Supporting information

Lanthanide-Catalyzed Deamidative Cyclization of Secondary Amides and Ynones through Tandem C–H and C–N Activation

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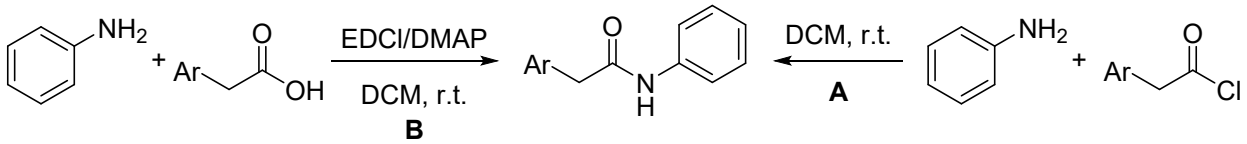
1. General information

All manipulations involving air- and moisture-sensitive compounds were performed under nitrogen atmosphere using Schlenk techniques or an Mbraun glovebox. All reagents were used directly without purification unless stated otherwise. Toluene, hexane and tetrahydrofuran (THF) were taken from a solvent purification system (PS-400-5, Unilab Mbraun, Inc.). $Ln[N(TMS)_2]_3$,[1] substituted phenylacetamide substrates[2,3] and substituted conjugated ynone substrates[4,5] were synthesized according to the corresponding literatures. Deuterated solvents were obtained from Cambridge Isotope. Toluene-d_8 was dried over sodium chips. ^{1}H NMR and ^{13}C NMR spectra were recorded on a JEOL ECA-400 NMR spectrometer (FT, 400 MHz for ^{1}H; 101 MHz for ^{13}C) at room temperature. All chemical shift values are quoted in ppm referenced to residual solvent ($CDCl_3$, δ = 7.26 ppm (^{1}H) and 77.16 ppm (^{13}C)) unless otherwise noted. The following abbreviations were used to describe peak splitting patterns when appropriate: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constant (*J*) was reported in Hz unit. High resolution mass spectra (HRMS) were obtained on a micro TOF II Instrument using ESI ionization sources. Column chromatography was performed using EM silica gel 60 (300−400 mesh).

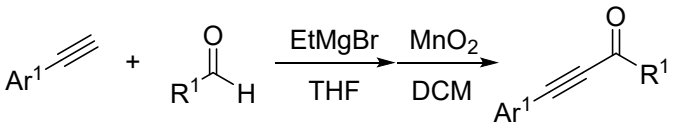
2. Preparation of substrates.

2.1 Synthesis of 2-arylacetamides



Method A:[2] To a solution of aniline (1.39 g, 15 mmol) in DCM (25 mL), 2-arylacetyl chloride (1.3 mL, 10 mmol) was added. After stirring at room temperature for 1 h, HCl (1 M) was added slowly and the mixture was extracted with ethyl acetate (3 × 30 mL). The combined extract was washed with saturated $NaHCO_3$ (2 × 10 mL) and brine, dried over anhydrous Na_2SO_4. After removal of solvent, the residue was purified by either recrystallization in ethyl acetate or by flash chromatography (Silica gel, petroleum ether : ethyl acetate = 50 : 1 as eluent) to afford the corresponding 2-arylacetamide.
Method B: To a solution of 2-phenylacetic acid (10 mmol), aniline (11 mmol) in DCM (25 mL) were added EDCI (2.493 g, 13 mmol) and DMAP (366.6 mg, 3.0 mmol). The reaction mixture was stirred at room temperature overnight, diluted with HCl (1 M) aqueous solution, and extracted with DCM (3 × 25 mL). The combined organic phase was washed with saturated $NaHCO_3$ aqueous solution and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated under vacuum. Purification by flash chromatography (Silica gel, petroleum ether: ethyl acetate = 50 : 1 as eluent) gave the corresponding phenylacetamides.[3]

2.2 Synthesis of conjugated ynones[4]



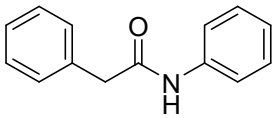
A solution of arylacetylene (10 mmol) in dry THF (15 mL) was cooled to 0 ℃ under N_2. To which CH_3CH_2MgBr (11 mmol) was added dropwise, the reaction mixture was stirred for 4 h at this temperature. After that, the corresponding aldehyde (15 mmol) was added dropwise, and the reaction

solution was allowed to warm to room temperature. After stirring for 4 h, the reaction mixture was quenched with water, and extracted with ethyl acetate. The combined extract was dried over $MgSO_4$, filtered and condensed to give the crude product alcohol, to which DCM (25 mL) and MnO_2 (0.10 mol) were added. The mixture was stirred at rt in the dark for 2 h. The reaction solution was filtered over celite, solvents were removed, and the crude product was purified via column chromatography on silica gel with hexane/EtOAc and concentrated to afford the corresponding conjugated ynone.

3. Characterization data for substrates

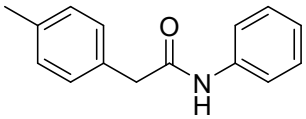
3.1 Characterization data of phenylacetamide

N,2-diphenylacetamide (1a)



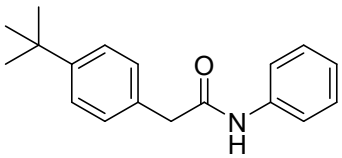
1.92 g, 91% yield; as a white solid. m.p. 116-117 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.50 (br, *J* = 10.8 Hz, 1H, NH), 7.42 (d, *J* = 7.7 Hz, 2H, Ar-H), 7.39-7.33 (m, 2H, Ar-H), 7.33-7.28 (m, 3H, Ar-H), 7.25 (t, *J* = 7.9 Hz, 2H, Ar-H), 7.06 (t, *J* = 7.6 Hz, 1H, Ar-H), 3.68 (s, 2H, CH_2). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 169.37, 137.76, 134.58, 129.51, 129.16, 128.94, 127.59, 124.47, 120.00, 44.74.

N-phenyl-2-(p-tolyl)acetamide (1b)



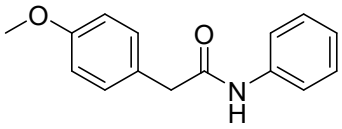
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.41 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.32 (s, 1H, NH), 7.25 (t, *J* = 7.8 Hz, 2H, Ar-H), 7.19 (d, *J* = 2.3 Hz, 4H, Ar-H), 7.06 (t, *J* = 7.4 Hz, 1H, Ar-H), 3.66 (s, 2H, CH_2), 2.35 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 169.53, 137.75, 137.37, 131.40, 129.91, 129.44, 128.92, 124.41, 119.89, 44.39, 21.13.

2-(4-(*tert*-Butyl)phenyl)-N-phenylacetamide (1c)



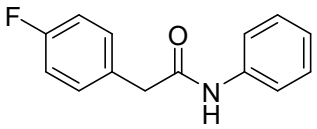
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.48-7.34 (m, 5H, Ar-H, NH), 7.26 (t, *J* = 7.7 Hz, 4H, Ar-H), 7.06 (t, *J* = 7.4 Hz, 1H, Ar-H), 3.67 (s, 2H, CH_2), 1.32 (s, 9H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm):169.56, 150.58, 137.80, 131.44, 129.22, 128.93, 126.13, 124.42, 120.00, 44.30, 34.57, 31.35.

2-(4-methoxyphenyl)-N-phenylacetamide (1d)



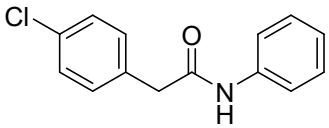
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.43-7.38 (m, 2H, Ar-H), 7.29 (d, *J* = 7.8 Hz, 2H, Ar-H), 7.25 (d, *J* = 6.6 Hz, 2H, Ar-H), 7.08 (t, *J* = 7.4 Hz, 2H, Ar-H), 6.97-6.90 (m, 2H, Ar-H, NH), 3.83 (s, 3H, OCH_3), 3.68 (s, 2H, CH_2). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 169.48, 130.68, 128.90, 126.26, 124.39, 119.73, 114.65, 55.30, 43.94.

2-(4-Fluorophenyl)-N-phenylacetamide (1e)



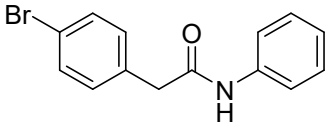
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.64 (s, 1H, NH), 7.42 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.31-7.20 (m, 4H, Ar-H), 7.05 (dt, *J* = 26.1, 8.0 Hz, 3H, Ar-H), 3.62 (s, 2H, CH_2). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 169.25, 163.43, 160.97, 137.66, 131.07, 130.99, 130.33, 128.97, 124.61, 120.10, 116.03, 115.81, 43.67.

2-(4-Chlorophenyl)-N-phenylacetamide (1f)



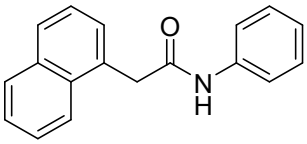
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.64 (s, 1H, NH), 7.42 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.31-7.20 (m, 4H, Ar-H), 7.05 (dt, *J* = 26.1, 8.0 Hz, 3H, Ar-H), 3.62 (s, 2H, CH_2). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 169.25, 163.43, 160.97, 137.66, 131.07, 130.99, 130.33, 128.97, 124.61, 120.10, 116.03, 115.81, 43.67.

2-(4-Bromophenyl)-N-phenylacetamide (1g)



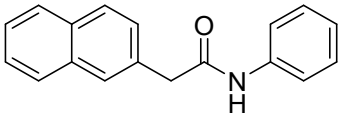
^{1}H NMR (400 MHz, DMSO) (δ, ppm): 10.17 (s, 1H, NH), 7.64-7.56 (m, 2H, Ar-H), 7.56-7.49 (m, 2H, Ar-H), 7.29 (dt, *J* = 8.0, 3.6 Hz, 4H, Ar-H), 7.04 (tt, *J* = 7.4, 1.3 Hz, 1H, Ar-H), 3.63 (s, 2H, CH_2). **^{13}C NMR** (101 MHz, DMSO) (δ, ppm): 169.05, 139.53, 135.83, 131.85, 131.57, 129.15, 123.70, 120.16, 119.53, 42.91.

2-(Naphthalen-1-yl)-N-phenylacetamide (1h)



^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.05-7.98 (m, 1H, Ar-H), 7.88 (ddd, *J* = 14.2, 6.8, 2.3 Hz, 2H, Ar-H), 7.58-7.45 (m, 4H, Ar-H), 7.32-7.27 (m, 2H, Ar-H), 7.25-7.18 (m, 2H, Ar-H), 7.13 (s, 1H, NH), 7.06-6.99 (m, 1H, Ar-H), 4.15 (s, 2H, CH_2). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 169.09, 137.47, 134.03, 132.04, 130.65, 128.88, 128.83, 128.48, 127.06, 126.35, 125.68, 124.44, 123.67, 119.93, 42.80.

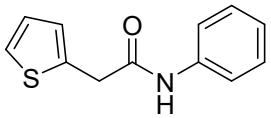
2-(Naphthalen-2-yl)-N-phenylacetamide (1i)



^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.93-7.72 (m, 4H, Ar-H, NH), 7.54-7.46 (m, 2H, Ar-H), 7.41

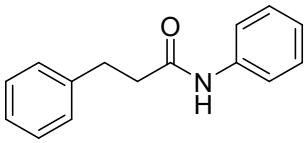
(t, J = 7.4 Hz, 3H, Ar-H), 7.32-7.19 (m, 3H, Ar-H), 7.06 (t, J = 7.4 Hz, 1H, Ar-H), 3.87 (s, 2H, CH_2). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 169.09, 137.59, 133.58, 132.63, 131.87, 129.04, 128.90, 128.42, 127.77, 127.69, 127.21, 126.55, 126.22, 124.47, 119.87, 44.97.

N-phenyl-2-(thiophen-2-yl)acetamide (1j)



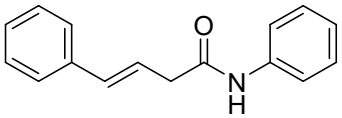
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.93 (s, 1H, NH), 7.45 (d, J = 7.9 Hz, 2H, Ar-H), 7.31-7.21 (m, 3H, Ar-H), 7.07 (t, J = 7.4 Hz, 1H, Ar-H), 6.99 (d, J = 4.7 Hz, 2H, Ar-H), 3.89 (s, 2H, CH_2). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 168.33, 137.59, 135.81, 128.92, 127.53, 127.41, 126.73, 125.77, 124.56, 120.12, 120.09, 38.36.

N,3-diphenylpropanamide (1k)



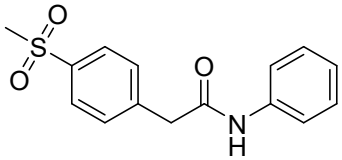
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.53-7.37 (m, 3H, Ar-H, NH), 7.32-7.15 (m, 7H, Ar-H), 7.07 (t, J = 7.4 Hz, 1H, Ar-H), 3.01 (t, J = 7.7 Hz, 2H, CH_2), 2.62 (t, J = 7.7 Hz, 2H, CH_2). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm):170.65, 140.62, 137.78, 128.92, 128.61, 128.37, 126.35, 124.30, 120.07, 39.32, 31.56.

(E)-N,4-diphenylbut-3-enamide (1l)



^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.15 (s, 1H, NH), 7.53 (d, J = 8.0 Hz, 2H, Ar-H), 7.25 (m, 7H, Ar-H), 7.06 (t, J = 7.4 Hz, 1H, Ar-H), 6.50 (d, J = 15.9 Hz, 1H, CH), 6.31 (dt, J = 15.2, 7.1 Hz, 1H, CH), 3.26 (d, J = 7.2 Hz, 2H, CH_2). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm):169.59, 137.99, 136.62, 134.77, 129.00, 128.68, 127.85, 126.41, 124.48, 122.23, 120.28, 41.75.

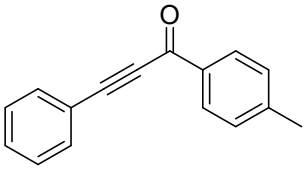
2-(4-(Methylsulfonyl)phenyl)-N-phenylacetamide (1s)



^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.89 (d, J = 8.4 Hz, 2H, Ar-H), 7.63-7.43 (m, 5H, Ar-H, NH), 7.30 (t, J = 7.9 Hz, 2H, Ar-H), 7.14-7.07 (m, 1H, Ar-H), 3.78 (s, 2H, CH_2), 3.05 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm):167.64, 140.98, 139.39, 137.45, 130.34, 129.02, 127.83, 124.75, 119.93, 44.51, 44.13.

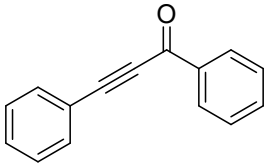
3.2 Characterization data of conjugated ynones

3-Phenyl-1-(p-tolyl)prop-2-yn-1-one (2a)



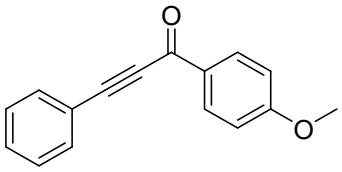
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.12 (d, *J* = 7.9 Hz, 2H, Ar-H), 7.73-7.64 (m, 2H, Ar-H), 7.52-7.38 (m, 3H, Ar-H), 7.31 (d, *J* = 7.9 Hz, 2H, Ar-H), 2.45 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 145.24, 134.68, 133.04, 130.68, 129.74, 129.37, 128.68, 92.61, 87.01, 21.85.

1,3-diphenylprop-2-yn-1-one (2b)



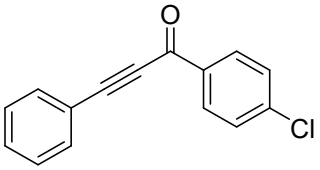
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.23 (d, *J* = 7.7 Hz, 2H, Ar-H), 7.69 (d, *J* = 7.5 Hz, 2H, Ar-H), 7.63 (t, *J* = 7.4 Hz, 1H, Ar-H), 7.55-7.39 (m, 6H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 178.00, 136.88, 134.11, 133.06, 130.79, 129.56, 128.69, 128.62, 120.12, 93.10, 86.89.

1-(4-methoxyphenyl)-3-phenylprop-2-yn-1-one (2c)



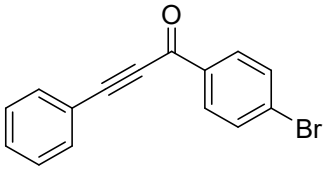
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.25-8.16 (m, 2H, Ar-H), 7.73-7.63 (m, 2H, Ar-H), 7.51-7.37 (m, 3H, Ar-H), 7.06-6.91 (m, 2H, Ar-H), 3.90 (s, 3H, OCH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 176.68, 164.52, 132.97, 132.00, 130.59, 130.36, 128.67, 120.40, 113.91, 92.31, 86.96, 55.61.

1-(4-Chlorophenyl)-3-phenylprop-2-yn-1-one (2d)



^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): δ 8.22-8.09 (m, 2H, Ar-H), 7.74-7.64 (m, 2H, Ar-H), 7.54-7.47 (m, 3H, Ar-H), 7.46-7.40 (m, 2H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 176.63, 140.69, 135.28, 133.08, 130.96, 130.84, 128.98, 128.72, 119.86, 93.62, 86.56.

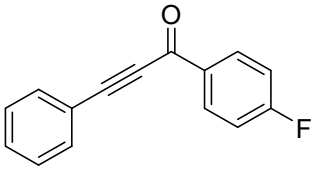
1-(4-Bromophenyl)-3-phenylprop-2-yn-1-one (2e)



^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.13-8.02 (m, 2H, Ar-H), 7.73-7.62 (m, 4H, Ar-H), 7.54-7.47 (m, 1H, Ar-H), 7.43 (dd, *J* = 8.2, 6.7 Hz, 2H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 176.86,

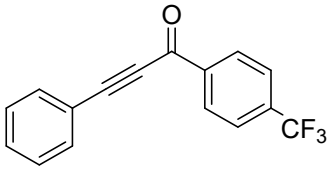
135.74, 133.13, 132.02, 131.01, 130.95, 129.58, 128.76, 119.91, 93.70, 86.59.

1-(4-Fluorophenyl)-3-phenylprop-2-yn-1-one (2f)



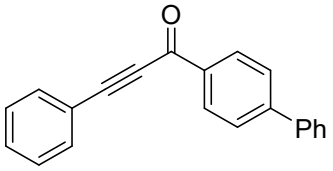
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.33-8.19 (m, 2H, Ar-H), 7.76-7.64 (m, 2H, Ar-H), 7.53-7.47 (m, 1H, Ar-H), 7.47-7.39 (m, 2H, Ar-H), 7.24-7.15 (m, 2H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 167.72, 133.04, 132.26, 132.17, 130.88, 128.70, 119.95, 115.85($^{2}J_{CF}$ = 21.9 Hz), 93.33, 86.57.

3-Phenyl-1-(4-(trifluoromethyl)phenyl)prop-2-yn-1-one (2g)



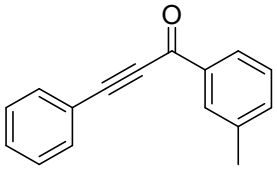
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.40-8.27 (m, 2H, Ar-H), 7.79 (d, J = 8.2 Hz, 2H, Ar-H), 7.74-7.65 (m, 2H, Ar-H), 7.57-7.49 (m, 1H, Ar-H), 7.48-7.40 (m, 2H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 176.74, 139.42, 133.21, 131.21, 129.82, 128.82, 125.73(q, $^{3}J_{CF}$ = 3.9 Hz), 119.70, 94.49, 86.60.

1-([1,1'-Biphenyl]-4-yl)-3-phenylprop-2-yn-1-one (2h)



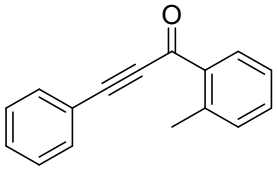
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.28 (d, J = 8.0 Hz, 2H, Ar-H), 7.76-7.60 (m, 6H, Ar-H), 7.52-7.36 (m, 6H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 177.57, 146.85, 139.77, 135.82, 133.13, 130.84, 130.21, 129.05, 128.75, 128.49, 127.38, 127.32, 120.23, 93.14, 87.09.

3-Phenyl-1-(m-tolyl)prop-2-yn-1-one (2i)



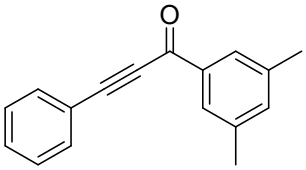
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.09-7.96 (m, 2H, Ar-H), 7.72-7.63 (m, 2H, Ar-H), 7.52-7.36 (m, 5H, Ar-H), 2.44 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 178.20, 138.49, 136.89, 134.99, 133.04, 130.75, 129.76, 128.68, 128.52, 127.11, 120.17, 92.88, 87.01, 21.32.

3-phenyl-1-(o-tolyl)prop-2-yn-1-one (2j)



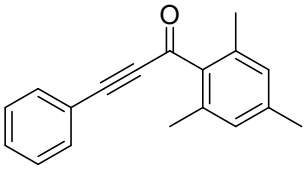
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.30 (dd, *J* = 7.8, 1.5 Hz, 1H, Ar-H), 7.68-7.61 (m, 2H, Ar-H), 7.51-7.32 (m, 5H, Ar-H), 7.29-7.23 (m, 1H, Ar-H), 2.67 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 179.75, 140.46, 135.70, 133.16, 132.90, 132.16, 130.58, 128.63, 125.88, 120.33, 91.80, 88.38, 21.93.

1-(3,5-dimethylphenyl)-3-phenylprop-2-yn-1-one (2k)



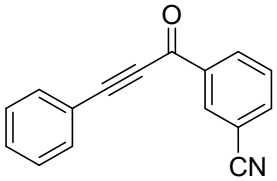
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.81 (s, 2H, Ar-H), 7.66 (dt, *J* = 6.9, 1.5 Hz, 2H, Ar-H), 7.50-7.36 (m, 3H, Ar-H), 7.23 (s, 1H, Ar-H), 2.39 (s, 6H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 178.35, 138.36, 137.02, 135.96, 133.02, 130.72, 128.70, 127.39, 120.31, 92.65, 87.19, 21.23.

1-mesityl-3-phenylprop-2-yn-1-one (2l)



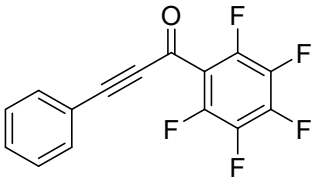
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.59-7.51 (m, 2H, Ar-H), 7.40 (d, *J* = 7.3 Hz, 1H, Ar-H), 7.34 (t, *J* = 7.5 Hz, 2H, Ar-H), 6.87 (s, 2H, Ar-H), 2.40 (s, 6H, CH_3), 2.28 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 184.21, 139.93, 137.47, 135.19, 133.15, 130.88, 129.15, 128.69, 120.17, 93.20, 89.69, 21.23, 19.84.

3-(3-phenylpropioloyl)benzonitrile (2m)



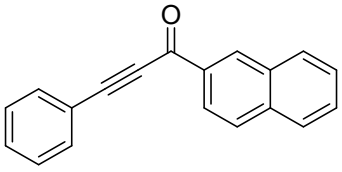
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.50 (s, 1H, Ar-H), 8.43 (d, *J* = 8.0 Hz, 1H, Ar-H), 7.92 (d, *J* = 7.7 Hz, 1H, Ar-H), 7.76-7.63 (m, 3H, Ar-H), 7.54 (t, *J* = 7.5 Hz, 1H, Ar-H), 7.46 (t, *J* = 7.5 Hz, 2H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 175.63, 137.61, 136.81, 133.29, 133.23, 133.14, 131.40, 129.74, 128.86, 119.42, 117.82, 113.30, 95.06, 86.17.

1-(perfluorophenyl)-3-phenylprop-2-yn-1-one (2n)



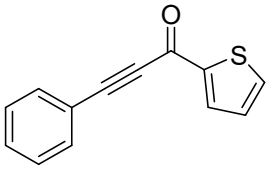
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.64 (d, *J* = 7.6 Hz, 2H, Ar-H), 7.52 (t, *J* = 7.5 Hz, 1H, Ar-H), 7.42 (t, *J* = 7.6 Hz, 2H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 167.79, 146.76, 144.19, 139.02, 136.48, 133.49, 131.71, 128.81, 119.10, 95.28, 88.67.

1-(Naphthalen-2-yl)-3-phenylprop-2-yn-1-one (2o)



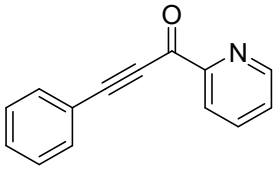
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.80 (d, *J* = 1.7 Hz, 1H, Ar-H), 8.22 (dd, *J* = 8.6, 1.8 Hz, 1H, Ar-H), 8.04 (d, *J* = 8.1 Hz, 1H, Ar-H), 7.93 (t, *J* = 9.0 Hz, 2H, Ar-H), 7.80-7.71 (m, 2H, Ar-H), 7.68-7.56 (m, 2H, Ar-H), 7.55-7.41 (m, 3H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 177.96, 136.20, 134.47, 133.11, 132.68, 132.46, 130.83, 129.93, 129.07, 128.76, 128.59, 127.97, 127.00, 124.01, 120.26, 93.10, 87.14.

3-Phenyl-1-(thiophen-2-yl)prop-2-yn-1-one (2p)



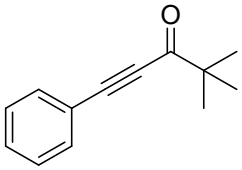
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.01 (dd, *J* = 3.8, 1.2 Hz, 1H, Ar-H), 7.73 (dd, *J* = 4.9, 1.1 Hz, 1H, Ar-H), 7.71-7.64 (m, 2H, Ar-H), 7.53-7.46 (m, 1H, Ar-H), 7.46-7.38 (m, 2H, Ar-H), 7.19 (dd, *J* = 4.9, 3.8 Hz, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 165.16, 135.23, 135.06, 133.06, 130.86, 128.72, 128.35, 119.99, 91.74, 86.52.

3-Phenyl-1-(pyridin-2-yl)prop-2-yn-1-one (2q)



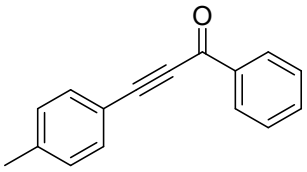
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.84 (t, *J* = 4.8 Hz, 1H, Ar-H), 8.19 (d, *J* = 7.6 Hz, 1H, Ar-H), 7.90 (d, *J* = 7.2 Hz, 1H, Ar-H), 7.73 (d, *J* = 7.4 Hz, 2H, Ar-H), 7.61-7.36 (m, 4H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 174.37, 149.95, 137.04, 133.40, 130.87, 128.58, 127.53, 123.38, 96.44, 87.94.

4,4-dimethyl-1-phenylpent-1-yn-3-one (2r)



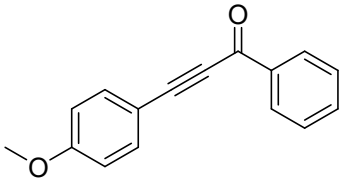
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.63-7.54 (m, 2H, Ar-H), 7.48-7.42 (m, 1H, Ar-H), 7.41-7.34 (m, 2H, Ar-H), 1.28 (s, 9H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 194.25, 132.92, 130.54, 128.58, 120.20, 92.20, 85.96, 44.84, 26.10.

1-Phenyl-3-(p-tolyl)prop-2-yn-1-one (2s)



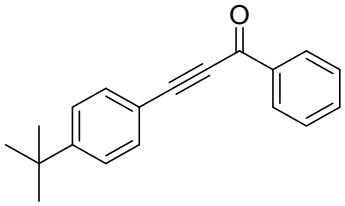
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.31-8.15 (m, 2H, Ar-H), 7.60 (dd, *J* = 11.6, 7.7 Hz, 3H, Ar-H), 7.51 (t, *J* = 7.6 Hz, 2H, Ar-H), 7.23 (d, *J* = 7.8 Hz, 2H, Ar-H), 2.41 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 178.09, 141.58, 137.03, 134.02, 133.15, 129.57, 129.51, 128.61, 117.06, 93.83, 86.81, 21.78.

3-(4-Methoxyphenyl)-1-phenylprop-2-yn-1-one (2t)



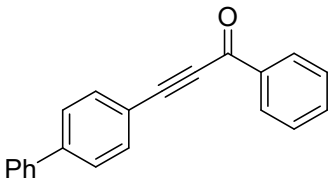
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.28-8.11 (m, 2H), 7.67-7.55 (m, 3H), 7.49 (dd, *J* = 8.4, 7.0 Hz, 2H), 6.97-6.80 (m, 2H), 3.82 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 177.99, 161.74, 137.01, 135.13, 133.91, 129.44, 128.56, 114.43, 111.80, 94.36, 86.88, 55.41.

3-(4-(tert-butyl)phenyl)-1-phenylprop-2-yn-1-one (2u)



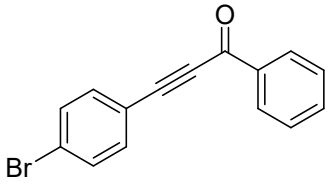
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.32-8.15 (m, 2H, Ar-H), 7.66-7.57 (m, 3H, Ar-H), 7.50 (dd, *J* = 8.4, 7.0 Hz, 2H, Ar-H), 7.46-7.40 (m, 2H, Ar-H), 1.33 (s, 9H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 178.05, 154.57, 136.99, 134.01, 132.99, 129.53, 128.59, 125.76, 117.01, 93.80, 86.75, 35.07, 31.04.

3-([1,1'-biphenyl]-4-yl)-1-phenylprop-2-yn-1-one (2v)



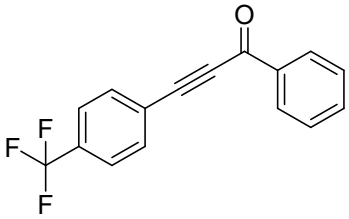
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.32-8.15 (m, 2H, Ar-H), 7.72 (d, *J* = 8.1 Hz, 2H, Ar-H), 7.65-7.54 (m, 5H, Ar-H), 7.50 (t, *J* = 7.6 Hz, 2H, Ar-H), 7.43 (t, *J* = 7.5 Hz, 2H, Ar-H), 7.36 (t, *J* = 7.3 Hz, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 178.00, 143.64, 139.76, 136.99, 134.18, 133.67, 129.63, 129.06, 128.71, 128.28, 127.37, 127.18, 118.86, 93.29, 87.72.

3-(4-Bromophenyl)-1-phenylprop-2-yn-1-one (2w)



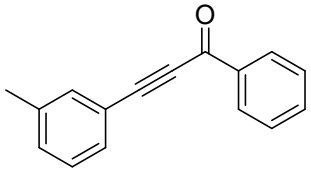
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.20 (d, *J* = 7.7 Hz, 2H, Ar-H), 7.64 (t, *J* = 7.4 Hz, 1H, Ar-H), 7.60-7.47 (m, 6H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 177.77, 136.69, 134.32, 134.25, 132.08, 129.55, 128.66, 125.58, 119.02, 91.61, 87.66.

1-Phenyl-3-(4-(trifluoromethyl)phenyl)prop-2-yn-1-one (2x)



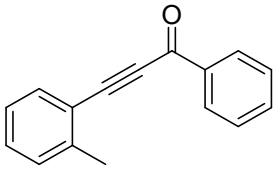
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.28-8.15 (m, 2H, Ar-H), 7.80 (d, *J* = 8.1 Hz, 2H, Ar-H), 7.67 (dd, *J* = 16.5, 7.9 Hz, 3H, Ar-H), 7.54 (t, *J* = 7.7 Hz, 2H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 177.64, 136.55, 134.44, 133.14, 132.40, 132.07, 129.61, 128.85, 128.72, 125.62 (q, $^3J_{CF}$ = 3.8 Hz), 124.88, 123.93, 90.43, 88.06.

1-Phenyl-3-(m-tolyl)prop-2-yn-1-one (2y)



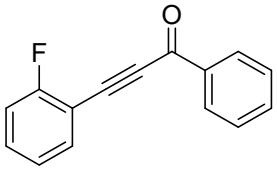
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.22 (dd, *J* = 8.3, 1.3 Hz, 2H, Ar-H), 7.69-7.59 (m, 1H, Ar-H), 7.55-7.45 (m, 4H, Ar-H), 7.29 (dd, *J* = 4.8, 2.6 Hz, 2H, Ar-H), 2.38 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 178.02, 138.51, 136.92, 134.06, 133.54, 131.75, 130.22, 129.55, 128.60, 128.58, 119.90, 93.50, 86.67, 21.17.

1-Phenyl-3-(o-tolyl)prop-2-yn-1-one (2z)



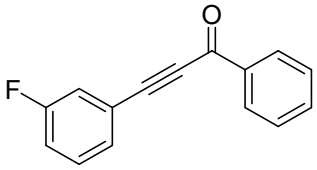
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.29-8.19 (m, 2H, Ar-H), 7.69-7.59 (m, 2H, Ar-H), 7.52 (dd, *J* = 8.4, 7.0 Hz, 2H, Ar-H), 7.36 (dd, *J* = 7.5, 1.4 Hz, 1H, Ar-H), 7.32-7.19 (m, 2H, Ar-H), 2.59 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 178.04, 142.15, 137.03, 134.02, 133.65, 130.80, 129.87, 129.52, 128.62, 125.93, 119.98, 92.17, 90.74, 20.87.

3-(2-Fluorophenyl)-1-phenylprop-2-yn-1-one (2aa)



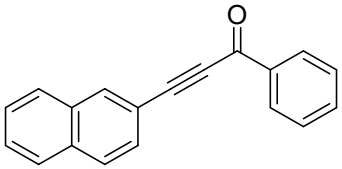
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): δ 8.27-8.14 (m, 2H, Ar-H), 7.69-7.61 (m, 1H, Ar-H), 7.57-7.45 (m, 3H, Ar-H), 7.44-7.33 (m, 2H, Ar-H), 7.20 (tdd, *J* = 8.4, 2.7, 1.1 Hz, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 177.75, 163.51, 136.66, 134.31, 130.45, 130.37, 129.58, 128.96, 128.68, 119.76, 118.11, 91.04, 87.09.

3-(3-Fluorophenyl)-1-phenylprop-2-yn-1-one (2ab)



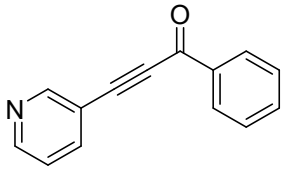
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.28-8.16 (m, 2H, Ar-H), 7.63 (q, *J* = 9.7, 8.5 Hz, 1H, Ar-H), 7.56-7.44 (m, 3H, Ar-H), 7.43-7.34 (m, 2H, Ar-H), 7.19 (td, *J* = 8.4, 2.7 Hz, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 177.77, 162.33 ($^1J_{CF}$ = 247.1 Hz), 136.72, 134.34, 130.46 ($^3J_{CF}$ = 8.7 Hz), 129.61, 128.95 ($^4J_{CF}$ = 3.2 Hz), 128.72, 119.67 ($^2J_{CF}$ = 21.2 Hz), 118.44 ($^2J_{CF}$ = 21.9 Hz), 91.11, 87.15.

3-(Naphthalen-2-yl)-1-phenylprop-2-yn-1-one (2ac)



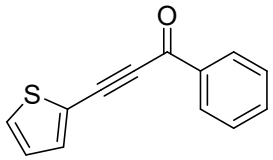
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.35-8.19 (m, 3H, Ar-H), 7.93-7.80 (m, 3H, Ar-H), 7.70-7.61 (m, 2H, Ar-H), 7.59-7.49 (m, 4H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 178.03, 137.00, 134.38, 134.16, 133.98, 132.73, 129.64, 128.68, 128.55, 128.44, 128.24, 128.07, 127.96, 127.10, 117.33, 93.68, 87.23.

1-Phenyl-3-(pyridin-3-yl)prop-2-yn-1-one (2ad)



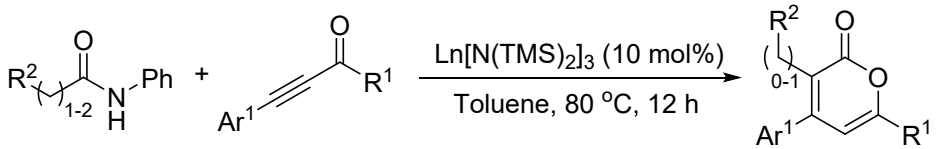
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.99-8.87 (m, 1H, Ar-H), 8.70 (d, *J* = 4.9 Hz, 1H, Ar-H), 8.22 (d, *J* = 7.7 Hz, 2H, Ar-H), 7.98 (d, *J* = 7.9 Hz, 1H, Ar-H), 7.66 (t, *J* = 7.4 Hz, 1H, Ar-H), 7.54 (t, *J* = 7.6 Hz, 2H, Ar-H), 7.39 (dd, *J* = 8.0, 5.0 Hz, 1H, Ar-H).**^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 177.53, 153.25, 150.76, 139.87, 136.48, 134.45, 129.59, 128.73, 123.30, 117.51, 89.36, 88.91.

1-Phenyl-3-(thiophen-2-yl)prop-2-yn-1-one (2ae)



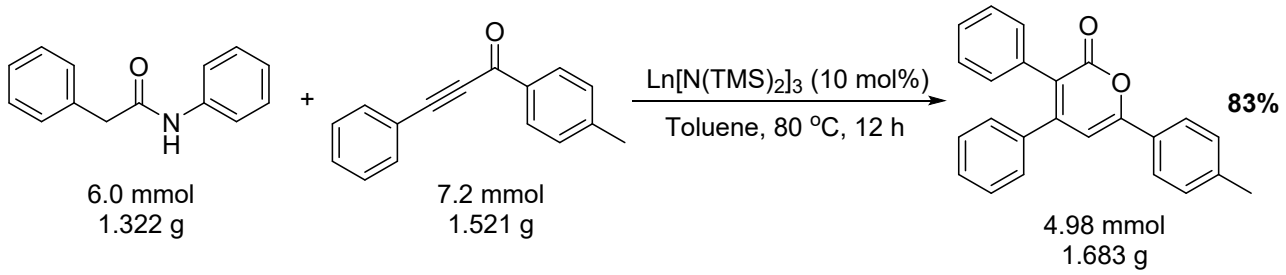
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.23-8.12 (m, 2H, Ar-H), 7.64-7.57 (m, 1H, Ar-H), 7.55 (dd, *J* = 3.8, 1.1 Hz, 1H, Ar-H), 7.53-7.45 (m, 3H, Ar-H), 7.07 (dd, *J* = 5.1, 3.7 Hz, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 177.47, 136.79, 134.14, 131.80, 129.43, 128.66, 127.84, 119.79, 91.69, 87.10.

3.3 General Procedure for the Synthesis of Trisubstituted 2-Pyrones



In a glovebox, $Y[N(TMS)_2]_3$ (11.4 mg, 0.02 mmol, 10 mol%) was added to a Schlenk tube equipped with a magnetic stirring bar and a Teflon cap. Then, a mixture of conjugated ynones (0.20 mmol) and phenylacetamides (0.24 mmol) in 2 mL toluene was added. The sealed tube was taken out from the glovebox, and stirred at 80 °C for 12 h. After completion of the reaction, the reaction mixture was quenched with water (2 mL), and extracted with ethyl acetate (3 × 5 mL). The organic layers were combined, and dried over anhydrous Na_2SO_4. The crude product was purified by flash column chromatography on silica gel with ethyl acetate/petroleum (10 : 1) as eluent.

4. Procedure of the Gram-Scale Reaction

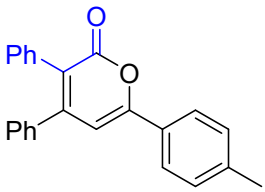


In a glovebox, a 50 mL Schlenk-type tube (with a Teflon screw cap) equipped with a magnetic stir bar was charged with $Y[N(TMS)_2]_3$ (0.60 mmol, 342.0 mg, 0.10 equiv), 3-phenyl-1-(p-tolyl)prop-2-yn-1-one (6.0 mmol, 1.322 g, 1.0 equiv), N,2-diphenylacetamide (7.2 mmol, 1.521 g, 1.2 equiv) and toluene (10 mL). The mixture was stirred at 80 °C (oil bath heating) for 12 h. After being cooled down to room

temperature, the reaction mixture was diluted with EtOAc (50 mL), washed with brine, dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (10 : 1) to afford **3a** (1.683 g, 83% yield) as a yellow solid.

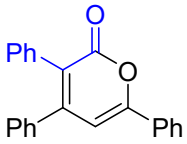
5. Characterization Data for Products

3,4-Diphenyl-6-(p-tolyl)-2H-pyran-2-one (3a)



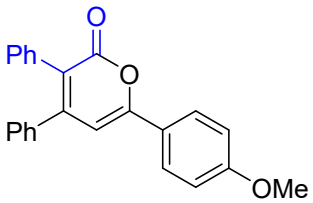
56.8 mg, 84% yield; yellow solid, mp 188-189 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.80 (d, *J* = 8.1 Hz, 2H, Ar-H), 7.27 (d, *J* = 8.2 Hz, 4H, Ar-H), 7.25-7.13 (m, 8H, Ar-H), 6.79 (s, 1H, Ar-H), 2.41 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.80, 158.53, 152.90, 141.24, 137.89, 133.90, 130.88, 129.67, 128.67, 128.60, 128.33, 127.93, 127.60, 125.53, 122.59, 104.33, 21.48.

3,4,6-Triphenyl-2H-pyran-2-one (3b)



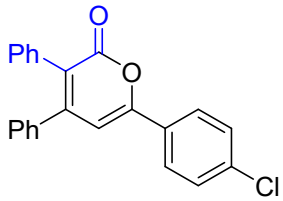
38.9 mg, 60% yield; yellow solid, mp 185-186 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.86 (d, *J* = 8.3 Hz, 2H, Ar-H), 7.46 (m, 3H, Ar-H), 7.26-7.15 (m, 10H, Ar-H), 6.84 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.68, 158.23, 152.74, 137.77, 133.83, 131.34, 130.89, 130.76, 128.96, 128.75, 128.70, 128.39, 127.97, 127.70, 125.58, 123.07, 105.00.

6-(4-Methoxyphenyl)-3,4-diphenyl-2H-pyran-2-one (3c)



48.1 mg, 68% yield; yellow solid, mp 180-181 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.87(d, *J* = 8.5 Hz, 2H, Ar-H), 7.27-7.21 (m, 10H, Ar-H), 6.98 (d, *J* = 8.5 Hz, 2H, Ar-H), 6.74 (s, 1H, Ar-H), 3.88 (s, 3H, OCH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.84, 161.71, 158.44, 153.10, 137.99, 133.97, 130.90, 128.65, 128.61, 128.31, 127.90, 127.52, 127.27, 123.90, 121.87, 114.35, 103.55, 55.44.

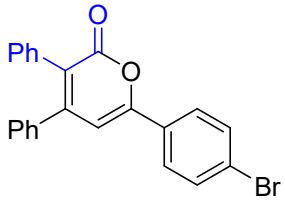
6-(4-Chlorophenyl)-3,4-diphenyl-2H-pyran-2-one (3d)



64.6 mg, 90% yield; yellow solid, mp 190-191 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.86(d, *J* = 8.3 Hz, 2H, Ar-H), 7.46 (d, *J* = 8.2 Hz, 2H, Ar-H), 7.30-7.28 (m, 2H, Ar-H), 7.25-7.18 (m, 8H, Ar-

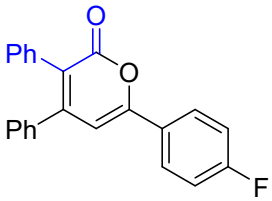
H), 6.84 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.39, 157.01, 152.58, 137.59, 136.82, 133.65, 130.83, 129.81, 129.24, 128.82, 128.67, 128.42, 127.99, 127.79, 126.82, 123.39, 105.19.

6-(4-bromophenyl)-3,4-diphenyl-2H-pyran-2-one (3e)



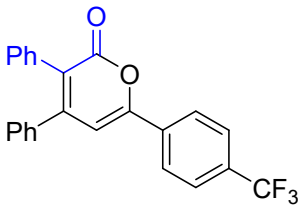
73.3 mg, 91% yield; yellow solid, mp 178-179 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.77 (d, *J* = 8.4 Hz, 2H, Ar-H), 7.61 (d, *J* = 8.4 Hz, 2H, Ar-H), 7.27-7.14 (m, 10H, Ar-H), 6.82 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.53, 157.27, 152.69, 137.77, 133.78, 132.37, 130.95, 130.43, 128.95, 128.79, 128.56, 128.14, 127.95, 127.14, 125.40, 123.71, 105.32.

6-(4-fluorophenyl)-3,4-diphenyl-2H-pyran-2-one (3f)



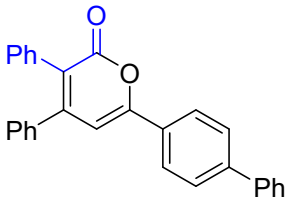
60.9 mg, 89% yield; yellow solid, mp 206-207 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.78-7.76(m, 2H, Ar-H), 7.62-7.60 (m, 2H, Ar-H), 7.27-7.14 (m, 10H, Ar-H), 6.82 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 165.49, 162.98, 162.54, 157.36, 152.74, 137.75, 133.72, 130.86, 128.79, 128.67, 128.41, 128.00, 127.79, 127.74($^{3}J_{CF}$ = 3.8 Hz), 123.05, 116.18($^{2}J_{CF}$ = 21.9 Hz), 104.73.

3,4-Diphenyl-6-(4-(trifluoromethyl)phenyl)-2H-pyran-2-one (3g)



51.7 mg, 66% yield; yellow solid, mp 182-184 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 8.01 (d, *J* = 8.4 Hz, 2H, Ar-H), 7.73 (d, *J* = 8.3 Hz, 2H, Ar-H), 7.32-7.13 (m, 10H, Ar-H), 6.92 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.21, 156.35, 152.29, 137.41, 134.57, 133.43, 132.19($^{2}J_{CF}$ = 35.7 Hz), 130.76, 128.91, 128.64, 128.46, 128.02, 127.93, 127.72($^{1}J_{CF}$ = 270.7 Hz), 125.94(q, $^{3}J_{CF}$ = 3.8 Hz), 125.79, 124.37, 106.33. **HRMS (ESI)** calcd for $C_{24}H_{16}F_3O_2^+$ $[M + H]^+$: 393.1097, found 393.1099.

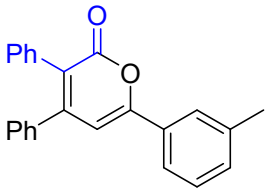
6-([1,1'-biphenyl]-4-yl)-3,4-diphenyl-2H-pyran-2-one (3h)



65.6 mg, 82% yield; yellow solid, mp 182-183 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.98 (d, *J*

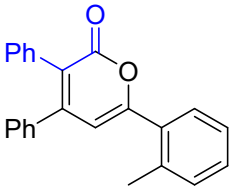
= 8.3 Hz, 2H, Ar-H), 7.71 (d, *J* = 8.2 Hz, 2H, Ar-H), 7.65 (d, *J* = 7.3 Hz, 2H, Ar-H), 7.47 (t, *J* = 7.5 Hz, 2H, Ar-H), 7.40 (d, *J* = 7.4 Hz, 1H, Ar-H), 7.23 (tt, *J* = 16.9, 3.6 Hz, 10H, Ar-H), 6.88 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.70, 158.08, 152.79, 143.48, 139.89, 137.88, 133.85, 130.91, 130.19, 128.97, 128.72, 128.41, 128.05, 127.99, 127.71, 127.57, 127.12, 126.06, 123.10, 104.94.

3,4-diphenyl-6-(o-tolyl)-2H-pyran-2-one (3i)



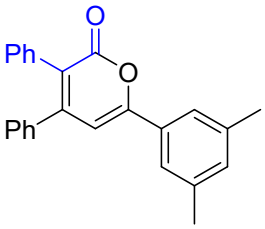
58.8 mg, 87% yield; yellow solid, mp 188-190 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.59 (d, *J* = 7.2 Hz, 1H, Ar-H), 7.41-7.23 (m, 11H, Ar-H), 7.21-7.13 (m, 2H, Ar-H), 6.54 (s, 1H, Ar-H), 2.60 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): δ 162.97, 160.36, 152.44, 137.65, 136.74, 133.78, 132.12, 131.31, 130.89, 130.25, 129.09, 128.74, 128.35, 127.97, 127.68, 126.10, 122.74, 108.93, 21.00. **HRMS (ESI)** calcd for $C_{24}H_{19}O_2^+$ $[M + H]^+$: 339.1380, found 339.1371.

3,4-Diphenyl-6-(o-tolyl)-2H-pyran-2-one (3j)



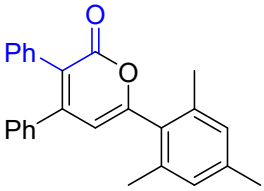
58.8 mg, 87% yield; yellow solid, mp 188-190 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.59 (d, *J* = 7.2 Hz, 1H, Ar-H), 7.41-7.23 (m, 11H, Ar-H), 7.21-7.13 (m, 2H, Ar-H), 6.54 (s, 1H, Ar-H), 2.60 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.97, 160.36, 152.44, 137.65, 136.74, 133.78, 132.12, 131.31, 130.89, 130.25, 129.09, 128.74, 128.35, 127.97, 127.68, 126.10, 122.74, 108.93, 21.00. **HRMS (ESI)** calcd for $C_{24}H_{19}O_2^+$ $[M + H]^+$: 339.1380, found 339.1361.

6-(3,5-Dimethylphenyl)-3,4-diphenyl-2H-pyran-2-one (3k)



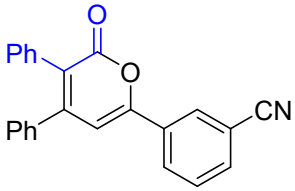
56.3 mg, 80% yield; yellow solid, mp 155-156 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.57 (s, 2H, Ar-H), 7.37-7.17 (m, 10H, Ar-H), 7.14 (s, 1H, Ar-H), 6.85 (s, 1H, Ar-H), 2.41 (s, 6H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.87, 158.73, 152.90, 138.64, 137.95, 133.94, 132.59, 131.27, 130.94, 128.72, 128.38, 127.98, 127.66, 126.42, 123.45, 122.83, 104.86, 21.33. **HRMS (ESI)** calcd for $C_{25}H_{21}O_2^+$ $[M + H]^+$: 353.1536, found 353.1521.

6-Mesityl-3,4-diphenyl-2H-pyran-2-one (3l)



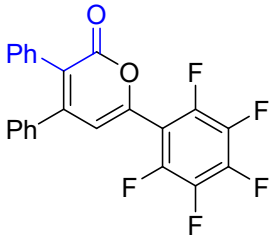
59.3 mg, 81% yield; yellow solid, mp 191-192 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.25-7.20 (m, 8H, Ar-H), 7.17-7.11 (m, 2H, Ar-H), 6.93 (s, 2H, Ar-H), 6.35 (s, 1H, Ar-H), 2.34 (s, 6H, CH_3), 2.31 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 163.64, 159.73, 152.10, 139.67, 137.49, 137.03, 133.81, 130.95, 129.73, 128.83, 128.77, 128.52, 128.31, 127.97, 127.67, 122.65, 110.26, 21.19, 20.21. **HRMS (ESI)** calcd for $C_{26}H_{23}O_2^+$ $[M + H]^+$: 367.1693, found 367.1702.

3-(2-Oxo-3,4-diphenyl-2H-pyran-6-yl)benzonitrile (3m)



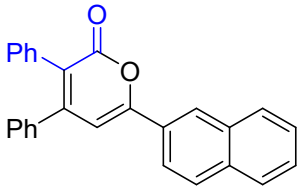
57.2 mg, 82% yield; yellow solid, mp 184-185 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 8.18 (t, J = 1.7 Hz, 1H, Ar-H), 8.14 (dt, J = 8.1, 1.5 Hz, 1H, Ar-H), 7.75 (dt, J = 7.8, 1.4 Hz, 1H, Ar-H), 7.62 (t, J = 7.9 Hz, 1H, Ar-H), 7.34-7.26 (m, 4H, Ar-H), 7.25-7.14 (m, 6H, Ar-H), 6.89 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 155.44, 152.18, 137.24, 133.59, 133.29, 132.63, 130.72, 129.94, 129.42, 128.99, 128.96, 128.61, 128.50, 128.05, 128.00, 124.53, 117.91, 113.53, 106.19. **HRMS (ESI)** calcd for $C_{24}H_{16}NO_2^+$ $[M + H]^+$: 350.1176, found 350.1186.

6-(Perfluorophenyl)-3,4-diphenyl-2H-pyran-2-one (3n)



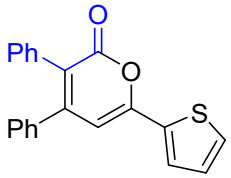
62.1 mg, 75% yield; yellow solid, mp 151-153 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.30-7.19 (m, 8H, Ar-H), 7.16-7.12 (m, 2H, Ar-H), 6.69 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 161.68, 151.24, 145.97, 136.69, 132.99, 130.67, 129.10, 128.69, 128.50, 128.17, 128.09, 125.56, 113.05(dd, $^4J_{CF}$ = 3.6 Hz). **HRMS (ESI)** calcd for $C_{23}H_{12}F_5O_2^+$ $[M + H]^+$: 415.0752, found 415.0757.

6-(Naphthalen-2-yl)-3,4-diphenyl-2H-pyran-2-one (3o)



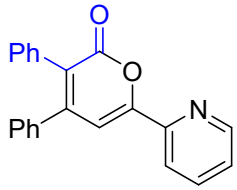
63.6 mg, 85% yield; yellow solid, mp 209-210 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 8.49 (s, 1H, Ar-H), 7.96-7.90 (m, 1H, Ar-H), 7.88-7.80 (m, 3H, Ar-H), 7.59-7.49 (m, 2H, Ar-H), 7.29-7.15 (m, 10H, Ar-H), 6.93 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.77, 158.19, 152.83, 137.89, 134.28, 133.88, 133.16, 130.94, 129.05, 128.79, 128.75, 128.44, 128.02, 127.78, 127.76, 127.68, 127.02, 126.10, 123.21, 122.03, 105.43.

3,4-Diphenyl-6-(thiophen-2-yl)-2H-pyran-2-one (3p)



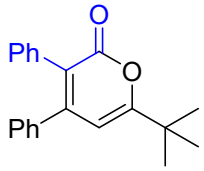
36.3 mg, 55% yield; yellow solid, mp 208-210 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.67 (d, *J* = 3.8 Hz, 1H, Ar-H), 7.46 (d, *J* = 5.0 Hz, 1H, Ar-H), 7.30-7.10 (m, 12H, Ar-H), 6.66 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): δ 162.03, 154.04, 152.81, 137.58, 135.11, 133.78, 130.85, 128.72, 128.69, 128.62, 128.36, 128.35, 127.93, 127.65, 127.36, 122.55, 104.07.

3,4-diphenyl-6-(pyridin-2-yl)-2H-pyran-2-one (3q)



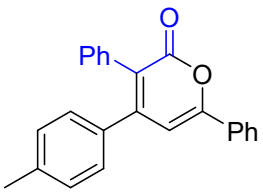
33.8 mg, 52% yield; yellow solid, mp 217-219 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 8.66 (ddd, *J* = 4.7, 1.8, 0.9 Hz, 1H, Ar-H), 8.11 (dt, *J* = 8.0, 1.1 Hz, 1H, Ar-H), 7.85 (td, *J* = 7.8, 1.8 Hz, 1H, Ar-H), 7.57 (s, 1H, Ar-H), 7.36 (ddd, *J* = 7.6, 4.7, 1.1 Hz, 2H, Ar-H), 7.26-7.18 (m, 9H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): δ 152.77, 149.78, 137.16, 130.80, 128.85, 128.74, 128.25, 127.99, 127.82, 124.78, 120.52, 106.72. **HRMS (ESI)** calcd for $C_{22}H_{16}NO_2^+$ $[M + H]^+$: 326.1176, found 326.1189.

6-(Tert-butyl)-3,4-diphenyl-2H-pyran-2-one (3r)



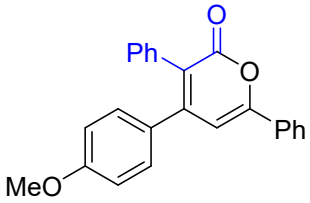
34.9 mg, 57% yield; white solid, mp 204-205 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.25-7.18 (m, 6H, Ar-H)), 7.15 (dt, *J* = 6.8, 2.5 Hz, 2H, Ar-H)), 7.12-7.07 (m, 2H, Ar-H)), 6.19 (s, 1H, Ar-H)), 1.35 (s, 9H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): δ 170.51, 163.30, 152.60, 138.02, 133.98, 130.82, 128.65, 128.51, 128.23, 127.89, 127.47, 122.05, 103.08, 36.09, 27.98.

3,6-Diphenyl-4-(p-tolyl)-2H-pyran-2-one (3s)



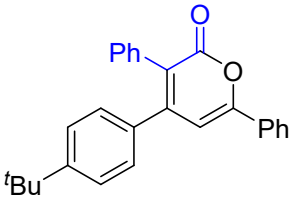
60.8 mg, 90% yield; yellow solid, mp 180−182 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.95-7.85 (m, 2H, Ar-H), 7.47 (dd, *J* = 5.1, 1.8 Hz, 3H, Ar-H), 7.28-7.18 (m, 5H, Ar-H), 7.06 (s, 4H, Ar-H), 6.83 (s, 1H, Ar-H), 2.31 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.78, 158.11, 152.66, 138.86, 134.78, 134.03, 131.43, 130.84, 130.65, 129.05, 128.91, 128.66, 127.97, 127.58, 125.56, 122.77, 105.05, 21.24. **HRMS (ESI)** calcd for $C_{24}H_{19}O_2^+$ $[M + H]^+$: 339.1380, found 339.1386.

4-(4-Methoxyphenyl)-3,6-diphenyl-2H-pyran-2-one (3t)



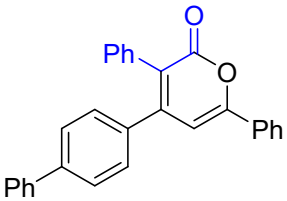
47.4 mg, 67% yield; yellow solid, mp 166−168 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.88 (td, *J* = 8.0, 3.5 Hz, 2H, Ar-H), 7.54-7.34 (m, 3H, Ar-H), 7.31-7.14 (m, 5H, Ar-H), 7.13-6.99 (m, 2H, Ar-H), 6.87-6.58 (m, 3H, Ar-H), 3.77 (s, 3H, OCH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.83, 159.94, 158.04, 152.22, 134.21, 131.46, 130.86, 130.65, 130.33, 129.79, 128.91, 128.06, 127.56, 125.56, 122.31, 113.77, 105.00, 55.24.

4-(4-(Tert-butyl)phenyl)-3,6-diphenyl-2H-pyran-2-one (3u)



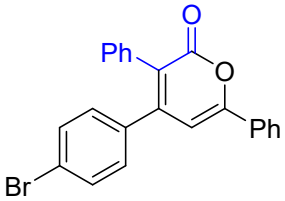
60.0 mg, 79% yield; yellow solid, mp 135−137 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.90 (dd, *J* = 6.7, 3.0 Hz, 2H, Ar-H), 7.46 (dd, *J* = 5.0, 1.9 Hz, 3H, Ar-H), 7.27-7.19 (m, 7H, Ar-H), 7.14-7.07 (m, 2H, Ar-H), 6.85 (s, 1H, Ar-H), 1.28 (s, 9H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.81, 158.05, 152.56, 152.06, 134.70, 134.03, 131.45, 130.88, 130.64, 128.90, 128.50, 127.93, 127.59, 125.55, 125.26, 122.77, 105.12, 34.66, 31.15. **HRMS (ESI)** calcd for $C_{27}H_{25}O_2^+$ $[M + H]^+$: 381.1849, found 339.1858.

4-([1,1'-Biphenyl]-4-yl)-3,6-diphenyl-2H-pyran-2-one (3v)



63.2 mg, 79% yield; yellow solid, mp 166−168 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.96-7.89 (m, 2H, Ar-H), 7.61-7.54 (m, 2H, Ar-H), 7.52-7.40 (m, 7H, Ar-H), 7.38-7.32 (m, 1H, Ar-H), 7.28-7.20 (m, 7H, Ar-H), 6.89 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.70, 158.30, 152.24, 141.45, 139.93, 136.59, 133.86, 131.38, 130.87, 130.74, 129.25, 128.94, 128.85, 128.07, 127.78, 127.74, 126.98, 126.94, 125.59, 123.03, 104.85. **HRMS (ESI)** calcd for $C_{29}H_{21}O_2^+$ $[M + H]^+$: 401.1536, found 401.1536.

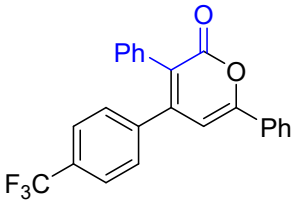
4-(4-bromophenyl)-3,6-diphenyl-2H-pyran-2-one (3w)



60.1 mg, 77% yield; yellow solid, mp 169-170 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.97-7.83 (m, 2H, Ar-H), 7.47 (dd, *J* = 5.5, 2.2 Hz, 3H, Ar-H), 7.42-7.35 (m, 2H, Ar-H), 7.30-7.22 (m, 3H, Ar-

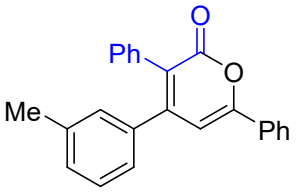
H), 7.18 (dd, J = 6.2, 2.7 Hz, 2H, Ar-H), 7.08-7.00 (m, 2H, Ar-H), 6.78 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.44, 158.57, 151.39, 136.64, 133.42, 131.63, 131.18, 130.88, 130.76, 130.30, 128.97, 128.16, 127.92, 125.59, 123.23, 123.13, 104.38. **HRMS (ESI)** calcd for $C_{23}H_{16}BrO_2^+$ $[M + H]^+$: 403.0328, found 403.0332.

3,6-Diphenyl-4-(4-(trifluoromethyl)phenyl)-2H-pyran-2-one (3x)



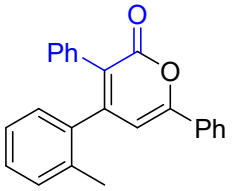
54.1 mg, 69% yield; yellow solid, mp 168-170 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.94-7.86 (m, 2H, Ar-H), 7.57-7.44 (m, 5H, Ar-H), 7.32-7.22 (m, 5H, Ar-H), 7.17 (dd, J = 6.7, 3.0 Hz, 2H, Ar-H), 6.79 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.27, 158.78, 151.10, 141.44, 133.11, 131.09, 130.99, 130.75, 129.07, 129.01, 128.17, 128.09, 125.41 (q, $^3J_{CF}$ = 3.8 Hz), 125.32, 123.76, 104.24. **HRMS (ESI)** calcd for $C_{24}H_{16}F_3O_2^+$ $[M + H]^+$: 392.1097, found 392.1103.

3,6-Diphenyl-4-(o-tolyl)-2H-pyran-2-one (3y)



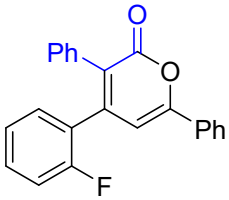
60.1 mg, 89% yield; yellow solid, mp 180-182 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.90 (dd, J = 6.3, 2.8 Hz, 2H, Ar-H), 7.54-7.40 (m, 3H, Ar-H), 7.28-7.17 (m, 5H, Ar-H), 7.14-7.04 (m, 2H, Ar-H), 7.00 (s, 1H, Ar-H), 6.96-6.88 (m, 1H, Ar-H), 6.83 (d, J = 1.3 Hz, 1H, Ar-H), 2.25 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.74, 158.14, 152.86, 138.10, 137.68, 133.91, 131.40, 130.81, 130.68, 129.45, 129.23, 128.92, 128.16, 127.90, 127.62, 125.88, 125.57, 123.01, 105.02, 21.32. **HRMS (ESI)** calcd for $C_{24}H_{19}O_2^+$ $[M + H]^+$: 339.1380, found 339.1388.

3,6-Diphenyl-4-(o-tolyl)-2H-pyran-2-one (3z)



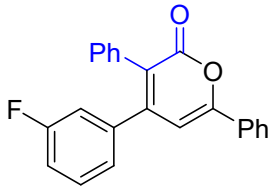
56.1 mg, 83% yield; yellow solid, mp 180−182 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.89 (dd, J = 6.7, 2.9 Hz, 2H, Ar-H), 7.47 (q, J = 2.9 Hz, 3H, Ar-H), 7.25-7.01 (m, 9H, Ar-H), 6.70 (s, 1H, Ar-H), 2.10 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 130.72, 130.34, 130.26, 128.92, 128.44, 127.66, 127.63, 125.79, 125.55, 105.31, 29.67. **HRMS (ESI)** calcd for $C_{24}H_{19}O_2^+$ $[M + H]^+$: 339.1380, found 339.1387.

4-(3-Fluorophenyl)-3,6-diphenyl-2H-pyran-2-one (3aa)



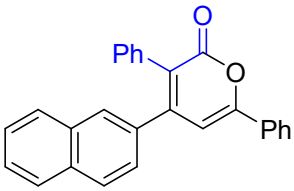
51.3 mg, 75% yield; yellow solid, mp 197-198 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.95-7.86 (m, 2H, Ar-H), 7.53-7.44 (m, 3H, Ar-H), 7.27-7.16 (m, 6H, Ar-H), 7.03-6.85 (m, 3H, Ar-H), 6.80 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.56 ($^1J_{CF}$ = 245.4Hz), 158.57, 151.28, 139.93, 133.32, 131.18, 130.88, 130.69, 130.03 ($^3J_{CF}$ = 8.4Hz), 128.97, 128.09, 127.97, 125.61, 124.49 ($^4J_{CF}$ = 3.1Hz), 123.50, 115.70 ($^2J_{CF}$ = 22.0Hz), 104.42. **HRMS (ESI)** calcd for $C_{23}H_{16}FO_2^+$ [M + H]$^+$: 343.1129, found 343.1145.

4-(3-Fluorophenyl)-3,6-diphenyl-2H-pyran-2-one (3ab)



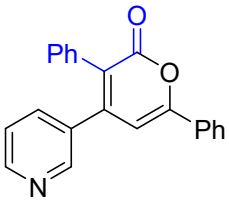
45.1 mg, 66% yield; yellow solid, mp 196−198 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.91 (dd, *J* = 6.7, 3.0 Hz, 2H, Ar-H), 7.58-7.42 (m, 3H, Ar-H), 7.32-7.13 (m, 6H, Ar-H), 7.01-6.86 (m, 3H, Ar-H), 6.80 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.49 ($^1J_{CF}$ = 246.3Hz), 158.62, 151.32, 133.37, 131.23, 130.93, 130.74, 130.07 ($^3J_{CF}$ = 8.4Hz), 129.01, 128.12, 128.01, 125.65, 124.29($^4J_{CF}$ = 2.8Hz), 115.84 ($^2J_{CF}$ = 40.1Hz), 115.62, 104.47. **HRMS (ESI)** calcd for $C_{23}H_{16}FO_2^+$ [M + H]$^+$: 343.1129, found 343.1134.

4-(Naphthalen-2-yl)-3,6-diphenyl-2H-pyran-2-one (3ac)



59.8 mg, 80% yield; yellow solid, mp 189−190 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): δ 7.93 (dd, *J* = 6.5, 2.9 Hz, 2H, Ar-H), 7.76 (d, *J* = 9.3 Hz, 3H, Ar-H), 7.63 (d, *J* = 8.5 Hz, 1H, Ar-H), 7.48 (dq, *J* = 7.7, 2.4 Hz, 5H, Ar-H), 7.21 (ddd, *J* = 14.4, 7.4, 3.7 Hz, 5H, Ar-H), 7.11 (dd, *J* = 8.6, 1.7 Hz, 1H, Ar-H), 6.94 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): δ 162.73, 158.29, 152.51, 135.32, 133.80, 132.94, 131.39, 130.93, 130.76, 128.96, 128.29, 128.05, 127.86, 127.73, 127.03, 126.58, 126.16, 125.62, 124.46, 123.21, 105.17. **HRMS (ESI)** calcd for $C_{27}H_{19}O_2^+$ [M + H]$^+$: 375.1380, found 375.1386.

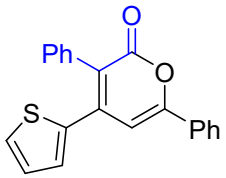
3,6-Diphenyl-4-(pyridin-3-yl)-2H-pyran-2-one (3ad)



39.3 mg, 60% yield; yellow solid, mp 194−196 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 8.59-8.48 (m, 2H, Ar-H), 7.94 (dd, *J* = 6.6, 3.1 Hz, 2H, Ar-H)), 7.57-7.48 (m, 3H, Ar-H), 7.45 (dt, *J* = 8.1, 2.0

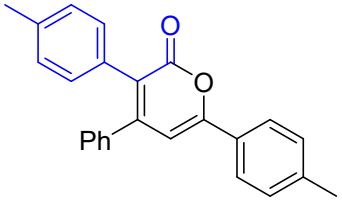
Hz, 1H, , Ar-H), 7.28 (d, J = 3.0 Hz, 3H, , Ar-H), 7.20 (dt, J = 7.8, 4.1 Hz, 3H, Ar-H), 6.84 (s, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 159.04, 149.76, 149.24, 136.12, 133.79, 133.03, 131.07, 130.87, 129.05, 128.29, 128.20, 125.69, 124.14, 123.04, 104.07. **HRMS (ESI)** calcd for $C_{22}H_{15}NO_2^+$ $[M + H]^+$: 326.1176, found 326.1174.

3,6-Diphenyl-4-(thiophen-2-yl)-2H-pyran-2-one (3ae)



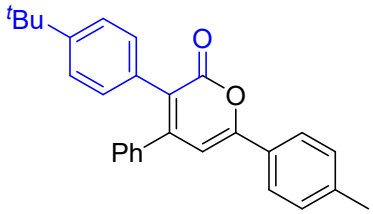
39.2 mg, 59% yield; yellow solid, mp 183−184 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.91 (dd, J = 6.6, 2.9 Hz, 2H, Ar-H), 7.48 (dd, J = 5.1, 2.0 Hz, 3H, Ar-H), 7.40 (dt, J = 4.9, 2.5 Hz, 3H, Ar-H), 7.35 (d, J = 5.1 Hz, 1H, Ar-H), 7.33-7.28 (m, 2H, Ar-H), 7.06 (d, J = 3.8 Hz, 1H, Ar-H), 7.00 (s, 1H, Ar-H), 6.94 (dd, J = 5.1, 3.8 Hz, 1H, Ar-H). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 158.11, 144.48, 134.34, 131.48, 130.80, 130.59, 130.28, 130.00, 128.97, 128.92, 128.59, 127.29, 125.68, 103.15. **HRMS (ESI)** calcd for $C_{21}H_{14}O_2S^+$ $[M + H]^+$: 331.0788, found 331.0786.

4-Phenyl-3,6-di-p-tolyl-2H-pyran-2-one (3af)



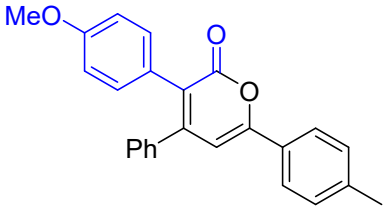
59.8 mg, 85% yield; yellow solid, mp 212−214 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.90-7.68 (m, 2H, Ar-H), 7.27 (dt, J = 9.1, 2.8 Hz, 5H, Ar-H), 7.17 (dt, J = 7.2, 2.1 Hz, 2H, Ar-H), 7.10-6.98 (m, 4H, Ar-H), 6.78 (d, J = 3.2 Hz, 1H, Ar-H), 2.41 (s, 3H, CH_3), 2.29 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.96, 158.26, 152.47, 141.12, 138.08, 137.36, 130.81, 130.69, 129.63, 128.69, 128.65, 128.53, 128.32, 125.48, 104.35, 21.45, 21.26. **HRMS (ESI)** calcd for $C_{25}H_{21}O_2^+$ $[M + H]^+$: 353.1536, found 353.1547.

3-(4-(Tert-butyl)phenyl)-4-phenyl-6-(p-tolyl)-2H-pyran-2-one (3ag)



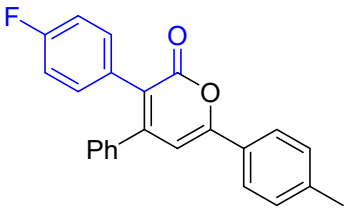
50.1 mg, 64% yield; yellow solid, mp 165−166 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.88-7.72 (m, 2H, Ar-H), 7.32-7.21 (m, 8H, Ar-H), 7.19-7.14 (m, 2H, Ar-H), 7.14-7.08 (m, 2H, Ar-H), 6.79 (s, 1H, Ar-H), 2.41 (s, 3H, CH_3), 1.27 (s, 9H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.98, 158.23, 152.51, 150.46, 141.09, 138.10, 130.69, 130.44, 129.62, 128.69, 128.52, 128.23, 125.48, 124.83, 104.42, 31.22, 21.45. **HRMS (ESI)** calcd for $C_{28}H_{27}O_2^+$ $[M + H]^+$: 395.2006, found 395.2028.

