Supporting Information

NHC-Catalyzed [4+2] Annulations of Allenoates and 2,3-Dioxopyrrolidine Derivatives

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1 General methods

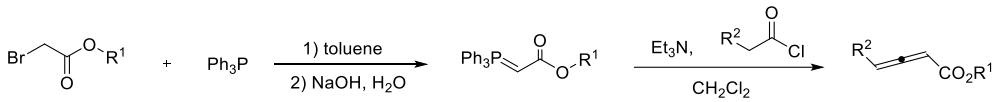
Common reagents and materials were purchased from commercial sources and purified by recrystallization or distillation. ^{1}H NMR spectra were measured on a Bruker DPX 400 MHz spectrometer in $CDCl_3$ (100 MHz**,** ^{13}C NMR) with chemical shift (δ) given in ppm relative to TMS as internal standard. Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). Coupling constants were reported in Hertz (Hz). IR spectra were taken on a FT-IR-Tensor 27 spectrometer in KBr pellets and reported in cm^{-1}. High-resolution mass spectra (HRMS) were obtained on a micrOTOF-QⅡ HRMS/MS instrument (Bruker). HPLC analysis was performed on high performance liquid chromatography, UV detection monitored at 254 nm, using an IC column with hexane and ^{i}PrOH as the eluent.

1.1 Syntheses of NHC precursors

These precatalysts were prepared according to the known reports[1].

1.2 Syntheses of allenoates

These allenoates were prepared according to the known reports[2].

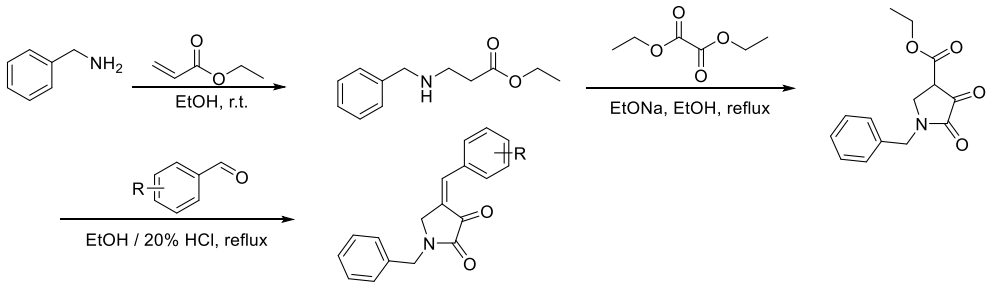


A 250 mL flask was charged with PPh_3 (13.104 g, 50 mmol) and 100 mL of toluene. To this solution was added bromoacetic esters (50 mmol) over 15 minutes. The solution was stirred for 12 hours and the precipitate was filtered, washed with cold toluene (3 × 5 mL) and dried. The collected phosphonium salt was dissolved in H_2O (150 mL). The mixture was cooled in an ice bath and saturated aqueous NaOH was added dropwise. The solid was filtered and washed with cold H_2O, collected and dried in vacuo to afford stabilized ylides.

A flask was charged with stabilized ylide, CH_2Cl_2 and Et_3N. After 10 minutes the appropriate acid chloride was added as a solution (5 mL) in CH_2Cl_2 slowly such that the temperature of the reaction remained constant. After 30 minutes, the color of the reaction mixture changes to clear yellow. After the completion of the reaction (TLC), the solution was concentrated to afford a gummy residue. This was treated with hexane (25 mL), stirred well, and allowed to sit undisturbed for 2 hours. The mixture was filtered and the filtrate evaporated and passed through a short pad of silica gel (petroleum ether/EtOAc = 20/1) to afford the pure allenic esters in 80–90% yield.

1.3 Syntheses of 2,3-dioxopyrrolidine derivatives

These 2,3-dioxopyrrolidines were prepared according to the known reports[3].

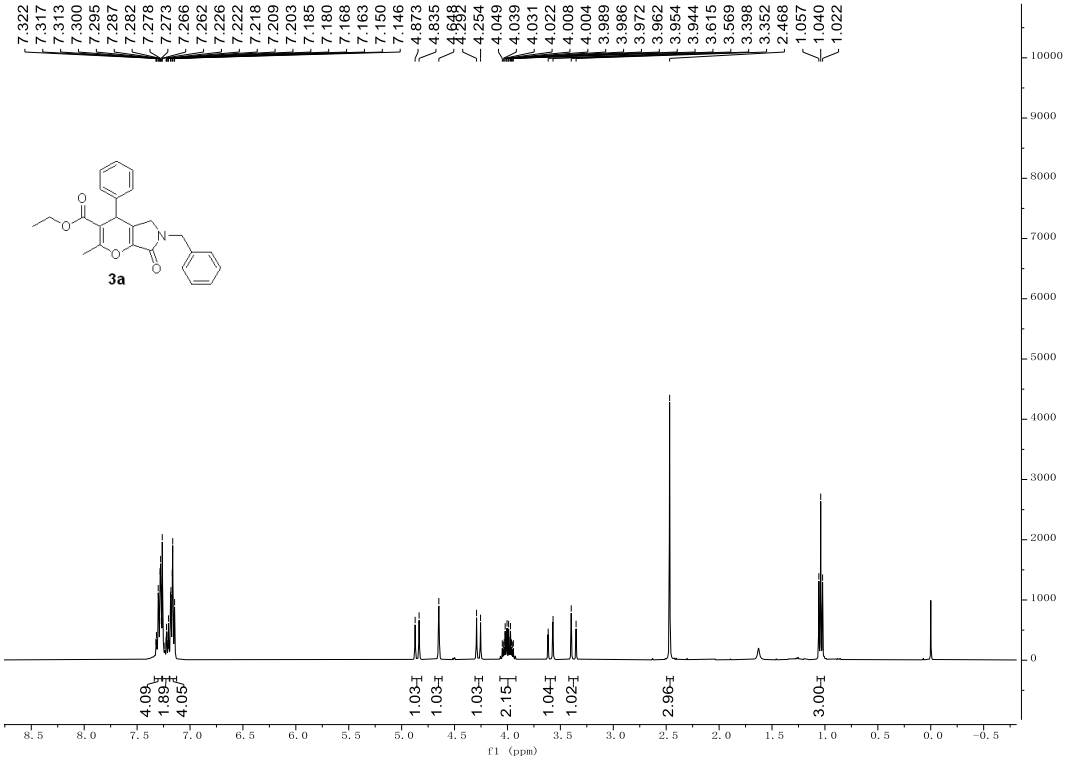


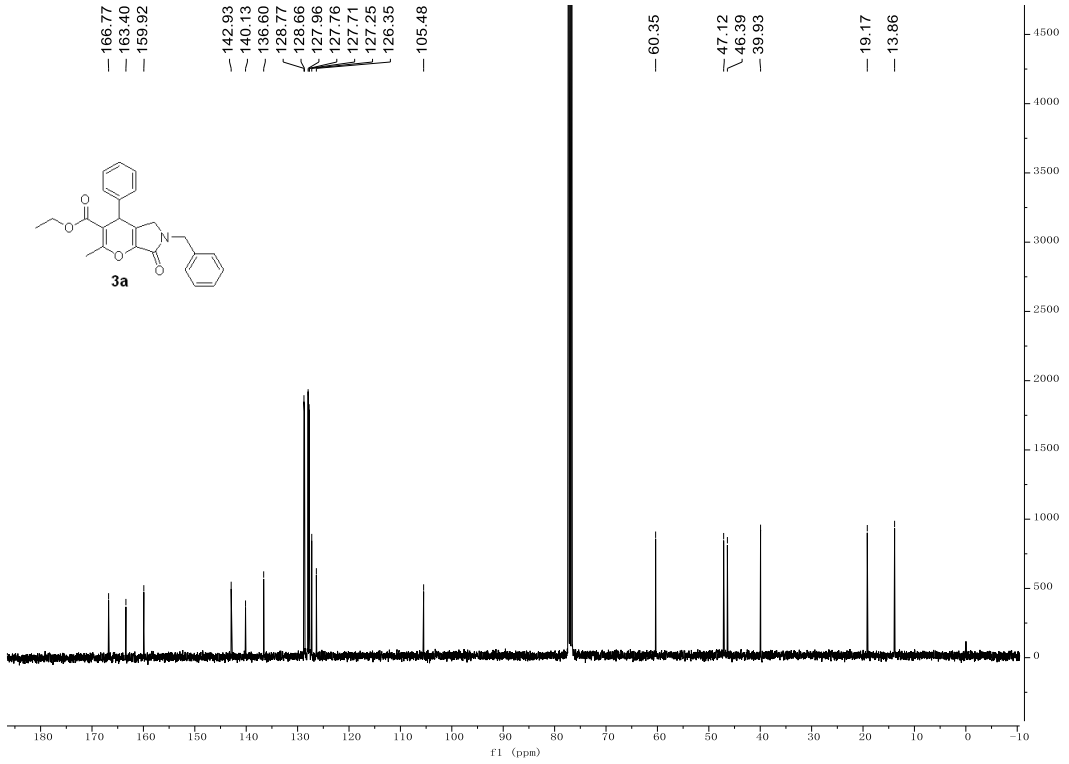
A solution of benzylamine (66.0 mmol), ethyl acrylate (66.0 mmol) in EtOH (20 mL) was allowed to stand at room temperature for 16 h. Diethyl oxalate (66.0 mmol) and freshly-made sodium ethoxide solution in EtOH (generated from 2.0 g of sodium metal, 80.0 mmol, in 30

mL EtOH) was added, and the mixture was heated at reflux for 1 h. The solvent was removed in vacuo. The crude product was diluted with H_2O (80 mL) and following acidification of the mixture and several hours standing to complete the precipitation. The resultant ethyl 1-benzyl-4,5-dioxopyrrolidine-3-carboxylate was collected by filtration and washed with diethyl ether as a white solid.

A single necked 250 mL round-bottom flask equipped with a magnetic stirring bar was charged with ethyl 1-benzyl-4,5-dioxopyrrolidine-3-carboxylate (10 mmol) and aldehyde (11 mmol) in 20 mL EtOH and 20% HCl 50 mL. The mixture was heated at reflux for 5 h and evaporate in vacuo to remove EtOH. The residue was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were washed with brine solution, dried over Na_2SO_4, filtered and concentrated in vacuo. The product was purified by column chromatography (PE/EA/CH_2Cl_2 = 10/1/1 to 2/1/1).

