

## Supporting Information

# Tandem Nucleophilic Addition-Oppenauer Oxidation of Aromatic Aldehydes to Aryl Ketones with Triorganoaluminium Reagents

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### General Experimental Section:

**Analytic methods.** All the reactions were carried out under argon or nitrogen atmosphere. <sup>1</sup>H NMR (400 MHz), and <sup>13</sup>C NMR (100 MHz) were recorded on Bruker AV400 NMR spectrometer with CDCl<sub>3</sub> as solvent. Chemical shifts of <sup>1</sup>H and <sup>13</sup>C NMR spectra are reported in parts per million (ppm) with TMS as an internal standard. Column chromatography was performed on silica gel 300 - 400 mesh. Analytical thin layer chromatography (TLC) was performed on pre-coated, glass-backed silica gel plates. Visualization of the developed chromatogram was performed by UV absorbance (254 nm). IR spectra were recorded as KBr disks on an Alpha Centauri FT-IR spectrometer. EI mass spectra and HRMS were done on QP-1000A GCMS spectrometer by EI ionization at 70 eV. ESI mass spectra were recorded on a Bruker Esquire 6000.

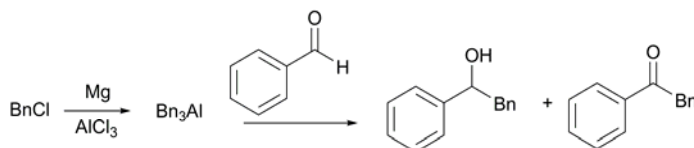
### General Procedure A: in situ preparation of triorganoaluminium reagents

To a dry, three-necked 100 mL round-bottomed flask equipped with an argon inlet, a thermometer, and an addition funnel was added magnesium turnings (0.36 g, 15.0 mmol), AlCl<sub>3</sub> (0.46g, 3.5 mmol) and a small piece of iodine added. After changed the air in the reaction system to argon or nitrogen atmosphere, 2 mL of THF was added and the reaction mixture was stirred until the brown colour disappeared. Organo halides (12.0 mmol) in THF (10 mL) was then added under ice-water bath and the reaction mixture was stirred overnight, allowing the reaction temperature raise to room temperature.

### General Procedure B: tandem triorganoaluminium reagent addition-Oppenauer oxidation of aldehydes:

To the triorganoaluminium reagent prepared above was added a solution of aldehyde (3.5 mmol) in pinacolone (10 mL) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{L}$ ) was then added. The reaction mixture was stirred for 2 days and then quenched by aqueous  $\text{NH}_4\text{Cl}$  (10 mL). 10 mL of ethyl acetate was then added and the organic phase was separated, washed with 10 mL of water and then with 10 mL of brine. The water phase was extracted with ethyl acetate (2 $\times$ 10 mL). The organic phase was combined, dried ( $\text{Na}_2\text{SO}_4$ ) and concentrated under reduced pressure. Ketones were obtained by column chromatography on silica gel using petroleum/ethyl acetate as an eluent. All products were characterized by IR,  $^1\text{H}$  NMR, and  $^{13}\text{C}$  NMR.

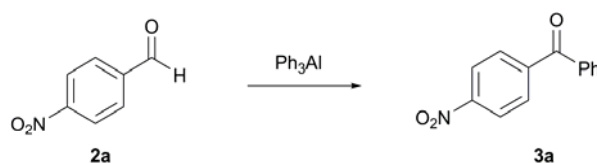
### Reaction of the benzyl magnesium reagent with benzaldehyde in the presence of $\text{AlCl}_3$



To a dry, three-necked 100 mL round-bottomed flask equipped with an argon inlet, a thermometer, and an addition funnel was added magnesium turnings (0.36 g, 15.0 mmol),  $\text{AlCl}_3$  (0.46g, 3.5 mmol) and a small piece of iodine. After changed the air in the reaction system to nitrogen atmosphere, 2 mL of THF was added and the reaction mixture was stirred until the brown colour disappeared. Benzyl chlorides (3.5 mmol) in THF (10 mL) was then added under ice-water bath for 3 hours, allowing the reaction temperature raise to room temperature. A solution of benzaldehyde (0.37g, 3.5 mmol) in THF (10 mL) was then added under ice-water bath and the reaction mixture was then stirred at room temperature overnight. After usual work-up, 0.45 g of 1,2-diphenylethanol (2.27 mmol) and 0.16 g of 1,2-diphenylethanone (0.8 mmol) was obtained.

### Synthesis and characterization of products

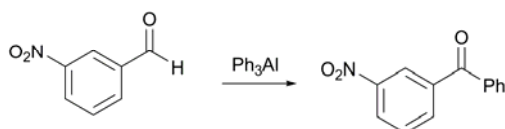
#### 3a: 4-Nitrobenzophenone



According to general procedure B: A solution of 4-nitrobenzaldehyde (0.53g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{L}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure

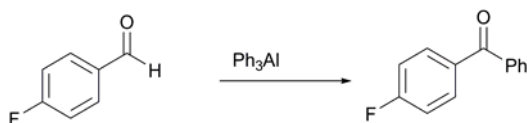
product was obtained as a yellowish solid (0.73 g, 3.22 mmol, 92 %) after purification on silica gel (10:1~3:1 petroleum ether : EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 3101, 2924, 1649, 1595, 1512, 1445, 1356, 1314, 1276, 1145, 1105, 928;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 7.53 (t,  $J = 8.0$  Hz, 2H), 7.66 (t,  $J = 7.6$  Hz, 1H), 7.80(d,  $J = 7.2$  Hz, 2H), 7.94 (dt,  $J = 8.8, 2.0$  Hz, 2H), 8.34 (dt,  $J = 8.8, 2.0$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 123.49, 128.64, 130.05, 130.64, 133.42, 136.25, 142.84, 149.79, 194.72.

