

Electronic Supplementary Information

High-Valent Cu(III)-CF₃ Compound-Mediated Esterification Reaction

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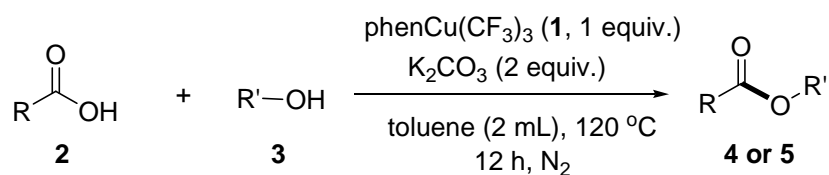
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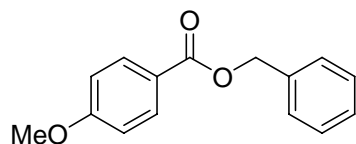
1. General experimental details

All of the chemicals were purchased commercially except complex **1** (phenCu(CF₃)₃) which was prepared according to the method we developed (S.-L. Zhang, W.-F Bie, *RSC Adv.* **2016**, *6*, 70902), and were used as received without further purification. All of the reactions were performed in a sealed Schlenk tube under N₂ atmosphere which was realized through evacuation/backfill techniques after three times. The reactions were monitored by TLC analysis with stains visualized by UV irradiation or iodine vapor until the substrates were completely consumed. Column chromatography on silica gel was used to obtain purified products that are suitable for NMR spectroscopic characterization. NMR spectra were recorded on a 400 MHz spectrometer for ¹H NMR, 101MHz for ¹³C NMR, and 376 MHz for ¹⁹F NMR. Chemical shifts are reported in ppm and referenced to residual solvent peaks (¹H and ¹³C are referenced to HCCl₃; ¹⁹F are relative to CFCl₃). NMR signals are reported as follows to delineate possible splitting: s, singlet; br s, broad singlet; d, doublet; t, triplet; q, quartet; and m, multiplet. Coupling constants are reported in Hertz where present. All of the ¹³C and ¹⁹F NMR spectra were obtained with proton decoupling for clarity. Elemental analyses were performed by the Analytic Laboratory of Jiangnan University. High resolution mass spectra (HRMS) were determined on Thermo Scientific LTQ Orbit-rap XL with ESI ionization mode.

2. General procedure for (phen)Cu(III)(CF₃)₃-mediated reaction of carboxylic acids and alcohols/phenols to produce esters

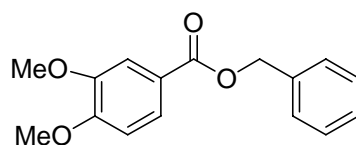


In an oven-dried 25-mL Schlenk tube equipped with a magnetic stir bar were added a phenCu(CF₃)₃ (**1**) (0.2 mmol), carboxylic acid (**2**) (0.2 mmol), alcohol/phenol (**3**, 0.2 mmol), K₂CO₃ (0.4 mmol). The Schlenk tube was evacuated and refilled with dry nitrogen. Toluene (2 ml) was then added by syringe. The contents in the tube were vigorously stirred for 12 h at 120 °C (seated in an oil bath). The reaction mixture was then allowed to cool to temperature. The resulting mixture was extracted by dichloromethane. The combined organic layers were washed with a large amount of water for 4 times, with brine for once and then dried over magnesium sulfate. The solvent was removed under vacuum and the residuals were purified by column chromatography on silica using a mixture of petroleum ether (PE) and ethyl acetate (EA) as the eluent to give purified esters **4** or **5**.

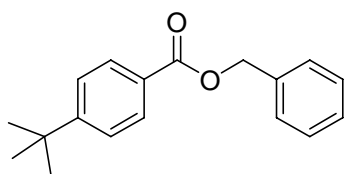


Benzyl 4-methoxybenzoate (4a); 41.1 mg, 85%). Eluted with a mixture of petroleum ether/ethyl acetate = 5:1 (v/v). Yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, *J* = 8.9 Hz, 2H), 7.48 – 7.31 (m, 5H), 6.92 (d, *J* = 8.9 Hz, 2H), 5.34 (s, 2H), 3.86 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 166.2, 163.4, 136.3, 131.7, 128.6, 128.12, 128.09, 122.6, 113.6, 66.4, 55.4.

These data are in good agreement with literature report.^{S1}

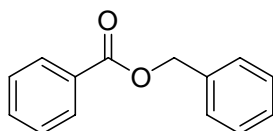


Benzyl 3,4-dimethoxybenzoate (4b; 24.3 mg, 45%). Eluted with a mixture of petroleum ether/ethyl acetate = 5:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.73 (dd, $J = 8.4, 2.0$ Hz, 1H), 7.58 (d, $J = 1.9$ Hz, 1H), 7.48 – 7.31 (m, 5H), 6.88 (d, $J = 8.5$ Hz, 1H), 5.35 (s, 2H), 3.93 (s, 3H), 3.92 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.2, 152.1, 147.6, 135.3, 127.6, 127.2, 127.1, 122.7, 121.6, 111.1, 109.2, 65.5, 55.0. These data are in good agreement with literature report.^{S2}



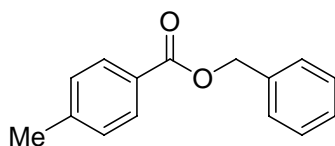
Benzyl 4-(tert-butyl)benzoate (4c; 36.1 mg, 67%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400MHz, CDCl_3) δ 8.03 (d, $J = 8.7$ Hz, 2H), 7.50 – 7.43 (m, 4H), 7.42–7.31 (m, 3H), 5.37 (s, 2H), 1.35 (s, 9H). ^{13}C NMR (101 MHz, CDCl_3) δ 166.4, 156.7, 136.3, 129.6, 128.5, 128.1, 128.0, 127.3, 125.3, 66.4, 35.0, 31.1.

These data are in good agreement with literature report.^{S1}



Benzyl benzoate (4d; 33.2 mg, 79%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 8.10 (d, $J = 7.1$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.50 – 7.33 (m, 7H), 5.39 (s, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 166.4, 136.0, 133.0, 130.1, 129.7, 128.6, 128.3, 128.2, 128.1, 66.7.

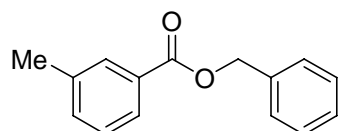
These data are in good agreement with literature report.^{S1}



Benzyl 4-methylbenzoate (4e; 42.5 mg, 95%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J =$

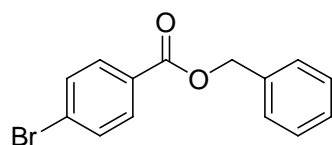
8.2 Hz, 2H), 7.49 – 7.34 (m, 5H), 7.24 (d, $J = 8.0$ Hz, 2H), 5.37 (s, 2H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 166.5, 143.7, 136.2, 129.7, 129.1, 128.5, 128.13, 128.08, 127.4, 66.5, 21.6.

These data are in good agreement with literature report.^{S3}



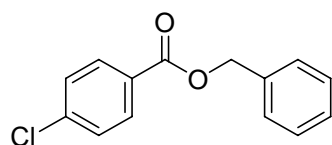
Benzyl 3-methylbenzoate (4f); 32.3 mg, 72%). Eluted with petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.94 – 7.87 (m, 2H), 7.49 – 7.44 (m, 2H), 7.43 – 7.30 (m, 5H), 5.38 (s, 2H), 2.41 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 166.6, 138.1, 136.1, 133.8, 130.1, 130.0, 128.6, 128.24, 128.17, 128.14, 126.8, 66.6, 21.2.

These data are in good agreement with literature report.^{S3}



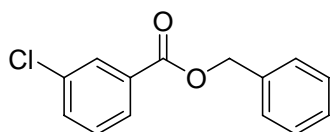
Benzyl 4-bromobenzoate (4g); 46.6 mg, 81%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J = 8.6$ Hz, 2H), 7.58 (d, $J = 8.6$ Hz, 2H), 7.48 – 7.34 (m, 5H), 5.37 (s, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.7, 135.7, 131.2, 129.7, 129.0, 128.6, 128.3, 128.2, 128.1, 66.9.

These data are in good agreement with literature report.^{S3}



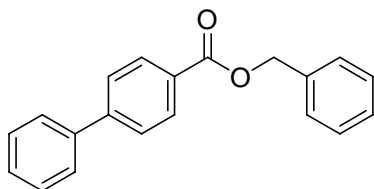
Benzyl 4-chlorobenzoate (4h); 35.3 mg, 77%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 8.01 (d, $J = 8.7$ Hz, 2H), 7.46 – 7.36 (m, 7H), 5.37 (s, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.5, 139.5, 135.8, 131.1, 128.7, 128.6, 128.5, 128.3, 128.2, 66.9.

These data are in good agreement with literature report.^{S3}



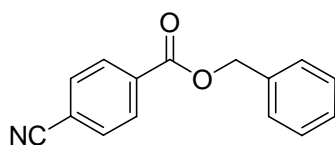
Benzyl 3-chlorobenzoate (4i); 33.1 mg, 67%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.06 (t, *J* = 1.7 Hz, 1H), 7.97 (dt, *J* = 7.8, 1.2 Hz, 1H), 7.53 (ddd, *J* = 8.0, 2.0, 1.0 Hz, 1H), 7.47 – 7.35 (m, 6H), 5.38 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 165.2, 135.6, 134.5, 133.0, 131.9, 129.71, 129.67, 128.6, 128.4, 128.3, 127.8, 67.1.

These data are in good agreement with literature report.^{S2}



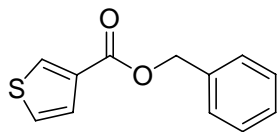
Benzyl [1,1'-biphenyl]-4-carboxylate (4j); 51.3 mg, 88%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). White solid; ¹H NMR (400 MHz, CDCl₃) δ 8.17 (d, *J* = 8.5 Hz, 2H), 7.68 (d, *J* = 8.5 Hz, 2H), 7.64 (d, *J* = 7.1 Hz, 2H), 7.48 (t, *J* = 7.5 Hz, 4H), 7.45 – 7.36 (m, 4H), 5.41 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 166.3, 145.7, 139.9, 136.1, 130.2, 128.9, 128.8, 128.6, 128.21, 128.14, 128.12, 127.2, 127.0, 66.7.

These data are in good agreement with literature report.^{S4}



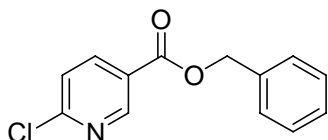
Benzyl 4-cyanobenzoate (4k); 11 mg, 23%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.17 (d, *J* = 8.6 Hz, 2H), 7.74 (d, *J* = 8.6 Hz, 2H), 7.46–7.32 (m, 5H), 5.39 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 164.8, 135.3, 133.9, 132.2, 130.2, 128.7, 128.6, 128.4, 117.9, 116.5, 67.5.

These data are in good agreement with literature report.^{S3}



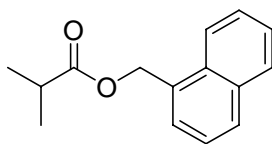
Benzyl thiophene-3-carboxylate (4l); 24.6 mg, 56%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oli; ¹H NMR (400 MHz, CDCl₃) δ 8.15 (dd, *J* = 3.1, 1.1 Hz, 1H), 7.56 (dd, *J* = 5.1, 1.1 Hz, 1H), 7.46 – 7.34 (m, 5H), 7.31 (dd, *J* = 5.1, 3.1 Hz, 1H), 5.33 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 162.6, 136.0, 133.5, 132.9, 128.6, 128.21, 128.16, 127.9, 126.0, 66.4.

These data are in good agreement with literature report.^{S3}



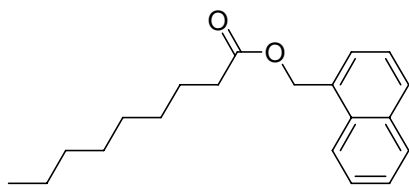
Benzyl 6-chloronicotinate (4m); 41.3 mg, 84%). Eluted with a mixture of petroleum ether/ethyl acetate = 10:1 (v/v). Yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 9.02 (d, *J* = 2.3 Hz, 1H), 8.25 (dd, *J* = 8.3, 2.4 Hz, 1H), 7.47 – 7.35 (m, 6H), 5.39 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 164.2, 155.7, 151.2, 139.6, 135.2, 128.63, 128.58, 128.4, 125.0, 124.1, 67.3.

These data are in good agreement with literature report.^{S5}



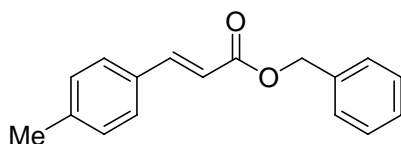
Naphthalen-1-ylmethyl isobutyrate (4n); 21.1 mg, 54%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.01 (d, *J* = 8.1 Hz, 1H), 7.91 – 7.83 (m, 2H), 7.58 – 7.43 (m, 4H), 5.58 (s, 2H), 2.62 (hept, *J* = 7.0 Hz, 1H), 1.19 (d, *J* = 7.0 Hz, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 177.0, 133.7, 131.7, 131.6, 129.1, 128.7, 127.2, 126.5, 125.9, 125.3, 123.6, 64.5, 34.1, 19.0.

These data are in good agreement with literature report.^{S6}



Naphthalen-1-ylmethyl nonanoate (4o; 41.2 mg, 69%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 8.02 (d, $J = 8.2$ Hz, 1H), 7.92 – 7.84 (m, 2H), 7.60 – 7.43 (m, 4H), 5.59 (s, 2H), 2.37 (t, $J = 7.5$ Hz, 2H), 1.65 (quartet, $J = 7.2$ Hz, 2H), 1.34 – 1.21 (m, 10H), 0.88 (t, $J = 7.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 173.8, 133.7, 131.6, 129.2, 128.7, 127.4, 126.5, 125.9, 125.2, 123.6, 64.4, 34.4, 31.8, 29.17, 29.10, 29.07, 25.0, 22.6, 14.1.

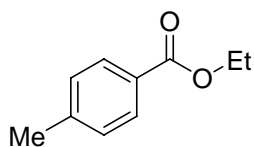
These data are in good agreement with literature report.^{S7}



(E)-Benzyl 3-(*p*-tolyl)acrylate (4p; 38.5 mg, 77%). Eluted with petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.72 (d, $J = 16.0$ Hz, 1H), 7.48 – 7.33 (m, 7H), 7.19 (d, $J = 7.9$ Hz, 2H), 6.45 (d, $J = 16.0$ Hz, 1H), 5.26 (s, 2H), 2.38 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 167.0, 145.2, 140.8, 136.1, 131.6, 129.6, 128.5, 128.3, 128.2, 128.1, 116.7, 66.2, 21.4.

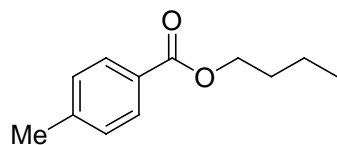
These data are in good agreement with literature report.^{S8}

Reaction of *p*-toluic acid with various alcohols and phenols:



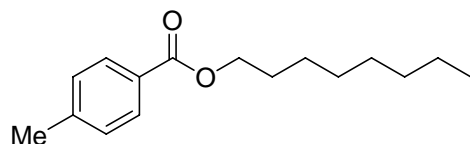
Ethyl 4-methylbenzoate (5a; 14.2 mg, 43%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.93 (d, $J = 8.2$ Hz, 2H), 7.23 (d, $J = 8.0$ Hz, 2H), 4.36 (q, $J = 7.1$ Hz, 2H), 2.41 (s, 3H), 1.39 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 166.7, 143.4, 129.5, 129.0, 127.8, 60.7, 21.6, 14.3.

These data are in good agreement with literature report.^{S9}



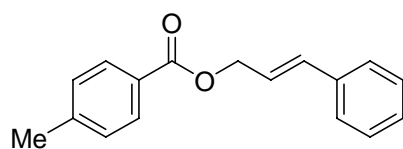
Butyl 4-methylbenzoate (5b); 13.1 mg, 35%). Eluted with petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 7.9 Hz, 2H), 4.31 (t, *J* = 6.6 Hz, 2H), 2.41 (s, 3H), 1.79 – 1.70 (m, 2H), 1.53 – 1.42 (m, 2H), 0.98 (t, *J* = 7.4 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 166.8, 143.4, 129.5, 129.0, 127.8, 64.6, 30.8, 21.6, 19.3, 13.8.

These data are in good agreement with literature report.^{S10}



Octyl 4-methylbenzoate (5c); 38.4 mg, 75%). Eluted with a mixture of petroleum ether/ethyl acetate = 40:1 (v/v). Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 4.30 (t, *J* = 6.7 Hz, 2H), 2.41 (s, 3H), 1.81 – 1.70 (m, 2H), 1.48 – 1.21 (m, 10H), 0.89 (t, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 166.8, 143.4, 129.5, 129.0, 127.8, 64.9, 31.8, 29.24, 29.18, 28.7, 26.0, 22.6, 21.6, 14.1.

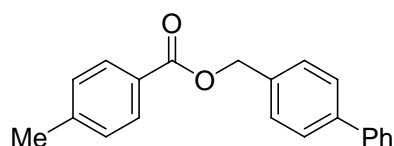
These data are in good agreement with literature report.^{S11}



Cinnamyl 4-methylbenzoate (5d); 32.5 mg, 65%). Eluted with a mixture of petroleum ether/ethyl acetate = 40:1 (v/v). Yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, *J* = 8.2 Hz, 2H), 7.43 (d, *J* = 7.2 Hz, 2H), 7.34 (t, *J* = 7.4 Hz, 2H), 7.30 – 7.23 (m, 3H), 6.75 (d, *J* = 15.9 Hz, 1H), 6.42 (dt, *J* = 15.9, 6.4 Hz, 1H), 4.98 (dd, *J* = 6.4,

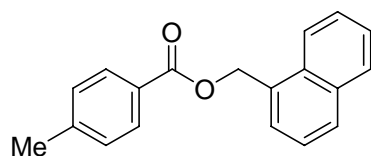
1.3 Hz, 2H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 166.4, 143.6, 136.3, 134.1, 129.7, 129.1, 128.6, 128.0, 127.4, 126.6, 123.4, 65.3, 21.6.

These data are in good agreement with literature report.^{S12}

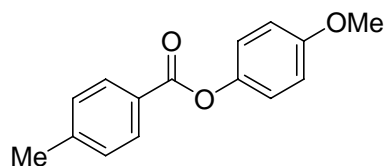


[1,1'-Biphenyl]-4-ylmethyl 4-methylbenzoate (5e; 27.0 mg, 45%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 8.01 (d, J = 8.2 Hz, 2H), 7.66 – 7.58 (m, 4H), 7.53 (d, J = 8.3 Hz, 2H), 7.45 (t, J = 7.5 Hz, 2H), 7.36 (t, J = 7.3 Hz, 1H), 7.25 (d, J = 8.0 Hz, 2H), 5.41 (s, 2H), 2.41 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 166.5, 143.7, 141.1, 140.7, 135.2, 129.7, 129.1, 128.8, 128.6, 127.37, 127.34, 127.28, 127.1, 66.2, 21.6.

These data are in good agreement with literature report.^{S5}



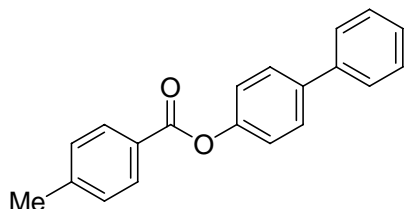
Naphthalen-1-ylmethyl 4-methylbenzoate (5f; 32.6 mg, 60%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 8.13 (d, J = 8.2 Hz, 1H), 7.96 (d, J = 8.2 Hz, 2H), 7.94 - 7.86 (m, 2H), 7.65 (d, J = 6.9 Hz, 1H), 7.61 - 7.46 (m, 3H), 7.21 (d, J = 8.0 Hz, 2H), 5.82 (s, 2H), 2.40 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 166.5, 143.7, 133.7, 131.7, 131.6, 129.8, 129.2, 129.0, 128.7, 127.4, 126.6, 125.9, 125.3, 123.6, 64.9, 21.6. HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{17}\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$ 277.1229, found 277.1220.



4-Methoxyphenyl 4-methylbenzoate (5g; 21.9 mg, 52%). Eluted with a mixture of petroleum ether/ethyl acetate = 15:1 (v/v). White solid; ^1H NMR (400 MHz, CDCl_3) δ

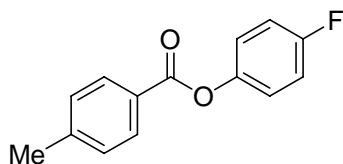
8.09 (d, $J = 8.2$ Hz, 2H), 7.30 (d, $J = 8.0$ Hz, 2H), 7.13 (d, $J = 9.1$ Hz, 2H), 6.94 (d, $J = 9.1$ Hz, 2H), 3.83 (s, 3H), 2.45 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.6, 157.2, 144.5, 144.3, 130.1, 129.2, 126.9, 122.5, 114.5, 55.6, 21.7.

These data are in good agreement with literature report.^{S13}



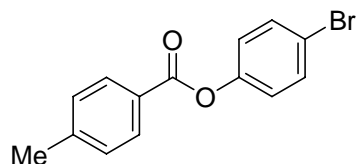
[1,1'-Biphenyl]-4-yl 4-methylbenzoate (5h); 49.6 mg, 86%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). White solid; ^1H NMR (400 MHz, CDCl_3) δ 8.14 (d, $J = 8.1$ Hz, 2H), 7.66 (d, $J = 8.5$ Hz, 2H), 7.62 (d, $J = 7.8$ Hz, 2H), 7.47 (d, $J = 7.6$ Hz, 2H), 7.41–7.29 (m, 5H), 2.48 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.3, 150.4, 144.4, 140.4, 138.9, 130.2, 129.3, 128.8, 128.2, 127.3, 127.1, 126.7, 121.9, 21.7.

These data are in good agreement with literature report.^{S14}



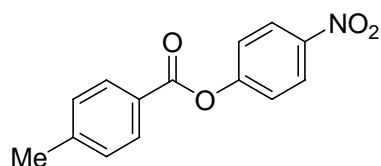
4-Fluorophenyl 4-methylbenzoate (5i); 29.4 mg, 64%). Eluted with a mixture of petroleum ether/ethyl acetate = 15:1 (v/v). White solid; ^1H NMR (400 MHz, CDCl_3) δ 8.08 (d, $J = 8.2$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 7.20–7.15 (m, 2H), 7.14–7.07 (m, 2H), 2.46 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.2, 160.2 (d, $J = 244.1$ Hz), 146.8 (d, $J = 2.9$ Hz), 144.6, 130.2, 129.3, 126.5, 123.1 (d, $J = 8.5$ Hz), 116.1 (d, $J = 23.5$ Hz), 21.7. ^{19}F NMR (376 MHz, CDCl_3) δ -117.1 (s).

These data are in good agreement with literature report.^{S15}



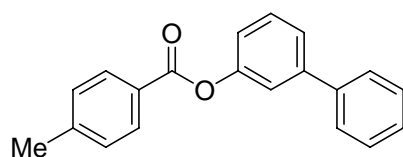
4-Bromophenyl 4-methylbenzoate (5j; 51.9 mg, 90%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). White solid; ^1H NMR (400 MHz, CDCl_3) δ 8.08 (d, $J = 8.2$ Hz, 2H), 7.54 (d, $J = 8.9$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 7.11 (d, $J = 8.9$ Hz, 2H), 2.46 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 164.9, 150.0, 144.7, 132.4, 130.2, 129.3, 126.4, 123.6, 118.8, 21.7.

These data are in good agreement with literature report.^{S16}

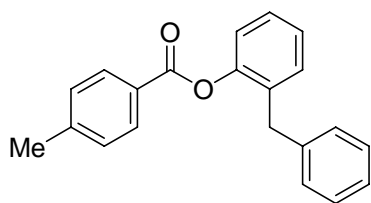


4-Nitrophenyl 4-methylbenzoate (5k; 17 mg, 32%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). White solid; ^1H NMR (400 MHz, CDCl_3) δ 8.32 (d, $J = 9.0$ Hz, 2H), 8.09 (d, $J = 8.1$ Hz, 2H), 7.41 (d, $J = 9.0$ Hz, 2H), 7.34 (d, $J = 8.1$ Hz, 2H), 2.47 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 164.3, 155.9, 145.33, 145.27, 130.4, 129.5, 125.8, 125.2, 122.6, 21.8.

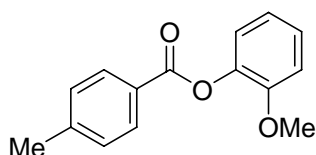
These data are in good agreement with literature report.^{S17}



[1,1'-Biphenyl]-3-yl 4-methylbenzoate (5l; 44.2 mg, 76%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 8.15 (d, $J = 8.2$ Hz, 2H), 7.65 – 7.61 (m, 2H), 7.53 – 7.43 (m, 5H), 7.40 – 7.35 (m, 1H), 7.34 (d, $J = 8.0$ Hz, 2H), 7.25 – 7.20 (m, 1H), 2.48 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.2, 151.4, 144.4, 142.8, 140.2, 130.2, 129.7, 129.3, 128.8, 127.6, 127.2, 126.8, 124.5, 120.51, 120.48, 21.7. HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{17}\text{O}_2^+$ ($\text{M}+\text{H}$)⁺ 289.1229, found 289.1223.

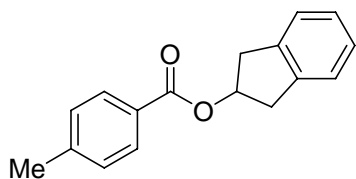


2-Benzylphenyl 4-methylbenzoate (5m; 34.2 mg, 57%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 8.02 (d, $J = 8.2$ Hz, 2H), 7.32 – 7.28 (m, 3H), 7.25 – 7.14 (m, 8H), 3.97 (s, 2H), 2.46 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 164.9, 149.2, 144.4, 139.8, 133.2, 130.9, 130.2, 129.2, 128.9, 128.4, 127.5, 126.6, 126.09, 126.08, 122.6, 36.4, 21.7. HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{19}\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$ 303.1385, found 303.1378.



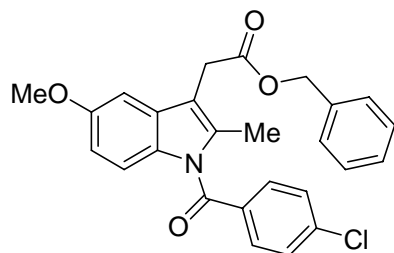
2-Methoxyphenyl 4-methylbenzoate (5n; 18.2 mg, 38%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 8.11 (d, $J = 8.2$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 7.25 – 7.21 (m, 1H), 7.15 (dd, $J = 7.8, 1.6$ Hz, 1H), 7.03 – 6.96 (m, 2H), 3.82 (s, 3H), 2.45 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 164.8, 151.4, 144.2, 140.1, 130.3, 129.2, 126.8, 126.7, 123.0, 120.8, 112.5, 55.9, 21.7.

These data are in good agreement with literature report.^{S14}



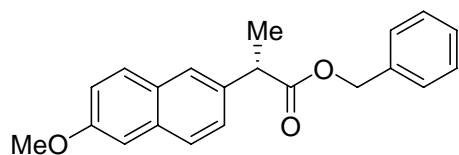
2,3-Dihydro-1H-inden-2-yl 4-methylbenzoate (5o; 33 mg, 65%). Eluted with petroleum ether/ethyl acetate = 10:1 (v/v). Yellow solid; ^1H NMR (400 MHz, CDCl_3) δ 7.95 (d, $J = 8.1$ Hz, 2H), 7.36–7.21 (m, 6H), 5.82 (tt, $J = 6.6, 3.3$ Hz, 1H), 3.49 (dd, $J = 17.0, 6.6$ Hz, 2H), 3.22 (dd, $J = 16.9, 3.2$ Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 166.6, 143.5, 140.5, 129.6, 128.9, 127.7, 126.7, 124.6, 75.6, 39.7, 21.6. HRMS calcd for $\text{C}_{17}\text{H}_{17}\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$ 253.1229; found 253.1224.

3. Late-stage functionalization of biologically active acids or alcohols



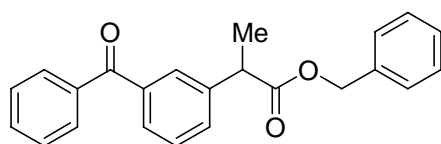
Benzyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetate (6a; 47.7 mg, 53%). Eluted with a mixture of petroleum ether/ethyl acetate = 5:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.65 (d, $J = 8.6$ Hz, 2H), 7.46 (d, $J = 8.6$ Hz, 2H), 7.35 – 7.29 (m, 5H), 6.94 (d, $J = 2.5$ Hz, 1H), 6.89 (d, $J = 9.0$ Hz, 1H), 6.68 (dd, $J = 9.0, 2.5$ Hz, 1H), 5.15 (s, 2H), 3.76 (s, 3H), 3.72 (s, 2H), 2.37 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.6, 168.2, 156.0, 139.2, 135.9, 135.7, 133.9, 131.1, 130.8, 130.6, 129.1, 128.5, 128.3, 128.1, 114.9, 112.5, 111.8, 101.2, 66.8, 55.6, 30.4, 13.3.

These data are in good agreement with literature report.^{S18}



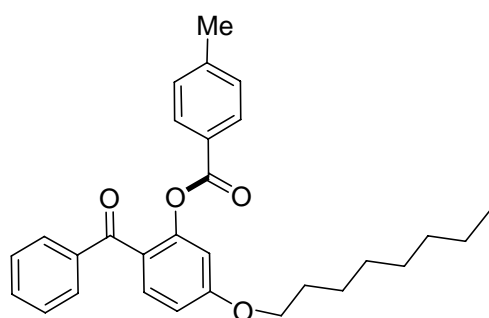
Benzyl 2-(6-methoxynaphthalen-2-yl)propanoate (6b; 52.3 mg, 82%). Eluted with a mixture of petroleum ether/ethyl acetate = 8:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.64 – 7.57 (m, 3H), 7.33 (dd, $J = 8.5, 1.8$ Hz, 1H), 7.22 – 7.14 (m, 5H), 7.09 – 7.02 (m, 2H), 5.05 (q, $J = 12.5$ Hz, 2H), 3.88 – 3.80 (m, 4H), 1.52 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 174.4, 157.6, 136.0, 135.5, 133.7, 129.3, 128.9, 128.4, 128.0, 127.9, 127.1, 126.3, 126.0, 118.9, 105.6, 66.4, 55.3, 45.4, 18.5.

