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Supporting Information

Transition-metal-free and base promoted C–C bond formation via C–N bond cleavage of organoammonium salts

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1. General Information

The reactions were carried out in Schlenk tubes of 25 mL under N₂ atmosphere. For reactions that require heating, heating mantle was used as the heat source. Organoammonium salts **1** were prepared according to the reported literatures.¹ All solvents were purified according to standard operation procedures. All solvents and reagents were purchased from Tansoole, Meryer, Heowns, Energy Chemical, Alfa Aesar, and Aladdin. Column chromatography was performed using Silica Gel 60 (300-400 mesh). The reactions were monitored by GC and GC-MS, GC-MS results were recorded on GC-MS QP2010, and GC analysis was performed on GC 2014. The ¹H, ¹³C NMR spectra were recorded on a Brucker ADVANCE III spectrometer at 400 MHz, 100 MHz respectively, and chemical shifts were reported in parts per million (ppm). The electron ionization (EI) method was used as the ionization method for the HRMS measurement, and the mass analyzer type is TOF for EI.

2. Experimental Procedure

2.1 General Experimental Procedure for the Synthesis of C-C Bond formation via C-N bond cleavage of organoammonium salts.

In an oven dried 25 mL Schlenk tube charged with **1** (0.2 mmol), **2** (0.2 mmol, 1.0 equiv), and KO'Bu (0.22 mmol, 1.1 equiv), after charging N_2 for three times, toluene (2 mL) were added. The reaction mixture was reacted at 100 °C for 2 h. The experiment was conducted in two sets, and the reaction mixtures of two sets were combined and concentrated after completion of the reaction. The desired product was isolated by column chromatography over silica gel (300-400 mesh) using petroleum ether/ethyl acetate (PE/EA) as eluent.

2.2 Procedure for the Synthesis of α , β -unsaturated ketones.



In an oven-dried 25 mL Schlenk tube was charged with 1,3-diphenylpropan-1-one **3a** (0.2 mmol), KI (10 mol%), I₂ (2.5 mol%), and DMSO (3 mL) under N₂. The reaction mixture was reacted at 140 °C for 16 h. After completion of the reaction, the reaction mixture was washed with NaCl saturated solution, dried over anhydrous Na₂SO₄ and concentrated under vacuum. The desired product was isolated by column chromatography over silica gel (300-400 mesh) using petroleum ether/ethyl acetate = 50:1 as eluent to afford a pale-yellow solid **4a** in 90% yield (51.1 mg).²

3. Characterization Data for the Products

1,2,3-Triphenylpropan-1-one (3a)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford a white solid in 86% yield (98.5 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.90–7.88 (m, 2H), 7.44 (t, *J* = 7.6 Hz, 1H), 7.34 (t, *J* = 8.0 Hz, 2H), 7.27–7.17 (m, 7H), 7.15–7.07 (m, 3H) 4.81 (t, *J* = 7.2 Hz, 1H), 3.56 (dd, *J* = 12.0, 8.8 Hz, 1H), 3.06 (dd, *J* = 14.0, 8.4 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 199.2, 139.8, 139.2, 136.7, 132.8, 129.1, 128.9, 128.7, 128.4, 128.3, 128.2, 127.1, 126.1, 55.9, 39.7. This compound is known.³

1,2-Diphenyl-3-(o-tolyl)propan-1-one (3b)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford a pale-yellow solid in 84% yield (101.3 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.89–7.87 (m, 2H), 7.45 (t, J = 7.2 Hz, 1H), 7.34 (t, J = 7.6 Hz, 2H), 7.25–7.17 (m, 5H), 7.09–6.96 (m, 4H), 4.80 (t, J = 7.6 Hz, 1H), 3.55 (dd, J = 14.0, 7.6 Hz, 1H), 3.07 (dd, J = 14.4, 7.6 Hz, 1H), 2.22 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 199.3, 139.2, 137.9, 136.7, 136.3, 132.8, 130.2, 129.7, 128.9, 128.7, 128.5, 128.2, 127.1, 126.2, 125.7, 54.5, 37.1, 19.5. This compound is known.⁴

1,2-Diphenyl-3-(p-tolyl)propan-1-one (3c)



The title compound was prepared according to the Experimental Procedure, and purified by

column chromatography on silica gel with PE/EA = 50:1(v/v) to afford white solid in 82% yield (98.7 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.90–7.88 (m, 2H), 7.43 (t, *J* = 7.6 Hz, 1H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.26–7.17 (m, 5H), 7.01–6.96 (m, 4H), 4.79 (t, *J* = 7.2 Hz, 1H), 3.53 (dd, *J* = 13.2, 7.6 Hz, 1H), 3.02 (dd, *J* = 13.6, 6.8 Hz, 1H), 2.26 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 199.2, 139.2, 136.2, 136.7, 135.5, 132.8, 129.0, 128.9, 128.9, 128.7, 128.4, 128.3, 127.1, 56.0, 39.7, 21.0. This compound is known.⁵

