

Supporting Information

Visible-Light Dual Photoredox/Nickel Catalyzed Hydroacylation of Ethylene with Aromatic Acids

Table of Contents

| | |
|---|----|
| 1 General information | 2 |
| 2 Optimization of the reaction conditions..... | 3 |
| 3 General procedure for hydroacylation of ethylene..... | 6 |
| 4 Investigation of the reaction mechanism..... | 7 |
| 5 Characterization data of products..... | 10 |
| 6 NMR spectra | 22 |
| 7 References..... | 60 |

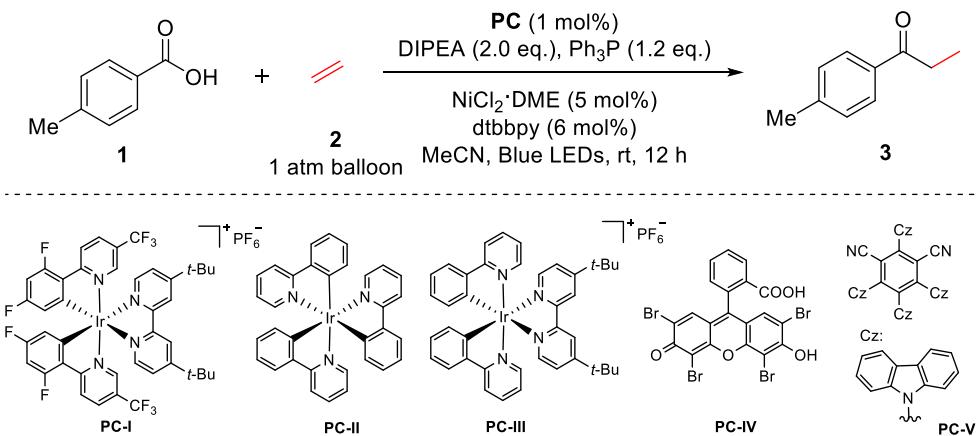
1 General information

General information. Reactions were monitored by TLC on silica gel plates (GF₂₅₄). Column chromatography was performed on silica gel (300-400 mesh). NMR spectra were recorded on a Bruker Ultra-shield 400 MHz spectrometer. ¹H NMR, ¹³C NMR and ¹⁹F NMR are recorded on an NMR spectrometer with CDCl₃ as solvent. Chemical shifts of ¹H, ¹³C, and ¹⁹F NMR spectra are reported in parts per million (ppm) from tetramethylsilane. The residual solvent signals were used as standard, and the chemical shifts were converted to the corresponding scale (CDCl₃: δ H = 7.26 ppm, δ C = 77.00 ppm). Data are represented as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz). The high-resolution mass spectra (HRMS) were detected with the ESI mode of the Micromass Q-TOF instrument. Gas chromatographic (GC) analyses were performed on a GC equipped with a flameionization detector and an Rtx@-65 (30 m × 0.32 mm ID × 0.25 μm df) column. GC-MS analyses were performed on a GC-MS with an EI mode. Melting points (mp) were determined with a digital electrothermal apparatus without further correction. IR spectra were recorded on a Thermo Scientific Nicolet 380 FT-IR spectrometer. The 40 W blue LEDs light was purchased from Kessil (A360NE/WE) with maximum wavelength as 440 nm. Schlenk tubes (10 mL and 100 mL) were purchased from synthware. The compound names were generated by the computer program ChemDraw according to the guidelines specified.

Starting materials. All aromatic acids, solvents, nickel catalysts, ligands and additives were from commercial sources and used without purification unless otherwise noted. The photocatalysts were prepared following the literature procedures.¹

2 Optimization of the reaction conditions

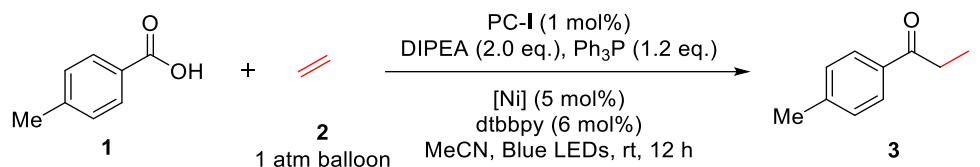
Table S1. Screening of photocatalyst



| Entry ^a | PC | Yield ^b |
|--------------------|------------|--------------------|
| 1 | I | 11% |
| 2 | II | ND |
| 3 | III | ND |
| 4 | IV | ND |
| 5 | V | ND |

^a Unless noted, **1** (0.2 mmol), **2** (ethylene balloon), **PC** (1 mol%), DIPEA (2.0 equiv.), Ph₃P (1.2 equiv.), NiCl₂·DME (5 mol%), dtbbpy (6 mol%), MeCN (2.0 mL), blue light-emitting diodes (440 nm), rt, 12 h. ^b GC yield, biphenyl as internal standard.

Table S2. Screening of nickel catalyst

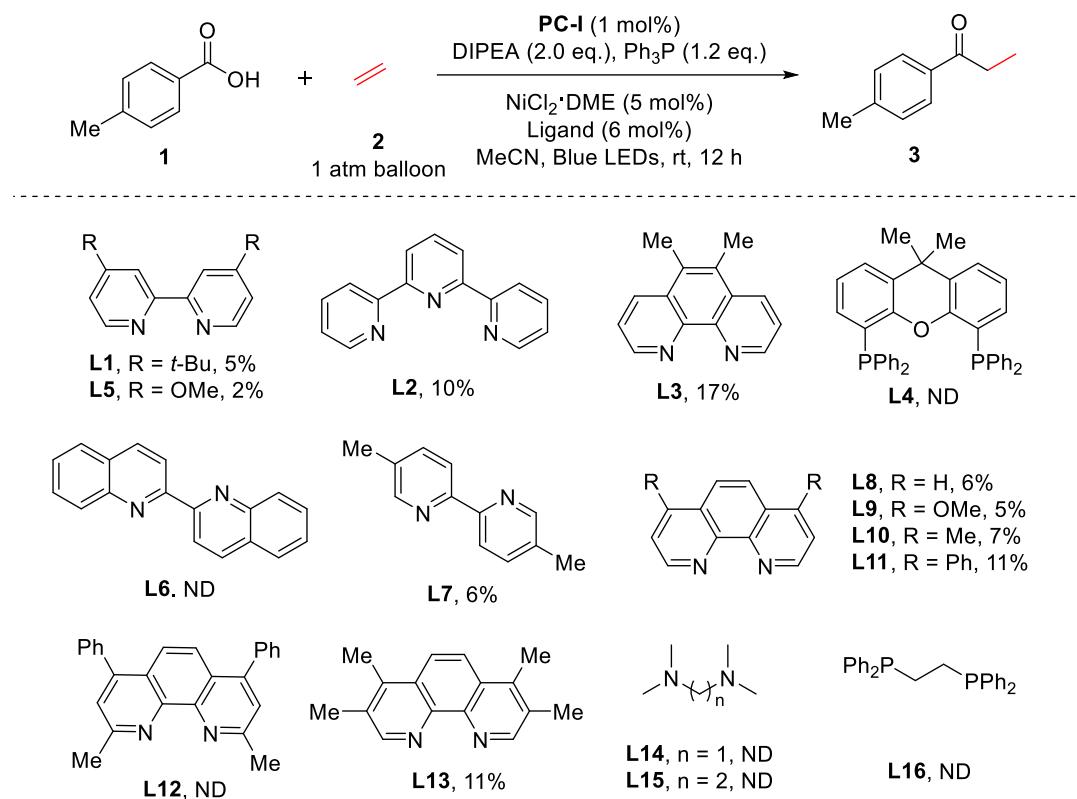


| Entry ^a | [Ni] | Yield ^b |
|--------------------|------------------------|--------------------|
| 1 | NiCl ₂ ·DME | 11% |
| 2 | NiBr ₂ ·DME | trace |
| 3 | NiBr ₂ | trace |
| 4 | NiCl ₂ | trace |
| 5 | Ni(COD) ₂ | trace |

^a Unless noted, **1** (0.2 mmol), **2** (ethylene balloon), **PC-I** (1 mol%), DIPEA (2.0 equiv.), Ph₃P (1.2

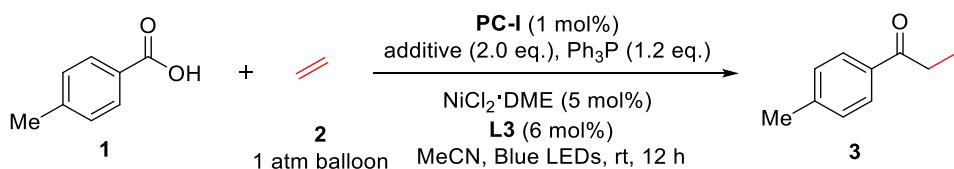
equiv.), [Ni] (5 mol%), dtbbpy (6 mol%), MeCN (2.0 mL), blue light-emitting diodes (440 nm), rt, 12 h. ^b GC yield, biphenyl as internal standard.

Table S3. Screening of ligand



Unless noted, **1** (0.2 mmol), **2** (ethylene balloon), **PC-I** (1 mol%), DIPEA (2.0 equiv.), Ph₃P (1.2 equiv.), NiCl₂·DME (5 mol%), Ligand (6 mol%), MeCN (2.0 mL), blue light-emitting diodes (440 nm), rt, 12 h. GC yield, biphenyl as internal standard.

Table S4. Screening of additive

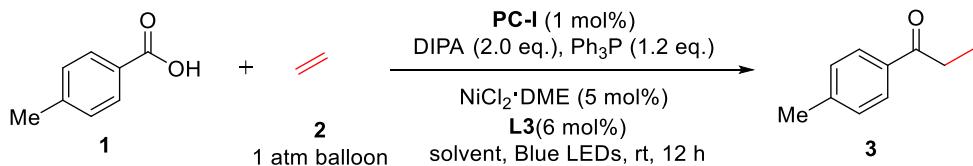


| Entry ^a | Additive | Yield ^b |
|--------------------|----------|--------------------|
| 1 | TEA | 11% |
| 2 | DBU | trace |
| 3 | DABCO | 4% |

| | | |
|----|-------------------------------|-----|
| 4 | 2,6-Lutidine | 28% |
| 5 | 2,4,6-Collidine | 14% |
| 6 | 2,6-di-tert-butylpyridine | 18% |
| 7 | DMAP | 43% |
| 8 | N,N-Dimethylaniline | ND |
| 9 | TMG | 11% |
| 10 | Diethylamine | 42% |
| 11 | Diisopropylamine (DIPA) | 65% |
| 12 | Diisobutylamine | 53% |
| 13 | Dicyclohexylamine | 34% |
| 14 | Diphenylamine | ND |
| 15 | Pyrrolidine | 16% |
| 16 | 2,2,6,6-Tetramethylpiperidine | 23% |

^a Unless noted, **1** (0.2 mmol), **2** (ethylene balloon), **PC-I** (1 mol%), additive (2.0 equiv.), Ph₃P (1.2 equiv.), NiCl₂·DME (5 mol%), **L3** (6 mol%), MeCN (2.0 mL), blue light-emitting diodes (440 nm), rt, 12 h. ^b GC yield, biphenyl as internal standard.

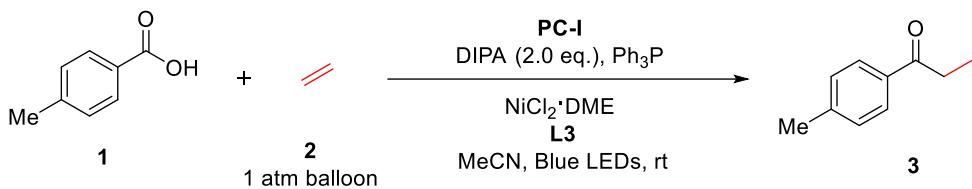
Table S5. Screening of solvent



| Entry ^a | Solvent | Yield ^b |
|--------------------|---------|--------------------|
| 1 | MeCN | 65% |
| 2 | Toluene | ND |
| 3 | DMF | 44 |
| 4 | DMA | 39 |
| 5 | NMP | 32 |
| 6 | MeOH | ND |
| 7 | THF | ND |
| 8 | DCM | ND |

^a Unless noted, **1** (0.2 mmol), **2** (ethylene balloon), **PC-I** (1 mol%), DIPA (2.0 equiv.), Ph₃P (1.2 equiv.), NiCl₂·DME (5 mol%), **L3** (6 mol%), solvent (2.0 mL), blue light-emitting diodes (440 nm), rt, 12 h. ^b GC yield, biphenyl as internal standard.

Table S6. Screening of catalyst dosage



| Entry ^a | PC-I | NiCl ₂ ·DME | L3 | Yield ^b |
|--------------------|--------|------------------------|---------|------------------------|
| 1 | 1 mol% | 5 mol% | 6 mol% | 65% |
| 2 | 1 mol% | 10 mol% | 12 mol% | 51% |
| 3 | 1 mol% | 2.5 mol% | 3 mol% | 75% |
| 4 | 2 mol% | 5 mol% | 6 mol% | 82% (72%) ^c |
| 5 ^d | 2 mol% | 2.5 mol% | 3 mol% | 90% (80%) ^c |

^a Unless noted, **1** (0.2 mmol), **2** (ethylene balloon), PC-I (1 mol%), DIPA (2.0 equiv.), Ph₃P (1.2 equiv.), NiCl₂·DME (5 mol%), L3 (6 mol%), MeCN (2.0 mL), blue light-emitting diodes (440 nm), rt, 12 h. ^b GC yield, biphenyl as internal standard. ^c Isolated yield. ^d 2 h.

3 General procedure for hydroacylation of ethylene

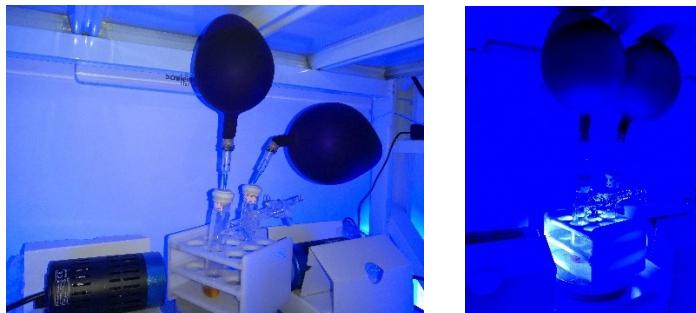
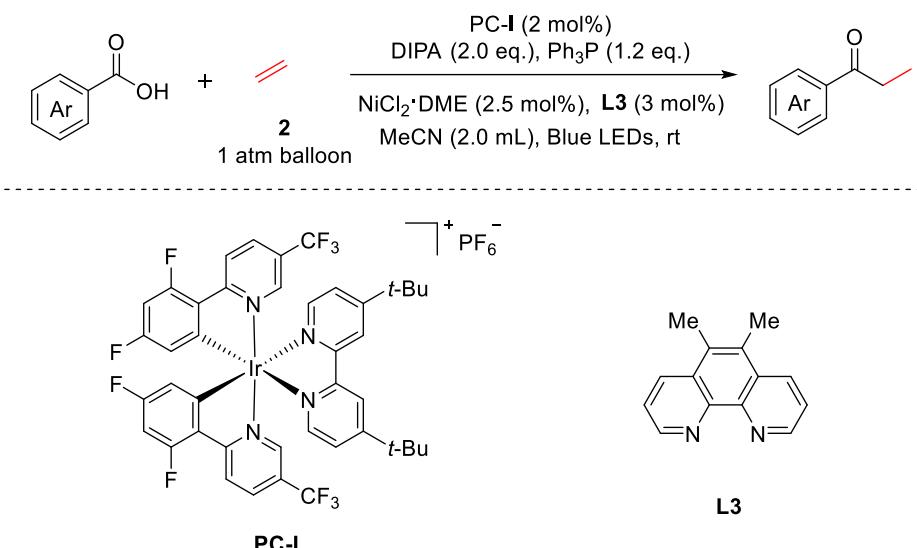


Figure S1. Reaction setup for batch experiments using ethylene balloon.



In the nitrogen-filled glove box, an oven-dried vial (4 mL screw-cap threaded) was successively added stirring bar, $\text{NiCl}_2\cdot\text{DME}$ (1.1 mg, 2.5 mol%), 5,6-dimethyl-1,10-phenanthroline (1.2 mg, 3.0 mol%) and CH_3CN (2.0 mL), and then it was sealed with a Teflon-lined plastic screw-cap and stirred about 30 min. Aromatic carboxylic acid (0.2 mmol, 1.0 equiv.), Ph_3P (62.9 mg, 0.24 mmol, 1.2 equiv, or 78.6 mg, 0.30 mmol, 1.5 equiv) and photocatalyst $[\text{Ir}(\text{dF}(\text{CF}_3)\text{ppy})_2(\text{dtbbpy})]\text{PF}_6$ (4.5 mg, 2 mol%) was added to a 10 mL schlenk flask equipped with a magnetic stirrer bar. This resulting mixture was sealed and degassed via vacuum evacuation and subsequent backfill with ethylene for three times. Then, diisopropylamine, DIPA (56 μL , 2.0 equiv.) and nickel-catalyst solution were subsequently added in this order. The mixture was then irradiated with blue LED (2 x 40 W) with ethylene balloon for 2-6 hours at room temperature (Figure S1, air fan was used to keep the temperature). After completion, the reaction mixture was removed from the light and added water (10 mL) and extracted with ethyl acetate (5 mL) three times. The combined organic layer was successively washed with brine three times and dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel chromatography (hexane/ethyl acetate) to afford the corresponding desired product.

4 Investigation of the reaction mechanism

a) Control Experiment with Additives



In the nitrogen-filled glove box, an oven-dried vial (4 mL screw-cap threaded) was successively added stirring bar, $\text{NiCl}_2 \cdot \text{DME}$ (1.1 mg, 2.5 mol%), 5,6-dimethyl-1,10-phenanthroline (1.2 mg, 3.0 mol%) and CH_3CN (2.0 mL), and then it was sealed with a Teflon-lined plastic screw-cap and stirred about 30 min. *p*-Toluic acid (0.2 mmol, 1.0 equiv.), photocatalyst $[\text{Ir}(\text{dF}(\text{CF}_3)\text{ppy})_2(\text{dtbbpy})]\text{PF}_6$ (4.5 mg, 2 mol%), Ph_3P (62.9 mg, 0.24 mmol, 1.2 equiv) and additives TEMPO (0.8 mmol, 4.0 equiv.) was added to a 10 mL schlenk flask equipped with a magnetic stirrer bar. This resulting mixture was sealed and degassed via vacuum evacuation and subsequent backfill with ethylene for three times. Then, diisopropylamine, DIPA (56 μL , 2.0 equiv.) and nickel-catalyst solution were subsequently added in this order. The mixture was then irradiated with blue LED (2 x 40 W) with ethylene balloon for 2 hours at room temperature. After completion, the reaction mixture was removed from the light. The corresponding product **3** was not detected according to both TLC and GC-Mass analysis. The product 2,2,6,6-tetramethylpiperidin-1-yl 4-methylbenzoate detected by ESI-HRMS, which demonstrates the generation of acyl radical during the reaction process.

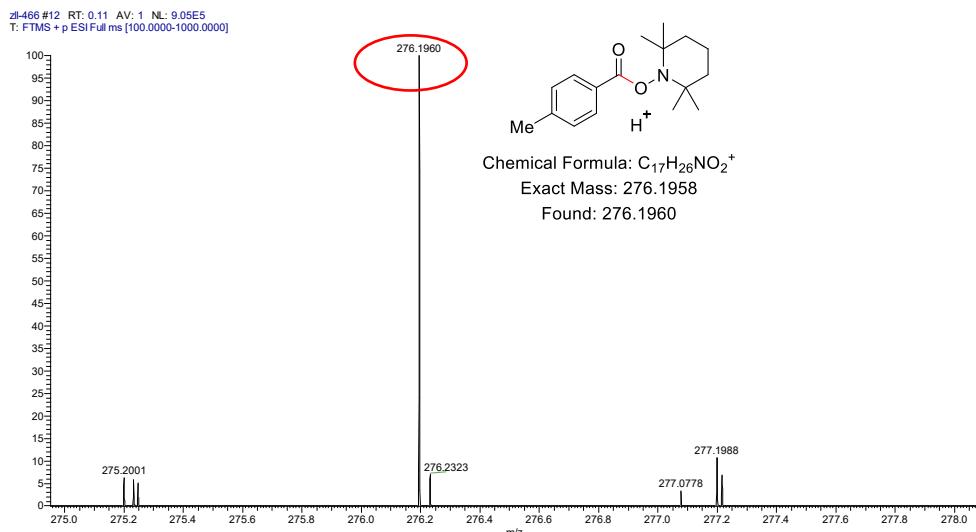
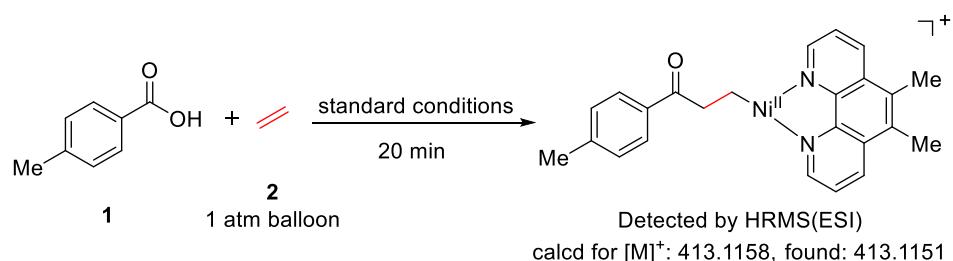
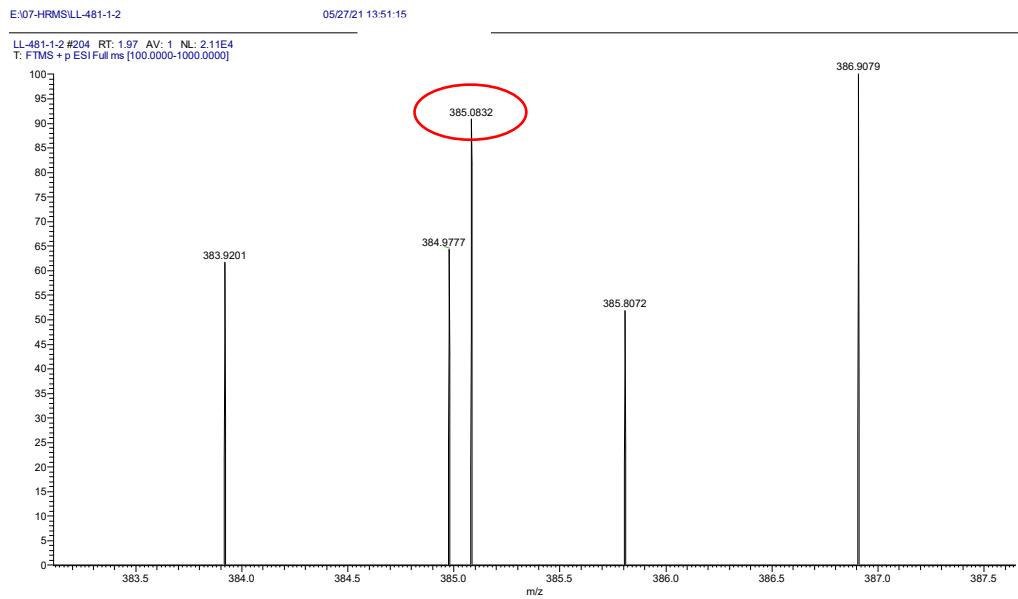
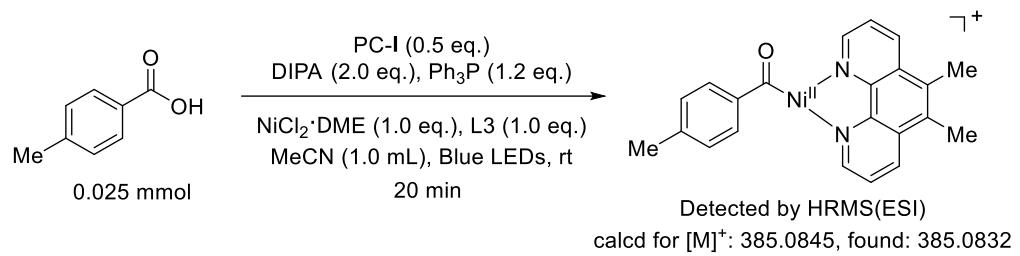
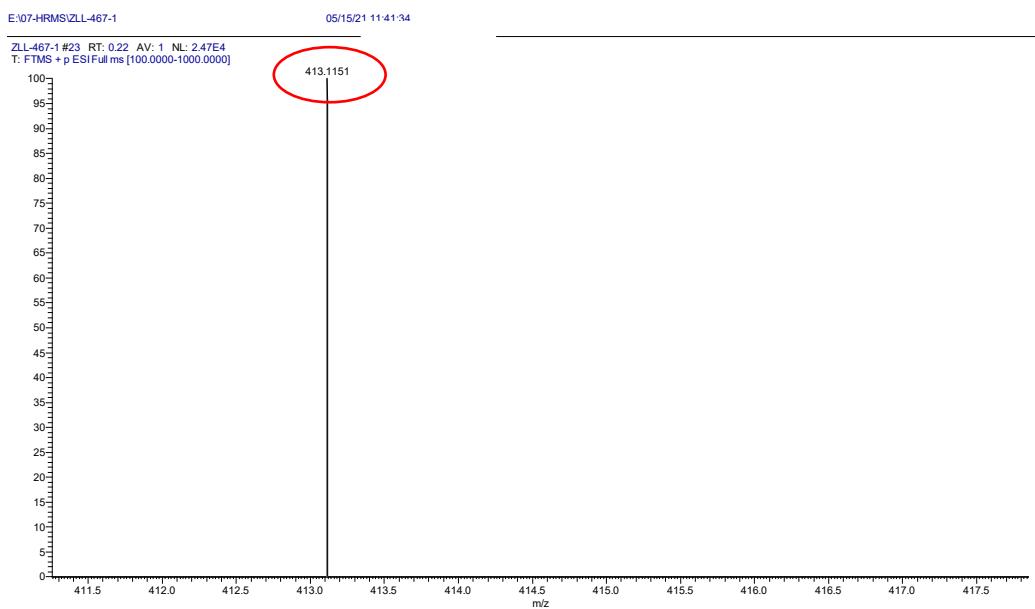


Figure S2. The HRMS -ESI spectra.

b) Detection of reaction intermediate





c) Light ON/OFF Experiments

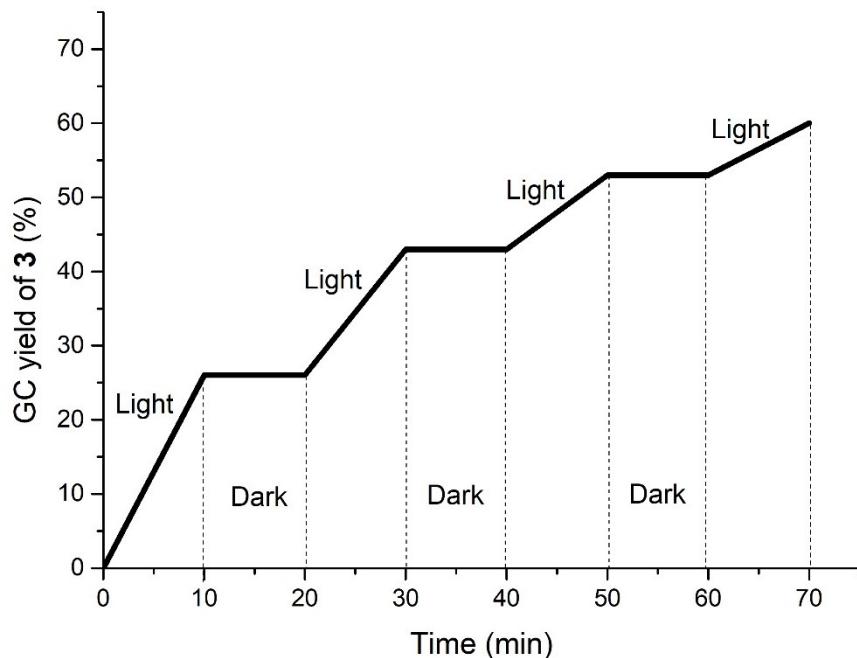
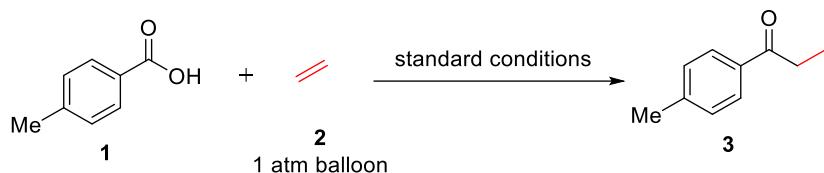


Figure S3. Light on/off experiments.

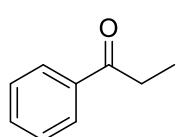
It was found that the reactions need continuous irradiation of light.