### 3b: 3-Nitrobenzophenone



According to general procedure B: A solution of 3-nitrobenzaldehyde (0.53g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a yellowish solid (0.62 g, 2.73 mmol, 78 %) after purification on silica gel (10:1~3:1 petroleum ether : EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 3091, 2924, 2862, 1988, 1834, 1654, 1613, 1535, 1471, 1441, 1350, 1279, 1154, 1082, 971;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 7.54 (t,  $J = 7.2$  Hz, 2H), 7.67 (tt,  $J = 7.6, 2.0$  Hz, 2H), 7.71(t,  $J = 8.0$  Hz, 1H), 7.79-7.83 (m, 1H), 8.15 (ddd,  $J = 7.6, 1.6, 1.2$  Hz, 1H), 8.45 (ddd,  $J = 8.4, 2.4, 1.2$  Hz, 1H), 8.63 (td,  $J = 2.0, 0.4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 124.70, 126.70, 128.71, 129.61, 129.98, 133.34, 135.40, 136.24, 139.05, 148.07, 194.13;

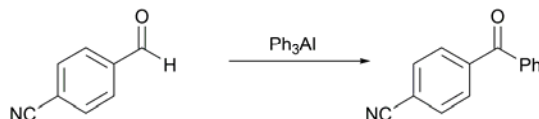
### 3c: 4-Fluorobenzophenone



According to general procedure B: A solution of *p*-fluorobenzaldehyde (0.44g, 3.5 mmol) in pinacolone (10 mL) was slowly added to triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.59 g, 2.94 mmol, 84 %) after purification on silica gel (10:1~5:1 petroleum ether : EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 2926, 2857, 1651, 1598, 1503, 1446, 1404, 1279, 1227, 1151, 1097, 937, 851;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 7.16 (t,  $J = 8.4$  Hz, 2H), 7.49 (t,  $J = 8.0$  Hz, 2H), 7.60 (t,  $J = 7.6$  Hz, 1H), 7.77 (tt,  $J = 6.8, 1.6, 0.4$  Hz, 2H), 7.82-7.87 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$

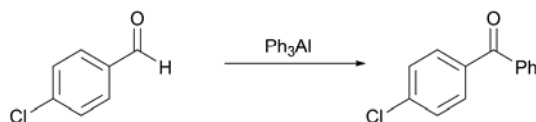
(ppm): 115.30, 115.52, 128.32, 129.82, 132.42, 132.57, 132.66, 133.75, 133.78, 137.46, 164.08, 166.60, 195.19.

### 3d: 4-Cyanobenzophenone



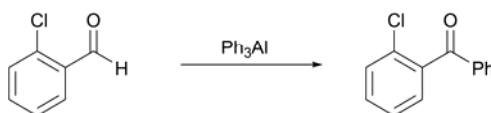
According to general procedure B: A solution of *p*-cyanobenzaldehyde (0.46g, 3.5 mmol) in pinacolone (10 mL) was slowly added to triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours, CF<sub>3</sub>CO<sub>2</sub>H (0.175 mmol, 14 μl) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.41 g, 2.0 mmol, 57 %) after purification on silica gel (10:1~5:1 petroleum ether : EtOAc). IR (KBr)  $\nu$ (cm<sup>-1</sup>): 2992, 2853, 2224, 1647, 1595, 1444, 1401, 1314, 1280, 1140, 1112, 1071, 928, 857 cm<sup>-1</sup>. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 7.52 (t, *J* = 7.6 Hz, 2H), 7.65 (tt, *J* = 7.6, 1.6 Hz, 1H), 7.77-7.83 (m, 4H), 7.89 (dt, *J* = 8.8, 2.0 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 115.62, 117.96, 128.64, 130.05, 130.19, 132.01, 133.32, 136.33, 141.30, 194.97.

### 3e: 4-Chlorobenzophenone



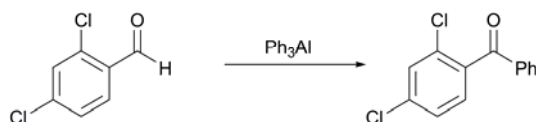
According to general procedure B: A solution of *p*-chlorobenzaldehyde (0.49g, 3.5 mmol) in pinacolone (10 mL) was slowly added to triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours, CF<sub>3</sub>CO<sub>2</sub>H (0.175 mmol, 14 μl) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.69 g, 3.2 mmol, 91 %) after purification on silica gel (10:1~5:1 petroleum ether : EtOAc). IR (KBr)  $\nu$ (cm<sup>-1</sup>): 2924, 2854, 1649, 1582, 1483, 1443, 1398, 1281, 1173, 1087, 1010, 922; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 7.44-7.53 (m, 4H), 7.61 (tt, *J* = 7.6, 2.0 Hz, 1H), 7.75-7.80 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 128.38, 128.61, 129.91, 131.45, 132.63, 135.81, 137.19, 138.87, 195.51;

