Preparation of CF₃-Containing 1,3-Di- and 1,1,3-Trisubstituted Allenes

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General Methods. Unless otherwise noted, materials were obtained from commercial suppliers and were used without further purification. All manipulations involving air-sensitive materials were performed under argon, with such materials being exposed only to thoroughly dried and degassed solvents. Anhydrous ether, THF, and CH₂Cl₂ were purchased from commercial supplier and were used without further purification.

¹H and ¹³C NMR spectra were recorded in CDCl₃ (300 and 75 MHz, respectively) unless otherwise noted and chemical shifts were reported in parts per million (ppm) downfield from internal tetramethylsilane (Me₄Si). Data were tabulated in the following order: multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; quint, quintet; sex, sextet; m, multiplet; br, broad peak), number of protons, coupling constants (in hertz). Infrared (IR) spectra were obtained as thin films on NaCl plates or KBr pelets, and all spectra were reported in wave numbers (cm⁻¹). Melting points, mp, were measured without correction. Column chromatography was conducted with silica gel (Silica gel 60, spherical, 100~210 μm) by using a mixture of *n*-hexane and AcOEt (v/v).

4,4,4-trifluoro-1-(1-naphthyl)but-2-yn-1-ol (2a): Yield 68%. R_f=0.36 (Hexane:AcOEt=4:1). mp 100~101 °C. ¹H NMR δ 2.44 (1H, br), 6.23 (1H, d, *J*=2.7 Hz), 7.48~7.64 (3H, m), 7.78 (1H, d, *J*=6.6 Hz), 7.90~7.93 (2H, m), 8.19 (1H, d, *J*=8.7 Hz). ¹³C NMR δ 65.3, 74.0 (q, *J*=52.8 Hz), 86.3 (q, *J*=6.5 Hz), 114.1 (q, *J*=257.4 Hz), 123.2, 125.0, 125.2, 126.3, 127.0, 128.9, 130.2, 130.3, 132.9, 134.0. IR (neat) ν 660, 740, 960, 1020, 1040, 1150, 1220, 1280, 1520, 2250, 3000 cm⁻¹. Anal. Calcd for C₁₄H₉F₃O: C, 67.20; H, 3.63. Found: C, 66.83; H, 3.87.

6,6,6-Trifluoro-1-phenylhex-4-yn-3-ol[#] (2b): Yield 89%. R_f=0.49 (Hexane: AcOEt=4:1). ¹H NMR δ 1.98~2.16 (2H, m), 2.37 (1H, br), 2.76 (2H, t, *J*=7.5 Hz), 4.38 (1H, sex, *J*=3.2 Hz), 7.14~7.32 (5H, m). ¹³C NMR δ 30.9, 38.0, 60.8, 72.2 (q, *J*=52.6 Hz), 87.8 (q, *J*=6.4 Hz), 114.0 (q, *J*=270.1 Hz), 126.3, 128.4, 128.6, 140.3. IR (neat) ν 600, 700, 750, 820, 860, 940, 1030, 1130, 1220, 1260, 1360, 1430, 1500, 1600, 1640, 1740, 2950, 3050 cm⁻¹.

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1,1,1-Trifluoro-2-hexadecyn-4-ol (2c): Yield 98%. R_f=0.47 (Hexane: AcOEt=6:1). ¹H NMR δ 0.859 (3H, t, *J*=6.7 Hz), 1.20~1.35 (20H, m), 1.72~1.80 (2H, m), 3.10 (1H, br), 4.41 (1H, m). ¹³C NMR δ 14.1, 22.7, 24.9, 29.1, 29.4, 29.4, 29.5, 29.6, 29.6, 29.7, 31.9, 36.7, 61.6, 70.8 (q, *J*=52.4 Hz), 88.2 (q, *J*=6.4 Hz), 109.0 (q, *J*=256.1

Hz). IR (neat) ν 710, 1020, 1130, 1260, 1440, 2225, 2825, 2900, 3050 cm^{-1} . Anal. Calcd for $\text{C}_{16}\text{H}_{27}\text{F}_3\text{O}$: C, 65.73; H, 9.31. Found: C, 65.77; H, 9.55.

