Supplementary Material for

Thiol Ester - Boronic Acid Coupling. A Mechanistically Unprecedented and General Ketone Synthesis.

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^{1}H NMR spectra were recorded on a Varian Inova 400 spectrometer using chloroform as a internal standard. IR spectra were recorded on ASI ReactIR^R 1000 FT-IR spectrometer with a silicone probe. TLC plates used for monitoring of reactions were Kieselgel 60 F_{254}, 0.25mm, purchased from E.Merck, Darmstad. Ethyl thiol, *p*-thiocresol, lauroyl chloride, 4-nitrobenzoyl chloride, triethylphosphine (1M solution in THF), diphenyl disulfide, acetoxyacetyl chloride, trifluoroacetic anhydride, chloroacetyl chloride, trimethylaluminum (2 M solution in hexanes), adamantoyl chloride, *p*-hydroxy ethylbenzoate, tris-2-furylphosphine, pyrazine-2-carboxylic acid were purchased from Aldrich. N,N-dimethylchloroacetamide was purchased from Fluka. All boronic acids and Cu(I) thiophene-2-carboxylate (CuTC) were obtained from Frontier Scientific, Inc. Solvents were purged with nitrogen for several hours, then dried and stored over 4Å sieves. All reactions were carried out under argon.

General Protocol for the Synthesis of Thiol Esters From Acyl Chlorides: The acyl chloride (0.10 mol) and thiol (0.10 mol) were dissolved in hexanes (150 mL) and the mixture was cooled to 0 °C. Triethylamine (0.11 mol) dissolved in hexanes (50 mL) was added drop wise within 30 minutes to the magnetically stirred reaction mixture. The reaction mixture was stirred overnight and allowed to reach ambient temperature. The white precipitate was filtered and washed with a 1:1 mixture of hexanes and Et_2O (2 x 20 mL). The filtrate was evaporated under reduced pressure to give analytically clean thiol ester.

***S*-(*p*-Nitrobenzoyl)mercaptoethane, 2:** Yield 68%. Yellowish crystals. M.p. 68-70 °C (lit.[1] 68.5 °C). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.30 (d, *J*(H,H) = 9.2 Hz, 2H), 8.11 (d, *J*(H,H) = 9.3 Hz, 2H), 3.13 (q, *J*(H,H) = 7.7 Hz, 2H), 1.38 (t, *J*(H,H) = 7.7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 195.7, 190.6, 141.7, 128.2, 123.8, 24.0, 14.5; IR(neat): ν~= 1664 cm^{-1}.(lit.[1]: 1660 cm^{-1}); Calcd for $C_9H_9NO_3S$: C, 51.17; H, 4.29; S, 15.18; Found: C, 51.15; H, 4.30; S, 15.05.

***S*-Lauroyl-*p*-mercaptotoluene, 5:** Yield 98%. Colorless oil. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.29 (d, *J*(H,H) = 8.4 Hz, 2H), 7.22 (d, *J*(H,H) = 8.0 Hz, 2H), 2.63 (t, *J*(H,H) = 7.2 Hz, 2H), 2.37 (s, 3H), 1.70 (q, *J*(H,H) = 8.0 Hz, 2H), 1.26 (br s, 16H), 0.88 (t, *J*(H,H) = 6.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 198.2, 139.5, 134.4, 129.9, 124.4, 43.6, 31.9, 29.6, 29.4, 29.3, 29.2, 28.9, 25.6, 22.7, 21.3, 14.1. IR(neat):ν~= 1710 cm^{-1} (C=O); HRMS(EI) Calcd for $C_{19}H_{30}OS$: 306.2017. Found: 306.2011 (M^+).

***S*-Adamantoyl-*p*-mercaptotoluene, 6:** Yield 78%. White crystals. M.p. 111-112 °C (ether/hexanes). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.27 - 7.19 (m, 4H), 2.37 (s, 3H), 2.09 (br s, 3H), 1.99 (d, *J*(H,H) = 2.7 Hz, 6H), 1.75 (d, *J*(H,H) = 2.4 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 204.6, 139.2, 136.9, 129.9, 48.9, 39.2, 36.4, 28.2, 21.3; IR(neat): ν~= 1691 cm^{-1}(C=O); Anal. Calcd for $C_{18}H_{22}OS$: C, 75.48; H, 7.74; S, 11.19; Found: C, 75.46; H, 7.64; 11.02.