### 3f: 2-Chlorobenzophenone



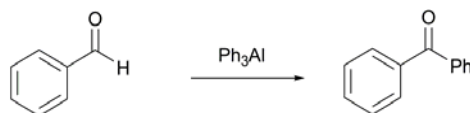
According to general procedure B: A solution of *o*-chlorobenzaldehyde (0.49g, 3.5 mmol) in pinacolone (10 mL) was slowly added to triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours, CF<sub>3</sub>CO<sub>2</sub>H (0.175 mmol, 14 μl) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.40 g, 1.86 mmol, 53 %) after purification on silica gel (10:1~5:1 petroleum ether : EtOAc). IR (KBr)  $\nu$  (cm<sup>-1</sup>): 2923, 2852, 1745, 1673, 1595, 1455, 1377, 1316, 1288, 1255, 1057, 929; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 7.36-7.40 (m, 2H), 7.45-7.50 (m, 4H), 7.61 (t, *J* = 7.2 Hz, 1H), 7.82 (dd, *J* = 8.4, 1.2 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 126.65, 128.58, 129.10, 130.05, 131.09, 133.67, 136.46, 138.60, 195.26;

### 3g: 2,4-diChlorobenzophenone



According to general procedure B: A solution of 2,4-dichlorobenzaldehyde (0.61 g, 3.5 mmol) in pinacolone (10 mL) was slowly added to triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours, CF<sub>3</sub>CO<sub>2</sub>H (0.175 mmol, 14 μl) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.59 g, 2.35 mmol, 67%) after purification on silica gel (10:1~5:1 petroleum ether : EtOAc). IR (KBr)  $\nu$  (cm<sup>-1</sup>): 2924, 2854, 1654, 1580, 1516, 1454, 1376, 1276, 1103, 1054, 929; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 7.33 (d, *J* = 8.0 Hz, 1H), 7.36 (dt, *J* = 8.4, 1.6 Hz, 1H), 7.46 (t, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 0.8 Hz, 1H), 7.62 (t, *J* = 8.0 Hz, 1H), 7.790 (d, *J* = 8.0 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 127.085, 128.670, 129.973, 130.141, 132.396, 133.867, 136.192, 136.611, 136.908, 194.194.

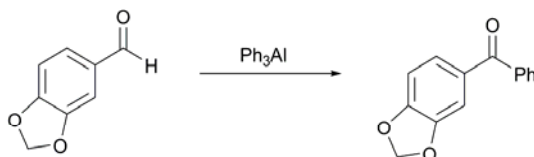
### 3h: benzophenone



According to general procedure B: A solution of benzaldehyde (0.37g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours, CF<sub>3</sub>CO<sub>2</sub>H (0.175 mmol, 14 μl) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.50 g, 2.73 mmol, 78 %) after purification on silica gel (10:1~5:1 petroleum ether : EtOAc). IR (KBr)  $\nu$  (cm<sup>-1</sup>): 2924, 2867, 1651, 1593, 1447, 1317, 1277, 1174, 1150, 1074, 1026, 997, 939, 916, 864, 813, 764, 702; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 7.48-7.51 (t, *J* = 7.7 Hz, 4H),

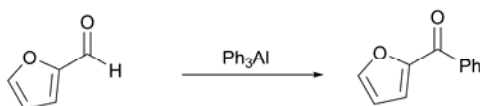
7.59-7.61 (m, 2H), 7.81-7.82 (t,  $J = 4.1$  Hz, 4H);  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ): 128.3, 130.1, 132.4, 137.6, 196.8.

**3i: benzo[d][1,3]dioxol-5-yl(phenyl)methanone**



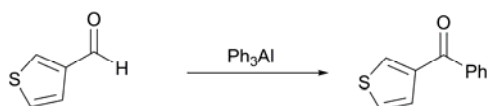
According to general procedure B: A solution of Piperonyl aldehyde (0.53 g, 3.5 mmol) in pinacolone (10 mL) was slowly added to triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.57 g, 2.52 mmol, 72%) after purification on silica gel (10:1~3:1 petroleum ether : EtOAc). IR (KBr)  $\nu(\text{cm}^{-1})$ : 2904, 2783, 1651, 1601, 1485, 1442, 1353, 1280, 1260, 1175, 1122, 1091, 1038, 955, 932;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 6.07 (s, 2H), 6.85 (dt,  $J = 8.4, 0.8$  Hz, 1H), 7.30-7.40 (m, 2H), 7.47 (t,  $J = 8.0$  Hz, 2H), 7.57 (t,  $J = 7.2$  Hz, 1H), 7.74 (d,  $J = 7.2$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 101.90, 107.93, 109.77, 126.67, 127.77, 128.75, 129.61, 138.05, 147.68, 151.46, 195.14;