3-(4-Methoxyphenyl)-4-phenyl-6-(p-tolyl)-2H-pyran-2-one (3ah)



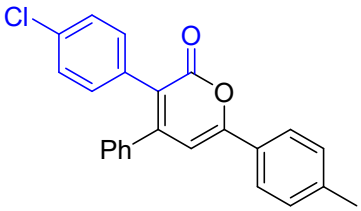
53.0 mg, 72% yield; yellow solid, mp 209−212 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.79 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.30-7.25 (m, 5H, Ar-H), 7.18 (dt, *J* = 7.4, 3.7 Hz, 2H, Ar-H), 7.12 (d, *J* = 8.4 Hz, 2H, Ar-H), 6.81-6.72 (m, 3H, Ar-H), 3.76 (s, 3H, CH_3), 2.41 (s, 3H, OCH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 163.06, 158.97, 158.16, 152.28, 141.12, 138.22, 132.17, 129.67, 128.70, 128.54, 128.41, 126.07, 125.50, 122.34, 113.50, 104.43, 55.17, 21.48. **HRMS (ESI)** calcd for $C_{25}H_{21}O_3^+$ [M + H]$^+$: 368.1485, found 368.1499.

3-(4-Fluorophenyl)-4-phenyl-6-(p-tolyl)-2H-pyran-2-one (3ai)



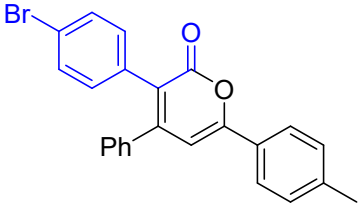
56.2 mg, 79% yield; yellow solid, mp 204-206 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.80 (d, *J* = 8.0 Hz, 1H, Ar-H), 7.33-7.24 (m, 3H, Ar-H), 7.20-7.12 (m, 2H, Ar-H), 6.92 (t, *J* = 8.6 Hz, 1H, Ar-H), 2.41 (s, 1H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.12($^1J_{CF}$ = 246.3Hz), 158.73, 153.15, 141.40, 137.79, 132.73 ($^3J_{CF}$ = 8.0Hz), 129.72, 128.81, 128.63, 128.55, 128.51, 125.58, 121.57, 115.05 ($^2J_{CF}$ = 21.5Hz), 104.30, 21.50. **HRMS (ESI)** calcd for $C_{24}H_{18}FO_2^+$ [M + H]$^+$: 357.1286, found 357.1294.

3-(4-Chlorophenyl)-4-phenyl-6-(p-tolyl)-2H-pyran-2-one (3aj)



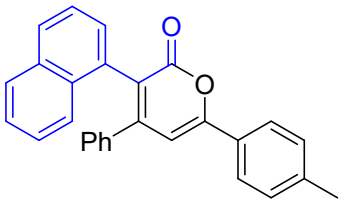
49.2 mg, 66% yield; yellow solid, mp 245-247 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.79 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.28 (dq, *J* = 9.4, 4.4, 4.0 Hz, 5H, Ar-H), 7.22-7.09 (m, 6H, Ar-H), 6.79 (s, 1H, Ar-H), 2.41 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.55, 158.89, 153.31, 141.48, 137.65, 133.58, 132.42, 132.33, 129.74, 128.91, 128.62, 128.58, 128.50, 128.24, 125.61, 121.32, 104.31, 21.50. **HRMS (ESI)** calcd for $C_{24}H_{17}ClO_2^+$ [M + H]$^+$: 373.0990, found 373.0998.

3-(4-Bromophenyl)-4-phenyl-6-(p-tolyl)-2H-pyran-2-one (3ak)



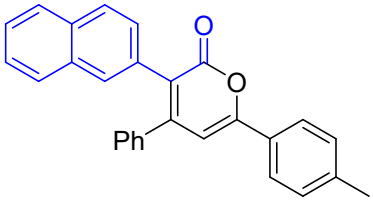
67.5 mg, 81% yield; yellow solid, mp 237-239 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.88-7.71 (m, 2H, Ar-H), 7.40-7.33 (m, 2H, Ar-H), 7.32-7.24 (m, 5H, Ar-H), 7.19-7.13 (m, 2H, Ar-H), 7.11-7.02 (m, 2H, Ar-H), 6.79 (s, 1H, Ar-H), 2.42 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 158.87, 153.26, 141.47, 137.57, 132.84, 132.58, 131.15, 129.70, 128.89, 128.57, 128.56, 128.43, 125.57, 121.84, 104.28, 21.48. **HRMS (ESI)** calcd for $C_{24}H_{17}BrO_2^+$ $[M + H]^+$: 417.0485, found 417.0491.

3-(Naphthalen-1-yl)-4-phenyl-6-(p-tolyl)-2H-pyran-2-one (3al)



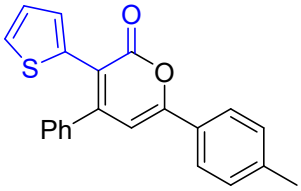
45.0 mg, 58% yield; yellow solid, mp 178-179 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.90-7.72 (m, 5H, Ar-H), 7.49-7.40 (m, 2H, Ar-H), 7.35-7.27 (m, 3H, Ar-H), 7.21-7.03 (m, 6H, Ar-H), 6.90 (s, 1H, Ar-H), 2.43 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.49, 159.27, 154.81, 141.37, 137.64, 133.49, 132.34, 132.09, 129.71, 128.85, 128.75, 128.64, 128.56, 128.51, 128.14, 127.81, 126.32, 125.79, 125.63, 125.32, 125.27, 121.67, 103.86, 21.50.

3-(Naphthalen-2-yl)-4-phenyl-6-(p-tolyl)-2H-pyran-2-one (3am)



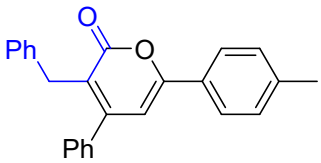
42.7 mg, 55% yield; yellow solid, mp 181-183 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.87-7.79 (m, 2H, Ar-H), 7.79-7.73 (m, 2H, Ar-H), 7.68 (dd, J = 11.9, 7.8 Hz, 2H, Ar-H), 7.42 (ddd, J = 7.2, 4.9, 1.8 Hz, 2H, Ar-H), 7.29 (d, J = 8.0 Hz, 2H, Ar-H), 7.25-7.17 (m, 6H, Ar-H), 6.84 (s, 1H, Ar-H), 2.42 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 158.63, 153.11, 141.28, 137.87, 133.01, 132.60, 131.35, 130.49, 129.68, 128.69, 128.61, 128.47, 128.43, 128.20, 127.52, 127.33, 126.18, 125.80, 125.55, 122.40, 104.42, 21.47. **HRMS (ESI)** calcd for $C_{28}H_{21}O_2^+$ $[M + H]^+$: 389.1536, found 389.1544.

4-Phenyl-3-(thiophen-2-yl)-6-(p-tolyl)-2H-pyran-2-one (3an)



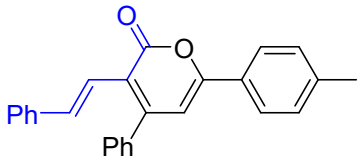
44.0 mg, 64% yield; yellow solid, mp 251−253 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.83-7.72 (m, 2H, Ar-H), 7.37 (dd, J = 5.0, 1.9 Hz, 3H, Ar-H), 7.32-7.21 (m, 5H, Ar-H), 6.92 (dd, J = 3.7, 1.2 Hz, 1H, Ar-H), 6.84 (dd, J = 5.1, 3.7 Hz, 1H, Ar-H), 6.73 (s, 1H, Ar-H), 2.40 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 161.88, 157.73, 152.49, 141.38, 138.51, 134.65, 130.15, 129.70, 128.96, 128.78, 128.32, 128.14, 127.25, 126.22, 125.51, 116.17, 104.72, 21.49. **HRMS (ESI)** calcd for $C_{22}H_{17}O_2S^+$ $[M + H]^+$: 345.0944, found 345.0947.

3-Benzyl-4-phenyl-6-(p-tolyl)-2H-pyran-2-one (3ao)



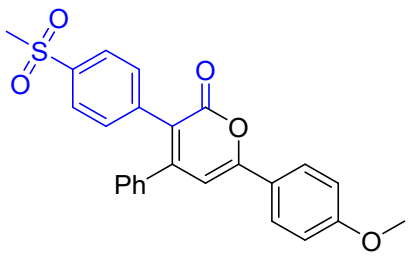
43.6 mg, 62% yield; yellow solid, mp 165−167 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.77-7.66 (m, 2H, Ar-H), 7.44 (dd, *J* = 4.9, 2.0 Hz, 3H, Ar-H), 7.34-7.28 (m, 2H, Ar-H), 7.25-7.06 (m, 7H, Ar-H), 6.62 (s, 1H, Ar-H), 3.87 (s, 2H, CH_2), 2.39 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 163.49, 157.52, 153.83, 140.97, 139.63, 137.92, 129.60, 128.90, 128.72, 128.63, 128.32, 127.64, 126.08, 125.34, 121.85, 104.03, 33.07, 21.42. **HRMS (ESI)** calcd for $C_{25}H_{21}O_2^+$ $[M + H]^+$: 353.1536, found 353.1545.

(E)-4-Phenyl-3-styryl-6-(p-tolyl)-2H-pyran-2-one (3ap)



36.4 mg, 50% yield; orange solid, mp 163-165 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 8.00 (d, *J* = 16.2 Hz, 1H, Ar-H), 7.78 (d, *J* = 7.9 Hz, 2H, Ar-H), 7.57-7.41 (m, 5H, Ar-H), 7.33 (d, *J* = 7.6 Hz, 2H, Ar-H), 7.26 (d, *J* = 6.9 Hz, 4H, Ar-H), 7.21 (d, *J* = 7.1 Hz, 1H, CH), 6.90 (d, *J* = 16.1 Hz, 1H, CH), 6.72 (s, 1H, Ar-H), 2.41 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.00, 158.20, 152.63, 141.18, 138.10, 137.95, 134.30, 129.70, 129.14, 128.76, 128.64, 128.57, 127.76, 126.82, 125.47, 121.44, 104.71, 21.49. **HRMS (ESI)** calcd for $C_{25}H_{21}O_2^+$ $[M + H]^+$: 365.1536, found 365.1512.

6-(4-Methoxyphenyl)-3-(4-(methylsulfonyl)phenyl)-4-phenyl-2H-pyran-2-one (5)



50.1 mg, 58% yield; yellow solid, mp 226-227 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) (δ, ppm): 7.92-7.84 (m, 2H, Ar-H), 7.82-7.76 (m, 2H, Ar-H), 7.45-7.36 (m, 2H, Ar-H), 7.35-7.26 (m, 3H, Ar-H), 7.13 (dt, *J* = 6.8, 1.6 Hz, 2H, Ar-H), 7.04-6.96 (m, 2H, Ar-H), 6.76 (s, 1H, Ar-H), 3.89 (s, 3H, CH_3), 3.02 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) (δ, ppm): 162.23, 162.12, 159.59, 154.76, 140.18, 139.04, 137.07, 132.02, 129.31, 128.74, 128.49, 127.52, 126.98, 123.44, 119.56, 114.47, 103.51, 55.48, 44.50. **HRMS (ESI)** calcd for $C_{25}H_{21}O_5S^+$ $[M + H]^+$: 433.1104, found 433.1109.

6. Synthesis and Structural Characterization of Y-2

A mixture of **Y-1** (235 mg, 0.50 mmol) and **1a** (317 mg, 1.5 mmol) in 5 mL toluene was stirred at room temperature for 12 h. Slow evaporation of solvent afforded **Y-2** as pale yellow crystals (143 mg, 35%). X-ray diffraction data for compound **Y-2** was collected on a SMART APEX CCD diffractometer (graphite-monochromated Mo*K*α radiation, ϕ-ω scan technique, λ = 0.71073 Å). The intensity data were integrated by means of the SAINT program. SADABS was used to perform area-detector scaling and absorption corrections. The structure was solved by direct methods and was refined against F^2 using all reflections with the aid of the SHELXTL package. All non-hydrogen atoms were found from the difference Fourier syntheses and refined anisotropically. The H atoms were included in calculated positions with isotropic thermal parameters related to those of the supporting carbon atoms but were not included in the refinement. All calculations were performed using the Bruker Smart program. CCDC 2225598 (**Y-2**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (+44)-1223-336033; or deposit@ccdc.cam.ac.uk).

Table S1. Crystal and Data Collection Parameters of Complex **Y-2**.

Identification code	ga_210320b_a
Empirical formula	$C_{140}N_{10}O_{10}Y_3$
Formula weight	2248.23
Temperature/K	173.0
Crystal system	triclinic
Space group	P-1
a/Å	14.227(2)
b/Å	18.221(4)
c/Å	26.440(5)
α/°	98.237(15)
β/°	92.972(15)
γ/°	106.495(13)
Volume/Å^3	6473(2)
Z	2
ρ_{calc}g/cm^3	1.153
μ/mm^{-1}	1.485
F(000)	2214.0
Crystal size/mm^3	0.18 × 0.12 × 0.06
Radiation	GaKα (λ = 1.34138)
2θ range for data collection/°	6.79 to 114.876
Index ranges	-17 ≤ h ≤ 17, -22 ≤ k ≤ 22, -33 ≤ l ≤ 33
Reflections collected	238625
Independent reflections	26621 [R_{int} = 0.0863, R_{sigma} = 0.0396]
Data/restraints/parameters	26621/3243/1456
Goodness-of-fit on F^2	1.053
Final R indexes [I>=2σ (I)]	R_1 = 0.0630, wR_2 = 0.1902

Final R indexes [all data]	R_1 = 0.0714, wR_2 = 0.2002
Largest diff. peak/hole / e Å^{-3}	1.38/-1.13

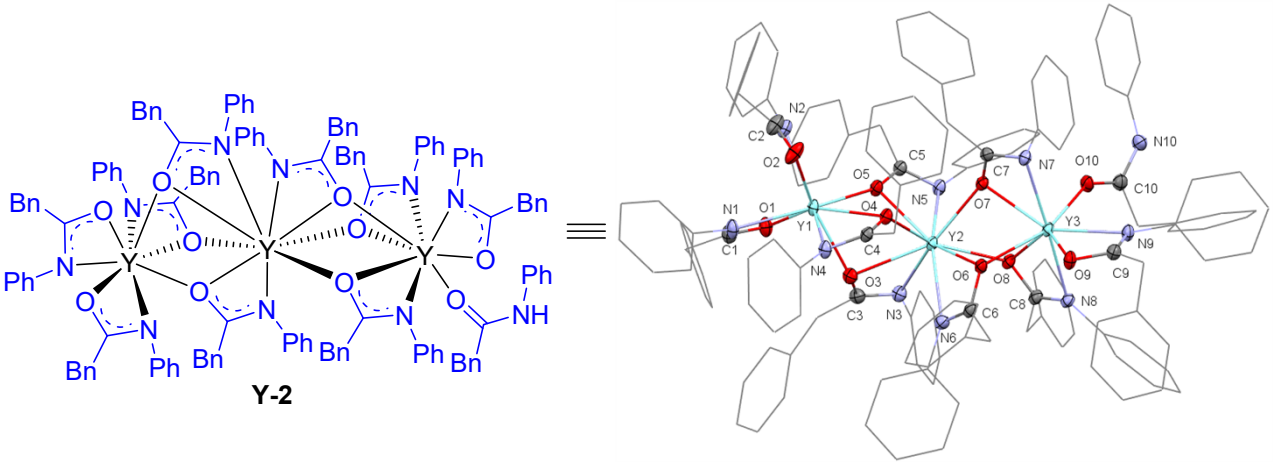


Figure S1. Molecular structure of complex **Y-2**. Molecular structures of **Y-2** with thermal ellipsoids at 30% probability. All hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (deg): Y1–O3 2.349(3), Y2–O3 2.553(3), Y1–O4 2.444(2), Y2–O4 2.322(2), Y1–O5 2.298(2), Y2–O5 2.384(2), Y2–O5 2.384(2), Y2–O6 2.501(2), Y3–O6 2.295(2), Y2–O7 2.379(2), Y3–O7 2.393(2), Y2–O8 2.377(2), Y3–O8 2.459(3), Y1–N1 2.434(3), Y1–N2 2.469(3), Y1–N4 2.475(3), Y2–N3 2.469(3), Y2–N5 2.559(3), Y2–N6 2.494(3), Y3–N7 2.428(3), Y3–N8 2.438(3), Y3–N9 2.457(3),Y1–C1 2.786(4), Y1–C2 2.742(4), Y1–C4 2.856(4), Y2–C3 2.939(4), Y2–C5 2.896(3), Y2–C6 2.930(4), Y3–C7 2.825(3), Y1–O3–Y2 95.5(1), Y2–O4–Y1 99.2(1), Y1–O5–Y2 101.7(1), Y3–O6–Y2 98.2(1), Y2–O7–Y3 98.9(1), Y2–O8–Y3 97.1(1).

7. ^{1}H NMR monitoring the reaction of Y-2 and 1a

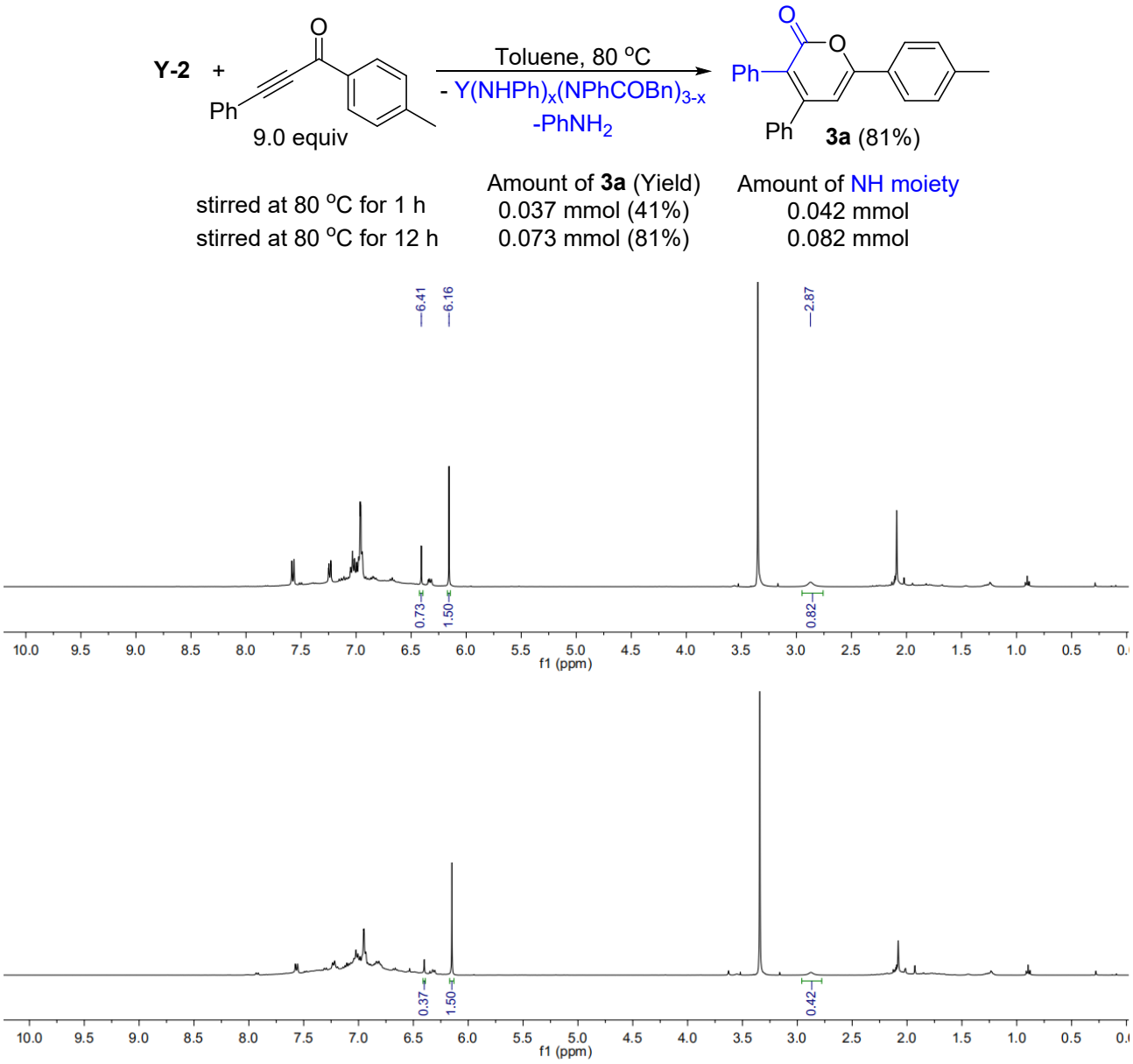
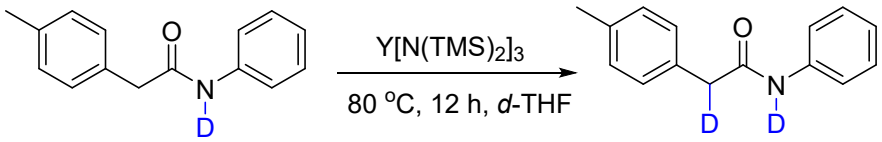


Figure S2. ^{1}H NMR monitoring spectra for the progress of the reaction of ynone **1a** with **Y-2**. In a glovebox, **Y-2** (0.010 mmol, 24.4 mg), **1a** (0.090 mmol, 19.8 mg), 1,3,5-trimethoxybenzene (0.050 mmol, 8.4 mg) and toluene-d_8 (1.0 mL) were added to a NMR tube equipped with a Teflon valve (J-Young). Subsequently, the reaction mixture was heated at 80 °C. The reaction was monitored by ^{1}H NMR technique, the yield of **3a** was determined by the integration of its ring C(sp^2)–H resonance absorption. The results indicate that the molar amount of product **3a** is slightly lower than that of the resulting NH moiety (2.87 ppm) during reaction proceeding, while the NH peak (7.92 ppm) of coordinated neutral amide decreases gradually. Evidently, with the formation of compound **3a**, the yttrium amido complex [$(PhNH)_xY(N(Ph)COBn)_{3-x}$] and small amount of aniline were also produced, although we have not determined their composition ratio accurately. With the increase of **1a** used, the metal-containing byproduct released will become $(PhNH)_3Y$ when the reaction is complete.

8. Deuterium labeling experiment



Integration of N-H		Integration of C-H	
1b' at rt	0.01	**1b'** at rt	2.00
stirred at rt for 15 min	0.12	stirred at rt for 15 min	1.74
stirred at 80 °C for 12 h	0.34	stirred at 80 °C for 12 h	1.23

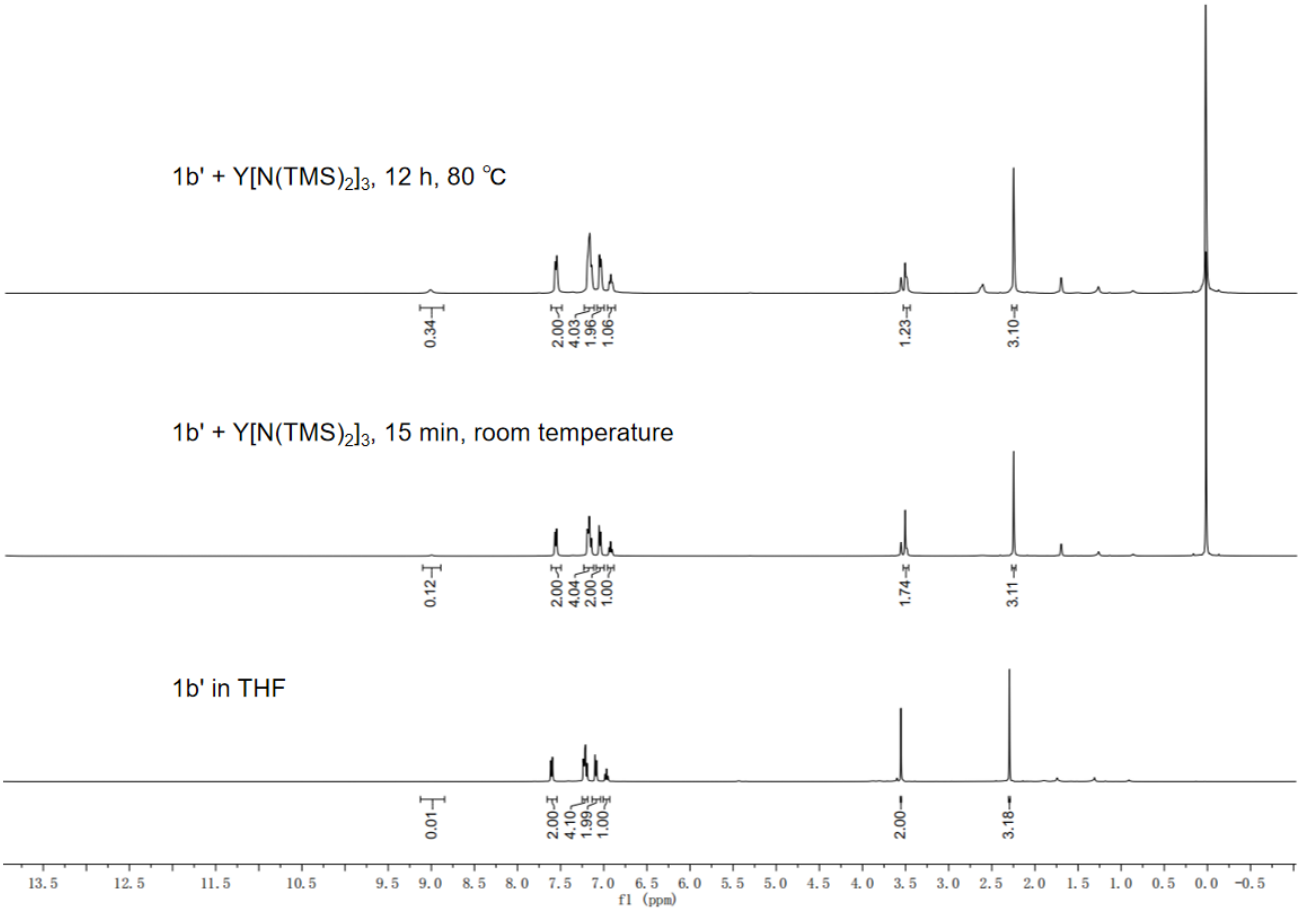


Figure S3. ^{1}H NMR monitoring spectra for the progress of the reaction of N-deuterated **1b'** with Y[(N(TMS)$_2$]$_3$. In a glovebox, a solution of **1b'** (22.6 mg, 0.010 mmol) in 0.4 mL THF-d_8 was injected into a NMR tube equipped with a Teflon valve (J-Young). After the original ^{1}H NMR determination, Y[(N(TMS)$_2$]$_3$ (10 mol%) was added to the NMR tube, the reaction mixture was then heated at 80 °C. The ^{1}H NMR monitoring data (Figure S2) reveal that addition of 10 mol% **Y-1** to a solution of **1b'** in THF-d_8 at room temperature led to the complete liberation of $(Me_3Si)_2N$ ligand from the precatalyst rapidly, giving the free $(Me_3Si)_2NH$, and **Y-1** was cleanly converted into yttrium amido complex within 15 minutes. With the extension of reaction time, the deuterium on nitrogen atom decreases continuously, while the deuterium on the α-carbon increases continuously, featuring the occurrence of the tautomerization of yttrium amido complex and yttrium α-amino enolate complex.

9. Reference

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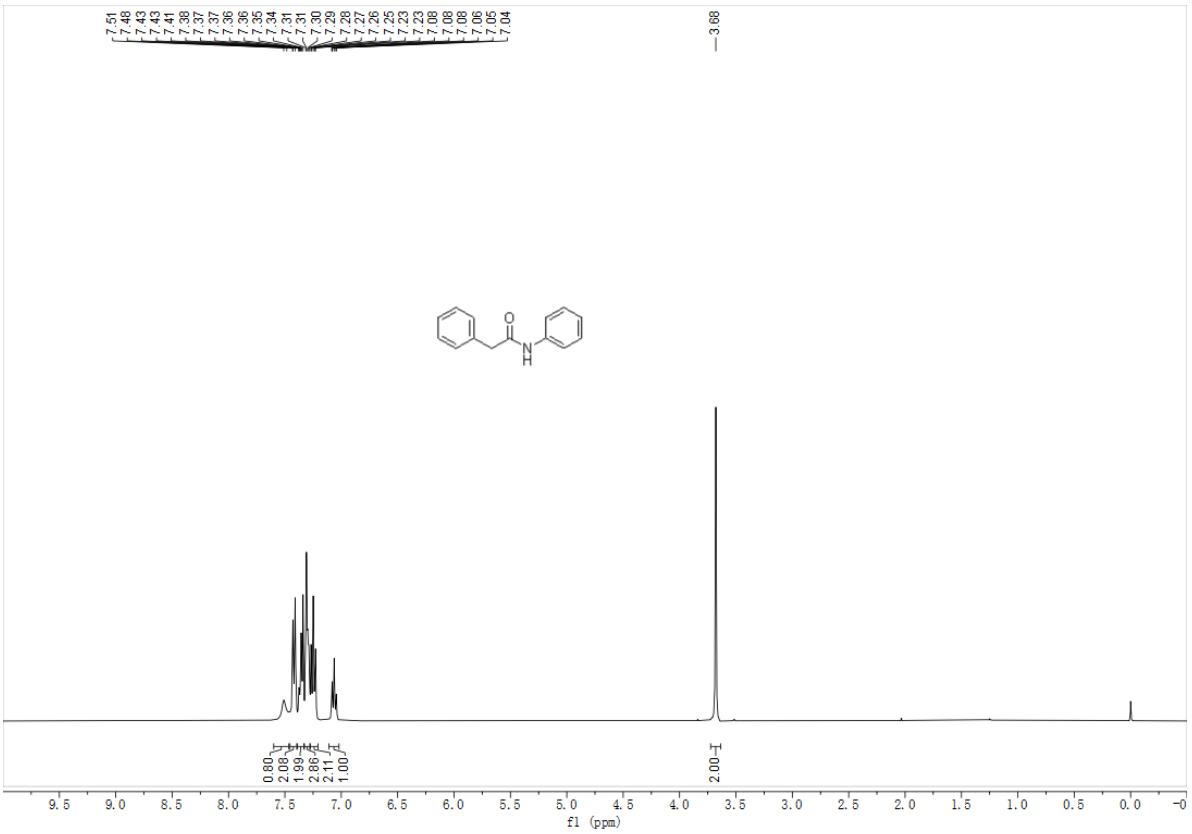
2 J. Shao, X. Huang, S. Wang and B. Liu, *Tetrahedron*, 2012, **68**, 573−579.

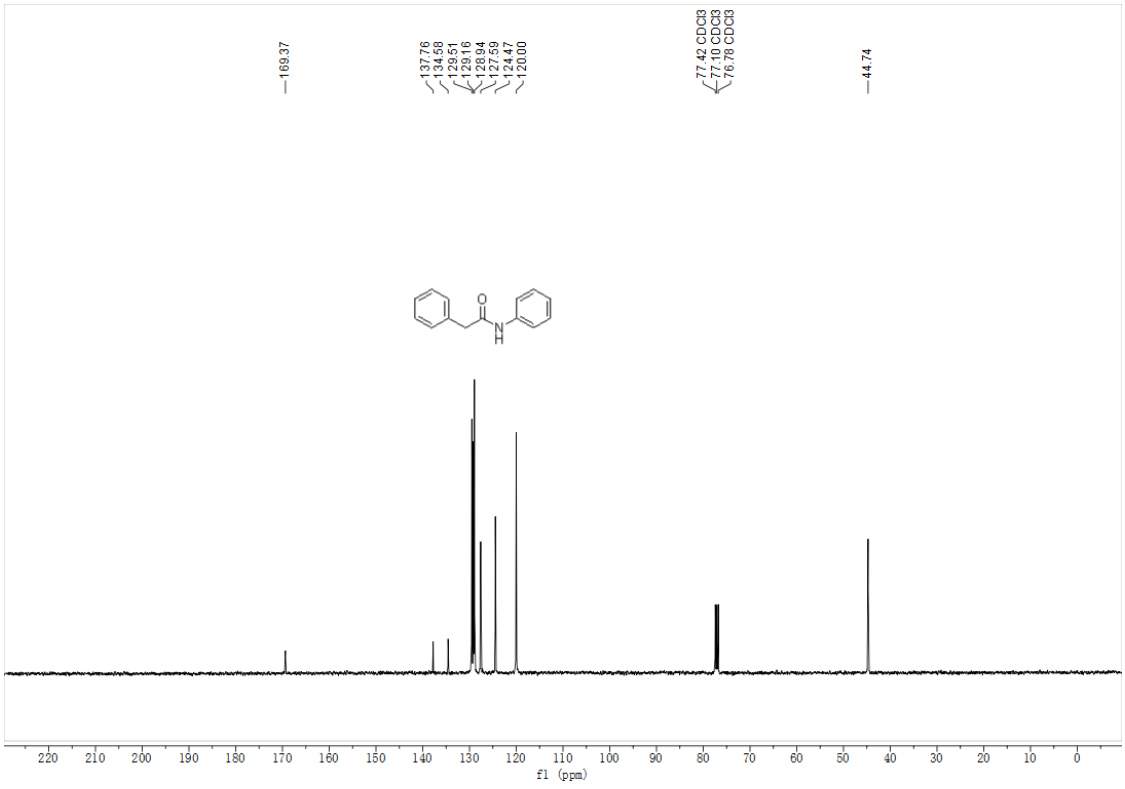
3 X. Zhao, J. Zhao, X. Sun, J. Liu and B. Wu, *Tetrahedron*, 2017, **73**, 3463−3477.

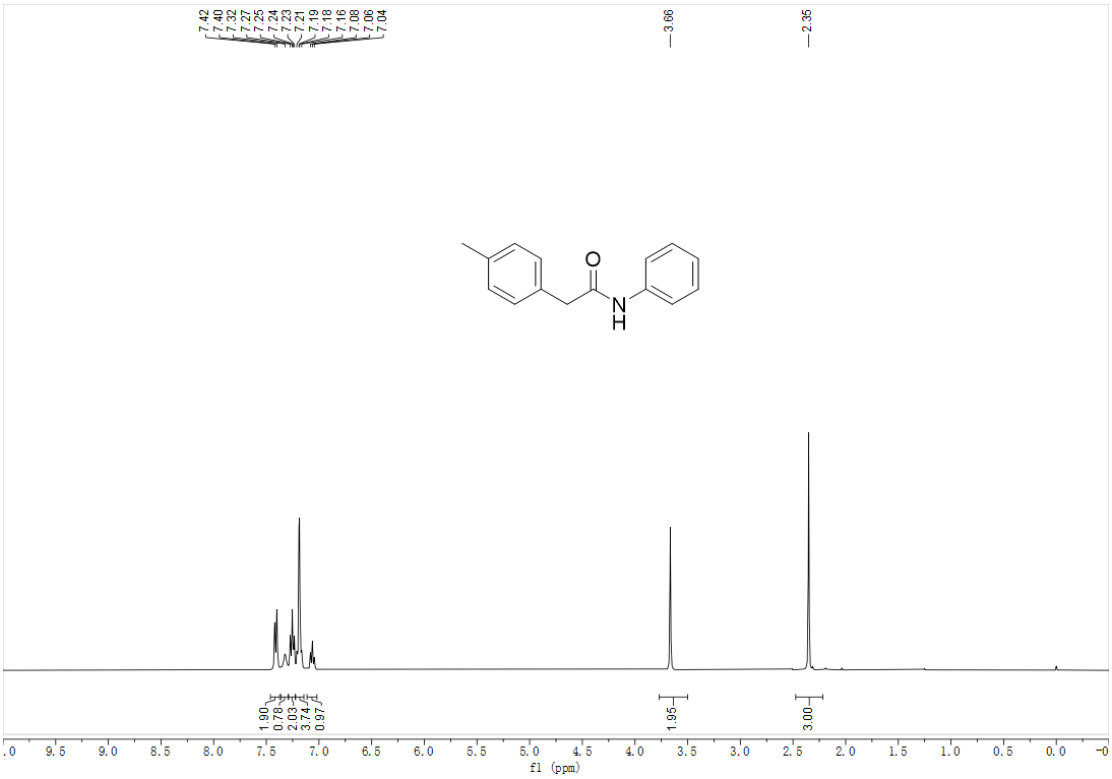
4 T. E. Glotova, M. Y. Dvorko, A. I. Albanov, O. N. Kazheva, G. V. Shilov and O. A. D'yachenko, *Russ J. Org. Chem.*, 2008, **44**, 1532 –1537.