These data are in good agreement with literature report.^{S19}

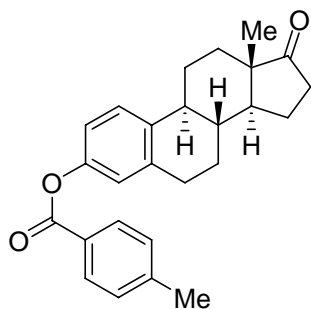


Benzyl 2-(3-benzoylphenyl)propanoate (6c; 58.6 mg, 85%). Eluted with a mixture of petroleum ether/ethyl acetate = 10:1 (v/v). Yellow oil; ^1H NMR (400 MHz, CDCl_3) δ 7.81 – 7.74 (m, 3H), 7.69 (d, $J = 7.7$ Hz, 1H), 7.59 (t, $J = 7.4$ Hz, 1H), 7.55 (d, $J = 7.8$ Hz, 1H), 7.47 (t, $J = 7.6$ Hz, 2H), 7.44 (t, $J = 7.7$ Hz, 1H), 7.32 – 7.28 (m, 3H), 7.27 – 7.23 (m, 2H), 5.13 (m, 2H), 3.86 (q, $J = 7.2$ Hz, 1H), 1.57 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 196.4, 173.8, 140.6, 137.9, 137.4, 135.7, 132.4, 131.5, 130.0, 129.2, 128.9, 128.5, 128.4, 128.2, 128.1, 127.9, 66.6, 45.4, 18.3.

These data are in good agreement with literature report.^{S20}



2-Benzoyl-5-(octyloxy)phenyl 4-methylbenzoate (6d; 40.3 mg, 45%). Eluted with a mixture of petroleum ether/ethyl acetate = 20:1 (v/v). Colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.77 – 7.69 (m, 4H), 7.57 (d, $J = 8.4$ Hz, 1H), 7.43 (t, $J = 7.4$ Hz, 1H), 7.35 (t, $J = 7.4$ Hz, 2H), 7.15 (d, $J = 8.0$ Hz, 2H), 6.89 - 6.83 (m, 2H), 4.03 (t, $J = 6.5$ Hz, 2H), 2.38 (s, 3H), 1.87–1.75 (m, 2H), 1.51–1.26 (m, 10H), 0.90 (t, $J = 6.9$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 194.2, 164.7, 162.6, 151.0, 144.2, 138.6, 132.5, 132.3, 130.0, 129.5, 129.0, 128.1, 126.2, 123.8, 111.9, 109.3, 68.5, 31.8, 29.3, 29.2, 29.0, 25.9, 22.6, 21.6, 14.1. HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{33}\text{O}_4^+$ ($\text{M}+\text{H}$) $^+$ 445.2379, found 445.2373.



(8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthren-3-yl 4-methylbenzoate (6e; 47.5 mg, 71%). Eluted with petroleum ether/ethyl acetate = 5:1 (v/v). White solid; ¹H NMR (400 MHz, CDCl₃) δ 8.08 (d, *J* = 8.1 Hz, 2H), 7.37 – 7.27 (m, 3H), 6.98 (dd, *J* = 8.5, 2.1 Hz, 1H), 6.94 (d, *J* = 2.0 Hz, 1H), 2.97 – 2.90 (m, 2H), 2.58 - 2.40 (m, 5H), 2.31 (td, *J* = 10.7, 3.0 Hz, 1H), 2.21 – 1.94 (m, 4H), 1.70 - 1.41 (m, 6H), 0.93 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 220.8, 165.4, 148.8, 144.2, 137.9, 137.2, 130.1, 129.2, 126.8, 126.3, 121.7, 118.8, 50.4, 47.9, 44.1, 37.9, 35.8, 31.5, 29.3, 26.3, 25.7, 21.7, 21.5, 13.8. HRMS (ESI) *m/z* calcd for C₂₆H₂₉O₃⁺ (M+H)⁺ 389.2117, found 389.2106.

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4. Radical trapping experiment

Radical trapping experiment was performed in the presence of three equivalents of TEMPO. The yield of **4a** was isolated yield after column chromatography, and TEMPO-CF₃ adduct was observed by ¹⁹F NMR analysis of the crude reaction mixture.

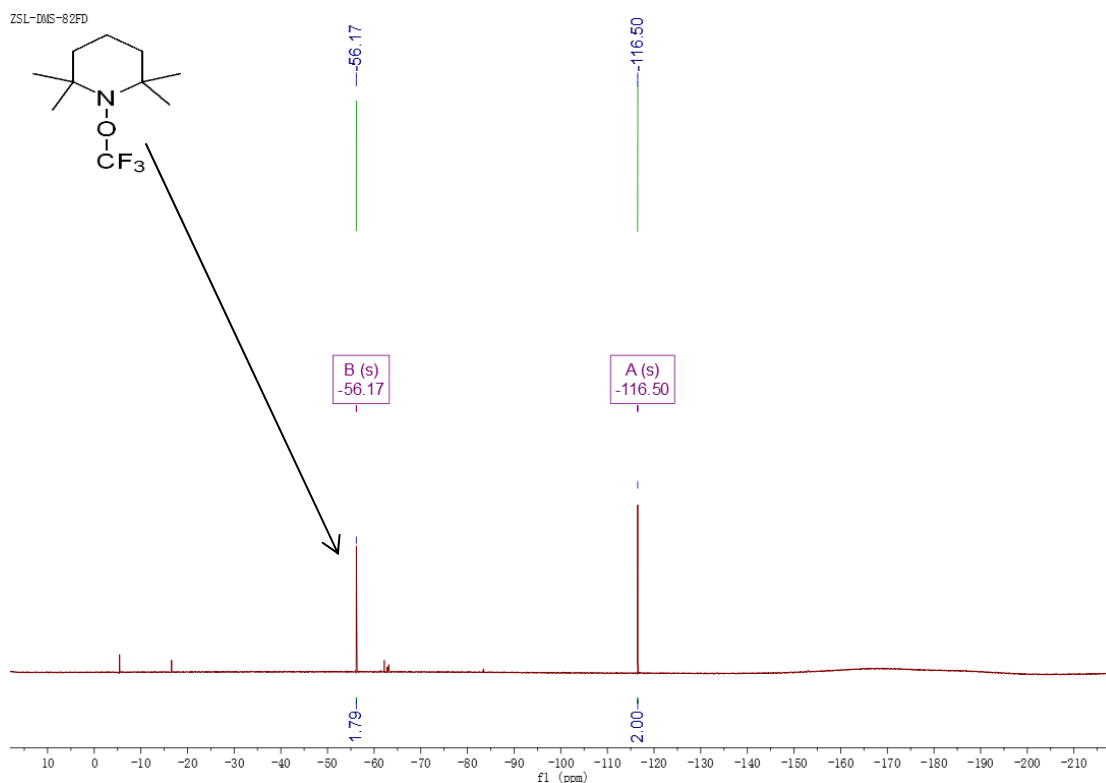
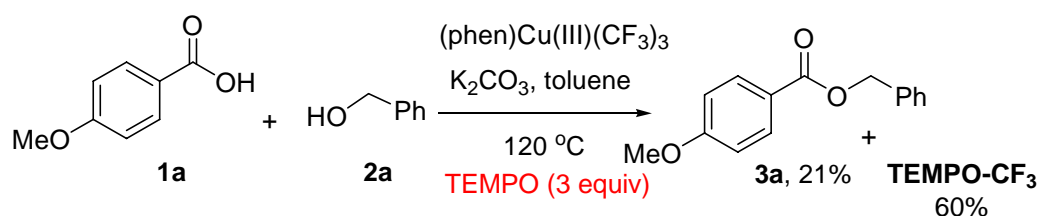


Figure S1. ¹⁹F NMR spectrum of the reaction crude mixture with radical scavenger TEMPO.

5. ^{19}F NMR monitoring of reaction course of **2a** in the absence of alcohols

The reaction course of **2a** and $(\text{phen})\text{Cu}(\text{III})(\text{CF}_3)_3$ under the optimized conditions albeit without alcohols was monitored by ^{19}F NMR analysis of the crude mixtures at a reaction time of 10 mins, 0.5, 1, 2, 3 and 6 hours.

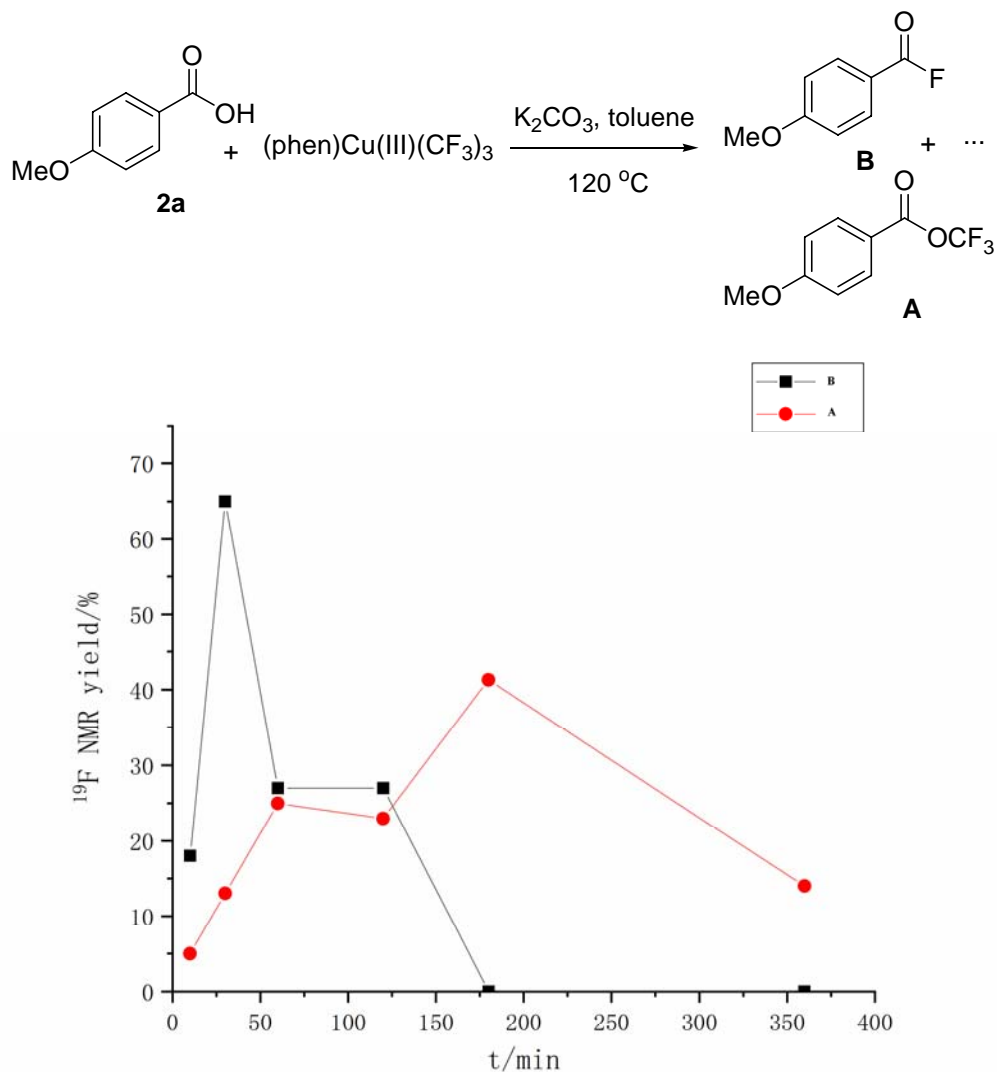
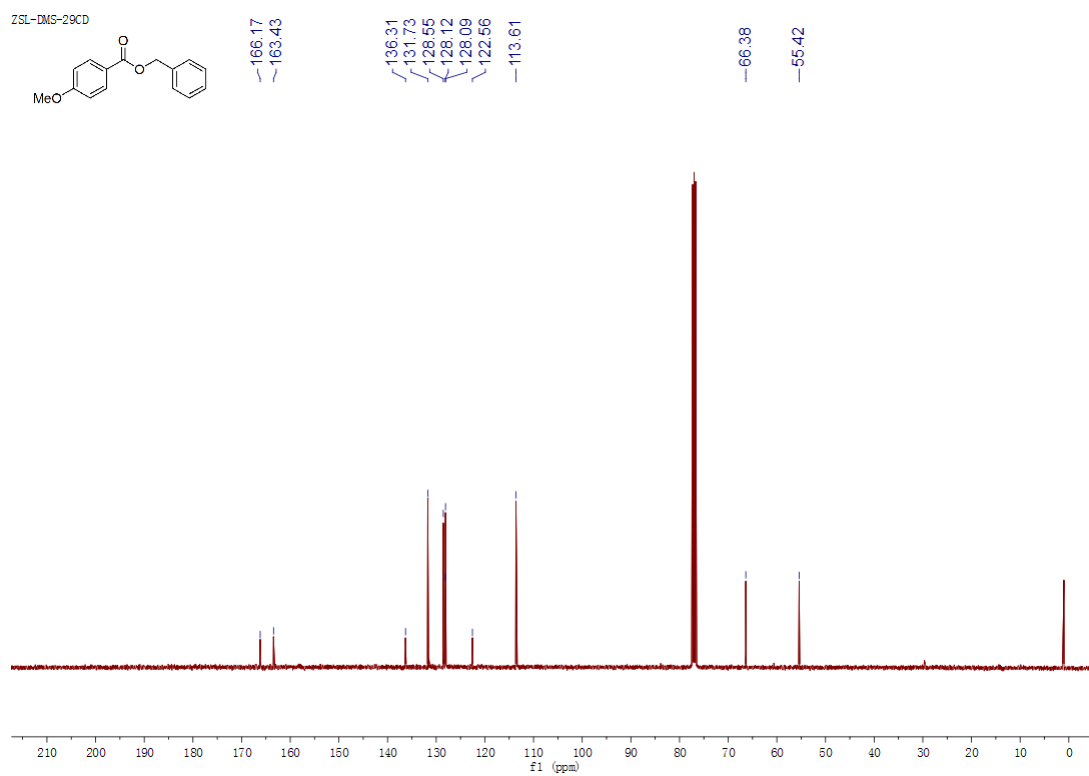
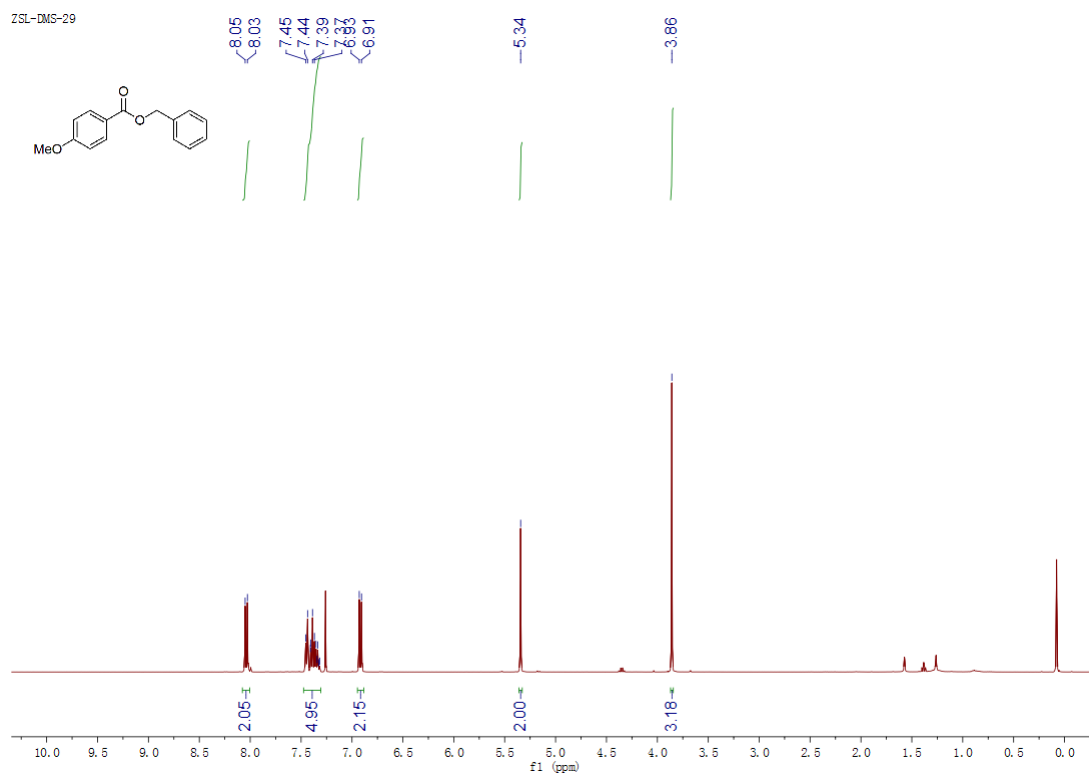


Figure S2. ^{19}F NMR monitoring of the crude reaction mixtures in 10 mins, 0.5, 1, 2, 3 and 6 hours.

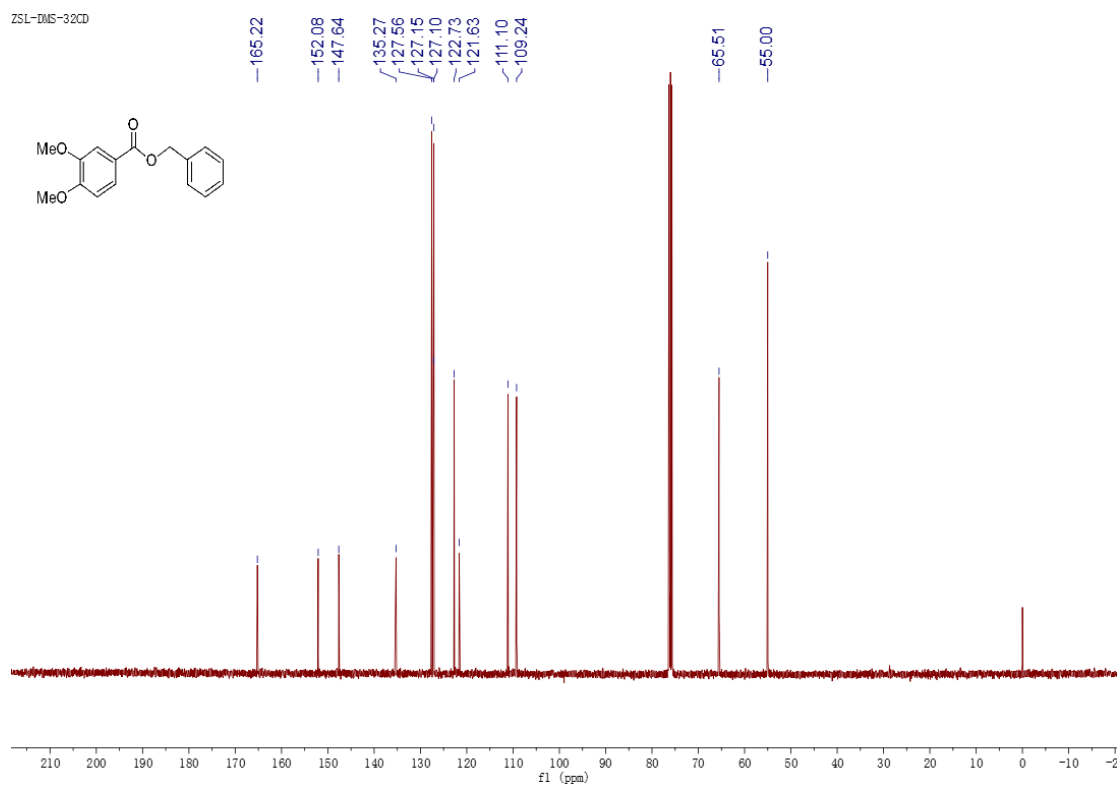
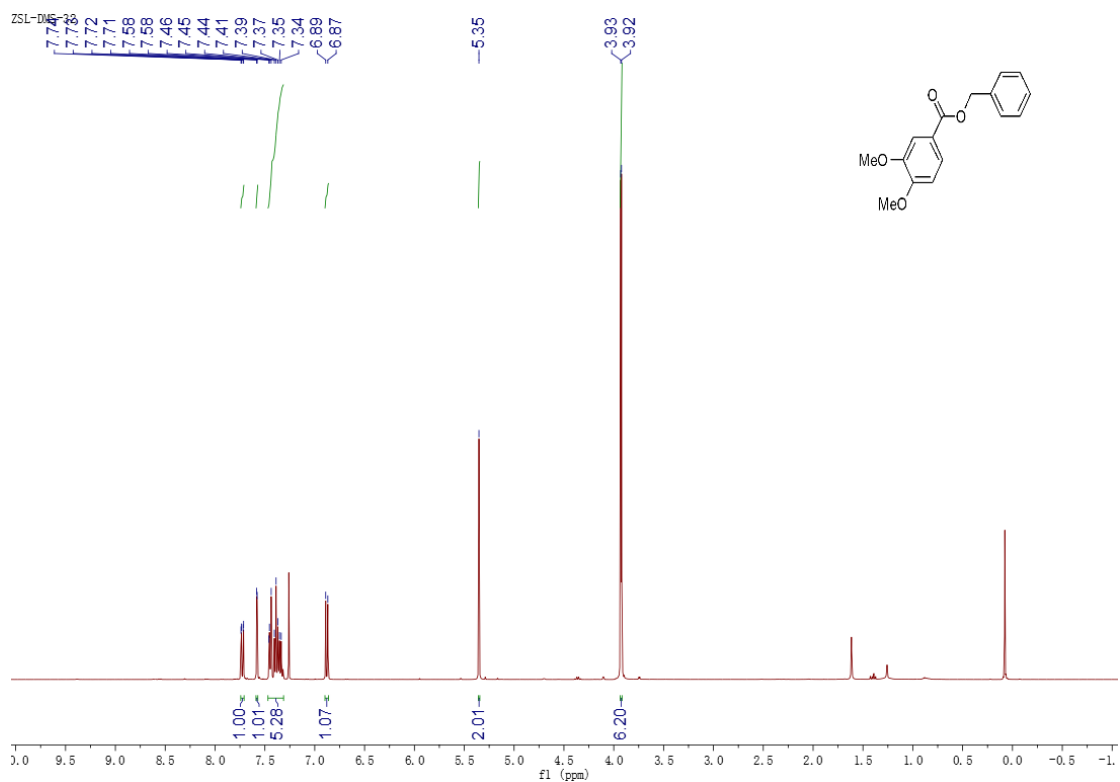
Into the above crude mixture at a reaction time of 1 hour was added benzyl alcohol, and the mixture was then reacted for 12 hours. ^{19}F NMR analysis of the reaction mixture showed the complete disappearance of signals corresponding to **A** and **B**. TLC indicated the formation of ester **4a**, which was isolated in 50% yield.

6. Copies of NMR spectra for all of the products

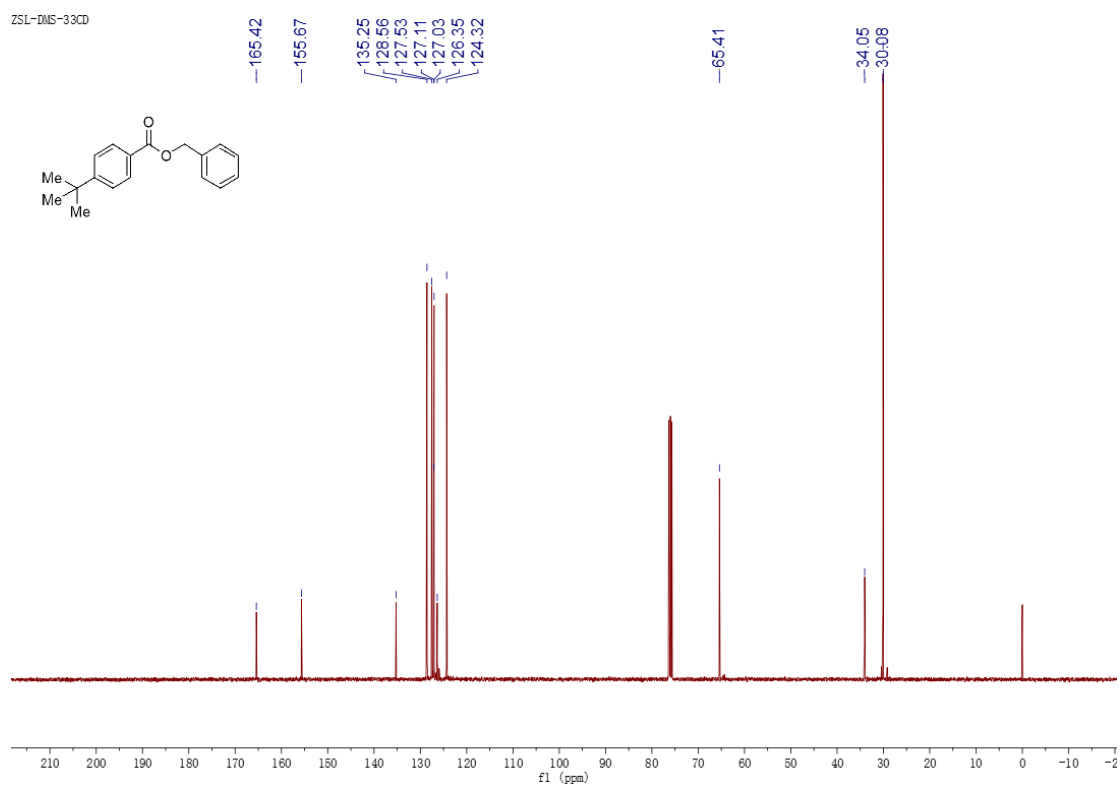
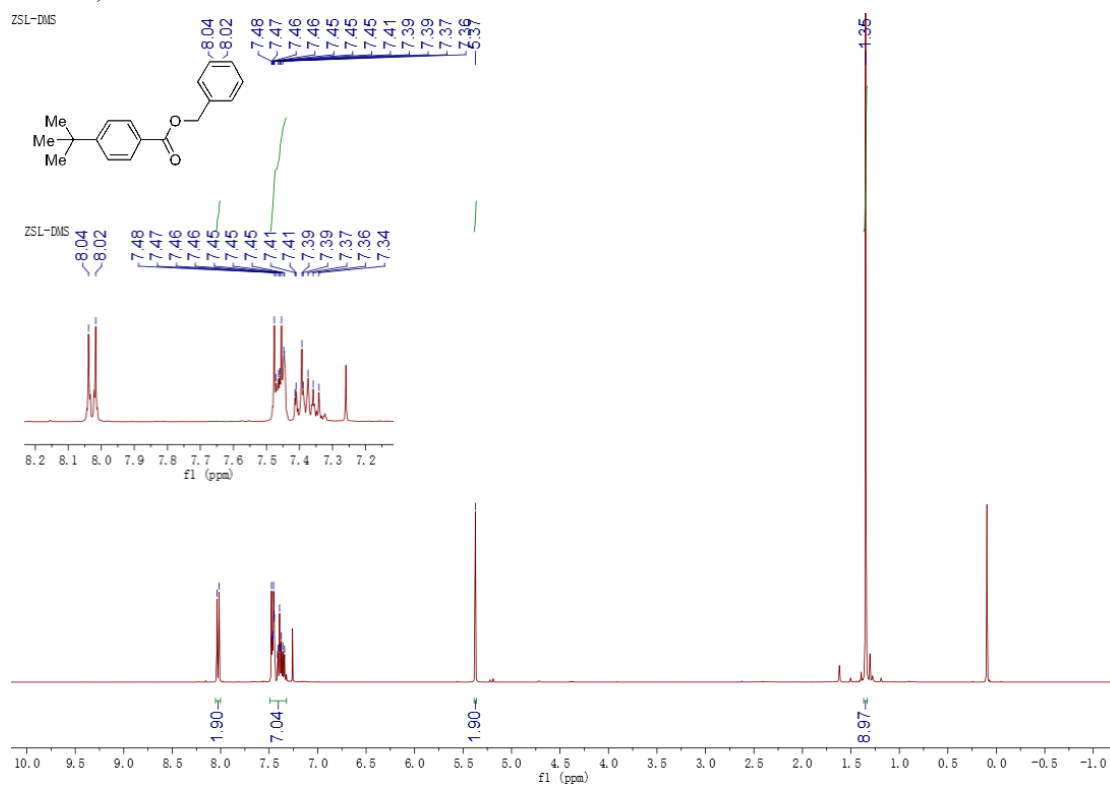
Benzyl 4-methoxybenzoate (4a). ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3



