Electronic Supplementary Material (ESI) for RSC Advances.

Mechanically Induced Solvent-Free Esterification Method at Room Temperature

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1. General Information

All reagents were obtained from commercial sources (purity > 99%) and used without fu rther purification unless otherwise indicated. All of the HSBM reactions were performed i n a Mixer Mill (MM 400 RetschGmbh, Hann, Germany) with 50 mL and milled with stai nless-steel balls. Thin-layer chromatography (TLC) was used to monitor the reaction. Melt ing points (mp) were obtained on a digital melting point apparatus (OptiMelt MPA100) a nd are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance II I instruments using tetramethylsilane (TMS, δ = 0 ppm) as the internal standard. Chemic al shifts (δ) were reported in ppm referenced to the $CDCl_3$ residual peak (δ 7.26) or the DMSO-d_6 residual peak (δ 2.50) for ^{1}H NMR. Chemical shifts of ^{13}C NMR were reported relative to $CDCl_3$ (δ 77.0) or DMSO- d_6 (δ 39.5). The abbreviations used are: s = singlet, d = doublet, t = triplet, dd = doublet of doublet, m = multiplet. Coupling constants (*J*) are given in Hz. Mass spectra were recorded with a high-resolution MS instrument (Bruk er Daltonics micro TOF II) and a low-resolution MS instrument (Finnigan Trace DSQ) usin g an ESI ion source.

2. General Procedure

I_2 catalytic procedure for the synthesis of Phenyl benzoate **3aa**:

A mixture of benzoic acid **1a** (0.5 mmol, 1.0 equiv), phenol **2a** (0.6 mmol, 1.2 equiv), I_2 (0.5 mmol, 1.0 equiv), KH_2PO_2 (0.5 mmol, 1.0 equiv.) and anhydrous sodium sulfate (0.4 g) was placed in a 50 mL stainless-steel vessel, along with two stainless-steel balls (ø=1.2 cm, Φ_{MB}=0.036). Then, the vessel was placed in the mixer mill, and the contents were milled at 25 Hz for 20 min. At the end of the experiment, the mixture was scratched from the vessel and purified by column chromatography (petroleum ether/ethyl acetate, 100:1) to give the desired product.

KI catalytic procedure for the synthesis of Phenyl benzoate **3aa**:

A mixture of benzoic acid **1a** (0.5 mmol, 1.0 equiv), phenol **2a** (0.6 mmol, 1.2 equiv), KI (0.75 mmol, 1.5 equiv), P(OEt)3 (0.5 mmol, 1.0 equiv.) and anhydrous sodium sulfate (0.4 g) was placed in a 50 mL stainless-steel vessel, along with two stainless-steel balls (ø=1.2 cm, *Φ*MB=0.036). Then, the vessel was placed in the mixer mill, and the contents were milled at 25 Hz for 60 min. At the end of the experiment, the mixture was scratc hed from the vessel and purified by column chromatography (petroleum ether/ethyl acet ate, 100:1) to give the desired product.

I_2-catalytic procedure for the synthesis of Inositol niacinate:

A mixture of niacin (369mg, 3.0 mmol.), inositol (108mg, 0.6 mmol.), I_2 (381mg, 3.0 mmol.), KH_2PO_2 (416mg 4.0 mmol.) and anhydrous sodium sulfate (0.4 g) was placed in a 50 mL stainless-steel vessel, along with two stainless-steel balls (ø=1.4 cm). Then, the vessel was placed in the Retsch MM400 mixer mill, and the contents were milled at 25 Hz for 90 min. At the end of the

experiment, the 20mL water was added to the vessel. Next, the vessel was placed in the Retsch PM400 mixer mill the contents were milled at 100 rpm for 20 min to fully dissolve the impurities. The mixture purified by suction filtration to give the crude inositol nicotinate **3df** (251mg, 62%).

3. Characterization Data of Products 3

Phenyl benzoate **(3aa)**[1]**.** White solid (Path A: 90mg, 91%; Path B: 81mg, 82%), mp 67-68 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.25 (d, *J* = 8.0 Hz, 2H), 7.67 (t, *J* = 8.0 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 2H), 7.47 (t, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.2, 151.0, 133.6, 130.2, 129.5, 128.6, 125.9, 121.7.

Phenyl 4-chlorobenzoate **(3ab)**[2]. White solid (Path A: 96mg, 83%; Path B: 94mg, 81%), m.p. 104-105 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.18 (d, *J* = 8.0 Hz, 2H), 7.57 – 7.43 (m, 4H), 7.32 (t, *J* = 8.0 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.34, 150.80, 140.14, 131.56, 129.56, 128.97, 128.06, 126.06, 121.64.

Phenyl 4-bromobenzoate **(3ac)**[3]. White solid (Path A: 103mg, 74%; Path B: 104mg, 75%), mp 103-104 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.09 (d, *J* = 8.0 Hz, 2H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.46 (t, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.5, 150.8, 132.0, 131.7, 129.5, 128.8, 128.5, 126.1, 121.6.

Phenyl 4-nitrobenzoate **(3ad)**[4]. Faint yellow solid (Path A: 76mg, 63%; Path B: 55mg, 45%) , ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.45 – 8.36 (m, 4H), 7.49 (t, *J* = 7.9 Hz, 2H), 7.34 (t, *J* = 7.4 Hz, 1H), 7.28 (d, *J* = 4.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 163.3, 150.9, 150.5, 135.0, 131.3, 129.7, 126.4, 123.7, 121.4.

Phenyl 4-cyanobenzoate **(3ae)**[20]. White solid (Path A: 78mg, 35%; Path B: 56mg, 25%), mp 165-166 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (d, *J* = 8.3 Hz, 2H), 7.84 (d, *J* = 8.2 Hz, 2H), 7.48 (t, *J* = 7.8 Hz, 2H), 7.33 (t, *J* = 7.4 Hz, 1H), 7.25 (d, *J* = 7.8 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.60, 150.54, 133.43, 132.42, 130.65, 129.68, 126.38, 121.46, 117.88, 117.01.

Phenyl 4-acetylbenzoate **(3af)**[21]. White solid (Path A: 185mg, 77%; Path B: 151mg, 63%), mp 132−134 °C; ^{1}H NMR (400 MHz, CDCl3) δ 8.32 (d, J = 8.5 Hz, 2H), 8.10 (d, J = 8.5 Hz, 2H), 7.48 (t, J = 7.9 Hz, 2H), 7.32 (t, J = 7.4 Hz, 1H), 7.26 (d, J = 7.6 Hz, 2H), 2.70 (s, 3H). ^{13}C NMR (101 MHz, CDCl3) δ 197.47, 164.34, 150.74, 140.71, 133.34, 130.43, 129.60, 128.37, 126.17, 121.58, 26.97.

Phenyl 3-methoxybenzoate **(3ag)**[5]. Colorless solid (Path A: 96mg, 84%; Path B: 84mg, 78%) , mp 64–66 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.84 (d, *J* = 8.0 Hz, 1H), 7.74 (s, 1H), 7.50 – 7.42 (m, 3H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.27 – 7.19 (m, 3H), 3.92 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.1, 159.7, 151.0, 131.0, 129.6, 129.5, 125.9, 122.6, 121.7, 120.2, 114.5, 55.5.

Phenyl 4-methylbenzoate **(3ah)**[6]. White solid (Path A: 93mg, 88%; Path B: 73mg, 69%), mp 77.5−78 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.13 (d, *J* = 8.0 Hz, 2H), 7.51 – 7.43 (m, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.3, 151.1, 144.4, 130.2, 129.5, 129.3, 126.8, 125.8, 121.8.

Phenyl 3-methylbenzoate **(3ai)**[7]**.** White solid (Path A: 68mg, 64%; Path B: 62mg, 58%), mp 61− 62 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.12 (d, *J* = 8.0 Hz, 2H), 7.53 – 7.43 (m, 4H), 7.39 – 7.28 (m, 3H), 2.51 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.4, 151.1, 138.5 , 134.4, 130.8, 129.6, 129.6, 128.6, 127.4, 125.9, 121.8, 21.4.

Phenyl 2-naphthoate **(3aj)**[3]. White solid (Path A: 105mg, 85%; Path B: 55mg, 44%), mp 93−94 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 9.13 (s, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.47 (q, *J* = 7.0, 6.2 Hz, 4H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.34 (d, *J* = 7.1 Hz, 1H), 7.29 (d, *J* = 8.2 Hz, 3H), 7.22 (t, *J* = 7.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 160.5, 150.5, 137.3, 12 9.6, 127.5, 126.4, 126.1, 126.0, 122.8, 121.7, 121.1, 112.0, 110.3.

Phenyl nicotinate **(3ak)**[5]**.** Faint yellow solid (Path A: 83mg, 83%; Path B: 85mg, 85%), m p 74–75 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 9.43 (s, 1H), 8.87 (d, *J* = 4.0 Hz, 1H), 8.48 (d, *J* = 8.0 Hz, 1H), 7.52 – 7.43 (m, 3H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 163.8, 153.9, 151.3, 150.5, 137.7, 129.6, 126.3, 125. 7, 123.6, 121.6.

Phenyl butyrate **(3al)**[8]**.** Colorless oil (Path A: 44mg, 53%; Path B: 0mg, 0%), ^{1}H NMR (4 00 MHz, $CDCl_3$) δ = 7.40 (t, *J* = 8.0 Hz, 2H), 7.25 (t, *J* = 8.0 Hz, 1H), 7.11 (d, *J* = 8.0 Hz, 2H), 2.57 (t, *J* = 8.0 Hz, 2H), 1.88 – 1.76 (m, 2H), 1.08 (t, *J* = 8.0 Hz, 3H). ^{13}C NM R (100 MHz, $CDCl_3$) δ = 172.1, 150.8, 129.4, 125.7, 121.6, 36.3, 18.5, 13.7.

p-Tolyl benzoate **(3ba)**[9]**.** White solid (Path A: 92mg, 87%; Path B: 80mg, 75%), mp 70-7 1.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.24 (d, *J* = 8.0 Hz, 2H), 7.66 (t, *J* = 8.0 Hz, 1H), 7.54 (t, *J* = 8.0 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.4, 148.7, 135.5, 133.5, 130.2, 130.0, 129.7, 128.5, 12 1.4, 20.9.

4-(*Tert*-butyl)phenyl benzoate **(3bb)**[17]. White solid (Path A: 97mg, 76%; Path B: 83mg, 6 5%), mp 76-77 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.24 (d, *J* = 8.0 Hz, 2H), 7.66 (t, *J* = 8.0 Hz, 1H), 7.54 (t, *J* = 8.0 Hz, 2H), 7.47 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 1.37 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.3, 148.7, 148.6, 133.5, 130.3, 129.8, 12 8.5, 126.4, 121.0, 34.5, 31.5.

4-Fluorophenyl benzoate **(3bc)**[5]. White solid (Path A: 95mg, 88%; Path B: 70mg, 65%), mp 55-56 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.22 (d, *J* = 8.0 Hz, 2H), 7.68 (t, *J* = 8.0 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 2H), 7.24 – 7.19 (m, 2H), 7.19 – 7.09 (m, 2H). ^{13}C NMR (1 00 MHz, $CDCl_3$) δ = 165.2, 160.3 (d, J_{C-F} = 244.2 Hz), 146.8 (d, J_{C-F} = 2.9 Hz), 133.8, 13 0.2, 129.3, 128.6, 123.1 (d, J_{C-F} = 8.5 Hz), 116.2 (d, J_{C-F} = 23.5 Hz).

4-Chlorophenyl benzoate **(3bd)**[10]**.** White solid (Path A: 103mg, 92%; Path B: 78mg, 67%), mp 88–89 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.22 (d, *J* = 8.0 Hz, 2H), 7.68 (t, *J* = 8.0 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 2H), 7.42 (d, *J* = 12.0 Hz, 2H), 7.20 (d, *J* = 12.0 Hz, 2H). ^{1} ^{3}C NMR (100 MHz, $CDCl_3$) δ = 165.0, 149.4, 133.8, 131.3, 130.2, 129.6, 129.2, 128.7, 12 3.1.

4-Bromophenyl benzoate **(3be)**[5]**.** White solid (Path A: 132mg, 95%; Path B: 105mg, 76%), mp 99-100.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.22 (d, *J* = 8.0 Hz, 2H), 7.68 (t, *J* = 8. 0 Hz, 1H), 7.60 – 7.51 (m, 4H), 7.15 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.9, 150.0, 133.8, 132.6, 130.2, 129.2, 128.7, 123.6, 119.0.

4-(Trifluoromethyl)phenyl benzoate **(3bf)**[11]**.** White solid (Path A: 125mg, 94%; Path B: 71 mg, 53%), mp 107-108.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.24 (d, *J* = 8.0 Hz, 2H), 7.

74 (d, *J* = 8.0 Hz, 2H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.56 (t, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ = 164.6, 153.5, 134.0, 130.3, 129.0, 128.7, 128.2 (q, J_{C-F} = 33.0 Hz), 126.9 (q, J_{C-F} = 3.7 Hz), 123.9 (q, J_{C-F} = 270.3 Hz), 122.3.

4-nitrophenyl benzoate **(3bg)**[20]. White solid (Path A: 163mg, 67%; Path B: 104mg, 43%), mp 139-140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (d, *J* = 9.1 Hz, 2H), 8.23 (d, *J* = 7.3 Hz, 2H), 7.71 (t, *J* = 7.4 Hz, 1H), 7.58 (d, *J* = 7.9 Hz, 2H), 7.45 (d, *J* = 9.1 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.25, 155.75, 145.40, 134.28, 130.34, 128.82, 128.54, 125.29, 122.66.

4-cyanophenyl benzoate **(3bh)**[20]. White solid (Path A: 192mg, 86%; Path B: 151mg, 68%), mp 98-99.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 7.1 Hz, 2H), 7.76 (d, *J* = 8.7 H z, 2H), 7.70 (t, *J* = 7.5 Hz, 1H), 7.56 (t, *J* = 7.8 Hz, 2H), 7.40 (d, *J* = 8.7 Hz, 2H). ^{13}C N MR (101 MHz, $CDCl_3$) δ 164.34, 154.27, 134.20, 133.76, 130.31, 128.79, 128.65, 122.96, 118.31, 109.83.

4-acetylphenyl benzoate **(3bi)**[21]. White solid (Path A: 84mg, 35%; Path B: 130mg, 54%),mp 120-121 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, *J* = 7.1 Hz, 2H), 8.08 (d, *J* = 8.7 Hz, 2H), 7.69 (t, *J* = 7.4 Hz, 1H), 7.56 (t, *J* = 7.7 Hz, 2H), 7.36 (d, *J* = 8.7 Hz, 2H), 2.66 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.91, 164.65, 154.70, 134.81, 133.95, 130.27, 130.03, 129.05, 128.70, 121.96, 26.67.

4-Methoxyphenyl benzoate **(3bj)**[12]. White solid (Path A: 86mg, 75%; Path B: 80mg, 70%), mp 89-90 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.23 (d, *J* = 8.0 Hz, 2H), 7.66 (t, *J* = 8.0 Hz, 1H), 7.54 (t, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 8.0 Hz, 2H), 3.8 5 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.6, 157.3, 144.4, 133.5, 130.1, 129.7, 128.6, 122.5, 114.5, 55.6.

2, 4-Dimethoxyphenyl benzoate **(3bk)**. Colorless oil (Path A: 74mg, 57%; Path B: 41mg, 32%), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.27 (d, *J* = 8.0 Hz, 2H), 7.65 (t, *J* = 8.0 Hz, 1H), 7.53 (t, *J* = 8.0 Hz, 2H), 7.20 (t, *J* = 8.0 Hz, 1H), 6.69 (d, *J* = 12.0 Hz, 2H), 3.84 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.5, 152.6, 133.3, 130.4, 129.4, 128.9, 128.4, 126.3, 10 5.0, 56.2. ESI-HRMS m/z: 281.2607 $[M+Na]^+$; $C_{15}H_{14}O_4$: 281.2622.

Methyl benzoate **(3ca)**[13]**.** Colorless oil (Path A: 43mg, 63%; Path B: 16mg, 24%), ^{1}H NM R (400 MHz, $CDCl_3$) δ = 8.07 (d, *J* = 8.0 Hz, 2H), 7.58 (t, *J* = 8.0 Hz, 1H), 7.46 (t, *J* = 8.0 Hz, 2H), 3.95 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.1, 132.9, 130.2, 129.6, 128.4 , 52.1.

Ethyl benzoate **(3cb)**[13]**.** Colorless oil (Path A: 54mg, 72%; Path B: 24mg, 32%), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.07 (d, *J* = 8.0 Hz, 2H), 7.57 (t, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 8.0 Hz, 2H), 4.40 (q, *J* = 8.0 Hz, 2H), 1.42 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.6, 132.8, 130.5, 129.5, 128.3, 60.9, 14.3.

Butyl benzoate **(3cc)**[14]**.** Colorless oil (Path A: 67mg, 75%; Path B: 38mg, 43%), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.07 (d, *J* = 8.0 Hz, 2H), 7.56 (t, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 8.0 Hz, 2H), 4.35 (t, *J* = 8.0 Hz, 2H), 1.77 (dt, *J* = 16.0, 8.0 Hz, 2H), 1.57 – 1.42 (m, 2H), 1.0 0 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.7, 132.8, 130.5, 129.5, 128.3, 64.8, 30.8, 19.3, 13.8.

(2,2-dimethyl-1,3-dioxolan-4-yl)methyl benzoate **(3cd).** Colorless oil (Path A: 53mg, 45%; Path B: 0mg, 0%), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.05 (d, *J* = 8.0 Hz, 2H), 7.55 (t, *J* = 8.0 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 2H), 4.54 – 4.24 (m, 3H), 4.13 (dd, *J* = 8.0, 8.0 Hz, 1H), 3.87 (dd, *J* = 8.0, 4.0 Hz, 1H), 1.45 (s, 3H), 1.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ

= 166.3, 133.1, 129.8, 129.7, 128.4, 109.8, 73.7, 66.4, 65.0, 26.7, 25.4. ESI-HRMS m/z: 259.2577 $[M+Na]^+$; $C_{13}H_{16}O_4$: 236.2670.

(*S*)-1-phenylethyl benzoate **(*S*-3da)**[24]**.**

$[\alpha]_D^{25}$= + 22.2° (*c* 1.0, CH_2Cl_2, *S*, 58% *ee*)

Colorless oil (Path A: 194mg, 86%)**;** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 6.8 Hz, 2H), 7.59 (t, *J* = 6.8 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 4H), 7.41 (t, *J* = 7.4 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 1H), 6.18 (q, *J* = 6.6 Hz, 1H), 1.72 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.82, 141.81, 132.94, 130.54, 129.67, 128.57, 128.35, 127.91, 126.07, 72.94, 22.45. Chiral HPLC: column chiralpak AD-H (4.6 mm x 250 mm, 5 µm), mobile phase: 95:5 i-hexane:i-PrOH, flow rate 1 mL/min, λ = 210 nm, temperature: 30 °C, retention time: t_R (minor) = 5.7 min, t_R (major) = 6.9 min.

(*R*)-1-phenylethyl benzoate **(*R*-3da)**[24]**.**

$[\alpha]_D^{25}$= - 32.0° (*c* 1.0, CH_2Cl_2, *R*, 83% *ee*)

Colorless oil (Path B: 147mg, 65%)**;** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 6.8 Hz, 2H), 7.59 (t, *J* = 6.8 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 4H), 7.41 (t, *J* = 7.4 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 1H), 6.18 (q, *J* = 6.6 Hz, 1H), 1.72 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.82, 141.81, 132.94, 130.54, 129.67, 128.57, 128.35, 127.91, 126.07, 72.94, 22.45. Chiral HPLC: column chiralpak AD-H (4.6 mm x 250 mm, 5 µm), mobile phase: 95:5 i-hexane:i-PrOH, flow rate 1 mL/min, λ = 210 nm, temperature: 30 °C, retention time: t_R (major) = 5.8 min, t_R (minor) = 7.0 min.

1-(*tert*-butyl) 2-phenyl (*S*)-pyrrolidine-1,2-dicarboxylate **(3db)**[25].

$[\alpha]_D^{20}$=+ 17.8 (*c* 1.0, CH_2Cl_2, *S*, 75% *ee*)

Colorless oil (Path A: 67mg, 23%,; Path B: trace); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.32 (m, 2H), 7.24 (dd, *J* = 11.9, 7.2 Hz, 1H), 7.12 (t, *J* = 9.8 Hz, 2H), 4.51 (m, 1H), 3.73 – 3.40 (m, 2H), 2.39 (m, 1H), 2.27 – 1.89 (m, 3H), 1.50 (d, *J* = 4.5 Hz, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.62, 153.76, 150.59, 129.50, 129.35, 125.93, 125.78, 121.47, 121.14, 80.22, 79.97, 59.20, 46.46, 31.07, 30.03, 28.44, 24.50, 23.72. Chiral HPLC: column chiralpak AD-H (4.6 mm x 250 mm, 5 µm), mobile phase: 95:5 i-hexane:i-PrOH, flow rate 1 mL/min, λ = 210 nm, temperature: 30 °C, retention time: t_R (major) = 8.1 min, t_R (minor) = 9.2 min.

Phenyl (*tert*-butoxycarbonyl)-*L*-valinate **(3dc)**[25].

$[\alpha]_D^{20}$=+ 18.7 (c=1.0, CH_2Cl_2, *S*, 78% *ee*)

Colorless oil (Path A: 79mg, 27%,; Path B: 17 mg, 6%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (t, *J* = 7.9 Hz, 2H), 7.26 (d, *J* = 7.4 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 2H), 5.11 (d, *J* = 8.5 Hz, 1H), 4.50 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.36 (dt, *J* = 13.0, 6.5 Hz, 1H), 1.49 (s, 9H), 1.11 (d, *J* = 6.9 Hz, 3H), 1.05 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.13, 155.72, 150.44, 129.51, 126.08, 121.39, 80.00, 58.67, 31.42, 28.34, 19.12, 17.68. Chiral HPLC: column chiralpak AD-H (4.6 mm x 250 mm, 5 µm), mobile phase: 95:5 i-hexane:i-PrOH, flow rate 1 mL/min, λ = 210 nm, temperature: 30 °C, retention time: t_R (major) = 11.3 min, t_R (minor) = 16.3 min.

2-Isopropyl-5-methylphenyl benzoate **(3ea).** White solid (Path A: 83mg, 65%), mp 34-36 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.32 (s, 2H), 7.71 (t, *J* = 8.0 Hz, 1H), 7.59 (t, *J* = 8.0 Hz, 2H), 7.42 – 7.31 (m, 1H), 7.16 (s, 1H), 7.04 (s, 1H), 3.30 – 3.04 (m, 1H), 2.43 (s, 3 H), 1.39 – 1.26 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.4, 148.3, 137.3, 136.7, 133. 6, 130.2, 129.7, 128.7, 127.3, 126.6, 122.98, 122.96, 27.4, 23.1, 20.9. ESI-HRMS m/z: 277.3 215 $[M+Na]^+$; $C_{17}H_{18}O_2$: 277.3182.

2-Acetyl-5-methoxyphenyl benzoate **(3eb)**[15]**.** Faint yellow oil (Path A: 99mg, 73%), ^{1}H N MR (400 MHz, $CDCl_3$) δ = 8.24 (d, *J* = 8.0 Hz, 2H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.68 (t, *J*

= 8.0 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 2H), 6.90 (dd, *J* = 8.0, 2.5 Hz, 1H), 6.75 (d, *J* = 2.5 Hz, 1H), 3.89 (s, 3H), 2.52 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 195.7, 165.1, 163.8, 151.7, 133.8, 132.4, 130.3, 129.4, 128.7, 123.6, 112.0, 109.3, 55.8, 29.5. ESI-HRMS m/z: 293.2713 $[M+Na]^+$; $C_{16}H_{14}O_4$: 293.2732.

4-Allyl-2-methoxyphenyl benzoate **(3ec)**[16]**.** White solid (Path A: 62mg, 46%), mp 67-68.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.25 (d, *J* = 8.0 Hz, 2H), 7.65 (t, *J* = 8.0 Hz, 1H), 7.53 (t, *J* = 8.0 Hz, 2H), 7.10 (d, *J* = 8.0 Hz, 1H), 6.91 – 6.82 (m, 2H), 6.08 – 5.96 (m, 1H), 5.34 – 4.82 (m, 2H), 3.83 (s, 3H), 3.44 (d, *J* = 4.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.0, 151.2, 139.1, 138.3, 137.1, 133.4, 130.3, 129.6, 128.5, 122.7, 120.8, 116.1, 112.9, 55.9, 40.1. ESI-HRMS m/z: 291.3025 $[M+Na]^+$; $C_{17}H_{16}O_3$: 291.3012.

(3*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl benzoate **(3ed)**[17]**.** White solid (Path A: 130mg, 53%), mp 190.5-192 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.07 (d, *J* = 8.0 Hz, 2H), 7.57 (t, *J* = 8.0 Hz, 1H), 7.46 (t, *J* = 8.0 Hz, 2H), 5.45 (d, *J* = 3.6 Hz, 1H), 4.97 – 4.81 (m, 1H), 2.49 (d, *J* = 7.6 Hz, 2H), 2.09 – 1.11 (m, 24H), 1.10 (s, 3H), 1.07 – 1.02 (m, 2H), 0.95 (d, *J* = 6.5 Hz, 3H), 0.89 (d, *J* = 6.6, 6H), 0.72 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.0, 139.7, 132.7, 130.9, 129.5, 128.3, 122.8, 74.6, 56.7, 56.2, 50.1, 42.3, 39.8, 39.5, 38.2, 37.1, 36.7, 36.2, 35.8, 32.0, 31.9, 28.2, 28.0, 27.9, 24.3, 23.8, 22.8, 22.6, 21.1, 19.4, 18.7, 11.9. ESI-HRMS m/z: 513.7607 $[M+Na]^+$; $C_{34}H_{50}O_2$: 513.7612.

Phenyl(4a*S*,6a*S*,6b*R*,8a*R*,10*S*,12a*R*,12b*R*,14b*S*)-10-hydroxy-2,2,6a,6b,9,9,12a-heptamethyl-1,3,4,5,6,6a,6b,7,8,8a,9,10,11,12,12a,12b,13,14b-octadecahydropicene-4a(2*H*)-carboxylate **(3ee)**[18]**.** White solid (Path A: 61mg, 23%), mp 156-159 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.07 (d, *J* = 7.1 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 5.32 (s, 1H), 4.77 (dd, *J* = 10.7, 5.4 Hz, 1H), 2.86 (dd, *J* = 13.7, 3.9 Hz, 1H), 2.09 – 1.29 (m, 18H), 1.29 – 1.09 (m, 7H), 1.03 (d, *J* = 10.2 Hz, 6H), 0.99 – 0.91 (m, 10H), 0.80 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.3, 143.6, 132.7, 131.0, 129.5, 128.3, 122.6, 81.6, 55.4, 47.6, 46.5, 45.9, 41.6, 41.0, 39.3, 38.1, 37.0, 33.8, 33.1, 32.6, 32.4, 30.7, 28.2, 27.7, 26.0, 23.6, 23.4, 22.9, 18.2, 17.2, 17.0, 15.4.

Inositol niacinate **(3ef).** White solid; mp 254-256 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ = 9.39 (d, *J* = 1.7 Hz, 1H), 9.02 (d, *J* = 1.7 Hz, 1H), 8.98 (dd, *J* = 4.8, 1.6 Hz, 1H), 8.88 (dd, *J* = 30.4, 1.7 Hz, 4H), 8.73 (d, *J* = 4.8 Hz, 5H), 8.51 (d, *J* = 8.0 Hz, 1H), 8.20 (d, *J* = 8.0 Hz, 1H), 8.09 (t, *J* = 8.4 Hz, 4H), 7.74 (dd, *J* = 7.9, 4.9 Hz, 1H), 7.49 (dt, *J* = 8.8, 4.7 Hz, 5H), 6.57 – 6.46 (m, 1H), 6.42 (t, *J* = 10.1 Hz, 2H), 6.34 (d, *J* = 8.2 Hz, 3H), 2.51 (s, 6H). ^{13}C NMR (100 MHz, DMSO-d_6) δ = 164.8, 164.7, 164.4, 164.0, 154.7, 154.6, 150.3, 137.9, 137.4, 137.2, 137.2, 124.9, 124.8, 124.5, 124.5, 71.3, 71.2, 70.3, 70.0. ESI-HRMS m/z: 811.7367 $[M+H]^+$; $C_{34}H_{50}O_2$: 811.7395.

N-phenylbenzamide **(3fa)**[22]. White solid; mp 117-119 °C; (Path A: 69mg, 35%; Path B: 130mg, 66%), ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.1 Hz, 3H), 7.67 (d, *J* = 7.7 Hz, 2H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.51 (t, *J* = 7.4 Hz, 2H), 7.40 (t, *J* = 7.9 Hz, 2H), 7.18 (t, *J* = 7.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.76, 137.93, 135.01, 131.86, 129.12, 128.81, 127.03, 124.59, 120.21.

S-(*m*-tolyl) benzothioate **(3fb)**[23]. Colorless oil; (Path A: 36mg, 16%; Path B: 130mg, 57%), ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 7.9 Hz, 2H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.7 Hz, 2H), 7.41 – 7.34 (m, 3H), 7.29 (d, *J* = 7.8 Hz, 1H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, CDCl3) δ 190.3, 139.1, 136.7, 135.6, 133.5, 132.1, 130.4, 129.0, 128.7, 127.4, 126.9, 21.3.

4. Control Experiments

Synthesis ^{18}O-labeled phenol[19]

Aniline (0.91 mL, 10 mmol) was dissolved in water (3.5 mL) and 50% tetrafluoroboric acid (3.5 mL) was added. The solution was cooled to 0 °C, and a solution of sodium nitrite (700 mg, 10.1 mmol) in water (1.5 mL) was added drop wise. The suspension was stirred keeping 0 °C for 30 min, and the mixture was tfiltered, solid materials was purified by re-precipitation from acetone/diethyl ether (5:1) solution. Benzenediazonium tetrafluoroborate was obtained in 63% yield (1.2g, 6.32 mmol) dried under vacuum. Concentrated sulfuric acid (62.5 µL) was added to a stirred paste of benzenediazonium tetrafluoroborate (0.5g, 2.6 mmol) in 98% [^{18}O]water (0.5 mL). The mixture was then heated to 65 °C until evolution of nitrogen ceased. The solution was extracted with diethyl ether several times. The ethereal layer was washed with 1 M HCl and saturated brine, dried over magnesium sulfate, and concentrated under reduced pressure. The crystaline residue was purified by sublimation to give the ^{18}O-labelled phenol as colourless solid (106.8 mg, 43%). The ^{18}O-enrichment of phenol was found by ESI-MS to be 59%.

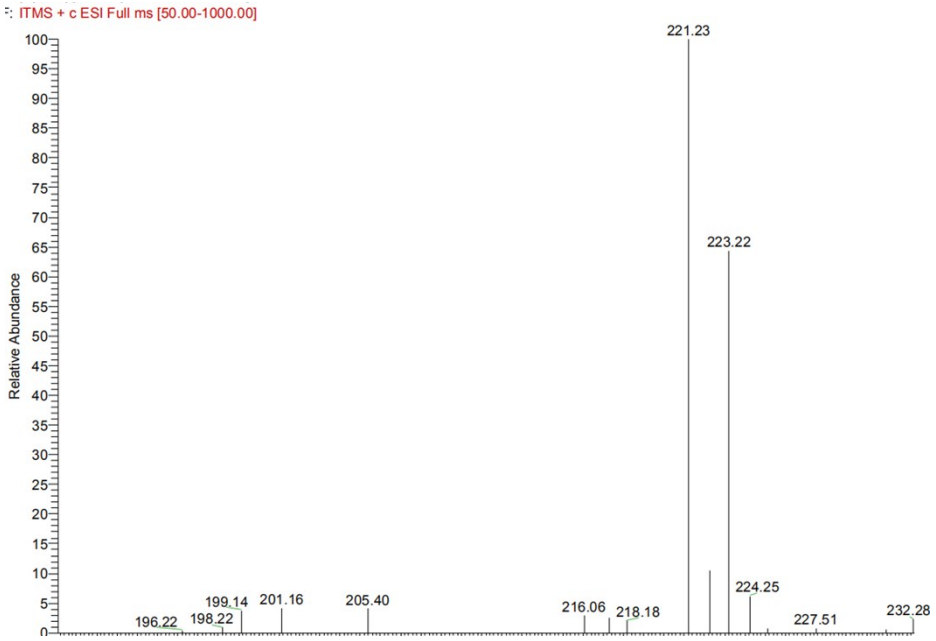
The I_2-catalytic reaction of benzoic acid 1a and ^{18}O-labeled phenol

A mixture of benzoic acid **1a** (0.5 mmol, 1.0 equiv.), ^{18}O-labeled phenol **2a** (0.6 mmol, 1.2 equiv.), I2 (0.5 mmol, 1.0 equiv.), KH_2PO_2 (0.5 mmol, 1.0 equiv.) and anhydrous sodium sulfate (0.4 g) was placed in a 50 mL stainless-steel vessel, along with two stainless-steel balls (ø=1.2 cm, ΦMB=0.036). Then, the vessel was placed in the mixer mill, and the contents were milled at 25 Hz for 20 min. At the end of the experiment, the mixture was scratched from the vessel and purified by column chromatography (petroleum ether/ethyl acetate, 100:1) to give the desired product. The ^{18}O-enrichment of **3aa** was found by ESI-MS to be 0%.