**3j: furan-2-yl(phenyl)methanone**



According to general procedure B: A solution of furfuraldehyde (0.34 g, 3.5 mmol) in pinacolone (10 mL) was slowly added to triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a colourless liquid (0.25 g, 1.47 mmol, 42%) after purification on silica gel (10:1~3:1 petroleum ether : EtOAc). IR (KBr)  $\nu(\text{cm}^{-1})$ : 2924, 2852, 1647, 1599, 1561, 1463, 1390, 1297, 1226, 1178, 1148, 1080, 1020, 956;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 6.60 (q,  $J = 2.0$  Hz, 1H), 7.24 (dd,  $J = 3.2, 0.8$  Hz, 1H), 7.50 (t,  $J = 7.2$  Hz, 2H), 7.60 (tt,  $J = 7.2, 0.8$  Hz, 1H), 7.715-7.717(m, 1H), 7.94-7.99(dd,  $J = 7.6, 1.2$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 112.16, 120.53, 128.37, 129.23, 132.53, 137.20, 147.06, 152.22, 182.52;

**3k: phenyl(thiophen-3-yl)methanone**



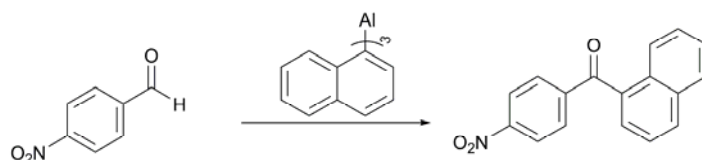
According to general procedure B: A solution of 3-Thiophenecarboxaldehyde (0.39 g, 3.5 mmol) in pinacolone (10 mL) was slowly added to triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a colourless liquid (0.45 g, 2.38 mmol, 68%) after purification on silica gel (10:1~3:1 petroleum ether : EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 3109, 2920, 2851, 1636, 1594, 1575, 1511, 1446, 1412, 1386, 1276, 1196, 1174, 1135, 1076, 999, 966;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 7.39 (dd,  $J = 4.8, 3.2$  Hz, 1H), 7.49 (t,  $J = 7.6$  Hz, 2H), 7.58 (dt,  $J = 7.6, 2.4$  Hz, 1H), 7.61 (dd,  $J = 4.8, 0.8$  Hz, 1H), 7.85 (dt,  $J = 8.0, 1.6$  Hz, 2H), 7.94 (dd,  $J = 3.6, 1.2$  Hz, 1H);  $^{13}\text{C}$  NMR (400 Hz,  $\text{CDCl}_3$ ): 126.16, 128.32, 128.54, 128.64, 129.31, 132.26, 132.36, 133.90, 138.55, 141.21, 189.96; ESI-MS  $m/z$  ( $\text{M}+\text{H}^+$ ) 189.1.

### 3l: (E)-chalcone



According to general procedure B: A solution of cinnamaldehyde (0.46g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared triphenyl aluminium reagent under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a yellowish solid (0.35 g, 1.68 mmol, 48 %) after purification on silica gel (10:1~5:1 petroleum ether: EtOAc). IR (KBr),  $\nu$  ( $\text{cm}^{-1}$ ): 3028, 1662, 1606, 1573, 1447, 1400;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 7.43 (t,  $J = 2.6$  Hz, 3H), 7.50-7.54 (m, 2H), 7.57-7.62 (m, 2H), 7.65-7.67 (m, 2H), 7.83 (d,  $J = 15.7$  Hz, 1H), 8.02-8.05 (m, 2H);  $^{13}\text{C}$  NMR (100 Hz,  $\text{CDCl}_3$ ): 121.99, 128.41, 128.46, 128.54, 128.59, 128.66, 128.92, 130.52, 132.76, 134.81, 138.13, 144.82, 190.52;

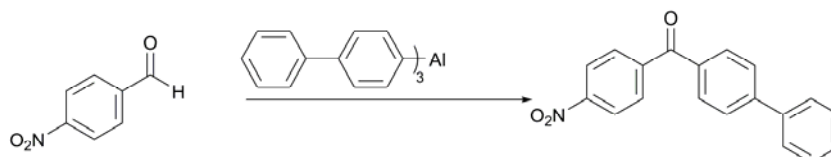
### 5a: naphthalen-1-yl(4-nitrophenyl)methanone



According to general procedure B: A solution of 4-nitrobenzaldehyde (0.53g, 3.5 mmol) in pinacolone (10 mL) was added to a previously prepared tri(1-naphthyl)aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.55 g, 2.0 mmol,

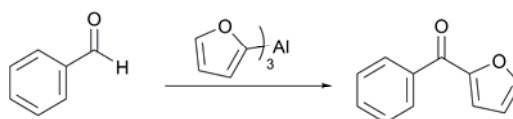
57%) after purification on silica gel (10:1~5:1 petroleum ether: EtOAc). IR (KBr)  $\nu$  (cm<sup>-1</sup>): 2924, 2853, 1678, 1598, 1523, 1460, 1346, 1315, 1278, 1246, 1199, 1105, 1045, 1012, 911; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 7.52-7.62 (m, 4H), 7.95-7.98 (m, 1H), 7.99-8.04 (m, 2H), 8.08 (d, *J* = 8.0 Hz, 1H), 8.15-8.20 (m, 1H), 8.30-8.34 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 123.55, 124.17, 125.28, 126.75, 127.79, 128.53, 128.84, 130.68, 131.06, 132.53, 133.72, 134.55, 143.25, 150.12, 195.91;