2 ^{1}H NMR and ^{13}C NMR Spectra

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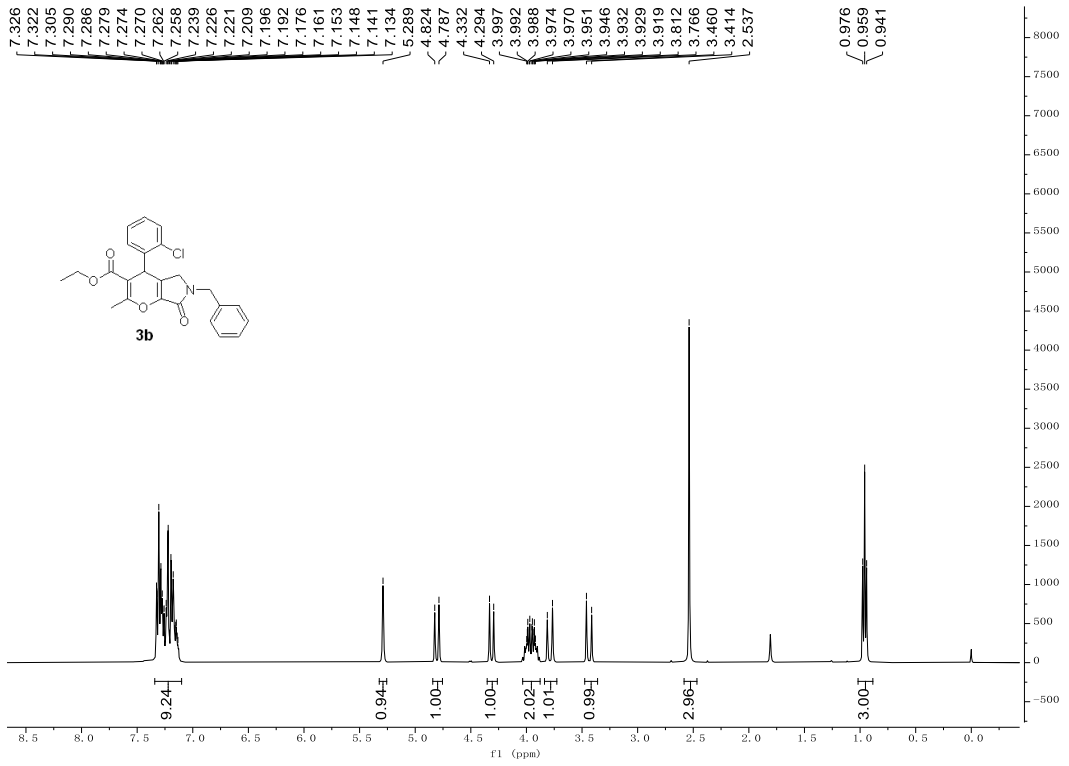
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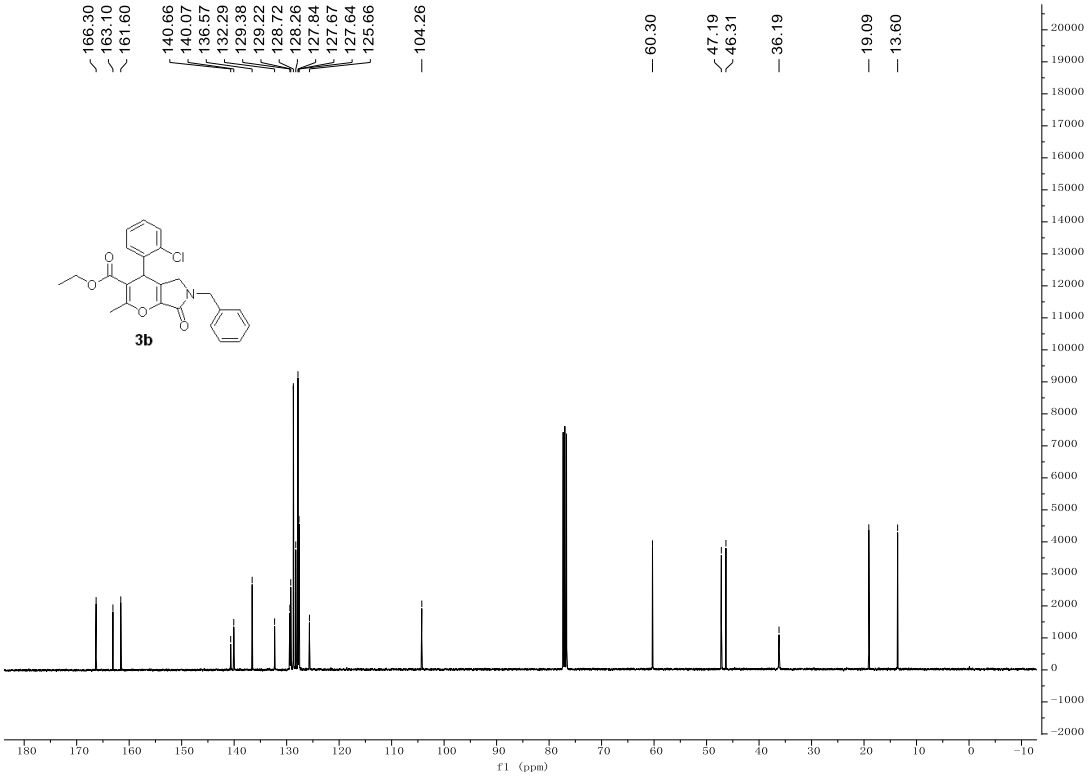
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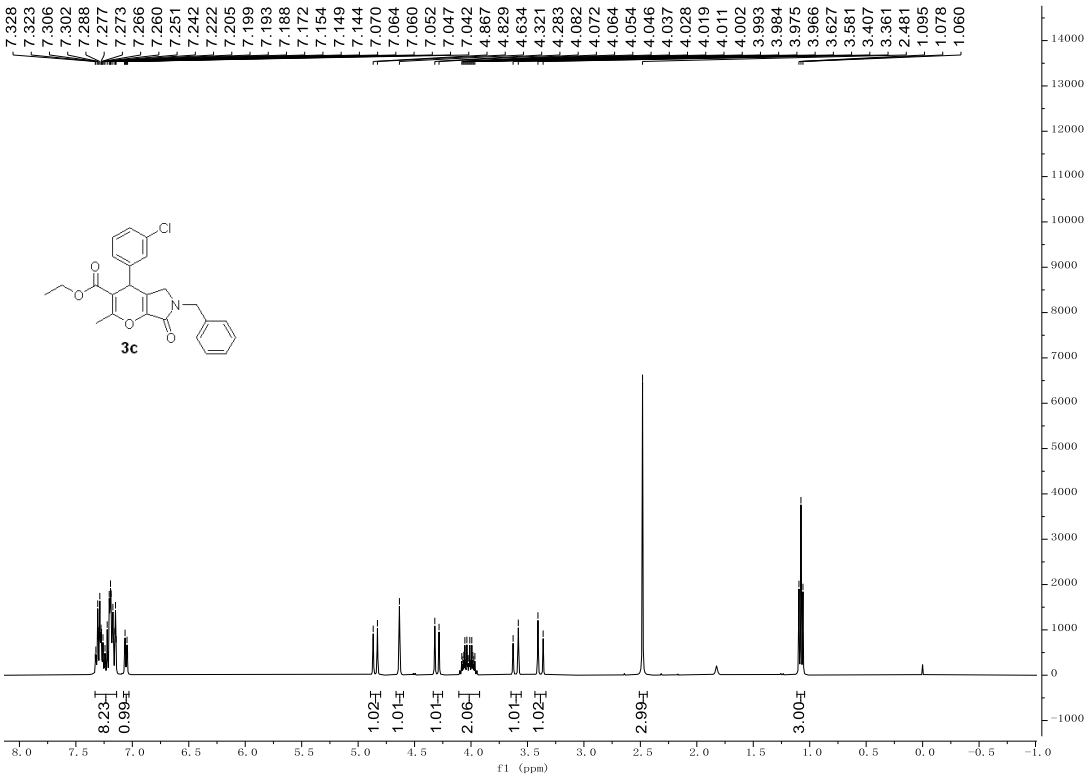
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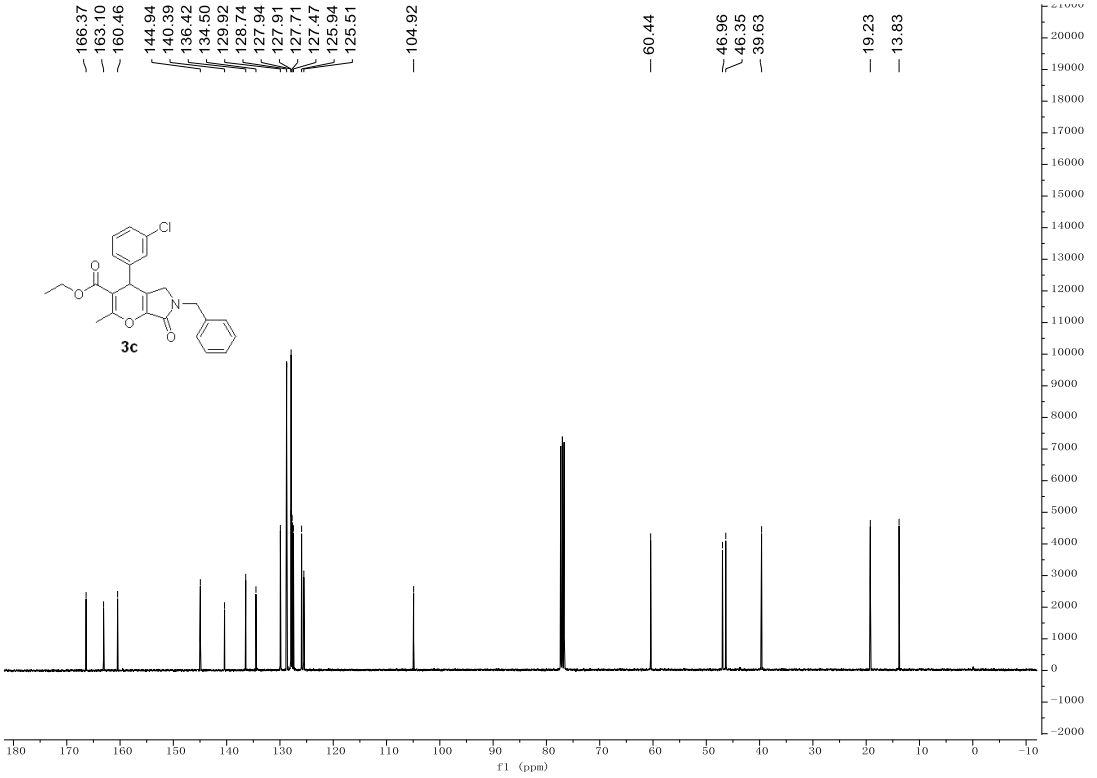
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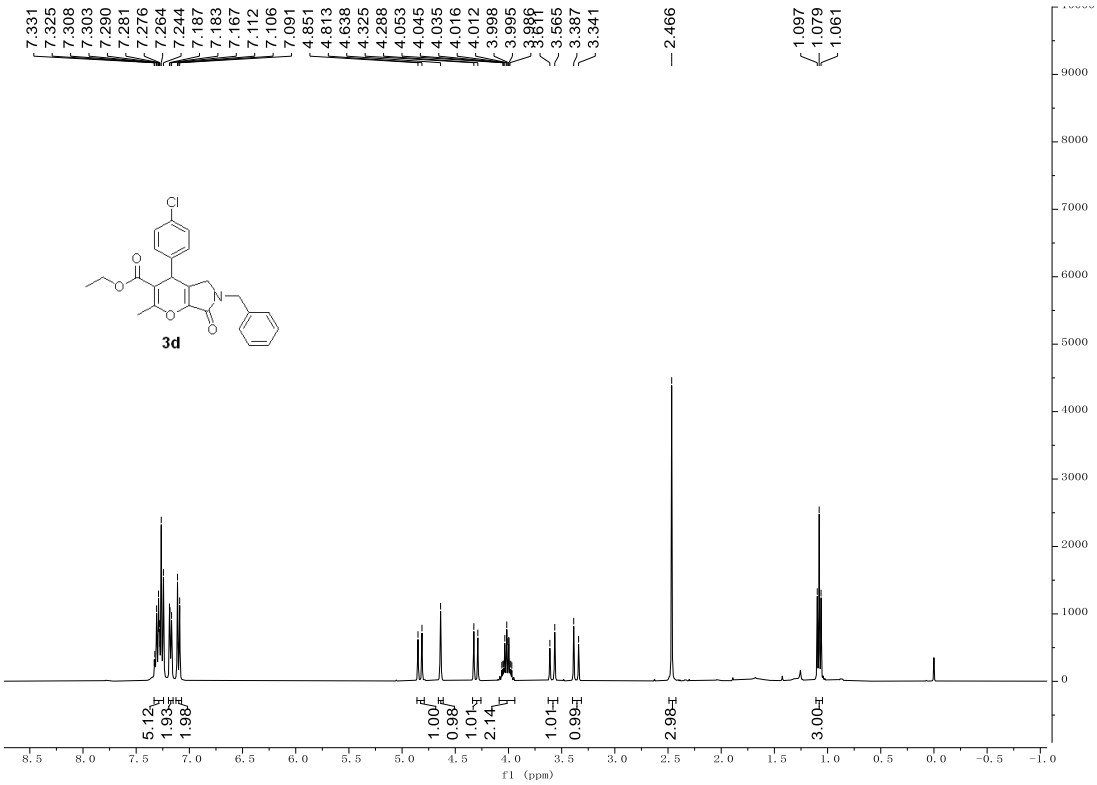
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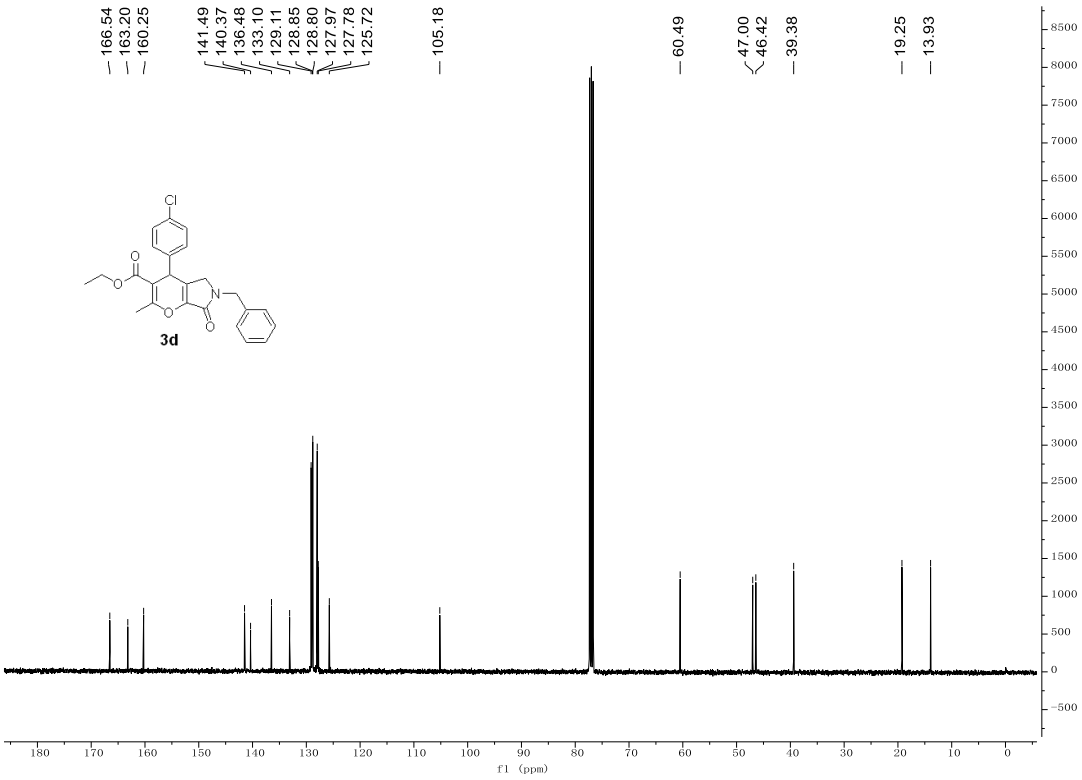
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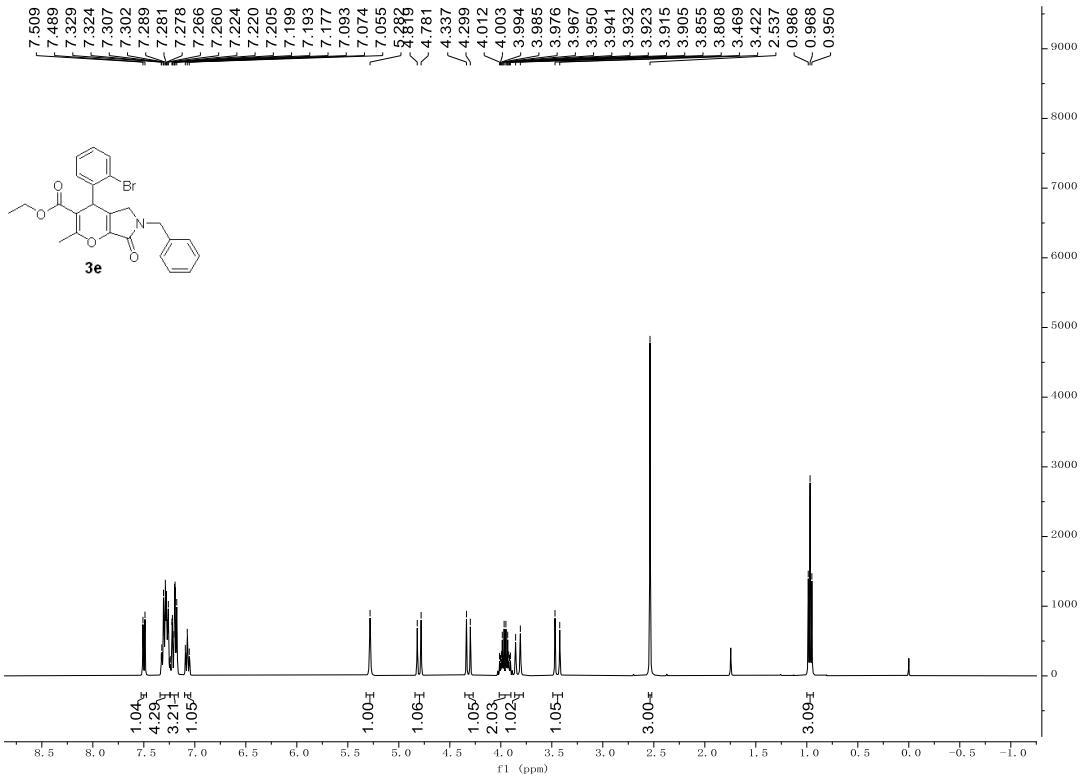
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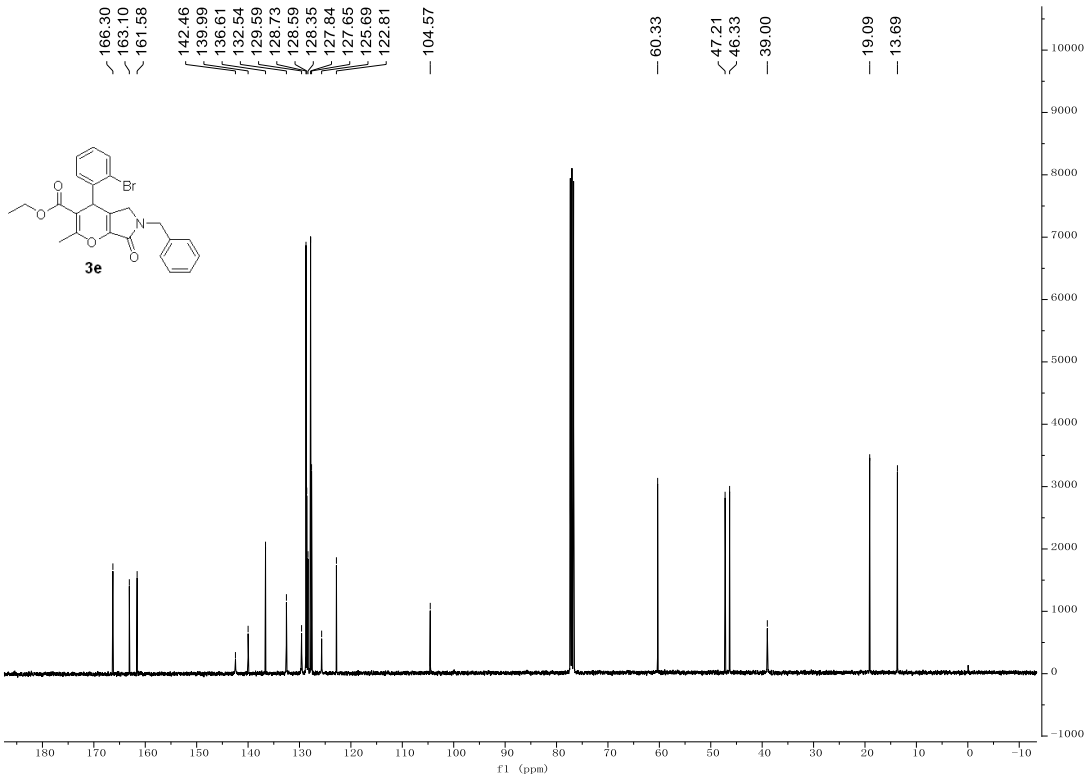
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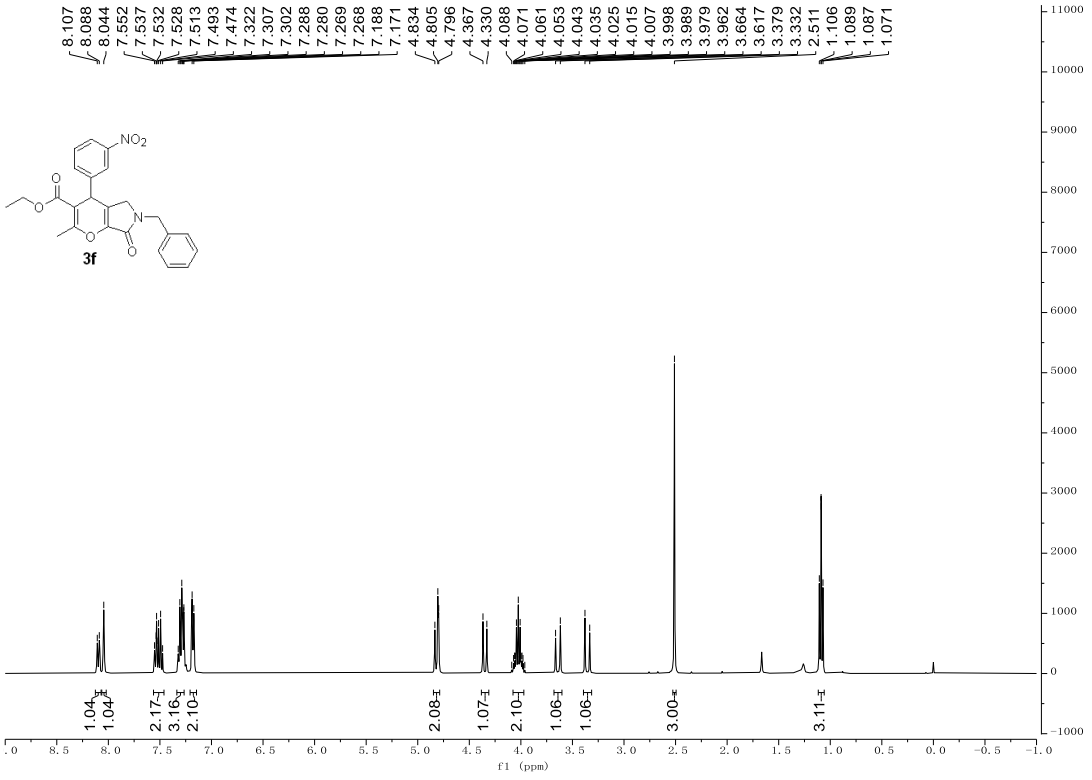
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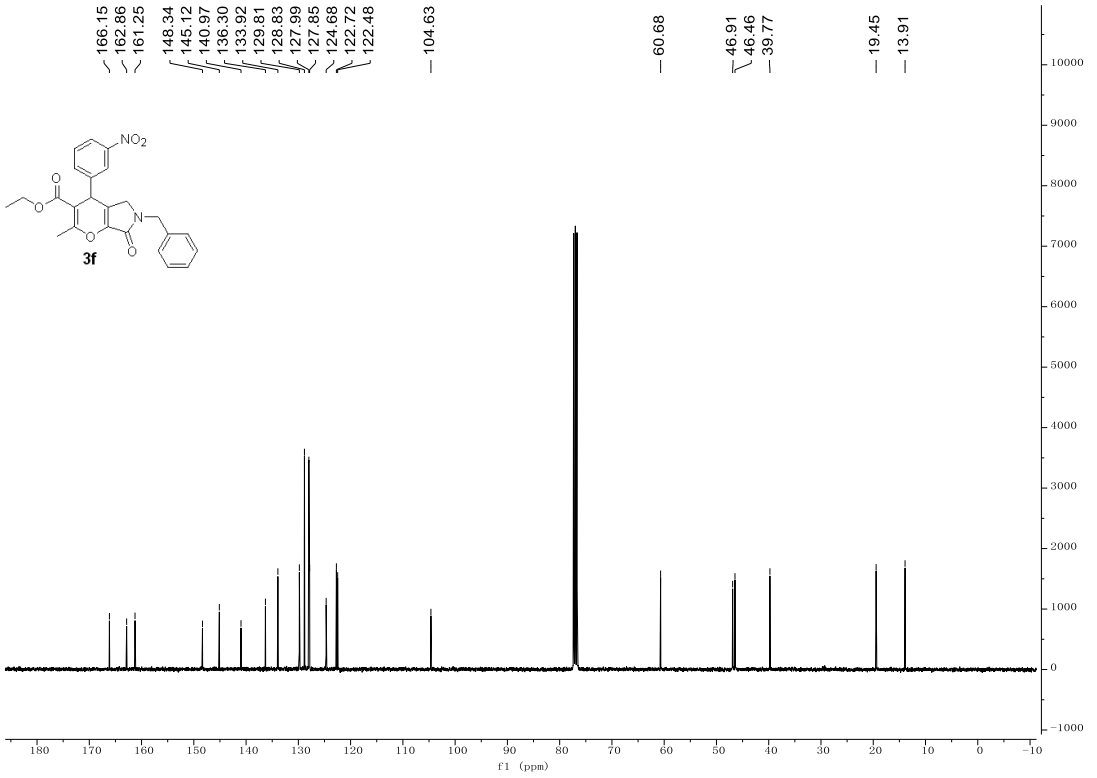