The KI-catalytic reaction of benzoic acid 1a and ^{18}O-labeled phenol

A mixture of benzoic acid **1a** (0.5 mmol, 1.0 equiv.), phenol **2a** (0.6 mmol, 1.2 equiv.), KI (0.75 mmol, 1.5 equiv.), $P(OEt)_3$ (0.5 mmol, 1.0 equiv.) and anhydrous sodium sulfate (0.4 g) was placed in a 50 mL stainless-steel vessel, along with two stainless-steel balls (ø=1.2 cm, Φ_{MB}=0.036). Then, the vessel was placed in the mixer mill, and the contents were milled at 25 Hz for 60 min. At the end of the experiment, the mixture was scratched from the vessel and purified by column chromatography (petroleum ether/ethyl acetate, 100:1) to give the desired product. The ^{18}O-enrichment of **3aa** was found by ESI-MS to be 43%.

Figure S1 : ESI-MS for ^{18}O-enrichment of **3aa**



m/z	Intensity	Relative
221.23	33153	100
223.22	24864.8	75
237.16	6715	20.26
295.38	11451.6	34.54
419.08	28703.8	86.58
420.4	12682.8	38.25

Figure S2: Depiction of the ^{31}P-NMR spectrum for **Scheme 3c**

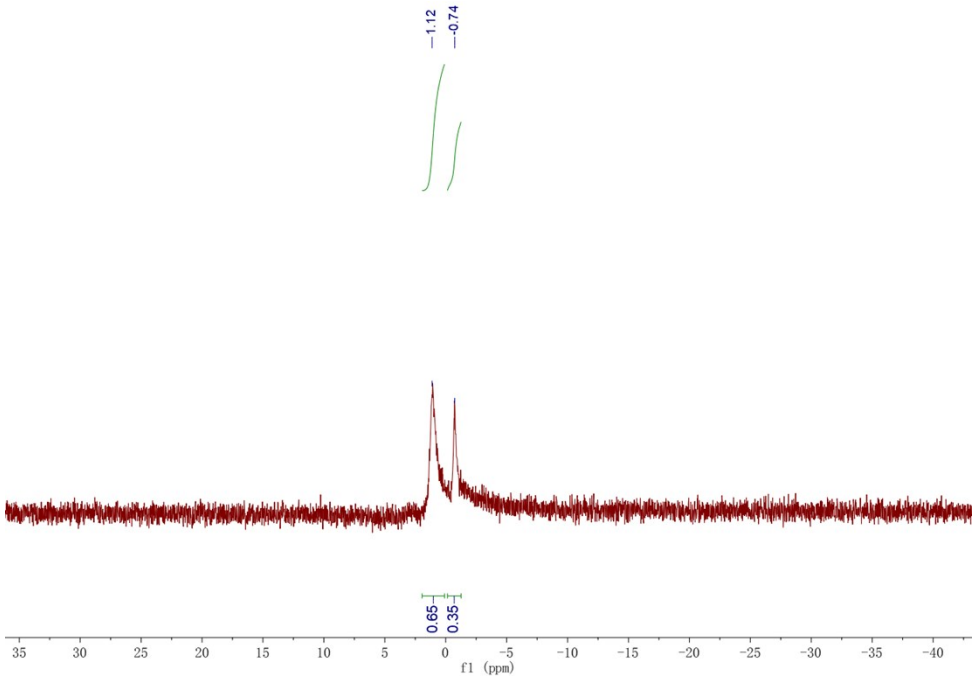
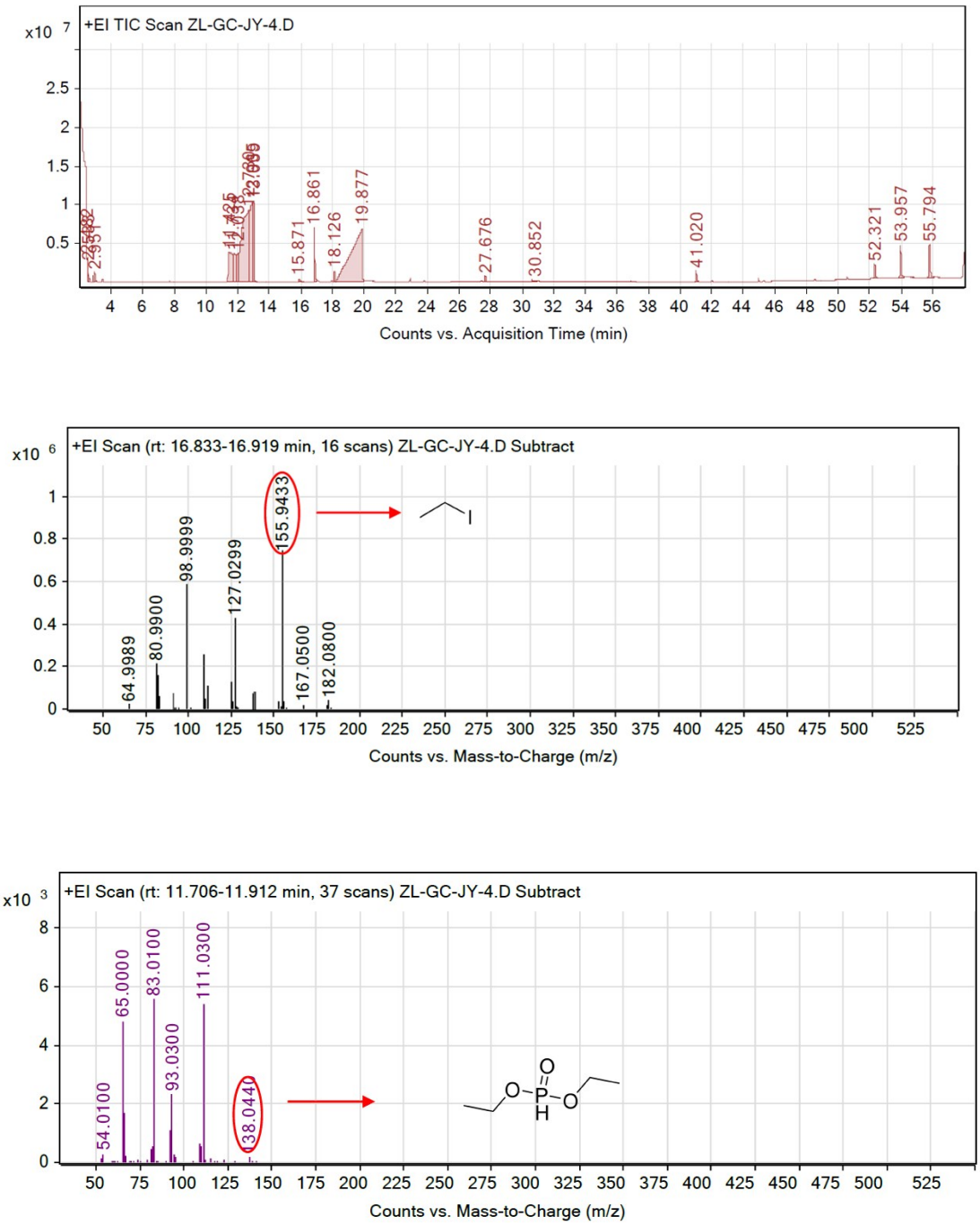
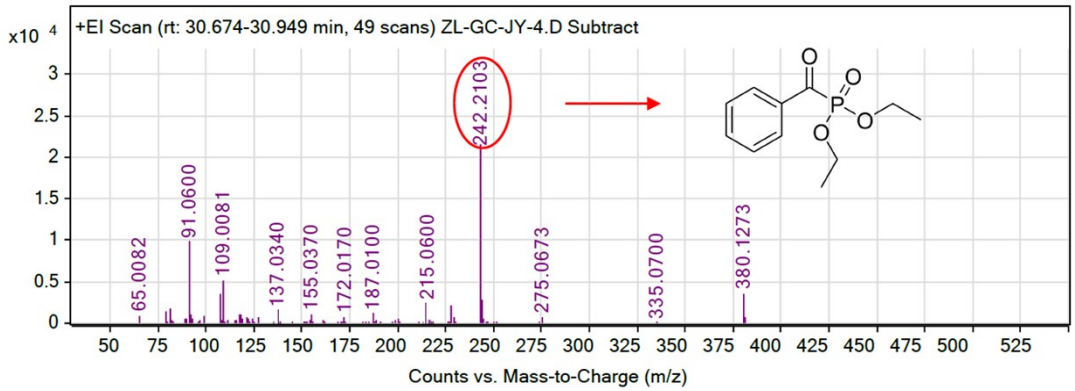


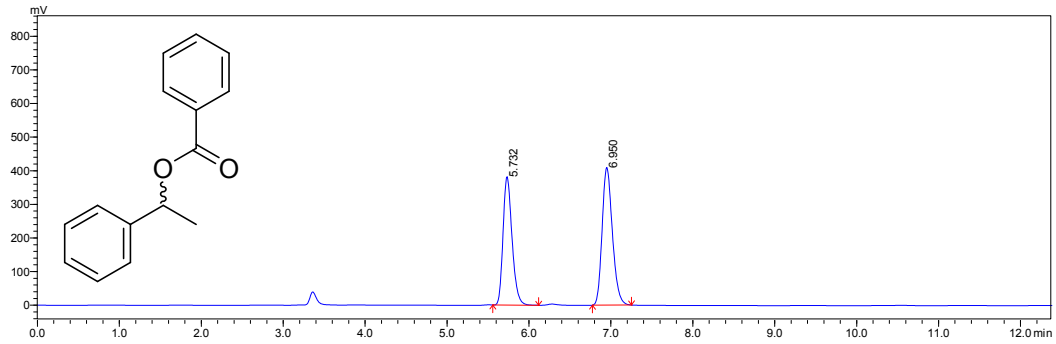
Figure S3: GC-MS for **Scheme 4 (Path B)**





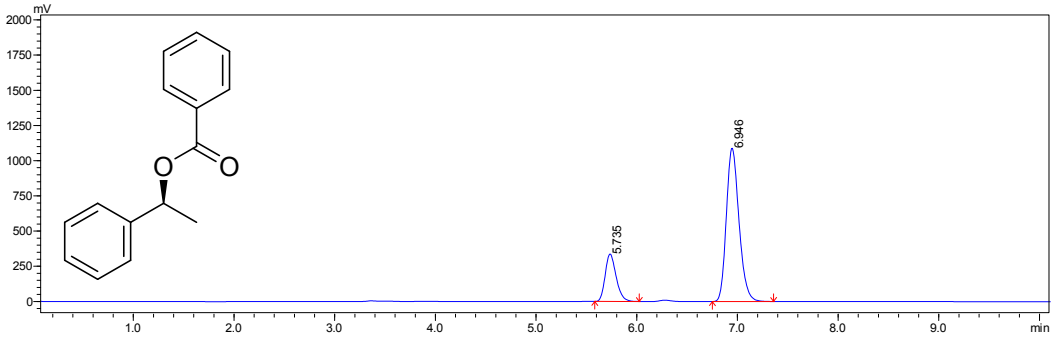
5. HPLC and SFC Chromatograms

Racemic **3da**



RT [min]	Area	Height	Area%
5.732	3923.471	381.772	45.058
6.950	3564.773	409.406	54.942

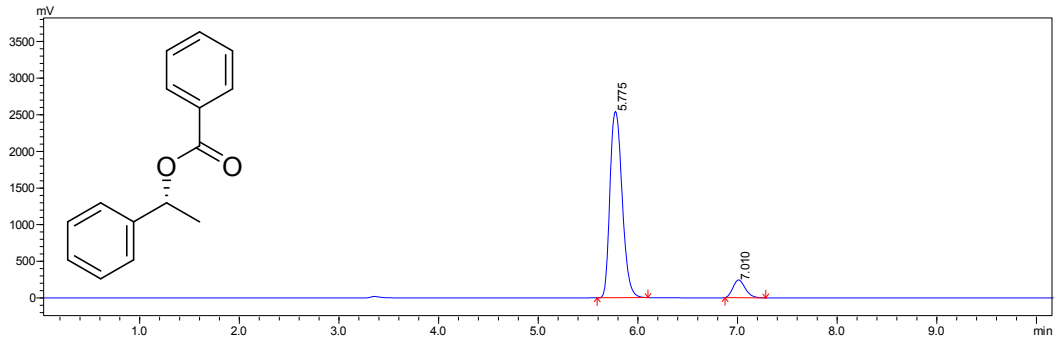
S-**3da**



RT [min]	Area	Height	Area%
5.735	2551.941	337.262	21.131

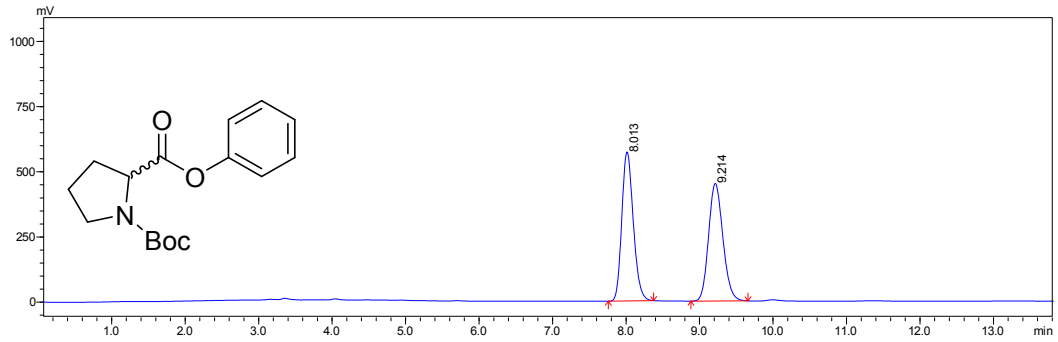
6.946	9524.575	1088.411	78.869

R-**3da**



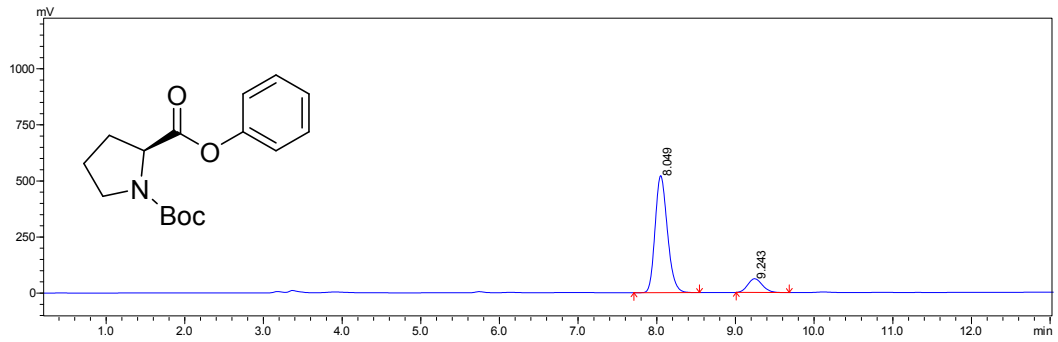
RT [min]	Area	Height	Area%
5.775	21630.721	2543.264	91.345
7.010	2049.453	240.967	8.655

Racemic **3db**



RT [min]	Area	Height	Area%
8.013	6211.930	571.570	50.405
9.214	6112.082	450.938	49.595

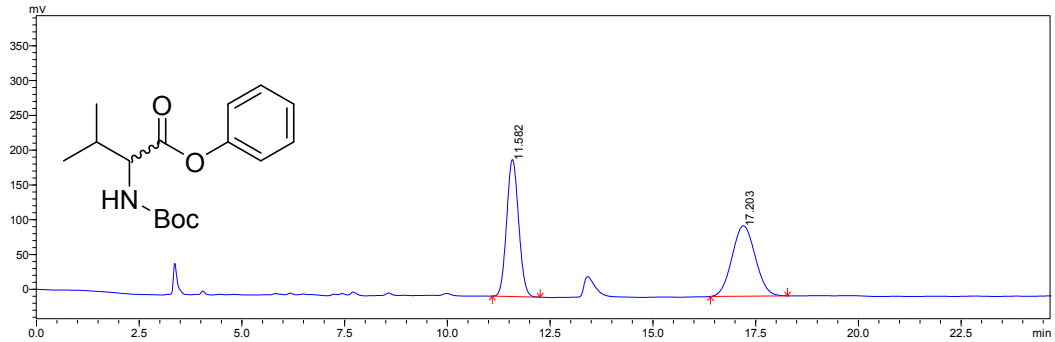
S-**3db**



RT [min]	Area	Height	Area%

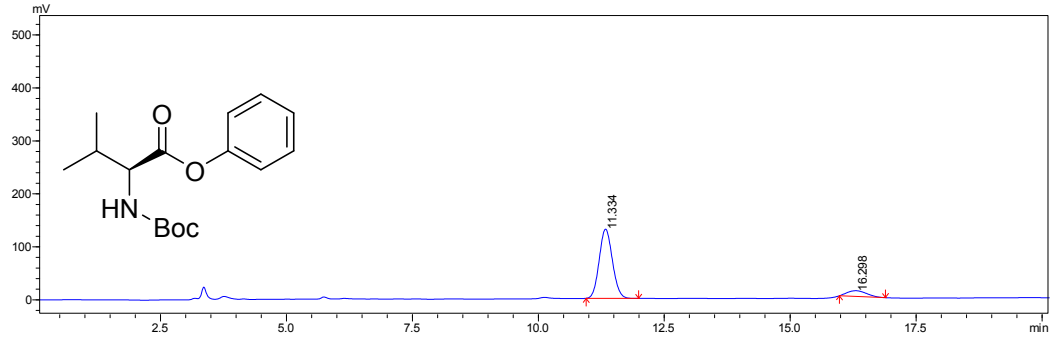
8.049	5652.303	522.201	87.694
9.243	793.175	61.336	12.306

Racemic **3dc**



RT [min]	Area	Height	Area%
11.582	4000.235	196.932	50.158
17.203	3975.108	101.087	49.842

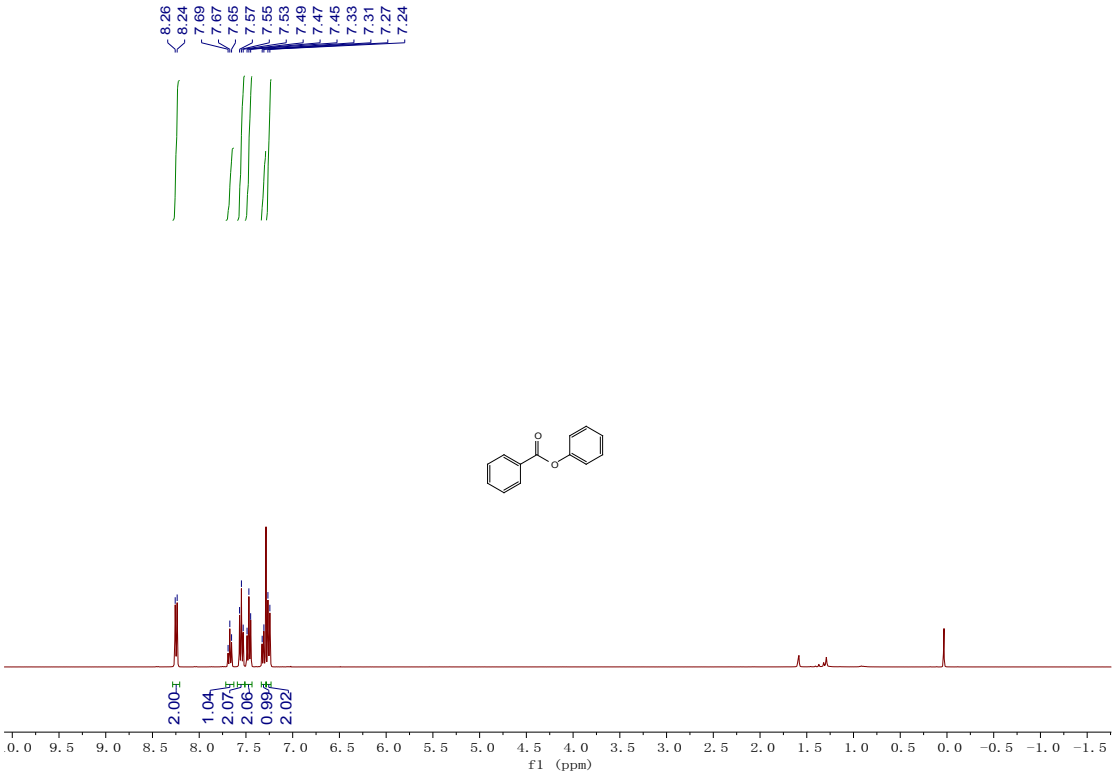
S-**3dc**

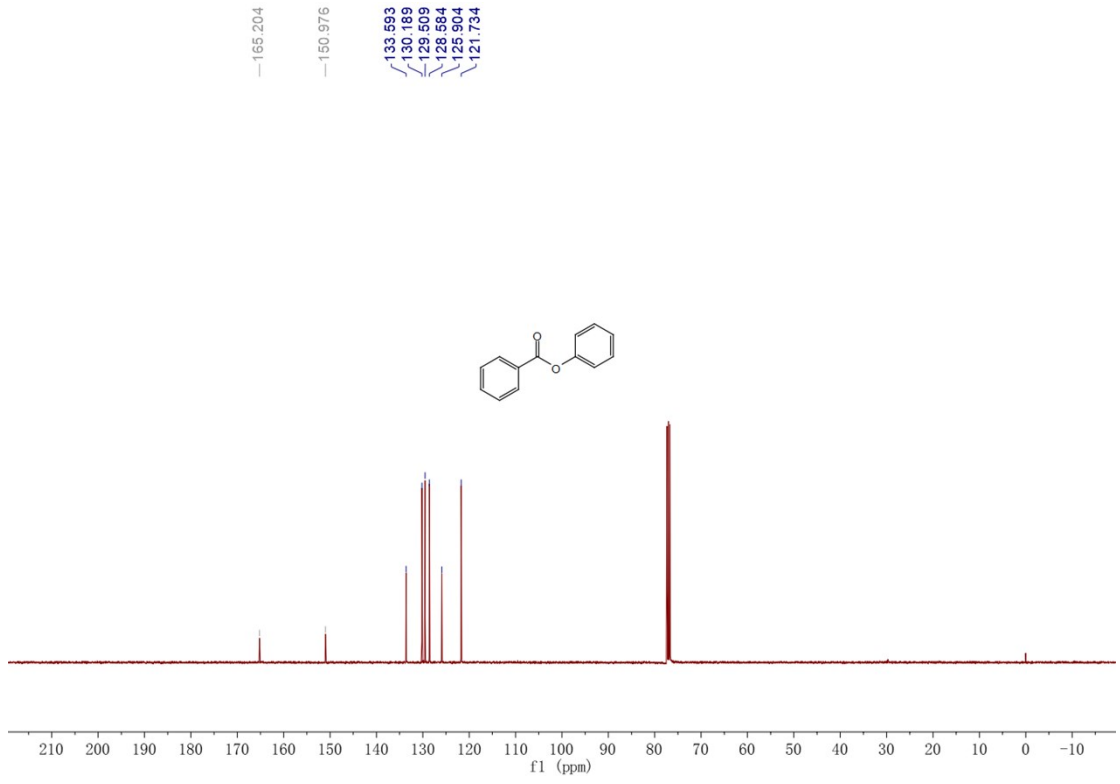


RT [min]	Area	Height	Area%
11.334	2380.201	130.727	89.173
16.298	288.991	10.809	10.827

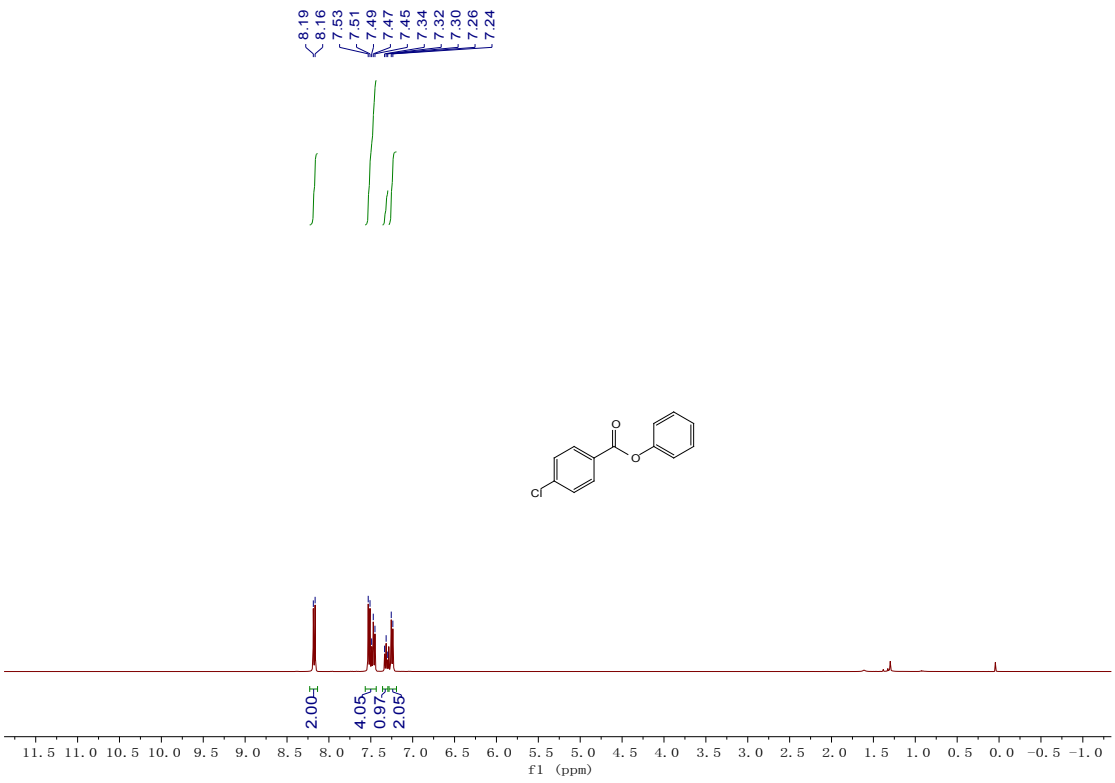
6. Copies of ^{1}H and ^{13}C NMR spectrum

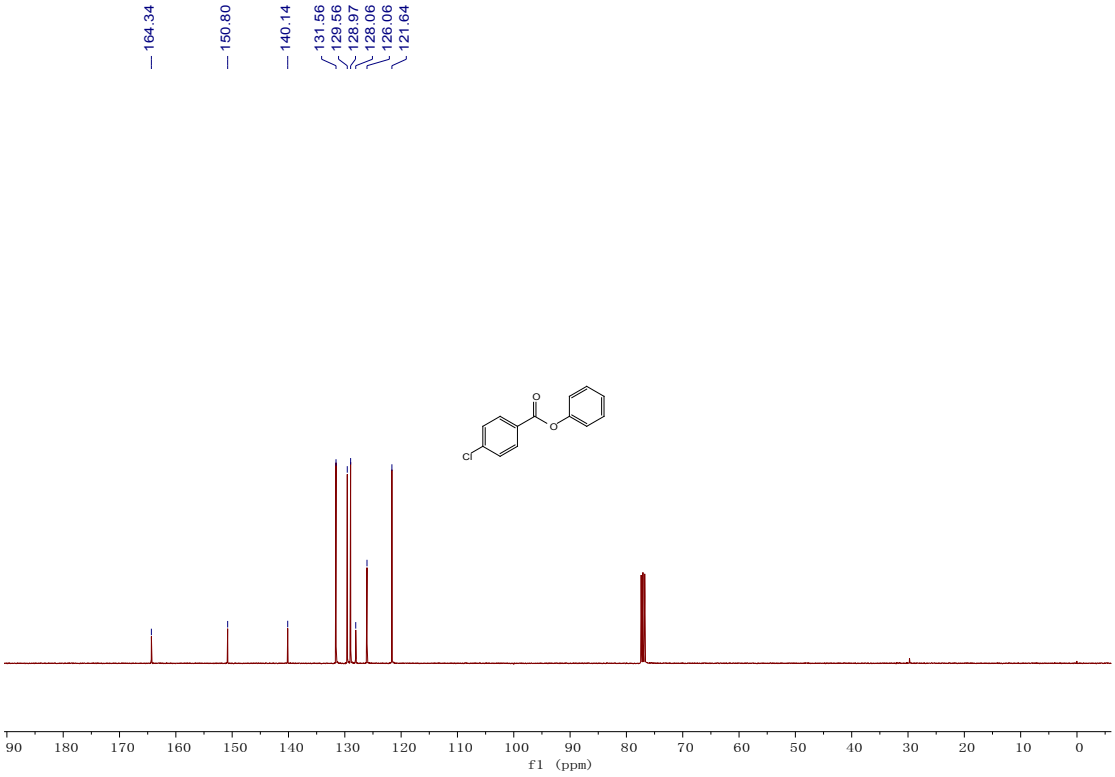
Phenyl benzoate (3aa)



