Supplementary Material (ESI) for Photochemical & Photobiological Sciences


# Synthesis of Tricyclic Vinylcyclobutanes and their Application in Photoinduced Electron Transfer Chemistry

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## Supplementary Materials

### Synthesis of Vinylcyclobutanes 1a-j by Peterson olefination of tricyclic ketones 2a-j

The synthesis of tricyclic ketones **2a-j** has been reported separately.[1,2]

**General procedure A of synthesis of (trimethylsilyl)methyllithium (4)[3]:** To a refluxing suspension of lithium sand (982 mg, 141 mmol) in 75 mL *n*-pentane under argon atmosphere (cloromethyl)trimethylsilane (8.66 g, 71 mmol) was added dropwise. The mixture was heated under reflux for 8 h and cooled to r.t. After 12 h without stirring the mixture had separated into a colorless solution and a purple residue. The colorless solution of **4** was taken out with a syringe immediately before use.

**General procedure B of synthesis of tricyclic β-trimethylsilylalcohols 3a-j[3]:** 17 mL of the solution of **4,** made according to procedure A, were diluted with 50 mL anhyd $Et_2O$. A solution of the ketone (3 mmol) in 10 mL anhyd $Et_2O$ was added dropwise. The mixture was heated under reflux for 3-6 h. Conversion was controlled by GC. Under cooling to 0°C aq $NH_4Cl$ (25%, 10 mL) was added. After addition of $H_2O$ (100 mL) the mixture was extracted with $Et_2O$ (3 x 75 mL). The combined organic layers were washed with $H_2O$ (50 mL) and dried ($Na_2SO_4$). The solvents were evaporated under reduced pressure. The residue was purified by Kugelrohr destillation and flash chromatography (cyclohexane).

**General procedure C of synthesis of vinylcyclobutanes 1a-j[4]:** A suspension of KH in paraffin (1 g) was washed under an argon atmosphere with *n*-pentane (2 x 10 mL) and suspended in anhyd THF (15 mL). A solution of alcohol **3** (1.5 mmol) in anhyd THF (10 mL) was added dropwise. After stirring at r.t. for 2 h the mixture was added to cold aq $NH_4Cl$ (10%, 50 mL) overlaid with $Et_2O$ (100 mL). The organic layer was separated and dried ($MgSO_4$). The solvents were evaporated under reduced pressure. The residue was purified by Kugelrohr destillation and flash chromatography (cyclohexane).

#### *cis,anti,cis*-3-[(Trimethylsilyl)methyl]-tricyclo[5.3.0.0$^{2,6}$]decan-3-ol (3a)

**3a** was obtained from **2a** according to procedure B.

Yield: 81 %, colorless oil.

IR (film): 3488, 2948, 2870, 1465, 1447, 1415, 1361, 1287, 1246, 1175, 1150, 1118, 1056, 1043, 1018, 996, 891, 839, 762, 688 cm$^{-1}$.

$^1$H-NMR ($C_6H_6$): δ = 0.169 (s, 9 H, $Si(CH_3)_3$), 1.067 (s, 1 H, OH), 0.728 (d, 1 H, *J* = 14.8 Hz, 11-H), 0.811 (d, 1 H, *J* = 14.8 Hz, 11-H), 1.327-1.435 (m, 3 H), 1.457-1.529 (m, 2 H), 1.546-1.660 (m, 3 H), 1.676-1.738 (m, 2 H), 1.790 (ddd, 1 H, *J* = 6.8 Hz, 6.8 Hz, 3.2 Hz), 1.812-1.880 (m, 1 H), 1.932 (ddd, 1 H, *J* = 7.0 Hz, 7.0 Hz, 3.0 Hz), 2.264 (ddd, 1 H, *J* = 6.9 Hz, 6.9 Hz, 3.6 Hz).

$^{13}$C-NMR ($C_6D_6$): □ = 0.85 ($Si(CH_3)_3$), 25.50 ($CH_2$), 30.30 ($CH_2$), 30.59 ($CH_2$), 33.41 ($CH_2$), 33.63 ($CH_2$), 36.09 (CH), 40.44 ($CH_2$), 40.69 (CH), 42.29 (CH), 53.21 (CH), 81.97 ($C_q$, C-3).

