75.20; H, 9.18.

1-(Trimethylsilylethynyl)-1,2,3,4-tetrahydronaphthalen-1-ol (**5**): IR (film) cm^{-1}: 3450 (OH), 2170 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.17 (9H, s, TMS), 2.15—2.20 (4H, m, alkyl H), 2.32 (1H, br s, OH), 2.80—2.82 (2H, m, alkyl H), 7.09 (1H, m, ArH), 7.20—7.24 (2H, m, ArH), 7.73—7.77 (1H, m, ArH). *Anal.* Calcd for $C_{15}H_{20}OSi$: C, 73.71; H, 8.25. Found: C, 73.58; H, 8.05.

3-Methyl-1-(trimethylsilyl)-1-tridecyn-3-ol (**6**): IR (film) cm^{-1}: 3400 (OH), 2170 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.16 (9H, s, TMS), 0.72 (3H, t, J=7 Hz, Me), 1.30 (16H, br s, alkyl H), 1.46 (3H, s, Me), 1.60—1.67 (2H, m, alkyl H). *Anal.* Calcd for $C_{17}H_{34}OSi$: C, 77.19; H, 12.19. Found: C, 77.27; H, 12.15.

3,9-Diphenyl-1-(trimethylsilyl)-1,5-nonadiyn-3-ol (**7**): IR (film) cm^{-1}: 3550, 3460 (OH), 2160 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.22 (9H, s, TMS), 1.67—1.84 (4H, m, alkyl H), 2.53 (2H, t, J=8 Hz, alkyl H), 2.84 (1H, br s, OH), 5.07 (2H, t, J=3 Hz, 4-CH_2), 7.06—7.36 (8H, m, ArH), 7.62—7.68 (2H, m, ArH). MS *m/z*: 286 (small M^+).

9-Phenyl-1-(trimethylsilyl)-1,5-nonadiyn-3-ol (**8**): IR (film) cm^{-1}: 3400 (OH), 2160 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.67 (9H, s, TMS), 1.77—1.88 (2H, m, alkyl H), 2.13—2.20 (3H, m, alkyl H), 2.59—2.75 (3H, m, alkyl H), 4.81 (1H, br s, OH), 4.96—4.99 (1H, m, alkyl H), 7.17—7.30 (5H, m, ArH). MS *m/z*: 284 (small M^+).

A Reaction of Propargyl Alcohol 1 with PPSE (Typical Procedure) A $ClCH_2CH_2Cl$ (3 ml) solution of **1** (0.28 g, 1.0 mmol) was added dropwise to a PPSE solution (prepared from P_2O_5 (0.9 g, 6.3 mmol) and hexamethyldisiloxane (4 ml) in $ClCH_2CH_2Cl$ (3 ml)) under an Ar atmosphere and the reaction mixture was refluxed for 0.5 h. It was then cooled to room temperature and poured into saturated $NaHCO_3$ solution. The organic layer was separated and the aqueous layer was extracted with $CHCl_3$. The organic layer and the extracts were combined and dried over $MgSO_4$. The solvent was removed under reduced pressure. The residue was purified by preparative TLC on silica-gel with hexane to afford (*Z*)-1-(trimethylsilylethynyl)-1-cyclodecene (**9**) (0.24 g, 91%) as a pale yellow oil. IR (film) cm^{-1}: 2120 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.02 (9H, s, TMS), 1.11—1.20 (12H, m, alkyl H), 1.36—1.40 (4H, m, alkyl H), 1.95—1.99 (2H, m, alkyl H), 2.02—2.16 (2H, m, alkyl H), 5.63 (1H, t, J=8 Hz, olefinic H). ^{13}C-NMR (67.5 MHz, $CDCl_3$) δ: 0.00 (q), 24.31 (t), 24.50 (t), 24.72 (t), 25.08 (t), 25.71 (t), 25.98 (t), 26.81 (t), 30.11 (t), 36.05 (t), 73.08 (s), 80.09 (s), 98.15 (s), 104.48 (s), 122.36 (s), 141.08 (d). *Anal.* Calcd for $C_{17}H_{30}Si$: C, 77.78; H, 11.52. Found: C, 77.96; H, 11.62.