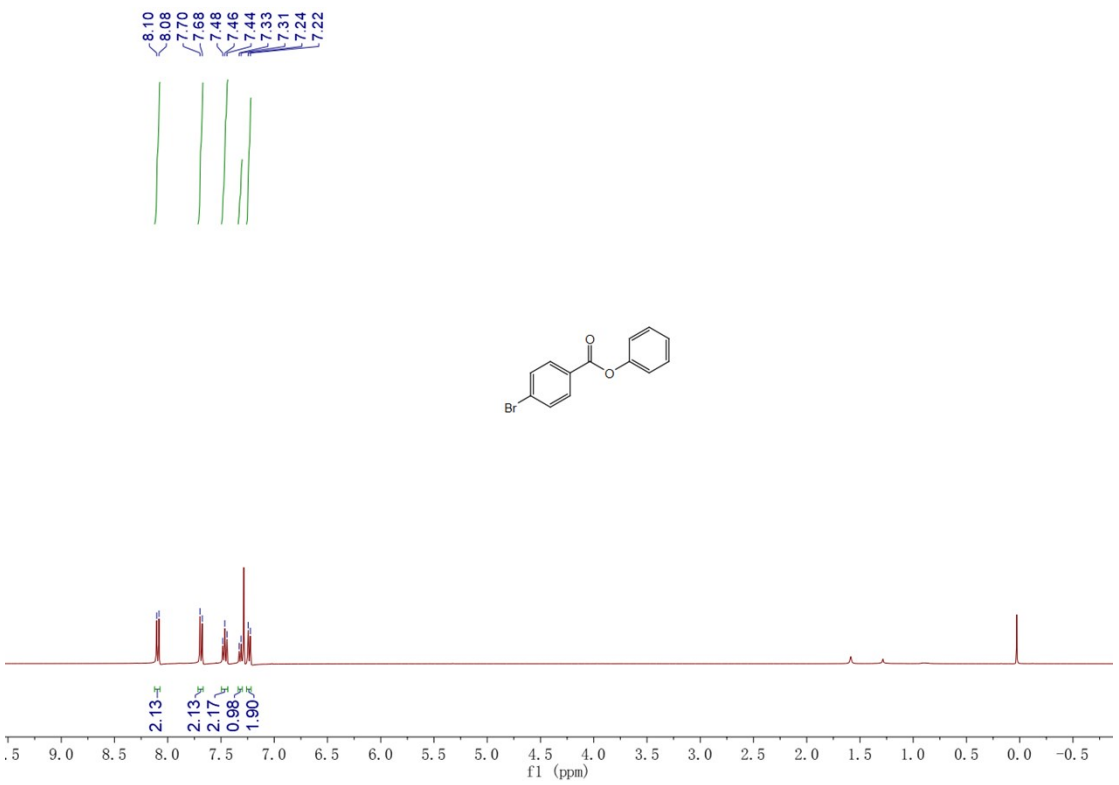


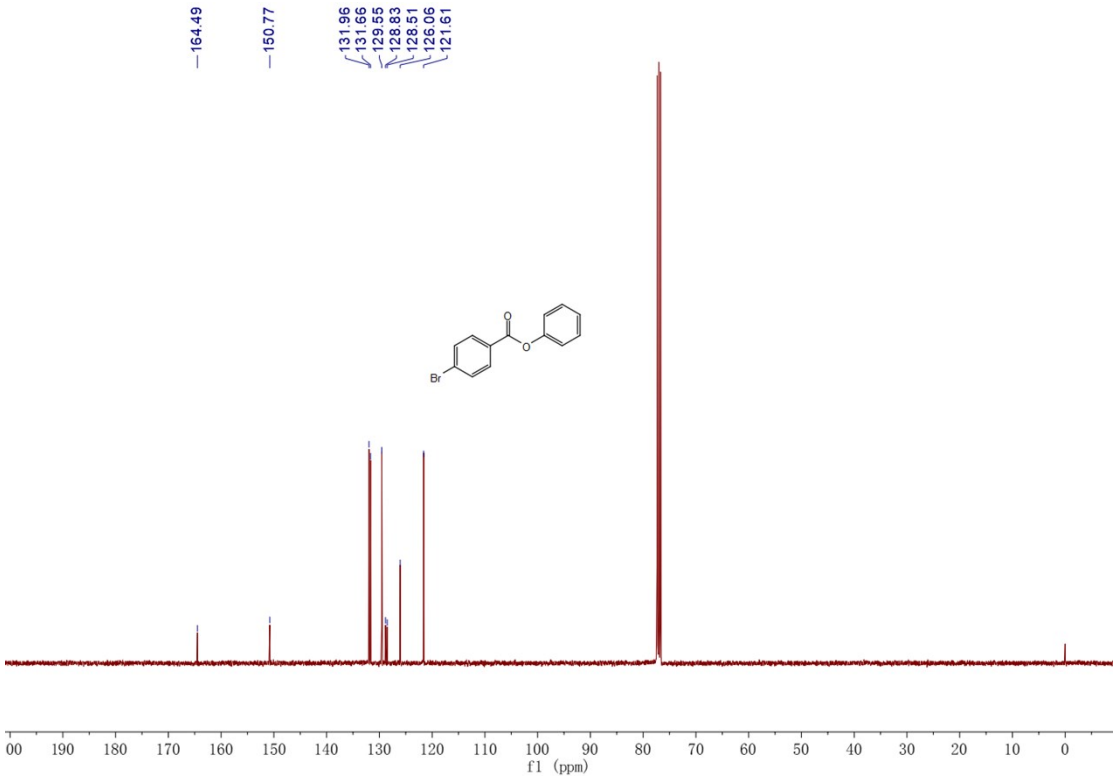
Phenyl 4-chlorobenzoate (3ab)



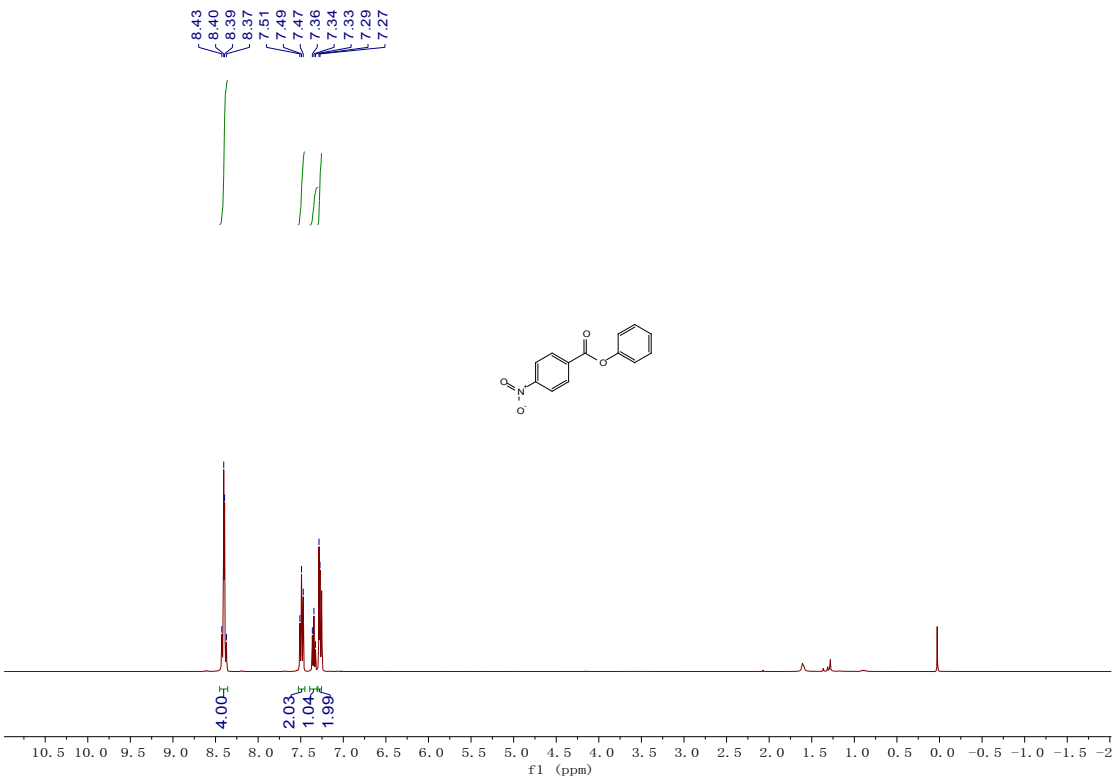


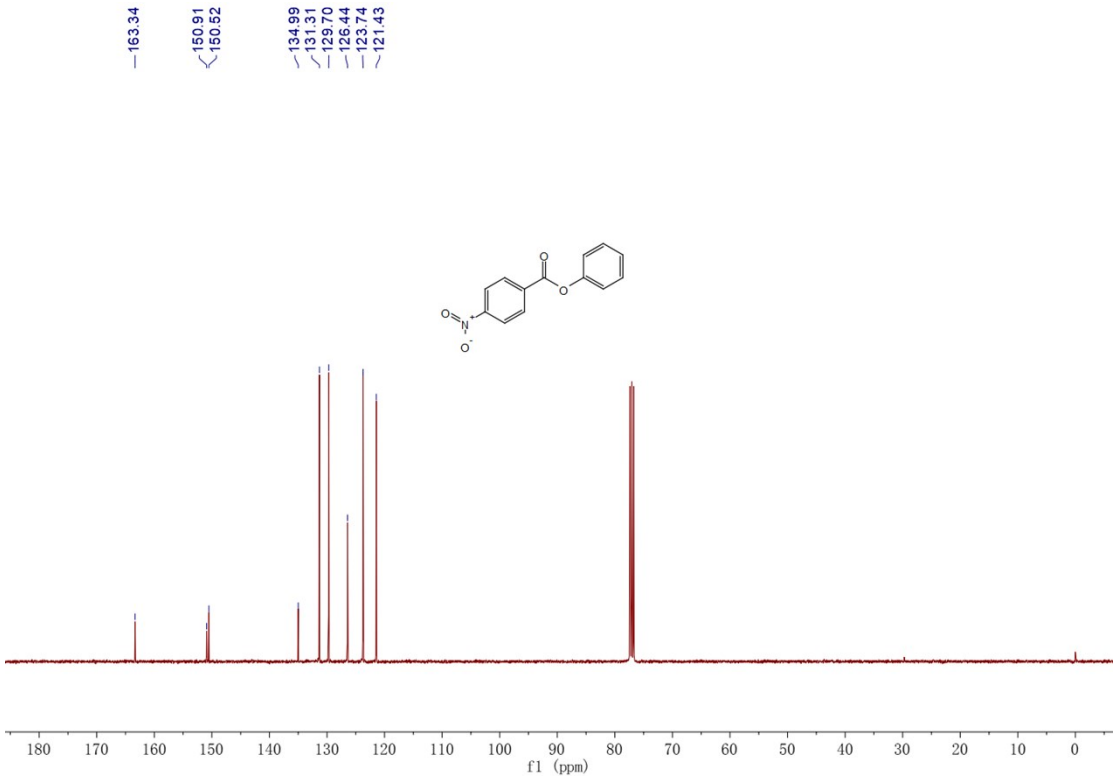
Phenyl 4-bromobenzoate (3ac)



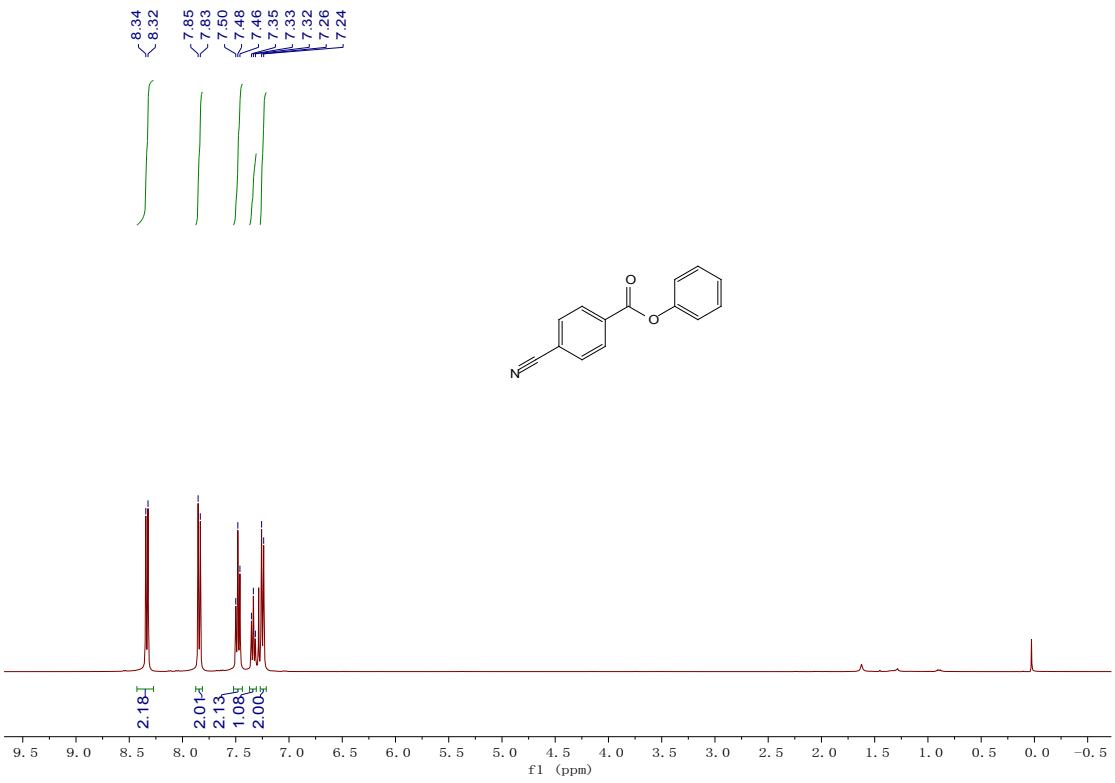


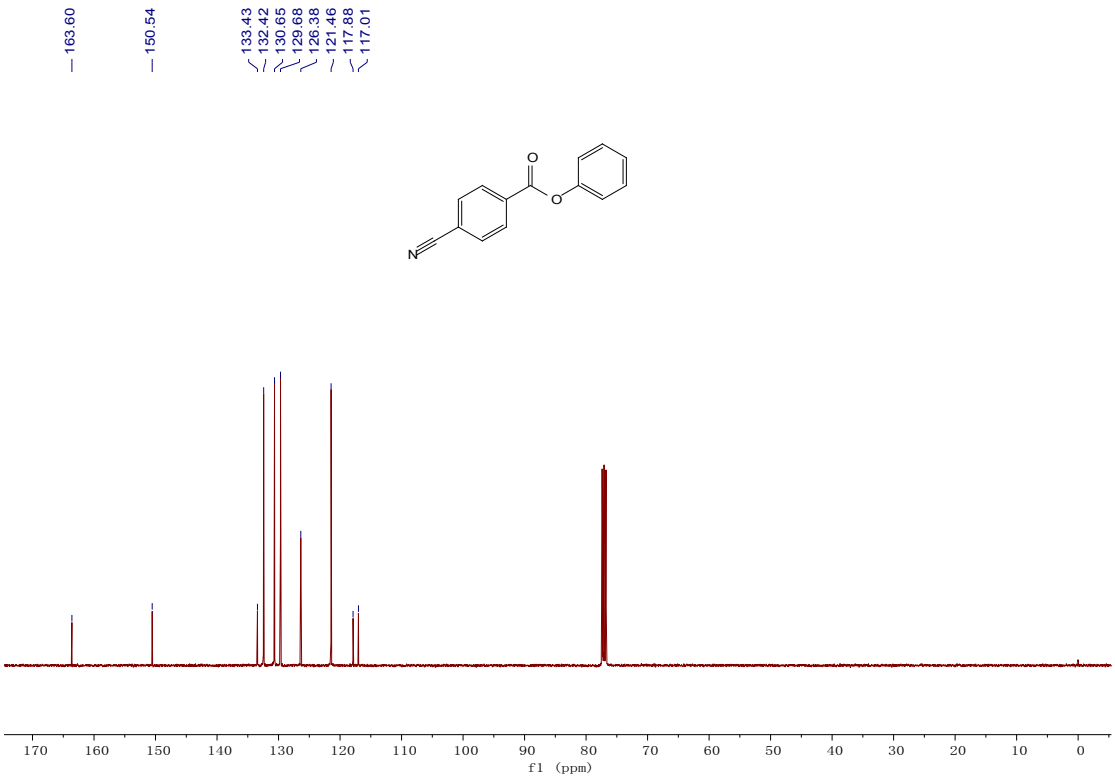
Phenyl 4-nitrobenzoate (3ad)



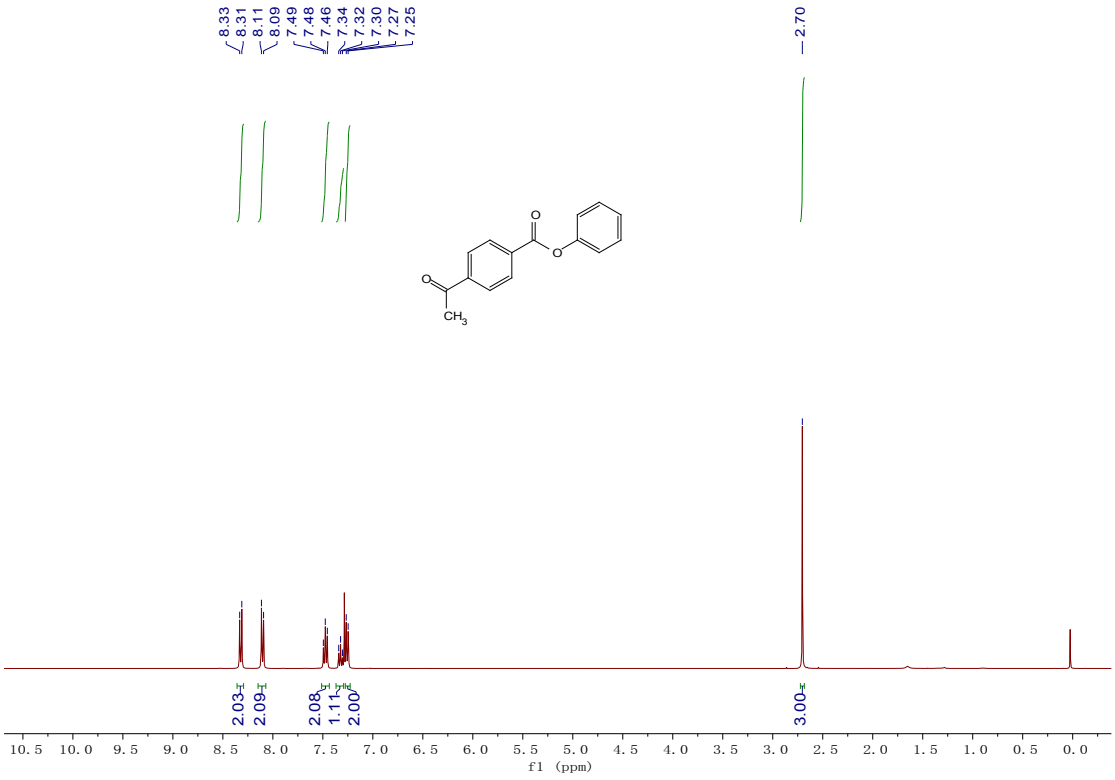


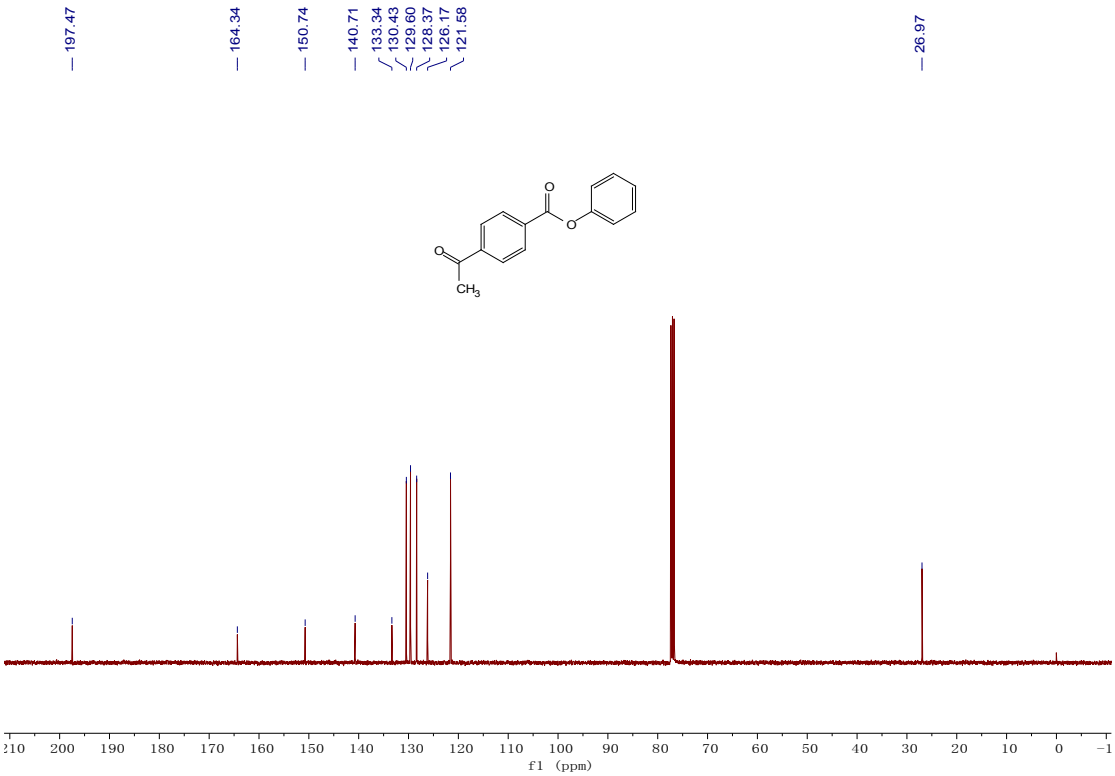
Phenyl 4-cyanobenzoate (3ae)



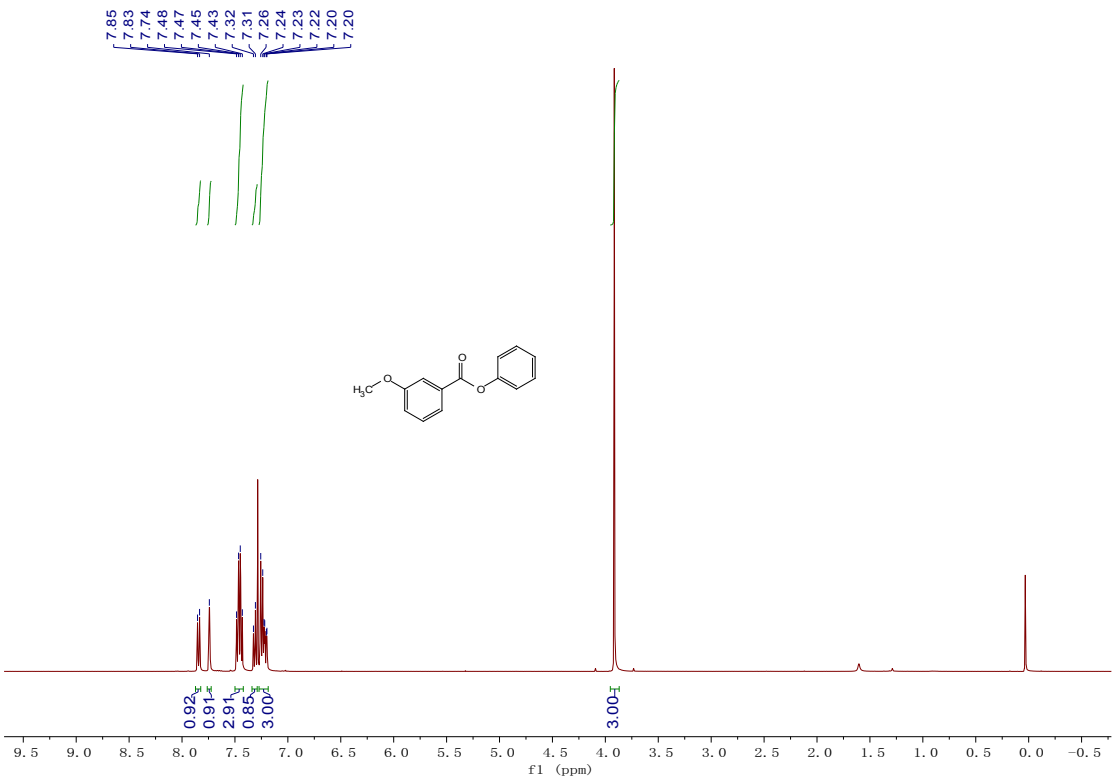


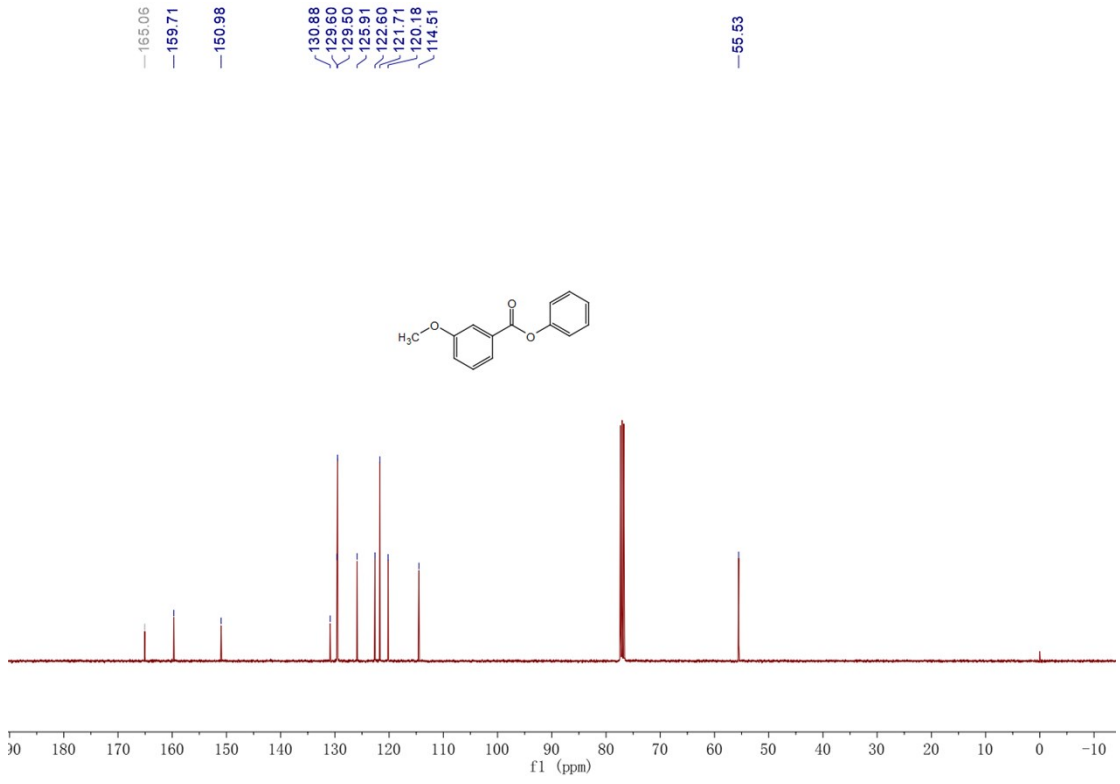
Phenyl 4-acetylbenzoate (3af)



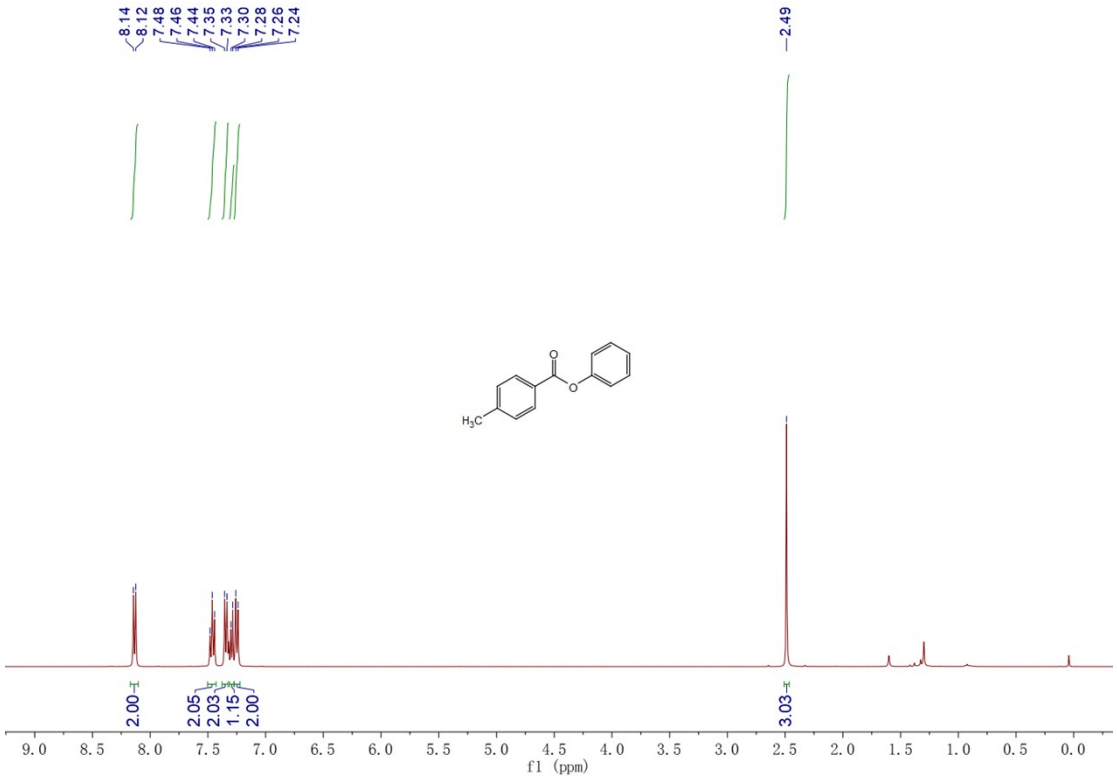


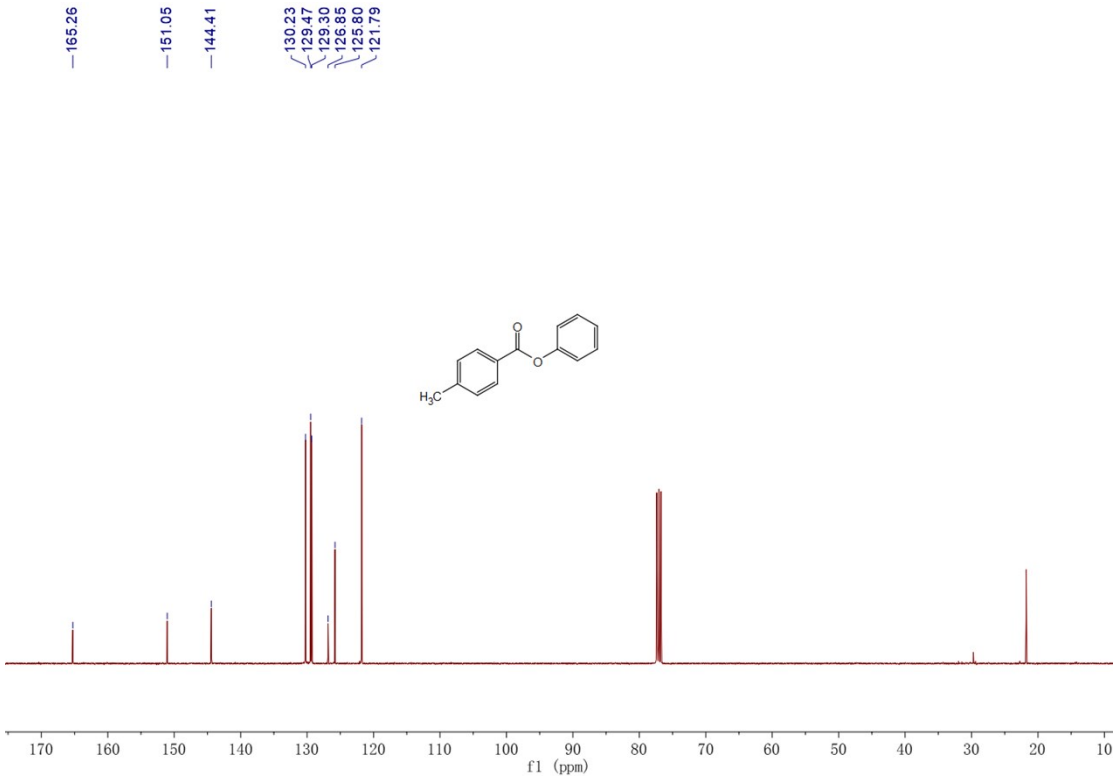
Phenyl 3-methoxybenzoate (3af)



