Electronic Supplementary Information

Palladium-Catalyzed [4+2] Cycloaddition of Amido-Tethered Allylic Carbonates with Oxazol-5-(4*H*)-ones: Synthesis of Piperidine-2,6-dione Derivatives

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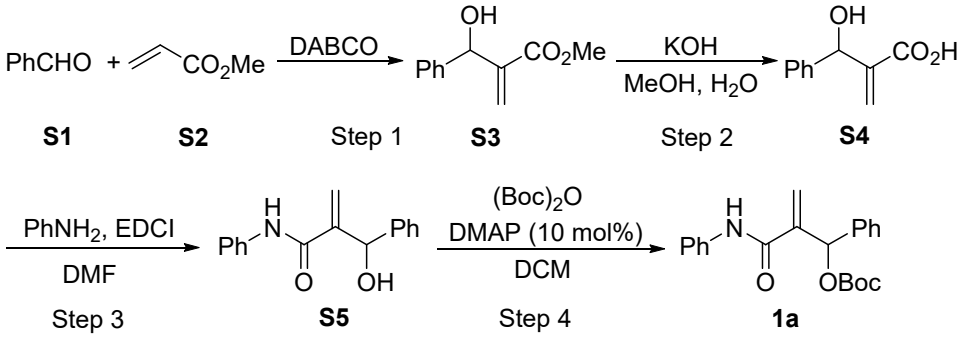
General Information

All reactions were performed under N_2 atmospheres in glassware with magnetic stirring. Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. All solvents were purified and dried according to standard methods prior to use. Organic solutions were concentrated under reduced pressure on a rotary evaporator or an oil pump. Reactions were monitored through thin layer chromatography (TLC) on silica gel–precoated glass plates. Chromatograms were visualized by fluorescence quenching with UV light at 254 nm. Flash column chromatography was performed using Qingdao Haiyang flash silica gel (200-300 mesh). ^1H, ^{13}C NMR spectra were recorded in $CDCl_3$ using a 300MHz or 500MHz NMR instrument (referenced internally to Me_4Si). Chemical shifts (δ, ppm) are relative to tetramethylsilane (TMS) with the resonance of the non-deuterated solvent or TMS as the internal standard. ^1H NMR data are reported as follows: chemical shift, multiplicity (s = singlet; d = doublet; t = triplet; m = multiplet), coupling constant (Hz), and integral. Data for ^{13}C NMR is reported in terms of chemical shift. Optical rotation was obtained on an Autopol V Plus polarimeter. Accurate mass measurements were performed with an Agilent instrument equipped with the ESI-MS technique.

Preparation of Starting Materials

Representative Procedure for the Preparation of tert-butyl (1-phenyl-2-(phenylcarbamoyl)allyl) carbonate[1]



(1) The DABCO (100 mmol, 11.2 g) was dissolved into the mixture of benzaldehyde **S1** (100 mmol, 10.2 mL) and methyl acrylate **S2** (150 mmol, 13.4 mL) and the mixture was stirred at room

[1] (a) H. L. Cui, L. Jiang, H. Tan, S. Liu, *Adv. Synth. Catal.* **2019**, *361*, 4772–4780. (b) S. W. Liu, D. D. Ma, X. X. Zhu, H. L. *Cui. Chem Asian J.* **2020**, *9*, 1617–1622.

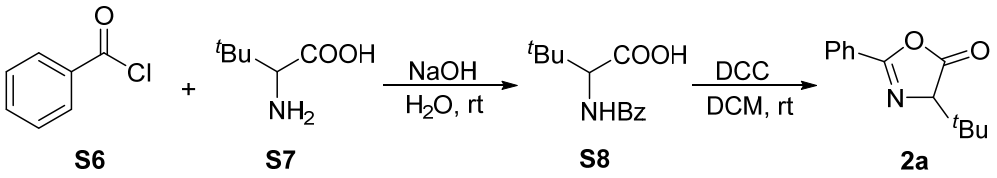
temperature for 3 days or until complete consumption of the starting material as determined by TLC. Then, the product was extracted with EtOAc (50 mL × 3) and the organic layers were combined, washed with saturated brine and dried with Na_2SO_4. The solvent was removed under vacuum and the crude product was purified by column chromatography (Petroleum ether: EtOAc = 5: 1) to give the product methyl 2-(hydroxy(phenyl)methyl)acrylate **S3** as colorless oily liquid (18.94 g, 98.6% yield).

(2) The product methyl 2-(hydroxy(phenyl)methyl)acrylate **S3** (18.94 g, 98.6 mmol) was dissolved into 60 mL of MeOH, then KOH (aqueous) (6.7 g of KOH dissolved into 120 mL of H_2O) was added via dropper. The mixture was stirred at room temperature for 15 hours. After the reaction was complete, the pH of solution was adjusted to 1 with HCl (aqueous). Then, the solution was extracted with EtOAc (50 mL × 3) and the organic layers were combined, washed with saturated brine, dried with Na_2SO_4. The solvent was removed under vacuum to give the product **S4** as colorless oily liquid (17.2 g, 98% yield) without further purification.

(3) The EDCI (35.7 mmol, 6.8 g) was dissolved into the mixture of **S4** (29.8 mmol, 5.3 g) and aniline (35.7 mmol, 3.2 mL) in 60 mL of DMF. The resulting mixture was stirred at room temperature overnight, and then 50 mL of water was added into the mixture. The resulting mixture was extracted with EtOAc. The organic layer was washed with water for three times and dried with Na_2SO_4. The solvent was removed under vacuum. The crude product was purified by flash column (Petroleum ether : EtOAc = 2: 1) to give the product **S5** as white solid (5.3 g, 71% yield).

(4) The product **S5** (21 mmol, 5.3 g) was dissolved into 30 mL of CH_2Cl_2, then the mixture of $(Boc)_2O$ (23.1 mmol, 5.5 mL) and DMAP (2.1 mmol, 258 mg) in 15 mL of CH_2Cl_2 was added into the former solution via dropper. The resulting mixture was stirred at 0 ℃ for 20 min and then was monitored through TLC. Once the reaction was complete, the reaction was quenched with 1N HCl and the reaction mixture was washed with $NaHCO_3$ (aqueous) and the organic layer was dried with Na_2SO_4. After the solvent was removed under vacuum, the crude product was purified by column chromatography (Petroleum ether : EtOAc = 8: 1) to give the final product **1a** as white solid (5.0 g, 66% yield).

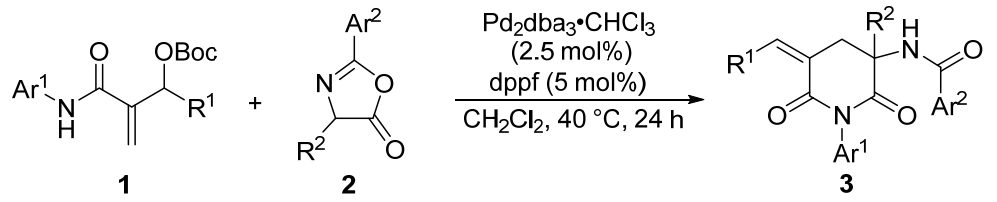
Representative Procedure for the Preparation of 4-(tert-butyl)-2-phenyloxazol-5(4*H*)-one 2a[2]



S6 **S7** **S8** **2a**

The NaOH (3.4 g, 85 mmol) and benzoyl chloride **S6** (4.9 mL, 42 mmol) sequentially were added to the aqueous solution of *tert*-leucine **S7** (5.03 g, 38.3 mmol). This heterogeneous mixture was stirred overnight. Once the reaction mixture became homogeneous, the product amide was then precipitated by the slow addition of aqueous HCl (2 N), isolated by filtration, and dried thoroughly under high vacuum. This chalky white solid was dissolved in CH_2Cl_2 (50 mL), and then 1,3-dicyclohexylcarbodiimide (8.05 g, 39.0 mmol) was added to the mixture. The resulting heterogeneous mixture was stirred at rt overnight and then was filtered. The filtrate was washed with aqueous HCl (2 N; 100 mL), dried with anhydrous Na_2SO_4, and concentrated. The desired product was purified by flash chromatography through a short pad of silica gel (5% ethyl acetate in hexanes), affording final product **2a** as a white solid (6.95 g, 84%, two steps).

General Procedure for Annulation Reaction

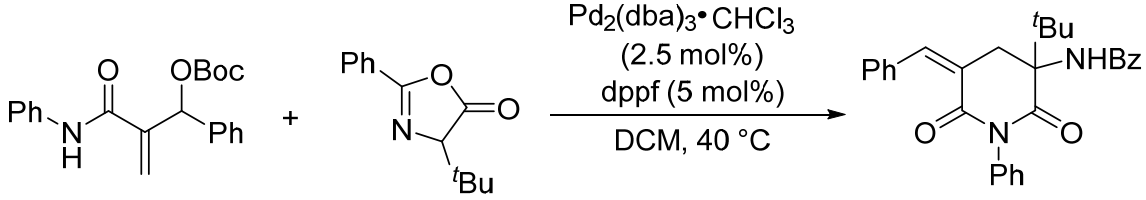


1 **2** **3**

Under a nitrogen atmosphere, an oven-dried 10 mL of Schlenk tube was charged with *tert*-butyl (1-phenyl-2-(phenylcarbamoyl)allyl)carbonate **1** (0.12 mmol), 4-(*tert*-butyl)-2-phenyloxazol-5(4*H*)-one **2** (0.1 mmol), $Pd_2(dba)_3 \cdot CHCl_3$ (2.5 mol%, 2.6 mg), dppf (5 mol%, 2.8 mg) and 1 mL of CH_2Cl_2. The reaction solution was then vigorously stirred at 40 °C. Once the starting material was completely consumed (monitored by TLC), the mixture was concentrated to dryness. The residue was purified by flash column to afford the product **3**.

[2] Forrest O. Arp and Gregory C. Fu. *J. Am. Chem. Soc.* **2006**, 128, 44, 14264–14265

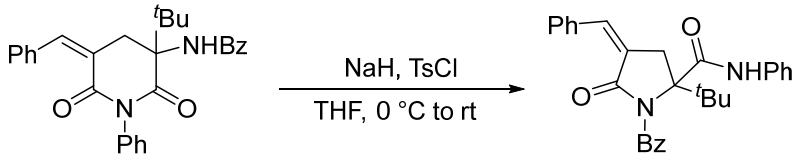
The Gram-scale Annulation Reaction



1a **2a** (0.5 g, 2.5 mmol) **3aa** (0.89 g, 79% yield)

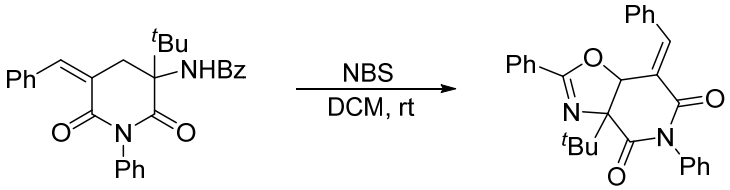
Under a nitrogen atmosphere, an oven-dried 50 mL of Schlenk tube was charged with *tert*-butyl (1-phenyl-2-(phenylcarbamoyl)allyl)carbonate **1a** (1.1 g), 4-(*tert*-butyl)-2-phenyloxazol-5(4*H*)-one **2a** (0.5 g), $Pd_2(dba)_3 \cdot CHCl_3$ (2.5 mol%), dppf (5 mol%) and 25 mL of CH_2Cl_2. The reaction solution was then vigorously stirred at 40 °C. Once the starting material was completely consumed (monitored by TLC), the mixture was concentrated to dryness. The residue was purified by flash column to afford the product **3aa** (0.89 g, 79% yield).

Further Transformations



3aa **4aa**, 75% yield

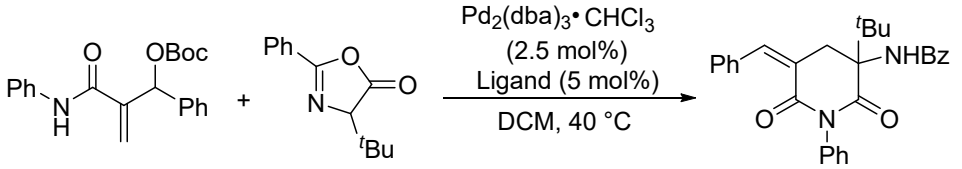
The NaH (1.1 equiv.) was slowly added to the solution of **3aa** (45.2 mg, 0.1 mmol) in 1mL of THF in an oven-dried 10 mL of Schlenk tube at 0 °C. The temperature was allowed to warm to room temperature, and the mixture was further stirred at rt for 30 min. The TsCl (1.5 equiv.) was added and the resulting mixture was stirred for 4 h. Once the starting material was completely consumed (monitored by TLC), the mixture was concentrated to dryness. The residue was purified by flash column to afford the product **4aa** (33.8 mg, 75% yield).



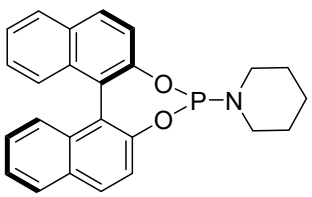
3aa **5aa**, 42% yield

An oven-dried 10 mL of Schlenk tube was charged with **3aa** (45.2 mg, 0.1 mmol), NBS (3.0 equiv.) and 1 mL of CH_2Cl_2. The resulting mixture was stirred at rt for 48 h. Once the starting material was completely consumed (monitored by TLC), the mixture was concentrated to dryness. The residue was purified by flash column to afford the product **5aa** (18.9 mg, 42% yield).