GC/MS (EI, 70 eV): m/z (%) = 238 (0.9) [$M^+$], 155 (10), 115 (33), 91 (15), 80 (13), 79 (21), 75 (72), 74 (12), 73 (100) [$Si(CH_3)_3^+$], 67 (17), 66 (11), 45 (16), 41 (10).

HRMS: *m/z* calcd for $C_{14}H_{26}OSi$ ($M^+$), 238.17529; found, 238.17556.

#### *cis,anti,cis*-6-Methyl-3-[(trimethylsilyl)methyl]-tricyclo[5.3.0.0$^{2,6}$]decan-3-ol (3c)

**3c** was obtained from **2c** according to procedure B.

Yield: 82 %, colorless oil.

IR (film): 3491, 2948, 2867, 1730, 1448, 1415, 1373, 1289, 1246, 1220, 1155, 1107, 1059, 1021, 1006, 883, 838, 689 $cm^{-1}$.

$^1$H-NMR ($C_6D_6$): δ = 0.181 (s, 9 H, $Si(CH_3)_3$), 0.754 (dd, 1 H, *J* = 14.8 Hz, 1.1 Hz, 11-H), 0.850 (s, 3 H, 12-H), 0.870 (d, 1 H, *J* = 14.8 Hz, 11-H), 1.018 (s, 1 H, OH), 1.260 (dddd, 1 H, *J* = 12.9 Hz, 12.9 Hz, 7.7 Hz, 7.1 Hz), 1.327 (d, 1 H, *J* = 3.9 Hz), 1.353-1.420 (m, 3 H), 1.462 (dd, 1 H, *J* = 12.4 Hz, 6.2 Hz), 1.559 (ddddd, 1 H, *J* = 12.5 Hz, 12.5 Hz, 12.4 Hz, 6.2 Hz, 6.2 Hz), 1.655-1.721 (m, 3 H), 1.917 (dd, 1 H, *J* = 10.4 Hz, 10.4 Hz), 1.968 (ddd, 1 H, *J* = 7.7 Hz, 7.7 Hz, 7.7 Hz), 2.207 (ddd, 1 H, *J* = 7.1 Hz, 7.1 Hz, 4.2 Hz).

$^{13}$C-NMR ($C_6D_6$):δ = 0.97 ($CH_3$, $Si(CH_3)_3$), 21.16 ($CH_3$, C-12), 26.78 ($CH_2$), 28.56 ($CH_2$), 30.52 ($CH_2$), 33.04 ($CH_2$), 34.98 (CH), 40.08 ($CH_2$), 40.66 ($CH_2$), 41.02 ($C_q$, C-6), 45.19 (CH), 60.18 (CH), 82.09 ($C_q$, C-3).

GC/MS (EI, 70 eV): m/z (%) = 252 (<1) [$M^+$], 115 (49), 97 (28), 91 (21), 79 (40), 77 (15), 75 (87), 73 (100) [$Si(CH_3)_3^+$], 67 (20), 66 (15), 59 (10), 55 (13), 45 (20), 43 (10), 41 (14).

HRMS: *m/z* calcd for $C_{15}H_{28}OSi$ ($M^+$), 252.1909; found, 252.1910.

***cis,syn,trans*-6-Methyl-3-[(trimethylsilyl)methyl]-tricyclo[5.4.0.0$^{2,6}$]undecan-3-ol (3d)**

**3d** was obtained from **2d** according to procedure B.

Yield: 87 %, colorless oil.

IR (film): 3503, 2931, 2858, 1444, 1247, 999, 839, 687, 664 $cm^{-1}$.

$^1$H-NMR ($C_6D_6$): δ = 0.169 (s, 9 H, $Si(CH_3)_3$), 0.921 (s, 2 H, 12-H), 1.074 (s, 1 H, OH), 1.111 (s, 3 H, 13-H), 1.192-1.237 (m, 3 H), 1.288 (ddd, 1 H, *J* = 12.8 Hz, 10.0 Hz, 7.4 Hz), 1.506-1.534 (m, 1 H), 1.561 (ddd, 1 H, *J* = 12.8 Hz, 7.0 Hz, 3.6 Hz), 1.650-1.739 (m, 5 H), 1.779-1.818 (m, 1 H), 1.847 (ddd, 1 H, *J* = 13.0 Hz, 7.1 Hz, 3.6 Hz), 2.000 (d, 1 H, *J* = 7.4 Hz), 2.060 (ddd, 1 H, *J* = 13.0 Hz, 10.0 Hz, 7.1 Hz).