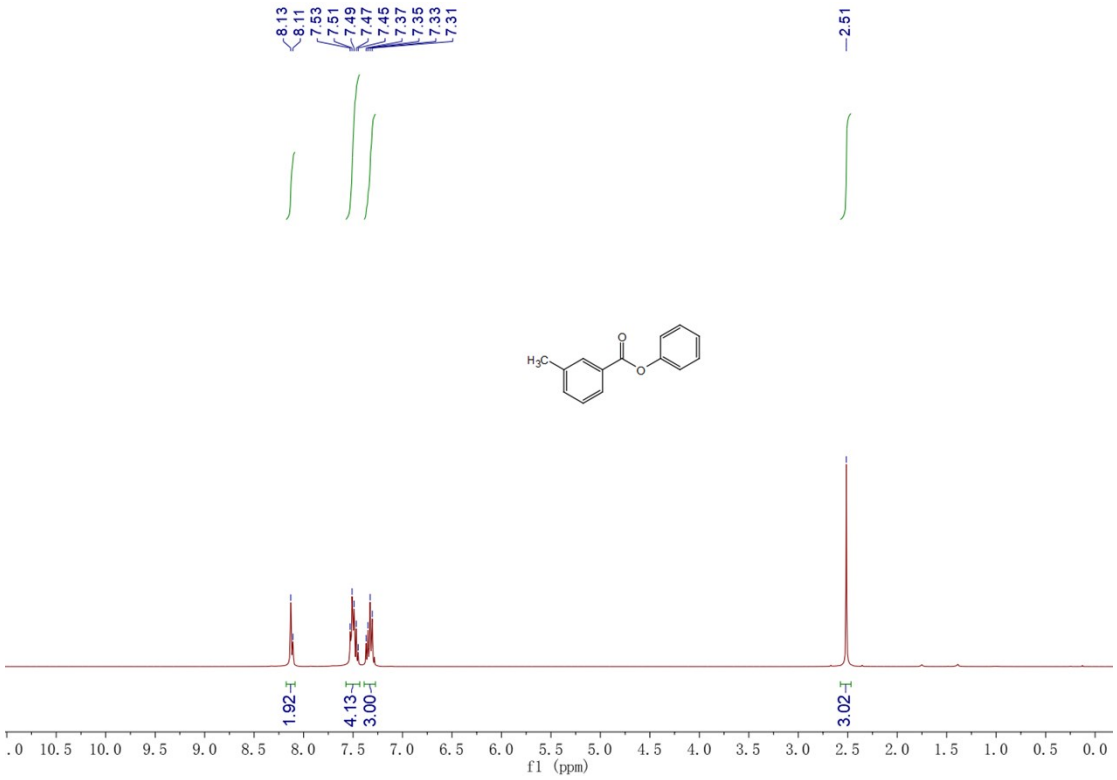


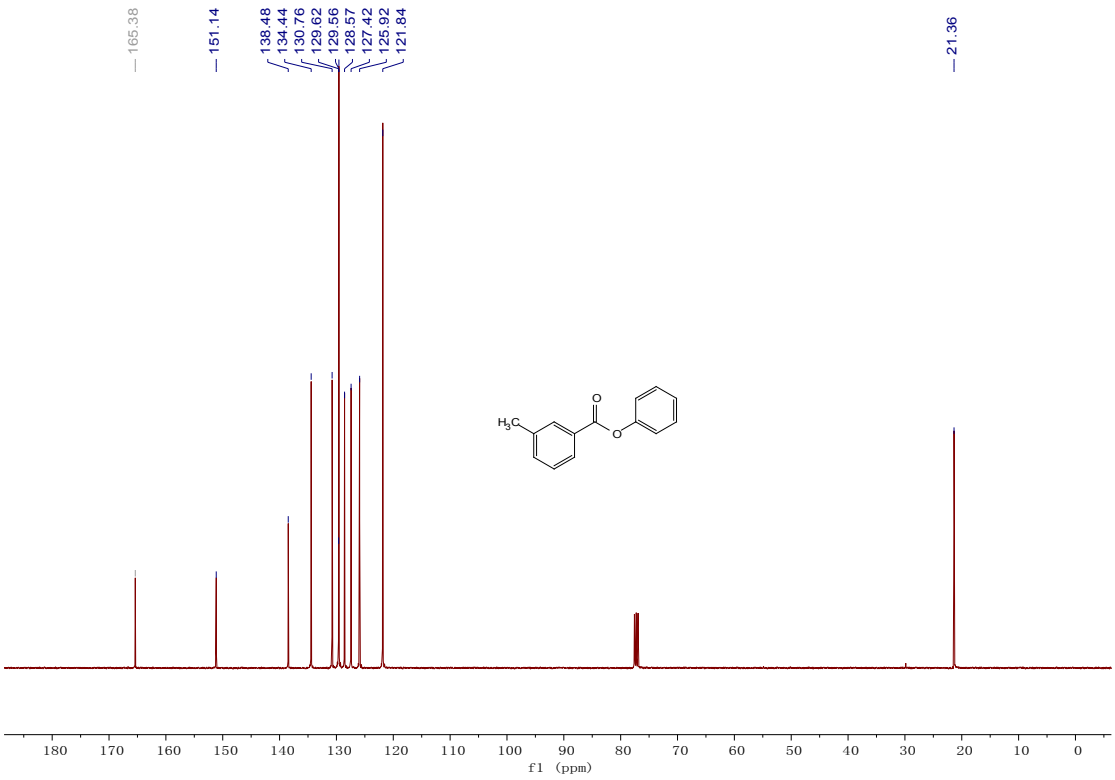
Phenyl 4-methylbenzoate (3ag)



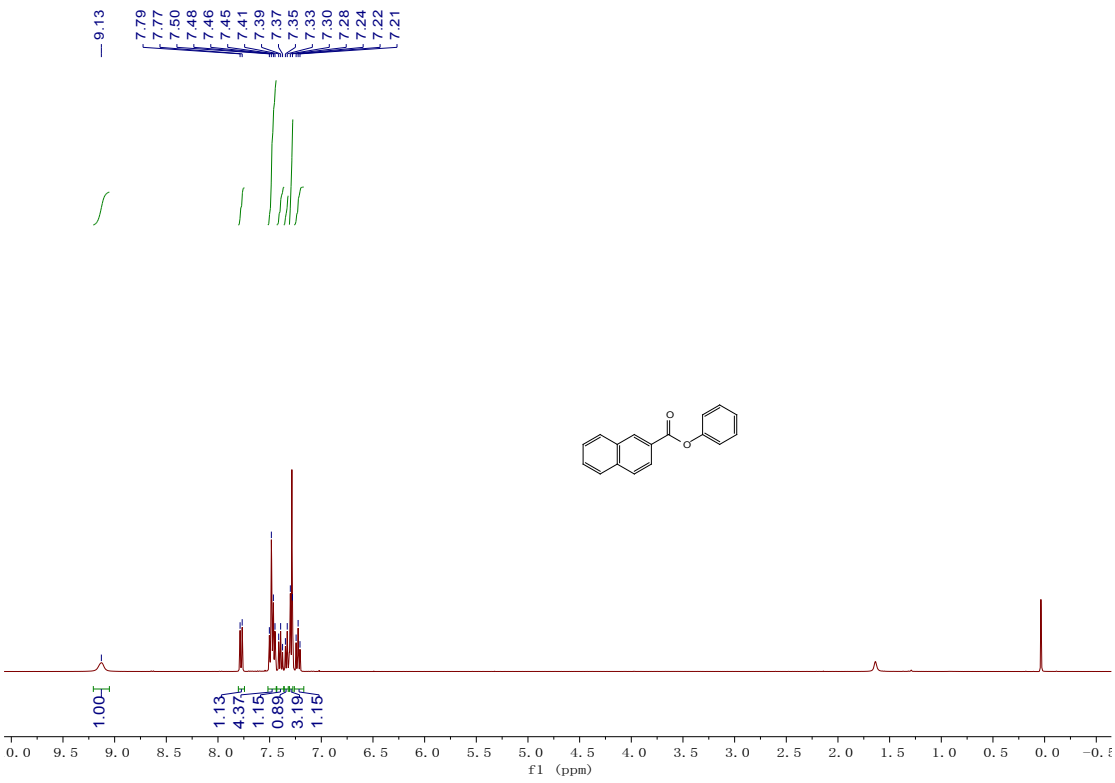


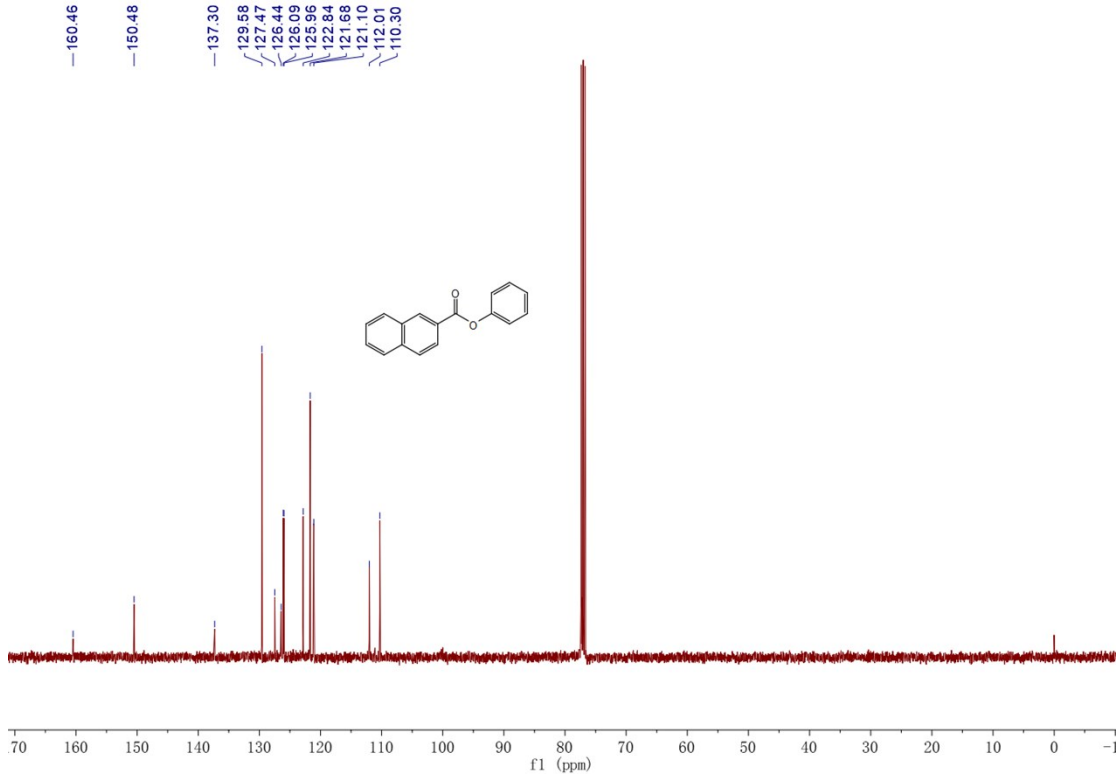
Phenyl 3-methylbenzoate (3ah)



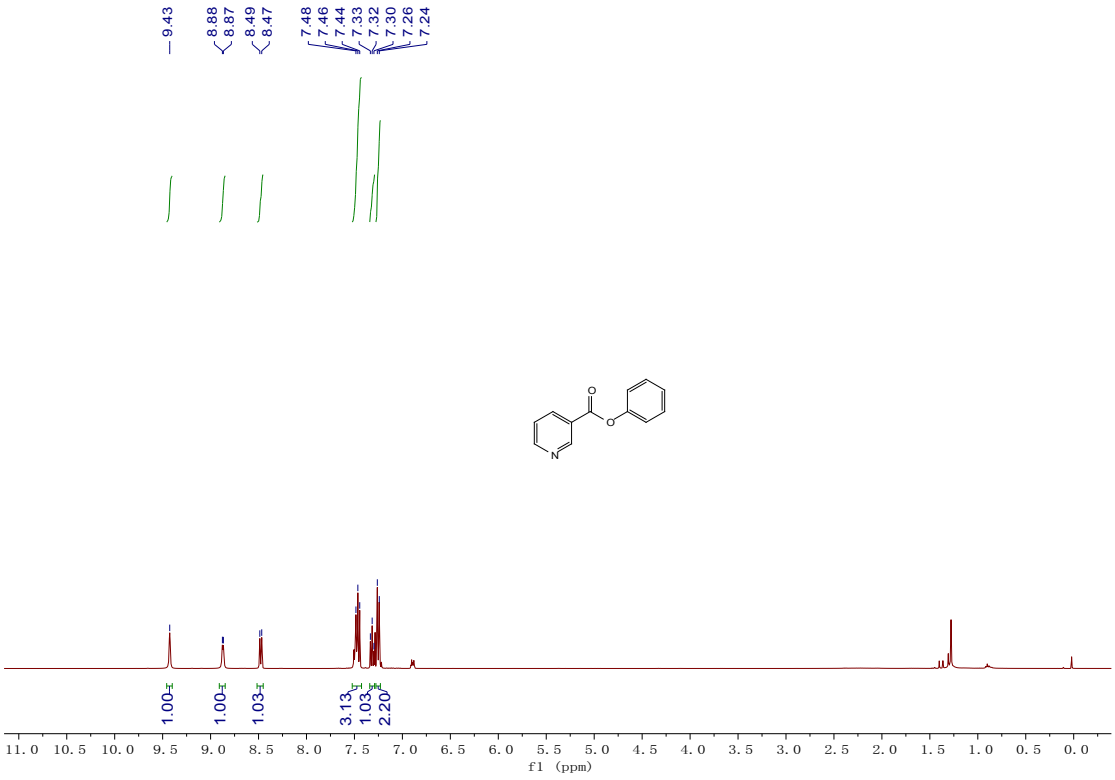


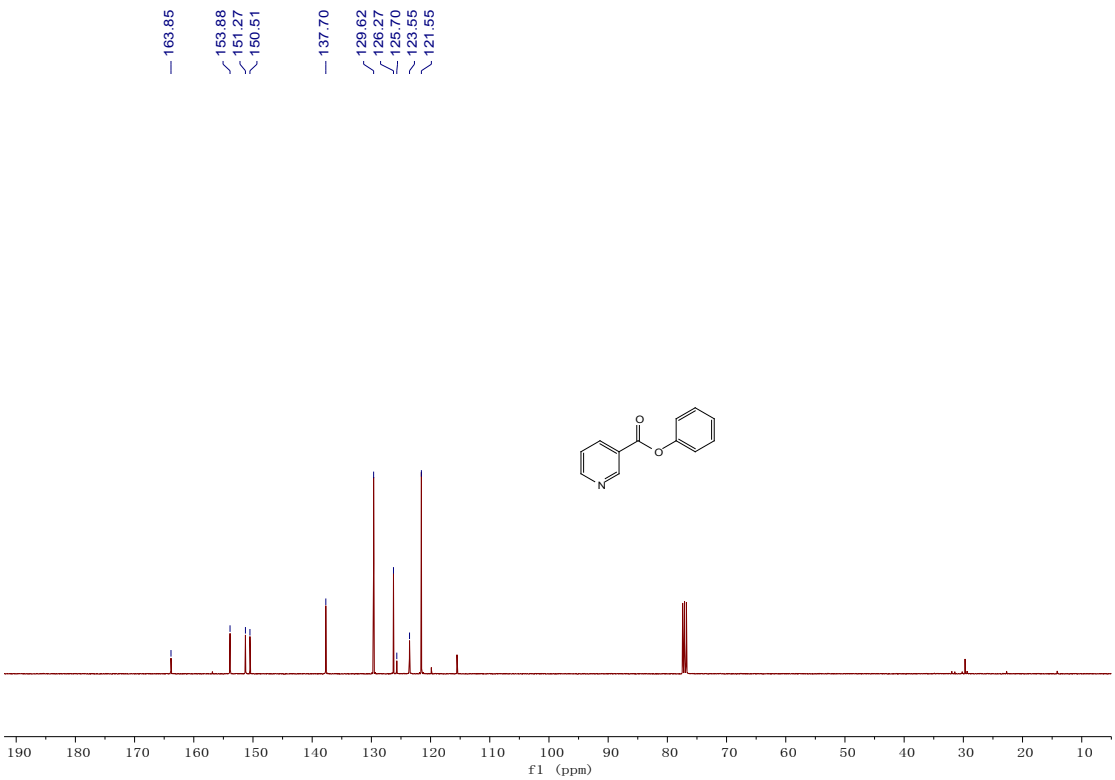
Phenyl 2-naphthoate (3ai)



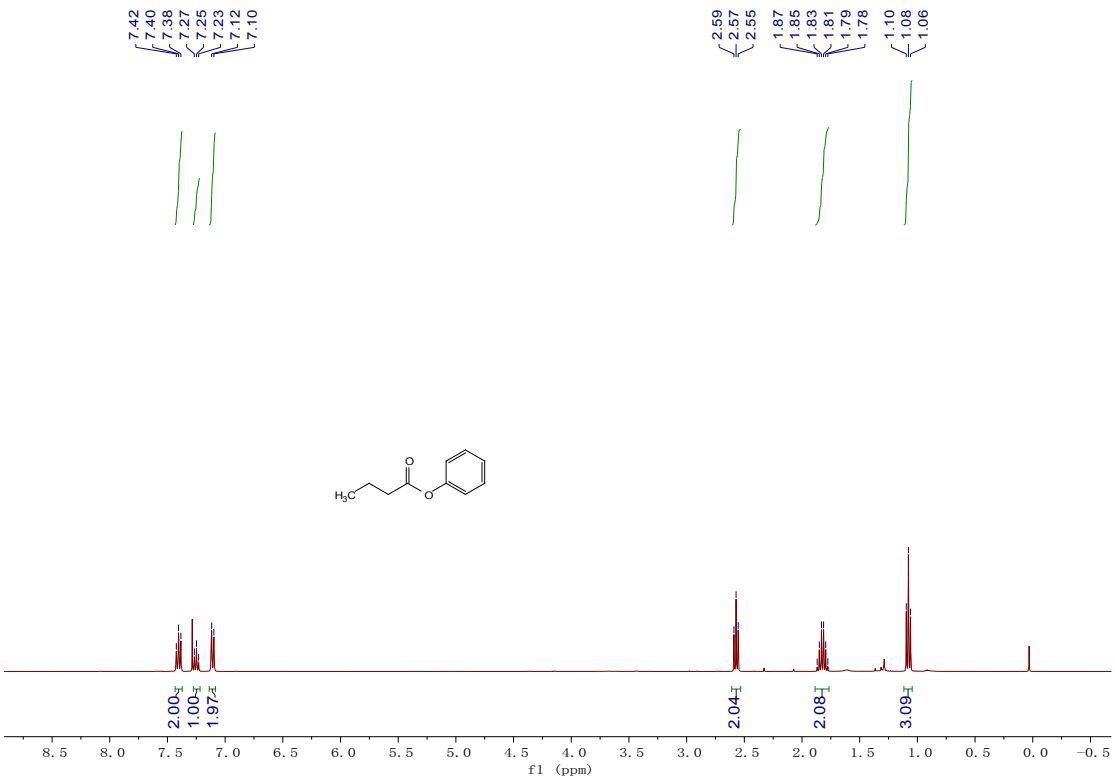


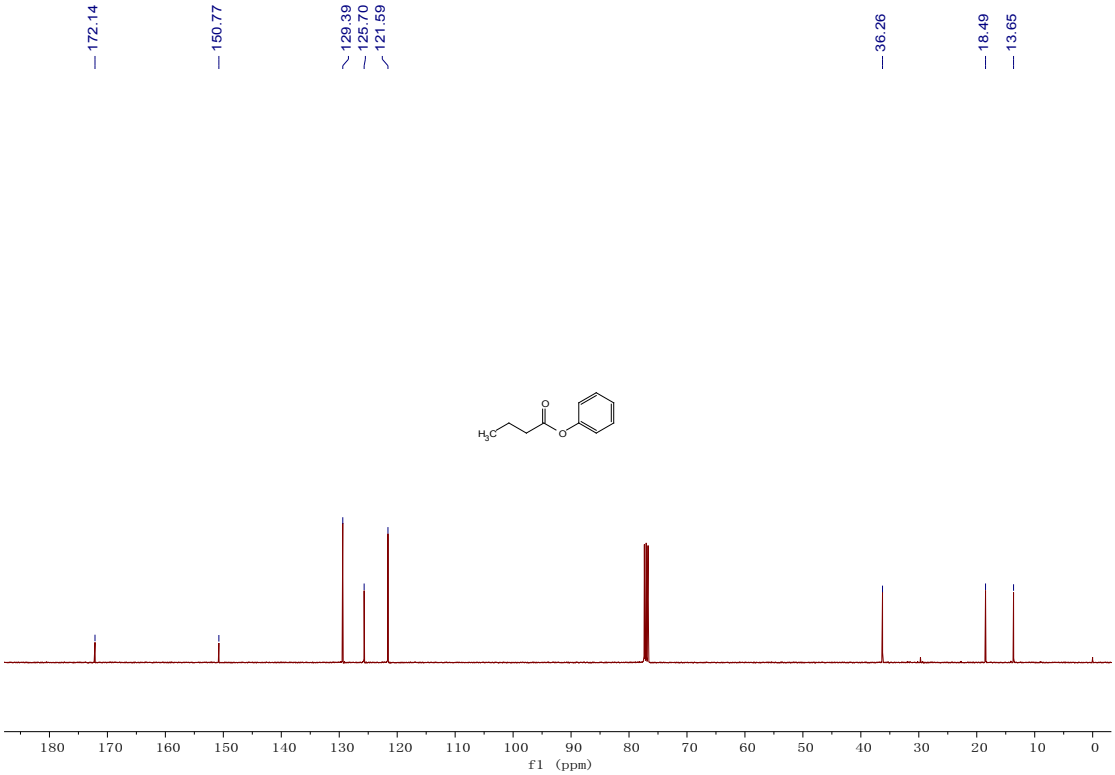
Phenyl nicotinate (3aj)



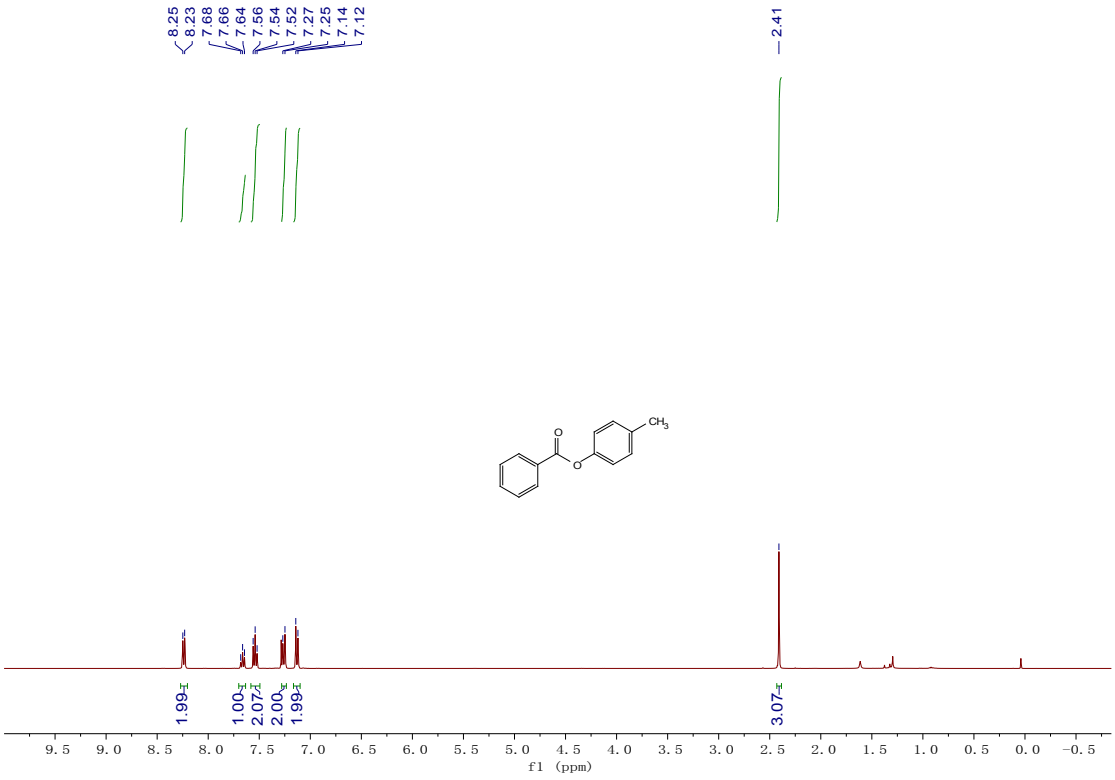


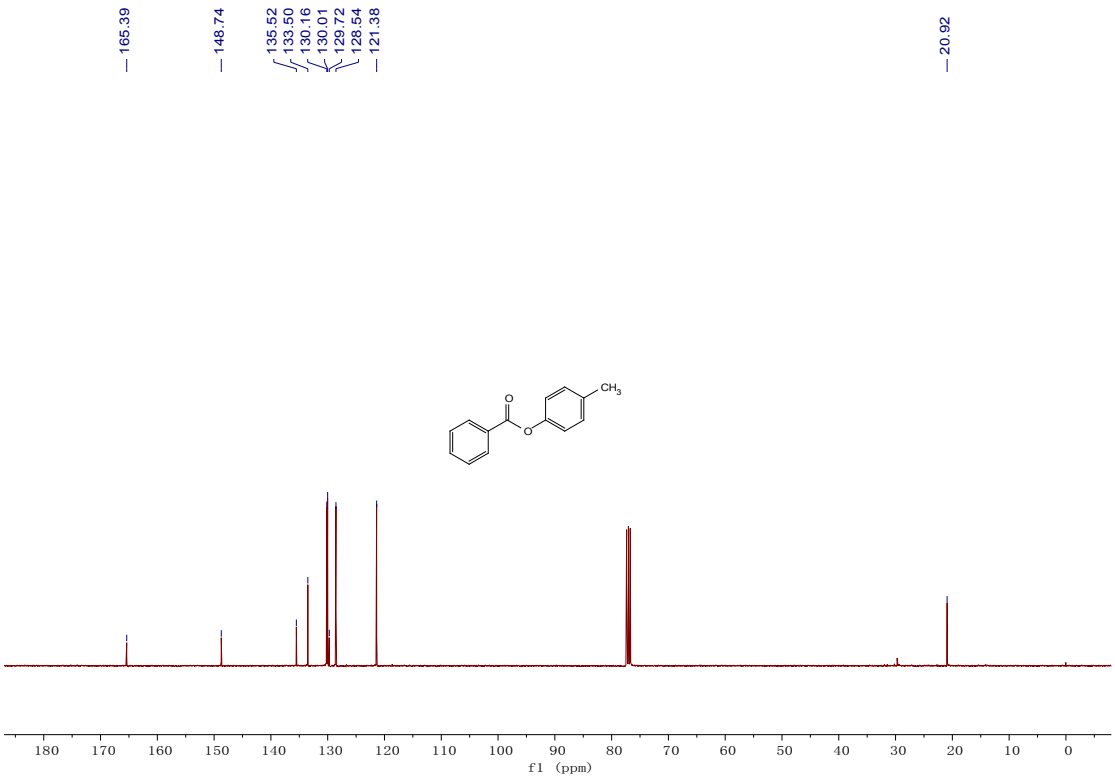
Phenyl butyrate (3al)



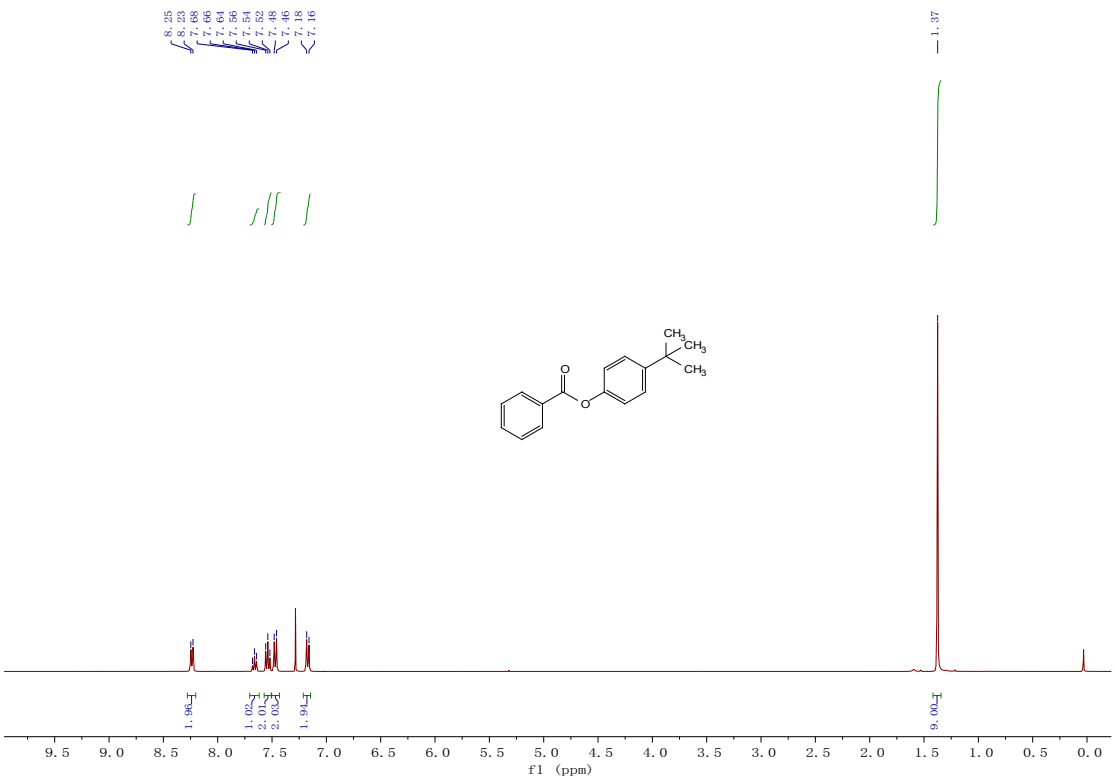


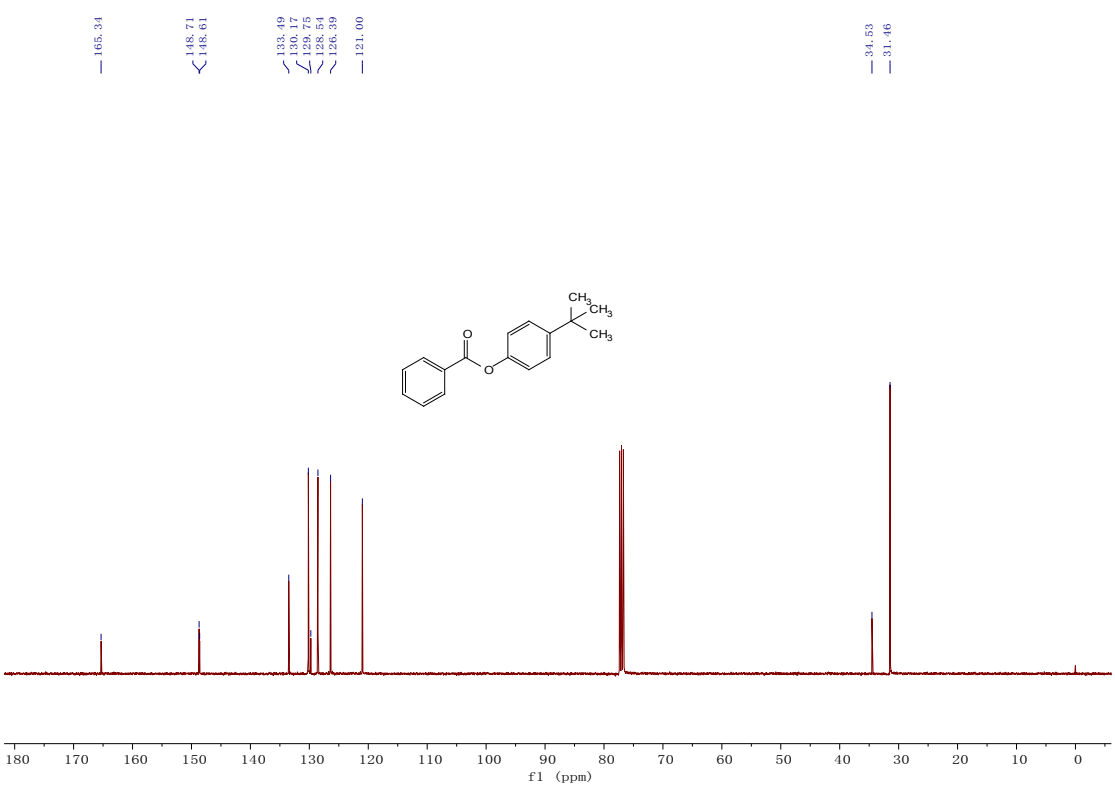
p-tolyl benzoate (3ba)