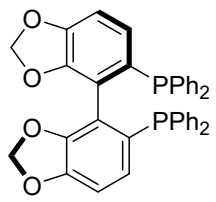
Table S1. Investigation of Asymmetric [4+2] Cycloaddition



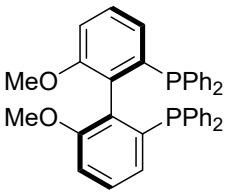
1a **2a** **3aa**



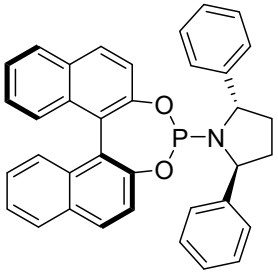
L1, 24 h, trace



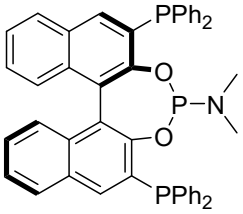
L2, 24 h, trace



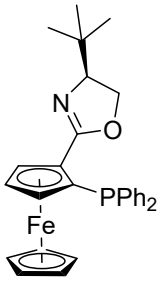
L3, 24 h, trace



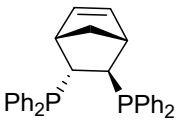
L4, 24 h, trace



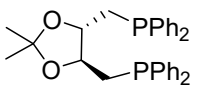
L5, 24 h, trace



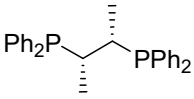
L6, 24 h, trace



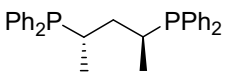
L7, 24 h, trace



L8, 24 h
40% yield, 25% ee



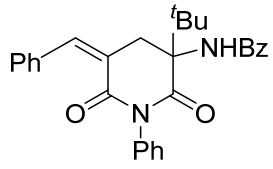
L9, 24 h, trace



L10, 24 h, trace

Characterization Data of All Products

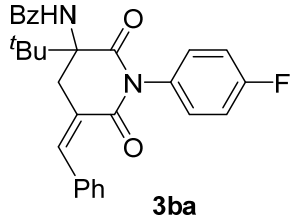
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3aa)



3aa

The title compound **3aa** was prepared according to the general procedure as described above in 86% yield (38.9 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 159 – 161 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.5 Hz, 2H), 7.55 (d, *J* = 6.9 Hz, 2H), 7.51 (d, *J* = 7.4 Hz, 2H), 7.49 – 7.42 (m, 4H), 7.39 (t, *J* = 7.4 Hz, 1H), 7.35 – 7.28 (m, 3H), 7.24 (d, *J* = 7.4 Hz, 2H), 7.08 (s, 1H), 6.86 (s, 1H), 3.75 – 3.65 (m, 2H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.2, 165.8, 163.8, 140.9, 134.9, 133.7, 133.6, 130.8, 128.6, 128.2, 127.8, 127.7, 127.5, 127.4, 127.0, 125.8, 124.9, 63.6, 38.7, 36.9, 25.9. IR (film) ν_{max} 3391, 3057, 2977, 1660, 1517, 1265, 1175, 969, 827, 750, 730, 529 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{28}N_2O_3$ $[M+H]^+$ calcd.: 453.2173, found: 453.2166.

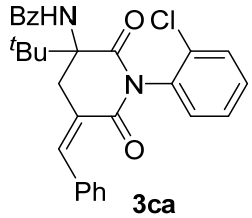
(Z)-N-(5-benzylidene-3-(tert-butyl)-1-(4-fluorophenyl)-2,6-dioxopiperidin-3-yl)benzamide (3ba)



3ba

The title compound **3ba** was prepared according to the general procedure as described above in 84% yield (39.5 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 110 – 112 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 – 7.71 (m, 2H), 7.55 – 7.49 (m, 3H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.35 – 7.28 (m, 3H), 7.22 (dd, *J* = 8.9, 4.9 Hz, 2H), 7.13 (t, *J* = 8.7 Hz, 2H), 7.05 (s, 1H), 6.74 (s, 1H), 3.70 (dd, *J* = 15.1, 2.4 Hz, 1H), 3.53 (d, *J* = 14.8 Hz, 1H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.1, 167.0, 165.0, 162.4 (d, *J* = 247.5 Hz), 141.9, 134.7, 134.5, 131.9, 130.3, 130.3, 129.6, 128.9, 128.8, 128.1, 126.9, 125.9, 116.3 (d, *J* = 22.7 Hz), 64.5, 39.4, 37.9. ^{19}F NMR (471 MHz, $CDCl_3$) δ -114.43; IR (film) ν_{max} 3391, 3057, 2965, 1661, 1507, 1265, 1175, 964, 827, 750, 731, 529 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}FN_2O_3$ $[M+H]^+$ calcd.: 471.2079, found: 471.2076.

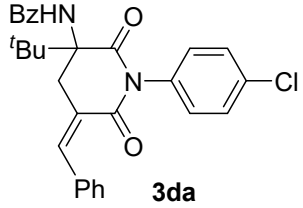
(Z)-N-(5-benzylidene-3-(tert-butyl)-1-(2-chlorophenyl)-2,6-dioxopiperidin-3-yl)benzamide (3ca)



3ca

The title compound **3ca** was prepared according to the general procedure as described above in 52% yield (25.3 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 120 – 122 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 – 7.72 (m, 2H), 7.55 – 7.50 (m, 3H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.36 – 7.29 (m, 3H), 7.23 (dd, *J* = 8.9, 4.9 Hz, 2H), 7.14 (t, *J* = 8.7 Hz, 2H), 7.06 (s, 1H), 6.75 (s, 1H), 3.71 (dd, *J* = 15.1, 2.4 Hz, 1H), 3.53 (d, *J* = 14.8 Hz, 1H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.9, 166.1, 163.9, 140.9, 133.7, 133.49, 133.46, 130.9, 129.0, 128.6, 128.5, 128.0, 127.8, 127.1, 125.9, 124.9, 63.4, 38.4, 37.0, 25.8. IR (film) ν_{max} 3395, 3050, 2975, 1661, 1507, 1265, 1176, 964, 827, 750, 731, 530 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}ClN_2O_3$ $[M+H]^+$ calcd.: 487.1783, found: 487.1782.

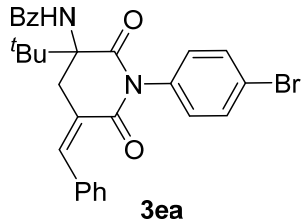
(Z)-N-(5-benzylidene-3-(tert-butyl)-1-(4-chlorophenyl)-2,6-dioxopiperidin-3-yl)benzamide (3da)



3da

The title compound **3da** was prepared according to the general procedure as described above in 75% yield (36.5 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 187 – 189 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.0 Hz, 2H), 7.52 (m, 3H), 7.47 – 7.39 (m, 4H), 7.35 – 7.27 (m, 3H), 7.19 (d, *J* = 8.6 Hz, 2H), 7.05 (s, 1H), 6.72 (s, 1H), 3.69 (dd, *J* = 15.1, 2.3 Hz, 1H), 3.50 (d, *J* = 15.9 Hz, 1H), 1.32 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.9, 167.1, 164.9, 141.9, 134.7, 134.5, 134.4, 131.9, 130.0, 129.6, 129.5, 128.9, 128.8, 128.1, 126.9, 125.9, 64.4, 39.4, 38.0, 26.8. IR (film) ν_{max} 3392, 3056, 2966, 1663, 1489, 1264, 1175, 964, 765, 750, 746, 731 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}ClN_2O_3$ $[M+H]^+$ calcd.: 487.1783, found: 487.1785.

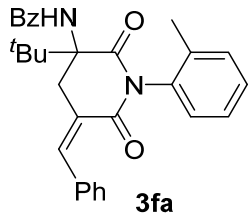
(Z)-N-(5-benzylidene-1-(4-bromophenyl)-3-(tert-butyl)-2,6-dioxopiperidin-3-yl)benzamide (3ea)



3ea

The title compound **3ea** was prepared according to the general procedure as described above in 70% yield (37.1 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 139 – 141 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 – 7.71 (m, 2H), 7.57 (d, *J* = 8.7 Hz, 2H), 7.54 – 7.50 (m, 3H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.30 (dd, *J* = 9.9, 7.1 Hz, 3H), 7.13 (d, *J* = 8.5 Hz, 2H), 7.05 (s, 1H), 6.72 (s, 1H), 3.69 (dd, *J* = 15.1, 2.4 Hz, 1H), 3.50 (d, *J* = 15.2 Hz, 1H), 1.32 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.8, 167.1, 164.8, 141.9, 135.0, 134.7, 134.4, 132.5, 131.9, 130.3, 129.6, 128.9, 128.8, 128.1, 126.9, 125.9, 122.6, 64.4, 39.4, 38.0, 26.8. IR (film) ν_{max} 3307, 2981, 1742, 1488, 1251, 1153, 1072, 823, 765, 750, 735, 506 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}BrN_2O_3$ $[M+H]^+$ calcd.: 531.1278, found: 531.1276.

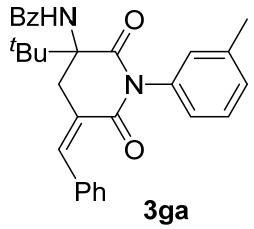
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-(o-tolyl)piperidin-3-yl)benzamide (3fa)



3fa

The title compound **3fa** was prepared according to the general procedure as described above in 79% yield (36.8 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 200 – 202 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 – 7.74 (m, 2H), 7.56 (d, *J* = 6.5 Hz, 2H), 7.54 – 7.49 (m, 1H), 7.44 (t, *J* = 7.7 Hz, 1H), 7.33 – 7.27 (m, 4H), 7.11 (d, *J* = 8.3 Hz, 2H), 7.08 (s, 1H), 6.89 (s, 1H), 3.82 – 3.61 (m, 2H), 2.37 (s, 3H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3,165.8, 163.8, 140.9, 138.2, 134.7, 133.73, 133.68, 130.7, 128.7, 128.4, 128.1, 127.9, 127.8, 127.7, 127.0, 125.8, 124.9, 124.3, 63.7, 38.8, 36.9, 26.0, 20.3. IR (film) ν_{max} 3370, 2962, 1656, 1510, 1486, 1464, 1176, 961, 758, 750, 746, 730 cm^{-1}; HRMS (ESI): m/z for $C_{30}H_{29}N_2O_3$ $[M+H]^+$ calcd.: 467.2329, found: 467.2322.

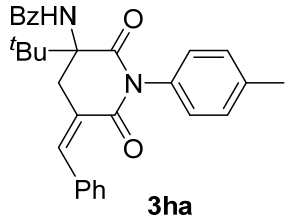
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-(m-tolyl)piperidin-3-yl)benzamide (3ga)



3ga

The title compound **3ga** was prepared according to the general procedure as described above in 78% yield (36.3 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 160 – 162 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 – 7.74 (m, 2H), 7.55 (d, *J* = 6.7 Hz, 2H), 7.53 – 7.49 (m, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.37 – 7.26 (m, 5H), 7.19 (d, *J* = 7.7 Hz, 1H), 7.08 (s, 1H), 7.02 (d, *J* = 6.9 Hz, 2H), 6.88 (s, 1H), 3.70 (t, *J* = 2.2 Hz, 2H), 2.37 (s, 3H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 165.8, 163.8, 141.0, 138.2, 134.7, 133.74, 133.69, 130.8, 128.7, 128.5, 128.1, 127.9, 127.8, 127.7, 127.0, 125.9, 124.9, 124.3, 63.7, 38.8, 36.9, 26.0, 20.3. IR (film) ν_{max} 3371, 2962, 1660, 1510, 1486, 1464, 1176, 961, 758, 750, 736 cm^{-1}; HRMS (ESI): m/z for $C_{30}H_{29}N_2O_3$ $[M+H]^+$ calcd.: 467.2329, found: 467.2326.

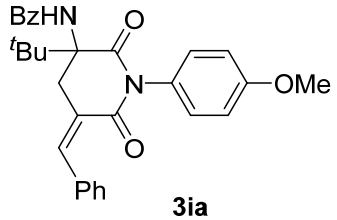
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-(p-tolyl)piperidin-3-yl)benzamide (3ha)



3ha

The title compound **3ha** was prepared according to the general procedure as described above in 80% yield (37.3 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc=5:1) to afford white solid. mp = 161 – 163 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 – 7.73 (m, 2H), 7.55 (d, *J* = 6.5 Hz, 2H), 7.53 – 7.49 (m, 1H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.33 – 7.26 (m, 4H), 7.10 (d, *J* = 8.3 Hz, 2H), 7.07 (s, 1H), 6.88 (s, 1H), 3.75 – 3.62 (m, 2H), 2.36 (s, 3H), 1.32 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.4, 166.8, 165.0, 141.9, 138.5, 134.8, 133.2, 131.8, 130.0, 129.8, 128.9, 128.8, 128.1, 128.0, 126.9, 126.0, 64.7, 39.9, 38.0, 27.0, 21.2. IR (film) ν_{max} 3369, 2966, 1727, 1666, 1486, 1264, 1178, 963, 765, 757, 753, 751, 751, 750, 749, 746, 731 cm^{-1}; HRMS (ESI): m/z for $C_{30}H_{29}N_2O_3$ $[M+H]^+$ calcd.: 467.2329, found: 467.2322.