5 Characterization data of products

1-(*p*-tolyl)propan-1-one (3)

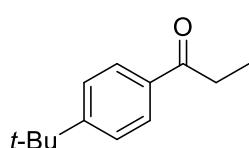
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 23.8 mg, 80% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 7.87 (d, J = 8.2 Hz, 2H), 7.25 (d, J = 7.3 Hz, 2H), 2.98 (q, J = 7.3 Hz, 2H), 2.41 (s, 3 H), 1.22 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 200.6, 143.6, 134.5, 129.2, 128.1, 31.7, 21.6, 8.3. IR (ATR): ν = 2922, 1657, 1606, 1460, 1277, 1174, 1020, 749 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{10}\text{H}_{13}\text{O}$ [$\text{M}+\text{H}]^+$: 149.0961, found: 149.0955. These date are consistent with those reported in the literature.²

Propiophenone (4)



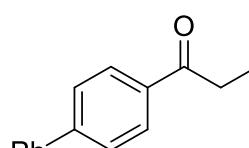
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 18.6 mg, 69% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 7.98-7.95 (m, 2H), 7.57-7.53 (m, 1H), 7.48-7.43 (m, 2H), 3.01 (q, J = 7.2 Hz, 2H), 1.23 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 200.9, 136.9, 132.9, 128.6, 128.0, 31.8, 8.3. IR (ATR): ν = 2971, 1683, 1448, 1217, 950, 743, 689 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_9\text{H}_{11}\text{O} [\text{M}+\text{H}]^+$: 135.0804, found: 135.0805. These date are consistent with those reported in the literature.³

1-(4-(*tert*-butyl)phenyl)propan-1-one (5)



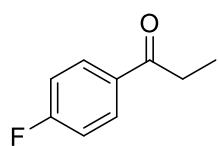
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 34.5 mg, 91% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 7.91 (d, J = 8.6 Hz, 2H), 7.47 (d, J = 8.6 Hz, 2H), 2.98 (q, J = 7.3 Hz, 2H), 1.34 (s, 9H), 1.22 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 200.5, 156.5, 134.4, 128.0, 125.5, 35.1, 31.7, 31.1, 8.4. IR (ATR): ν = 2964, 1682, 1605, 1460, 1225, 1011, 952, 799, 577 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{13}\text{H}_{19}\text{O} [\text{M}+\text{H}]^+$: 191.1430, found: 191.1427. These date are consistent with those reported in the literature.⁴

1-([1,1'-biphenyl]-4-yl)propan-1-one (6)



Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 25.4 mg, 60% yield, white solid, M.P. 90-92 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 8.03 (d, J = 8.4 Hz, 2H), 7.67 (d, J = 8.4 Hz, 2H), 7.63-7.61 (m, 2H), 7.48-7.44 (m, 2H), 7.41-7.37 (m, 1H), 3.03 (q, J = 7.2 Hz, 2H), 1.25 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 200.5, 145.6, 139.9, 135.6, 129.0, 128.6, 128.2, 127.3, 127.2, 31.9, 8.3. IR (ATR): ν = 2929, 1683, 1600, 1348, 1216, 1168, 951, 802, 756, 696 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{15}\text{H}_{15}\text{O} [\text{M}+\text{H}]^+$: 211.1117, found: 211.1121. These date are consistent with those reported in the literature.⁵

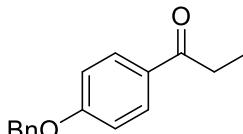
1-(4-fluorophenyl)propan-1-one (7)



Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 19.9 mg, 65% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 8.01-7.98 (m, 2H), 7.15-7.11 (m, 2H), 7.48-7.43 (m, 2H), 2.98 (q, J = 7.2 Hz, 2H), 1.23 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 199.2, 165.6 (d, J = 255.1 Hz), 133.3 (d, J = 3.1 Hz), 130.6 (d, J = 9.2 Hz), 115.6 (d, J = 21.9 Hz), 37.1, 8.2. ^{19}F NMR (376 MHz, CDCl_3) δ -105.79. IR (ATR): ν = 2980, 1685, 1596, 1351, 1217, 1156, 848, 798, 591 cm^{-1} . HRMS (ESI)

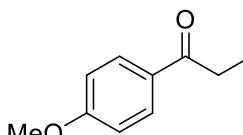
Calculated for C₉H₁₀FO [M+H]⁺: 153.0710, found: 153.0709. These date are consistent with those reported in the literature.²

1-(4-(benzyloxy)phenyl)propan-1-one (8)



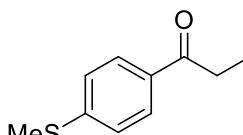
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 35.5 mg, 74% yield, white solid, M.P. 94-96 °C. ¹H NMR (400 MHz, CDCl₃): δ (ppm) 7.94 (d, *J* = 8.9 Hz, 2H), 7.43-7.31 (m, 5H), 6.99 (d, *J* = 8.9 Hz, 2H), 5.11 (s, 2H), 2.93 (q, *J* = 7.3 Hz, 2H), 1.20 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃): δ (ppm) 199.5, 162.5, 136.2, 130.2, 130.2, 128.7, 128.2, 127.5, 114.5, 70.1, 31.4, 8.5. IR (ATR): ν = 2937, 1676, 1595, 1385, 1222, 1011, 950, 756, 699 cm⁻¹. HRMS (ESI) Calculated for C₁₆H₁₇O₂ [M+H]⁺: 241.1223, found: 241.1218. These date are consistent with those reported in the literature.⁶

1-(4-methoxyphenyl)propan-1-one (9)



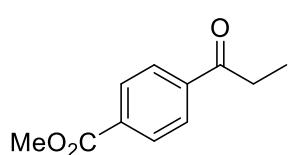
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 30.3 mg, 92% yield, yellow oil. ¹H NMR (400 MHz, CDCl₃): δ (ppm) 7.95 (d, *J* = 9.0 Hz, 2H), 6.93 (d, *J* = 8.9 Hz, 2H), 3.86 (s, 3H), 2.95 (q, *J* = 7.3 Hz, 2H), 1.21 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃): δ (ppm) 199.5, 163.3, 130.2, 130.0, 113.7, 55.4, 31.4, 8.4. IR (ATR): ν = 2937, 1675, 1598, 1508, 1351, 1223, 1168, 1029, 951, 797, 632 cm⁻¹. HRMS (ESI) Calculated for C₁₀H₁₃O₂ [M+H]⁺: 165.0910, found: 165.0907. These date are consistent with those reported in the literature.³

1-(4-(methylthio)phenyl)propan-1-one (10)



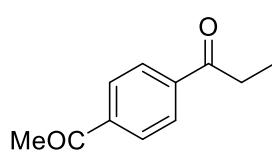
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 25.0 mg, 69% yield, white solid, M.P. 64-67 °C. ¹H NMR (400 MHz, CDCl₃): δ (ppm) 7.87 (d, *J* = 8.6 Hz, 2H), 7.26 (d, *J* = 8.6 Hz, 2H), 2.96 (q, *J* = 7.2 Hz, 2H), 2.5 (s, 3H), 1.23 (t, *J* = 7.2 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃): δ (ppm) 199.9, 145.5, 133.2, 128.4, 125.0, 31.6, 14.8, 8.3. IR (ATR): ν = 2937, 1669, 1586, 1401, 1221, 1092, 948, 791, 524 cm⁻¹. HRMS (ESI) Calculated for C₁₀H₁₃OS [M+H]⁺: 181.0682, found: 181.0679. These date are consistent with those reported in the literature.⁷

methyl 4-propionylbenzoate (11)



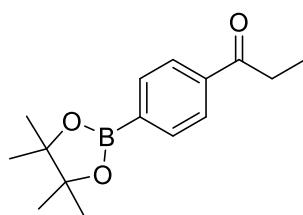
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 20:1. 23.4 mg, 61% yield, white solid, M.P. 77-79 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 8.12 (d, J = 8.4 Hz, 2H), 8.01 (d, J = 8.4 Hz, 2H), 4.0 (s, 3H), 3.04 (q, J = 7.2 Hz, 2H), 1.24 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 200.2, 166.3, 140.1, 133.7, 129.8, 127.9, 52.4, 32.2, 8.1. IR (ATR): ν = 2936, 1720, 1677, 1281, 1109, 951, 759, 696 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{11}\text{H}_{13}\text{O}_3$ [$\text{M}+\text{H}]^+$: 193.0859, found: 193.0856. These date are consistent with those reported in the literature.⁸

1-(4-acetylphenyl)propan-1-one (12)



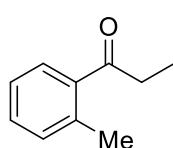
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 10:1. 20.8 mg, 59% yield, white solid, M.P. 63-66 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 8.06-8.01 (m, 4H), 3.04 (q, J = 7.2 Hz, 2H), 2.65 (s, 3H), 1.25 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 200.2, 197.5, 140.1, 140.0, 128.5, 128.2, 32.2, 26.9, 8.1. IR (ATR): ν = 2918, 1672, 1351, 1218, 950, 796, 593 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{11}\text{H}_{13}\text{O}_2$ [$\text{M}+\text{H}]^+$: 177.0910, found: 177.0908. These date are consistent with those reported in the literature.⁹

1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)propan-1-one (13)



Eluent for flash column chromatography: petroleum ether/ethyl acetate = 5:1. 31.4 mg, 60% yield, yellow solid, M.P. 72-74 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 7.94 (d, J = 8.2 Hz, 2H), 7.89 (d, J = 8.2 Hz, 2H), 3.01 (q, J = 7.2 Hz, 2H), 1.36 (s, 12H), 1.22 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 201.1, 138.8, 134.9, 127.0, 84.2, 32.0, 24.9, 8.2. IR (ATR): ν = 2980, 1687, 1506, 1357, 1215, 1088, 954, 852, 734, 651 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{15}\text{H}_{22}\text{BO}_3$ [$\text{M}+\text{H}]^+$: 260.1657, found: 261.1649. These date are consistent with those reported in the literature.¹⁰

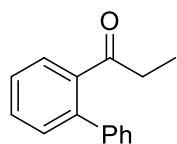
1-(*o*-tolyl)propan-1-one (14)



Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 21.3 mg, 72% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 7.55-7.53 (m, 1H), 7.30-7.26 (m, 1H), 7.19-7.15 (m, 2H), 2.84 (q, J = 7.3 Hz, 2H), 2.41 (s, 3H), 1.12 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 205.2, 138.1, 137.8, 131.9, 131.0, 128.2, 125.6, 34.7, 21.3, 8.4. IR (ATR): ν = 2927, 1687, 1457, 1217, 950, 752 cm^{-1} . HRMS (ESI) Calculated for

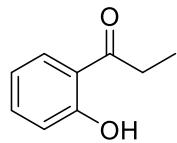
$C_{10}H_{13}O$ $[M+H]^+$: 149.0961, found: 149.0959. These date are consistent with those reported in the literature.¹¹

1-([1,1'-biphenyl]-2-yl)propan-1-one (15)



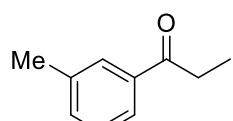
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 33.0 mg, 79% yield, yellow oil. 1H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.51-7.45 (m, 2H), 7.44-7.37 (m, 5H), 7.36-7.31 (m, 2H), 2.25 (q, J = 7.3 Hz, 2H), 0.90 (t, J = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 208.8, 141.1, 140.7, 139.9, 130.4, 130.1, 128.8, 128.7, 127.8, 127.6, 127.5, 36.2, 8.6. IR (ATR): ν = 2937, 1686, 1596, 1347, 1212, 1113, 1077, 1009, 946, 778, 747 cm^{-1} . HRMS (ESI) Calculated for $C_{15}H_{15}O$ $[M+H]^+$: 211.1117, found: 211.1121. These date are consistent with those reported in the literature.¹²

1-(2-hydroxyphenyl)propan-1-one (16)



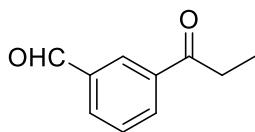
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 17.1 mg, 57% yield, yellow oil. 1H NMR (400 MHz, $CDCl_3$): δ (ppm) 12.36 (s, 1H), 7.78-7.76 (m, 1H), 7.48-7.44 (m, 1H), 6.99-6.97 (m, 1H), 6.91-6.87 (m, 1H), 3.05 (q, J = 7.2 Hz, 2H), 1.24 (t, J = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 207.1, 162.4, 136.1, 129.8, 119.2, 118.9, 118.5, 31.6, 8.2. IR (ATR): ν = 2984, 1641, 1488, 1265, 1206, 906, 272, 649 cm^{-1} . HRMS (ESI) Calculated for $C_9H_{11}O_2$ $[M+H]^+$: 151.0754, found: 151.0752. These date are consistent with those reported in the literature.¹³

1-(*m*-tolyl)propan-1-one (17)



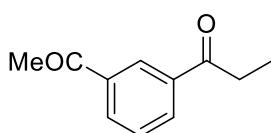
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 26.1 mg, 88% yield, yellow oil. 1H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.78-7.74 (m, 2H), 7.37-7.31 (m, 2H), 2.99 (q, J = 7.2 Hz, 2H), 2.41 (s, 3H), 1.22 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 201.1, 138.3, 137.0, 133.6, 128.5, 128.4, 125.2, 31.8, 21.4, 8.3. IR (ATR): ν = 2977, 1684, 1459, 1347, 1249, 1165, 965, 772, 689 cm^{-1} . HRMS (ESI) Calculated for $C_{10}H_{13}O$ $[M+H]^+$: 149.0961, found: 149.0959. These date are consistent with those reported in the literature.¹⁴

3-propionylbenzaldehyde (18)



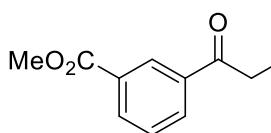
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 20:1. 14.4 mg, 44% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 10.08 (s, 1H), 8.44-8.43 (m, 1H), 8.24-8.21 (m, 1H), 8.08-8.05 (m, 1H), 7.66-7.62 (m, 1H), 3.06 (q, J = 7.2 Hz, 2H), 1.24 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 199.7, 191.5, 137.6, 136.6, 133.5, 133.4, 129.5, 129.2, 32.0, 8.1. IR (ATR): ν = 2979, 1685, 1597, 1379, 1151, 974, 784, 682 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{10}\text{H}_{11}\text{O}_2$ [$\text{M}+\text{H}]^+$: 163.0754, found: 163.0751. These date are consistent with those reported in the literature.¹⁵

1-(3-acetylphenyl)propan-1-one (19)



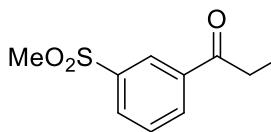
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 20:1. 32.2 mg, 91% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 8.50-8.49 (m, 1H), 8.14-8.10 (m, 2H), 7.57-7.53 (m, 1H), 3.03 (q, J = 7.2 Hz, 2H), 2.63 (s, 3H), 1.21 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 200.0, 197.4, 137.4, 137.2, 132.3, 132.2, 129.0, 127.7, 32.0, 26.7, 8.1. IR (ATR): ν = 2978, 1682, 1357, 1271, 1196, 976, 787, 685 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{11}\text{H}_{13}\text{O}_2$ [$\text{M}+\text{H}]^+$: 177.0910, found: 177.0907. These date are consistent with those reported in the literature.¹⁶

methyl 3-propionylbenzoate (20)



Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 35.3 mg, 92% yield, yellow solid, M.P. 44-46 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 8.57-8.56 (m, 1H), 8.20-8.18 (m, 1H), 8.15-8.12 (m, 1H), 7.54-7.50 (m, 1H), 3.93 (s, 3H), 3.03 (q, J = 7.2 Hz, 2H), 1.22 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 199.9, 166.3, 137.1, 133.7, 132.1, 130.6, 129.1, 128.8, 52.4, 32.0, 8.1. IR (ATR): ν = 2916, 1729, 1677, 1427, 1292, 1202, 952, 741, 680 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{11}\text{H}_{13}\text{O}_3$ [$\text{M}+\text{H}]^+$: 193.0859, found: 193.0857. These date are consistent with those reported in the literature.¹⁷

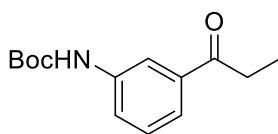
1-(3-(methylsulfonyl)phenyl)propan-1-one (21)



Eluent for flash column chromatography: petroleum ether/ethyl acetate = 1:1. 30.5 mg, 72% yield, yellow solid, M.P. 68-71 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 8.48-8.47 (m, 1H), 8.23-8.21 (m, 1H), 8.12-8.09 (m, 1H), 7.70-7.66 (m, 1H), 3.08 (s, 3H), 3.04 (q, J = 7.2 Hz, 2H), 1.23 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 198.9, 141.4, 137.9, 132.8, 131.2, 130.0, 126.8, 44.4, 32.1, 8.0. IR

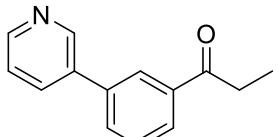
(ATR): ν = 3020, 2942, 1684, 1423, 1290, 1144, 972, 754, 673, 535 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{10}\text{H}_{13}\text{SO}_3$ [M+H]⁺: 213.0580, found: 213.0588. These date are consistent with those reported in the literature.¹⁸

tert-butyl (3-propionylphenyl)carbamate (22)



Eluent for flash column chromatography: petroleum ether/ethyl acetate = 20:1. 43.0 mg, 86% yield, yellow solid, M.P. 76-79 °C. ¹H NMR (400 MHz, CDCl_3): δ (ppm) 7.97-7.96 (m, 1H), 7.68-7.66 (m, 1H), 7.61-7.58 (m, 1H), 7.37-7.33 (m, 1H), 7.00 (s, 1H), 2.97 (q, J = 7.2 Hz, 2H), 1.51 (s, 9H), 1.19 (t, J = 7.2 Hz, 3H). ¹³C NMR (101 MHz, CDCl_3): δ (ppm) 200.8, 152.8, 139.0, 137.6, 129.2, 122.9, 122.5, 117.9, 80.8, 31.9, 28.3, 8.2. IR (ATR): ν = 3323, 2975, 1724, 1674, 1536, 1230, 1154, 776, 684 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{14}\text{H}_{20}\text{NO}_3$ [M+H]⁺: 250.1438, found: 250.1433.

1-(3-(pyridin-3-yl)phenyl)propan-1-one (23)



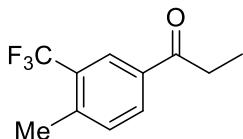
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 5:1. 29.7 mg, 70% yield, yellow oil. ¹H NMR (400 MHz, CDCl_3): δ (ppm) 8.85-8.84 (m, 1H), 8.60-8.59 (m, 1H), 8.15-8.14 (m, 1H), 7.97-7.95 (m, 1H), 7.90-7.87 (m, 1H), 7.75-7.72 (m, 1H), 7.57-7.53 (m, 1H), 7.38-7.35 (m, 1H), 3.03 (q, J = 7.2 Hz, 2H), 1.23 (t, J = 7.2 Hz, 3H). ¹³C NMR (101 MHz, CDCl_3): δ (ppm) 200.4, 148.9, 148.2, 138.3, 137.7, 135.8, 134.5, 131.4, 129.4, 127.6, 126.6, 123.7, 32.0, 8.2. IR (ATR): ν = 2977, 1683, 1432, 1349, 1204, 1023, 959, 780, 710, 692 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{14}\text{H}_{14}\text{NO}$ [M+H]⁺: 212.1070, found: 212.1067.

2-(3-propionylphenyl)propanenitrile (24)



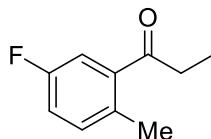
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 10:1. 33.2 mg, 89% yield, yellow oil. ¹H NMR (400 MHz, CDCl_3): δ (ppm) 7.93-7.88 (m, 2H), 7.58-7.58 (m, 1H), 7.50-7.46 (m, 1H), 3.97 (q, J = 7.3 Hz, 1H), 3.00 (q, J = 7.2 Hz, 2H), 1.66 (d, J = 7.3 Hz, 3H), 1.21 (t, J = 7.2 Hz, 3H). ¹³C NMR (101 MHz, CDCl_3): δ (ppm) 200.1, 137.7, 131.1, 129.5, 127.7, 126.2, 121.1, 32.0, 31.2, 21.3, 8.1. IR (ATR): ν = 2982, 1686, 1455, 1352, 1230, 1165, 784, 693 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{12}\text{H}_{14}\text{NO}$ [M+H]⁺: 188.1070, found: 188.1066. These date are consistent with those reported in the literature.¹⁹

1-(4-methyl-3-(trifluoromethyl)phenyl)propan-1-one (25)



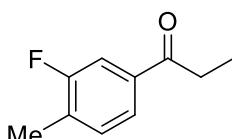
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 20.9 mg, 48% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 8.20 (s, 1H), 8.01 (d, J = 7.9 Hz, 1H), 7.32 (d, J = 8.0 Hz, 1H), 3.00 (q, J = 7.2 Hz, 2H), 2.55-2.54 (m, 3H), 1.24 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 199.2, 141.9, 134.7, 132.3, 131.0, 129.3 (d, J = 30.5 Hz), 125.5 (q, J = 5.7 Hz), 122.7, 31.8, 19.5, 8.1. ^{19}F NMR (376 MHz, CDCl_3) δ -61.97. IR (ATR): ν = 2982, 1691, 1315, 1201, 1116, 1054, 799, 669 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{11}\text{H}_{12}\text{F}_3\text{O}$ [$\text{M}+\text{H}]^+$: 217.0835, found: 217.0828.

1-(5-fluoro-2-methylphenyl)propan-1-one (26)



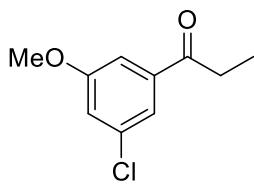
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 16.6 mg, 50% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 7.29-7.26 (m, 1H), 7.19-7.16 (m, 1H), 7.06-7.01 (m, 1H), 2.86 (q, J = 7.2 Hz, 2H), 2.41 (s, 3H), 1.17 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 203.7 (d, J = 2.1 Hz), 160.6 (d, J = 246.0 Hz), 139.2 (d, J = 5.5 Hz), 133.3, 133.2 (d, J = 7.3 Hz), 117.7 (d, J = 20.8 Hz), 114.9 (d, J = 22.3 Hz), 34.7, 20.4, 8.2. ^{19}F NMR (376 MHz, CDCl_3) δ -116.98. IR (ATR): ν = 2979, 1690, 1492, 1239, 1169, 887, 815 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{10}\text{H}_{12}\text{FO}$ [$\text{M}+\text{H}]^+$: 167.0867, found: 167.0865.

1-(3-fluoro-4-methylphenyl)propan-1-one (27)



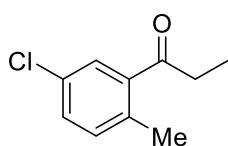
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 19.5 mg, 59% yield, white solid, M.P. 36-39 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 7.65-7.63 (m, 1H), 7.61-7.57 (m, 1H), 7.28-7.24 (m, 1H), 2.96 (q, J = 7.2 Hz, 2H), 2.33 (d, J = 1.8 Hz, 3H), 1.22 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 199.3 (d, J = 1.8 Hz), 161.2 (d, J = 247.1 Hz), 136.6 (d, J = 6.1 Hz), 131.5 (d, J = 4.9 Hz), 130.5 (d, J = 17.6 Hz), 123.5 (d, J = 3.3 Hz), 114.3 (d, J = 23.2 Hz), 31.8, 14.8 (d, J = 3.5 Hz), 8.2. ^{19}F NMR (376 MHz, CDCl_3) δ -116.46. IR (ATR): ν = 2925, 1677, 1573, 1415, 1247, 1159, 869, 790 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{10}\text{H}_{12}\text{FO}$ [$\text{M}+\text{H}]^+$: 167.0867, found: 167.0865. These date are consistent with those reported in the literature.²⁰

1-(3-chloro-5-methoxyphenyl)propan-1-one (28)



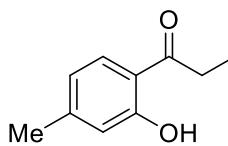
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 19.5 mg, 49% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 7.51-7.50 (m, 1H), 7.38-7.37 (m, 1H), 7.08-7.07 (m, 1H), 3.85 (s, 3H), 2.96 (q, J = 7.2 Hz, 2H), 1.22 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 199.3, 160.5, 139.1, 135.3, 120.7, 118.9, 111.4, 55.8, 32.0, 8.1. IR (ATR): ν = 2939, 1690, 1574, 1414, 1268, 1172, 1053, 801, 673 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{10}\text{H}_{12}\text{ClO}_2$ [$\text{M}+\text{H}]^+$: 199.0520, found: 199.0518. These date are consistent with those reported in the literature.²¹

1-(5-chloro-2-methylphenyl)propan-1-one (29)



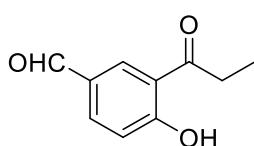
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 20.8 mg, 57% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 7.56 (d, J = 2.2 Hz, 1H), 7.32-7.30 (m, 1H), 7.17 (d, J = 8.2 Hz, 1H), 2.88 (q, J = 7.2 Hz, 2H), 2.43 (s, 3H), 1.18 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 203.8, 139.4, 136.1, 133.2, 131.3, 130.8, 128.1, 34.8, 20.6, 8.2. IR (ATR): ν = 2936, 1689, 1561, 1447, 1193, 1109, 957, 810 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{10}\text{H}_{12}\text{ClO}$ [$\text{M}+\text{H}]^+$: 183.0571, found: 183.0569. These date are consistent with those reported in the literature.²²

1-(2-hydroxy-4-methylphenyl)propan-1-one (30)



Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 17.1 mg, 52% yield, white solid, M.P. 39-40 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 12.38 (s, 1H), 7.64 (d, J = 8.2 Hz, 1H), 6.78 (s, 1H), 6.70 (d, J = 8.2 Hz, 1H), 3.00 (q, J = 7.3 Hz, 2H), 2.34 (s, 3H), 1.23 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 206.6, 162.5, 147.7, 129.7, 120.1, 118.5, 117.0, 31.4, 21.9, 8.4. IR (ATR): ν = 2918, 1633, 1356, 1206, 1011, 929, 788, 726 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{10}\text{H}_{13}\text{O}_2$ [$\text{M}+\text{H}]^+$: 165.0910, found: 165.0909. These date are consistent with those reported in the literature.²³

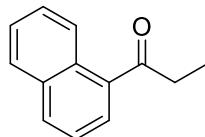
4-hydroxy-3-propionylbenzaldehyde (31)



Eluent for flash column chromatography: petroleum ether/ethyl acetate = 20:1. 18.2 mg, 51% yield, white solid, M.P. 105-108 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 12.94 (s, 1H), 9.90 (s, 1H), 8.34 (d, J = 2.0 Hz, 1H), 8.00-7.98 (m, 1H), 7.11 (d, J = 8.6 Hz, 1H), 3.15 (q, J = 7.2 Hz, 2H), 1.28 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3):

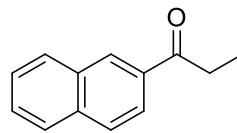
δ (ppm) 207.1, 189.8, 167.3, 136.7, 132.7, 128.2, 119.6, 119.0, 31.7, 8.0. IR (ATR): ν = 2930, 2850, 1688, 1642, 1604, 1565, 1381, 1164, 971, 815, 624 cm⁻¹. HRMS (ESI) Calculated for C₁₀H₁₁O₃ [M+H]⁺: 179.0703, found: 179.0700.

1-(naphthalen-1-yl)propan-1-one (32)



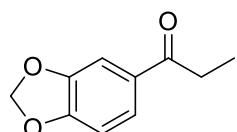
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 24.7 mg, 67% yield, colorless oil. ¹H NMR (400 MHz, CDCl₃): δ (ppm) 8.55 (d, J = 8.6 Hz, 1H), 7.97-7.95 (m, 1H), 7.87-7.82 (m, 2H), 7.59-7.46 (m, 3H), 3.07 (q, J = 7.3 Hz, 2H), 1.28 (t, J = 7.2 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃): δ (ppm) 205.4, 136.2, 134.0, 132.3, 130.1, 128.4, 127.8, 127.1, 126.4, 125.8, 124.4, 35.4, 8.7. IR (ATR): ν = 2936, 1678, 1507, 1229, 1109, 931, 771 cm⁻¹. HRMS (ESI) Calculated for C₁₃H₁₃O [M+H]⁺: 185.0961, found: 185.0958. These date are consistent with those reported in the literature.²

1-(naphthalen-2-yl)propan-1-one (33)



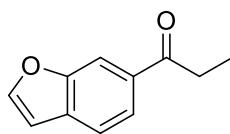
Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 22.3 mg, 61% yield, white solid, M.P. 53-55 °C. ¹H NMR (400 MHz, CDCl₃): δ (ppm) 8.47 (s, 1H), 8.05-8.02 (m, 1H), 7.95 (d, J = 8.0 Hz, 1H), 7.89-7.85 (m, 2H), 7.61-7.52 (m, 2H), 3.13 (q, J = 7.2 Hz, 2H), 1.29 (t, J = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ (ppm) 200.8, 135.5, 134.2, 132.6, 129.5, 129.5, 128.4, 128.3, 128.3, 127.8, 126.7, 123.9, 31.9, 8.4. IR (ATR): ν = 2938, 1680, 1375, 1187, 1126, 800, 755 cm⁻¹. HRMS (ESI) Calculated for C₁₃H₁₃O [M+H]⁺: 185.0961, found: 185.0958. These date are consistent with those reported in the literature.⁸

1-(benzo[d][1,3]dioxol-5-yl)propan-1-one (34)

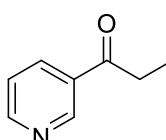


Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 31.0 mg, 87% yield, yellow solid, M.P. 33-35 °C. ¹H NMR (400 MHz, CDCl₃): δ (ppm) 7.58-7.55 (m, 1H), 7.44 (d, J = 1.7 Hz, 1H), 6.84 (d, J = 8.1 Hz, 1H), 6.04 (s, 2H), 2.92 (q, J = 7.3 Hz, 2H), 1.20 (t, J = 7.3 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ (ppm) 199.0, 151.5, 148.1, 131.8, 124.1, 107.9, 107.8, 101.8, 31.5, 8.5. IR (ATR): ν = 2906, 1670, 1438, 1245, 1037, 934, 792 cm⁻¹. HRMS (ESI) Calculated for C₁₀H₁₁O₃ [M+H]⁺: 179.0703, found: 179.0701. These date are consistent with those reported in the literature.²⁴

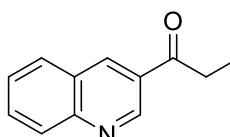
1-(benzofuran-6-yl)propan-1-one (35)


 Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 30.1 mg, 86% yield, white solid, M.P. 73-75 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 8.26 (d, J = 1.6 Hz, 1H), 7.98-7.96 (m, 1H), 7.68 (d, J = 2.2 Hz, 1H), 7.53 (d, J = 8.7 Hz, 1H), 6.85-6.84 (m, 1H), 3.06 (q, J = 7.2 Hz, 2H), 1.25 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 200.3, 157.4, 146.3, 132.5, 127.5, 124.7, 122.2, 111.4, 107.3, 31.9, 8.5. IR (ATR): ν = 3109, 2851, 1678, 1609, 1537, 1340, 1262, 1103, 1010, 964, 802, 772, 744 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{11}\text{H}_{11}\text{O}_2$ [$\text{M}+\text{H}]^+$: 175.0754, found: 175.0752.