***S*-Chloroacetyl-*p*-mercaptotoluene, 7:** Yield 92%. Yellowish solid. M.p. 42 °C (hexanes) (lit.[2] 40 °C). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.31 (d, *J*(H,H) = 8.4 Hz, 2H), 7.25 (d, *J*(H,H) = 8.4 Hz, 2H), 4.28 (s, 2H), 2.39(s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 192.9, 140.3, 134.5, 130.3, 122.8, 47.9, 21.4; IR(neat): ν~= 1695 cm^{-1}(C=O).

***S*-(2-Acetoxyacetyl)-*p*-mercaptotoluene, 8:** Yield 95%. Colorless oil. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.30 (d, *J* = 8.5 Hz, 2H), 7.23 (d, *J* = 8.5 Hz, 2H), 4.83 (s, 2H), 2.38 (s, 3H), 2.22 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 194.4, 169.8, 140.1, 134.7, 130.2, 121.9, 67.5, 21.3, 20.5; IR(neat): ν~= 1756, 1710 cm^{-1} (C=O); HRMS(EI) Calcd for $C_{11}H_{12}O_3S$: 224.0507. Found 224.0512 (M^+).

***S*-Benzoyl mercaptoacetic acid, *N*,*N*-dimethylamide, 1:** Thiobenzoic acid (6.90 g, 0.05 mol) was dissolved in THF (100 mL) together with *N*,*N*-dimethylchloroacetamide (6.87 g, 0.05 mol) and K_2CO_3 (13.00 g, 0.094 mol). The mixture was stirred for 18 h at room temperature under nitrogen. The suspension was filtered and volatiles were evaporated under reduced pressure. The viscous substance that formed after evaporation solidified and was recrystallized from Et_2O and hexanes yielding white crystals (8.14 g, 73%) M.p. 67-69 °C (hexane/ether); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.70 (dd, *J*(H,H) = 8.3 Hz, *J*(H,H) = 1.3 Hz, 2H), 7.57 (tt, *J*(H,H) = 7.7 Hz, *J*(H,H) = 1.3 Hz, 1H), 7.44 (t, *J*(H,H) = 8.0 Hz, 2H), 4.01 (s, 2H), 3.14 (s, 3H), 2.99 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 190.9, 167.4, 136.3, 133.6, 128.6, 127.3, 37.7, 36.0, 32.0; IR(neat): ν~= 1652 cm^{-1} (C=O); Anal. Calcd for $C_{11}H_{13}NO_2S$: C, 59.17; H, 5.87; N, 6.27; S, 14.36; Found: C, 59.00; H, 5.74; N, 6.27; S, 14.36.

***S*-(*p*-Hydroxybenzoyl)mercaptoethane, 3:** Trimethylaluminum (4 mL of 2M soln in hexanes, 0.008 mol) was added via syringe to a solution of ethanethiol (2 mL, 0.027 mol) dissolved in toluene (15 mL) at room temperature. After an immediate, vigorous evolution of gas, the mixture was stirred for 30 minutes at room temperature and additional 30 minutes at 50 °C under argon. Then, ethyl(*p*-hydroxy)benzoate (1.60 g, 0.010 mol) was added and the reaction mixture was stirred at 55 °C under argon for 18 h. It was cooled down to 0 °C and 5% aq. HCl (15 mL) was added. Extraction with Et_2O (30 + 15 mL) followed. The collected organic extracts were dried over $MgSO_4$, filtered, and evaporated under reduced pressure. A yellow oil solidified and was recrystallized from Et_2O/hexanes to give **3** as yellowish crystals (1.29 g, 74%). M.p. 67-70 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.90 (td, *J*(H,H) = 8.8 Hz, *J*(H,H) = 2.9 Hz, 2H), 6.89 (td, *J*(H,H) = 8.8 Hz, *J*(H,H) = 2.9 Hz, 2H), 3.06 (q, *J*(H,H) = 7.7 Hz, 2H), 1.34 (t, *J*(H,H) = 7.7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 192.1, 160.6, 129.9, 129.6,

115.4, 23.4, 14.7; IR(neat): ν~= 3358 cm^{-1}(OH), 1633 cm^{-1} (C=O); HRMS(EI) Calcd for $C_9H_{10}O_2S$: 182.0401. Found 182.0400 (M^+).