1-(4-Bromophenyl)-4,4,4-trifluorobut-2-yn-1-ol (2d): Yield 85%. $R_f=0.45$ (Hexane:AcOEt= 4:1). mp 62~63 °C. ^1H NMR δ 2.68 (1H, br), 5.52 (1H, s), 7.35~7.42 (2H, m), 7.53~7.56 (2H, m). ^{13}C NMR δ 63.1, 73.4 (q, $J=53.2$ Hz), 86.0 (q, $J=6.5$ Hz), 113.9 (q, $J=257.5$ Hz), 123.2, 128.2, 130.5, 132.0, 132.3, 136.8. IR (neat) ν 600, 780, 800, 840, 980, 1010, 1040, 1070, 1130, 1260, 1400, 1480, 1680, 1700, 1900, 2250, 2850, 3300 cm^{-1} . Because this material is unstable and partial decomposition was noticed both by chromatography and by distillation, this material was used for the next step without further purification.

4,4,4-Trifluoro-1-(2-methoxyphenyl)but-2-yn-1-ol (2e): Yield 90%. $R_f=0.33$ (Hexane:AcOEt=4:1). ^1H NMR δ 3.43 (1H, d, $J=7.5$ Hz), 3.88 (3H, s), 5.37 (1H, qd, $J=3.0, 7.1$ Hz), 6.84~7.05 (2H, m), 7.27~7.44 (2H, m). ^{13}C NMR δ 55.5, 60.8, 71.0 (q, $J=52.6$ Hz), 86.7 (q, $J=6.78$ Hz), 109.0 (q, $J=256.8$ Hz), 111.2, 121.1, 126.3, 127.8, 130.5, 156.7. IR (neat) ν 640, 740, 820, 920, 960, 1020, 1110, 1220, 1370, 1420, 1440, 1480, 1580, 2200, 2800, 2900, 3250 cm^{-1} . Anal. Calcd for $\text{C}_{11}\text{H}_9\text{F}_3\text{O}_2$: C, 57.40; H, 3.94. Found: C, 56.95; H, 3.87.

5,5,5-Trifluoro-2-phenyl-3-pentyn-2-ol[#] (2f): Yield 90%. $R_f=0.31$ (Hexane:AcOEt=10:1). ^1H NMR δ 1.80 (3H, s), 2.98 (1H, s), 7.23~7.46 (3H, m), 7.55~7.58 (2H, m). ^{13}C NMR δ 32.1, 69.6, 70.9 (q, $J=53.9$ Hz), 89.9 (q, $J=6.5$ Hz), 109.1 (q, $J=257.1$ Hz), 124.6, 128.4, 128.4, 128.6, 128.7, 143.9.

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4,4,4-Trifluoro-2-iodo-1-(1-naphthyl)but-2-en-1-ol (3a): Yield 81%. $R_f=0.59$ (Hexane:Ether=3:1, three times). mp 117~118 °C. ^1H NMR δ 2.61 (1H, d, $J=4.8$ Hz), 6.70 (1H, m), 7.01 (1H, dq, $J=1.8, 7.5$ Hz), 7.48~7.63 (4H, m), 7.89~7.93 (2H, m), 8.08 (1H, d, $J=8.4$ Hz). ^{13}C NMR δ 62.3, 114.1 (q, $J=255.9$ Hz), 118.5 (q, $J=5.9$ Hz), 124.5 (q, $J=35.7$ Hz), 123.2, 125.0, 125.2, 126.3, 126.9, 128.9, 130.1, 130.2, 132.6, 134.0. IR (KBr) ν 695, 800, 840, 862, 1710, 1965, 3025 cm^{-1} . Anal. Calcd for $\text{C}_{14}\text{H}_{10}\text{F}_3\text{IO}$: C, 44.47; H, 2.67. Found: C, 44.76; H, 2.66.