3-(4-Methoxyphenyl)-1,2-diphenylpropan-1-one (3d)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 150:1(v/v) to afford white solid in 80% yield (101.8 mg).¹H NMR (400 MHz, CDCl₃) δ 7.90–7.87 (m, 2H), 7.44 (t, J = 7.2 Hz, 1H), 7.33 (t, J = 8.0 Hz, 2H), 7.25–7.16 (m, 5H), 6.99 (d, J = 8.4 Hz, 2H), 6.73 (d, J = 8.8 Hz, 2H), 4.77 (t, J = 7.2 Hz, 1H), 3.73 (s, 3H), 3.51 (dd, J = 14.0, 7.6 Hz, 1H), 3.01 (dd, J = 13.6, 6.8 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 199.4, 157.7, 139.1, 136.8, 132.8, 131.8, 130.0, 128.8, 128.6, 128.4, 128.3, 127.1, 113.6, 56.1, 55.1, 39.3. This compound is known.⁶

3-(4-(Methylthio)phenyl)-1,2-diphenylpropan-1-one (3e)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 200:1(v/v) to afford white solid in 90% yield (119.7 mg). mp 122-123 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.90–7.88 (m, 2H), 7.44 (t, *J* = 7.2, Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.28–7.24 (m, 4H), 7.22–7.18 (m, 1H), 7.09 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 8.4 Hz, 2H), 4.77 (t, *J* = 7.2 Hz, 1H), 3.51 (dd, *J* = 13.6, 7.6 Hz, 1H), 3.02 (dd, *J* = 14.0, 7.6 Hz, 1H), 2.41 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 199.1, 138.9, 136.8, 136.6, 135.7, 132.9, 129.6, 128.9, 128.6, 128.5, 128.3, 127.2, 126.8, 55.9, 39.6, 16.0. HRMS (EI) m/z: [M]⁺ calcd. for C₂₂H₂₀OS: 332.1235; found: 332.1235.

3-(4-(Tert-butyl)phenyl)-1,2-diphenylpropan-1-one (3f)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 150:1(v/v) to afford a pale-yellow solid in 79% yield (108.2 mg). mp 116-118 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.91–7.88 (m, 2H), 7.44 (t, J = 6.8 Hz, 1H), 7.34 (t, J = 8.0 Hz, 2H), 7.27–7.25 (m, 4H), 7.22–7.18 (m, 3H), 7.03 (d, J = 8.4 Hz, 2H), 4.83 (dd, J= 8.0, 6.4 Hz, 1H), 3.57 (dd, J = 13.6, 8.0 Hz, 1H), 3.01 (dd, J = 13.6, 6.4 Hz, 1H), 1.26 (s, 9H). ¹³C NMR (100 MHz, CDCl₃): δ 199.2, 148.9, 139.3, 136.8, 136.7, 136.6, 132.6, 128.9, 128.7, 128.4, 128.3, 127.1, 125.1, 55.8, 39.6, 34.3, 31.3. HRMS (EI) m/z: [M]⁺ calcd. for C₂₅H₂₆O: 342.1984; found: 342.1983.

3-([1,1'-Biphenyl]-4-yl)-1,2-diphenylpropan-1-one (3g)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford white solid in 86% yield (124.1 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.93–7.90 (m, 2H), 7.54–7.52 (m, 2H), 7.45–7.37 (m, 5H), 7.36–7.31 (m, 2H), 7.30–7.26 (m, 5H), 7.23–7.14 (m, 3H), 4.85 (t, *J* = 7.2 Hz, 1H), 3.61 (dd, *J* = 13.6, 7.2 Hz, 1H), 3.10 (dd, *J* = 13.6, 6.8 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 199.2, 140.9, 139.1, 138.9, 138.9, 132.9, 129.6, 128.9, 128.7, 128.7, 128.5, 128.3, 127.2, 127.0, 126.9, 55.9, 39.8. This compound is known.⁷

3-(Benzo[d][1,3]dioxol-5-yl)-1,2-diphenylpropan-1-one (3h)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 150:1(v/v) to afford white solid in 90% yield

(118.8 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.91–7.88 (m, 2H), 7.45–7.41 (m, 1H), 7.34 (t, J = 7.6 Hz, 2H), 7.28–7.16 (m, 5H), 6.64–6.52 (m, 3H), 5.85 (s, 2H), 4.76 (t, J = 7.2 Hz, 1H), 3.48 (dd, J = 13.6, 7.6 Hz, 1H), 2.97 (dd, J = 14.0, 7.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 199.2, 147.4, 145.8, 139.0, 136.7, 133.5, 132.5, 128.9, 128.7, 128.6, 128.2.127.2, 122.1, 109.5, 108.0, 100.7, 56.1, 39.8. This compound is known.⁸

