# Nickel-Catalyzed Olefination of Unactivated Aliphatic Dithioacetals

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## Supporting Information

**General procedure for the olefination of dithioacetals.** Under Ar atmosphere, to a toluene solution (10 mL) of Ni(acac)$_2$ (5 mmol%) and dithioacetal (1 mmol) was added a toluene solution of the phosphine ligand (1.8 mL, 0.067 or 0.068 M, 12 mmol%). At 0 ºC, the Grignard reagent in ether (4 mL, 2 M, 8 mmol) prepared from the corresponding halide (1 eq) and magnesium turnings (1 eq) was then added dropwise. The mixture was stirred at 0 ºC for 10 min, gradually warmed to rt and then refluxed for 20 h. After cooling to rt, the mixture was filtered through celite/silica gel (1:1) and the filtrate was evaporated in vacuo. The residue was chromatographed on silica gel to give the olefination product.

**Synthesis of (β-naphthylmethyl)-1,3-dithiane 1.** At -40 $^{o}$C, to a THF solution (120 mL) of 1,3-dithiane (2.10 g, 17.0 mmol) was added *n*-butyl lithium (7.0 mL, 2.5 M in hexane, 17.5 mmol) dropwise under Ar atmosphere and the mixture was stirred at -40 $^{o}$C for 2h. To a THF solution of 2-bromomethylnaphthalene[1] (3.66 g, 16.6 mmol) at -78 $^{o}$C was added the above THF solution dropwise over 20 min. The mixture was stirred at -78 ºC for 1 h, gradually warmed to rt and quenched with water. Ether was added and the organic layer was separated. The aqueous layer was extracted with ether (50 mL). The combined organic solutions were washed with water (100 mL) and brine (150 mL), dried (MgSO$_4$) and filtered. The filtrate was evaporated in vacuo and the residue was chromatographed on silica gel (hexane/ethyl acetate = 1/1, then hexane/CH$_2$Cl$_2$ = 1/1) to give **1** as white powder (3.62 g, 84%). mp. 102-103 ºC; $^1$H NMR (CDCl$_3$, 300 MHz): δ 1.75-1.94 (m, 1 H), 2.00-2.18 (m, 1 H), 2.75-2.95 (m, 4 H), 3.18-3.26 (d, *J* = 7.3 Hz, 2 H), 4.34 (t, *J* = 7.3 Hz, 1 H), 7.34-7.52

(m, 3 H), 7.69 (br, 1 H), 7.72-7.88 (m, 3 H); $^{13}$C ($CDCl_3$, 75 MHz): δ 25.7, 30.5, 41.9, 48.6, 125.6, 126.0, 127.3, 127.7, 127.9, 128.0, 132.5, 133.3, 134.8. IR (KBr): ν 3051, 2929, 2896, 1559, 1508, 1421, 1365, 1275, 1241, 1179, 960, 950, 906, 856, 819, 786, 763, 742, 714, 664, 480 cm$^{-1}$; HRMS (EI) $(M+1)^+$ calcd for $C_{15}H_{17}S_2$ 261.0772, found 261.0775; Anal. calcd for $C_{15}H_{16}S_2$: C, 69.18; H, 6.19; S, 24.63, found: C, 69.57; H, 6.37; S, 24.15.

**Synthesis of *(E)*-(2-naphthyl)propene 2, *(Z)*-(2-naphthyl)propene 3 and 2-[3-(1-propenyl)]naphthalene 4.** According to the general procedure that described above, under Ar atmosphere, a toluene solution (10 mL) of (β-naphthylmethyl)-1,3-dithiane (260 mg, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_3$ (1.8 mL, 0.067 M, 12 mmol%) and methylmagnesium iodide (2.0 mL, 2.0 M, 4 mmol) was trandformed into an isomeric mixture of **2**, **3** and **4** (12/1/2.5) as colorless liquid (132 mg, 78%). $^1$H NMR ($CDCl_3$, 300 MHz): characteristic peaks for **2**: δ 1.95 (d, $J$ = 6.8 Hz, 3 H), 6.37 (dq, $J$ = 17.0, 6.8 Hz, 1 H), 6.57 (d, $J$ = 17.0 Hz, 1 H)[2]; for **3**: δ 1.99 (d, $J$ = 6.8 Hz, 3 H), 5.89 (dq, $J$ = 10.0, 6.8 Hz, 1 H); for **4**: δ 3.56 (d, $J$ = 6.8 Hz, 2 H), 5.10-5.18 (m, 2 H), 6.06 (ddt, $J$ = 16.8, 10.0, 6.8 Hz, 1 H).