$^{13}$C-NMR ($C_6D_6$):δ = 0.81 (3 C, $Si(CH_3)_3$), 20.05, 26.77, 28.22, 28.66, 29.79, 34.76, 37.51, 41.16, 46.04, 47.92, 50.58, 61.48, 83.77 (C-3).

GC/MS (EI, 70 eV): m/z (%) =: 266 (<1) [$M^+$], 248 (3) [$M^+$-$H_2O$], 233 (2) [$M^+$-$H_2O$-$CH_3$], 136 (14), 115 (35), 107 (10), 105 (10), 97 (31), 93 (14), 91 (28), 81 (12), 79 (25), 77 (10), 75 (62), 74 (12), 73 (100) [$Si(CH_3)_3^+$], 67 (13), 55 (16), 45 (19), 43 (10), 41 (17).

HRMS: *m/z* calcd for $C_{16}H_{30}OSi$ ($M^+$), 266.20659; found, 266.20662.

**6-Methyl-3-[(trimethylsilyl)methyl]-tricyclo[n.m.0.0$^{2,n+1}$]alkan-3-ols (3b,e-j)**

**3b,e-f** were obtained from **2b,e-f** according to procedure B in good yields (60-99 %) as colorless oils. Characterization was performed using GC/MS.

***cis-anti-cis*-3-Methylen-tricyclo[5.3.0.0$^{2,6}$]decane (1a)**

**1a** was obtained from **3a** according to procedure C.

Yield: 82 %, colorless oil.

IR (film): 3076, 2937, 2857, 1658, 1466, 1439, 1249, 1173, 1143, 1111, 1058, 1028, 1007, 880, 838, 664 $cm^{-1}$.

$^1$H-NMR ($C_6D_6$): δ = 1.285-1.410 (m, 2 H), 1.455-1.530 (m, 2 H), 1.549-1.628 (m, 2 H), 1.659-1.739 (m, 2 H), 1.998 (ddd, 1 H, *J* = 7.0 Hz, 7.0 Hz, 3.5 Hz), 2.068 (ddd, 1 H, *J* = 6.8 Hz, 6.8 Hz, 3.5 Hz), 2.192- 2.261 (m, 2 H), 2.301 (dd, 1 H, *J* = 6.6 Hz, 2.7 Hz), 2.599-2.682 (m, 1 H), 4.827 (dd, 1 H, *J* = 1.9 Hz, 1.9 Hz, 11-H), 4.886 (dd, 1 H, *J* = 2.1 Hz, 2.1 Hz, 11-H).

$^{13}$C-NMR ($C_6D_6$):δ = 25.27 ($CH_2$), 32.79 ($CH_2$, 2 C), 33.43 ($CH_2$), 33.54 ($CH_2$), 42.22 (CH), 42.87 (CH), 43.68 (CH), 48.13 (CH), 104.20 ($CH_2$, C-11), 157.49 ($C_q$, C-3).

GC/MS (EI, 70 eV): m/z (%) = 148 (13) [$M^+$], 133 (10), 120 (4), 119 (7), 117 (1), 107 (3), 106 (8), 105 (12), 103 (1), 93 (4), 92 (7), 91 (25), 82 (1), 81 (21), 79 (37), 79 (100), 78 (3), 77 (28), 69 (1), 68 (3), 67 (27), 66 (8), 65 (21), 63 (5), 62 (1), 59 (1), 58 (4), 55 (5), 54 (3), 53 (14), 52 (8), 51 (11), 50 (3), 42 (2), 41 (24), 40 (5), 39 (27), 29 (4), 27 (13).

HRMS: *m/z* calcd for $C_{11}H_{16}$ ($M^+$), 148.12520; found, 148.12520.