3-(Naphthalen-1-yl)-1,2-diphenylpropan-1-one (3i)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 120:1(v/v) to afford white solid in 81% yield (108.8 mg). ¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, *J* = 8.0 Hz, 1H), 7.84–7.82 (m, 3H), 7.66 (d, *J* = 8.4 Hz, 1H), 7.54–7.45 (m, 2H), 7.42–7.39 (m, 1H), 7.29 (t, *J* = 8.0 Hz, 2H), 7.26–7.19 (m, 6H), 7.11 (d, *J* = 6.8 Hz, 1H), 5.00 (t, *J* = 6.8 Hz, 1H), 4.09 (dd, *J* = 14.4, 7.2 Hz, 1H), 3.48 (dd, *J* = 14.0, 6.8 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 199.2, 139.4, 136.7, 135.5, 133.9, 132.8, 131.8, 129.0, 128.9, 128.7, 128.4, 128.1, 127.6, 127.2, 127.0, 126.0, 125.4, 123.4, 54.6, 36.9. This compound is known.⁸

3-(4-Fluorophenyl)-1,2-diphenylpropan-1-one (3j)



3-(4-Chlorophenyl)-1,2-diphenylpropan-1-one (3k)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford white solid in 90% yield (115.4 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.90–7.89 (m, 2H), 7.45 (t, J = 7.2 Hz, 1H), 7.34(t, J = 8.0 Hz, 2H), 7.28–7.24 (m, 2H), 7.2–7.14 (m, 5H), 7.00 (d, J = 8.4 Hz, 2H), 4.75(t, J = 7.2 Hz, 1H), 3.51 (dd, J = 14.0, 7.6 Hz, 1H), 3.03 (dd, J = 13.6, 7.6 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 198.9, 138.7, 138.2, 136.5, 133.0, 131.9, 130.5, 129.0, 128.7, 128.5, 128.3, 128.2, 127.3, 55.8, 39.4. This compound is known.⁶

3-(2-Chlorophenyl)-1,2-diphenylpropan-1-one (31)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford white solid in 94% yield (121 mg). mp 69-70 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.90–7.88 (m, 2H), 7.43 (t, J = 7.6 Hz, 1H), 7.33 (t, J = 8.0 Hz, 3H), 7.25–7.16 (m, 5H), 7.10–7.00 (m, 3H), 5.01 (t, J = 7.6 Hz, 1H), 3.65 (dd, J = 13.6, 7.6 Hz, 1H), 3.18 (dd, J = 13.6, 6.8 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 199.0, 139.0, 137.2, 136.6, 134.1, 132.9, 132.1, 129.3, 128.9, 128.7, 128.5, 128.2, 127.7, 127.2, 126.5, 53.1, 38.0. HRMS (EI) m/z: [M]⁺ calcd. for C₂₁H₁₇ClO: 320.0968; found: 320.0968.

3-(2-Bromophenyl)-1,2-diphenylpropan-1-one (3m)



The title compound was prepared according to the Experimental Procedure, and purified by

column chromatography on silica gel with PE/EA = 100:1(v/v) to afford a pale-yellow oil in 83% yield (121.8 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.91–7.89 (m, 2H), 7.51 (d, J = 8.0 Hz, 1H), 7.44 (t, J = 7.6 Hz, 1H), 7.33 (t, J = 7.6 Hz, 2H), 7.26–7.17 (m, 5H), 7.08–6.98 (m, 3H), 5.04 (d, J = 7.6 Hz, 1H), 3.66 (dd, J = 13.6, 7.6 Hz, 1H), 3.17 (dd, J = 13.6, 6.8 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 198.9, 138.9, 138.8, 136.6, 132.9, 132.7, 132.2, 128.9, 128.7, 128.5, 128.2, 128.0, 127.2, 127.1, 124.6, 53.2, 40.1. This compound is known.⁶

3-(3-Bromophenyl)-1,2-diphenylpropan-1-one (3n)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford ligh yellow in 80% yield (116.3 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.91–7.88 (m, 2H), 7.47–7.43 (m, 1H), 7.35 (t, *J* = 8.0 Hz, 2H), 7.28–7.25 (m, 4H), 7.22–7.20 (m, 3H), 7.06–6.96 (m, 2H), 4.76 (t, *J* = 7.6 Hz, 1H), 3.52 (dd, *J* = 14.0, 7.6 Hz, 1H), 3.03 (dd, *J* = 13.6, 7.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 198.7, 142.1, 138.7, 136.5, 133.0, 132.1, 129.7, 129.3, 129.0, 128.7, 128.5, 128.2, 127.8, 127.3, 122.2, 55.8, 39.7. HRMS (EI) m/z: [M]⁺ calcd. for C₂₁H₁₇BrO: 364.0463; found: 364.0464.