**Synthesis of *(E)*-(2-naphthyl)vinyl-trimethyl-silane 6.** According to the general procedure that described above, under Ar atmosphere, a toluene solution (10 mL) of (β-naphthylmethyl)-1,3-dithiane (260 mg, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_3$ (1.8 mL, 0.067 M, 12 mmol%) and trimethylsilylmethylmagnesium chloride (4.0 mL, 2.0 M, 8 mmol) was transformed into **6** and **7** as pale-yellow liquid (137 mg, 57%). Recrystallized with pentane to give **6** as pale-yellow solid: mp. 56-58 ºC; $^1$H NMR ($CDCl_3$, 300 MHz): δ 0.07 (s, 9 H), 1.68-1.80 (m, 2 H), 6.39 (br, 2 H), 7.30-7.48 (m, 3 H), 7.62 (s, 1 H), 7.70-7.86 (m, 3H); $^{13}$C ($CDCl_3$, 75 MHz): δ -1.81, -1.20, 24.2, 43.3, 123.5, 124.5, 125.1, 125.9, 126.1, 126.8, 127.6, 127.7, 127.9, 128.4, 132.4, 133.8, 136.0, 145.0. IR (KBr): ν 3052, 3011, 2946, 2893, 1940, 1920, 1695, 1630, 1597, 1503, 1434, 1405, 1356, 1295, 1246, 1144, 1021, 964, 899, 837, 809, 751, 690, 621, 474 cm$^{-1}$; HRMS (EI) $(M)^+$ calcd for $C_{16}H_{20}Si$ 240.1329, found 240.1334; Anal. calcd for $C_{16}H_{20}Si$: C, 79.93; H, 8.38, found: C, 79.96; H, 8.38.

**Synthesis of 2-(1-adamantyl)-1,3-dithiane 8.** To a chloroform solution (100 mL) of 1-adamantanecarbaldehyde (4.26 g, 25.9 mmol) was added 1,3-propanedithiol (2.7 mL, 27.2 mmol) under Ar atmosphere. At 0 $^o$C, the mixed solution was then added

$BF_3.OEt_2$ (1.3 mL, 10.4 mmol) dropwise. The mixture was stirred at 0 $^{o}$C for 10 min and gradually warmed to rt for 5 h. The stirred solution was treated with chloroform (30 mL) and washed with 10% NaOH (50 mL) twice at 0 ºC. The organic layer was washed with water (150 mL). The combined aqueous layers were extracted with chloroform. The combined organic solutions were dried ($MgSO_4$) and filtered. The filtrate was evaporated in vacuo to give **8** as white powder (6.19g, 94%): mp. 73-74 ºC (EtOH, lit.[3] 73-74 ºC); $^{1}$H NMR ($CDCl_3$, 400 MHz): δ 1.60-1.90 (m, 13 H), 1.94-2.15 (m, 4 H), 2.75-2.95 (m, 4 H), 3.89 (s, 1 H).

**Synthesis of (2-adamantan-1-yl-vinyl)-dimethyl-phenyl-silane 9**. According to the general procedure that described above, under Ar atmosphere, a toluene solution (10 mL) of 2-(1-Adamantyl)-1,3-dithiane (254 mg, 1 mmol), $Ni(acac)_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_2$Me (1.8 mL, 0.068 M, 12 mmol%) and dimethylphenylsilylmethylmagnesium chloride (4.0 mL, 2.0 M, 8 mmol) was transformed into **9** as colorless liquid (255 mg, 91%). $^{1}$H NMR ($CDCl_3$, 400 MHz): δ 0.33 (m, 6H), 1.58-1.80 (m, 13H), 2.01 (br, 1 H), 5.61 (d, $J$ = 18.9 Hz, 1 H), 6.00 (d, $J$ = 18.9 Hz, 1 H), 7.28-7.40 (m, 3 H), 7.46-7.60 (m, 2 H).[4]