Pyrazine-2-carbothioic acid, *S*-phenyl ester, 4: Triethylphosphine (11 mL, 0.011 mol) was added via syringe at room temperature to a solution of pyrazine-2-carboxylic acid (1.24 g, 0.010 mol) and diphenyl disulfide (2.18 g, 0.010 mol) dissolved in THF (20 ml). The reaction mixture was stirred for 8 h under nitrogen. An Et_2O:hexanes mixture (40 ml, 1:1) was then added and the product crystallized upon standing in a refrigerator at 4 °C as yellowish needles (1.04 g, 48 %). M.p. 150-152 °C). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.17 (s, 1H), 8.85 (d, *J*(H,H) = 2.4 Hz, 1H), 8.72 (d, *J*(H,H) = 2.4 Hz, 1H), 7.54-7.26 (m, 5H); IR(neat): ν~= 1683 cm^{-1} (C=O); Calcd for $C_{11}H_8N_2OS$: C, 61.09; H, 3.73; N, 12.95; S, 14.83; Found: C, 61.26; H, 3.79; N, 12.74; S, 14.83.

***S*-(Trifluoroacetyl)-*p*-methylthiophenol, 9**: Trifluoroacetyl anhydride (15.00 mL, 0.11 mol) was placed in a dry flask and cooled under argon to –20 °C. P-Thiocresol (12.40 g, 0.10 mol) was than added with KOAc (0.020 g, 0.2 mmol) as a catalyst. The slightly exothermic reaction was stirred and allowed to slowly warm to room temperature. After 5 h, the mixture was poured onto ice and extracted with hexanes/Et_2O (1:1 mixture, 100 mL) to give, after drying and evaporation, **9** as a viscous liquid (19.93 g, 91%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.34 (td, *J*(H,H) = 8.0 Hz, *J*(H,H) = 1.6 Hz, 2H), 7.29 (d, *J*(H,H) = 8.5 Hz, 2H), 2.41 (s, 3H); IR(neat): ν~= 1718 cm^{-1} (C=O).[3]

General Cross-coupling Procedure: Thiol ester (0.500 mmol), Cu (I) thiophene-2-carboxylate (0.143 g, 0.750 mmol), boronic acid (0.550 mmol), $Pd_2dba_3.CHCl_3$ (0.003 g, 0.003 mmol) and tris-2-furylphosphine (0.006 g, 0.006 mmol) were placed in reaction vessel that was flushed with argon. THF (6 mL) was added and the mixture was stirred for 18 h at 50 °C. Et_2O (15 mL) was added and the suspension was washed with 5% aq. HCl (10 mL) and water (10 mL). The organic layer was dried over $MgSO_4$, filtered, and evaporated. The resulting solid was subjected to radial chromatography (Chromatotron) on silica gel plate (4 mm) with a hexanes/Et_2O gradient as eluent.

(2-Methoxyphenylcarbonyl)benzene (Table entry 1): Yield 88%. Off-white solid. M.p. 35-37 °C (hexanes, Lit.[4] 35-36 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.82 (d, *J*(H,H) = 7.7 Hz, 2H), 7.57- 7.31 (m, 5H), 7.06- 6.98 (m, 2H), 3.72 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.5, 157.3, 137.7, 132.9, 131.9, 129.8, 129.6, 128.7, 128.2, 120.4, 111.4, 55.5; IR(neat): ν~= 1663 cm^{-1} (C=O, Lit.:[5] 1660 cm^{-1}).

(3-Methoxyphenylcarbonyl)benzene (Table entry 2): Yield 83%. White crystals. M.p. 39-40 °C (hexanes, Lit.[6] 37-38 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.82 (dd, *J*(H,H) = 8.0 Hz, *J*(H,H) = 1.3 Hz, 2H), 7.59 (tt, *J*(H,H) = 7.7 Hz, *J*(H,H) = 1.3 Hz, 1H), 7.48 (t, *J*(H,H) = 7.7 Hz, 2H), 7.38- 7.33 (m, 3H), 7.15- 7.12 (m, 1H), 3.86 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.6, 159.5, 138.8, 137.6, 132.4, 130.0, 129.2, 128.2, 122.9, 118.9, 114.3, 55.4; IR(neat): ν~= 1662 cm^{-1} (C=O), Lit.[7]).