3-(4-Bromophenyl)-1,2-diphenylpropan-1-one (30)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford a white solid in 85% yield (123.2 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.90–7.87 (m, 2H), 7.45 (d, J = 7.6 Hz, 1H), 7.35 (t, J = 8.0 Hz, 2H), 7.30 (d, J = 8.4 Hz, 2H), 7.26–7.24 (m, 2H), 7.22–7.19 (m, 3H), 6.95 (d, J = 6.8 Hz, 2H), 4.74 (t, J = 6.8 Hz, 1H), 3.49 (dd, J = 13.6, 7.6 Hz, 1H), 3.02 (dd, J = 14.0, 7.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 198.9, 138.7, 138.7, 136.5, 132.9, 131.3, 130.9, 129.0, 128.6, 128.5, 128.2, 127.3, 120.0, 55.7, 39.4. This compound is known.¹¹

3-(4-Iodophenyl)-1,2-diphenylpropan-1-one (3p)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 75:1(v/v) to afford white solid in 85% yield (140.6 mg). mp 120-122 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.89–7.87 (m, 2H), 7.51–7.44 (m, 3H), 7.37–7.27 (m, 3H), 7.24–7.18 (m, 4H), 6.82 (d, J = 8 Hz, 2H), 4.74 (t, J = 7.2 Hz, 1H), 3.48 (dd, J = 13.6, 7.6 Hz, 1H), 3.01 (dd, J = 14.0, 7.6 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 198.8, 139.4, 138.7, 137.2, 136.5, 133.0, 131.2, 129.0, 128.7, 128.5, 128.2, 127.3, 91.4, 55.8, 39.6. HRMS (EI) m/z: [M]⁺ calcd. for C₂₁H₁₇IO: 412.0324; found: 412.0326.

1,2-Diphenyl-3-(4-(trifluoromethyl)phenyl)propan-1-one (3q)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford white solid in 81% yield (115.7 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.90– 7.88 (m, 2H), 7.47–7.43 (m, 3H), 7.35 (t, *J* = 8.0 Hz, 2H), 7.29–7.25 (m, 2H), 7.22–7.17 (m, 5H), 4.79 (t, *J* = 7.2 Hz, 1H), 3.61 (dd, *J* = 13.6, 7.6 Hz, 1H), 3.12 (dd, *J* = 13.6, 7.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 198.6, 143.9, 138.6, 136.4, 133.0, 129.5, 129.1, 128.7, 128.5, 128.2, 127.4, 125.1 (q, *J* = 3.6 Hz), 124.2 (q, *J* = 270.2 Hz), 55.6, 39.7. ¹⁹F NMR (376 MHz, CDCl₃): δ -62.4. This compound is known.⁶

4-(3-Oxo-2,3-diphenylpropyl)benzonitrile (3r)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 125:1(v/v) to afford white solid in 82% yield (102.3 mg). mp 150-152 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.89–7.87 (m, 2H), 7.47 (t, *J* = 8.0 Hz, 3H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.29–7.25 (m, 2H), 7.22–7.16 (m, 5H), 4.76 (t, *J* = 7.2 Hz, 1H), 3.57 (dd, *J* = 13.6, 7.2 Hz, 1H), 3.13 (dd, *J* = 14.0, 7.6 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 198.4,

145.4, 138.3, 136.3, 133.1, 132.0, 130.0, 129.1, 128.7, 128.6, 128.2, 127.5, 118.9, 110.1, 55.5, 40.1. HRMS (EI) m/z: [M]⁺ calcd. for C₂₂H₁₇NO: 311.1310; found: 311.1316.

3-(5-Chlorobenzo[b]thiophen-3-yl)-1,2-diphenylpropan-1-one (3s)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford a pale-yellow solid in 81% yield (122.3 mg). mp 120-122 °C ¹H NMR (400 MHz, CDCl₃) δ 7.91–7.89 (m, 2H), 7.71–7.66 (m, 2H), 7.44 (t, J = 7.6 Hz, 1H), 7.36–7.27 (m, 4H), 4.25–4.21 (m, 4H), 6.96 (s, 1H), 4.90 (t, J = 7.6 Hz, 1H), 3.78 (dd, J = 14.0, 8.4 Hz, 1H), 3.23 (dd, J = 14.8, 6.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 198.1, 140.1, 139.0, 136.4, 133.4, 133.0, 130.4, 129.1, 128.7, 128.5, 128.1, 127.4, 125.2, 124.5, 123.8, 121.2, 53.6, 32.5. HRMS (EI) m/z: [M]⁺ calcd. for C₂₃H₁₇ClOS: 376.0689; found: 376.0688.