(Z)-6,6,6-Trifluoro-4-iodo-1-phenylhex-4-en-3-ol (3b): Yield 99%. $R_f=0.42$ (Hexane:AcOEt= 4:1). ^1H NMR δ 1.85~2.15 (2H, m), 2.33 (1H, br), 2.61~2.84 (2H, m), 3.93~3.97 (1H, m), 6.64 (1H, dq, $J=1.2, 7.5$ Hz), 7.14~7.32 (5H, m). ^{13}C NMR δ 31.0, 37.2, 78.0, 116.3 (q, $J=270.8$ Hz), 120.9 (q, $J=6.0$ Hz), 124.1 (q, $J=35.8$ Hz), 126.2, 128.4, 128.6, 140.6. IR (neat) ν 620, 690, 720, 730, 800, 840, 860, 900, 1040,

1080, 1100, 1240, 1420, 1480, 1590, 1620, 1790, 1930, 2800, 2875, 3000, 3320 cm^{-1} .
Anal. Calcd for $\text{C}_{12}\text{H}_{12}\text{F}_3\text{IO}$: C, 40.47; H, 3.40. Found: C, 40.12; H, 3.85.

(Z)-1,1,1-Trifluoro-3-iodohexadec-2-en-4-ol (3c): Yield 84%. $R_f=0.40$ (Hexane:AcOEt=6:1). ^1H NMR δ 0.881 (3H, t, $J=6.8$ Hz), 1.23~1.43 (20H, m), 1.50~1.66 (1H, m), 1.68~1.82 (1H, m), 2.48 (1H, d, $J=4.2$ Hz), 3.96 (1H, br), 6.70 (1H, dq, $J=1.5, 6.0$ Hz). ^{13}C NMR δ 14.1, 22.7, 24.9, 29.2, 29.3, 29.4, 29.5, 29.6, 29.6, 29.6, 31.9, 35.9, 78.9, 116.3 (q, $J=270.9$ Hz), 121.6 (q, $J=6.0$ Hz), 123.8 (q, $J=35.6$ Hz). IR (neat) ν 620, 810, 850, 870, 1120, 1250, 1340, 1440, 1630, 2025, 2900, 3300 cm^{-1} .
Anal. Calcd for $\text{C}_{16}\text{H}_{28}\text{F}_3\text{IO}$: C, 45.72; H, 6.71. Found: C, 45.80; H, 6.54.

(Z)-1-(4-Bromophenyl)-4,4,4-trifluoro-2-iodobut-2-en-1-ol (3d): Yield 85%. $R_f=0.46$ (Hexane: AcOEt=4:1). ^1H NMR δ 3.38 (1H, br), 5.17 (1H, t, $J=1.5$ Hz), 6.97 (1H, dq, $J=1.5, 7.5$ Hz), 7.21~7.26 (2H, m), 7.49~7.54 (2H, m). ^{13}C NMR δ 80.5, 118.2 (q, $J=6.2$ Hz), 116.4 (q, $J=270.6$ Hz), 113.0, 124.2 (q, $J=36.1$ Hz), 128.9, 131.9, 138.2. IR (neat) ν 600, 620, 660, 710, 720, 780, 820, 870, 1000, 1050, 1100, 1250, 1360, 1390, 1470, 1580, 1630, 1680, 1980, 2800, 3000, 3300 cm^{-1} . Anal. Calcd for $\text{C}_{10}\text{H}_7\text{BrF}_3\text{IO}$: C, 29.51; H, 1.73. Found: C, 29.30; H, 1.80.

(Z)-4,4,4-Trifluoro-2-iodo-1-(2-methoxyphenyl)but-2-en-1-ol (3e): Yield 81%. $R_f=0.56$ (Hexane:AcOEt=4:1). mp=55~56 $^{\circ}\text{C}$. ^1H NMR δ 3.14 (1H, s), 3.86 (3H, s), 5.48 (1H, br), 6.85~7.03 (3H, m), 7.25~7.37 (2H, m). ^{13}C NMR δ 55.5, 77.1, 111.2, 117.8 (q, $J=5.6$ Hz), 120.2 (q, $J=270.5$ Hz), 121.0, 124.1 (q, $J=36.1$ Hz), 128.9, 130.1, 156.9. IR (neat) ν 600, 740, 800, 840, 860, 920, 980, 1010, 1030, 1100, 1220, 1360, 1420, 1440, 1470, 1570, 1580, 1620, 1780, 2775, 2850, 3250 cm^{-1} . Anal. Calcd for $\text{C}_{11}\text{H}_{10}\text{F}_3\text{IO}_2$: C, 36.89; H, 2.81. Found: C, 36.66; H, 2.81.