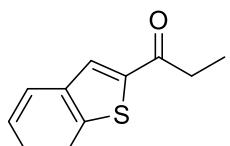
1-(pyridin-3-yl)propan-1-one (36)


 Eluent for flash column chromatography: petroleum ether/ethyl acetate = 3:1. 16.2 mg, 60% yield, yellow oil. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 9.16-9.15 (m, 1H), 8.76-8.75 (m, 1H), 8.24-8.21 (m, 1H), 7.42-7.39 (m, 1H), 3.02 (q, J = 7.2 Hz, 2H), 1.23 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 199.5, 153.3, 149.5, 135.3, 132.1, 123.6, 77.4, 77.0, 76.7, 32.2, 7.9. IR (ATR): ν = 2979, 1688, 1585, 1226, 949, 785, 702 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_8\text{H}_{10}\text{NO}$ [$\text{M}+\text{H}]^+$: 136.0757, found: 136.0755. These date are consistent with those reported in the literature.²⁵

1-(quinolin-3-yl)propan-1-one (37)


 Eluent for flash column chromatography: petroleum ether/ethyl acetate = 3:1. 18.7 mg, 51% yield, yellow solid, M.P. 69-73 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 9.42 (d, J = 2.2 Hz, 1H), 8.69 (d, J = 1.9 Hz, 1H), 8.13 (d, J = 8.5 Hz, 1H), 7.93-7.91 (m, 1H), 7.83-7.79 (m, 1H), 7.63-7.58 (m, 1H), 3.13 (q, J = 7.2 Hz, 2H), 1.28 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 199.4, 149.7, 149.1, 136.8, 131.8, 129.4, 129.3, 129.0, 127.5, 126.9, 32.2, 8.0. ^{19}F NMR (375 MHz, CDCl_3) δ -68.2. IR (ATR): ν = 2929, 1683, 1373, 1174, 908, 728 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{12}\text{H}_{12}\text{NO}$ [$\text{M}+\text{H}]^+$: 186.0913, found: 186.0912. These date are consistent with those reported in the literature.²⁶

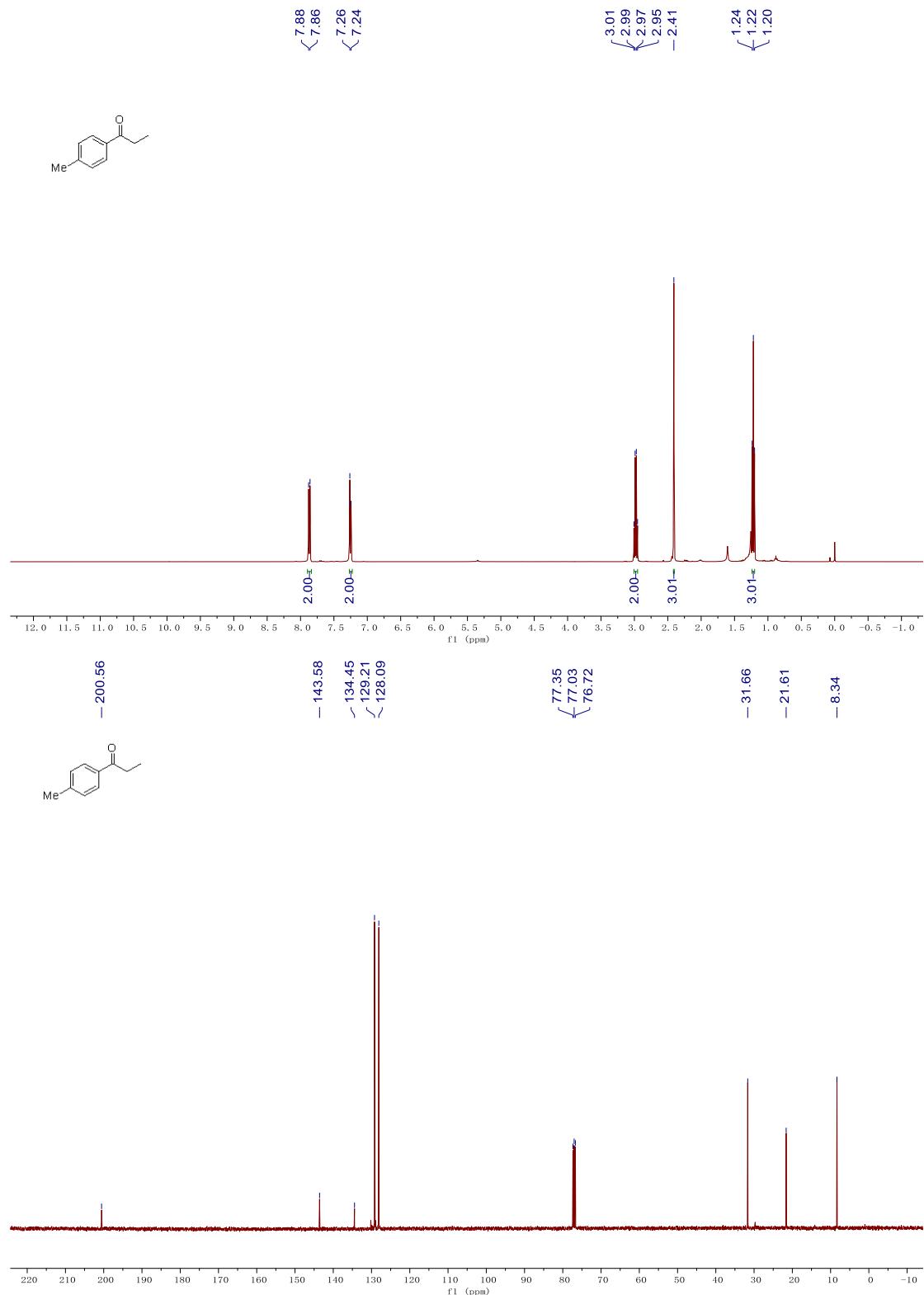
1-(benzo[b]thiophen-2-yl)propan-1-one (38)


 Eluent for flash column chromatography: petroleum ether/ethyl acetate = 50:1. 15.6 mg, 41% yield, yellow solid, M.P. 70-73 °C. ^1H NMR (400 MHz, CDCl_3): δ (ppm) 7.95 (s, 1H), 7.89-7.85 (m, 2H), 7.48-7.38 (m, 2H), 3.05 (q, J = 7.3 Hz, 2H), 1.28 (t, J = 7.3 Hz,

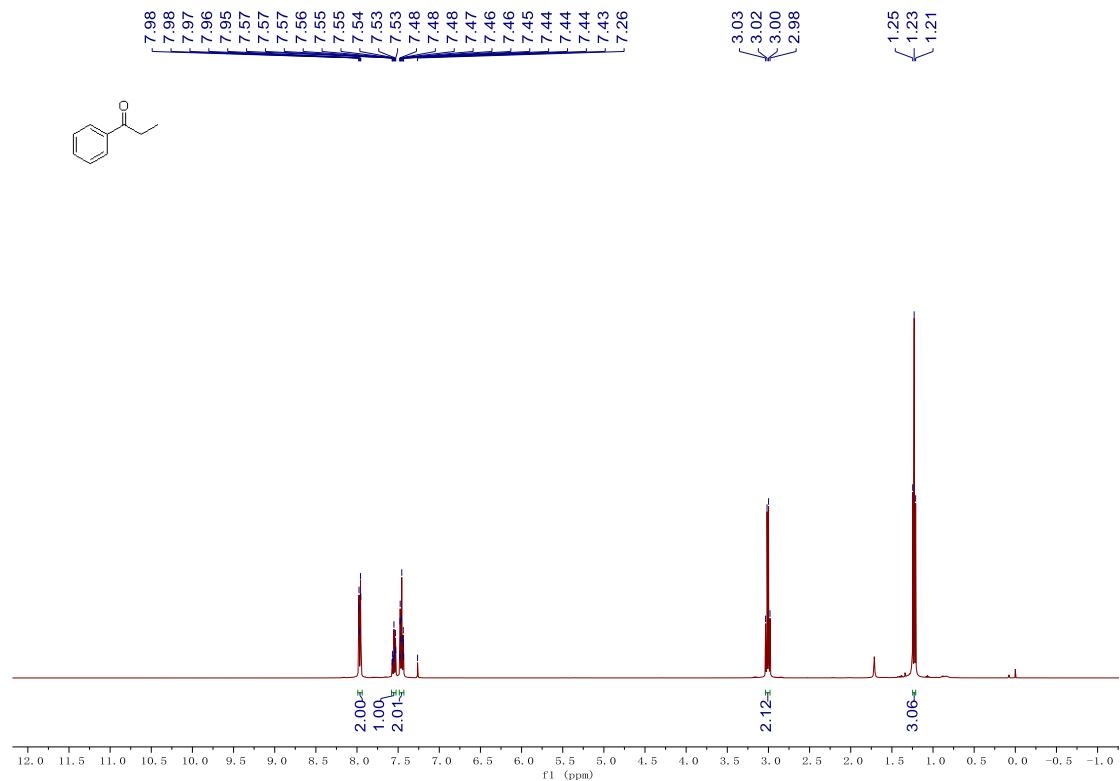
3H). ^{13}C NMR (101 MHz, CDCl_3): δ (ppm) 195.3, 143.6, 142.4, 139.2, 128.7, 127.3, 125.8, 125.0, 123.0, 32.5, 8.5. IR (ATR): ν = 2924, 1661, 1509, 1220, 1160, 895, 746, 728 cm^{-1} . HRMS (ESI) Calculated for $\text{C}_{11}\text{H}_{11}\text{SO} [\text{M}+\text{H}]^+$: 191.0525, found: 191.0522. These date are consistent with those reported in the literature.²⁷

6 NMR spectra

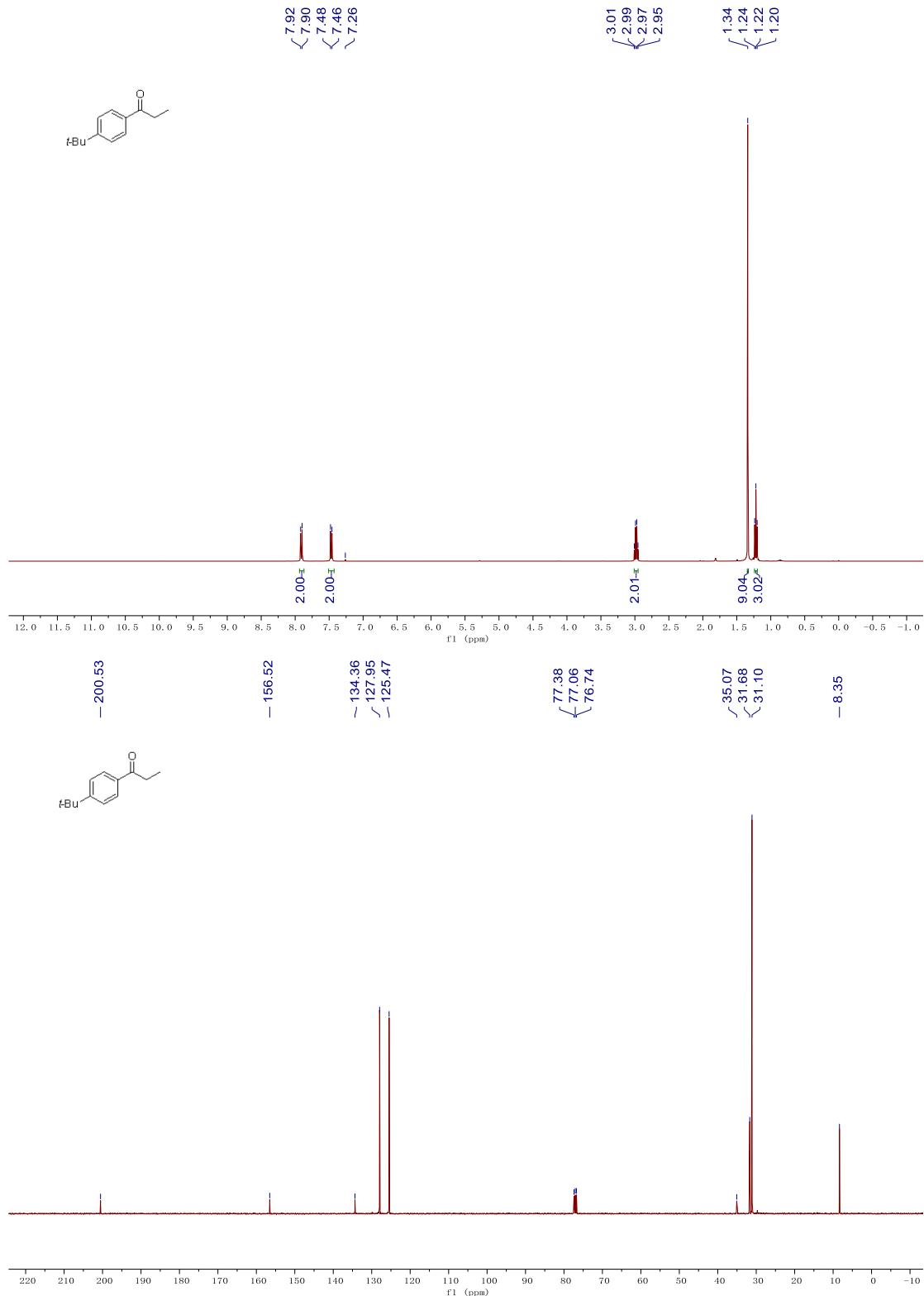
1-(*p*-tolyl)propan-1-one (3)



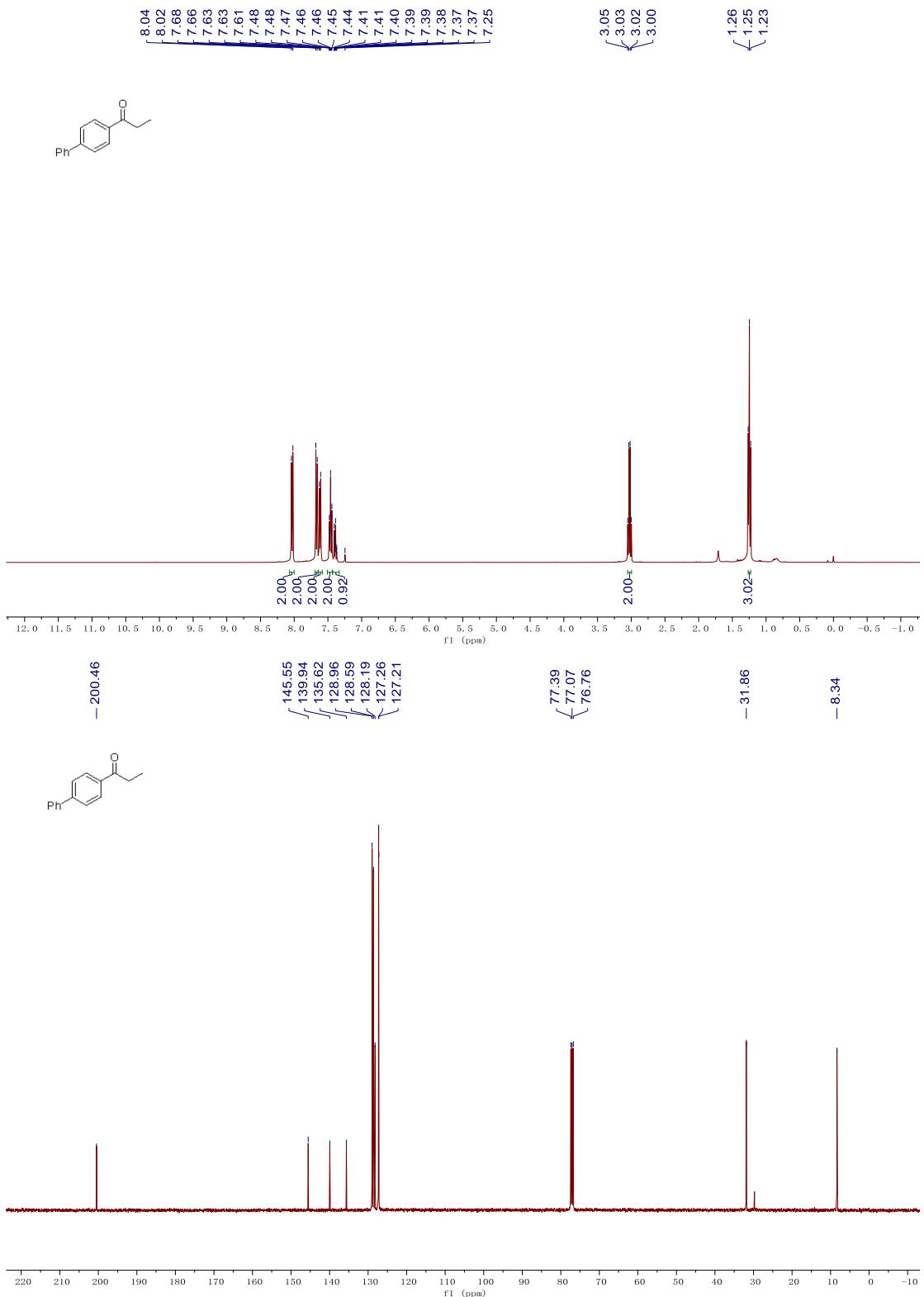
Propiophenone (4)



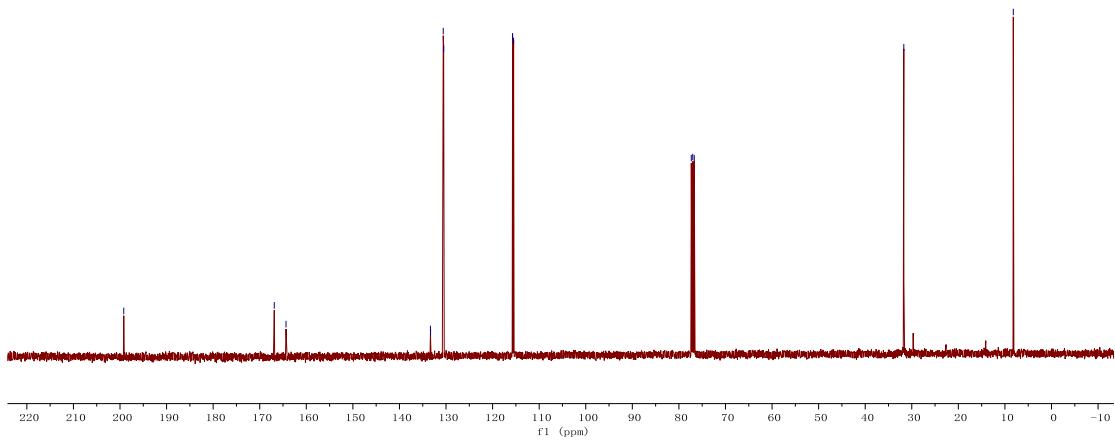
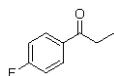
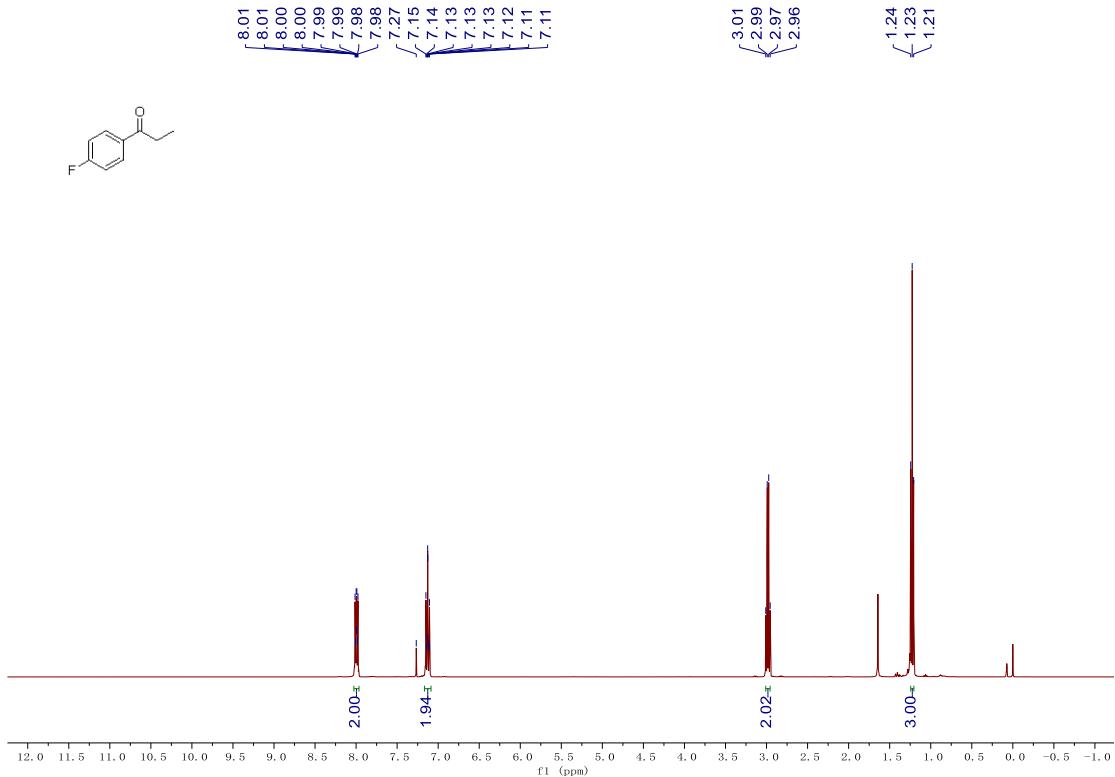
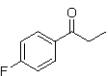
1-(4-(*tert*-butyl)phenyl)propan-1-one (5)

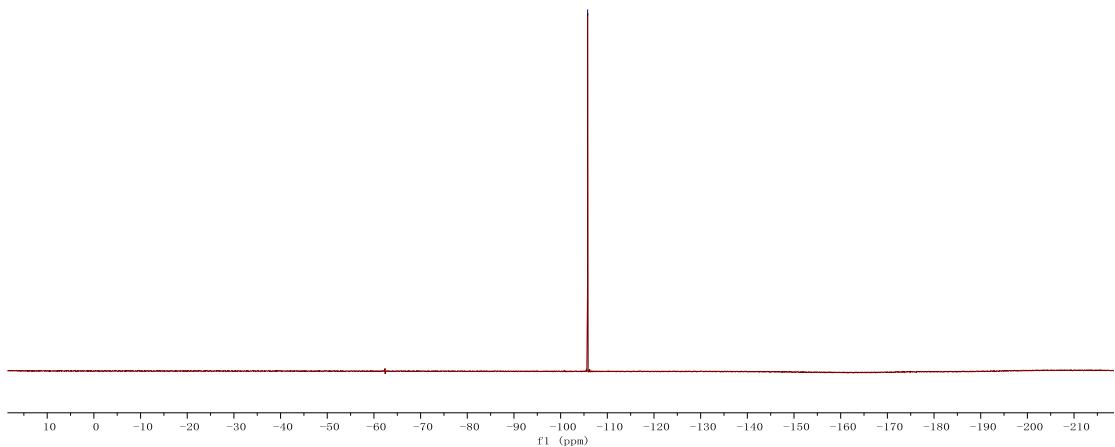
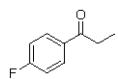


1-([1,1'-biphenyl]-4-yl)propan-1-one (6)

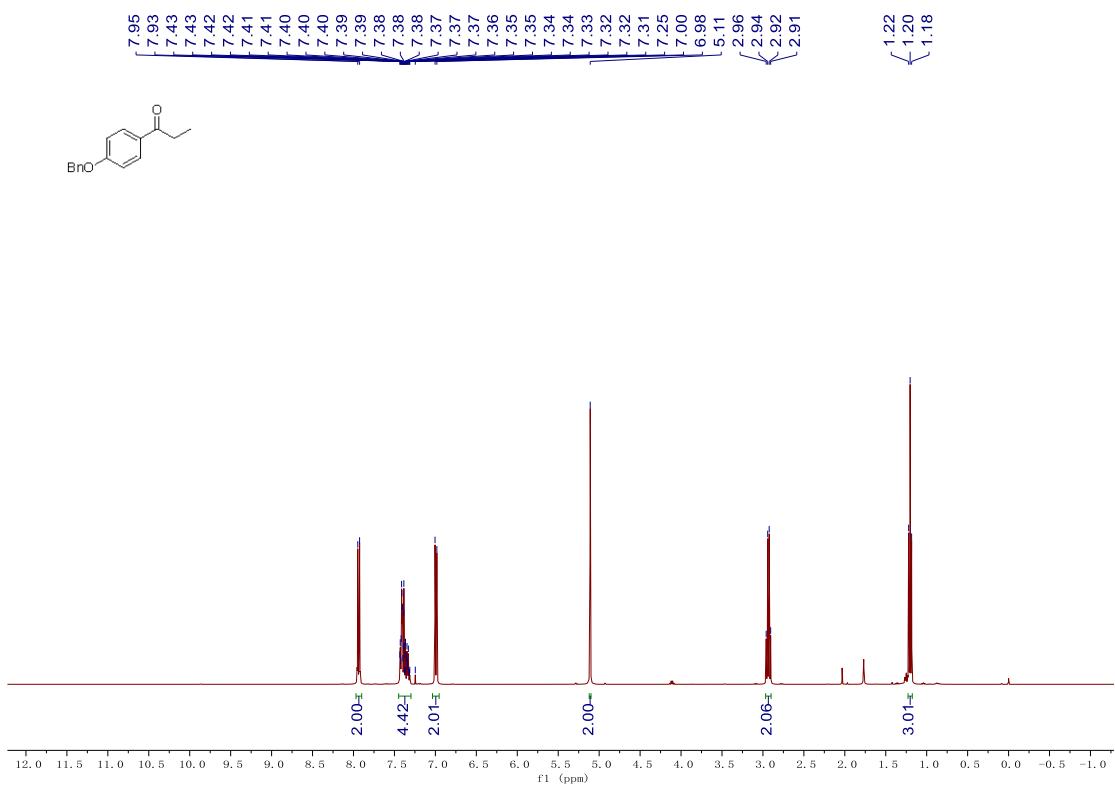


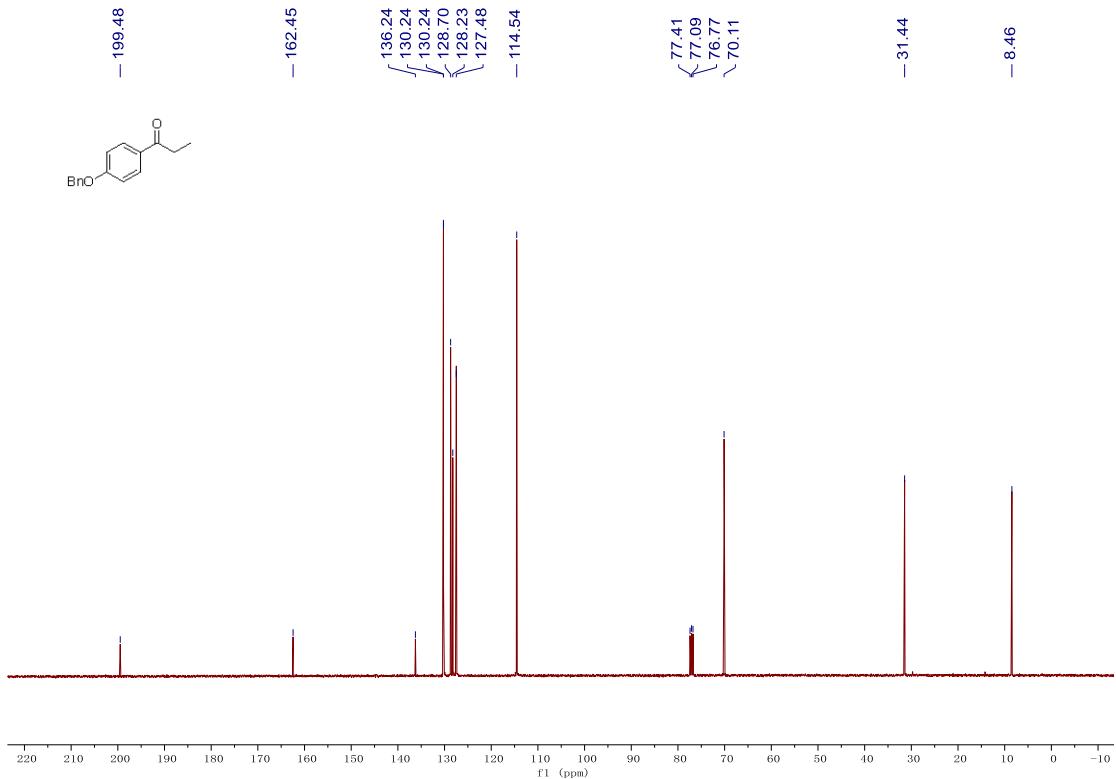
1-(4-fluorophenyl)propan-1-one (7)



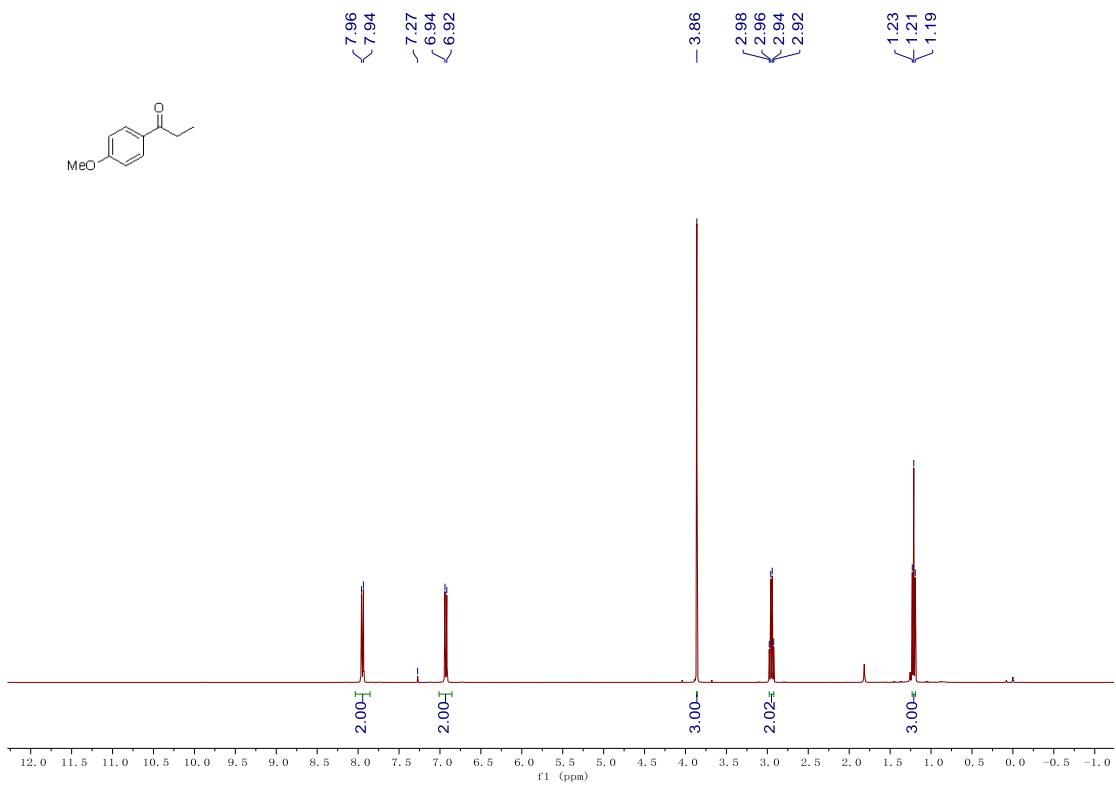


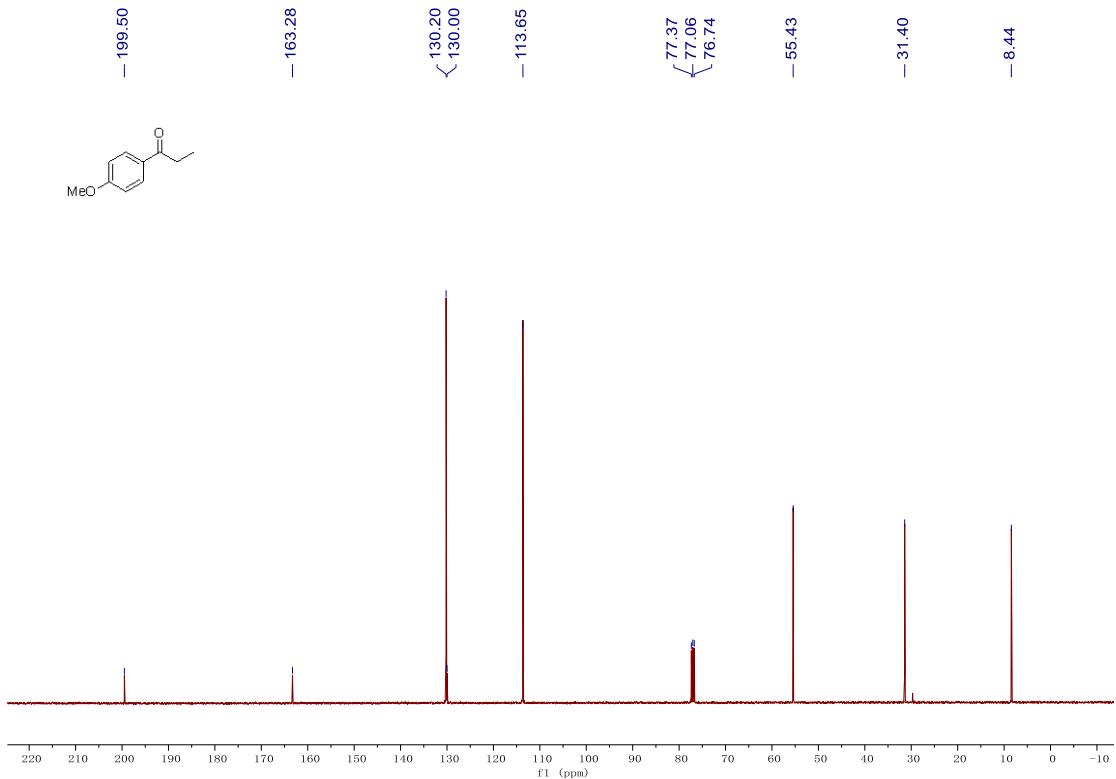
1-(4-(benzyloxy)phenyl)propan-1-one (8)