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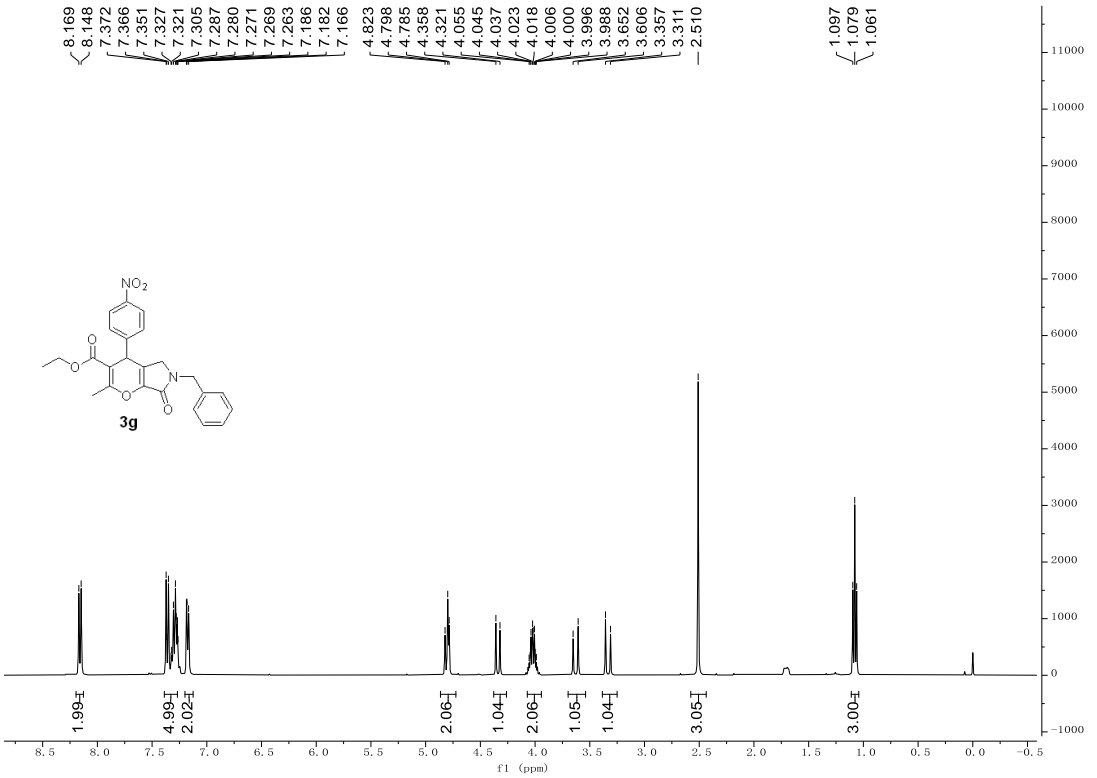
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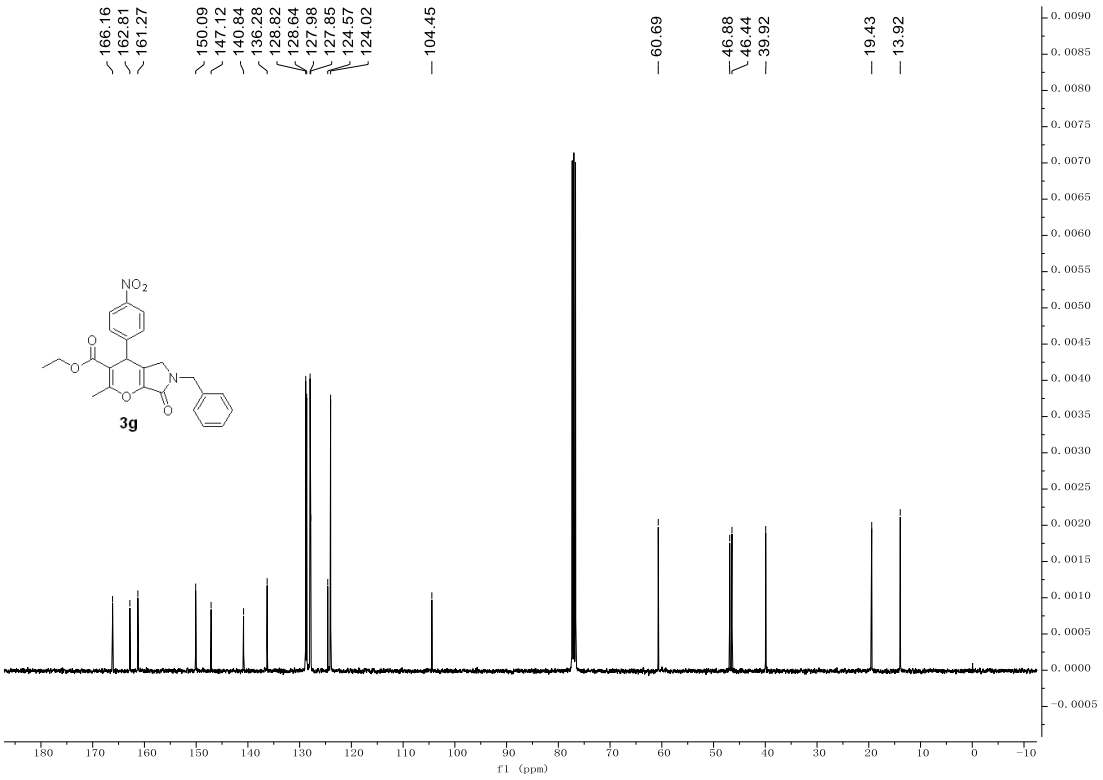
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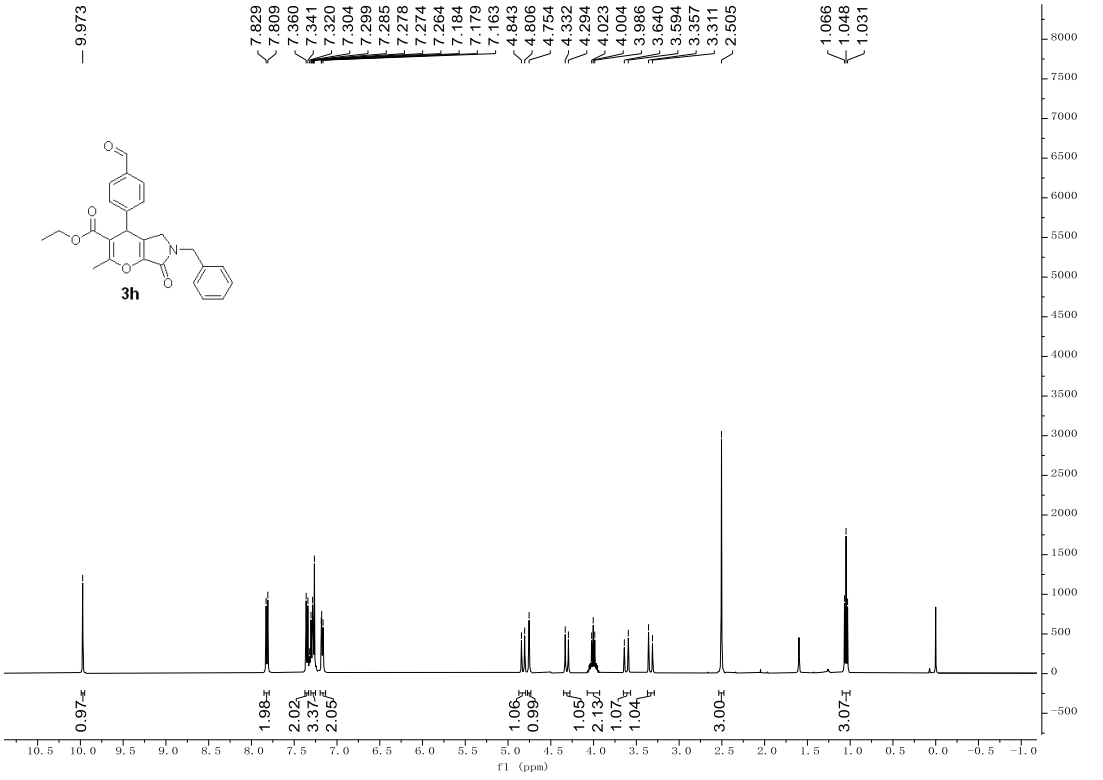
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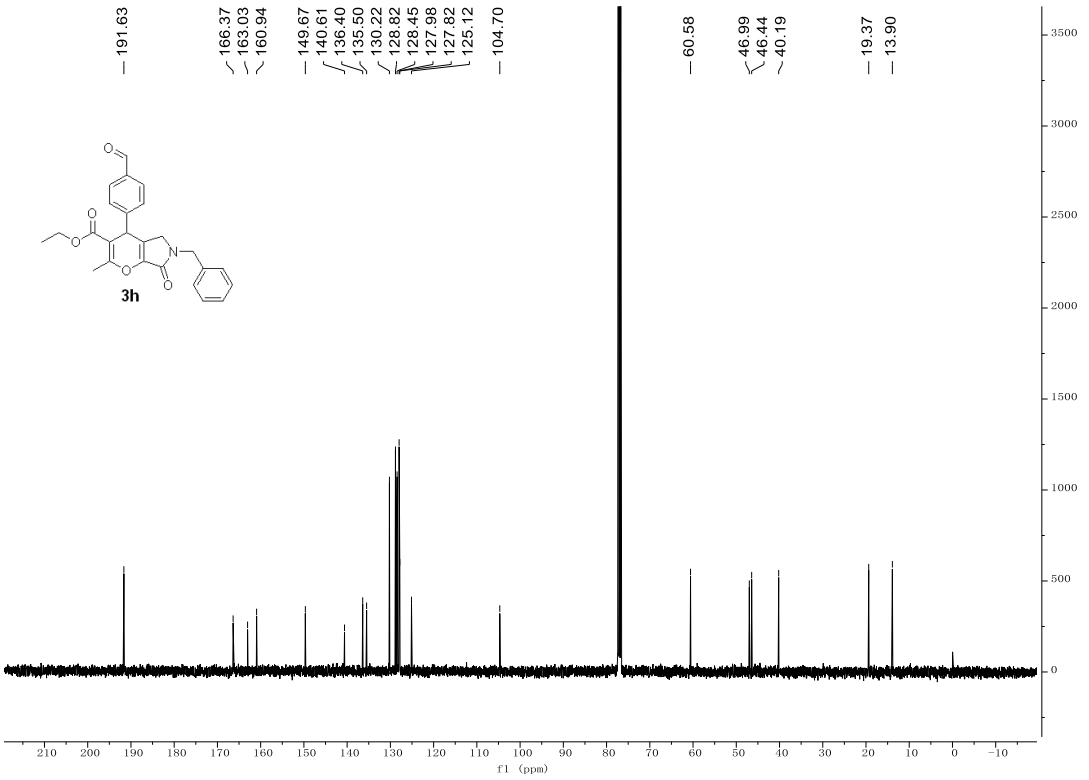
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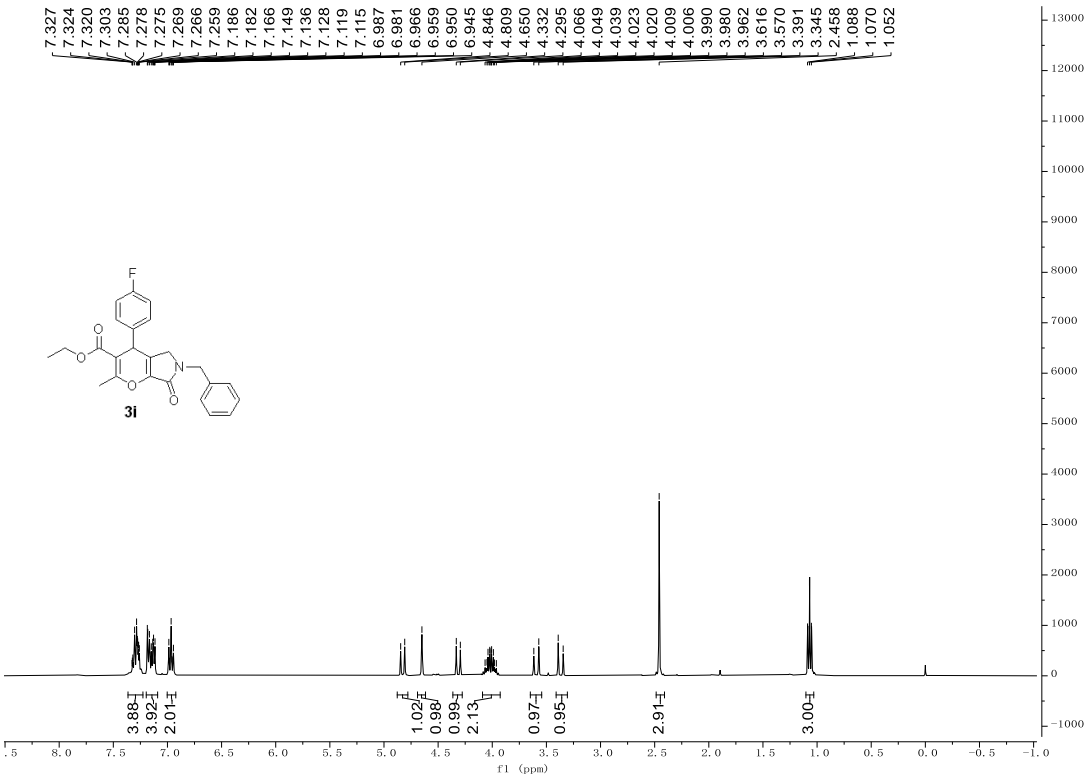
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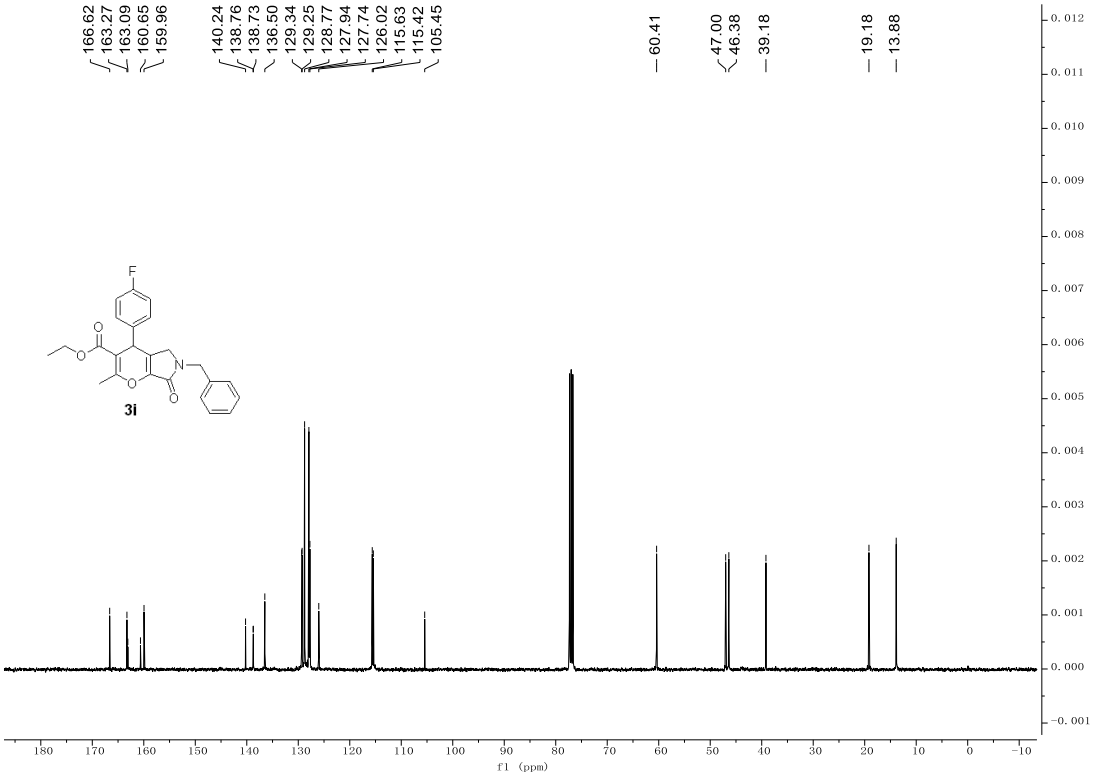
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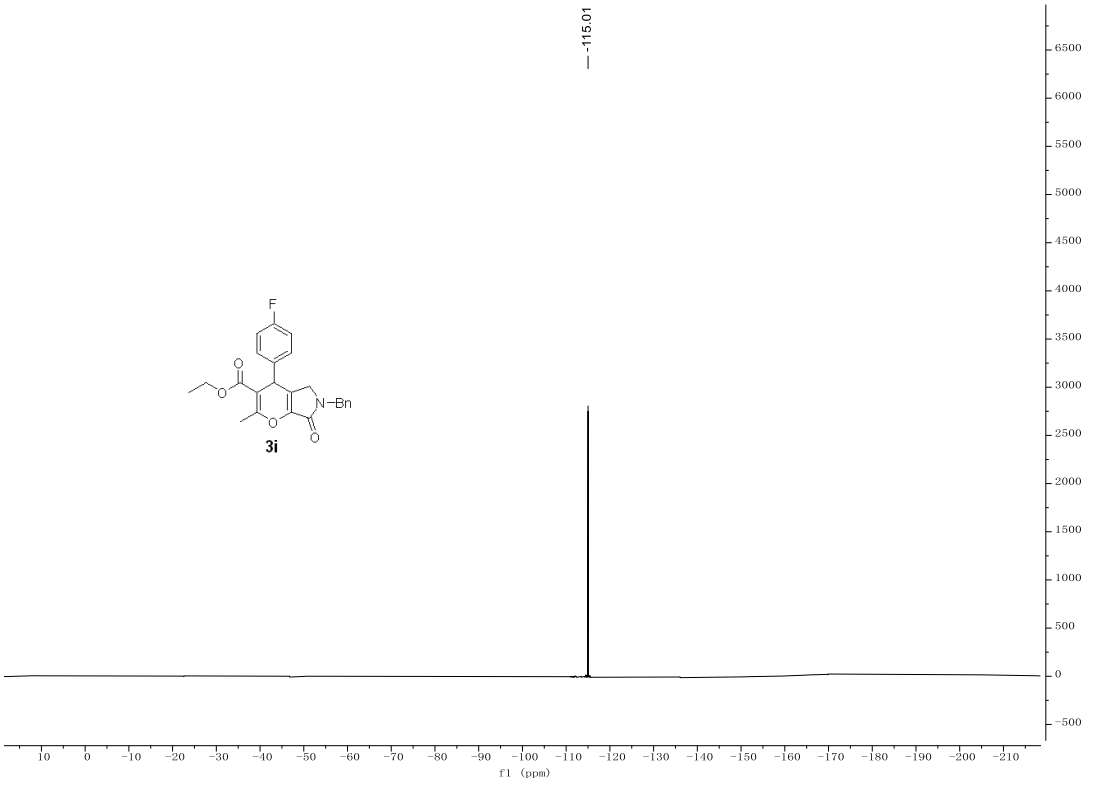
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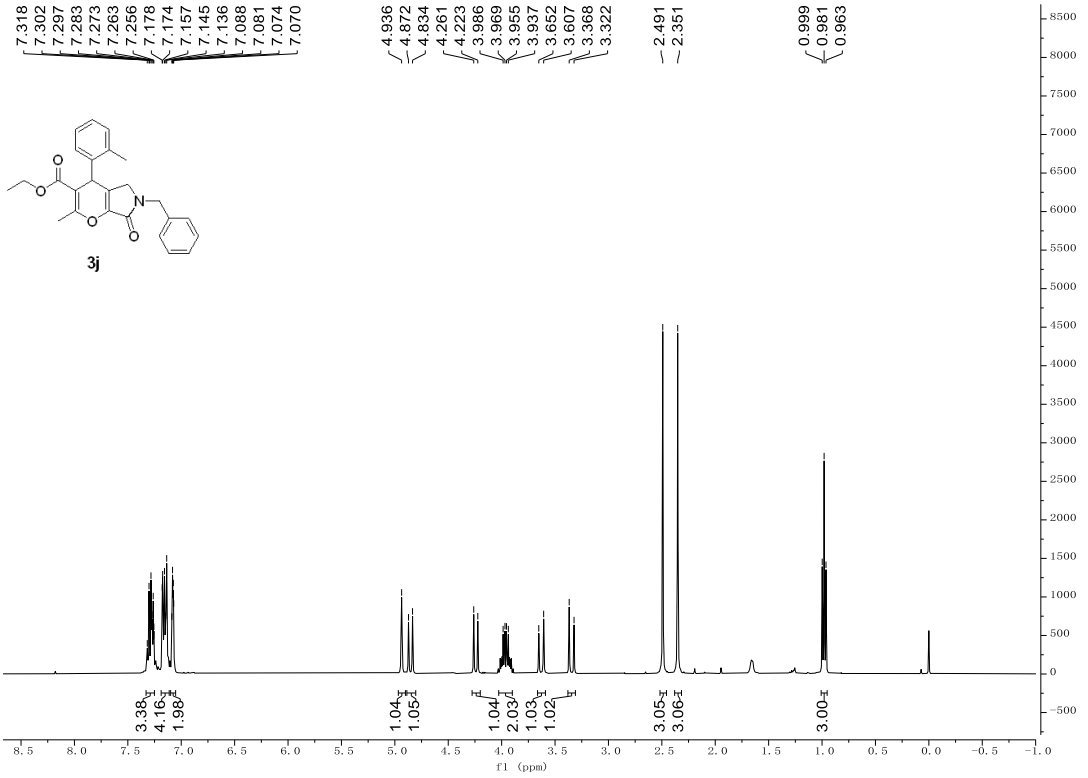
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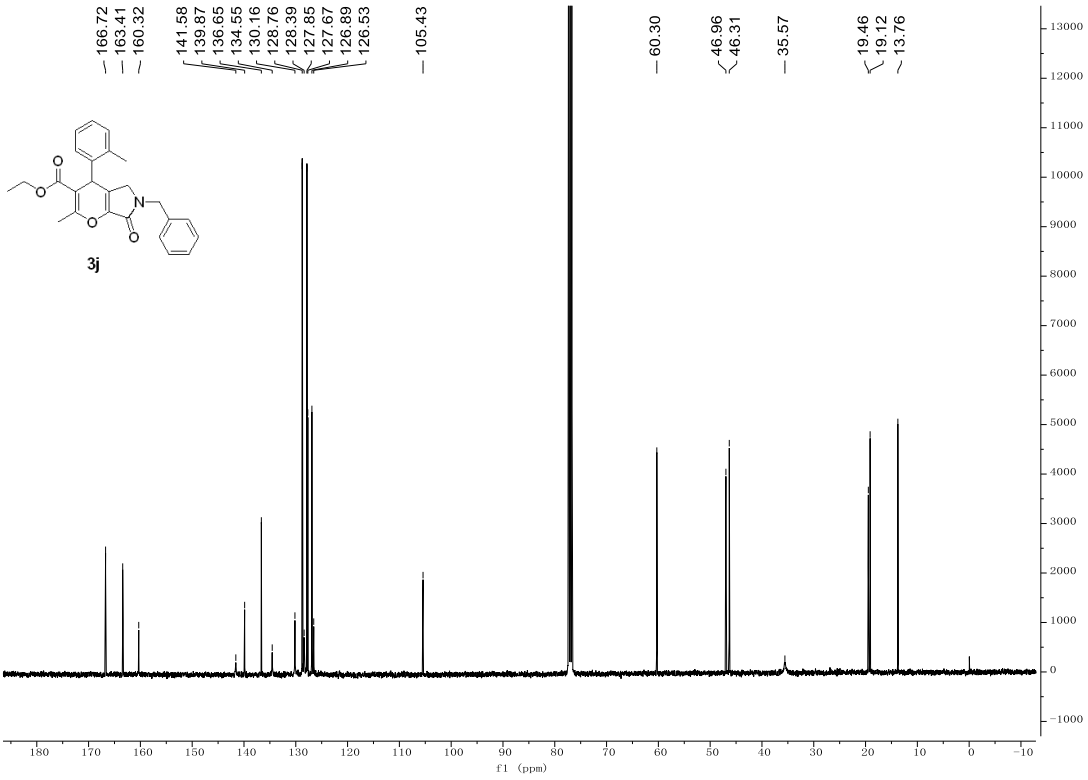
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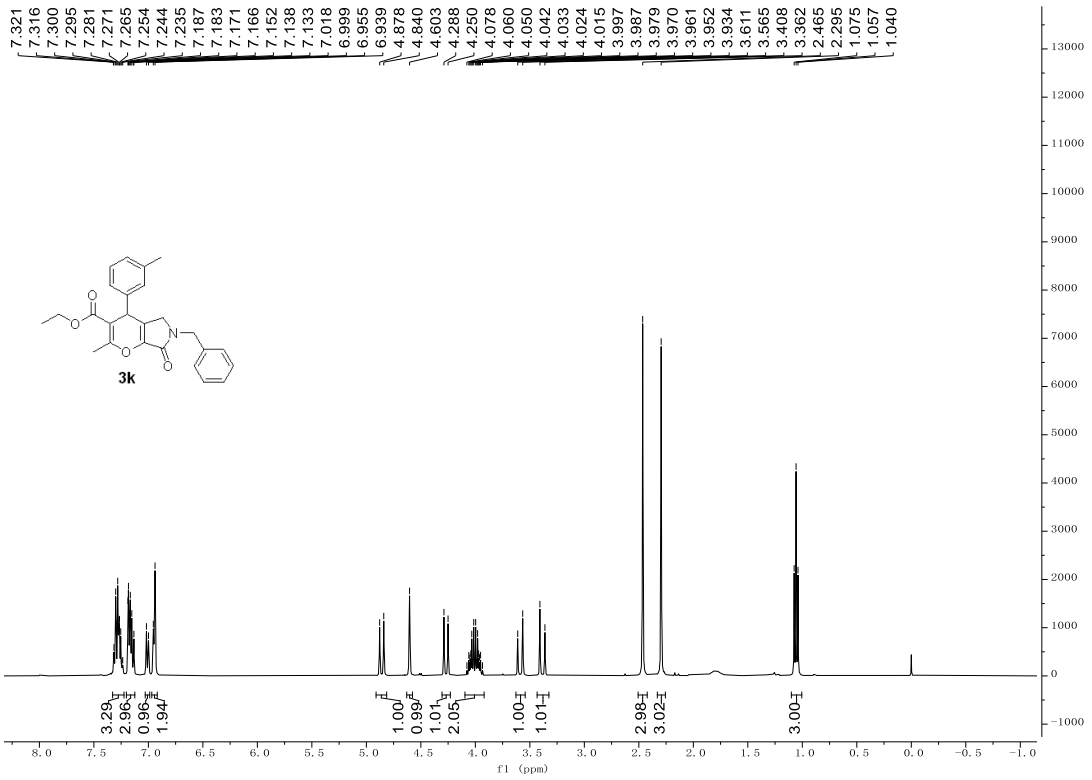