### 5b: biphenyl-4-yl(4-nitrophenyl)methanone



According to general procedure B: A solution of 4-nitrobenzaldehyde (0.53g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(4-biphenyl)aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours, CF<sub>3</sub>CO<sub>2</sub>H (0.175 mmol, 14  $\mu$ l) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.92 g, 3.05 mmol, 87%) after purification on silica gel (10:1~5:1 petroleum ether: EtOAc). IR (KBr)  $\nu$  (cm<sup>-1</sup>): 2920, 2851, 1652, 1600, 1518, 1454, 1400, 1350, 1317, 1286, 1103, 931, 850, 756, 730cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 7.44 (tt, *J* = 7.6, 1.2 Hz, 1H), 7.51 (tt, *J* = 7.2, 1.6 Hz, 2H), 7.66 (t, *J* = 7.2, 1.6 Hz, 2H), 7.75 (dt, *J* = 8.4, 2.0 Hz, 2H), 7.88 (dt, *J* = 8.4, 2.0 Hz, 2H), 7.97 (dt, *J* = 8.8, 2.0 Hz, 2H), 8.36 (dt, *J* = 8.8, 2.0 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 123.57, 127.33, 128.53, 129.01, 130.61, 130.78, 134.58, 139.82, 143.12, 146.34, 149.83, 194.35.

### 5c: 2-benzoylfurane



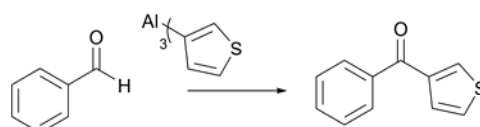
To a dry, three-necked 100 mL round-bottomed flask equipped with an argon inlet, an addition funnel was placed a magnetic stirring bar and the air in the reaction system was then changed to argon. Furan (0.82g, 0.86 mL, 12.0 mmol) and Tetramethylethylenediamine (2.8g, 3.6 mL, 24 mmol) in 5 ml of THF was added and the reaction system was cooled to -78 °C. BuLi in hexane (2.5 M, 5.8 mL, 14.4 mmol) was then added and the reaction temperature was raise to -40 °C gradually. AlCl<sub>3</sub> (0.46g, 3.5 mmol) in 5 mL of THF was added and the reaction mixture was stirred until the the reaction temperature raise to room temperature.

A solution of benzaldehyde (0.37g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(2-furanyl)aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours, CF<sub>3</sub>CO<sub>2</sub>H (0.175 mmol, 14  $\mu$ l)



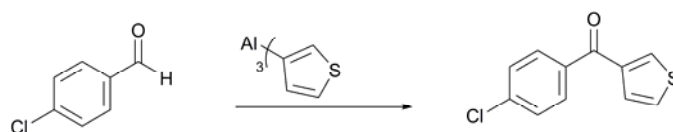
was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a colorless liquid (0.22 g, 1.27 mmol, 36%) after purification on silica gel (10:1~5:1 petroleum ether: EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 2924, 2852, 1647, 1599, 1561, 1463, 1390, 1297, 1226, 1178, 1148, 1080, 1020, 956;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 6.60 (q,  $J = 2.0$  Hz, 1H), 7.24 (dd,  $J = 3.2, 0.8$  Hz, 1H), 7.50 (t,  $J = 7.2$  Hz, 2H), 7.60 (tt,  $J = 7.2, 0.8$  Hz, 1H), 7.715-7.717(m, 1H), 7.94-7.99(dd,  $J = 7.6, 1.2$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 112.16, 120.53, 128.37, 129.23, 132.53, 137.20, 147.06, 152.22, 182.52.

### 5d: 3-Benzoylthiophene



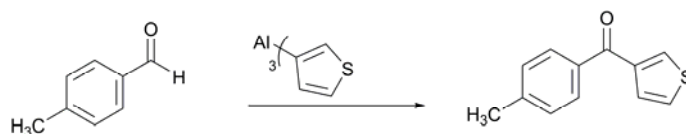
According to general procedure B: A solution of benzaldehyde (0.37g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(3-thienyl)aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a colorless liquid (0.44 g, 2.35 mmol, 67%) after purification on silica gel (10:1~5:1 petroleum ether: EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 2924, 2852, 1647, 1599, 1561, 1463, 1390, 1297, 1226, 1178, 1148, 1080, 1020, 956;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 6.60 (q,  $J = 2.0$  Hz, 1H), 7.24 (dd,  $J = 3.2, 0.8$  Hz, 1H), 7.50 (t,  $J = 7.2$  Hz, 2H), 7.60 (tt,  $J = 7.2, 0.8$  Hz, 1H), 7.715-7.717(m, 1H), 7.94-7.99(dd,  $J = 7.6, 1.2$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 112.16, 120.53, 128.37, 129.23, 132.53, 137.20, 147.06, 152.22, 182.52.