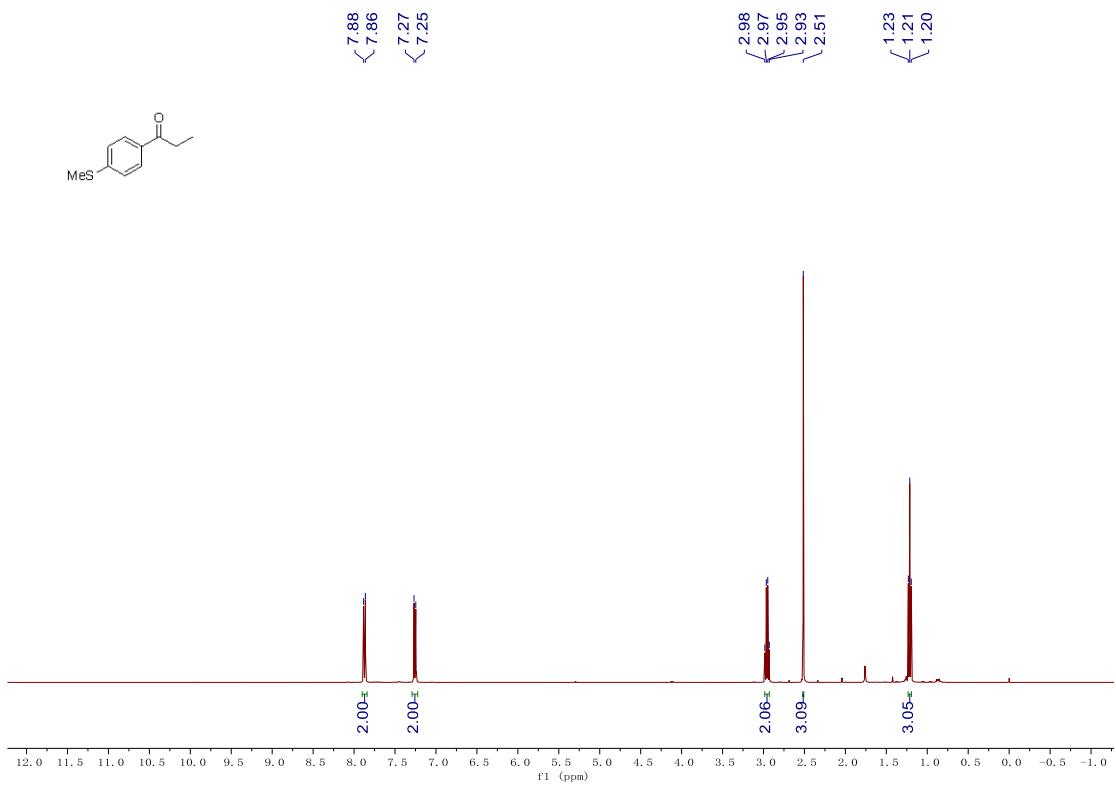


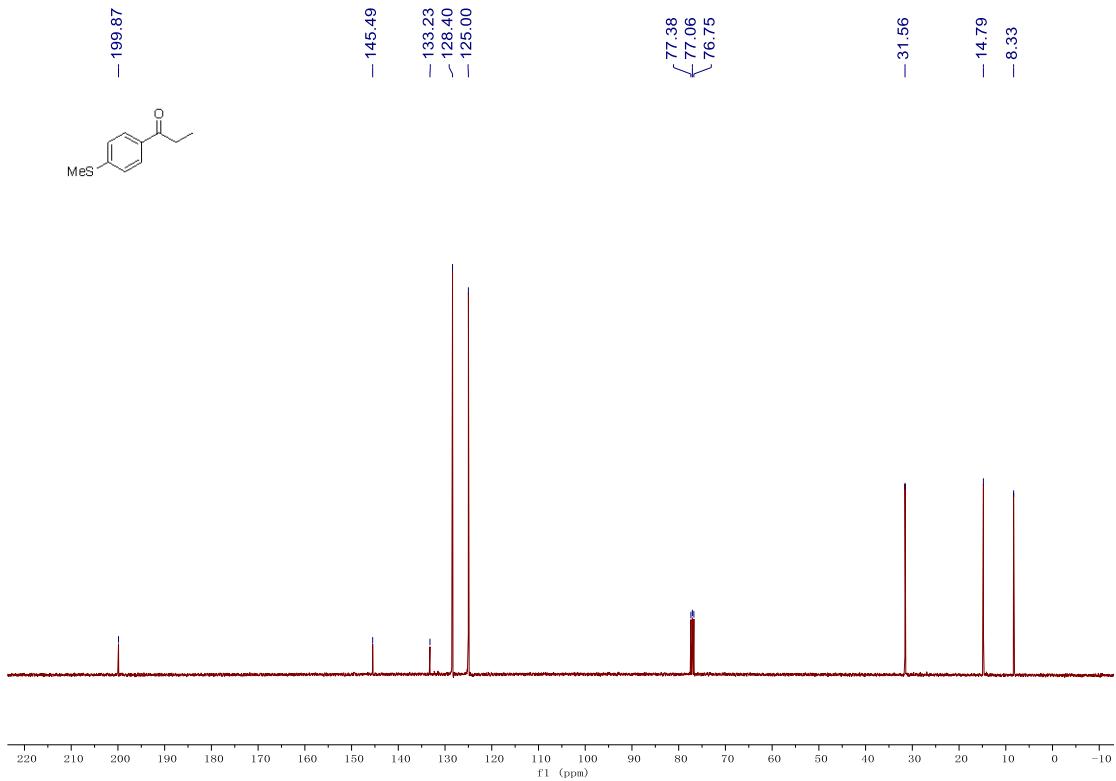
1-(4-methoxyphenyl)propan-1-one (9)



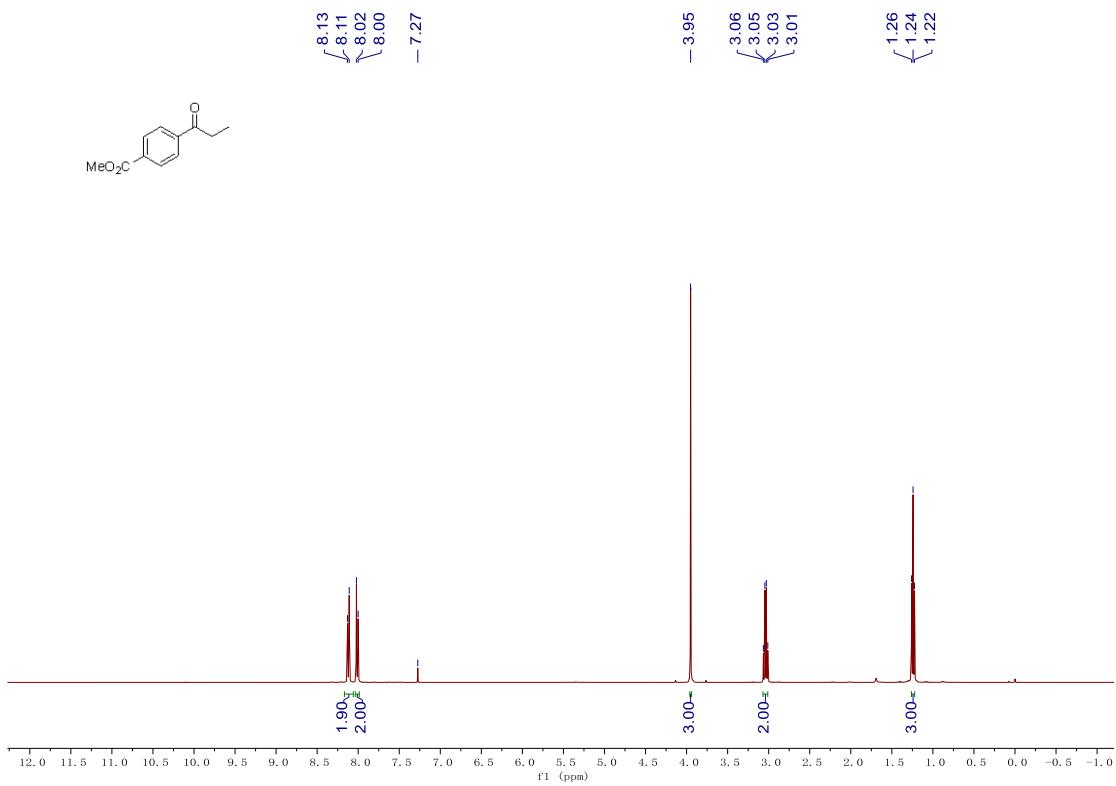


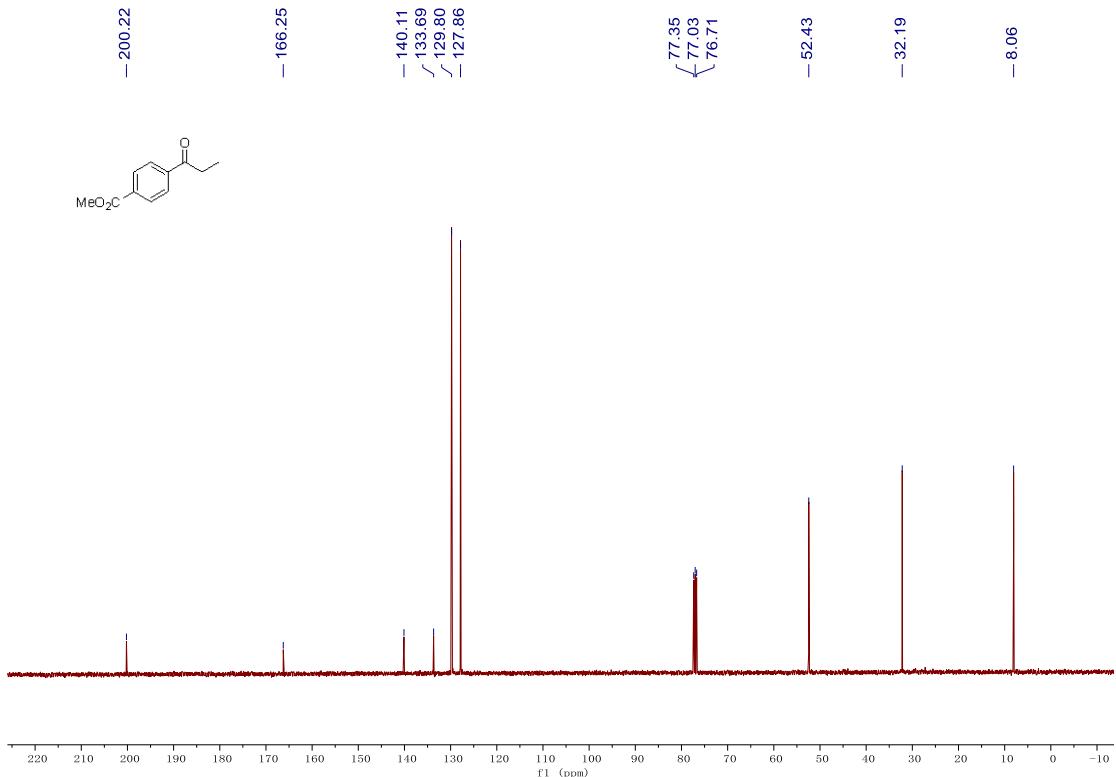
1-(4-(methylthio)phenyl)propan-1-one (10)



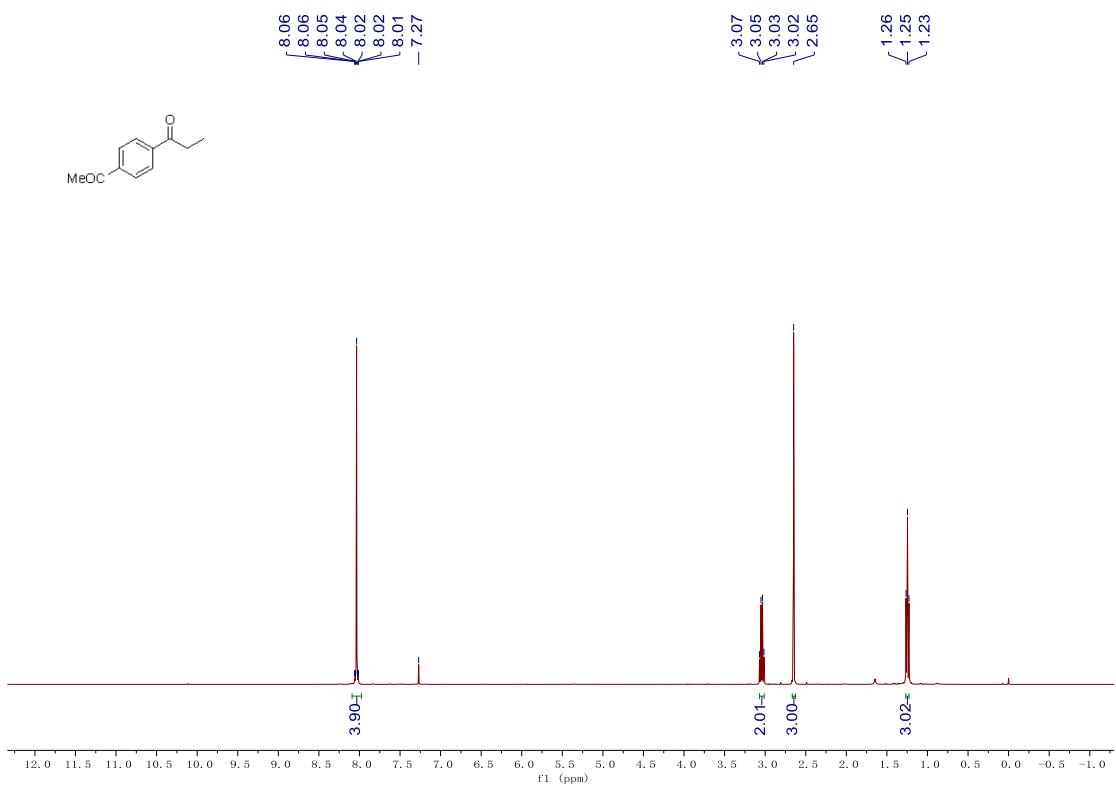


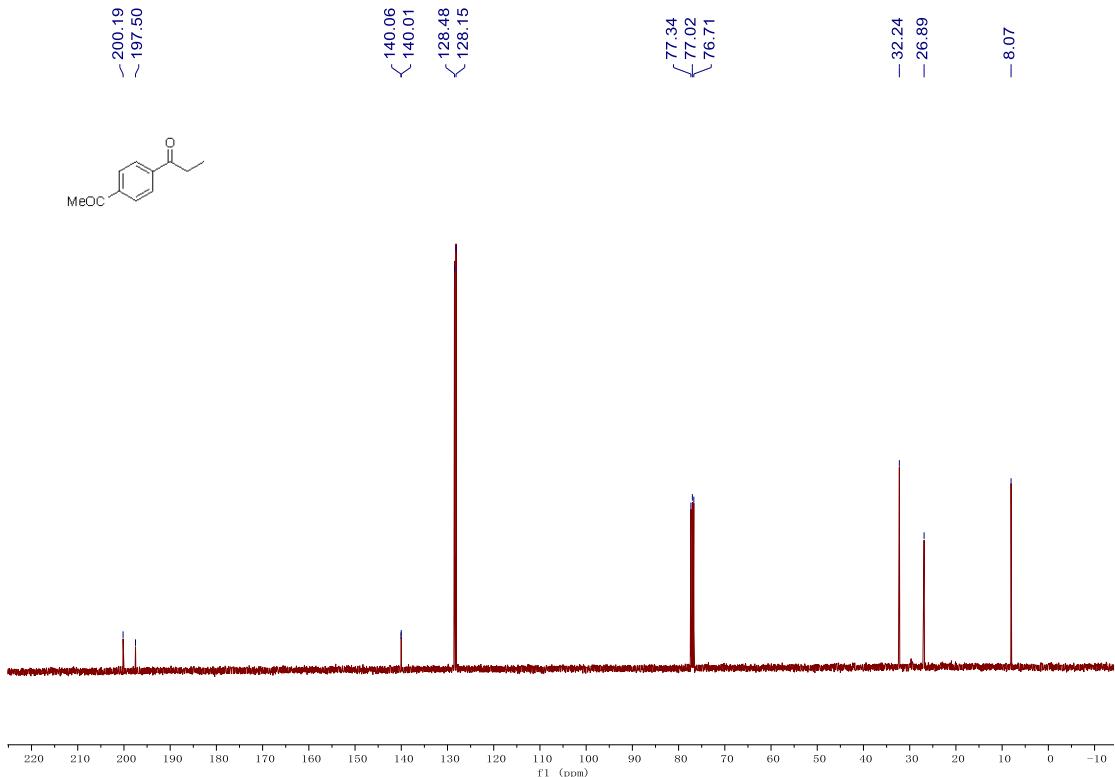
methyl 4-propionylbenzoate (11)



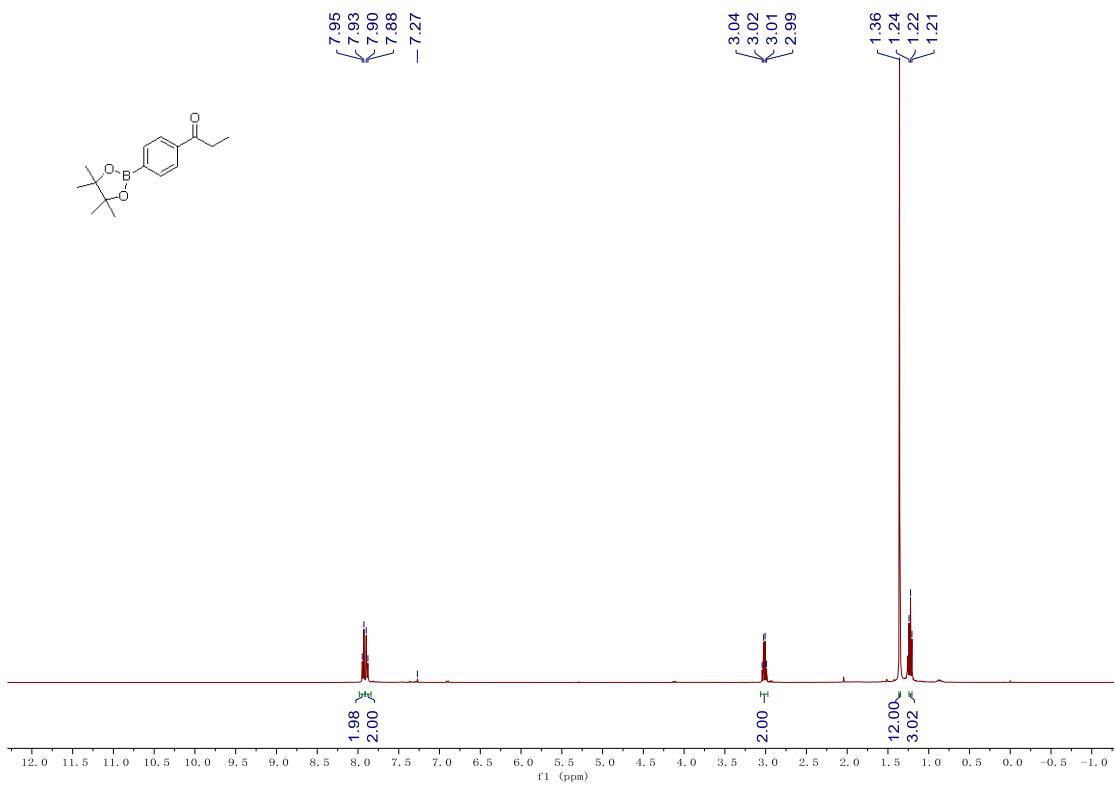


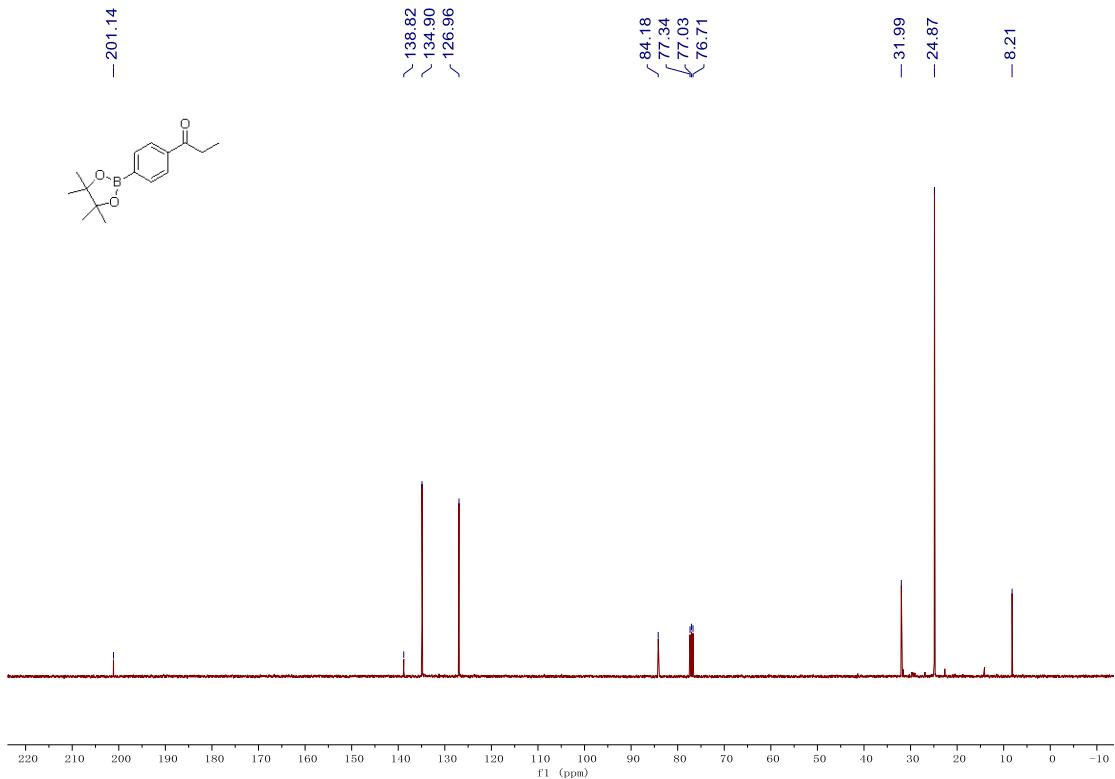
1-(4-acetylphenyl)propan-1-one (12)



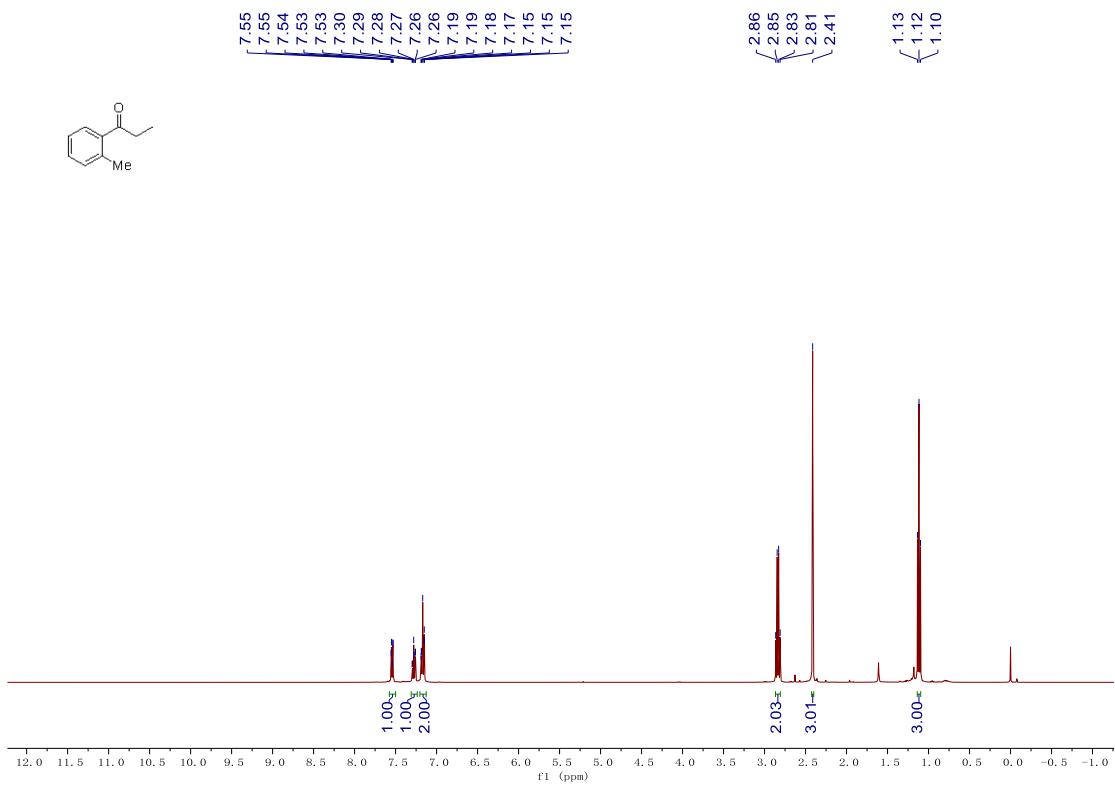


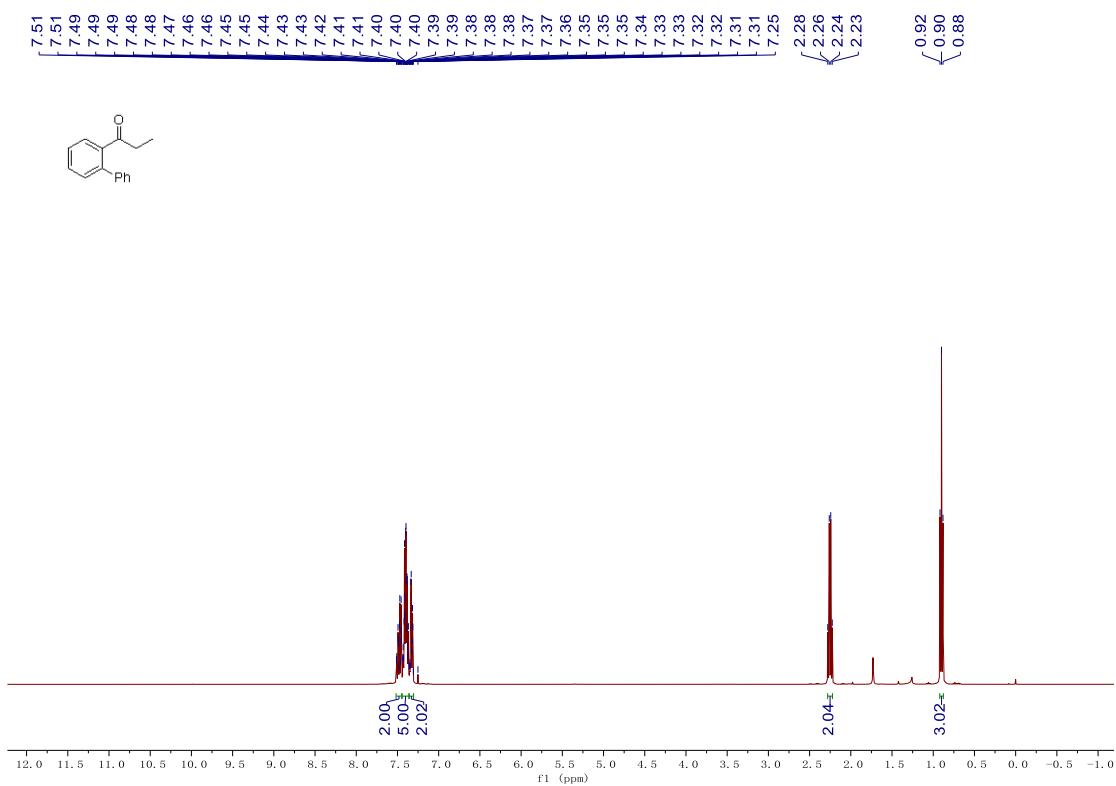
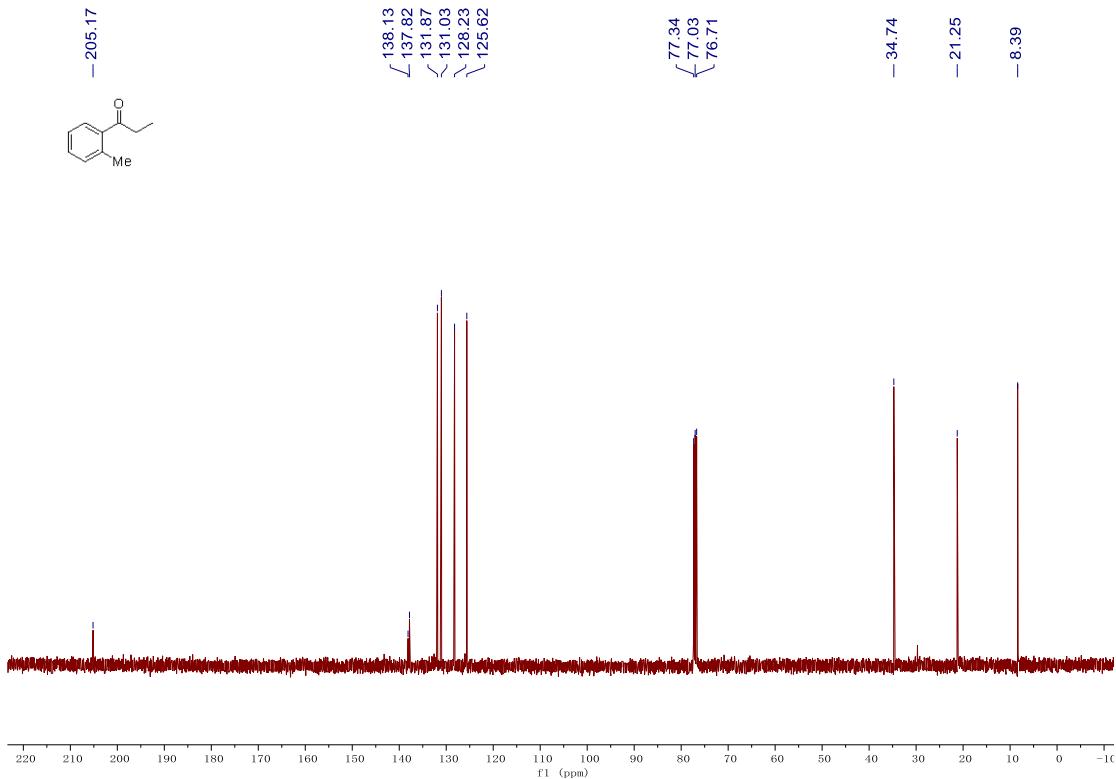
1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)propan-1-one (13)