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7.133
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4.060
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1.040
3k
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1.01
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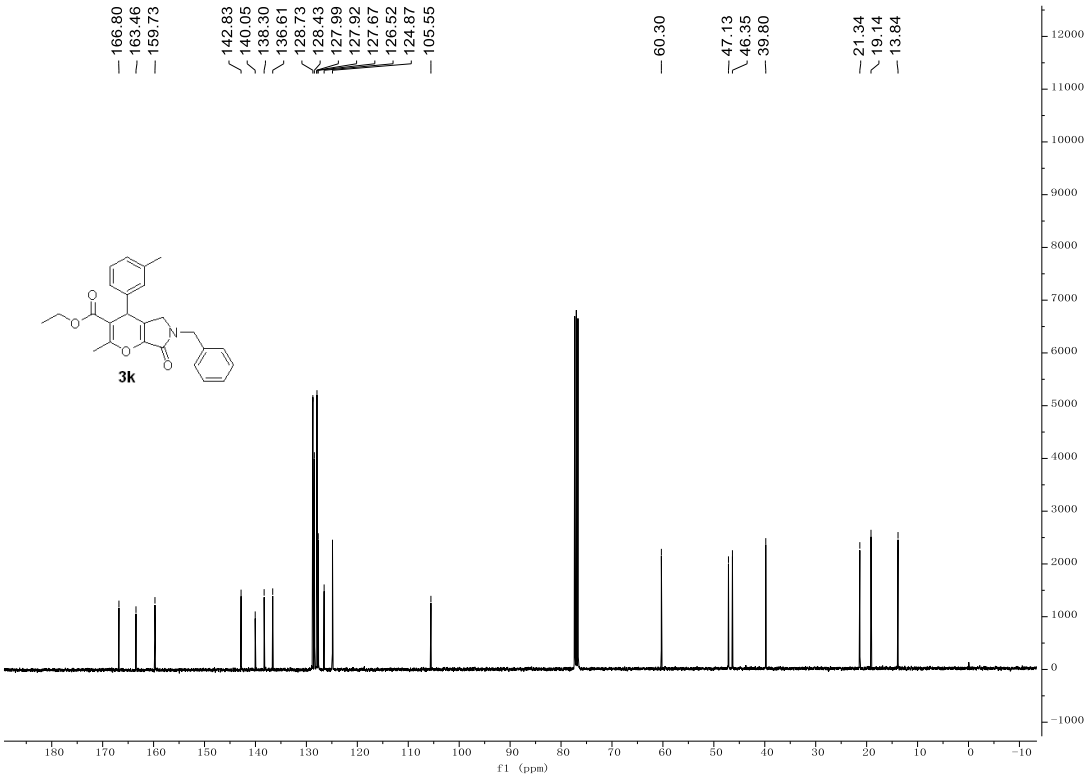
3.00
f1 (ppm)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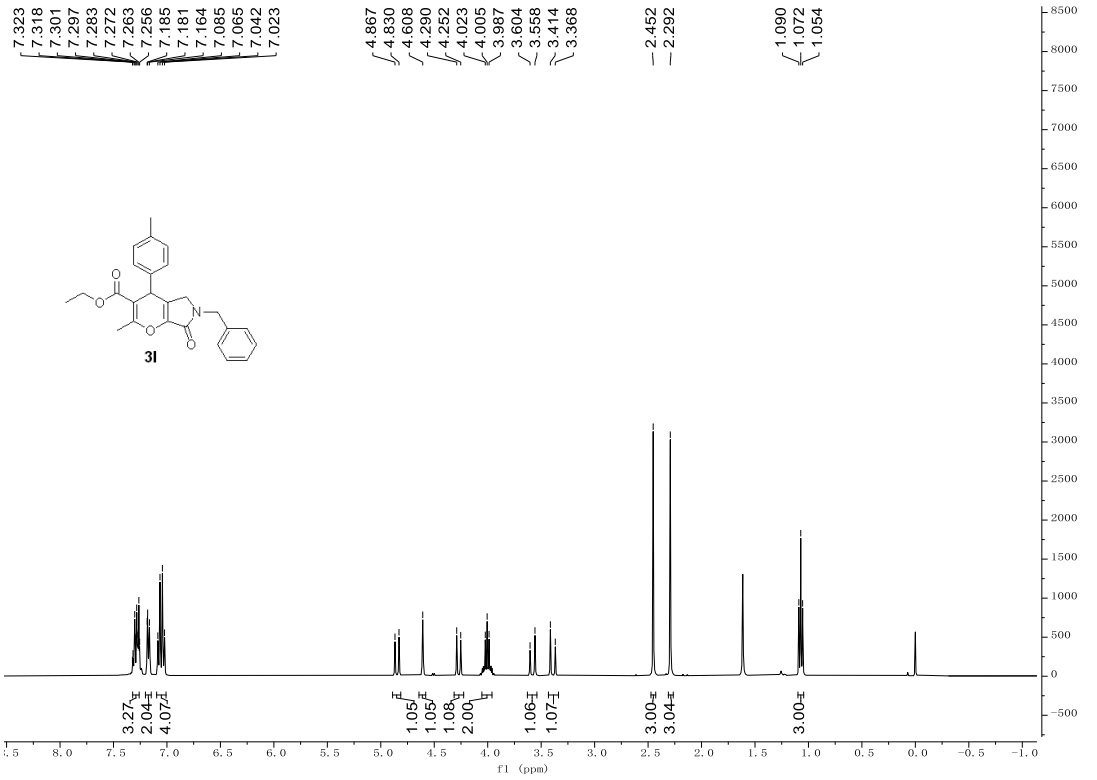
3k
f1 (ppm)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1.08
2.00
1.06
1.07
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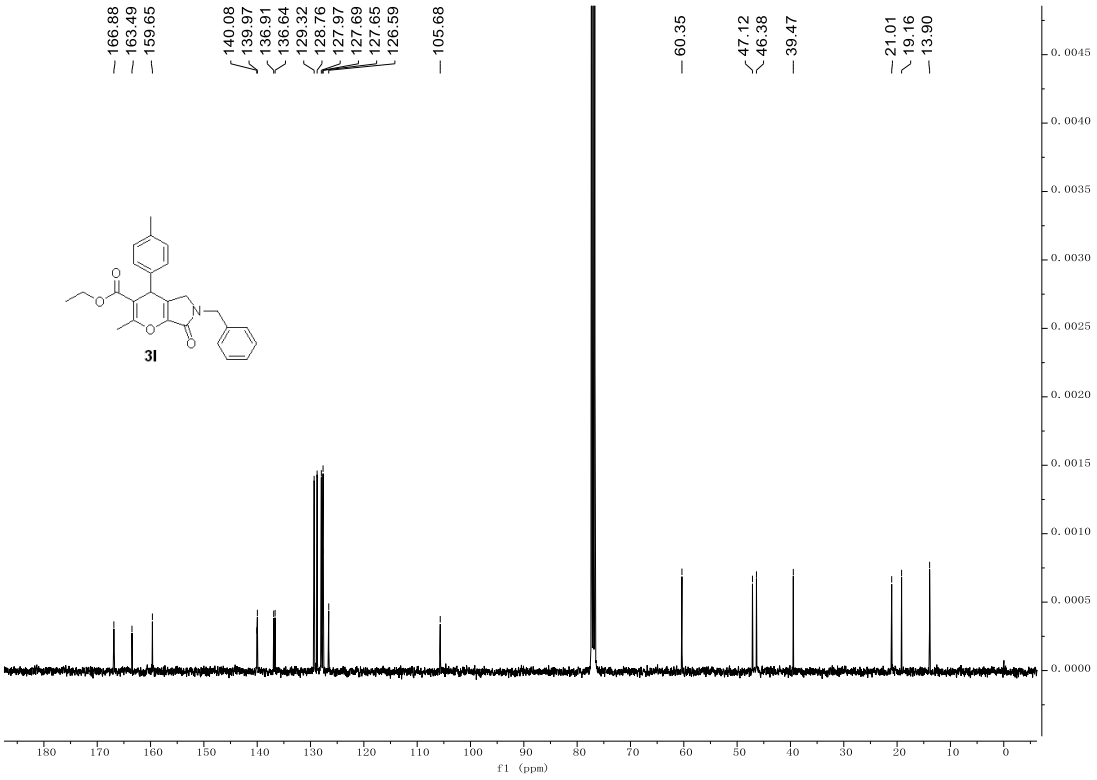
3.04
3.00
f1 (ppm)
3l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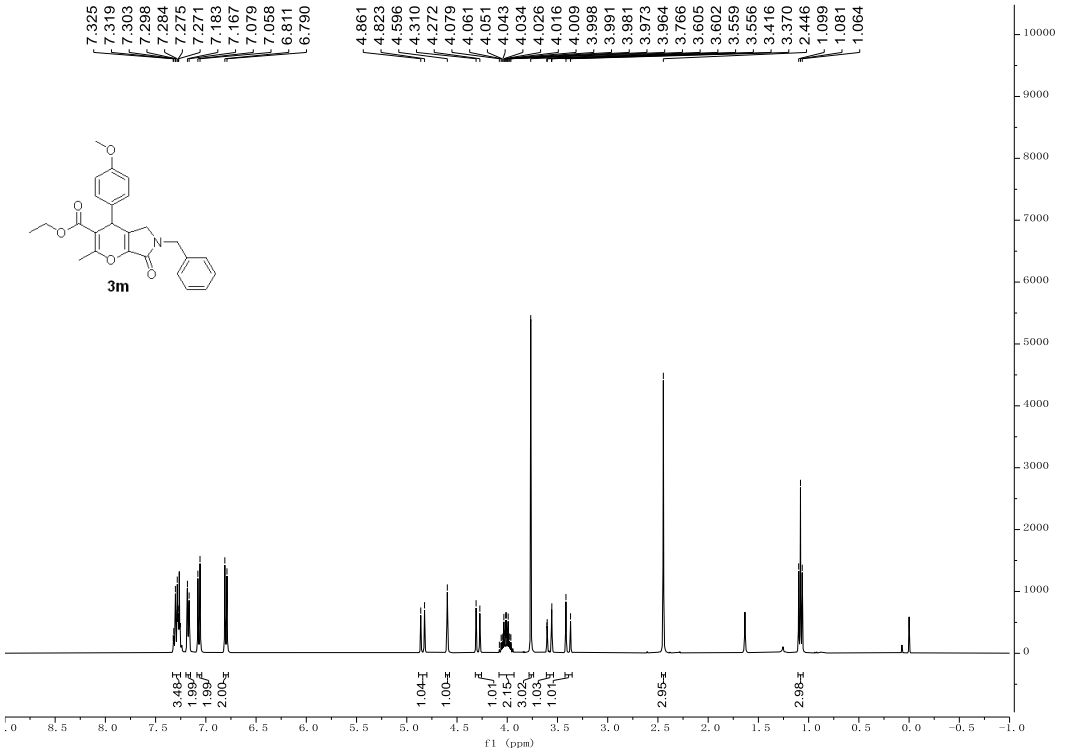
3l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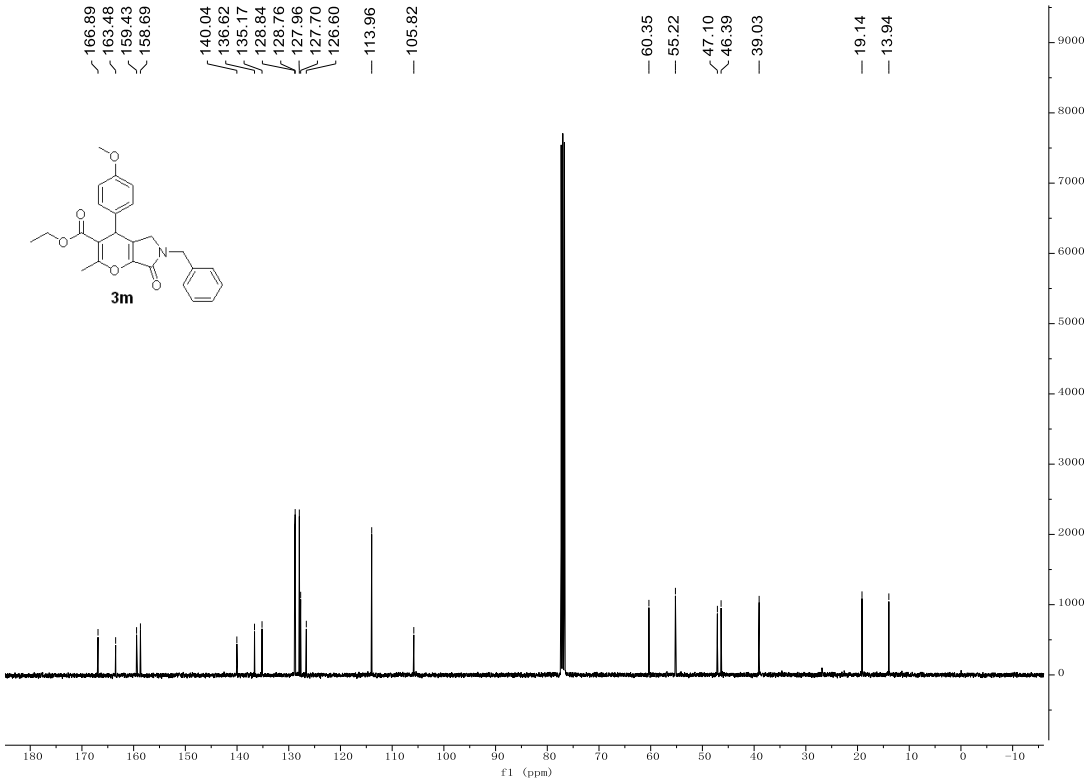
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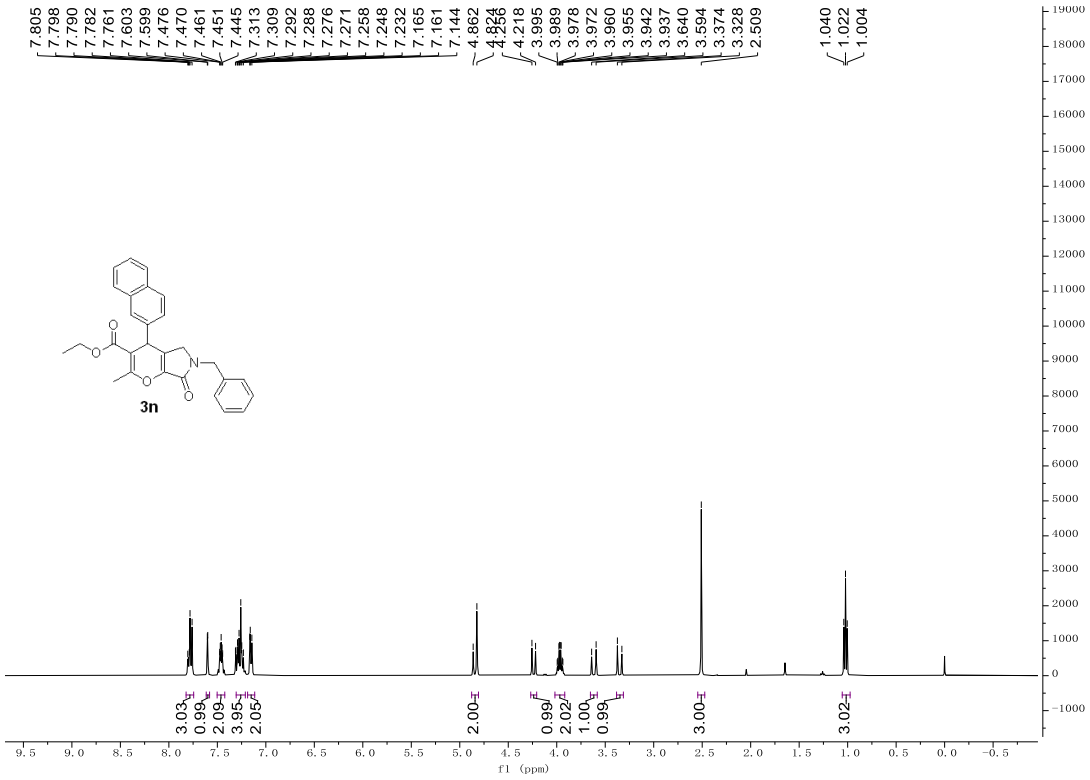
f1 (ppm)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7.161