### 5e: 3-(4-Chlorobenzoyl)thiophene



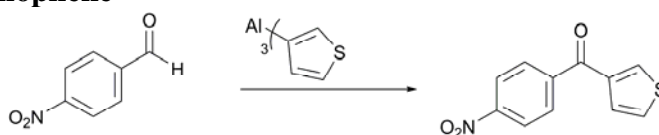
According to general procedure B: A solution of 4-chlorobenzaldehyde (0.49g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(3-thienyl)aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.59 g, 2.66 mmol, 76%) after purification on silica gel (10:1~5:1 petroleum ether: EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 3074, 2918, 2849, 1630, 1587, 1516, 1463, 1411, 1351, 1293, 1236, 1089, 1059, 1012, 948;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 7.17 (dd,  $J = 4.8, 4.0$  Hz, 1H), 7.48 (dd,  $J = 6.8, 2.0$  Hz, 2H), 7.63 (dd,  $J = 3.6, 0.8$  Hz, 1H), 7.74 (dd,  $J = 4.8, 1.2$  Hz, 1H), 7.82 (dt,  $J = 8.4, 2.0$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 128.03, 128.74, 130.57, 134.52, 134.75, 136.38, 138.26, 143.3, 193.93;

### 5f: 3-(4-methylbenzoyl) thiophene



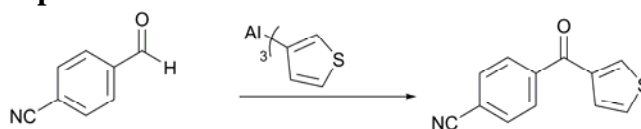
According to general procedure B: A solution of 4-methylbenzaldehyde (0.42g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(3-thienyl)aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours, CF<sub>3</sub>CO<sub>2</sub>H (0.175 mmol, 14 μl) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.43 g, 2.14 mmol, 61%) after purification on silica gel (10:1~5:1 petroleum ether: EtOAc). M.P.61-63 °C; IR (KBr)  $\nu$ (cm<sup>-1</sup>): 3097, 2921, 2852, 1828, 1730, , 1623, 1511, 1413, 1352, 1290, 1229, 1180, 1128, 1082, 1049; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 2.45 (2.45, s, 3H), 7.16 (dd, *J* = 4.8, 3.6 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.64 (dd, *J* = 2.8, 1.2 Hz, 1H), 7.71 (dd, *J* = 4.8, 1.2 Hz, 1H), 7.79 (dt, *J* = 8.4, 2.0 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 21.51, 127.839, 129.081, 129.379, 133.814, 134.462, 135.399, 143.028, 143.782, 187.923;

### 5g: 3-(4-nitrobenzoyl) thiophene



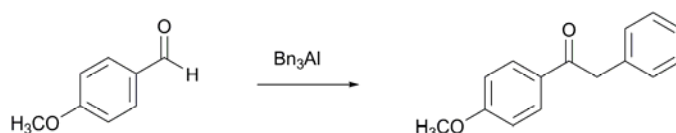
According to general procedure B: A solution of 4-nitrobenzaldehyde (0.53g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(3-thienyl)aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours, CF<sub>3</sub>CO<sub>2</sub>H (0.175 mmol, 14 μl) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.60 g, 2.59 mmol, 74%) after purification on silica gel (10:1~5:1 petroleum ether: EtOAc). IR (KBr)  $\nu$  (cm<sup>-1</sup>): 3099, 2925, 1943, 1626, 1597, 1514, 1442, 1411, 1351, 1303, 1107, 1058, 867, 837, 803, 725; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 7.21 (dd, *J* = 4.8, 4.0 Hz, 1H), 7.62 (dd, *J* = 3.6, 1.2 Hz, 1H), 7.83 (dd, *J* = 5.2, 1.2 Hz, 1H), 8.00 (dt, *J* = 8.0, 2.4 Hz, 2H), 8.37 (dt, *J* = 8.8, 2.0 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 123.69, 128.38, 129.80, 129.91, 135.45, 135.67, 142.62, 143.32, 149.818, 186.26

### 5h: 3-(4-cyanobenzoyl) thiophene



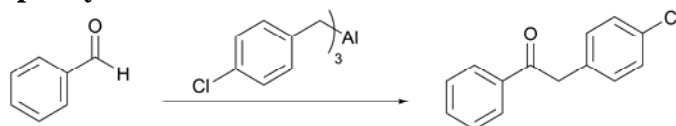
According to general procedure B: A solution of 4-cyanobenzaldehyde (0.46g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(3-thienyl)aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.42 g, 1.96 mmol, 56%) after purification on silica gel (10:1~3:1 petroleum ether: EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 2992, 2853, 1647, 1595, 1444, 1401, 1314, 1280, 1140, 1112, 1071, 928, 857, 792, 734  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 7.52 (t,  $J = 7.6$  Hz, 2H), 7.65 (tt,  $J = 7.6, 1.6$  Hz, 1H), 7.77-7.83 (m, 4H), 7.89 (dt,  $J = 8.8, 2.0$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 115.62, 117.96, 128.64, 130.05, 130.19, 132.01, 133.32, 136.33, 141.30, 194.97;

### 5i: 4-(thiophene-3-carbonyl)benzoinitrile



According to general procedure B: A solution of 4-methoxybenzaldehyde (0.48g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tribenzyl aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a colorless syrup (0.42 g, 1.86 mmol, 53%) after purification on silica gel (10:1~3:1 petroleum ether: EtOAc).  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 3.86 (s, 3H), 4.24 (s, 2H), 6.93 (d, 2H,  $J = 8.0$  Hz), 7.24-7.32 (m, 5H), 8.00 (d, 2H,  $J = 8.8$  Hz);  $^{13}\text{C}$  NMR(100Hz,  $\text{CDCl}_3$ ) : 45.26, 55.45, 113.78, 126.76, 128.62, 129.36, 129.65, 130.94, 134.97, 163.51, 196.21.