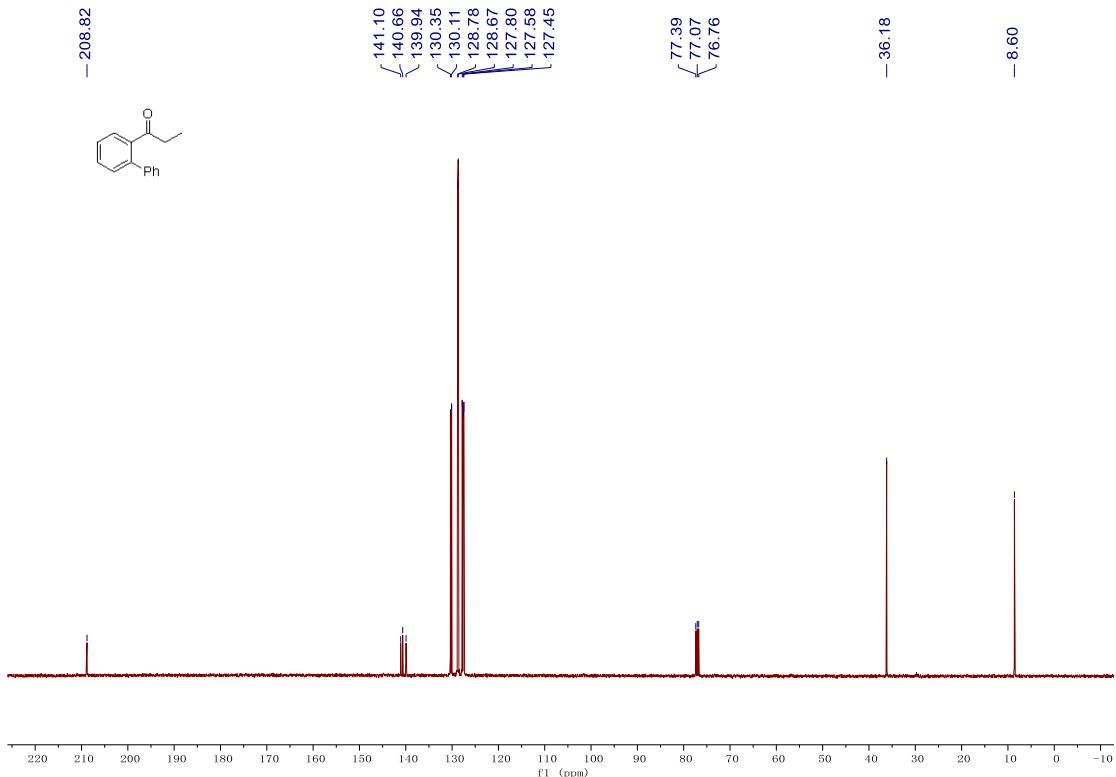




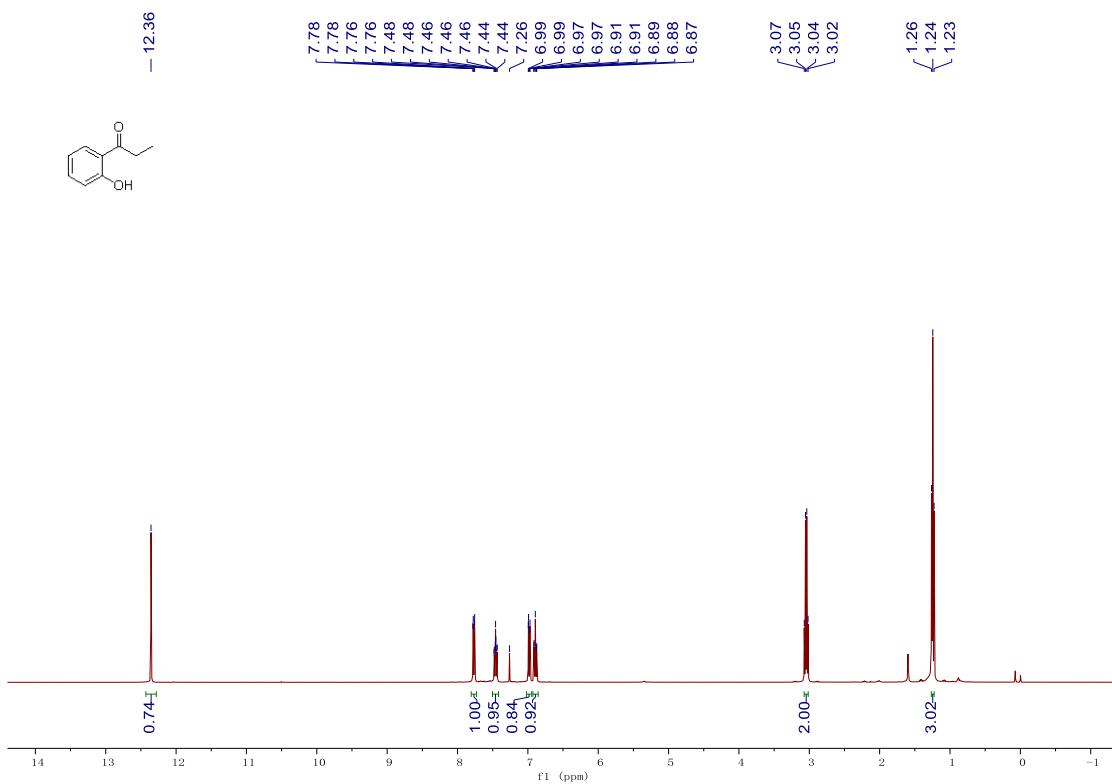
1-(*o*-tolyl)propan-1-one (14)

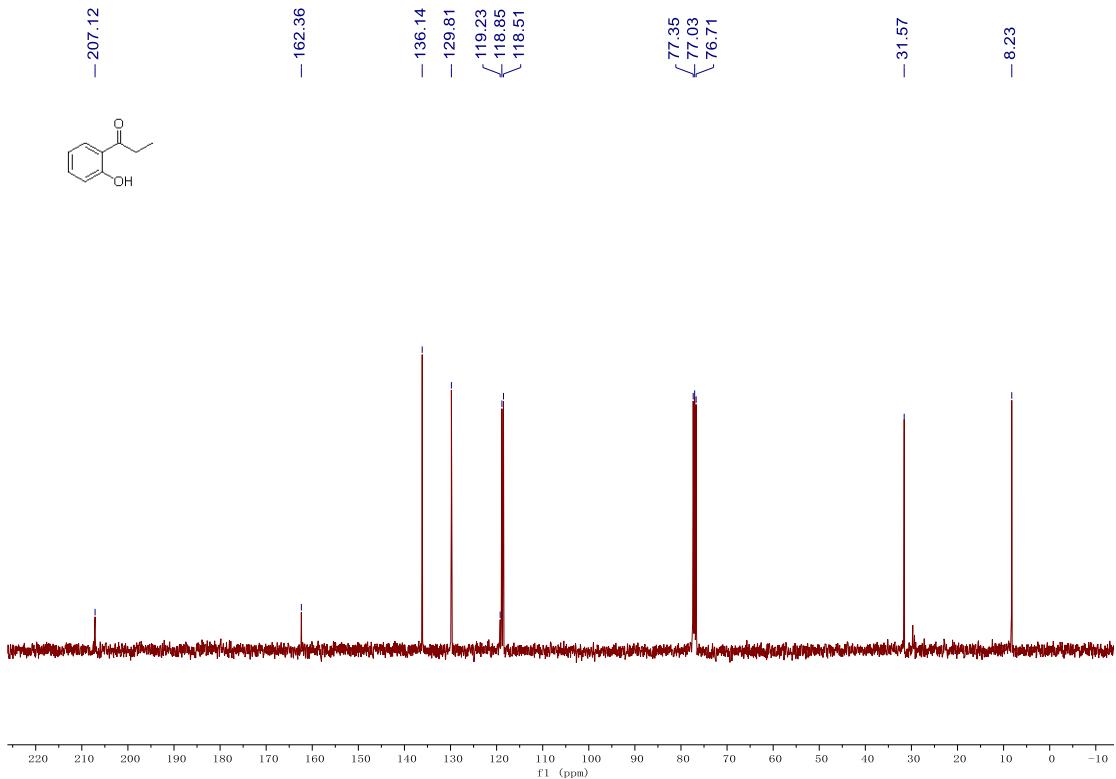




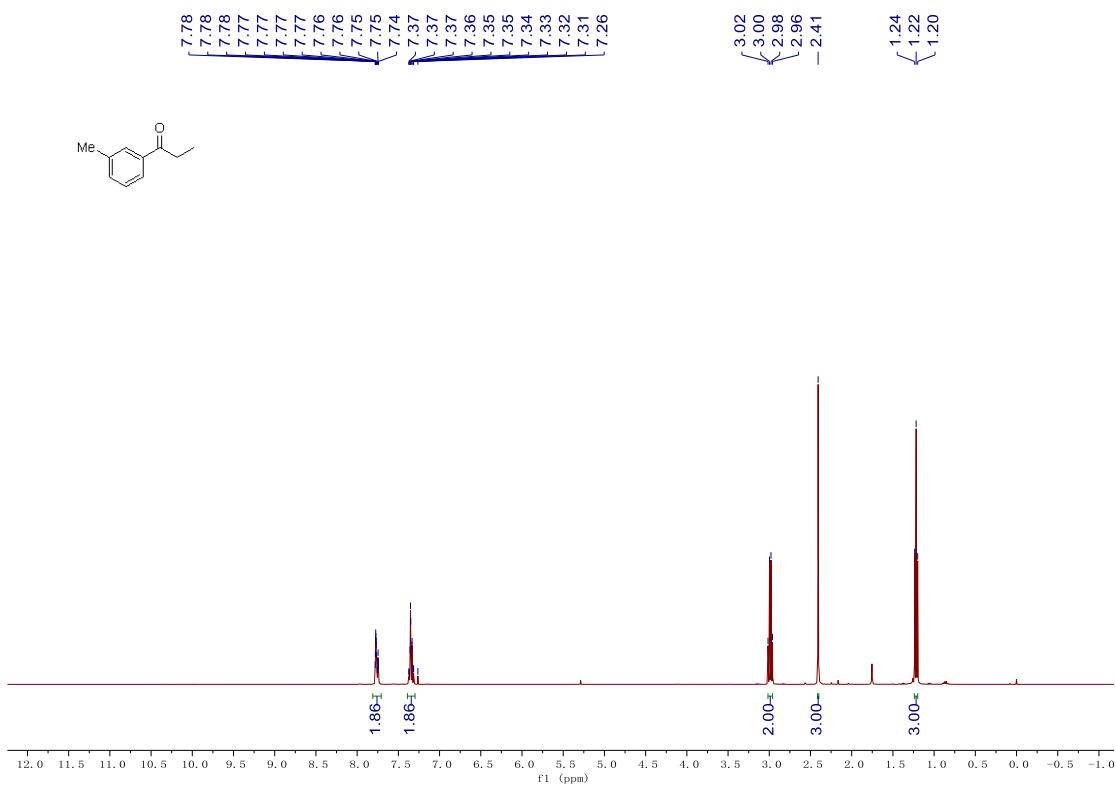


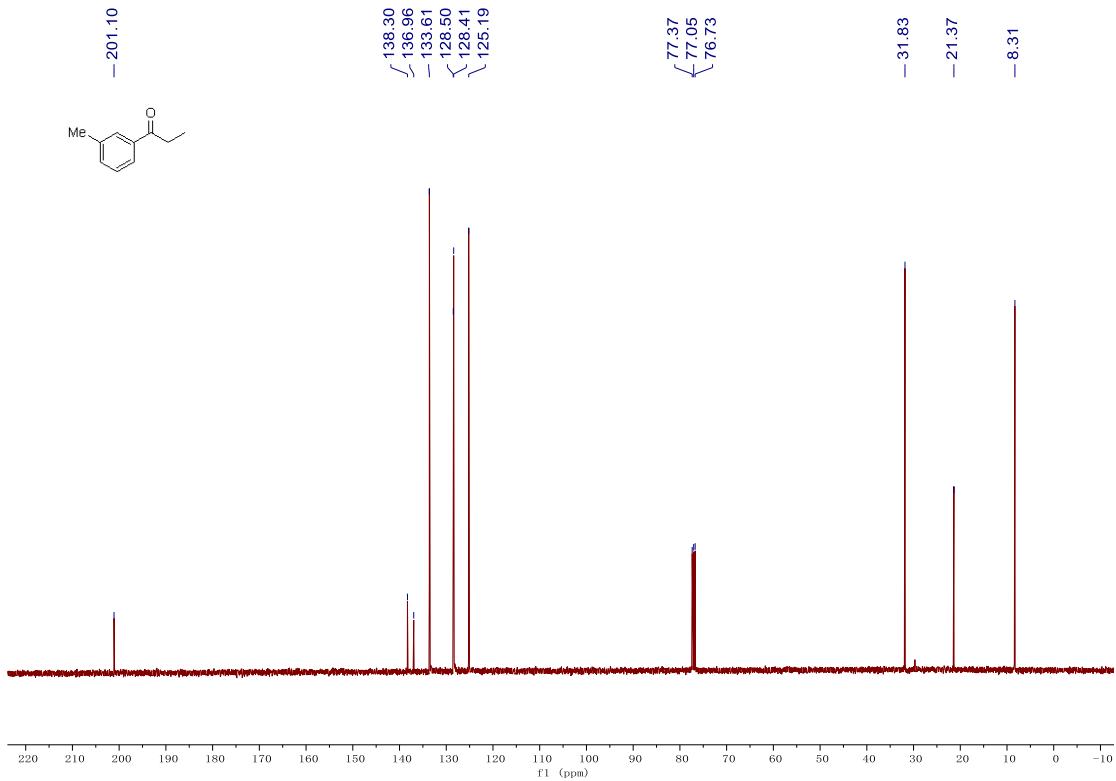
1-(2-hydroxyphenyl)propan-1-one (16)



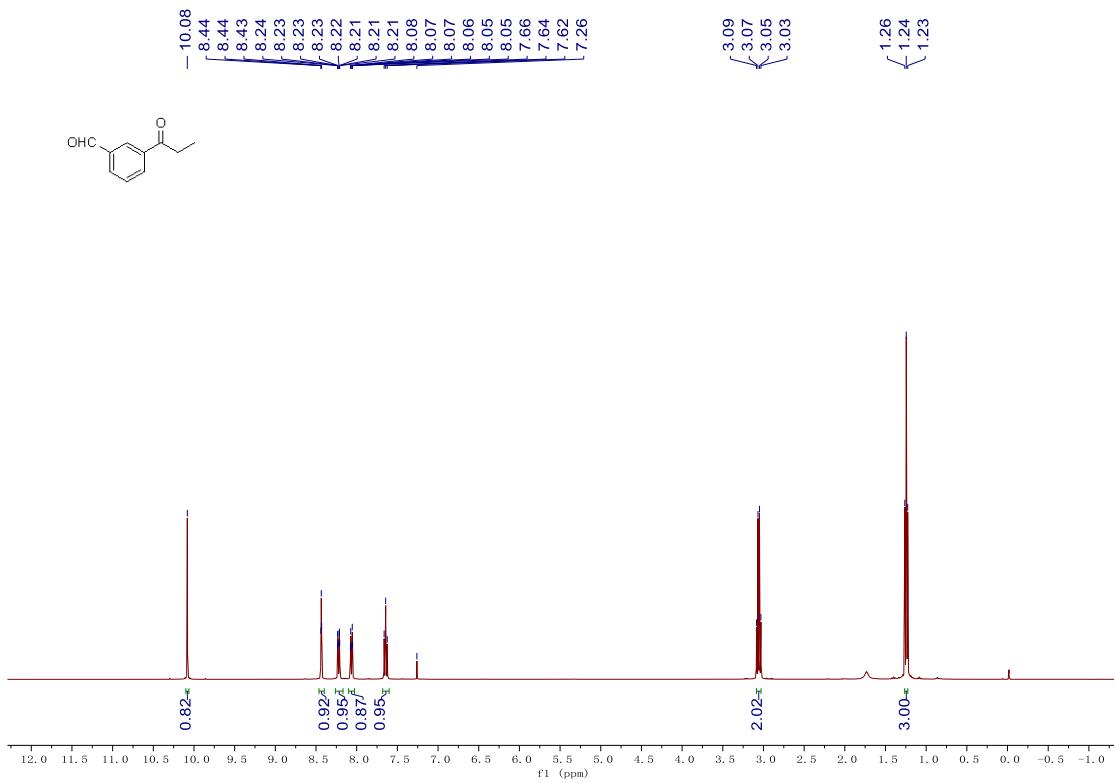


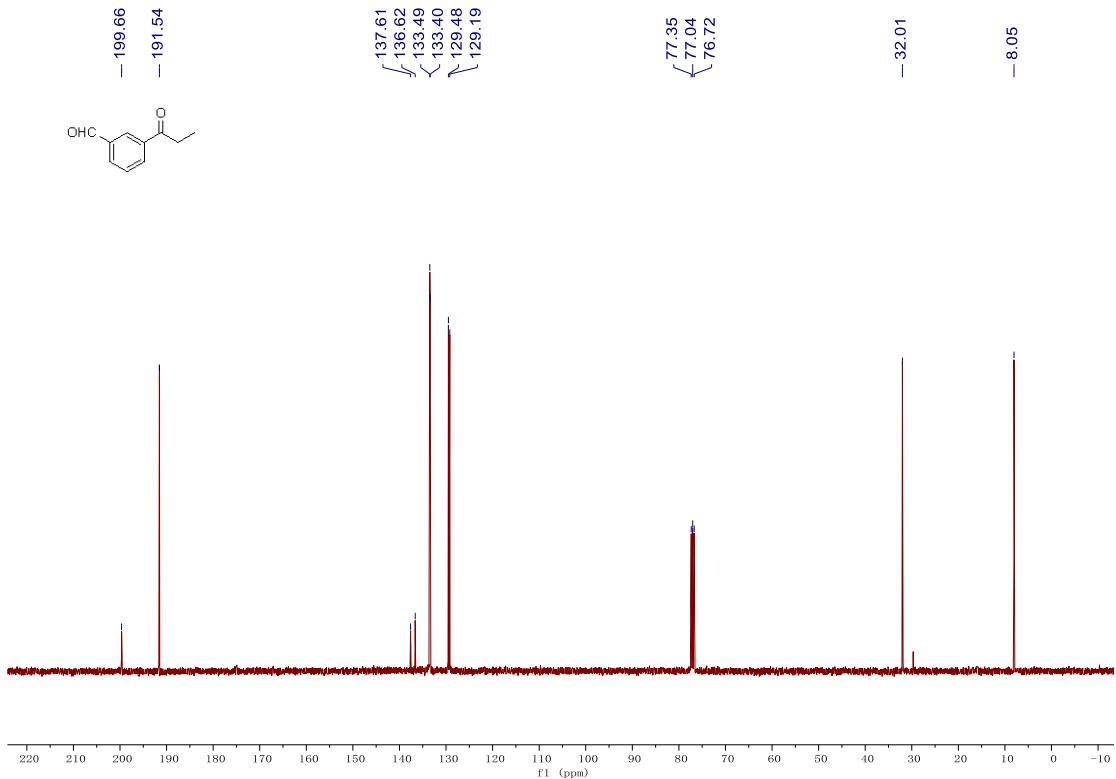
1-(*m*-tolyl)propan-1-one (17)



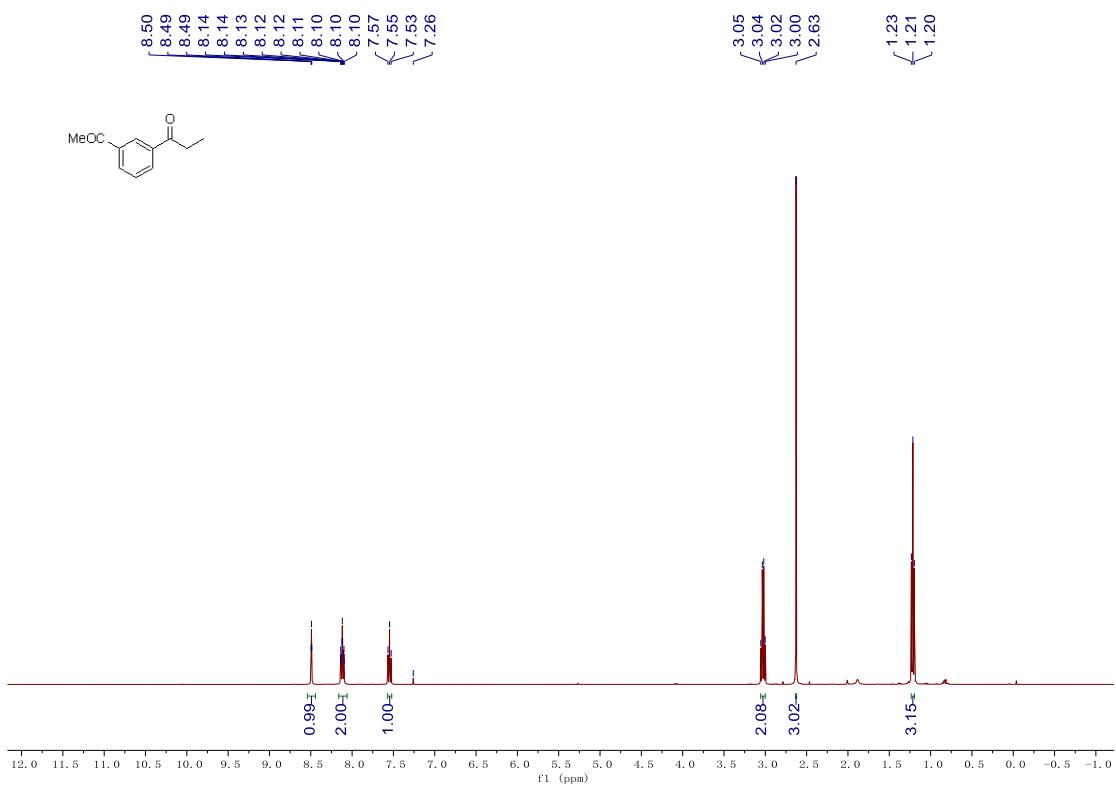


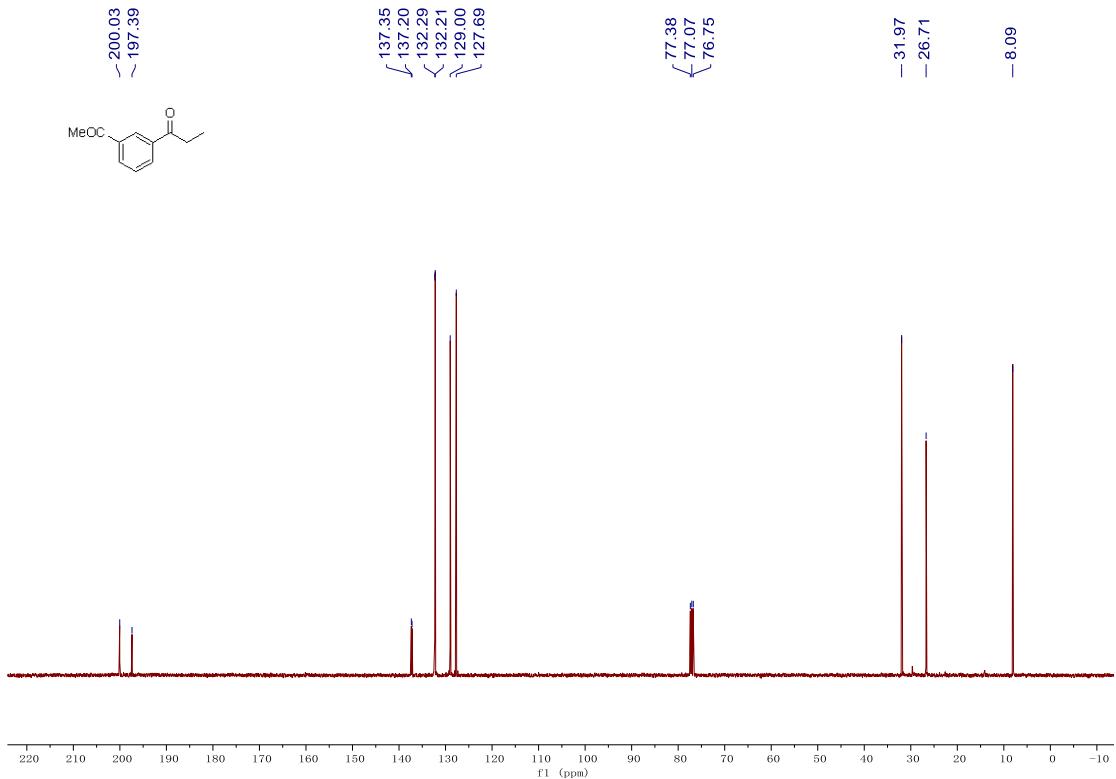
3-propionylbenzaldehyde (18)



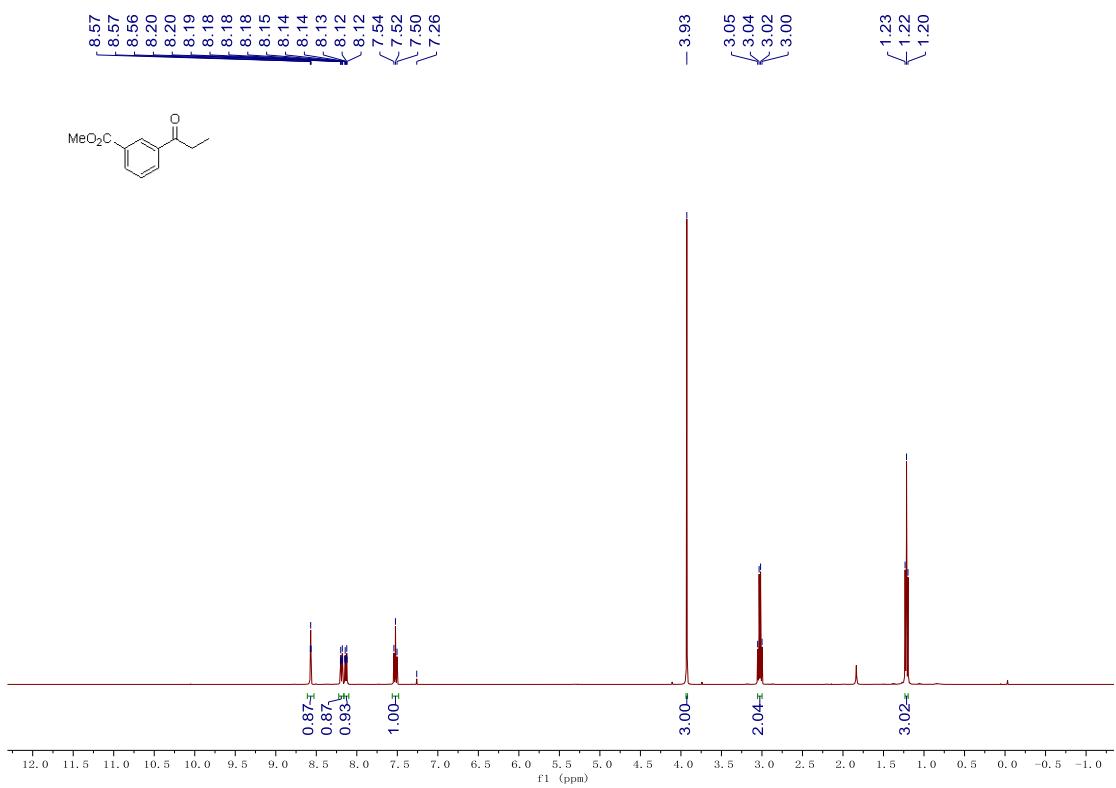


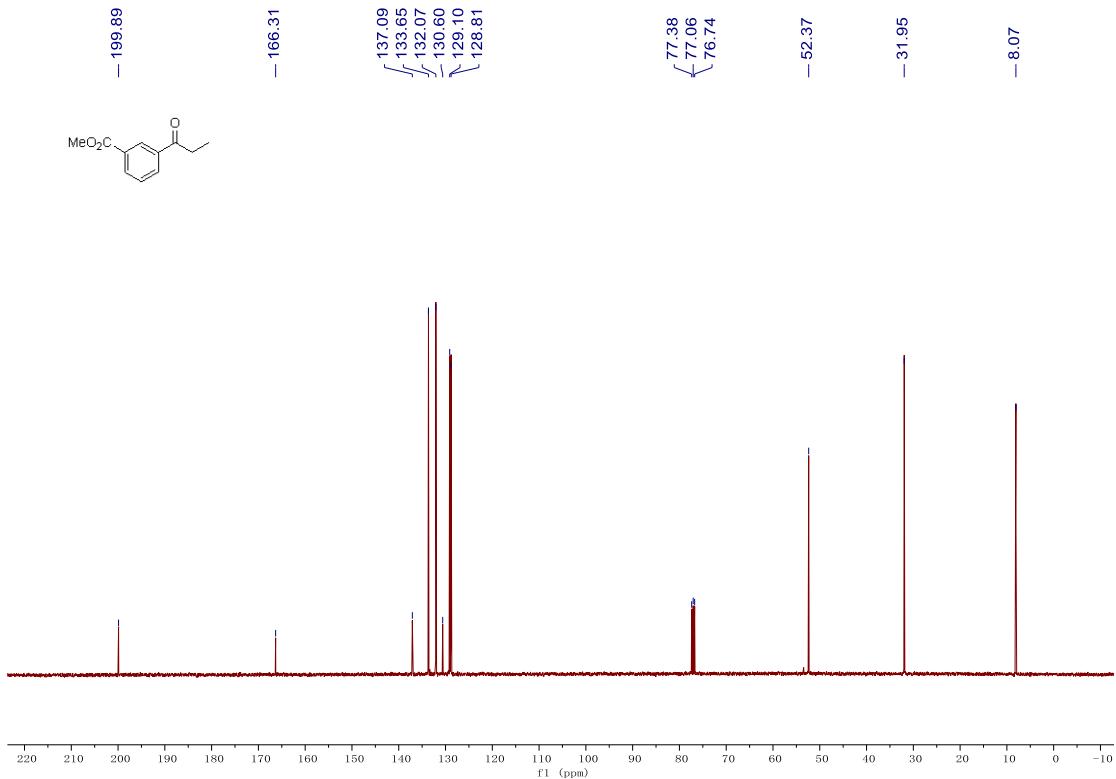
1-(3-acetylphenyl)propan-1-one (19)