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3.989
3.978
3.972
3.960
3.955
3.942
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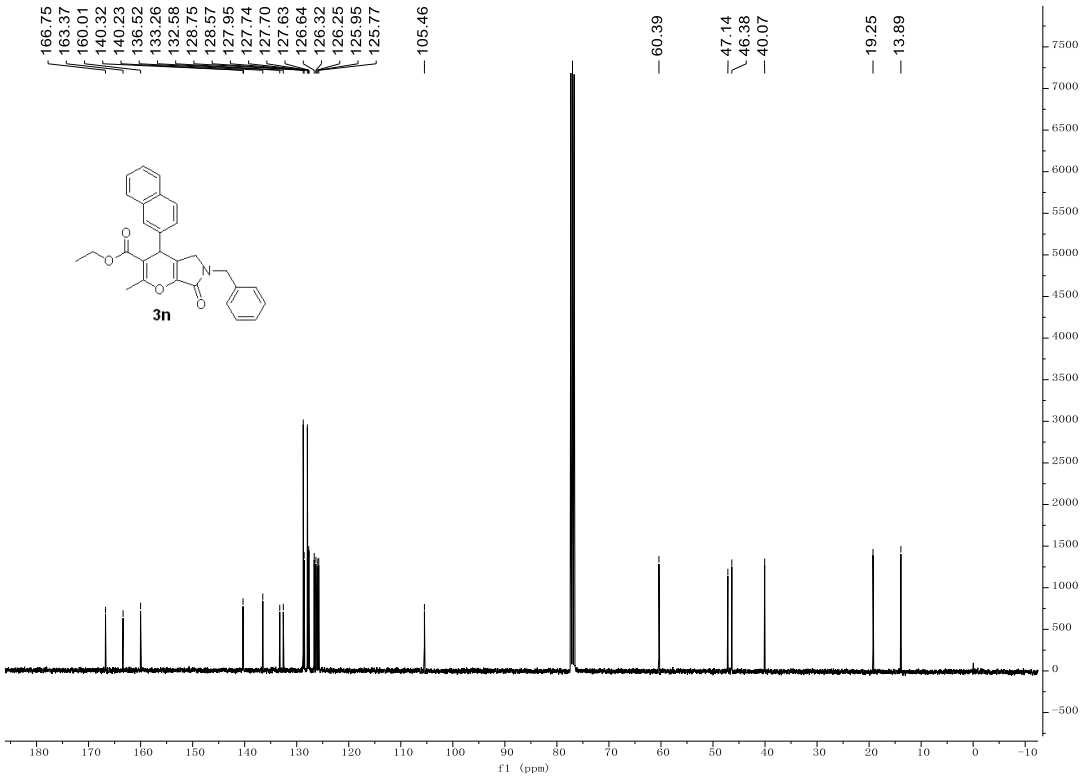
f1 (ppm)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126.32
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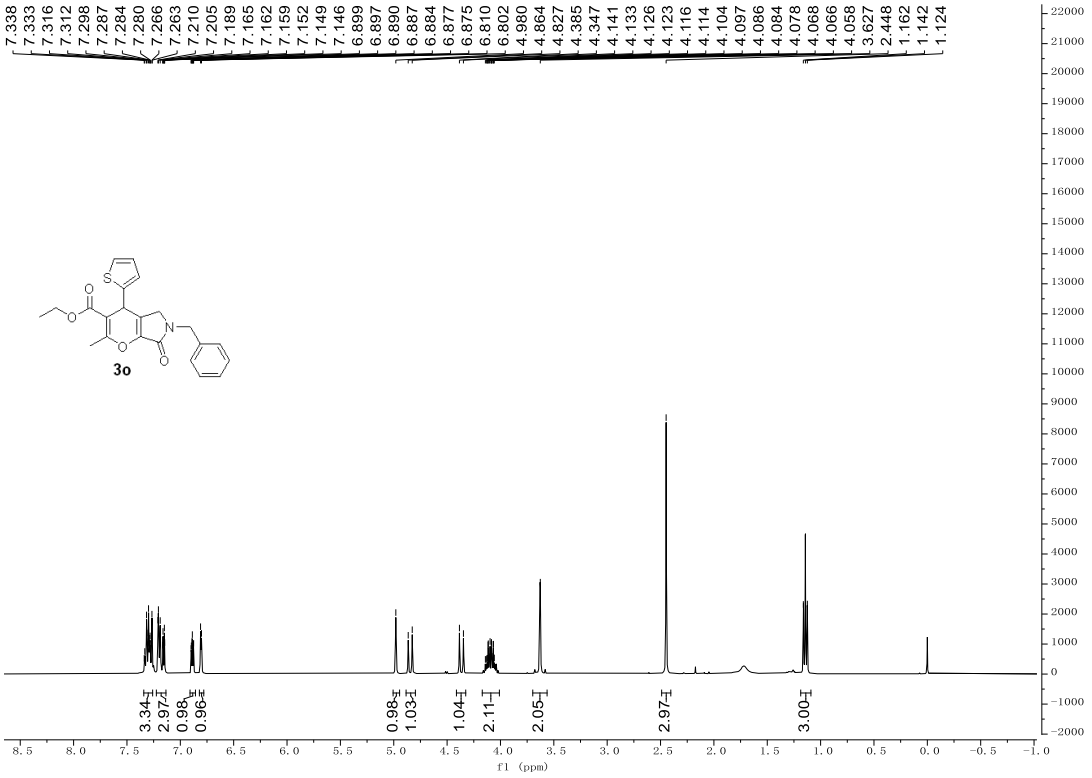
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6.884
6.877
6.875
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1.124
3o
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0.98
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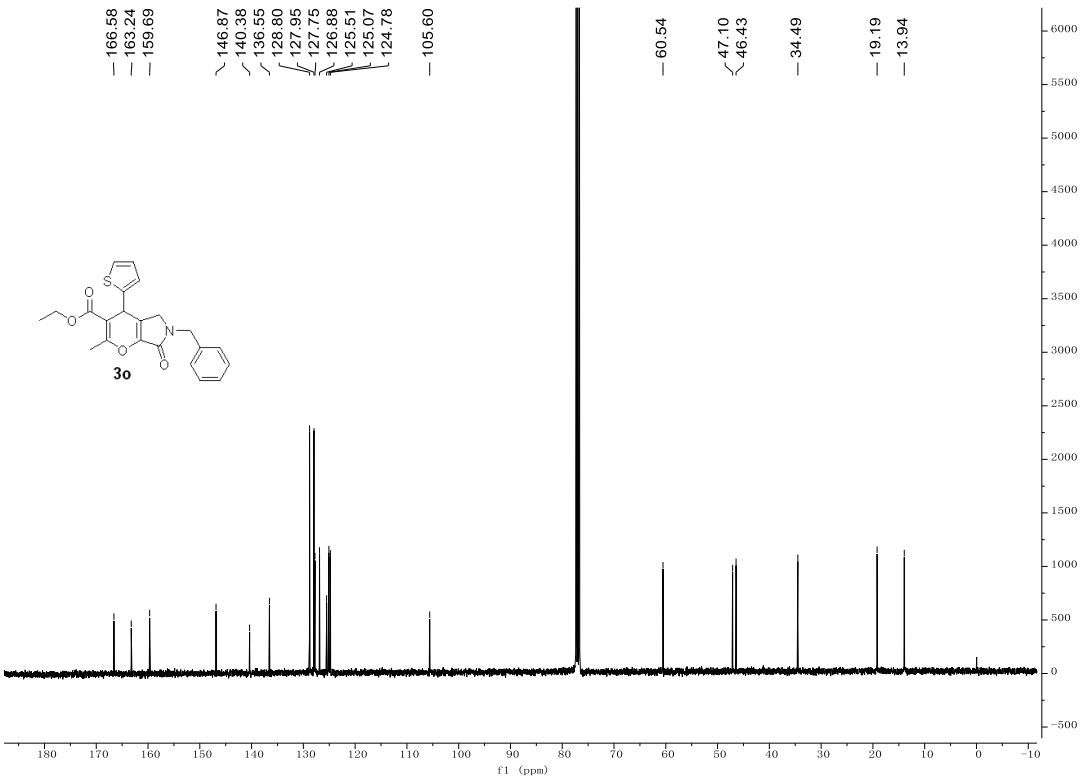
f1 (ppm)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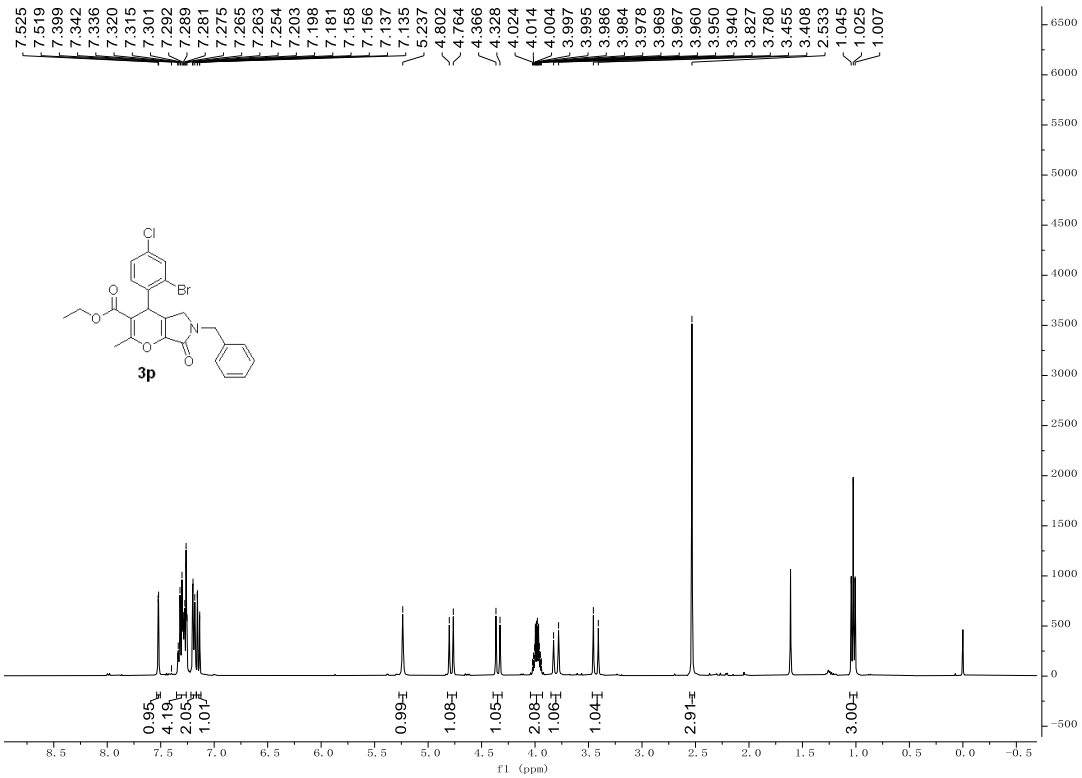
3o
f1 (ppm)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1.025
1.007
Cl
Br
O
O
N
O
O
3p
0.95
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1.05
2.08
1.06
1.04