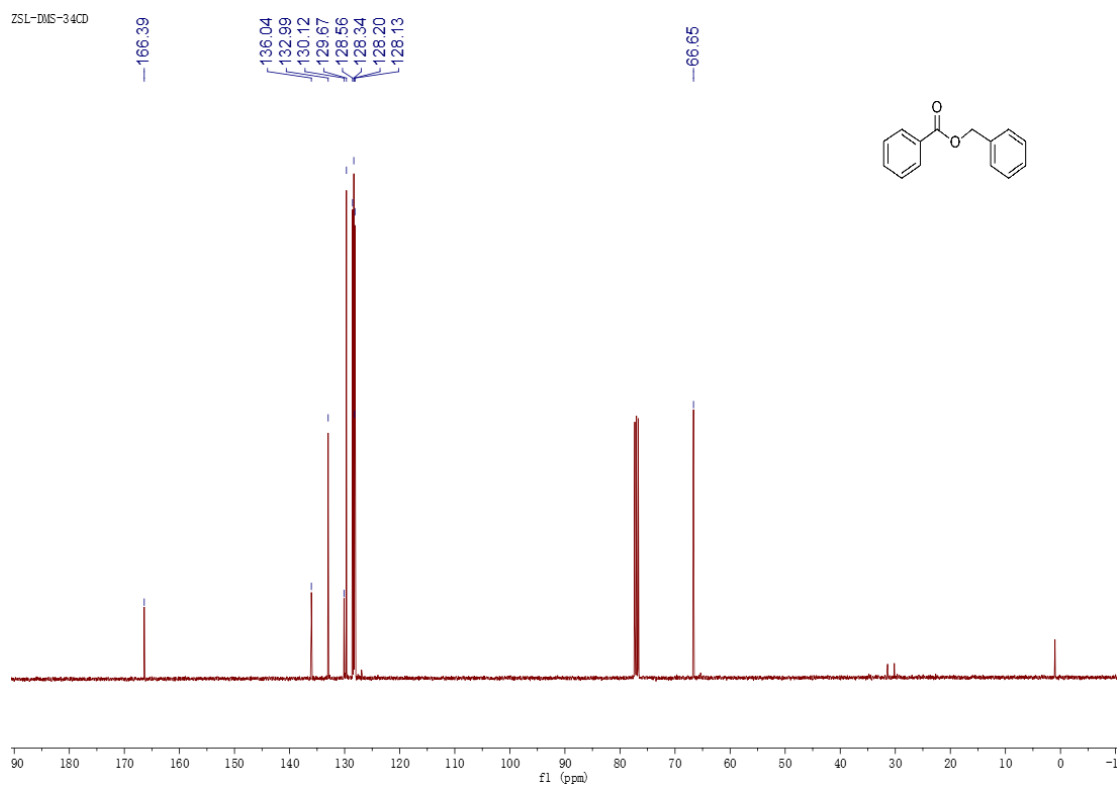
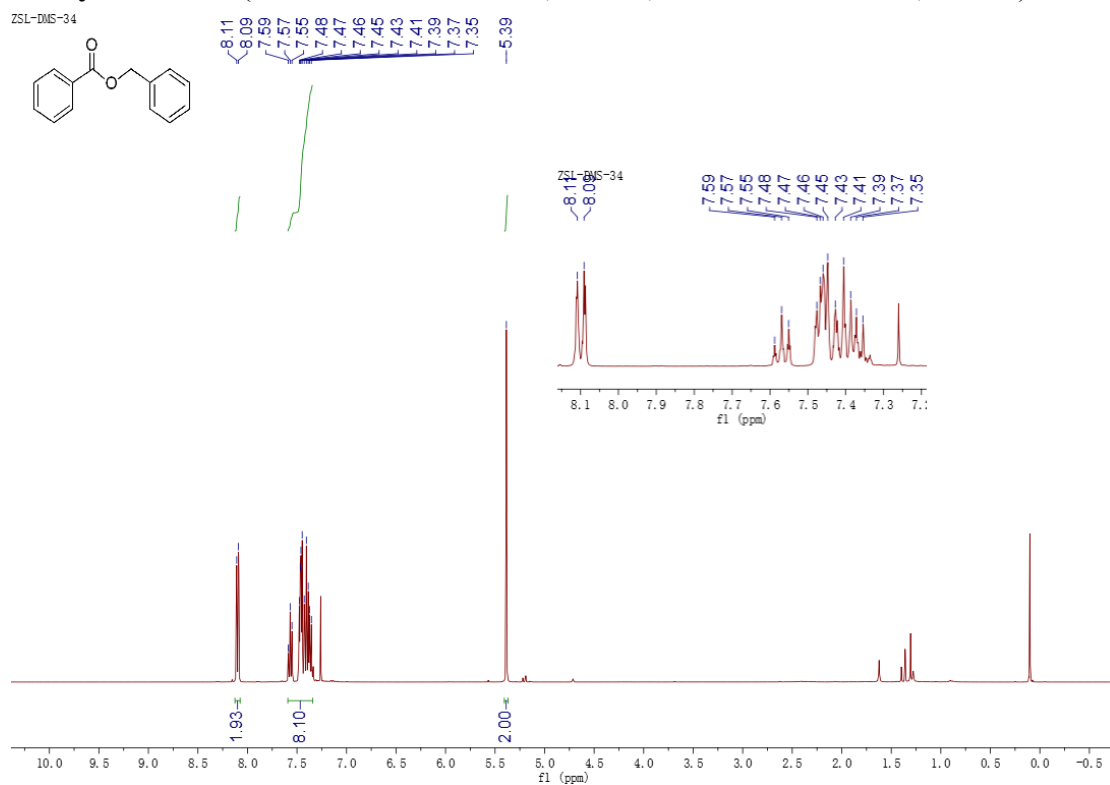
Benzyl 3,4-dimethoxybenzoate (4b). ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3



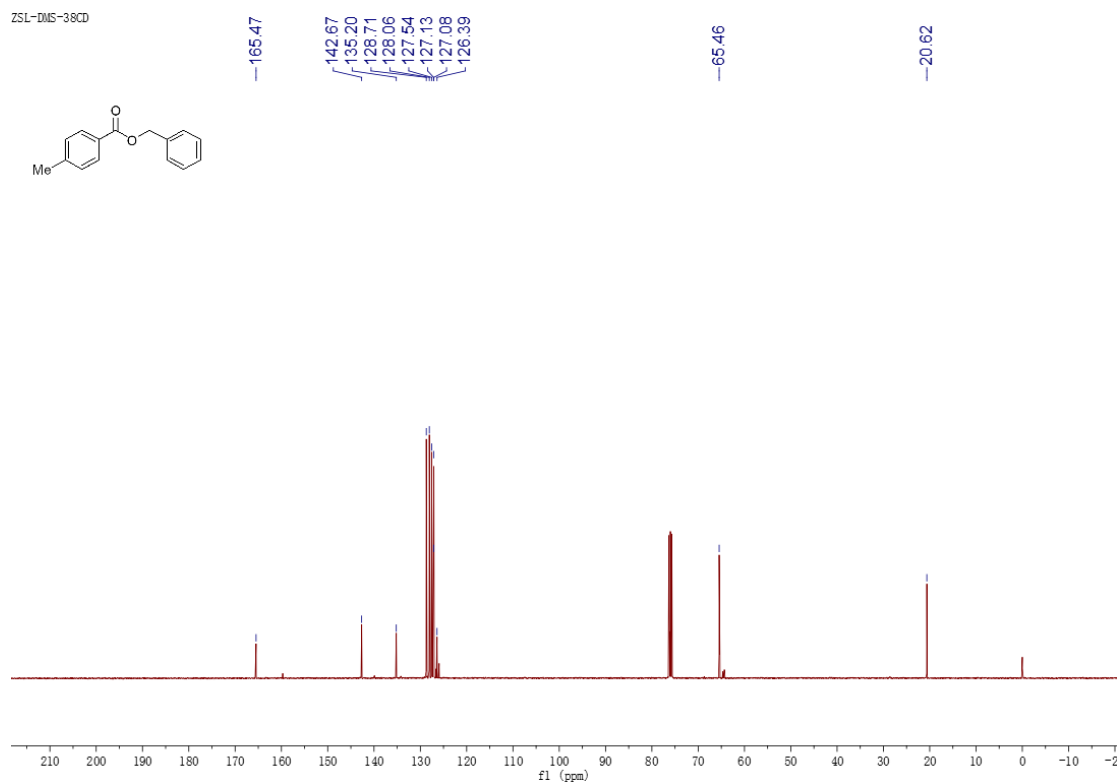
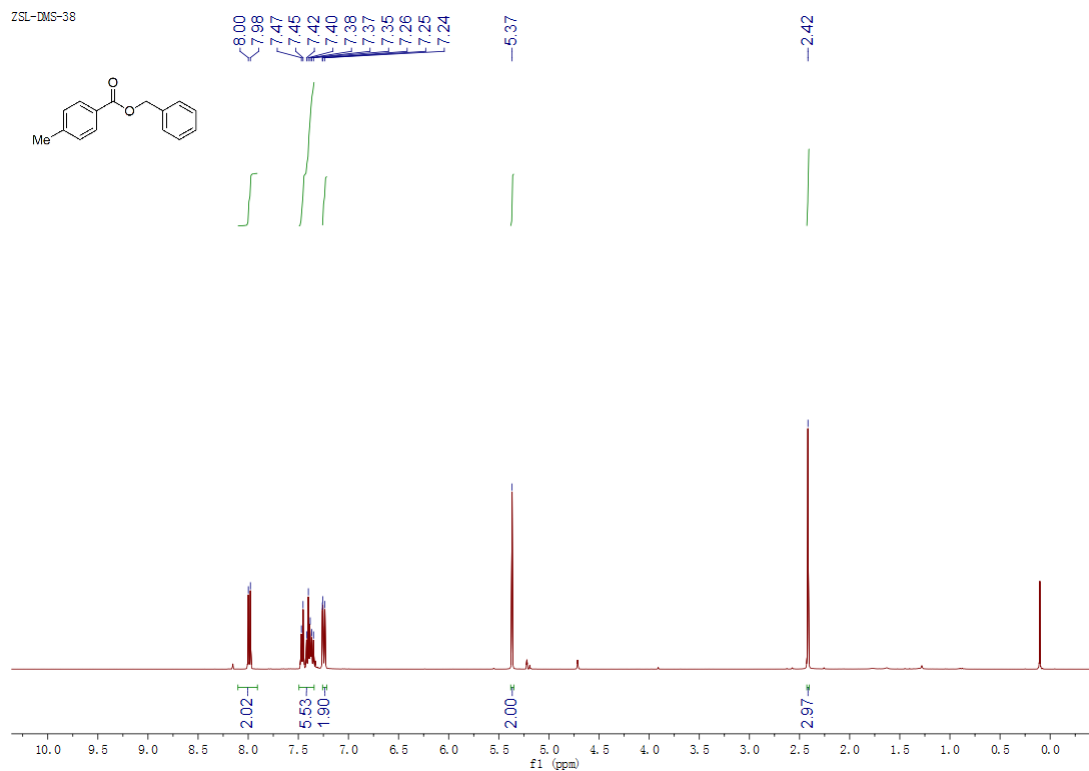
Benzyl 4-(tert-butyl)benzoate (4c. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)



Benzyl benzoate (4d). ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3

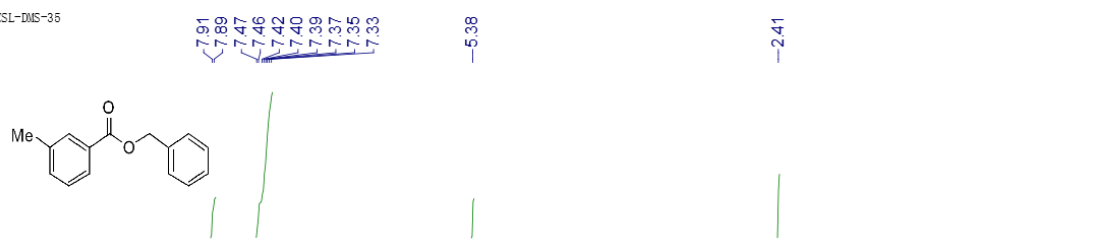


Benzyl 4-methylbenzoate (4e. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

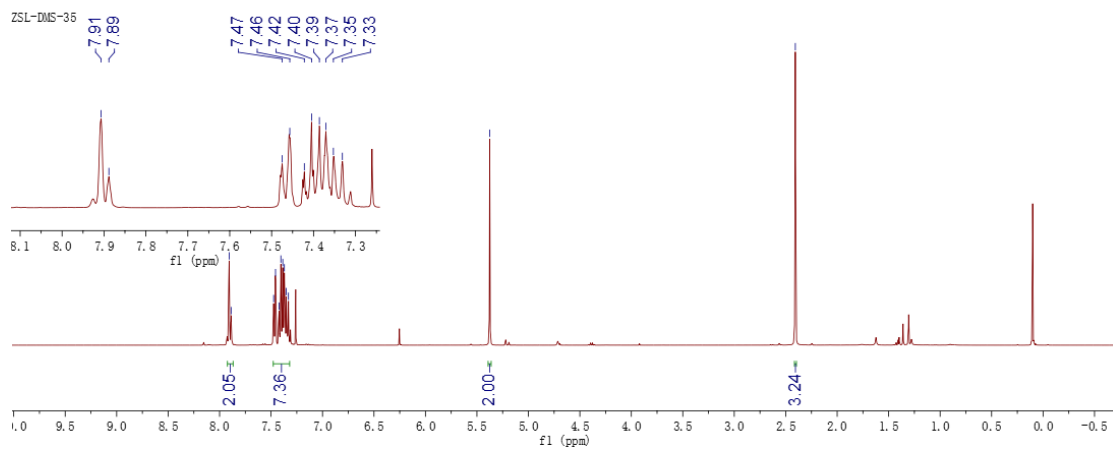


Benzyl 3-methylbenzoate (4f. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

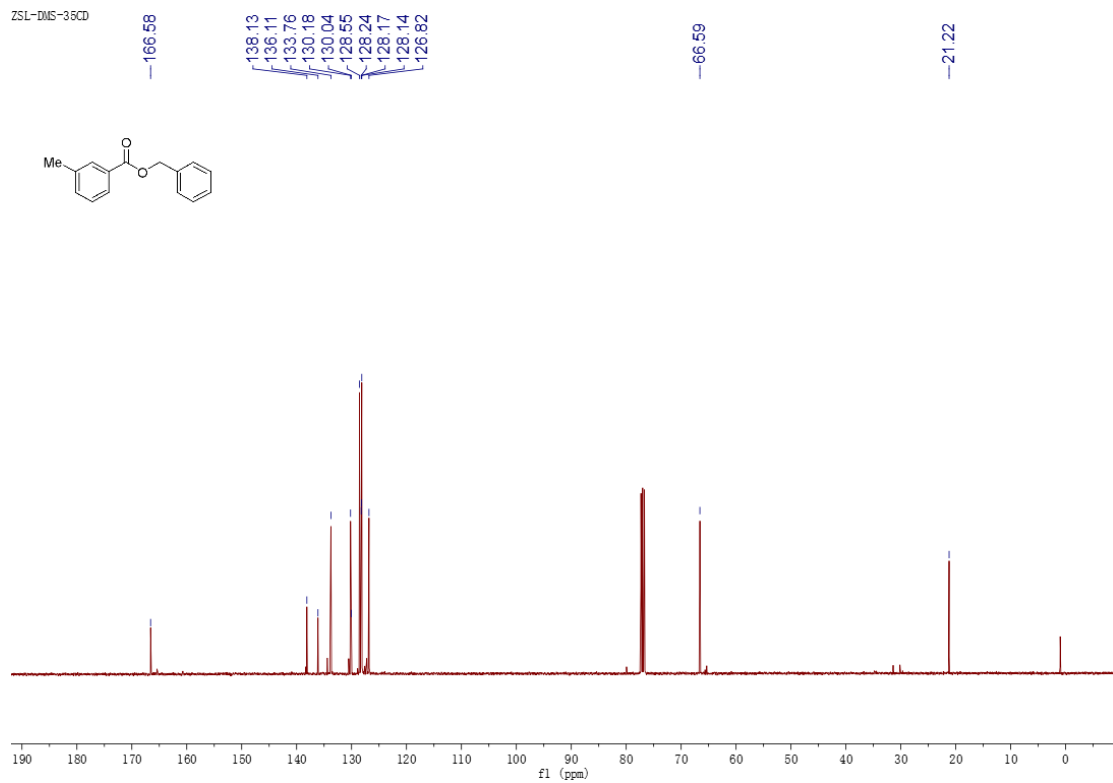
ZSL-DMS-35



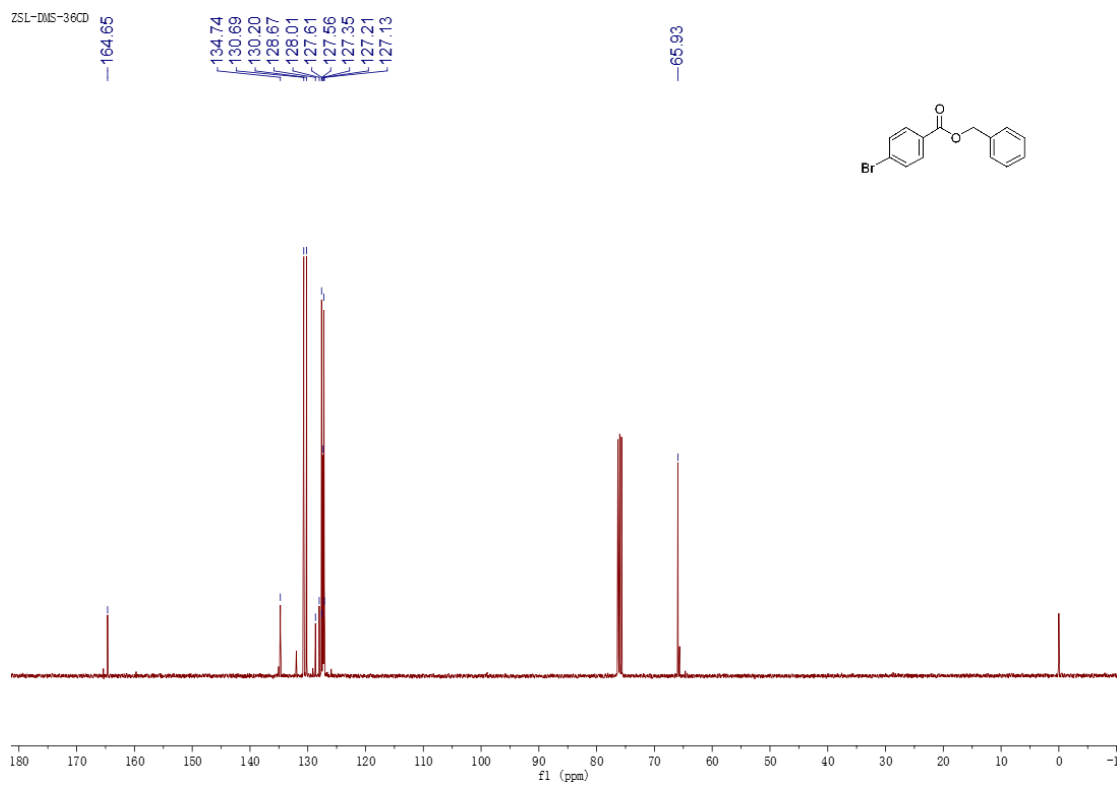
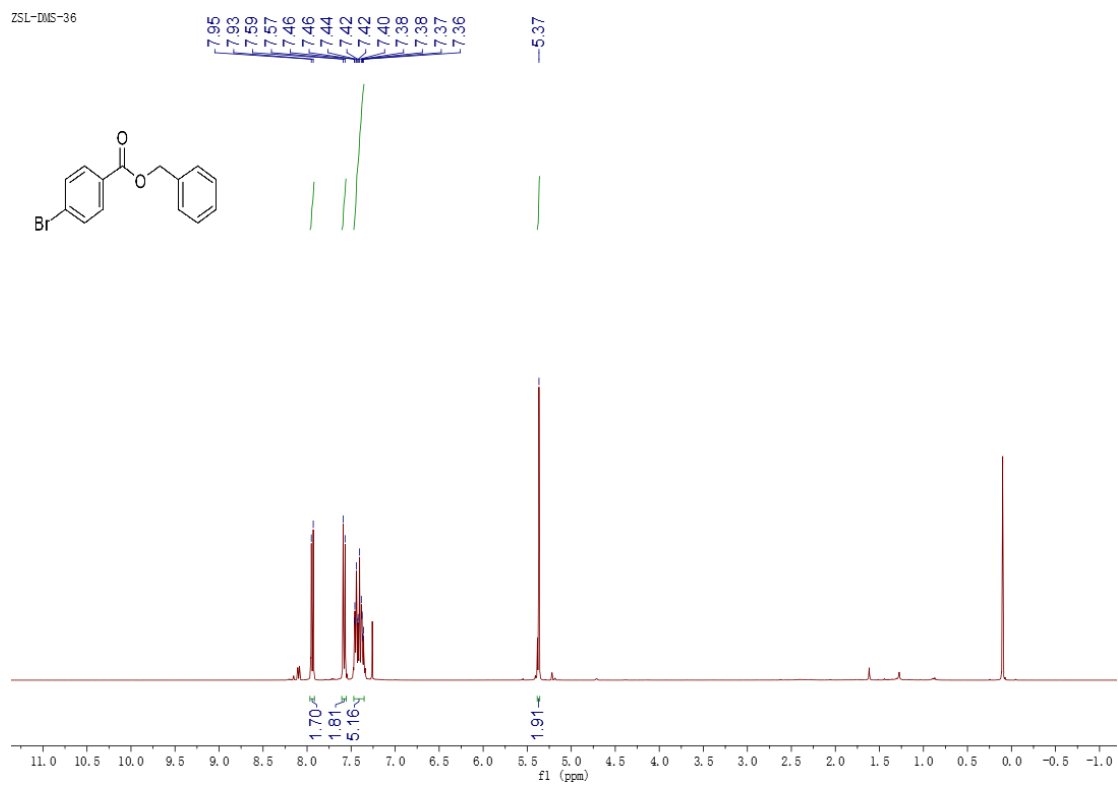
ZSL-DMS-35



ZSL-DMS-35CD

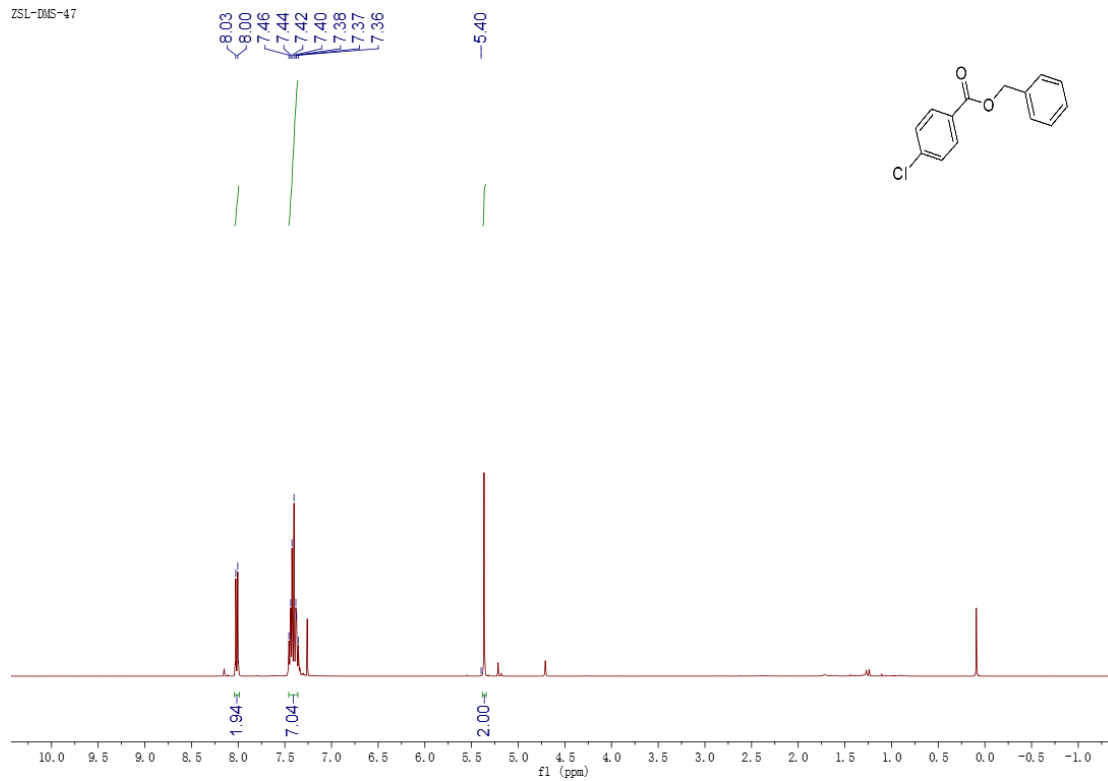


Benzyl 4-bromobenzoate (4g. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

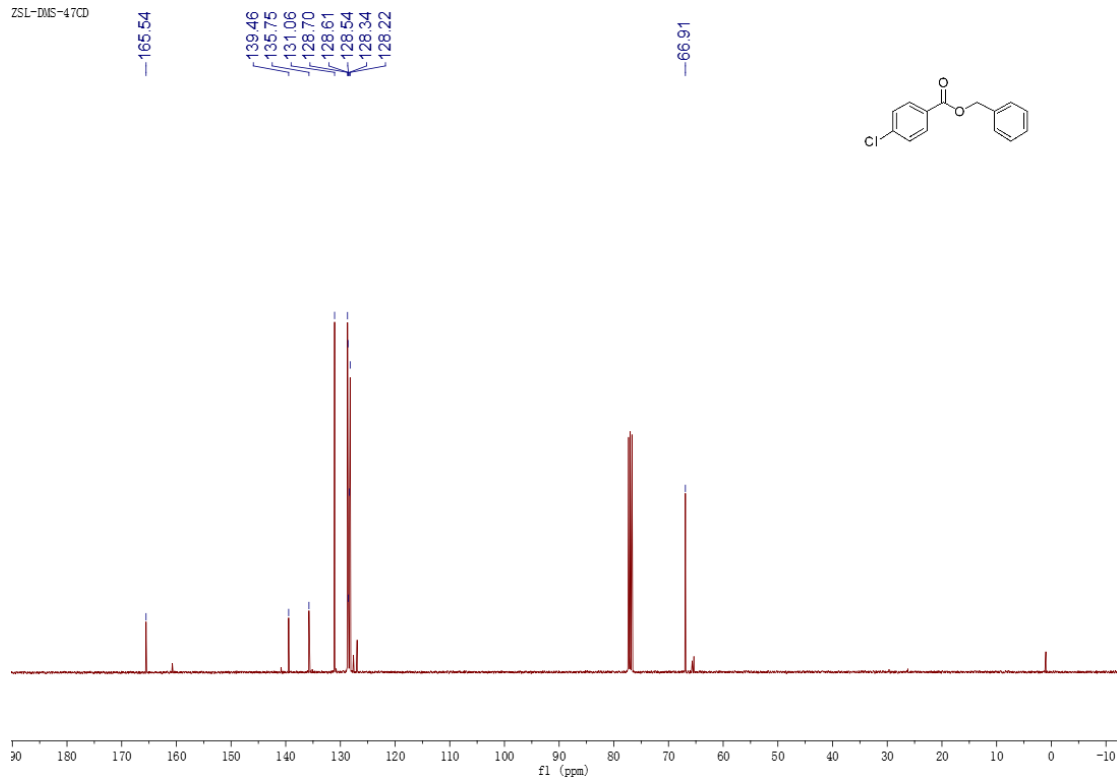


Benzyl 4-chlorobenzoate (4h. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

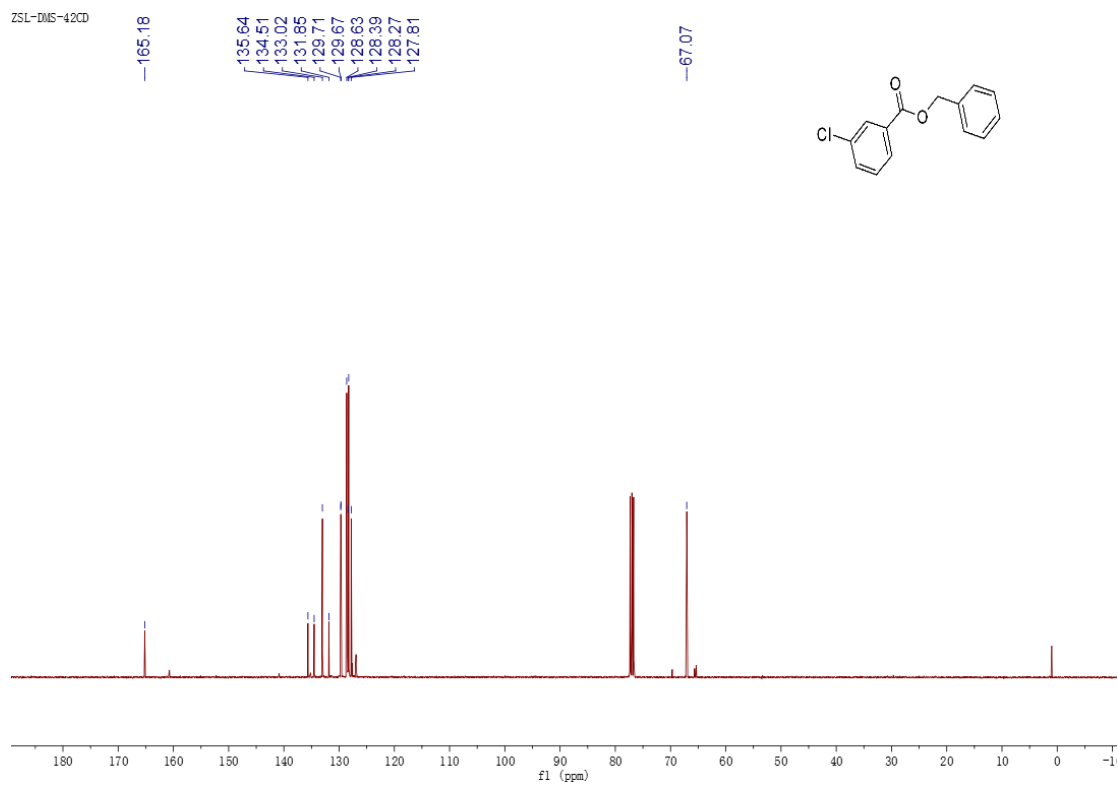
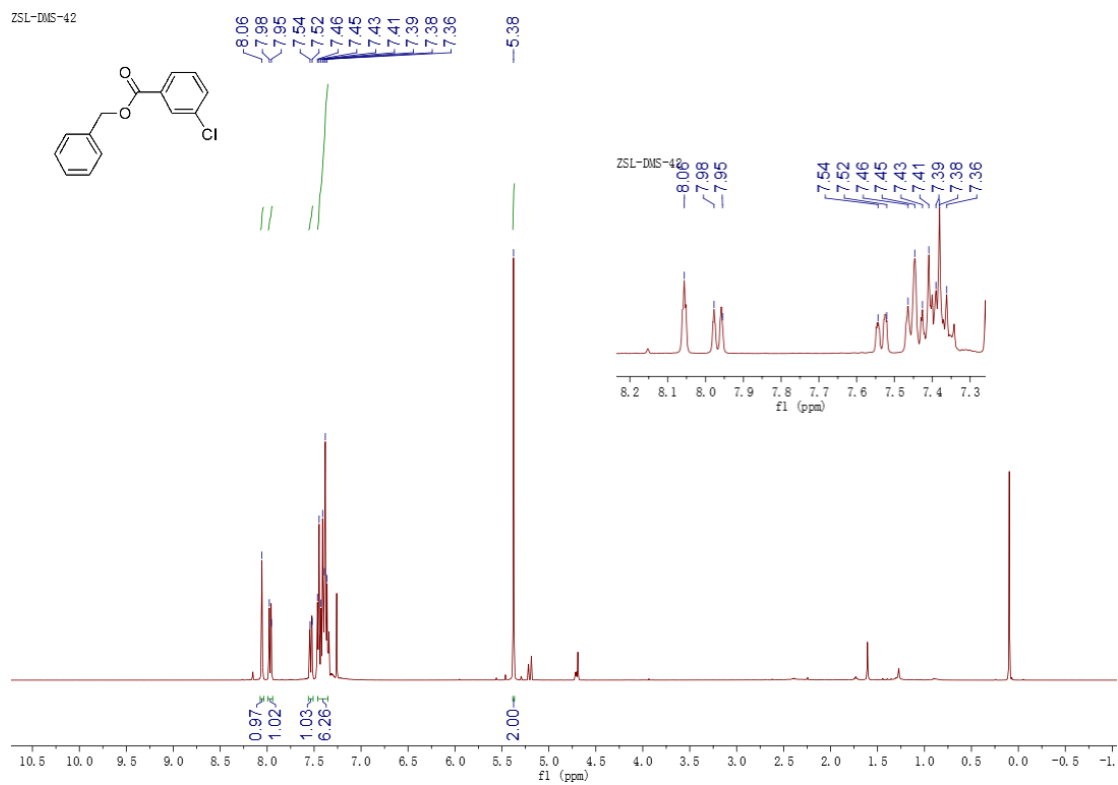
ZSL-DMS-47