(E)-1,2,5-Triphenylpent-4-en-1-one (3t)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford a pale-yellow solid in 80% yield (95.5 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.9–7.94 (m, 2H), 7.47–7.43 (m, 1H), 7.38–7.27 (m, 7H), 7.25–7.16 (m, 6H), 6.41 (d, *J* = 16 Hz, 1H), 6.17–6.10 (m, 1H), 4.67 (t, *J* = 7.6 Hz, 1H), 3.14–3.06 (m, 1H), 2.75–2.68 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 199.1, 139.0, 137.4, 136.6, 132.87, 132.0, 129.0, 128.7, 128.5, 128.4, 128.2, 127.7, 127.2, 127.0, 126.0, 54.1, 37.5. This compound is known.¹²

Methyl 2,3-diphenylpropanoate (3u)

The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford a yellow oil in 83% yield (95.1 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.31–7.29 (m, 4H), 7.27–7.21 (m, 3H), 7.17 (d, *J* = 7.2 Hz, 1H), 7.12–7.10 (m, 2H), 3.87–3.83 (m, 1H), 3.59 (s, 3H), 3.42 (dd, *J* = 13.6, 8.8 Hz, 1H), 3.03 (dd, *J* = 13.6, 6.4 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 173.8, 139.0, 138.6, 128.9, 128.6, 128.3, 127.9, 127.4, 126.4, 53.6, 52.0, 39.8. This compound is known.¹³

N,N-dimethyl-2,3-diphenylpropanamide (3v)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford white solid in 65% yield (65.4 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.27–7.15 (m, 8H), 7.09–7.06 (m, 2H), 3.97 (t, *J* = 7.2 Hz, 1H), 3.48 (dd, *J* = 13.6, 8.0 Hz, 1H), 2.95 (dd, *J* = 13.6, 6.8 Hz, 1H), 2.90 (s, 3H), 2.81 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 172.5, 140.1, 139.5, 129.1, 128.6, 128.1, 128.0, 126.9, 126.0, 51.2, 41.2, 37.1, 35.9. This compound is known.¹⁴

2,3,4-Triphenylbutanenitrile (3w)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford a yellow solid in 91% yield (102.9 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.33–7.27 (m, 10H), 7.21–7.12 (m, 3H), 6.90–6.85 (m, 2H), 3.66 (s, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 140.0, 134.6, 130.5, 128.7, 127.9, 127.9, 127.4, 127.3, 121.8, 52.9, 45.3. This compound is known. ¹⁵

Diphenyl(2-phenyl-1-(p-tolyl)ethyl)phosphine oxide (3x)



The title compound was prepared according to the Experimental Procedure, and purified by

column chromatography on silica gel with PE/EA = 10:1(v/v) to afford white solid in 76% yield (120.5 mg). mp 224-226 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.00–7.95 (m, 2H), 7.57– 7.55 (m, 3H), 7.48–7.43 (m, 2H), 7.33–7.31 (m, 1H), 7.25–7.21 (m, 2H), 7.08–7.04 (m, 5H) , 6.92 (d, *J* = 8.0 Hz, 2H), 6.82 (dd, *J* = 7.6, 2.0 Hz, 2H), 3.66–3.60 (m, 1H), 3.30–3.25 (m, 2H), 2.22 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 139.6 (d, *J* = 14.0 Hz), 136.5 (d, *J* = 2.2 Hz), 131.8 (d, *J* = 2.6 Hz), 131.4 (d, *J* = 8.2 Hz), 131.2 (d, *J* = 2.6 Hz), 131.0 (d, *J* = 8.7 Hz), 129.9 (d, *J* = 5.9 Hz), 128.9 (d, *J* = 1.3 Hz), 128.8 (d, *J* = 11.2 Hz), 128.7 (d, *J* = 58.0 Hz), 128.5 (d, *J* = 11.5 Hz), 126.1, 49.0 (d, *J* = 65.9 Hz), 35.9, 21.0 . HRMS (EI) m/z: [M]⁺ calcd. for C₂₇H₂₅OP: 396.1643; found: 396.1643.

Methyl 3-phenyl-2-(p-tolyl)propanoate (3y)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford a colorless oil in 76% yield (77 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.24–7.20 (m, 3H), 7.17 (d, *J* = 6.0 Hz, 2H), 7.13–7.10 (m, 4H), 3.82 (dd, *J* = 8.8, 6.8 Hz, 1H), 3.58 (s, 3H), 3.40 (dd, *J* = 13.6, 8.8 Hz, 1H), 3.01 (dd, *J* = 13.6, 6.4 Hz, 1H), 2.32 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 174.0, 139.2, 137.0, 135.6, 129.3, 128.9, 128.3, 127.8, 126.3, 53.2, 52.0, 39.8, 21.0. This compound is known.¹⁶

Methyl 2-(4-(tert-butyl)phenyl)-3-phenylpropanoate (3z)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford a colorless oil in 92% yield (109.2 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.34–7.32 (m, 2H), 7.26–7.23 (m, 4H), 7.20–7.14 (m, 3H), 3.85 (dd, J = 9.6, 6.0 Hz, 1H), 3.58 (s, 3H), 3.41 (dd, J = 13.6, 9.2 Hz, 1H), 3.00 (dd, J = 13.6, 6.0 Hz, 1H), 1.31 (s, 9H). ¹³C NMR (100 MHz, CDCl₃): δ 174.0, 150.3, 139.3, 135.7, 128.9, 128.3, 127.4, 126.3, 125.6, 53.1, 51.9, 40.0, 34.5, 31.3. This compound is known.¹⁷