**Synthesis of 2-(α-naphthylmethyl)-1,2-dithioacetal 10.** To a chloroform solution (80 mL) of [1]naphthyl-acetaldehyde[5](6.2 mL, 40 mmol) was added 1,2-ethanedithiol (3.6 mL, 42.8 mmol) under Ar atmosphere. At 0$^{o}$C, the mixed solution was then added $BF_3.OEt_2$ (12 mL, 95.3 mmol) dropwise. The mixture was stirred at 0 $^{o}$C for 10 min and gradually warmed to rt for 5 h. The stirred solution was added chloroform (40 mL) and washed with 10% NaOH (50 mL) at 0 ºC. The organic layer was washed with water (200 mL). The combined aqueous layers were extracted with some chloroform. The combined organic solutions were dried ($MgSO_4$) and filtered. The filtrate was evaporated in vacuo and the residue was chromatographed on silica gel (hexane/chloroform = 8/2 to 7/3) to give **10** as white powder (9.97 g, 77%). mp. 60-62 ºC; $^{1}$H NMR ($CDCl_3$, 300 MHz): δ 3.15-3.45 (m, 4 H), 3.59 (d, $J$ = 7.1 Hz, 2 H), 4.94 (t, $J$ = 7.1 Hz, 1 H), 7.35-7.62 (m, 4 H), 7.72-7.81 (m, 1 H), 7.87 (d, $J$ = 8.1 Hz, 1 H), 8.03 (d, $J$ = 8.1 Hz, 1 H); $^{13}$C ($CDCl_3$, 75 MHz): δ 38.6, 42.4, 54.0, 123.3, 125.3, 125.6, 126.0, 127.3, 127.7, 128.9, 131.5, 133.8, 135.1. IR (KBr): ν 3044, 2919, 2848, 1560, 1509, 1421, 1394, 1275, 1258, 1164, 1015, 850, 804, 782, 733, 584, 551 cm$^{-1}$; HRMS (EI) $(M+1)^+$ calcd for $C_{14}H_{15}S_2$ 247.0615, found 247.0616; Anal. calcd for $C_{14}H_{14}S_2$ (%): C, 68.24; H, 5.73; S, 26.03, found: C, 68.31; H, 5.81; S, 25.90.

**Synthesis of β-(1-naphthyl)styrene 11.** According to the general procedure that described above, under Ar atmosphere, a toluene solution (10 mL) of 2-(α-naphthylmethyl)-1,2-dithioacetal (246 mg, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_3$ (1.8 mL, 0.067 M, 12 mmol%) and phenylmagnesium bromide (2.0 mL, 2.0 M, 4 mmol) was transformed into **11** and a trace amount of cis isomer as pale-yellow liquid (115 mg, 50%). $^1$H NMR (CDCl$_3$, 300 MHz): characteristic peaks for **11**: δ 7.16 (d, *J* = 16.0 Hz, 1H), 7.26-7.32 (m, 2 H), 7.37-7.43 (m, 2H), 7.50-7.54 (m, 2 H),7.61 (d, *J* = 7.3 Hz, 2 H), 7.80 (d, *J* = 8.1 Hz, 2 H), 7.82-7.92 (m, 2 H), 8.23 (d, *J* = 8.1 Hz, 1 H).[6]

**Synthesis of 1-*p*-phenyl-cyclohexene 13 by utilizing different phosphine ligands.**

According to the general procedure that described above, under Ar atmosphere, a toluene solution (10 mL) of 1,4-dithiaspiro[4.5]decane[7] (0.15 mL, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_3$ (1.8 mL, 0.067 M, 12 mmol%) and phenylmagnesium bromide (4.0 mL, 2.0 M, 8 mmol) was transformed into **13** as colorless liquid (76 mg, 48%).

Under Ar atmosphere, a toluene solution (10 mL) of 1,4-dithiaspiro[4.5]decane[7] (0.15 mL, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_2$Me (1.8 mL, 0.068 M, 12 mmol%) and phenylmagnesium bromide (4.0 mL, 2.0 M, 8 mmol) was transformed into **13** as colorless liquid (89 mg, 56%). $^1$H NMR (CDCl$_3$, 300 MHz): δ 1.58-1.85 (m, 4H), 2.15-2.28 (m, 2H), 2.38-2.42 (m, 2H), 6.10 (br, 1H), 7.10-7.45 (m, 5H).[8]