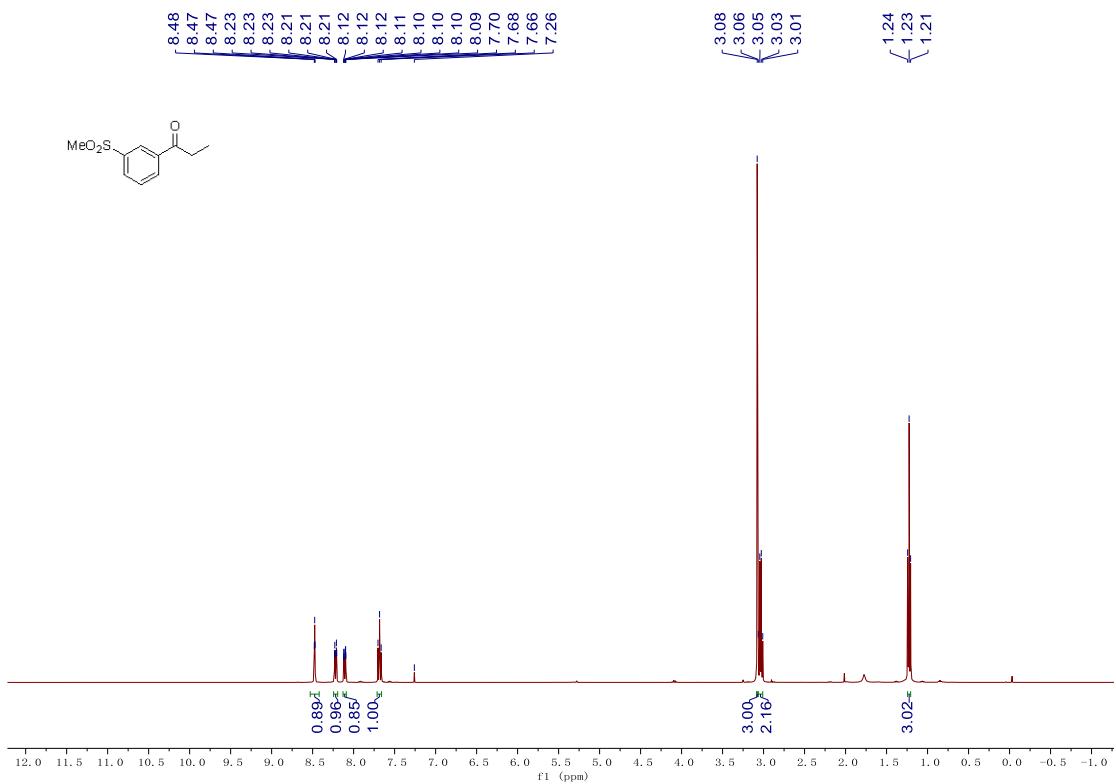


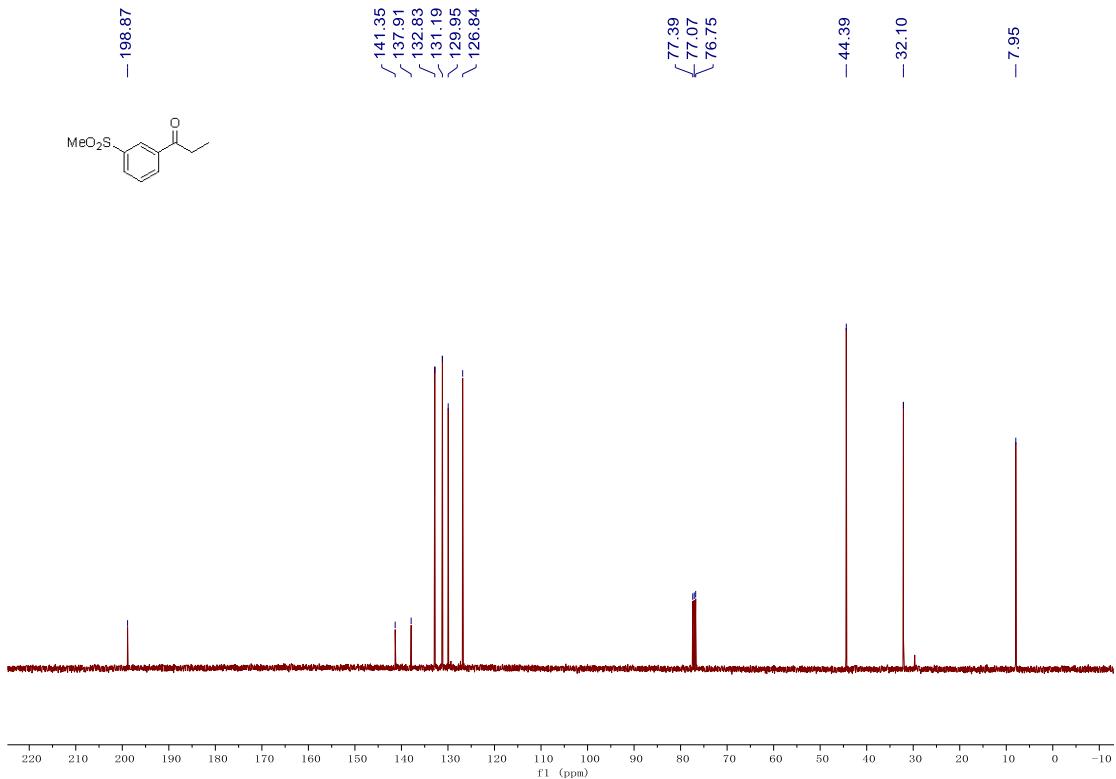
methyl 3-propionylbenzoate (20)



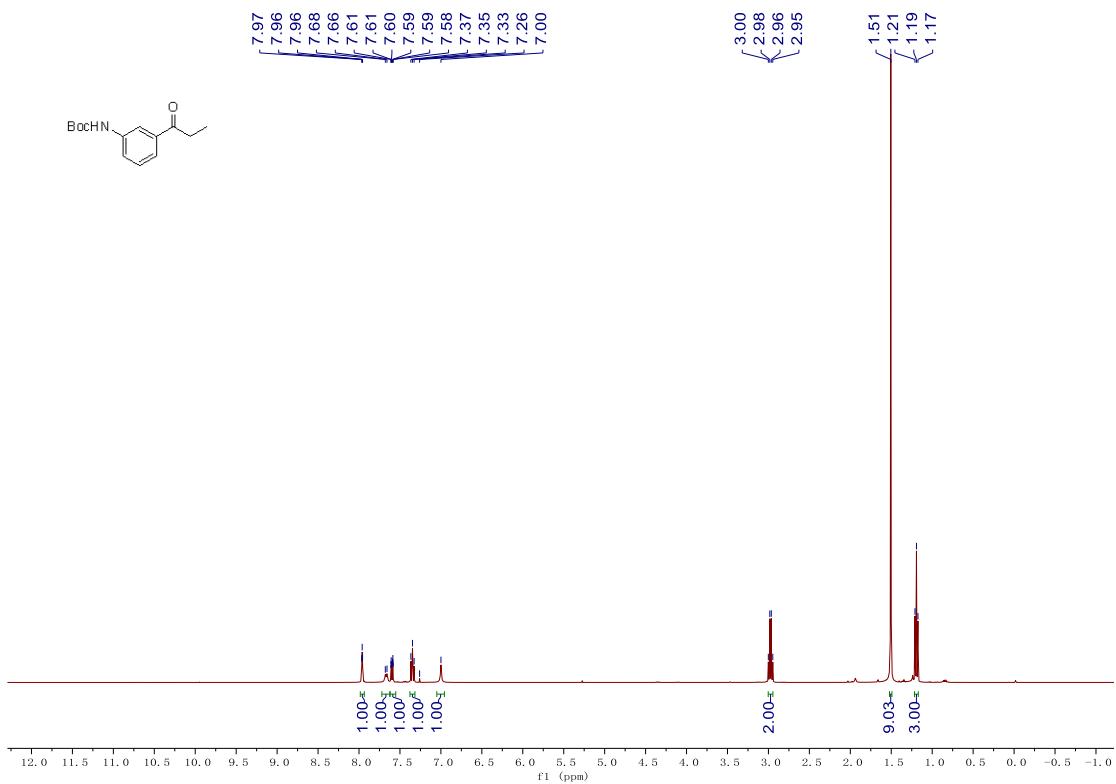


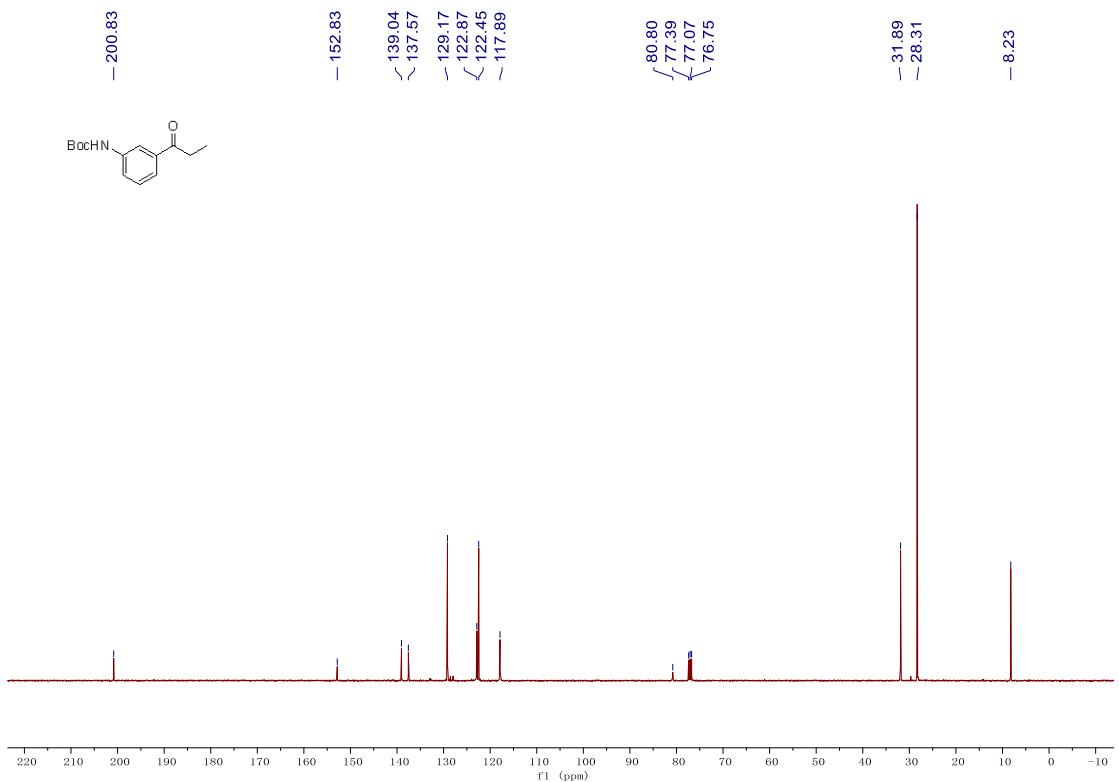
1-(3-(methylsulfonyl)phenyl)propan-1-one (21)



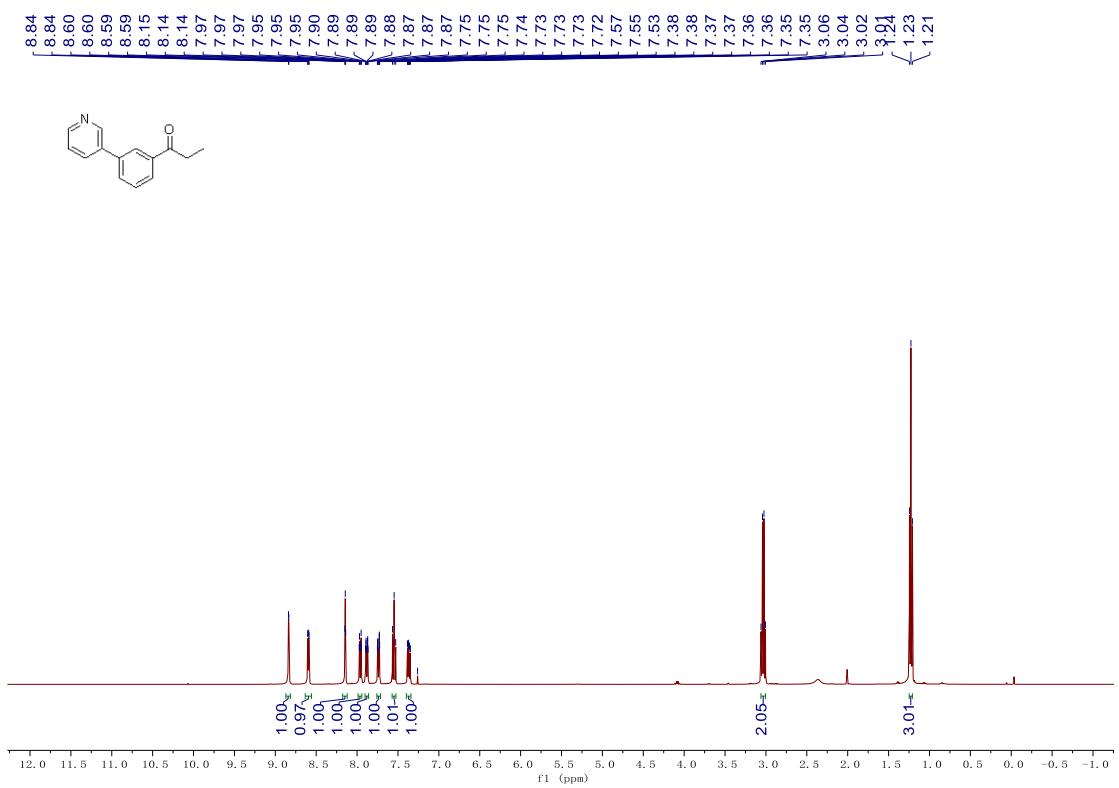


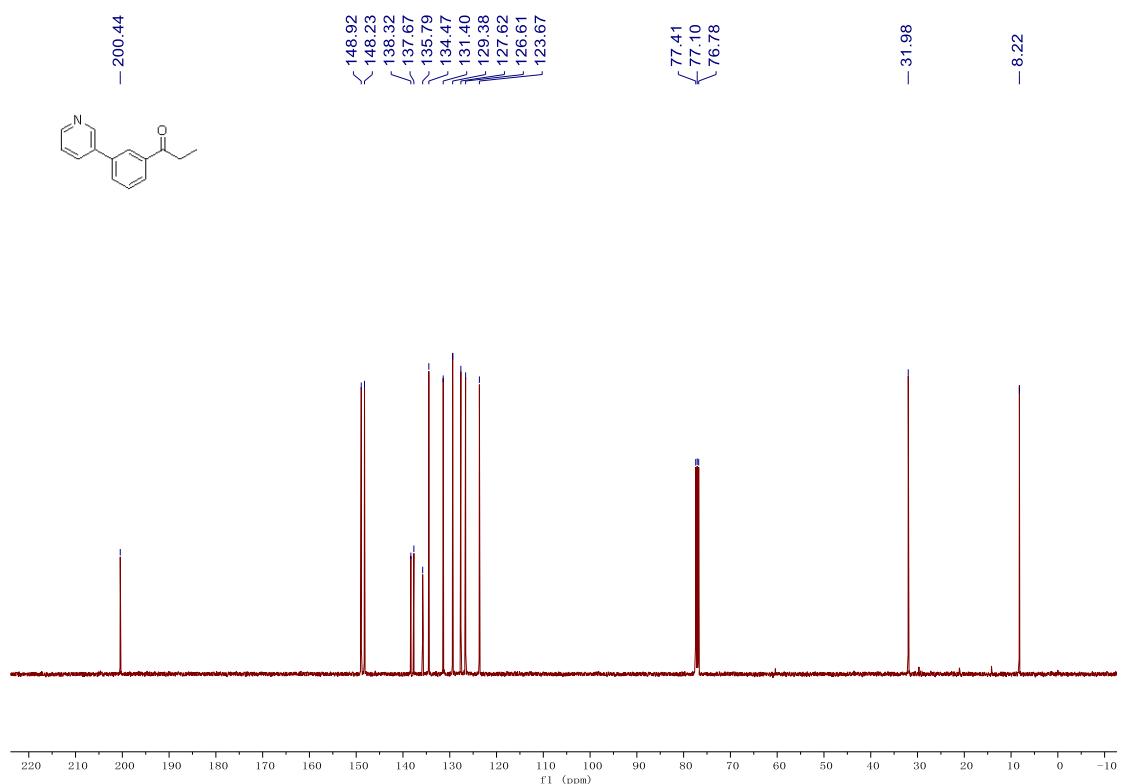
tert-butyl (3-propionylphenyl)carbamate (22)



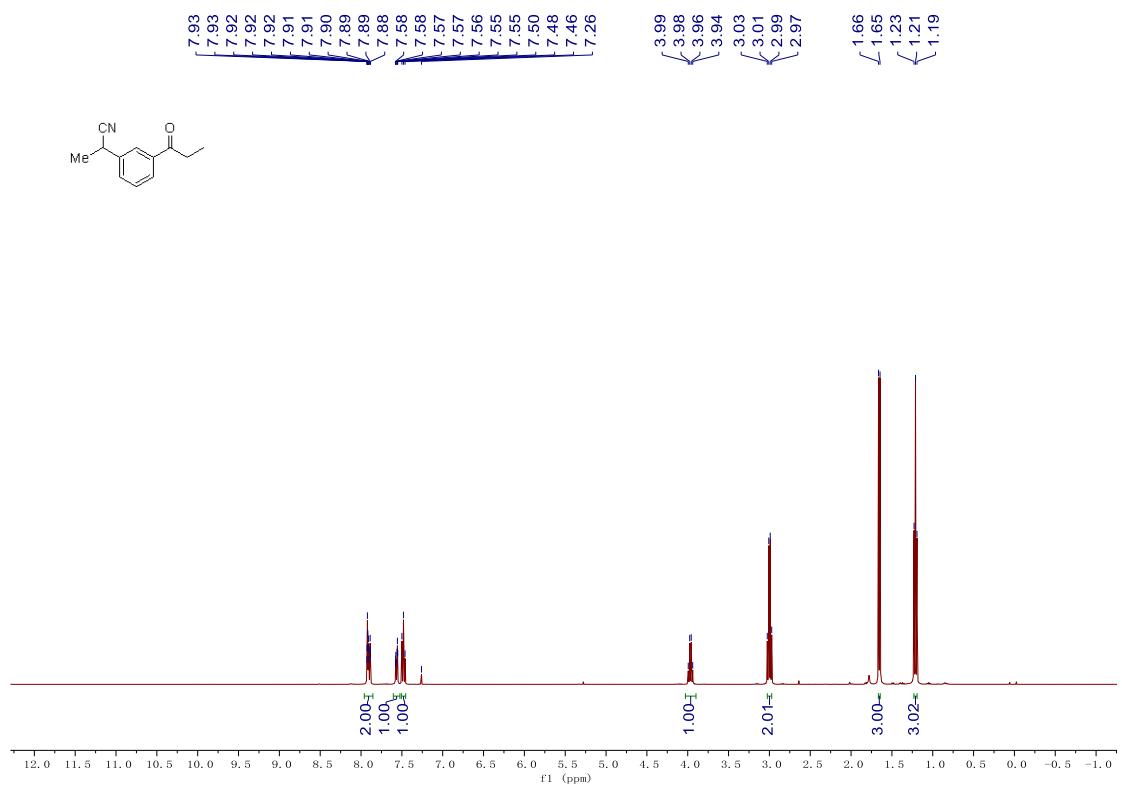


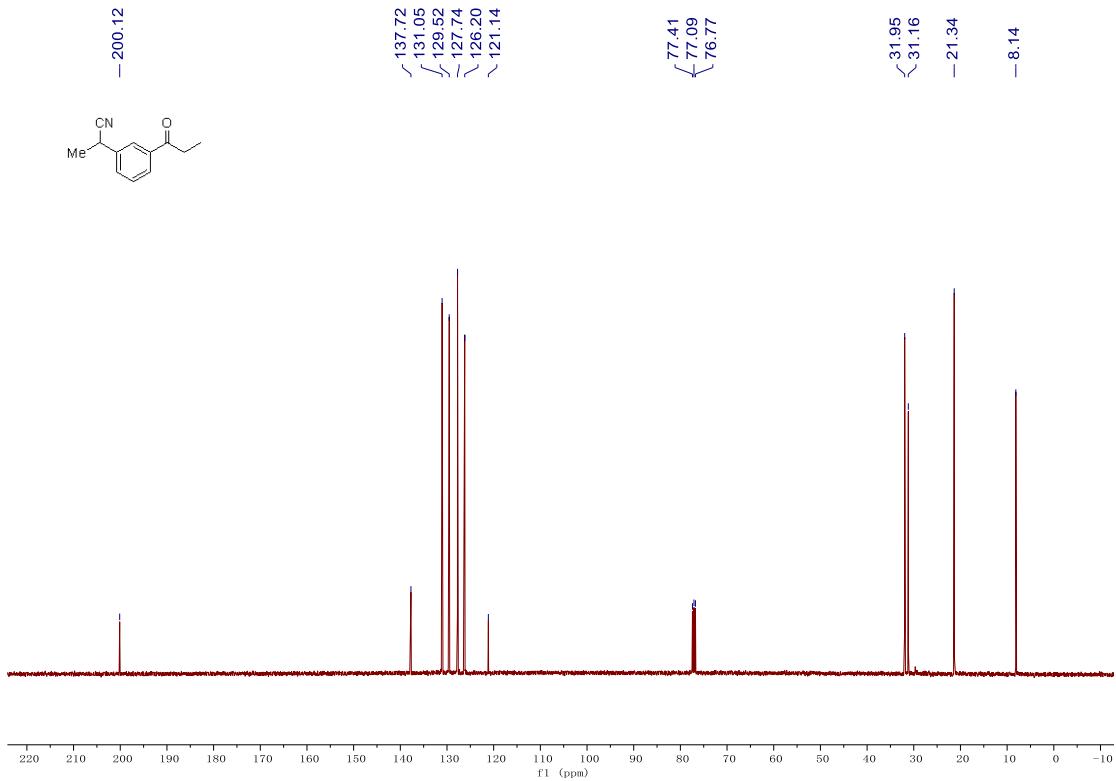
1-(3-(pyridin-3-yl)phenyl)propan-1-one (23)



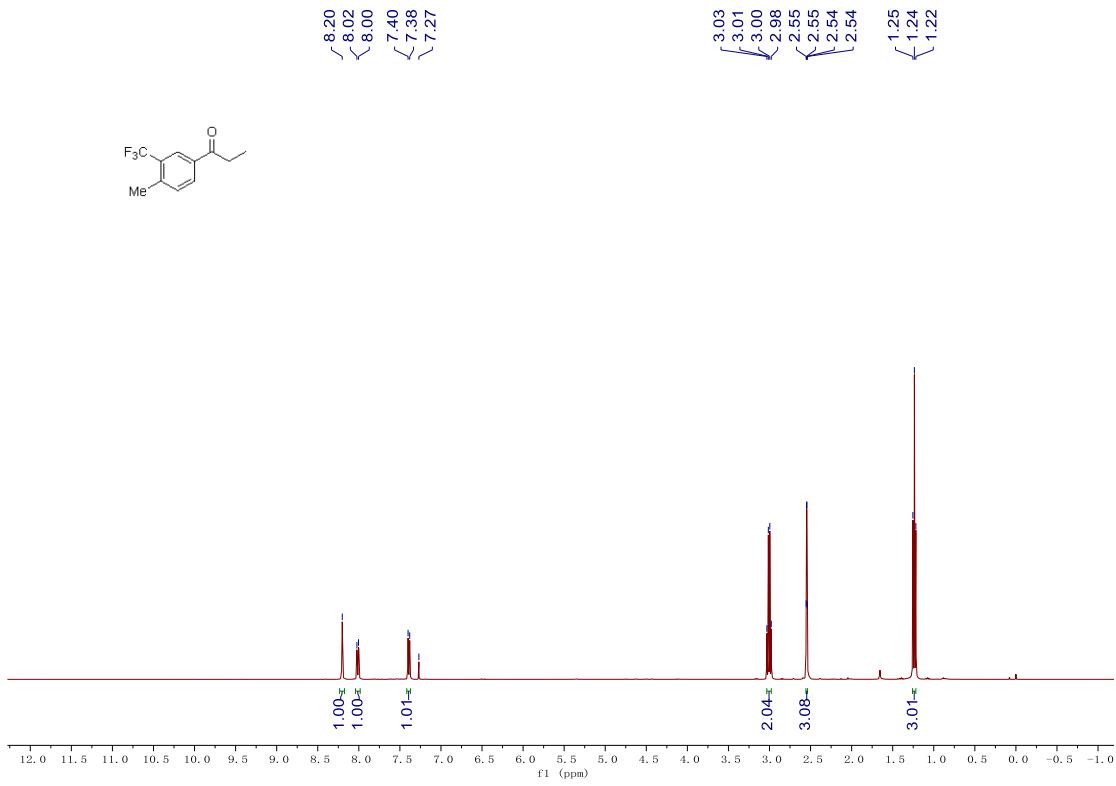


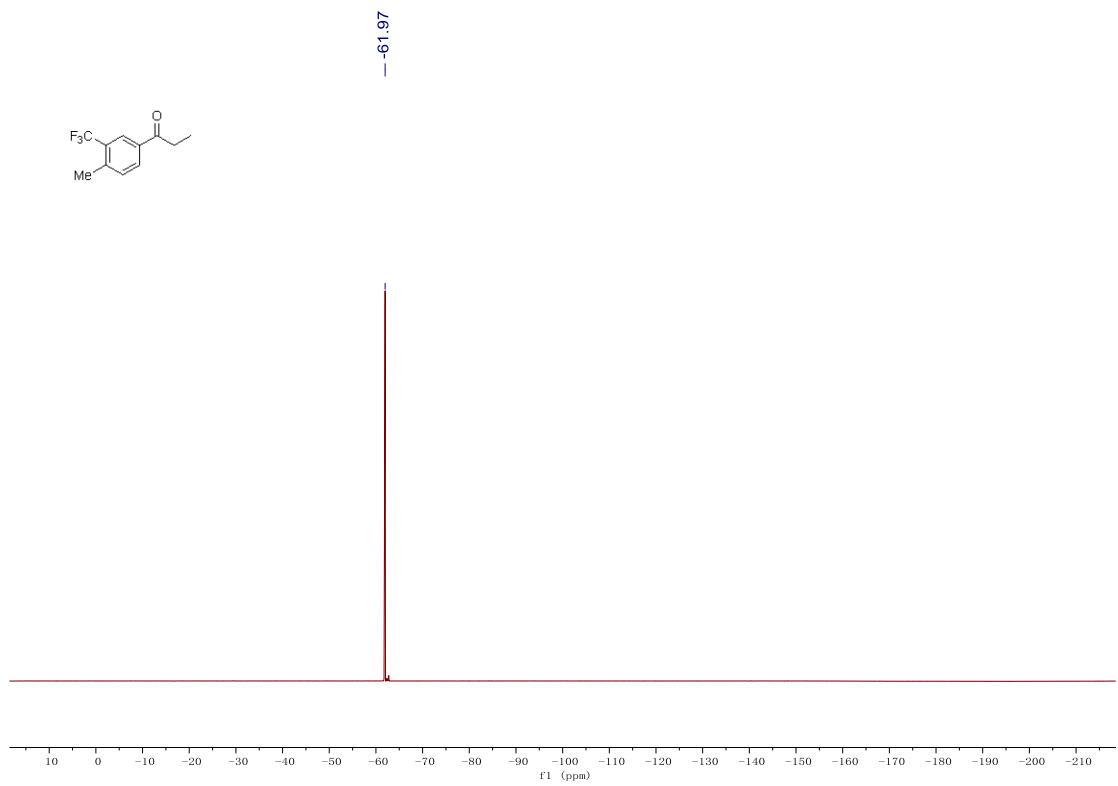
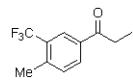
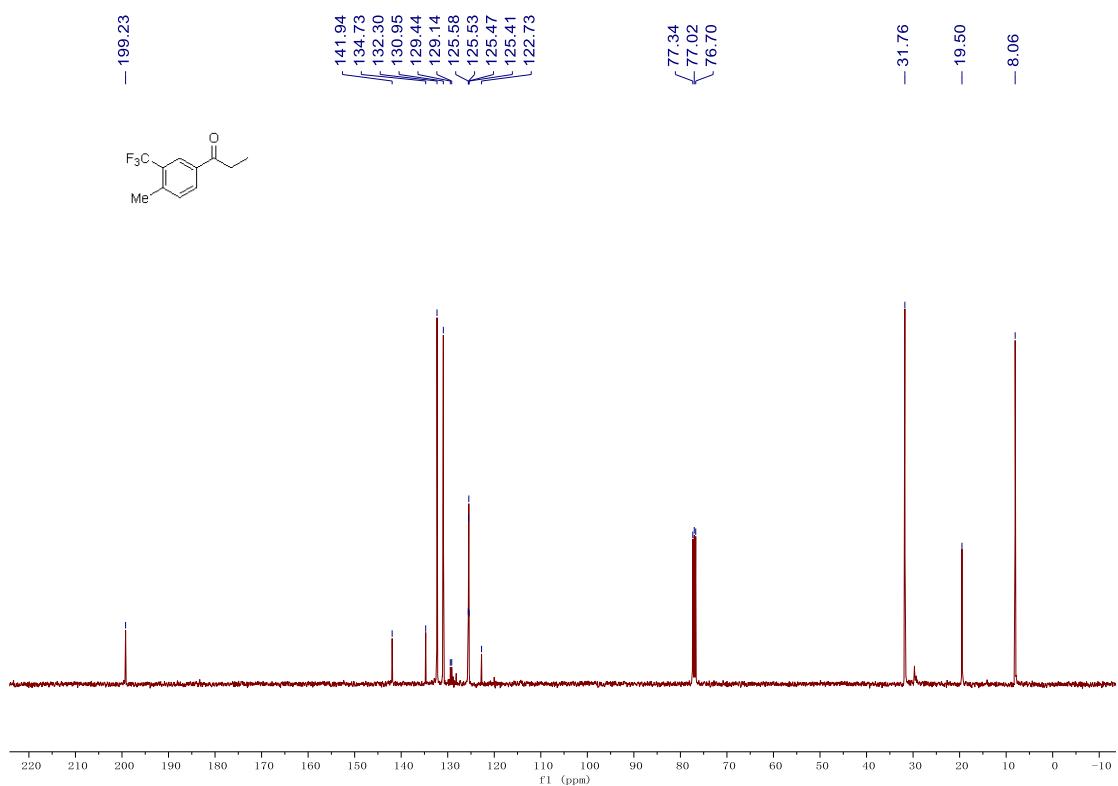
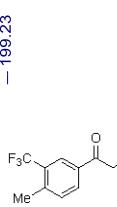
2-(3-propionylphenyl)propanenitrile (24)