***cis,syn,cis*-6-Methyl-3-methylen-tricyclo[5.3.0.0$^{2,6}$]decane (1b)**

**1b** was obtained from **3b** according to procedure C.

Yield: 94 %, colorless oil.

IR (film): 3075, 2949, 2862, 1449, 880, 664 $cm^{-1}$.

$^1$H-NMR ($C_6D_6$): δ = 1.093-1.153 (m, 1 H), 1.145 (s, 3 H, 12-H), 1.321-1.611 (m, 5 H), 1.724-1773 (m, 1 H), 1.730 (ddd, 1 H, *J* = 13.4 Hz, 8.9 Hz, 6.8 Hz), 2.265-2.429 (m, 3 H), 2.607 (ddddd, 1 H, *J* = 9.7 Hz, 1.9 Hz, 1.9 Hz, 1.9 Hz, 1.9 Hz), 2.741 (dddd, 1 H, *J* = 9.0 Hz, 9.0 Hz, 8.9 Hz, 2.7 Hz), 4.742 (dddd, 1 H, *J* = 2.0 Hz, 2.0 Hz, 2.0 Hz, 2.0 Hz, 11-H), 5.032 (dddd, 1 H, *J* = 2.0 Hz, 2.0 Hz, 2.0 Hz, 2.0 Hz, 11-H).

$^{13}$C-NMR ($C_6D_6$):δ = 27.40 ($CH_2$), 27.77 ($CH_2$), 28.04 ($CH_2$), 29.69 ($CH_3$, C-12), 33.07 ($CH_2$), 36.11 ($CH_2$), 36.31 (CH), 45.22 ($C_q$, C-6), 47.04 (CH), 50.13 (CH), 107.09 ($CH_2$, C-11), 154.33 ($C_q$, C-3).

GC/MS (EI, 70 eV): m/z (%) = 162 (9) [$M^+$], 147 (62) [$M^+$-$CH_3$], 134 (25), 133 (35), 120 (16), 119 (27), 107 (15), 106 (24), 105 (50), 95 (14), 94 (64), 93 (29), 92 (17), 91 (64), 81 (10), 80 (18), 79 (100), 78 (11), 77 (32), 67 (25), 65 (15), 55 (16), 53 (20), 51 (12), 41 (35), 39 (32), 29 (11), 27 (23).

HRMS: *m/z* calcd for $C_{12}H_{18}$ ($M^+$), 162.14085; found, 162.14059.

***cis,anti,cis*-6-Methyl-3-methylen-tricyclo[5.3.0.0$^{2,6}$]decane (1c)**

**1c** was obtained from **3c** according to procedure C.

Yield: 87 %, colorless oil.

IR (film): 3076, 2947, 2859, 1438, 1248, 879, 837 cm$^{-1}$.

$^1$H-NMR ($C_6D_6$): δ = 0.857 (s, 3 H, 12-H), 1.203-1.284 (m, 1 H, 8-H), 1.354 (dd, 1 H, *J* = 7.1 Hz, 7.1 Hz, 10-H), 1.426 (ddd, 1 H, *J* = 12.1 Hz, 12.1 Hz, 7.7 Hz, 5-H), 1.503-1.618 (m, 3 H, 5-H, 9-H, 10-H), 1.639-1.694 (m, 2 H, 8-H, 9-H), 1.905 (dddd, 1 H, *J* = 3.9Hz, 0.9 Hz, 0.9 Hz, 0.9 Hz, 2-H), 2.103 (dd, 1 H, *J* = 7.5 Hz, 7.5 Hz, 7-H), 2.214 (ddd, 1 H, *J* = 7.3 Hz, 7.2 Hz, 3.8 Hz, 1-H), 2.249 (dddddd, 1 H, *J* = 14.5 Hz, 7.7 Hz, 2.0 Hz, 1.0 Hz, 1.0 Hz, 1.0 Hz, 4-H), 2.672 (dddddd, 1 H, *J* = 14.4 Hz, 11.9 Hz, 8.1 Hz, 2.5 Hz, 2.5 Hz, 1.1 Hz, 4-H), 4.769 (ddddd, 1 H, *J* = 2.5 Hz, 1.7 Hz, 0.9 Hz, 0.9 Hz, 0.9 Hz, 11-H), 4.817 (dddd, 1 H, *J* = 2.1 Hz, 2.1 Hz, 1.0 Hz, 1.0 Hz, 11-H).