5 C. Nájera, L. K. Sydnes and M. Yus, *Chem. Rev.*, 2019, **119**, 11110 –11244.

10. Copies of ^{1}H and ^{13}C NMR spectra






7.42
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7.18
7.16
7.08
7.06
7.04
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N
H
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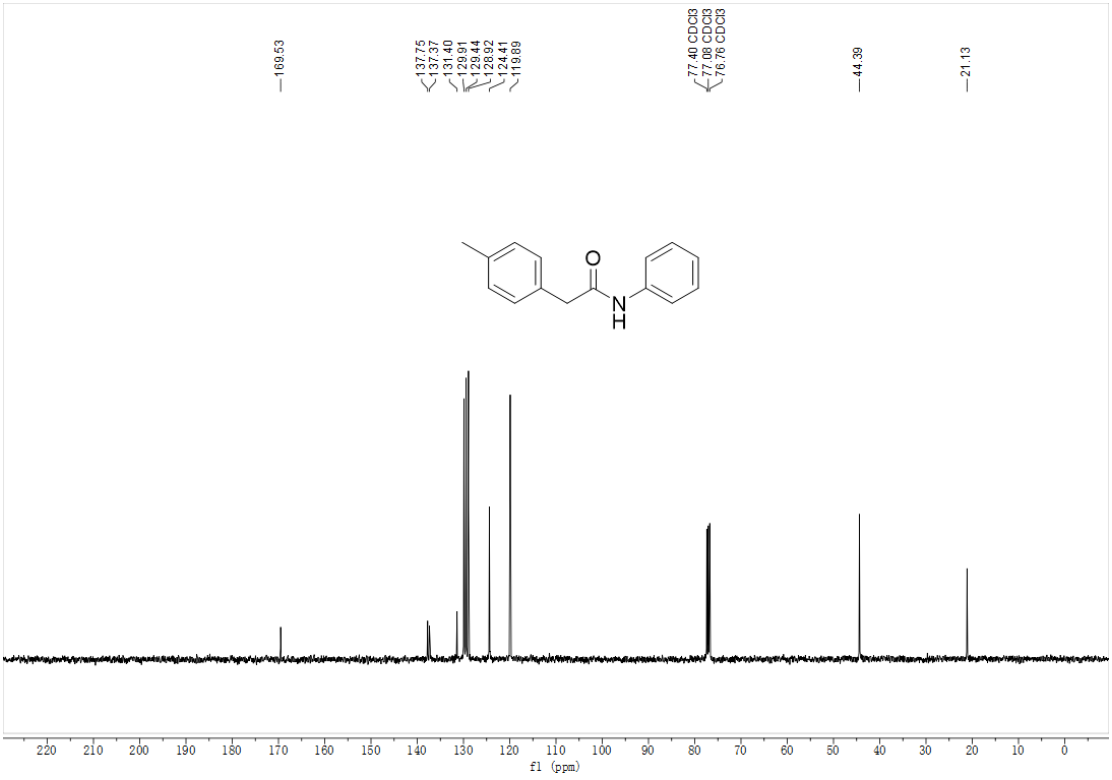
0.78
2.03
3.74
0.97
1.95
3.00
f1 (ppm)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128.92
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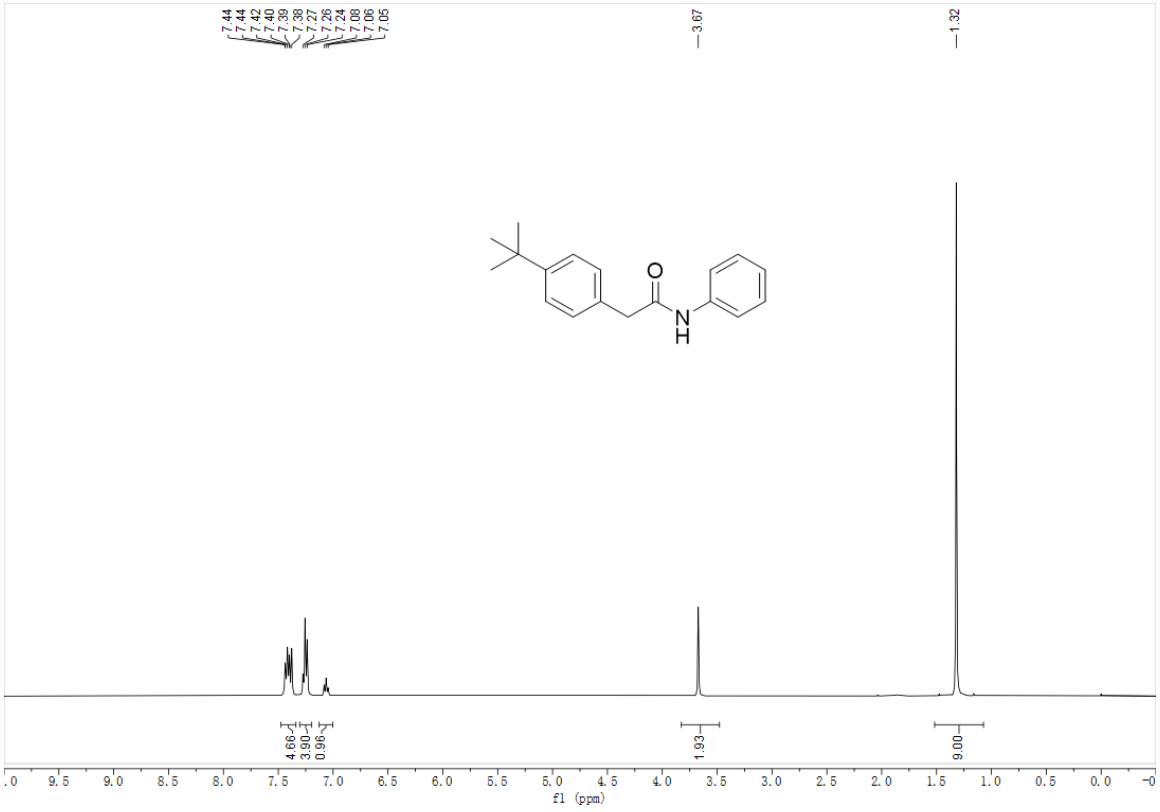
77.08 CDCl3
76.76 CDCl3
44.39
21.13
O
N
H
f1 (ppm)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7.26
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7.05
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4.66
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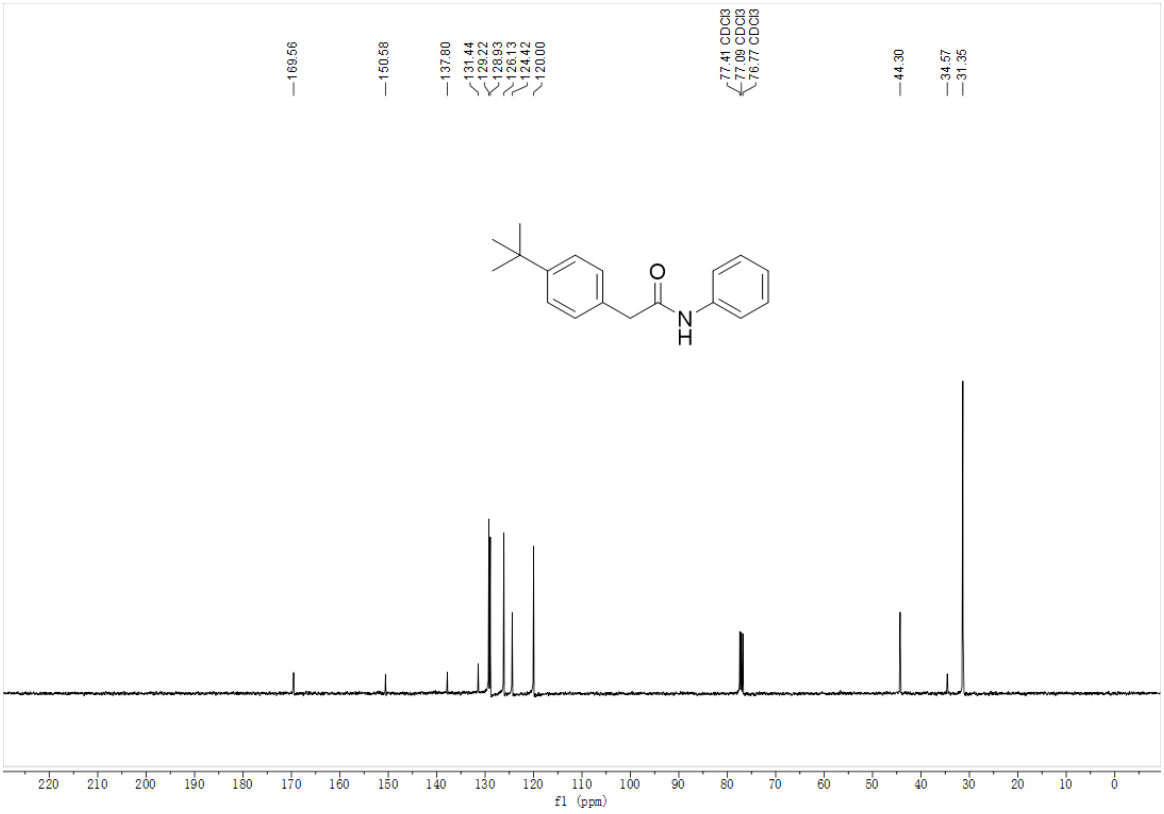
0.96
1.93
9.00
f1 (ppm)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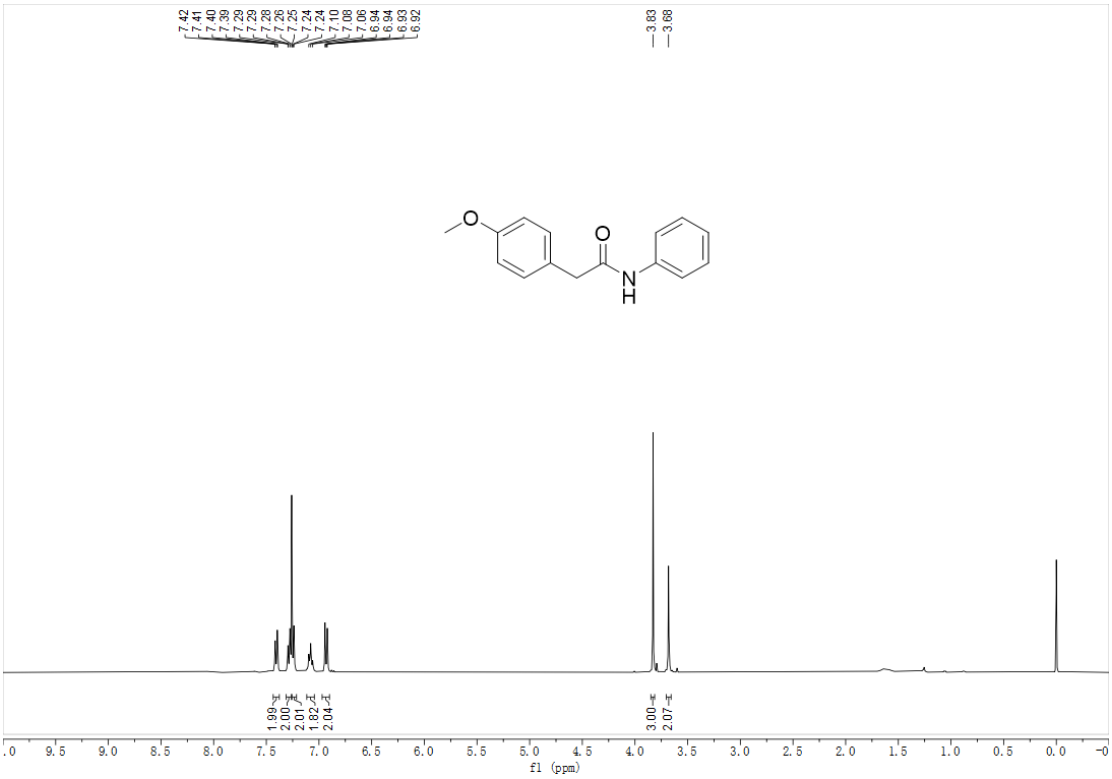
77.09 CDCl3
76.77 CDCl3
44.30
34.57
31.35
f1 (ppm)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7.24
7.10
7.08
7.06
6.94
6.94
6.93
6.92
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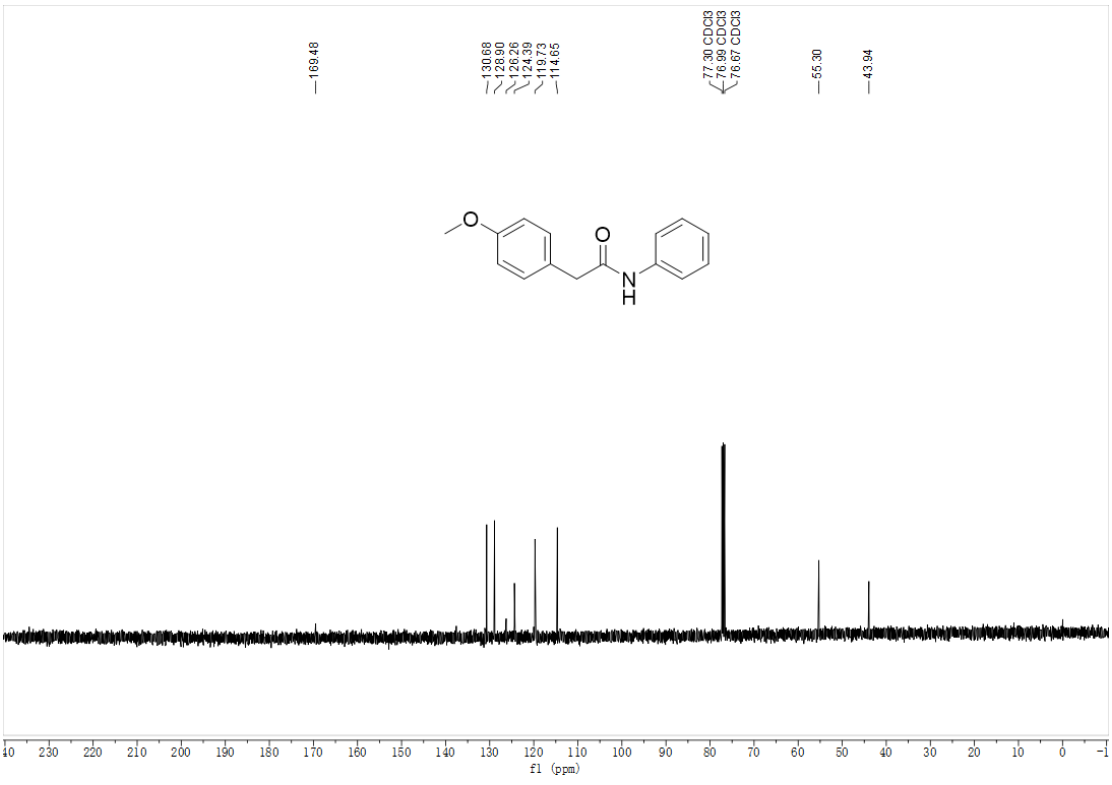
2.01
1.82
2.04
3.00
2.07
f1 (ppm)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76.99 CDCl3
76.67 CDCl3
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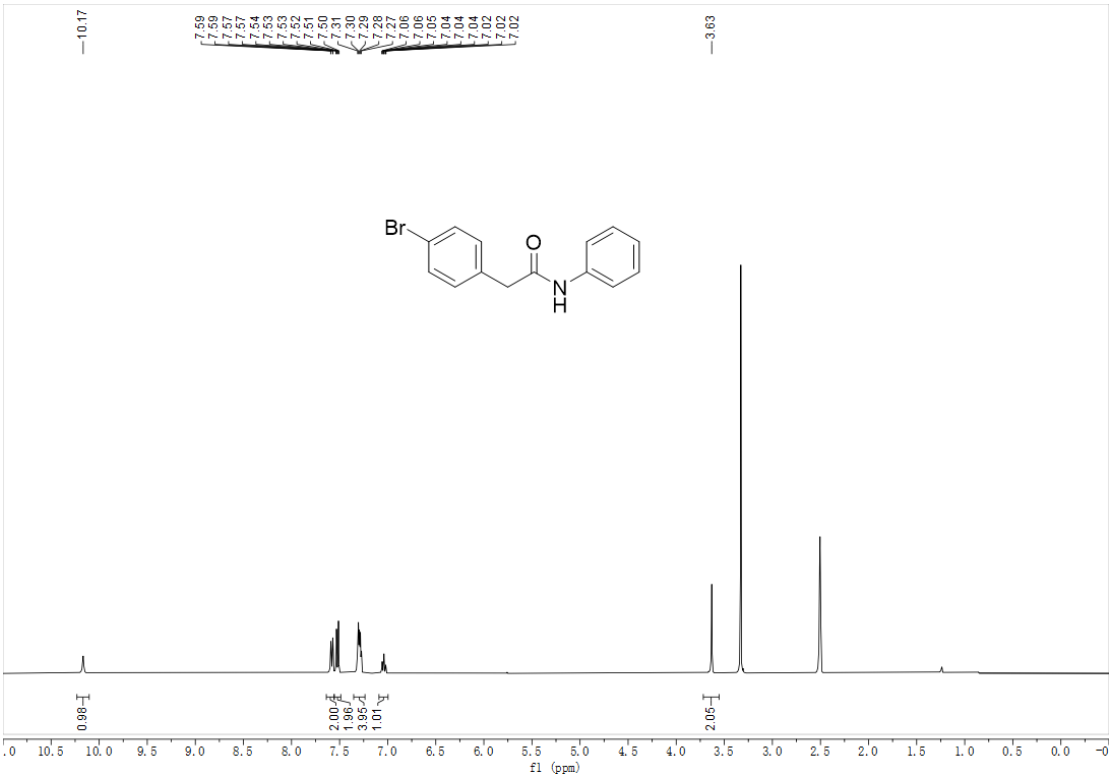
43.94
f1 (ppm)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7.28
7.27
7.06
7.05
7.04
7.04
7.02
7.02
7.02
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Br
O
N
H
0.98
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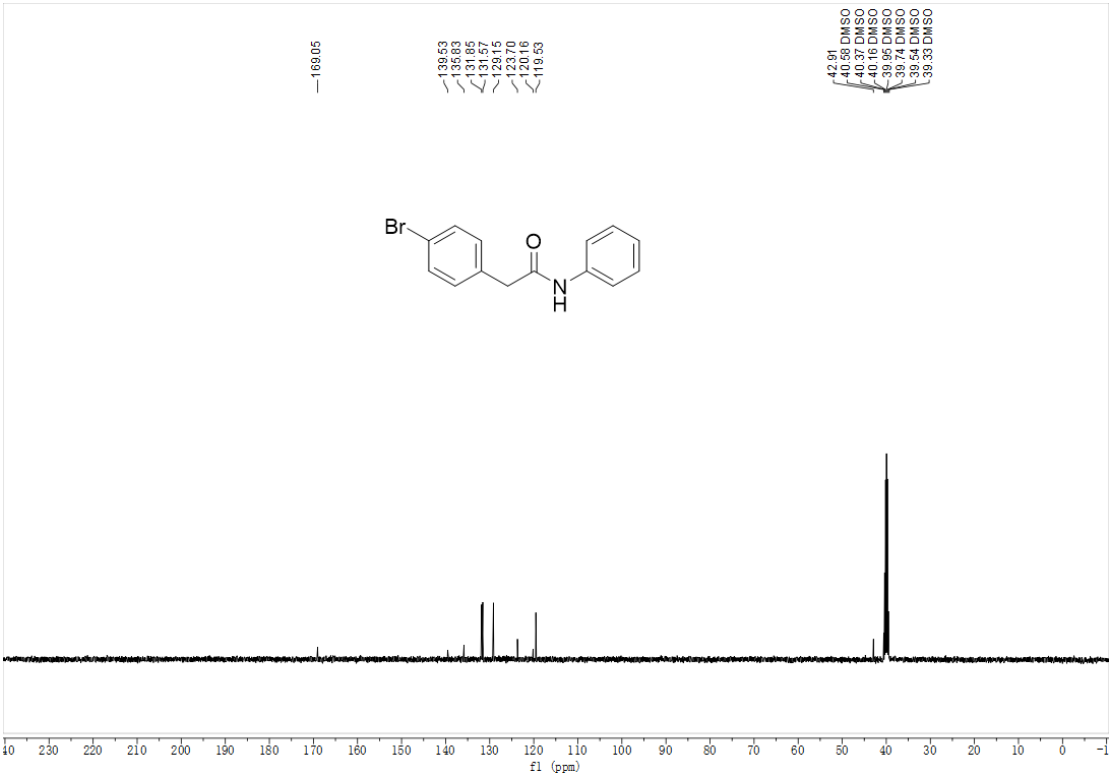
3.95
1.01
2.05
f1 (ppm)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120.16
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42.91
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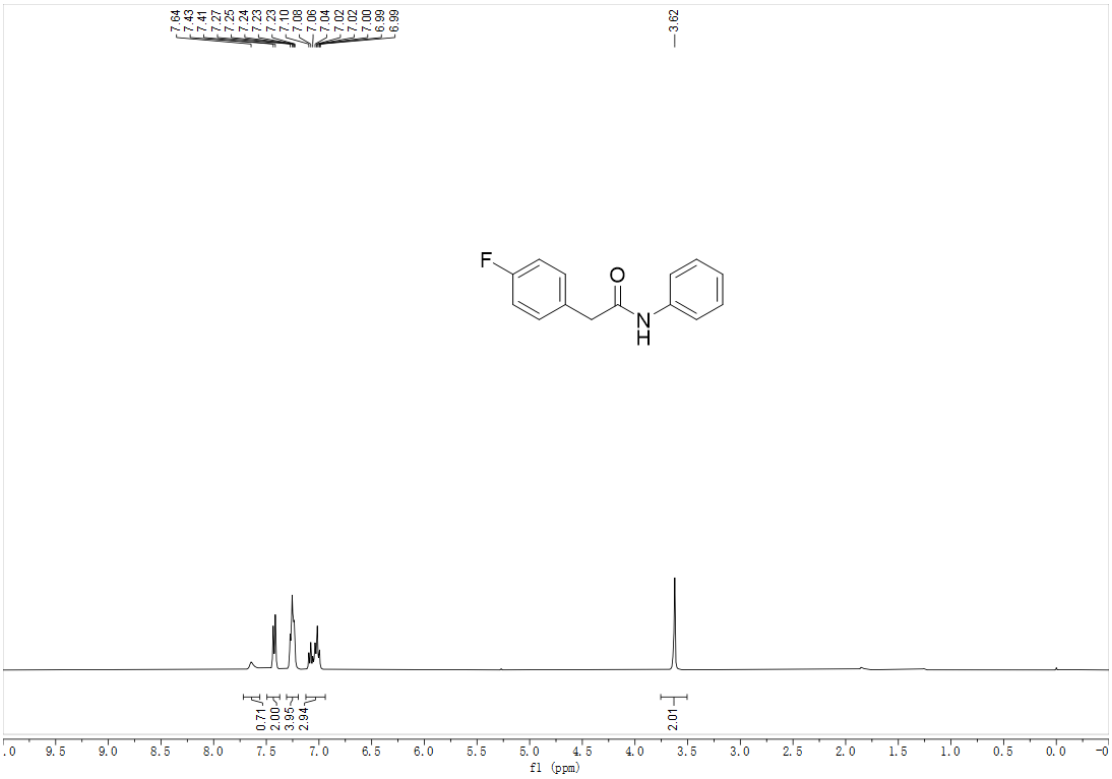
40.16 DMSO
39.95 DMSO
39.74 DMSO
39.54 DMSO
39.33 DMSO
Br
O
N
H
f1 (ppm)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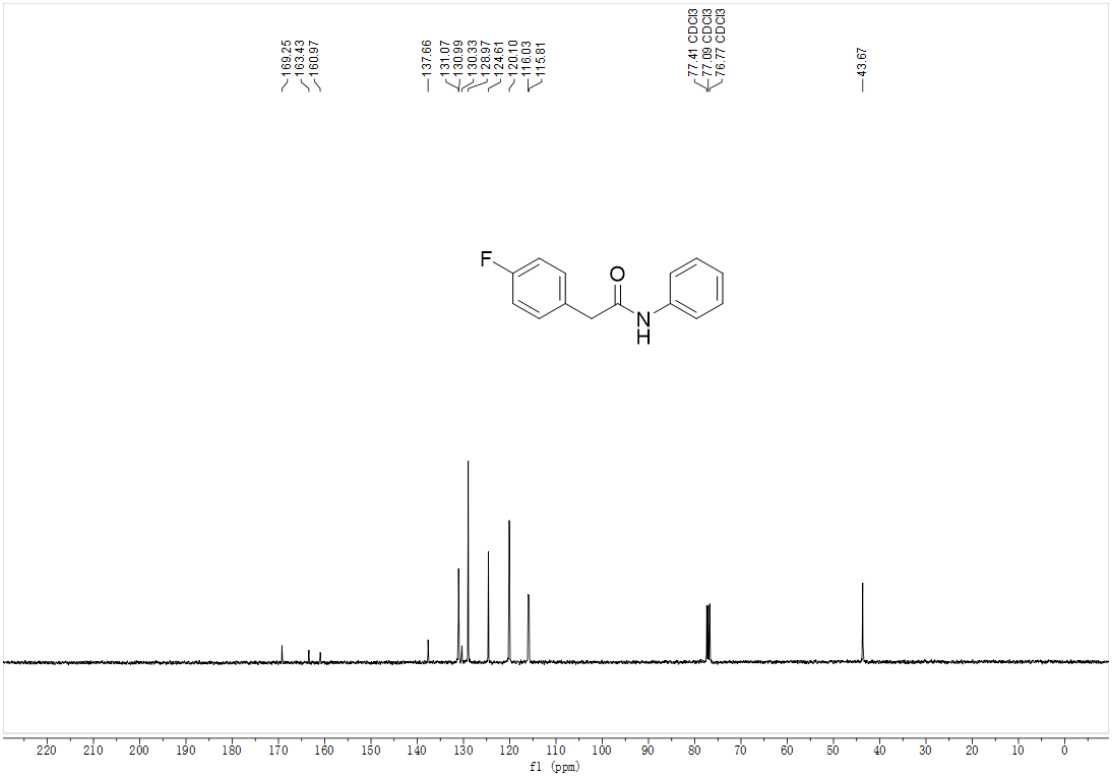
7.23
7.23
7.10
7.08
7.06
7.04
7.02
7.02
7.00
6.99
6.99
3.62
F
O
N
H
0.71
2.00
3.95
2.94
2.01
f1 (ppm)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128.97
124.61
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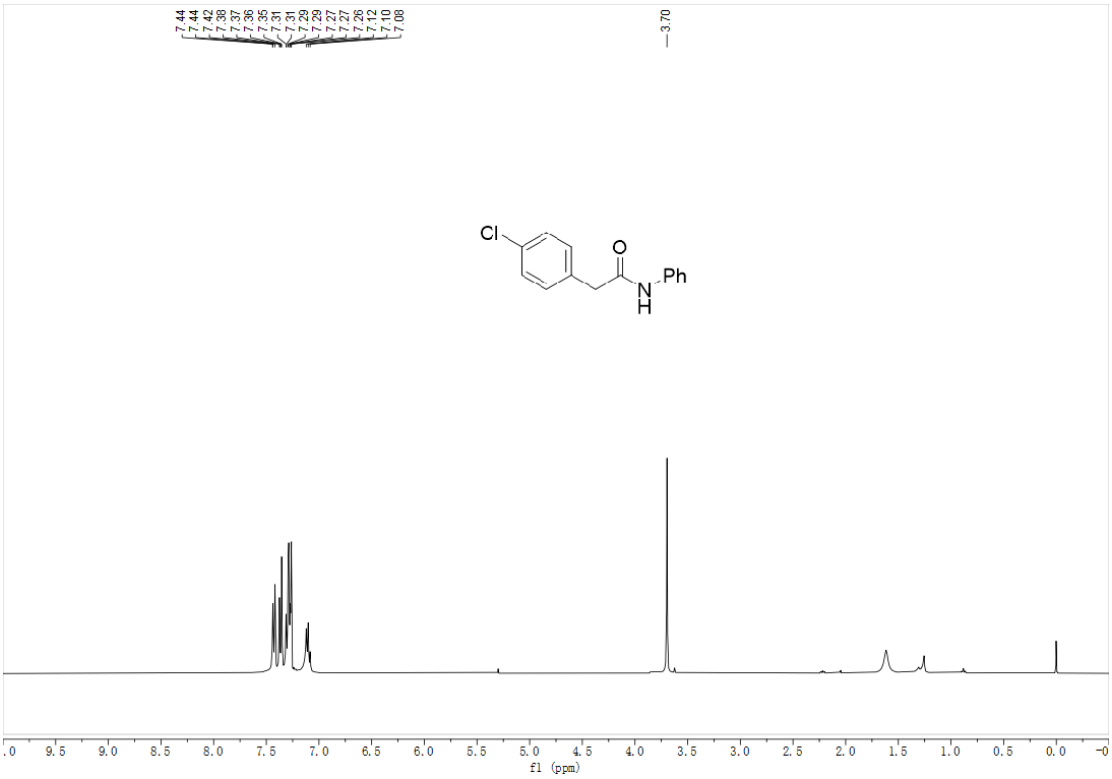
77.41 CDCl3
77.09 CDCl3
76.77 CDCl3
43.67
F
O
N
H
f1 (ppm)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7.27
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7.26
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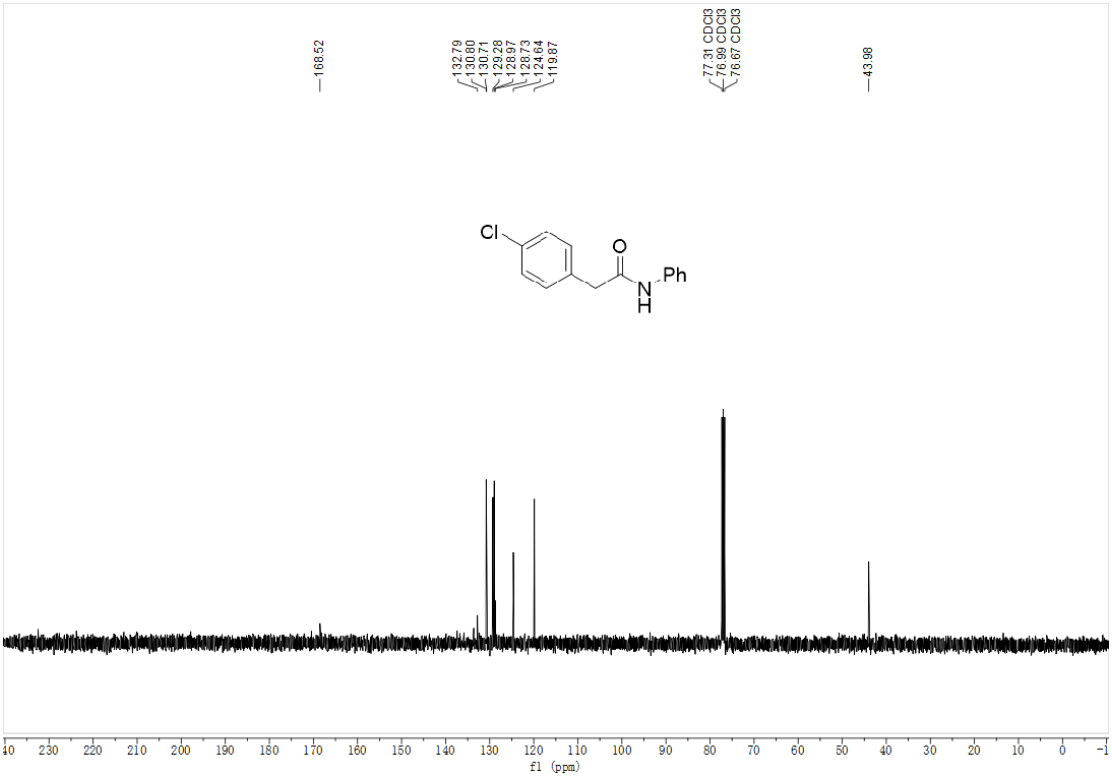
7.10
7.08
3.70
Cl
O
N
H
Ph
f1 (ppm)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128.97
128.73
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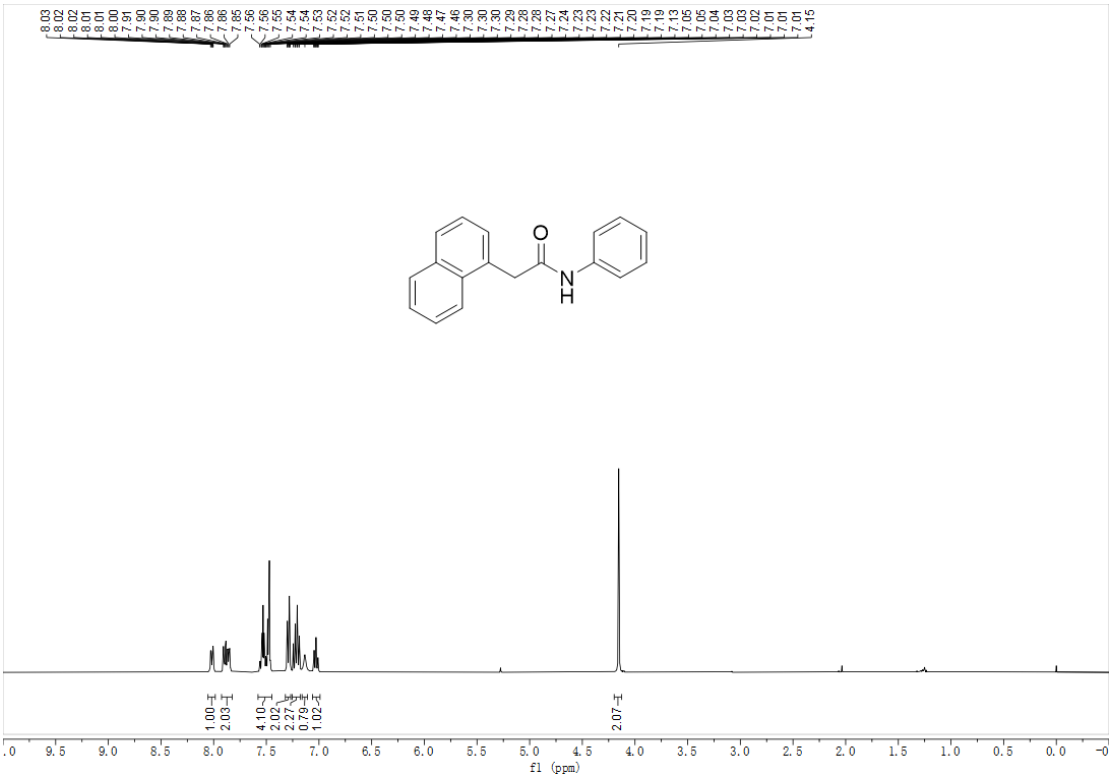
76.99 CDCl3
76.67 CDCl3
43.96
Cl
O
N
H
Ph
f1 (ppm)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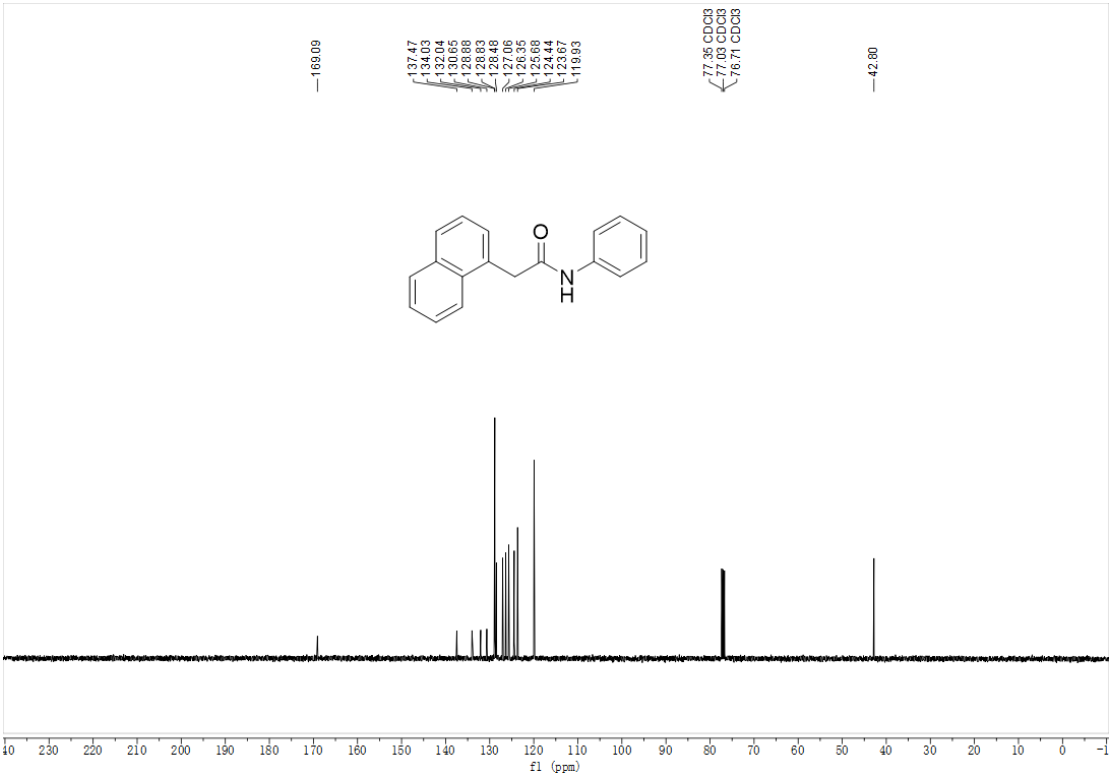
O
N
H
1.00
2.03
4.10
2.02
2.27
0.79
1.02
2.07
f1 (ppm)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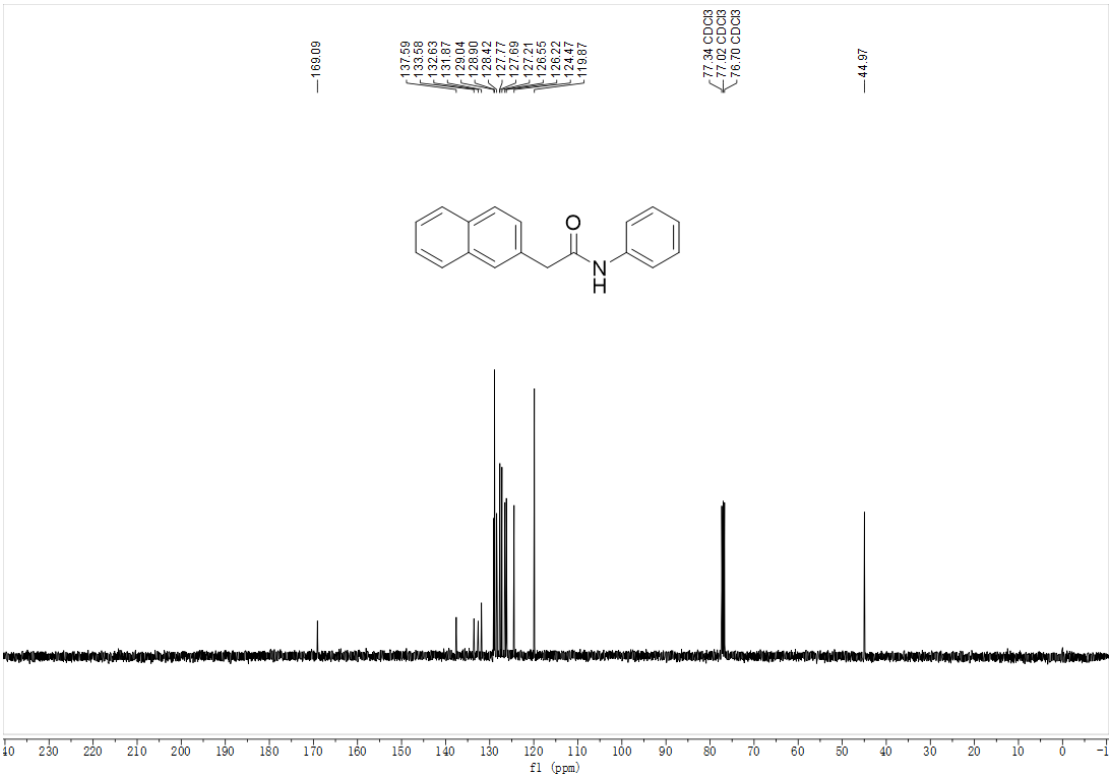
77.35 CDCl3
77.03 CDCl3
76.71 CDCl3
42.80
O
N
H
f1 (ppm)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126.55
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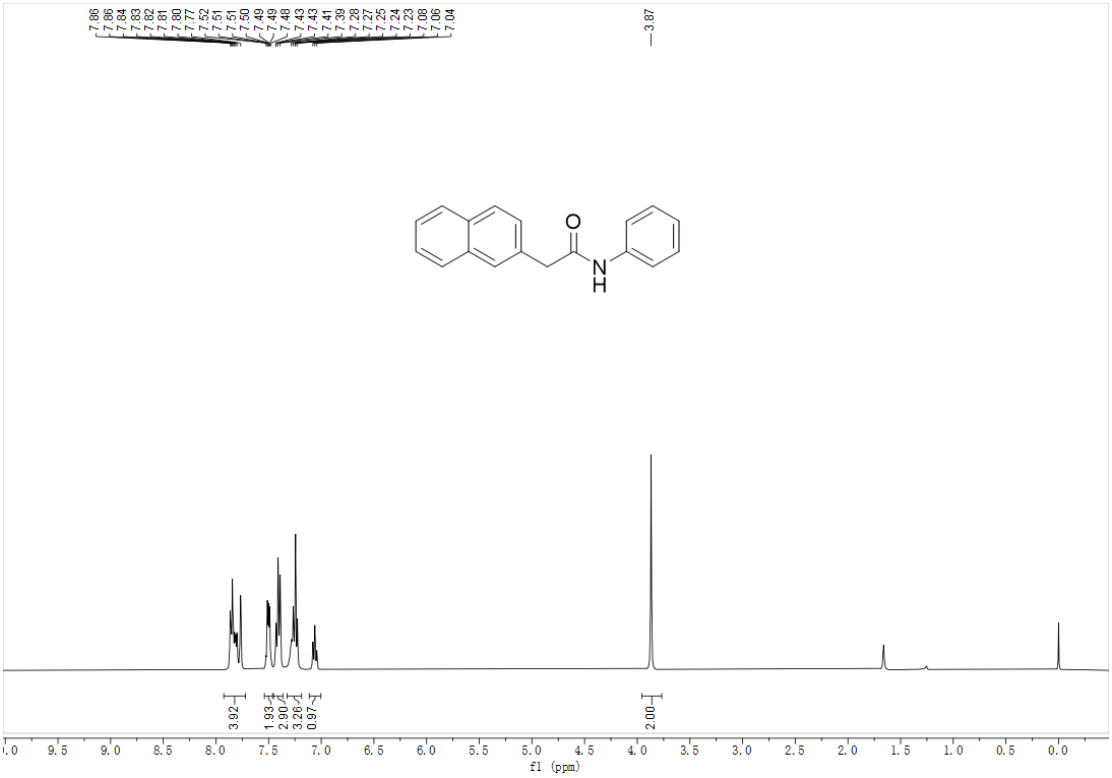
119.87
77.34 CDCl3
77.02 CDCl3
76.70 CDCl3
44.97
O
N
H
f1 (ppm)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7.43
7.41
7.39
7.28
7.27
7.25
7.24
7.23
7.08
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7.04
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O
N
H
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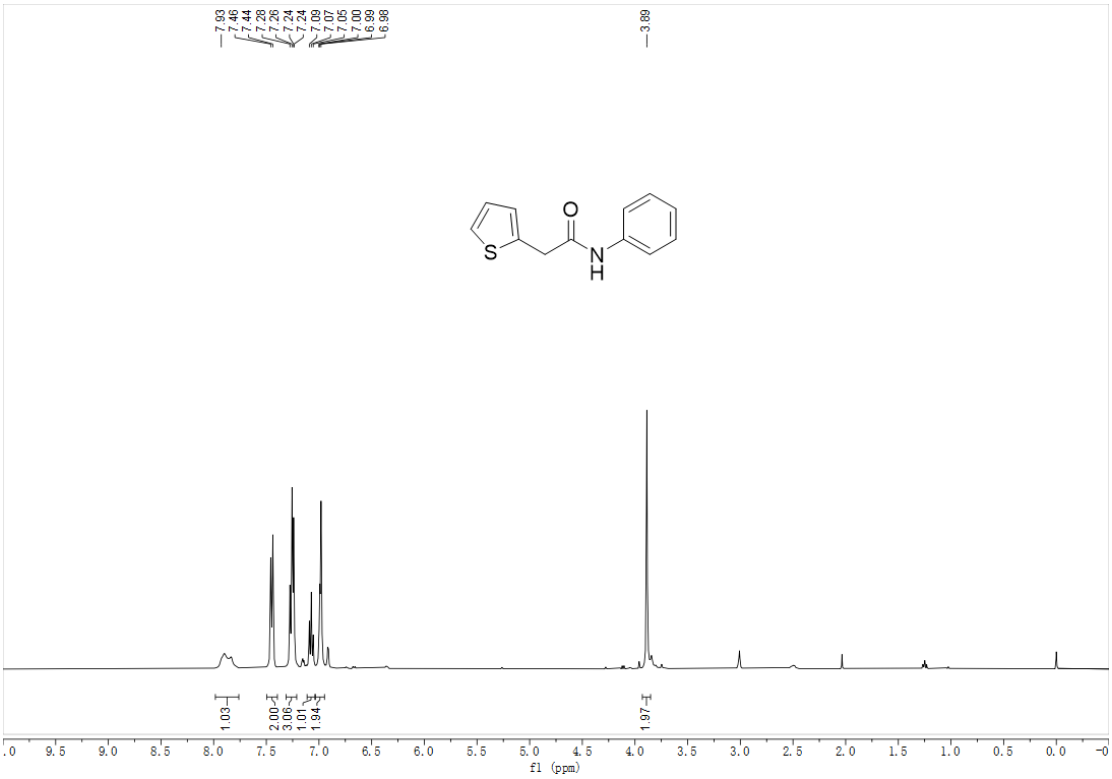
1.93
2.90
3.26
0.97
2.00
f1 (ppm)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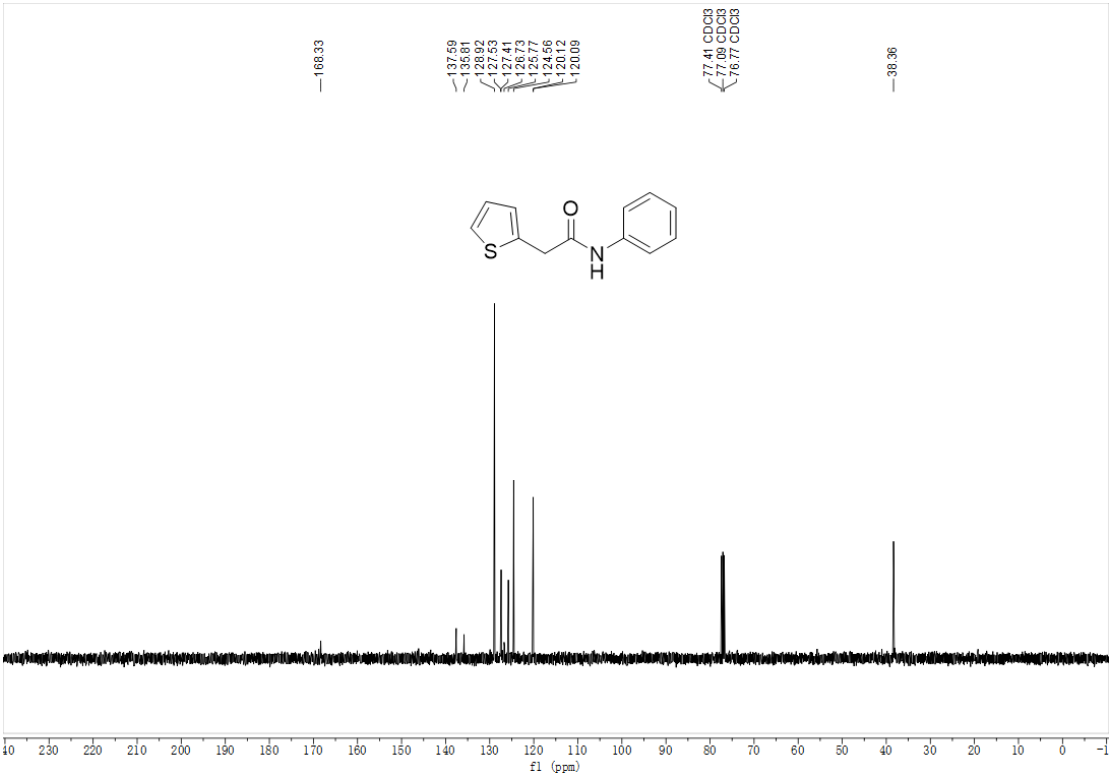
3.06
1.01
1.94
1.97
f1 (ppm)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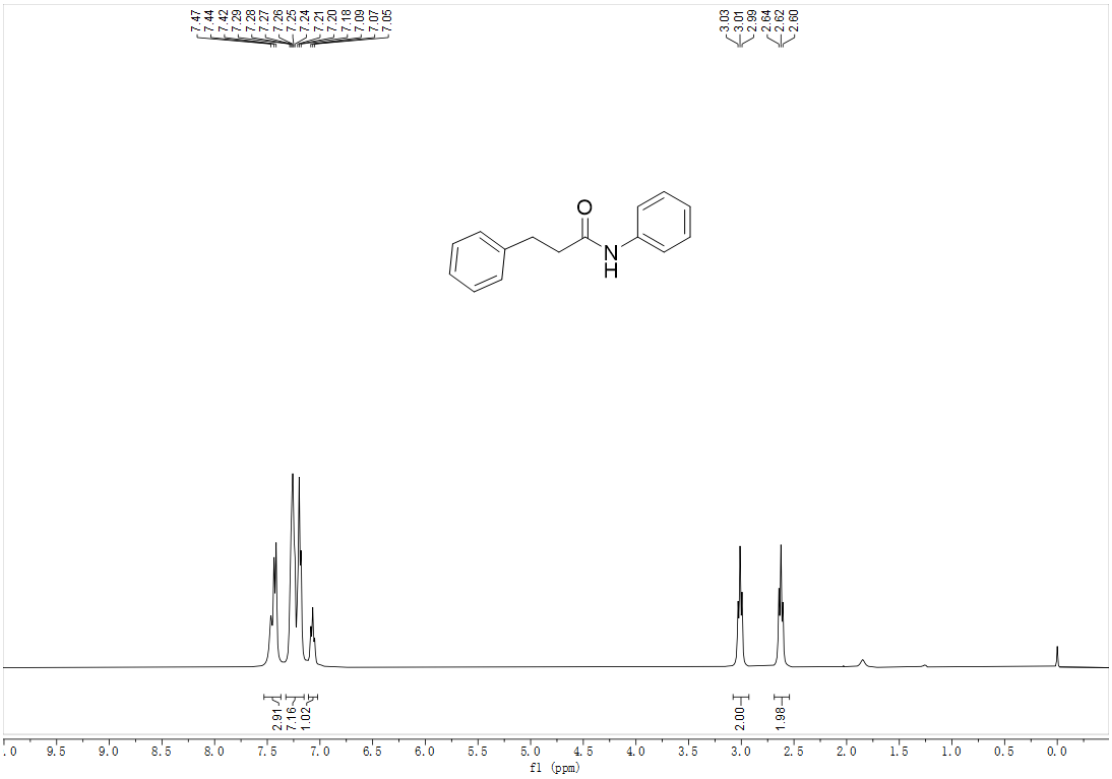
77.09 CDCl3
76.77 CDCl3
38.36
f1 (ppm)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7.18
7.09
7.07
7.05
3.03
3.01
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H
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1.02
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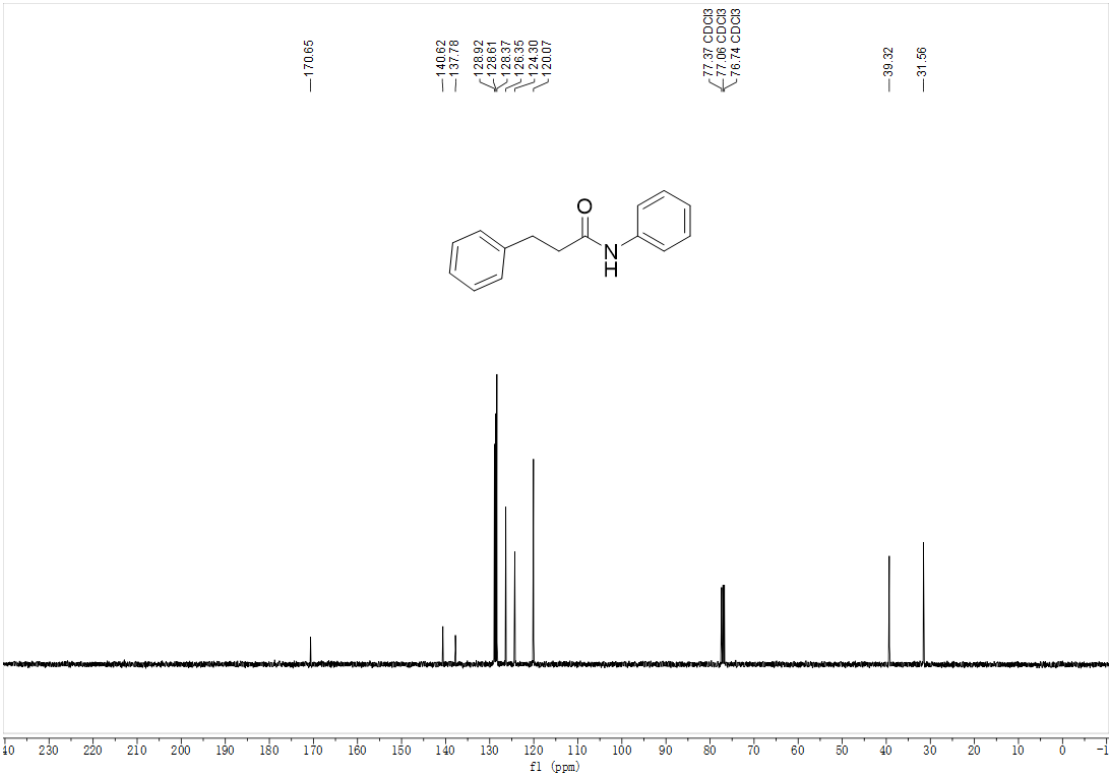
1.98
f1 (ppm)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77.06 CDCl3
76.74 CDCl3
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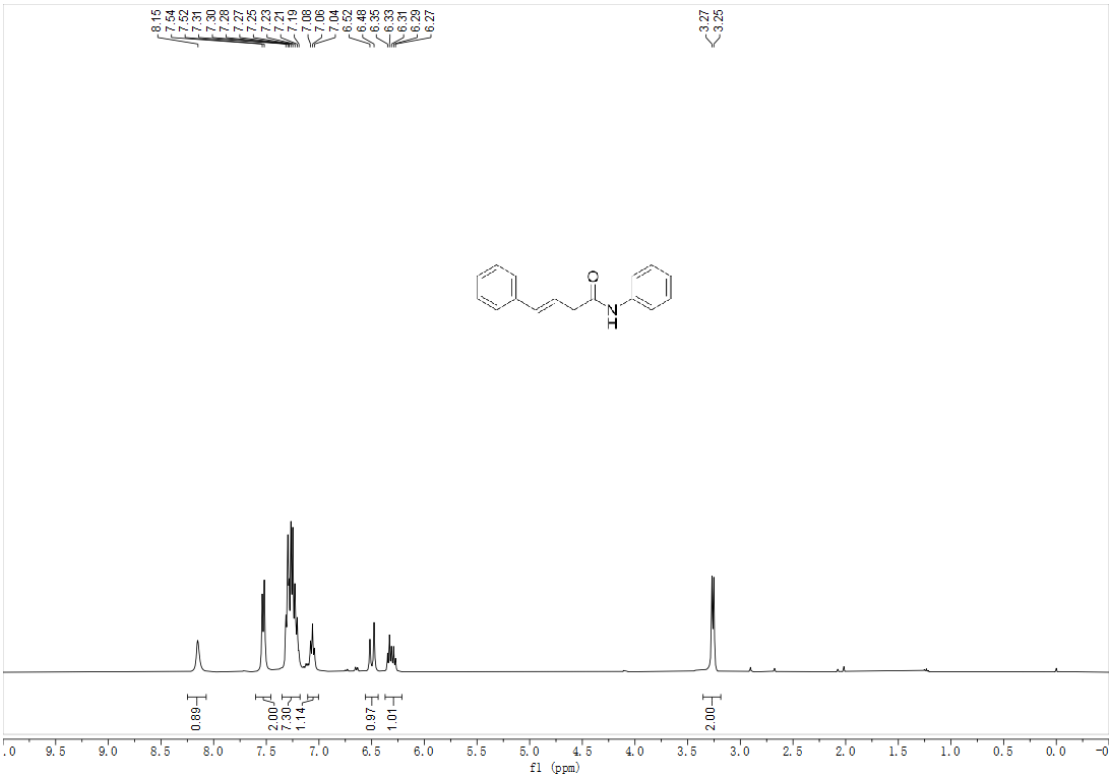
31.56
O
N
H
f1 (ppm)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7.06
7.04
6.52
6.48
6.35
6.33
6.31
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6.27
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3.25
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7.30
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0.97
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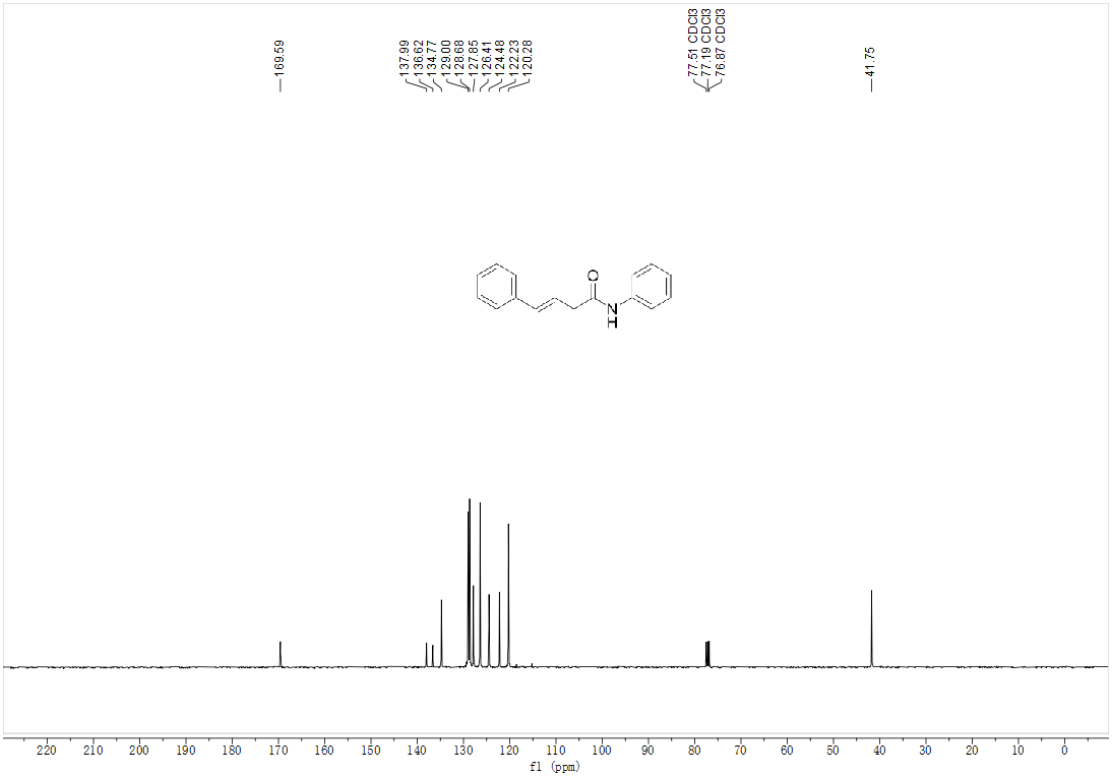
2.00
f1 (ppm)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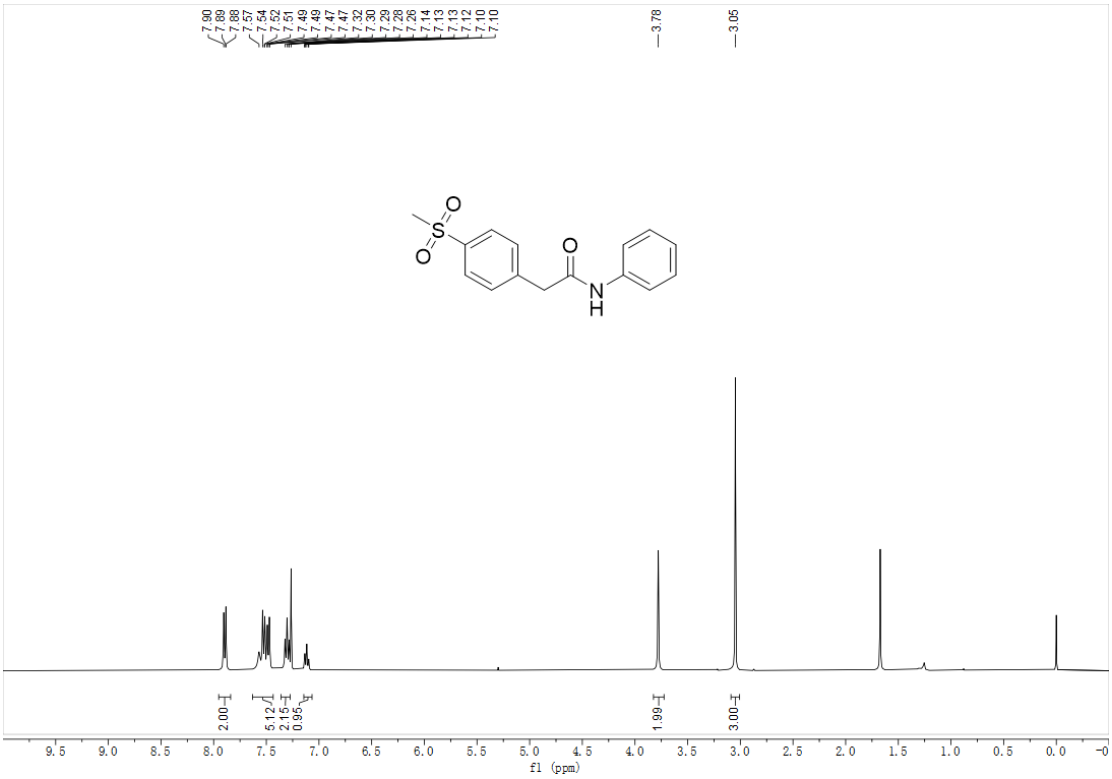
76.87 CDCl3
41.75
f1 (ppm)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7.28
7.26
7.14
7.13
7.13
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3.78
3.05
2.00
5.12
2.15
0.95
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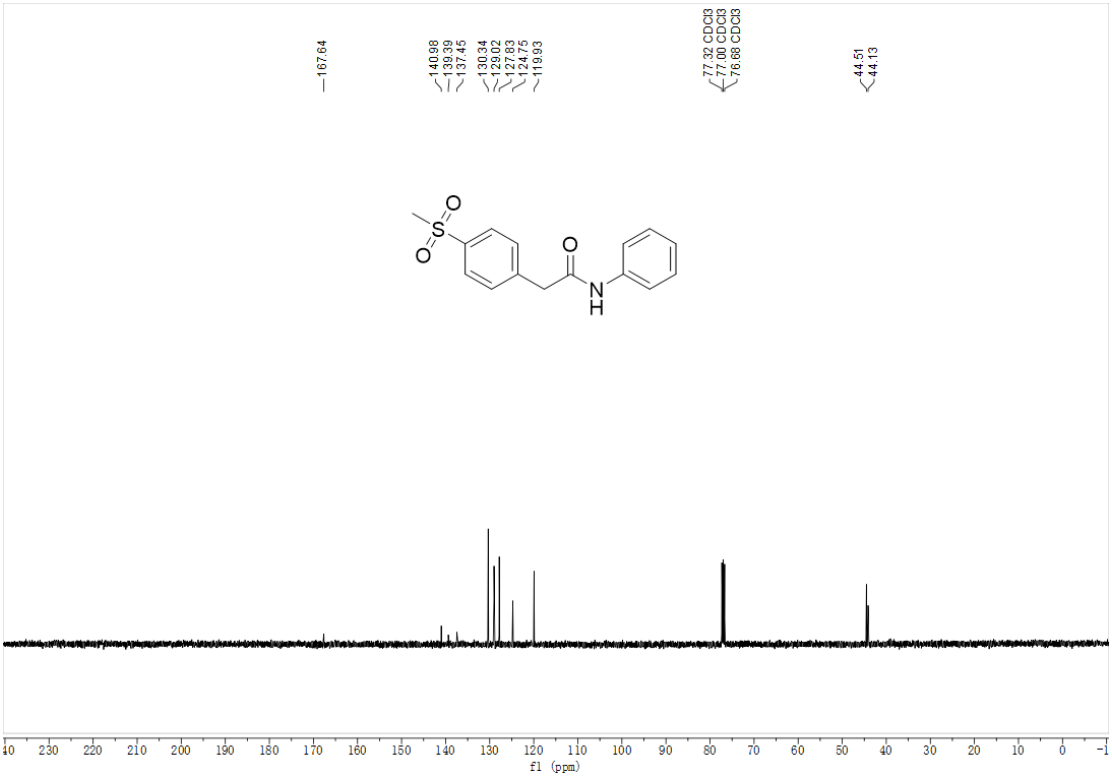
3.00
f1 (ppm)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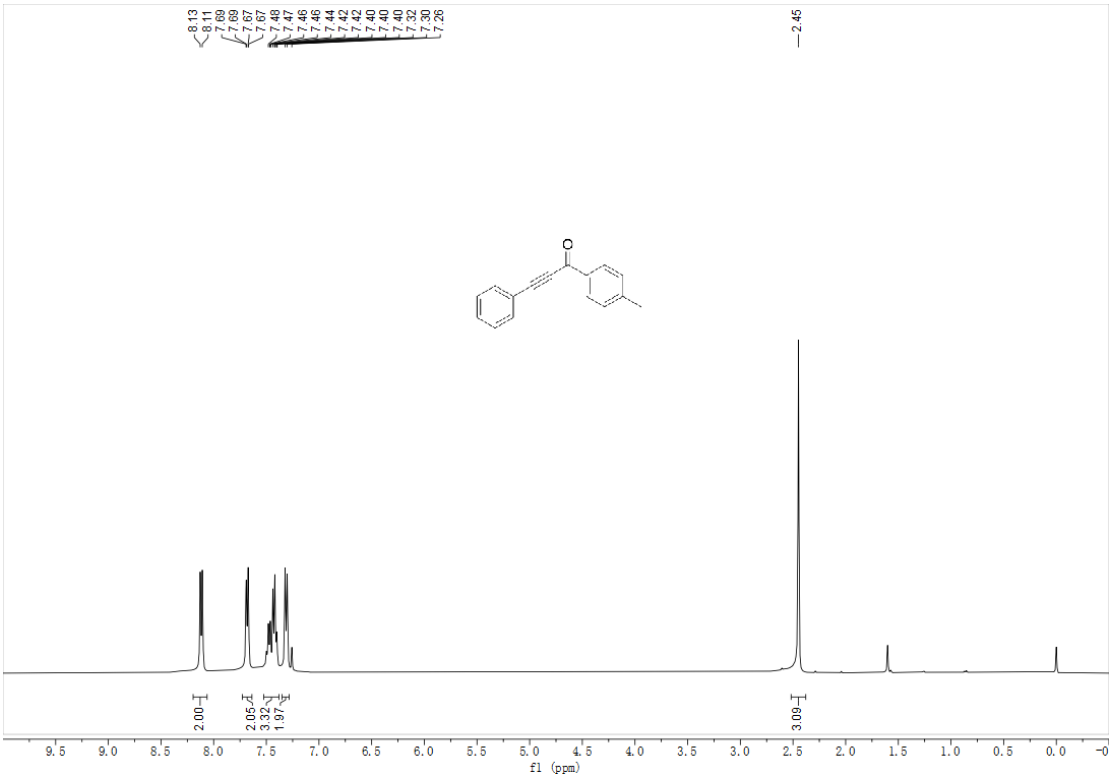
44.13
f1 (ppm)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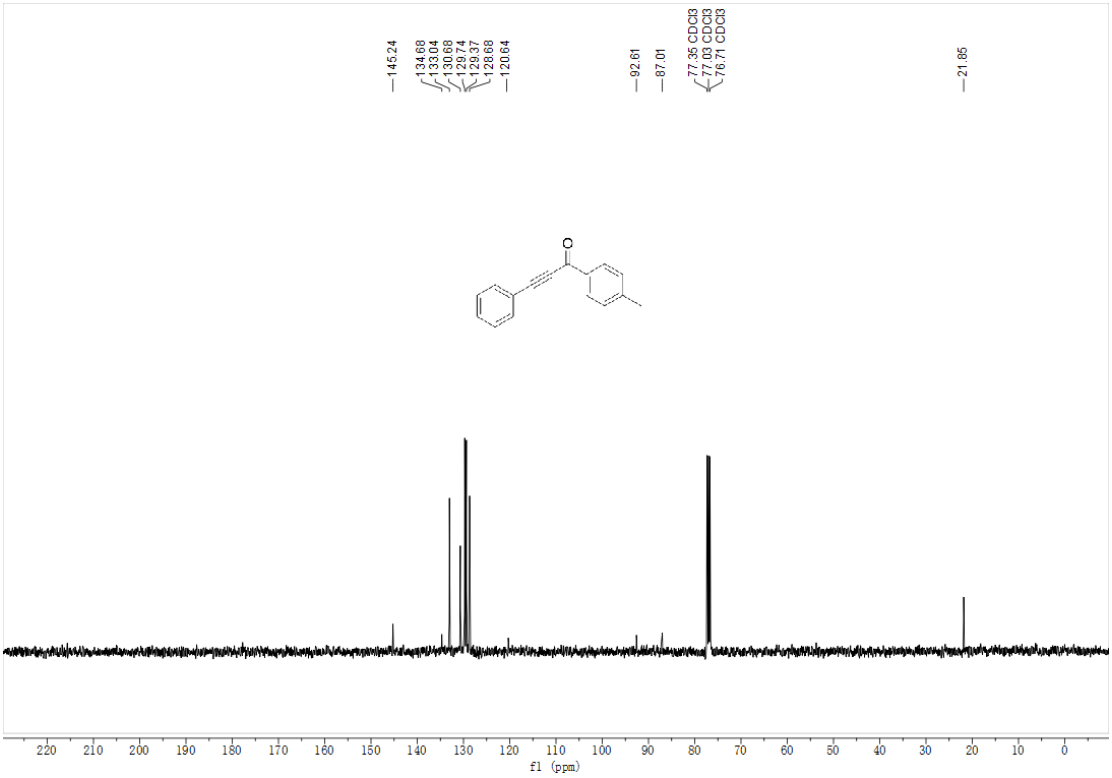
3.32
1.97
3.09
f1 (ppm)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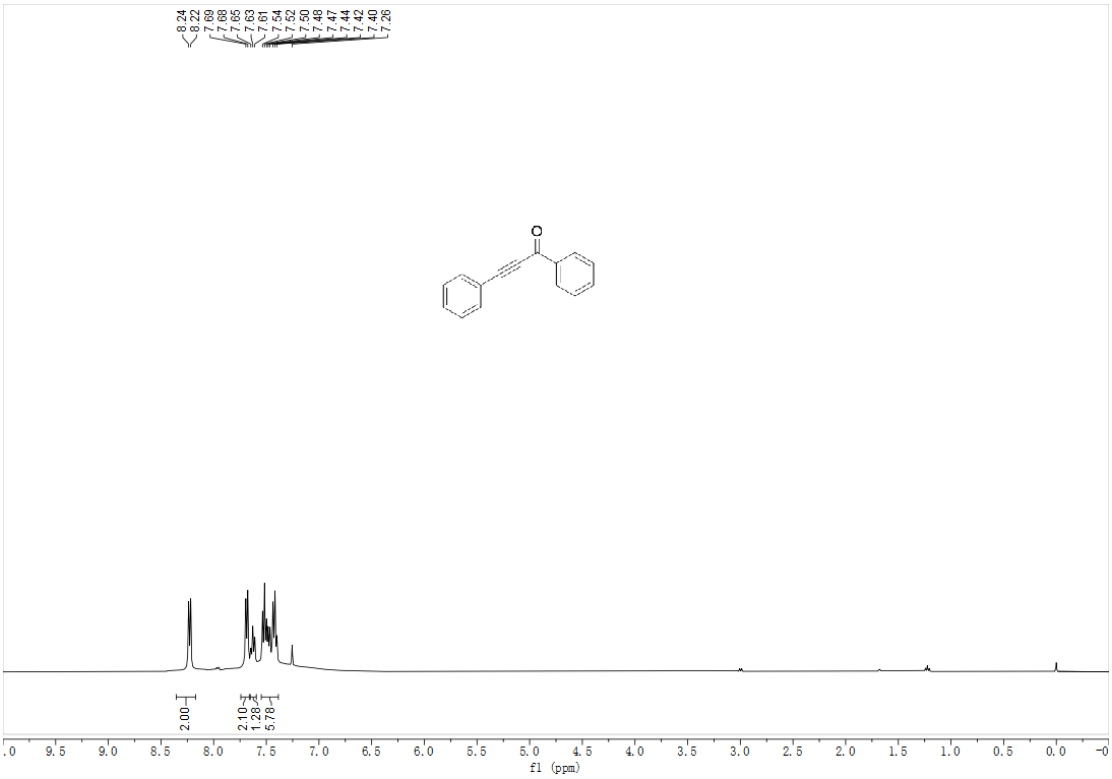
76.71 CDCl3
21.85
f1 (ppm)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7.50
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7.42
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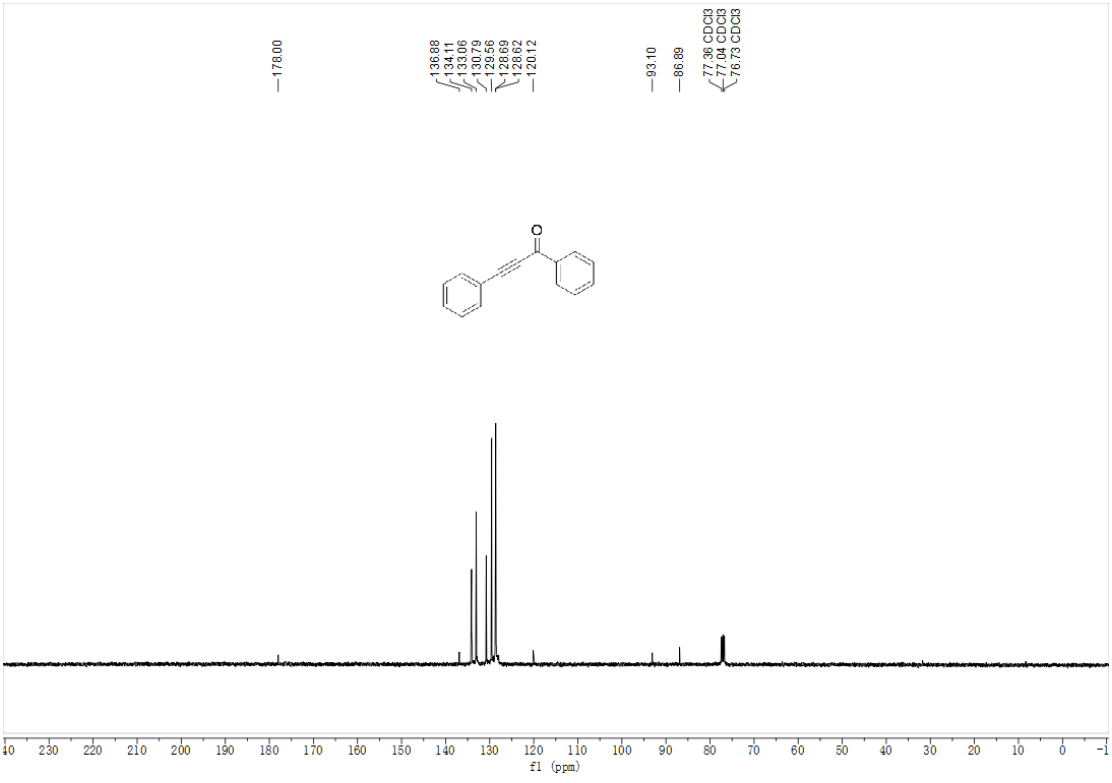
5.78
f1 (ppm)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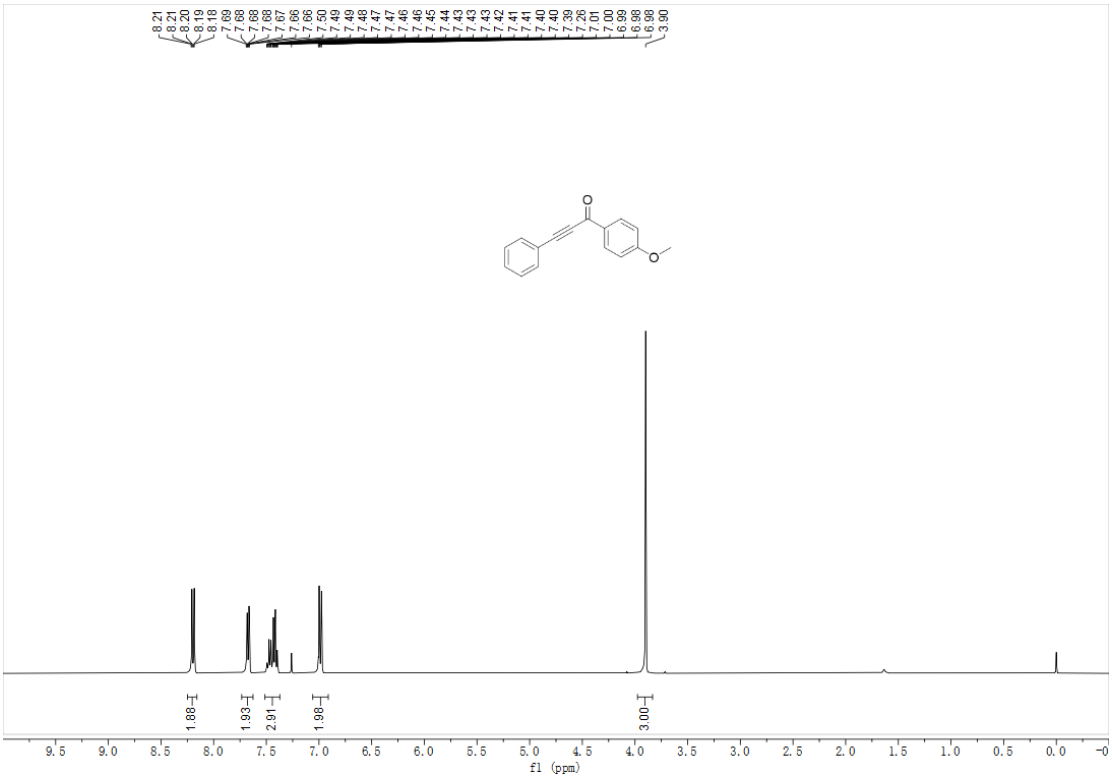
77.04 CDCl3
76.73 CDCl3
O
f1 (ppm)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O
O
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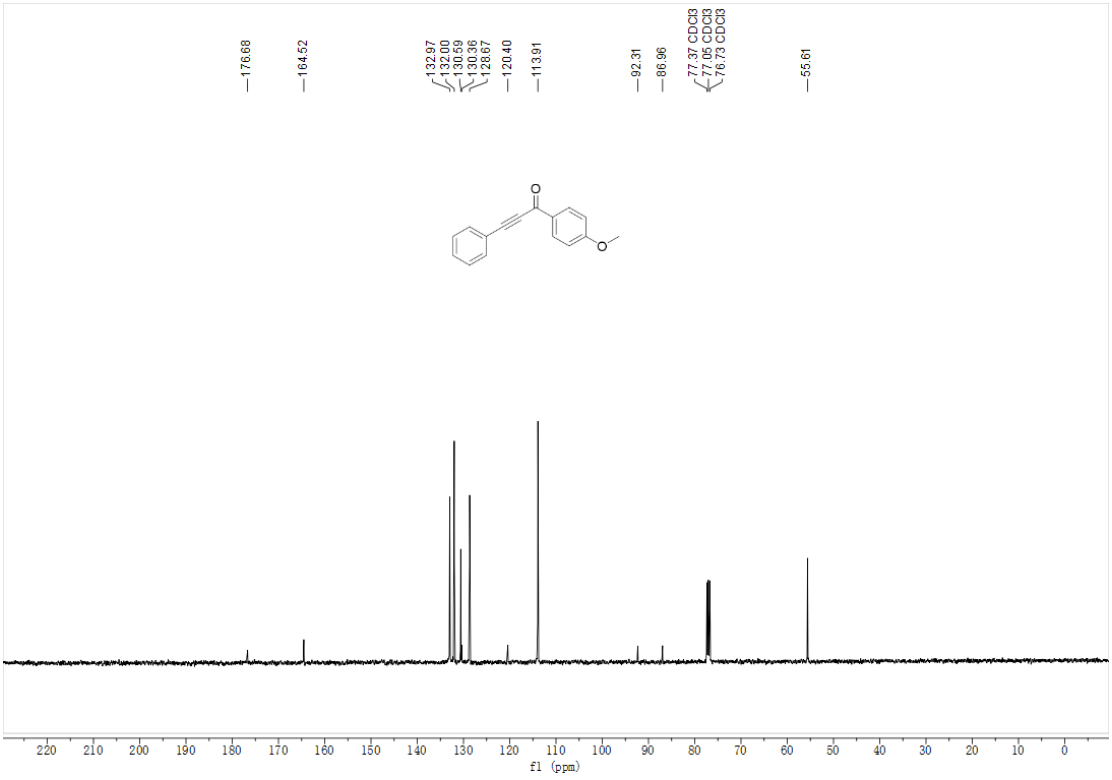