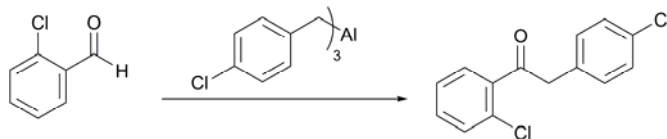
### 5j: 2-(4-chlorophenyl)-1-phenylethanone



According to general procedure B: A solution of benzaldehyde (0.37g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(4-chloro)benzyl aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.54 g, 2.34 mmol, 67%) after purification on silica gel (10:1~3:1 petroleum ether: EtOAc).  $^1\text{H}$  NMR (400MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 4.29 (s, 2H), 7.20 (d, 2H,  $J = 7.2$  Hz), 7.29-7.31 (m, 2H), 7.48 (t, 2H,  $J = 7.2$  Hz), 7.56-7.60 (m, 1H),

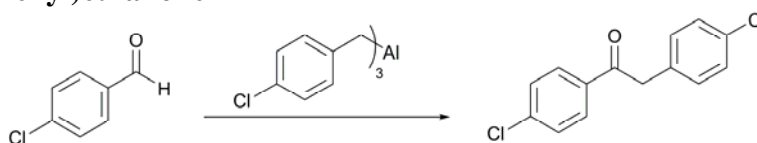
8.01 (d, 2H,  $J = 8$  Hz);  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ): 44.64, 128.48, 128.69, 128.75, 130.86, 132.84, 132.91, 133.31, 136.39, 197.03.

**5k: 1-(2-chlorophenyl)-2-(4-chlorophenyl)ethanone**



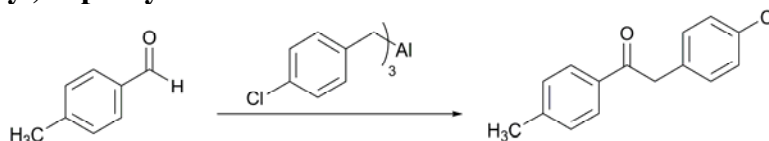
According to general procedure B: A solution of 2-chlorobenzaldehyde (0.49g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(4-chloro)benzyl aluminium (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.53 g, 2.0 mmol, 57%) after purification on silica gel (10:1~3:1 petroleum ether: EtOAc). IR (KBr)  $\nu(\text{cm}^{-1})$ : 2925, 2855, 1690, 1589, 1489, 1460, 1401, 1281, 1200, 1091, 1013, 813, 759  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 4.23 (s, 2H), 7.18 (d,  $J = 8.4$  Hz, 2H), 7.30 (d,  $J = 8.40$  Hz, 2H), 7.44 (d,  $J = 8.8$  Hz, 2H), 7.92 (d,  $J = 8.8$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 44.66, 128.46, 128.84, 129.03, 129.09, 129.90, 130.78, 132.50, 133.06, 134.60, 139.84, 195.86;

**5l: 1,2-bis(4-chlorophenyl)ethanone**



According to general procedure B: A solution of 4-chlorobenzaldehyde (0.49g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(4-chloro)benzyl aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.78 g, 2.94 mmol, 84%) after purification on silica gel (10:1~3:1 petroleum ether: EtOAc). M.P. 87~89  $^\circ\text{C}$ ; IR (KBr)  $\nu(\text{cm}^{-1})$ : 2923, 2854, 1906, 1687, 1635, 1585, 1489, 1462, 1401, 1338, 1281, 1195, 1089, 858, 812;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 4.22(s, 2H), 7.17 (d,  $J = 8.0$  Hz, 2H), 7.30 (d,  $J = 8.4$  Hz, 2H), 7.42 (d,  $J = 8.4$  Hz, 2H), 7.92 (d,  $J = 8.4$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 44.65, 128.83, 129.01, 129.88, 130.78, 132.49, 132.99, 134.58, 139.82, 196.856;

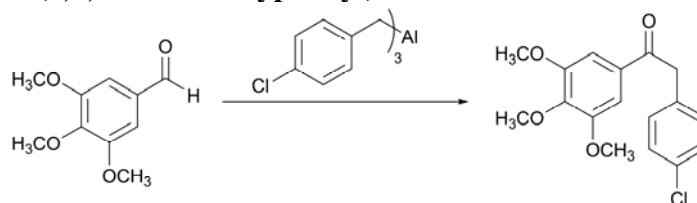
**5m: 2-(4-chlorophenyl)-1-p-tolyethanone**



According to general procedure B: A solution of 4-methylbenzaldehyde (0.42g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(4-chloro)benzyl aluminium reagent (about