(Z)-5,5,5-Trifluoro-3-iodo-2-phenylpent-3-en-1-ol (3f): Yield 72%. $R_f=0.18$ (Hexane: AcOEt=20:1). ^1H NMR δ 1.86 (3H, s), 2.61 (1H, s), 6.89 (1H, q, $J=7.2$ Hz), 7.28~7.46 (5H, m). ^{13}C NMR δ 27.9, 79.3, 116.4 (q, $J=270.9$ Hz), 124.3 (q, $J=35.8$ Hz), 125.7, 125.8 (q, $J=5.6$ Hz), 128.3, 128.6, 143.3. IR (neat) ν 560, 670, 690, 750, 810, 860, 890, 940, 1020, 1080, 1200, 1250, 1360, 1430, 1480, 1610, 1710, 1870, 1950, 2900, 2950, 3000, 3350 cm^{-1} . Anal. Calcd for $\text{C}_{11}\text{H}_{10}\text{F}_3\text{IO}$: C, 38.62; H, 2.95. Found: C, 38.70; H, 3.0.

(Z)-4,4,4-Trifluoro-2-iodo-1-(1-naphthyl)-2-butenyl trifluoroacetate (4a):
To a CH_2Cl_2 (5 mL) solution of 4,4,4-trifluoro-2-iodo-1-(1-naphthyl)but-2-en-1-ol (**3a**, 0.7562 g, 2.00 mmol) at 0 $^{\circ}\text{C}$ were successively added trifluoroacetic anhydride (0.42 mL, 3.0 mmol) and triethylamine (0.41 mL, 3.0 mmol), and the whole mixture was stirred at the same temperature for 5 h. After quenching the reaction by the addition of

H₂O, the usual workup and chromatographic separation afforded 0.9303 g (1.96 mmol) of the desired **4a**. Yield 98%. R_f=0.65 (Hexane:AcOEt=4:1). ¹H NMR δ 6.72 (1H, dq, *J*=1.5, 7.2 Hz), 7.24 (1H, t, *J*=1.5 Hz), 7.44~7.67 (4H, m), 7.87~8.00 (3H, m). ¹³C NMR δ 81.2, 109.0 (q, *J*=6.0 Hz), 114.4 (q, *J*=285.4 Hz), 121.3 (q, *J*=271.5 Hz), 122.5, 125.2, 126.5, 127.0, 127.5, 128.4 (q, *J*=36.5 Hz), 129.0, 129.3, 130.4, 131.2, 134.0, 155.8 (q, *J*=43.2 Hz). IR (neat) ν 560, 600, 620, 640, 700, 720, 760, 780, 840, 890, 920, 1000, 1020, 1040, 1100, 1200, 1250, 1280, 1340, 1500, 1580, 1620, 1770, 1940, 3000, 3700 cm⁻¹.

(Z)-4,4,4-Trifluoro-2-iodo-1-(1-naphthyl)-2-butenyl acetate (5a): The same procedure of the preparation of **4a** was employed except for the usage of acetyl chloride (0.21 mL, 3.0 mmol) instead of trifluoroacetic anhydride. After quenching the reaction by the addition of H₂O, the usual workup and chromatographic separation afforded 0.810 g (1.93 mmol) of the desired **5a**. Yield 96%. R_f=0.47 (Hexane:AcOEt=4:1). ¹H NMR δ 2.18 (3H, s), 6.66 (1H, dq, *J*=1.8, 7.2 Hz), 7.15 (1H, br), 7.48 (4H, m), 7.89 (2H, d, *J*=7.8 Hz), 7.99 (1H, d, *J*=8.4 Hz). ¹³C NMR δ 20.8, 77.6, 112.4 (q, *J*=6.0 Hz), 116.7 (q, *J*=251.9 Hz), 123.1, 125.2, 126.1 (q, *J*=36.1 Hz), 126.2, 126.8, 127.0, 129.0, 130.2, 130.9, 131.3, 133.9, 169.0. IR (neat) ν 620, 720, 760, 790, 850, 870, 910, 940, 1000, 1110, 1190, 1250, 1280, 1350, 1400, 1500, 1580, 1620, 1720, 1920, 2850, 3000 cm⁻¹.