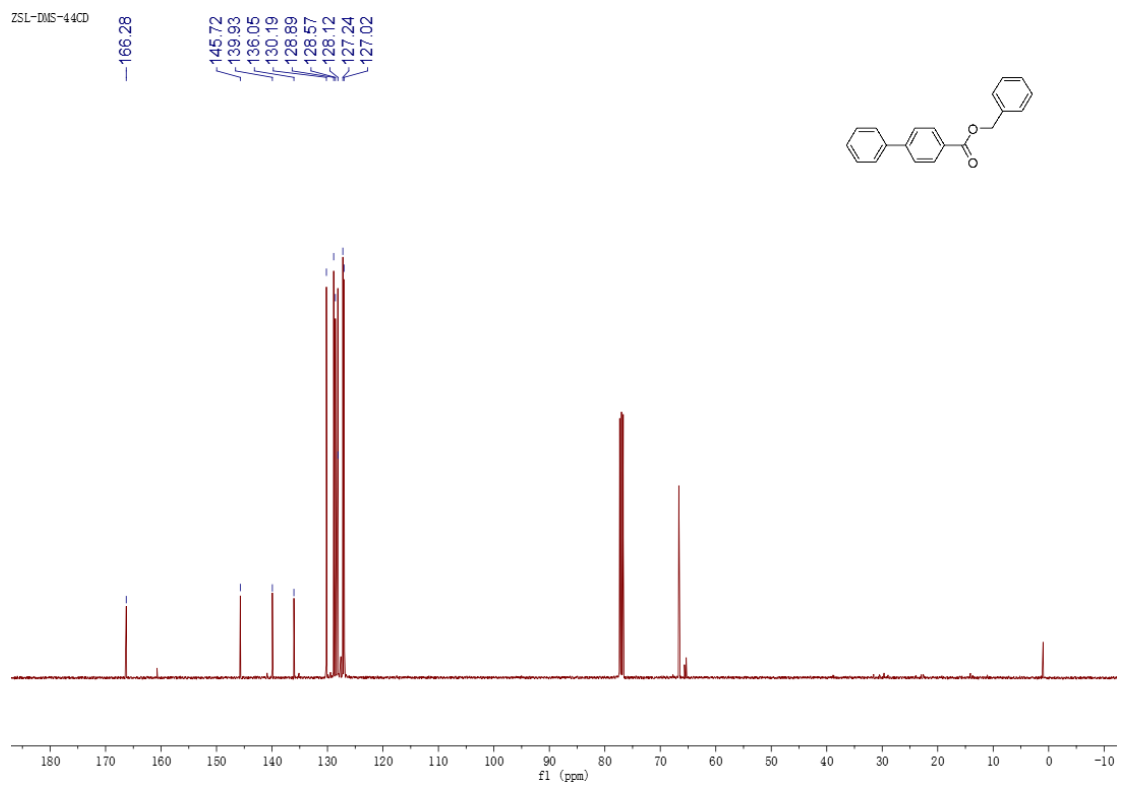
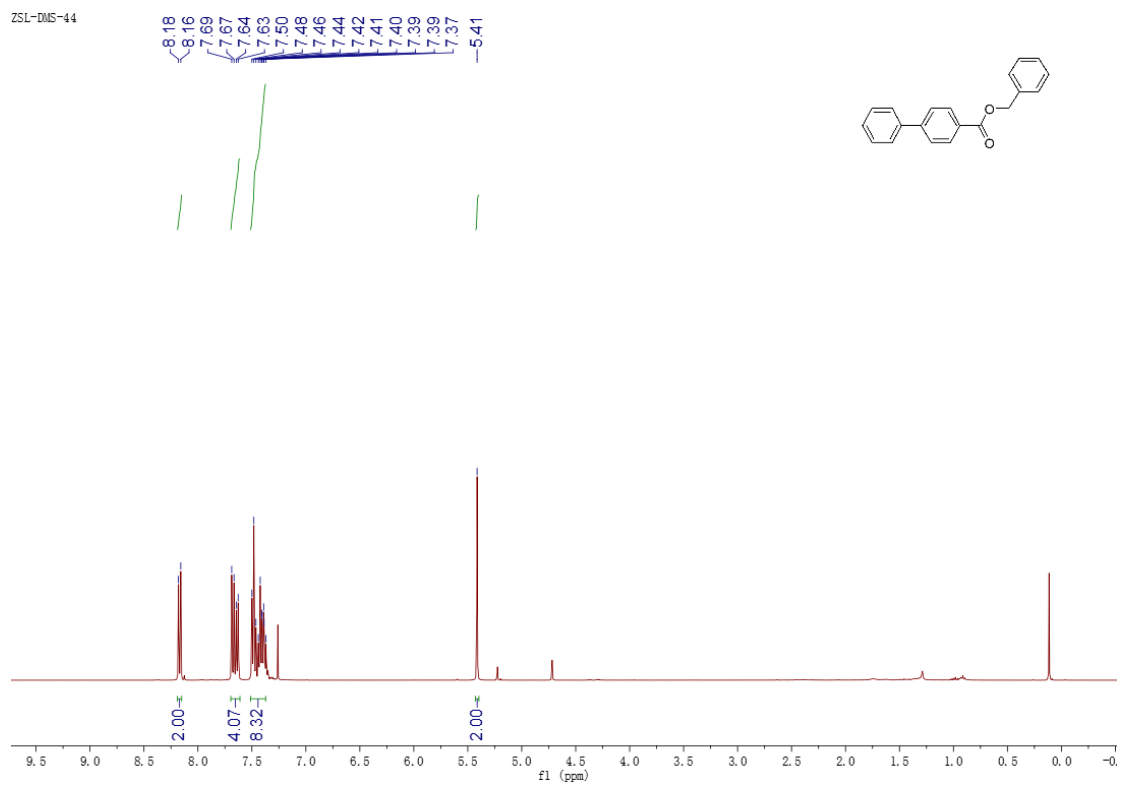
ZSL-DMS-47CD



Benzyl 3-chlorobenzoate (4i. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

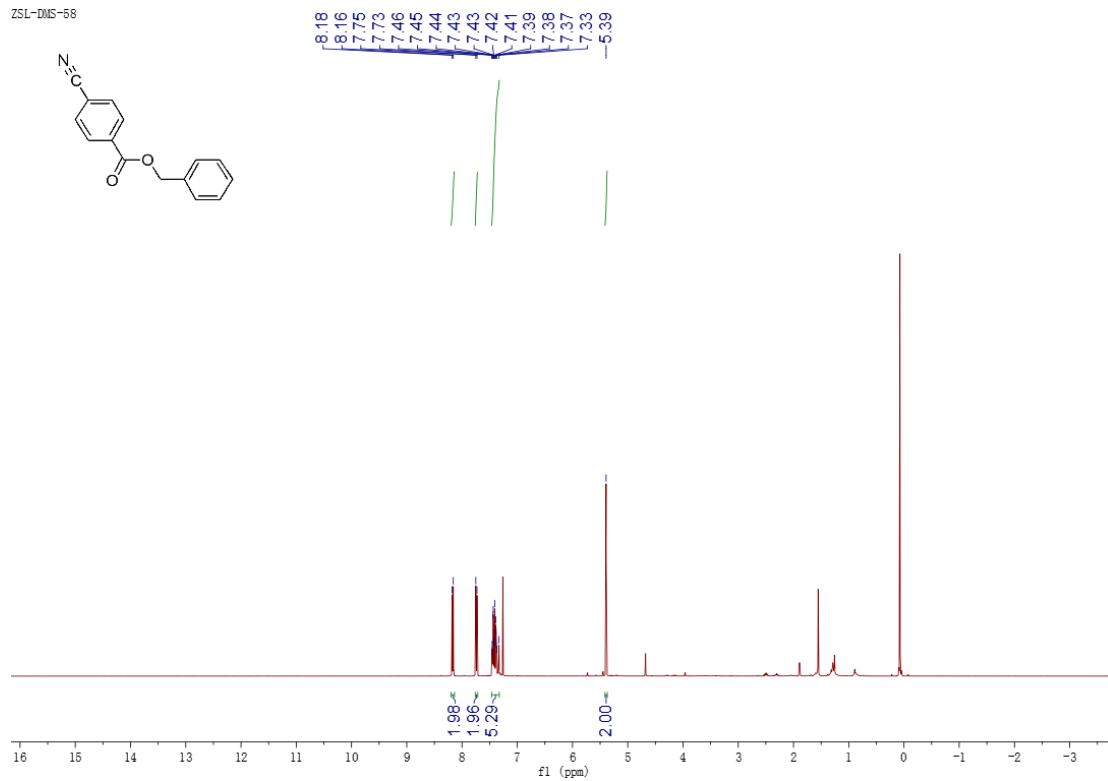


Benzyl [1,1'-biphenyl]-4-carboxylate (4j). ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3

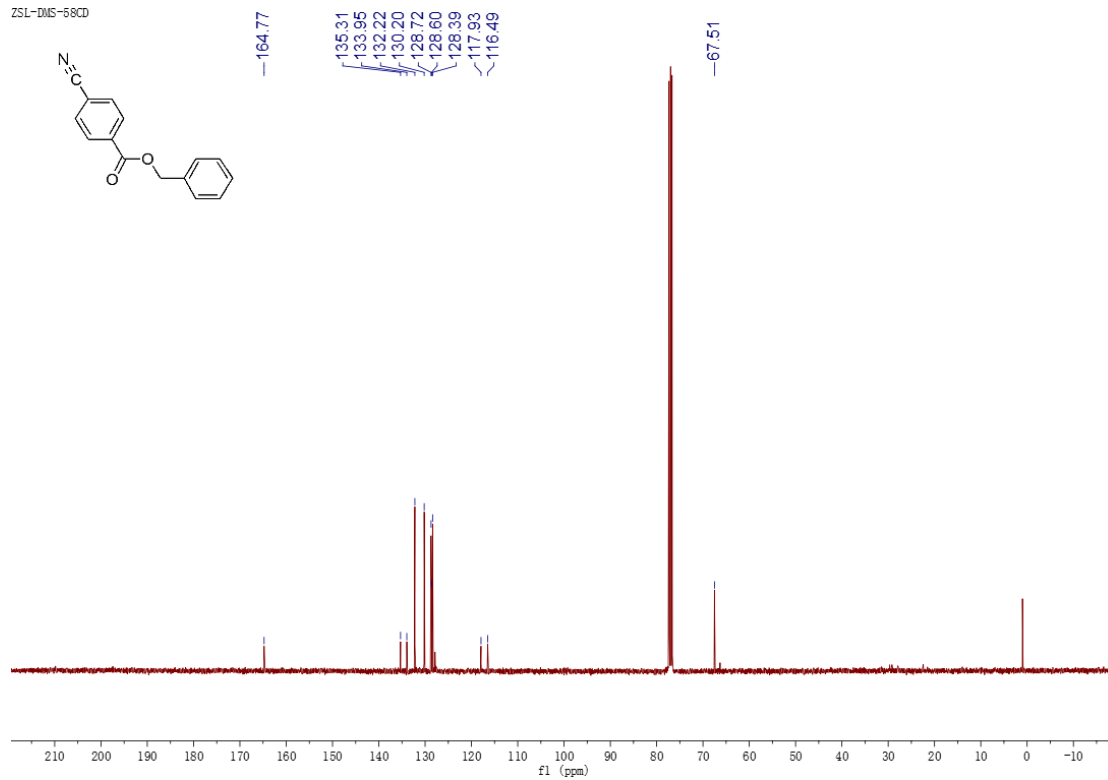


Benzyl 4-cyanobenzoate (4k. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

ZSL-DMS-58

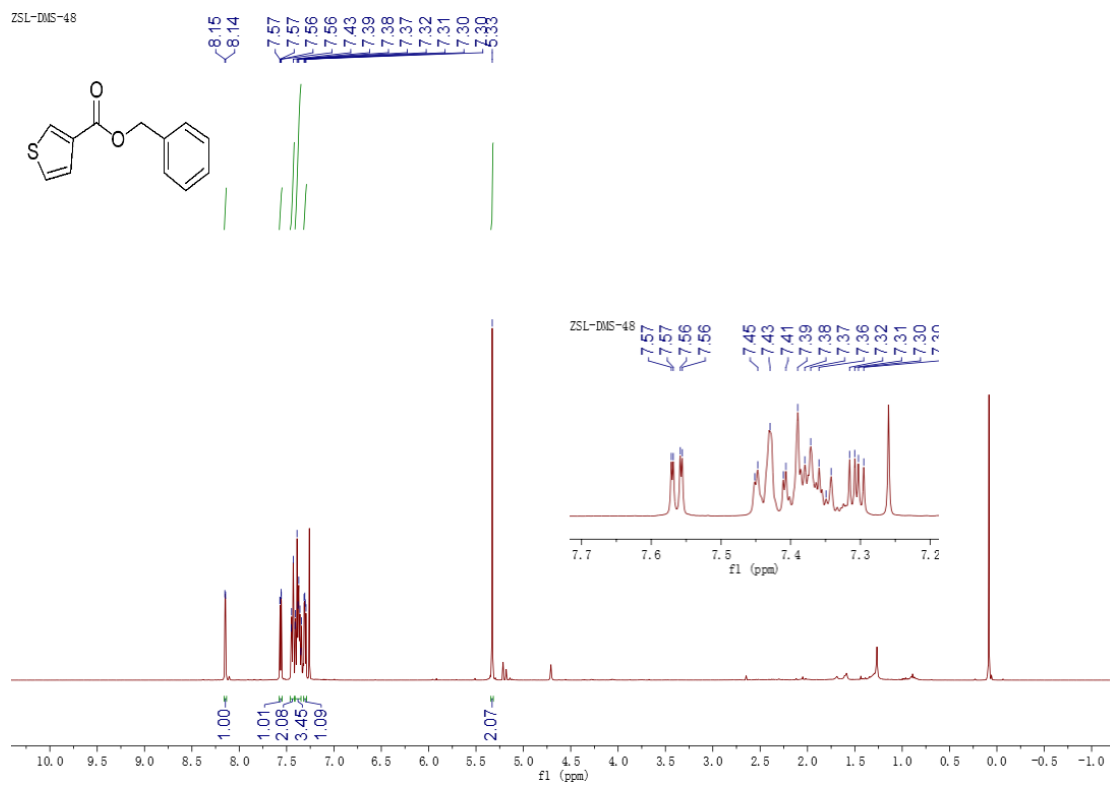


ZSL-DMS-68CD

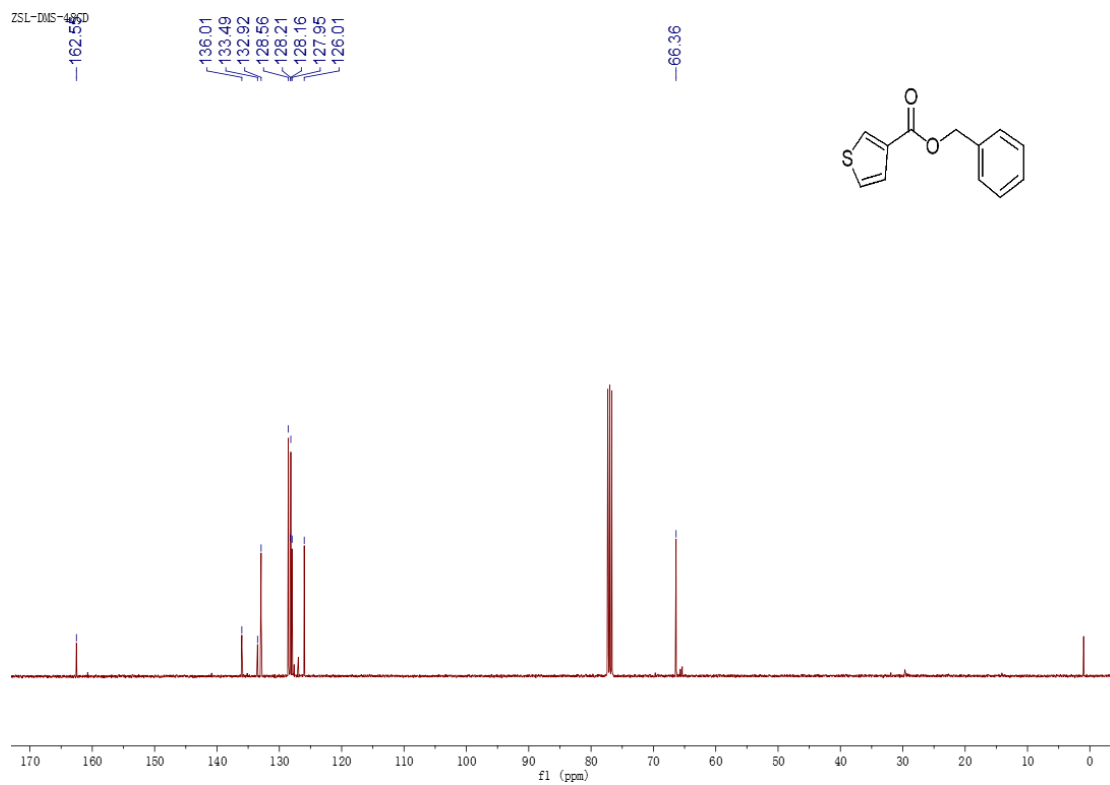


Benzyl thiophene-3-carboxylate (4l). ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

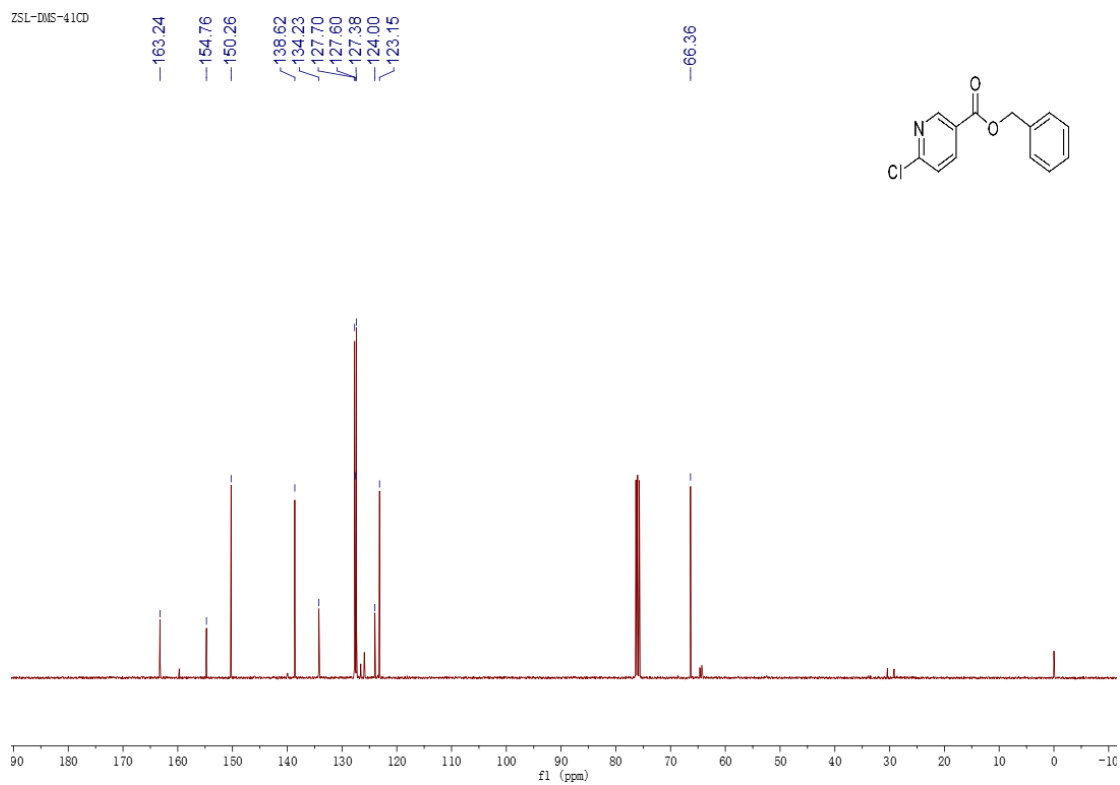
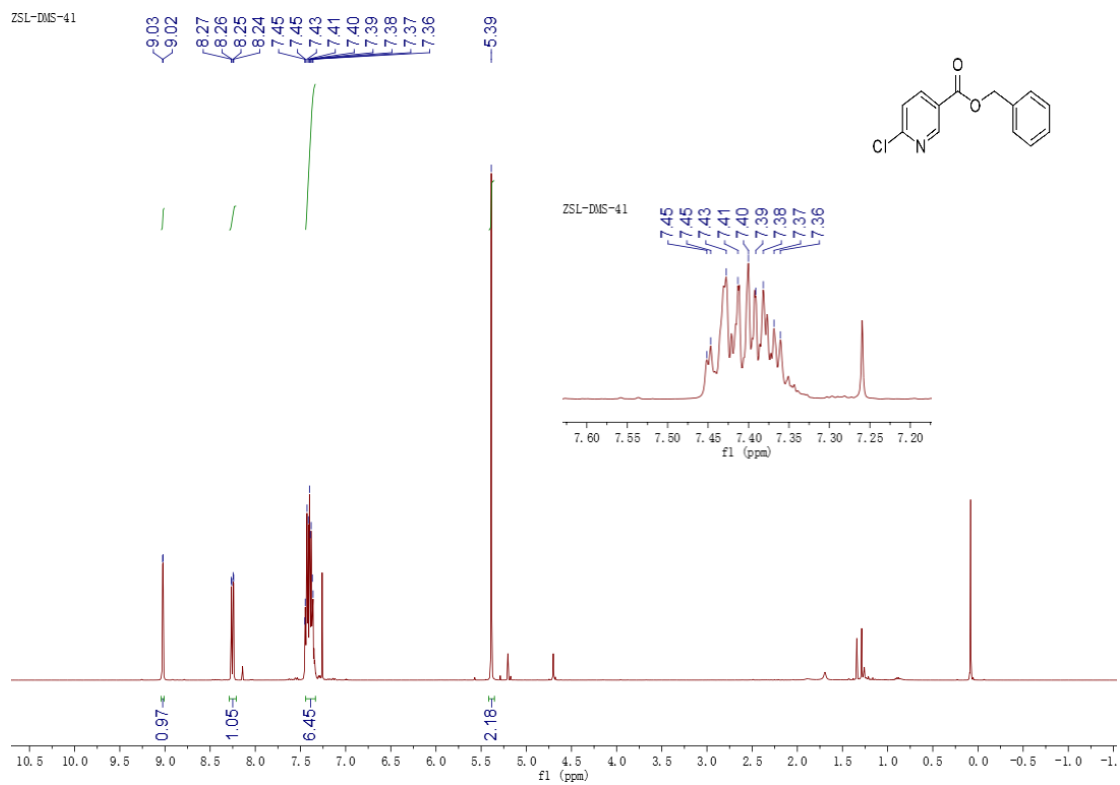
ZSL-DMS-48



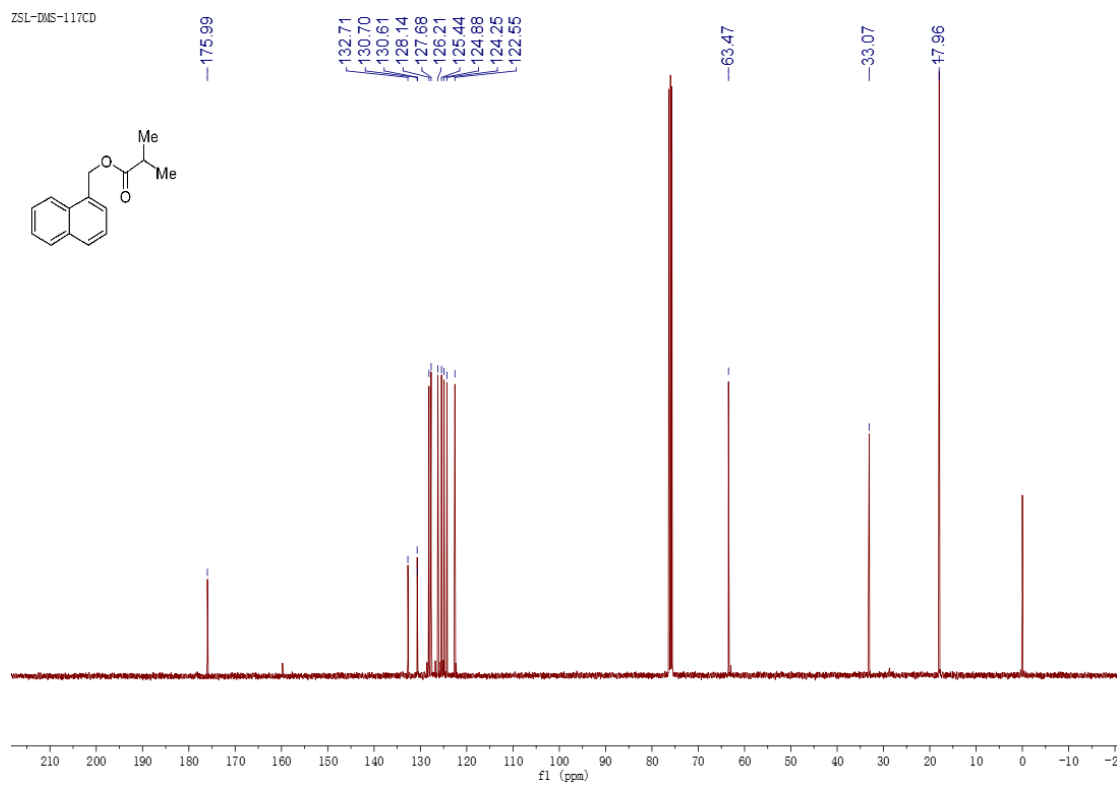
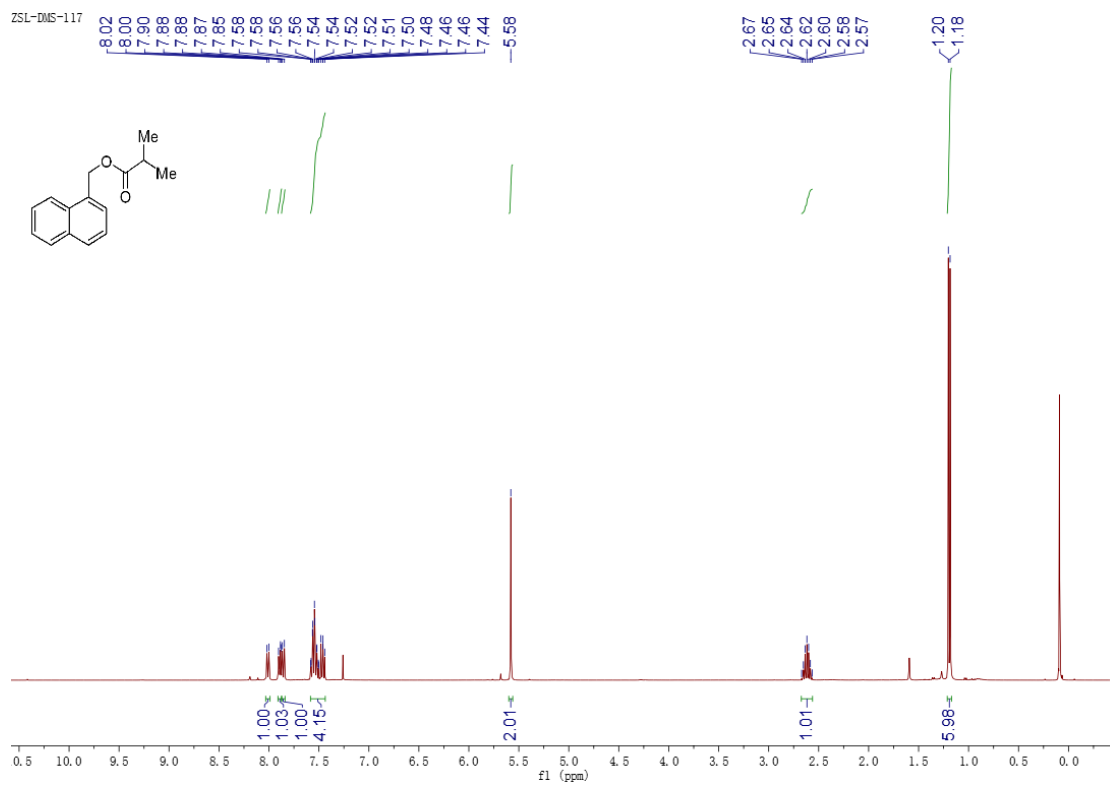
ZSL-DMS-465D