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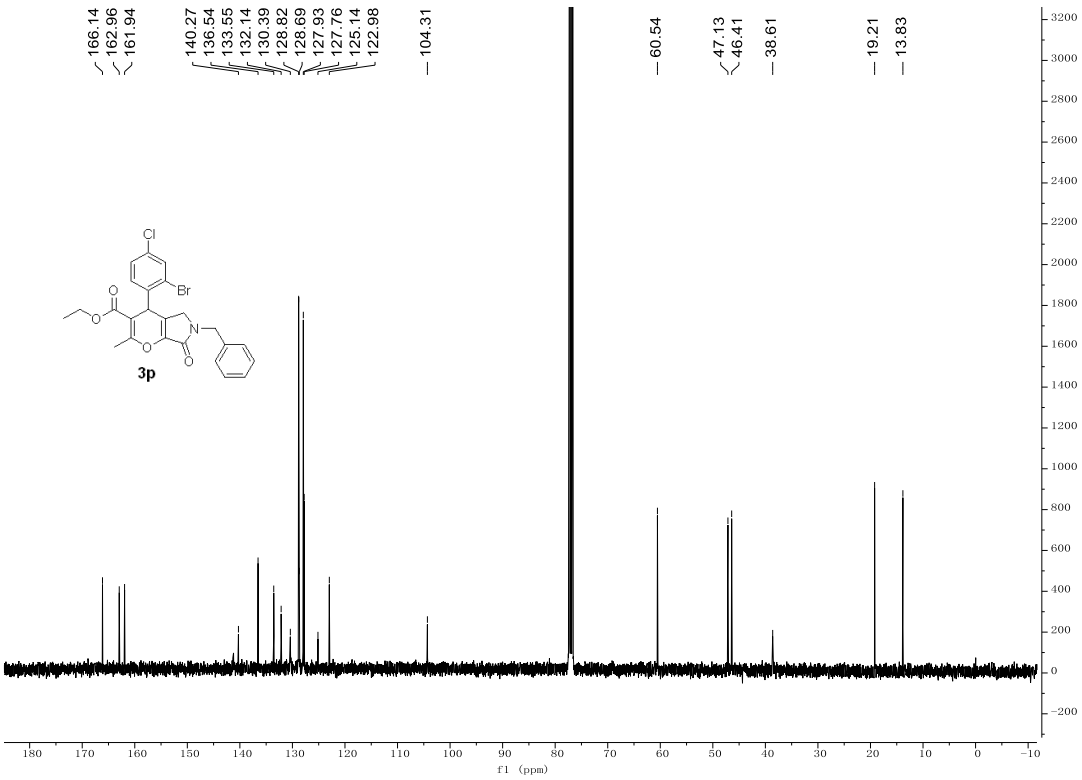
3.00
f1 (ppm)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104.31
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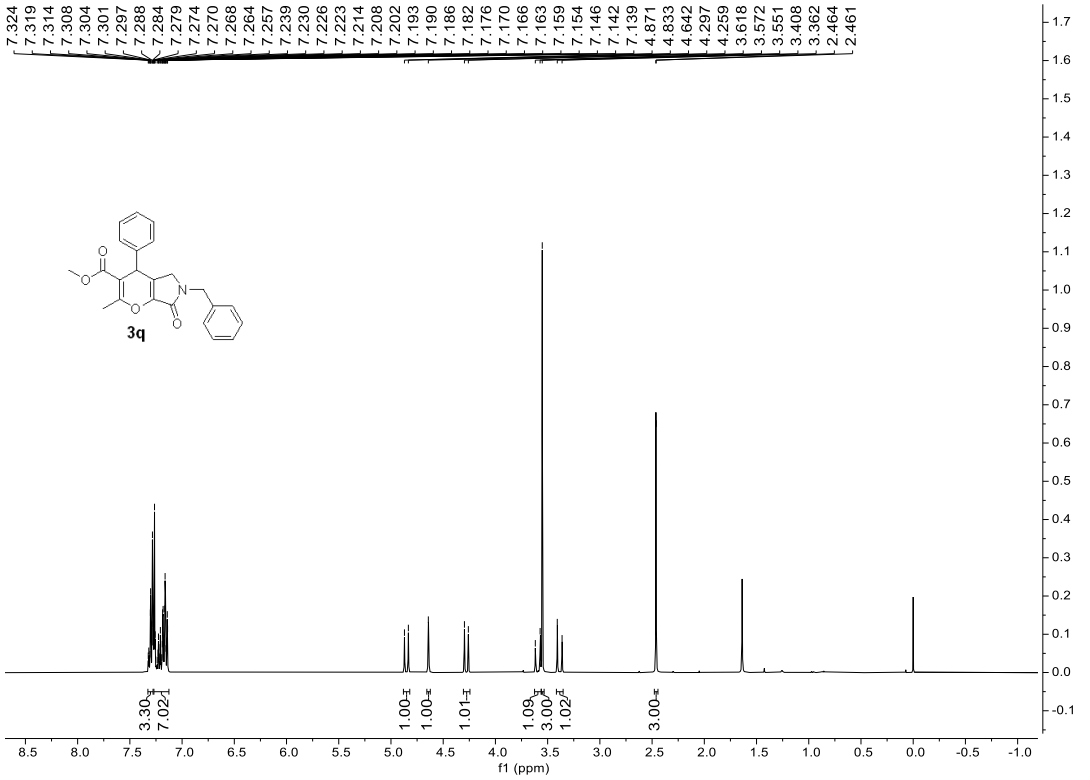
13.83
Cl
Br
O
O
N
O
O
3p
f1 (ppm)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7.182
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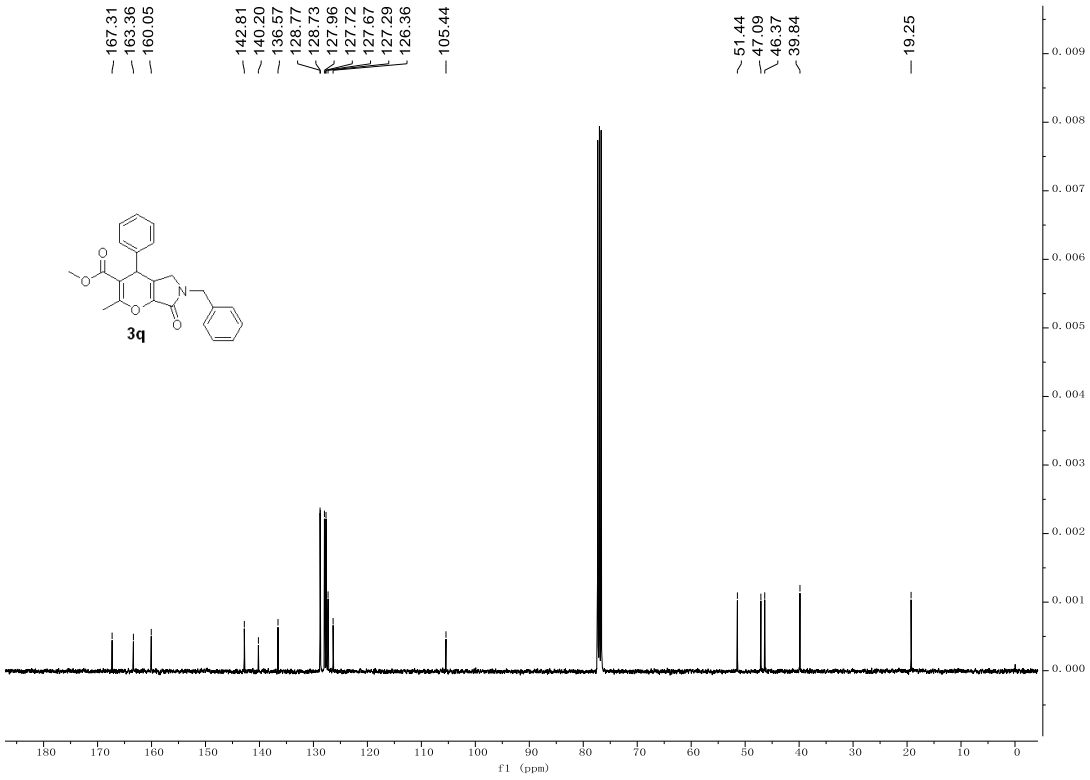
3.00
1.02
3.00
f1 (ppm)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126.36
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51.44
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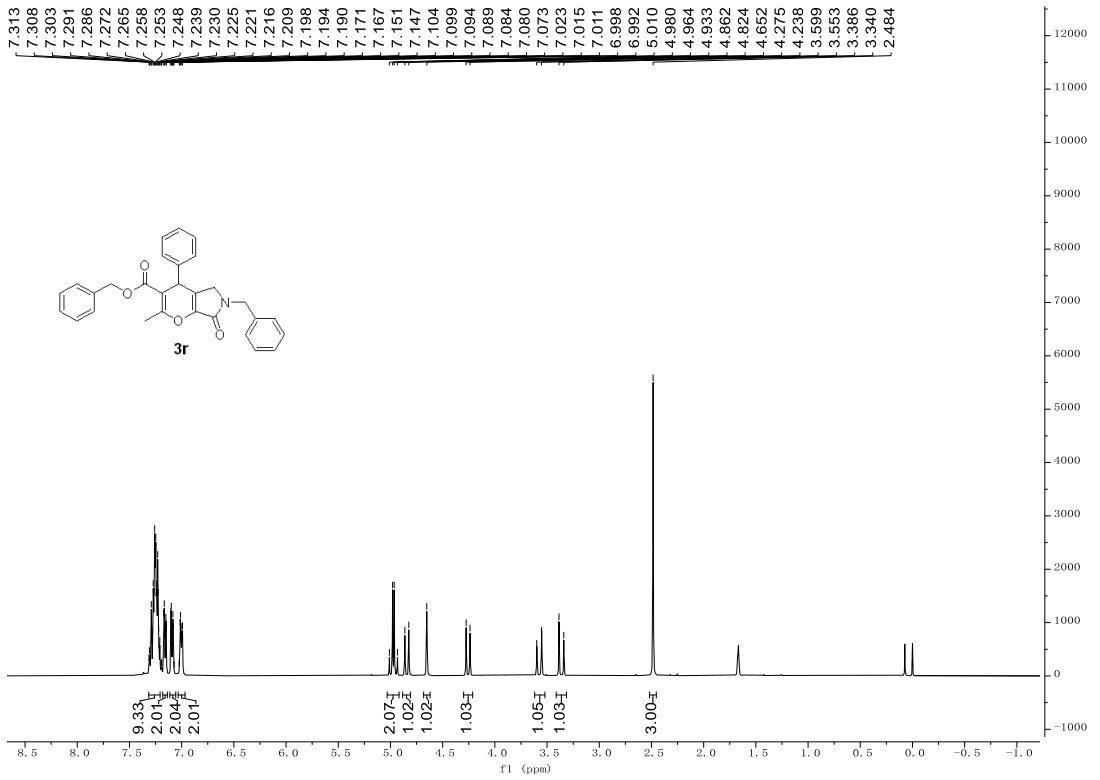
39.84
19.25
3q
f1 (ppm)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1.05
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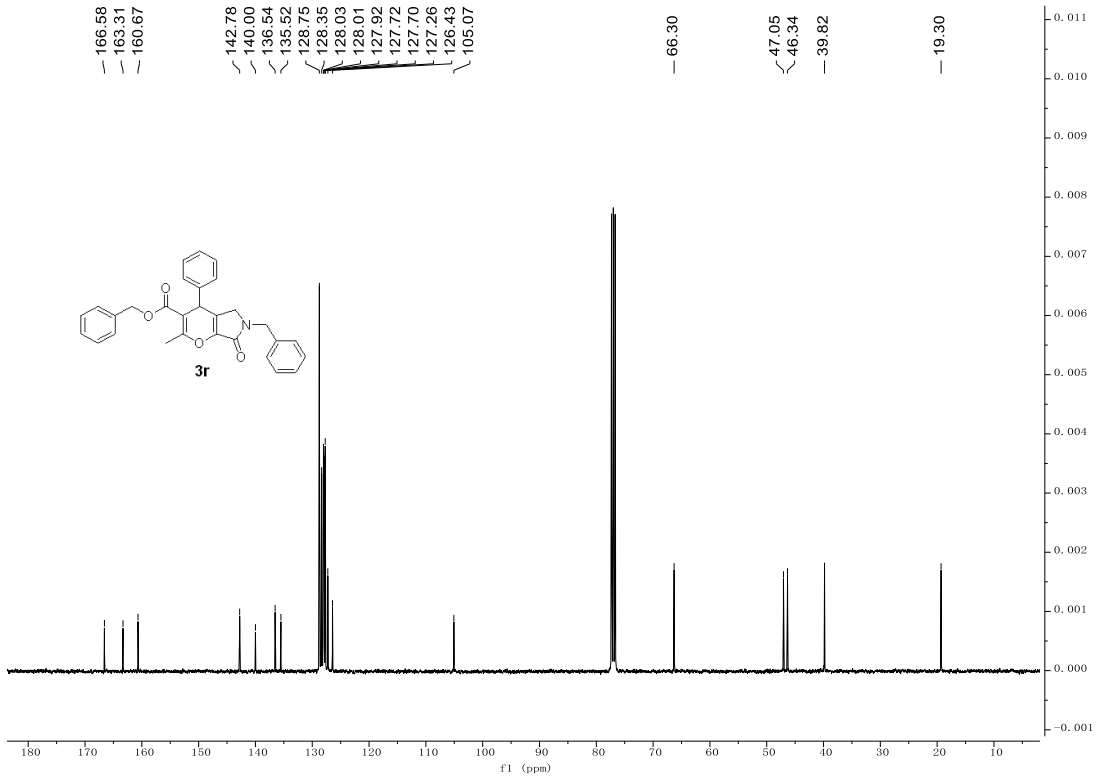
3.00
f1 (ppm)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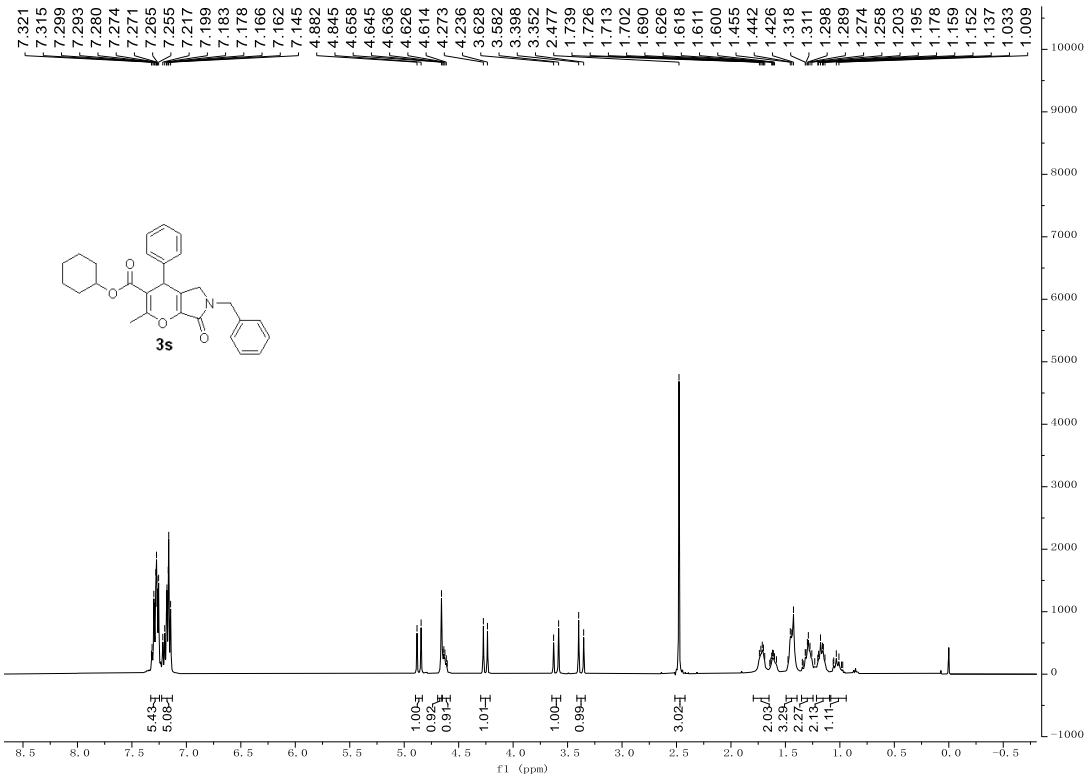
3r