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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132.00
130.59
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128.67
120.40
113.91
92.31
86.96
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
55.61
O
O
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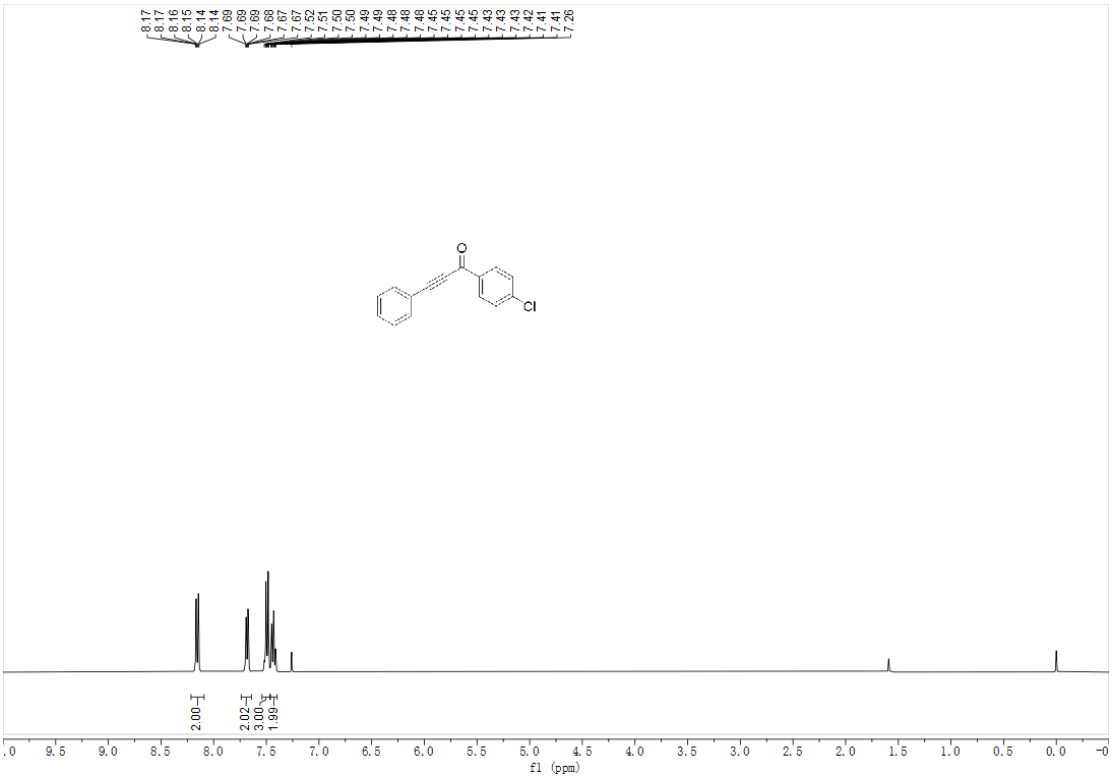
f1 (ppm)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7.48
7.48
7.45
7.45
7.45
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Cl
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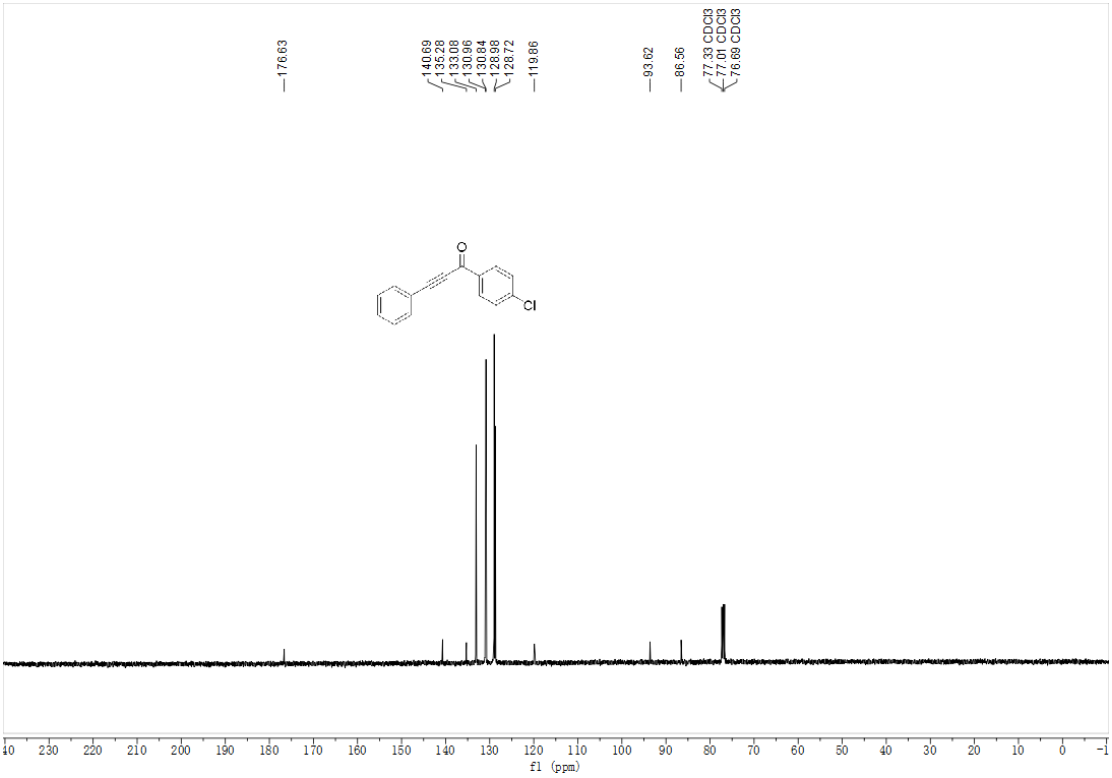
2.02
3.00
1.99
f1 (ppm)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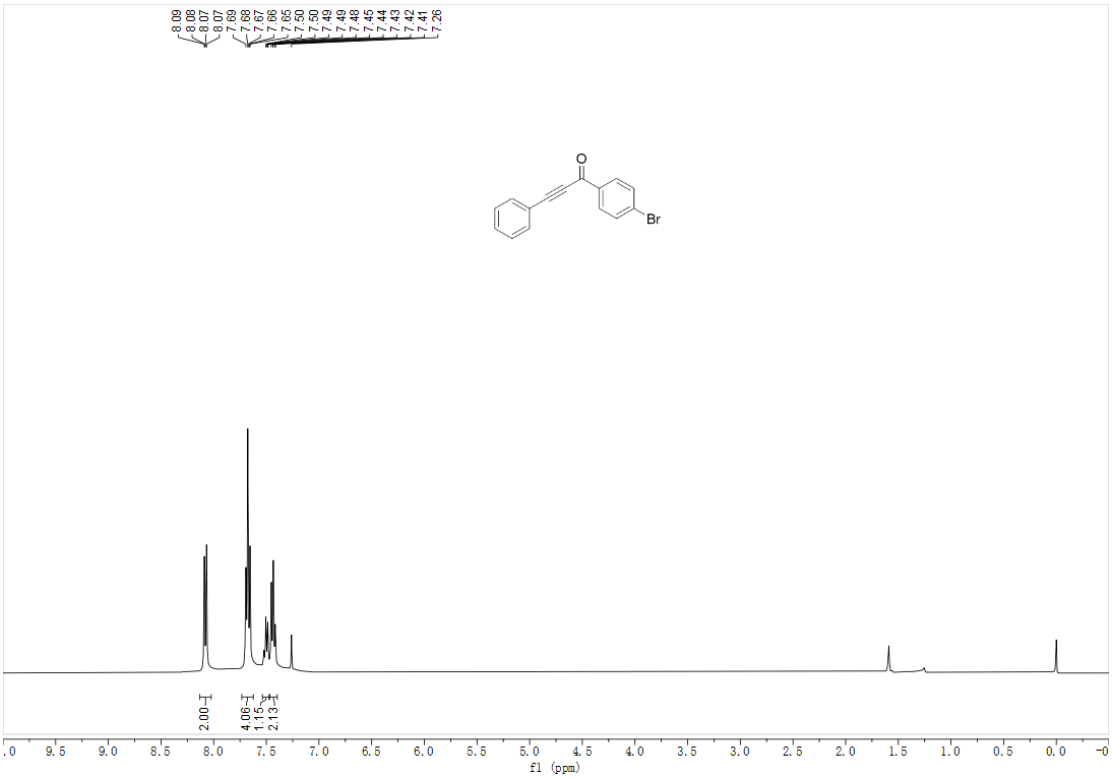
77.01 CDCl3
76.69 CDCl3
O
Cl
f1 (ppm)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7.49
7.48
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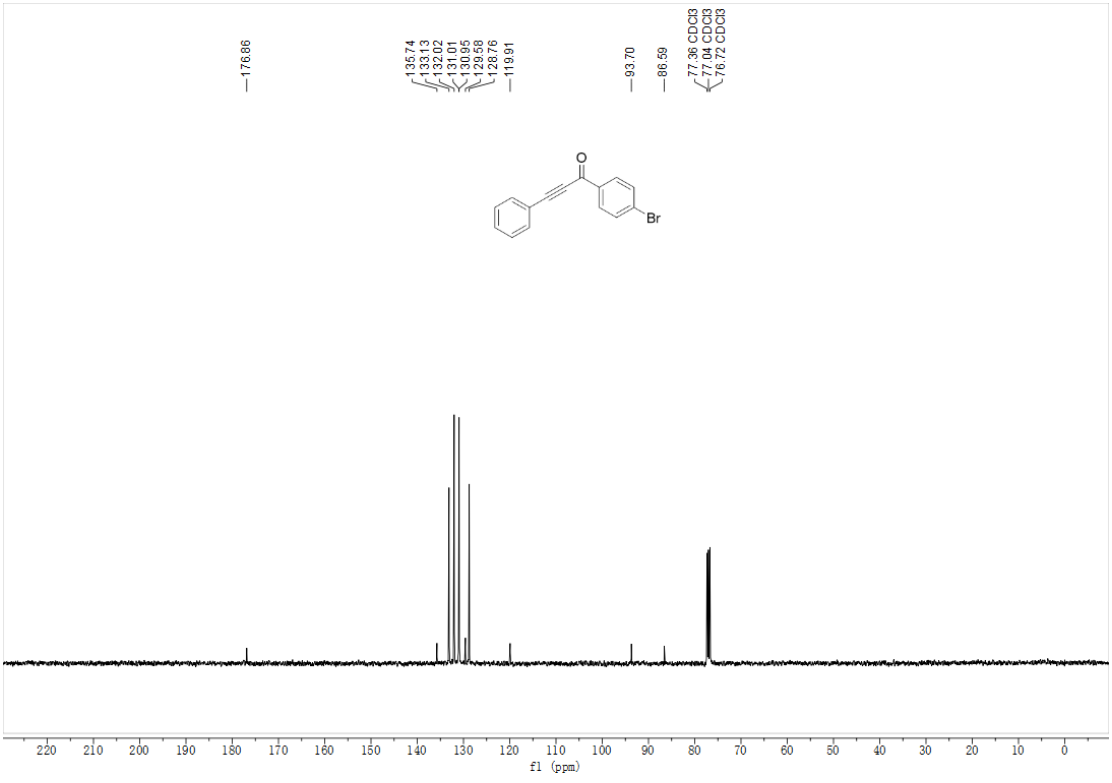
7.41
7.26
O
Br
2.00
4.06
1.15
2.13
f1 (ppm)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130.95
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128.76
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93.70
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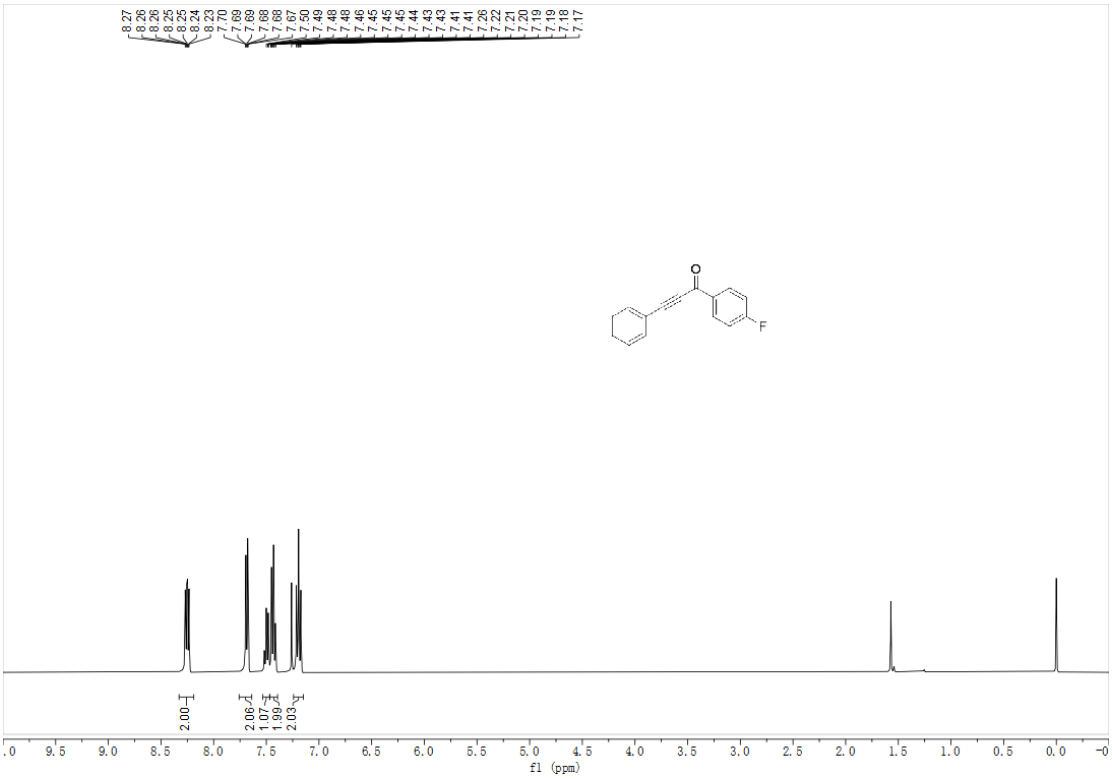
77.04 CDCl3
76.72 CDCl3
O
Br
f1 (ppm)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7.45
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7.43
7.41
7.41
7.26
7.22
7.21
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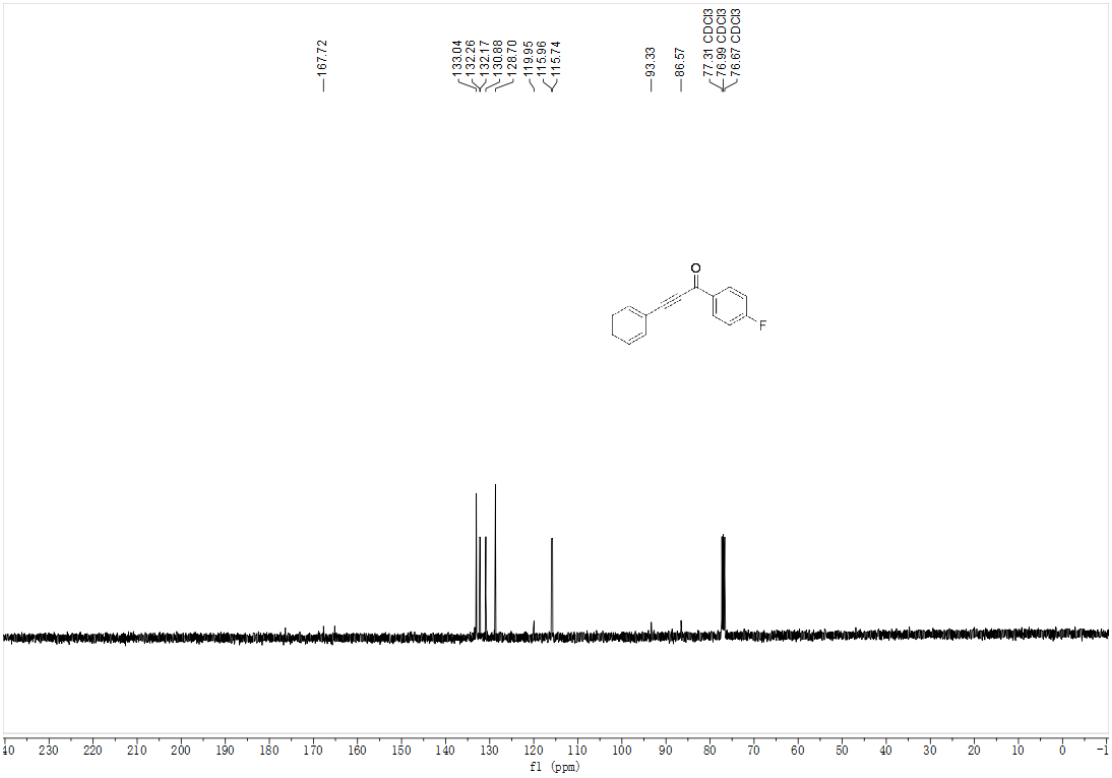
7.18
7.17
O
F
2.00
2.06
1.07
1.99
2.03
f1 (ppm)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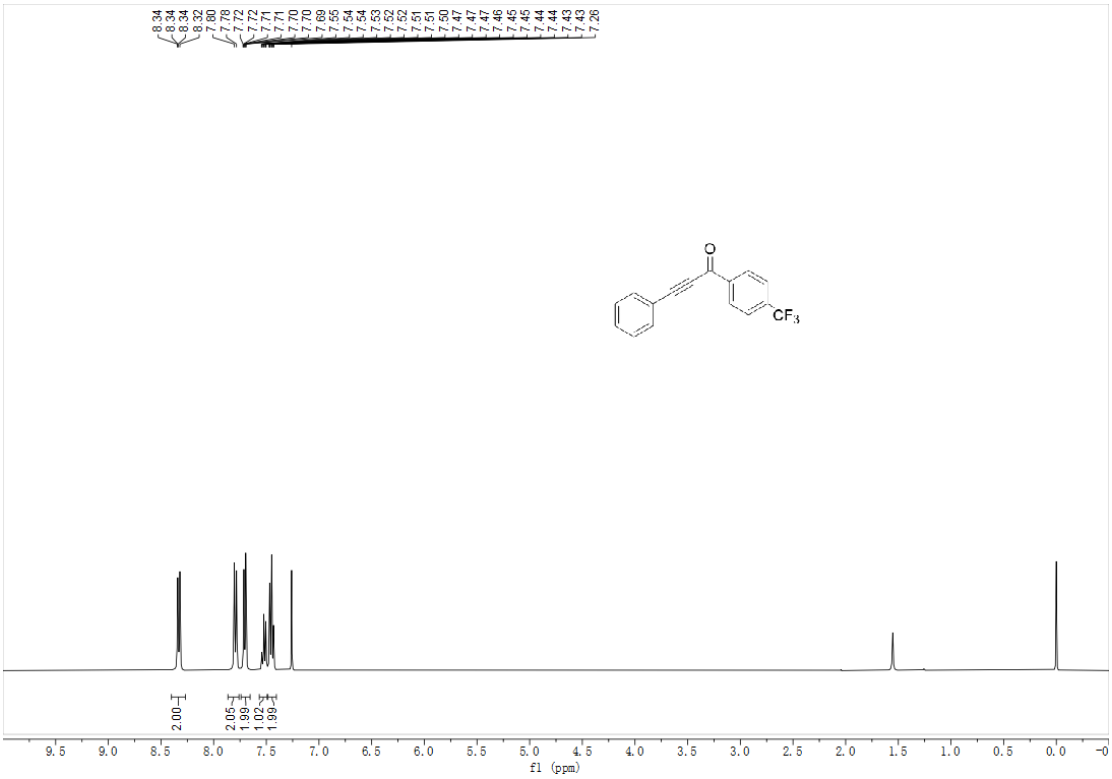
76.99 CDCl3
76.67 CDCl3
O
F
f1 (ppm)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7.50
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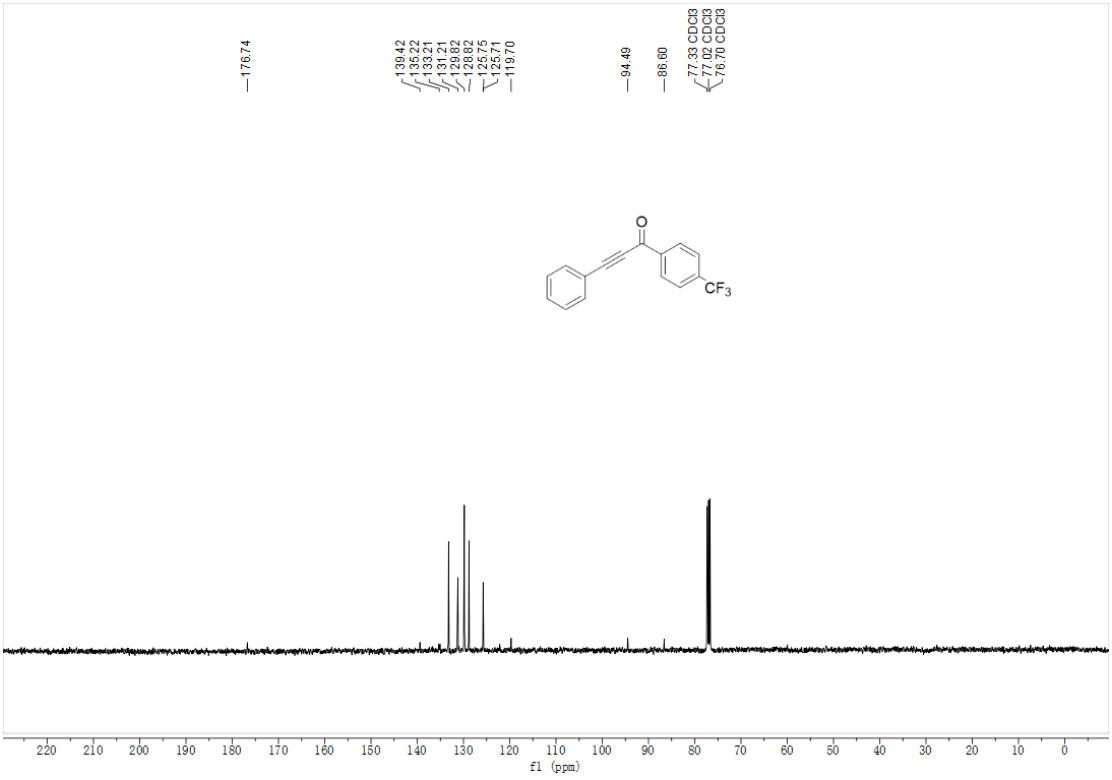
7.45
7.45
7.44
7.44
7.43
7.43
7.26
O
CF3
2.00
2.05
1.99
1.02
1.99
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)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94.49
86.60
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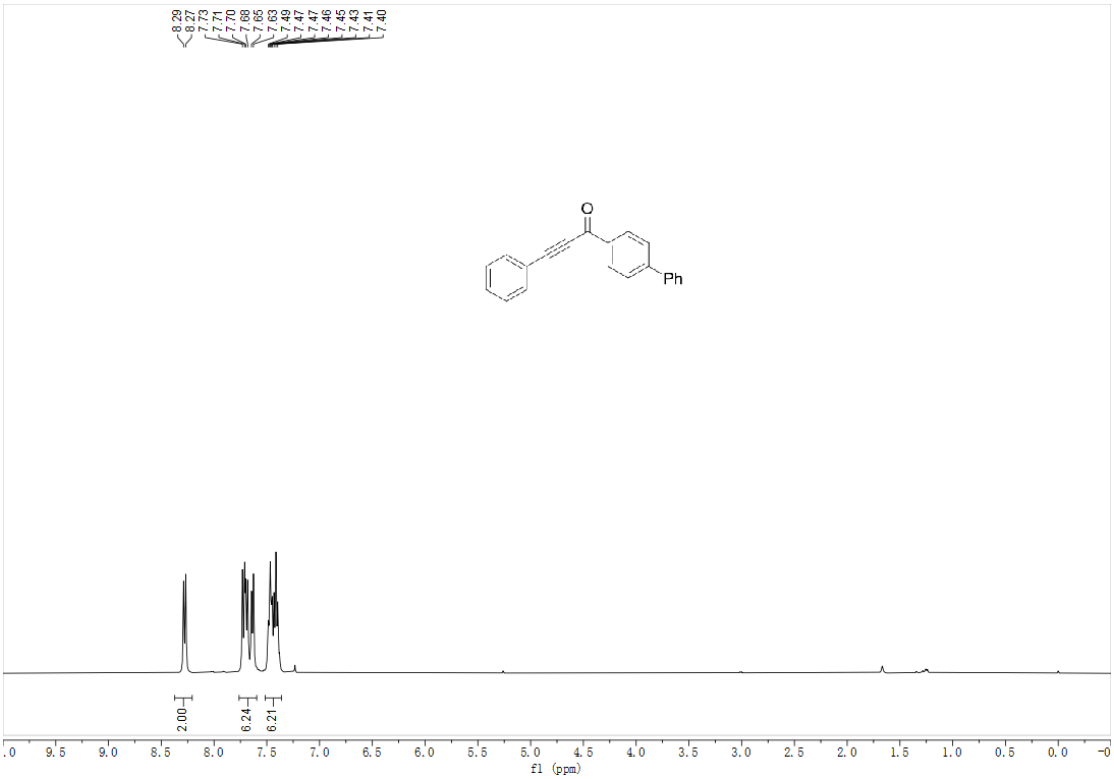
77.02 CDCl3
76.70 CDCl3
O
CF3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.47
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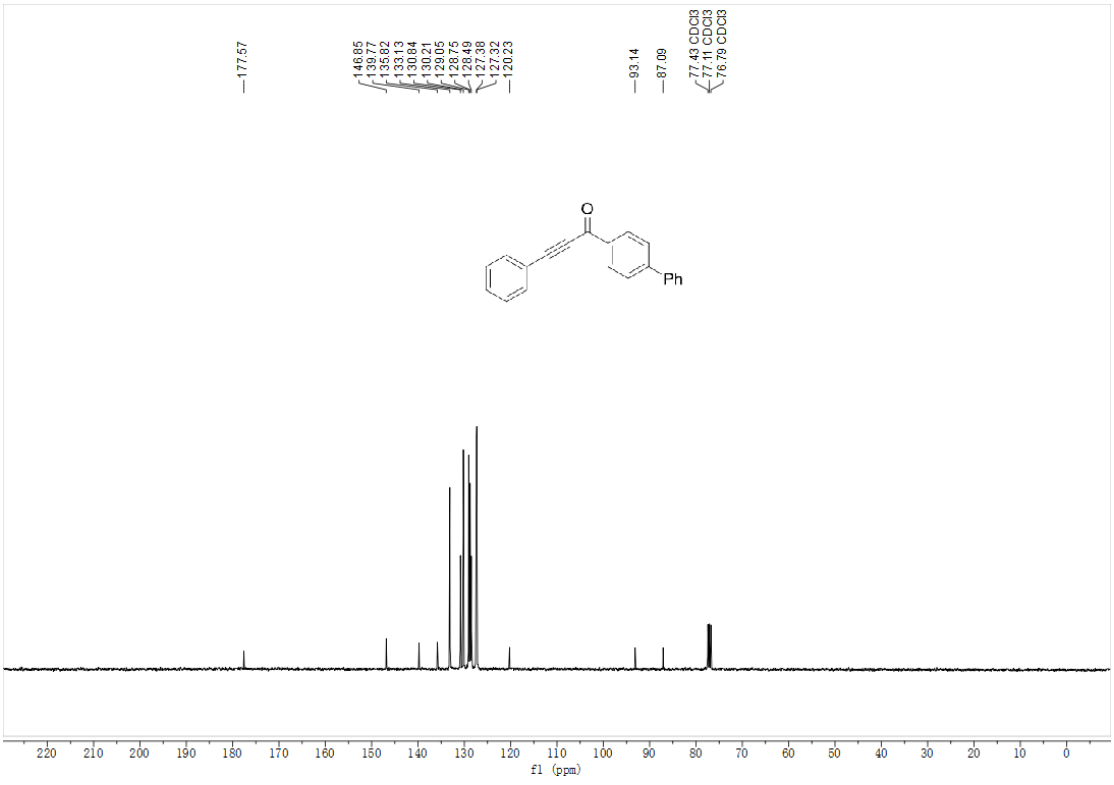
7.43
7.41
7.40
O
Ph
2.00
6.24
6.21
f1 (ppm)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128.75
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93.14
87.09
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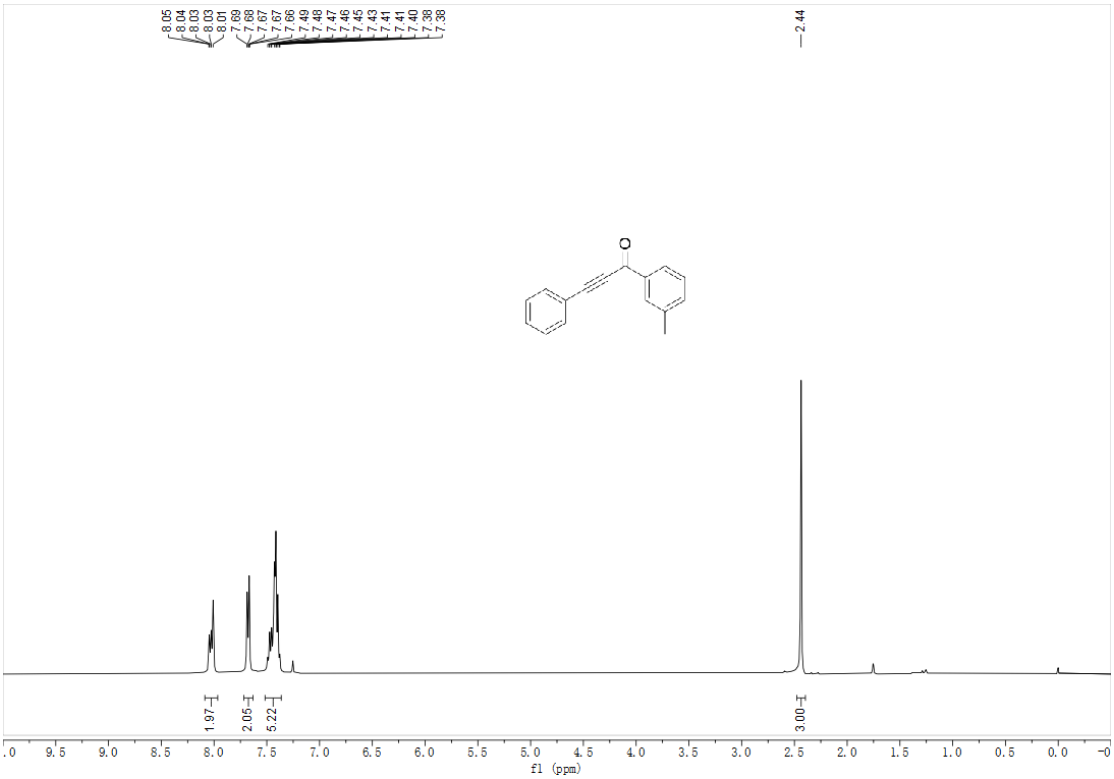
77.11 CDCl3
76.79 CDCl3
O
Ph
f1 (ppm)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7.47
7.46
7.45
7.43
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7.38
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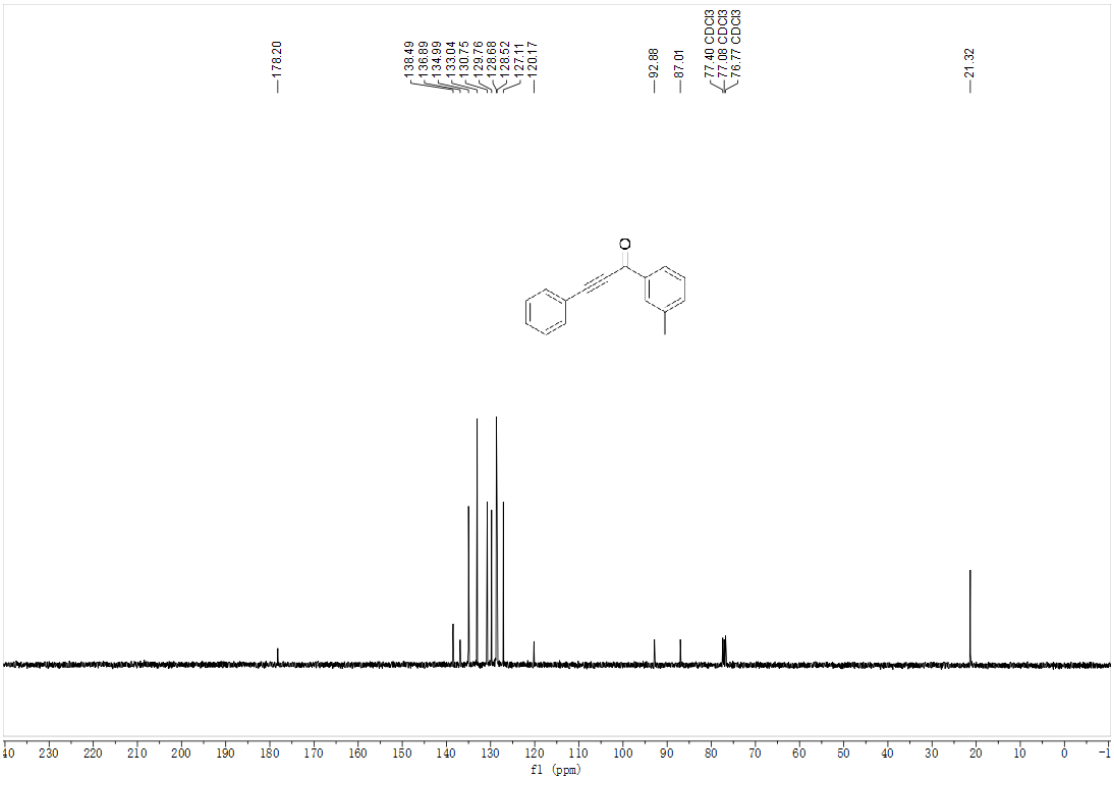
O
1.97
2.05
5.22
3.00
f1 (ppm)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128.68
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87.01
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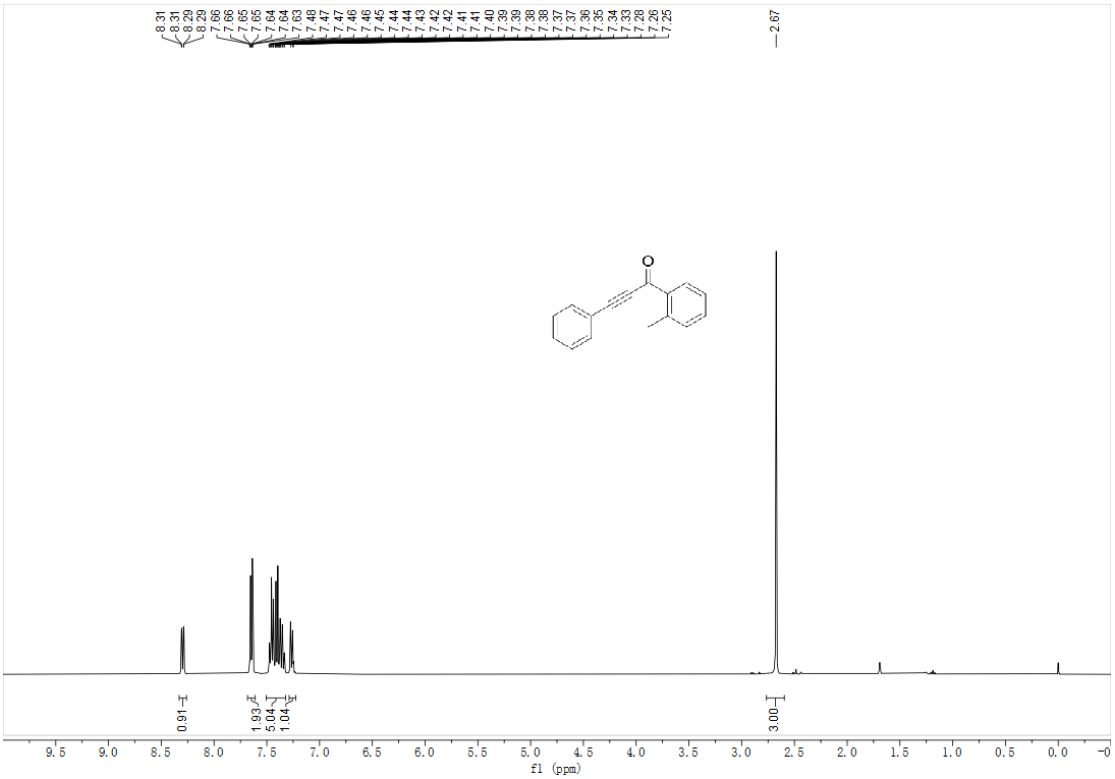
77.08 CDCl3
76.77 CDCl3
21.32
O
f1 (ppm)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7.38
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7.36
7.35
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7.25
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0.91
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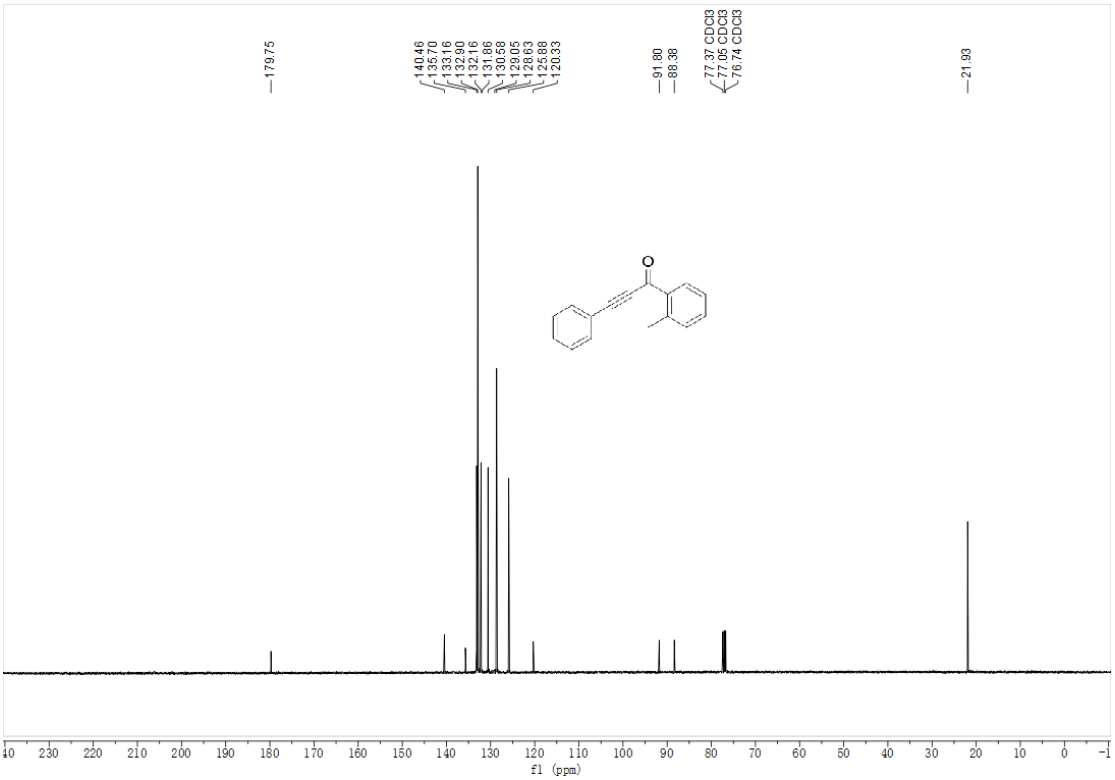
5.04
1.04
3.00
f1 (ppm)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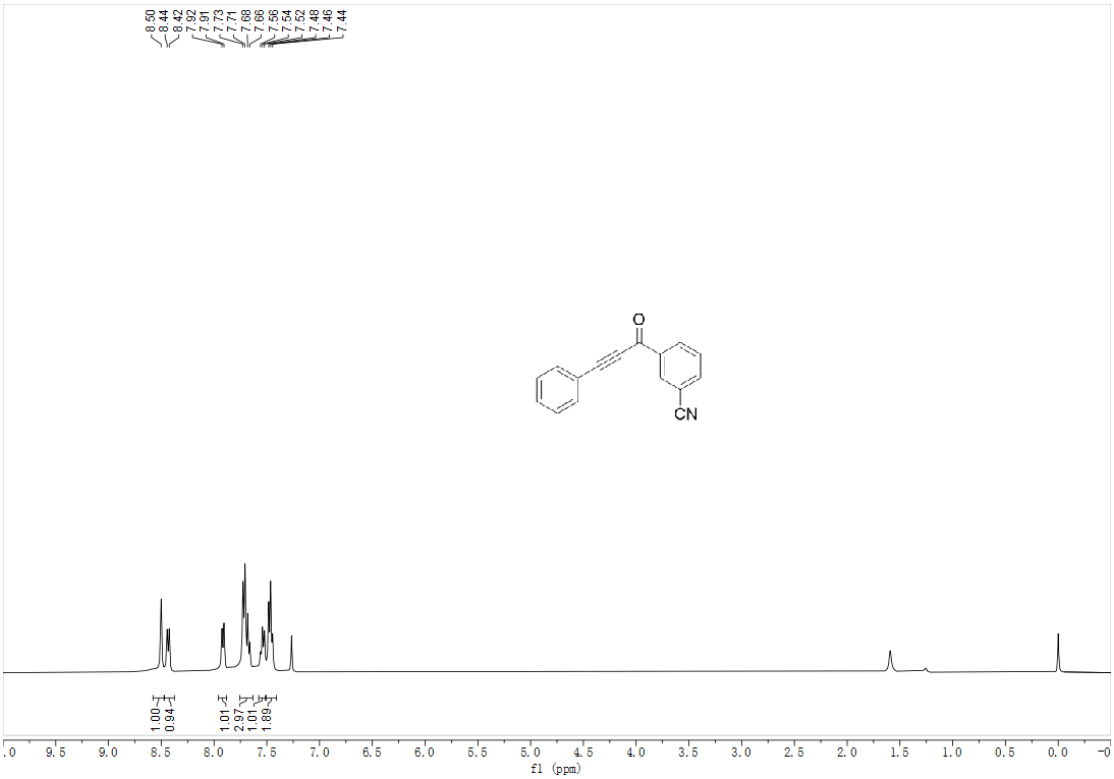
77.37 CDCl3
77.05 CDCl3
76.74 CDCl3
21.93
f1 (ppm)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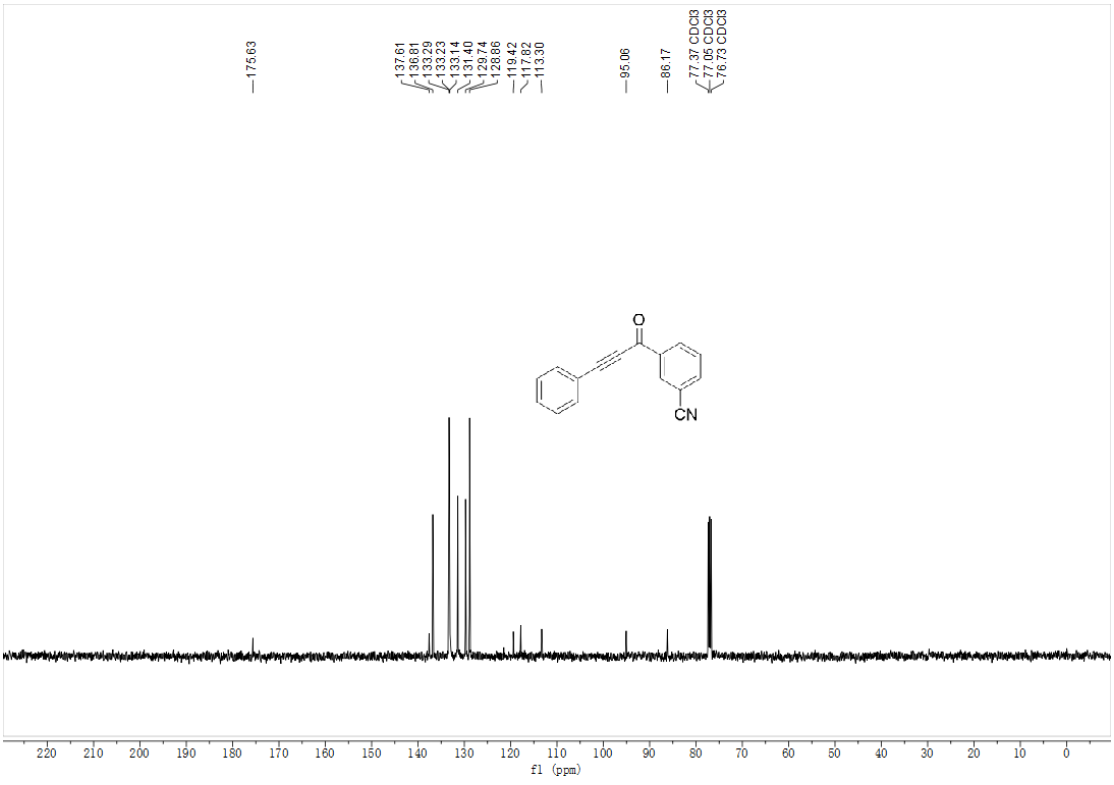
7.91
7.73
7.71
7.68
7.66
7.56
7.54
7.52
7.48
7.46
7.44
O
CN
1.00
0.94
1.01
2.97
1.01
1.89
f1 (ppm)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129.74
128.86
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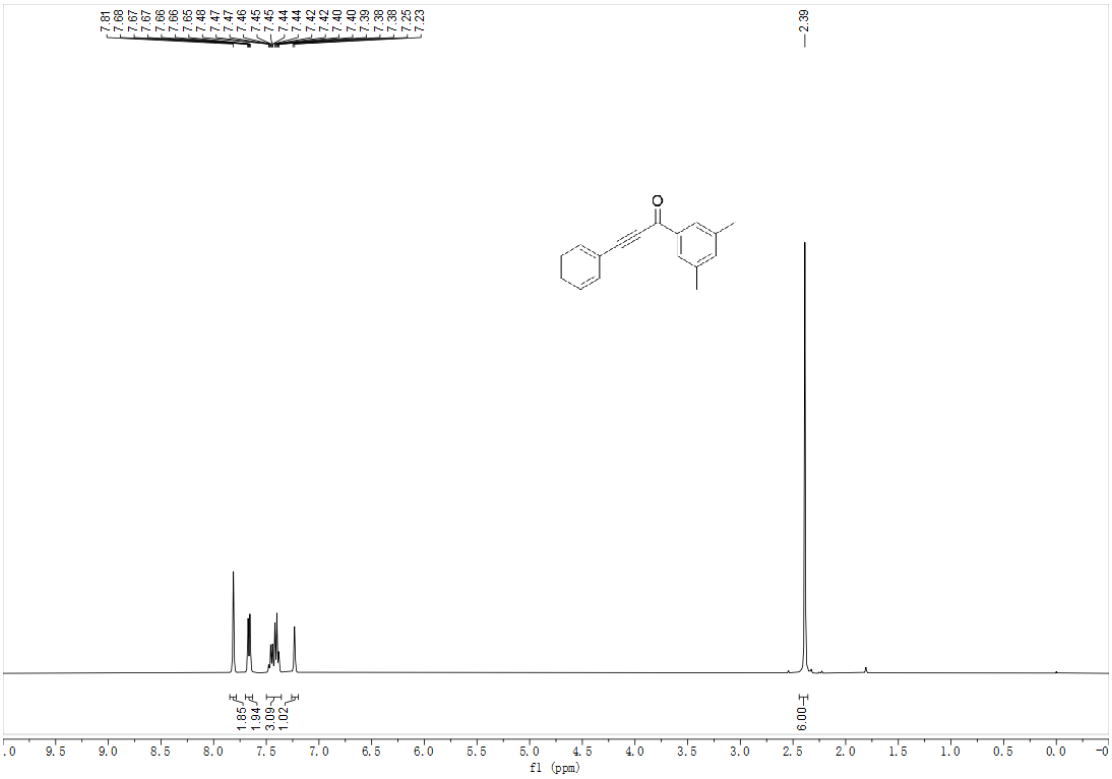
95.06
86.17
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
O
CN
f1 (ppm)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7.45
7.44
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7.42
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7.40
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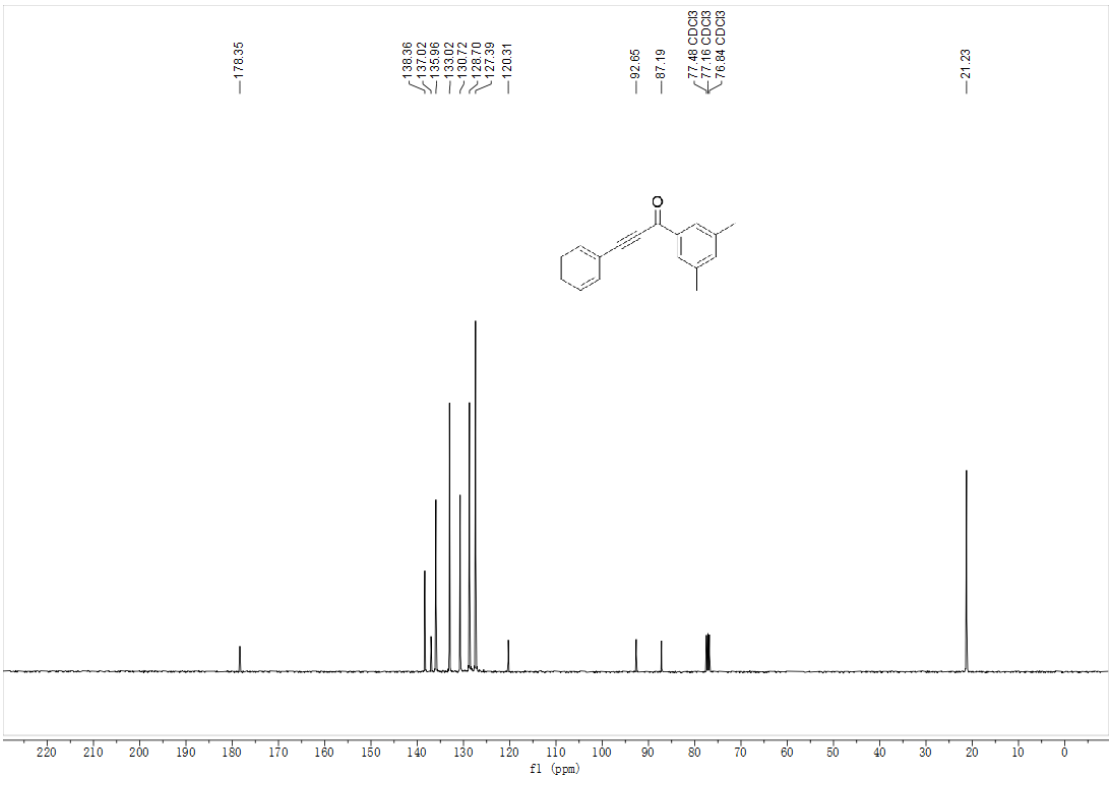
1.94
3.09
1.02
6.00
f1 (ppm)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133.02
130.72
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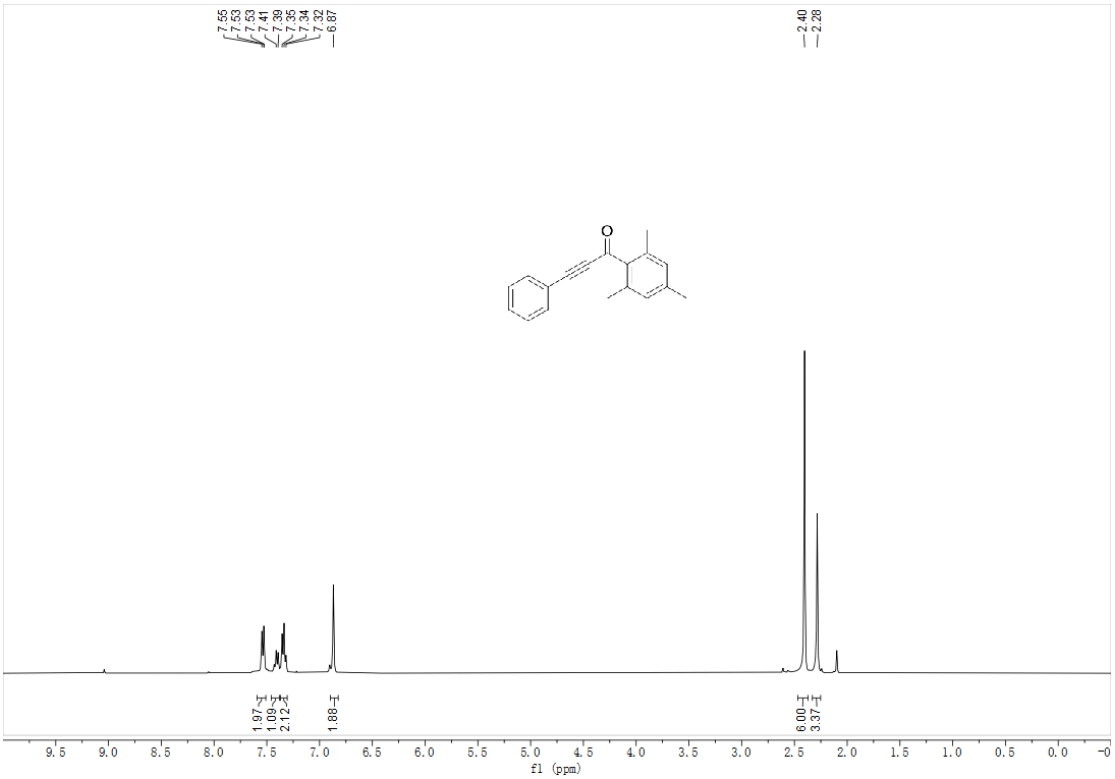
127.39
120.31
92.65
87.19
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
21.23
f1 (ppm)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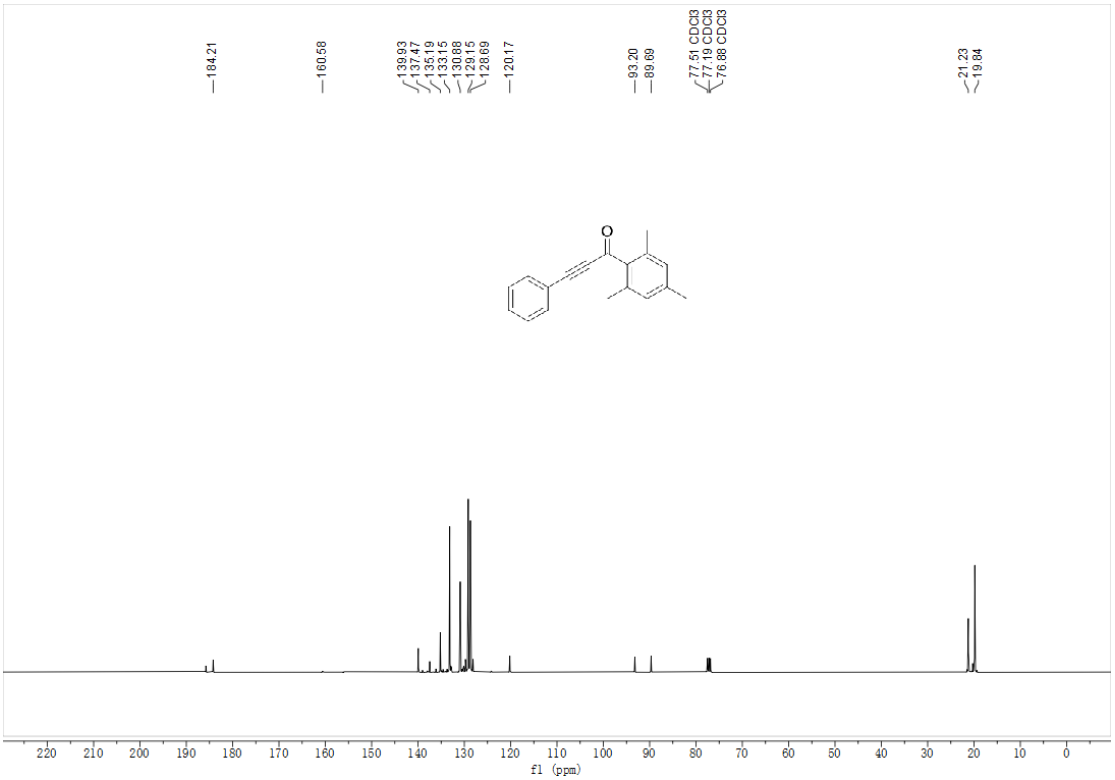
6.00
3.37
f1 (ppm)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128.69
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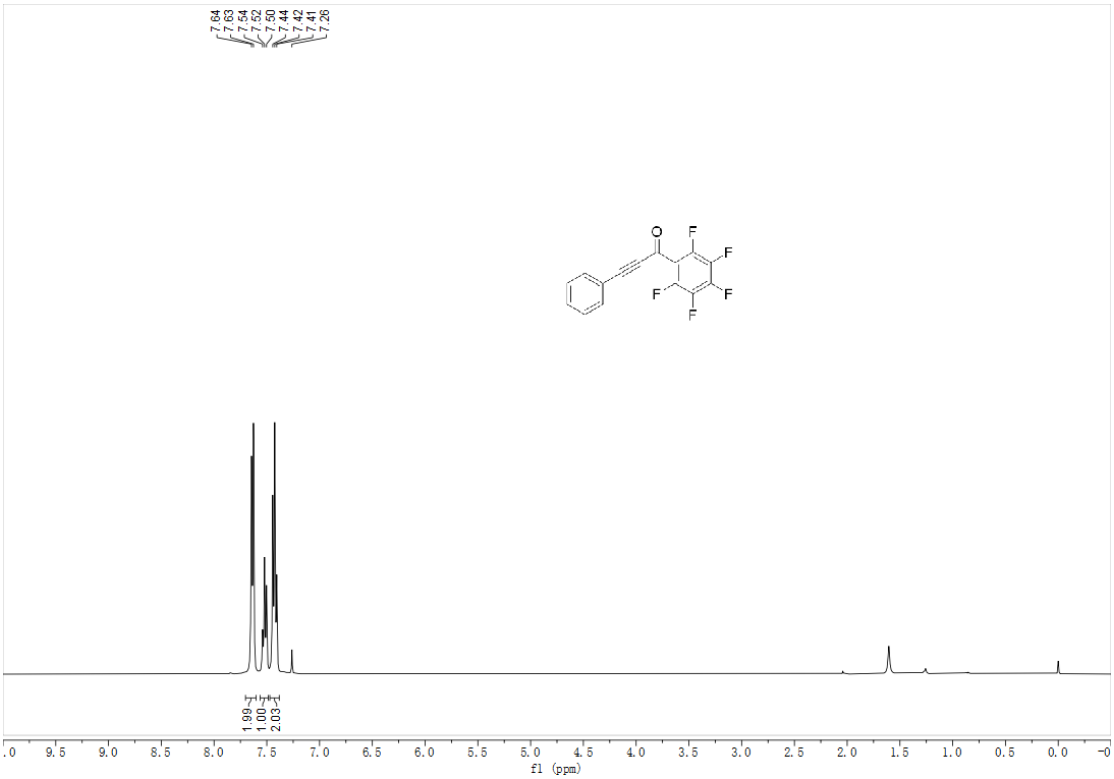
21.23
19.84
O
f1 (ppm)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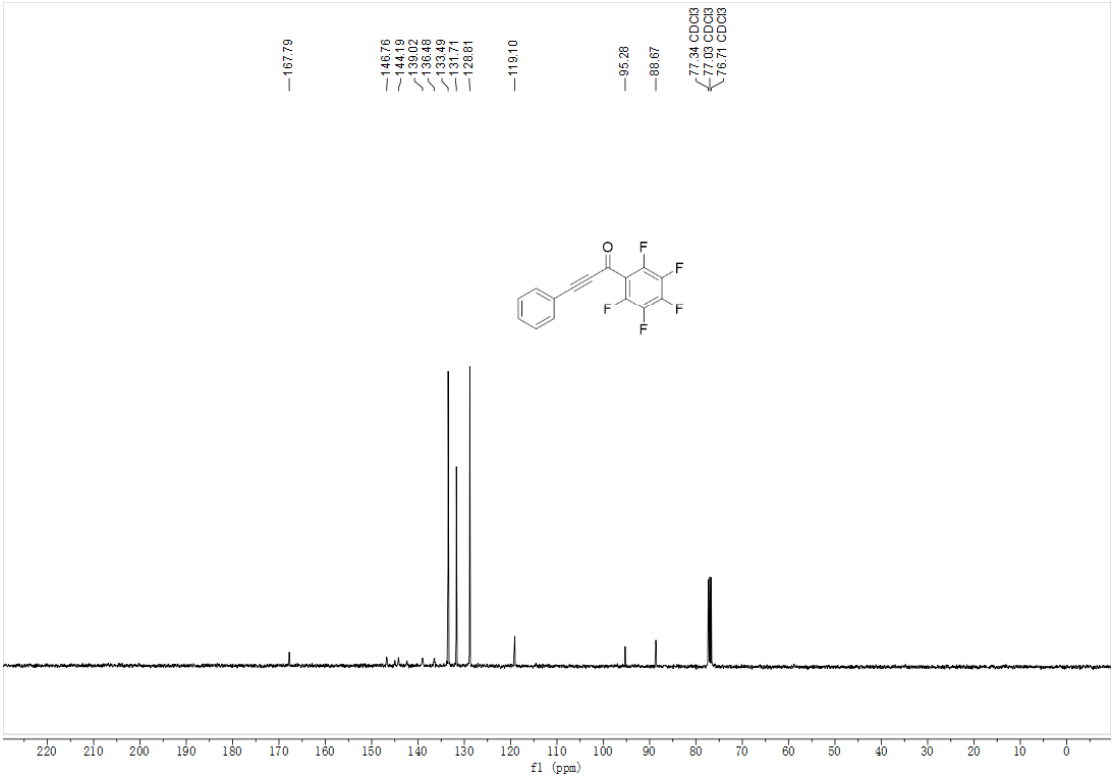
7.41
7.26
O
F
F
F
F
F
1.99
1.00
2.03
f1 (ppm)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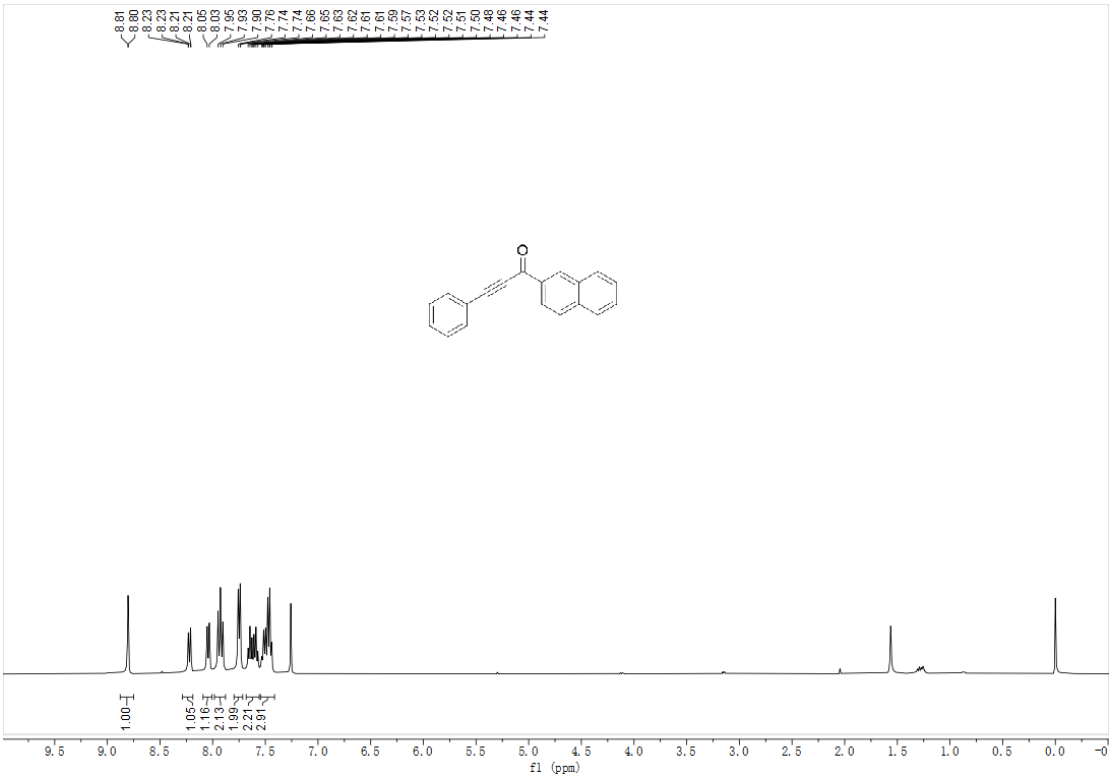
77.34 CDCl3
77.03 CDCl3
76.71 CDCl3
O
F
F
F
F
F
f1 (ppm)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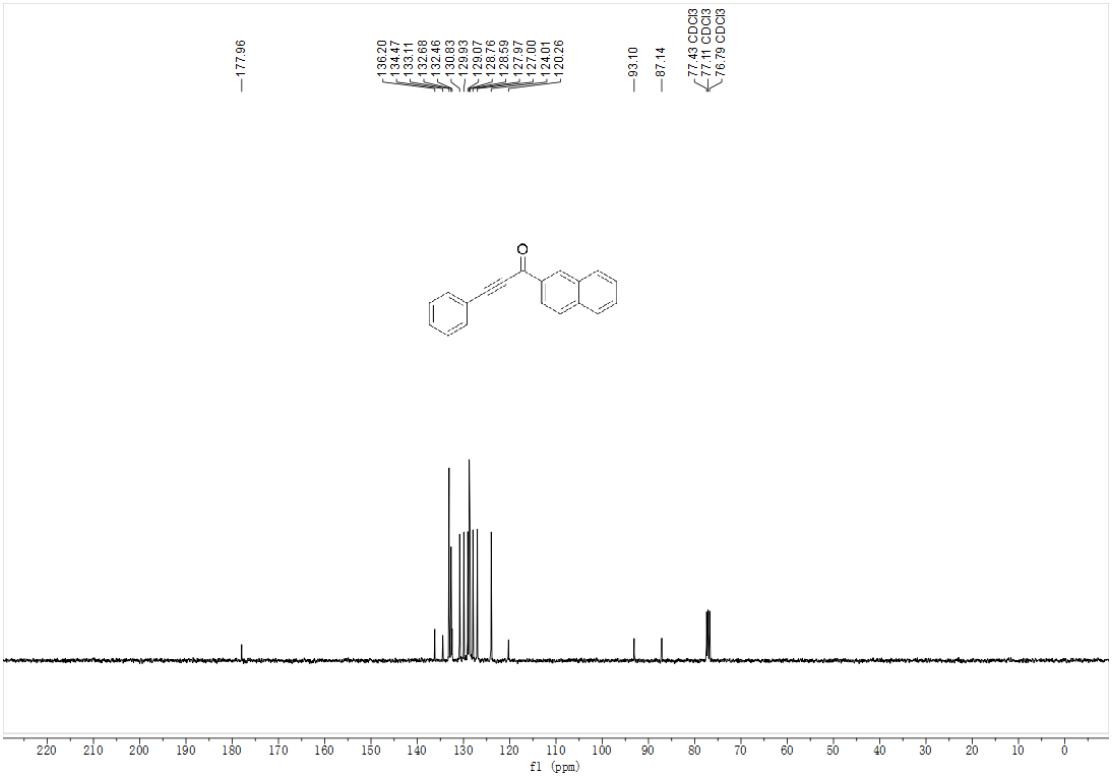
2.91
f1 (ppm)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120.26
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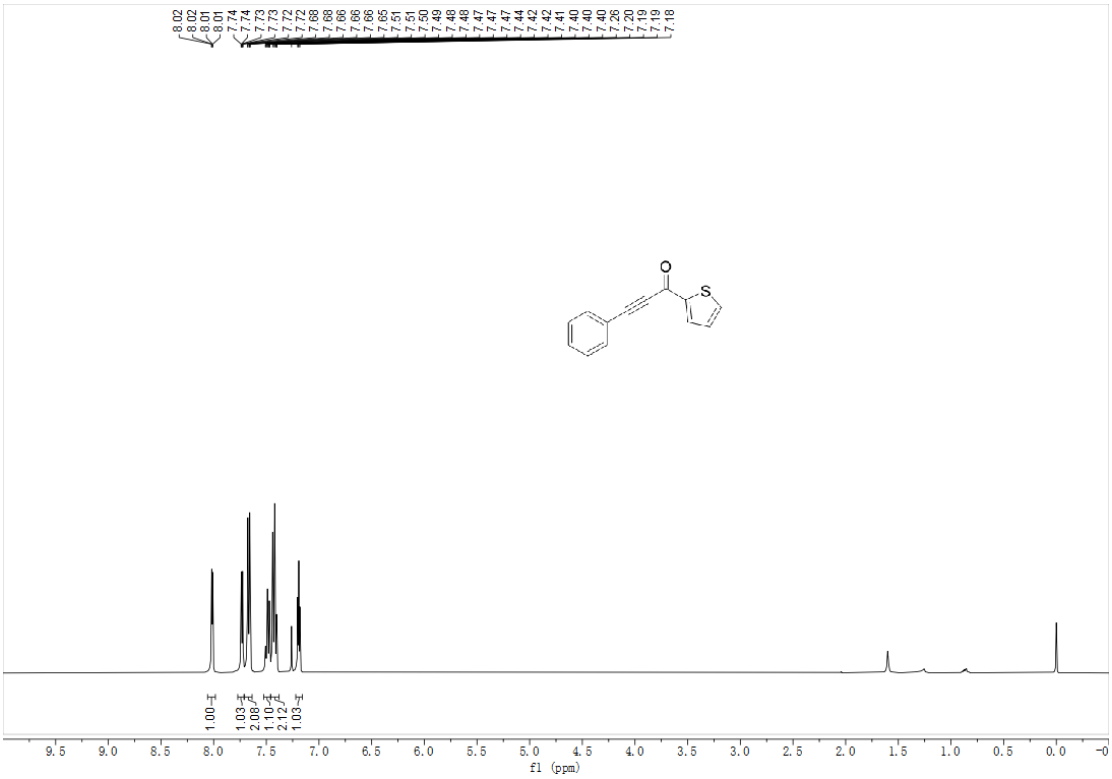
87.14
77.43 CDCl3
77.11 CDCl3
76.79 CDCl3
f1 (ppm)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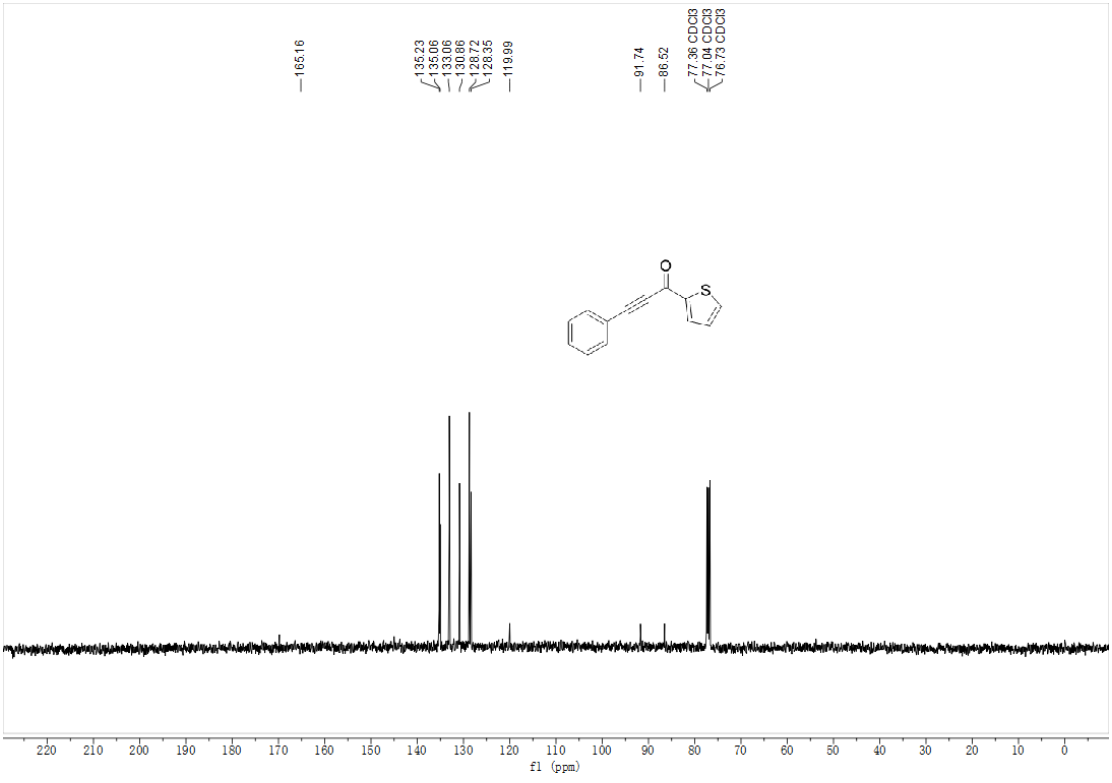
7.26
7.20
7.19
7.19
7.18
O
S
1.00
1.03
2.08
1.10
2.12
1.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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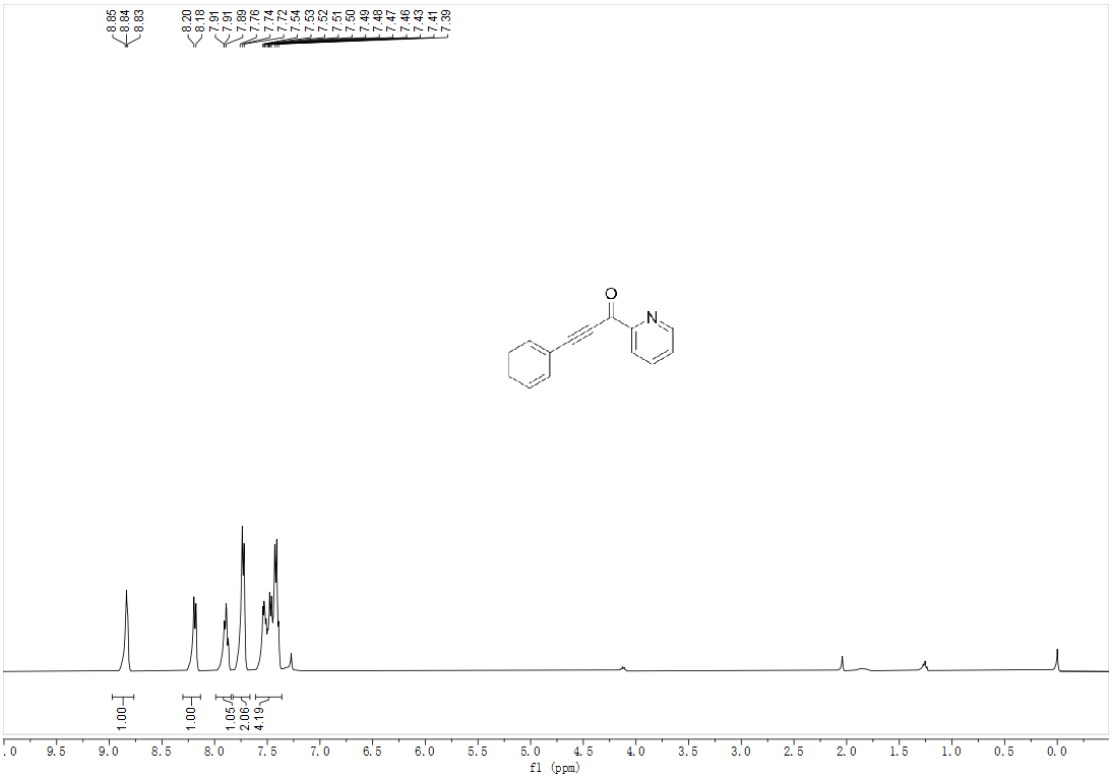
119.99
91.74
86.52
77.36 CDCl3
77.04 CDCl3
76.73 CDCl3
O
S
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.52
7.51
7.50
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7.48
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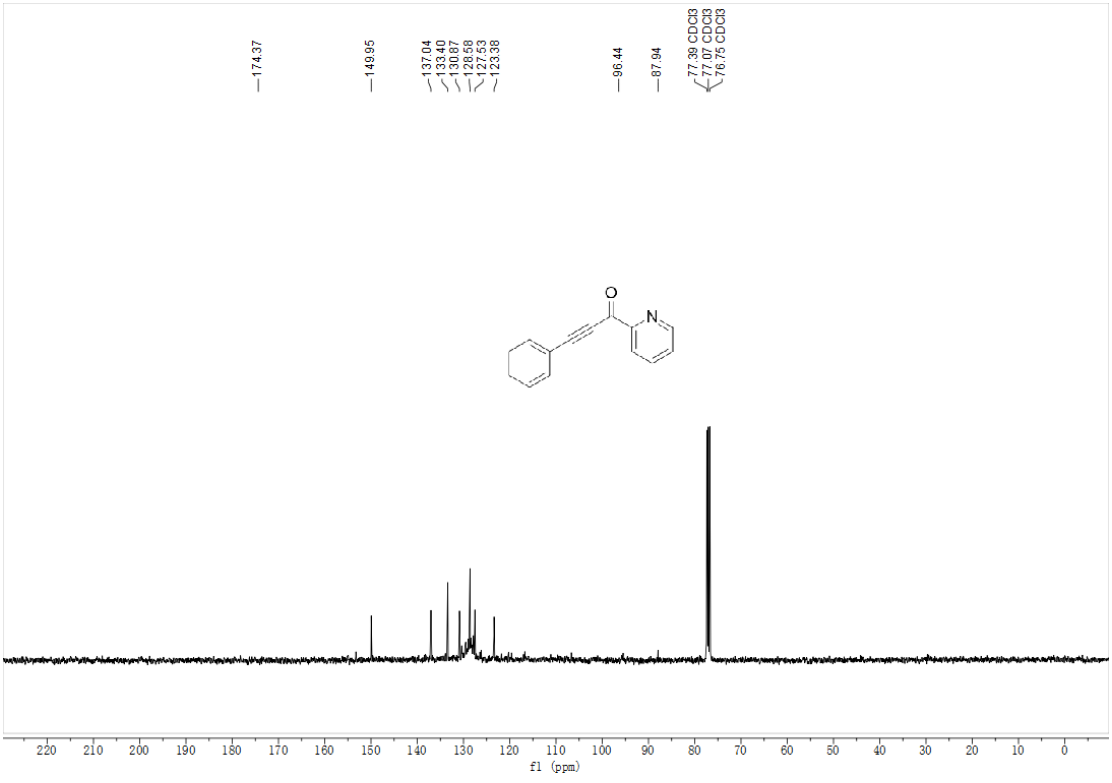
4.19
f1 (ppm)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87.94
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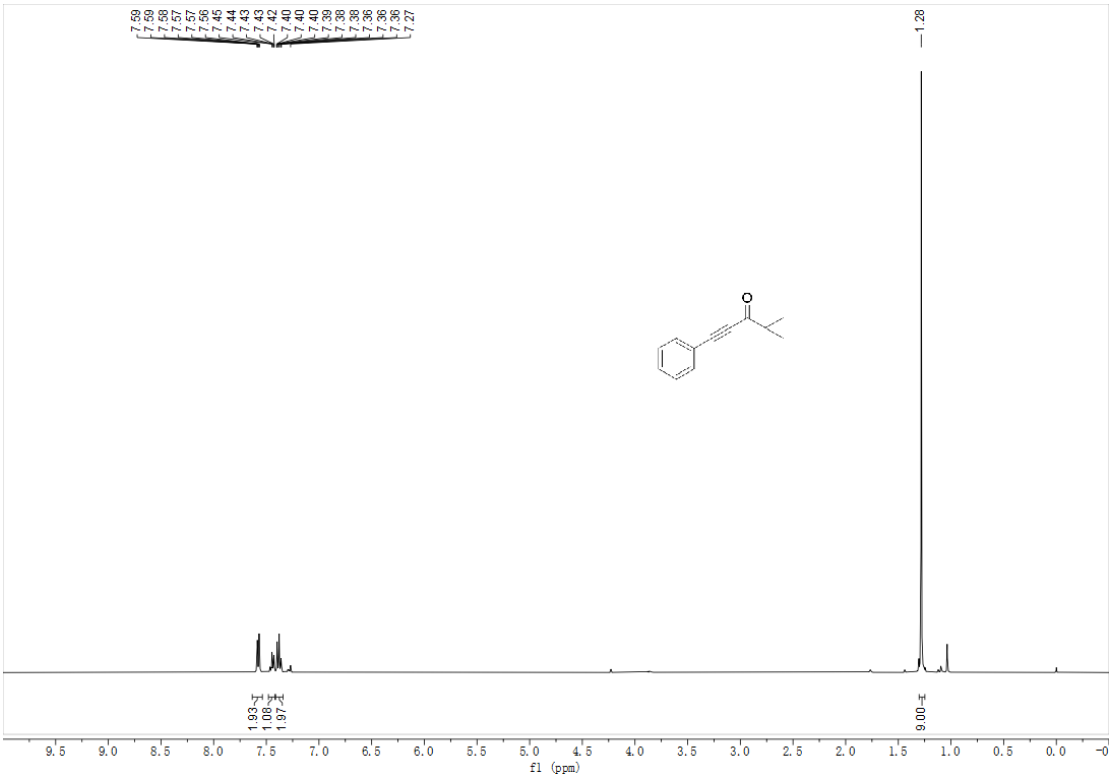
77.07 CDCl3