4-*tert*-Butyl-1-(trimethylsilylethynyl)-1-cyclohexene (**10**): IR (film) cm^{-1}: 2150 (acetylene). ^{1}H-NMR (200 MHz, $CDCl_3$) δ: 0.19 (9H, s, TMS), 0.86—0.87 (9H, m, *tert*-Bu), 1.09—1.31 (2H, m, alkyl H), 1.73—1.90 (2H, m, alkyl H), 2.04 (1H, m, alkyl H), 2.08—2.18 (2H, m, alkyl H), 6.18 (1H, br s, olefinic H). MS *m/z*: 234 (M^+).

[1-Phenyl-3-(trimethylsilyl)-2-propynylidene]cyclohexane (**11**): IR (film) cm^{-1}: 2140 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.33 (9H, s, TMS), 1.68—1.86 (6H, m, alkyl H), 2.37 (2H, br t, J=6 Hz, alkyl H), 2.81 (2H, br t, J=6 Hz, alkyl H), 7.35 (5H, m, ArH). ^{13}C-NMR (67.5 MHz, $CDCl_3$) δ: 0.00 (q), 26.29 (t), 27.69 (t), 27.94 (t), 31.10 (t), 33.94 (t), 79.98 (s), 96.63 (s), 105.41 (s), 116.23 (s), 126.55 (d), 127.84 (d), 127.97 (d), 128.99 (d), 129.10 (d), 138.78 (s), 151.62 (s), 151.97 (s). *Anal.* Calcd for $C_{18}H_{24}Si$: C, 80.53; H, 9.01. Found: C, 80.78; H, 8.57.

3,3′-Oxybis[cyclohexyl-3-phenyl-1-(trimethylsilyl)propyne] (**12**): IR (film) cm^{-1}: 2180 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.22 (18H, s, TMS), 1.17 (6H, m, alkyl H), 1.62—1.71 (14H, m, alkyl H), 2.30—2.33 (2H, m, alkyl H), 2.68—2.70 (2H, m, alkyl H), 7.23—7.35 (8H, m, ArH), 7.61—7.64 (2H, m, ArH). ^{13}C-NMR (67.5 MHz, $CDCl_3$) δ: 0.00 (q), 26.22 (t), 26.39 (t), 26.46 (t), 27.91 (t), 28.11 (t), 28.37 (t), 28.46 (t), 31.19 (t), 34.12 (t), 47.05 (s), 50.77 (d), 84.40 (s), 87.57 (s), 92.36 (s), 106.64 (s), 115.94 (s), 126.46 (d), 126.70 (d), 127.29 (d), 127.65 (d), 127.76 (d), 129.27 (d), 139.26 (s), 141.50 (s), 149.95 (s). MS *m/z*: 530 (M^+-O), 269 ($M^+-O/2$).

(*Z*)-3-Methyl-5-phenyl-1-(trimethylsilyl)-3,7-octadien-1-yne (**13**) and 4-Phenyl-2-(trimethylsilylethynyl)-1,6-heptadiene (**14**): IR (film) cm^{-1}: 2150 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.22 (s, TMS), 0.24 (s, TMS), 1.83 (s, Me), 1.84 (s, Me), 2.22—2.53 (m, alkyl H), 2.98—3.10 (m, **14**-benzyl H), 3.96—4.00 (m, **13**-benzyl H), 4.93—5.09 (m, **13**- and **14**-olefinic H), 5.10 (br s, **14**-olefinic H), 5.32 (br s, **14**-olefinic H), 5.63—5.76 (m, **13**- and **14**-olefinic H), 5.81 (dd, J=1, 9 Hz, **13**-4-H), 7.16—7.33 (5H, m, ArH). ^{13}C-NMR (67.5 MHz, $CDCl_3$) δ: −0.07 (q), 0.00 (q), 22.68 (q × 2), 39.84 (t), 40.01 (t), 43.51 (t), 44.04 (d), 46.61 (d), 94.32 (s), 97.93 (s), 104.73 (s), 105.45 (s), 115.96 (t), 116.07 (t), 123.46 (t), 126.10 (d), 126.13 (d), 127.42 (d), 127.73 (d), 128.11 (d), 128.35 (d), 128.48 (s), 129.85 (s), 136.27 (d), 136.55 (d), 141.92 (d), 143.96 (s), 144.14 (s). MS *m/z*: 268 (M^+). The yields of the products **13** and **14** were determined from the intensities of olefinic H in the ^{1}H-NMR spectrum.