(Z)-1,1,1-Trifluoro-3-iodo-4-(1-naphthyl)-4-(2-tetrahydropyranyloxy)but-2-ene (6a): To an Et₂O (5 mL) solution of 4,4,4-trifluoro-2-iodo-1-(1-naphthyl)but-2-en-1-ol (**3a**, 0.378 g, 1.00 mmol) and 3,4-dihydro-2*H*-pyran (0.14 mL, 1.5 mmol) at 0 °C was added a catalytic amount of *p*-toluenesulfonic acid monohydrate (1~1.5 mol%) and the whole mixture was stirred at room temperature overnight. After quenching the reaction by the addition of 5% NaHCO₃ aq, the usual workup and chromatographic separation afforded 0.4276 g (0.925 mmol) of the desired **6a** as a diastereomer mixture. Yield 93%. R_f=0.60 (Hexane:AcOEt=4:1). ¹H NMR δ 1.36~1.66 (4H, m), 1.68~1.96 (2H, m), 3.35~3.69 (1H, m), 3.70~4.06 (1H, m), 4.48 and 4.98 (1H, t, *J*=3.3 and 3.0 Hz), 6.00 and 6.05 (1H, m), 7.01 and 7.04 (1H, dq, *J*=1.5, 7.2 and 2.0, 7.4 Hz), 7.46~7.53 (3H, m), 7.57 and 7.66 (1H, dd, *J*=1.5, 5.1 and 1.2, 7.4 Hz), 7.88 (2H, m), 8.16 (1H, t, *J*=9.3 Hz). ¹³C NMR δ 18.5, 19.0, 25.1, 25.3, 30.2 (2C), 62.0, 62.5, 80.2, 80.3, 95.4, 97.1, 115.6 (q, *J*=6.0 Hz), 117.5 (q, *J*=5.8 Hz), 122.1 (q, *J*=271.2 Hz), 121.8 (q, *J*=273.0 Hz), 123.2, 123.6, 125.2, 125.2 (q, *J*=35.8 Hz), 125.3, 125.7, 125.9 (q, *J*=35.5 Hz), 125.9, 126.4 (2C), 128.0, 128.8, 128.9, 129.4, 129.7, 131.0,

131.7, 132.7, 133.3, 133.8, 133.9. IR (neat) ν 620, 650, 700, 730, 780, 810, 860, 900, 950, 1050, 1100, 1240, 1350, 1380, 1430, 1500, 1590, 1630, 1800, 1920, 2850, 2900, 3050 cm^{-1} .

(Z)-6,6,6-Trifluoro-4-iodo-1-phenylhex-4-en-3-yl methanesulfonate (7b):

The same procedure of the preparation of **4a** was employed except for the usage of methanesulfonyl chloride (0.12 mL, 1.5 mmol for 1 mmol of **3b**) instead of trifluoroacetic anhydride. After quenching the reaction by the addition of H_2O , the usual workup and chromatographic separation afforded 0.401 g (0.923 mmol) of the desired **7b**. Yield 92%. $R_f=0.28$ (Hexane:AcOEt=4:1). ^1H NMR δ 1.99~2.22 (2H, m), 2.44 (3H, s), 2.54~2.70 (2H, m), 4.71 (1H, t, $J=6.3$ Hz), 6.40 (1H, q, $J=7.1$ Hz), 7.11~7.14 (2H, m), 7.18~7.37 (5H, m), 7.70~7.79 (2H, m). ^{13}C NMR δ 21.6, 30.3, 36.0, 85.2, 111.9 (q, $J=6.0$ Hz), 120.9 (q, $J=271.3$ Hz), 126.4, 127.9 (q, $J=36.3$ Hz), 128.0, 128.3, 128.6, 129.9, 133.4, 139.5, 145.6. IR (neat) ν 670, 740, 920, 1150, 1180, 1210, 1270, 1350, 3025 cm^{-1} .