76.75 CDCl3
O
N
f1 (ppm)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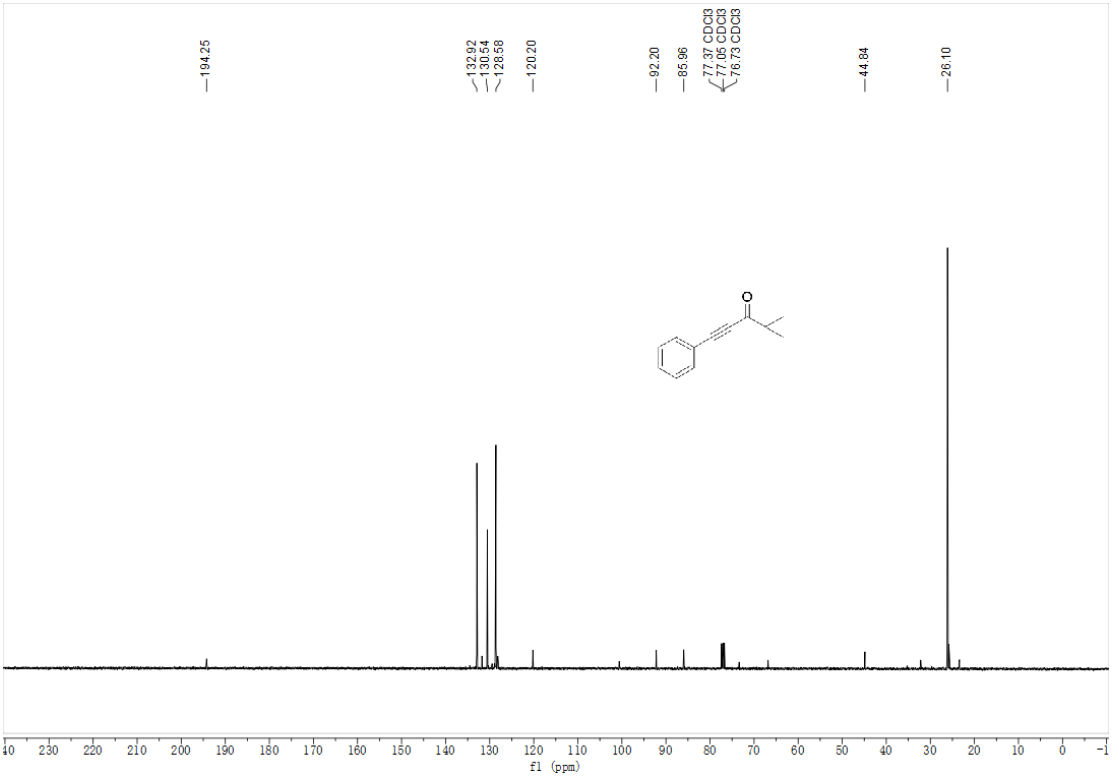
1.93
1.08
1.97
9.00
f1 (ppm)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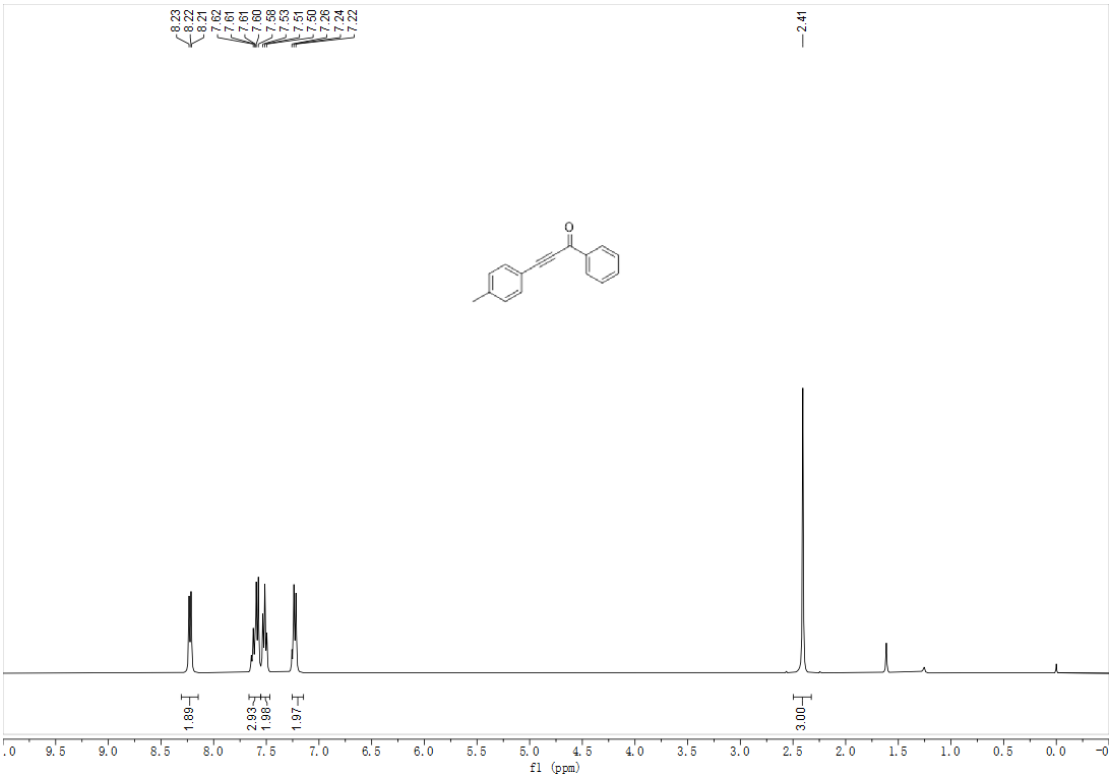
77.05 CDCl3
76.73 CDCl3
44.84
26.10
f1 (ppm)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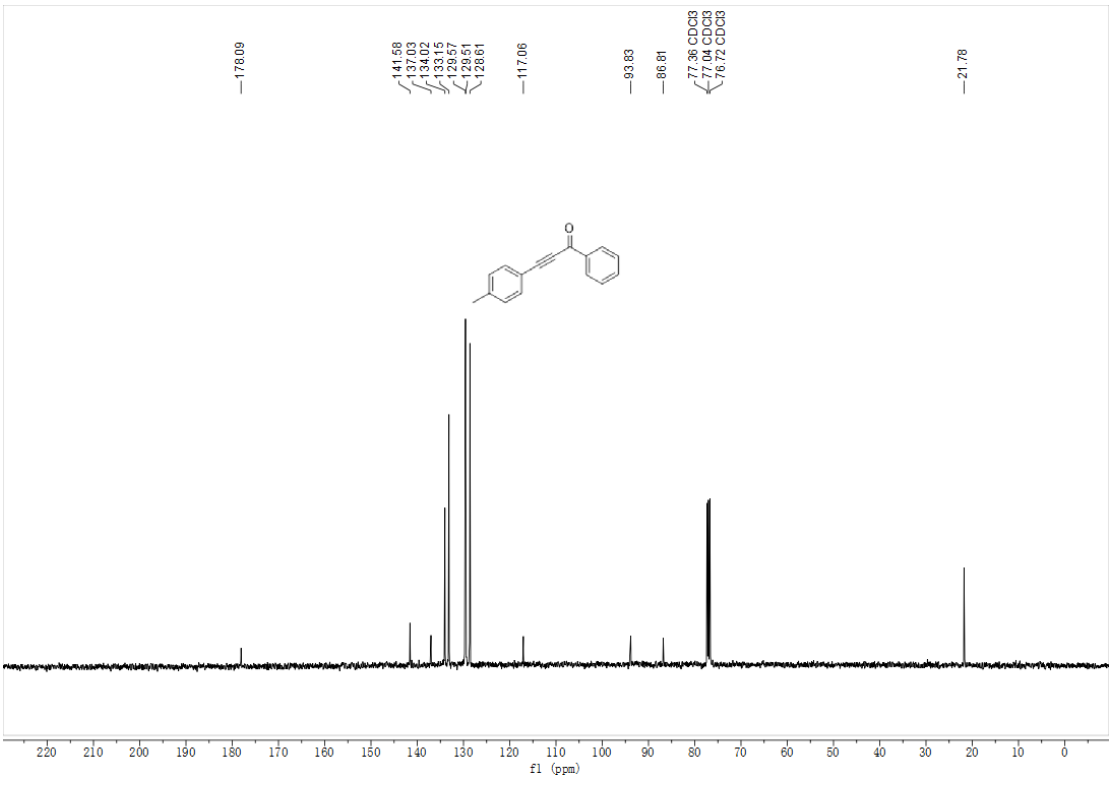
1.98
1.97
3.00
f1 (ppm)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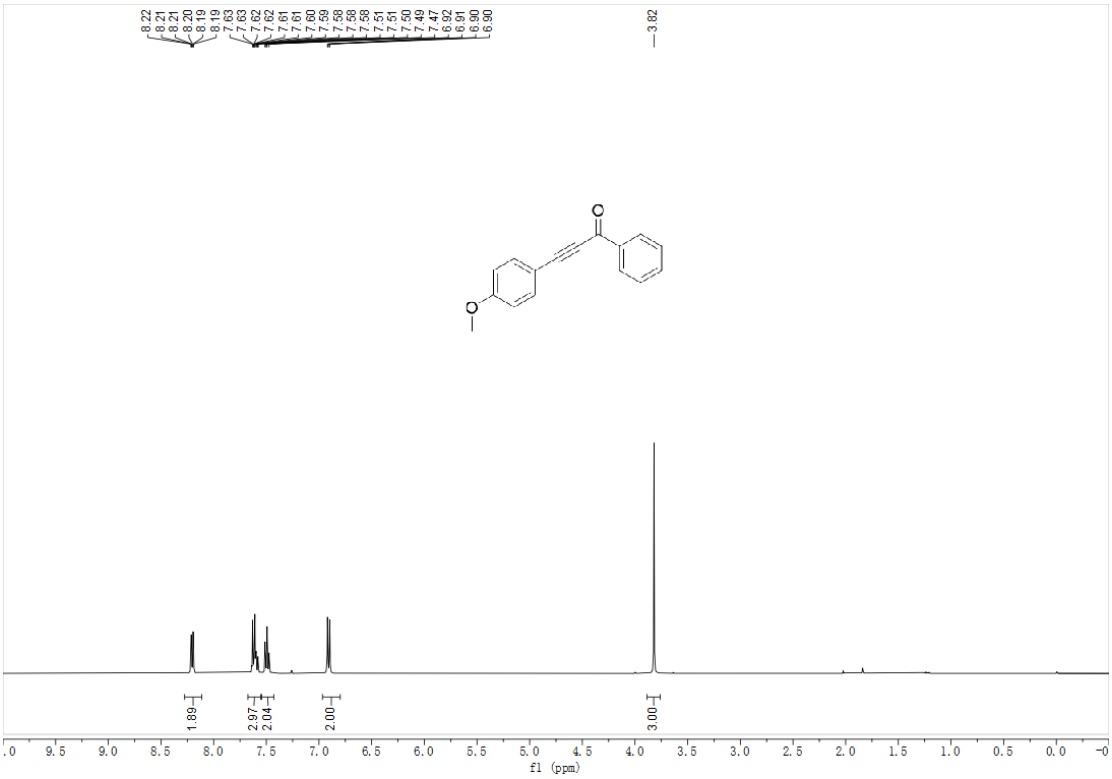
77.36 CDCl3
77.04 CDCl3
76.72 CDCl3
21.78
f1 (ppm)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7.47
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6.90
6.90
3.82
O
O
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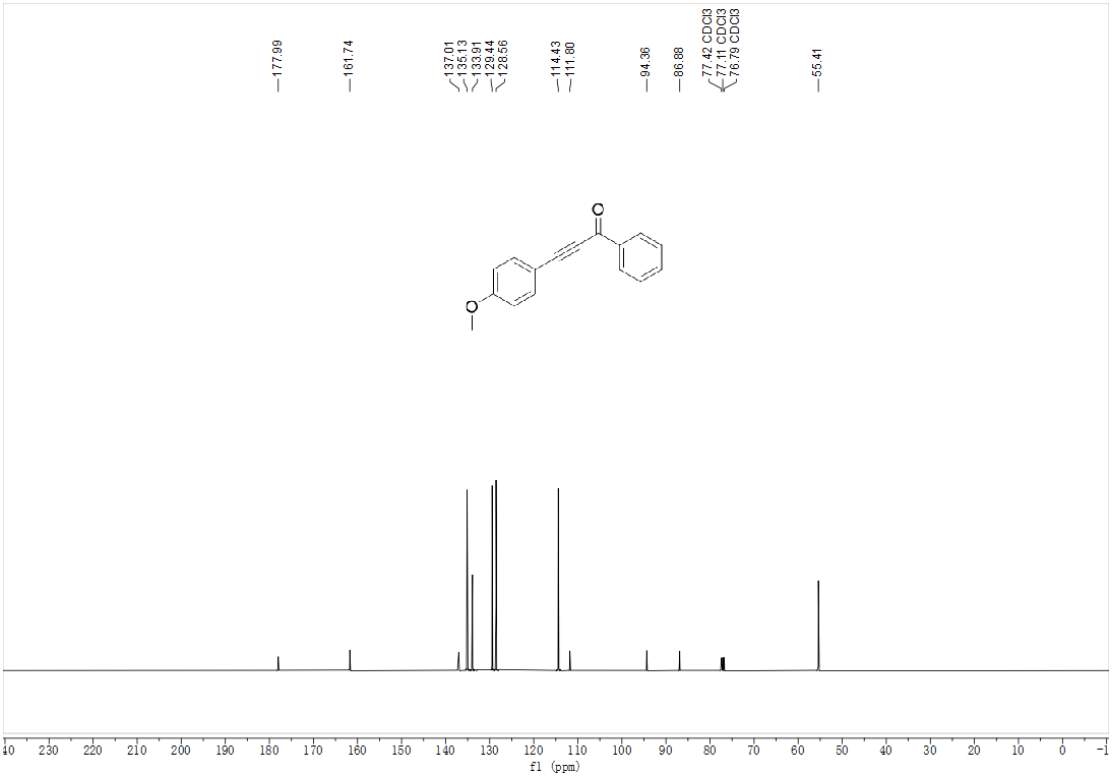
2.97
2.04
2.00
3.00
f1 (ppm)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128.56
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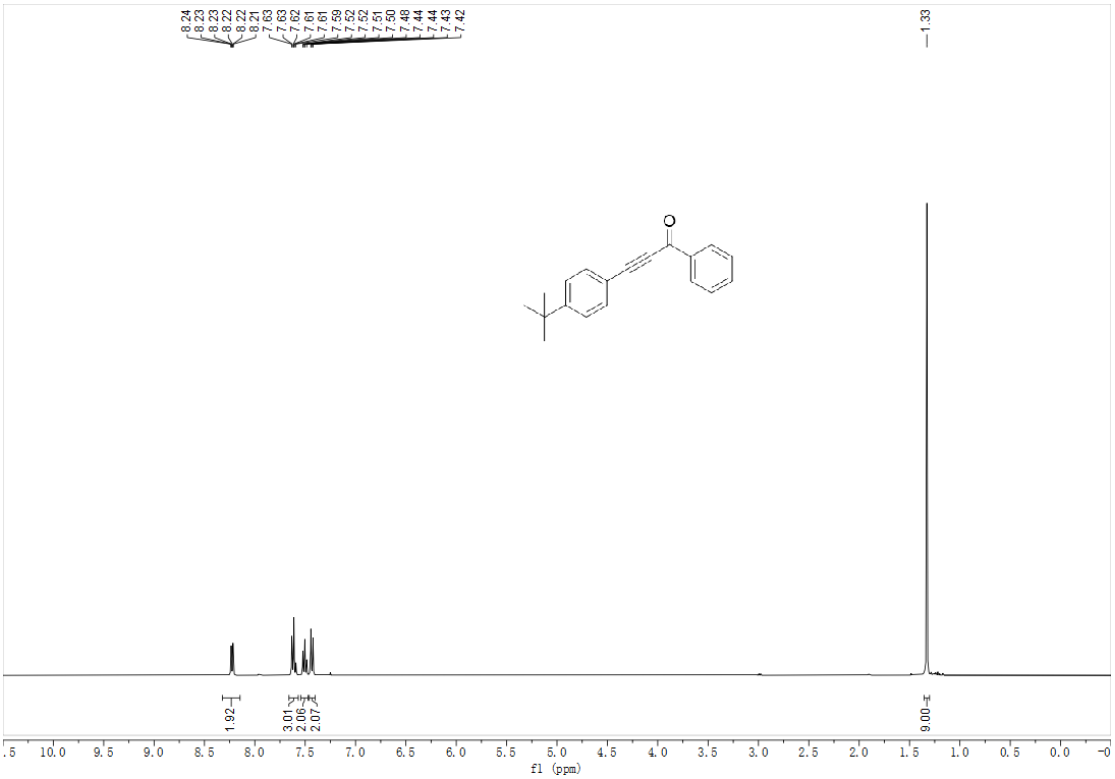
77.11 CDCl3
76.79 CDCl3
55.41
O
O
f1 (ppm)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7.51
7.50
7.48
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7.43
7.42
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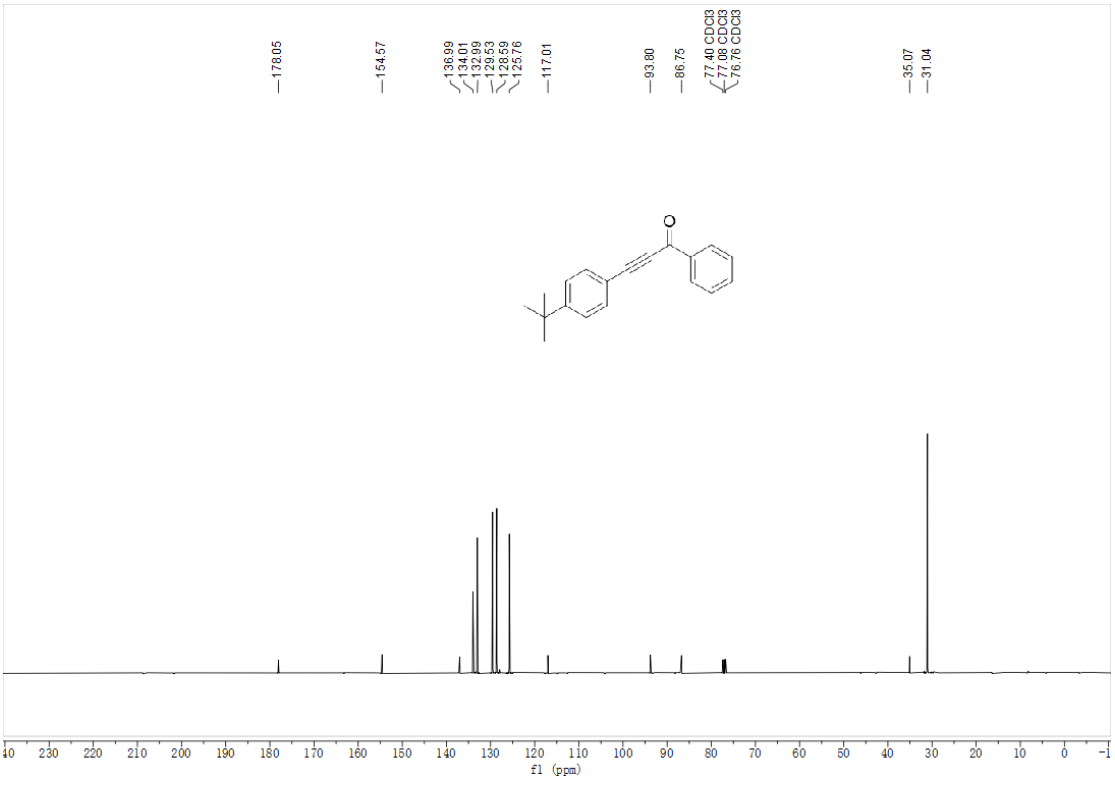
2.07
9.00
f1 (ppm)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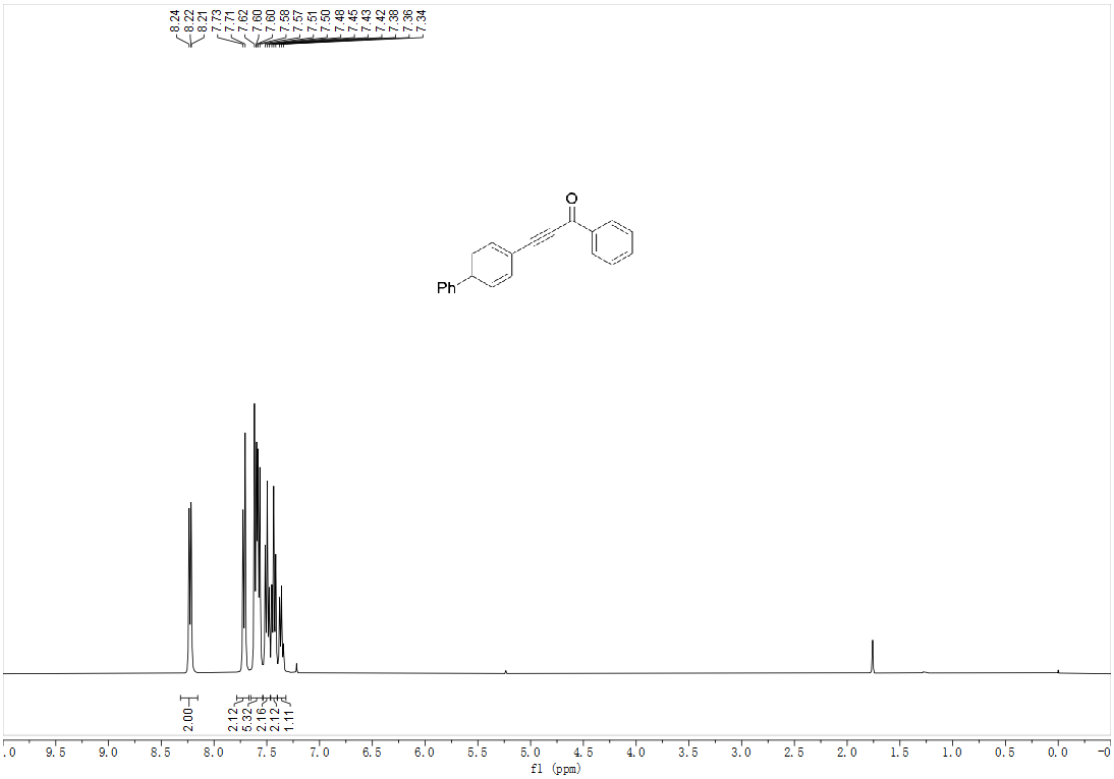
77.08 CDCl3
76.76 CDCl3
35.07
31.04
O
f1 (ppm)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7.45
7.43
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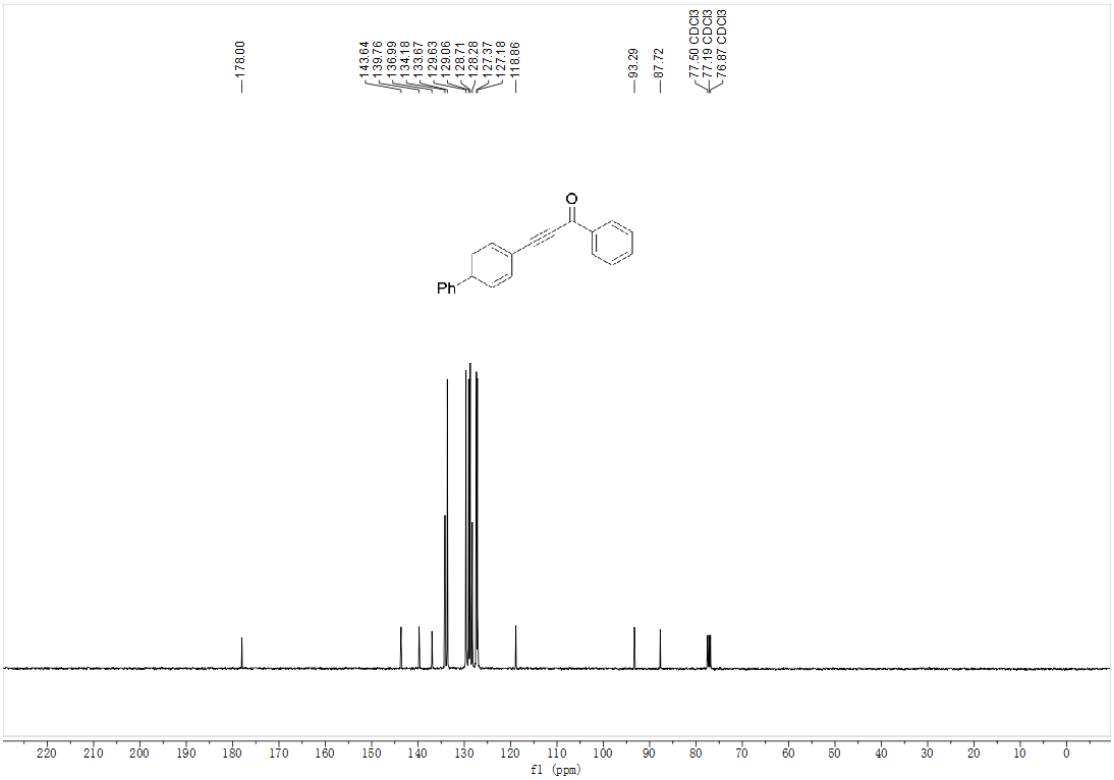
1.11
f1 (ppm)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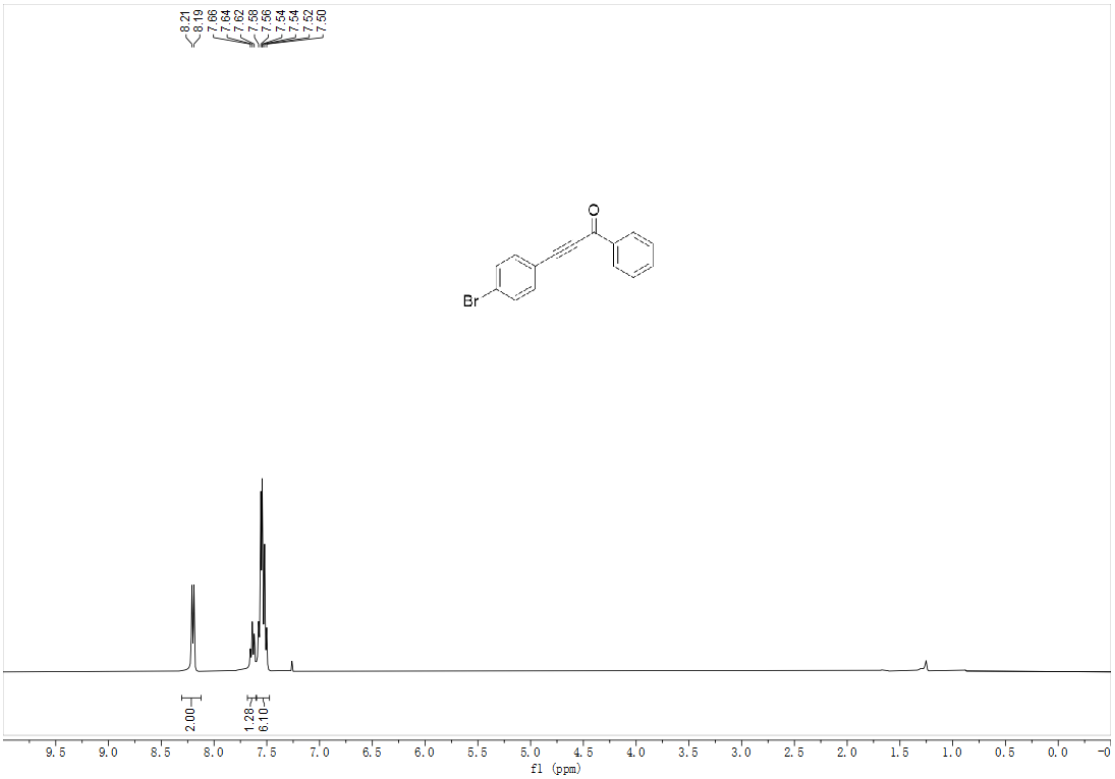
76.87 CDCl3
O
Ph
f1 (ppm)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7.52
7.50
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Br
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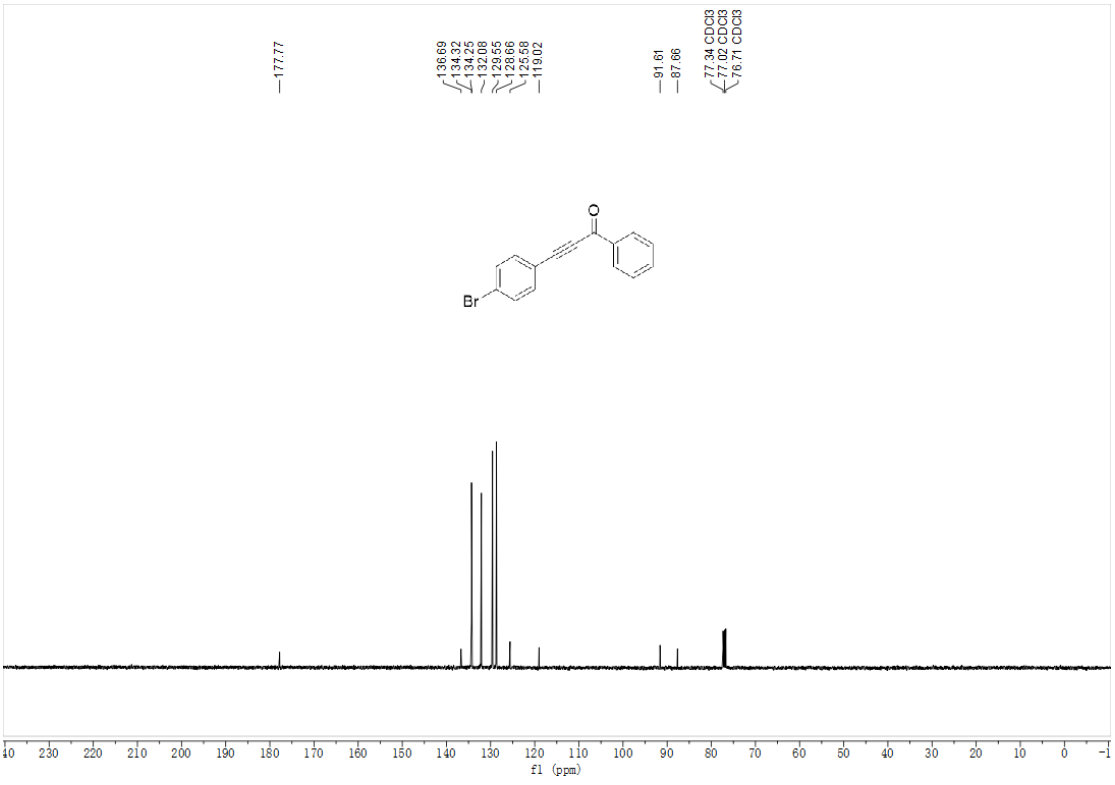
6.10
f1 (ppm)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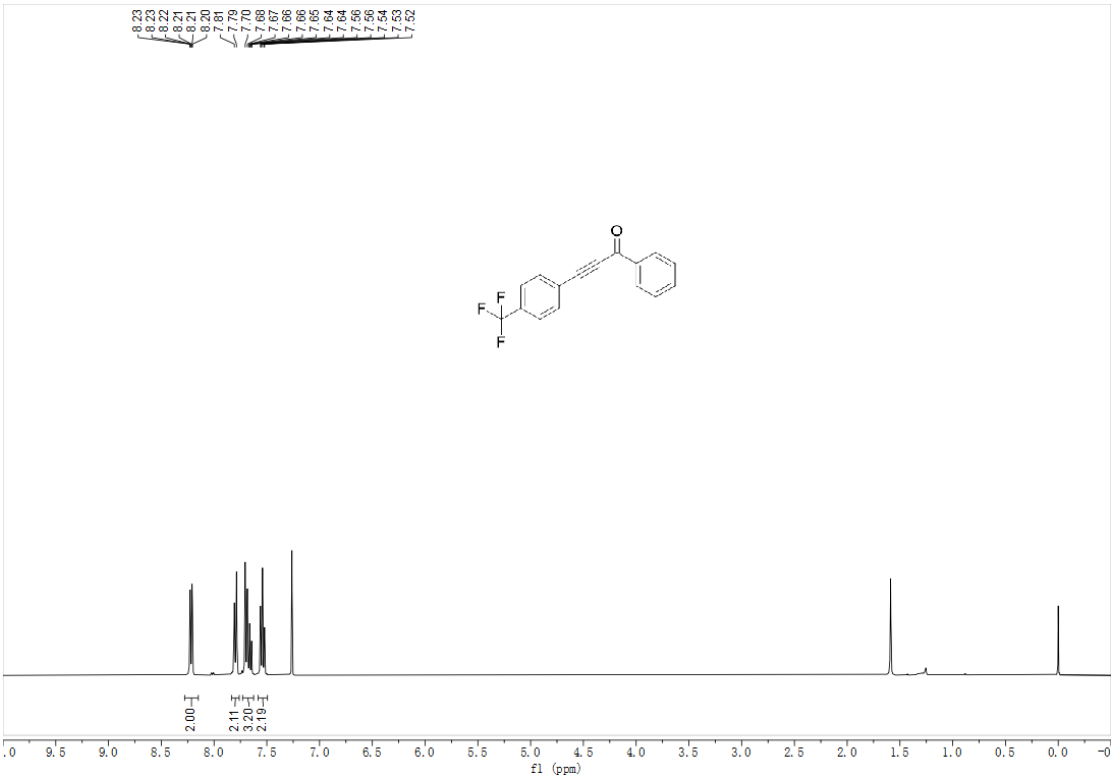
77.02 CDCl3
76.71 CDCl3
O
Br
f1 (ppm)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7.56
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7.52
O
F
F
F
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2.11
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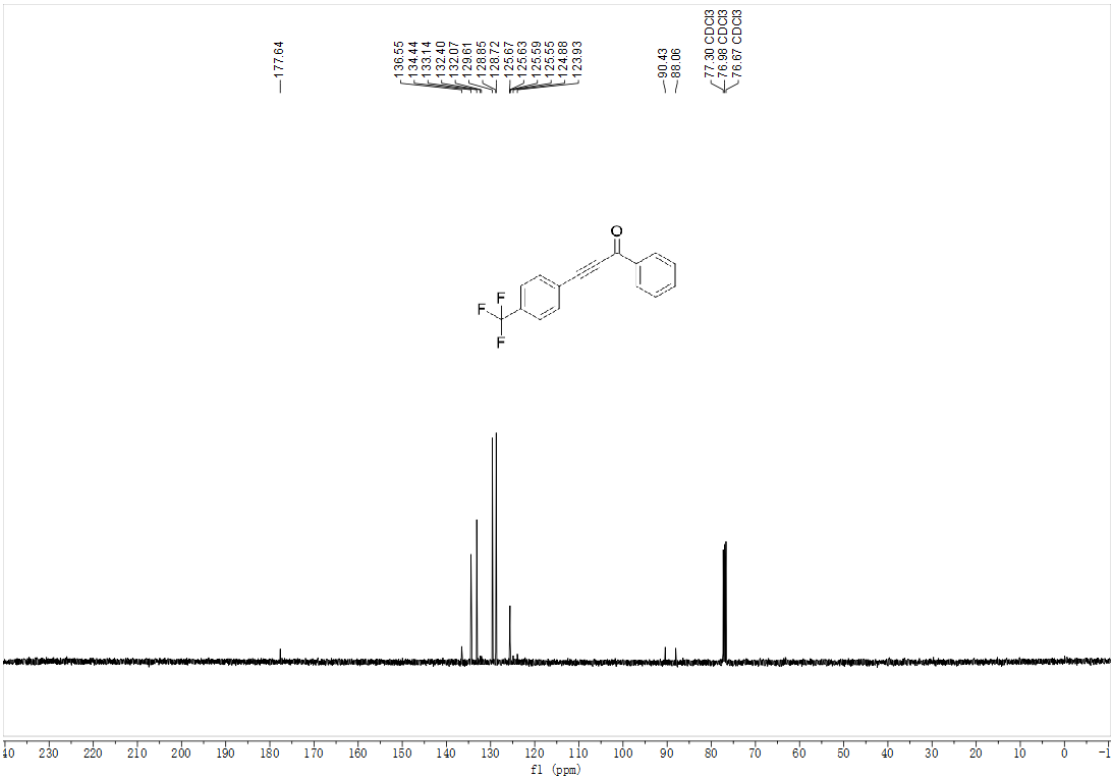
2.19
f1 (ppm)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125.55
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88.06
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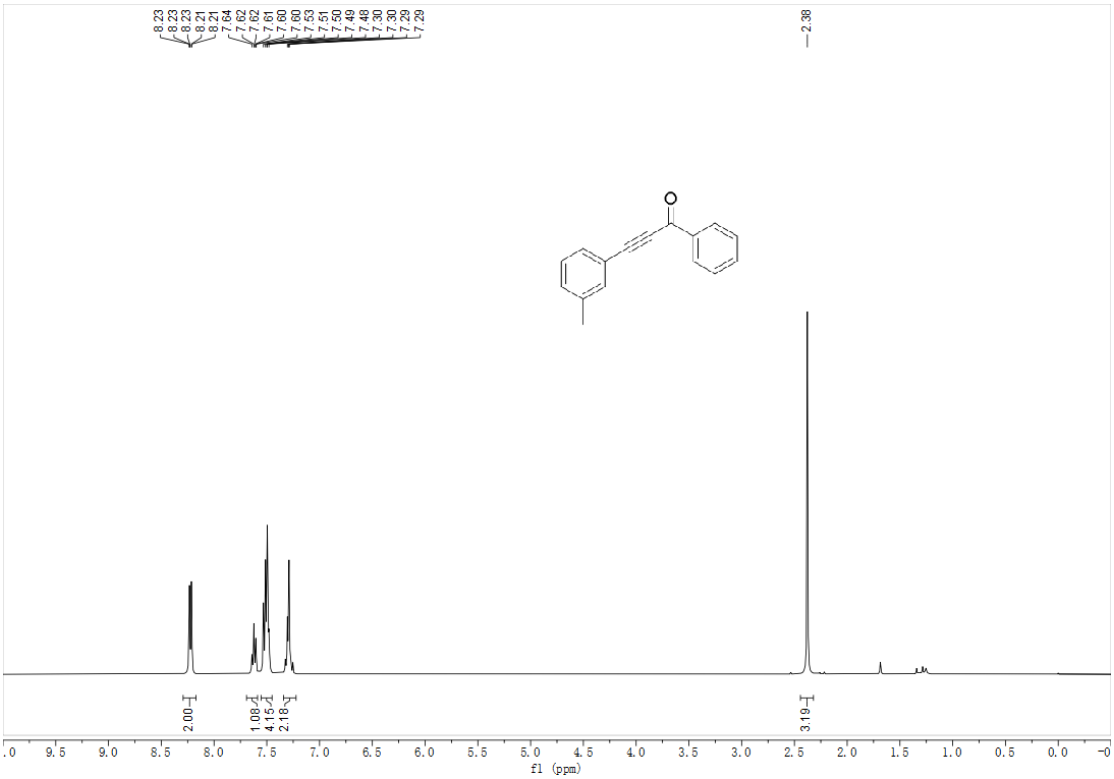
76.98 CDCl3
76.67 CDCl3
O
F
F
F
f1 (ppm)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7.50
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4.15
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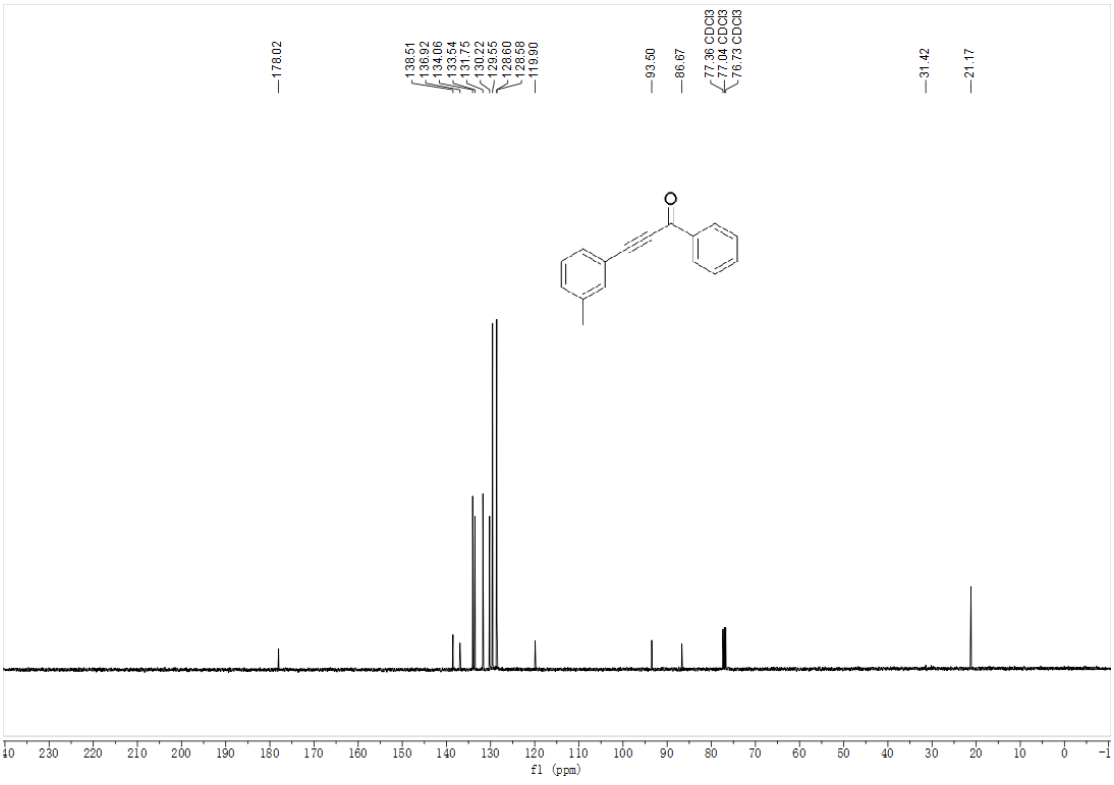
3.19
f1 (ppm)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77.04 CDCl3
76.73 CDCl3
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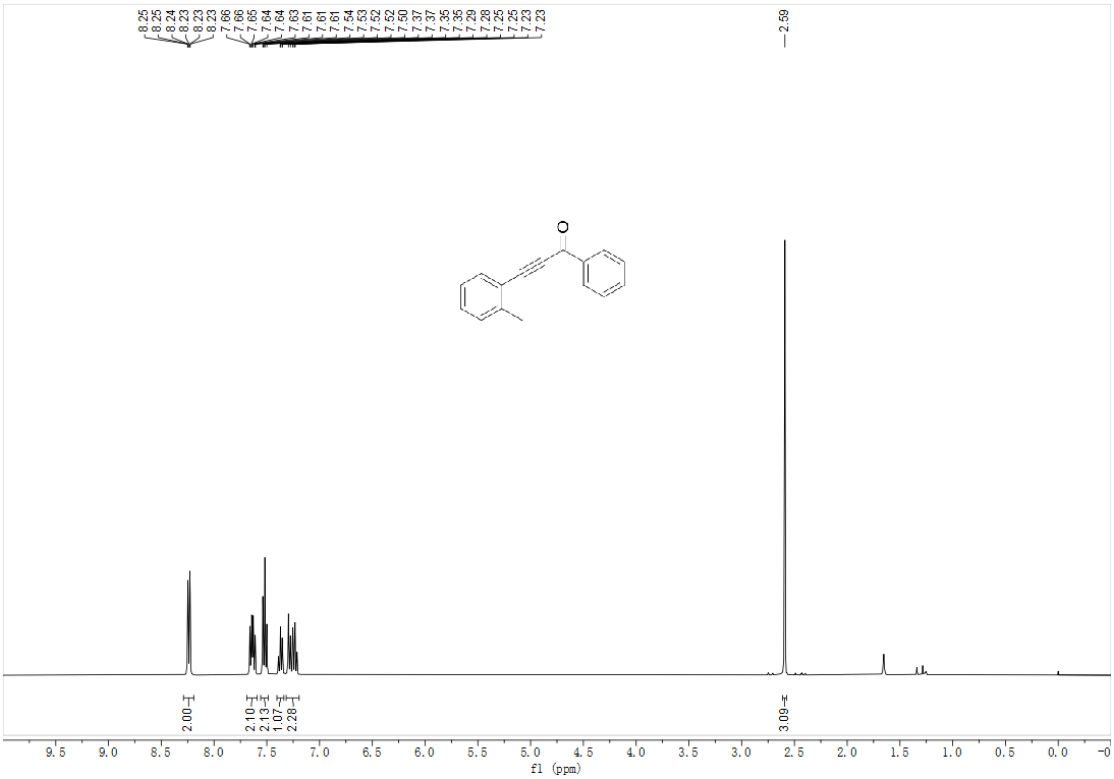
21.17
f1 (ppm)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1.07
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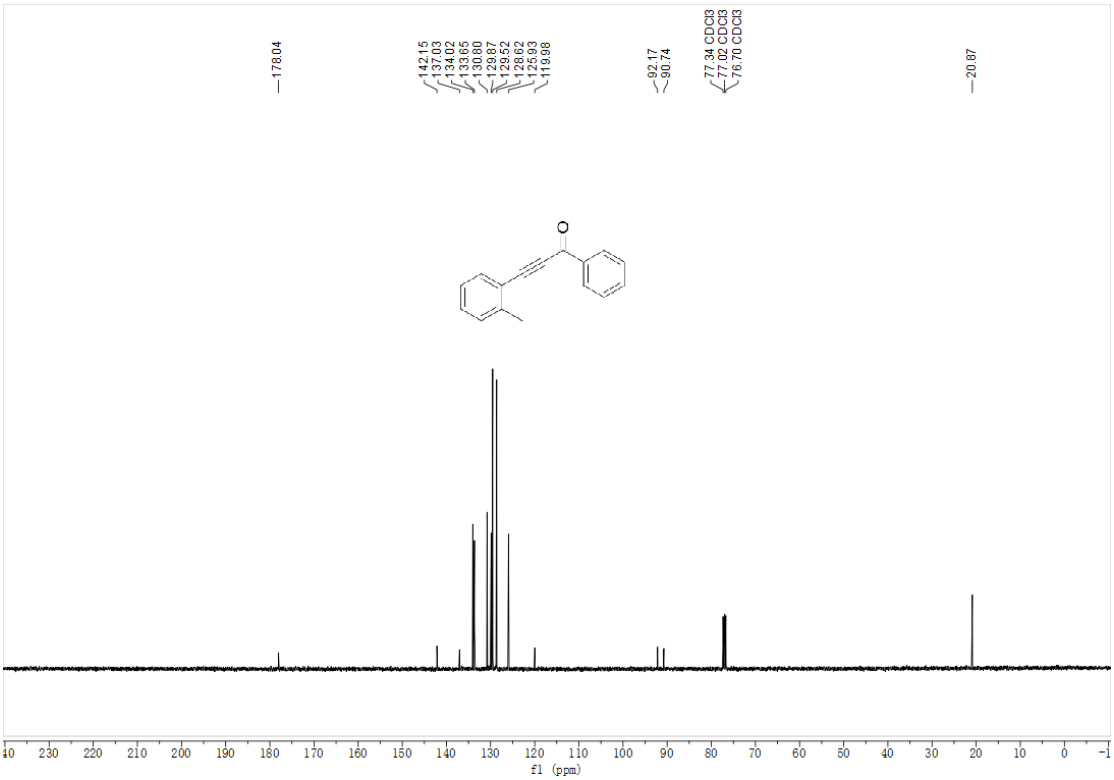
3.09
f1 (ppm)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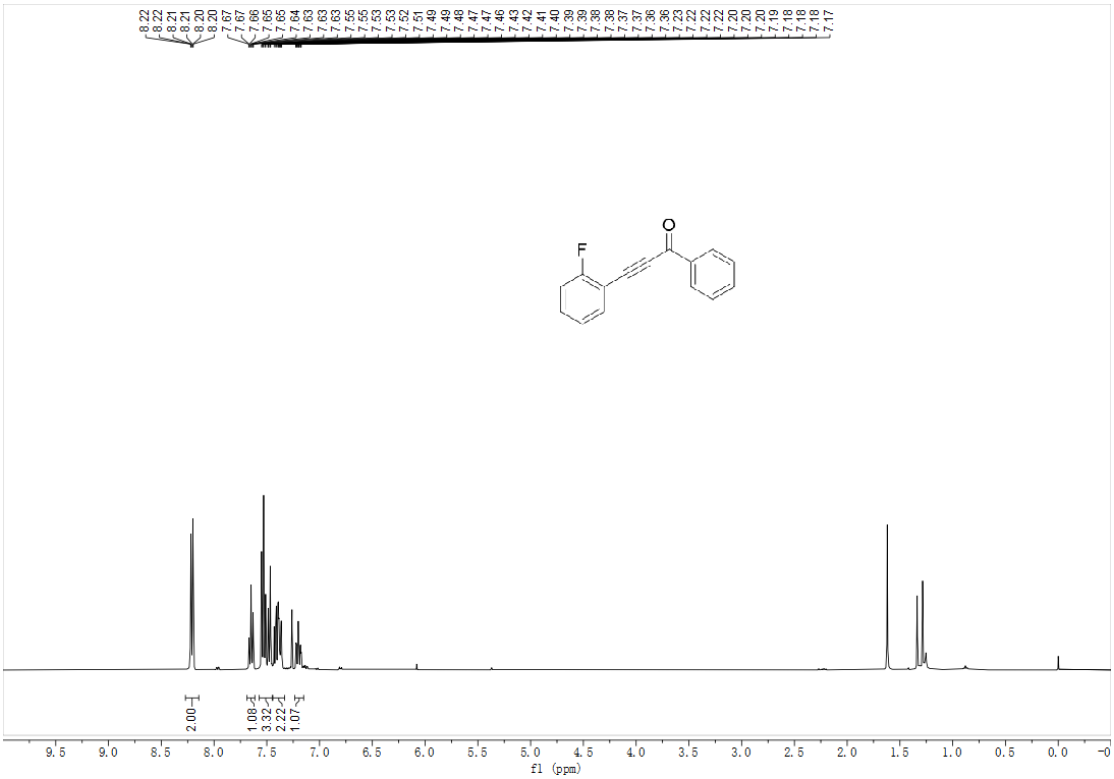
20.87
f1 (ppm)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7.17
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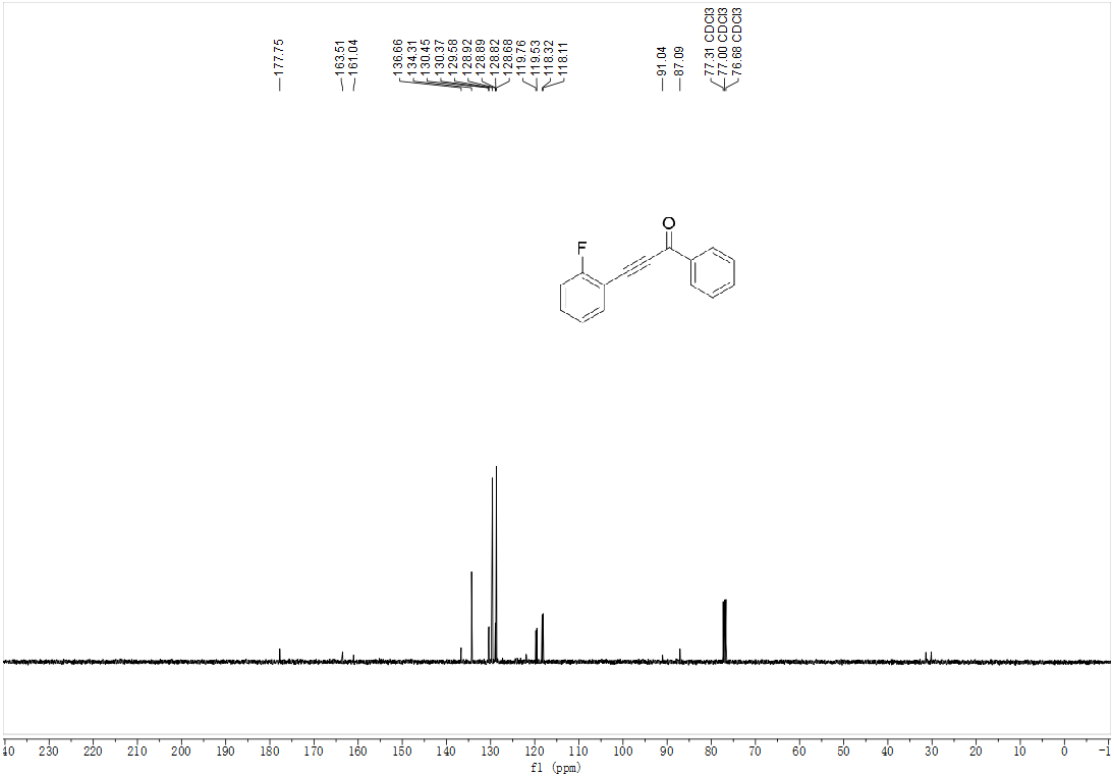
2.22
1.07
f1 (ppm)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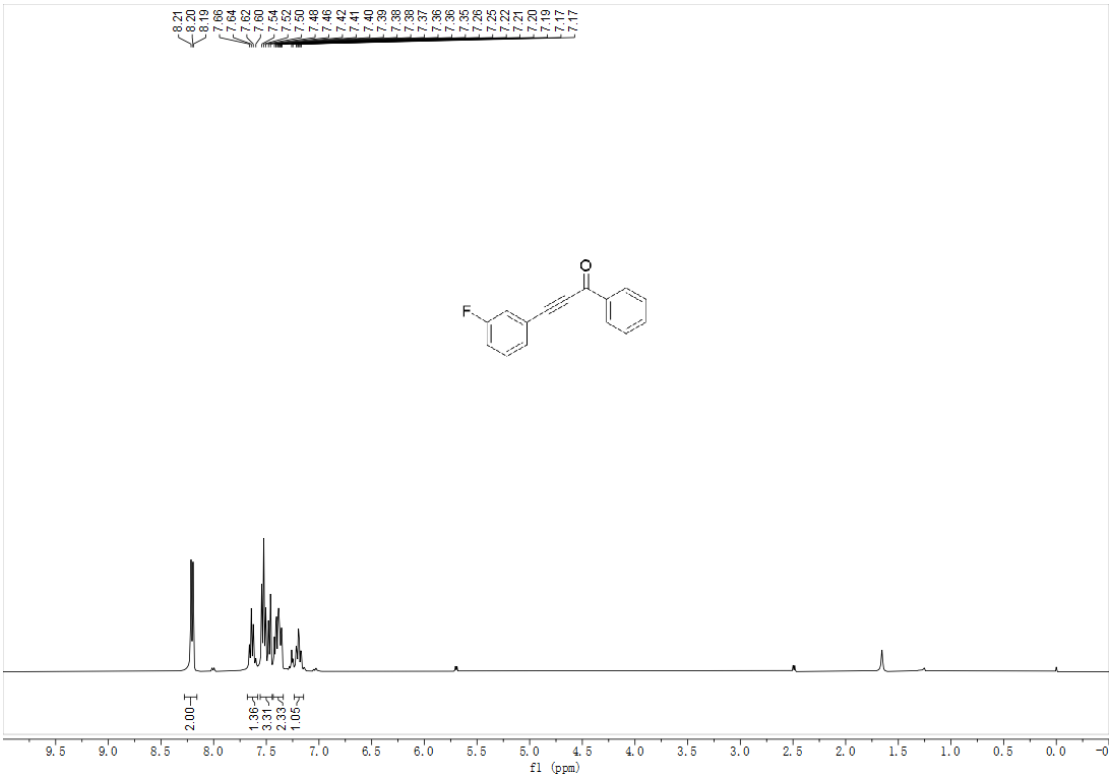
91.04
87.09
77.31 CDCl3
77.00 CDCl3
76.68 CDCl3
F
O
f1 (ppm)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7.36
7.36
7.35
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7.22
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7.17
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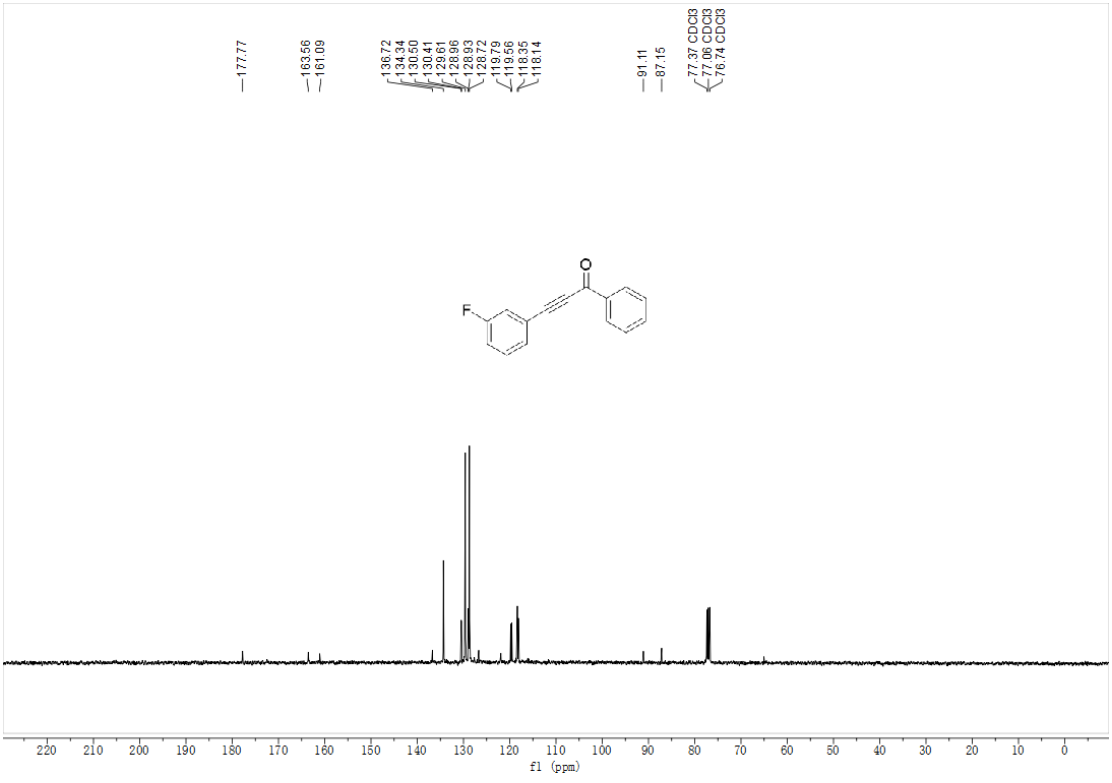
1.05
f1 (ppm)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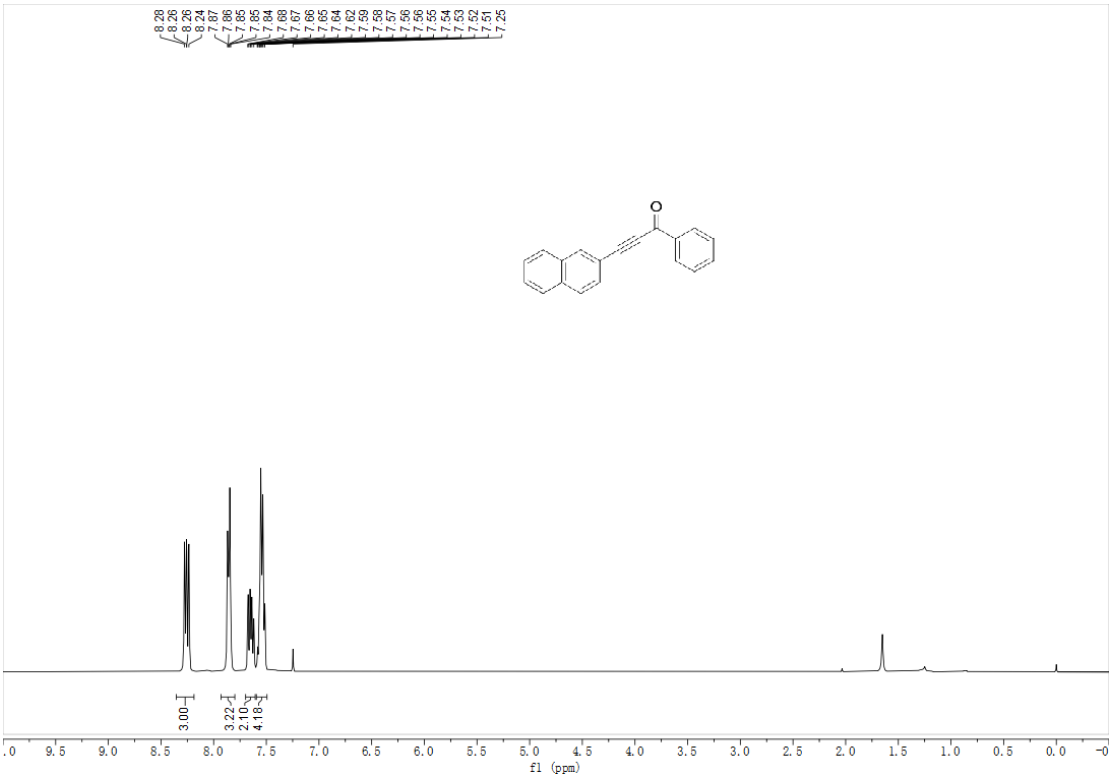
76.74 CDCl3
O
F
f1 (ppm)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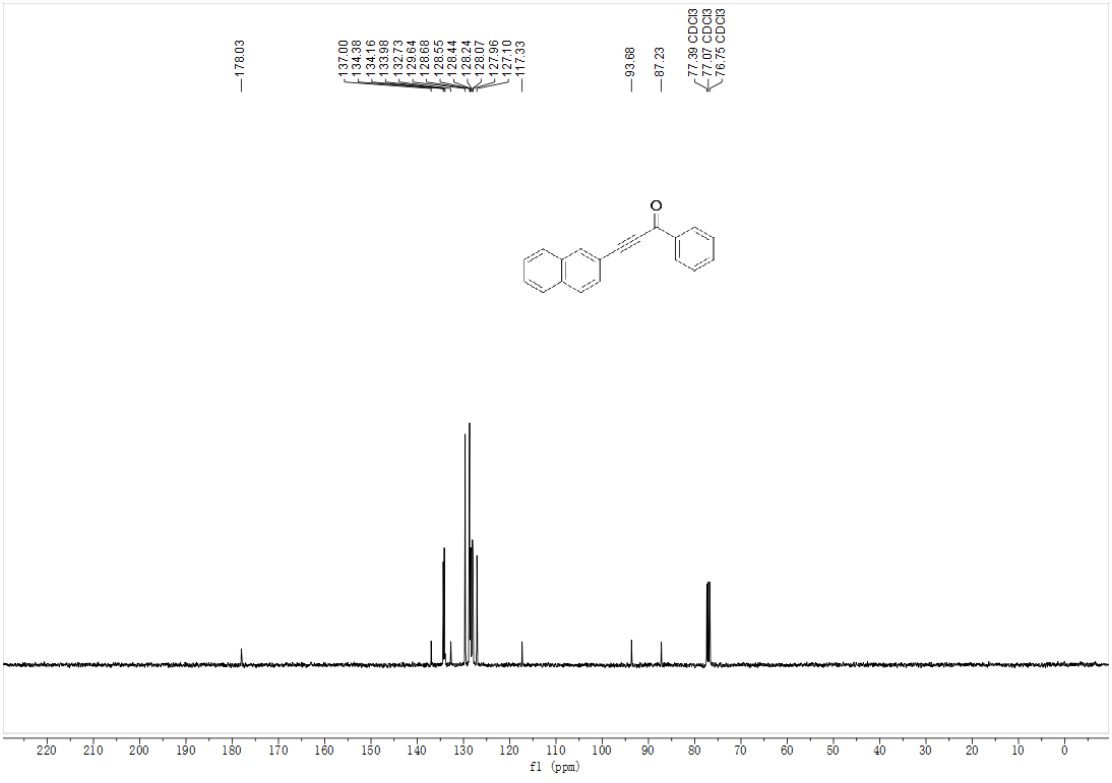
4.18
f1 (ppm)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93.68
87.23
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77.07 CDCl3
76.75 CDCl3
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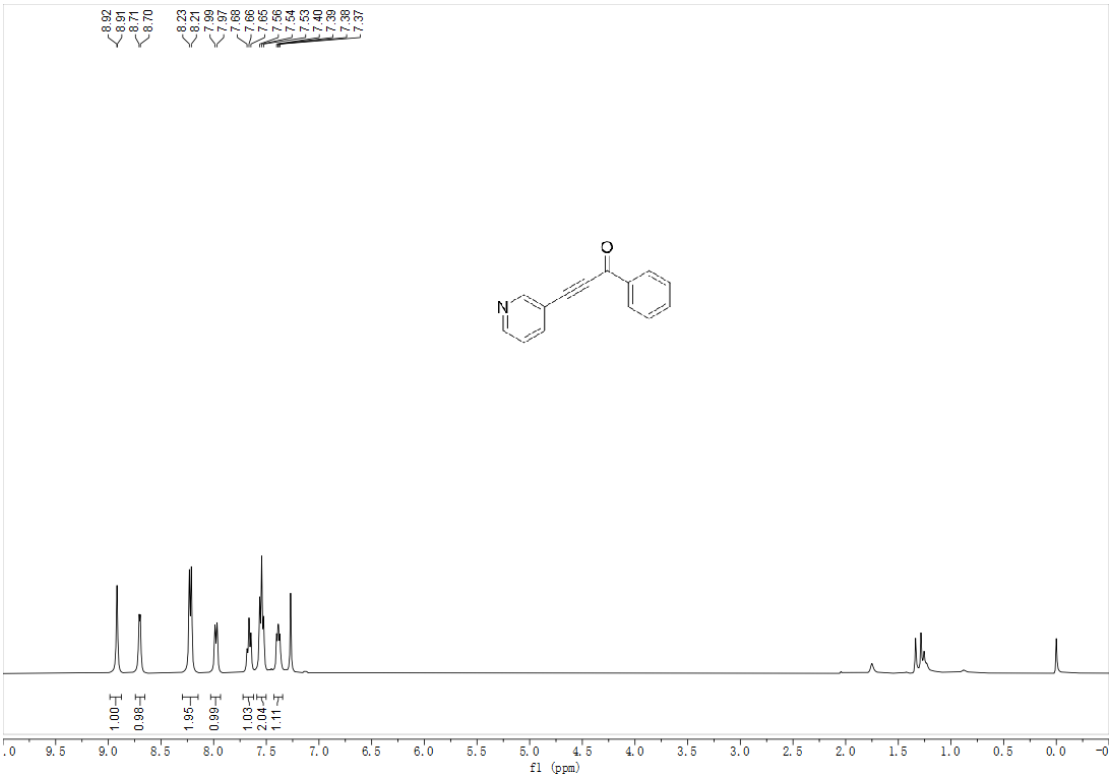
f1 (ppm)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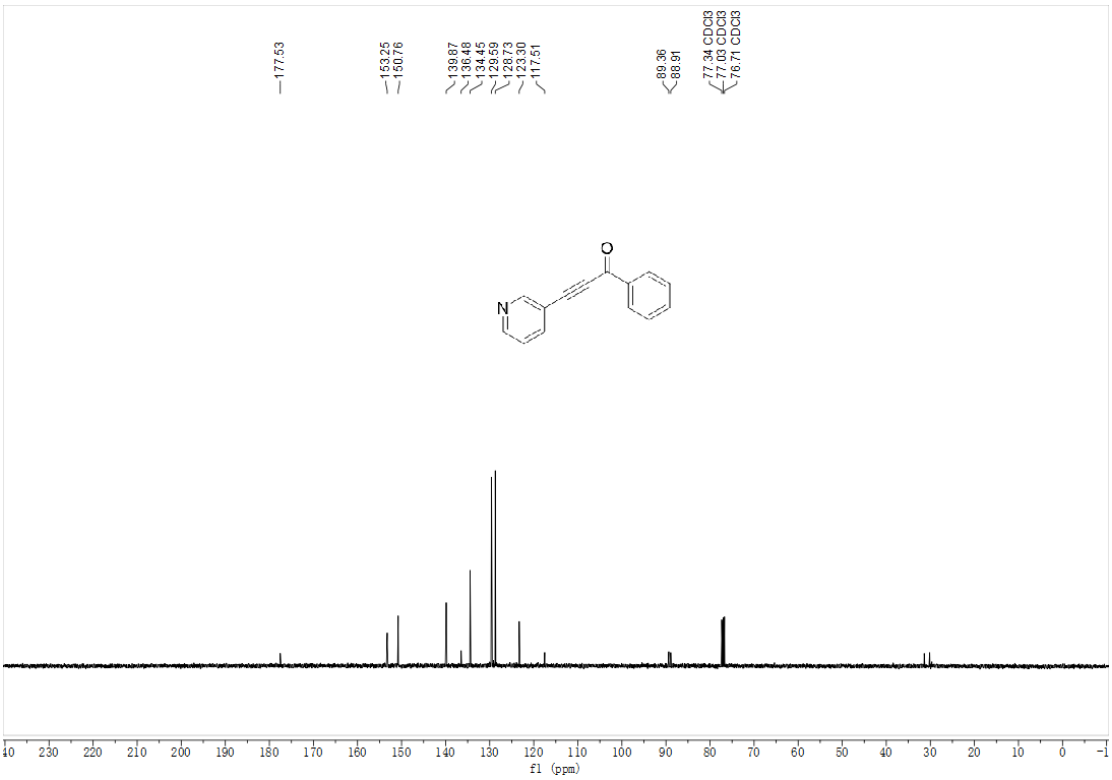
1.11
f1 (ppm)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76.71 CDCl3
O
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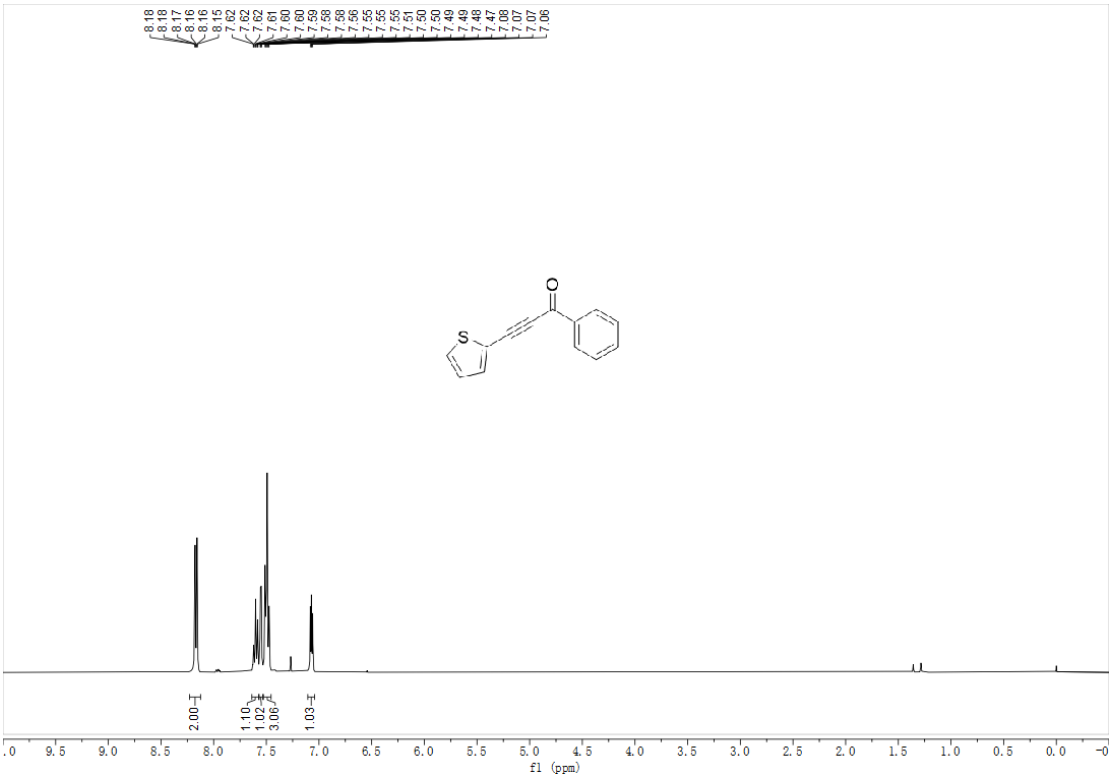
f1 (ppm)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7.51
7.50
7.49
7.49
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7.06
O
S
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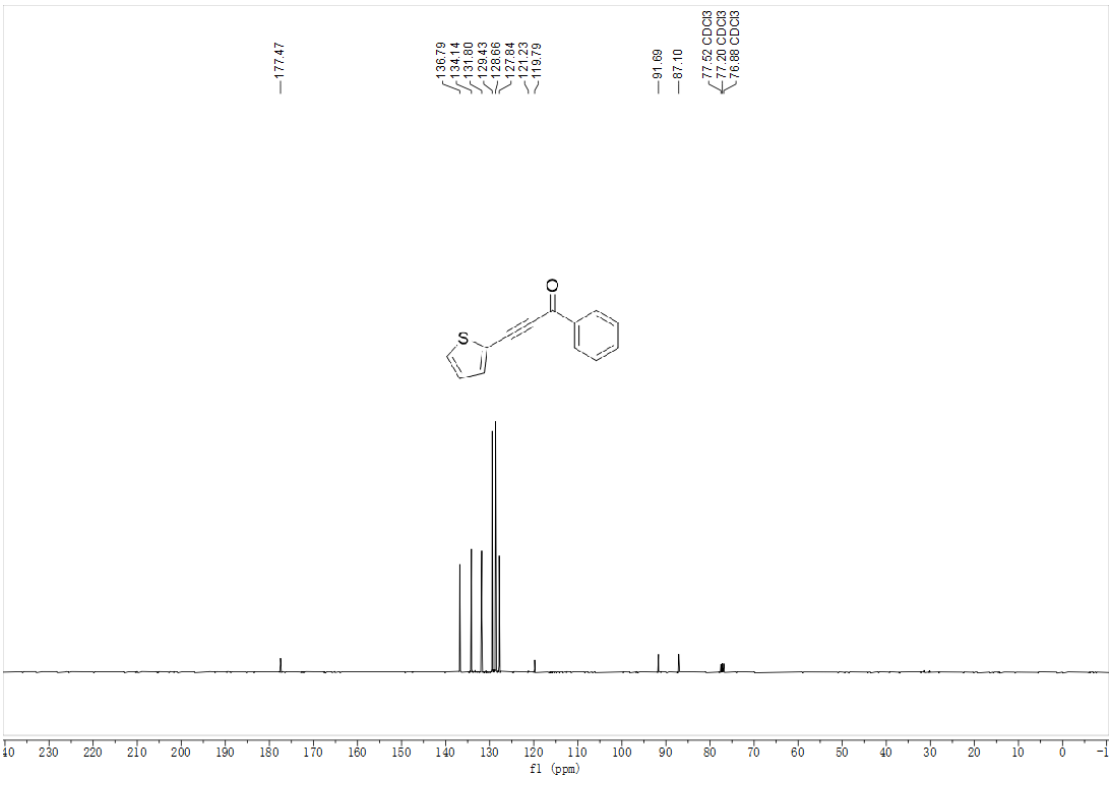
1.10
1.02
3.06
1.03
f1 (ppm)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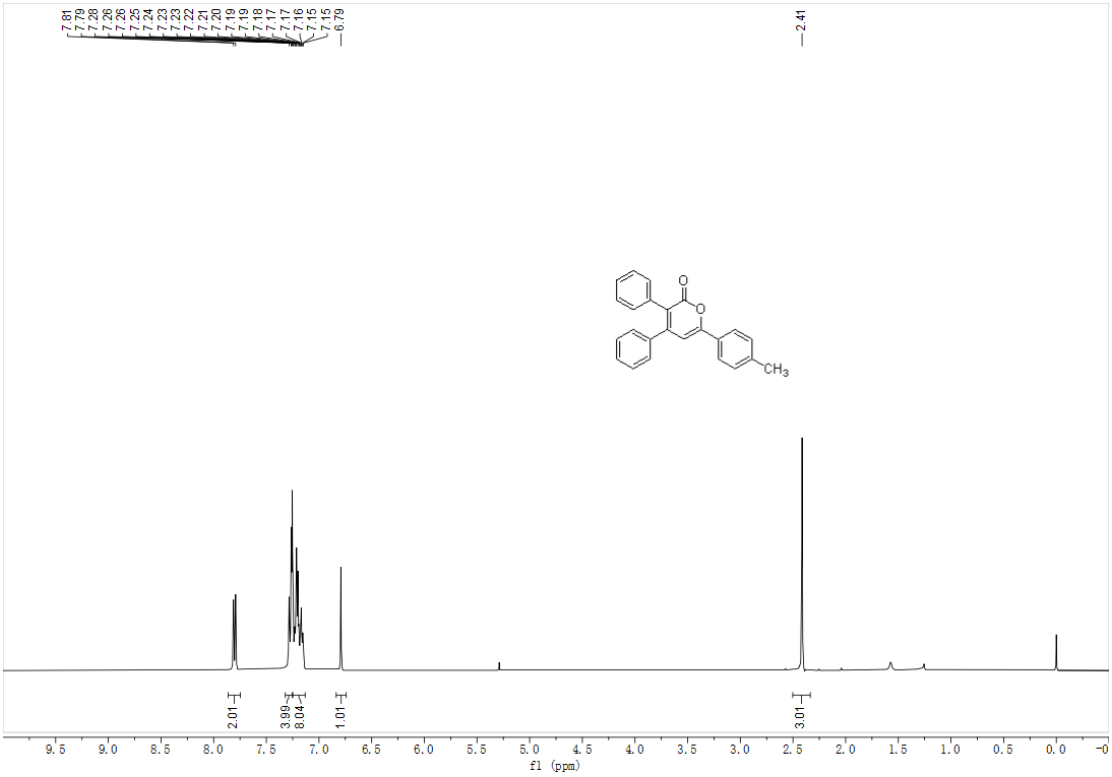
76.88 CDCl3
O
S
f1 (ppm)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7.17
7.17
7.16
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O
O
CH3
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8.04
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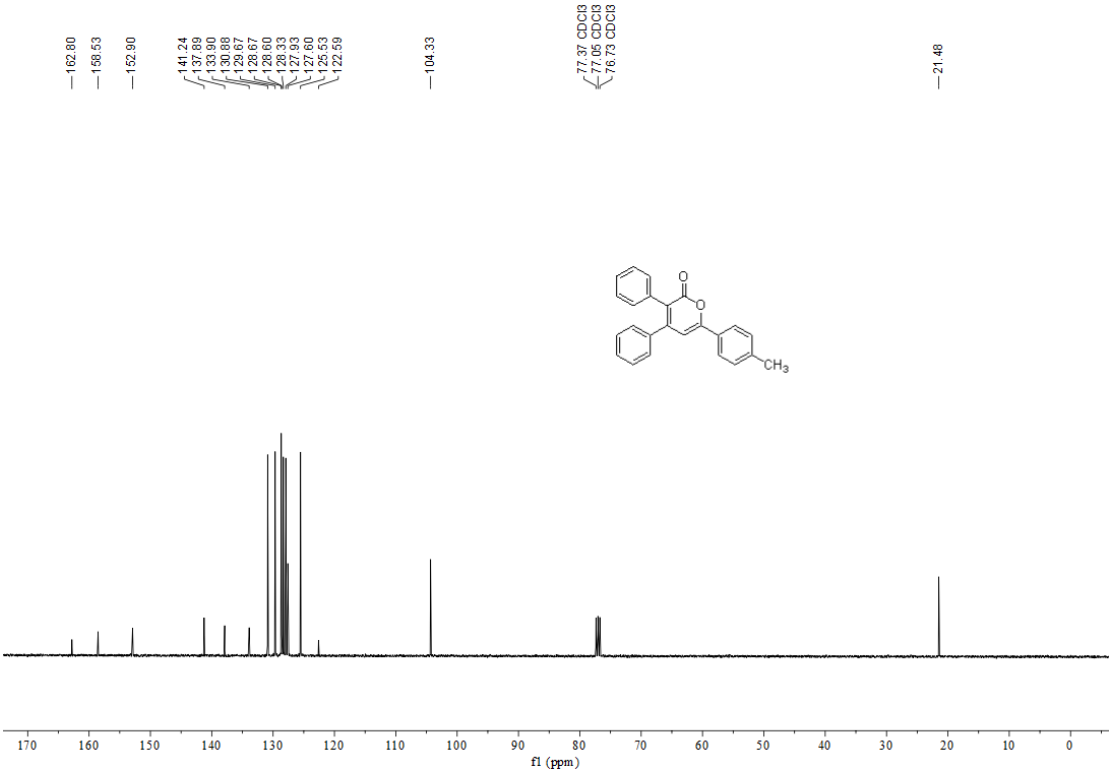
3.01
f1 (ppm)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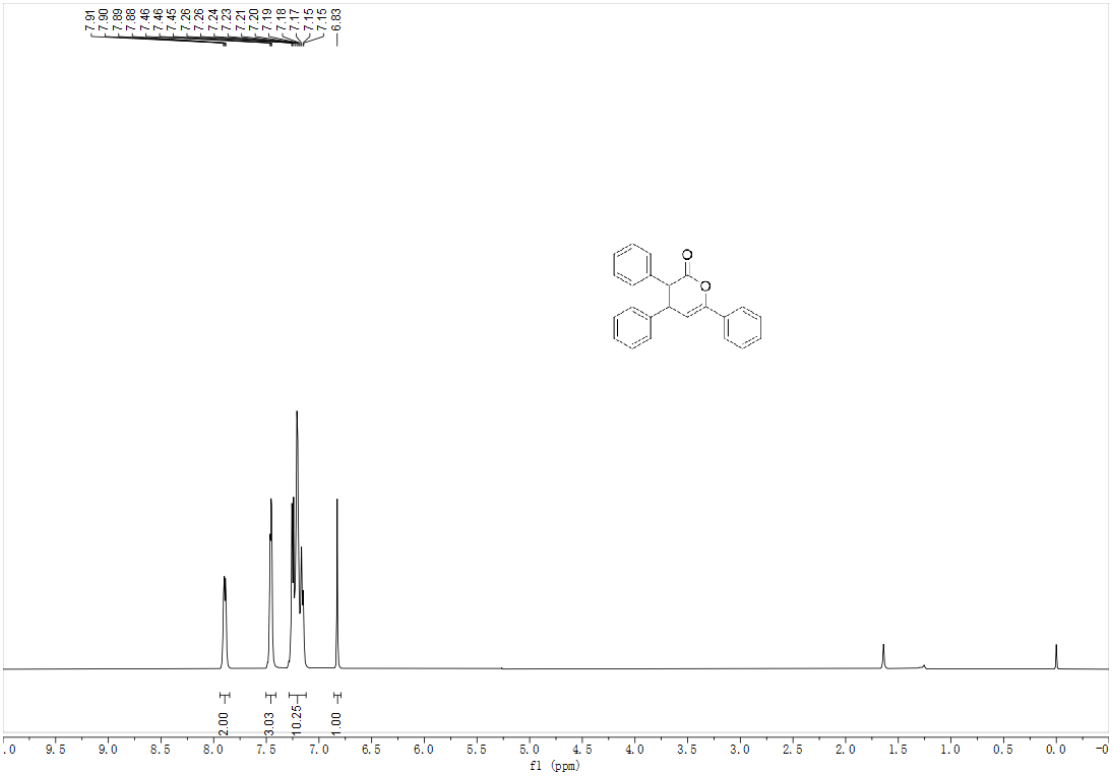
O
CH3
f1 (ppm)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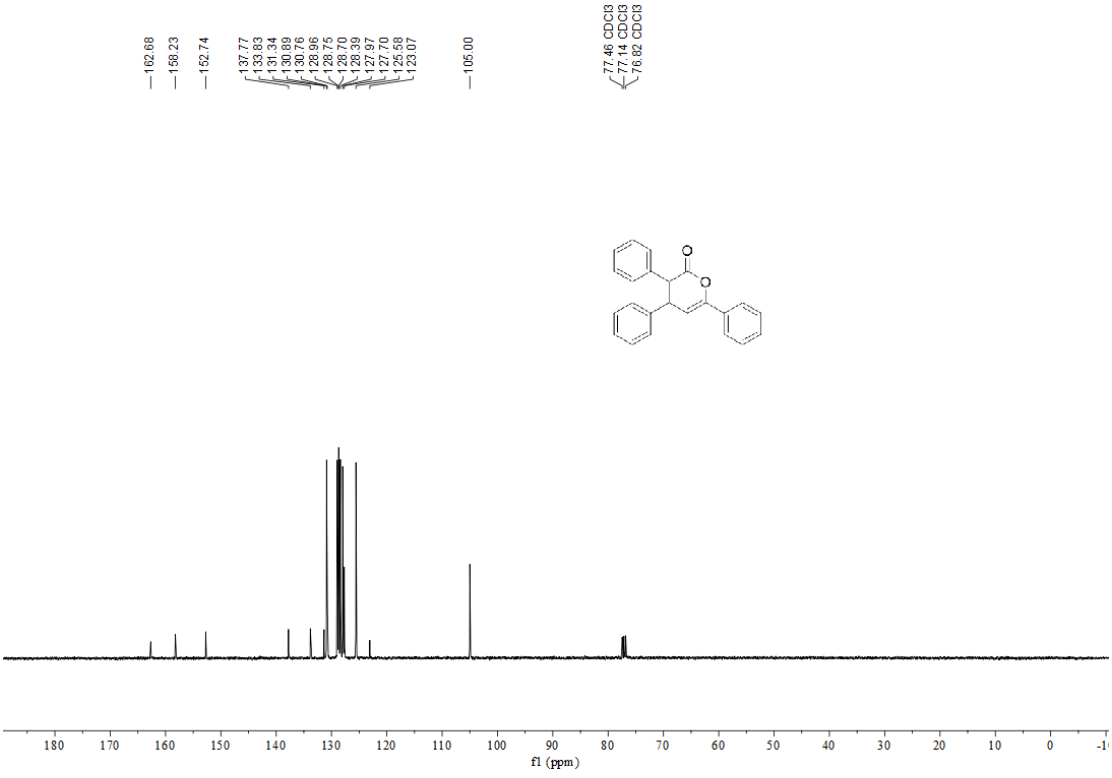
1.00
f1 (ppm)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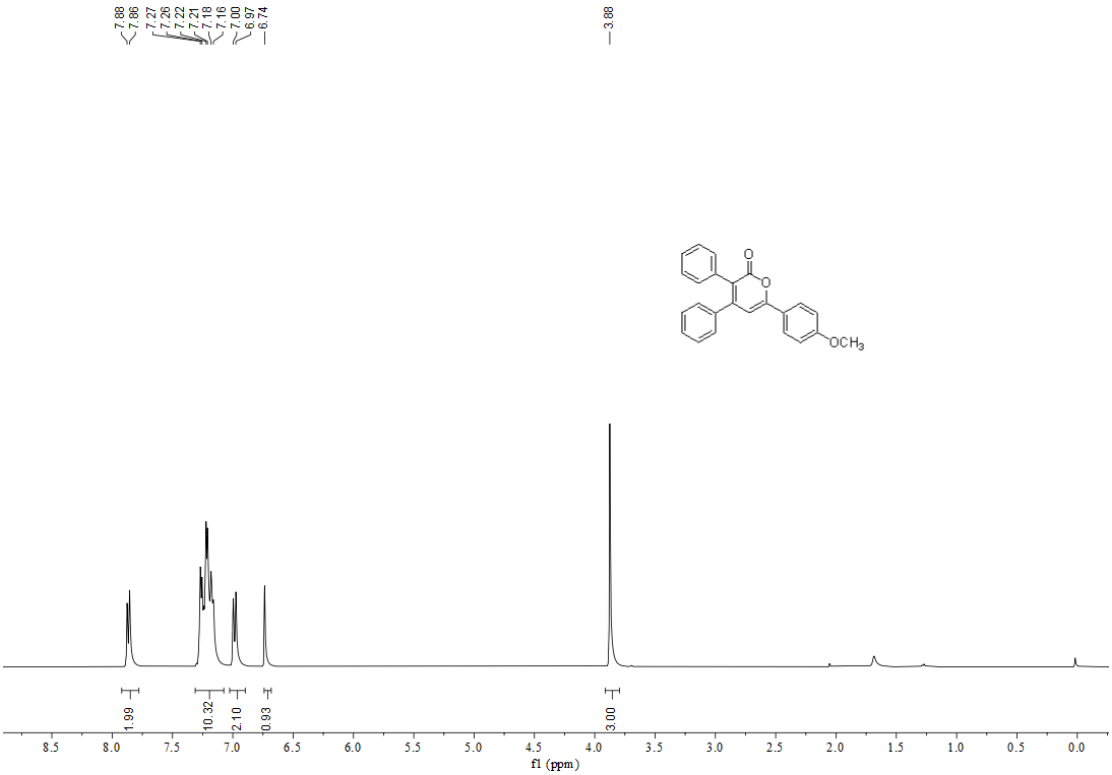
77.14 CDCl3
76.82 CDCl3
f1 (ppm)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7.16
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O
O
OCH3
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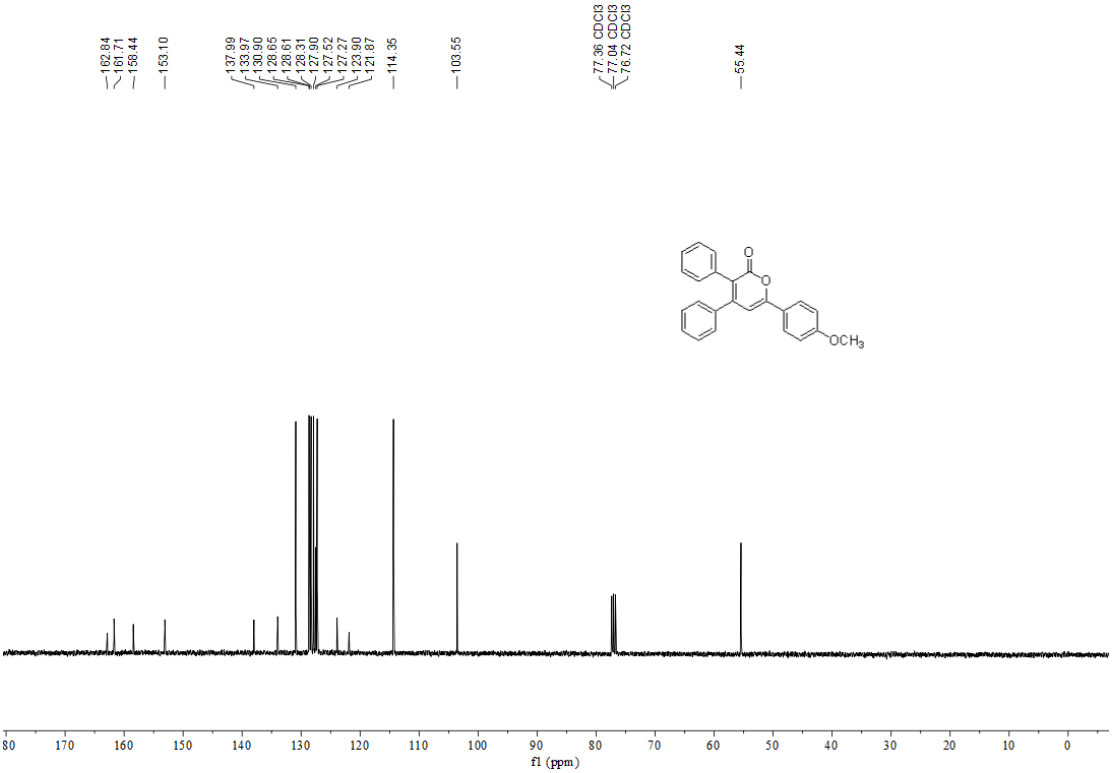
3.00
f1 (ppm)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123.90
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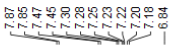
77.04 CDCl3
76.72 CDCl3
55.44
O
O
OCH3
f1 (ppm)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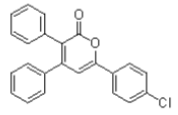
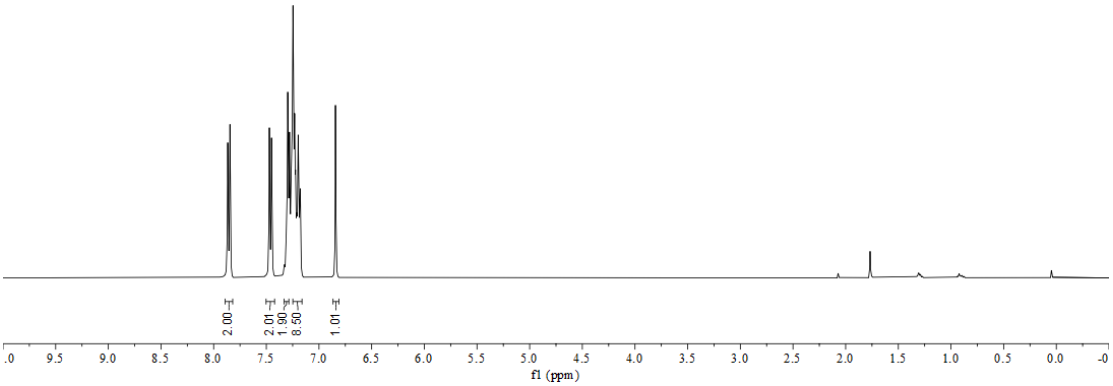