Phenyl-2-naphthylketone (Table entry 3): Yield 79%. Off white crystals. M.p. 79-80 °C (hexanes, commercial sample: 81 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.27 (s, 1H), 7.96- 7.86 (m, 6H), 7.65-7.50 (m, 5H); IR(neat): ν~= 1656 cm^{-1} (C=O). ^{1}H NMR spectra is consistent with that of the commercial sample.

3,4-Methylenedioxyphenyl-4-nitrophenylketone (Table entry 4): Yield 85%. Off-white crystals. M.p. 120-122 °C (hexanes); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.32 (d, *J*(H,H) = 8.0 Hz, 2H), 7.86 (d, *J*(H,H) = 9.3 Hz, 2H), 7.36 (d, *J*(H,H) = 1.6 Hz, 1H), 7.32 (dd, *J*(H,H) = 8.5 Hz ,*J*(H,H) = 2.0 Hz, 1H), 6.89 (d, *J*(H,H) = 8.4 Hz, 1H), 6.09 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 193.1, 152.4, 149.5, 148.4, 143.6, 130.6, 130.3, 127.3, 123.4, 109.5, 107.9, 102.1; IR(neat): ν~= 1656 cm^{-1} (C=O). HRMS(EI) Calcd for $C_{14}H_9NO_5$: 271.0490. Found: 271.0481 (M^+); Anal. Calcd for $C_{14}H_9NO_5$: C, 62.00; H, 3.34; N, 5.16; Found: C, 61.96; H, 3.36; N, 5.11.

4-Hydroxybenzophenone (Table entry 5): Yield 81%. White crystals (Et_2O:hexanes). M.p. 134-135 °C, Lit.[8] 136-137 °C). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.80-7.75 (m, 4H), 7.58 (t, *J*(H,H) = 7.7 Hz, 1H), 7.48 (t, *J*(H,H) = 7.7 Hz, 2H), 6.94 (d, *J*(H,H) = 8.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.6, 160.5, 137.9, 133.1, 132.2, 129.8, 129.7, 128.2, 115.3; IR(neat): ν~= 3212 cm^{-1} (OH), 1633 cm^{-1} (C=O).[8]

3-Phenyl-1-pyrazin-2-yl-propenone (Table entry 6): Yield 81%. Yellow crystals. M.p. 107-109 °C (Et_2O: hexanes, (Lit.[9] 106-107.5 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.38 (s, 1H), 8.78 (d, *J*(H,H) = 2.4 Hz, 1H), 8.70 (app s, 1H), 8.19 (d, *J*(H,H) = 15.7 Hz, 1H), 7.99 (d, *J*(H,H) = 16.0 Hz, 1H), 7.74- 7.71 (m, 2H), 7.45- 7.42 (m, 3H); IR(neat): ν~= 1671 cm^{-1} (C=O, Lit.:[9] 1673 cm^{-1}).

3-(1-Undecylcarbonyl)nitrobenzene (Table entry 7): Yield 79%. Off-white crystals. M.p. 40-42 °C (hexanes); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.77 (t, *J*(H,H) = 1.6 Hz, 1H), 8.43- 8.28 (m, 1H), 8.29 (dt, *J*(H,H) = 7.7 Hz, *J*(H,H) = 1.3 Hz, 1H), 7.68 (t, *J*(H,H) = 8.0 Hz, 1H), 3.02 (t, *J*(H,H) = 7.7 Hz, 2H), 1.76 (pent, *J*= 7.2 Hz, 2H), 1.37-1.26 (m, 16 H), 0.87 (t, *J*= 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 198.2, 148.4, 138.2, 133.6, 129.8, 127.2, 122.9, 38.8, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 24.0, 22.7, 14.1; IR(neat): ν~= 1698 cm^{-1} (C=O); HRMS(EI) Calcd for $C_{18}H_{27}O_3N$: 305.1991. Found 305.1999 (M^+).