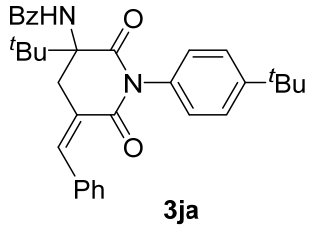
(Z)-N-(5-benzylidene-3-(tert-butyl)-1-(4-methoxyphenyl)-2,6-dioxopiperidin-3-yl)benzamide (3ia)



3ia

The title compound **3ia** was prepared according to the general procedure as described above in 76% yield (36.6 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 135 – 137 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 (d, J = 8.8 Hz, 2H), 7.55 (d, J = 6.7 Hz, 2H), 7.47 (t, J = 7.6 Hz, 2H), 7.39 (t, J = 7.5 Hz, 1H), 7.34 – 7.28 (m, 3H), 7.24 (d, J = 7.5 Hz, 2H), 7.07 (s, 1H), 6.93 (d, J = 8.8 Hz, 2H), 6.77 (s, 1H), 3.85 (s, 3H), 3.73 – 3.63 (m, 2H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.5, 165.8, 164.1, 158.4, 140.8, 133.74, 133.67, 130.8, 128.7, 128.3, 127.8, 127.7, 127.4, 127.0, 125.9, 125.0, 113.6, 63.6, 54.4, 38.7, 36.9, 25.9. IR (film) ν_{max} 3392, 3056, 2964, 2838, 1663, 1509, 1246, 1177, 1030, 963, 825 cm^{-1}; HRMS (ESI): m/z for $C_{30}H_{29}N_2O_4$ $[M+H]^+$ calcd.: 483.2278, found: 483.2277.

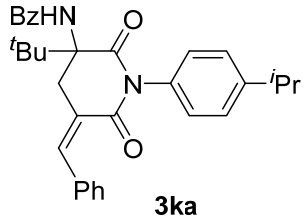
(Z)-N-(5-benzylidene-3-(tert-butyl)-1-(4-(tert-butyl)phenyl)-2,6-dioxopiperidin-3-yl)benzamide (3ja)



3ja

The title compound **3ja** was prepared according to the general procedure as described above in 92% yield (46.7 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 230 – 232 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.0 Hz, 2H), 7.45 (dd, *J* = 20.1, 7.2 Hz, 3H), 7.41 – 7.33 (m, 4H), 7.27 – 7.19 (m, 3H), 7.07 (d, *J* = 8.5 Hz, 2H), 7.01 (s, 1H), 6.83 (s, 1H), 3.68 – 3.60 (m, 2H), 1.26 (s, 9H), 1.24 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.6, 166.8, 164.9, 151.2, 142.0, 134.9, 134.7, 133.1, 131.8, 129.7, 128.8, 128.7, 128.04, 128.02, 127.7, 126.9, 126.4, 126.0, 64.7, 39.9, 37.9, 34.7, 31.4, 27.0. IR (film) ν_{max} 3400, 3056, 2965, 1667, 1511, 1264, 1178, 963, 746, 563 cm^{-1}; HRMS (ESI): m/z for $C_{33}H_{36}N_2O_3$ $[M+H]^+$ calcd.: 509.2799, found: 509.2795.

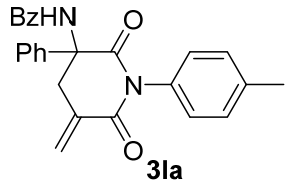
(Z)-N-(5-benzylidene-3-(tert-butyl)-1-(4-isopropylphenyl)-2,6-dioxopiperidin-3-yl)benzamide (3ka)



3ka

The title compound **3ka** was prepared according to the general procedure as described above in 95% yield (47.0 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 125 – 127 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 – 7.74 (m, 2H), 7.53 (dd, *J* = 20.3, 7.1 Hz, 3H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.35 – 7.28 (m, 5H), 7.17 – 7.13 (m, 2H), 7.09 (s, 1H), 6.91 (s, 1H), 3.80 – 3.61 (m, 2H), 2.98 – 2.89 (m, 1H), 1.34 (s, 9H), 1.25 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.6, 166.8, 164.9, 149.0, 142.0, 134.8, 134.7, 133.4, 131.8, 129.7, 128.8, 128.7, 128.1, 128.0, 127.4, 126.9, 126.0, 64.7, 39.9, 37.9, 33.9, 27.0, 23.93, 23.90. IR (film) ν_{max} 3392, 3057, 2962, 1663, 1510, 1265, 1176, 963, 823, 765, 731 cm^{-1}; HRMS (ESI): m/z for $C_{32}H_{34}N_2O_3$ $[M+H]^+$ calcd.: 495.2642, found: 495.2639.

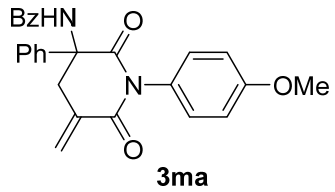
N-(5-methylene-2,6-dioxo-3-phenyl-1-(p-tolyl)piperidin-3-yl)benzamide (3la)



3la

The title compound **3la** was prepared according to the general procedure as described above in 86% yield (35.3 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford semi-oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 – 7.71 (m, 2H), 7.57 – 7.49 (m, 1H), 7.47 – 7.41 (m, 2H), 7.28 (d, *J* = 7.5 Hz, 2H), 7.10 (d, *J* = 8.3 Hz, 2H), 6.92 (s, 1H), 6.45 (dd, *J* = 2.9, 1.3 Hz, 1H), 5.75 (dd, *J* = 2.8, 1.3 Hz, 1H), 3.74 (d, *J* = 16.2 Hz, 1H), 3.62 (dt, *J* = 16.3, 2.9 Hz, 1H), 2.39 (s, 3H), 1.24 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 165.8, 164.1, 137.5, 134.3, 133.6, 132.1, 130.8, 129.0, 127.7, 126.9, 125.8, 124.8, 63.4, 38.9, 32.6, 25.8, 20.2. IR (film) ν_{max} 3390, 3061, 2962, 2924, 1728, 1684, 1511, 1403, 1364, 702, 570 cm^{-1}. HRMS (ESI): m/z for $C_{26}H_{22}N_2O_3$ $[M+H]^+$ calcd.: 404.1429, found: 404.1427.

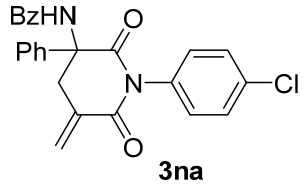
N-(1-(4-methoxyphenyl)-5-methylene-2,6-dioxo-3-phenylpiperidin-3-yl)benzamide (3ma)



3ma

The title compound **3ma** was prepared according to the general procedure as described above in 81% yield (35.5 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford semi-oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.55 – 7.48 (m, 1H), 7.44 (t, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 8.8 Hz, 2H), 6.99 (d, *J* = 9.1 Hz, 2H), 6.90 (s, 1H), 6.44 (dd, *J* = 2.8, 1.3 Hz, 1H), 5.75 (dd, *J* = 2.6, 1.1 Hz, 1H), 3.82 (s, 3H), 3.70 (d, *J* = 16.2 Hz, 1H), 3.61 (dt, *J* = 16.2, 2.8 Hz, 1H), 1.24 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.4, 165.8, 164.2, 158.5, 134.3, 133.6, 130.8, 128.2, 127.7, 127.3, 125.8, 124.8, 113.6, 63.4, 54.4, 38.8, 32.6, 25.8. IR (film) ν_{max} 3392, 3061, 2965, 2924, 1730, 1684, 1511, 1403, 1366, 1189, 801 cm^{-1}. HRMS (ESI): m/z for $C_{26}H_{22}N_2O_4$ $[M+H]^+$ calcd.: 404.1429, found: 404.1427.

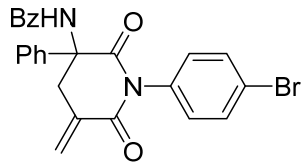
N-(1-(4-chlorophenyl)-5-methylene-2,6-dioxo-3-phenylpiperidin-3-yl)benzamide (3na)



3na

The title compound **3na** was prepared according to the general procedure as described above in 64% yield (27.5 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford semi-oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 – 7.72 (m, 2H), 7.55 – 7.50 (m, 1H), 7.47 – 7.41 (m, 4H), 7.18 (d, *J* = 8.6 Hz, 2H), 6.44 (dd, *J* = 2.7, 1.2 Hz, 1H), 5.89 – 5.61 (m, 1H), 3.67 (dt, *J* = 16.2, 2.8 Hz, 1H), 3.53 (d, *J* = 16.2 Hz, 1H), 1.24 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.8, 167.1, 164.9, 135.2, 134.5, 134.4, 134.3, 132.0, 129.8, 129.5, 128.8, 126.9, 125.9, 64.2, 39.4, 33.6, 26.7. IR (film) ν_{max} 3389, 3061, 2963, 2924, 1729, 1684, 1511, 1403, 1360, 1189, 801 cm^{-1}; HRMS (ESI): m/z for $C_{25}H_{19}ClN_2O_3$ $[M+H]^+$ calcd.: 404.1429, found: 404.1427.

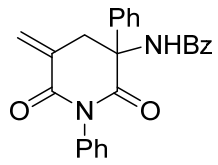
N-(1-(4-bromophenyl)-5-methylene-2,6-dioxo-3-phenylpiperidin-3-yl)benzamide (3oa)



3oa

The title compound **3oa** was prepared according to the general procedure as described above in 56% yield (26.5 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford semi-oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 – 7.71 (m, 2H), 7.60 (d, *J* = 8.7 Hz, 2H), 7.56 – 7.49 (m, 1H), 7.44 (t, *J* = 7.7 Hz, 1H), 7.12 (d, *J* = 8.6 Hz, 2H), 6.75 (s, 1H), 6.43 (dd, *J* = 2.7, 1.2 Hz, 1H), 5.75 – 5.73 (m, 1H), 3.67 (dt, *J* = 16.2, 2.9 Hz, 1H), 3.52 (d, *J* = 16.3 Hz, 1H), 1.24 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.7, 167.1, 164.9, 135.2, 134.9, 134.4, 132.5, 132.0, 130.2, 128.8, 126.9, 125.9, 122.6, 64.2, 39.4, 33.6, 26.7. IR (film) ν_{max} 3390, 3062, 2959, 2924, 1729, 1683, 1511, 1403, 1361, 1189, 801 cm^{-1}. HRMS (ESI): m/z for $C_{25}H_{19}BrN_2O_3$ $[M+H]^+$ calcd.: 404.1429, found: 404.1427.

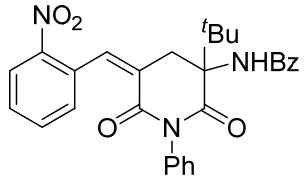
N-(5-methylene-2,6-dioxo-1,3-diphenylpiperidin-3-yl)benzamide (3pa)



3pa

The title compound **3pa** was prepared according to the general procedure as described above in 92% yield (46.7 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford semi-oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 – 7.73 (m, 2H), 7.54 – 7.46 (m, 3H), 7.46 – 7.39 (m, 3H), 7.22 (d, *J* = 7.2 Hz, 2H), 6.89 (s, 1H), 6.45 (dd, *J* = 2.5, 1.2 Hz, 1H), 5.76 (dd, *J* = 2.6, 1.3 Hz, 1H), 3.87 – 3.56 (m, 2H), 1.25 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.9, 164.0, 134.8, 134.3, 133.6, 130.8, 128.3, 127.7, 127.6, 127.2, 125.9, 124.8, 63.4, 32.6, 25.8. IR (film) ν_{max} 3391, 3063, 2961, 2924, 1729, 1683, 1511, 1486, 1272, 1189, 949, 801 cm^{-1}; HRMS (ESI): m/z for $C_{25}H_{20}N_2O_3$ $[M+H]^+$ calcd.: 404.1429, found: 404.1427.

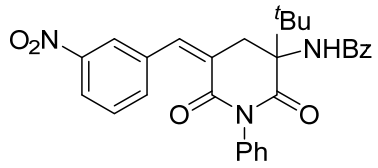
(Z)-N-(3-(tert-butyl)-5-(2-nitrobenzylidene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3qa)



3qa

The title compound **3qa** was prepared according to the general procedure as described above in 65% yield (32.3 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 183 – 185 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.1 Hz, 2H), 7.67 (d, *J* = 8.2 Hz, 2H), 7.51 (d, *J* = 6.5 Hz, 2H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.36 (t, *J* = 7.4 Hz, 1H), 7.32 – 7.26 (m, 3H), 7.19 (d, *J* = 7.4 Hz, 2H), 7.07 (s, 1H), 6.87 (s, 1H), 3.87 – 3.51 (m, 2H), 1.31 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.6, 166.8, 164.9, 151.2, 142.0, 134.9, 134.7, 133.1, 131.8, 129.7, 128.8, 128.7, 128.04, 128.02, 127.7, 126.9, 126.4, 126.0, 64.7, 39.9, 37.9, 34.7, 31.4, 27.0. IR (film) ν_{max} 3323, 3064, 2926, 1681, 1596, 1517, 1343, 1181, 797, 752 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}N_3O_5$ $[M+H]^+$ calcd.: 498.2024, found: 498.2019.