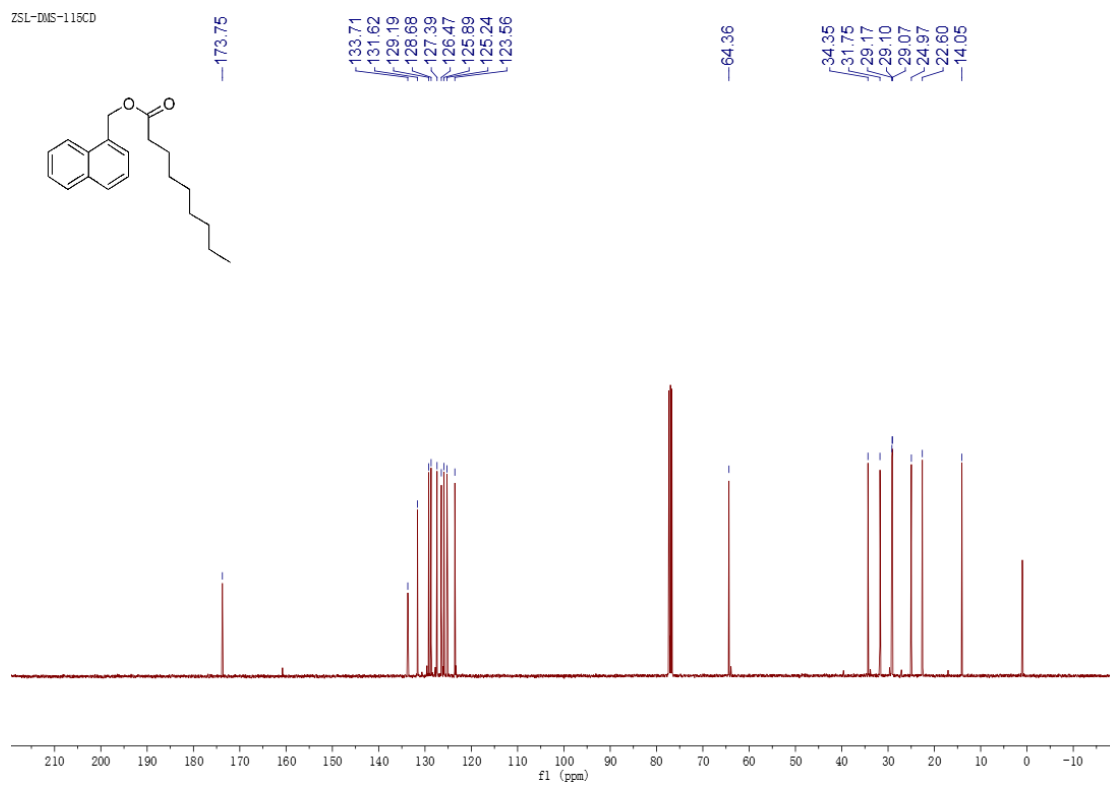
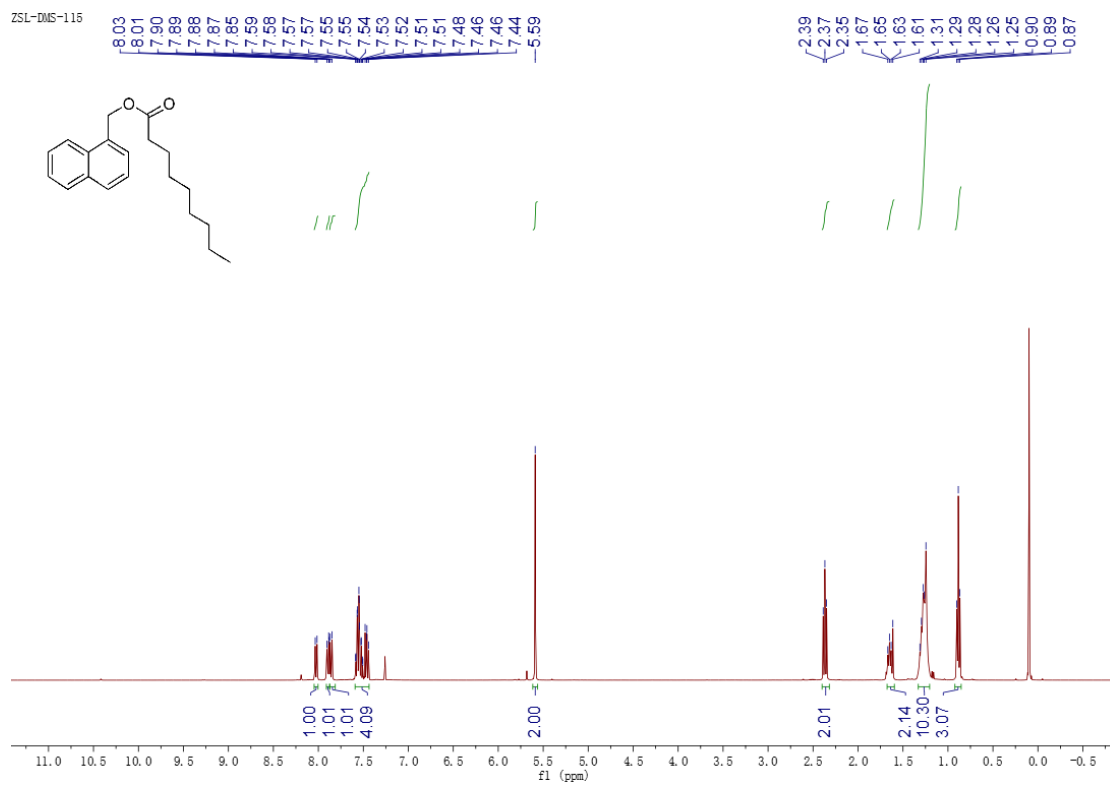
Benzyl 6-chloronicotinate (4m. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)



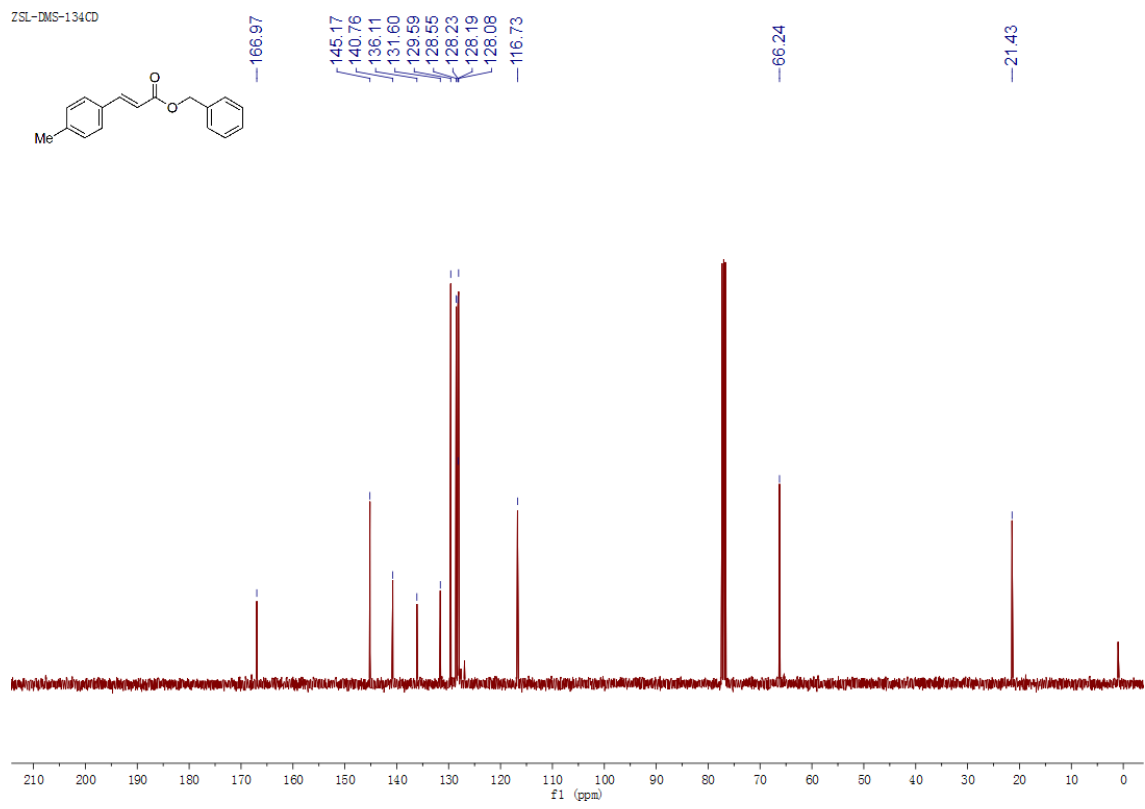
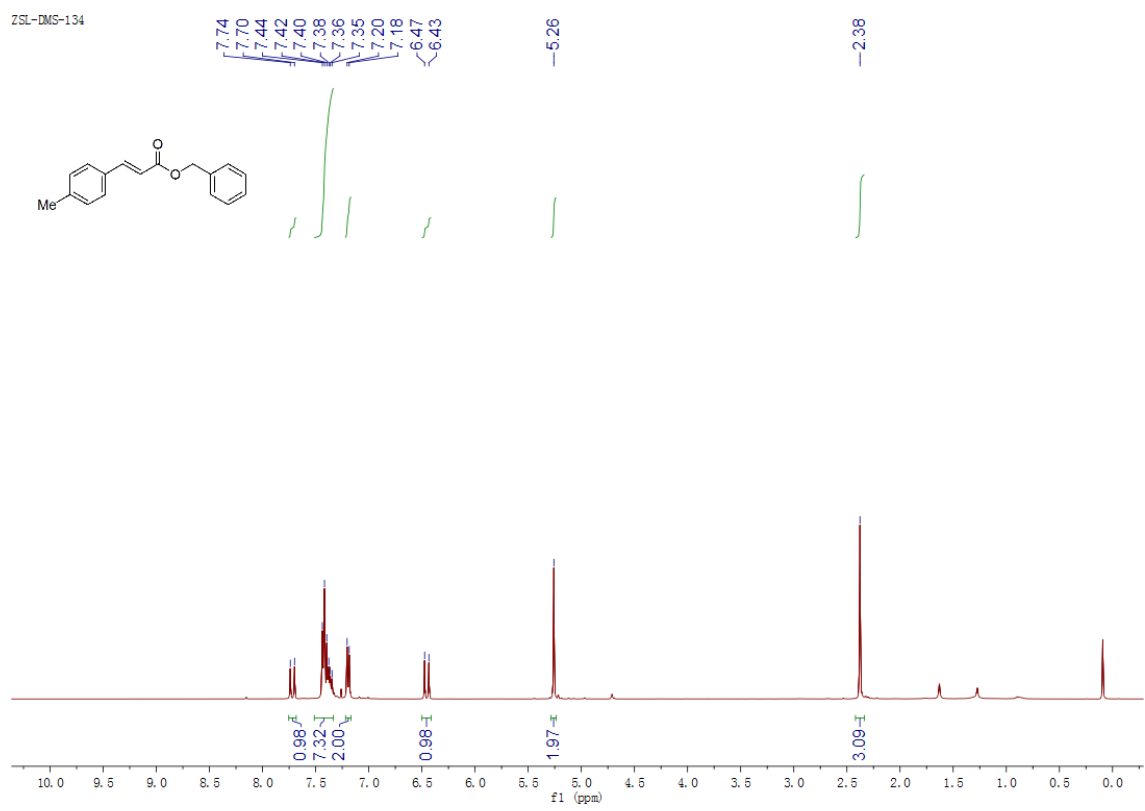
Naphthalen-1-ylmethyl isobutyrate (4n, ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)



Naphthalen-1-ylmethyl nonanoate (4o. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

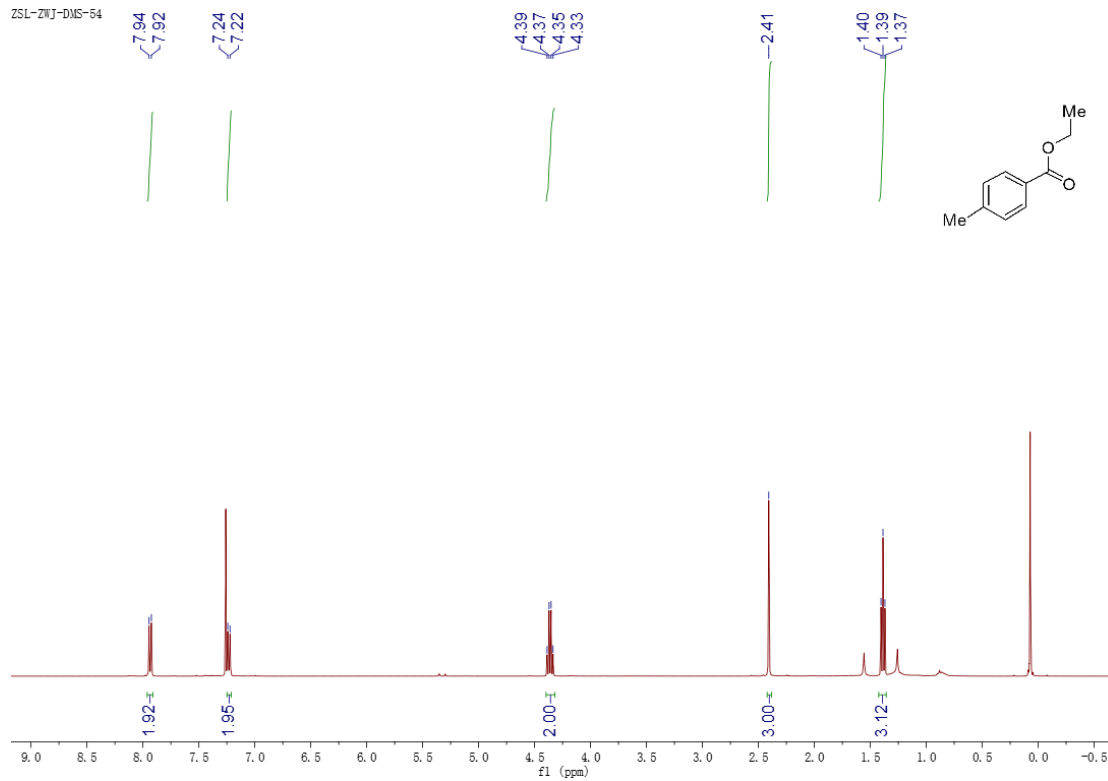


(E)-benzyl 3-*p*-tolylacrylate (4p), ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3

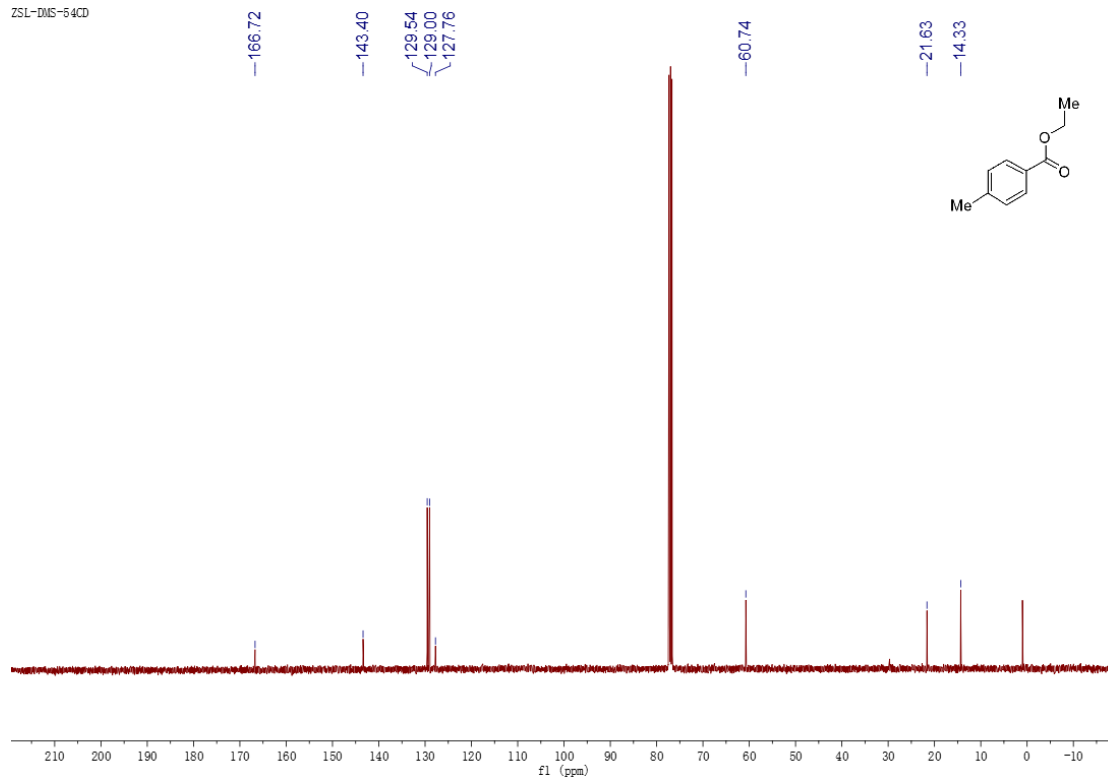


Ethyl 4-methylbenzoate (5a. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

ZSL-ZWJ-DMS-54

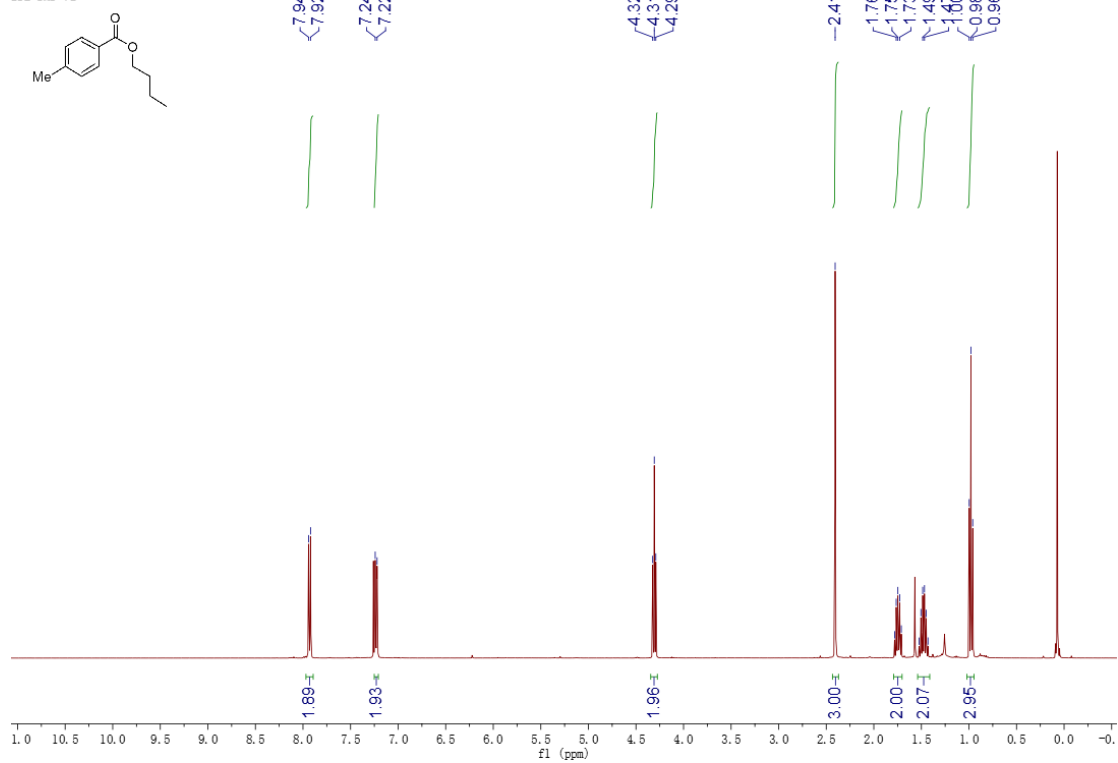


ZSL-DMS-54CD

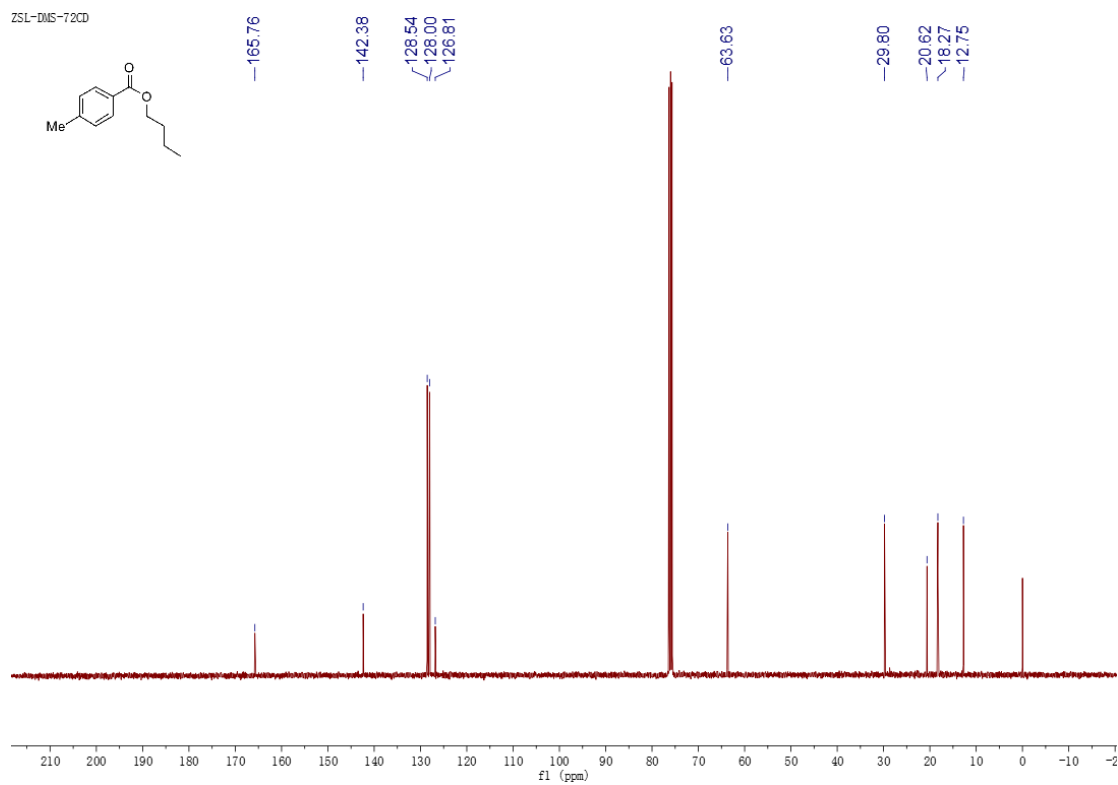


Butyl 4-methylbenzoate (5b). ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3

ZSL-DMS-72

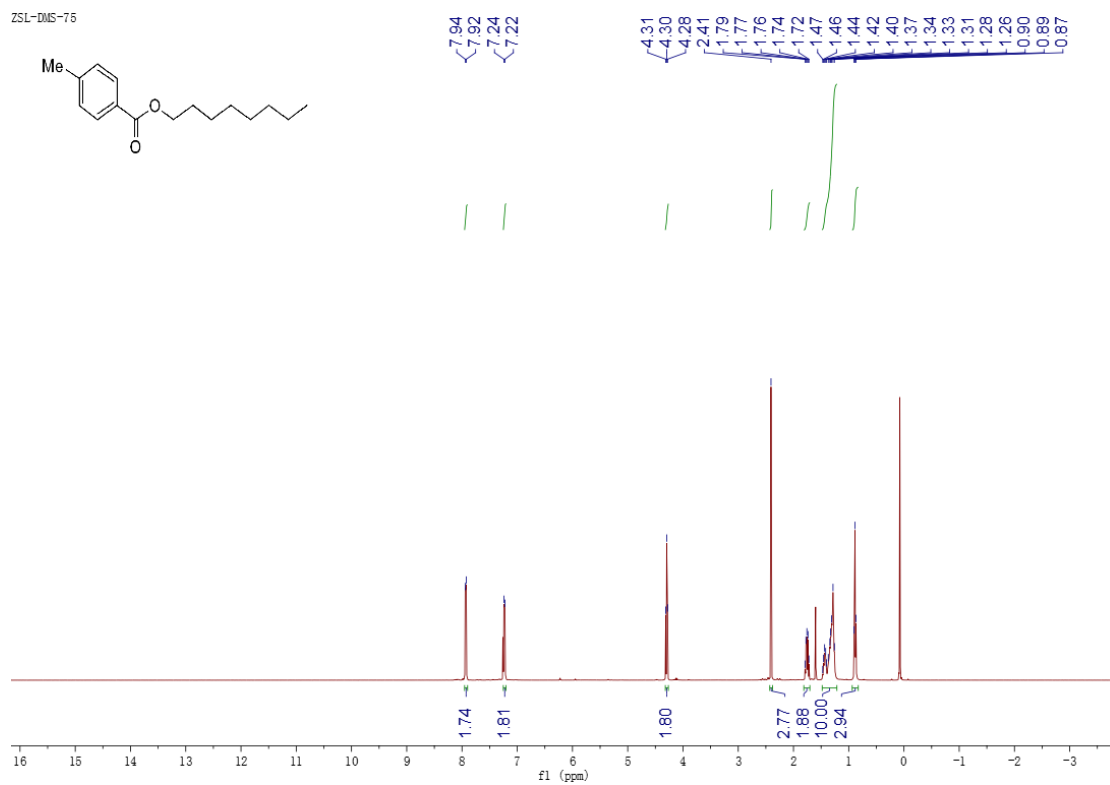


ZSL-DMS-72CD

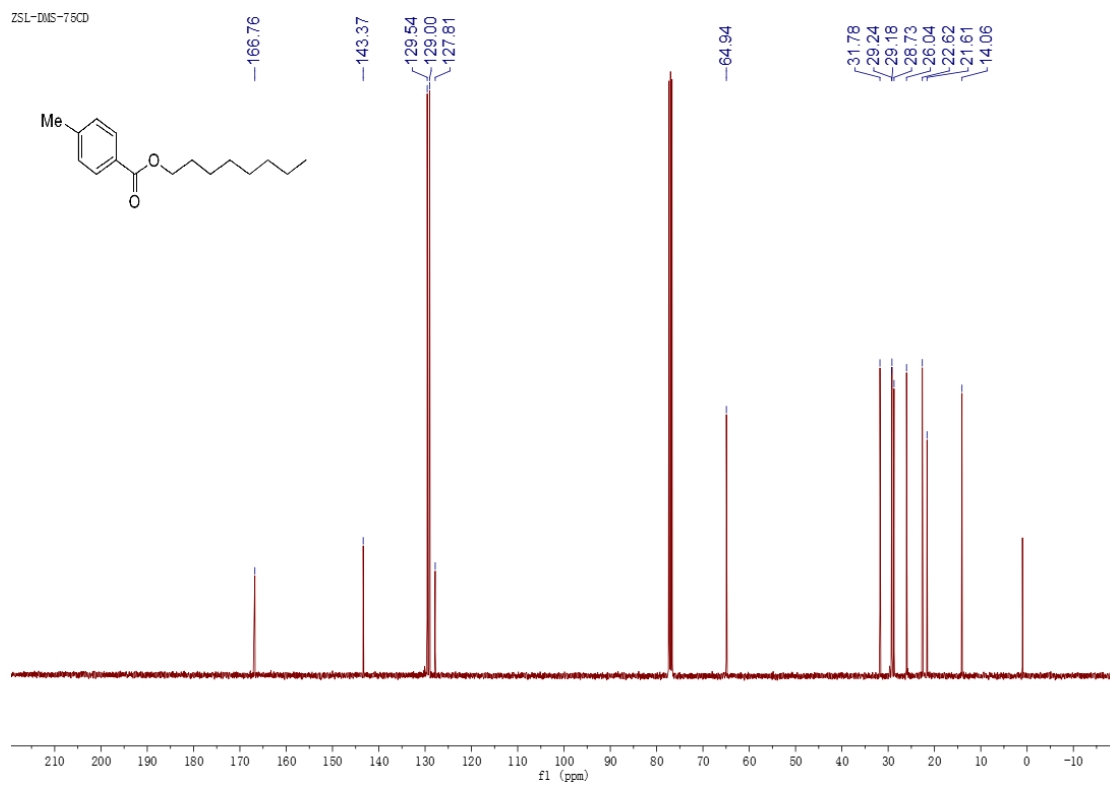


Octyl 4-methylbenzoate (5c. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

ZSL-DMS-75

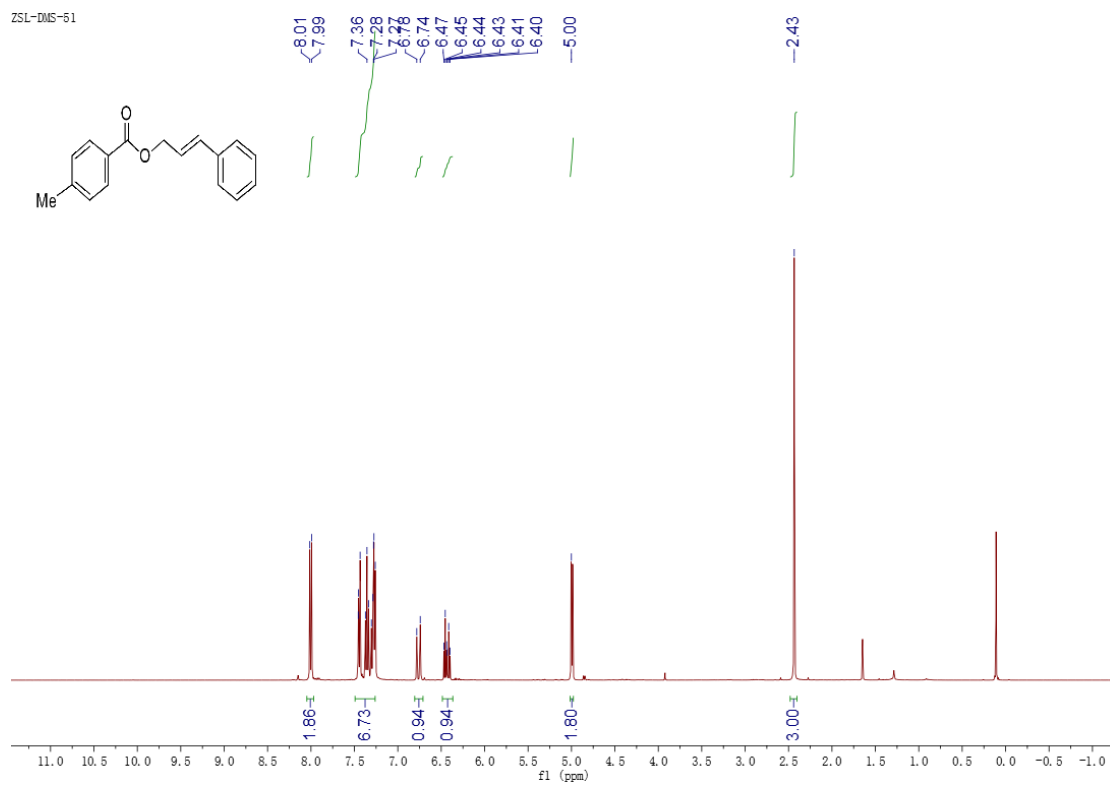


ZSL-DMS-75CD

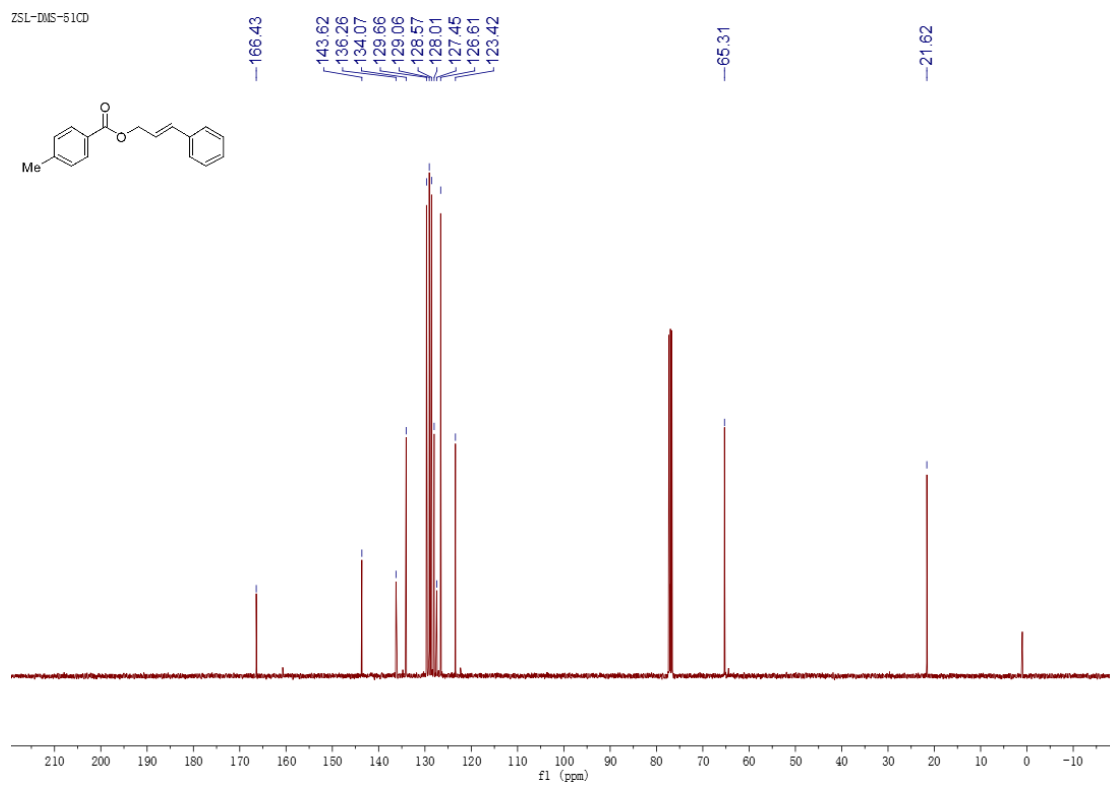


Cinnamyl 4-methylbenzoate (5d. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