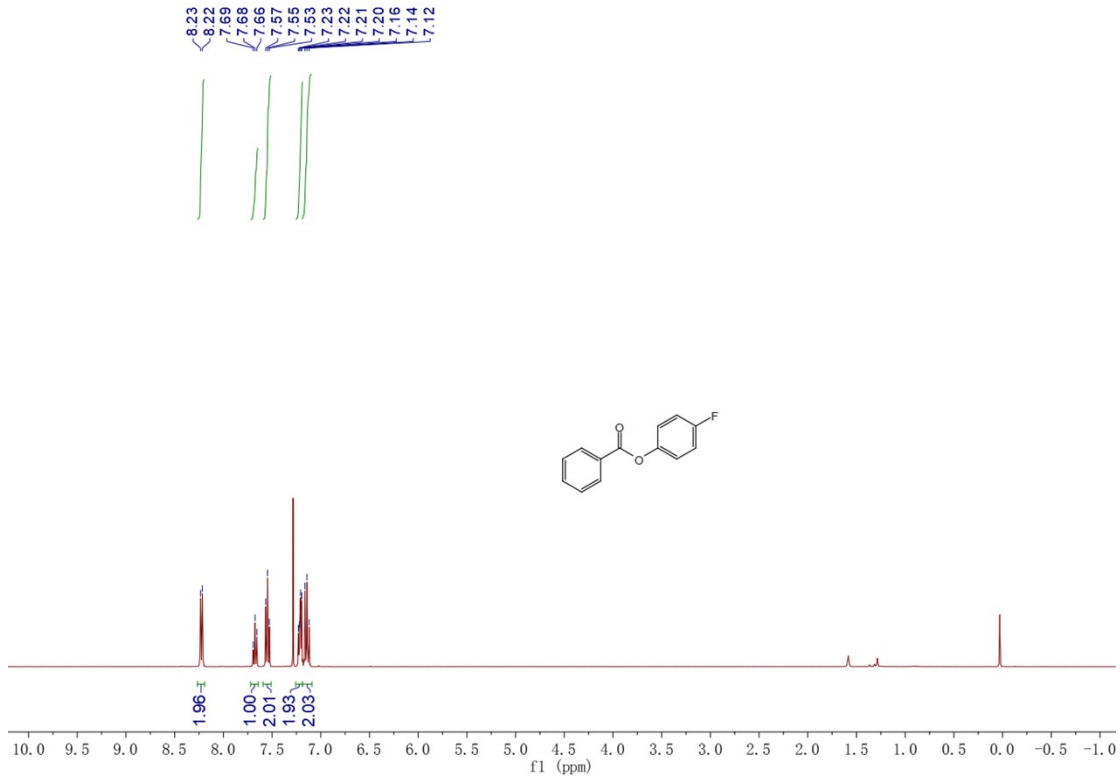


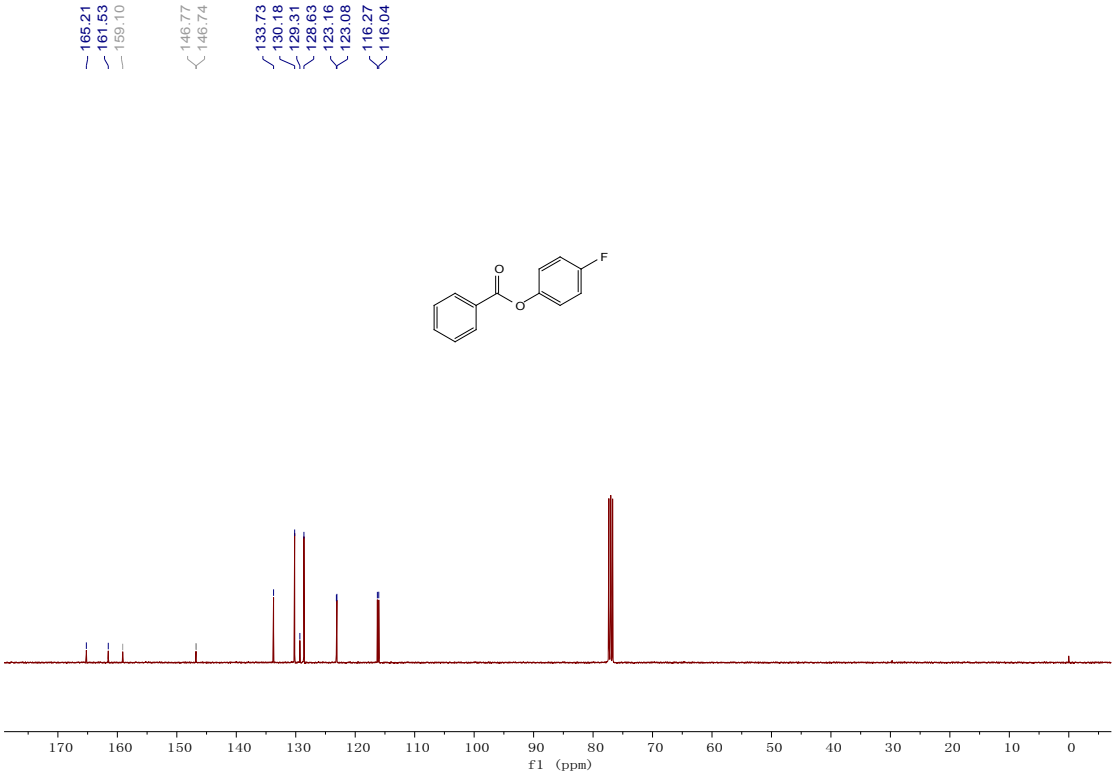
4-(tert-butyl)phenyl benzoate (3bb)



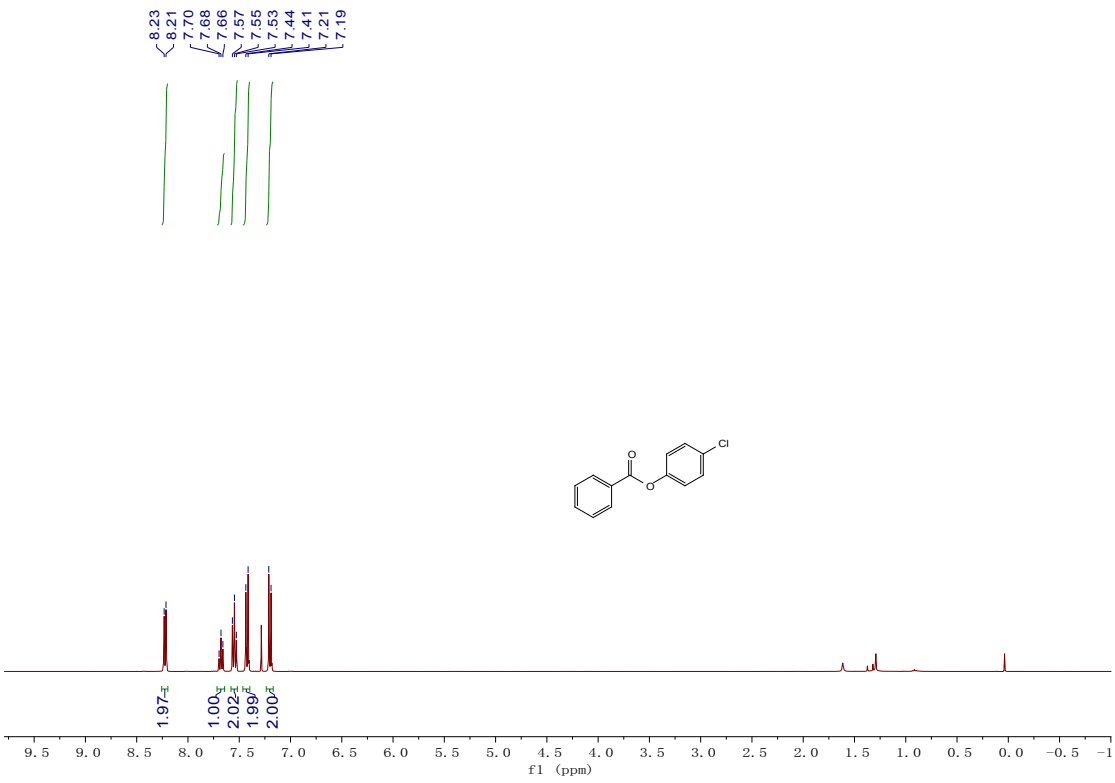


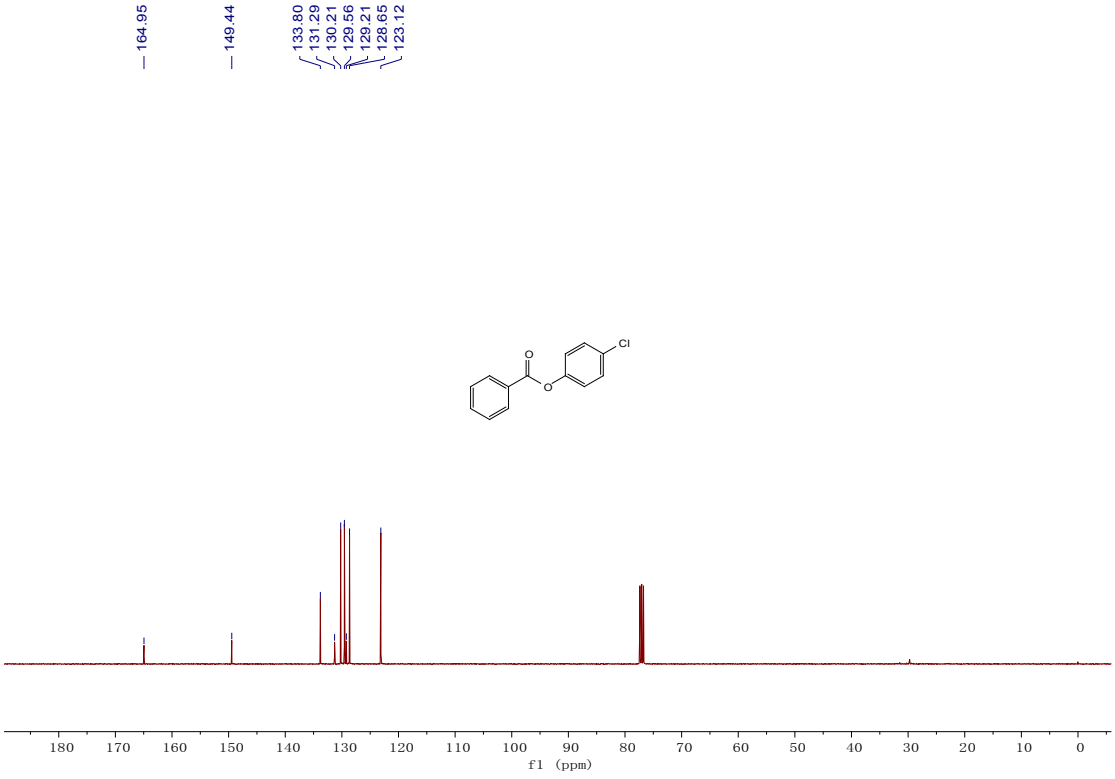
4-fluorophenyl benzoate (3bc)



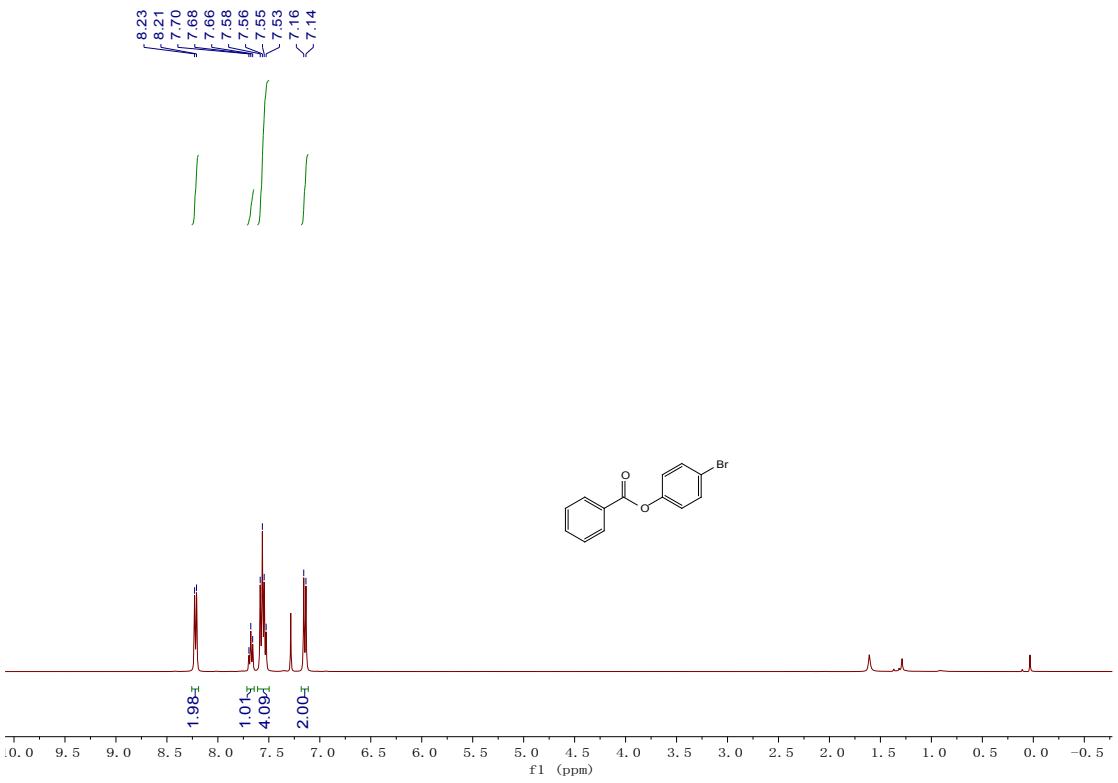


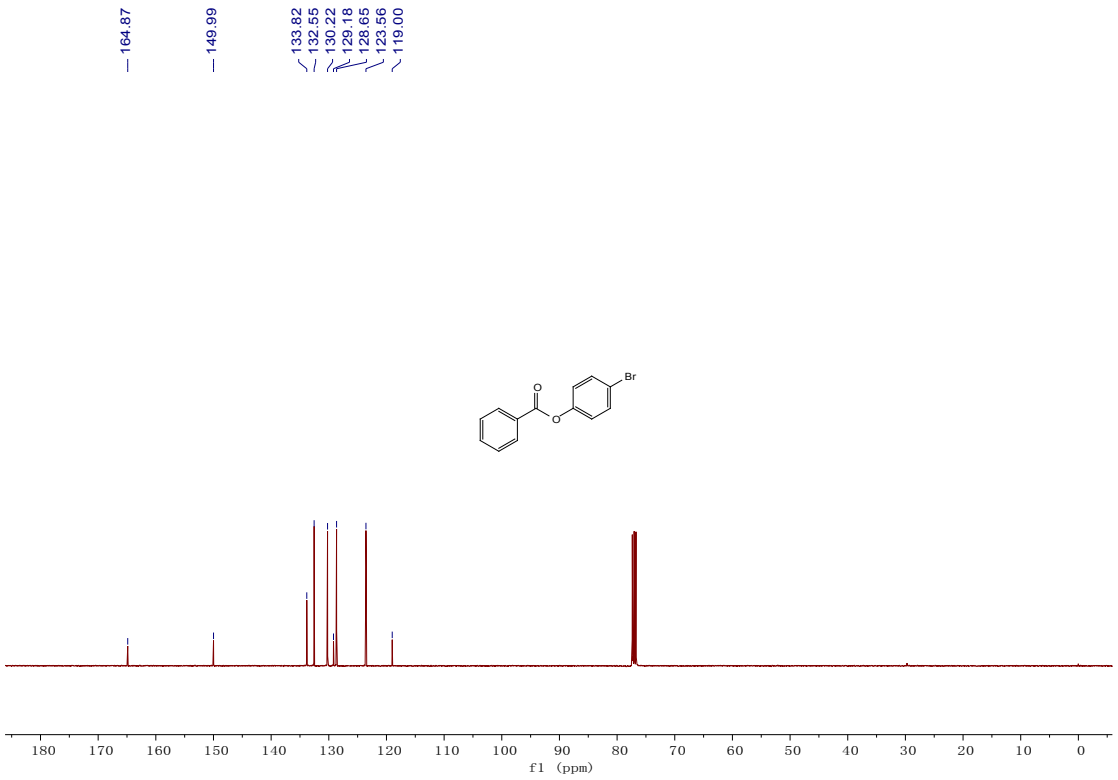
4-chlorophenyl benzoate (3bd)



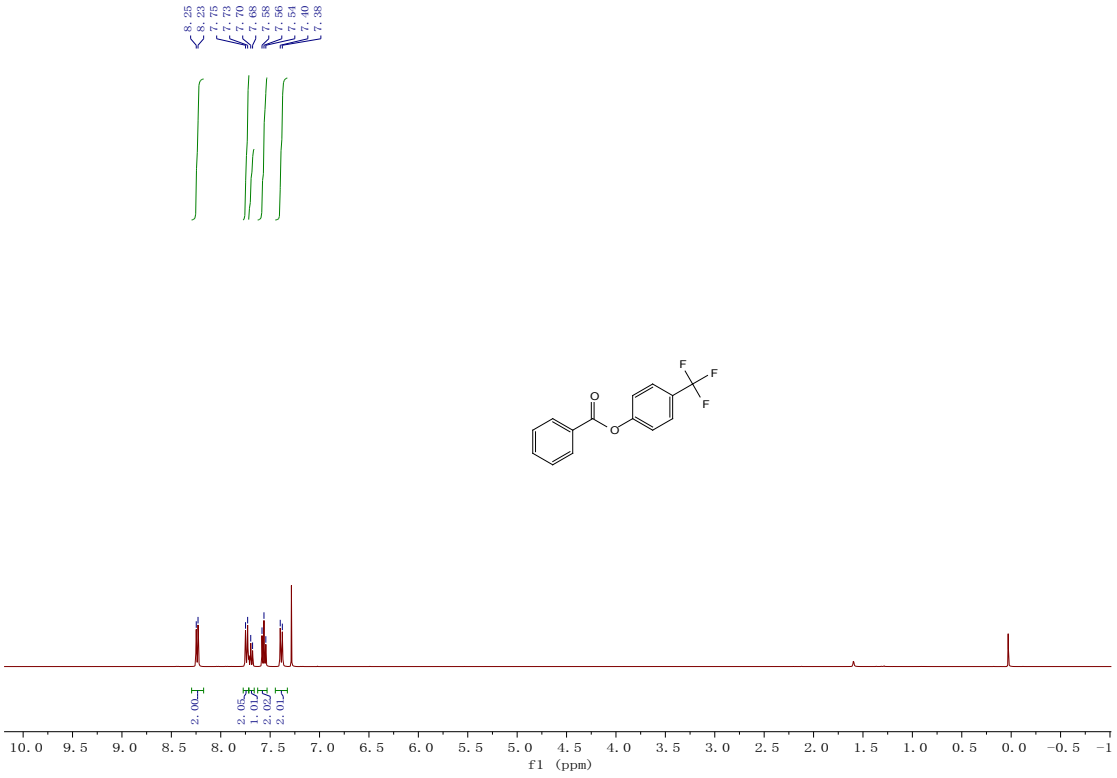


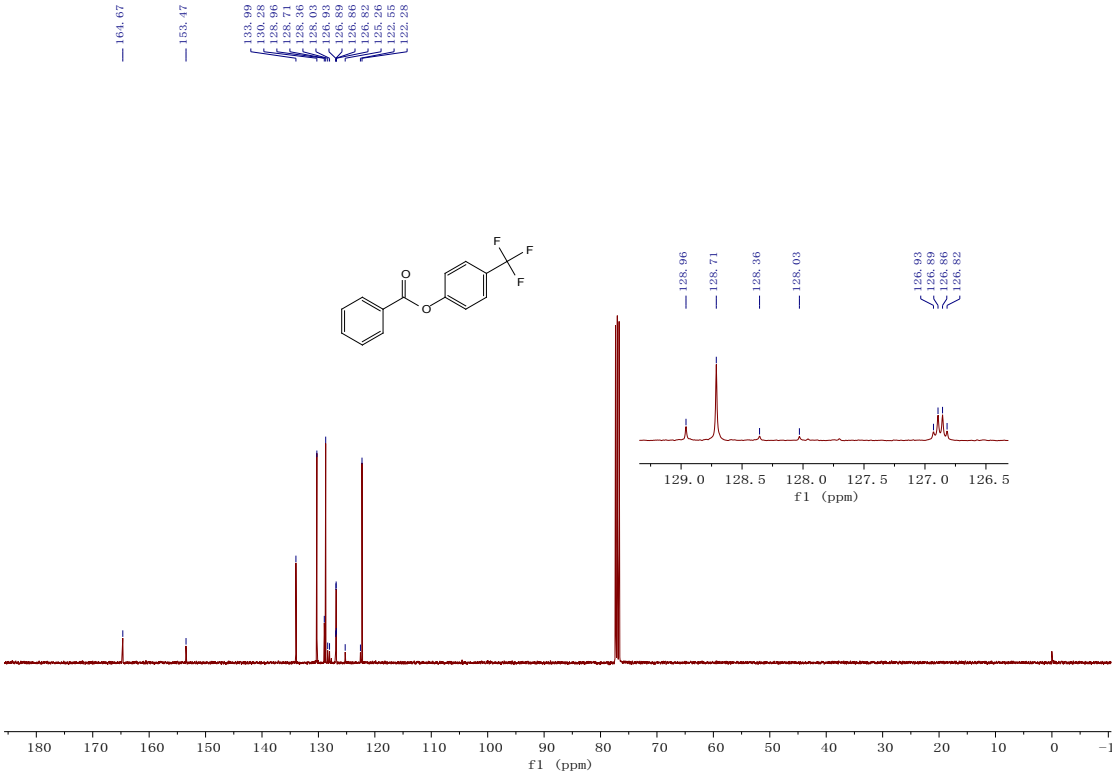
4-bromophenyl benzoate (3be)



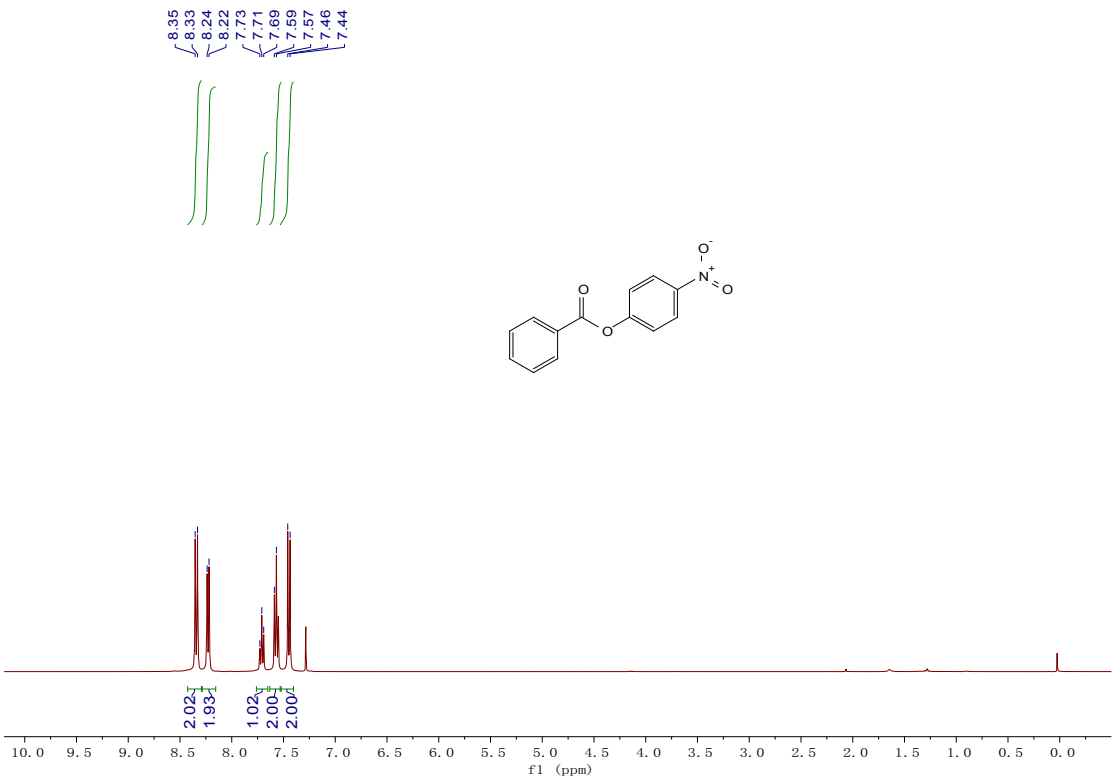


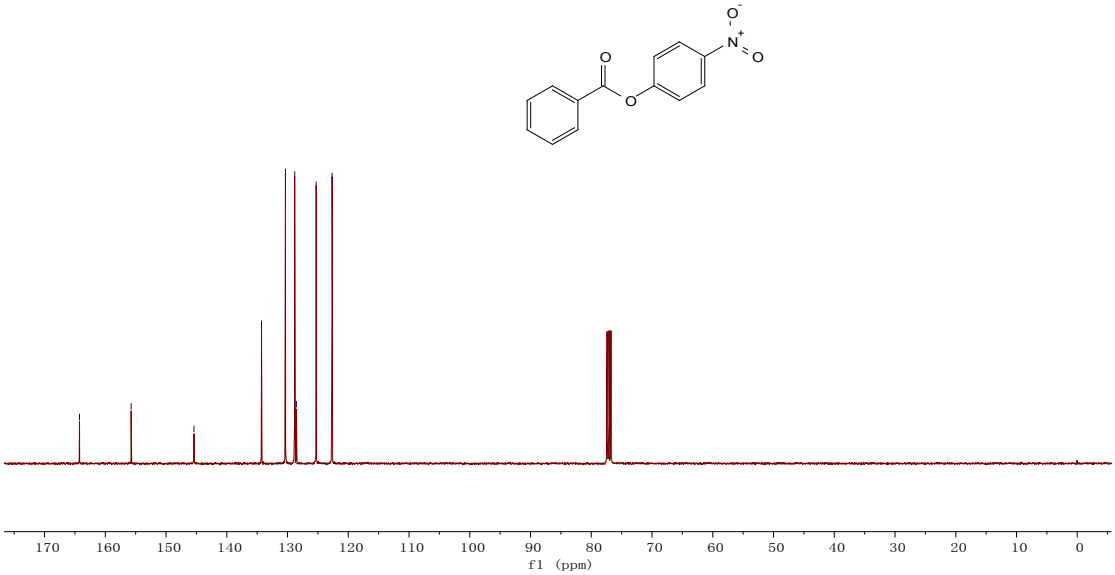
4-(trifluoromethyl)phenyl benzoate (3bf)



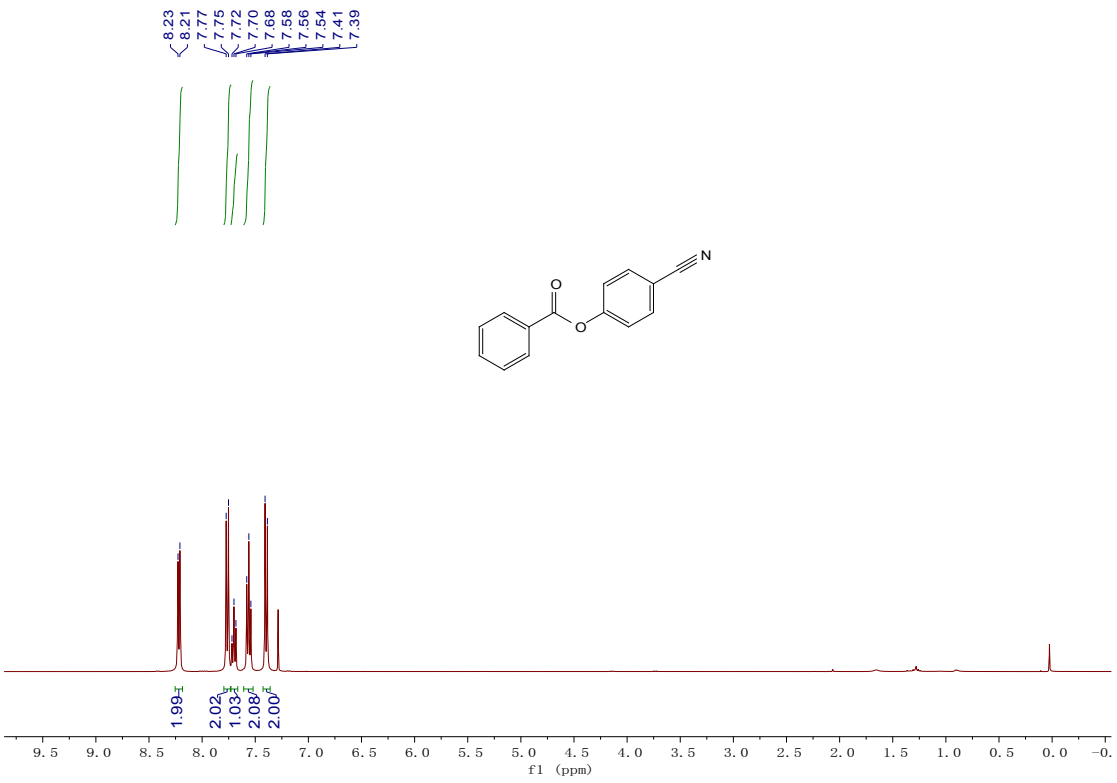


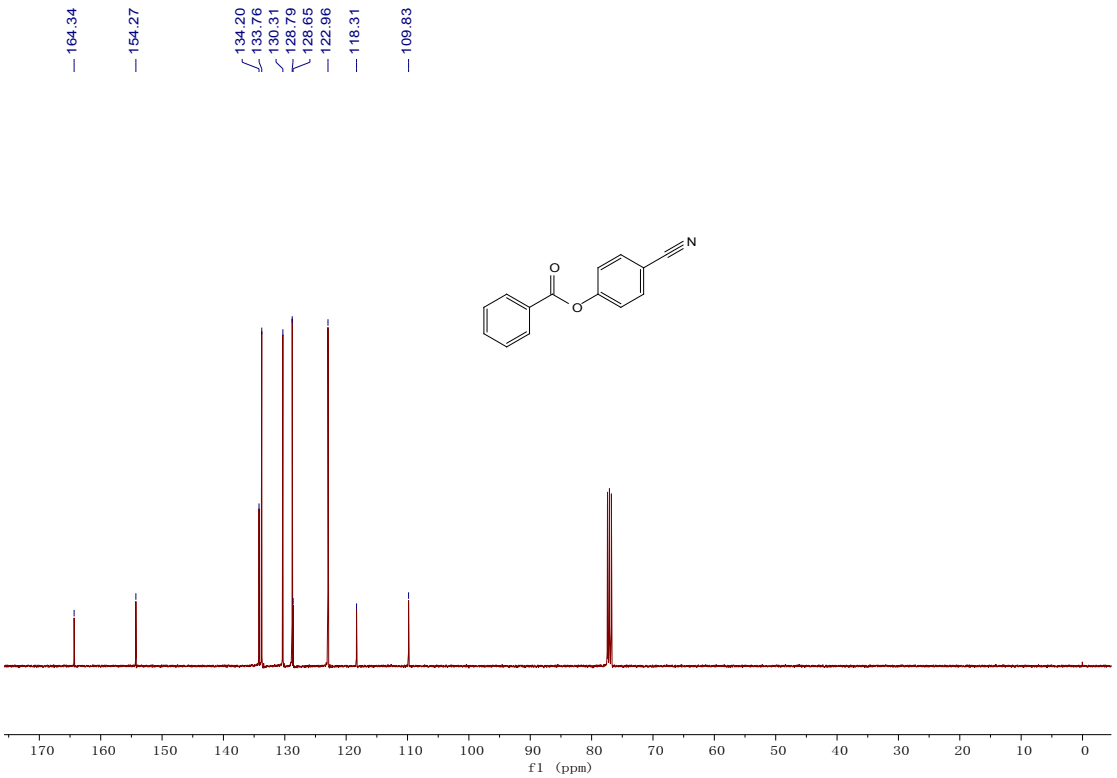
4-nitrophenyl benzoate (3bg)