Methyl 2-(4-fluorophenyl)-3-phenylpropanoate (3za)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford a colorless oil in 70% yield (72 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.27–7.25 (m, 2H), 7.23–7.17 (m, 3H), 7.08 (d, *J* = 6.4 Hz, 2H), 6.98 (t, *J* = 8.8 Hz, 2H), 3.83 (t, *J* = 8.0 Hz, 1H), 3.61 (s, 3H), 3.39 (dd, *J* = 13.6, 7.2 Hz, 1H), 3.00 (dd, *J* = 13.6, 7.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 173.7, 163.3 (d, *J* = 244.3 Hz) 138.7, 134.3 (d, *J* = 3.1 Hz), 129.6 (d, *J* = 8.0 Hz), 128.9, 128.4, 126.5, 115.6 (d, *J* = 21.3 Hz), 52.7 (d, *J* = 74.5 Hz), 39.8, 29.7. ¹⁹F NMR (376 MHz, CDCl₃) δ : -115.2. HRMS (EI) m/z: [M]⁺ calcd. for C₁₆H₁₅FO₂: 258.1056; found: 258.1059.

Methyl 2-(4-chlorophenyl)-3-phenylpropanoate (3zb)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford a colorless oil in 83% yield (92.1 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.29–7.25 (m, 1H), 7.24–7.17 (m, 6H), 7.09–7.07 (m, 2H), 3.82 (t, *J* = 8.0 Hz, 1H), 3.61 (s, 3H), 3.39 (dd, *J* = 13.6, 8.0 Hz, 1H), 3.00 (dd, *J* = 14.0, 7.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 173.5, 138.5, 136.9, 133.3, 129.4, 128.9, 128.8, 128.4, 126.5, 53.0, 52.1, 39.7. This compound is known.¹⁶

Methyl 2-(4-bromophenyl)-3-phenylpropanoate (3zc)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford a colorless oil in 70% yield (88.9 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.42 (d, J = 8.4 Hz, 2H), 7.25–7.21 (m, 2H), 7.19–7.15 (m, 3H), 7.09–7.07 (m, 2H), 3.80 (t, J = 7.6 Hz, 1H), 3.60 (s, 3H), 3.38 (dd, J = 13.6, 8.4 Hz,

1H), 2.99 (dd, J = 13.6, 7.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 173.4, 138.5, 137.5, 131.7,
129.7, 128.9, 128.4, 126.5, 121.4, 53.0, 52.1, 39.6. This compound is known.¹⁸

Methyl 2-(2-iodophenyl)-3-phenylpropanoate (3zd)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford pale-yellow oil in 78% yield (114.5 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, J = 8.8 Hz, 1H), 7.44–7.41 (m, 1H), 7.32 (t, J = 7.2 Hz, 1H), 7.27–7.22 (m, 4H), 7.20–7.18 (m, 1H),6.96–6.92 (m, 1H), 4.36 (dd, J = 9.2, 5.6 Hz, 1H), 3.60 (s, 3H), 3.29 (dd, J = 13.6, 9.2 Hz, 1H), 2.99 (dd, J = 12.8, 6.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 173.2, 141.5, 139.8, 138.5, 129.1, 129.0, 128.7, 128.3, 127.9, 126.9, 101.4, 56.9, 52.1, 39.6. This compound is known.¹⁹

Methyl 2-(4-cyanophenyl)-3-phenylpropanoate (3ze)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford a colorless solid in 54% yield (57.1 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.58 (d, J = 8.4 Hz, 2H), 7.38 (d, J = 8.0 Hz, 2H), 7.26–7.18 (m, 3H), 7.05 (d, J = 6.8 Hz, 2H), 3.90 (t, J = 8.0 Hz, 1H), 3.63 (s, 3H), 3.42 (dd, J = 13.6, 8.0 Hz, 1H), 3.02 (dd, J = 13.6, 7.6 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 172.7, 143.6, 137.9, 132.4, 128.9, 128.8, 128.5, 126.7, 118.6, 111.4, 53.6, 52.3, 39.6. This compound is known.²⁰

Methyl 4-(1-methoxy-1-oxo-3-phenylpropan-2-yl)benzoate (3zf)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 25:1(v/v) to afford a pale-yellow oil in 72% yield (86.2 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, J = 8.4 Hz, 2H), 7.35 (d, J = 8.4 Hz, 2H), 7.23–7.17 (m, 3H), 7.08 (d, J = 6.8 Hz, 2H), 3.90 (s, 3H), 3.69 –3.66 (m, 1H), 3.62 (s, 3H), 3.42 (dd, J = 13.6, 8.0 Hz, 1H), 3.03 (dd, J = 14.0, 7.6 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 173.2, 166.8, 143.6, 138.4, 129.9, 129.3, 128.9, 128.4, 128.1, 126.5, 53.6, 52.2, 52.1, 39.6. This compound is known.²¹