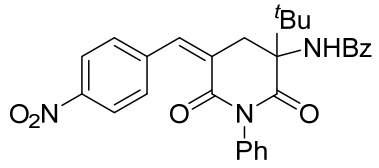
(Z)-N-(3-(tert-butyl)-5-(3-nitrobenzylidene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3ra)



3ra

The title compound **3ra** was prepared according to the general procedure as described above in 81% yield (40.2 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 183 – 185 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.1 Hz, 2H), 7.71 (d, *J* = 8.2 Hz, 2H), 7.55 (d, *J* = 6.5 Hz, 2H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.40 (t, *J* = 7.4 Hz, 1H), 7.36 – 7.29 (m, 3H), 7.22 (d, *J* = 7.4 Hz, 2H), 7.11 (s, 1H), 6.91 (s, 1H), 3.76 – 3.65 (m, 2H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.8, 166.0, 163.7, 140.9, 134.0, 133.6, 133.4, 131.4, 130.9, 129.3, 128.6, 127.9, 127.8, 127.0, 125.8, 124.8, 121.5, 63.4, 38.3, 36.9, 25.8. IR (film) ν_{max} 3305, 3067, 2925, 1746, 1674, 1593, 1527, 1349, 1254, 1156, 1014, 758 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}N_3O_5$ $[M+H]^+$ calcd.: 498.2024, found: 498.2022.

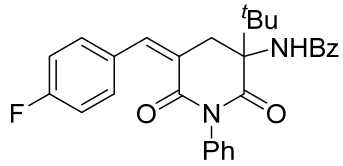
(Z)-N-(3-(tert-butyl)-5-(4-nitrobenzylidene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3sa)



3sa

The title compound **3sa** was prepared according to the general procedure as described above in 94% yield (46.7 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 185 – 187 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.4 Hz, 2H), 7.74 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 6.2 Hz, 2H), 7.47 (t, *J* = 7.6 Hz, 3H), 7.43 – 7.38 (m, 1H), 7.36 – 7.29 (m, 3H), 7.21 (d, *J* = 7.2 Hz, 2H), 7.11 (s, 1H), 6.91 (s, 1H), 3.80 – 3.60 (m, 2H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.0, 167.2, 165.0, 142.1, 134.9, 134.64, 134.60, 132.1, 130.2, 129.8, 129.7, 129.1, 129.0, 128.2, 127.1, 126.1, 64.6, 39.6, 38.1, 27.0. IR (film) ν_{max} 3389, 3063, 2962, 1683, 1596, 1343, 1259, 1106, 963, 799 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}N_3O_5$ $[M+H]^+$ calcd.: 498.2024, found: 498.2020.

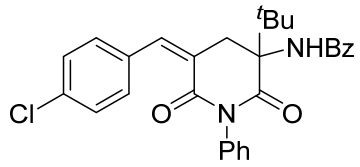
(Z)-N-(3-(tert-butyl)-5-(4-fluorobenzylidene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3ta)



3ta

The title compound **3ta** was prepared according to the general procedure as described above in 85% yield (40.0 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 135 – 137 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 – 7.70 (m, 2H), 7.57 – 7.49 (m, 3H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.34 – 7.28 (m, 3H), 7.24 – 7.20 (m, 2H), 7.13 (t, *J* = 8.7 Hz, 2H), 7.05 (s, 1H), 6.74 (s, 1H), 3.70 (dd, *J* = 15.1, 2.4 Hz, 1H), 3.53 (d, *J* = 14.8 Hz, 1H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.0, 166.0, 163.9, 161.3 (d, *J* = 247.5 Hz), 140.8, 133.6 (d, *J* = 30.5 Hz), 130.9, 129.3, 129.2, 128.5, 127.9, 127.7, 127.0, 125.8, 124.9, 115.2 (d, *J* = 22.7 Hz), 63.4, 38.4, 36.9, 25.8. ^{19}F NMR (471 MHz, $CDCl_3$) δ -112.32.IR (film) ν_{max} 3391, 3057, 2965, 1728, 1682, 1507, 1265, 964, 827, 758 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}FN_2O_3$ $[M+H]^+$ calcd.: 471.2079, found: 471.2062.

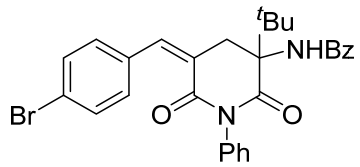
(Z)-N-(3-(tert-butyl)-5-(4-chlorobenzylidene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3ua)



3ua

The title compound **3ua** was prepared according to the general procedure as described above in 81% yield (39.4 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 135 – 137 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.1 Hz, 2H), 7.52 – 7.42 (m, 7H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 8.6 Hz, 2H), 7.22 (d, *J* = 7.4 Hz, 2H), 3.78 – 3.56 (m, 2H), 1.32 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.8, 166.0, 163.8, 140.9, 133.6, 133.4, 133.4, 130.9, 129.0, 128.5, 128.4, 127.9, 127.8, 127.0, 125.8, 124.8, 63.4, 38.3, 36.9, 25.8. IR (film) ν_{max} 3341, 3055, 2965, 1726, 1677, 1488, 1264, 1185, 955, 764 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}ClN_2O_3$ $[M+H]^+$ calcd.: 487.1783, found: 487.1785.

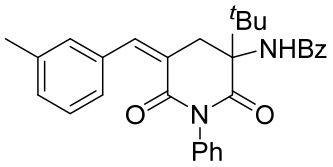
(Z)-N-(5-(4-bromobenzylidene)-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3va)



3va

The title compound **3va** was prepared according to the general procedure as described above in 80% yield (42.4 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 138 – 140 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 – 7.65 (m, 2H), 7.47 – 7.42 (m, 1H), 7.41 – 7.30 (m, 9H), 7.15 (dd, *J* = 7.3, 1.5 Hz, 2H), 6.89 (s, 1H), 6.73 (s, 1H), 3.67 – 3.22 (m, 2H), 1.25 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.0, 165.9, 163.9, 139.2, 134.8, 133.5, 132.7, 130.8, 130.2, 128.3, 127.74, 127.72, 127.6, 127.4, 125.9, 125.8, 122.0, 63.5, 38.7, 37.0, 25.8. IR (film) ν_{max} 3391, 3055, 2966, 1729, 1683, 1663, 1485, 1264, 1176, 1011, 754 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}BrN_2O_3$ $[M+H]^+$ calcd.: 531.1278, found: 531.1281.

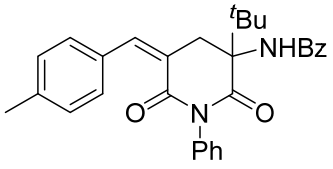
(Z)-N-(3-(tert-butyl)-5-(3-methylbenzylidene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3wa)



3wa

The title compound **3wa** was prepared according to the general procedure as described above in 92% yield (46.7 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 103 – 105 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.70 – 7.66 (m, 2H), 7.47 – 7.42 (m, 1H), 7.41 – 7.34 (m, 4H), 7.34 – 7.29 (m, 2H), 7.22 (s, 1H), 7.17 – 7.11 (m, 3H), 7.03 (d, *J* = 7.5 Hz, 1H), 6.99 (s, 1H), 6.79 (s, 1H), 3.78 – 3.46 (m, 2H), 2.26 (s, 3H), 1.26 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 165.8, 163.8, 141.2, 136.5, 134.9, 133.7, 133.6, 130.8, 129.2, 128.6, 128.2, 127.7, 127.5, 127.4, 126.9, 125.9, 125.7, 124.7, 63.7, 38.8, 36.8, 26.0, 20.4. IR (film) ν_{max} 3309, 2981, 2933, 1742, 1598, 1533, 1442, 1251, 1153, 1081, 792 cm^{-1}; HRMS (ESI): m/z for $C_{30}H_{30}N_2O_3$ $[M+H]^+$ calcd.: 467.2329, found: 467.2326.

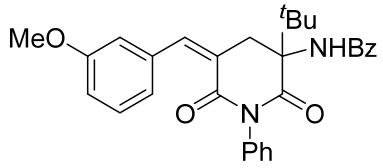
(Z)-N-(3-(tert-butyl)-5-(4-methylbenzylidene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3xa)



3xa

The title compound **3xa** was prepared according to the general procedure as described above in 74% yield (34.5 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 140 – 142 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 – 7.66 (m, 2H), 7.44 – 7.39 (m, 3H), 7.36 (dd, *J* = 15.0, 7.0 Hz, 4H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.15 (d, *J* = 7.3 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 6.98 (s, 1H), 6.79 (s, 1H), 3.67 – 3.56 (m, 2H), 2.25 (s, 3H), 1.25 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 165.8, 163.7, 141.2, 136.5, 134.9, 133.7, 133.6, 130.8, 129.2, 128.6, 128.2, 127.7, 127.5, 127.4, 126.9, 125.8, 125.7, 124.7, 63.6, 38.7, 36.8, 26.0, 20.4. IR (film) ν_{max} 3310, 2983, 2933, 1742, 1598, 1535, 1442, 1251, 1153, 755 cm^{-1}; HRMS (ESI): m/z for $C_{30}H_{30}N_2O_3$ $[M+H]^+$ calcd.: 467.2329, found: 467.2329.

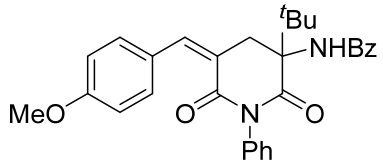
(Z)-N-(3-(tert-butyl)-5-(3-methoxybenzylidene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3ya)



3ya

The title compound **3ya** was prepared according to the general procedure as described above in 94% yield (45.3 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 103 – 105 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 – 7.73 (m, 2H), 7.54 – 7.49 (m, 1H), 7.47 – 7.41 (m, 4H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.23 (t, *J* = 7.8 Hz, 3H), 7.13 – 7.07 (m, 1H), 7.04 (s, 1H), 6.85 – 6.81 (m, 2H), 3.78 (s, 3H), 3.68 (dd, *J* = 15.1, 2.3 Hz, 1H), 3.64 (dd, *J* = 15.1, 1.2 Hz, 1H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.2, 166.9, 164.8, 159.2, 141.4, 136.2, 136.0, 134.6, 131.9, 129.3, 129.1, 128.8, 128.6, 128.5, 126.9, 126.5, 122.0, 115.0, 114.4, 64.7, 55.3, 39.7, 37.9, 26.9. IR (film) ν_{max} 3369, 3059, 2962, 1729, 1682, 1578, 1373, 1264, 1175, 906, 788 cm^{-1}; HRMS (ESI): m/z for $C_{30}H_{30}N_2O_4$ $[M+H]^+$ calcd.: 483.2278, found: 483.2277.

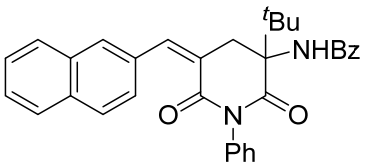
(Z)-N-(3-(tert-butyl)-5-(4-methoxybenzylidene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3za)



3za

The title compound **3za** was prepared according to the general procedure as described above in 97% yield (46.7 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 105 – 107 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 – 7.73 (m, 2H), 7.54 – 7.49 (m, 1H), 7.48 – 7.42 (m, 4H), 7.41 – 7.36 (m, 1H), 7.24 (t, *J* = 7.8 Hz, 3H), 7.13 – 7.08 (m, 2H), 7.04 (s, 1H), 6.84 (d, *J* = 8.1 Hz, 2H), 3.78 (s, 3H), 3.69 (dd, *J* = 15.1, 2.3 Hz, 1H), 3.64 (dd, *J* = 15.1, 1.2 Hz, 1H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.2, 166.9, 164.8, 159.2, 141.4, 136.2, 136.0, 134.6, 131.8, 129.3, 129.1, 128.8, 128.52, 128.47, 126.9, 126.5, 122.0, 115.0, 114.4, 64.7, 55.3, 39.7, 37.9, 26.9. IR (film) ν_{max} 3389, 3063, 2959, 1678, 1602, 1511, 1303, 1254, 1174, 1029, 760 cm^{-1}; HRMS (ESI): m/z for $C_{30}H_{30}N_2O_4$ $[M+H]^+$ calcd.: 483.2278, found: 483.2276.