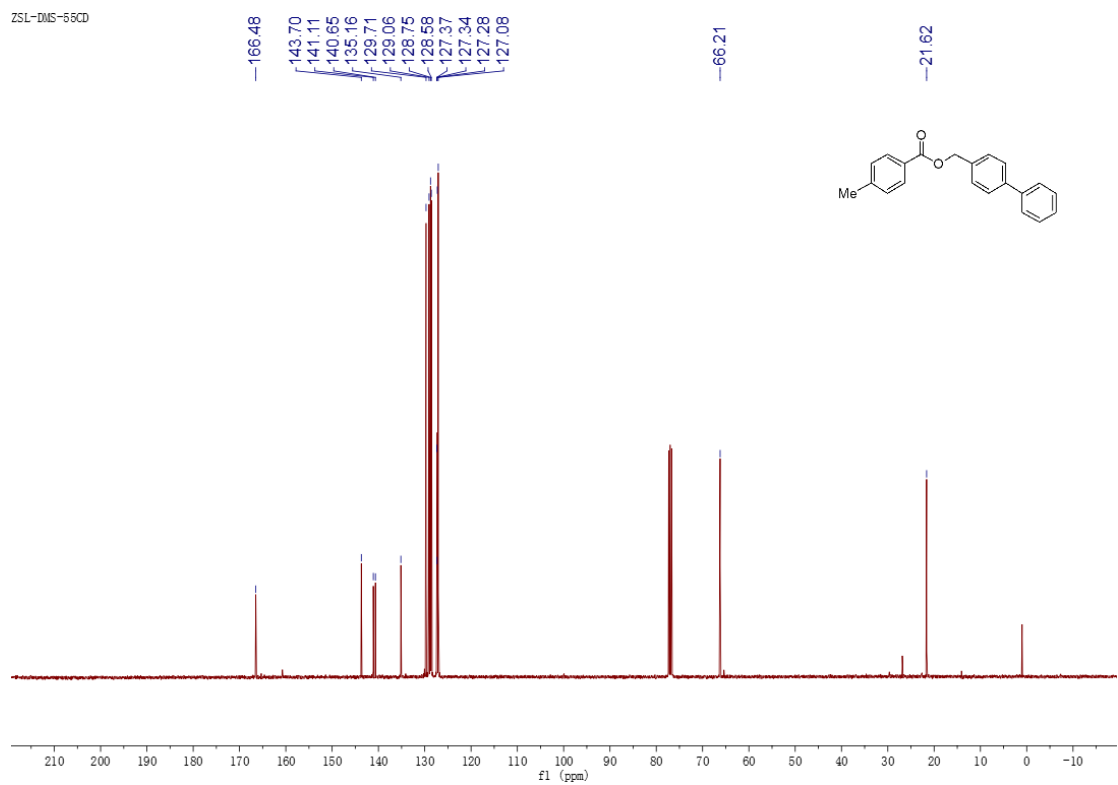
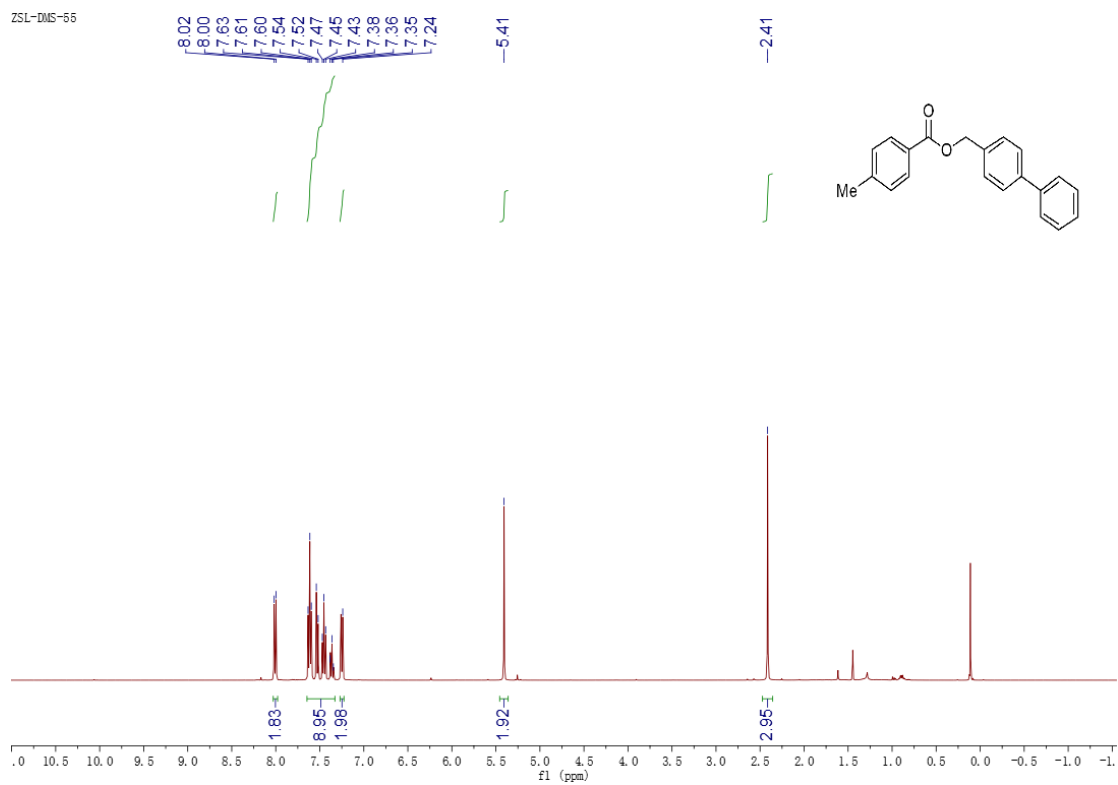
ZSL-DMS-51



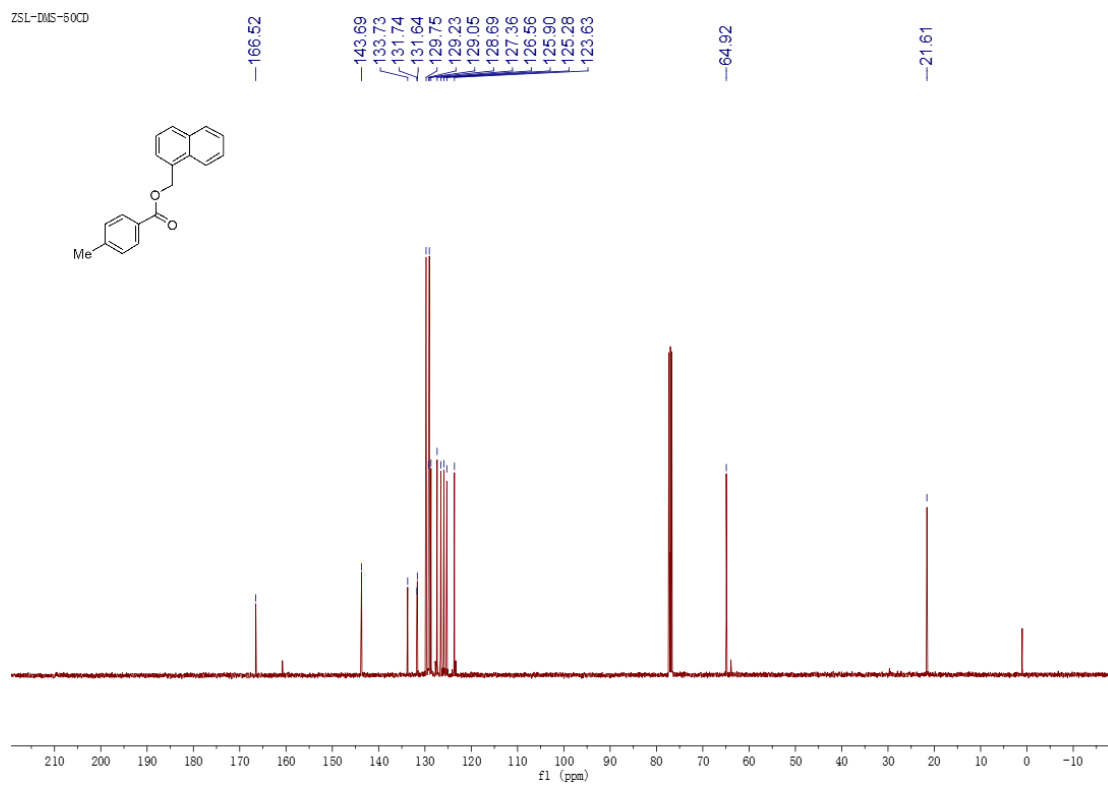
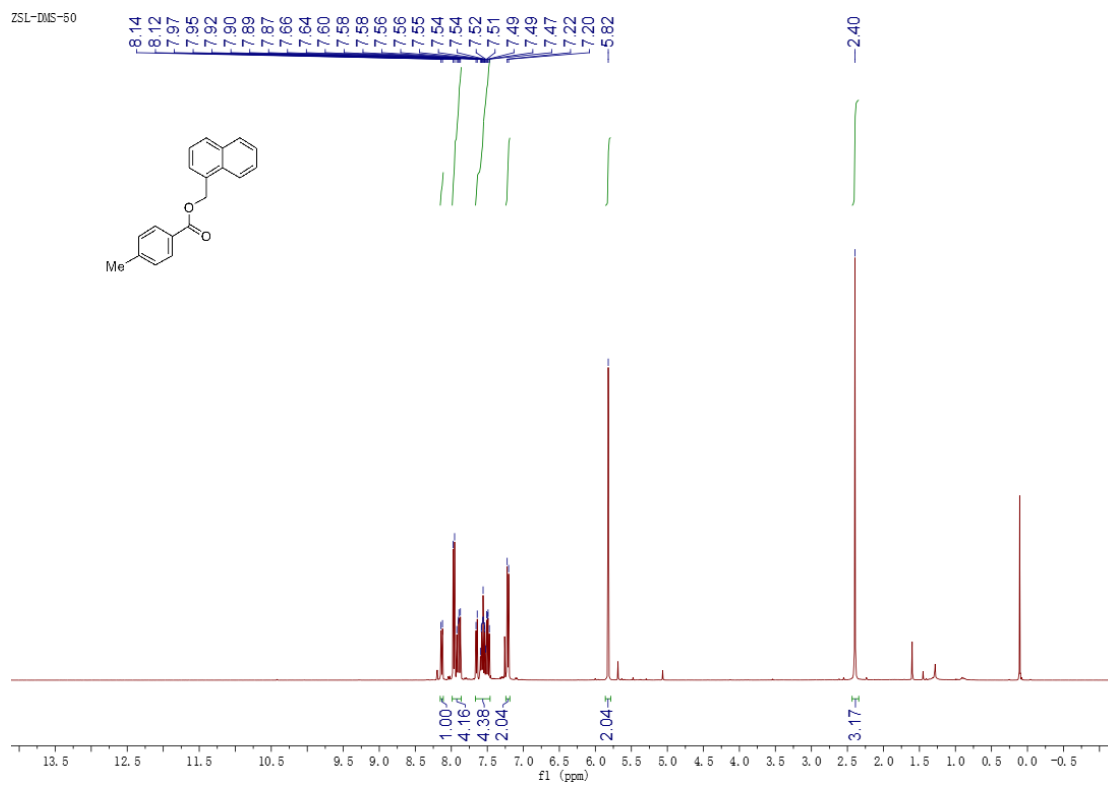
ZSL-DMS-51CD



[1,1'-Biphenyl]-4-ylmethyl 4-methylbenzoate (5e. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

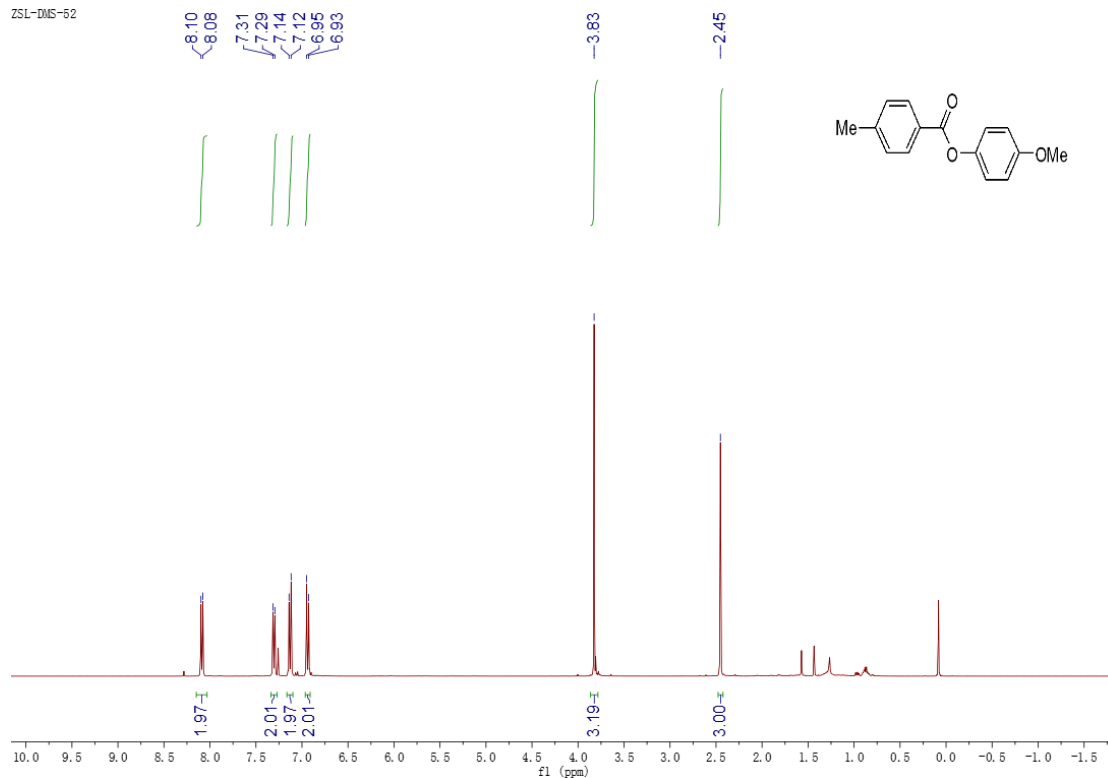


Naphthalen-1-ylmethyl 4-methylbenzoate (5f. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

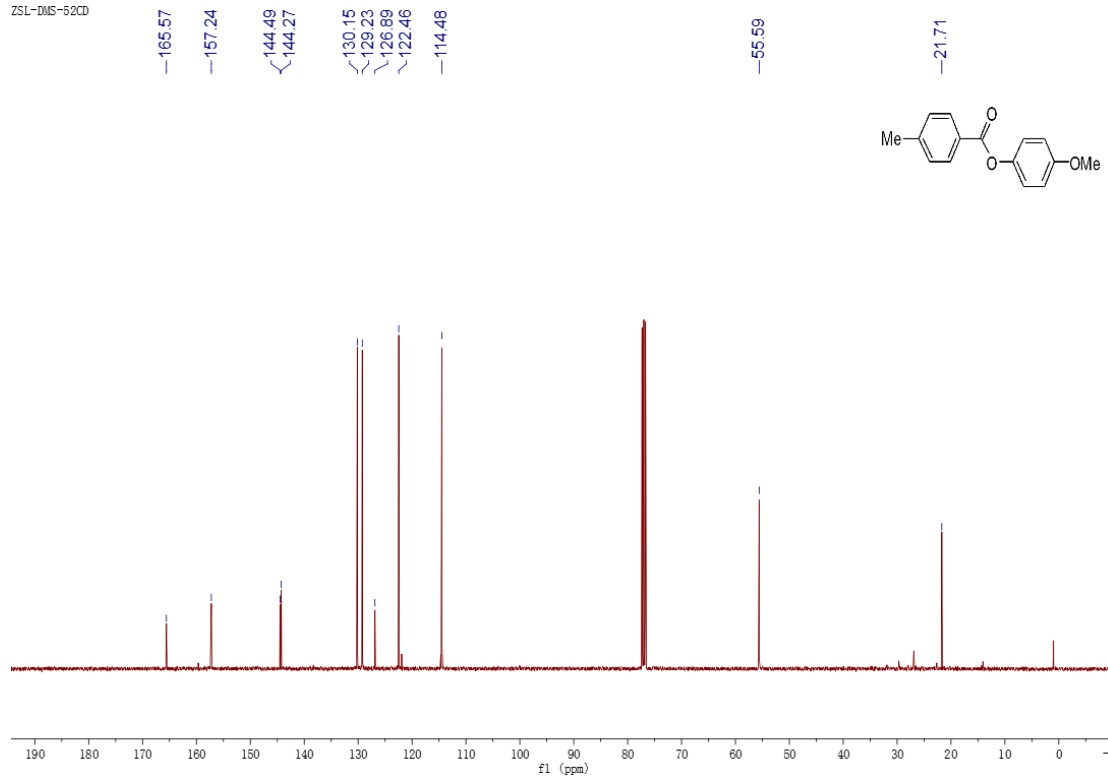


4-Methoxyphenyl 4-methylbenzoate (5g. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

ZSL-DMS-52

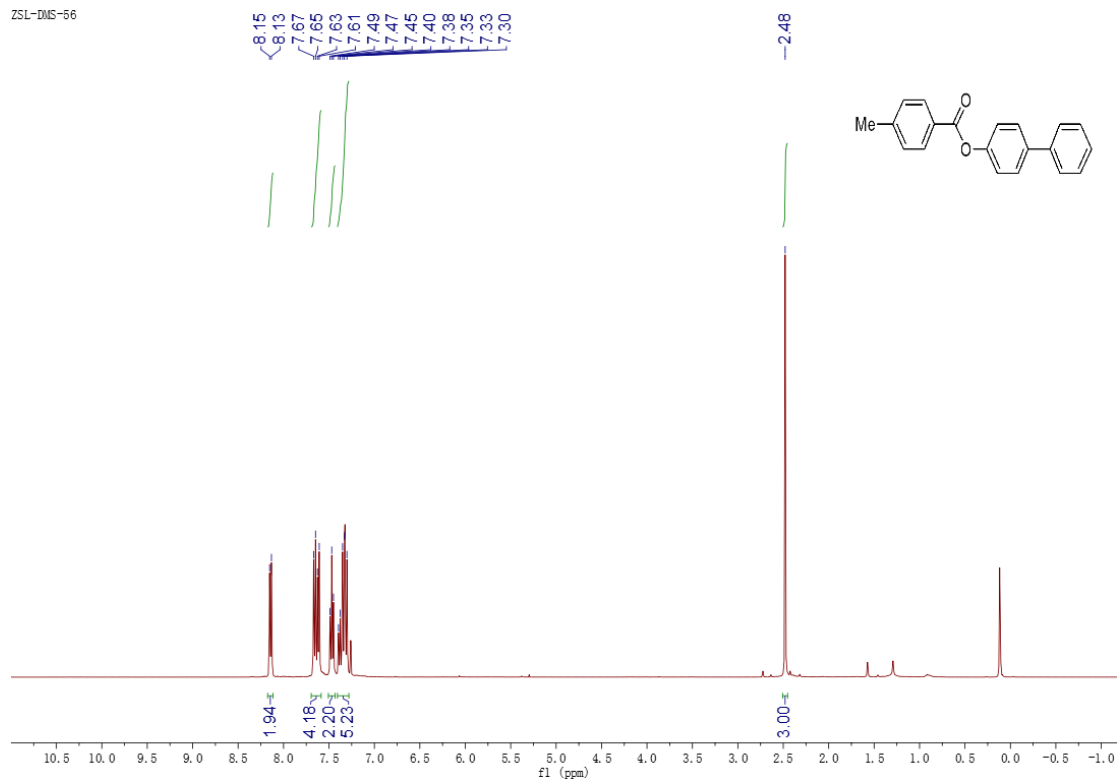


ZSL-DMS-82CD

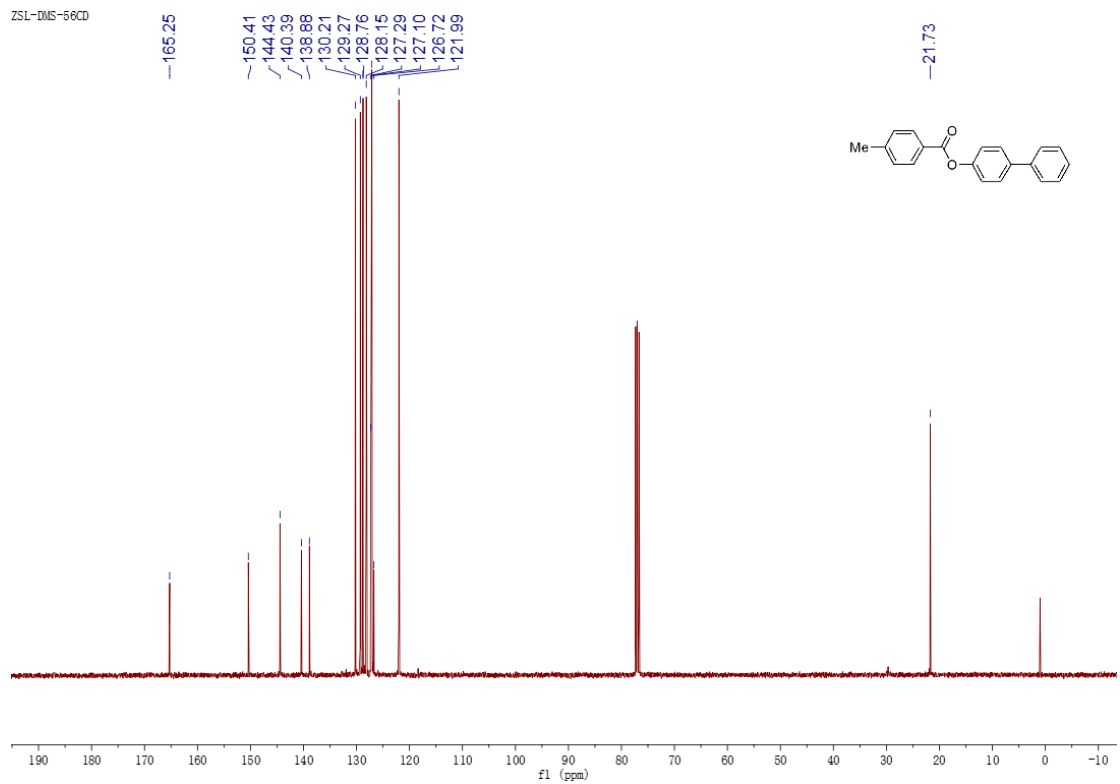


[1,1'-Biphenyl]-4-yl 4-methylbenzoate (5h). ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3

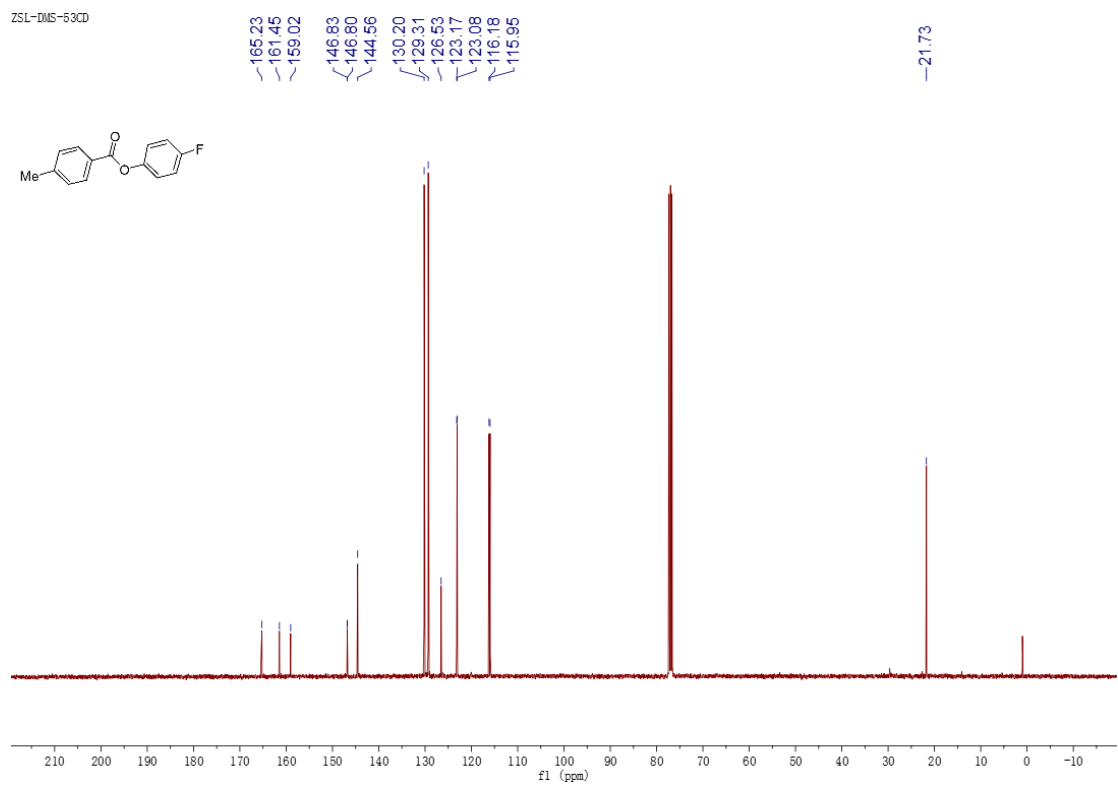
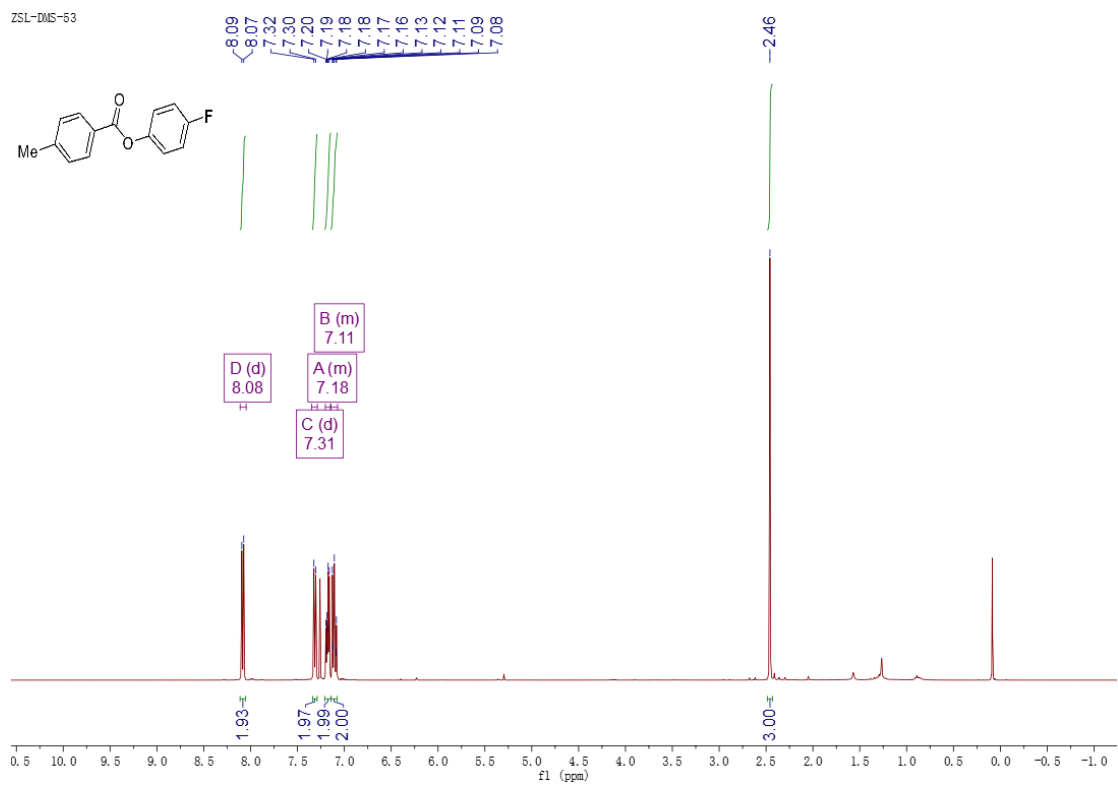
ZSL-DMS-56