O
O
Cl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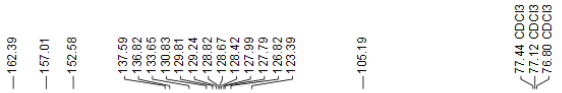
8.50
1.01
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)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129.81
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128.82
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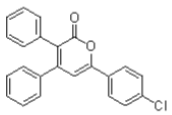
127.79
126.82
123.39
105.19
77.44 CDCl3
77.12 CDCl3
76.80 CDCl3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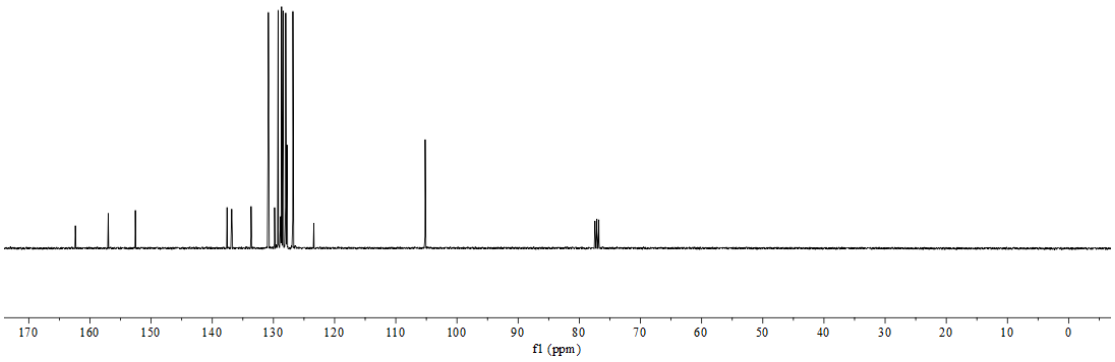
O
Cl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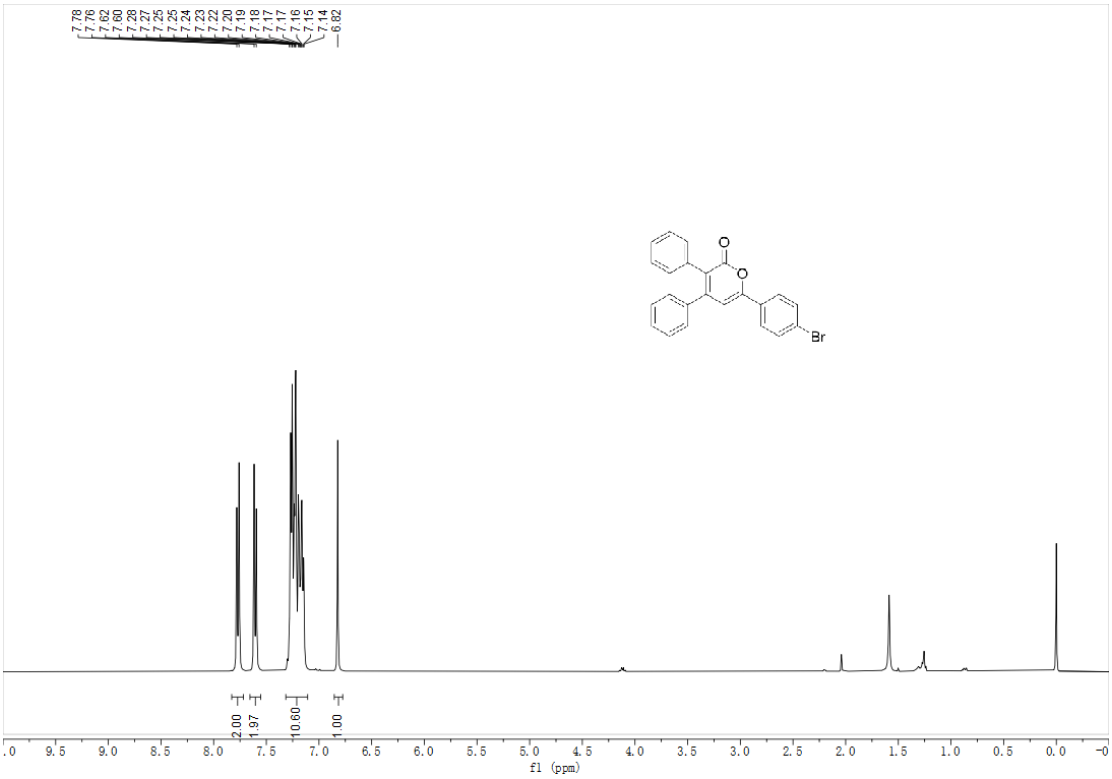
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7.25
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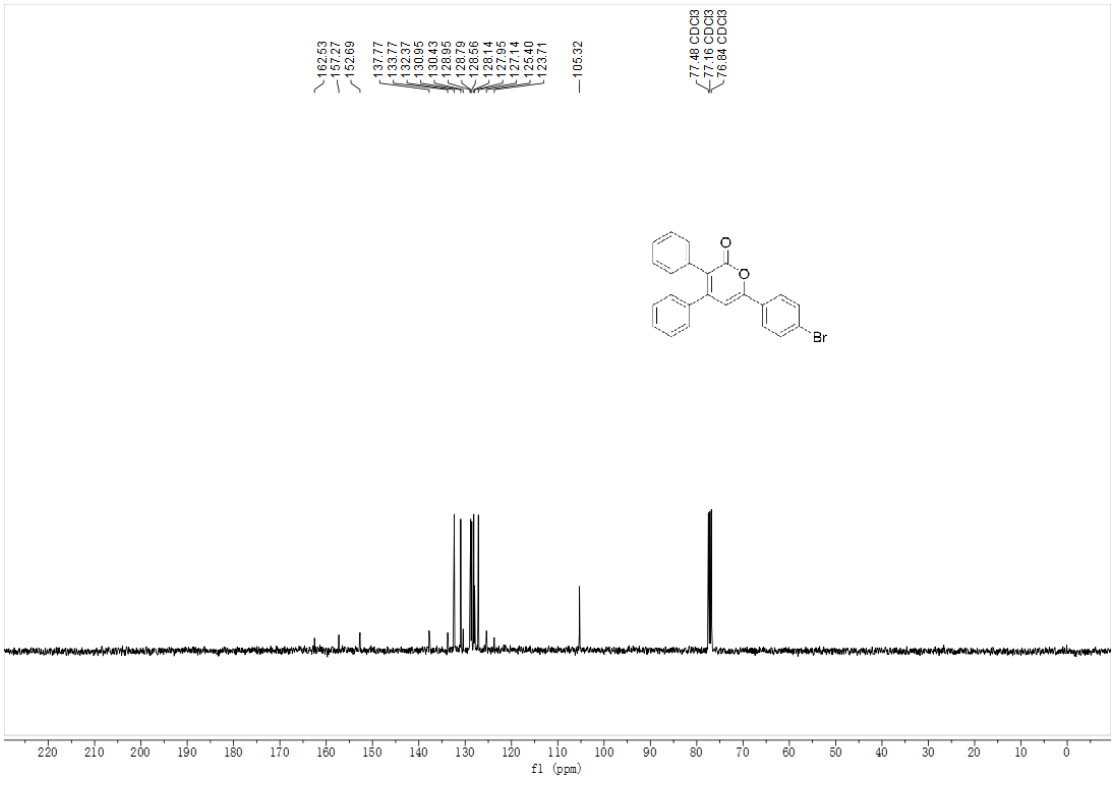
7.23
7.22
7.20
7.19
7.18
7.17
7.17
7.16
7.15
7.14
6.62
O
O
Br
2.00
1.97
10.60
1.00
f1 (ppm)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127.95
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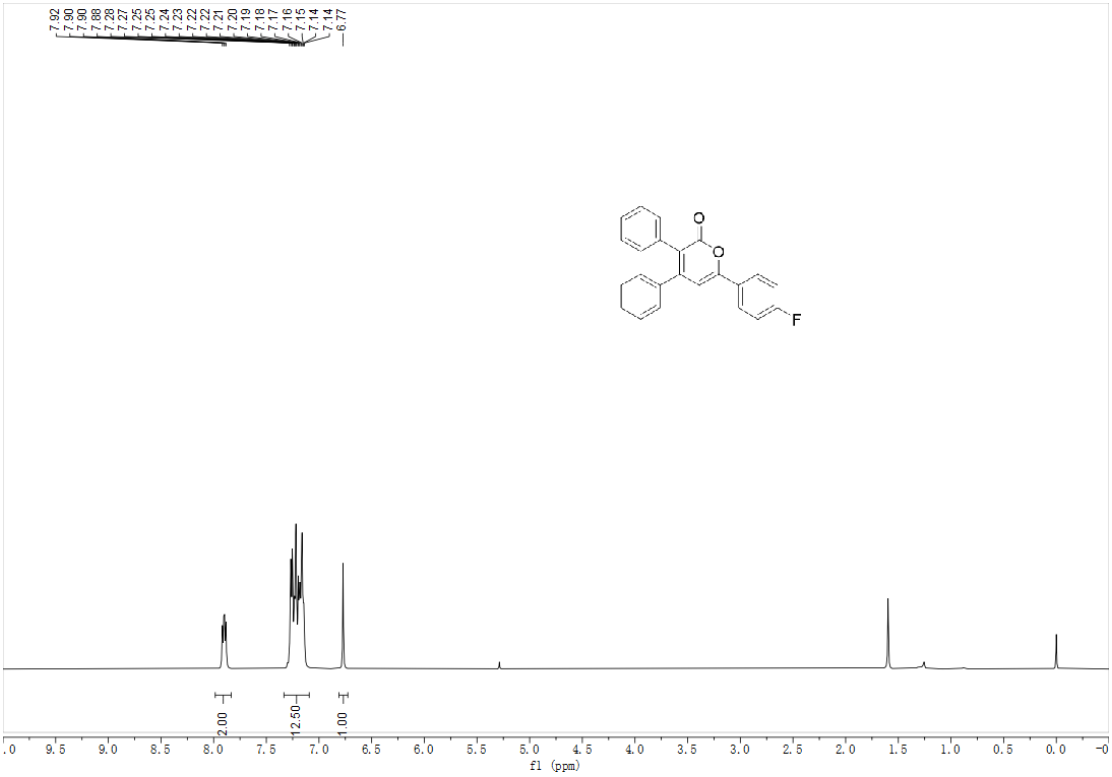
123.71
105.32
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
O
Br
f1 (ppm)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7.21
7.20
7.19
7.18
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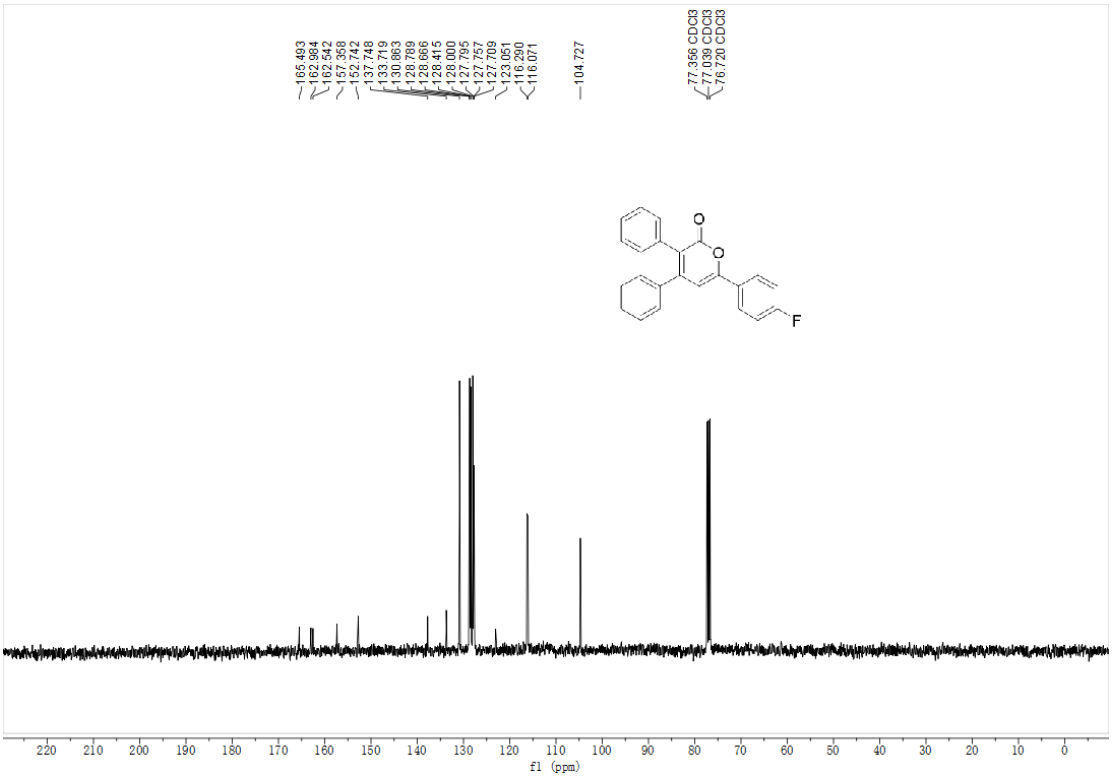
7.16
7.15
7.14
7.14
6.77
O
O
F
2.00
12.50
1.00
f1 (ppm)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127.795
127.757
127.709
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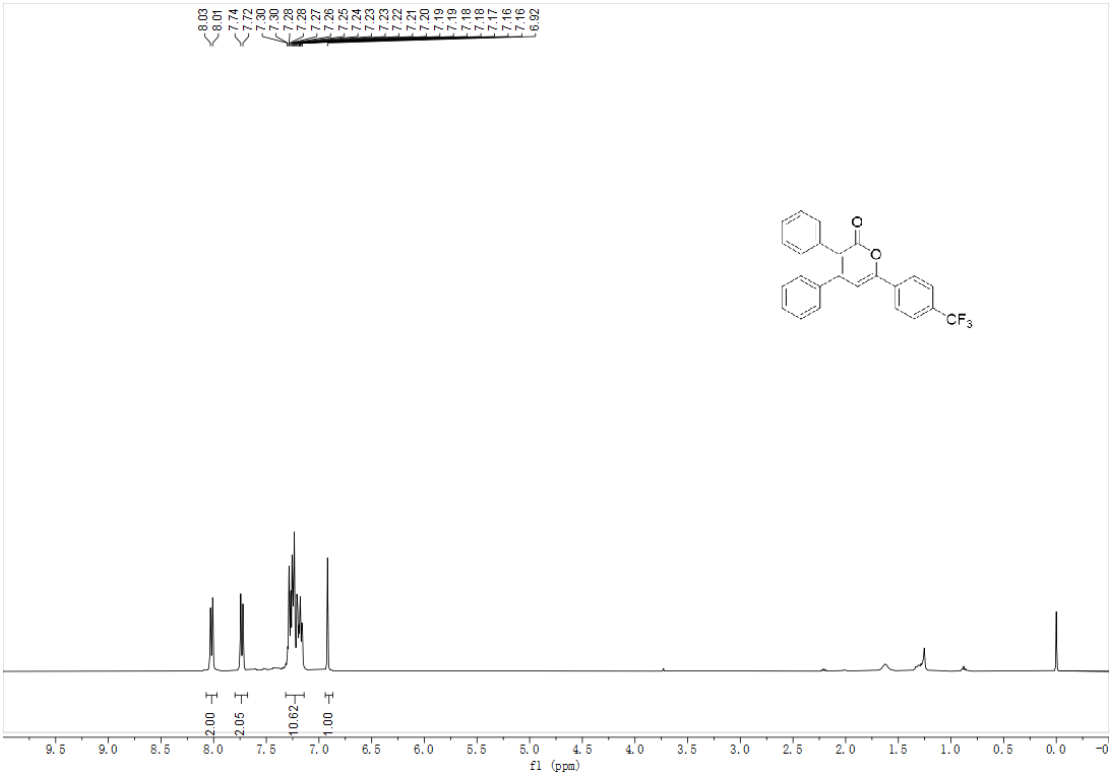
116.290
116.071
104.727
77.356 CDCl3
77.039 CDCl3
76.720 CDCl3
O
O
F
f1 (ppm)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7.22
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7.19
7.18
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2.05
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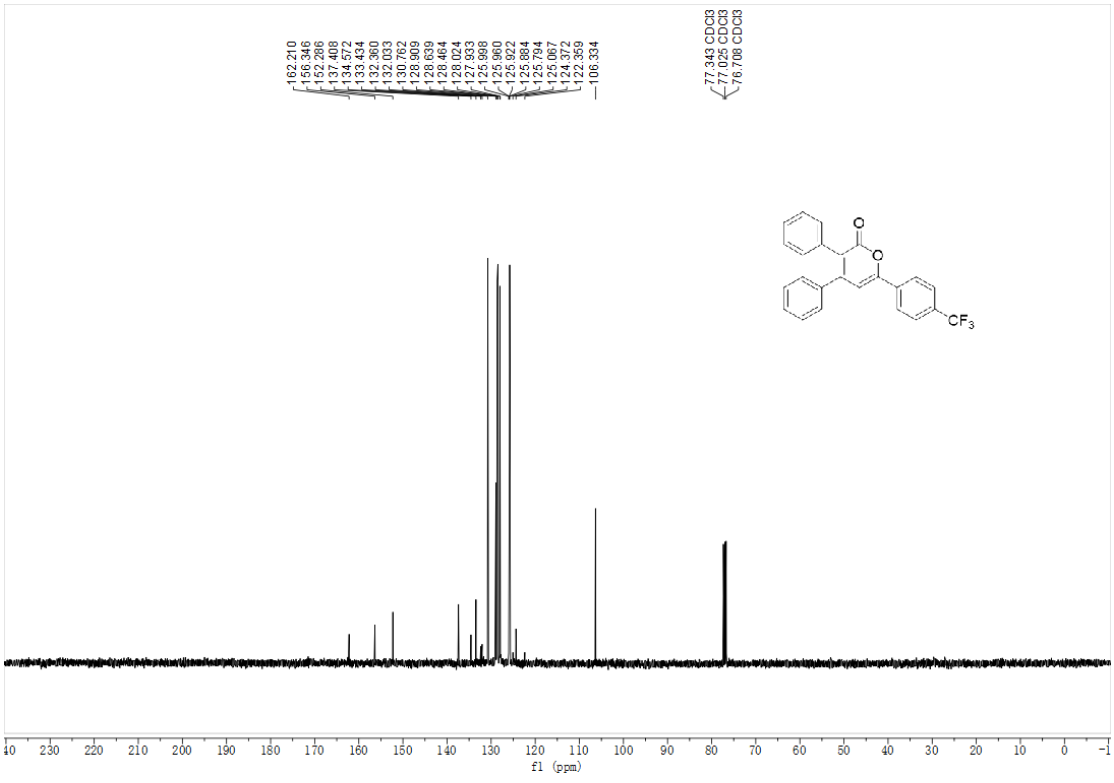
1.00
f1 (ppm)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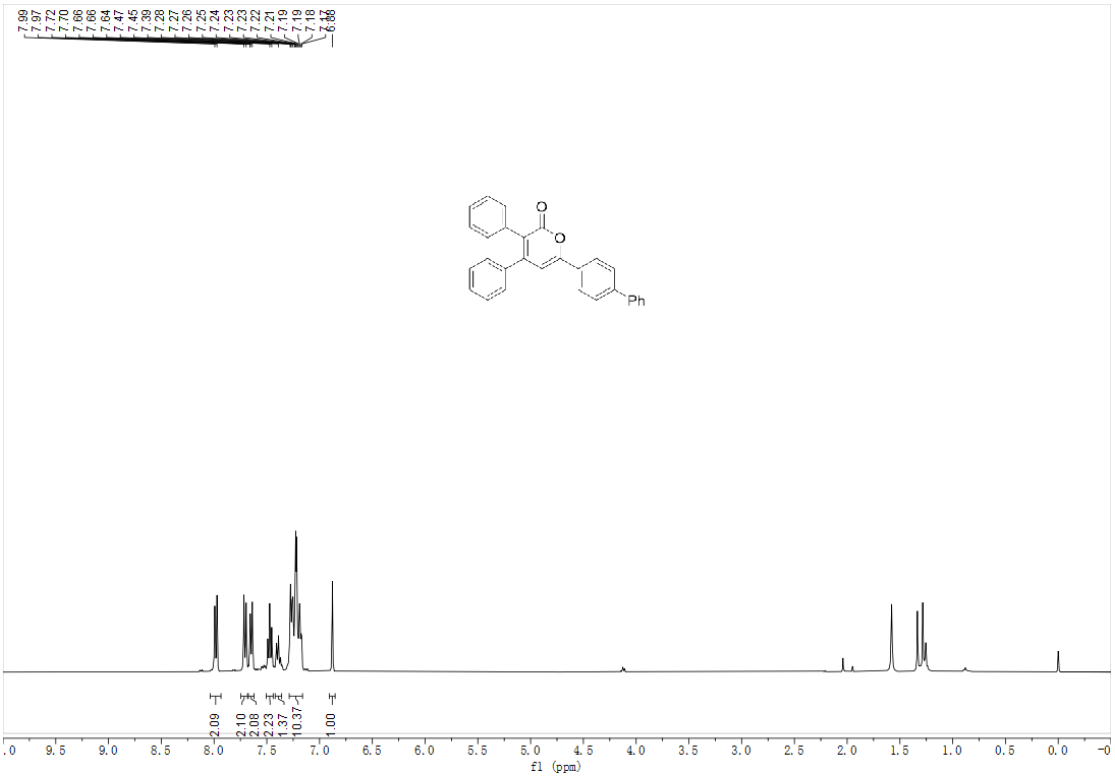
125.998
125.960
125.922
125.884
125.794
125.067
124.372
122.359
106.334
77.343 CDCl3
77.025 CDCl3
76.708 CDCl3
O
O
CF3
f1 (ppm)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7.26
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7.24
7.23
7.23
7.22
7.21
7.19
7.19
7.18
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6.88
O
O
Ph
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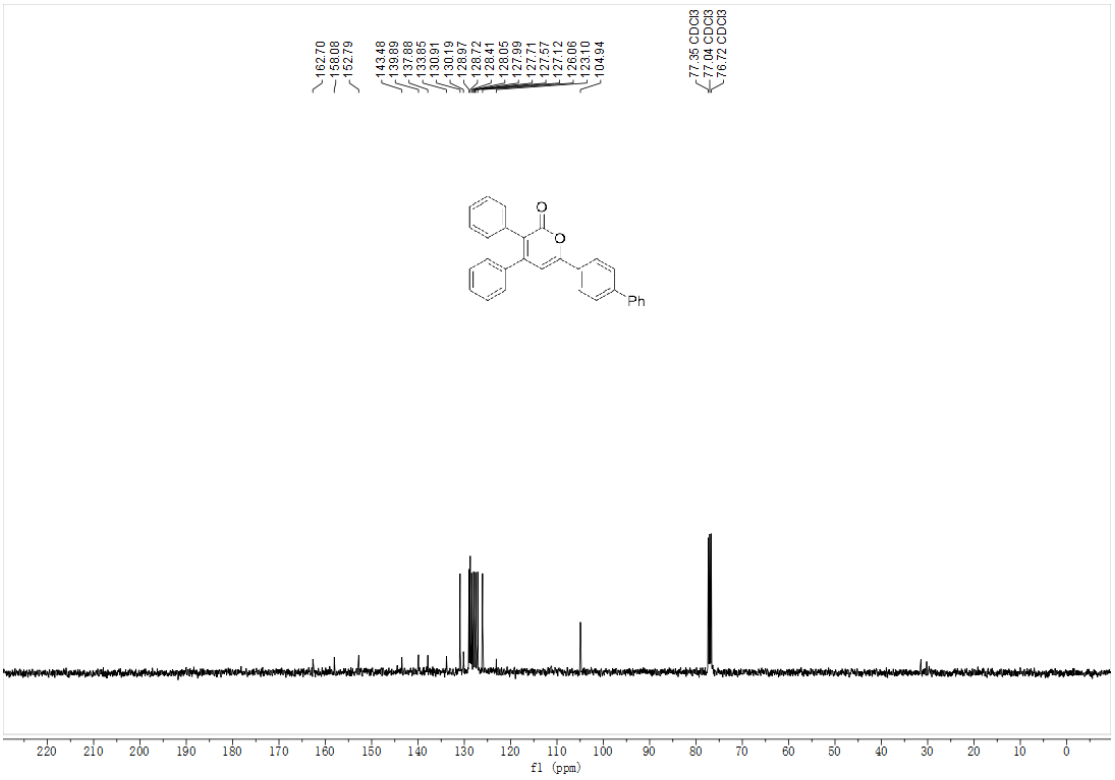
2.10
2.08
2.23
1.37
10.37
1.00
f1 (ppm)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128.41
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127.99
127.71
127.57
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126.06
123.10
104.94
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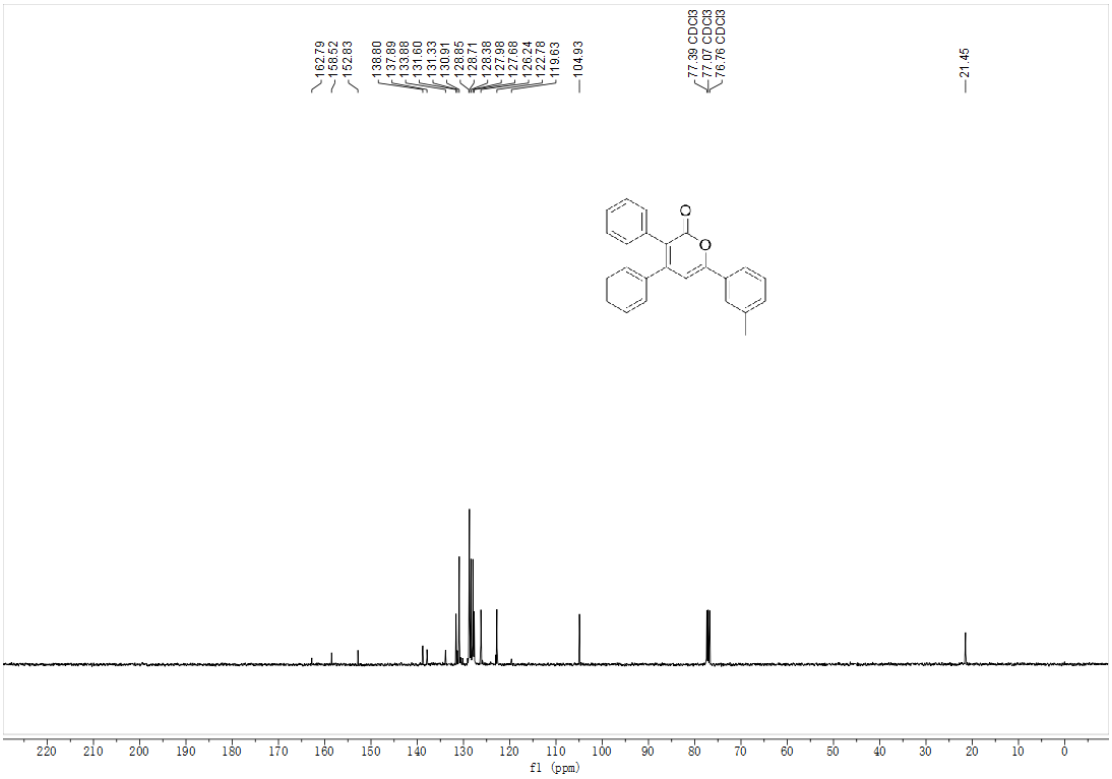
77.04 CDCl3
76.72 CDCl3
O
O
Ph
f1 (ppm)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126.24
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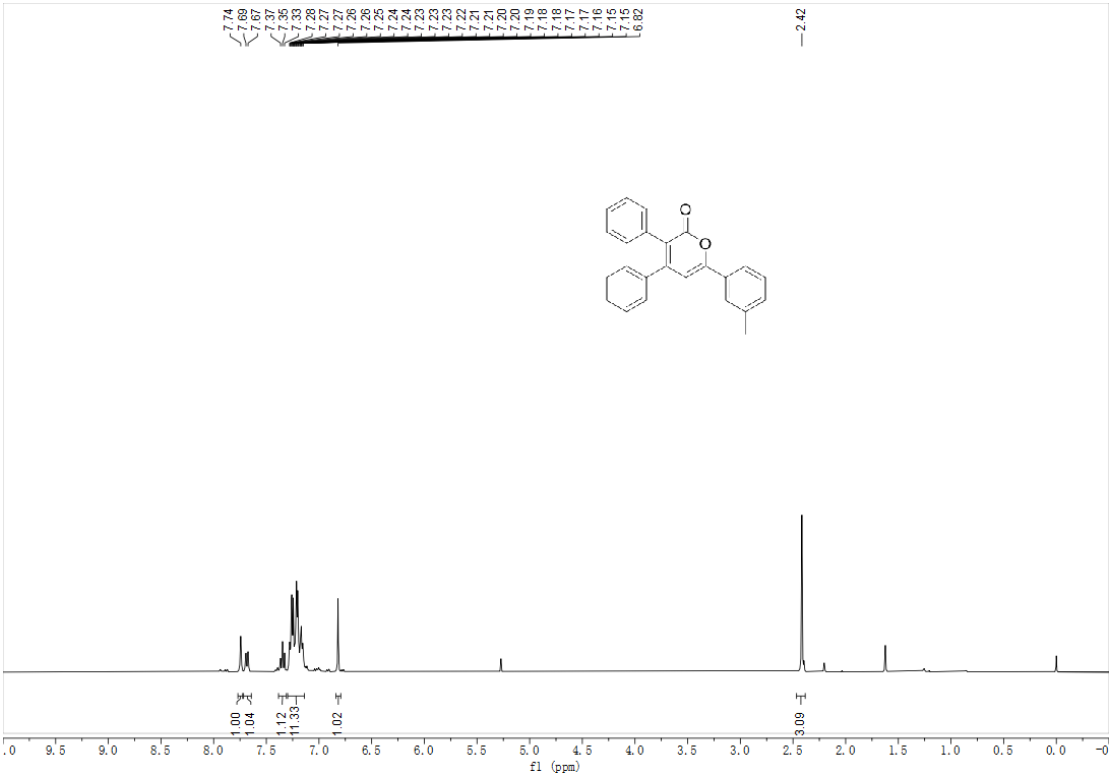
119.63
104.93
77.39 CDCl3
77.07 CDCl3
76.76 CDCl3
21.45
O
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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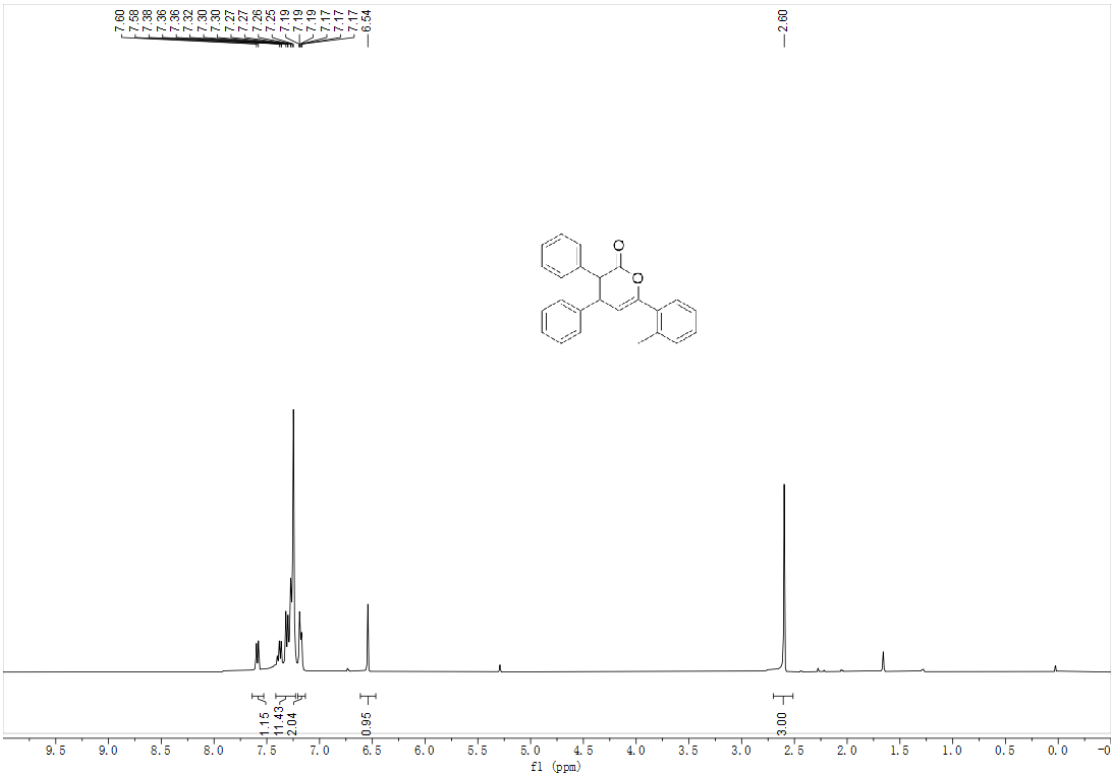
7.19
7.18
7.18
7.17
7.17
7.16
7.15
7.15
7.15
6.82
2.42
O
O
1.00
1.04
1.12
1.33
1.02
3.09
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)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7.19
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7.17
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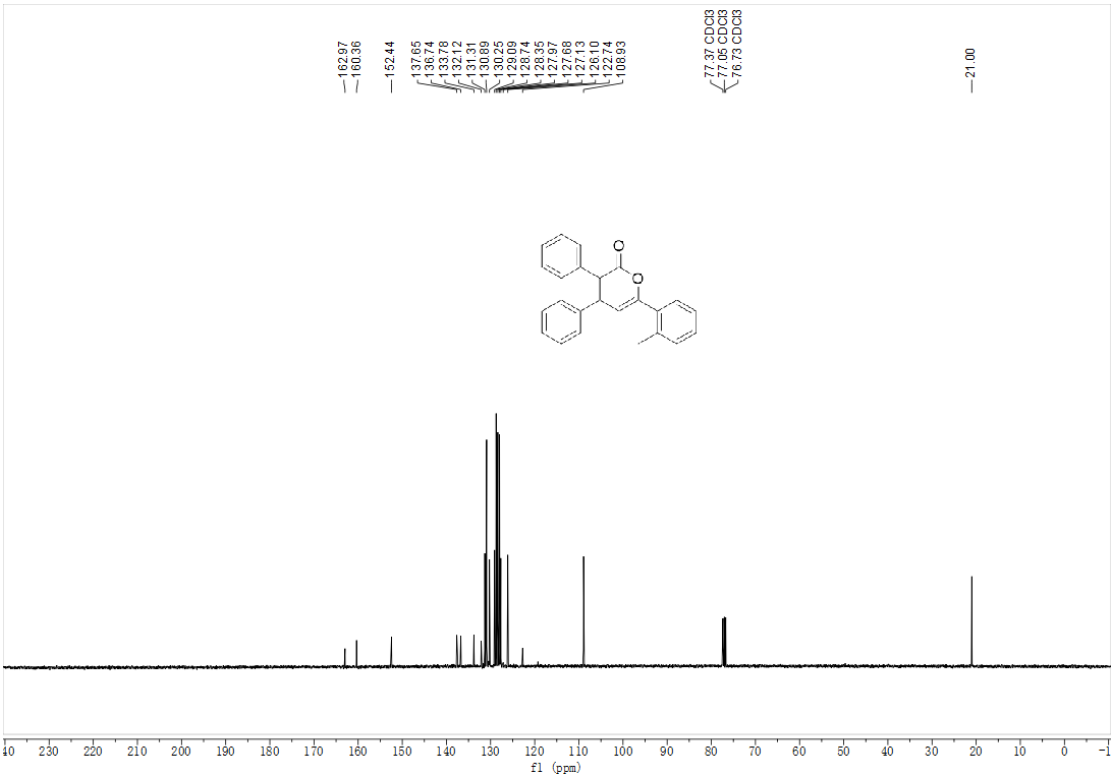
0.95
3.00
f1 (ppm)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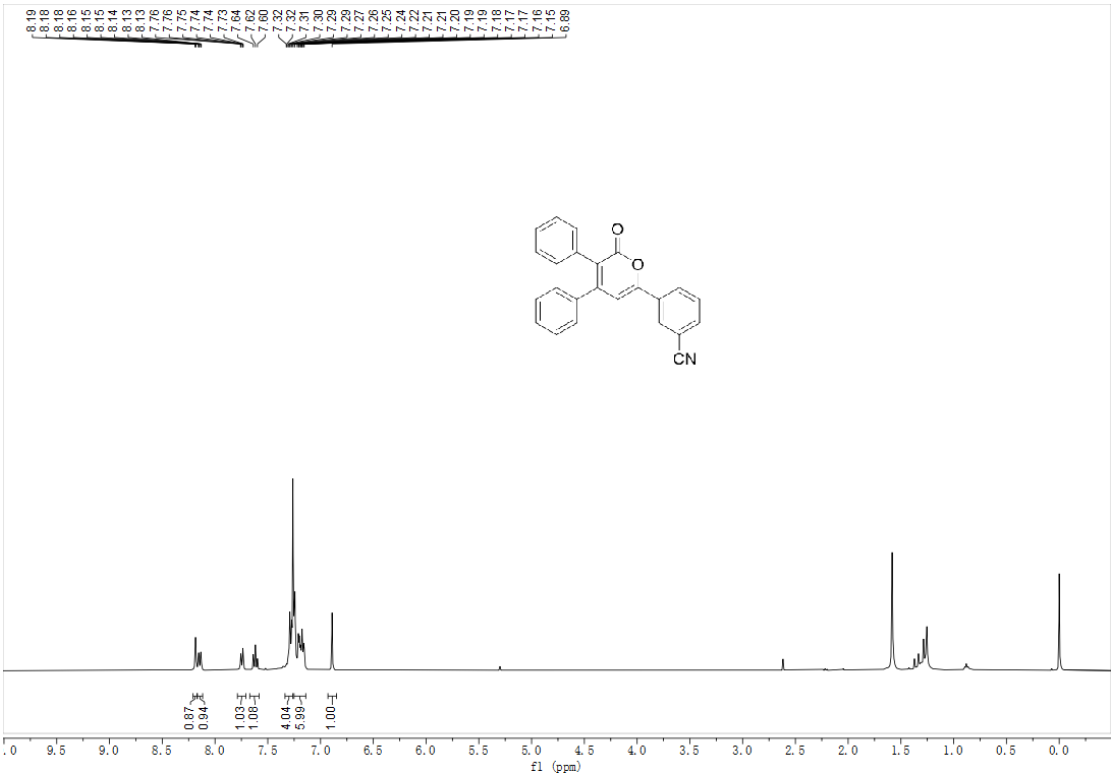
76.73 CDCl3
21.00
f1 (ppm)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7.21
7.20
7.19
7.19
7.18
7.17
7.17
7.16
7.15
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O
CN
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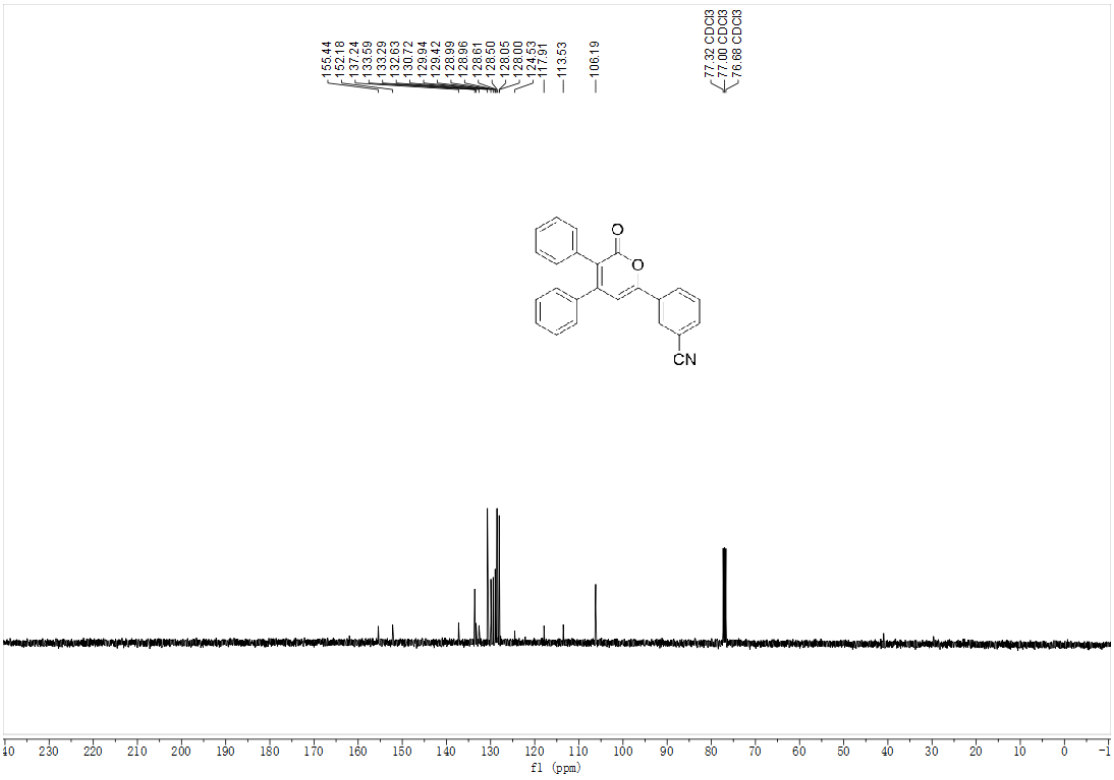
4.04
5.99
1.00
f1 (ppm)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128.96
128.61
128.51
128.05
128.00
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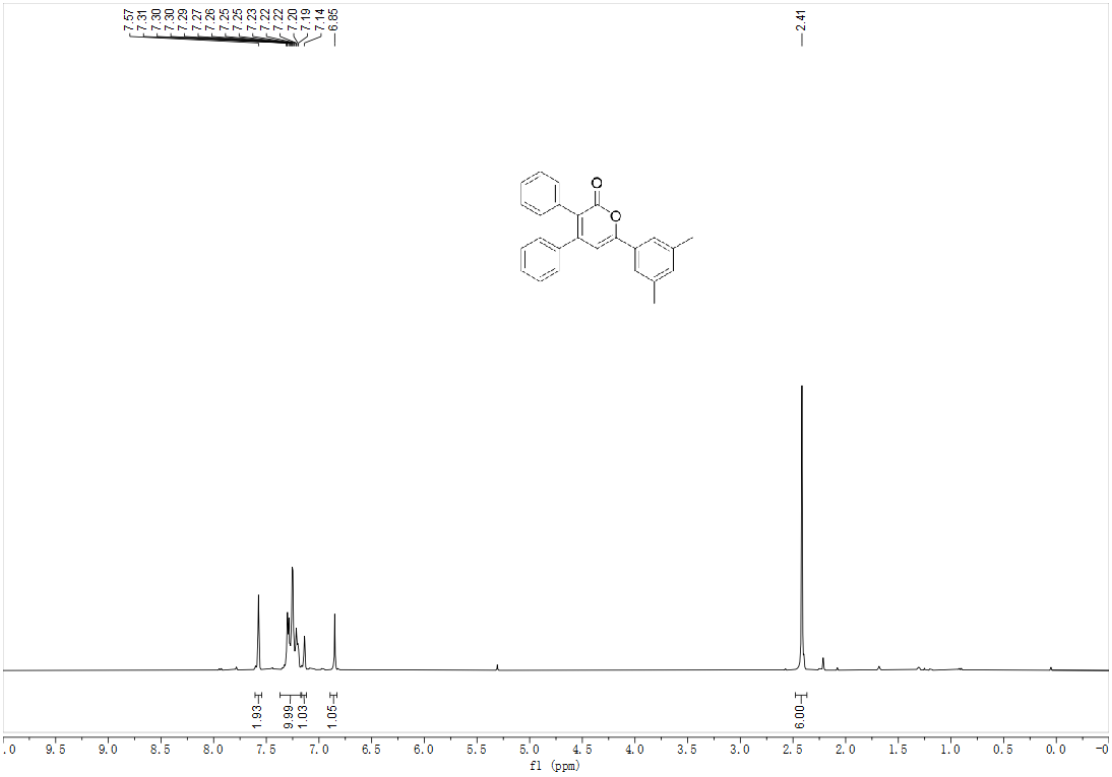
117.91
113.53
106.19
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
O
O
CN
f1 (ppm)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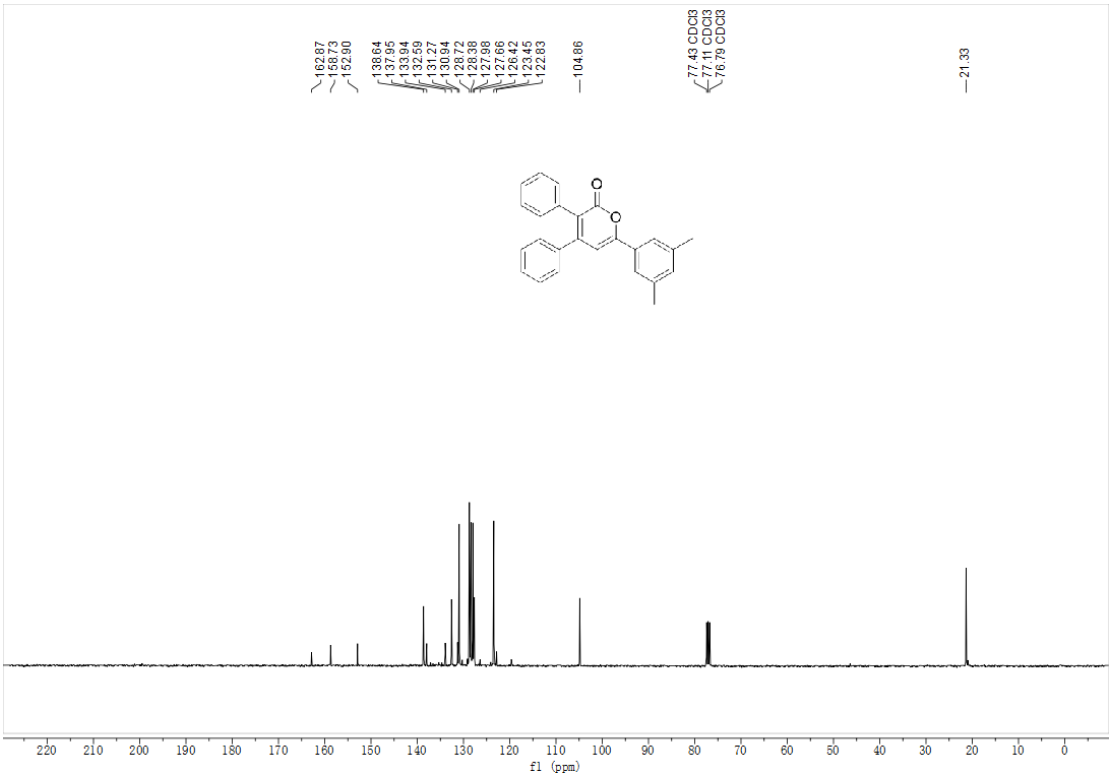
1.03
1.05
6.00
f1 (ppm)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77.11 CDCl3
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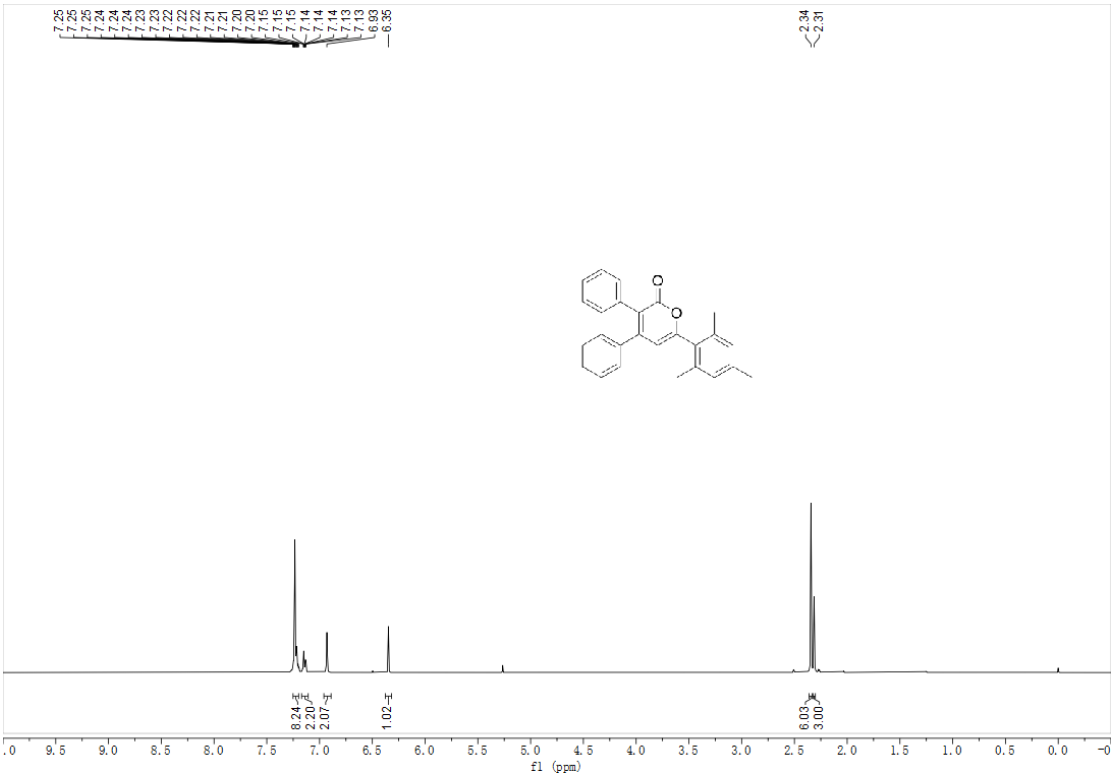
21.33
f1 (ppm)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7.15
7.14
7.14
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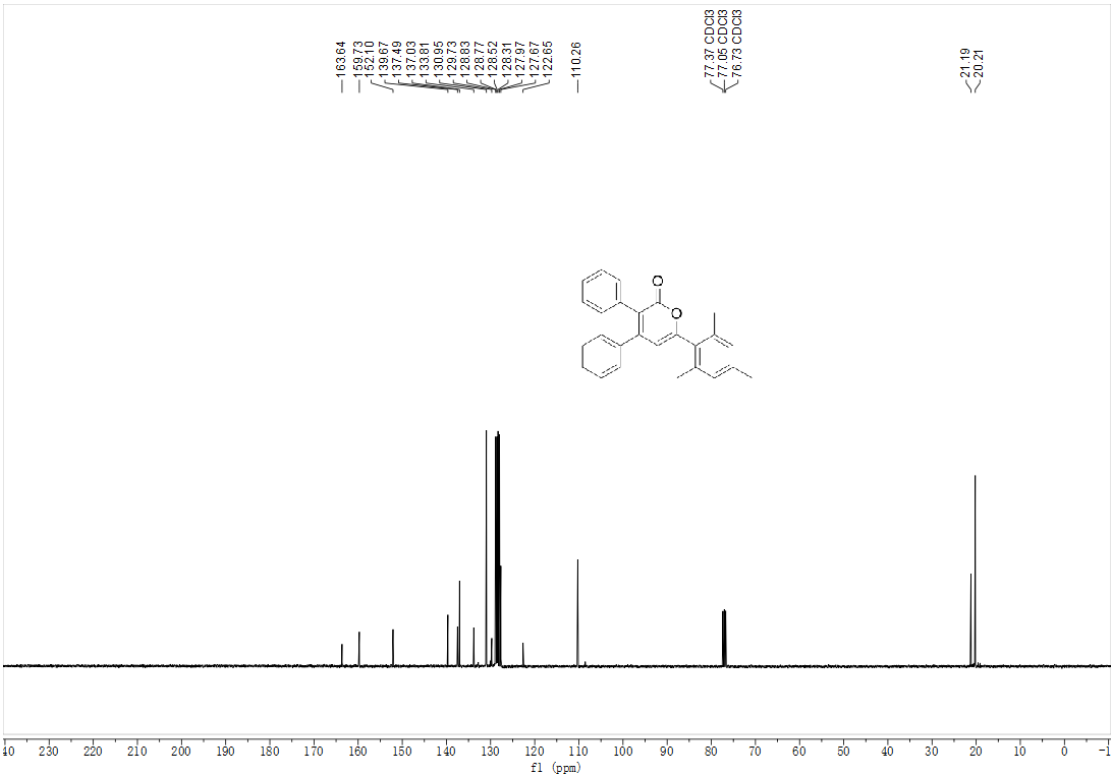
8.24
2.20
2.07
1.02
6.03
3.00
f1 (ppm)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77.05 CDCl3
76.73 CDCl3
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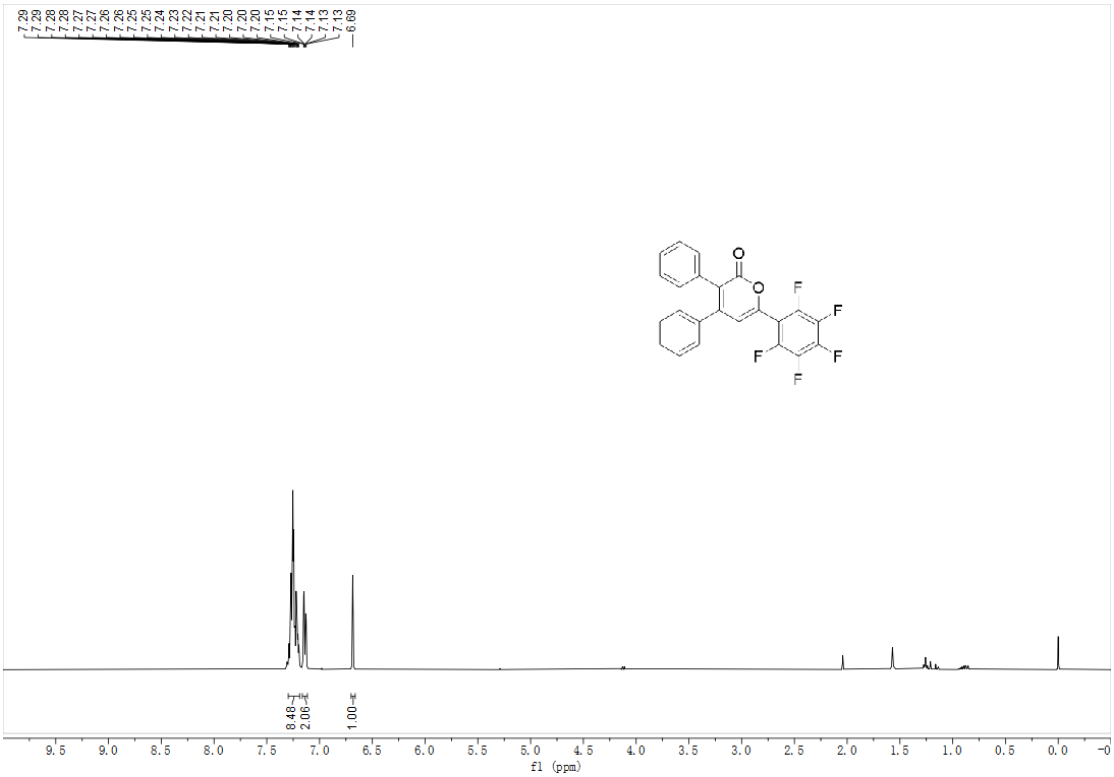
20.21
f1 (ppm)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7.20
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7.20
7.15
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7.14
7.13
7.13
6.69
O
O
F
F
F
F
F
8.48
2.06
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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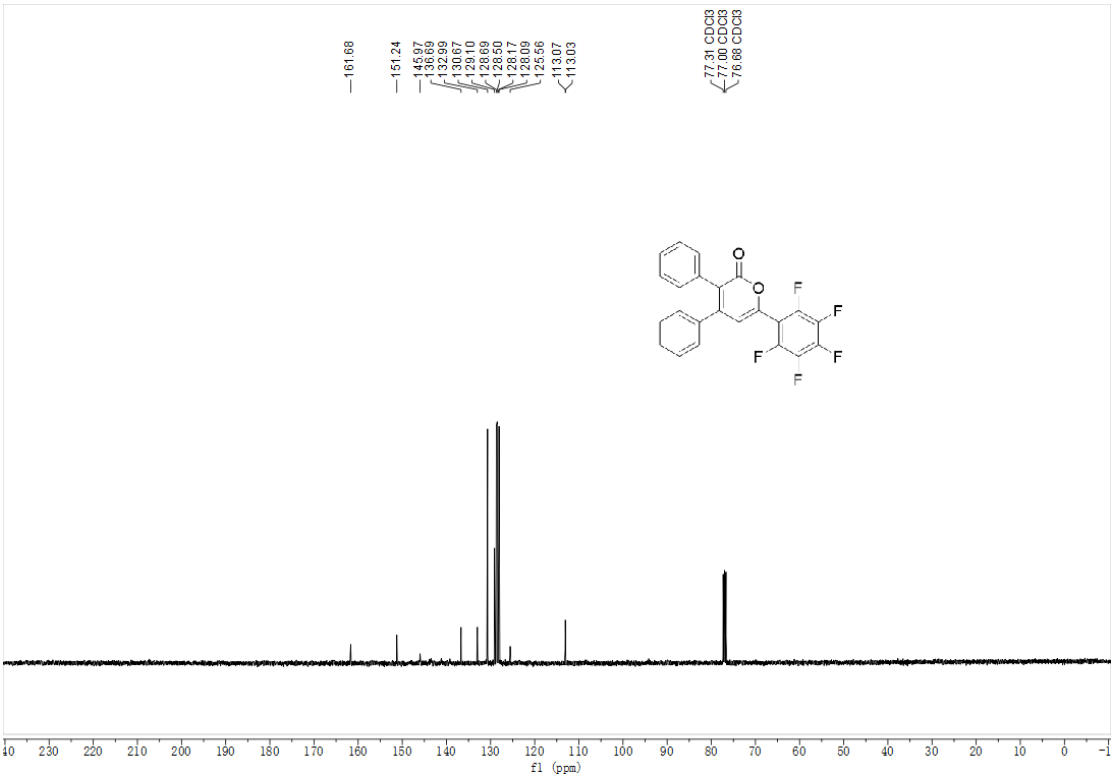
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77.00 CDCl3
76.68 CDCl3
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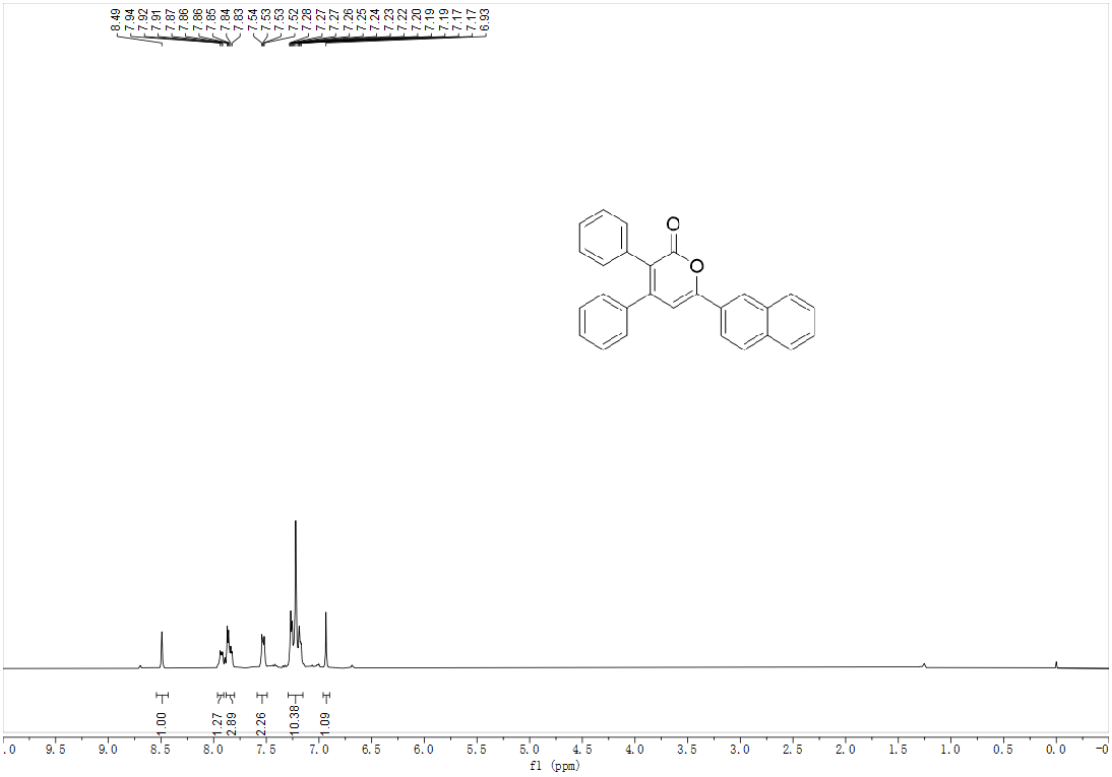
f1 (ppm)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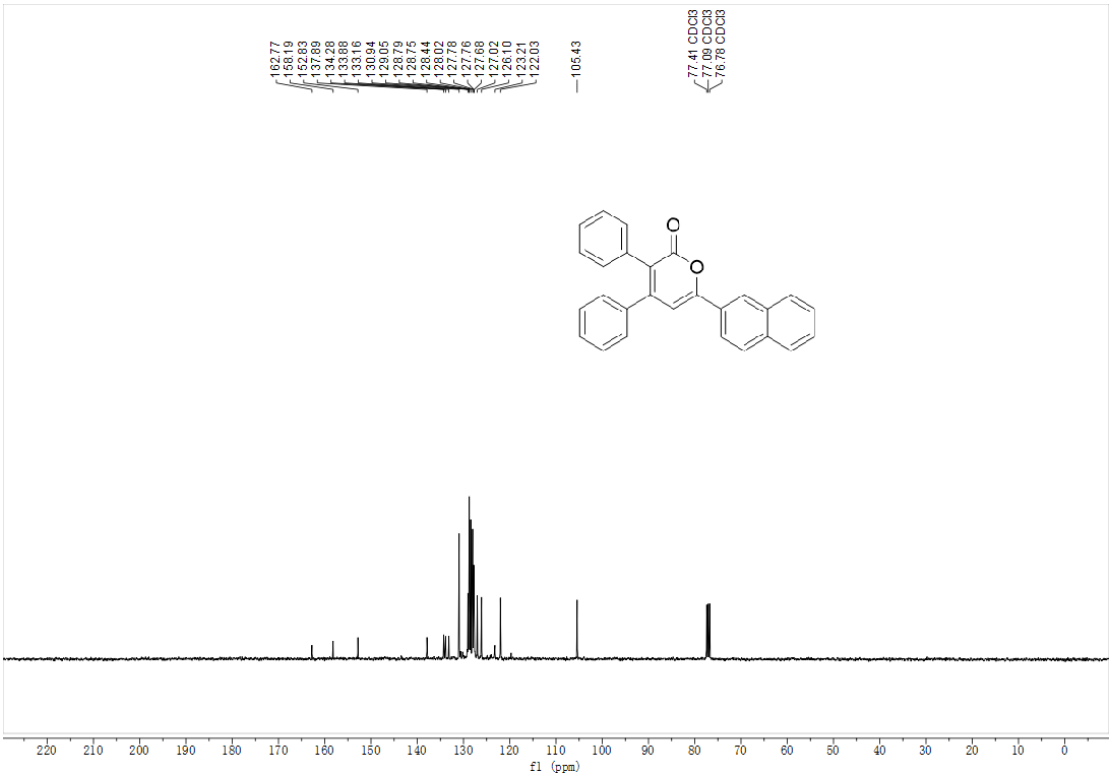
1.09
f1 (ppm)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127.76
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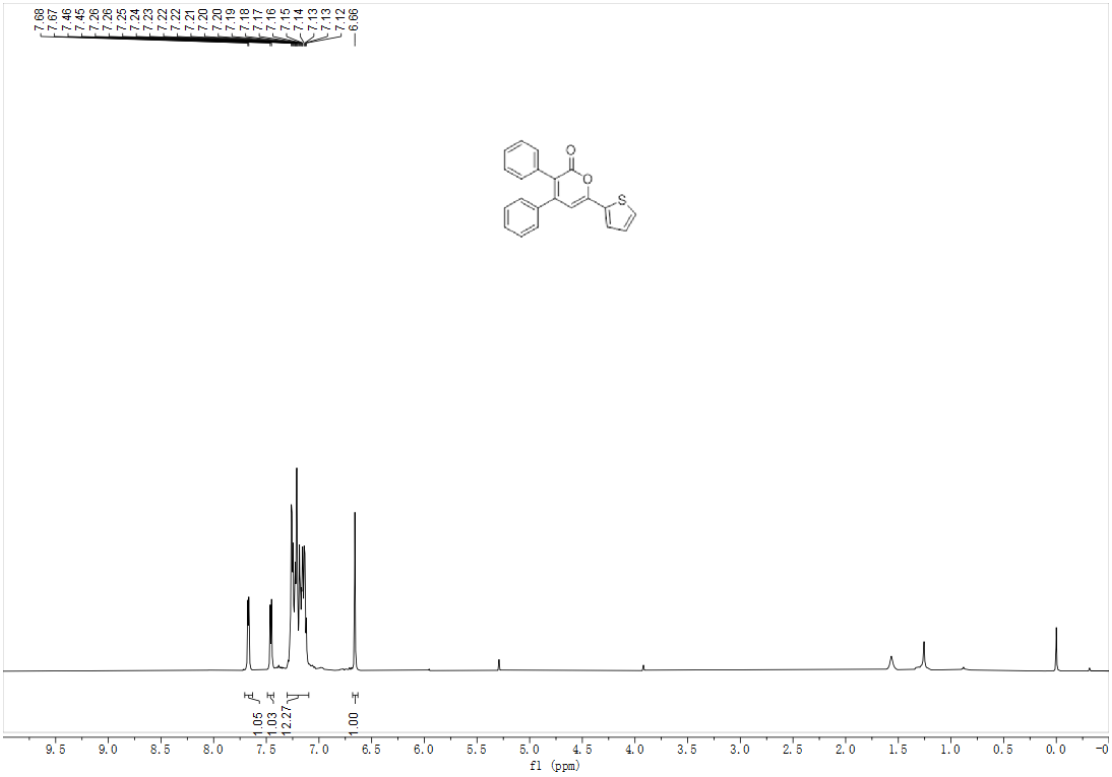
77.09 CDCl3
76.78 CDCl3
f1 (ppm)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7.15
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7.12
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O
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S