2-(1-Undecylcarbonyl)-4-methoxybenzaldehyde (Table entry 8): Yield 75%. White crystals. M.p. 45-46 °C (hexanes); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 10.24 (s, 1H), 7.77 (d, *J*(H,H) = 8.3 Hz, 1H), 7.34 (d, *J*(H,H) = 3.2 Hz, 1H), 7.10 (dd, *J*(H,H) = 8.0 Hz, *J*(H,H) = 2.8 Hz, 1H), 3.90 (s, 3H), 2.94 (t, *J*(H,H) = 8.0 Hz, 2H), 1.73 (m, 2H), 1.25 (br s, 16H), 0.87 (t, *J*(H,H) = 6.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 201.5, 192.4, 162.3, 139.6, 132.7, 130.8, 118.3, 112.8, 55.8, 40.1, 31.9, 29.6, 29.5, 29.4, 29.3, 24.5, 22.7, 14.1; IR(neat): ν~= 1695 cm^{-1}, 1675 cm^{-1} (C=O); HRMS(EI) Calcd for $C_{20}H_{30}O_3$: 318.2195. Found 318.2207 (M^+).

3,4-Methylenedioxyphenylcarbonyl-1-adamantane (Table entry 9): Yield 52%. White crystals. M.p. 114-115 °C (hexanes); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.30 (dd, *J*(H,H) = 8.0 Hz, *J*(H,H) = 1.6 Hz, 1H), 7.19 (d, *J*(H,H) = 1.6 Hz, 1H), 6.80 (d, *J*(H,H) = 8.0 Hz, 1H), 6.00 (s, 2H), 2.07 (s, 3H), 2.02 (d, *J*(H,H) = 2.9 Hz, 6H), 1.80-1.70 (m, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 207.1, 149.5, 147.3, 132.8, 123.1, 108.6, 107.5, 101.4, 46.8, 39.4, 36.5, 28.2;IR(neat): ν~= 1644 cm^{-1} (C=O); HRMS(EI) Calcd for $C_{18}H_{20}O_3$: 284.14124. Found: 284.14201; Anal. Calcd for $C_{18}H_{20}O_3$: C, 76.03; H, 7.09. Found: C, 75.99; H, 7.05.

2-Chloro-1-naphthalen-2-yl-ethanone (Table entry 10): Yield 57%. White crystals. M.p. 64-65 °C (hexanes, Lit.[10] 65 °C). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.47 (s, 1H), 8.03-7.88 (m, 4H), 7.66-7.56 (m, 2H), 4.85 (s, 2H); IR(neat): ν~= 1698 cm^{-1} (C=O).

4-(2-Acetoxycarbonyl)benzoic acid, methyl ester (Table entry 11): Yield 88%. White crystals. M.p.80-82 °C (hexane/ether). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.15 (d, *J*(H,H)= 5.3 Hz, 2H), 7.97 (d, *J*(H,H)= 4.8 Hz, 2H), 5.34 (s, 2H), 3.96 (s, 3H), 2.24 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 191.8, 170.4, 166.0, 137.2, 134.6, 130.0, 127.7, 66.1, 52.6, 20.5; IR(neat): ν~= 1752 cm^{-1} (C=O), 1720 cm^{-1} (C=O), 1702 cm^{-1} (C=O); Anal. Calcd for $C_{12}H_{12}O_5$: C, 61.01; H, 5.12. Found: C, 61.54; H, 5.09.

***m*-Nitrophenyltrifluoromethylketone (Table entry 12):** Yield 63%. Yellowish crystals. M.p. 54-56 °C (Et_2O/hexanes, Lit.[11] 58-59 °C). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.44 (app s, 1H), 8.19- 8.16 (m, 1H), 7.92-8.89 (m, 1H), 7.59 (t, *J*(H,H) = 8.4 Hz, 1H). The ^{1}H NMR is consistent with literature data. IR(neat): ν~= 1756 cm^{-1} (C=O).

2-(Trifluoromethylcarbonyl)styrene (Table entry 13): Yield 93%. Colorless oil. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.98 (d, *J*= 16.0 Hz, 1H), 7.65 (d, *J*= 6.9 Hz, 2H), 7.51-7.43 (m, 3H), 7.03 (d, *J*= 16.0 Hz, 1H); IR(neat): ν~= 1718 cm^{-1} (C=O).[12]

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