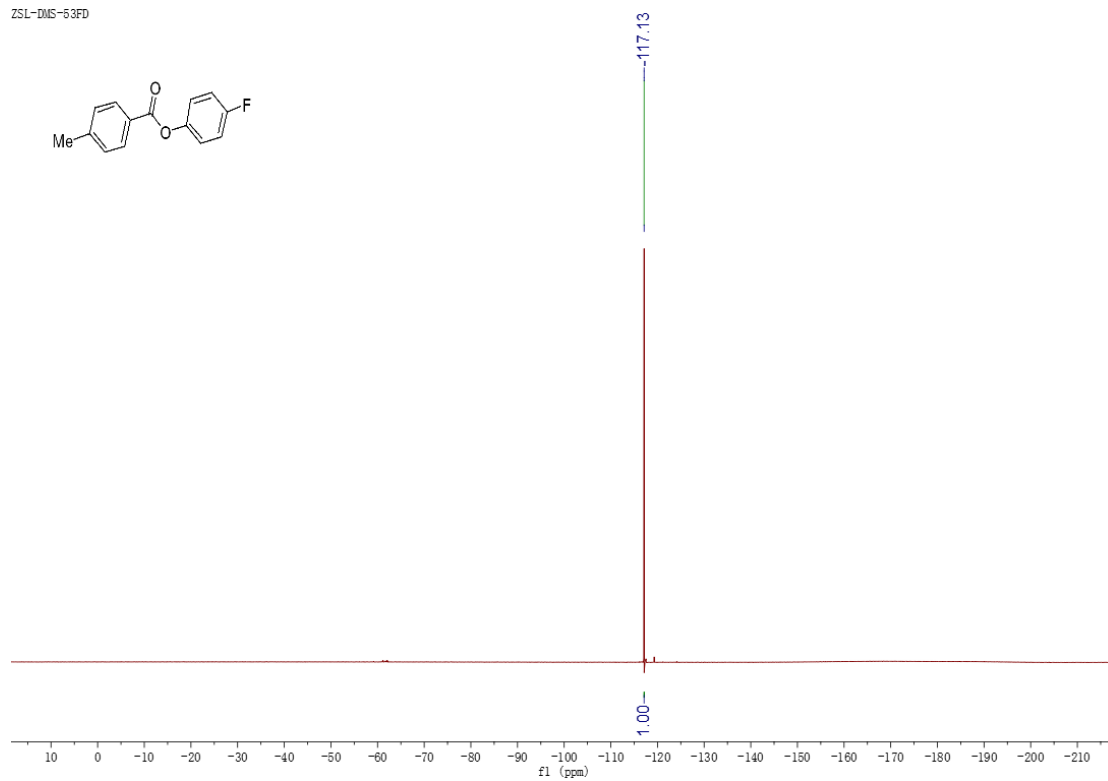
ZSL-DMS-56CD



4-Fluorophenyl 4-methylbenzoate (5i. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3 ; ^{19}F NMR 376 MHz, CDCl_3)

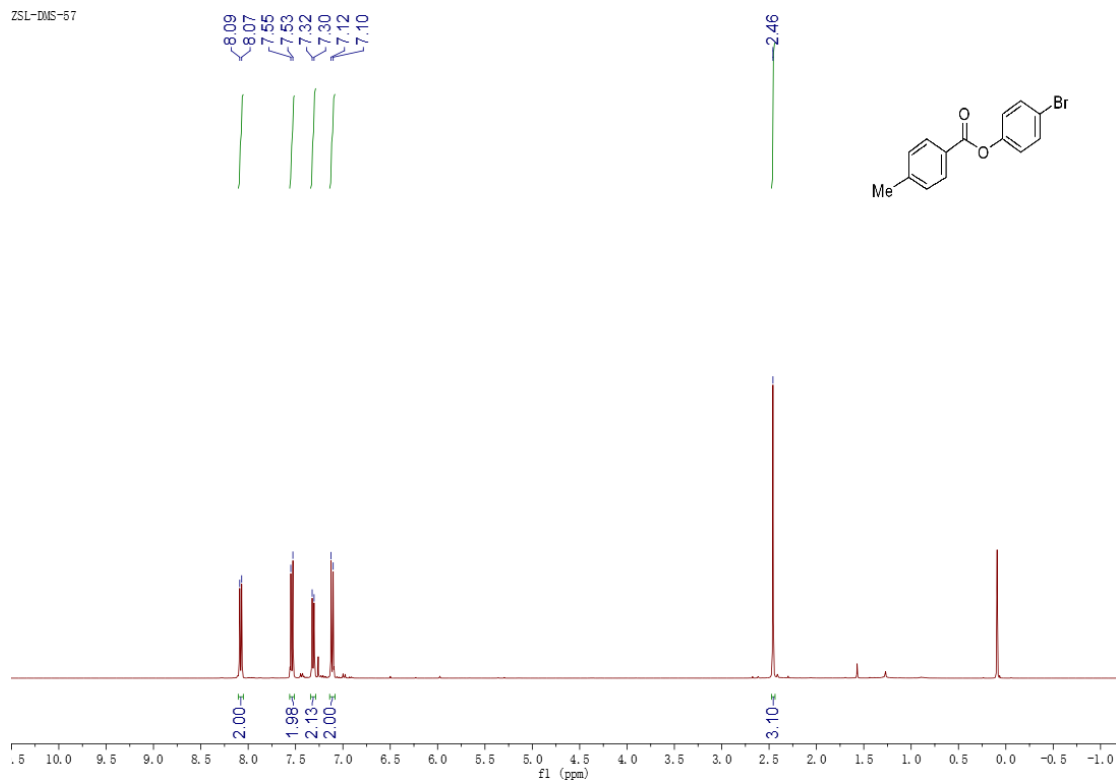


ZSL-DMS-53FD

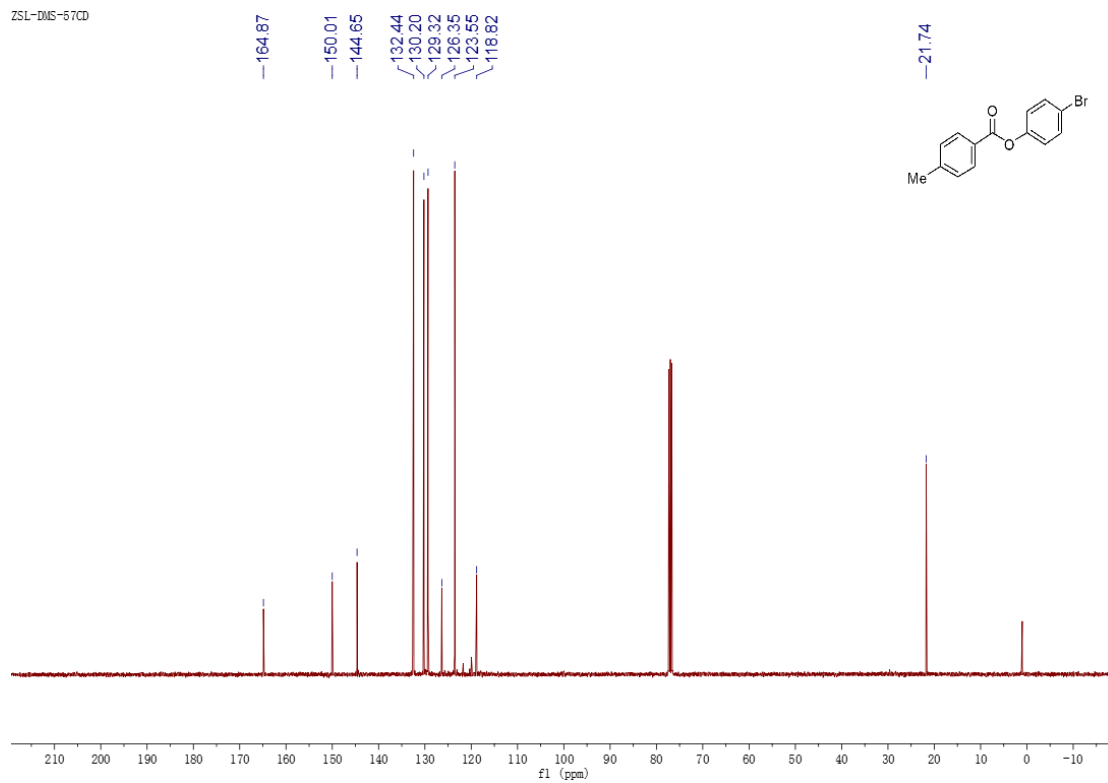


4-Bromophenyl 4-methylbenzoate (5j). ¹H NMR 400 MHz, CDCl₃; ¹³C NMR 101 MHz, CDCl₃)

ZSL-DMS-67

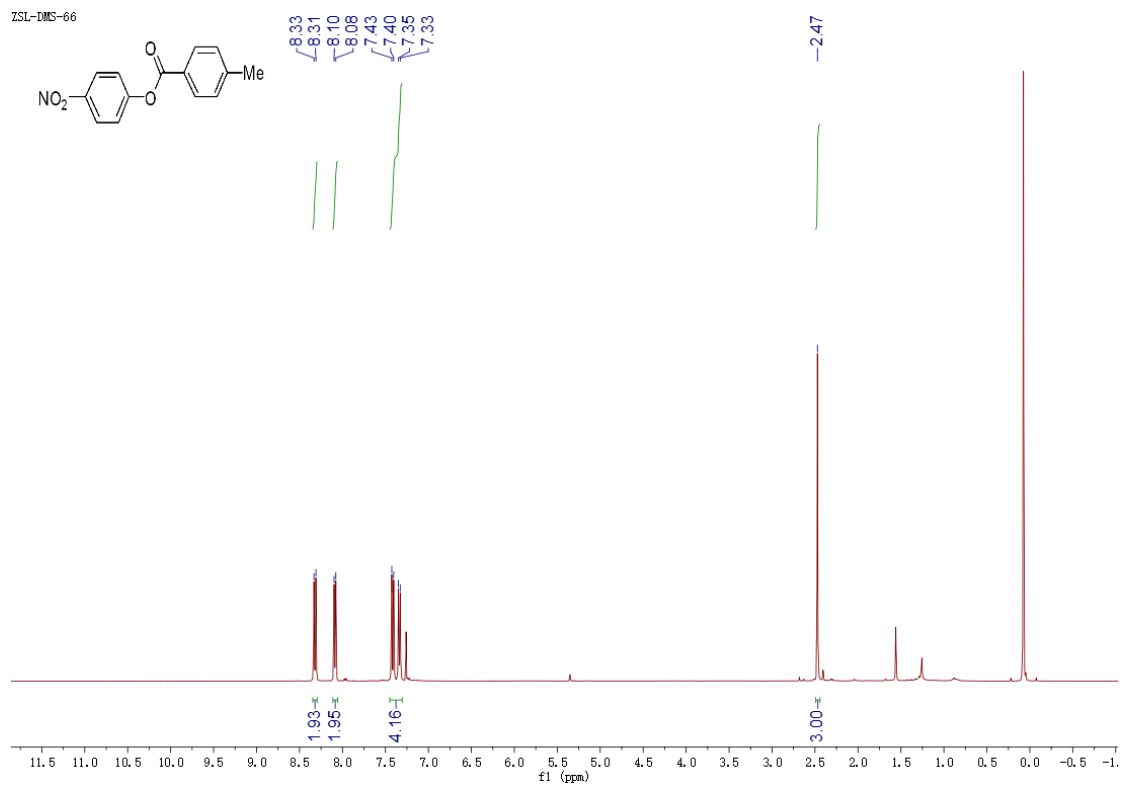


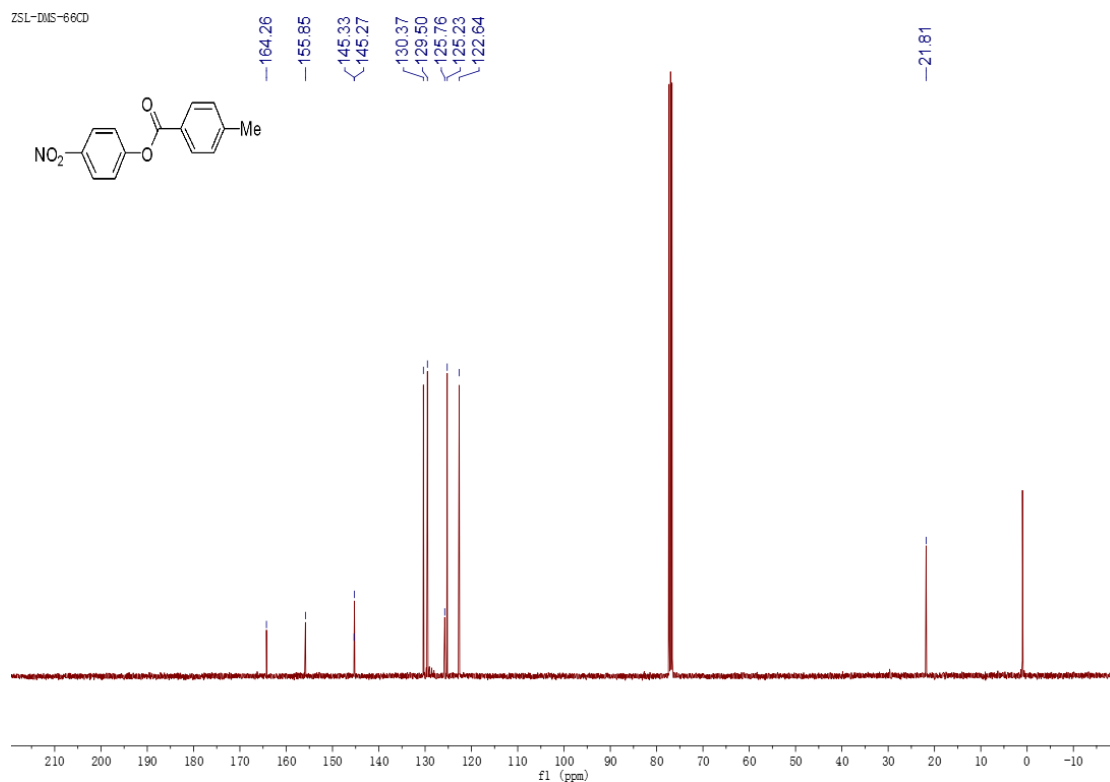
ZSL-DMS-57CD



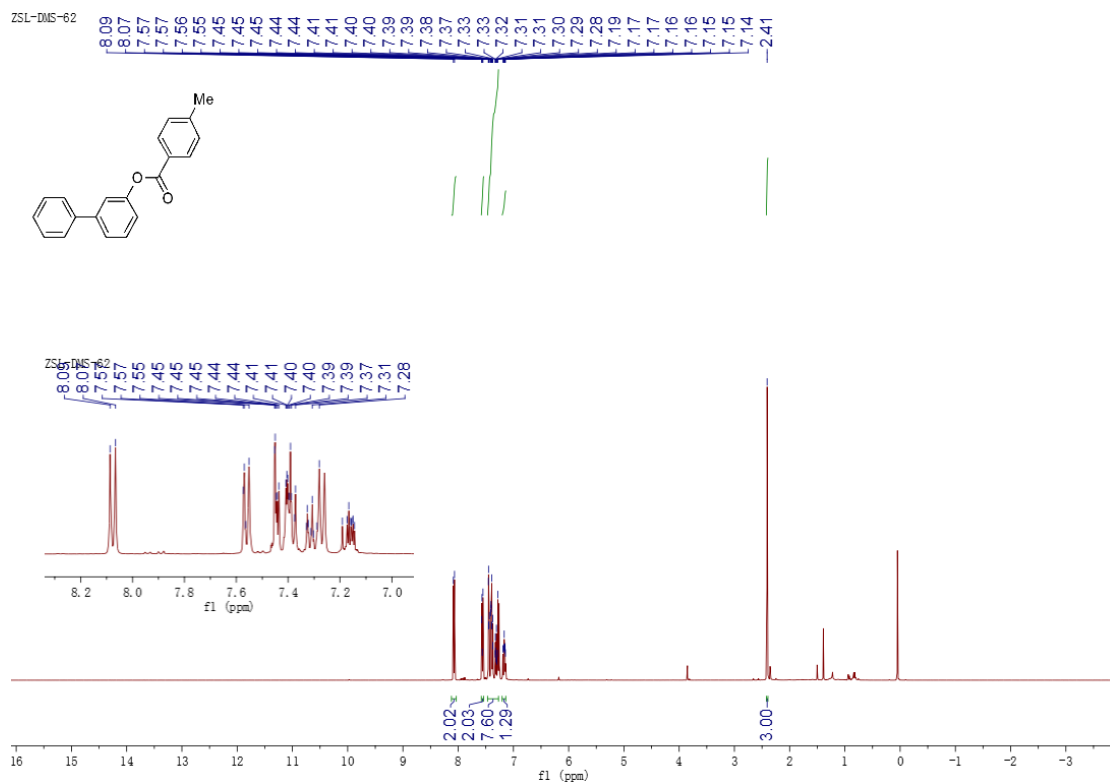
4-Nitrophenyl 4-methylbenzoate (5k. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

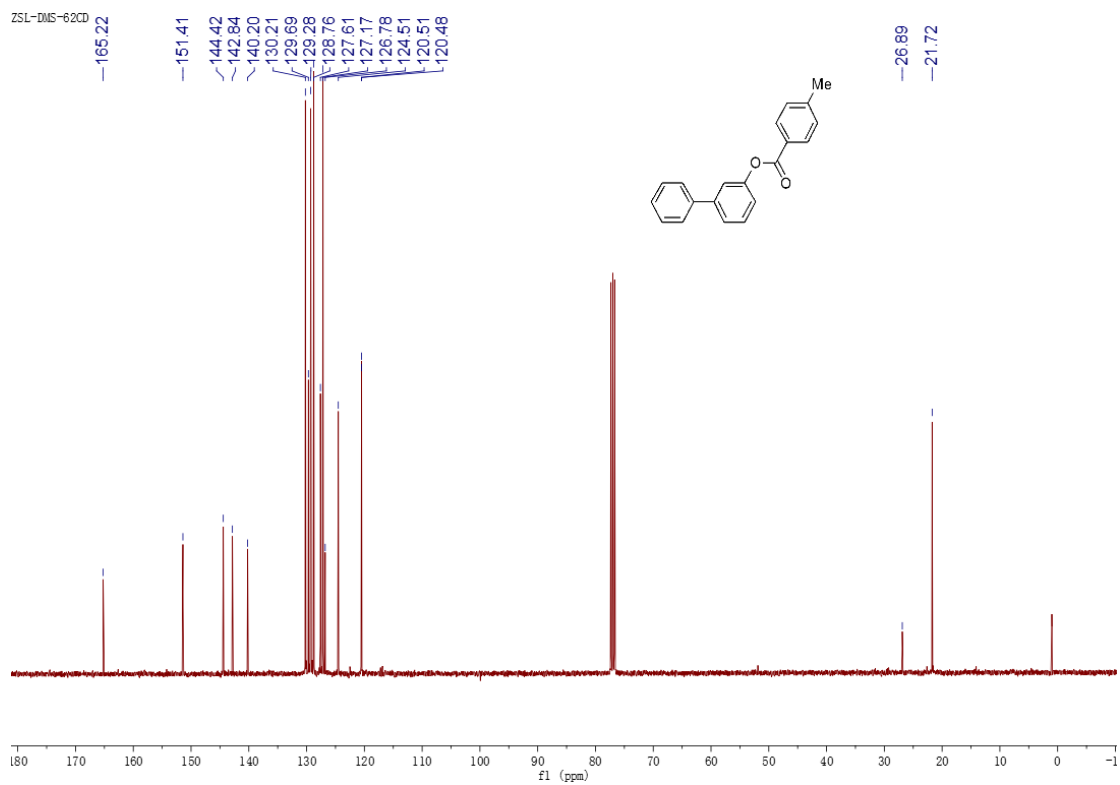
ZSL-DMS-66



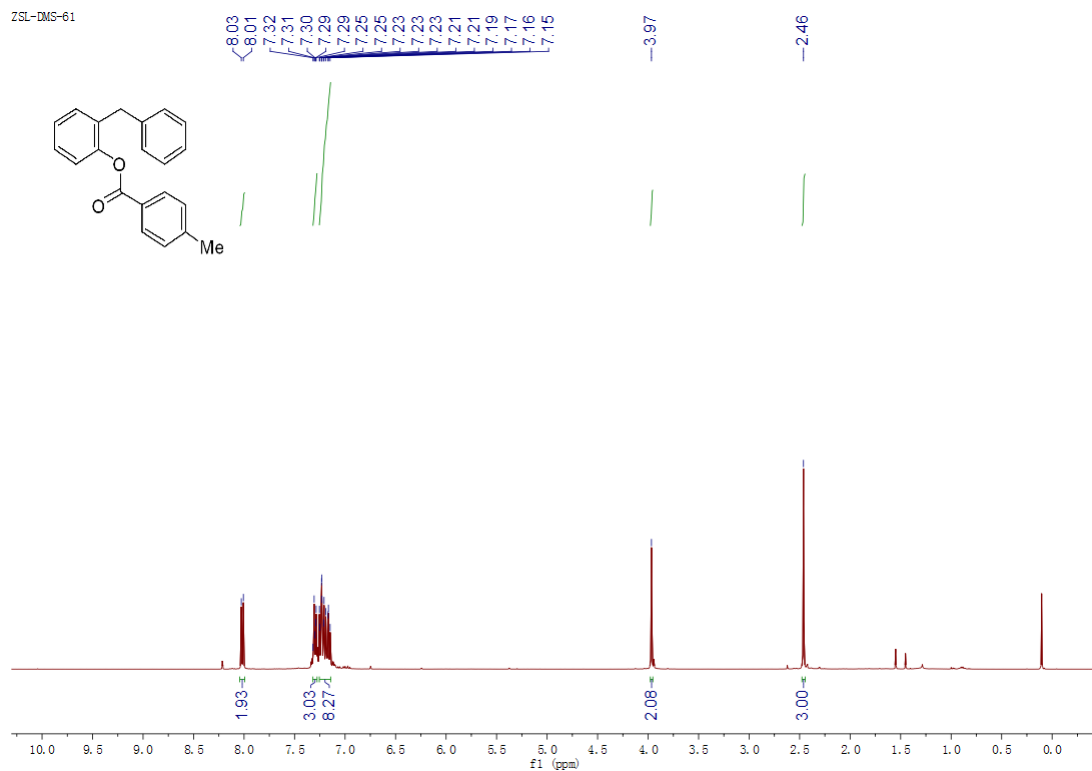


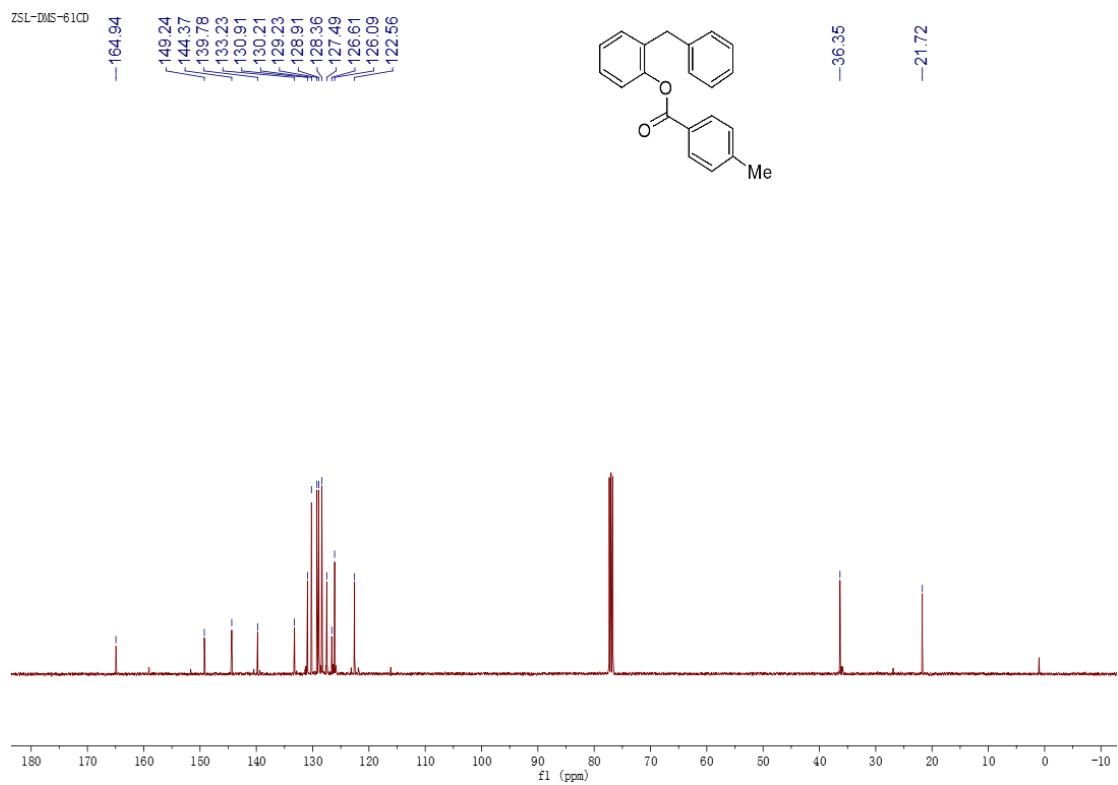
[1,1'-Biphenyl]-3-yl 4-methylbenzoate (5l). ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)



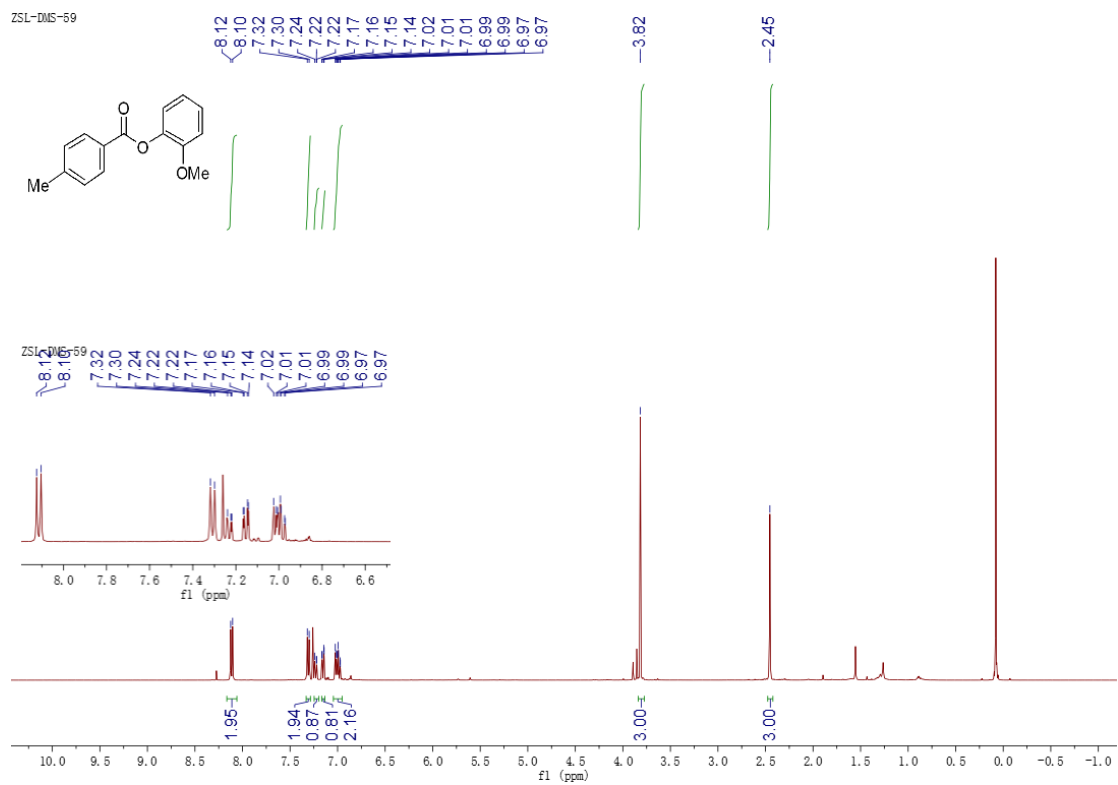


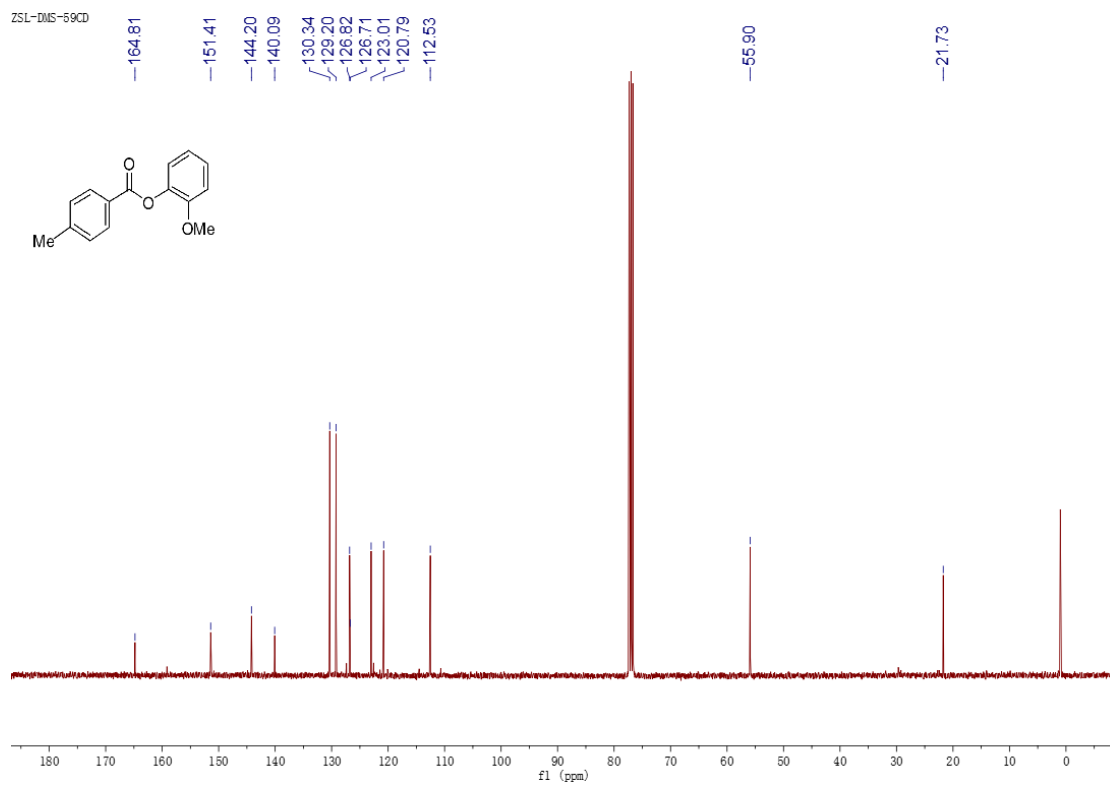
2-Benzylphenyl 4-methylbenzoate (5m. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)