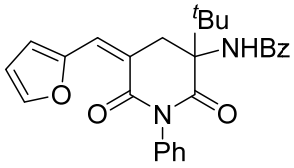
(Z)-N-(3-(tert-butyl)-5-(naphthalen-2-ylmethylene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3z1a)



3z1a

The title compound **3z1a** was prepared according to the general procedure as described above in 71% yield (35.6 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 185 – 187 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 (s, 1H), 7.76 – 7.71 (m, 1H), 7.69 (td, *J* = 8.4, 4.3 Hz, 4H), 7.61 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.44 (t, *J* = 7.4 Hz, 1H), 7.42 – 7.33 (m, 6H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.19 (d, *J* = 1.5 Hz, 1H), 7.16 (d, *J* = 7.6 Hz, 2H), 6.79 (s, 1H), 3.72 – 3.58 (m, 2H), 1.28 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 165.9, 163.9, 140.9, 134.9, 133.6, 132.3, 131.9, 131.4, 130.8, 128.7, 128.3, 127.7, 127.5, 127.5, 127.4, 126.6, 126.4, 125.93, 125.86, 125.7, 125.1, 63.6, 38.7, 37.0, 26.0. IR (film) ν_{max} 3399,3060, 2962, 2840, 1728,1681, 1651, 1606, 1346, 1028, 962 cm^{-1}; HRMS (ESI): m/z for $C_{33}H_{30}N_2O_3$ $[M+H]^+$ calcd.: 503.2329, found: 503.2328.

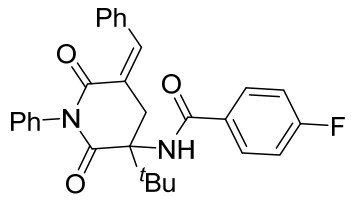
(Z)-N-(3-(tert-butyl)-5-(furan-2-ylmethylene)-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3z2a)



3z2a

The title compound **3z2a** was prepared according to the general procedure as described above in 69% yield (30.5 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford semi-oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 – 7.76 (m, 2H), 7.65 – 7.61 (m, 2H), 7.54 – 7.38 (m, 7H), 7.24 (s, 1H), 6.96 (s, 1H), 6.81 (d, *J* = 3.5 Hz, 1H), 6.56 (dd, *J* = 3.6, 1.8 Hz, 1H), 4.51 (d, *J* = 17.4 Hz, 1H), 3.67 (dd, *J* = 17.4, 3.1 Hz, 1H), 1.23 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.9, 167.0, 166.2, 151.5, 145.3, 136.3, 134.8, 131.8, 129.3, 128.73, 128.72, 128.5, 128.4, 126.9, 125.3, 123.0, 117.7, 112.6, 64.5, 39.9, 29.6, 26.8.IR (film) ν_{max} 3390,3063, 2962, 2840, 1728,1681, 1651, 1624, 1346, 1173, 1028, 969 cm^{-1}; HRMS (ESI): m/z for $C_{27}H_{26}N_2O_4$ $[M+H]^+$ calcd.: 443.1965, found: 443.1963.

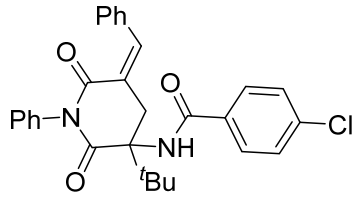
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-4-fluorobenzamide (3ab)



3ab

The title compound **3ab** was prepared according to the general procedure as described above in 85% yield (40.0 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 112 – 114 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (dd, *J* = 8.8, 5.2 Hz, 2H), 7.47 (d, *J* = 6.5 Hz, 2H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 7.27 – 7.22 (m, 3H), 7.15 (d, *J* = 7.2 Hz, 2H), 7.08 – 7.00 (m, 3H), 6.73 (s, 1H), 3.62 (d, *J* = 1.7 Hz, 2H), 1.26 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.8, 166.8, 164.7, 162.4 (d, *J* = 247.5 Hz), 141.6, 134.5, 134.2, 131.7, 131.5, 131.5, 130.1, 130.0, 129.3, 128.7, 128.6, 127.8, 126.6, 125.7, 116.0 (d, *J* = 22.7 Hz), 64.2, 39.2, 37.7, 26.6. ^{19}F NMR (471 MHz, $CDCl_3$) δ -113.99. IR (film) ν_{max} 3367, 3060, 2962, 1728, 1655, 1523, 1491, 1374, 1177, 962, 733 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}FN_2O_3$ $[M+H]^+$ calcd.:471.2079, found: 471.2080.

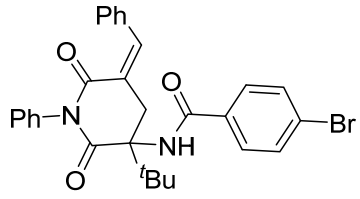
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-4-chlorobenzamide (3ac)



3ac

The title compound **3ac** was prepared according to the general procedure as described above in 86% yield (21.0 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford semi-oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.9 Hz, 2H), 7.77 – 7.72 (m, 2H), 7.59 (d, *J* = 8.8 Hz, 2H), 7.52 (d, *J* = 7.5 Hz, 1H), 7.49 – 7.42 (m, 4H), 7.41 – 7.36 (m, 1H), 7.22 (d, *J* = 7.1 Hz, 2H), 7.04 (s, 1H), 6.76 (s, 1H), 3.61 (dd, *J* = 6.9, 1.8 Hz, 2H), 1.34 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.8, 166.0, 163.8, 140.9, 133.6, 133.42, 133.39, 130.9, 129.0, 128.5, 128.4, 127.9, 127.8, 127.0, 125.8, 124.8, 63.4, 38.3, 36.9, 25.8. IR (film) ν_{max} 3366, 3065, 2962, 1728, 1650, 1523, 1491, 1374, 1346, 1177, 962, 848, 755 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}ClN_2O_3$ $[M+H]^+$ calcd.: 487.1783, found: 487.1768.

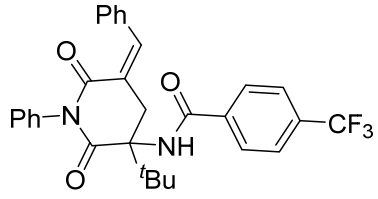
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-4-bromobenzamide (3ad)



3ad

The title compound **3ad** was prepared according to the general procedure as described above in 72% yield (38.2 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 225 – 227 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.6 Hz, 2H), 7.59 – 7.52 (m, 4H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.34 – 7.28 (m, 3H), 7.21 (d, *J* = 7.1 Hz, 2H), 7.08 (s, 1H), 6.82 (s, 1H), 3.68 (s, 2H), 1.32 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.3, 165.9, 164.8, 142.2, 135.8, 134.7, 133.5, 132.0, 129.7, 129.3, 129.0, 128.6, 128.5, 128.4, 128.1, 126.5, 125.8, 64.8, 39.8, 37.8, 27.0. IR (film) ν_{max} 3337, 3056, 2963, 2923,1726, 1655, 1590, 1480, 1375, 1174, 1070, 840 cm^{-1}; HRMS (ESI): m/z for $C_{29}H_{27}BrN_2O_3$ $[M+H]^+$ calcd.: 531.1278, found: 531.1276.

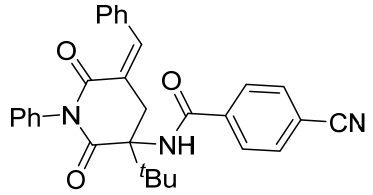
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-4-(trifluoromethyl) benzamide (3ae)



3ae

The title compound **3ae** was prepared according to the general procedure as described above in 64% yield (33.3 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 247 – 249 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.1 Hz, 2H), 7.63 (d, *J* = 8.2 Hz, 2H), 7.47 (d, *J* = 6.5 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.28 – 7.20 (m, 3H), 7.14 (d, *J* = 7.4 Hz, 2H), 7.03 (s, 1H), 6.83 (s, 1H), 3.86 – 3.51 (m, 2H), 1.26 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.2, 164.6, 163.7, 141.3, 136.9, 134.7, 133.6, 132.5 (d, *J* = 32.8 Hz), 128.6, 128.3, 127.8, 127.2, 126.4, 124.78 (d, *J* = 3.5 Hz), 124.6, 63.9, 38.8, 36.8, 25.9. ^{19}F NMR (471 MHz, $CDCl_3$) δ -113.99. IR (film) ν_{max} 3351, 3057, 2971, 1729, 1680, 1523, 1492, 1376, 1325, 1265, 1174, 1127, 1066, 854 cm^{-1}; HRMS (ESI): m/z for $C_{30}H_{27}F_3N_2O_3$ $[M+H]^+$ calcd.: 521.2047, found: 521.2047.

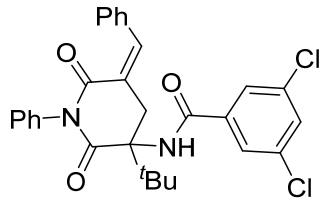
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-4-cyanobenzamide (3af)



3af

The title compound **3af** was prepared according to the general procedure as described above in 77% yield (35.3 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 2:1) to afford white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 6.2 Hz, 2H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.35 – 7.30 (m, 1H), 7.27 – 7.21 (m, 3H), 7.13 (d, *J* = 7.2 Hz, 2H), 7.03 (s, 1H), 6.83 (s, 1H), 3.72 – 3.51 (m, 2H), 1.26 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.2, 165.1, 164.6, 142.5, 138.5, 135.7, 134.6, 132.6, 129.7, 129.4, 129.1, 128.7, 128.4, 128.1, 127.7, 125.5, 117.9, 115.4, 65.1, 39.9, 37.7, 27.0. IR (film) ν_{max} 3360, 3062, 2964, 2230, 1730, 1662, 1519, 1492, 1375, 1347, 1283, 734 cm^{-1}. HRMS (ESI): m/z for $C_{30}H_{27}N_3O_3$ $[M+H]^+$ calcd.: 478.2125, found: 478.2122.

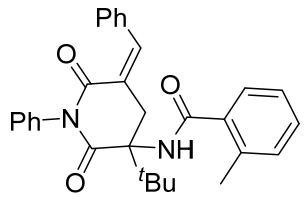
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-3,5-dichlorobenzamide (3ag)



3ag

The title compound **3ag** was prepared according to the general procedure as described above in 55% yield (28.6 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 239 – 241 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, *J* = 1.9 Hz, 2H), 7.47 (d, *J* = 6.3 Hz, 2H), 7.41 (dd, *J* = 14.3, 6.7 Hz, 3H), 7.33 (t, *J* = 7.4 Hz, 1H), 7.28 – 7.21 (m, 3H), 7.14 (d, *J* = 7.2 Hz, 2H), 7.04 (s, 1H), 6.75 (s, 1H), 3.66 (d, *J* = 15.3 Hz, 1H), 3.57 (dd, *J* = 15.3, 2.5 Hz, 1H), 1.26 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.2, 164.6, 164.3, 142.6, 137.5, 135.7, 135.7, 134.6, 131.7, 129.7, 129.4, 129.0, 128.7, 128.4, 128.1, 125.6, 125.5, 65.0, 39.9, 37.7, 27.0. IR (film) ν_{max} 3360, 3063, 2962, 1731, 1659, 1567, 1493, 1255, 1177, 961, 858, 804, 753, 734 cm^{-1}. HRMS (ESI): m/z for $C_{29}H_{26}Cl_2N_2O_3$ $[M+H]^+$ calcd.: 521.1393, found: 521.1394.

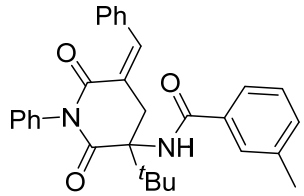
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-2-methylbenzamide (3ah)



3ah

The title compound **3ah** was prepared according to the general procedure as described above in 61% yield (28.4 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.3 Hz, 2H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.39 – 7.35 (m, 2H), 7.34 – 7.27 (m, 4H), 7.25 – 7.19 (m, 4H), 7.05 (s, 1H), 6.30 (s, 1H), 3.79 (dd, *J* = 15.0, 2.3 Hz, 1H), 3.46 (d, *J* = 16.1 Hz, 1H), 2.43 (s, 3H), 1.31 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 165.8, 163.8, 141.3, 140.8, 134.9, 133.8, 130.8, 128.6, 128.3, 128.2, 127.8, 127.5, 127.4, 127.0, 125.9, 125.0, 63.5, 38.7, 36.9, 25.9, 20.4. IR (film) ν_{max} 3339, 3058, 2963, 1730, 1655, 1491, 1374, 1264, 1176, 952, 731 cm^{-1}. HRMS (ESI): m/z for $C_{30}H_{30}N_2O_3$ $[M+H]^+$ calcd.: 467.2329, found: 467.2327.

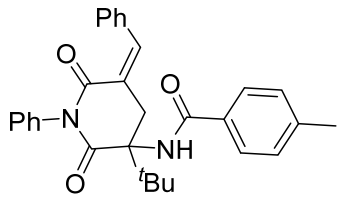
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-3-methylbenzamide (3ai)



3ai

The title compound **3ai** was prepared according to the general procedure as described above in 90% yield (41.9 mg). It was purified by flash column chromatography (Petroleum ether: EtOAc = 5:1) to afford white solid. mp = 194 – 196 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 – 7.50 (m, 4H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.34 – 7.27 (m, 5H), 7.23 (d, *J* = 7.1 Hz, 2H), 7.07 (s, 1H), 6.83 (s, 1H), 3.72 – 3.64 (m, 2H), 2.38 (s, 3H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.3, 167.1, 164.9, 141.9, 138.7, 136.0, 134.8, 134.7, 132.6, 129.7, 129.3, 128.9, 128.62, 128.55, 128.5, 128.1, 127.7, 126.0, 123.8, 64.6, 39.7, 38.0, 27.0, 21.4. IR (film) ν_{max} 3400, 3056, 2965, 1729, 1683, 1492, 1373, 1265, 1177, 962, 731 cm^{-1}. HRMS (ESI): m/z for $C_{30}H_{30}N_2O_3$ $[M+H]^+$ calcd.: 467.2329, found: 467.2331.