f1 (ppm)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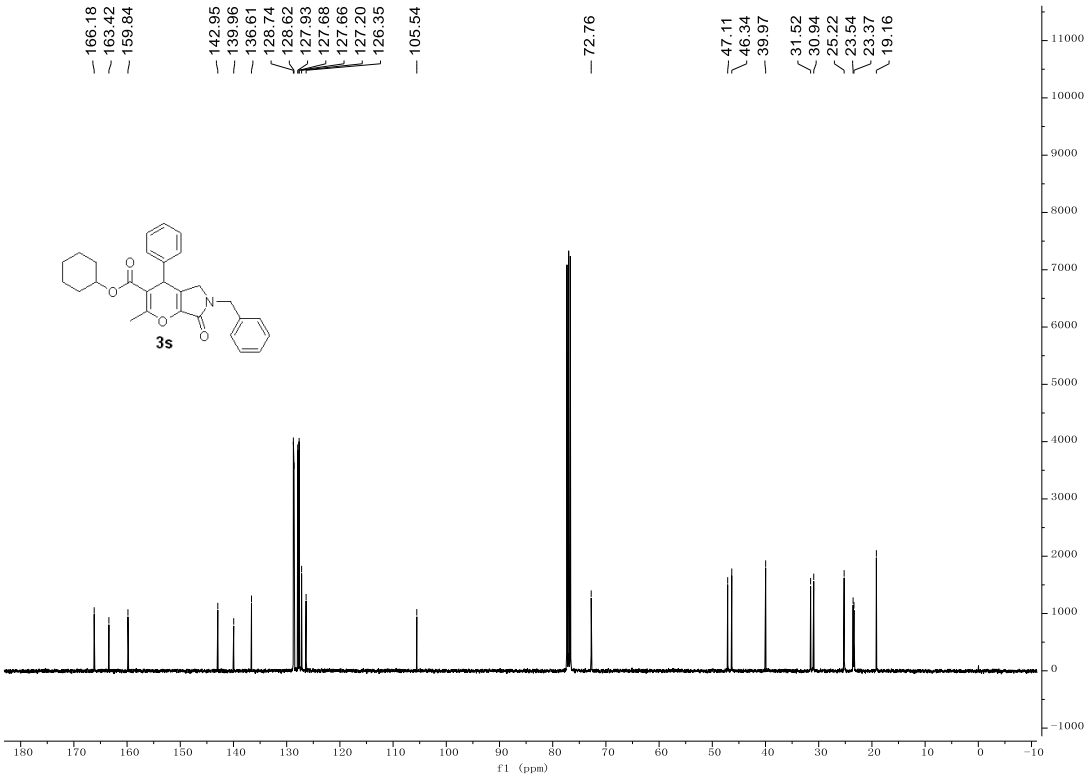
f1 (ppm)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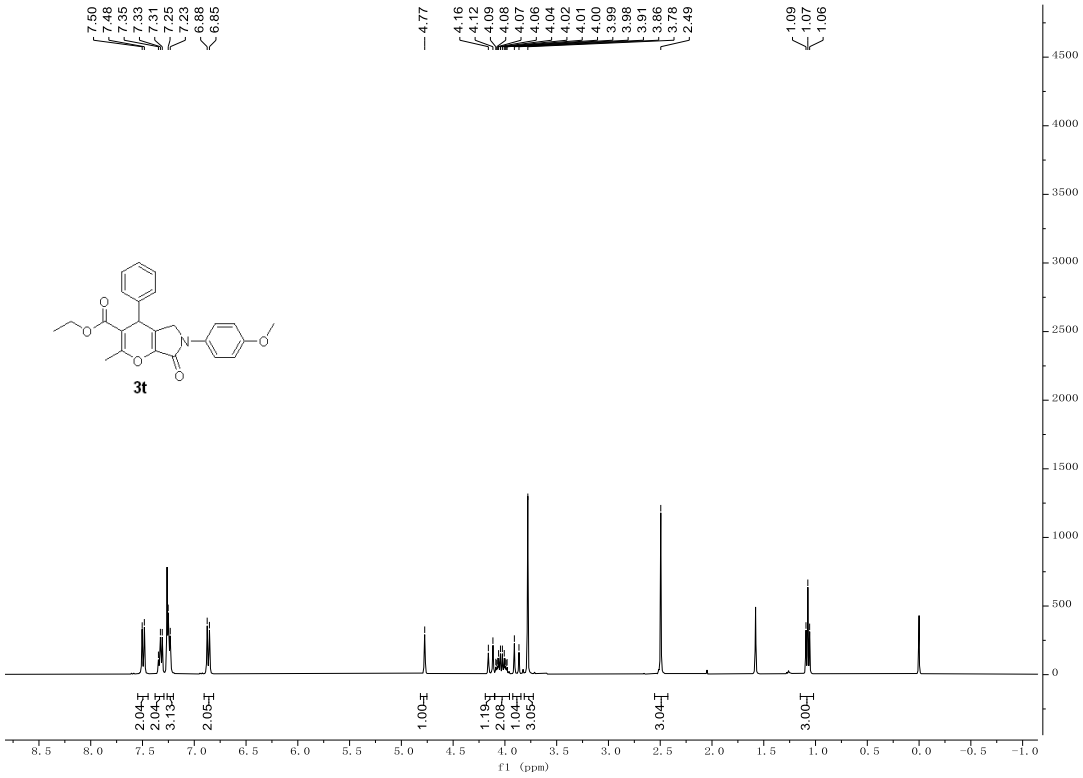
3s
f1 (ppm)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3t
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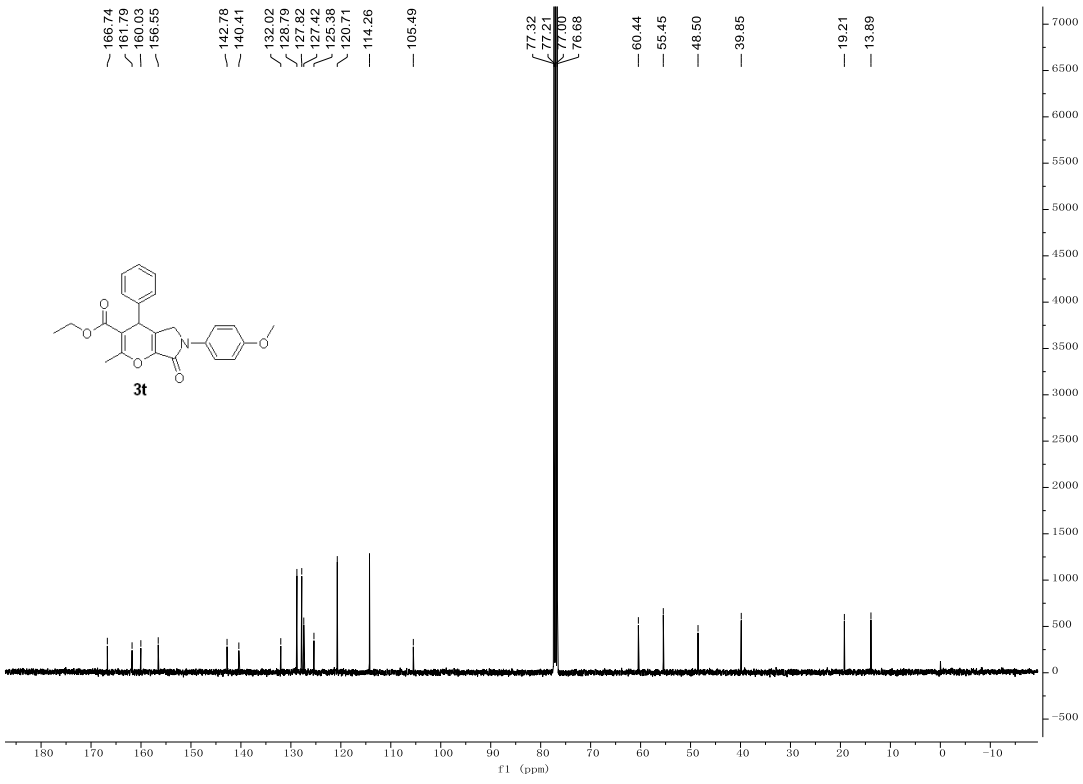
f1 (ppm)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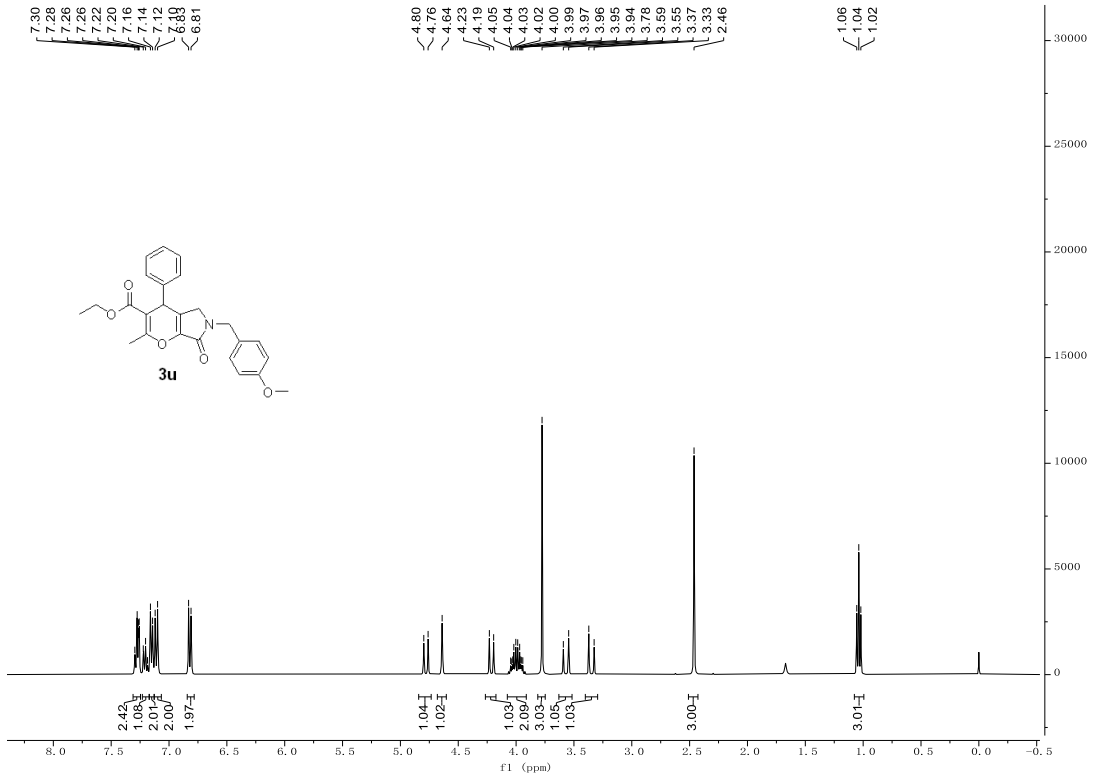
f1 (ppm)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4.05
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4.03
4.02
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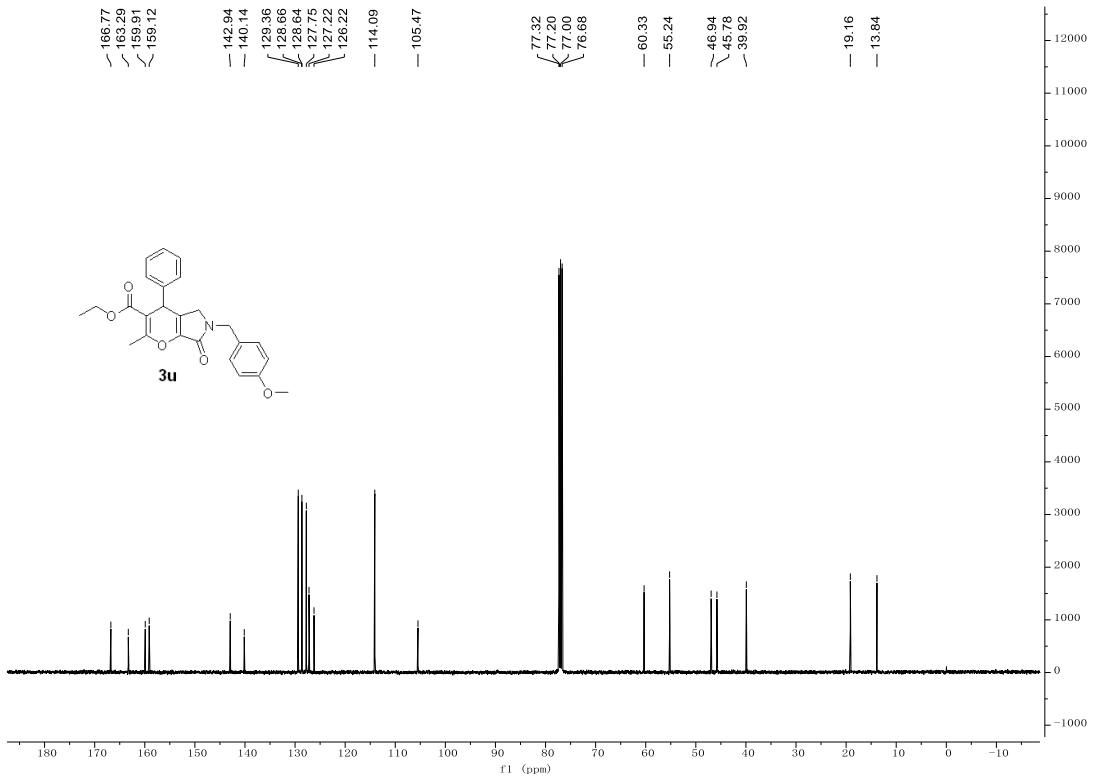
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126.22
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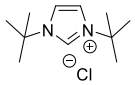
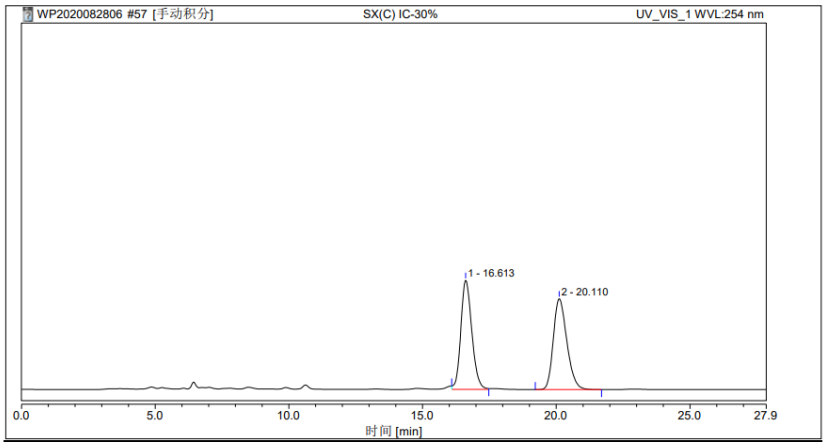
105.47
77.32
77.20
77.00
76.68
60.33
55.24
46.94
45.78
39.92
19.16
13.84
3u
f1 (ppm)