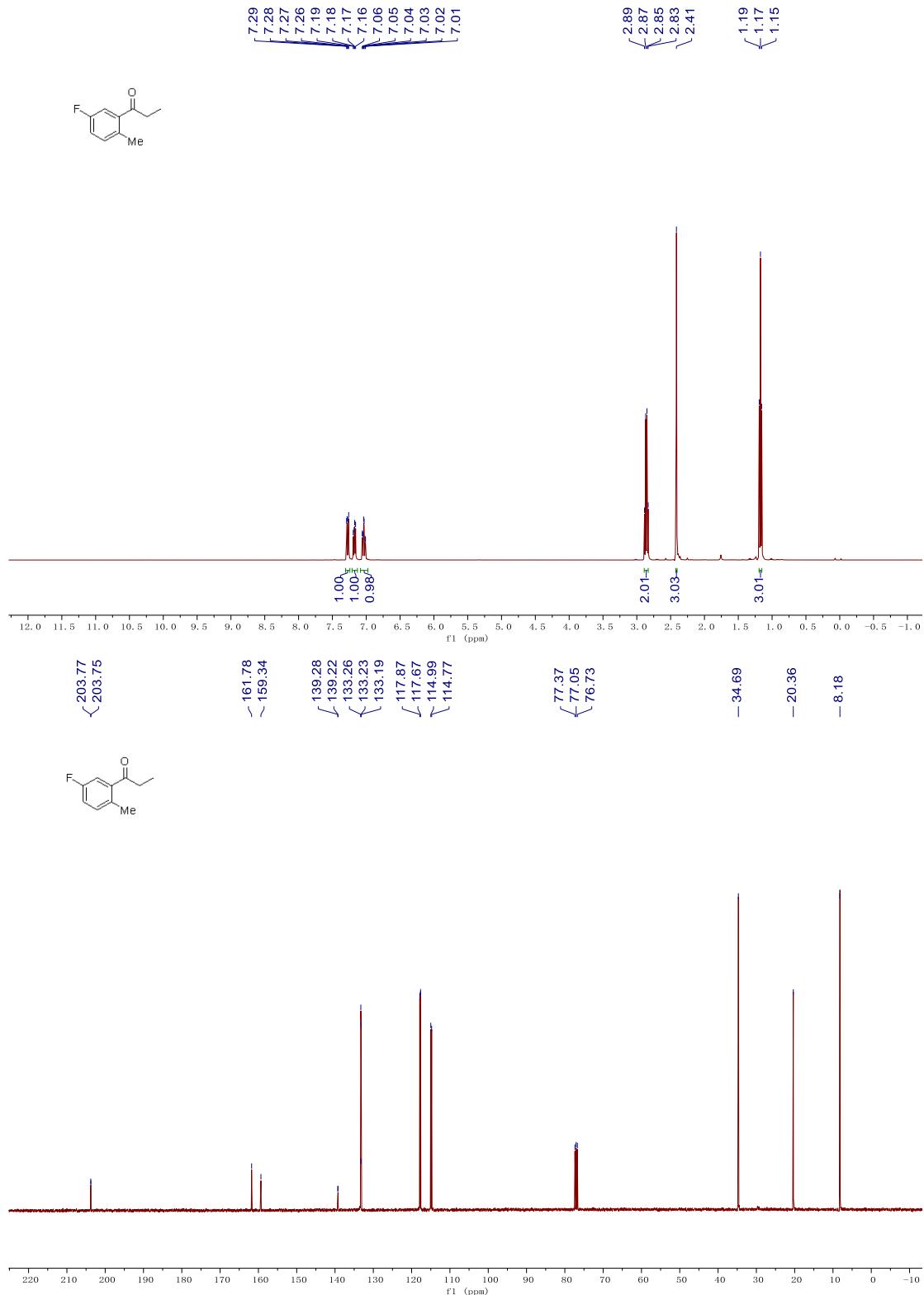


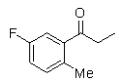
1-(4-methyl-3-(trifluoromethyl)phenyl)propan-1-one (25)



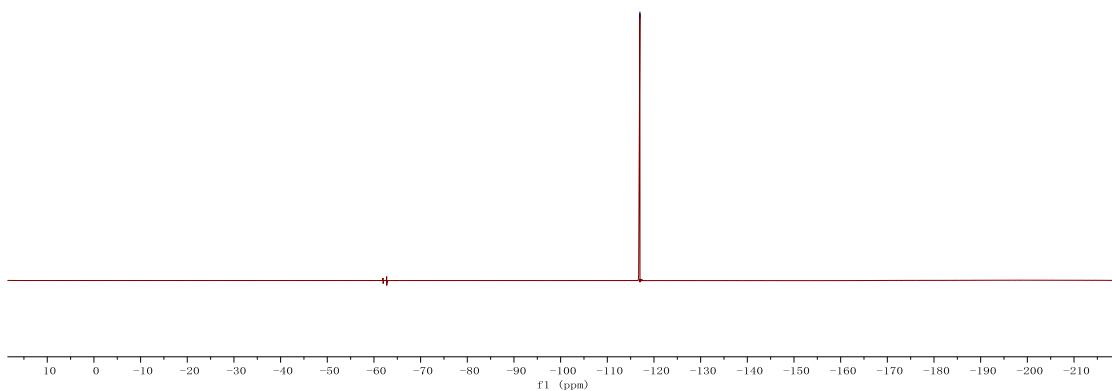


1-(5-fluoro-2-methylphenyl)propan-1-one (26)

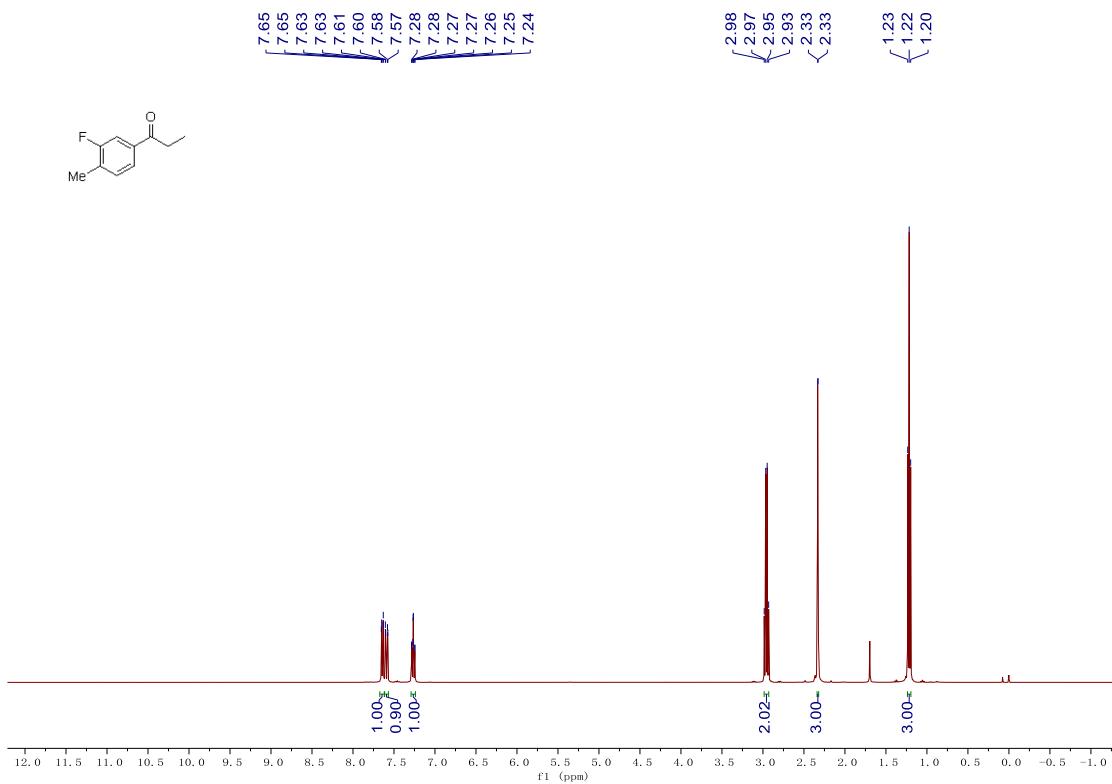


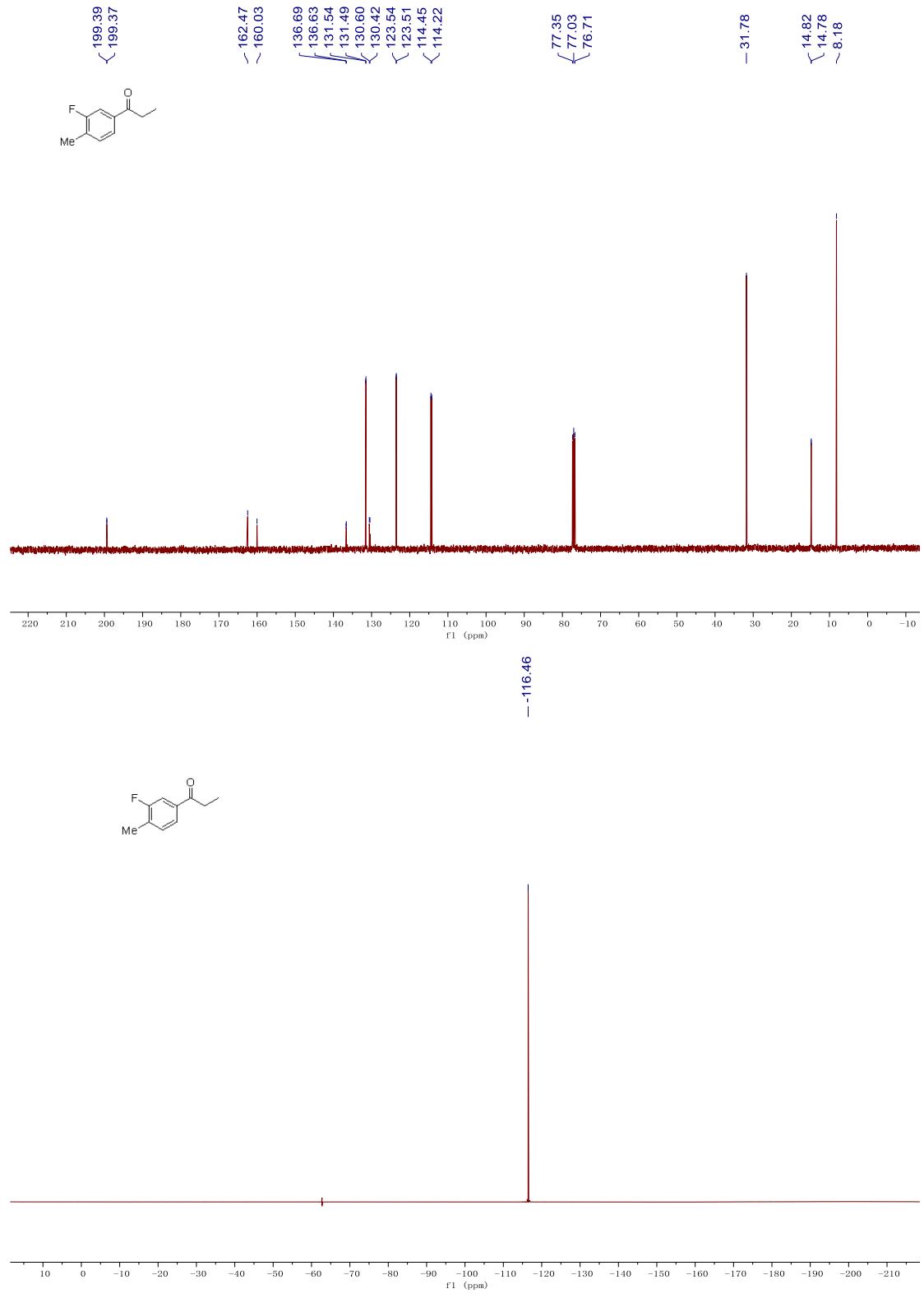


-116.98

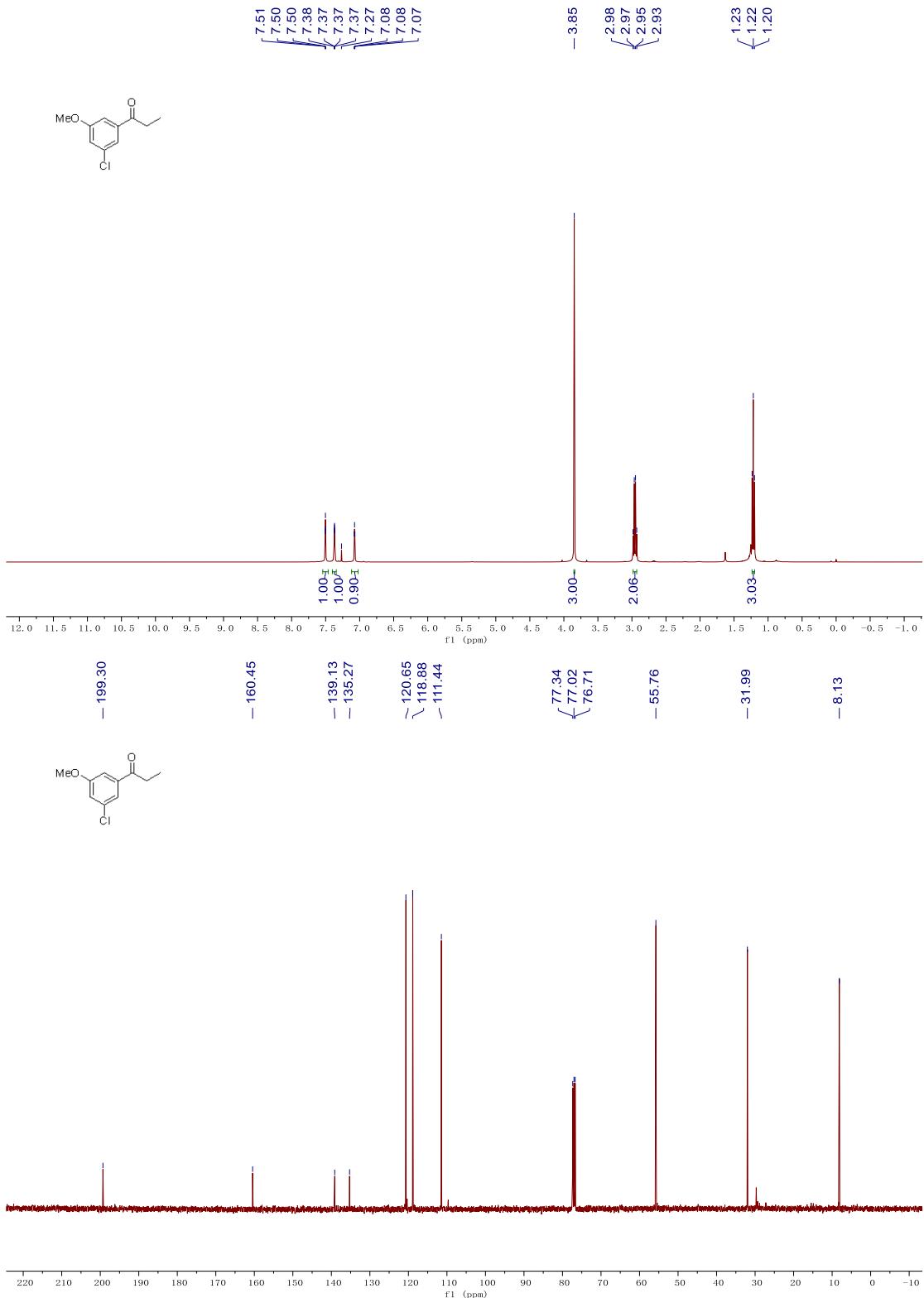


1-(3-fluoro-4-methylphenyl)propan-1-one (27)

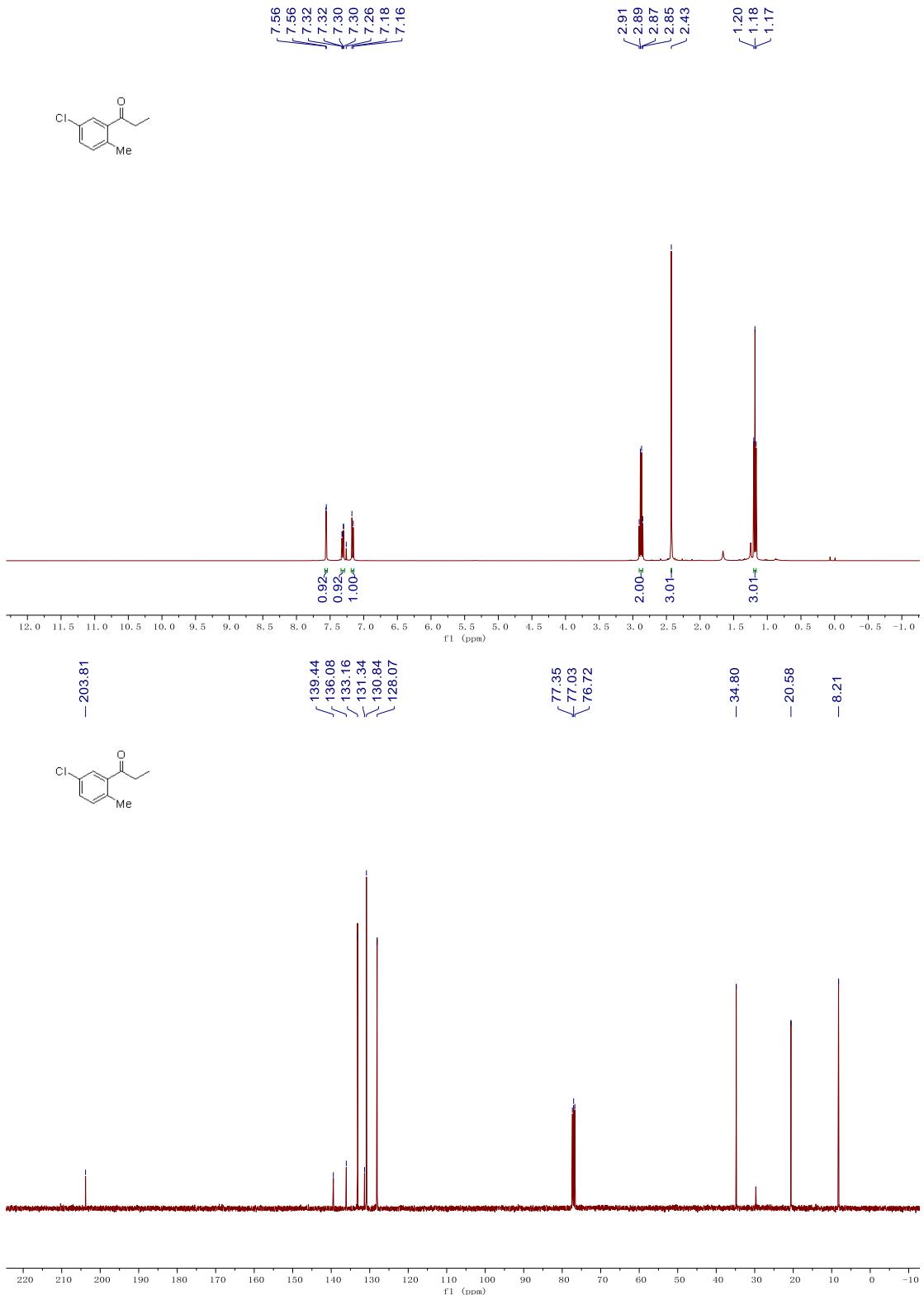




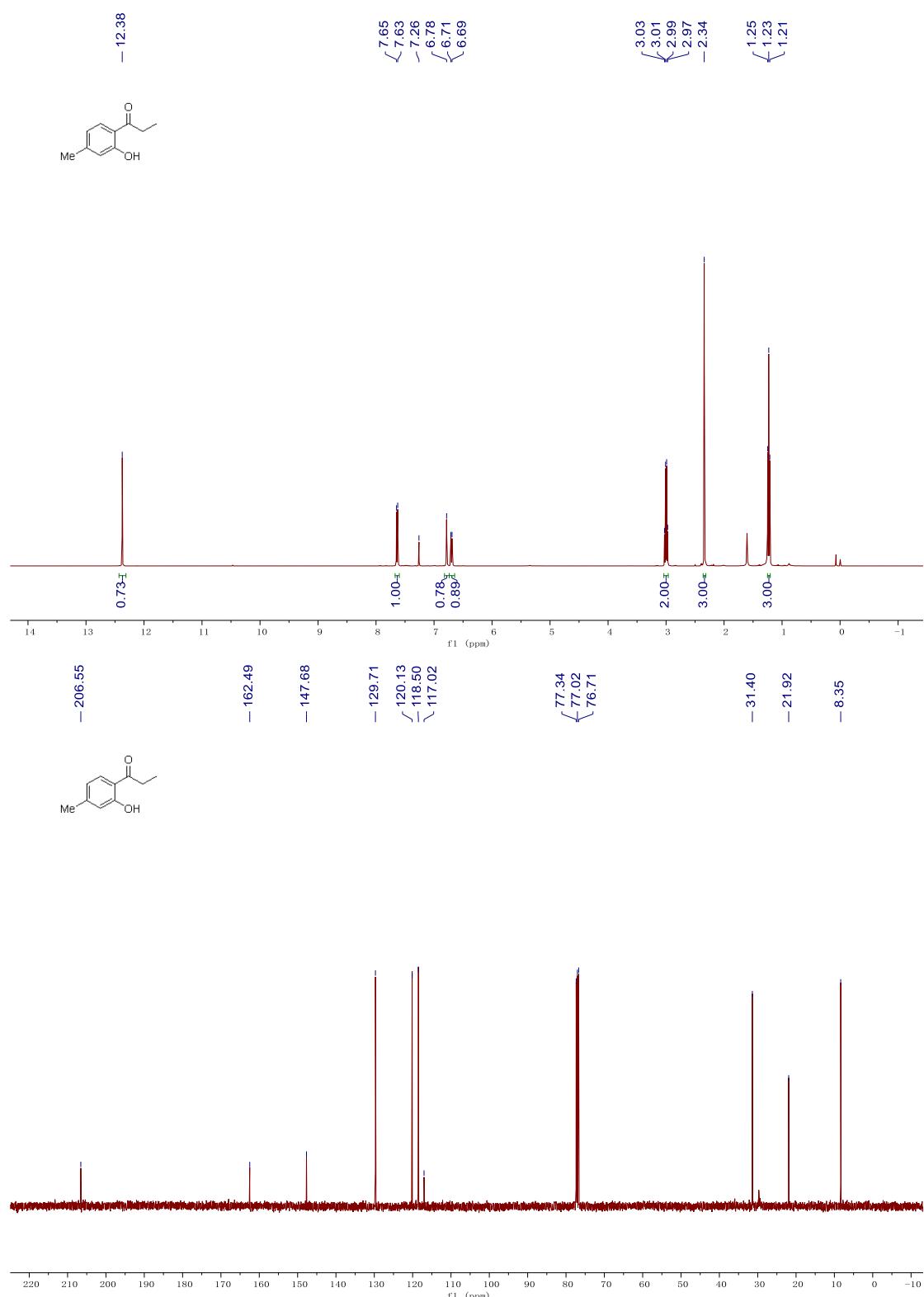
1-(3-chloro-5-methoxyphenyl)propan-1-one (28)



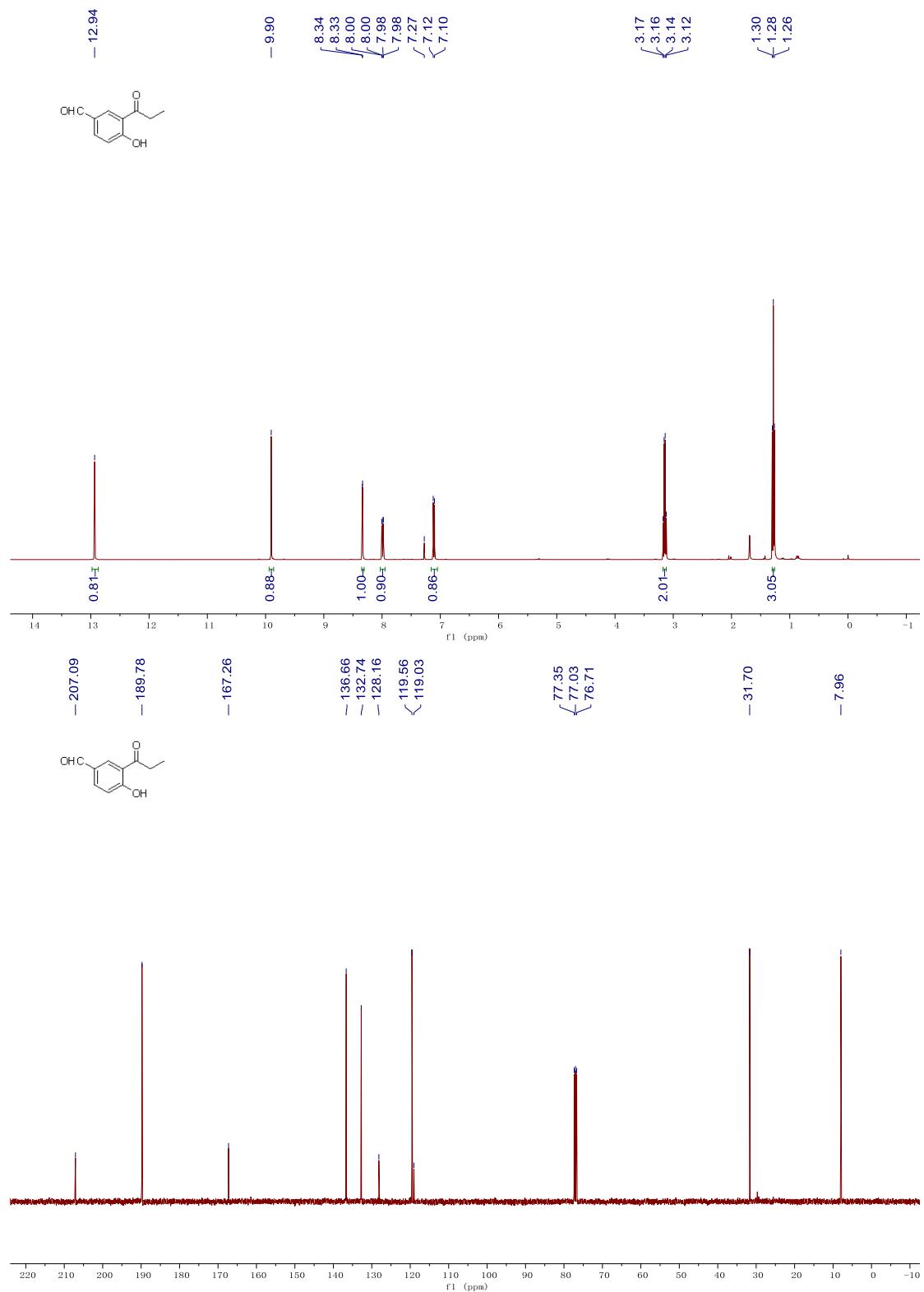
1-(5-chloro-2-methylphenyl)propan-1-one (29)



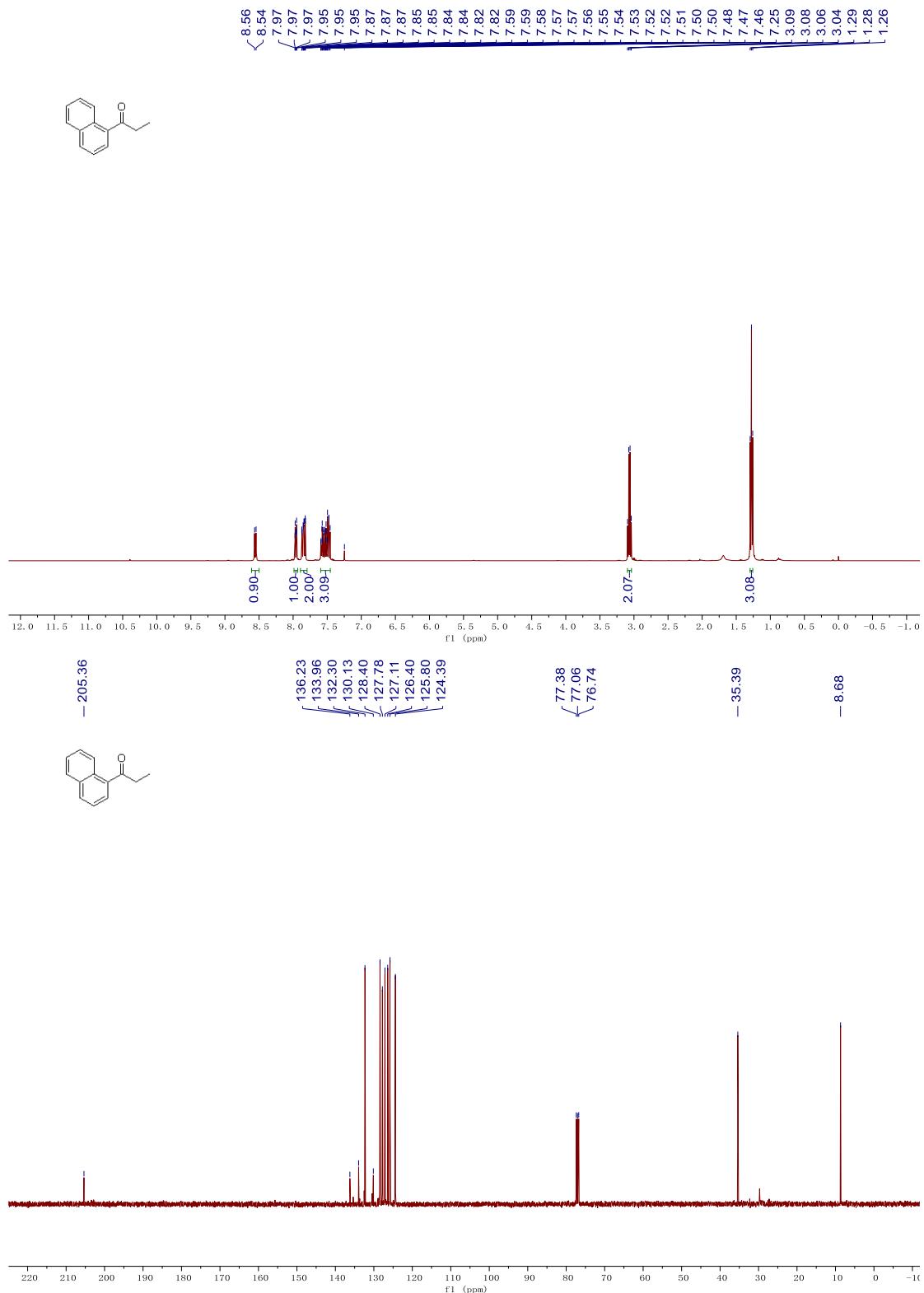
1-(2-hydroxy-4-methylphenyl)propan-1-one (30)