1-(Trimethylsilylethynyl)-3,4-dihydronaphthalene (**15**): IR (film) cm^{-1}: 2150 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.23 (9H, s, TMS), 2.24—2.32 (2H, m, alkyl H), 2.70 (2H, br t, J=8 Hz, alkyl H), 6.44 (1H, t, J=5 Hz, alkyl H), 7.00—7.20 (3H, m, ArH), 7.53 (1H, br d, J=7 Hz, ArH). *Anal.* Calcd for $C_{15}H_{18}Si$: C, 79.58; H, 8.01. Found: C, 79.73; H, 7.87.

(*Z*)-3-Methyl-1-(trimethylsilyl)-3-tridecen-1-yne (**16**) and 2-(Trimethylsilylethynyl)-1-dodecene (**17**): IR (film) cm^{-1}: 2150 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.21 (s, **17**-TMS), 0.22 (s, **16**-TMS), 0.88—0.93 (t, J=7 Hz, **16**- and **17**-Me), 1.03 (m, alkyl H), 1.84 (d, J=2 Hz, **16**-Me), 2.13 (br t, J=7 Hz, **17**-CH_2), 2.23—2.28 (m, **16**-CH_2), 5.24 (br s, **17**-olefinic H), 5.36 (br s, **17**-olefinic H), 5.72 (dt, J=1, 7 Hz, **16**-olefinic H). ^{13}C-NMR (67.5 MHz, $CDCl_3$) δ: −0.13 (q), 0.00 (q), 13.99 (q), 22.61 (q), 22.66 (t), 27.89 (t), 28.93 (t), 28.99 (t), 29.17 (t), 29.28 (t), 29.36 (t), 29.39 (t), 29.54 (t), 30.55 (t), 31.85 (t), 36.91 (t), 80.16

(s), 93.49 (s), 97.00 (s), 104.83 (s), 105.76 (s), 117.79 (s), 121.51 (t), 131.91 (s), 139.57 (d). MS *m/z*: 264 (M^+). The yields of the products **16** and **17** were determined from the intensities of olefinic H in the ^{1}H-NMR spectrum.

(*Z*)-3,9-Diphenyl-1-(trimethylsilyl)-3-nonen-1,5-diyne (*Z*-**18**): IR (film) cm^{-1}: 2200, 2140 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.24 (9H, s, TMS), 1.87—1.98 (2H, m, alkyl H), 2.44—2.50 (2H, dt, $J=2$, 7 Hz, alkyl H), 2.81 (2H, t, $J=7$ Hz, alkyl H), 6.32 (1H, t, $J=2$ Hz, olefinic H), 7.18—7.37 (8H, m, ArH), 7.59—7.62 (2H, m, ArH). ^{13}C-NMR (67.5 MHz, $CDCl_3$) δ: 0.00 (q), 19.40 (t), 30.35 (t), 34.82 (t), 80.18 (s), 99.73 (s), 115.48 (s), 115.52 (s), 115.57 (s), 125.95 (d), 128.35 (d), 128.46 (d), 128.61 (d), 132.00 (s), 136.71 (s), 141.53 (s), 148.90 (s). High-resolution mass Calcd for $C_{24}H_{26}Si$: 342.1804. Found: 342.1792.

(*E*)-3,9-Diphenyl-1-(trimethylsilyl)-3-nonen-1,5-diyne (*E*-**18**): IR (film) cm^{-1}: 2230 (acetylene). ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 0.21 (9H, s, TMS), 2.01—2.16 (2H, m, alkyl H), 2.67—2.75 (4H, m, alkyl H), 7.15—7.34 (9H, m, ArH and olefinic H), 7.70—7.74 (2H, m, ArH). High-resolution mass Calcd for $C_{24}H_{26}Si$: 342.1803. Found: 342.1813.

References and Notes

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