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1.03
2.27
1.00
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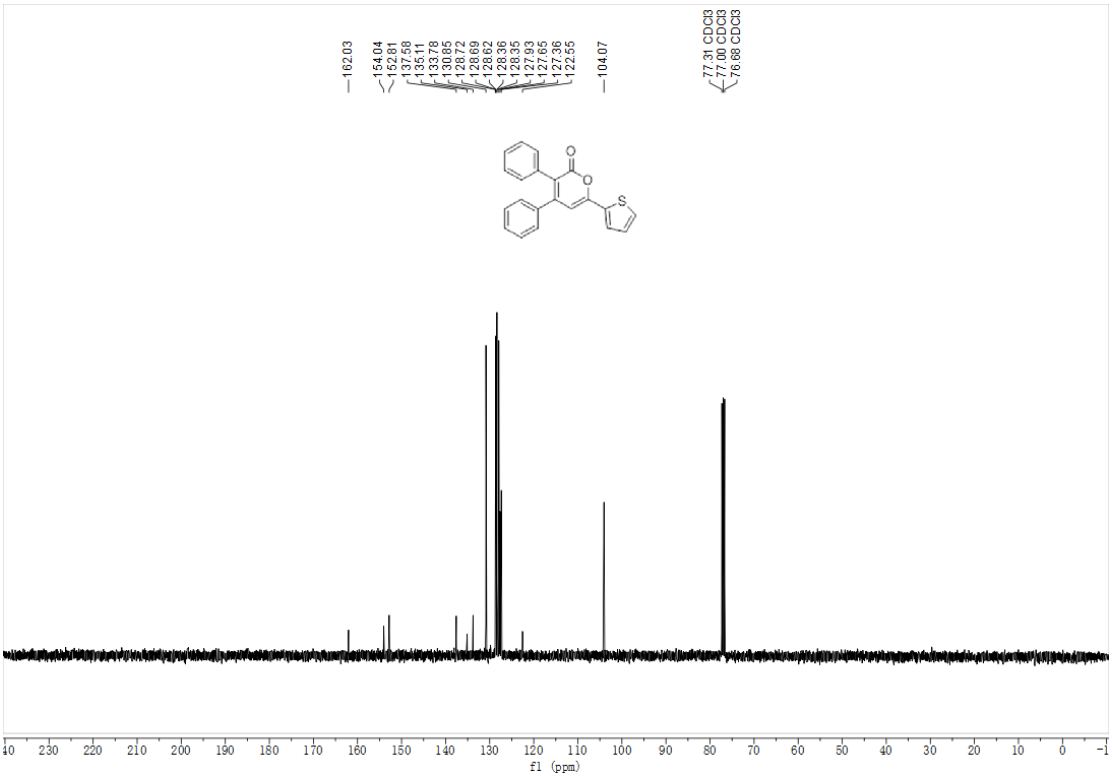
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104.07
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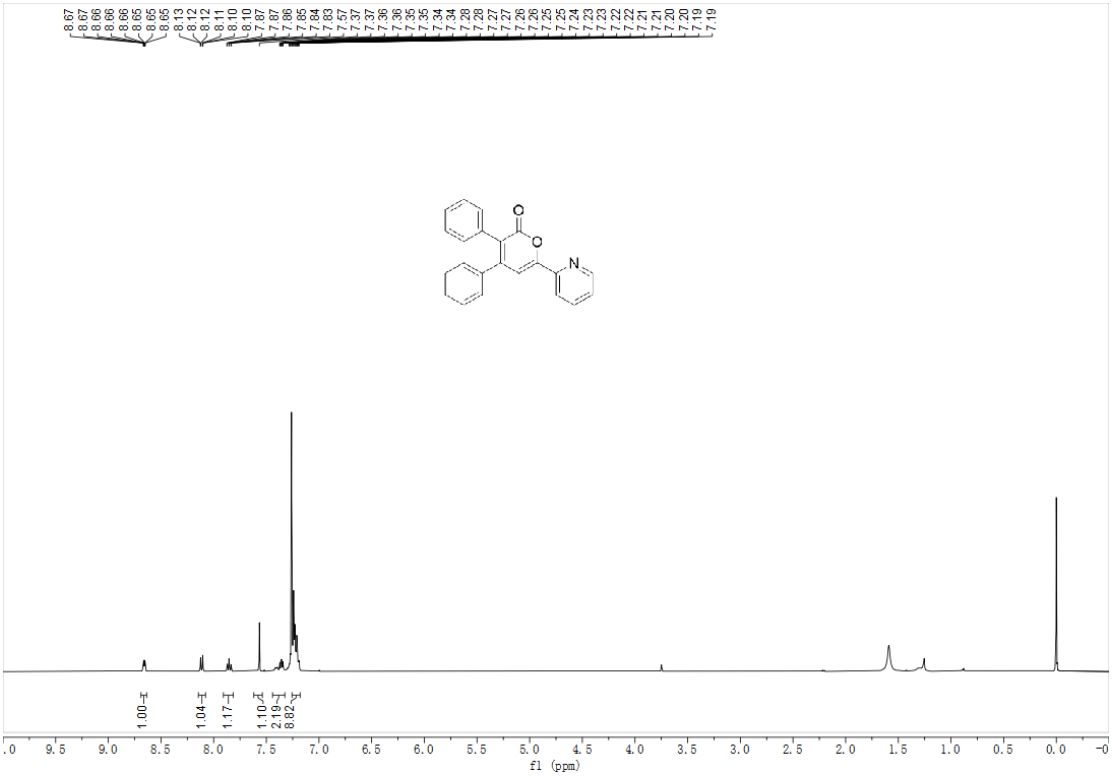
77.00 CDCl3
76.68 CDCl3
O
O
S
f1 (ppm)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1.10
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8.82
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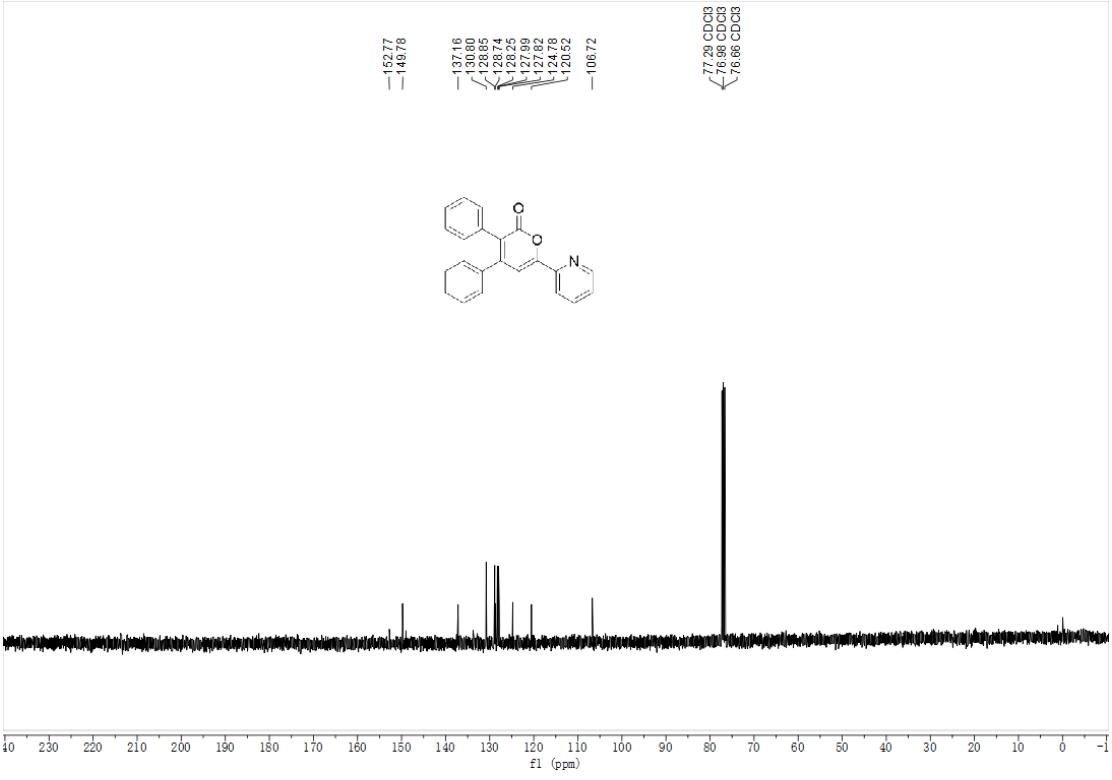
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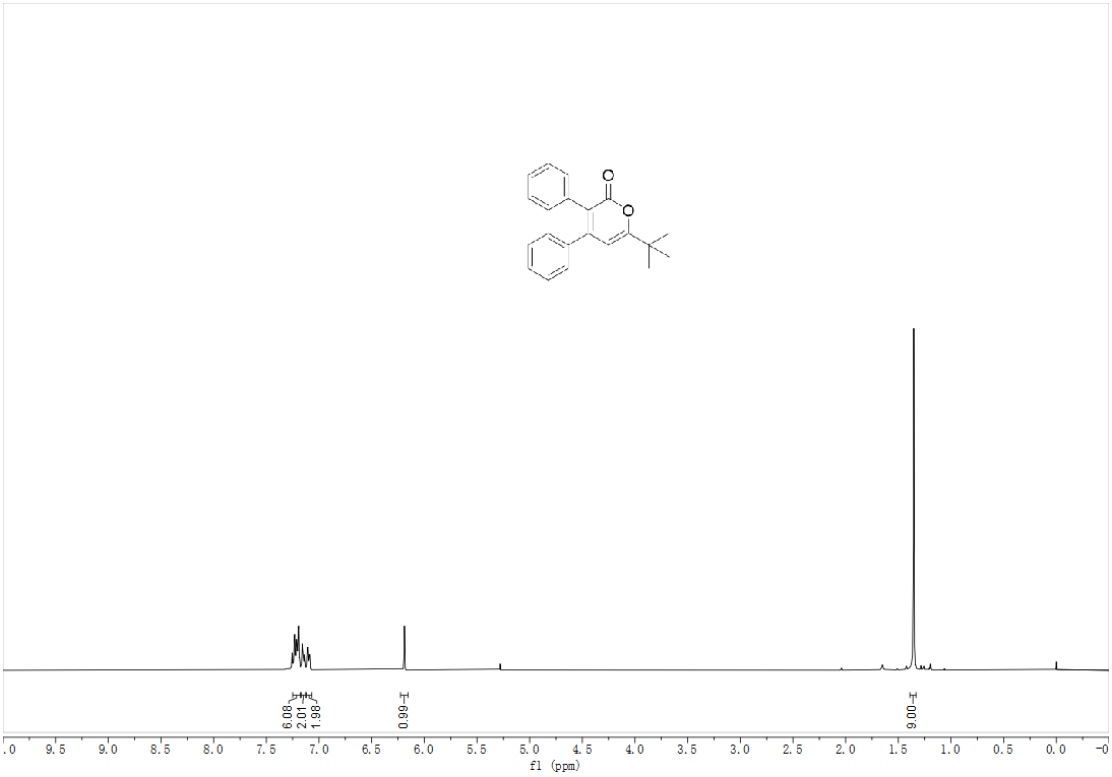
124.78
120.52
106.72
77.29 CDCl3
76.98 CDCl3
76.66 CDCl3
O
O
N
f1 (ppm)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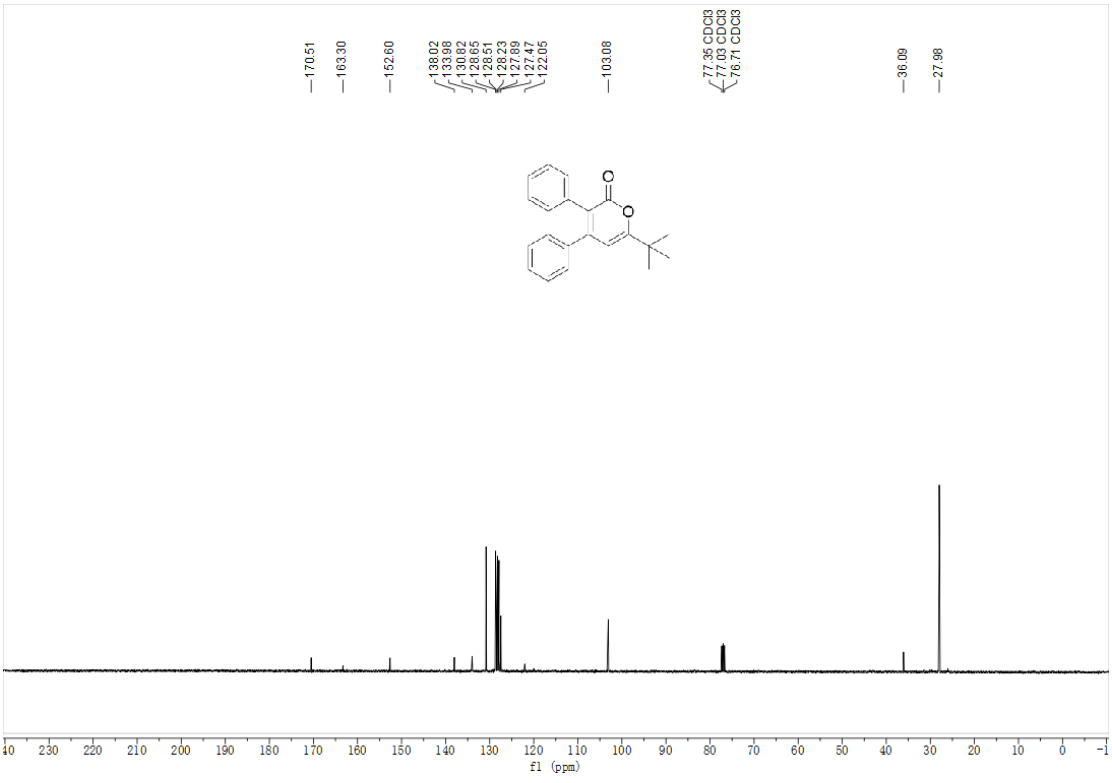
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103.08
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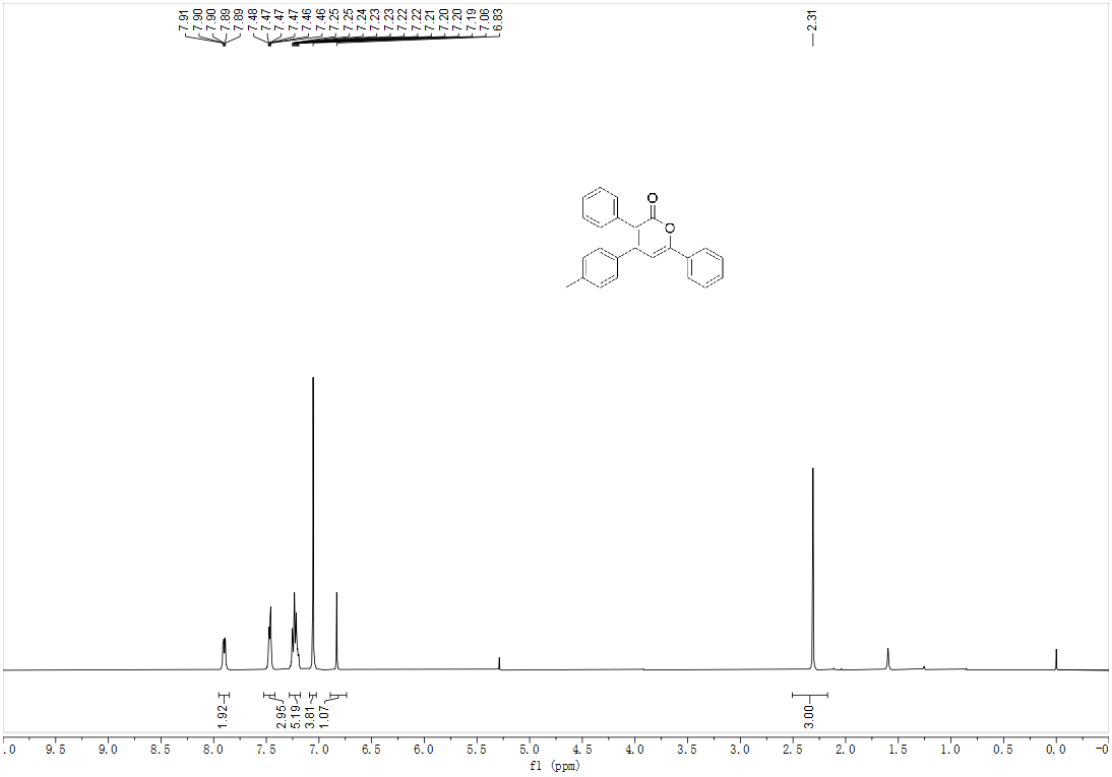
77.03 CDCl3
76.71 CDCl3
36.09
27.98
f1 (ppm)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7.22
7.22
7.21
7.20
7.20
7.19
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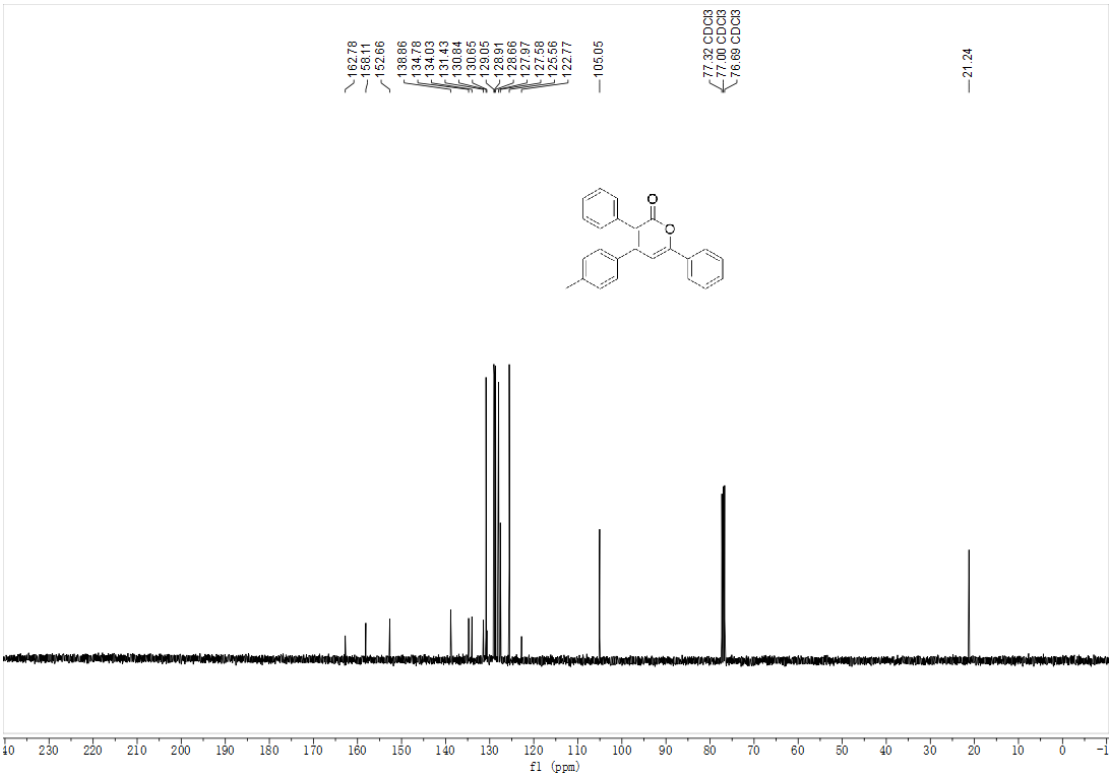
2.95
5.19
3.81
1.07
3.00
f1 (ppm)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127.97
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105.05
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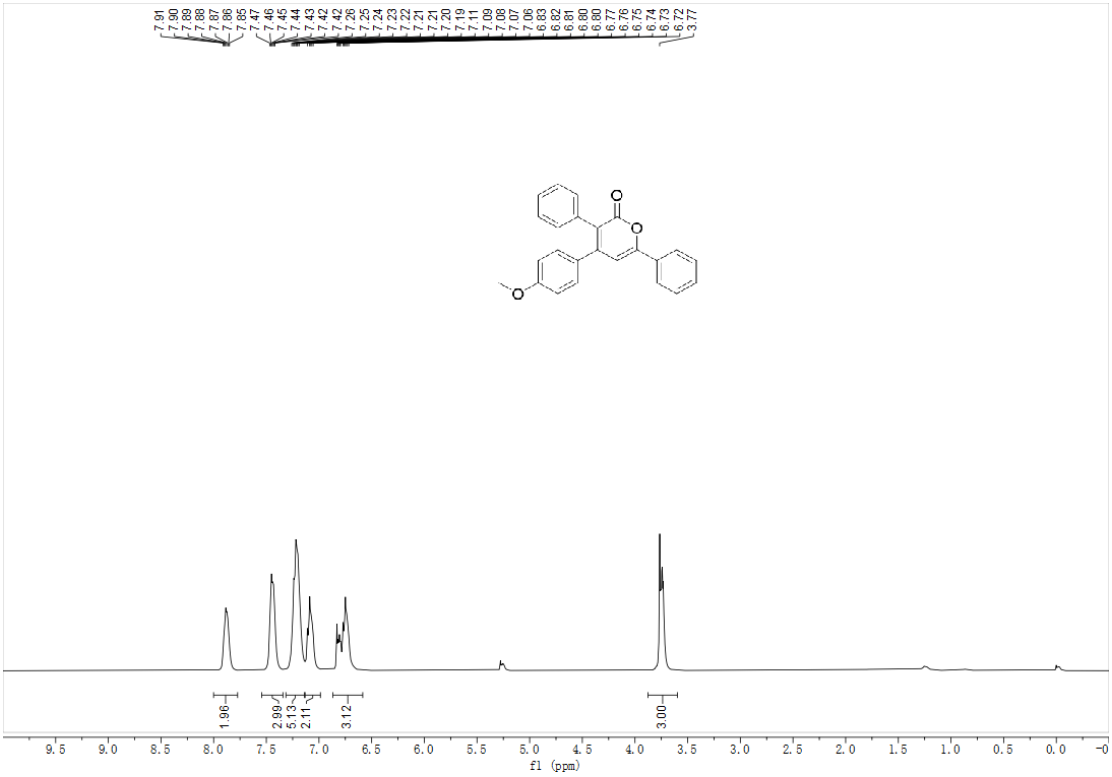
77.00 CDCl3
76.69 CDCl3
21.24
O
O
f1 (ppm)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7.08
7.07
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6.82
6.81
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6.80
6.77
6.76
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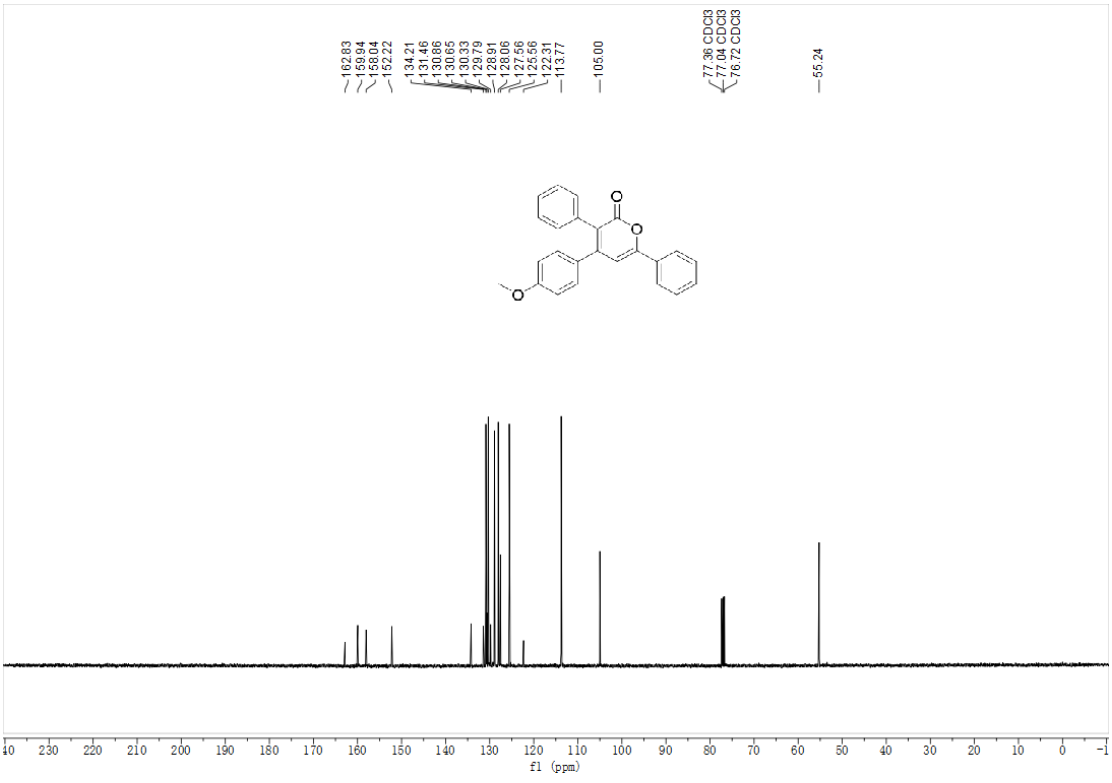
3.00
f1 (ppm)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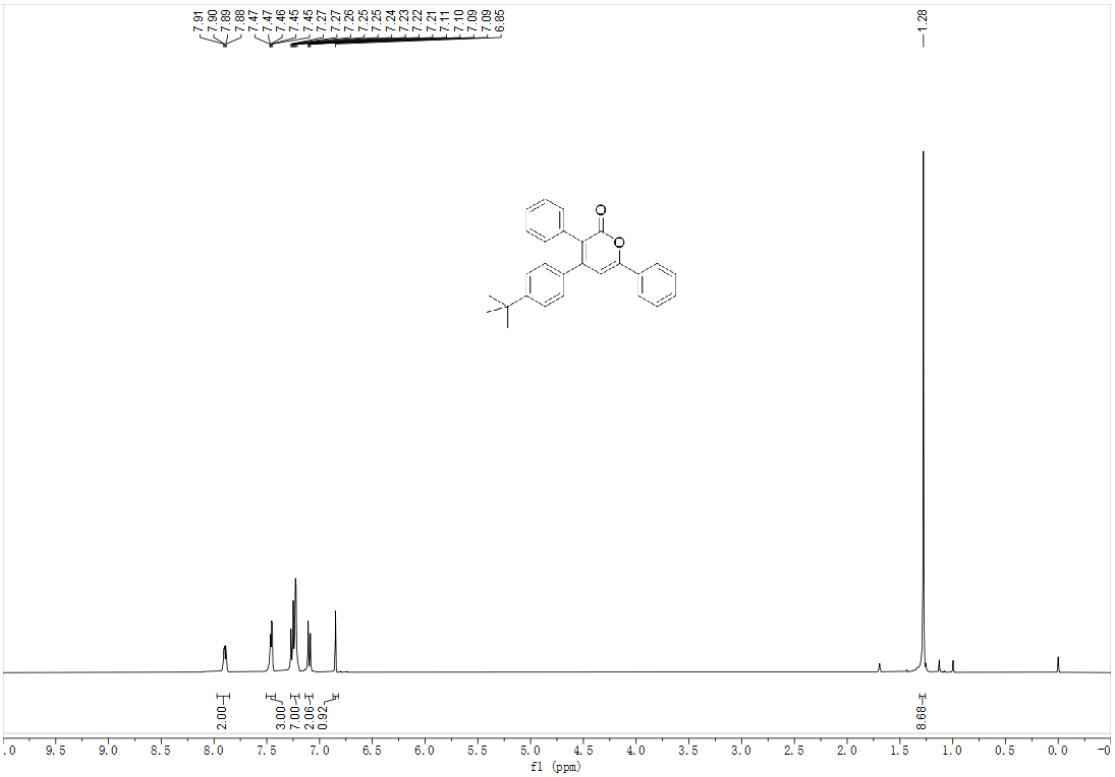
77.36 CDCl3
77.04 CDCl3
76.72 CDCl3
55.24
f1 (ppm)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7.11
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3.00
7.00
2.06
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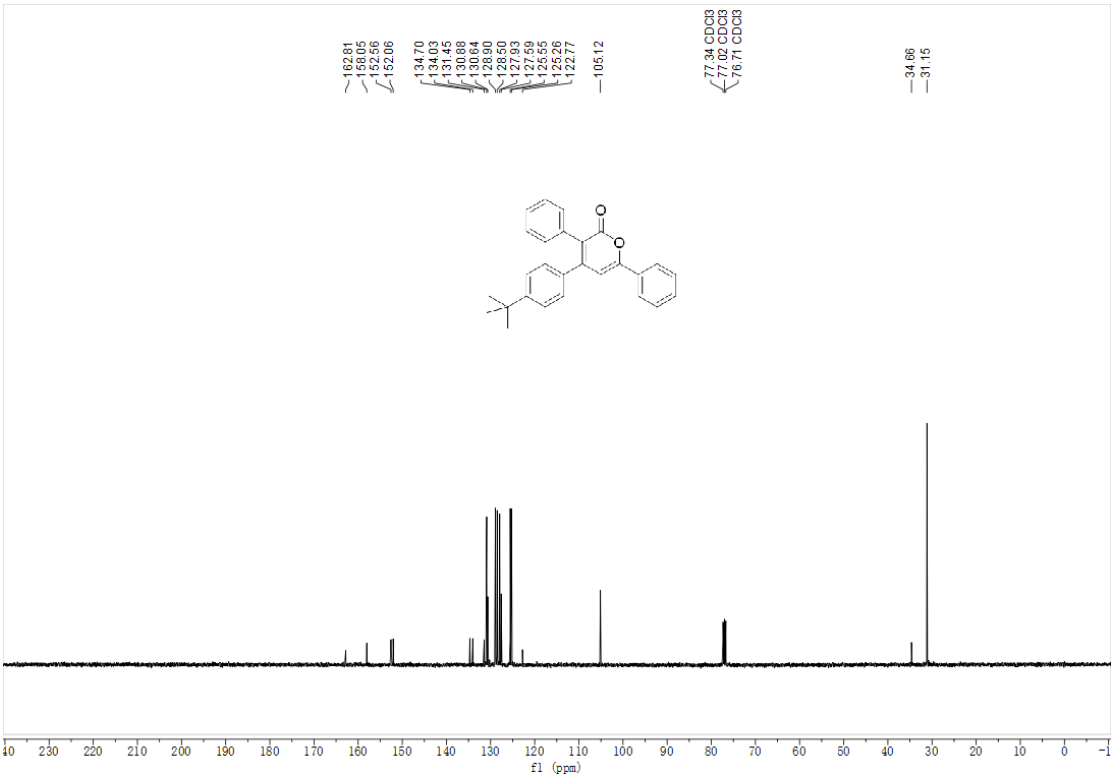
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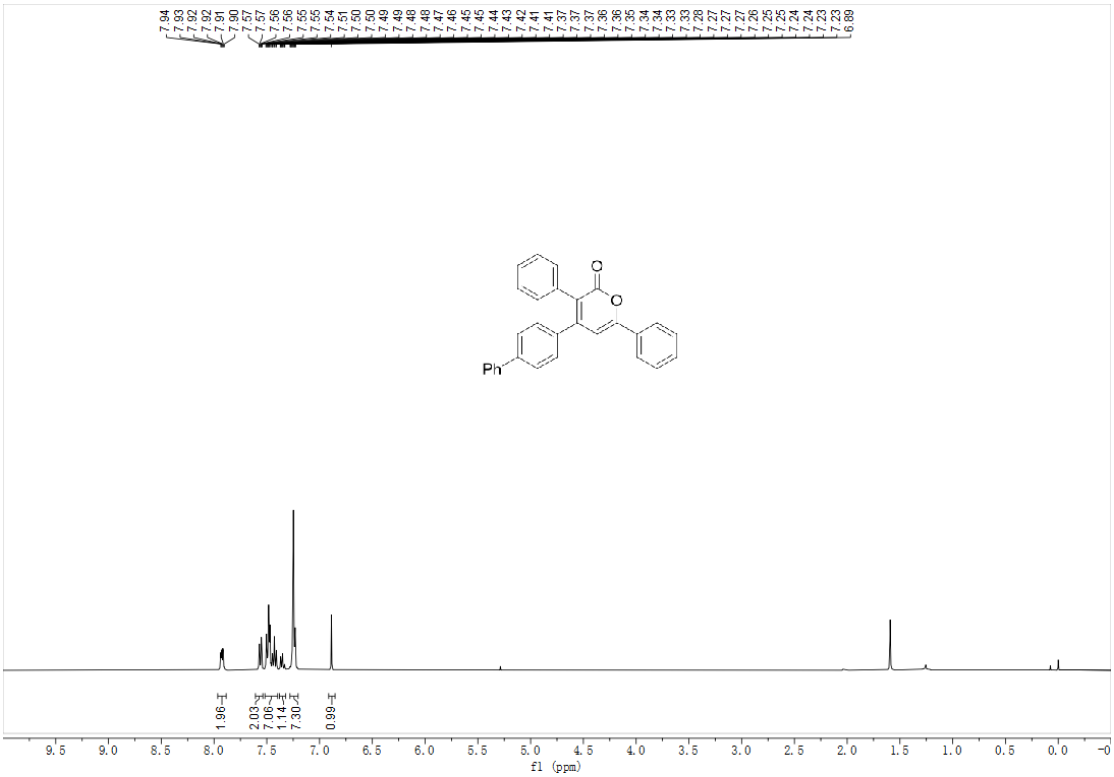
76.71 CDCl3
34.66
31.15
f1 (ppm)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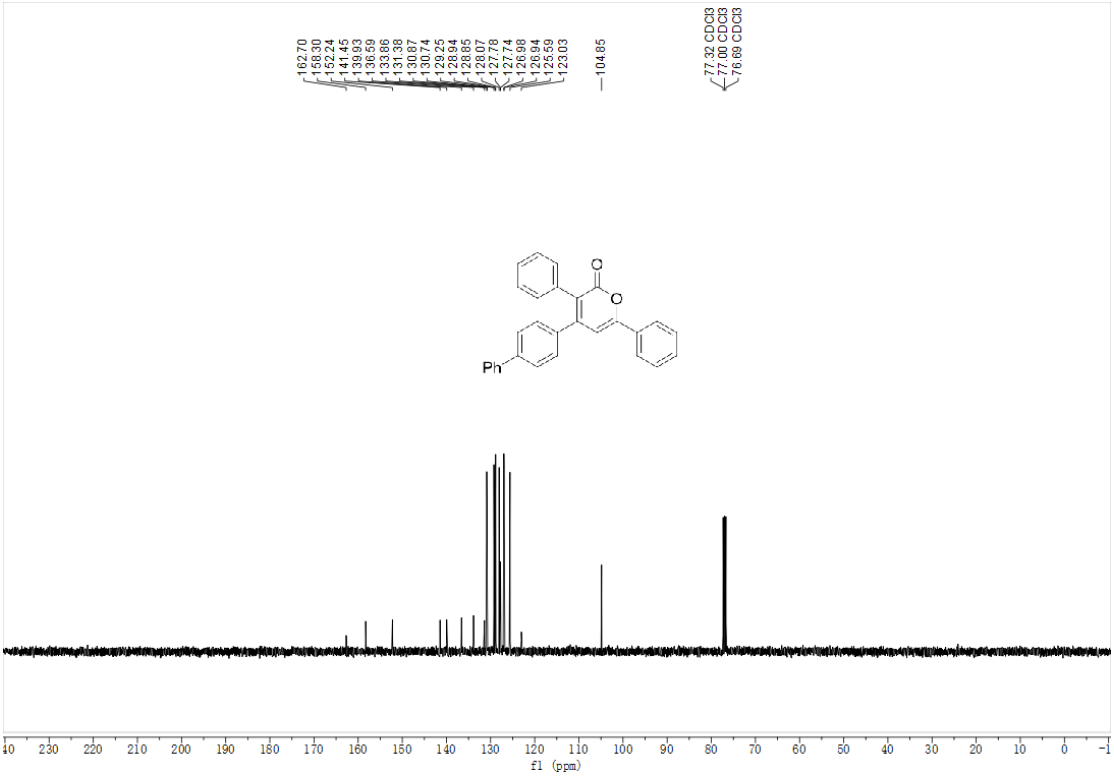
O
Ph
1.96
2.03
7.10
1.14
7.30
0.99
f1 (ppm)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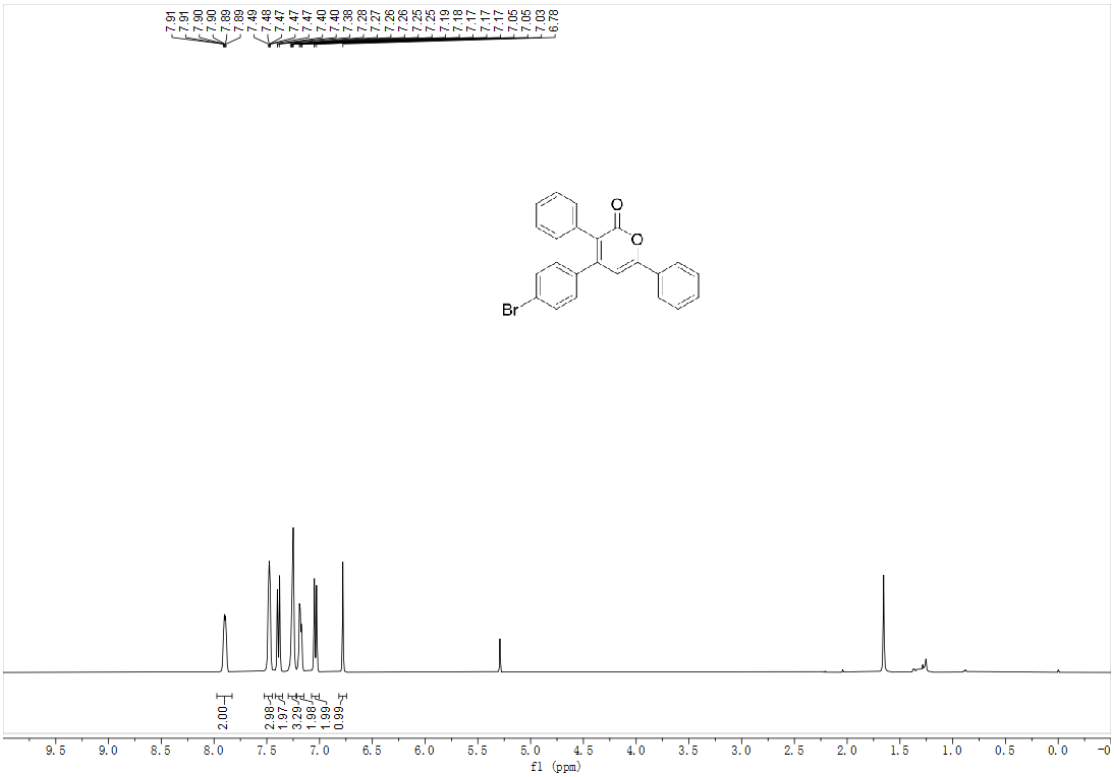
77.32 CDCl3 77.00 CDCl3 76.69 CDCl3
O
O
Ph
f1 (ppm)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7.26
7.25
7.25
7.19
7.18
7.17
7.17
7.17
7.05
7.03
6.78
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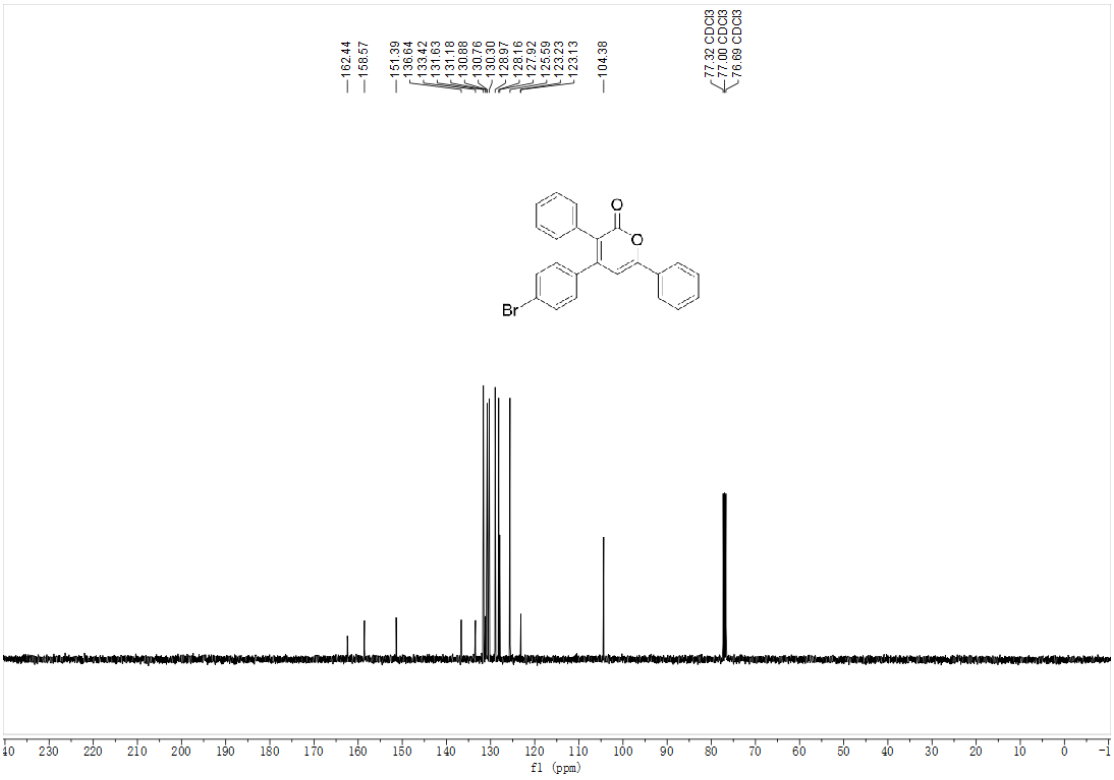
O
Br
2.00
2.98
1.97
3.29
1.90
1.99
0.99
f1 (ppm)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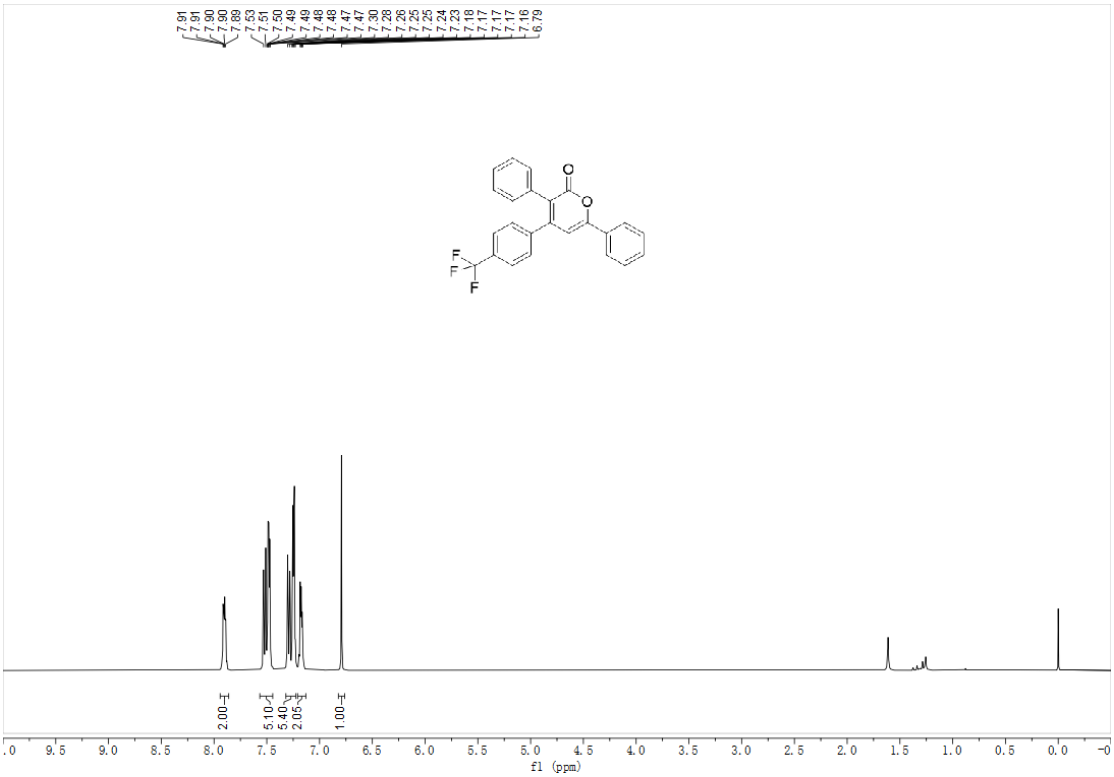
77.00 CDCl3
76.69 CDCl3
O
O
Br
f1 (ppm)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7.25
7.24
7.23
7.18
7.17
7.17
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F
F
F
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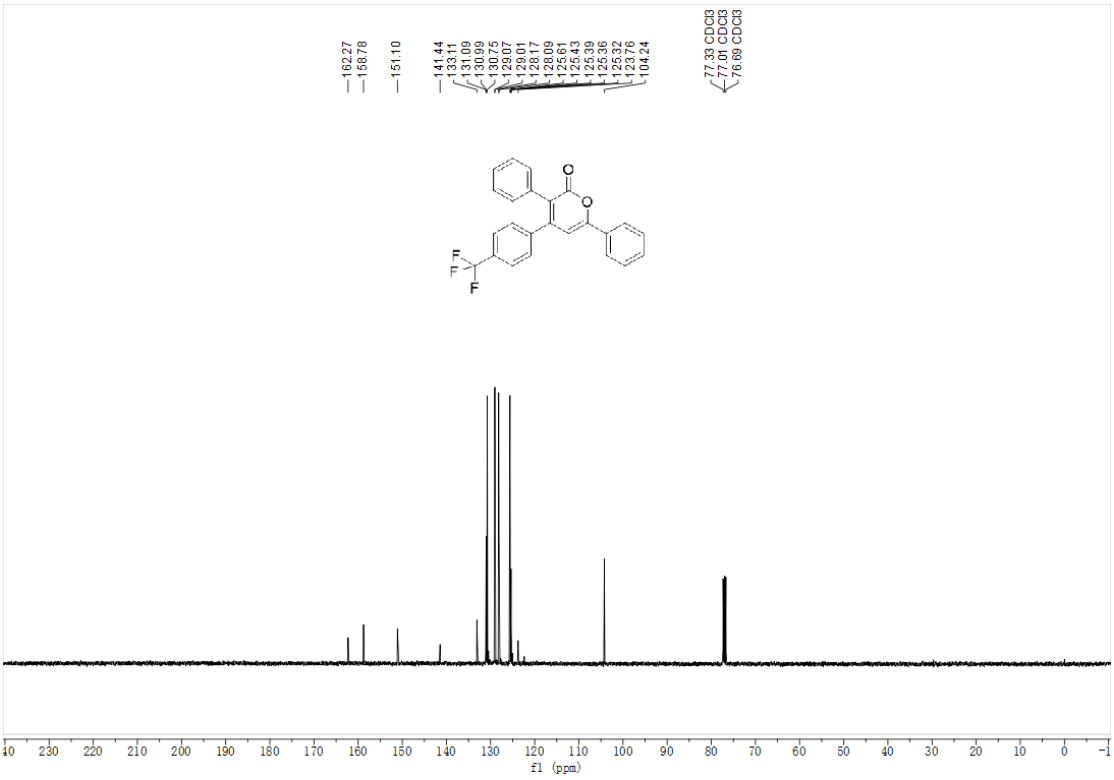
5.10
5.40
2.05
1.00
f1 (ppm)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125.36
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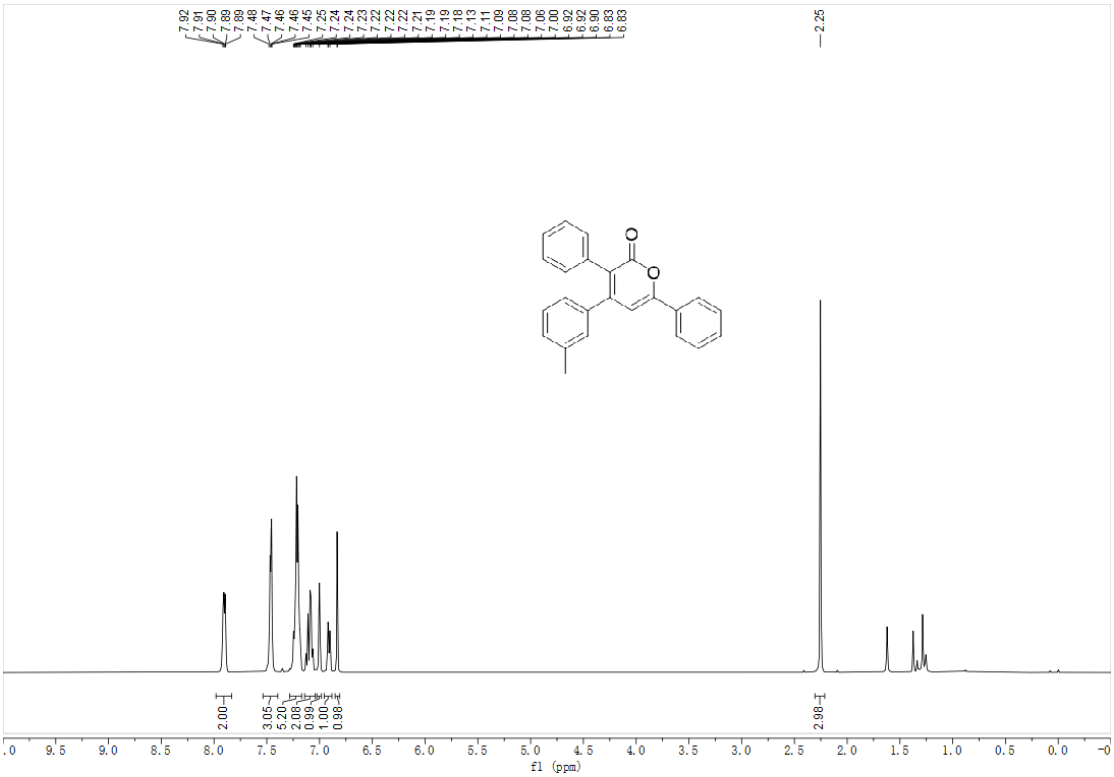
77.33 CDCl3
77.01 CDCl3
76.69 CDCl3
O
O
F
F
F
f1 (ppm)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7.13
7.11
7.09
7.08
7.08
7.06
7.00
6.92
6.92
6.90
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3.05
5.28
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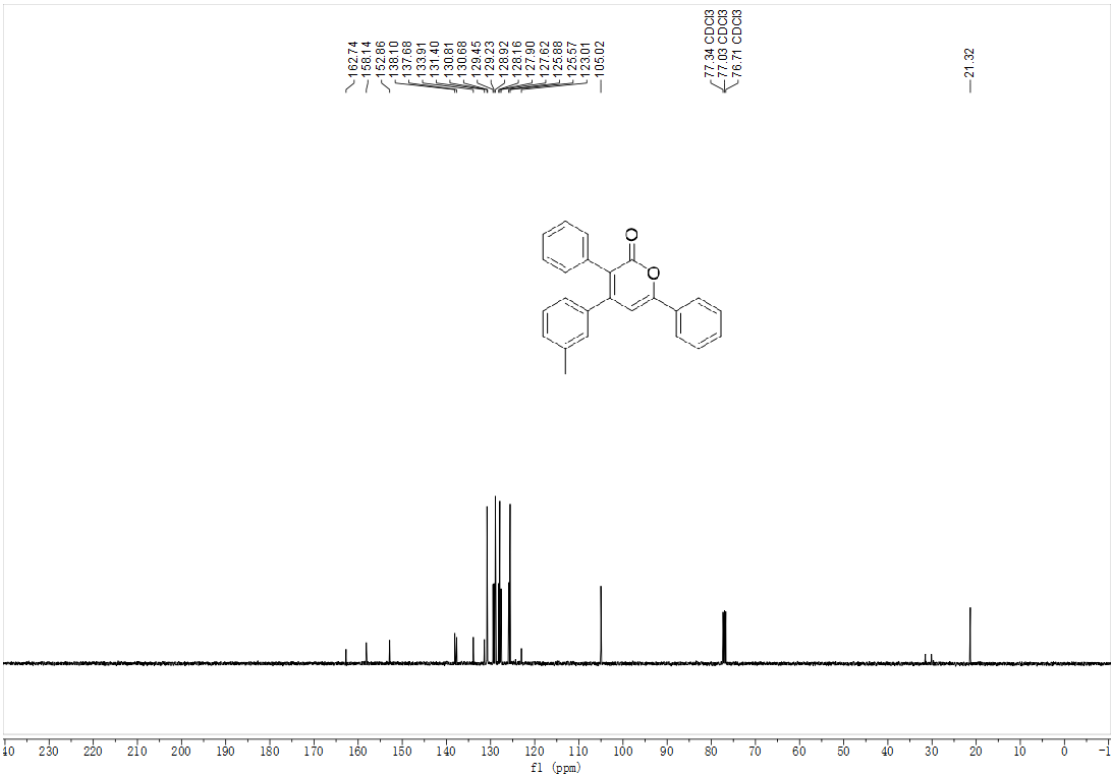
2.98
f1 (ppm)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127.90
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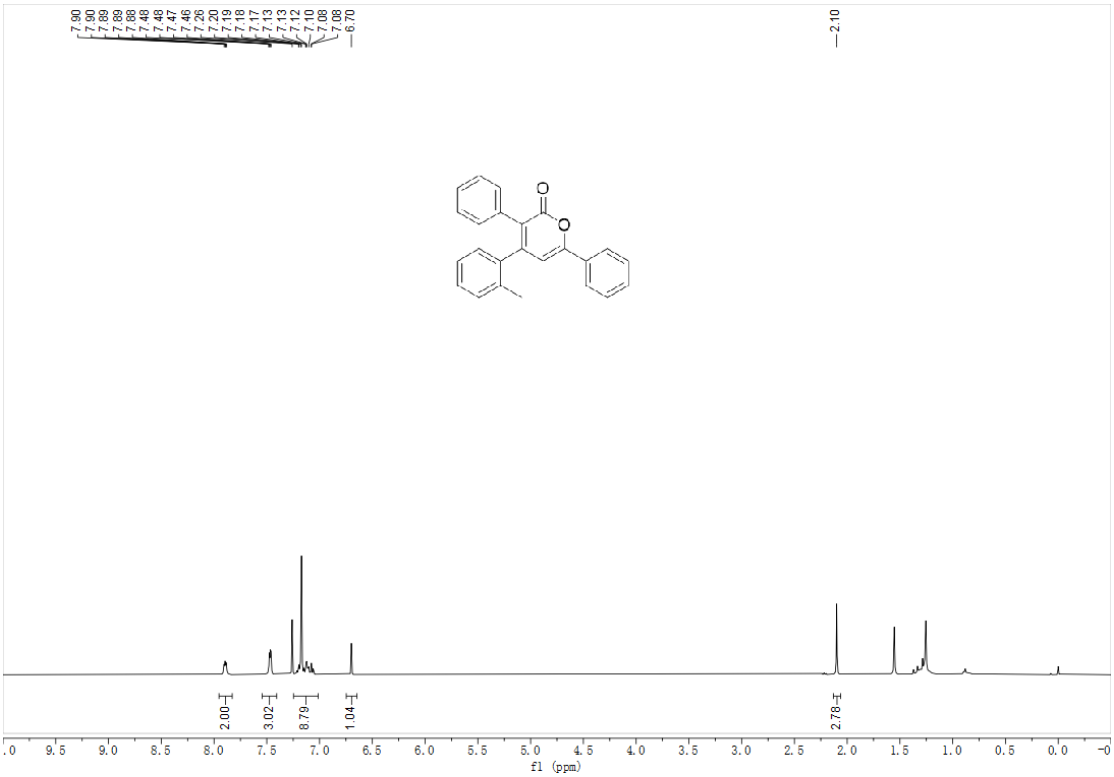
105.02
77.34 CDCl3
77.03 CDCl3
76.71 CDCl3
21.32
f1 (ppm)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7.18
7.17
7.13
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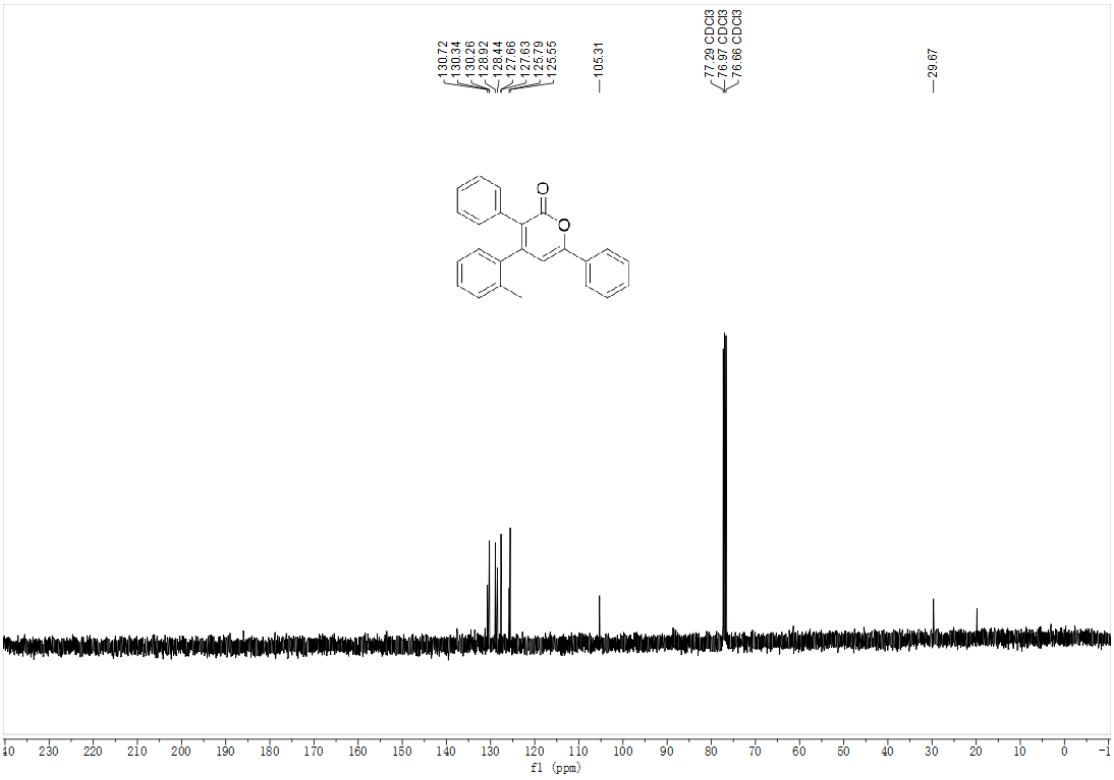
7.08
6.70
2.10
2.00
3.02
8.79
1.04
2.78
f1 (ppm)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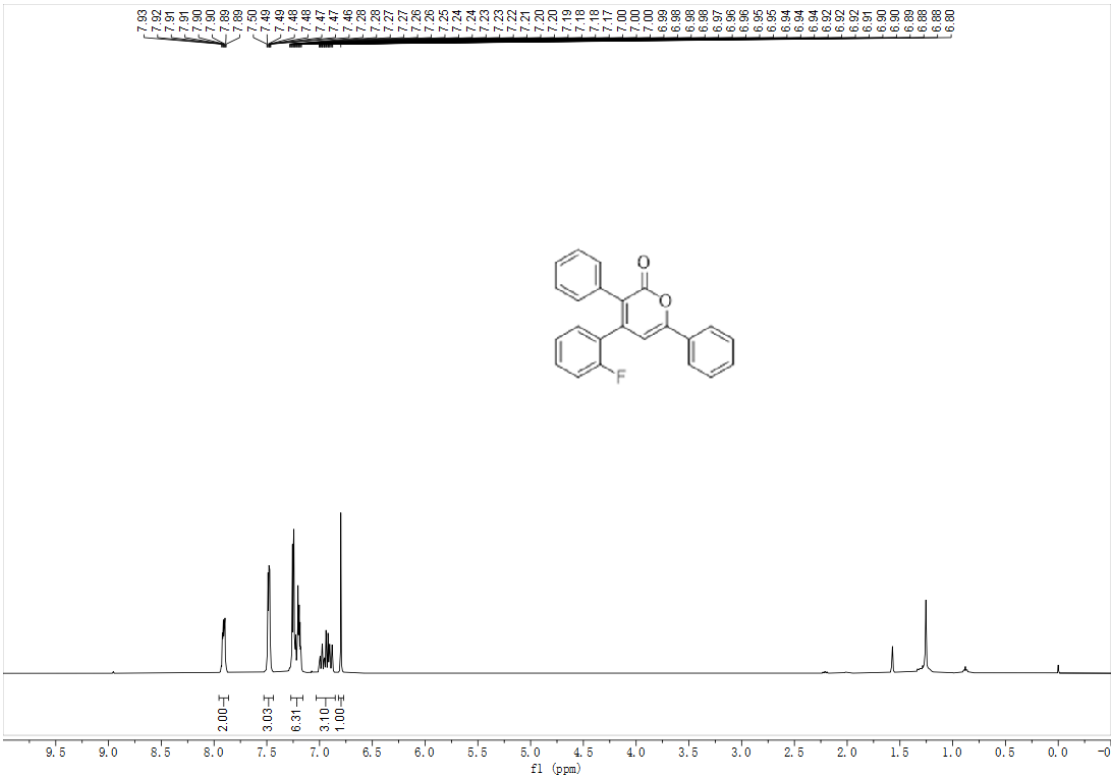
29.67
f1 (ppm)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O
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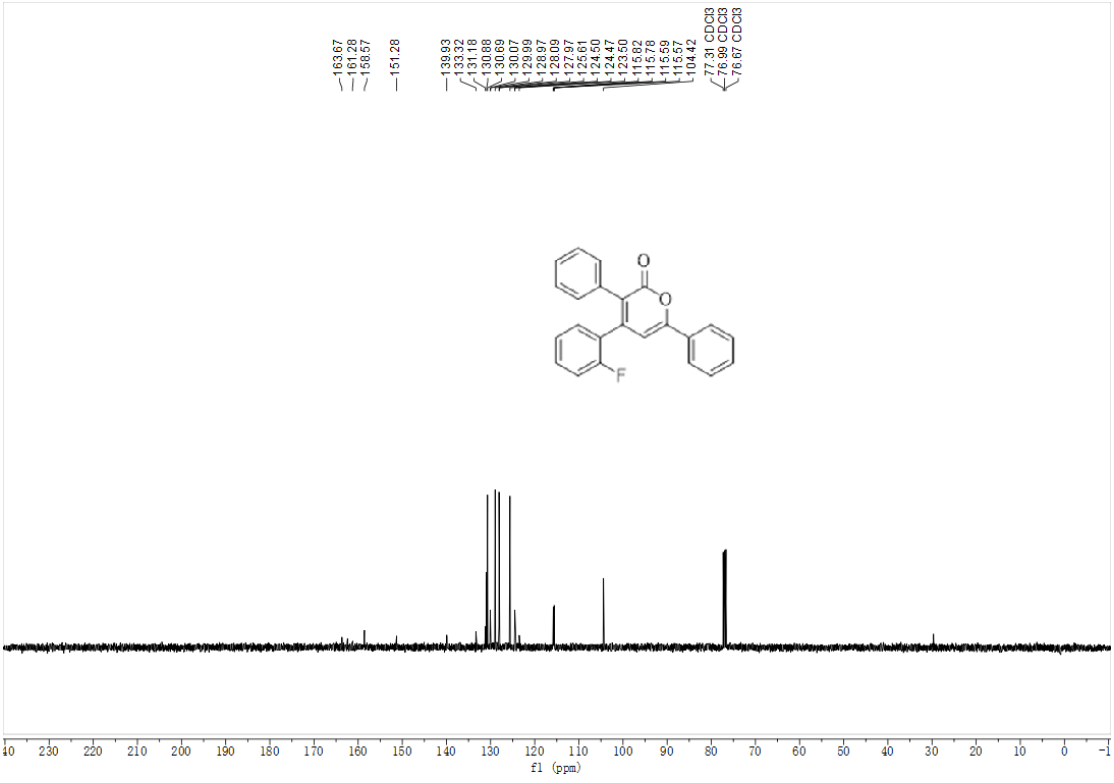
3.03
6.31
3.10
1.00
f1 (ppm)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76.67 CDCl3
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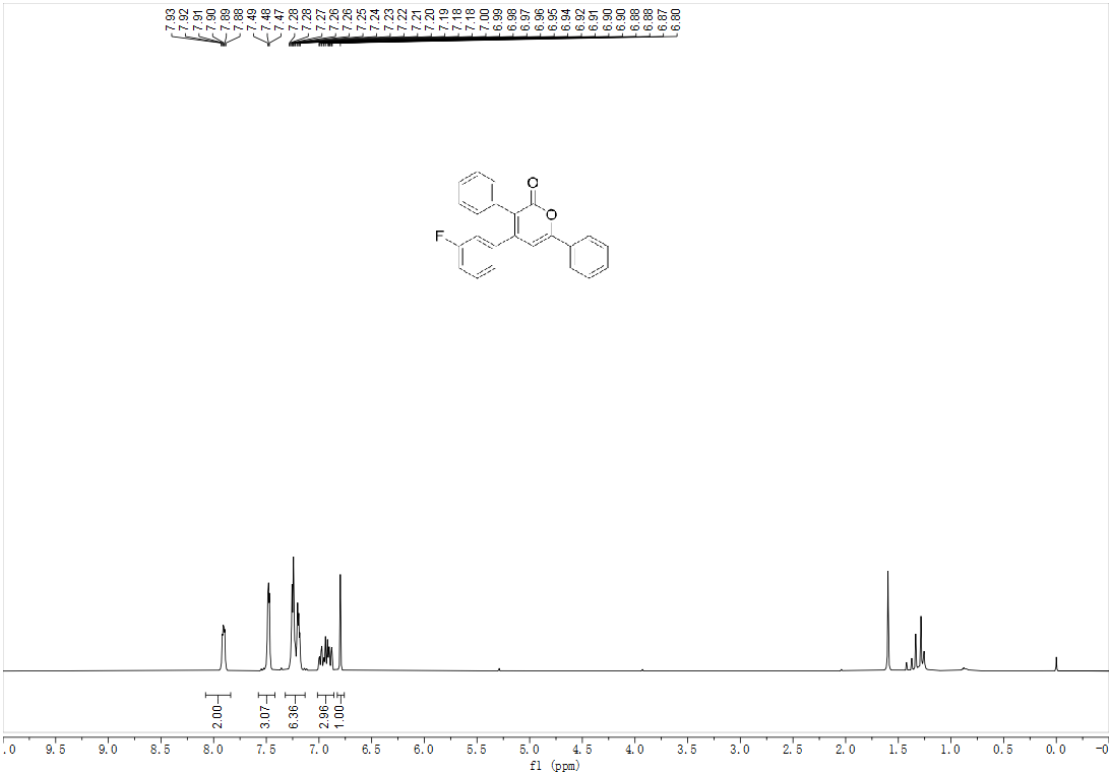
O
F
f1 (ppm)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7.19
7.18
7.18
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6.99
6.98
6.97
6.96
6.95
6.94
6.92
6.91
6.90
6.90
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6.88
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O
O
F
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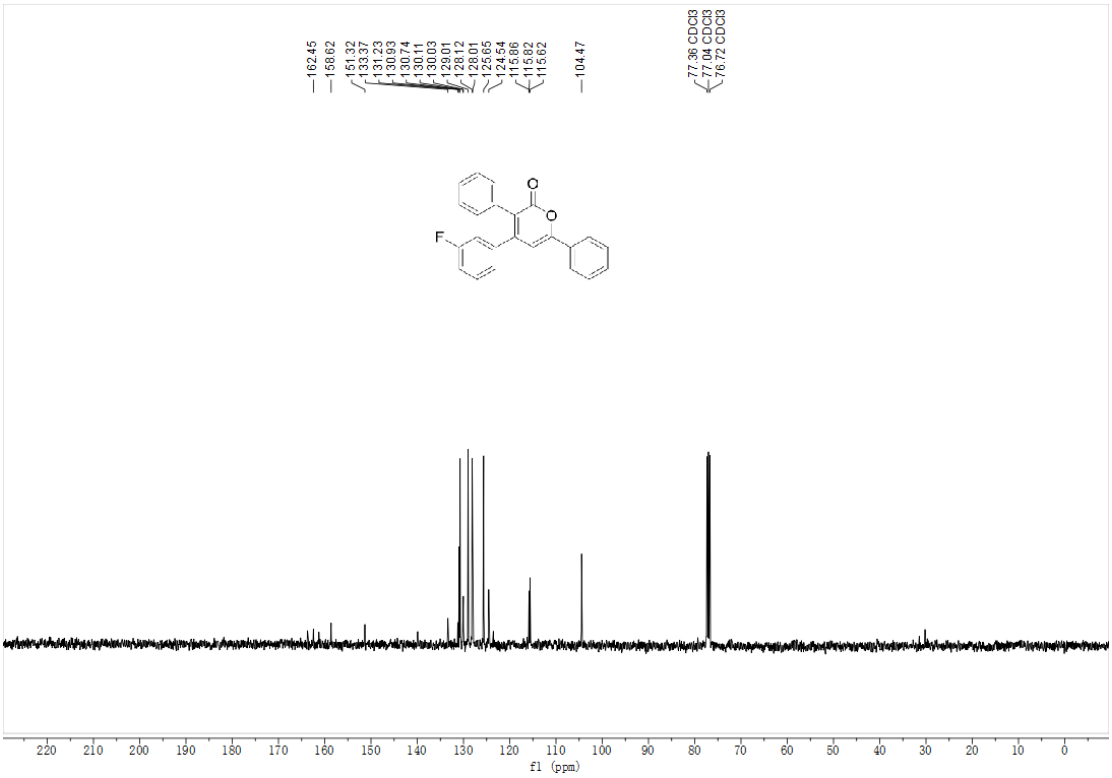
6.36
2.98
1.00
f1 (ppm)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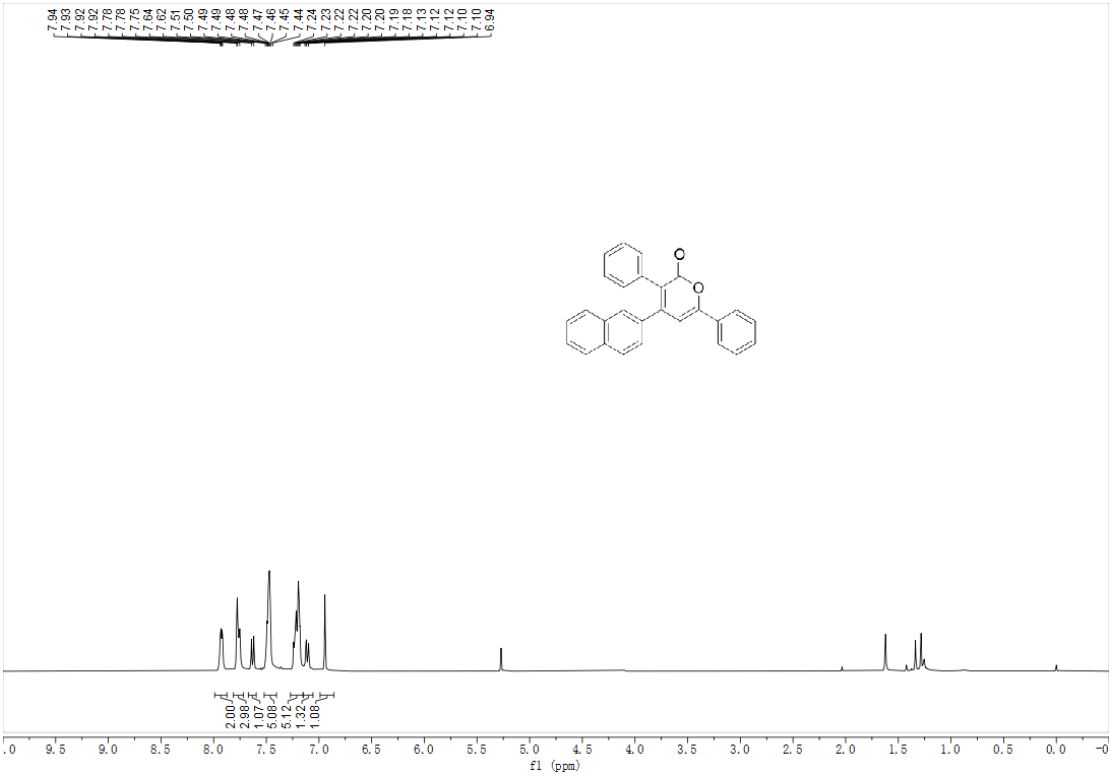
77.36 CDCl3
77.04 CDCl3
76.72 CDCl3
O
O
F
f1 (ppm)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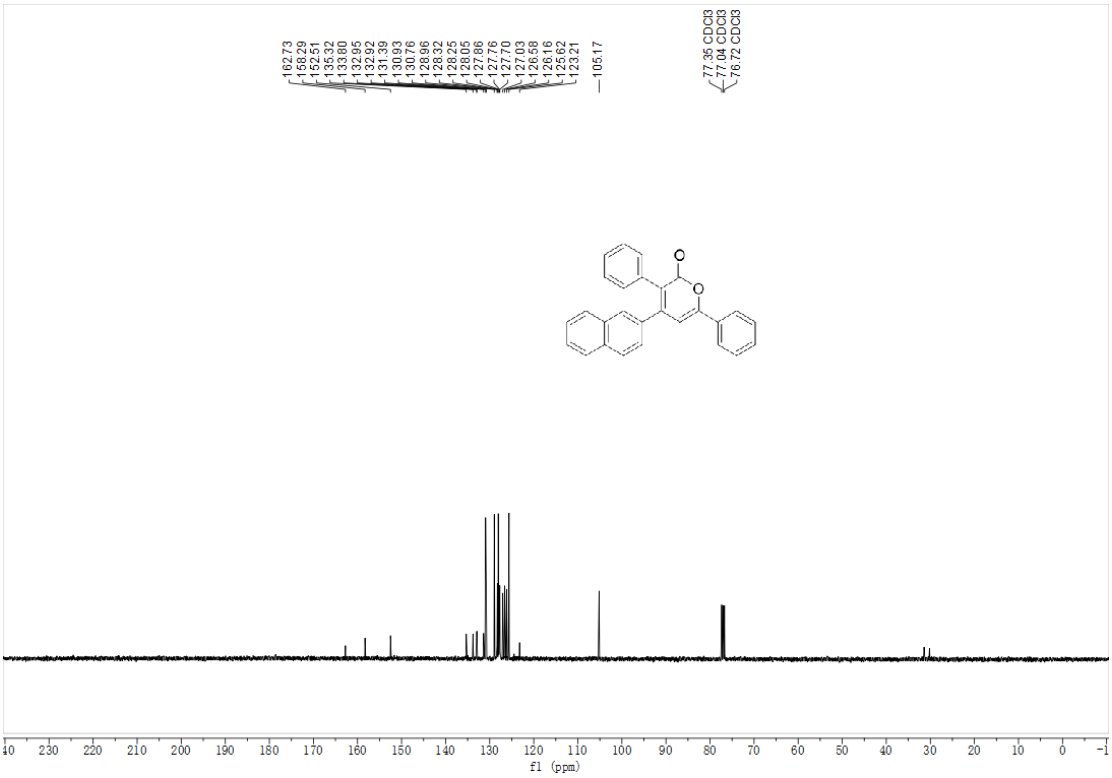
1.08
f1 (ppm)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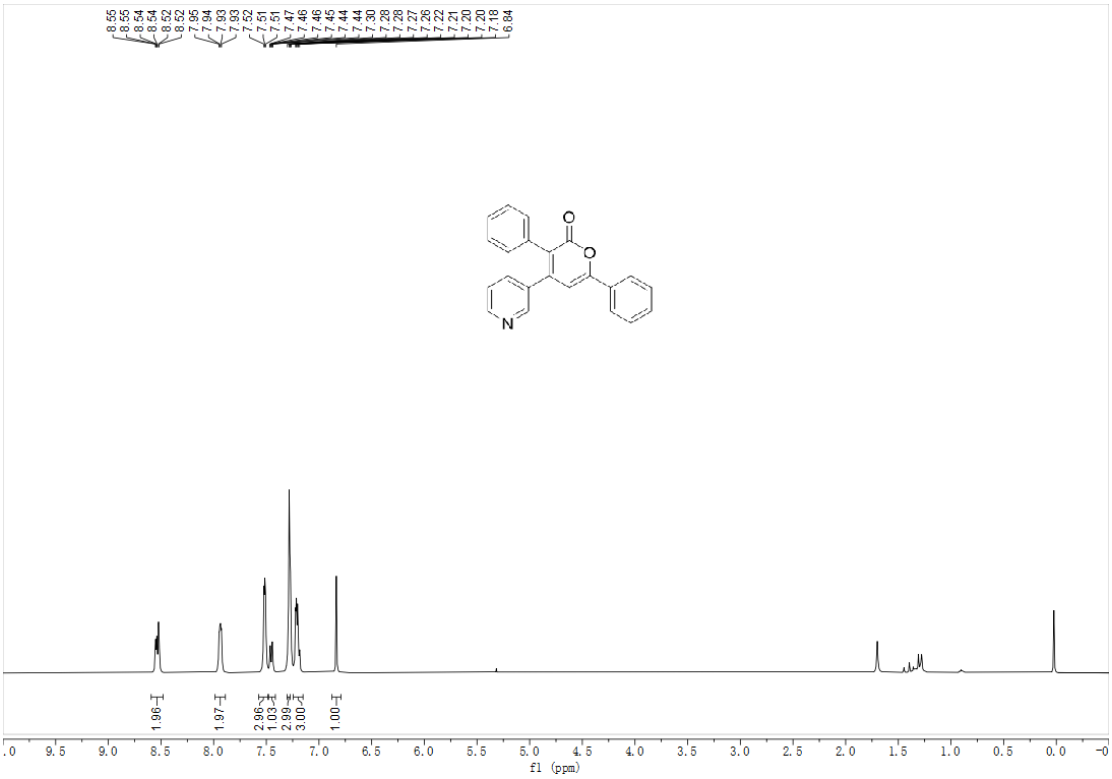
77.04 CDCl3
76.72 CDCl3
O
O
f1 (ppm)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7.20
7.20
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1.97
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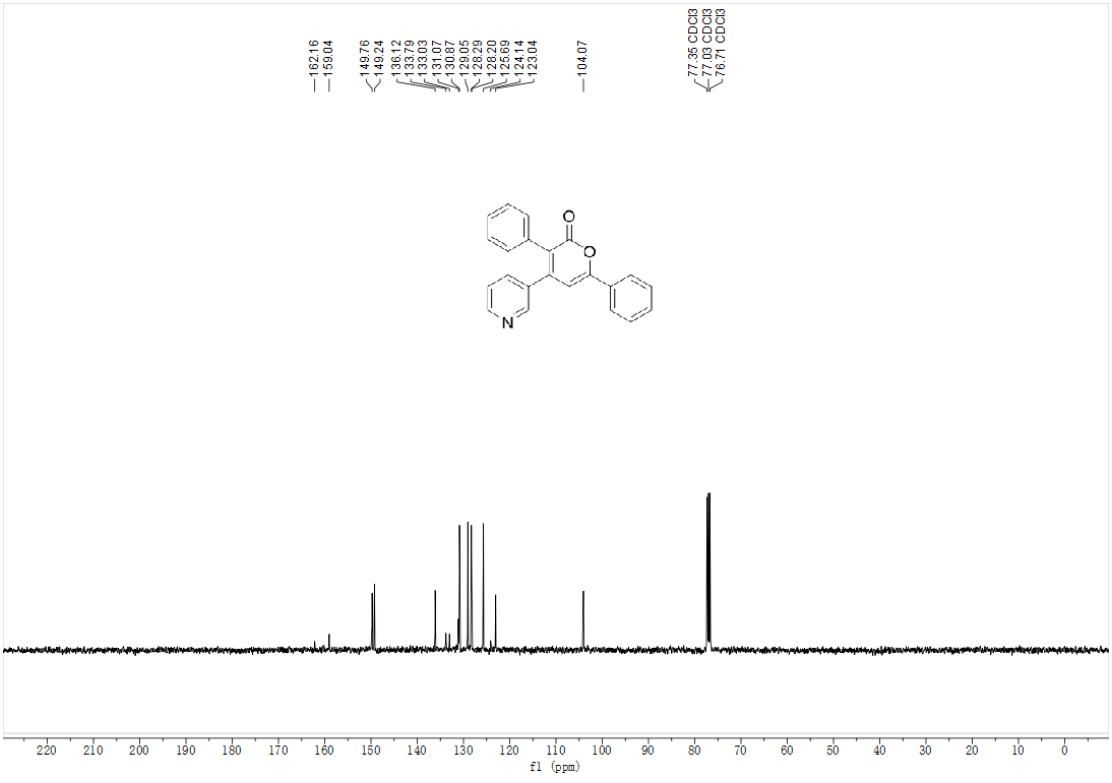
1.03
2.99
3.00
1.00
f1 (ppm)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128.20
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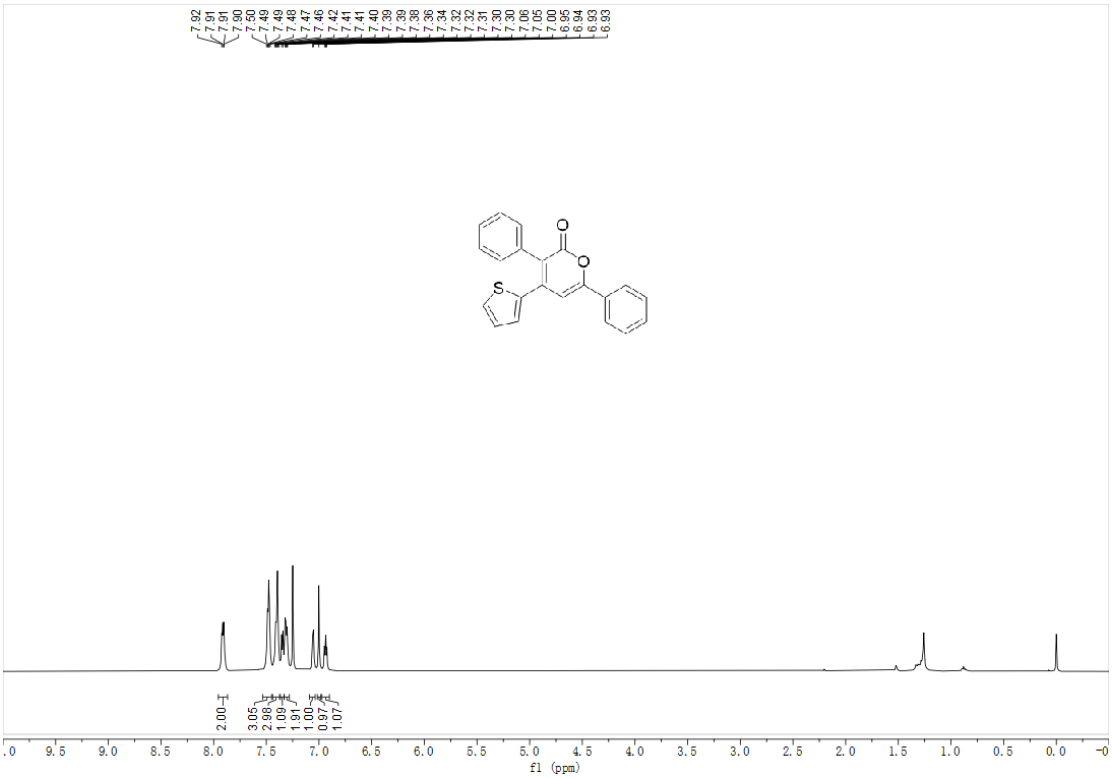
123.04
104.07
77.35 CDCl3
77.03 CDCl3
76.71 CDCl3
O
O
N
f1 (ppm)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7.30
7.30
7.06
7.05
7.00
6.95
6.94
6.93