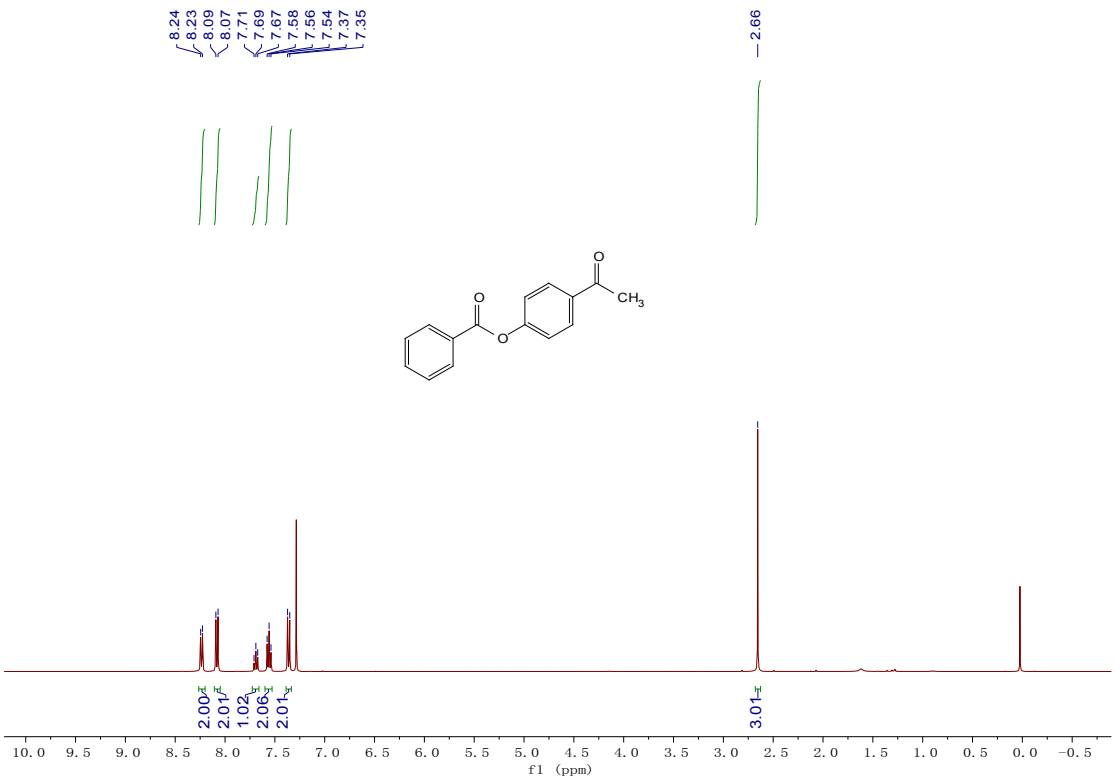


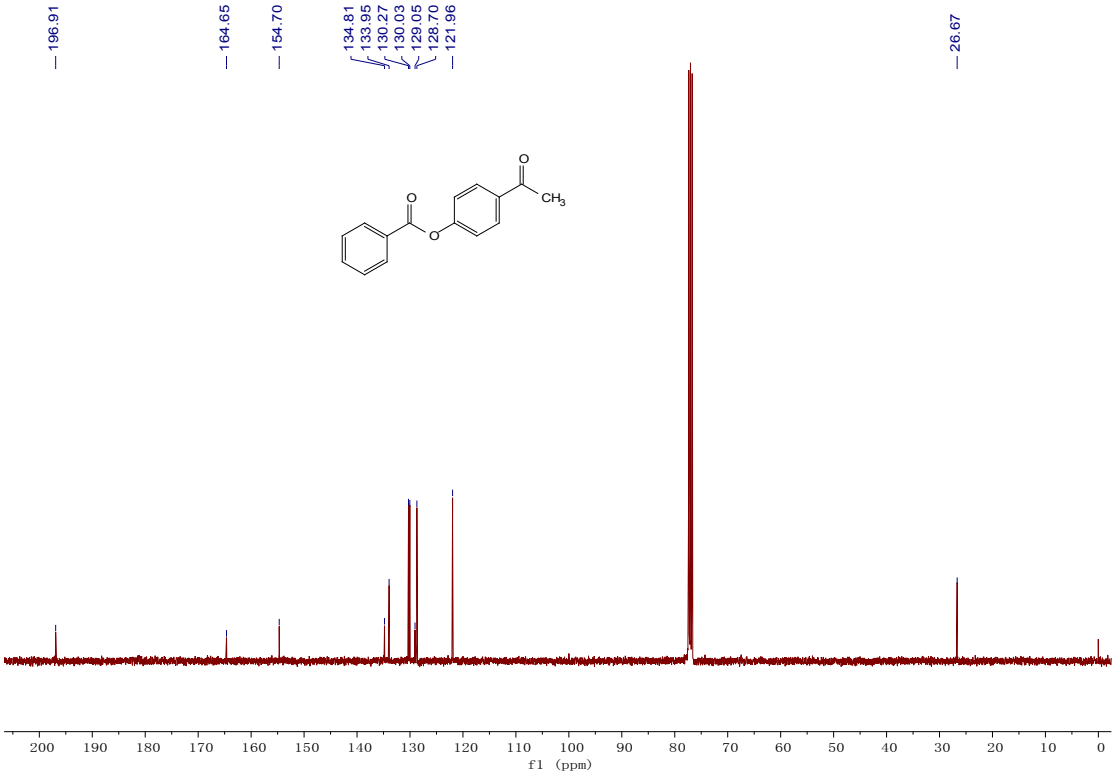
4-cyanophenyl benzoate (3bh)



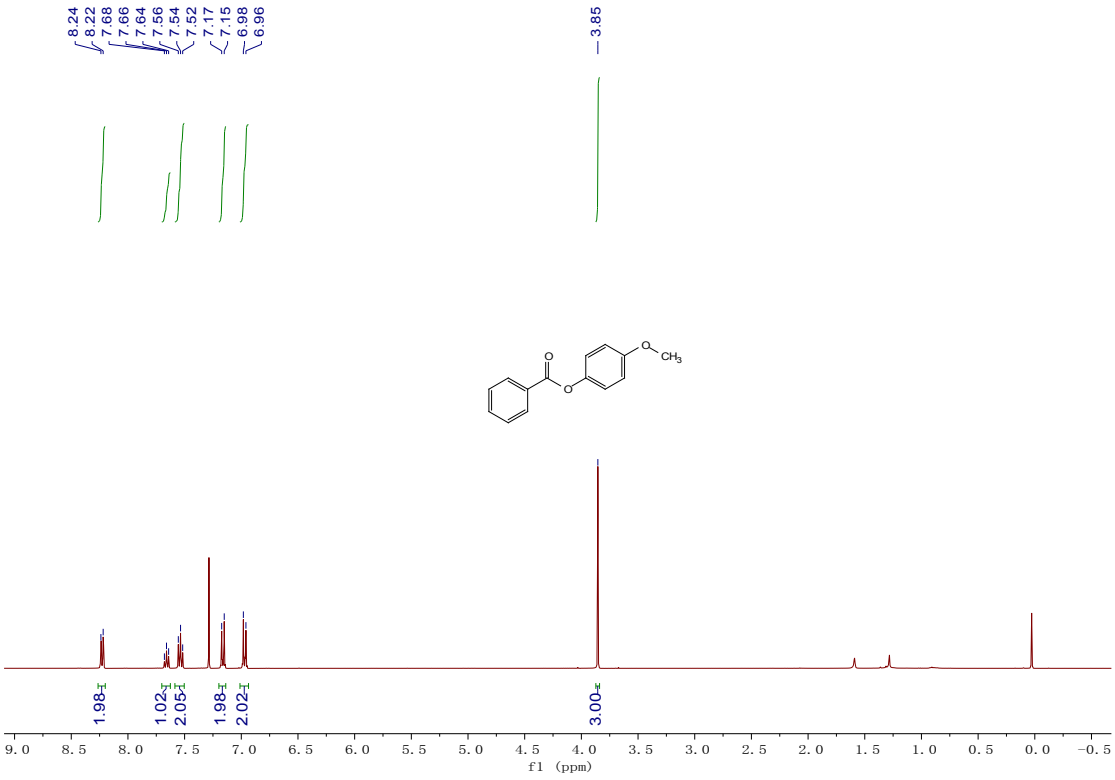


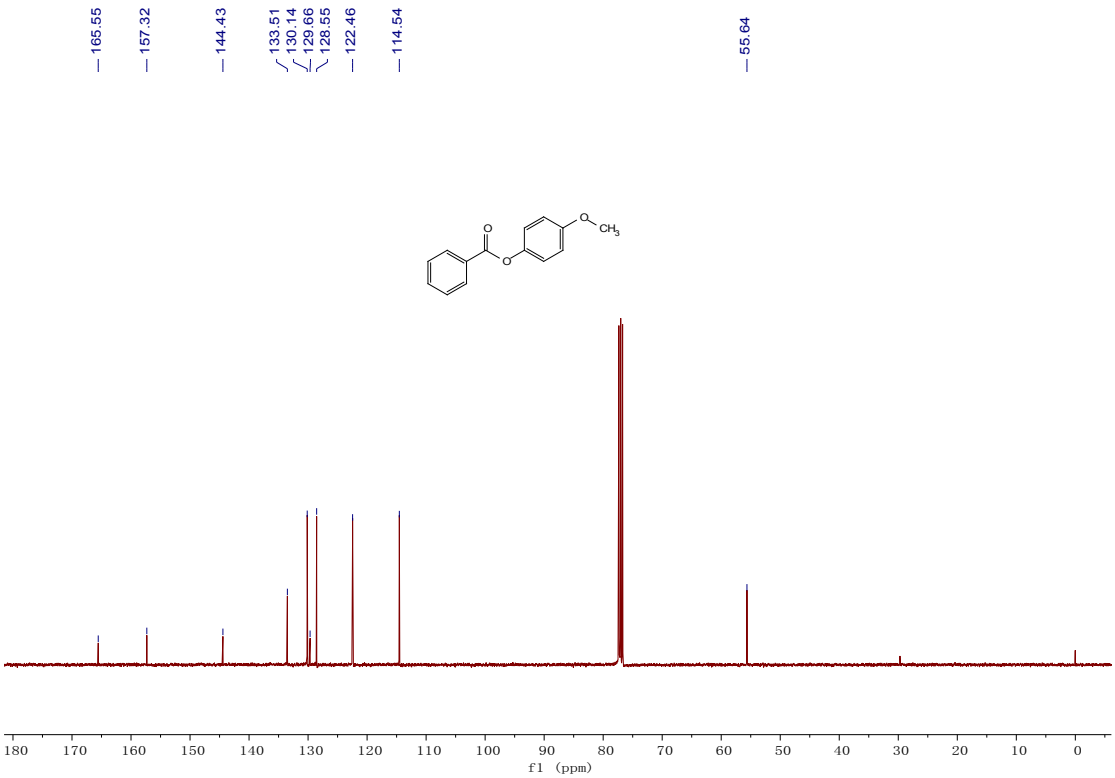
4-acetylphenyl benzoate (3bi)



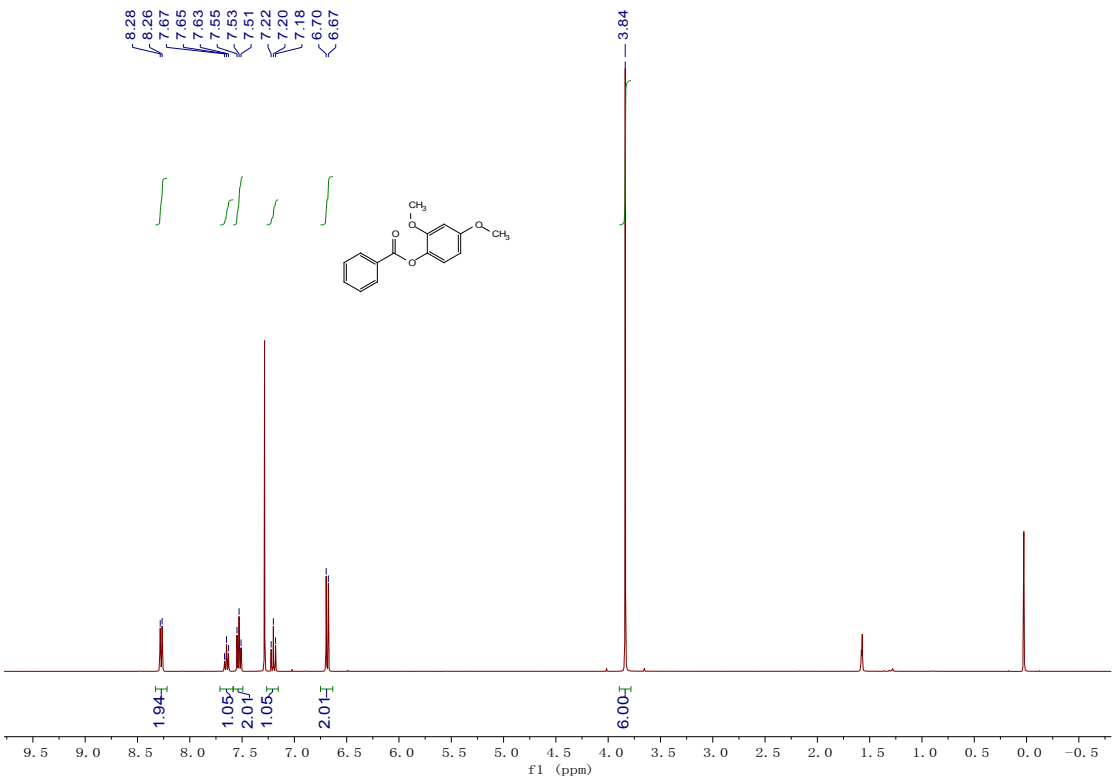


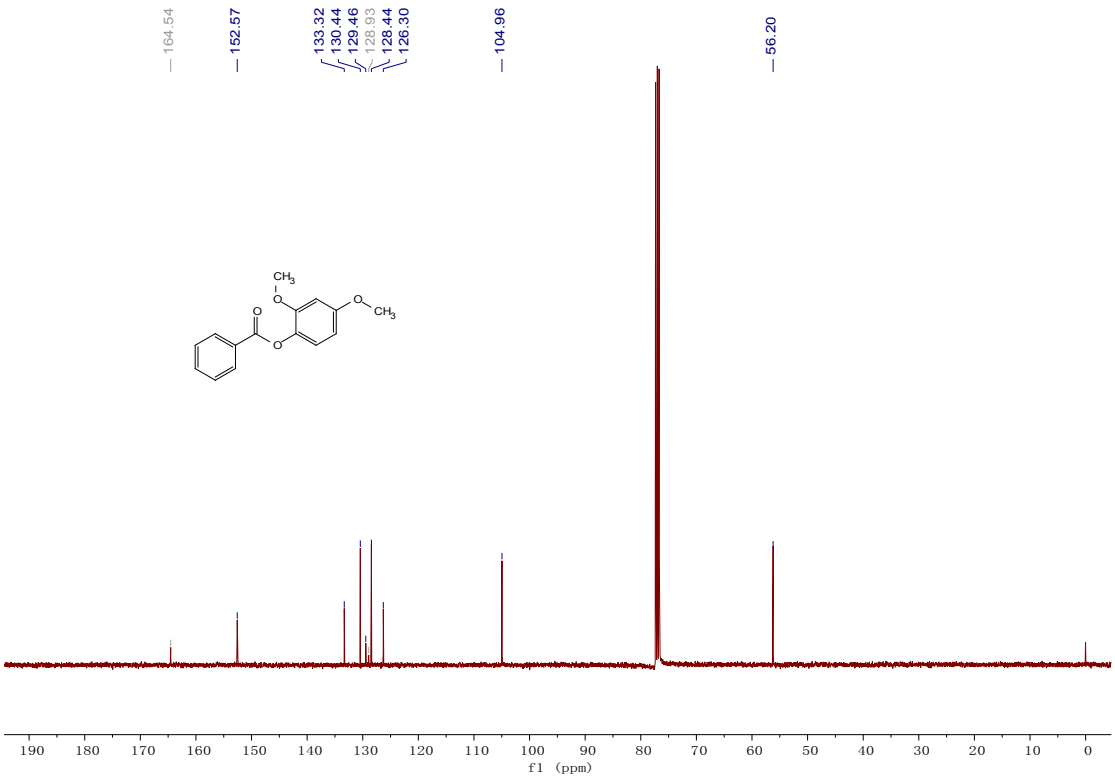
4-methoxyphenyl benzoate (3bj)



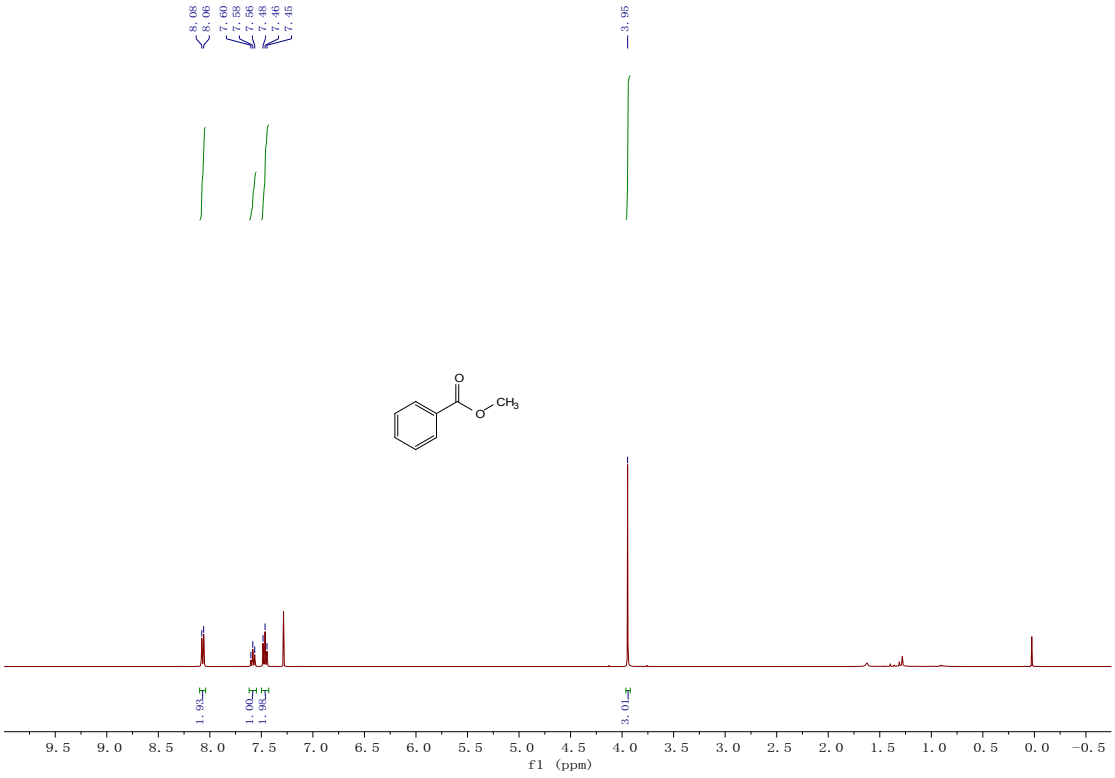


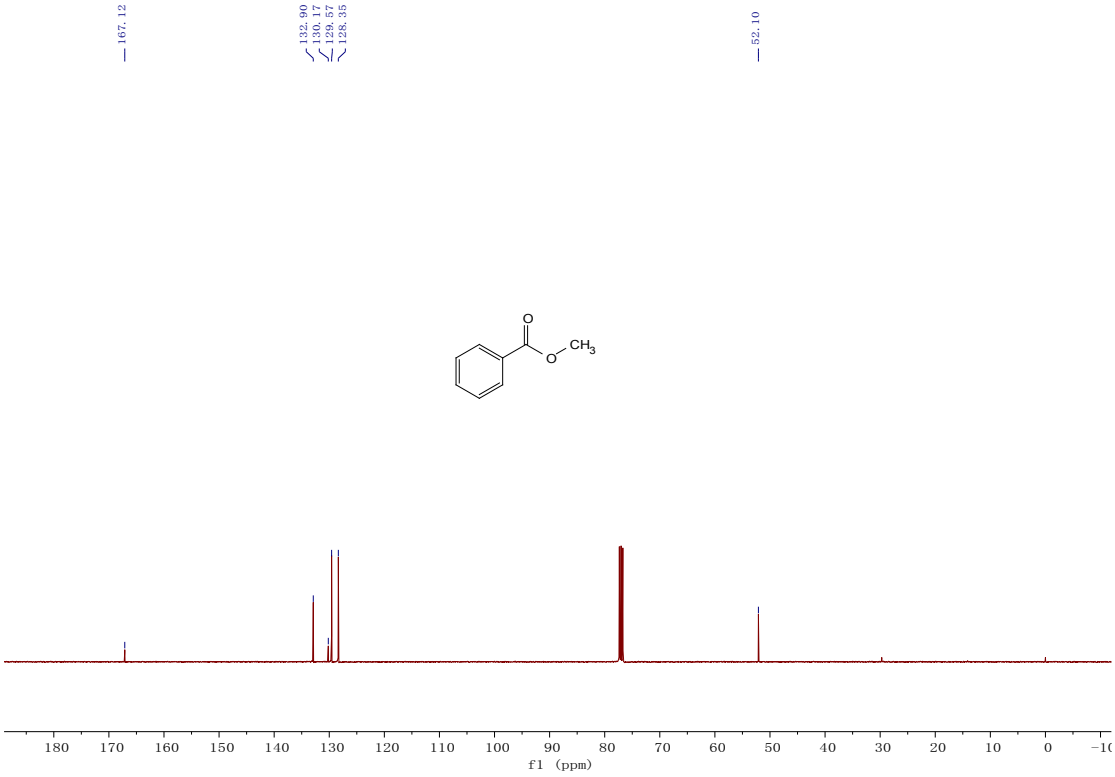
2,4-dimethoxyphenyl benzoate (3bk)



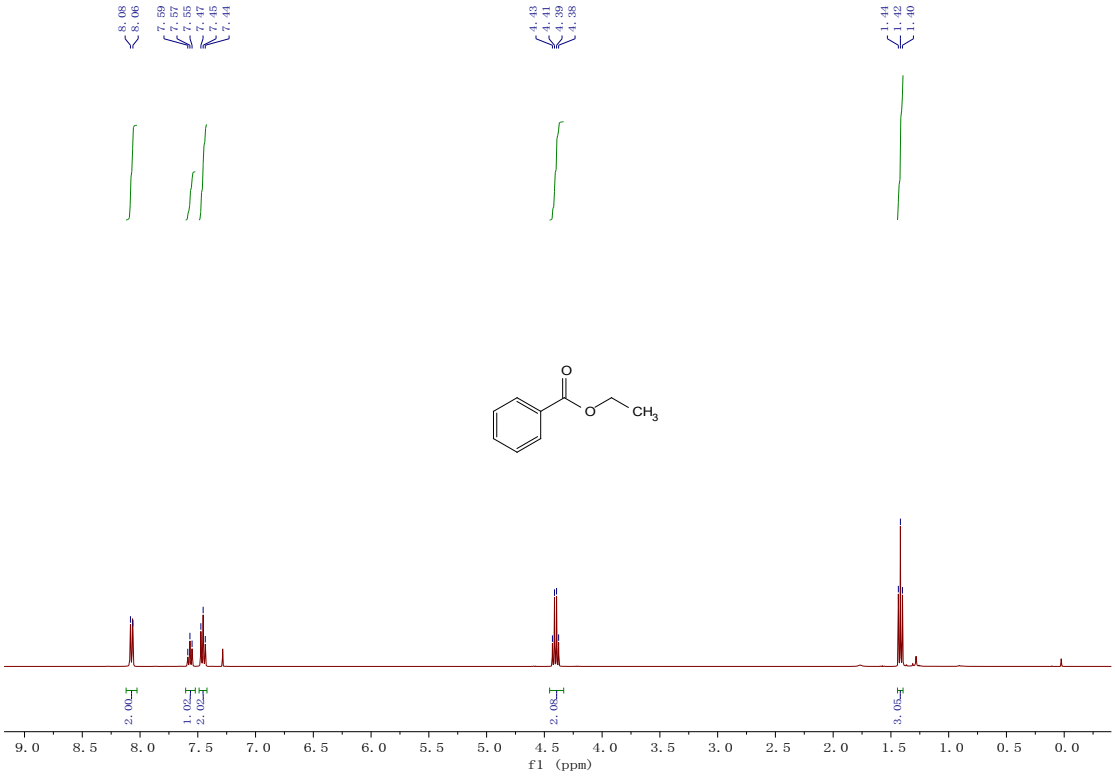


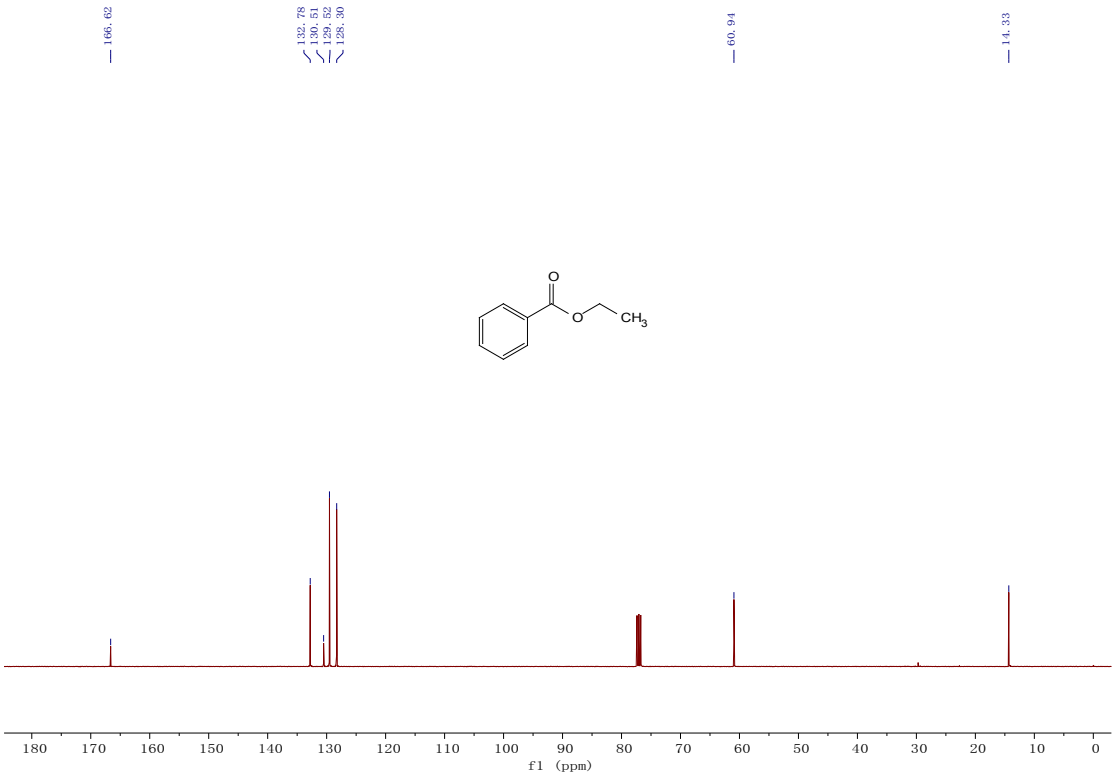
Methyl benzoate (3ca)



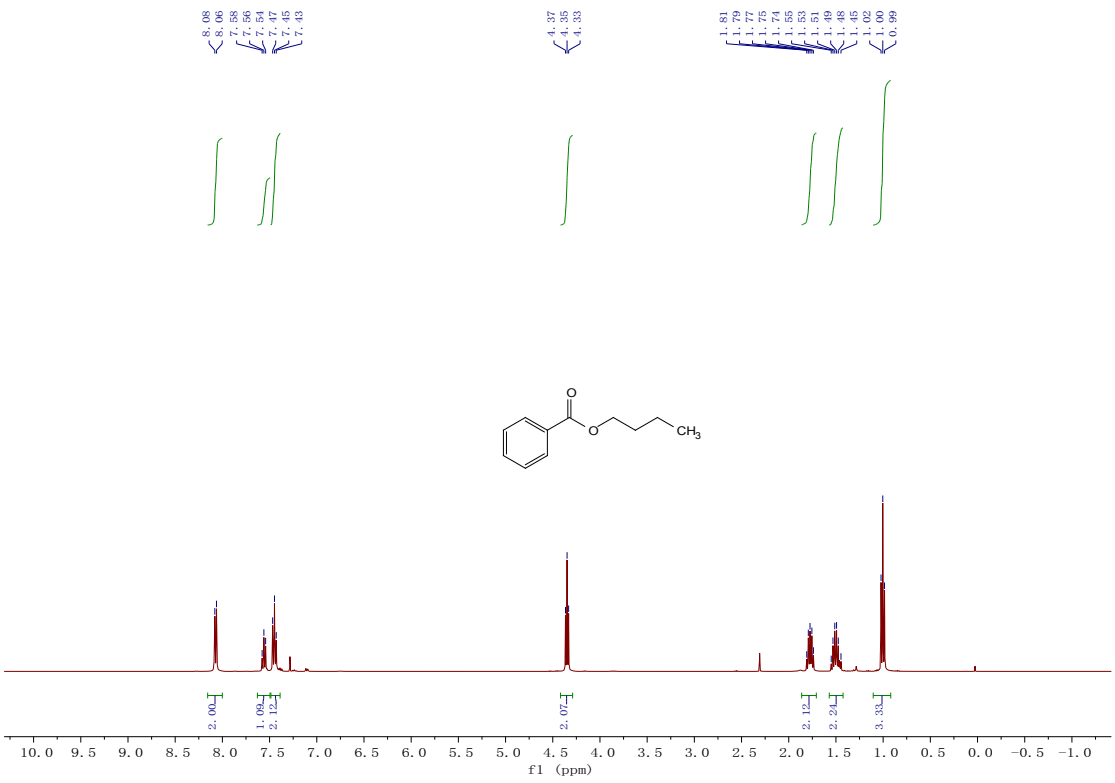


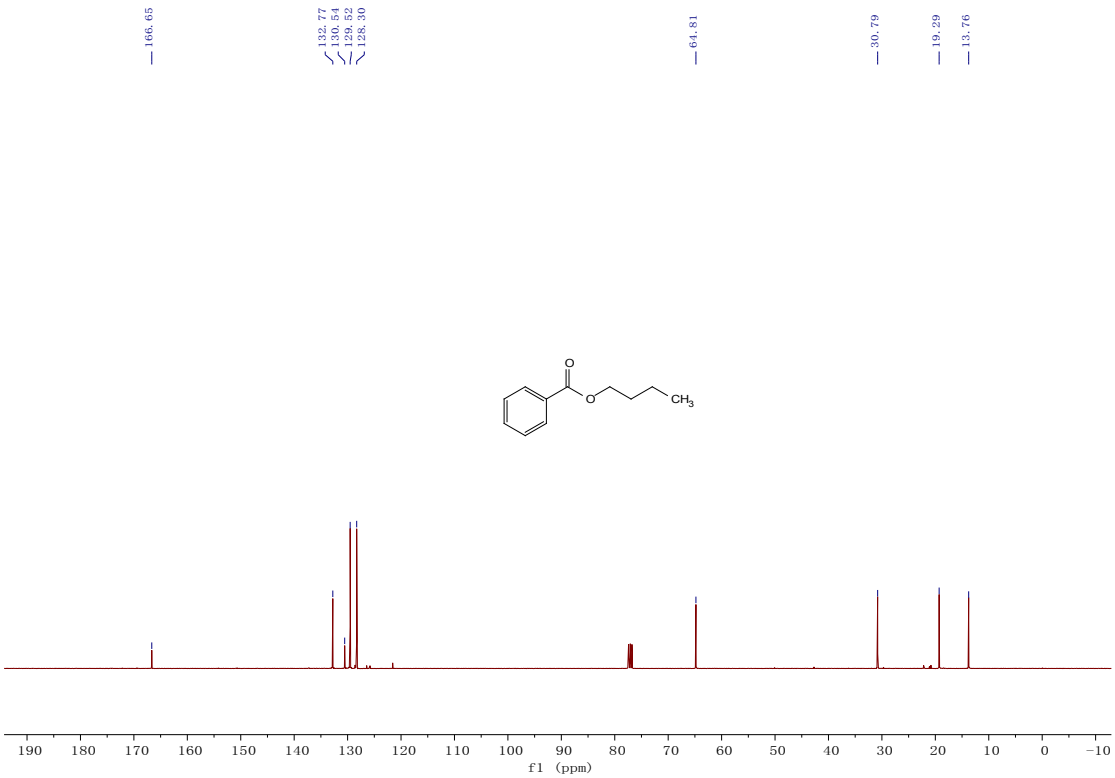
Ethyl benzoate (3cb)