**Synthesis of 1-*p*-tolyl-cyclohexene 14.** According to the general procedure that described above, under Ar atmosphere, a toluene solution (10 mL) of 1,4-dithiaspiro[4.5]decane[7] (0.15 mL, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_2$Me (1.8 mL, 0.068 M, 12 mmol%) and *p*-tolylmagnesium bromide (4.0 mL, 2.0 M, 8 mmol) was transformed into **14** as colorless liquid (100 mg, 59%). $^1$H NMR (CDCl$_3$, 300 MHz): δ 1.60-1.71 (m, 2 H), 1.71-1.82 (m, 2 H), 2.10-2.25 (m, 2 H), 2.26-2.45 (m, 5 H), 6.07 (br, 1 H), 7.09 (d, *J* = 8.1 Hz, 2 H), 7.26 (d, *J* = 8.1 Hz, 2 H).[9]

**Synthesis of 1-phenylcyclododecene 16 by utilizing different phosphine ligands.**

According to the general procedure that described above, under Ar atmosphere, a toluene solution (10 mL) of 1,4-dithiaspiro[4.11]hexadecane[10] (260 mg, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_3$ (1.8 mL, 0.067 M, 12 mmol%) and phenylmagnesium bromide (4.0 mL, 2.0 M, 8 mmol) was transformed into **16** as colorless liquid (124 mg, 52%).

Under Ar atmosphere, a toluene solution (10 mL) of 1,4-dithiaspiro[4.11]hexadecane[10] (260 mg, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_2$Me (1.8 mL, 0.068 M, 12 mmol%) and phenylmagnesium bromide (4.0 mL, 2.0 M, 8 mmol) was transformed into **16** as colorless liquid (160 mg, 67%). $^1$H NMR (CDCl$_3$, 300 MHz): δ *(E)* 1.11-1.49 (m, 16 H), 2.25 (q, *J* = 14.2 Hz, 7.2 Hz, 2 H), 2.58 (t, *J* = 6.7 Hz, 2 H), 5.57 (t, *J* = 7.8 Hz, 1 H), 7.15-7.38 (m, 5 H).[11]

**Synthesis of 1-(4-methylphenyl)cyclododencene 17.** According to the general procedure that described above, under Ar atmosphere, a toluene solution (10 mL) of 1,4-dithiaspiro[4.11]hexadecane (260 mg, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_2$Me (1.8 mL, 0.068 M, 12 mmol%) and *p*-tolylmagnesium bromide (4.0 mL, 2.0 M, 8 mmol) was transformed into **17** as colorless liquid (160 mg, 63%). $^1$H NMR (CDCl$_3$, 300 MHz): δ *(E)* δ 1.11-1.48 (m, 16 H), 2.24 (q, *J* = 13.8 Hz, 7.1 Hz, 2 H), 2.33 (s, 3 H), 2.55 (t, *J* = 6.7 Hz, 2 H), 5.53 (t, *J* = 7.7Hz, 1 H), 7.03-7.22 (m, 4 H).[12]

**Synthesis of 2-admantan-1-yl-2-methyl-1,3-dithhiolane 18.** To a chloroform solution (120 mL) of 1-adamantan-1-yl-ethanone (3.20 g, 18 mmol) was added 1,2-ethanedithiol (1.6 mL, 18.8 mmol) under Ar atmosphere. At 0 °C, the mixed solution was then added BF$_3$.OEt$_2$ (0.9 mL, 7.2 mmol) dropwise. The mixture was stirred at 0 °C for 10 min and gradually warmed to rt for 5 h. The stirred solution was treated with chloroform (30 mL) and washed with 10% NaOH (50 mL) at 0 °C. The organic layer was washed with water (150 mL). The combined aqueous layers were extracted with some chloroform. The combined organic solutions were dried (MgSO$_4$) and filtered. The filtrate was evaporated in vacuo to give white powder. Recrystallized with ethanol to give **18** as white powder (3.88g, 85%): mp. 99-101 °C; $^1$H NMR (CDCl$_3$, 400 MHz): δ 1.58-1.72 (m, 6 H), 1.74-1.90 (m, 9 H), 2.02 (br, 3 H), 3.14-3.30 (m, 4 H); $^{13}$C (CDCl$_3$, 100 MHz): δ 28.7, 29.2, 36.7, 38.4, 40.0, 41.0. IR (KBr): 2962, 2933, 2893, 2668, 2651, 1695, 1442, 1413, 1381, 1340, 1311, 1279, 1262, 1180, 1086, 1062, 1029, 972, 862, 731, 645 cm$^{-1}$; HRMS (EI) $(M+1)^+$ calcd for $C_{14}H_{23}S_2$ 255.1241, found 252.1238; Anal. calcd for $C_{14}H_{22}S_2$ (%): C, 66.08; H, 8.71, found: C, 65.78; H, 8.94.