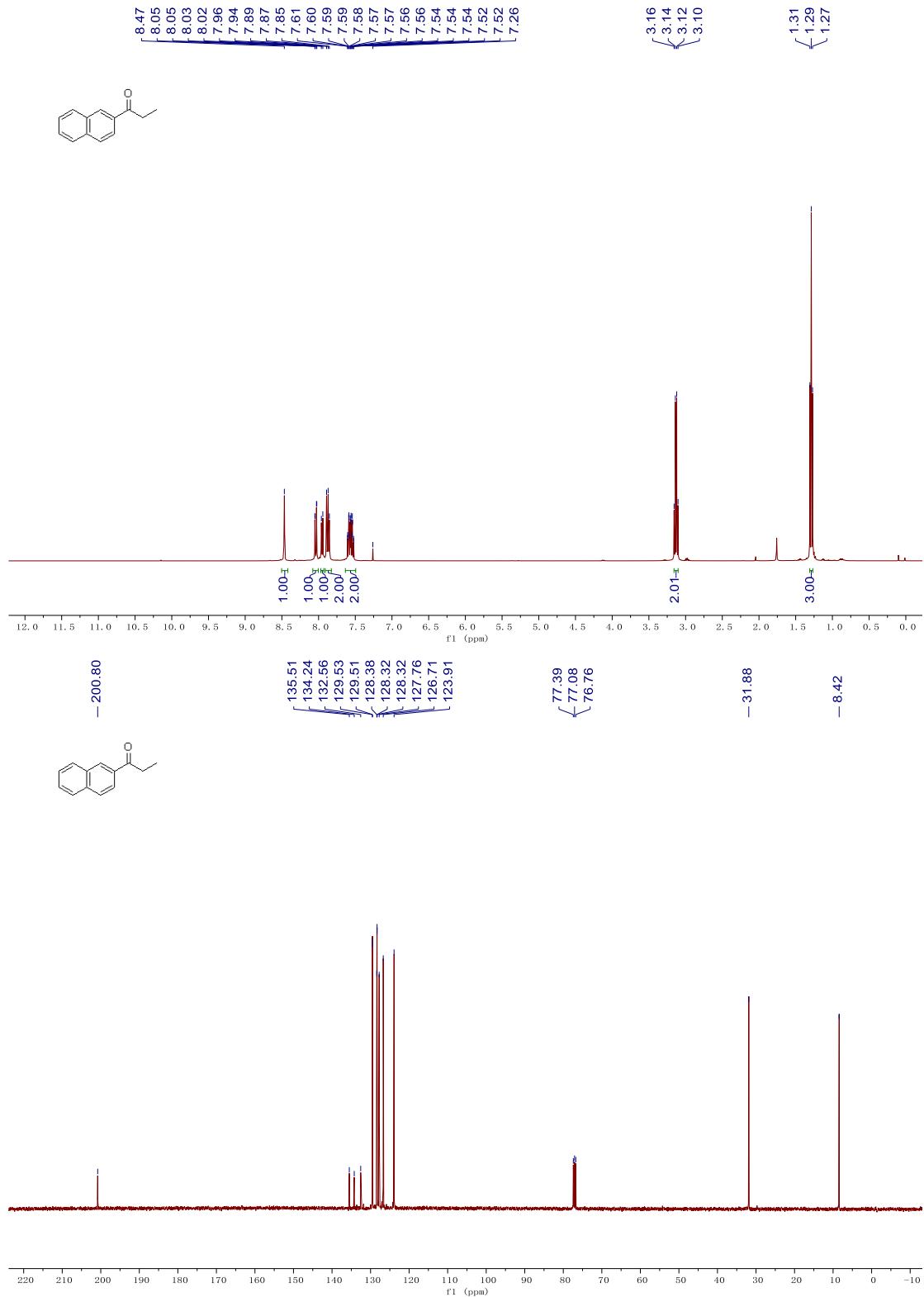
4-hydroxy-3-propionylbenzaldehyde (31)



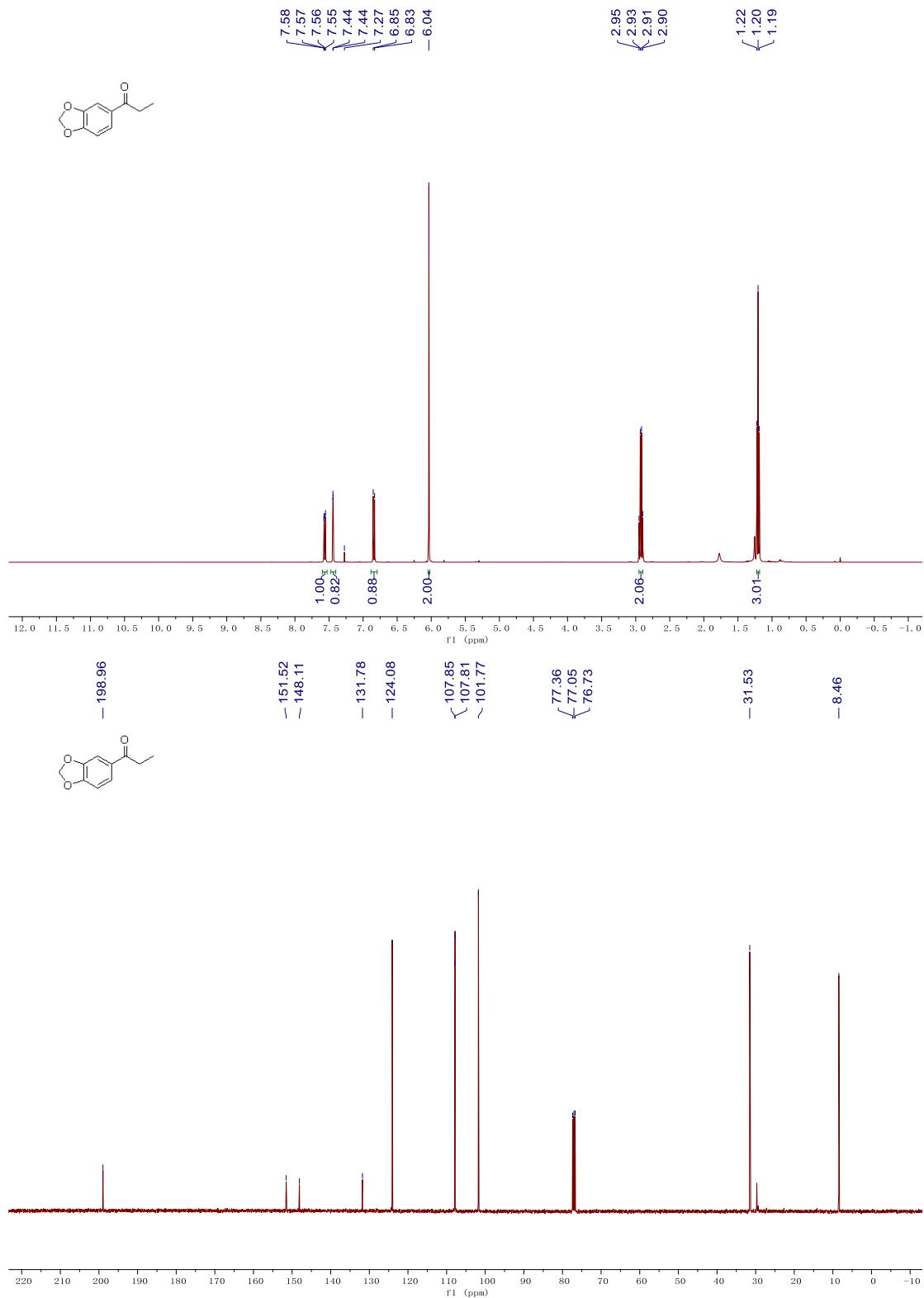
1-(naphthalen-1-yl)propan-1-one (32)



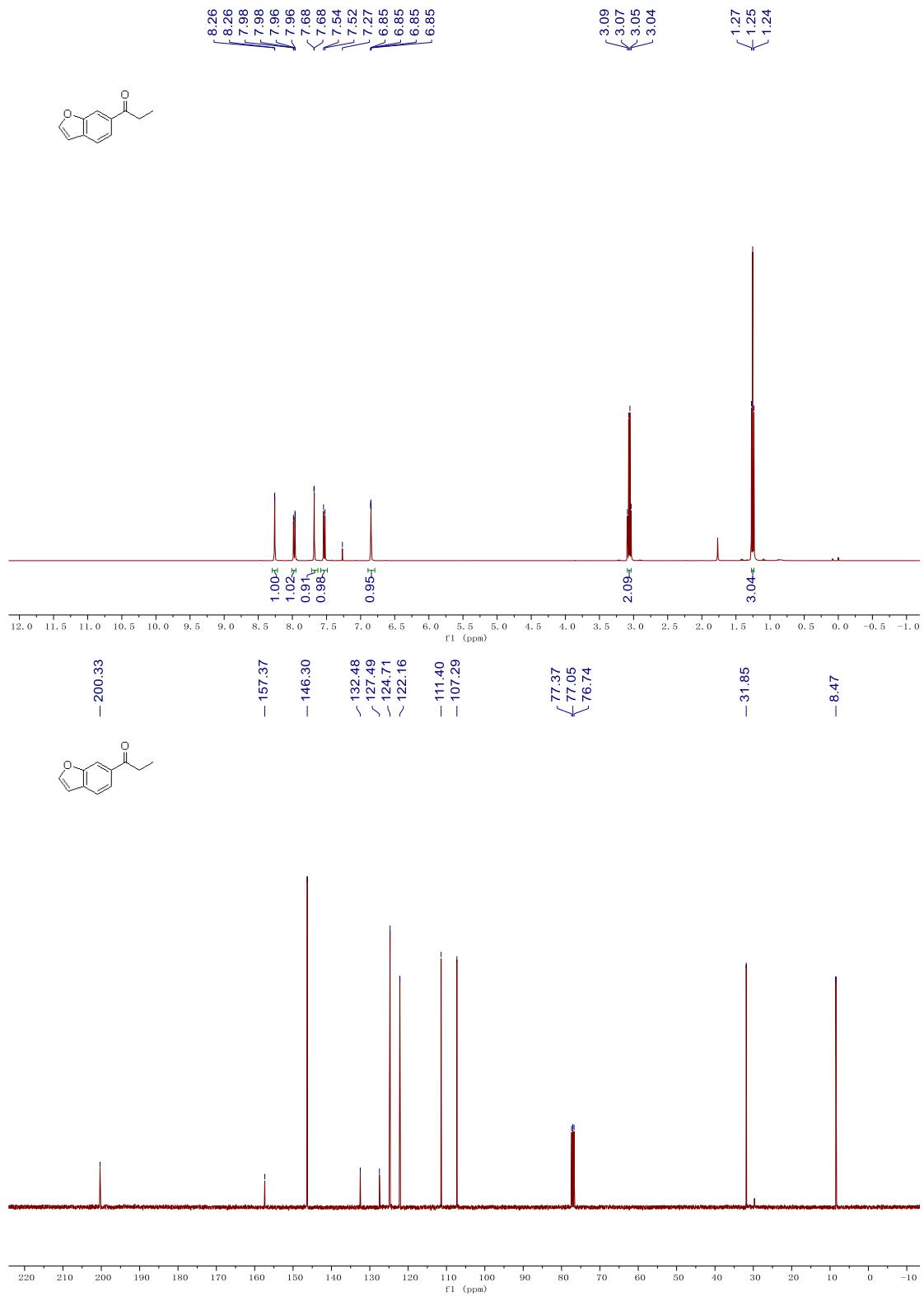
1-(naphthalen-2-yl)propan-1-one (33)



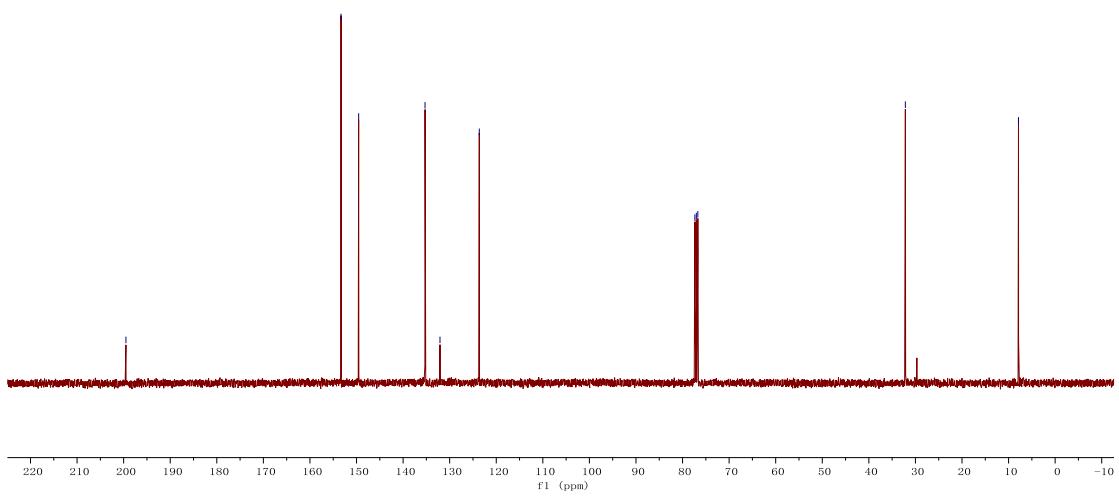
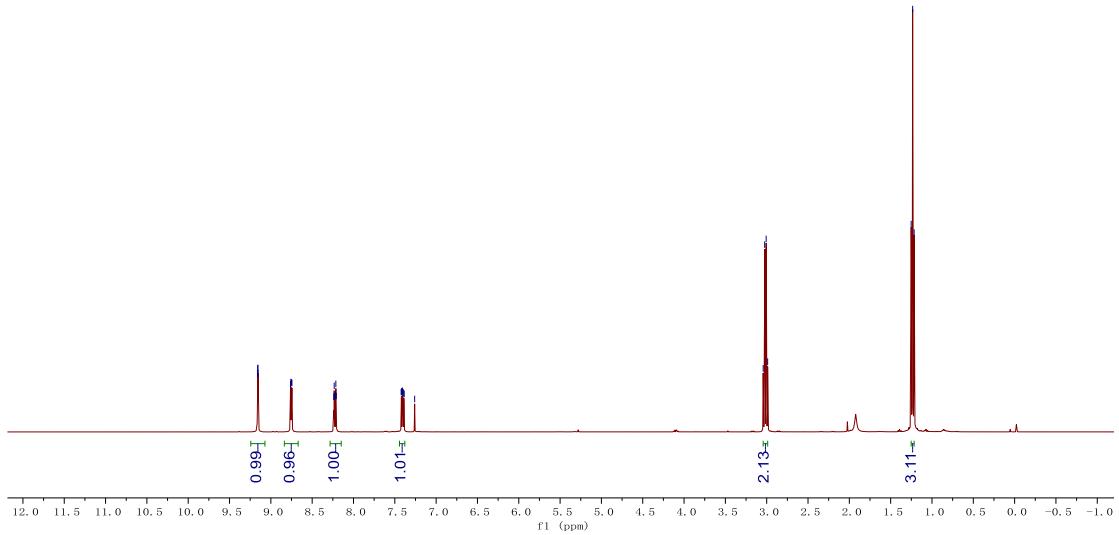
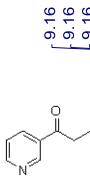
1-(benzo[*d*]1,3-dioxol-5-yl)propan-1-one (34)



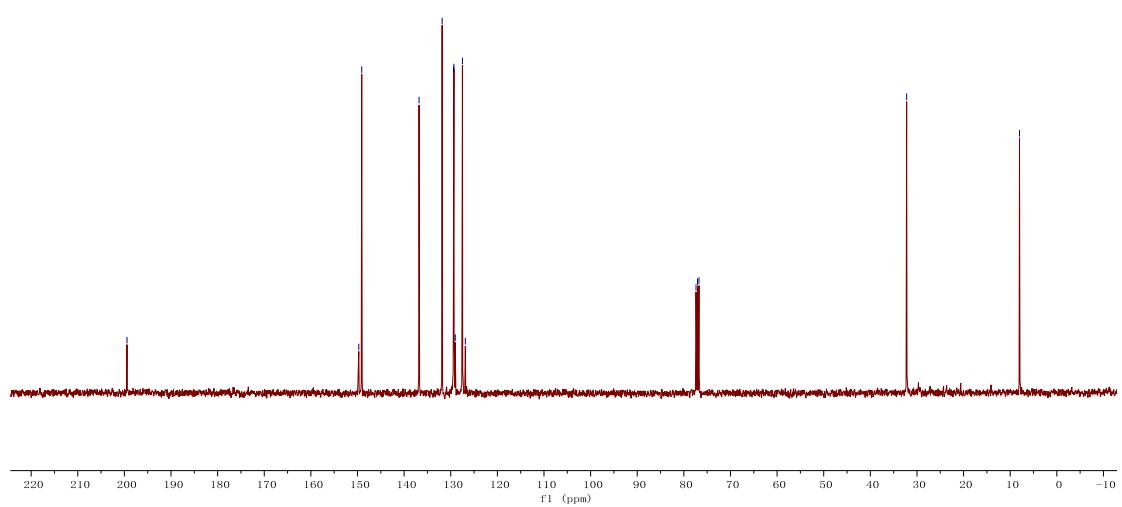
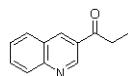
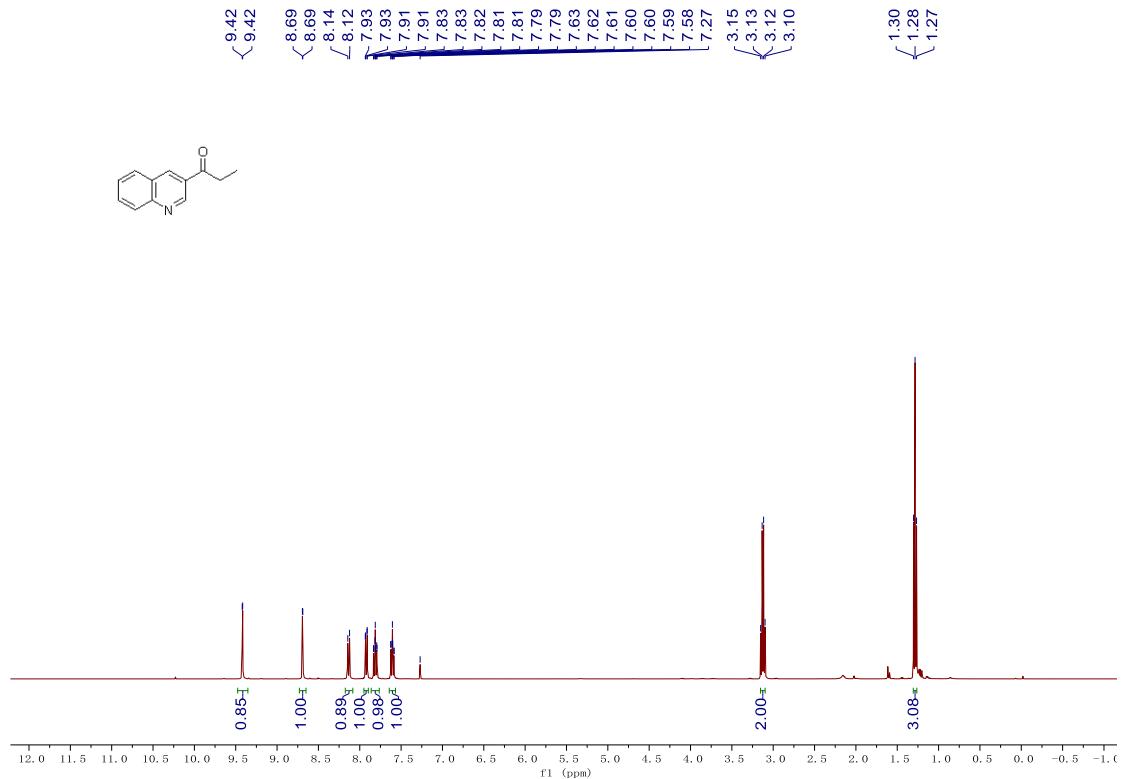
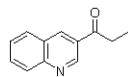
1-(benzofuran-6-yl)propan-1-one (35)



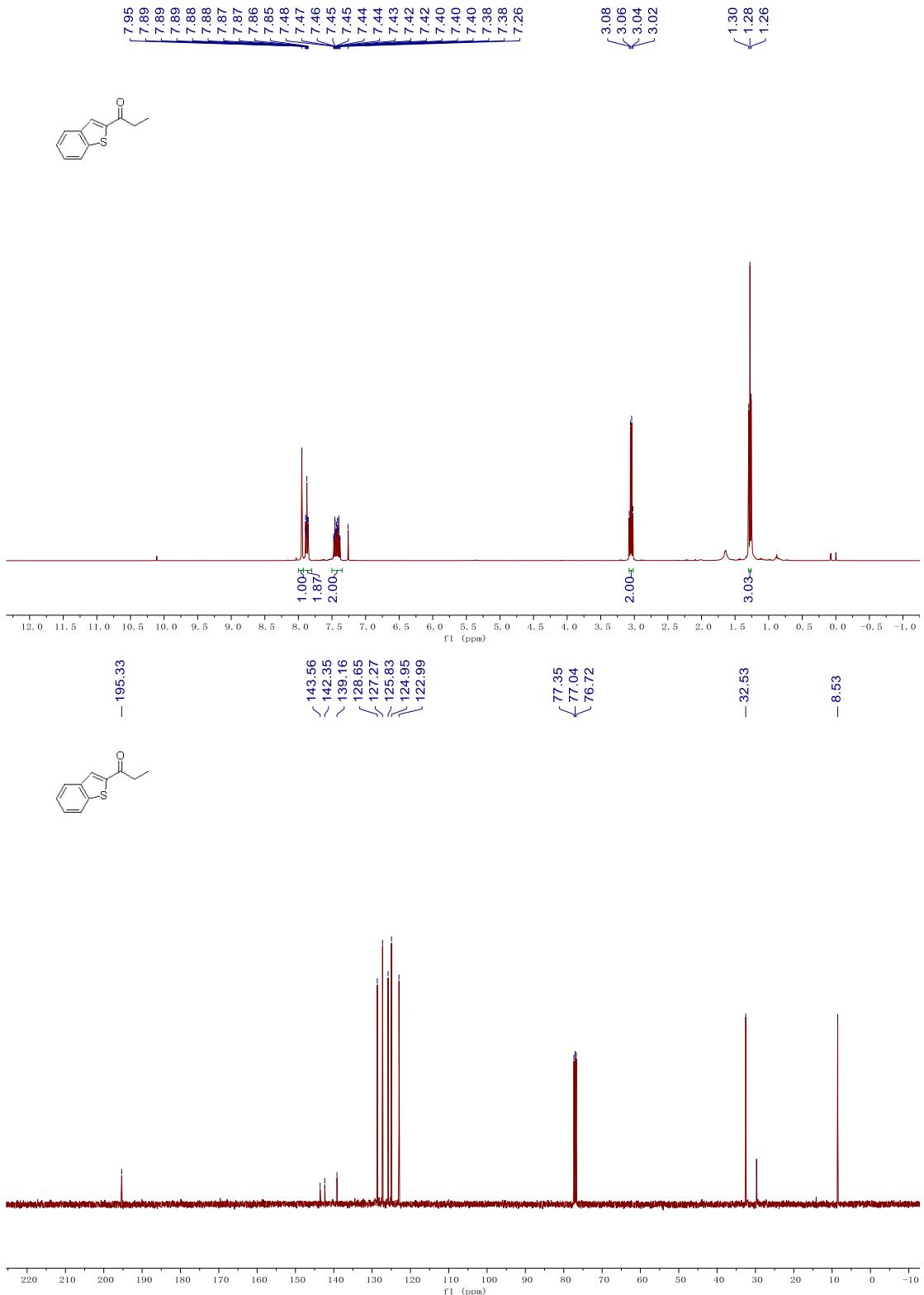
1-(pyridin-3-yl)propan-1-one (36)



1-(quinolin-3-yl)propan-1-one (37)



1-(benzo[b]thiophen-2-yl)propan-1-one (38)



7 References

- 1 M. S. Lowry, J. I. Goldsmith, J. D. Slinker, R. Rohl, R. A. Pascal, Jr., G. G. Malliaras and S. Bernhard, *Chem. Mater.* 2005, **17**, 5712-5719.
- 2 S. Ueno, R. Shimizu and R. Kuwano, *Angew. Chem., Int. Ed.*, 2009, **48**, 4543-4545.
- 3 J. Ruan, X. Li, O. Saidi and J. Xiao, *J. Am. Chem. Soc.*, 2008, **130**, 2424-2425.
- 4 J. Kubota, T. Ido, M. Kuroboshi, H. Tanaka, T. Uchida and K. Shimamura, *Tetrahedron*, 2006, **62**, 4769-4773.
- 5 S. -D. Cho, H. -K. Kim, H. -S. Yim, M. -R. Kim, J. -K. Lee, J. -J. Kimd and Y. -J. Yoon, *Tetrahedron*, 2007, **63**, 1345-1352.
- 6 A. Bouziane, M. Helou, B. Carboni, F. Carreaux, B. Demerseman, C. Bruneau and J. -L. Renaud, *Chem. Eur. J.*, 2008, **14**, 5630-5637.
- 7 P. Tamilselvan, Y. B. Basavaraju, R. Murugesan and E. Sampathkumar, *Catal. Commun.*, 2008, **10**, 300-303.
- 8 M. Liu, Z. Hyder, Y. Sun, W. Tang, L. Xu and J. Xiao, *Org. Biomol. Chem.*, 2010, **8**, 2012-2015.
- 9 N. Sato and N. Narita, *Synthesis*, 2001, **2001**, 1551-1555.
- 10 T. Kusukawa, Y. Kojima and F. Kannen, *Chem. Lett.*, 2019, **48**, 1213-1216.
- 11 B. M. Trost, Z. T. Ball and K. M. Laemmerhold, *J. Am. Chem. Soc.*, 2005, **127**, 10028-10038.
- 12 Z. Liu, H. Tan, L. Wang, T. Fu, Y. Xia, Y. Zhang and J. Wang, *Angew. Chem., Int. Ed.*, 2015, **54**, 3056-3060.
- 13 G. Shan, X. Yang, L. Ma and Y. Rao, *Angew. Chem., Int. Ed.*, 2012, **51**, 13070-13074.
- 14 P. Mamone, G. Danoun and L. J. Gooßen, *Angew. Chem., Int. Ed.*, 2013, **52**, 6704-6708.
- 15 T. Li, J. Li, Z. Zhu, W. Pan and S. Wu, *RSC Adv.*, 2019, **9**, 20879-20883.
- 16 R. Riemschneider and C. Weygand, *Monatshefte fuer Chemie*, 1955, **86**, 201-9.
- 17 I. M. Lagoja, C. Pannecouque, A. V. Aerschot, M. Witvrouw, Z. Debyser, J. Balzarini, P. Herdewijn and E. D. Clercq, *J. Med. Chem.*, 2003, **46**, 1546-1553.
- 18 T. Muto, T. Tanaka, H. Maruoka, S. Imajo, Y. Tomimori, K. Sato, T. Yagi, *PCT Int. Appl.*, 2007, WO 2007139230 A1 20071206.
- 19 A. Moriconi, M. C. Cesta, M. N. Cervellera, A. Aramini, S. Coniglio, S. Colagioia, A. R. Beccari, C. Bizzarri, M. R. Cavicchia, M. Locati, E. Galliera, P. D. Benedetto, P. Vigilante, R. Bertini and M. Allegretti, *J. Med. Chem.*, 2007, **50**, 3984-4002.
- 20 S. Ishibuchi, K. Saruta, M. Hamada, N. Matoba, T. Matsudaira, M. Seki, A. Tarao, T. Honjo, S. Ogata, A. Kawata, K. Morokuma, N. Fujie, Y. Aoyama, *PCT Int. Appl.*, 2017, WO 2017007008 A1 20170112.
- 21 R. W. Hartmann, A. Heindl and H. Schónenberger, *J. Med. Chem.*, 1984, **27**, 577-

- 22 F. I. Carroll, B. E. Blough, P. Abraham, A. C. Mills, J. A. Holleman, S. A. Wolckenhauer, A. M. Decker, A. Landavazo, K. T. McElroy, H. A. Navarro, M. B. Gatch and M. J. Forster, *J. Med. Chem.*, 2009, **52**, 6768-6781.
- 23 G. Shan, X. Han, Y. Lin, S. Yua and Y. Rao, *Org. Biomol. Chem.*, 2013, **11**, 2318-2322.
- 24 J. Ruan, J. A. Iggo, N. G. Berry and J. Xiao, *J. Am. Chem. Soc.*, 2010, **132**, 16689-16699.
- 25 M. Ito, S. Kitahara and T. Ikariya, *J. Am. Chem. Soc.*, 2005, **127**, 6172-6173.
- 26 A. K. Chaturvedia and N. Rastogi, *Org. Biomol. Chem.*, 2018, **16**, 8155-8159.
- 27 H. Wu, W. Tang, M. Jin, Q. Ge, X. Zhang, S. Chen, B. Fan, *Faming Zhuanli Shengqing*, 2020, CN 111393367 A 20200710.