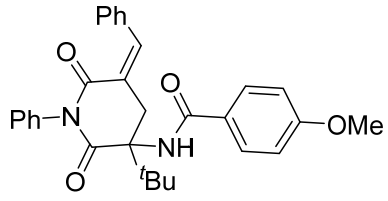
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-4-methylbenzamide (3aj)



3aj

The title compound **3aj** was prepared according to the general procedure as described above in 92% yield (42.8 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 143 – 145 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.9 Hz, 2H), 7.54 (d, *J* = 7.5 Hz, 2H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.40 – 7.36 (m, 1H), 7.33 – 7.27 (m, 3H), 7.23 (d, *J* = 8.0 Hz, 4H), 7.07 (s, 1H), 6.81 (s, 1H), 3.69 (dd, 2H), 2.39 (s, 3H), 1.33 (s, 9H).^{13}C NMR (126 MHz, $CDCl_3$) δ 172.4, 166.8, 164.9, 142.3, 141.9, 136.0, 134.8, 131.8, 129.7, 129.4, 129.3, 128.8, 128.53, 128.47, 128.0, 126.9, 126.1, 64.6, 39.7, 38.0, 27.0, 21.5. IR (film) ν_{max} 3349, 3059, 2962, 1728, 1682, 1653, 1522, 1492, 1374, 1282, 1177, 962, 754 cm^{-1}. HRMS (ESI): m/z for $C_{30}H_{30}N_2O_3$ $[M+H]^+$ calcd.: 467.2329, found: 467.2332.

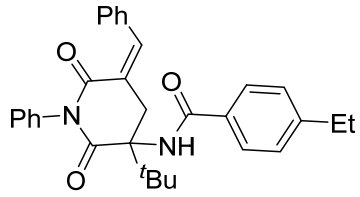
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-4-methoxybenzamide (3ak)



3ak

The title compound **3ak** was prepared according to the general procedure as described above in 96% yield (46.3 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford yellow solid. mp = 177 – 179 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.8 Hz, 2H), 7.54 (d, *J* = 6.7 Hz, 2H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.33 – 7.27 (m, 3H), 7.23 (d, *J* = 7.5 Hz, 2H), 7.06 (s, 1H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.76 (s, 1H), 3.84 (s, 3H), 3.71 (dd, *J* = 15.2, 2.3 Hz, 1H), 3.65 (d, *J* = 15.4 Hz, 1H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.4, 166.4, 164.9, 162.4, 141.8, 136.0, 134.8, 129.67, 129.65, 129.3, 128.82, 128.75, 128.52, 128.47, 128.0, 126.9, 126.1, 113.9, 64.5, 55.5, 39.7, 38.0, 27.0. IR (film) ν_{max} 3390, 3060, 2962, 2840, 1728, 1681, 1651, 1606, 1491, 1374, 1346, 1252, 1173, 1028, 962, 842 cm^{-1}. HRMS (ESI): m/z for $C_{30}H_{30}N_2O_4$ $[M+H]^+$ calcd.: 483.2278, found: 483.2274.

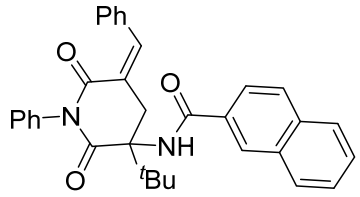
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-4-ethylbenzamide (3al)



3al

The title compound **3al** was prepared according to the general procedure as described above in 74% yield (35.5 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 185 – 187 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.3 Hz, 2H), 7.46 (d, *J* = 6.8 Hz, 2H), 7.41 – 7.37 (m, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.26 – 7.21 (m, 2H), 7.20 – 7.15 (m, 5H), 6.99 (s, 1H), 6.75 (s, 1H), 3.73 – 3.37 (m, 2H), 2.61 (q, *J* = 7.6 Hz, 2H), 1.25 (s, 9H), 1.17 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 165.8, 163.9, 147.5, 140.8, 134.9, 133.8, 131.0, 128.6, 128.2, 127.8, 127.5, 127.4, 127.2, 127.0, 126.0, 125.0, 63.5, 36.9, 27.8, 25.9, 14.3. IR (film) ν_{max} 3391, 3028, 2964, 1729, 1683, 1655, 1522, 1492, 1374, 1177, 962, 849 cm^{-1}. HRMS (ESI): m/z for $C_{31}H_{32}N_2O_3$ $[M+H]^+$ calcd.: 481.2486, found: 481.2482.

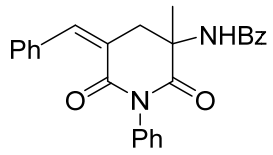
(Z)-N-(5-benzylidene-3-(tert-butyl)-2,6-dioxo-1-phenylpiperidin-3-yl)-2-naphthamide (3am)



3am

The title compound **3am** was prepared according to the general procedure as described above in 73% yield (36.6 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 195 – 197 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 (d, *J* = 11.0 Hz, 1H), 7.83 (dd, *J* = 19.2, 10.8 Hz, 3H), 7.73 (d, *J* = 6.5 Hz, 1H), 7.49 (d, *J* = 6.7 Hz, 4H), 7.41 (t, *J* = 7.9 Hz, 3H), 7.33 (d, *J* = 7.4 Hz, 1H), 7.24 (dd, *J* = 11.0, 8.2 Hz, 3H), 7.05 (s, 1H), 6.96 (s, 1H), 3.69 (s, 2H), 1.31 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.2, 165.9, 163.9, 140.9, 134.9, 133.6, 132.3, 131.8, 131.4, 130.8, 128.7, 128.2, 127.7, 127.51, 127.46, 127.4, 126.6, 126.4, 125.93, 125.85, 125.7, 125.1, 63.6, 38.7, 37.0, 25.9. IR (film) ν_{max} 3367, 3059, 2959, 1729, 1682, 1497, 1374, 1290, 1179, 962, 778 cm^{-1}. HRMS (ESI): m/z for $C_{33}H_{30}N_2O_3$ $[M+H]^+$ calcd.: 503.2329, found: 503.2327.

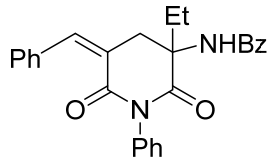
(Z)-N-(5-benzylidene-3-methyl-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3an)



3an

The title compound **3an** was prepared according to the general procedure as described above in 74% yield (30.4 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 211 – 213 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (d, J = 2.7 Hz, 1H), 7.82 – 7.76 (m, 2H), 7.53 – 7.49 (m, 3H), 7.48 – 7.45 (m, 4H), 7.45 – 7.41 (m, 3H), 7.24 (d, J = 1.5 Hz, 1H), 7.22 (s, 1H), 7.17 (s, 1H), 4.13 (d, J = 15.3 Hz, 1H), 3.45 (dd, J = 15.3, 2.9 Hz, 1H), 1.74 (s, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 174.4, 167.0, 166.0, 142.9, 135.6, 134.4, 134.2, 131.9, 130.0, 129.6, 129.4, 128.9, 128.8, 128.7, 128.3, 127.0, 125.1, 57.1, 33.4, 23.3. IR (film) ν_{max} 3350, 3057, 2926, 2854, 1732, 1678, 1523, 1488, 1447, 1349, 1263, 1193, 968 cm^{-1}. HRMS (ESI): m/z for $C_{26}H_{22}N_2O_3$ $[M+H]^+$ calcd.: 411.1703, found: 411.1701.

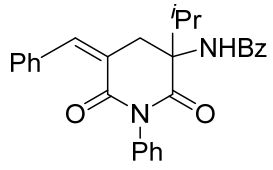
(Z)-N-(5-benzylidene-3-ethyl-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3ao)



3ao

The title compound **3ao** was prepared according to the general procedure as described above in 64% yield (27.1 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 149 – 151 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (d, *J* = 2.8 Hz, 1H), 7.81 (d, *J* = 7.5 Hz, 2H), 7.51 (dd, *J* = 13.8, 5.9 Hz, 5H), 7.48 – 7.41 (m, 6H), 7.36 (s, 1H), 7.20 (d, *J* = 8.2 Hz, 2H), 4.44 (dd, *J* = 15.6, 2.2 Hz, 1H), 3.32 (dd, *J* = 15.6, 3.0 Hz, 1H), 2.58 – 2.47 (m, 1H), 2.01 – 1.91 (m, 1H), 0.91 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.7, 164.9, 142.0, 134.6, 133.34, 133.28, 130.9, 129.0, 128.7, 128.4, 127.9, 127.8, 127.7, 127.3, 125.9, 123.8, 120.0, 59.7, 31.7, 27.2, 7.2. IR (film) ν_{max} 3351, 3059, 2973, 1728, 1678, 1518, 1486, 1254, 1193, 1028, 957, 802 cm^{-1}. HRMS (ESI): m/z for $C_{27}H_{24}N_2O_3$ $[M+H]^+$ calcd.: 425.1860, found: 425.1858.

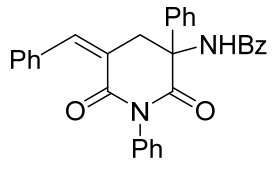
(Z)-N-(5-benzylidene-3-isopropyl-2,6-dioxo-1-phenylpiperidin-3-yl)benzamide (3ap)



3ap

The title compound **3ap** was prepared according to the general procedure as described above in 69% yield (30.2 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 5:1) to afford white solid. mp = 161 – 163 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.2 Hz, 2H), 7.59 (d, *J* = 6.2 Hz, 2H), 7.52 (d, *J* = 7.3 Hz, 1H), 7.49 – 7.40 (m, 5H), 7.32 (q, *J* = 8.5, 7.5 Hz, 3H), 7.21 – 7.16 (m, 3H), 7.00 (s, 1H), 3.92 (d, *J* = 14.8 Hz, 1H), 3.62 (dd, *J* = 14.9, 2.4 Hz, 1H), 2.51 (p, *J* = 6.8 Hz, 1H), 1.28 (d, *J* = 6.8 Hz, 3H), 1.14 (d, *J* = 6.9 Hz, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 173.5, 167.3, 164.4, 144.0, 135.5, 134.7, 134.6, 131.8, 129.9, 129.4, 129.1, 128.72, 128.68, 128.4, 128.0, 127.0, 124.5, 63.2, 37.3, 34.5, 17.7, 17.3. IR (film) ν_{max} 3351, 3060, 2968, 1730, 1682, 1598, 1580, 1515, 1487, 1285, 1241, 1199, 1073, 959, 735 cm^{-1}. HRMS (ESI): m/z for $C_{28}H_{26}N_2O_3$ $[M+H]^+$ calcd.: 439.2016, found: 439.2016.

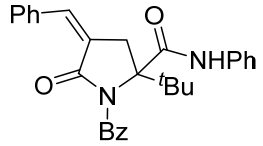
(Z)-N-(5-benzylidene-2,6-dioxo-1,3-diphenylpiperidin-3-yl)benzamide (3aq)



3aq

The title compound **3aq** was prepared according to the general procedure as described above in 58% yield (27.3 mg). It was purified by flash column chromatography (Petroleum ether: EtOAc = 5:1) to afford white solid. mp = 188 – 189 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 (s, 1H), 7.88 (s, 1H), 7.83 – 7.78 (m, 2H), 7.56 – 7.49 (m, 3H), 7.43 (t, *J* = 7.8 Hz, 3H), 7.37 – 7.32 (m, 5H), 7.34 – 7.28 (m, 4H), 7.13 (t, *J* = 7.4 Hz, 1H), 7.01 (d, *J* = 6.4 Hz, 1H), 5.80 (d, *J* = 6.3 Hz, 1H), 5.18 (d, *J* = 12.6 Hz, 1H), 5.08 (d, *J* = 12.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.4, 167.0, 165.0, 143.4, 138.0, 135.6, 134.2, 133.3, 132.1, 129.6, 129.4, 129.3, 129.1, 128.9, 128.8, 128.7, 127.5, 127.2, 124.5, 120.4, 61.3, 57.6. IR (film) ν_{max} 3367, 3059, 2959, 2925, 1729, 1682, 1497, 1348, 1290, 1179, 762 cm^{-1}. HRMS (ESI): m/z for $C_{31}H_{24}N_2O_3$ $[M+H]^+$ calcd.:473.1860, found: 473.1861.