**Synthesis of 1-(1-phenyl-vinyl)-adamantane 19.** According to the general procedure that described above, a toluene solution of 2-admantan-1-yl-2-methyl-1,3-

dithhiolane (250 mg, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_2$Me (1.8 mL, 0.068 M, 12 mmol%) and phenylmagnesium bromide (4.0 mL, 2.0 M, 8 mmol) was transformed into **19** as colorless liquid (145 mg, 61%). $^1$H NMR (CDCl$_3$, 300 MHz): $\delta$ 1.56-1.85 (m, 12 H), 1.99 (br, 3 H), 4.77 (s, 1H), 5.11 (s, 1 H), 7.00-7.35 (m, 5 H).[13]

**Synthesis of 1-(1-tolyl-vinyl)-adamantane 20.** According to the general procedure that described above, a toluene solution of 2-admantan-1-yl-2-methyl-1,3-dithhiolane (250 mg, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_2$Me (1.8 mL, 0.068 M, 12 mmol%) and *p*- tolylmagnesium bromide (4.0 mL, 2.0 M, 8 mmol) was transformed into **20** as white solid (138 mg, 55%). mp. 59-61 ºC; $^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ 1.55-1.82 (m, 12 H), 1.97 (br, 3 H), 2.34 (s, 3 H), 4.74 (s, 1 H), 5.08 (s, 1 H), 6.97 (d, *J* = 7.9 Hz, 2 H), 7.08 (d, *J* = 7.9 Hz, 2 H); $^{13}$C (CDCl$_3$, 100 MHz): $\delta$ 21.1, 28.6, 36.7, 37.7, 41.2, 111.2, 127.8, 129.1, 135.6, 139.8, 160.4. IR (KBr) 3081, 3048, 3019, 2901, 2848, 2676, 2652, 1908, 1814, 1622, 1507, 1442, 1344, 1287, 1246, 1180, 1103, 1066, 1013, 972, 899, 821, 727, 670, 621, 588, 531, 486 cm$^{-1}$; HRMS (EI) $(M+1)^+$ calcd for $C_{19}H_{25}$ 252.1873, found 252.1878; Anal. calcd for $C_{19}H_{24}$ (%): C, 90.42; H, 9.58, found: C, 90.18; H, 9.82.

**Synthesis of 2-adamantone thioketal 21.** To a chloroform solution (80 mL) of 2-adamantanone (3.00 g, 20.0 mmol) was added 1,2-ethanedithiol (1.8 mL, 21.0 mmol) under Ar atmosphere. At 0 $^o$C, the mixed solution was then added BF$_3$.OEt$_2$ (1.0 mL, 8.0 mmol) dropwise. The mixture was stirred at 0 $^o$C for 10 min and gradually warmed to rt for 5 h. The stirred solution was treated with chloroform (30 mL) and washed with 10% NaOH (50 mL) at 0 ºC. The organic layer was washed with water (150 mL). The combined aqueous layers were extracted with chloroform. The combined organic solutions were dried (MgSO$_4$) and filtered. The filtrate was evaporated in vacuo to give **21** as white powder (3.39g, 75%): mp. 55-56 ºC (EtOH, lit.[14] 55-56 ºC); $^1$H NMR (CDCl$_3$, 300 MHz): $\delta$ 1.65-1.90 (m, 8 H), 2.04 (br, 2 H), 2.08-2.25 (m, 4 H), 3.22 (s, 4 H).

**Synthesis of 2-(trimethylsilylmethylene)adamantane 22.** According to the general procedure that described above, a toluene solution of 2-adamantone thioketal (226 mg, 1 mmol), Ni(acac)$_2$ (13 mg, 5 mmol%), P(*t*-Bu)$_2$Me (1.8 mL, 0.068 M, 12 mmol%) and trimethylsilylmethylmagnesium chloride (4.0 mL, 2.0 M, 8 mmol) was transformed into **22** as colorless liquid (160mg, 73 %). $^1$H NMR (CDCl$_3$, 300 MHz): $\delta$ 0.07 (s, 9 H), 1.70-2.10 (m, 12 H), 2.40 (br, 1 H), 2.72 (br, 1 H), 5.01 (s, 1 H).[15]

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