Methyl 3-phenyl-2-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)propanoate (3zg)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford a colorless oil in 60% yield (87.9 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 7.25–7.20 (m, 2H), 7.18–7.09 (m, 3H), 3.88–3.88 (m, 1H), 3.56 (s, 3H), 3.42 (dd, J = 13.6, 8.4 Hz, 1H), 3.03 (dd, J = 13.6, 6.8 Hz, 1H), 1.33 (s, 12H). ¹³C NMR (100 MHz, CDCl₃): δ 173.6, 141.7, 138.9, 135.1, 128.9, 128.3, 128.2, 127.4, 126.4, 83.8, 53.7, 52.0, 39.6, 24.8. This compound is known.²⁰

Methyl 2-(naphthalen-2-yl)-3-phenylpropanoate (3zh)



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 100:1(v/v) to afford white solid in 52% yield (60.9 mg). mp 51-53°C. ¹H NMR (400 MHz, CDCl₃) δ 7.82–7.73 (m, 4H), 7.48–7.43 (m, 3H), 7.24–7.20 (m, 2H), 7.18–7.13 (m, 3H), 4.02 (dd, J = 8.4, 7.2 Hz, 1H), 3.61 (s, 3H), 3.52 (dd, J = 13.6, 8.4 Hz, 1H), 3.14 (dd, J = 13.6, 6.8 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 173.8, 139.0, 136.0, 133.4, 132.7, 130.5, 128.9, 128.4, 127.9, 127.8, 127.6, 126.9, 126.4, 126.2, 125.9, 53.7,

52.0, 39.7. HRMS (EI) m/z: [M]⁺ calcd. for C₂₀H₁₈O₂: 290.1307; found: 290.1306.

Methyl 2-(6-methoxynaphthalen-2-yl)-2-methyl-3-phenylpropanoate (3zi) MeO



The title compound was prepared according to the Experimental Procedure, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford white solid in 80% yield (107.1 mg). mp 118.6-120°C. ¹H NMR (400 MHz, CDCl₃) δ 7.69 (dd, J = 14.8, 8.4 Hz, 2H), 7.60 (d, J = 2.0 Hz, 1H), 7.42 (dd, J = 8.4, 2.0 Hz, 1H), 7.15–7.12 (m, 5H), 6.91–6.89 (m, 2H), 3.91 (s, 3H), 3.67 (s, 3H), 3.52 (d, J = 13.6 Hz, 1H), 3.29 (d, J = 14.2 Hz, 1H), 1.55 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 176.5, 157.8, 138.3, 137.4, 133.4, 130.5, 129.6, 128.6, 127.8, 126.8, 126.4, 125.4, 124.6, 118.9, 105.4, 55.3, 52.2, 51.2, 45.2, 22.1. HRMS (EI) m/z: [M]⁺ calcd. for C₂₂H₂₂O₃: 334.1569; found: 334.1571.

(E)-1,2,3-Triphenylprop-2-en-1-one (4a)



The title compound was prepared according to the Procedure for the Synthesis of α , β unsaturated ketones, and purified by column chromatography on silica gel with PE/EA = 50:1(v/v) to afford a pale-yellow solid in 90% yield (51.1 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, J = 7.2 Hz, 2H), 7.48 (t, J = 8.0 Hz, 3H), 7.37–7.33 (m, 4H), 7.31–7.28 (m, 3H), 7.20–7.14 (m, 4H). ¹³C NMR (100 MHz, CDCl₃): δ 199.4, 140.8, 138.0, 136.3, 135.4, 133.6, 130.1, 129.7, 128.9, 128.8, 128.7, 128.4, 128.2, 128.0, 126.4. This compound is known.²²

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5. Copies of ¹H, ¹³C NMR Spectra of the Products







¹H NMR Spectrum of 1,2-diphenyl-3-(o-tolyl)propan-1-one (**3b**)



¹³C NMR Spectrum of 1,2-diphenyl-3-(p-tolyl)propan-1-one (3c)





¹H NMR Spectrum of 3-(4-methoxyphenyl)-1,2-diphenylpropan-1-one (3d)

¹³C NMR Spectrum of 3-(4-methoxyphenyl)-1,2-diphenylpropan-1-one (3d)





¹H NMR Spectrum of 3-(4-(methylthio)phenyl)-1,2-diphenylpropan-1-one (3e)



¹³C NMR Spectrum of 3-(4-(tert-butyl)phenyl)-1,2-diphenylpropan-1-one (3f)



¹H NMR Spectrum of 3-(4-(tert-butyl)phenyl)-1,2-diphenylpropan-1-one (3f)