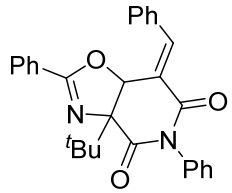
(Z)-N-(1-benzoyl-4-benzylidene-2-(tert-butyl)-5-oxopyrrolidin-2-yl)benzamide (4aa)



4aa

The title compound **4aa** was prepared according to the general procedure as described above in 75% yield (33.8 mg). It was purified by flash column chromatography (Petroleum ether: EtOAc = 10:1) to afford white solid. mp = 176 – 178 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.60 (s, 1H), 7.88 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.74 (dd, *J* = 6.9, 2.9 Hz, 2H), 7.64 – 7.56 (m, 3H), 7.52 – 7.45 (m, 2H), 7.37 – 7.31 (m, 2H), 7.24 (d, *J* = 3.0 Hz, 2H), 7.11 (t, *J* = 7.4 Hz, 1H), 7.03 (d, *J* = 1.8 Hz, 1H), 3.78 (dd, *J* = 14.7, 1.2 Hz, 1H), 3.23 (dd, *J* = 14.8, 2.9 Hz, 1H), 1.24 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.3, 169.5, 168.3, 140.6, 138.0, 134.0, 133.63, 133.59, 130.9, 130.5, 129.6, 129.0, 128.7, 128.1, 126.1, 124.5, 120.5, 80.6, 40.2, 38.1, 27.6. IR (film) ν_{max} 3057, 2929, 1782, 1656, 1492, 1474, 1265, 1090, 1006, 731 cm^{-1}. HRMS (ESI): m/z for $C_{29}H_{28}N_2O_3$ $[M+H]^+$ calcd.: 453.2173, found: 453.2171.

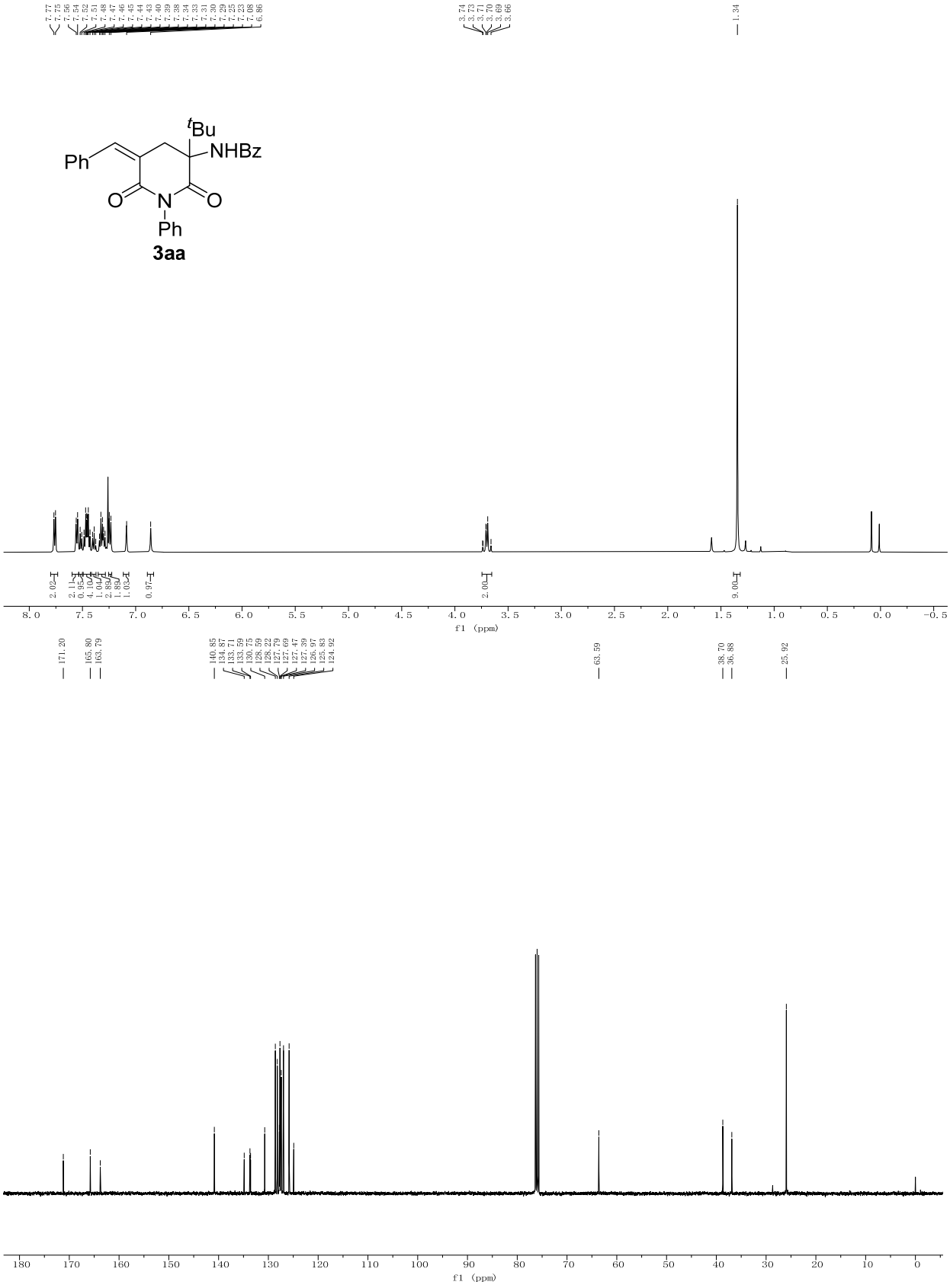
(E)-7-benzylidene-3a-(tert-butyl)-2,5-diphenyl-7,7a-dihydrooxazolo[4,5-c]pyridine-4,6(3aH,5H)-dione (5aa)

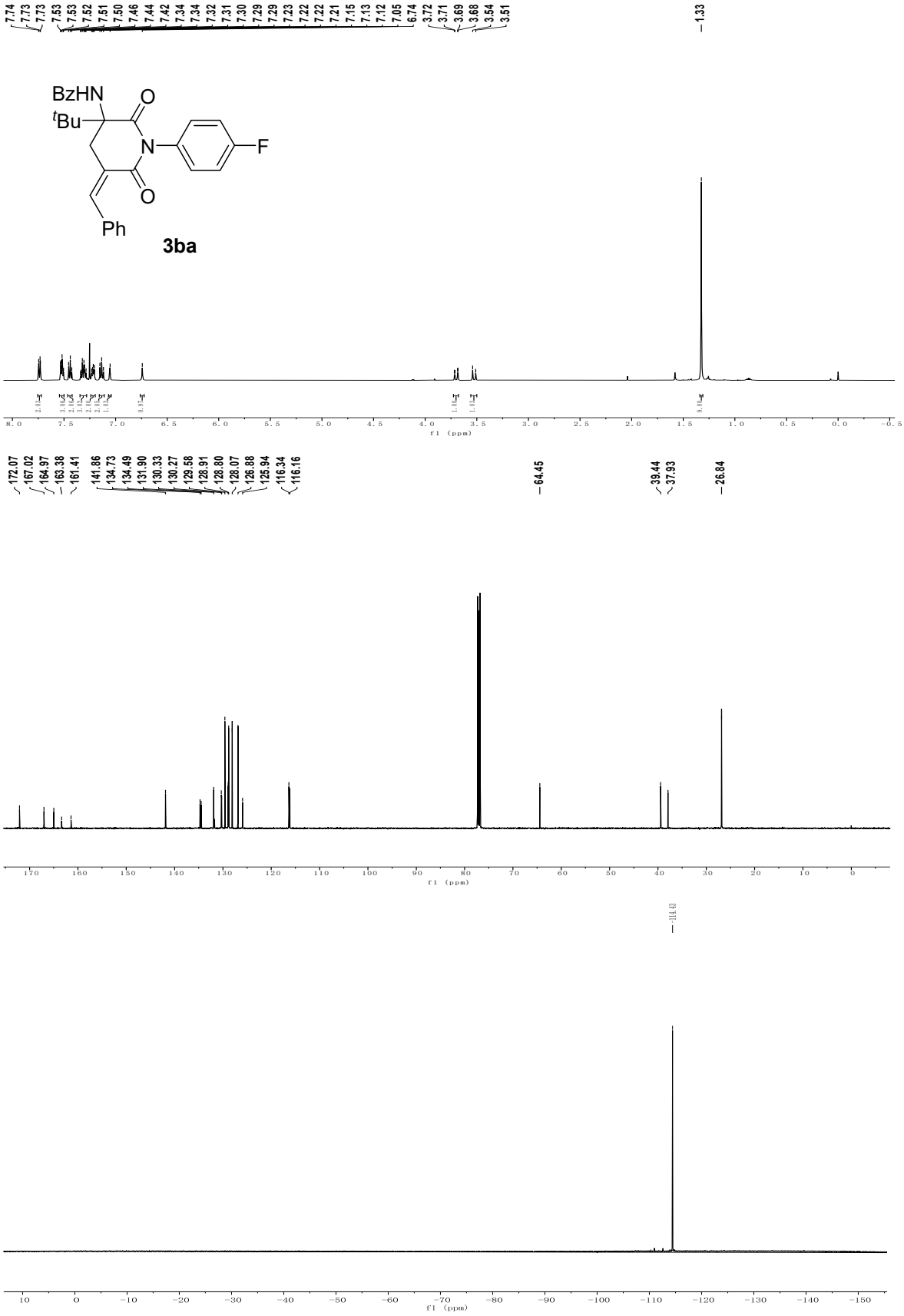


5aa

The title compound **5aa** was prepared according to the general procedure as described above in 42% yield (18.9 mg). It was purified by flash column chromatography (Petroleum ether : EtOAc = 15:1) to afford white solid. mp = 198 – 199 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.42 (s, 1H), 8.15 – 8.08 (m, 2H), 7.75 (dd, *J* = 6.6, 3.1 Hz, 2H), 7.60 – 7.52 (m, 4H), 7.50 – 7.45 (m, 4H), 7.41 (t, *J* = 7.4 Hz, 1H), 7.17 – 7.09 (m, 2H), 5.56 (s, 1H), 1.66 (s, 1H), 1.10 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.4, 164.5, 164.2, 149.4, 135.9, 133.4, 132.2, 131.0, 130.4, 129.3, 129.2, 128.8, 128.6, 128.5, 128.4, 126.9, 122.3, 81.2, 76.0, 37.0, 26.4. IR (film) ν_{max} 3062, 2961, 2871, 1724, 1680, 1355, 1286, 1196, 970, 731 cm^{-1}. HRMS (ESI): m/z for $C_{29}H_{26}N_2O_3$ $[M+H]^+$ calcd.: 450.1943, found: 450.1944.

^{1}H and ^{13}C NMR Spectra of All Products

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N
F
O
Ph
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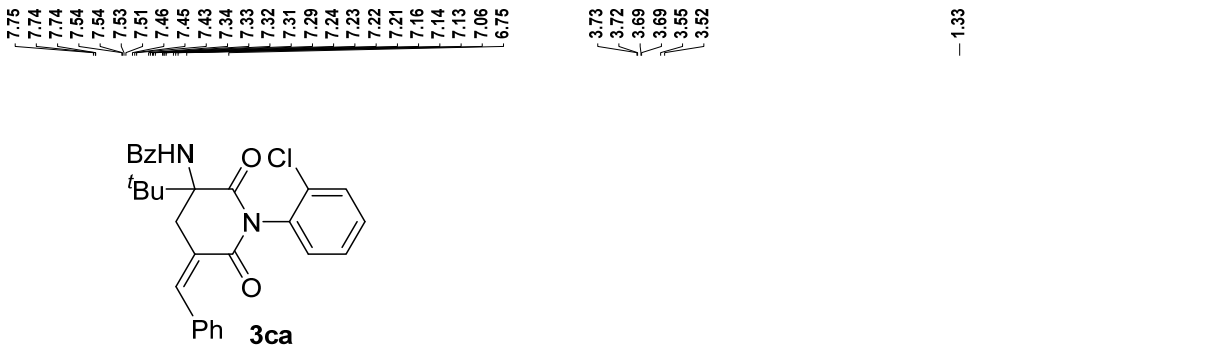
f1 (ppm)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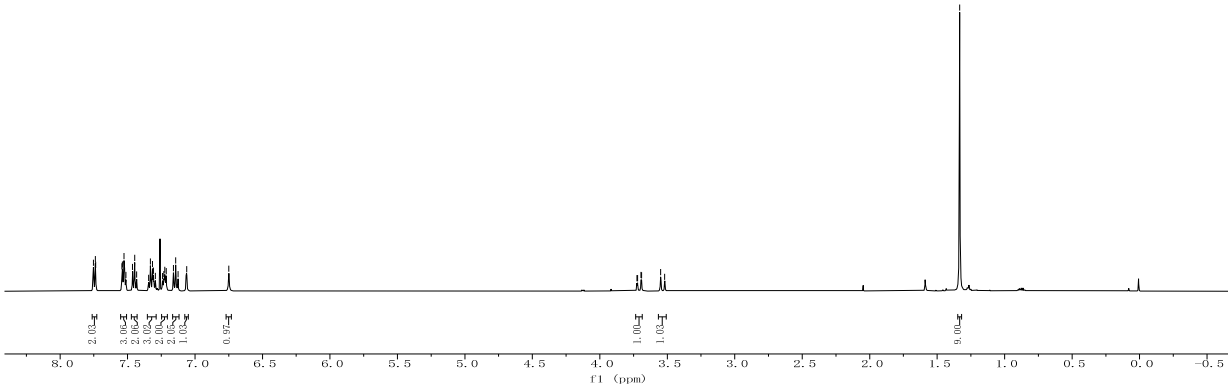
tBu
N
O
Ph