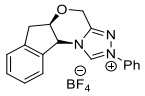
3 HPLC data of 3a

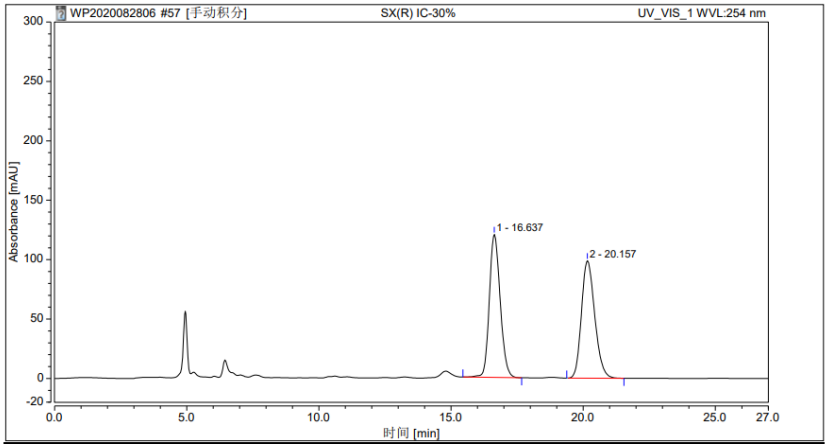
We measured the enantiomeric excess of several samples when chiral NHCs were employed. The enantiomeric excesses (ee) were determined by HPLC with a chiral IC column.

NHC: 

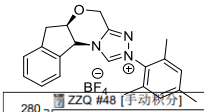


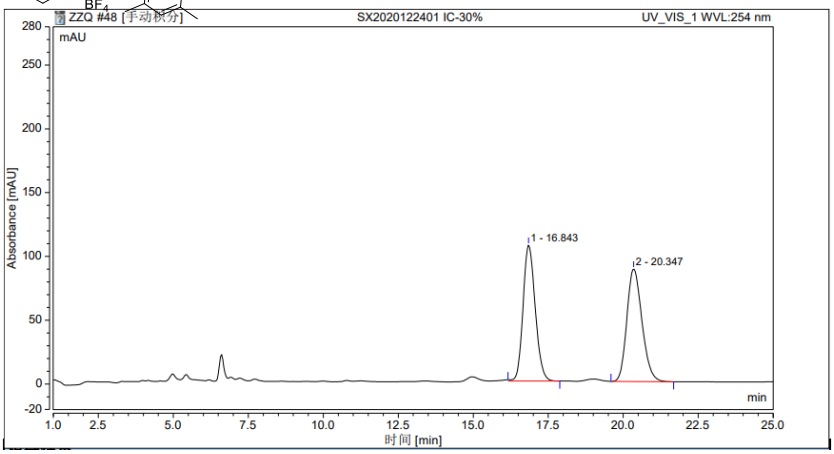
Peak	Processed Channel	Reatention Time (min)	Peak Area (mAU*min)	Peak Height (mAU)	Peak Area (%)
1	DAD 245.0 nm	16.613	94.119	196.830	49.47
2	DAD 245.0 nm	20.110	96.117	163.787	50.53

NHC: 

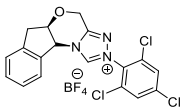


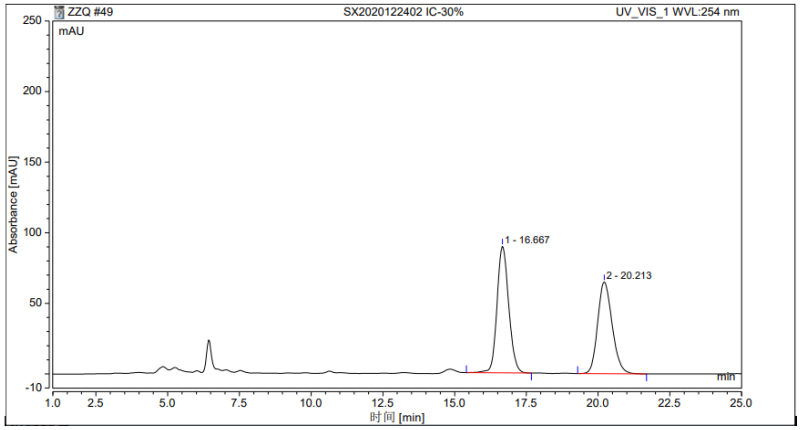
Peak	Processed Channel	Reatention Time (min)	Peak Area (mAU*min)	Peak Height (mAU)	Peak Area (%)
1	DAD 245.0 nm	16.637	57.370	120.394	49.87
2	DAD 245.0 nm	20.157	57.662	98.899	50.13

NHC: 

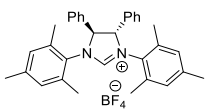


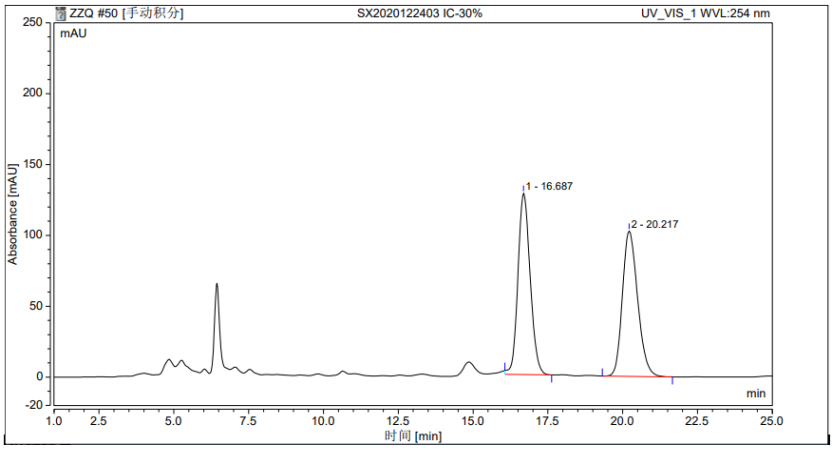
Peak	Processed Channel	Reatention Time (min)	Peak Area (mAU*min)	Peak Height (mAU)	Peak Area (%)
1	DAD 245.0 nm	16.843	50.228	106.569	49.29
2	DAD 245.0 nm	20.347	51.667	88.276	50.71

NHC: 



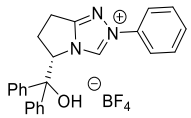
Peak	Processed Channel	Reatention Time (min)	Peak Area (mAU*min)	Peak Height (mAU)	Peak Area (%)
1	DAD 245.0 nm	16.667	42.477	89.717	52.75
2	DAD 245.0 nm	20.213	38.042	64.989	47.25

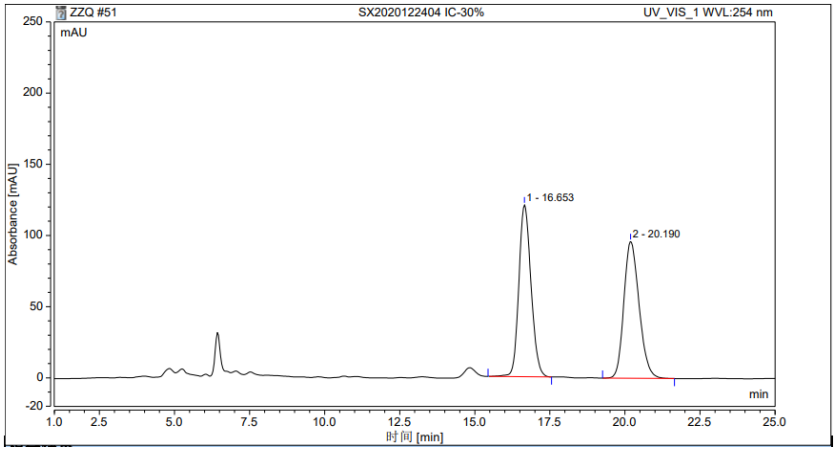
NHC: 



Peak	Processed Channel	Reatention Time (min)	Peak Area (mAU*min)	Peak Height (mAU)	Peak Area (%)
1	DAD 245.0 nm	16.687	60.618	128.036	50.01
2	DAD 245.0 nm	20.217	60.598	102.561	49.99

NHC:





Peak	Processed Channel	Reatention Time (min)	Peak Area (mAU*min)	Peak Height (mAU)	Peak Area (%)
1	DAD 245.0 nm	16.653	56.729	120.883	50.07
2	DAD 245.0 nm	20.190	56.575	96.108	49.93

4 References

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