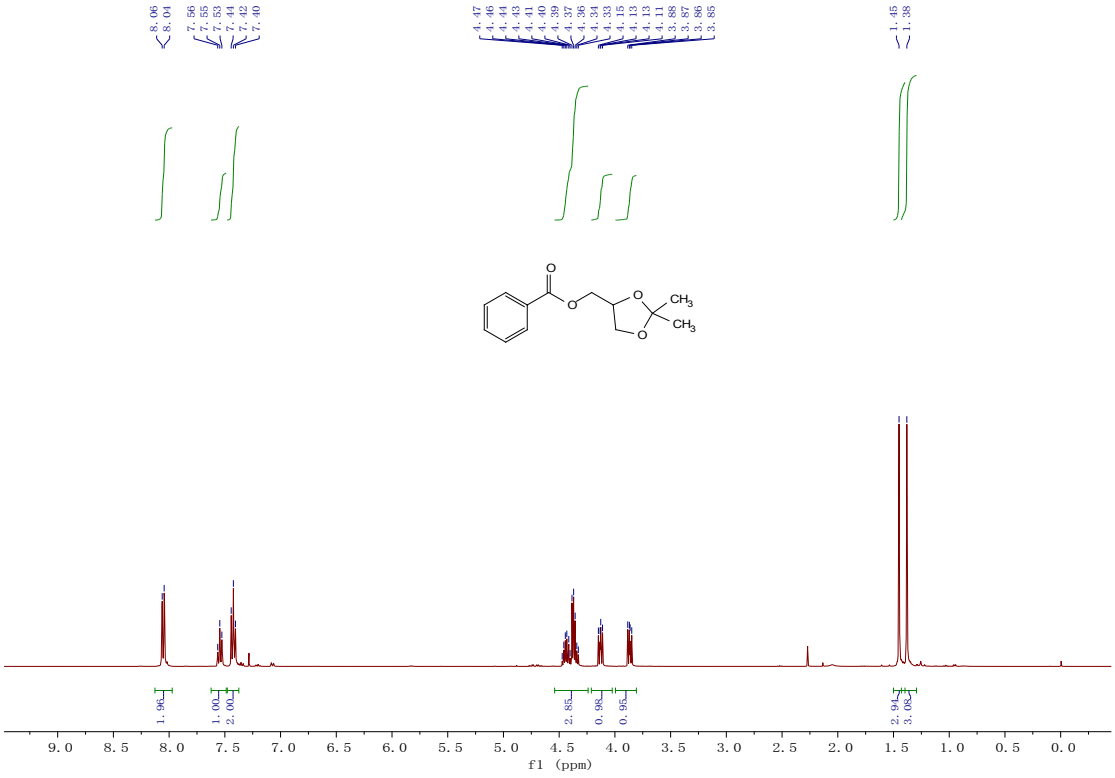


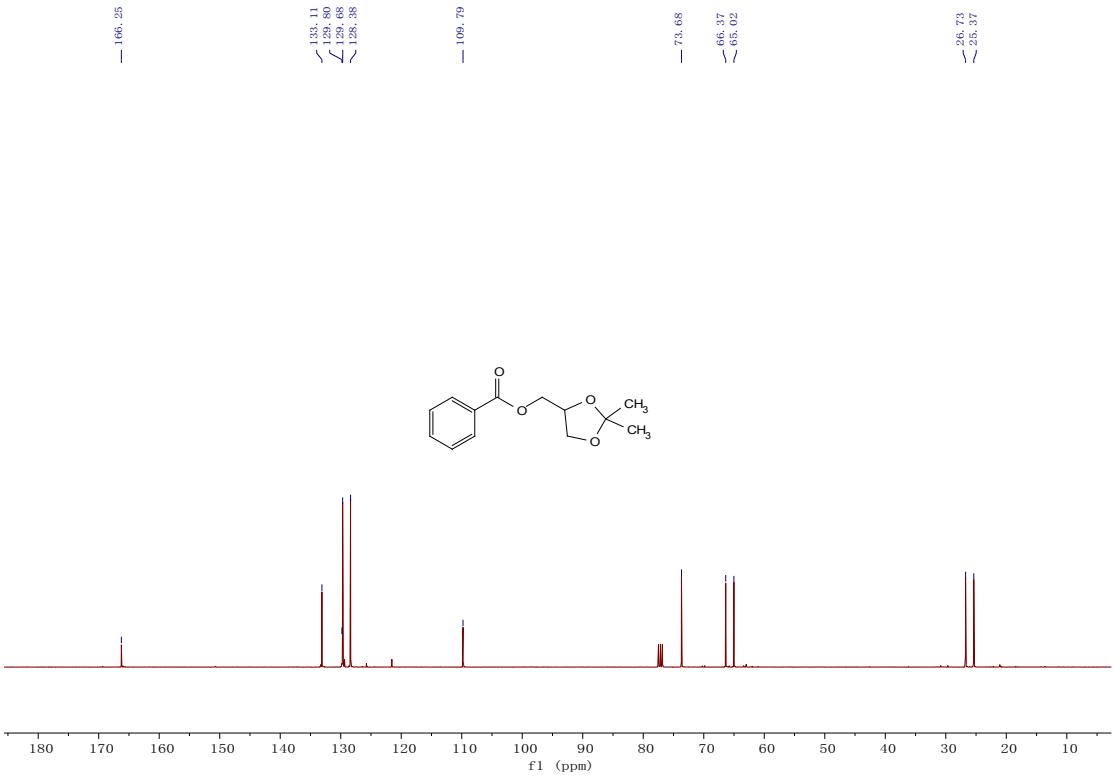
Butyl benzoate (3cc)



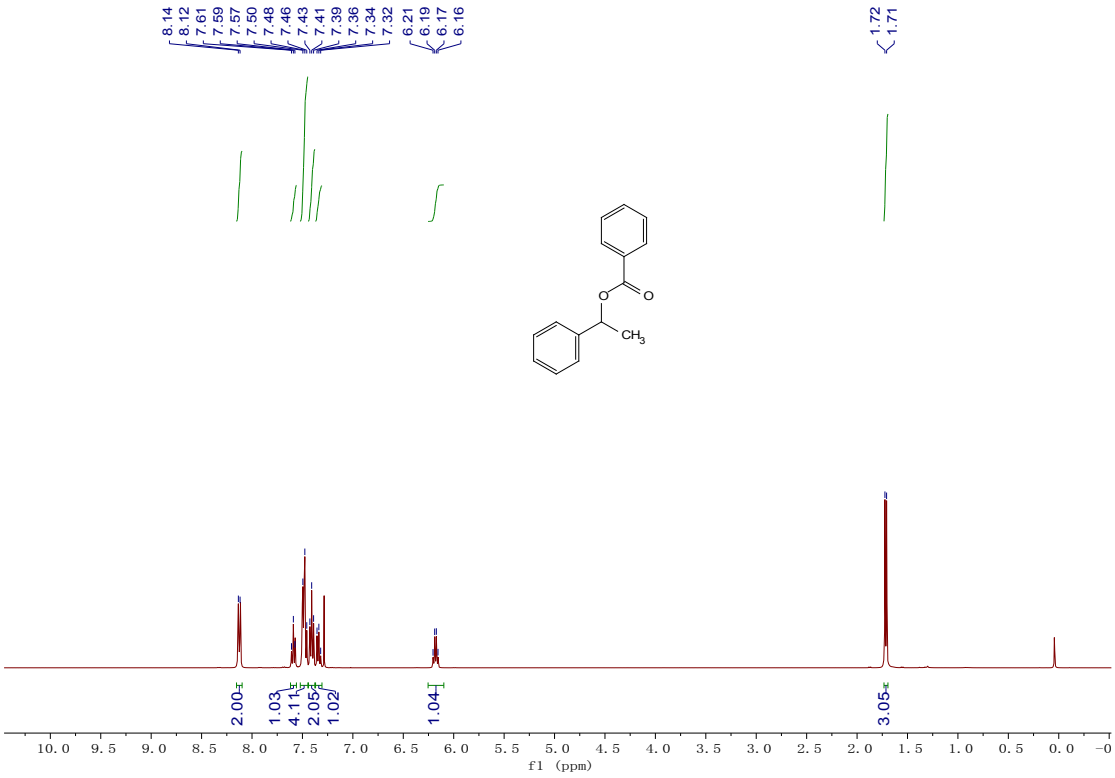


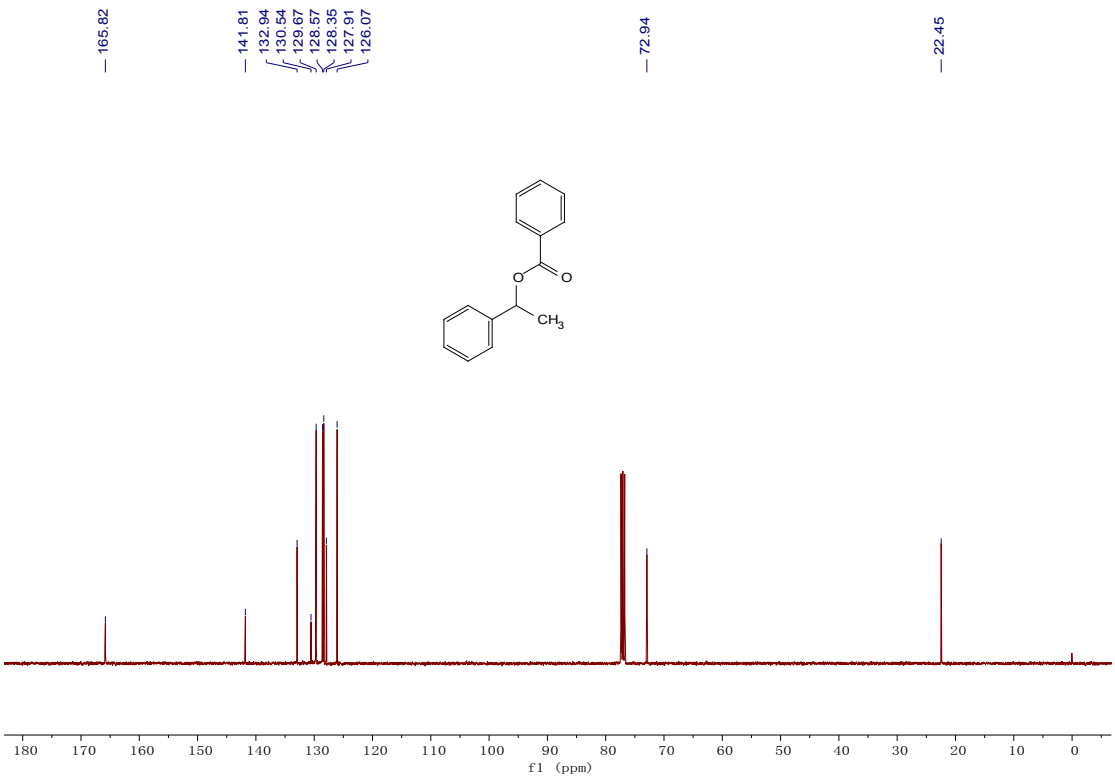
(2,2-dimethyl-1,3-dioxolan-4-yl)methyl benzoate (3cd)



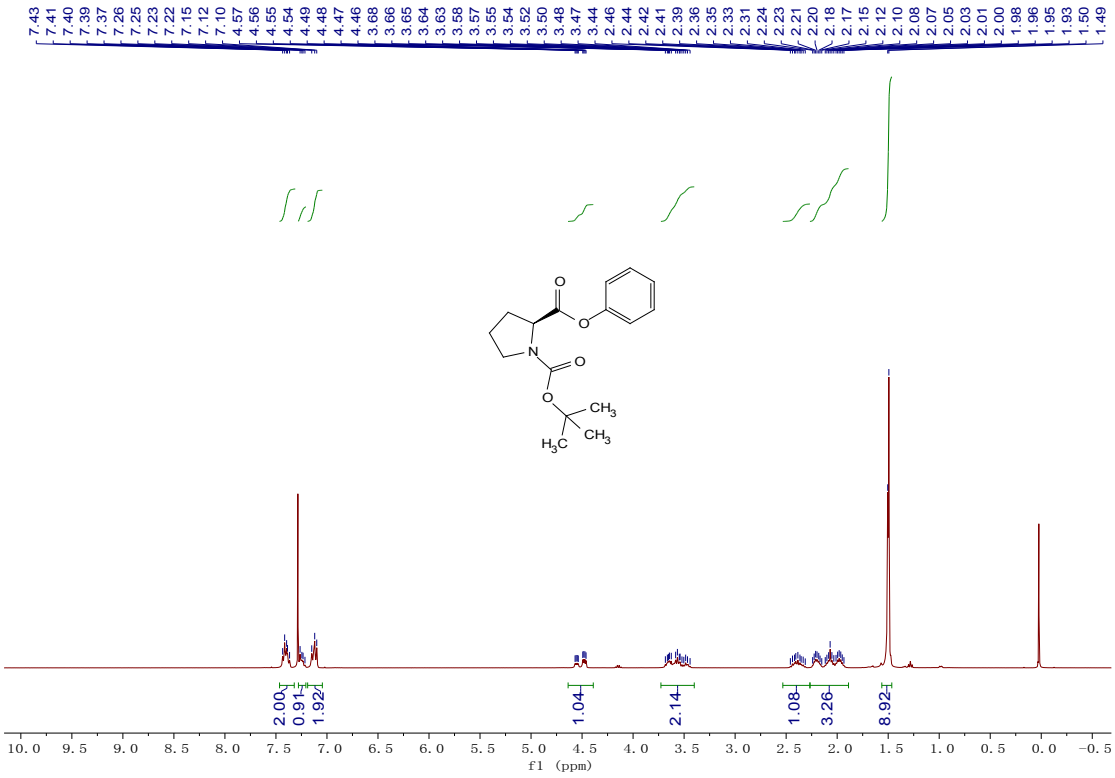


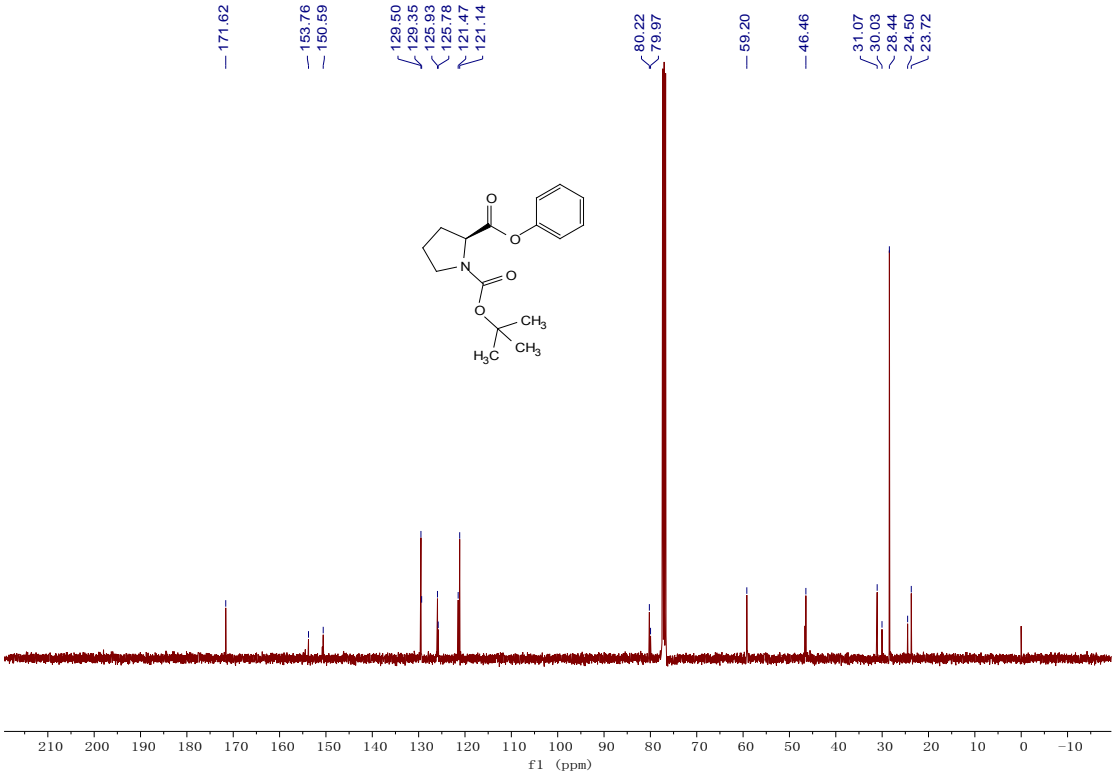
1-phenylethyl benzoate (3da)



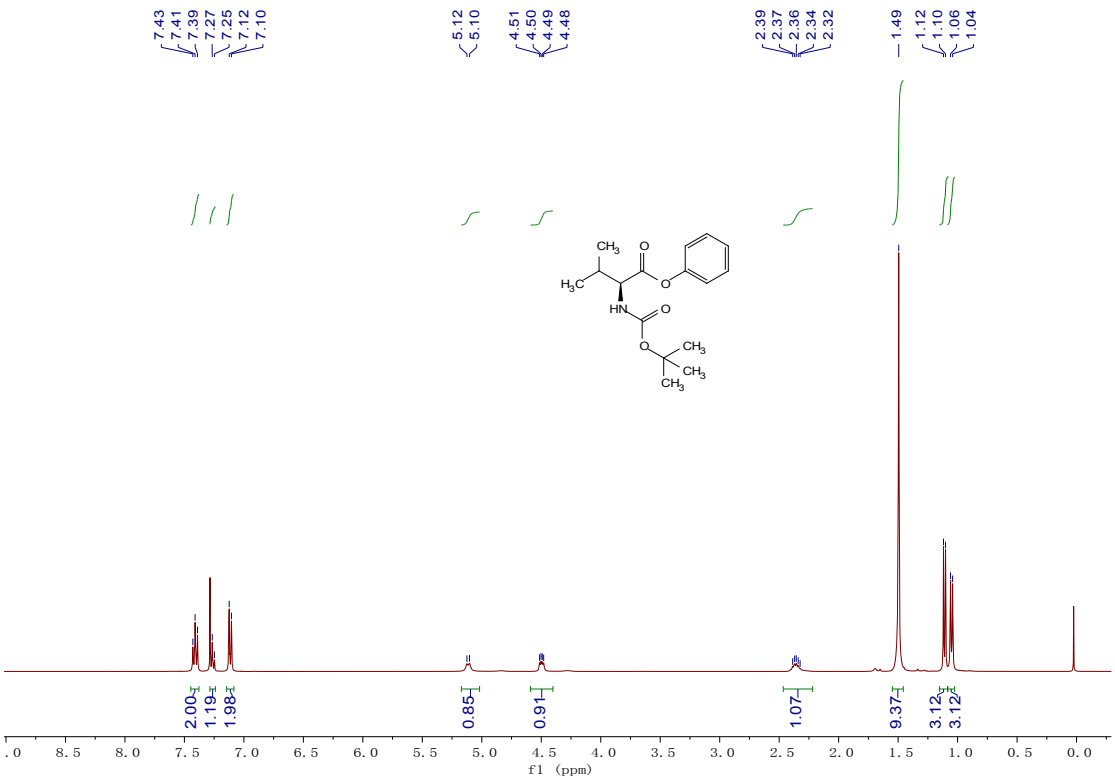


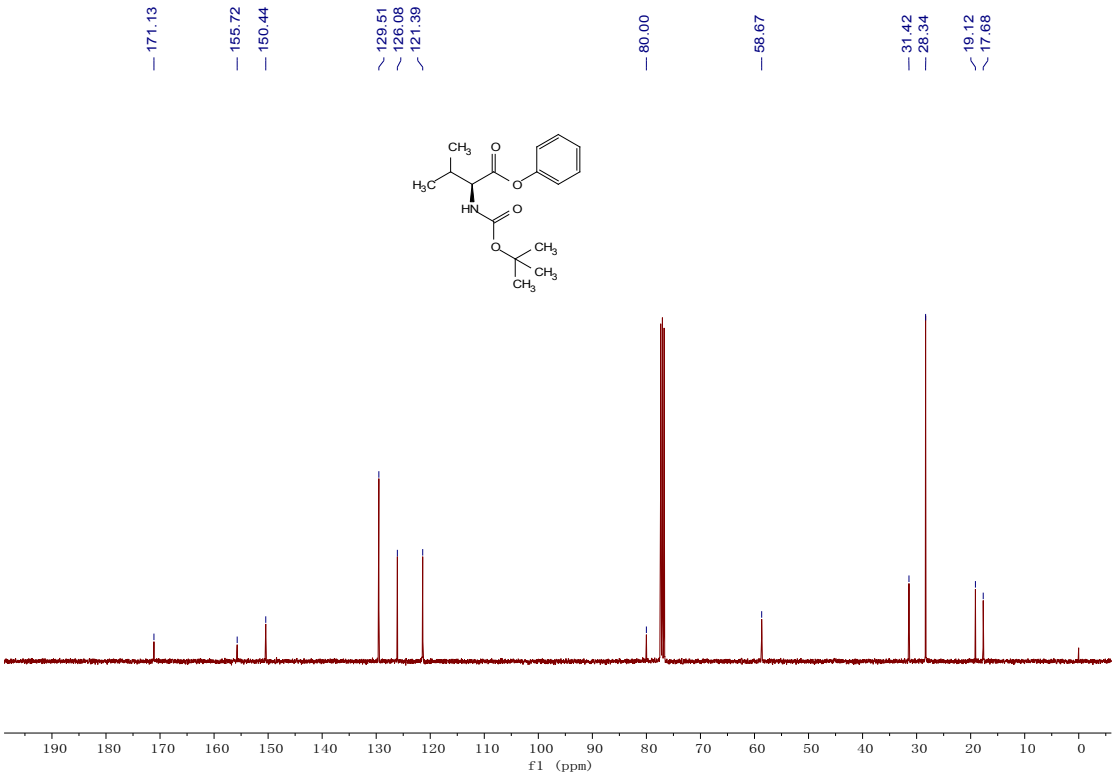
1-(*tert*-butyl) 2-phenyl (*S*)-pyrrolidine-1,2-dicarboxylate (3db).



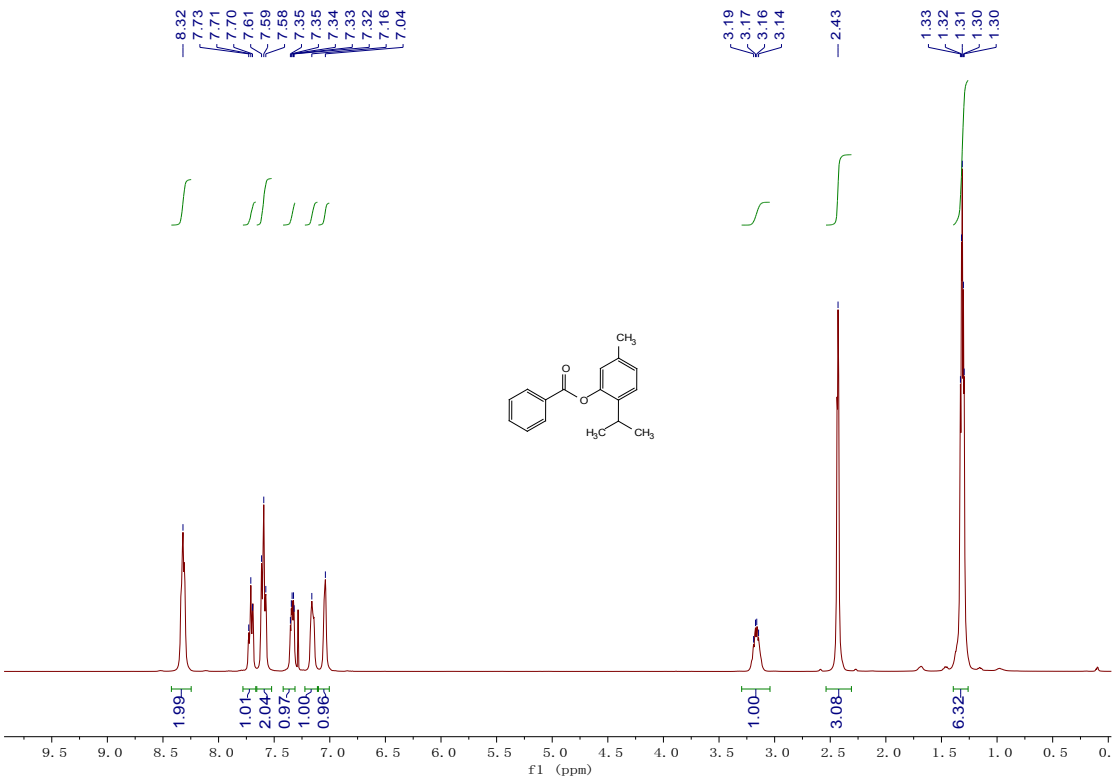


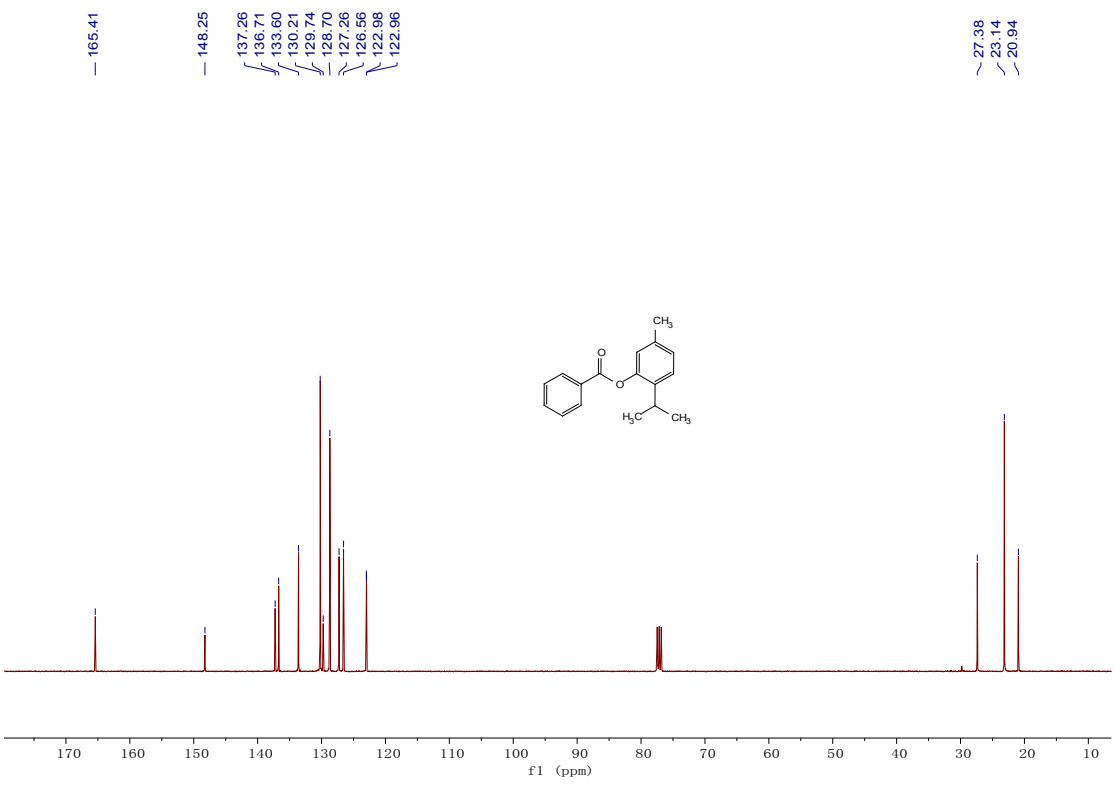
Phenyl (*tert*-butoxycarbonyl)-*L*-valinate (3dc)



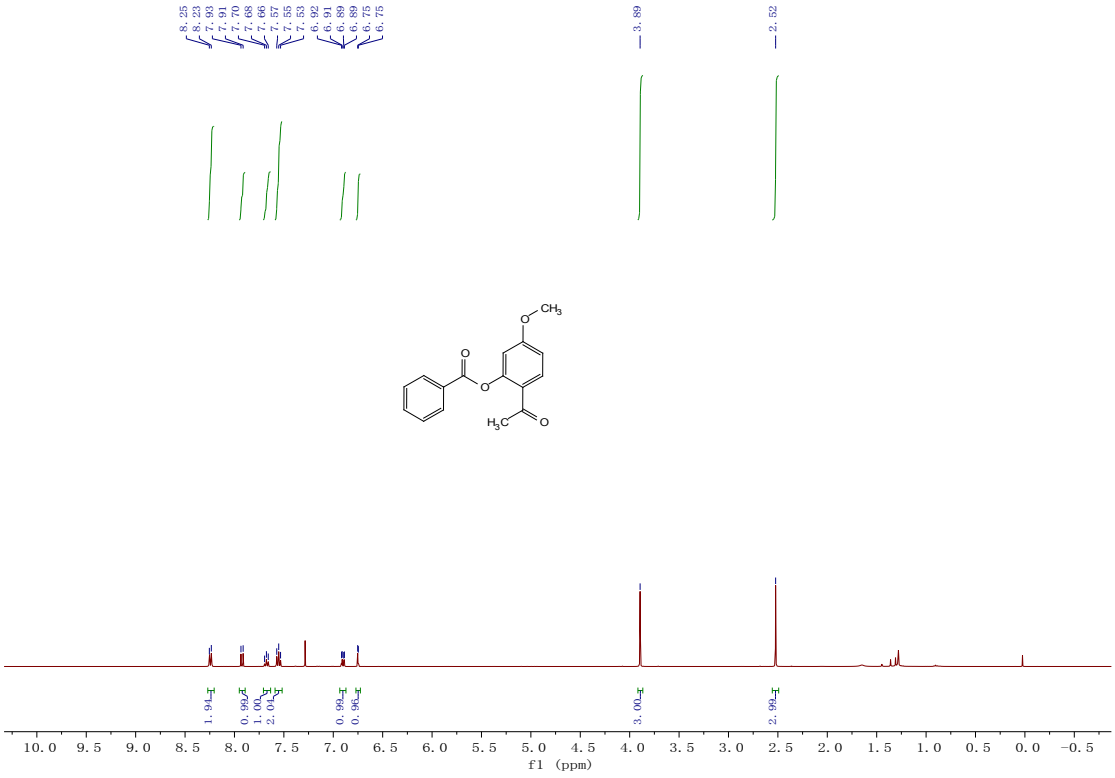


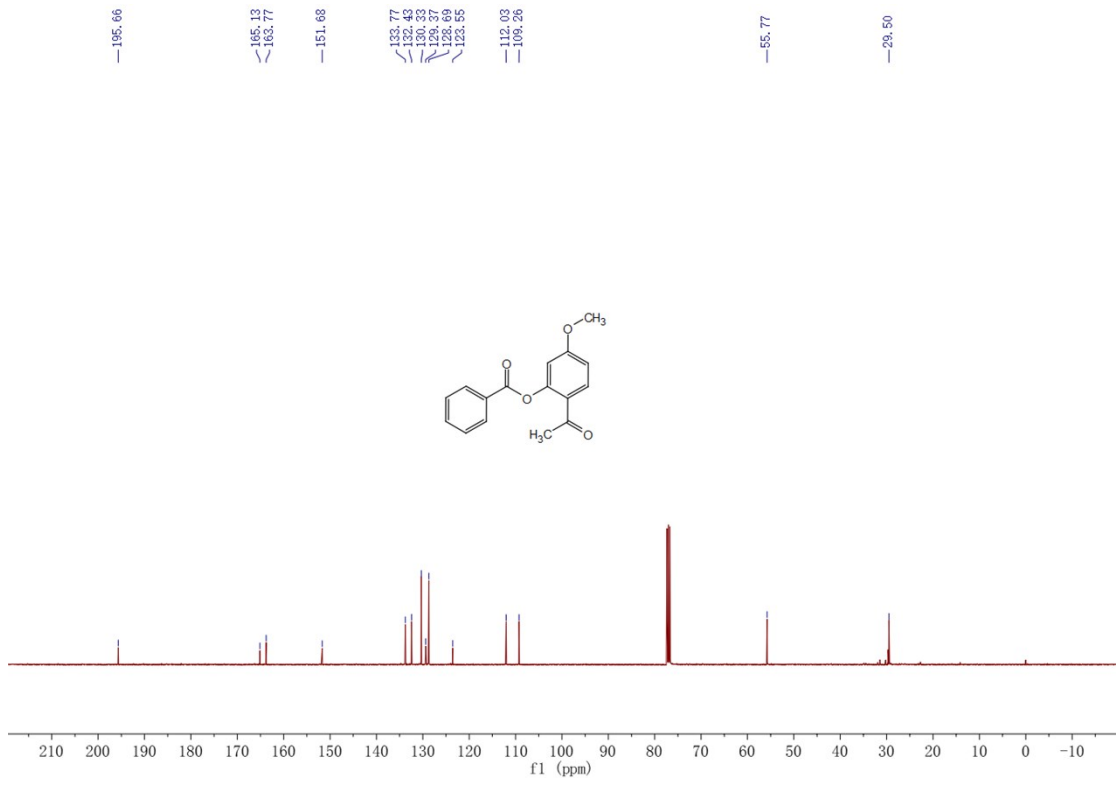
2-isopropyl-5-methylphenyl benzoate (3ea)