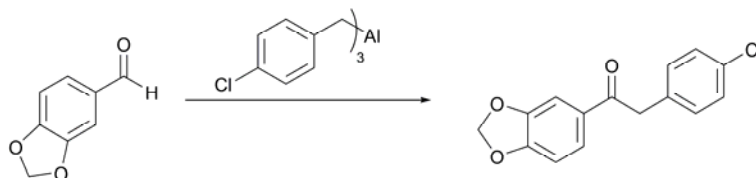
3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.64 g, 2.63 mmol, 75%) after purification on silica gel (10:1~3:1 petroleum ether: EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 2923, 2857, 1938, 1677, 1603, 1493, 1453, 1410, 1330, 1260, 1223, 1198, 1090, 1012, 866, 812, 767;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 2.40 (s, 3H), 4.22 (s, 2H), 7.18 (d,  $J = 8.4$  Hz, 2H), 7.25 (dd,  $J = 8.0, 2.0$  Hz, 2H), 7.28 (dt,  $J = 8.4, 2.0$  Hz, 2H), 7.89 (d,  $J = 8.4$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 21.63, 44.54, 128.60, 128.70, 129.36, 130.82, 132.70, 133.11, 133.82, 144.19, 196.69; ESI-MS  $m/z$  ( $\text{M}+\text{H}^+$ ) 245.2.

**5n: 2-(4-chlorophenyl)-1-(3,4,5-trimethoxyphenyl)ethanone**



According to general procedure B: A solution of 3,4,5-trimethoxybenzaldehyde (0.69g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(4-chloro)benzyl aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.36 g, 1.12 mmol, 32%) after purification on silica gel (10:1~3:1 petroleum ether: EtOAc). IR (KBr),  $\nu$  ( $\text{cm}^{-1}$ ): 2917, 2843, 2644, 1679, 1585, 1500, 1462, 1412, 1336, 1230, 1186, 1153, 1126, 1090, 1060, 1000, 885, 833, 810, 778, 750, 729;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 3.889(s, 9H), 3.957(s, 2H), 7.211(s, 1H), 7.262(s, 1H), 7.495-7.515(m, 2H), 7.912-7.933(m, 2H);  $^{13}\text{C}$  NMR(100Hz,  $\text{CDCl}_3$ ): 44.65, 56.14, 56.24, 56.35, 60.92, 106.05, 128.80, 130.68, 131.45, 132.85, 133.09, 142.73, 153.03, 196.86;

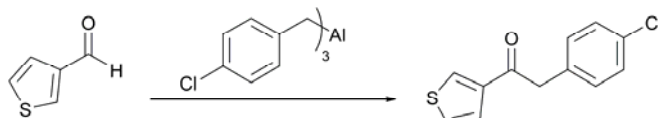
**5o: 1-(benzo[d][1,3]dioxol-5-yl)-2-(4-chlorophenyl)ethanone**



According to general procedure B: A solution of piperonal (0.53g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(4-chloro)benzyl aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.49 g, 1.78 mmol, 51%) after

purification on silica gel (10:1~3:1 petroleum ether: EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 2899, 1676, 1601, 1490, 1446, 1335, 1256, 1200, 1107, 1043, 1015, 934, 889, 808,  $774\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 4.17 (s, 2H), 6.05 (s, 2H), 6.85 (d,  $J = 8.4$  Hz, 1H), 7.18 (d,  $J = 8.4$  Hz, 2H), 7.30 (dt,  $J = 8.4$ , 2.0 Hz, 2H), 7.45 (d,  $J = 1.715\text{Hz}$ , 1H), 7.60 (dd,  $J = 8.4$ , 2.0 Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 44.49, 101.90, 107.92, 108.24, 124.90, 128.75, 130.74, 131.19, 132.79, 133.16, 148.28, 151.96, 195.14;

**5p: 2-(4-chlorophenyl)-1-(thiophen-3-yl)ethanone**



According to general procedure B: A solution of 3-thiophenecarbaldehyde (0.39g, 3.5 mmol) in pinacolone (10 mL) was added to the previously prepared tri(4-chloro)benzyl aluminium reagent (about 3.5 mmol in 10 ml of THF) under ice-water bath. After the reaction mixture was stirred at room temperature for 3 hours,  $\text{CF}_3\text{CO}_2\text{H}$  (0.175 mmol, 14  $\mu\text{l}$ ) was then added. The reaction mixture was stirred at room temperature for 2 day. The pure product was obtained as a white solid (0.38 g, 1.61 mmol, 46%) after purification on silica gel (10:1~5:1 petroleum ether: EtOAc). IR (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 3097, 2924, 2854, 1917, 1676, 1492, 1404, 1322, 1230, 1172, 1089, 1011, 947, 898;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 4.17 (s, 2H), 7.14 (dd,  $J = 4.8$ , 3.6 Hz, 1H), 7.24 (d,  $J = 8.0$  Hz, 2H), 7.29 (dt,  $J = 8.4$ , 2.0 Hz, 2H), 7.66 (dd,  $J = 4.8$ , 1.2 Hz, 1H), 7.77 (dd,  $J = 3.6$ , 1.2 Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 45.99, 126.57, 127.17, 128.78, 130.78, 132.65, 132.74, 132.90, 141.59, 191.25.