6.93
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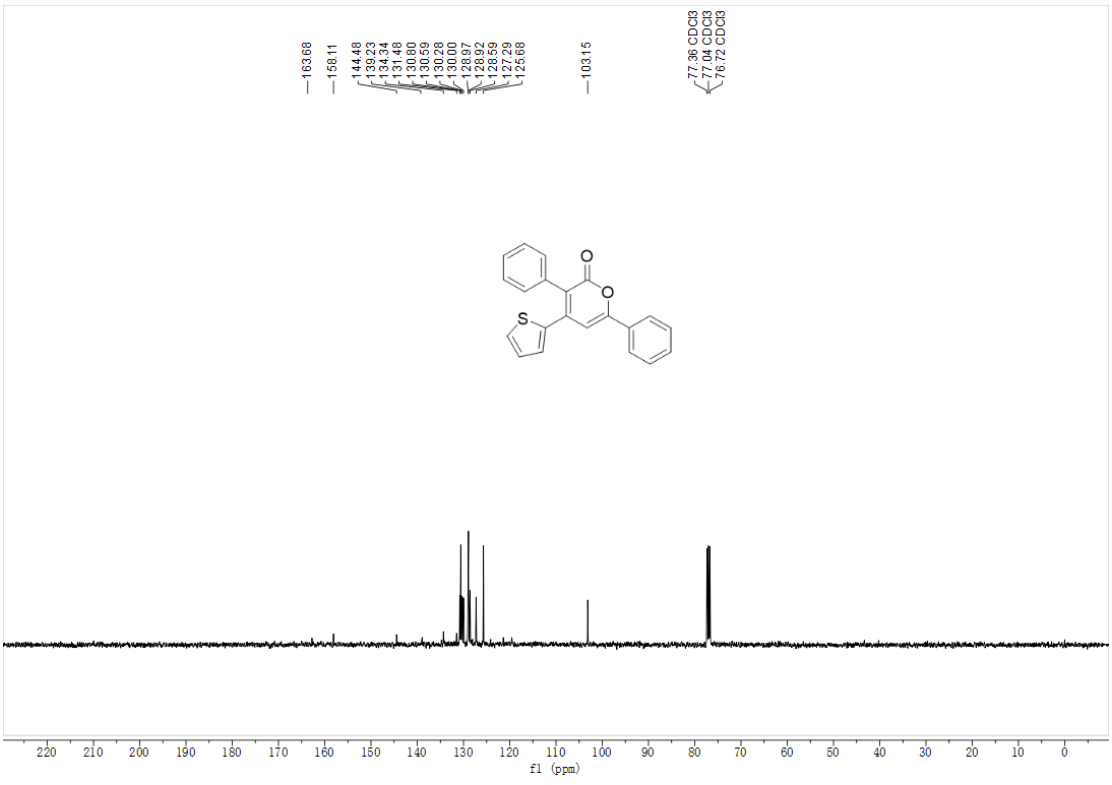
O
S
2.00
3.05
2.98
1.09
1.91
1.00
0.97
1.07
f1 (ppm)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128.92
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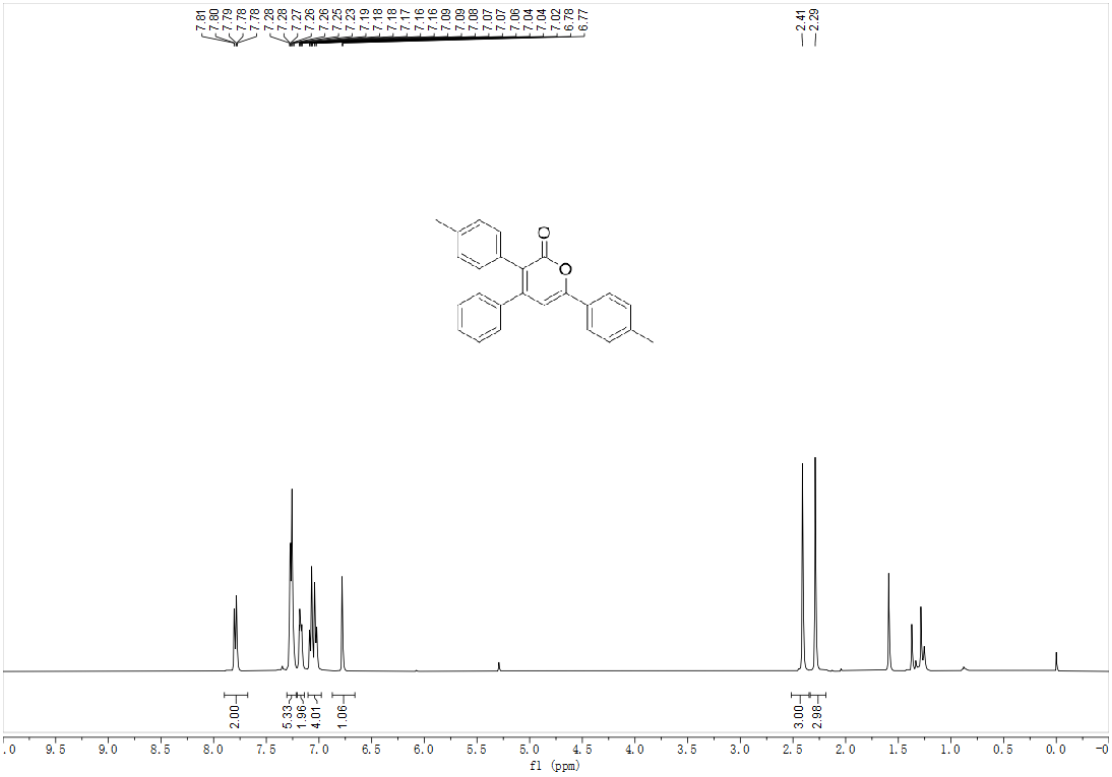
125.68
103.15
77.36 CDCl3
77.04 CDCl3
76.72 CDCl3
O
O
S
f1 (ppm)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7.09
7.08
7.07
7.07
7.06
7.04
7.04
7.02
6.78
6.77
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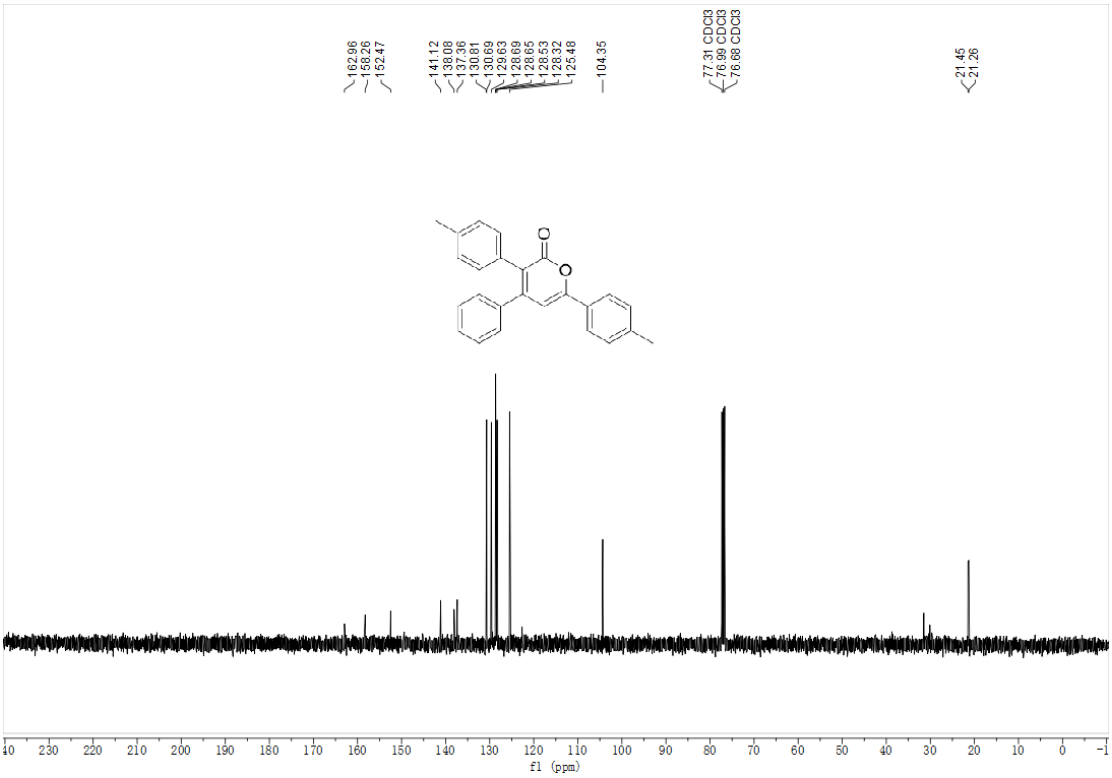
1.06
3.00
2.98
f1 (ppm)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76.99 CDCl3
76.68 CDCl3
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21.26
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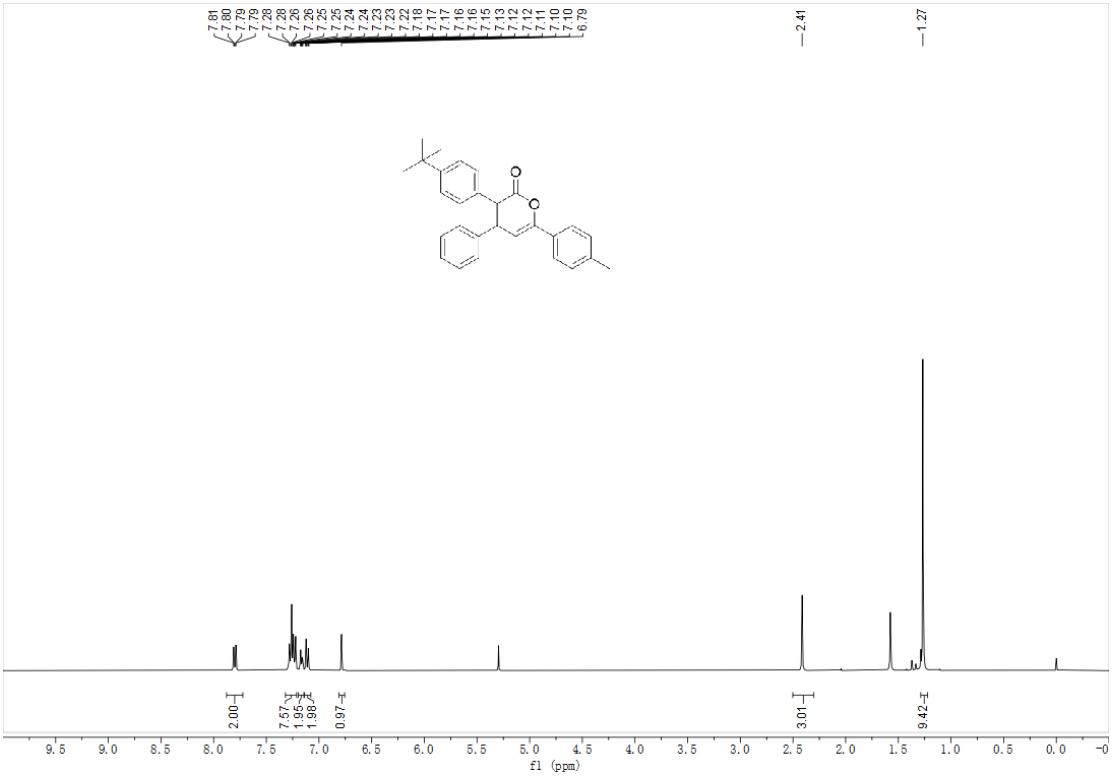
O
f1 (ppm)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7.18
7.17
7.17
7.16
7.15
7.13
7.12
7.12
7.11
7.10
7.10
6.79
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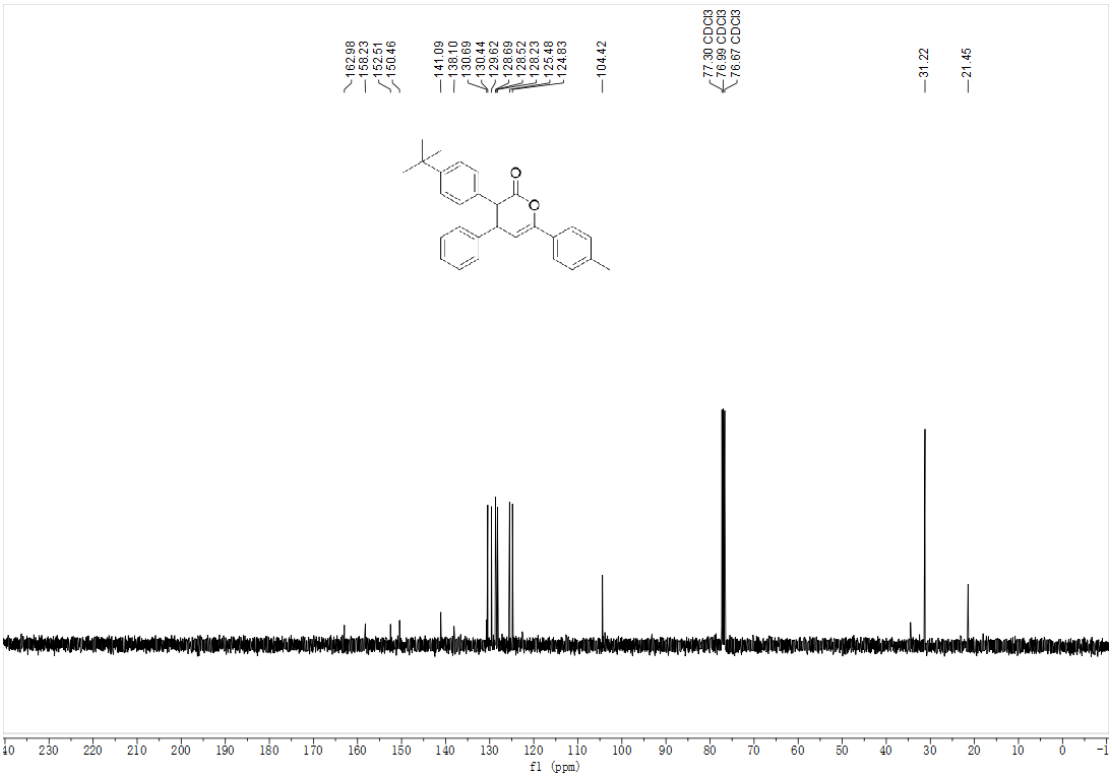
1.98
0.97
3.01
9.42
f1 (ppm)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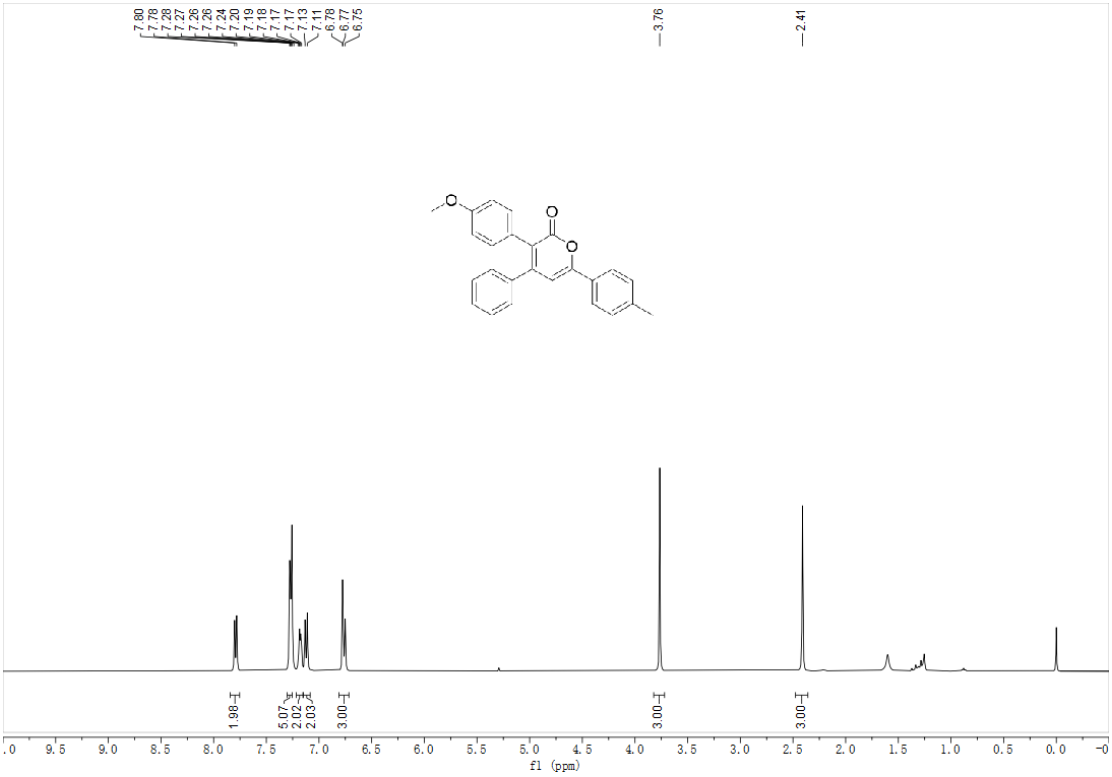
76.99 CDCl3
76.67 CDCl3
31.22
21.45
f1 (ppm)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7.17
7.17
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7.11
6.78
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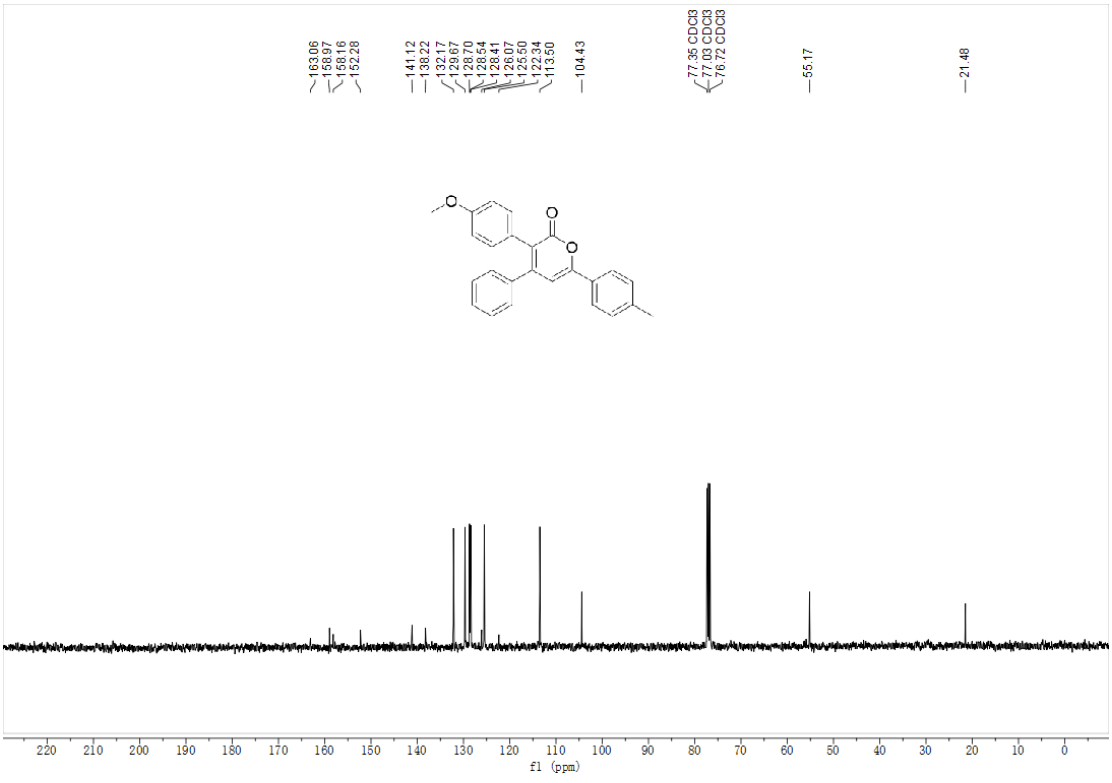
5.07
2.02
2.03
3.00
3.00
3.00
f1 (ppm)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129.67
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128.54
128.41
126.07
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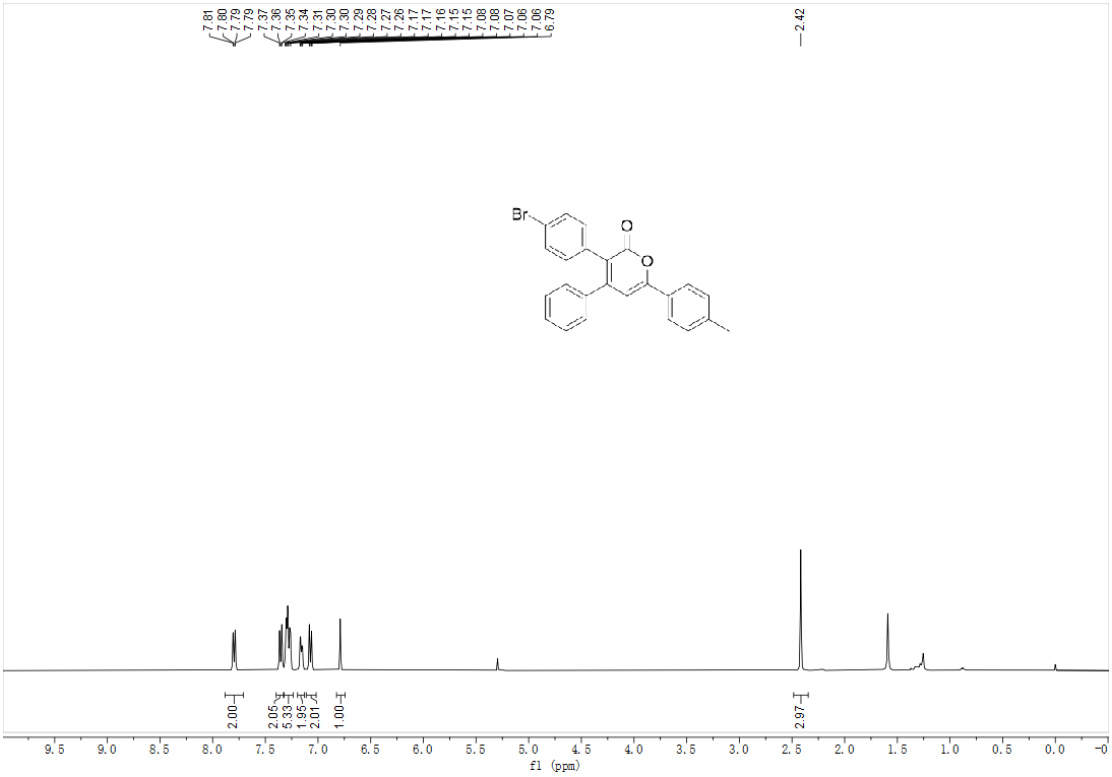
77.03 CDCl3
76.72 CDCl3
55.17
21.48
f1 (ppm)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7.29
7.28
7.27
7.26
7.17
7.17
7.16
7.15
7.15
7.08
7.08
7.07
7.06
7.06
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O
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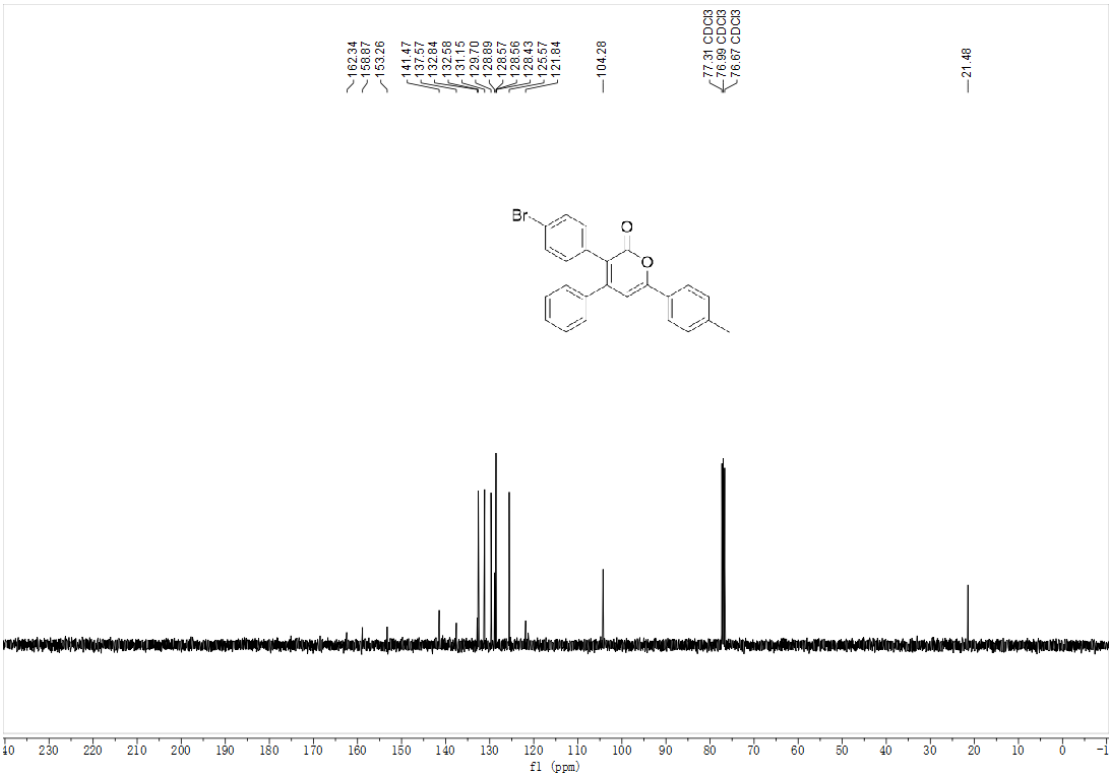
2.05
5.33
1.95
2.01
1.00
2.97
f1 (ppm)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128.56
128.43
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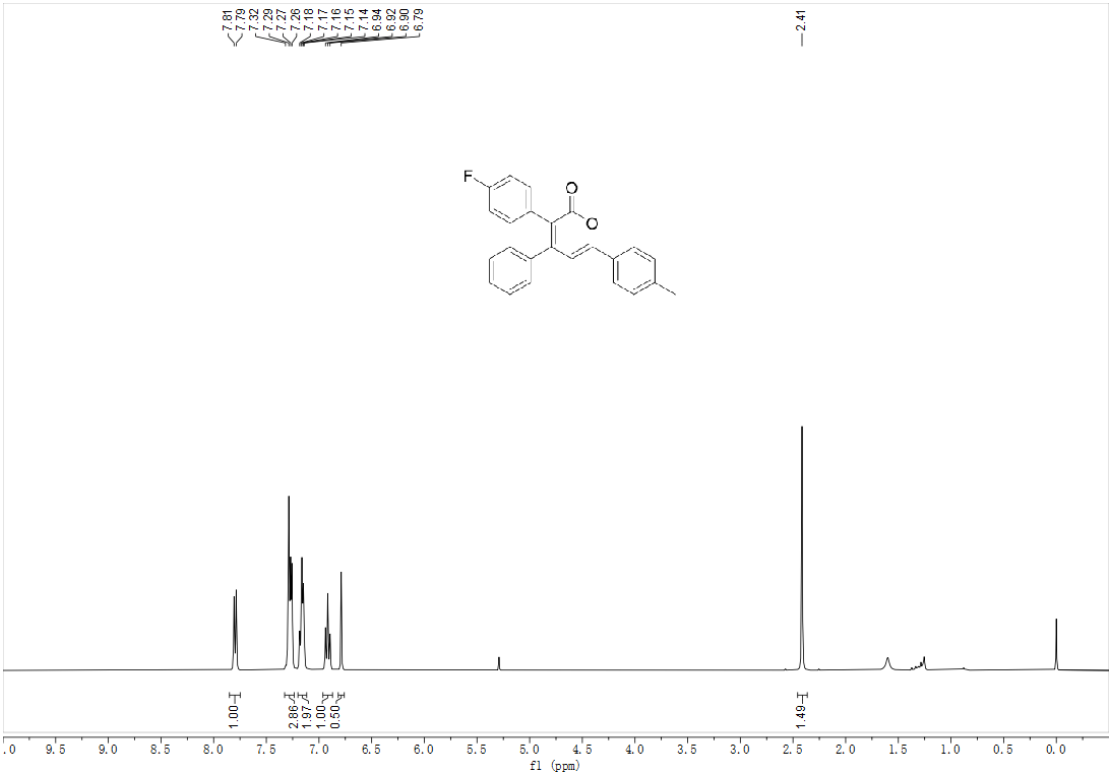
104.28
77.31 CDCl3
76.99 CDCl3
76.67 CDCl3
21.48
Br
O
O
f1 (ppm)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7.17
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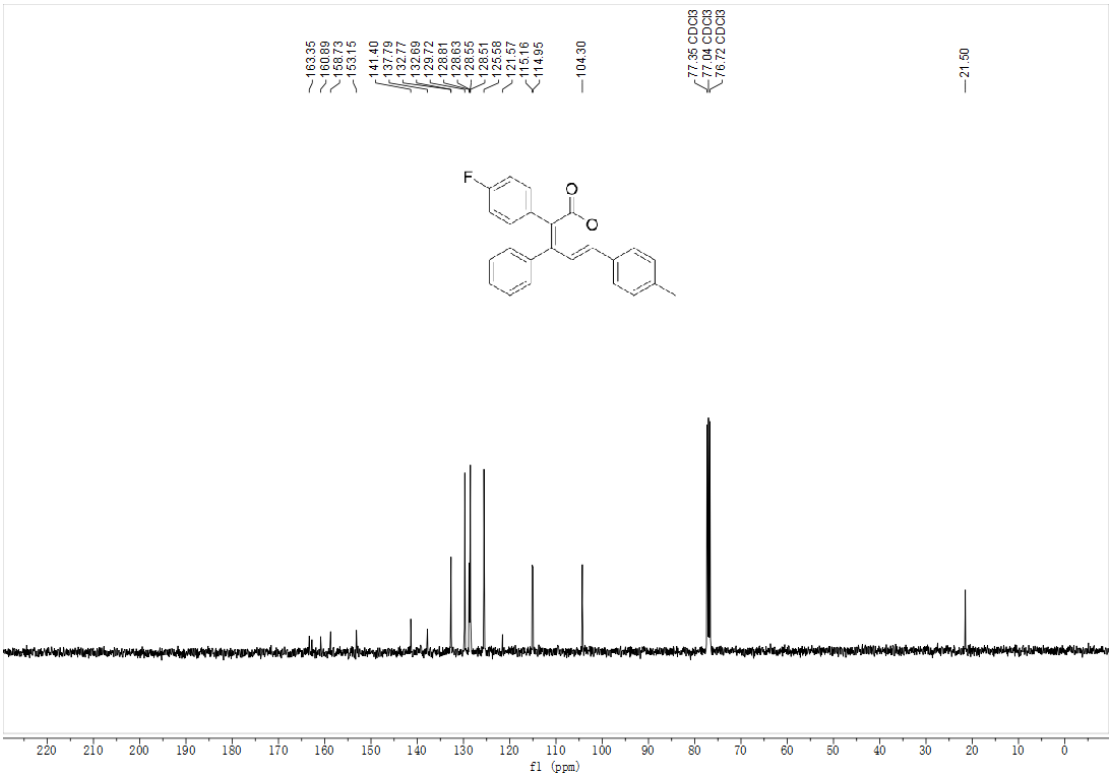
7.16
7.15
7.14
6.94
6.92
6.90
6.79
2.41
F
O
O
1.00
2.86
1.97
1.00
0.50
1.49
f1 (ppm)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128.81
128.63
128.55
128.51
125.58
121.57
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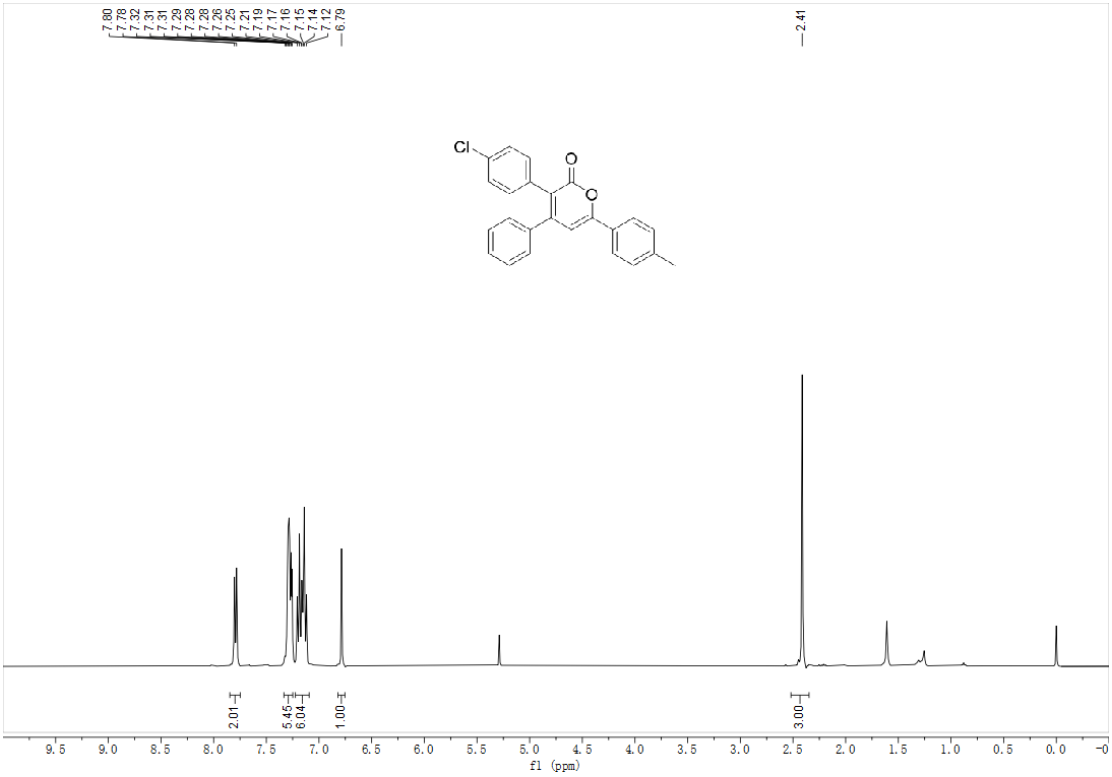
114.95
104.30
77.35 CDCl3
77.04 CDCl3
76.72 CDCl3
21.50
F
O
O
f1 (ppm)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7.21
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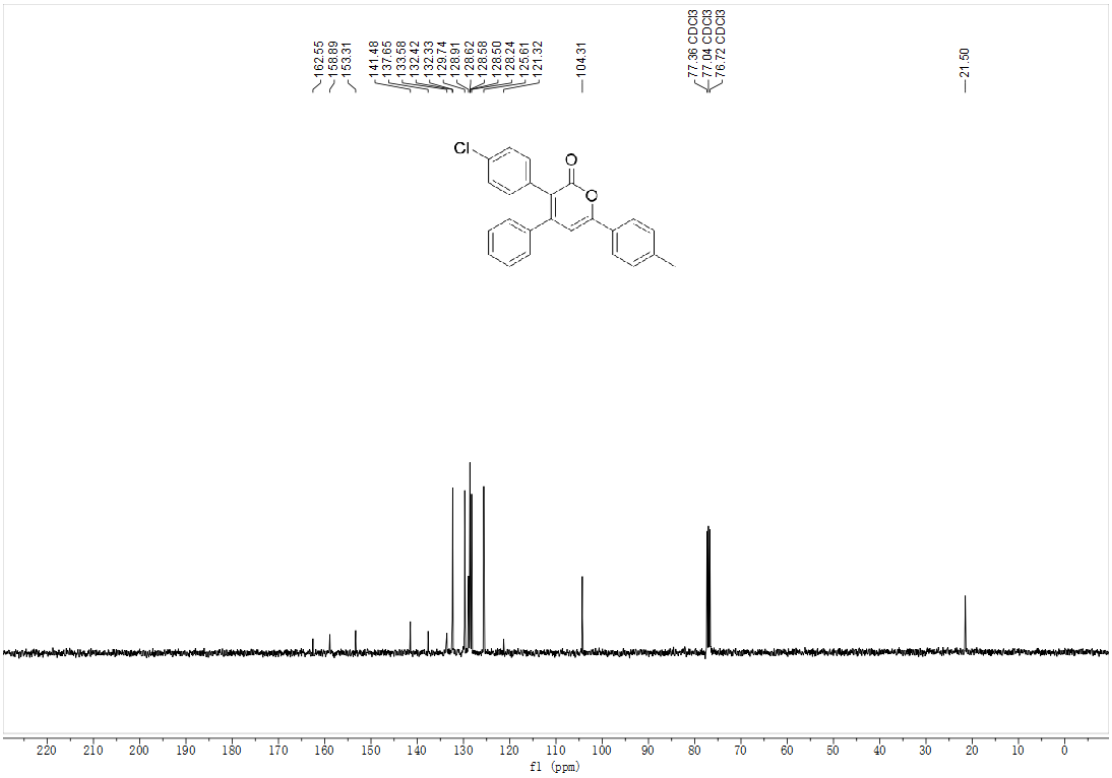
7.17
7.16
7.15
7.14
7.12
6.79
2.41
Cl
O
O
2.01
5.45
6.04
1.00
3.00
f1 (ppm)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128.24
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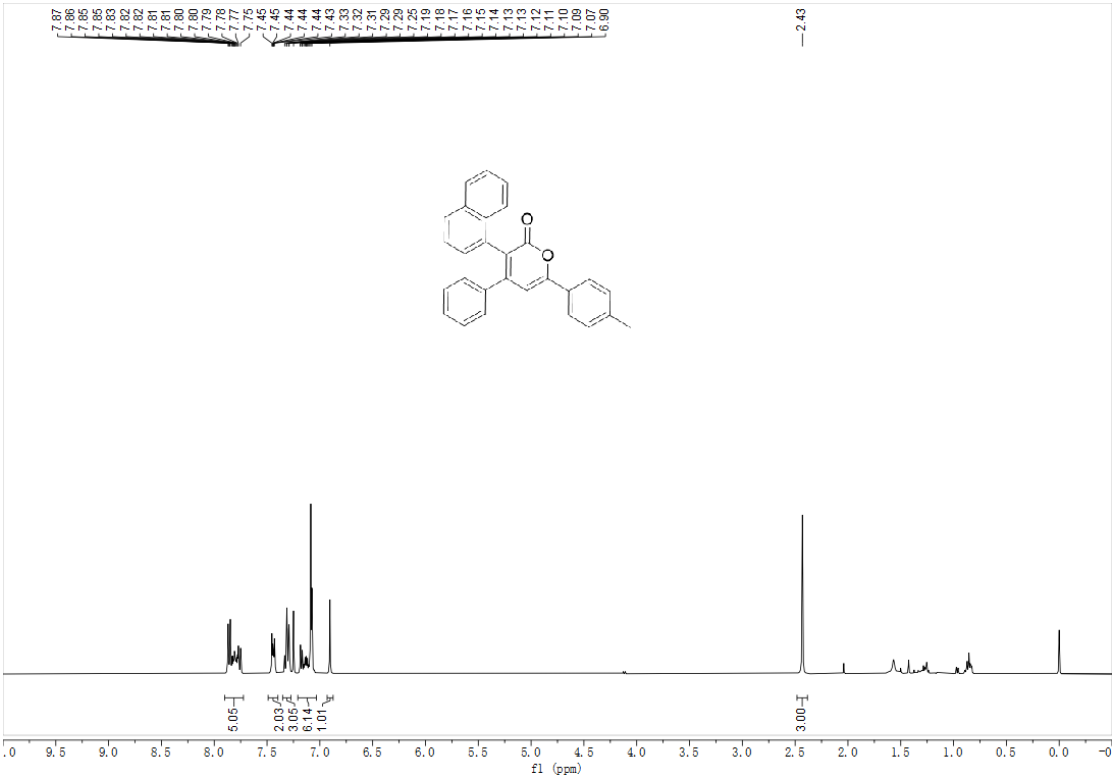
77.04 CDCl3
76.72 CDCl3
21.50
Cl
O
O
f1 (ppm)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7.77
7.75
7.45
7.44
7.44
7.44
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7.14
7.13
7.13
7.12
7.11
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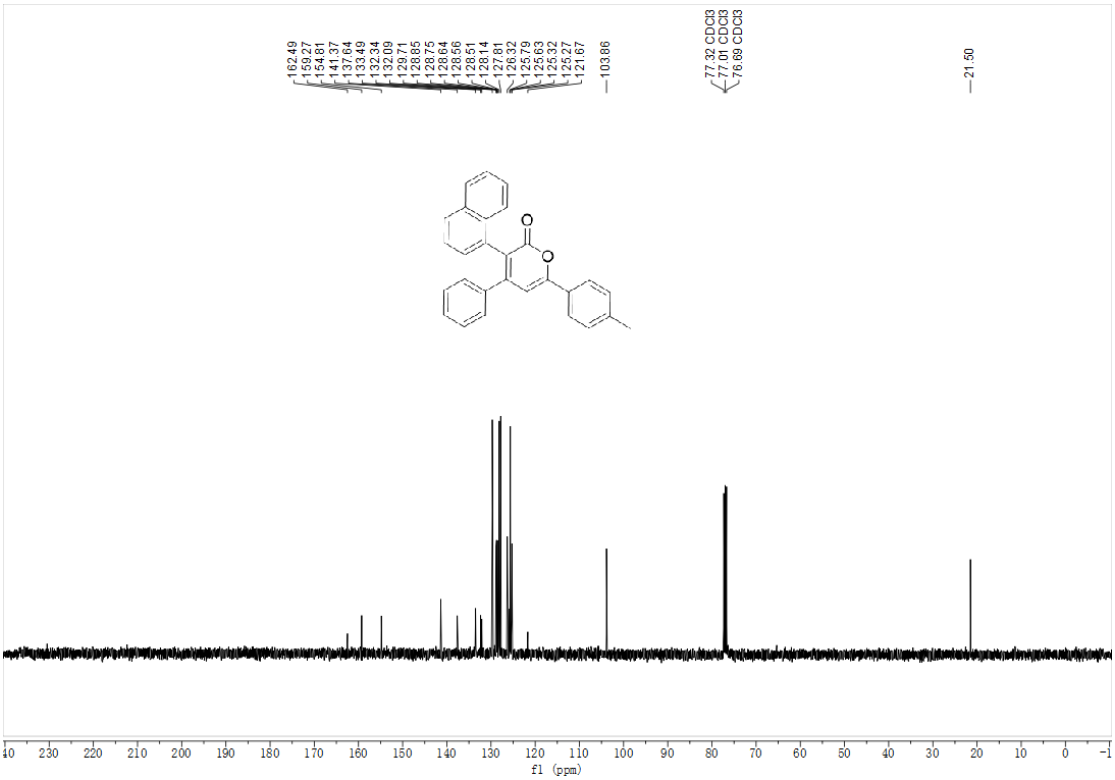
2.03
3.05
6.14
1.01
3.00
f1 (ppm)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128.56
128.51
128.14
127.81
126.32
125.79
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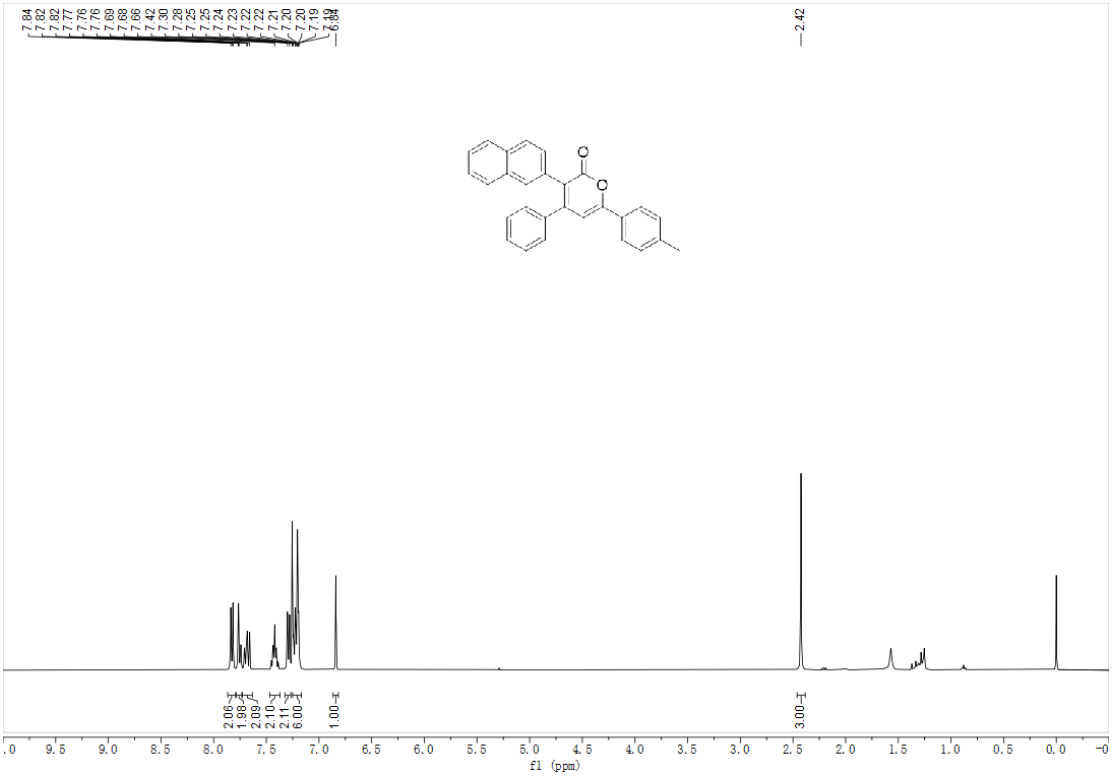
125.32
125.27
121.67
103.86
77.32 CDCl3
77.01 CDCl3
76.69 CDCl3
21.50
f1 (ppm)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7.23
7.22
7.21
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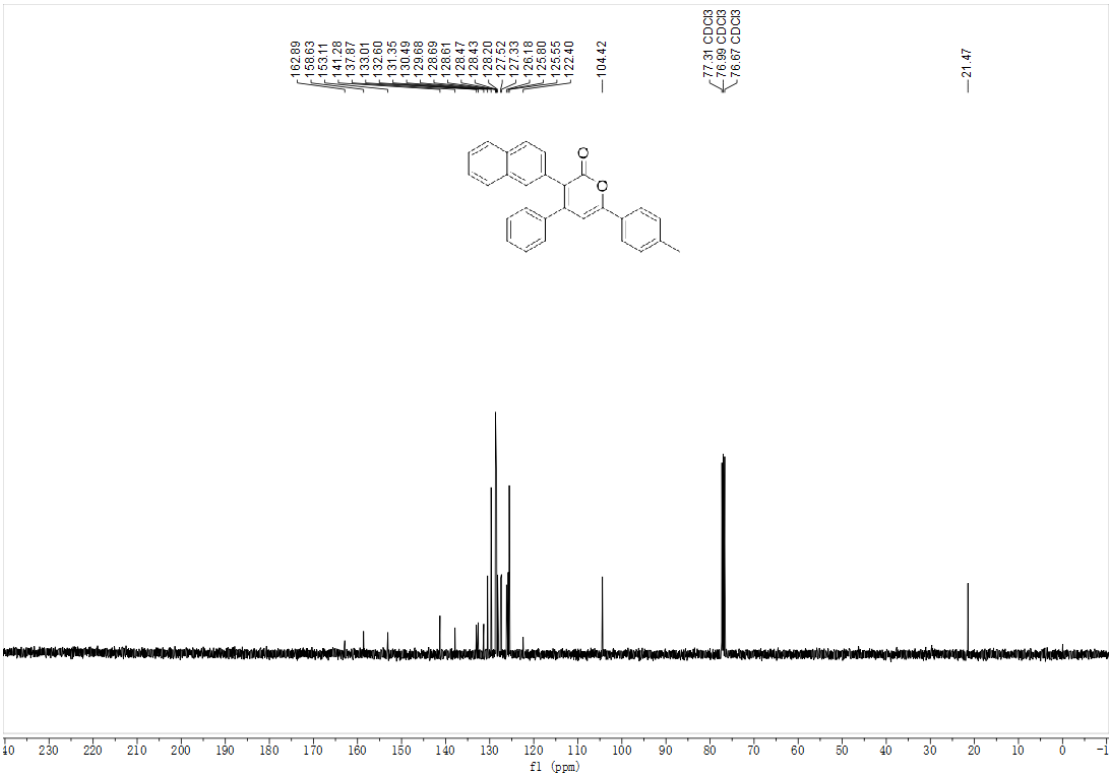
2.06
1.98
2.09
2.10
2.11
6.00
1.00
3.00
f1 (ppm)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127.52
127.33
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77.31 CDCl3
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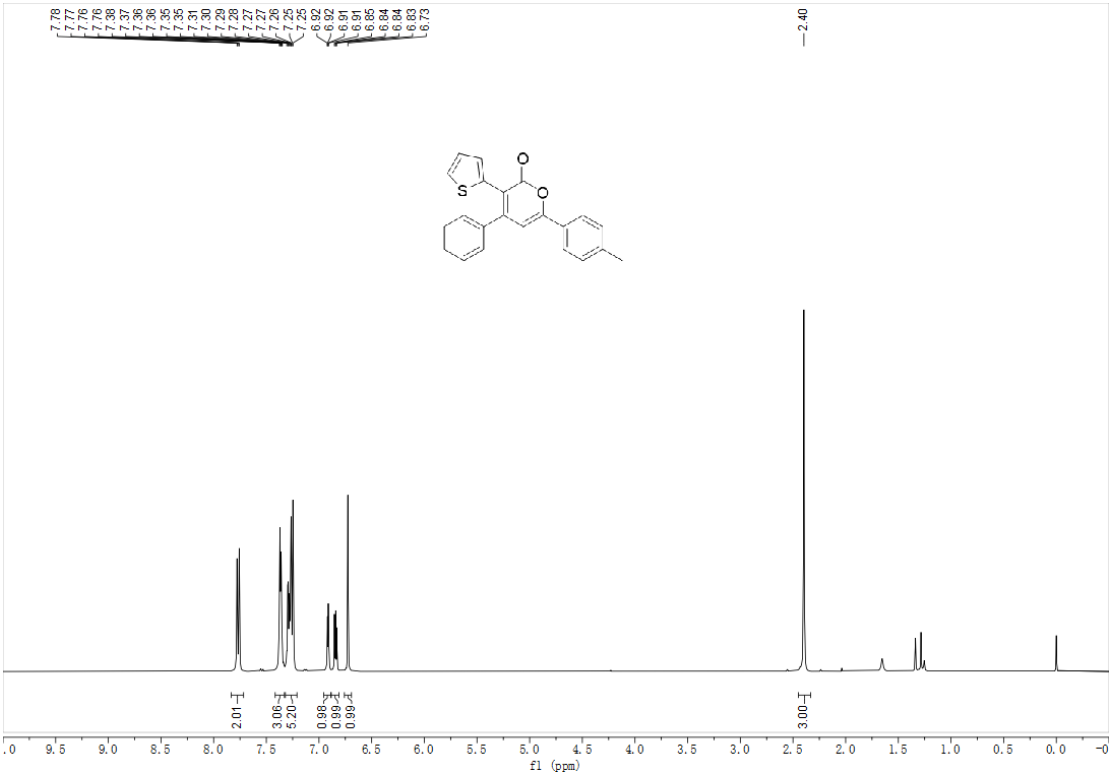
76.67 CDCl3
21.47
f1 (ppm)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6.92
6.91
6.91
6.85
6.84
6.84
6.83
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O
2.01
3.06
5.20
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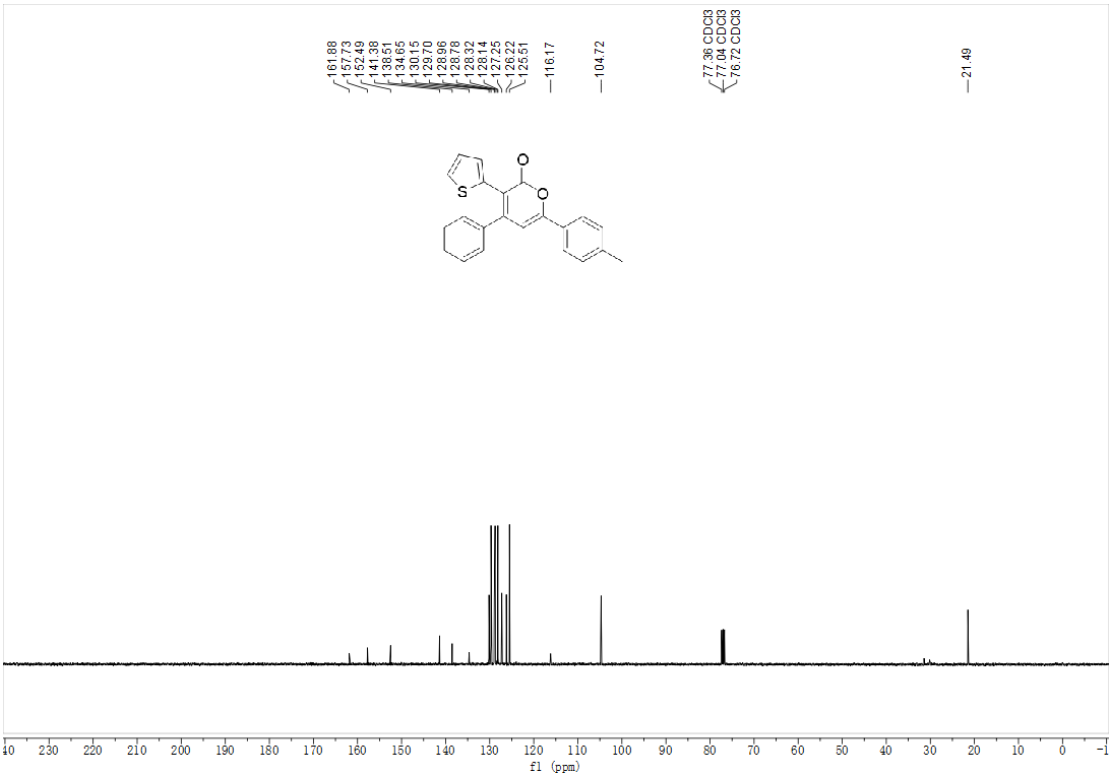
0.99
0.99
3.00
f1 (ppm)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126.22
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21.49
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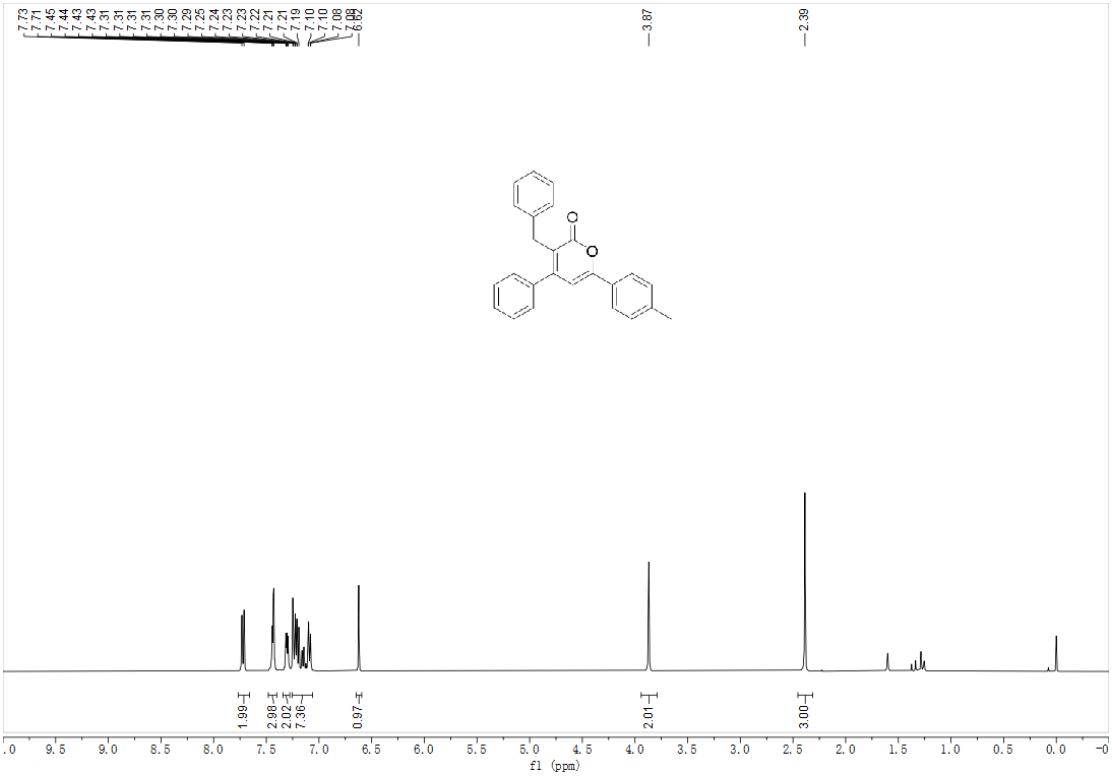
O
O
f1 (ppm)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7.22
7.21
7.21
7.19
7.10
7.10
7.08
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2.98
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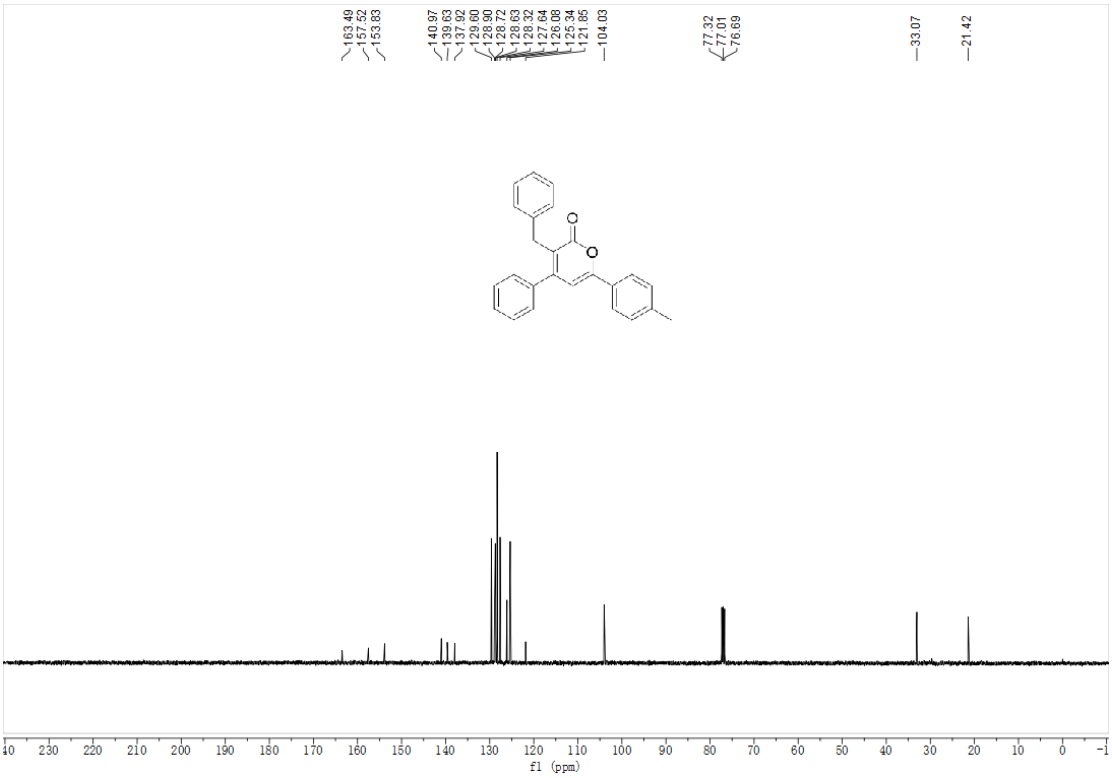
0.97
2.01
3.00
f1 (ppm)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121.85
104.03
77.32
77.01
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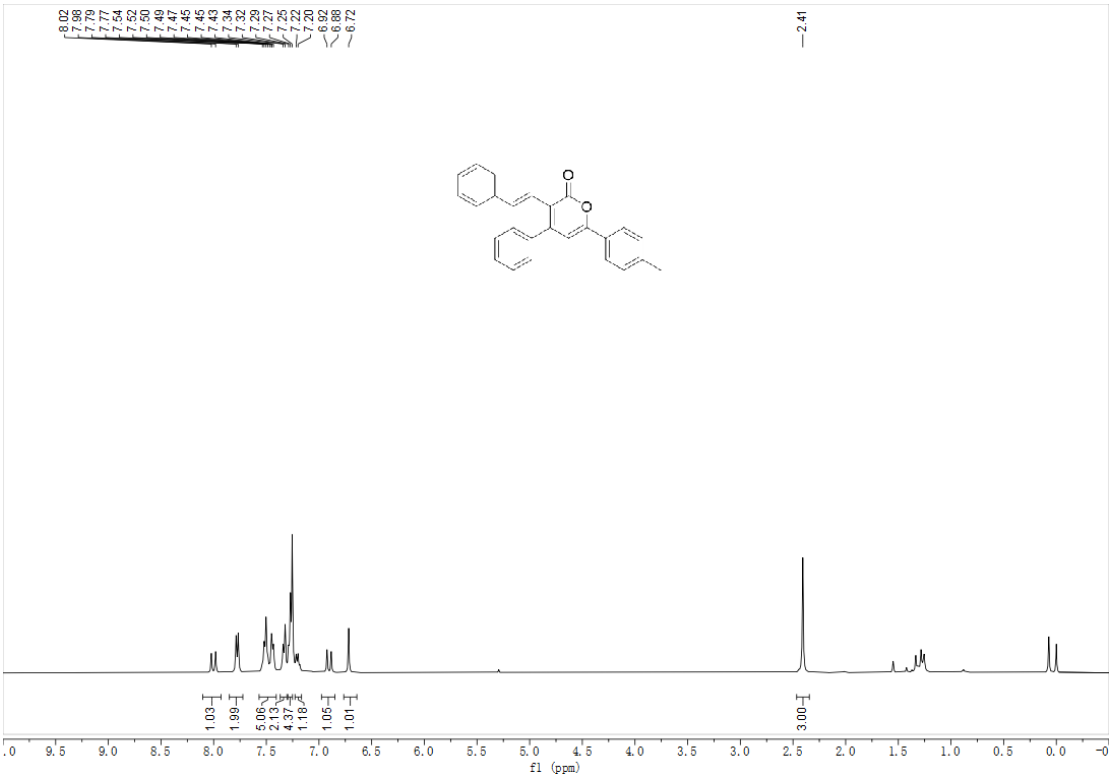
33.07
21.42
O
O
f1 (ppm)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7.29
7.27
7.25
7.22
7.20
6.92
6.88
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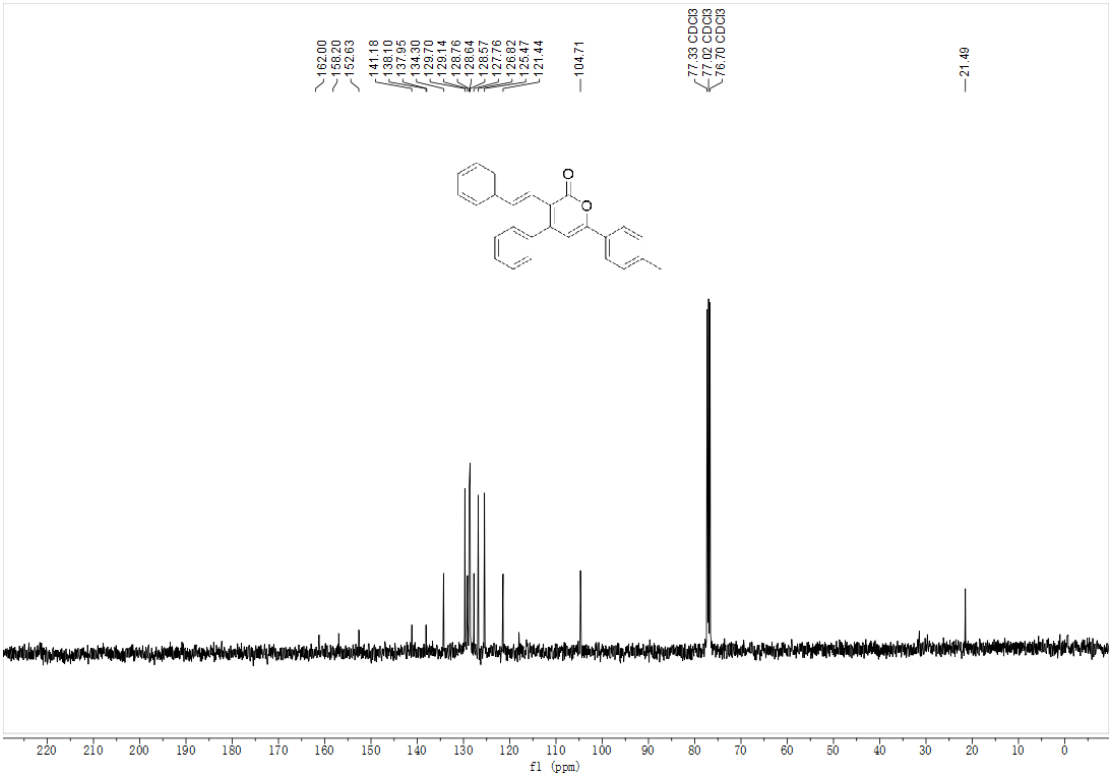
1.05
1.01
3.00
f1 (ppm)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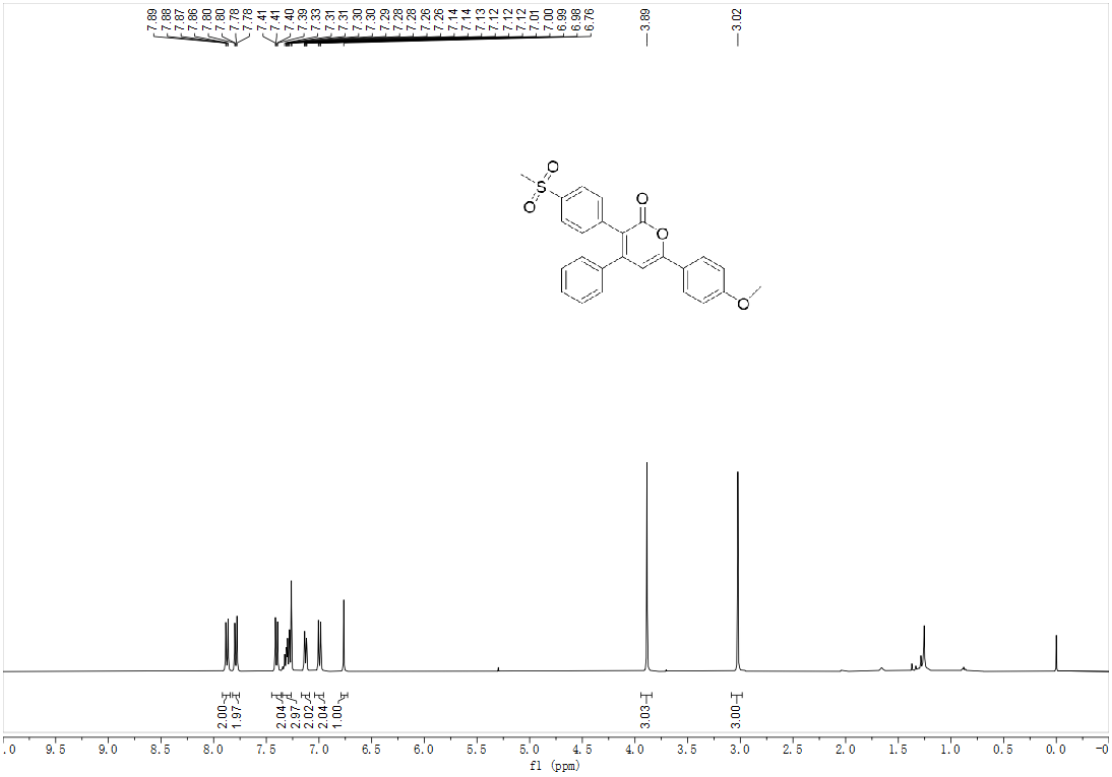
121.44
104.71
77.33 CDCl3
77.02 CDCl3
76.70 CDCl3
21.49
f1 (ppm)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6.98
6.76
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2.07
2.02
2.04
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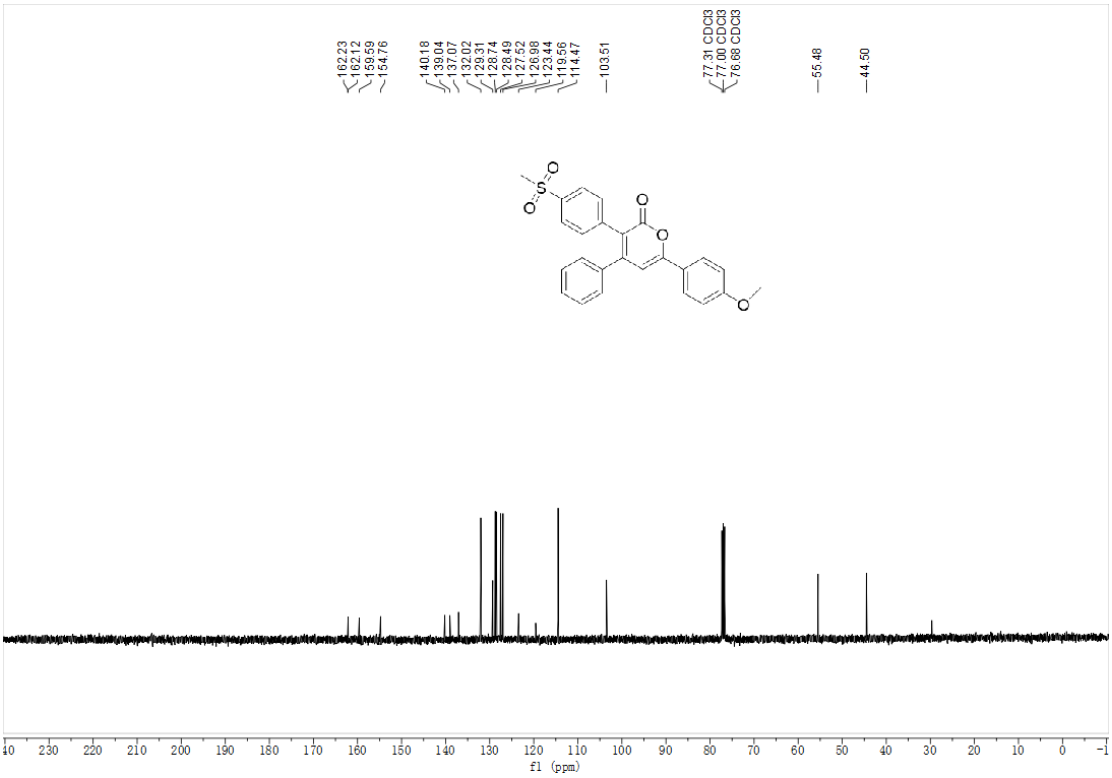
3.03
3.00
f1 (ppm)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123.44
119.56
114.47
103.51
77.31 CDCl3
77.00 CDCl3
76.68 CDCl3
55.48
44.50
f1 (ppm)