$^{13}$C-NMR ($C_6D_6$):δ = 20.39 ($CH_3$, C-12), 26.72 ($CH_2$, C-9), 28.69 ($CH_2$, C-8), 32.78 ($CH_2$, C-10), 33.38 ($CH_2$, C-4), 42.14 (CH, C-1), 42.51 ($CH_2$, C-5), 43.31 ($C_q$, C-6), 45.29 (CH, C-7), 54.97 (CH, C-2), 103.41 ($CH_2$, C-11), 157.58 ($C_q$, C-3).

GC/MS (EI, 70 eV): m/z (%) = 162 (17) [$M^+$], 147 (53) [$M^+$-$CH_3$], 134 (18), 133 (24), 119 (18), 106 (10), 105 (29), 94 (31), 93 (17), 92 (11), 91 (42), 80 (14), 79 (100), 78 (14), 77 (38), 67 (29), 65 (21), 55 (14), 53 (20), 51 (12), 41 (30), 39 (30), 27 (19).

HRMS: *m/z* calcd for $C_{12}H_{18}$ ($M^+$), 162.1409; found, 162.1414.

***cis,syn,trans*-6-Methyl-3-methylen-tricyclo[5.4.0.0$^{2,6}$]undecane (1d)**

**1d** was obtained from **3d** according to procedure C.

Yield: 93 %, colorless oil.

IR (film): 3074, 2931, 2859, 1452, 887, 875, 835 cm$^{-1}$.

$^1$H-NMR ($C_6D_6$): δ = 1.136 (s, 3 H, 13-H), 1.147-1.590 (m, 9 H), 1.648-1.721 (m, 3 H), 2.482 (ddddd, 1 H, *J* = 17.3 Hz, 9.3 Hz, 1.8 Hz, 1.8 Hz, 1.8 Hz), 2.606-2.694 (m, 2 H), 4.930-4.948 (m, 1 H, 12-H), 5.143 (dddd, 1 H, *J* = 2.2 Hz, 2.2 Hz, 2.1 Hz, 2.1 Hz, 12-H).

$^{13}$C-NMR ($C_6D_6$):δ = 18.54 ($CH_3$, C-13), 26.78 ($CH_2$), 26.87 ($CH_2$), 27.98 ($CH_2$), 28.32 ($CH_2$), 34.80 ($CH_2$), 37.50 ($CH_2$), 42.15 (CH), 46.40 (CH), 52.85 ($C_q$, C-6), 54.01 (CH), 108.53 ($CH_2$, C-12), 153.34 ($C_q$, C-3).

GC/MS (EI, 70 eV): m/z (%) = 176 (10) [$M^+$], 161 (11) [$M^+$-$CH_3$], 148 (22), 147 (19), 133 (16), 119 (20), 108 (19), 107 (66), 106 (18), 105 (38), 95 (15), 94 (86), 93 (44), 92 (14), 91 (55), 81 (14), 80 (16), 79 (100), 77 (29), 67 (21), 65 (12), 55 (23), 41 (36), 39 (24), 29 (13), 27 (20).

HRMS: *m/z* calcd for $C_{13}H_{20}$ ($M^+$), 176.15650; found, 176.15618.

***cis,anti,trans*-6-Methyl-3-methylen-tricyclo[5.4.0.0$^{2,6}$]undecane (1e) and *cis,anti,cis*-6-Methyl-3-methylen-tricyclo-[5.4.0.0$^{2,6}$]undecane (1f)**

A mixture of **1e-f** (30:70) was obtained from a mixture (30:70) of **3e-f** according to procedure C in 96 % yield. Analytical pure samples of **1e** and **1f** were obtained by preparative GC.

**1e**: colorless oil.

IR (film): 3075, 2931, 2746, 1651, 1451, 1372, 1309, 1247, 1191, 1091, 1014, 917, 907, 876, 834, 747, 686, 665 cm$^{-1}$.