¹H NMR Spectrum of 3-([1,1'-biphenyl]-4-yl)-1,2-diphenylpropan-1-one (**3g**)

¹³C NMR Spectrum of 3-([1,1'-biphenyl]-4-yl)-1,2-diphenylpropan-1-one (**3g**)





¹H NMR Spectrum of 3-(benzo[d][1,3]dioxol-5-yl)-1,2-diphenylpropan-1-one (**3h**)

¹³C NMR Spectrum of 3-(benzo[d][1,3]dioxol-5-yl)-1,2-diphenylpropan-1-one (3h)







¹H NMR Spectrum of 3-(naphthalen-1-yl)-1,2-diphenylpropan-1-one (3i)

100 90 fl (ppm) 70

60

80

50 40

30 20 10

-10

0

110

150

130 120

140

200

190 180 170 160



¹H NMR Spectrum of **3-(4-fluorophenyl)-1,2-diphenylpropan-1-one (3j)**

¹³C NMR Spectrum of **3-(4-fluorophenyl)-1,2-diphenylpropan-1-one (3j)**







¹H NMR Spectrum of 3-(4-chlorophenyl)-1,2-diphenylpropan-1-one (3k)





100 90 fl (ppm) 70

80

50

60

40

30 20

10 0

110

-10

¹H NMR Spectrum of 3-(2-chlorophenyl)-1,2-diphenylpropan-1-one (3)

130 120

150 140

200

190

170 160

180





¹³C NMR Spectrum of 3-(2-chlorophenyl)-1,2-diphenylpropan-1-one (3l)



S33



S34



S35





¹H NMR Spectrum of 1,2-diphenyl-3-(4-(trifluoromethyl)phenyl)propan-1-one (3q)





¹³C NMR Spectrum of 1,2-diphenyl-3-(4-(trifluoromethyl)phenyl)propan-1-one (3q)

¹⁹F NMR Spectrum of 1,2-diphenyl-3-(4-(trifluoromethyl)phenyl)propan-1-one (3q)





¹H NMR Spectrum of 4-(3-oxo-2,3-diphenylpropyl)benzonitrile (3r)

¹³C NMR Spectrum of 4-(3-oxo-2,3-diphenylpropyl)benzonitrile (3r)





¹H NMR Spectrum of 3-(5-chlorobenzo[b]thiophen-3-yl)-1,2-diphenylpropan-1-one (3s)

¹³C NMR Spectrum of 3-(5-chlorobenzo[b]thiophen-3-yl)-1,2-diphenylpropan-1-one (3s)





¹H NMR Spectrum of (E)-1,2,5-triphenylpent-4-en-1-one (**3t**)



¹H NMR Spectrum of methyl 2,3-diphenylpropanoate (3u)





¹H NMR Spectrum of N,N-dimethyl-2,3-diphenylpropanamide (3v)

¹H NMR Spectrum of 2,3,4-triphenylbutanenitrile (**3w**)



¹³C NMR Spectrum of 2,3,4-triphenylbutanenitrilen (3w)





¹H NMR Spectrum of diphenyl(2-phenyl-1-(p-tolyl)ethyl)phosphine oxide (3x)

¹³C NMR Spectrum of diphenyl(2-phenyl-1-(p-tolyl)ethyl)phosphine oxide (3x)







¹³C NMR Spectrum of methyl 3-phenyl-2-(p-tolyl)propanoate (3y)





¹H NMR Spectrum of methyl 2-(4-(tert-butyl)phenyl)-3-phenylpropanoate (3z)

¹³C NMR Spectrum of methyl 2-(4-(tert-butyl)phenyl)-3-phenylpropanoate (3z)





¹H NMR Spectrum of methyl 2-(4-fluorophenyl)-3-phenylpropanoate (3za)

¹³C NMR Spectrum of methyl 2-(4-fluorophenyl)-3-phenylpropanoate (3za)



¹⁹F NMR Spectrum of methyl 2-(4-fluorophenyl)-3-phenylpropanoate (3za)









¹H NMR Spectrum of methyl 2-(4-bromophenyl)-3-phenylpropanoate (3zc)



¹³C NMR Spectrum of methyl 2-(4-bromophenyl)-3-phenylpropanoate (3zc)





¹H NMR Spectrum of methyl 2-(4-cyanophenyl)-3-phenylpropanoate (3ze)







¹H NMR Spectrum of methyl 4-(1-methoxy-1-oxo-3-phenylpropan-2-yl)benzoate (3zf)





¹H NMR Spectrum of methyl 3-phenyl-2-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)propanoate **(3zg)**



¹³C NMR Spectrum of methyl 3-phenyl-2-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2yl)phenyl)propanoate (3zg)



3.5 9.0 8.5 8.0 7.5 6.5 6.0 5.0 4.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 7.0 5.5







¹H NMR Spectrum of (E)-1,2,3-triphenylprop-2-en-1-one (4a)