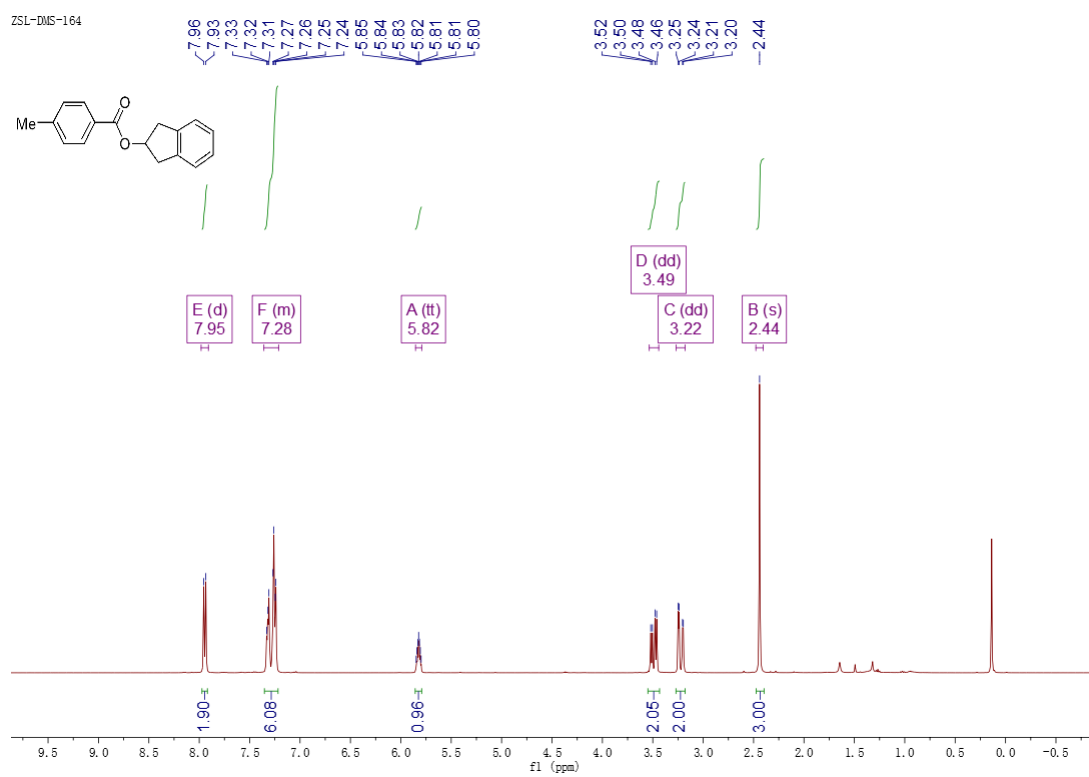


2-Methoxyphenyl 4-methylbenzoate (5n. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

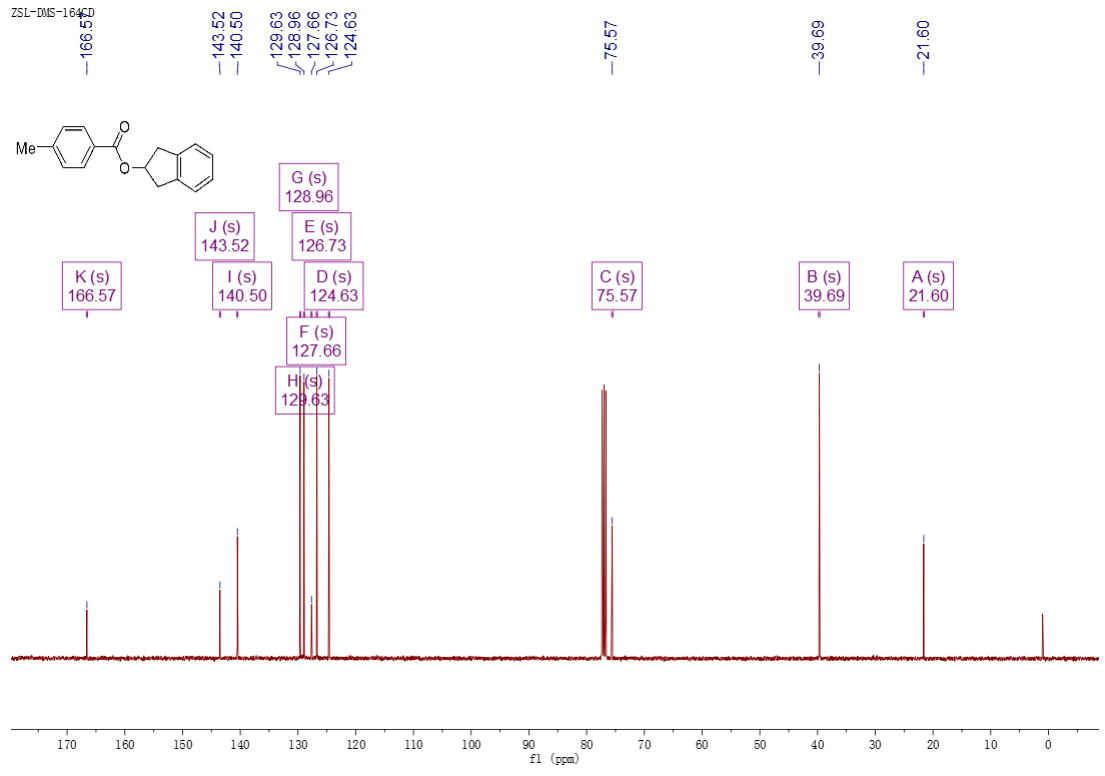
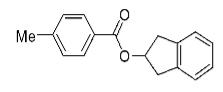




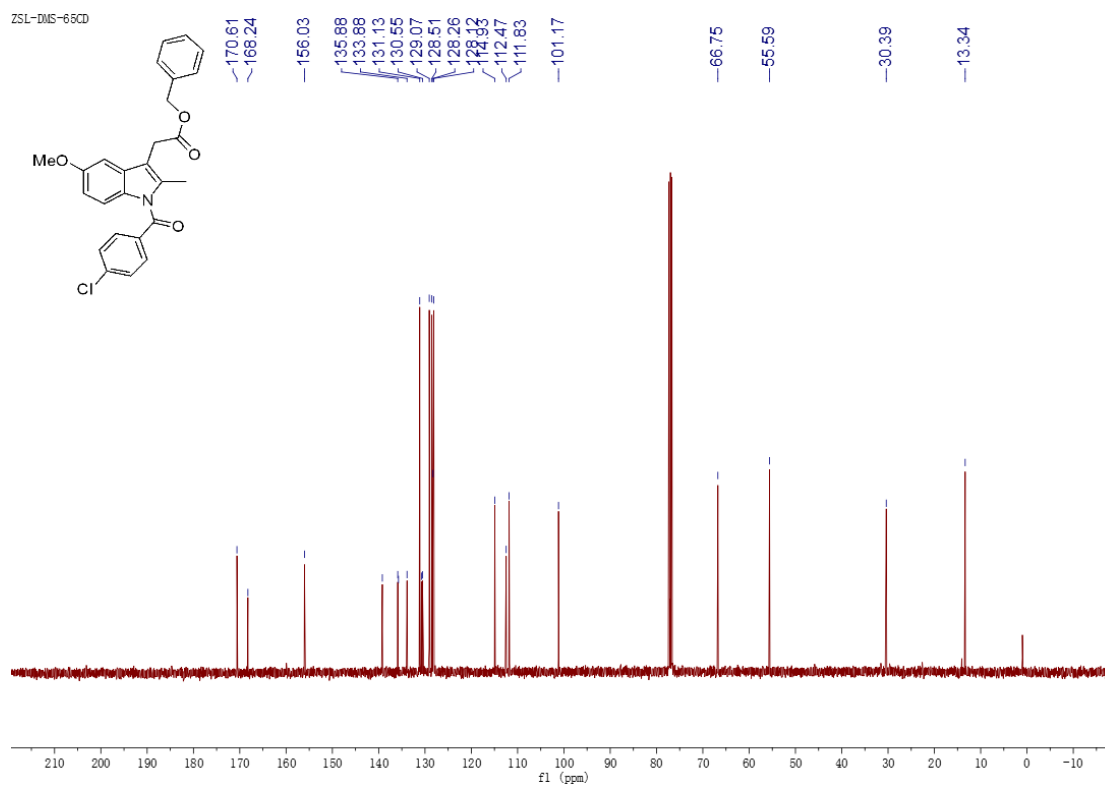
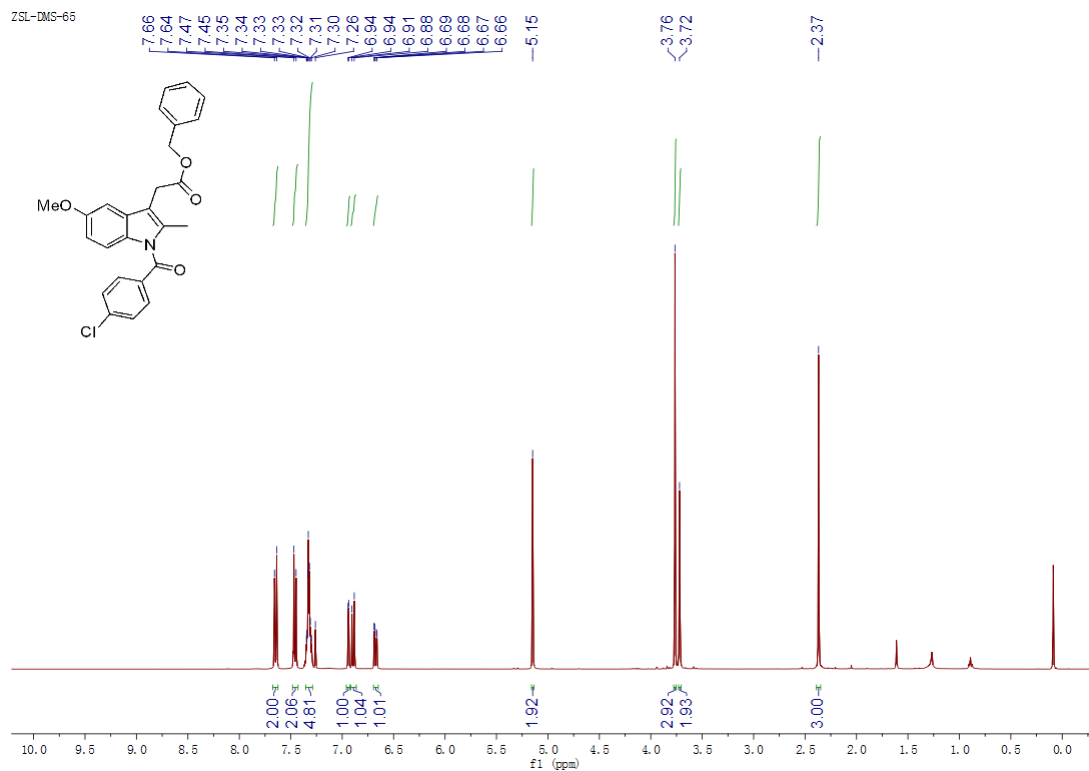
2,3-Dihydro-1*H*-inden-2-yl 4-methylbenzoate (**50**, ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)



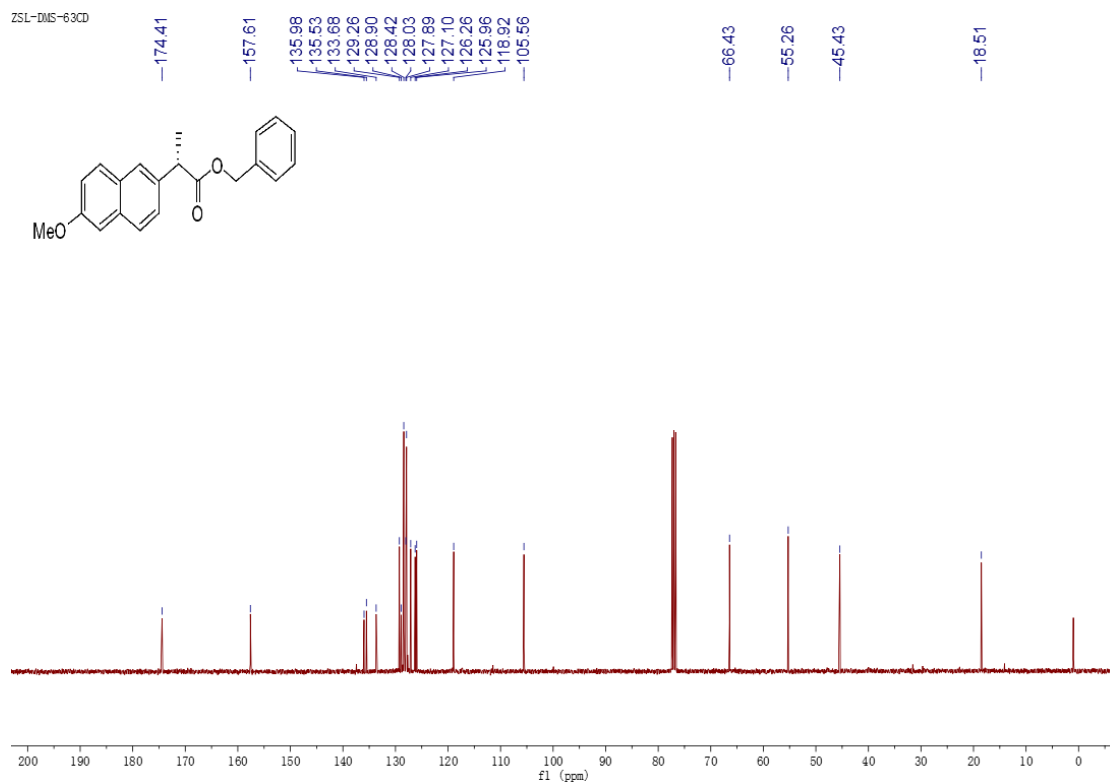
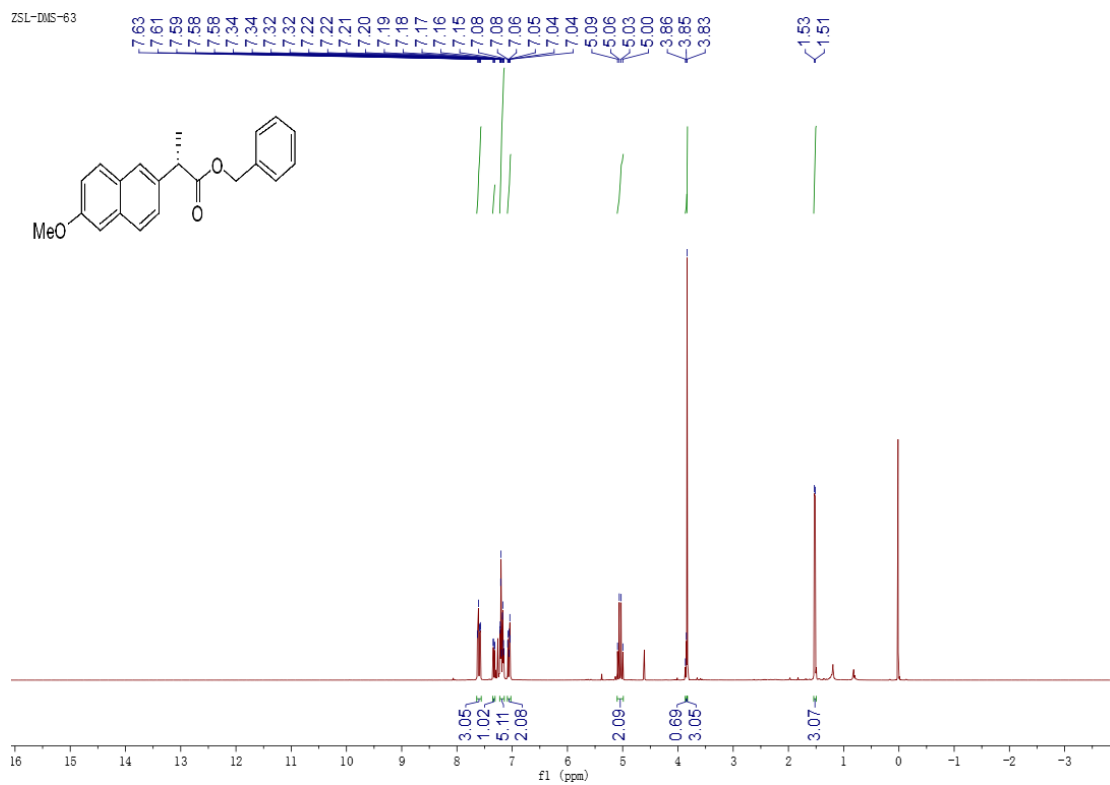
ZSL-DMS-1643



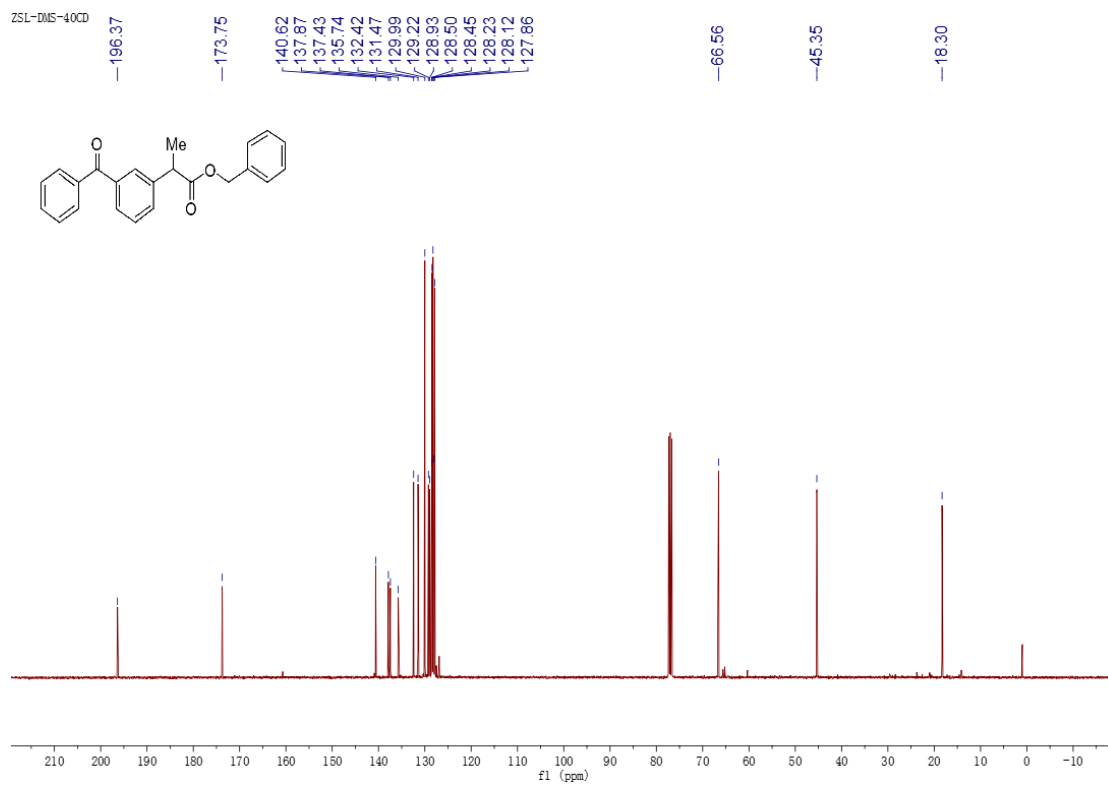
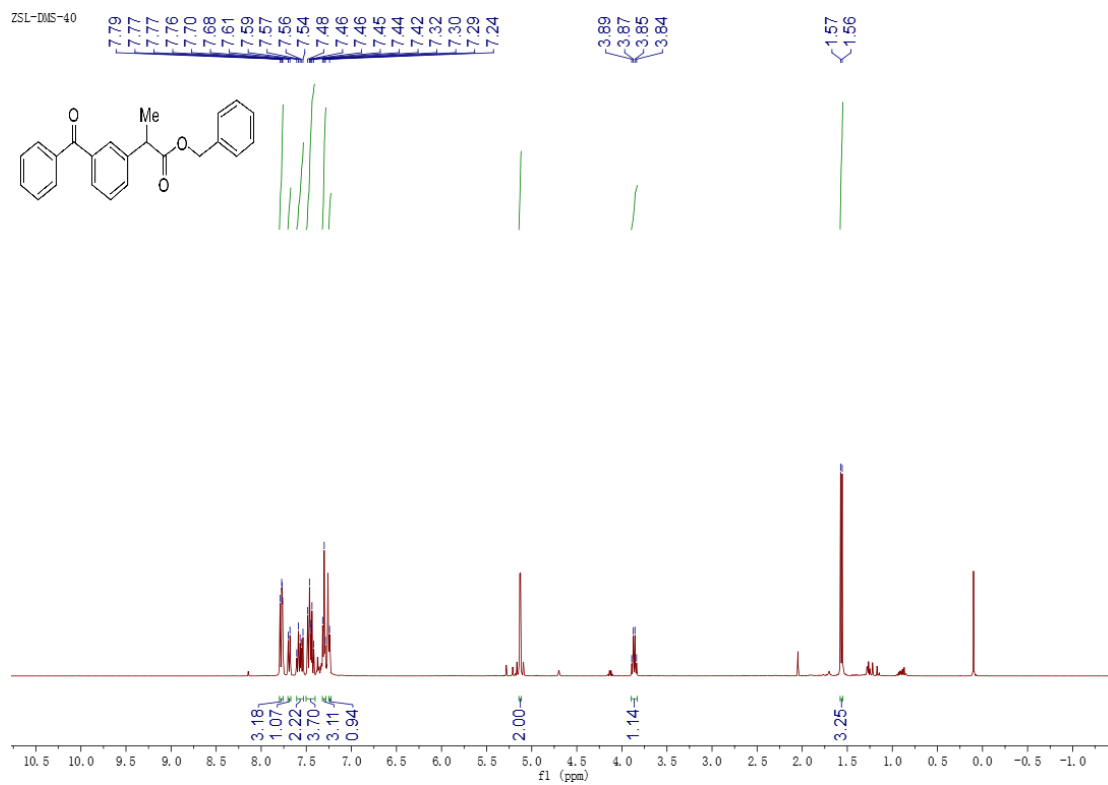
Benzyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (6a. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)



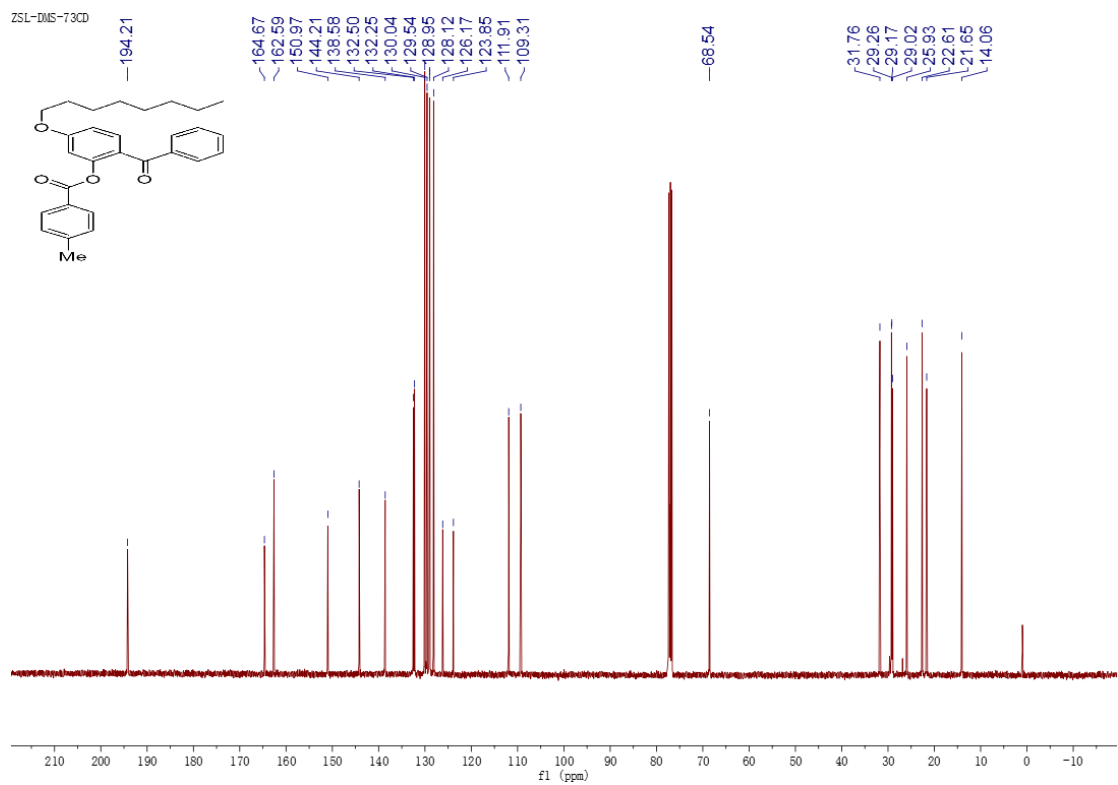
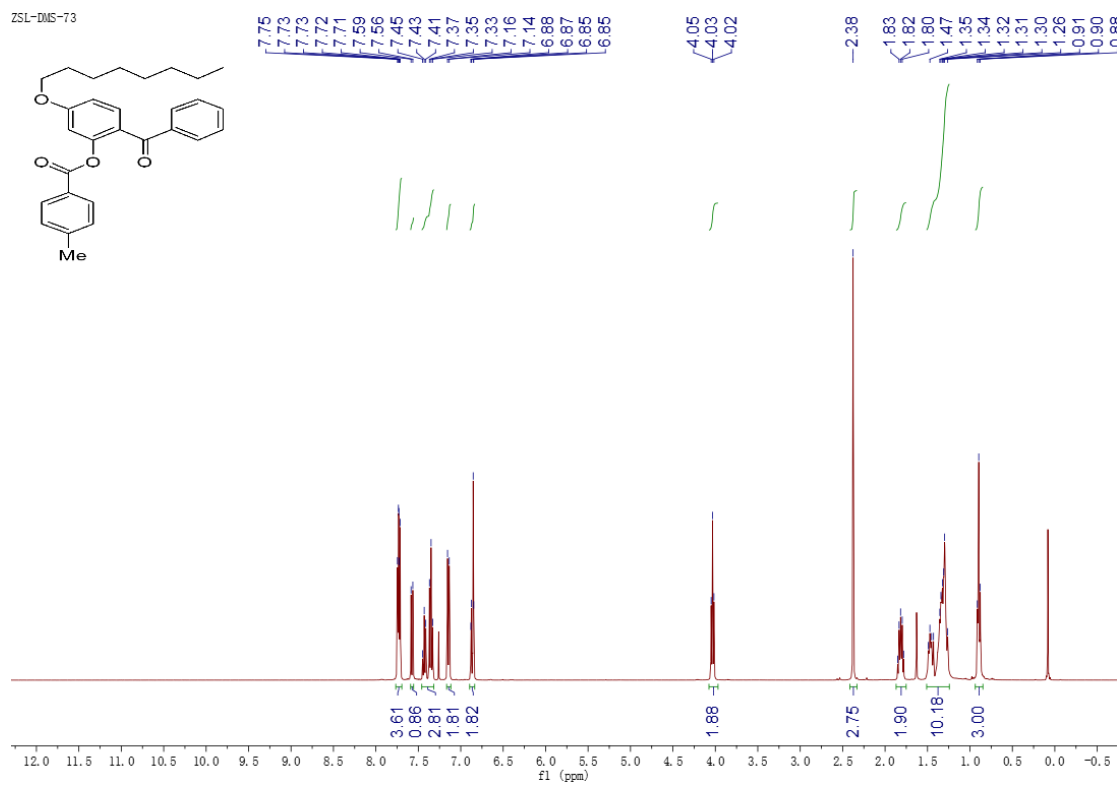
Benzyl 2-(6-methoxynaphthalen-2-yl)acetate (6b. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)



Benzyl 2-(3-benzoylphenyl)propanoate (6c. ^1H NMR 400 MHz, CDCl_3 ; ^{13}C NMR 101 MHz, CDCl_3)

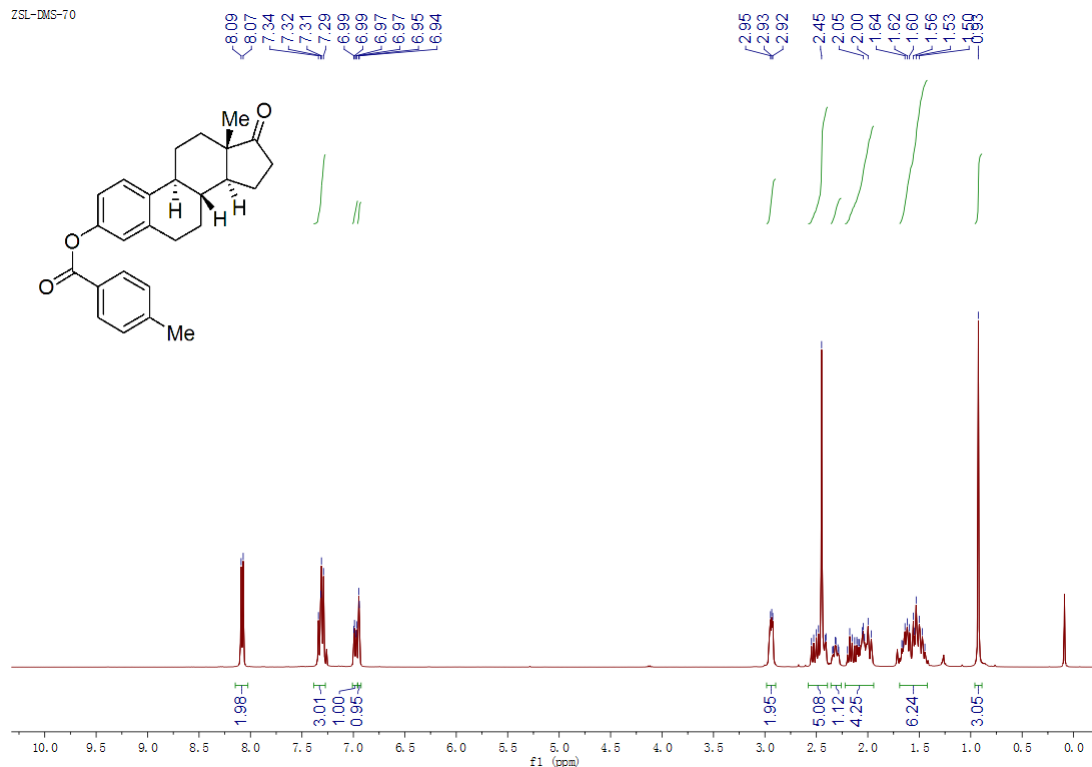


2-Benzoyl-5-(octyloxy)phenyl 4-methylbenzoate (6d). ^1H NMR 400 MHz, CDCl_3 ;
 ^{13}C NMR 101 MHz, CDCl_3



(8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthren-3-yl 4-methylbenzoate (6e. ¹H NMR 400 MHz, CDCl₃; ¹³C NMR 101 MHz, CDCl₃)

ZSL-DMS-70



ZSL-DMS-70CD