(Z)-6,6,6-Trifluoro-4-iodo-1-phenylhex-4-en-3-yl *p*-toluenesulfonate (8b):

The same procedure of the preparation of **7b** was employed except for the usage of *p*-toluenesulfonyl chloride (0.286 g, 1.50 mmol for 1 mmol of **3b**) instead of methanesulfonyl chloride. After quenching the reaction by the addition of H_2O , the usual workup and chromatographic separation afforded 0.134 g (0.263 mmol) of the desired **8b**. Yield 26%. $R_f=0.29$ (Hexane:AcOEt=8:1). ^1H NMR δ 2.03~2.30 (2H, m), 2.69 (2H, t, $J=7.8$ Hz), 3.01 (3H, s), 4.79 (1H, t, $J=6.5$ Hz), 6.75 (1H, dq, $J=0.9, 6.9$ Hz), 7.12 (2H, d, $J=6.9$ Hz), 7.18~7.34 (5H, m), 7.75 (2H, d, $J=8.4$ Hz). ^{13}C NMR δ 30.4, 36.0, 39.4, 84.5, 112.7 (q, $J=5.8$ Hz), 121.0 (q, $J=271.5$ Hz), 126.5, 128.3, 128.7, 128.7 (q, $J=36.1$ Hz), 139.3. IR (neat) ν 620, 660, 700, 730, 810, 900, 960, 1000, 1040, 1100, 1120, 1260, 1350, 1440, 1500, 1600, 1690, 2950, 3000 cm^{-1} .

4,4,4-Trifluoro-1-(1-naphthyl)-2,3-butadiene (9a): Yield 89%. $R_f=0.59$ (Hexane:AcOEt=8:1). ^1H NMR δ 5.91 (1H, quint, $J=6.0$ Hz), 7.33 (1H, qd, $J=3.9, 7.8$ Hz), 7.41~7.64 (4H, m), 7.75~7.92 (2H, m), 8.09 (1H, d, $J=8.1$ Hz). ^{13}C NMR δ 88.6 (q, $J=39.0$ Hz), 98.1, 122.6 (q, $J=268.9$ Hz), 123.2, 125.6, 126.1, 126.6, 126.7, 127.1, 128.8, 129.2, 130.7, 133.9, 207.9 (q, $J=6.0$ Hz). IR (neat) ν 688, 775, 800, 840, 865, 1120, 1260, 1295, 1342, 1388, 1420, 1525, 1595, 1720, 1962, 3050 cm^{-1} . Anal. Calcd for $\text{C}_{14}\text{H}_9\text{F}_3$: C, 71.79; H, 3.87. Found: C, 71.31; H, 3.86.

1,1,1-Trifluoro-6-phenyl-2,3-hexadiene (9b): Yield 98%. R_f=0.67 (Hexane: AcOEt=8:1). ¹H NMR δ 2.31~2.39 (2H, m), 2.65~2.70 (2H, t, *J*=7.8 Hz), 5.27 (1H, m), 5.56 (1H, m), 7.05~7.29 (5H, m). ¹³C NMR δ 31.9, 34.8, 85.5 (q, *J*=39.0 Hz), 97.7, 117.4 (q, *J*=269.9 Hz), 126.2, 128.4, 140.7, 205.2, 205.2. IR (neat) ν 685, 695, 735, 840, 850, 960, 1120, 1250, 1300, 1420, 1495, 1600, 1975, 2840, 2900, 3020 cm⁻¹. Anal. Calcd for C₁₂H₁₁F₃: C, 67.92; H, 5.22. Found: C, 67.98; H, 5.19.