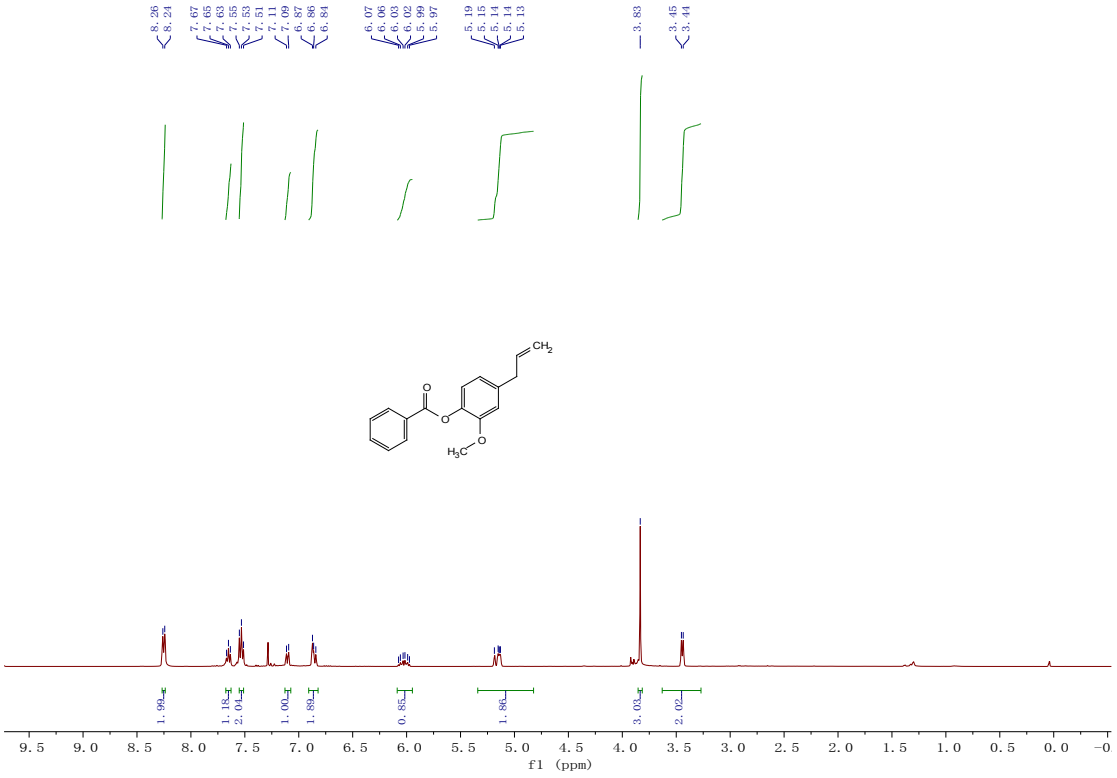


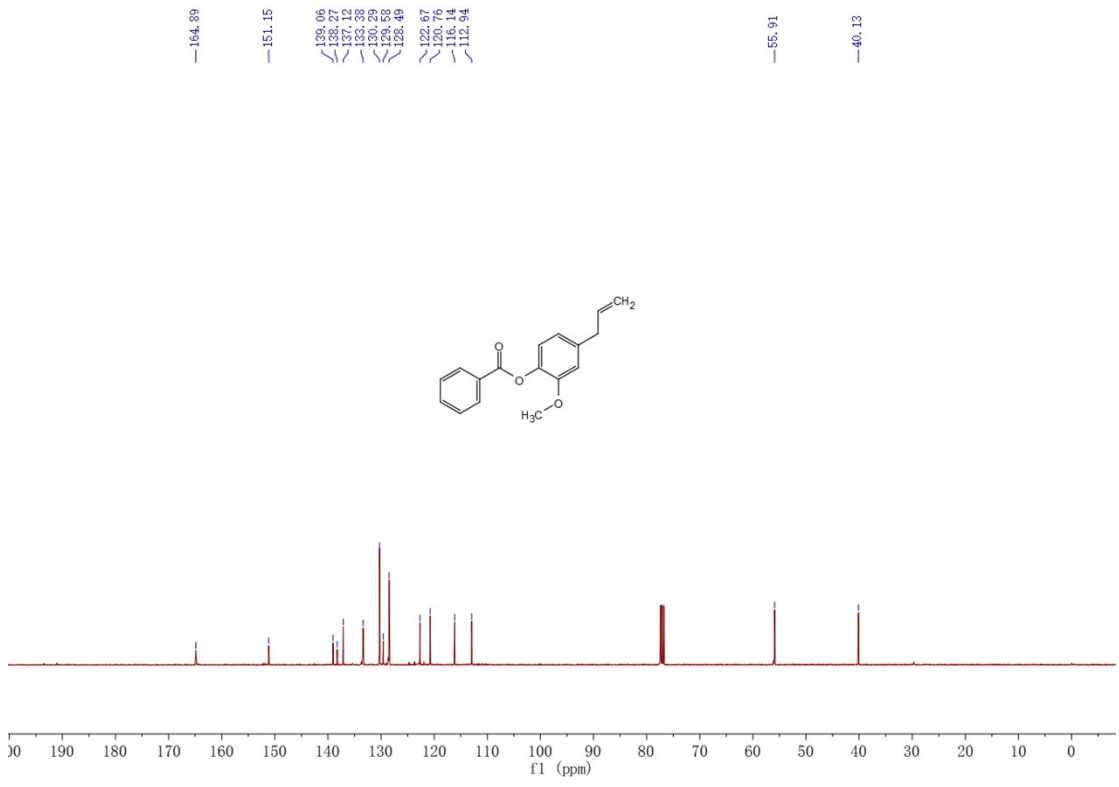
2-acetyl-5-methoxyphenyl benzoate (3eb)



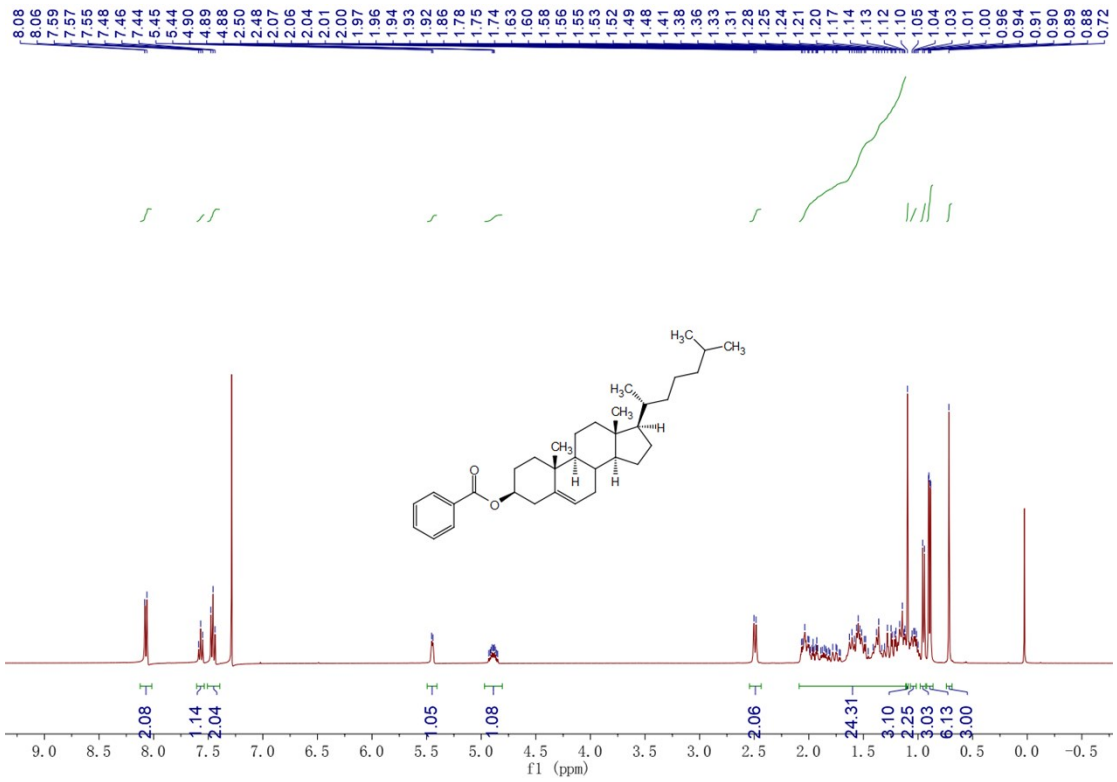


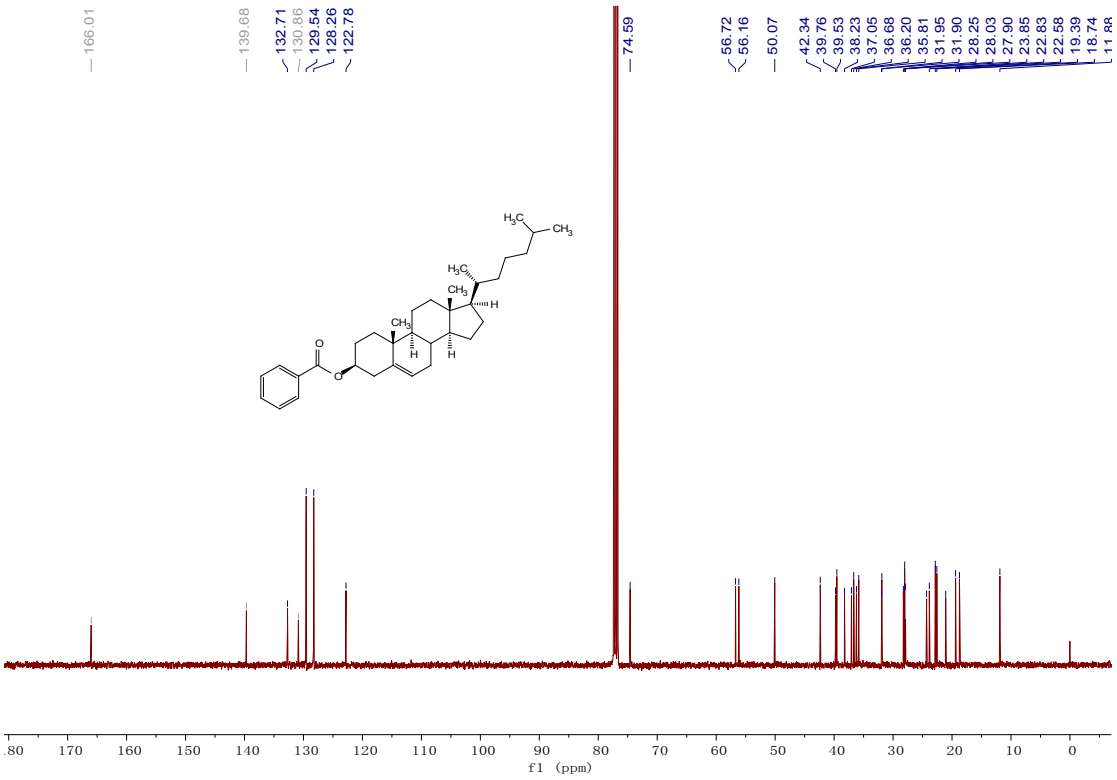
4-allyl-2-methoxyphenyl benzoate (3ec)



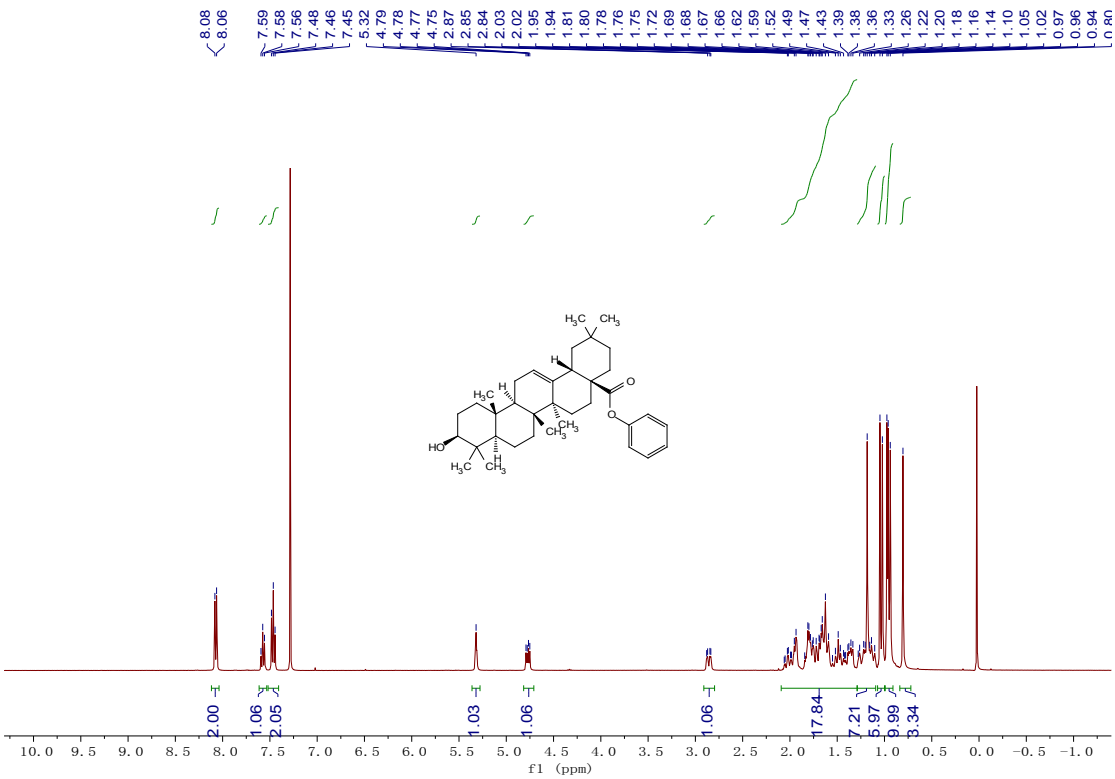


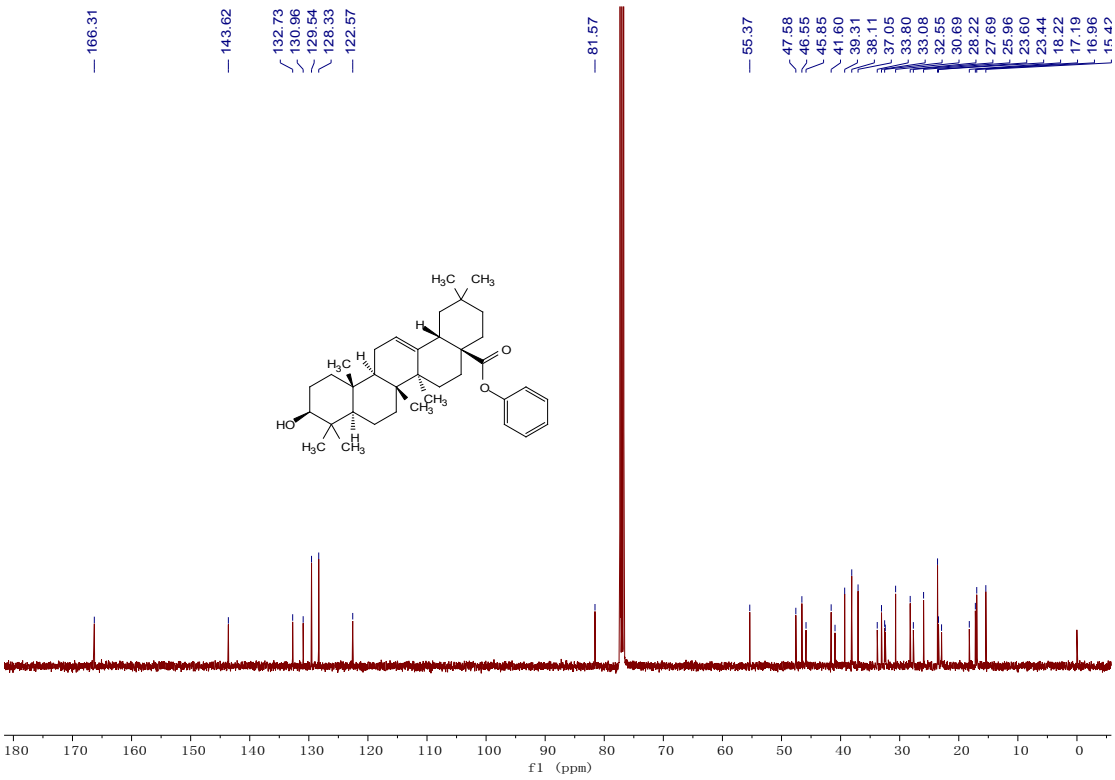
(3*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl benzoate (3ed)



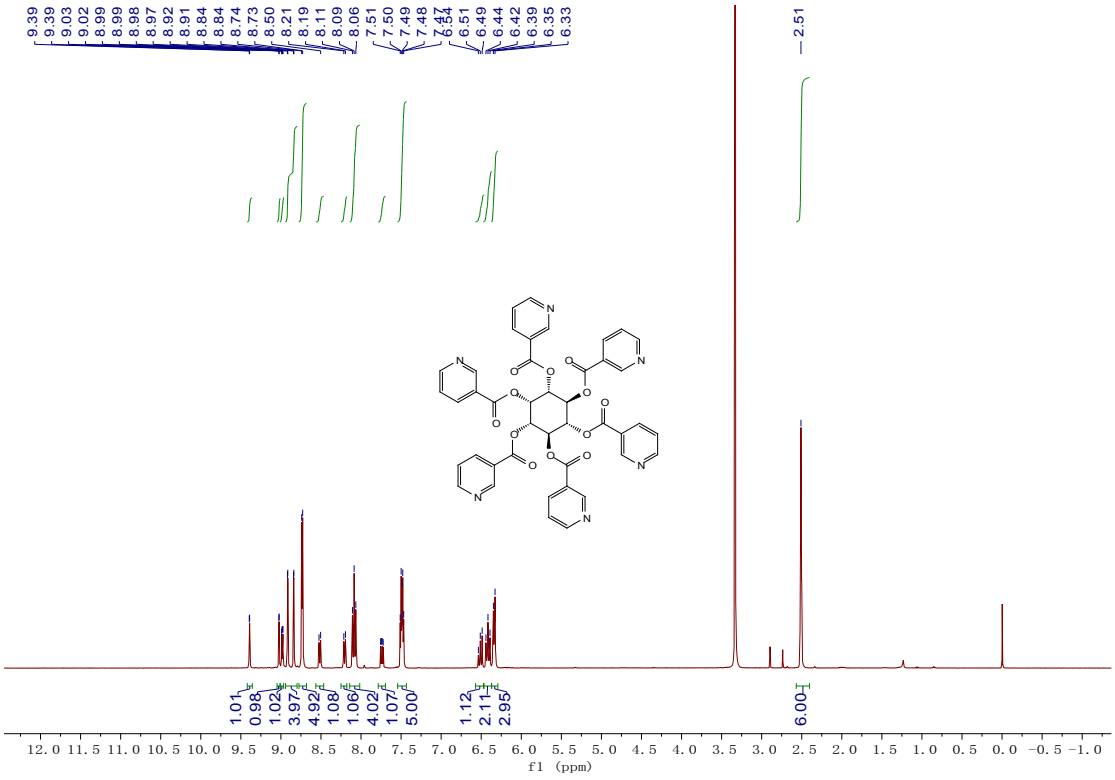


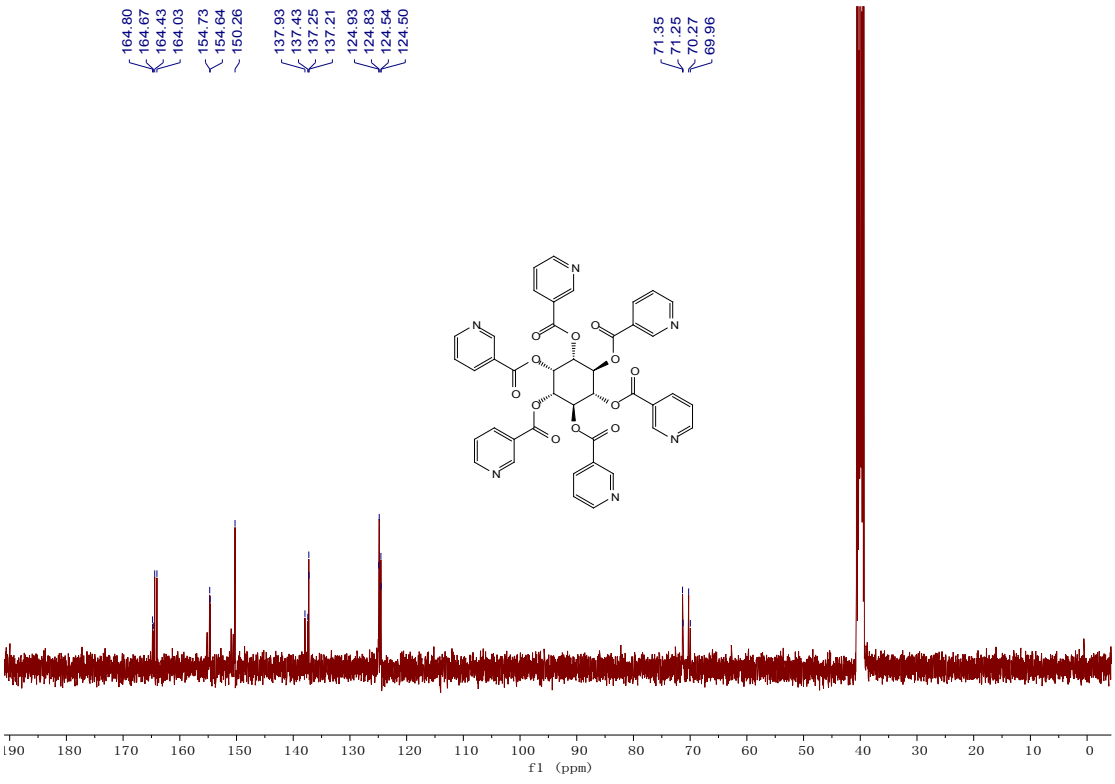
Phenyl(4a*S*,6a*S*,6b*R*,8a*R*,10*S*,12a*R*,12b*R*,14b*S*)-10-hydroxy-2,2,6a,6b,9,9,12a-heptamethyl-1,3,4,5,6,6a,6b,7,8,8a,9,10,11,12,12a,12b,13,14b-octadecahydropicene-4a(2*H*)-carboxylate (3ee)



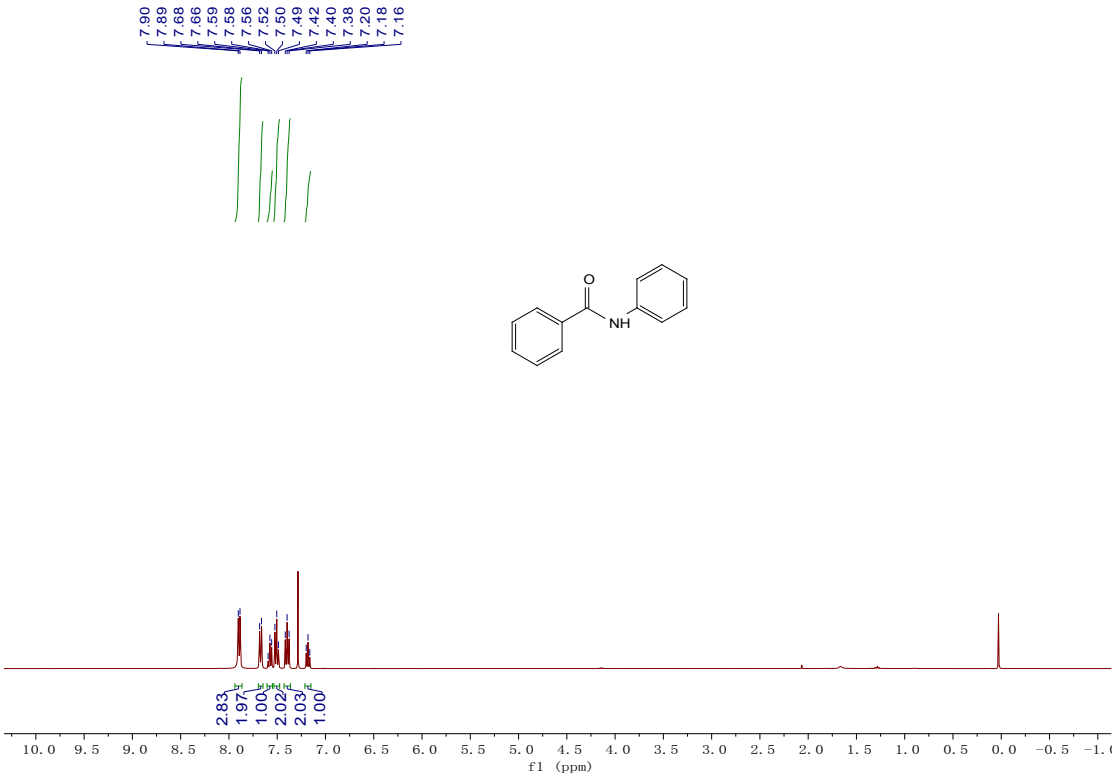


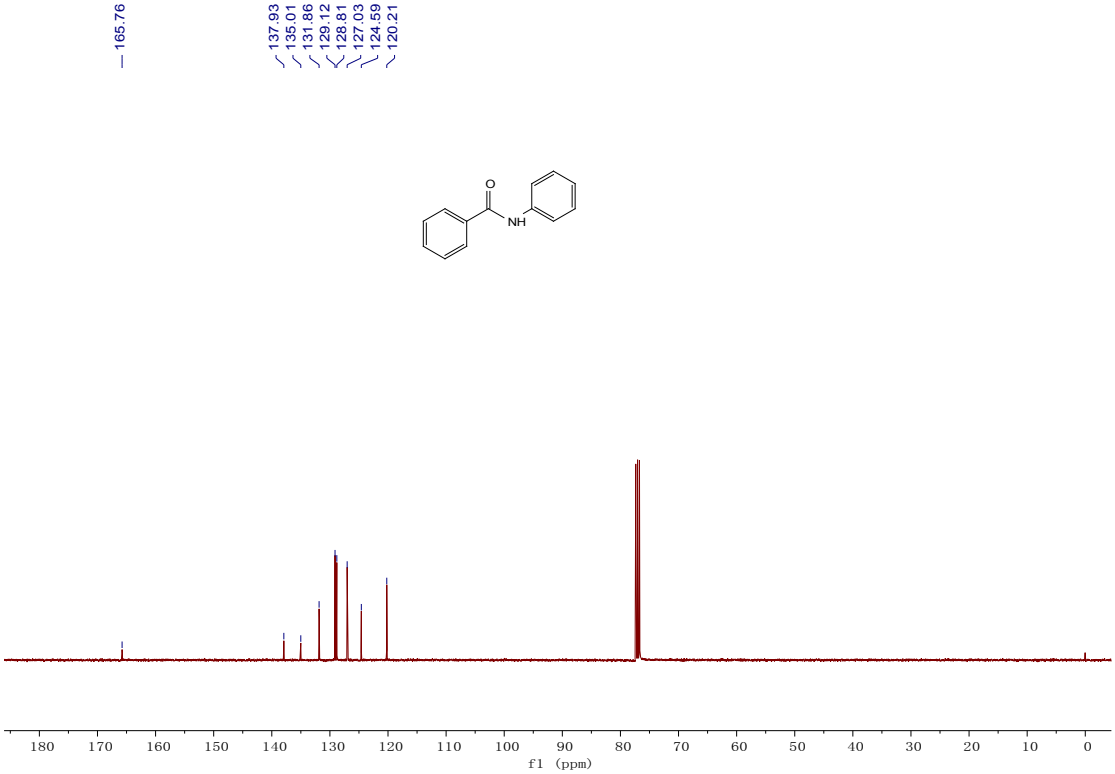
Inositol niacinate (3ef)



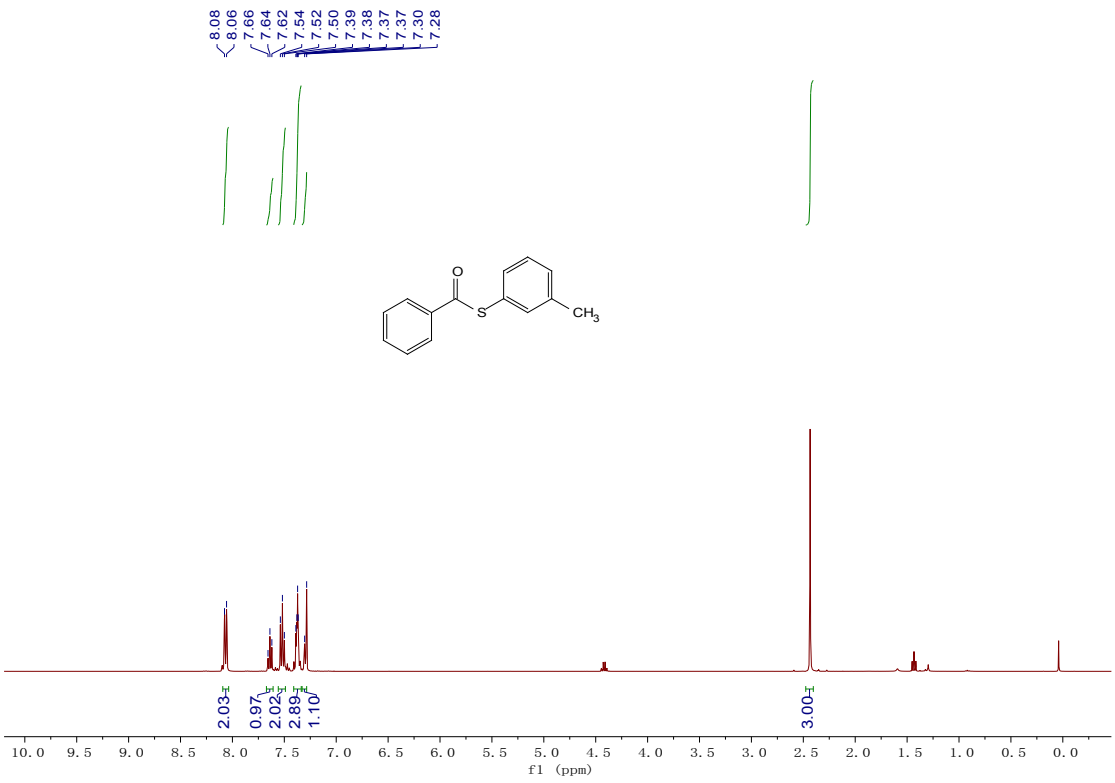


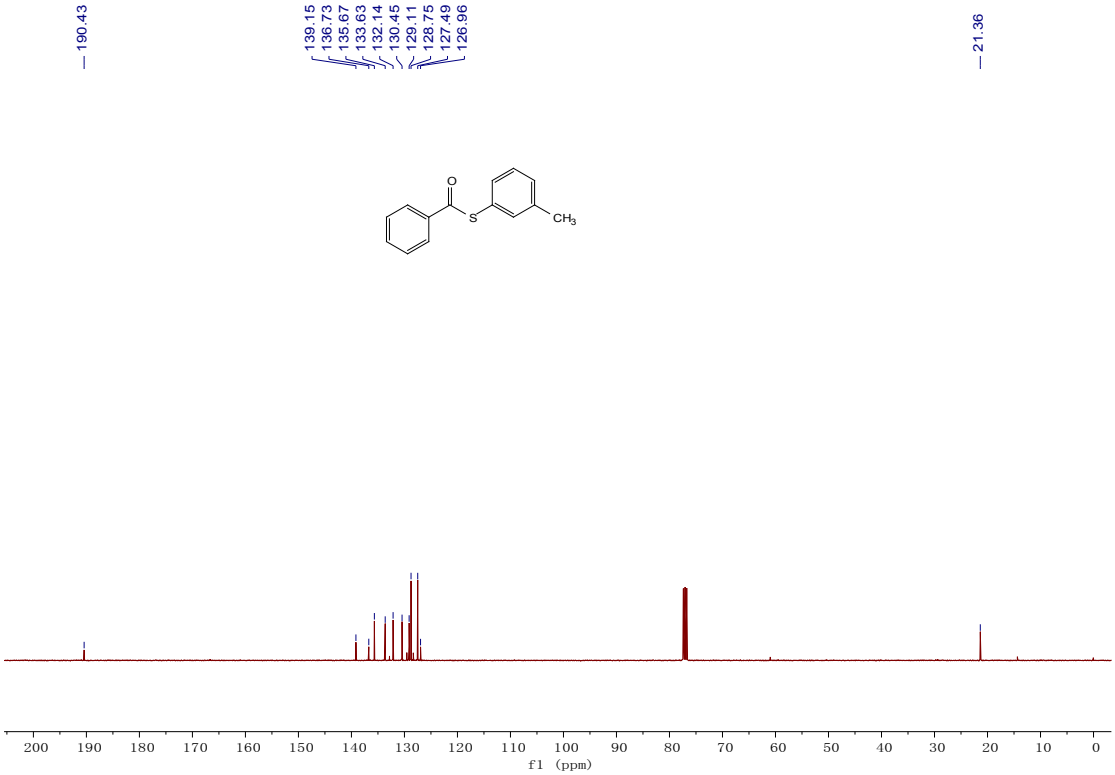
N-phenylbenzamide (3fa)





S-(*m*-tolyl) benzothioate (3fb)





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