$^1$H-NMR ($C_6D_6$): δ = 1.068 (s, 3 H, C-13), 1.106-1.305 (m, 6 H), 1.401 (dddd, 1 H, *J* = 11.3 Hz, 11.3 Hz, 8.2 Hz, 3.2 Hz), 1.517-1.551 (m, 1 H), 1.646-1.680 (m, 2 H), 1.762-1.796 (m, 1 H), 2.014 (ddd, 1 H, *J* = 13.2 Hz, 9.6 Hz, 9.6 Hz), 2.431 (d, 1 H, *J* = 8.2 Hz), 2.567 (dddd, 2 H, *J* = 9.7 Hz, 7.4 Hz, 2.3 Hz, 2.3 Hz), 4.869 (ddd, 2 H, *J* = 1.7 Hz, 1.7 Hz, 1.7 Hz, 12-H).

$^{13}$C-NMR ($C_6D_6$):δ = 26.63 ($CH_3$, C-13), 26.68 ($CH_2$), 27.29 ($CH_2$), 27.76 ($CH_2$), 31.04 ($CH_2$), 31.41 ($CH_2$), 35.97 ($CH_2$), 46.58 (CH), 50.97 (CH), 51.46 ($C_q$, C-6), 60.00 (CH), 103.94 ($CH_2$, C-12), 155.57 ($C_q$, C-3).

GC/MS (EI, 70 eV): m/z (%) = 176 (7) [$M^+$], 161 (10) [$M^+$-$CH_3$], 148 (20), 147 (13), 133 (15), 119 (20), 108 (19), 107 (78), 106 (18), 105 (40), 95 (12), 94 (66), 93 (44), 92 (15), 91 (62), 81(15), 80 (18), 79 (100), 78 (10), 77 (34), 67 (23), 65 (14), 55 (24), 53 (24), 51 (11), 41 (43), 39 (31), 29 (17), 27 (25).

HRMS: *m/z* calcd for $C_{13}H_{20}$ ($M^+$), 176.15650; found, 176.15601.

**1f**: colorless oil.

IR (film): 3074, 2933, 2866, 1656, 1449, 1375, 878, 665 $cm^{-1}$.

$^1$H-NMR ($C_6D_6$): δ = 1.077 (s, 3 H, 13-H), 1.157-1.273 (m, 2 H), 1.351-1.518 (m, 6 H), 1.603-1.684 (m, 2 H), 1.921-2.001 (m, 2 H), 2.332 (d, 1 H, *J* = 3.9 Hz), 2.382 (ddddd, 1 H, *J* = 15.7 Hz, 8.1 Hz, 4.6 Hz, 1.6 Hz, 1.6 Hz), 2.604-2.682 (m, 1 H), 4.851 (dddd, 1 H, *J* = 2.5 Hz, 1.4 Hz, 1.4 Hz, 1.4 Hz, 12-H), 4.888 (dddd, 1 H, *J* = 1.7 Hz, 1.7 Hz, 1.7 Hz, 1.7 Hz, 12-H).

$^{13}$C-NMR ($C_6D_6$):δ = 22.00 ($CH_2$), 22.10 ($CH_3$, C-13), 22.23 ($CH_2$), 22.38 ($CH_2$), 27.36 ($CH_2$), 34.34 ($CH_2$), 36.27 (CH), 37.88 (CH), 41.17 ($CH_2$), 47.33 ($C_q$, C-6), 55.57 (CH), 104.45 ($CH_2$, C-12), 157.42 ($C_q$, C-3).

GC/MS (EI, 70 eV): m/z (%) = 176 (9) [$M^+$], 161 (10) [$M^+$-$CH_3$], 148 (23), 147 (14), 133 (12), 119 (15), 108 (11), 107 (34), 106 (11), 105 (26), 95 (13), 94 (75), 93 (31), 92 (10), 91 (39), 81 (13), 80 (14), 79 (100), 77 (28), 67 (16), 65 (10), 55 (16), 53 (19), 41 (30), 39 (24), 29 (11), 27 (18).

HRMS: *m/z* calcd for $C_{13}H_{20}$ ($M^+$), 176.15650; found, 176.15614.

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