1,1,1-Trifluoro-2,3-hexadecadiene (9c): Yield 86%. R_f=0.89 (Hexane: AcOEt=6:1). ¹H NMR δ 0.88 (3H, t, *J*=6.6 Hz), 1.26 (18H, br), 1.40~1.47 (2H, m), 2.11 (2H, qd, *J*=3.0, 7.2 Hz), 5.40 (1H, m), 5.66 (1H, m). ¹³C NMR δ 14.1, 22.7, 22.7, 27.6, 27.7, 28.5, 28.9, 29.4, 29.4, 29.6, 29.7, 31.7, 85.0 (q, *J*=38.8 Hz), 98.5, 117.6 (q, *J*=269.6 Hz), 205.1 (q, *J*=5.6 Hz). IR (neat) ν 690, 720, 850, 870, 1130, 1260, 1300, 1420, 1460, 1980, 2900, 2950 cm⁻¹. Anal. Calcd for C₁₆H₂₇F₃: C, 69.53; H, 9.85. Found: C, 69.50; H, 9.78.

1-(4-Bromophenyl)-4,4,4-trifluoro-2,3-butadiene (9d): Yield 87%. R_f=0.66 (Hexane: AcOEt=0.66). ¹H NMR δ 5.74 (1H, quint, *J*=6.0 Hz), 6.48 (1H, qd, *J*=3.9, 7.5 Hz), 7.40~7.12 (2H, m), 7.31~7.40 (2H, m). ¹³C NMR δ 90.1 (q, *J*=39.4 Hz), 100.5, 122.2 (q, *J*=270.5 Hz), 122.6, 128.9, 129.8, 132.2, 207.0 (q, *J*=5.6 Hz). IR (neat) ν 700, 780, 820, 880, 1020, 1080, 1120, 1260, 1290, 1310, 1390, 1430, 1490, 1590, 1700, 1740, 1970, 2950 cm⁻¹. Anal. Calcd for C₁₀H₆BrF₃: C, 45.66; H, 2.30. Found: C, 45.27; H, 2.50.

4,4,4-Trifluoro-1-(2-methoxyphenyl)-2,3-butadiene (9e): Yield 93%. R_f=0.73 (Hexane: AcOEt=8:1). ¹H NMR δ 3.84 (3H, s), 5.75 (1H, quint, *J*=6.8 Hz), 6.87~6.96 (2H, m), 6.98 (1H, qd, *J*=4.2, 8.1 Hz), 7.23~7.31 (2H, m). ¹³C NMR δ 55.5, 87.6 (q, *J*=38.9 Hz), 95.7, 111.1, 114.7 (q, *J*=265.4 Hz), 120.9, 128.7, 129.8, 156.6, 207.7 (q, *J*=6.0 Hz). IR (neat) ν 640, 690, 750, 850, 880, 940, 1030, 1070, 1120, 1250, 1320, 1420, 1440, 1460, 1500, 1590, 1600, 1970, 2850, 2950, 3000 cm⁻¹. Anal. Calcd for C₁₁H₉F₃O: C, 61.68; H, 4.24. Found: C, 61.38; H, 4.15.

1,1,1-Trifluoro-4-phenyl-2,3-pentadiene (9f): Yield 76%. R_f=0.75 (Hexane: AcOEt=8:1). ¹H NMR δ 2.18 (3H, d, *J*=2.7 Hz), 5.72 (1H, m), 7.21~7.41 (5H, m). ¹³C NMR δ 16.3, 87.4 (q, *J*=38.9 Hz), 108.5, 122.7 (q, *J*=270.7 Hz), 126.2, 128.2, 128.7, 134.0, 205.9 (q, *J*=5.6 Hz). IR (neat) ν 700, 760, 845, 1030, 1075, 1125, 1260, 1305, 1405, 1450, 1495, 1720, 1805, 1970, 2860, 2950 cm⁻¹.

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STD1H
1H
300.40 MHz
130.00 KHz
1150.00 Hz
5.00 usec
97.50 usec
0.20000 msec
1.0000 msec
32768
32768
8
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6009.60 Hz
66.70 usec
5.4526 sec
1.5470 sec
16
16
0.10 Hz
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NON:Single.coupled:PW1_ACQTM.
1H
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1150.00 Hz
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511
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STD-TH5
25.60 KHz
52.0 Hz
200
24
105
1396
12 Hz

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STD1H
 1H
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 NON:Single.coupled:PW1_ACQTM.
 1H
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