3ca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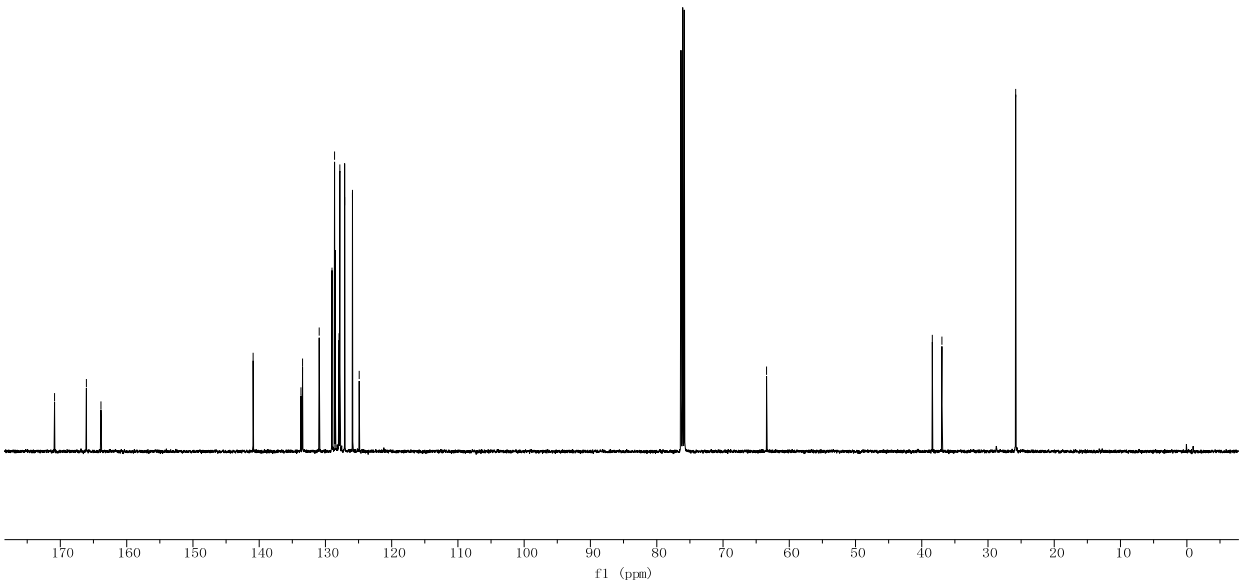
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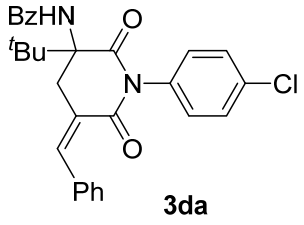
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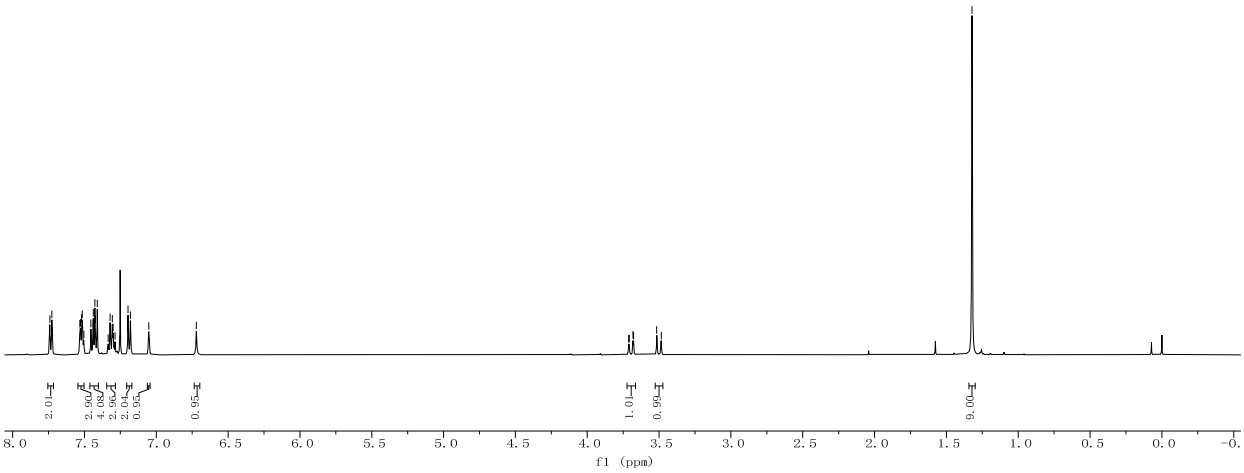
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6.0
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1.5
1.0
0.5
0.0
f1 (ppm)


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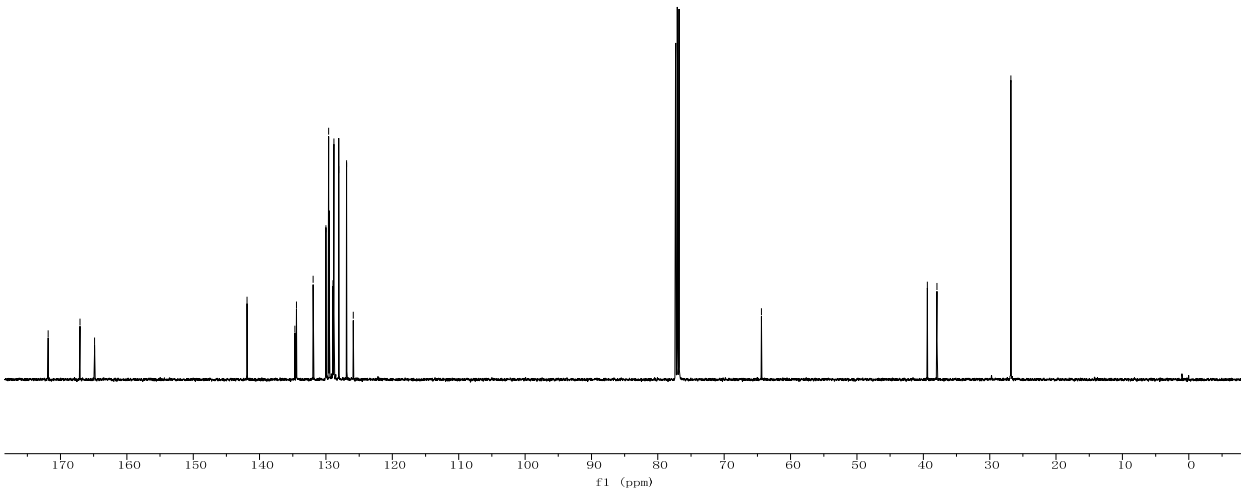
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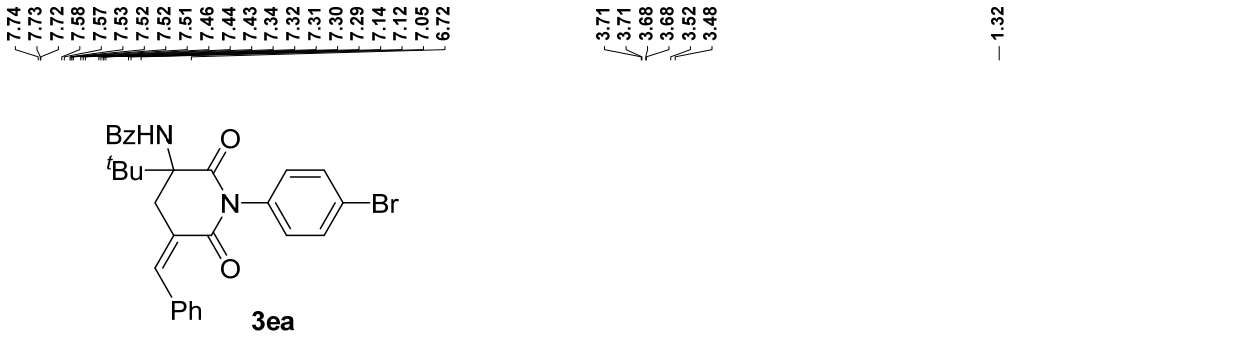
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Br
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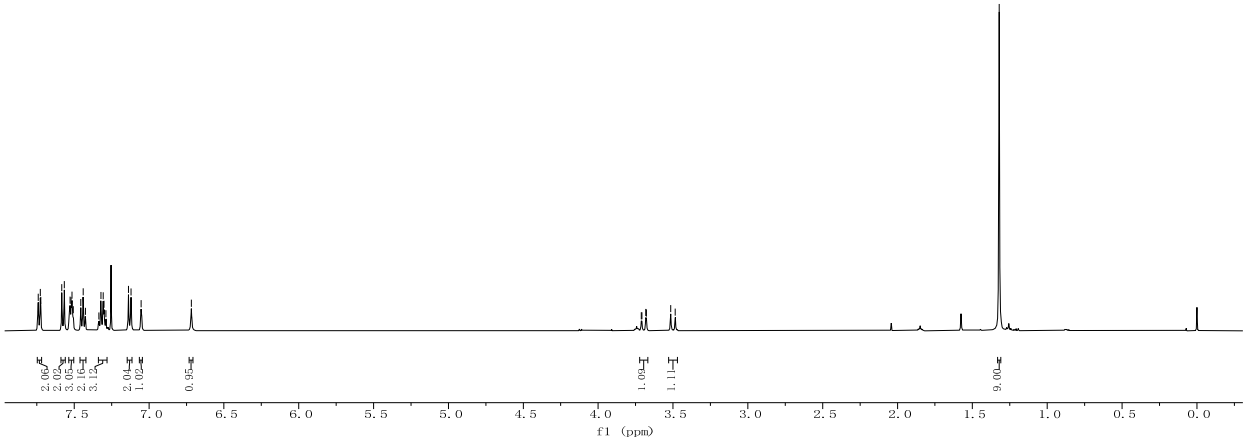
Ph

3ea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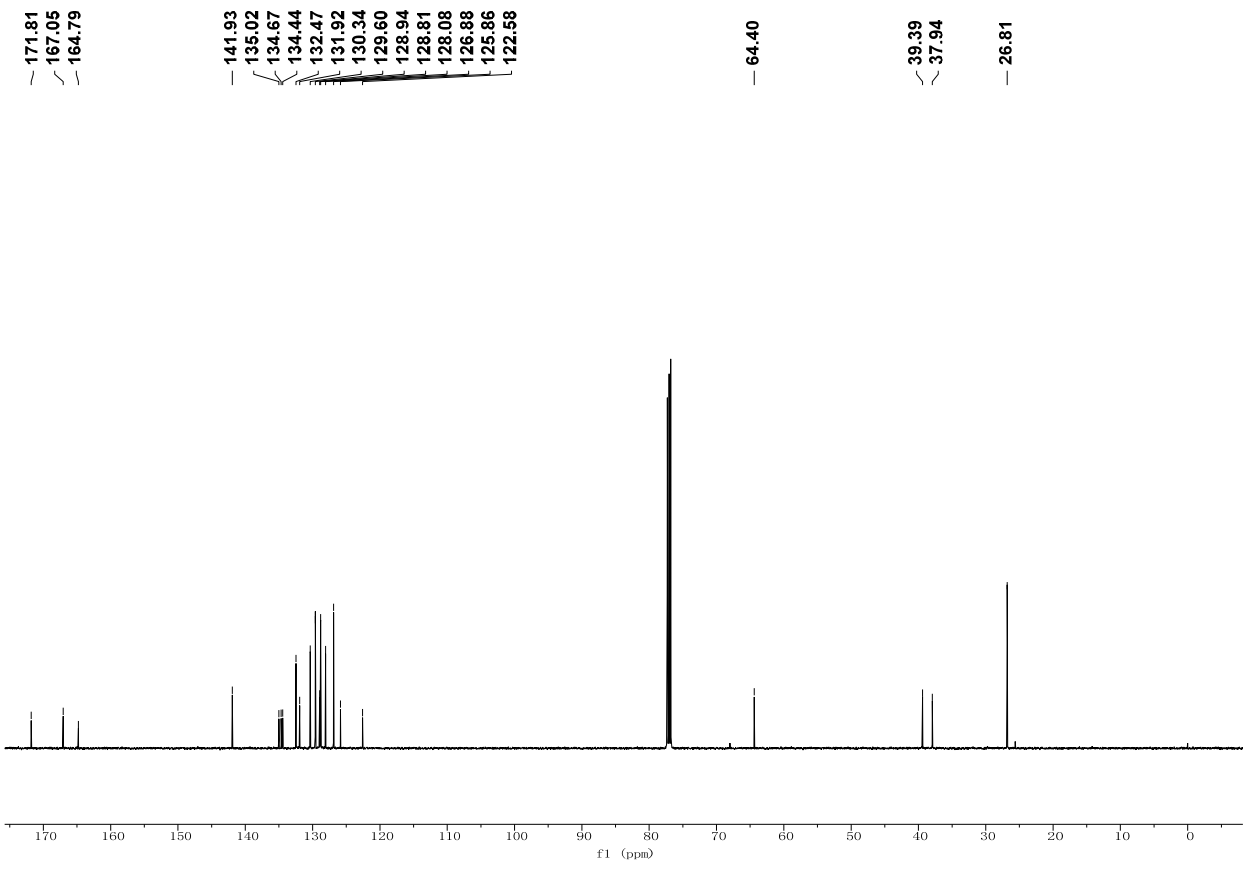
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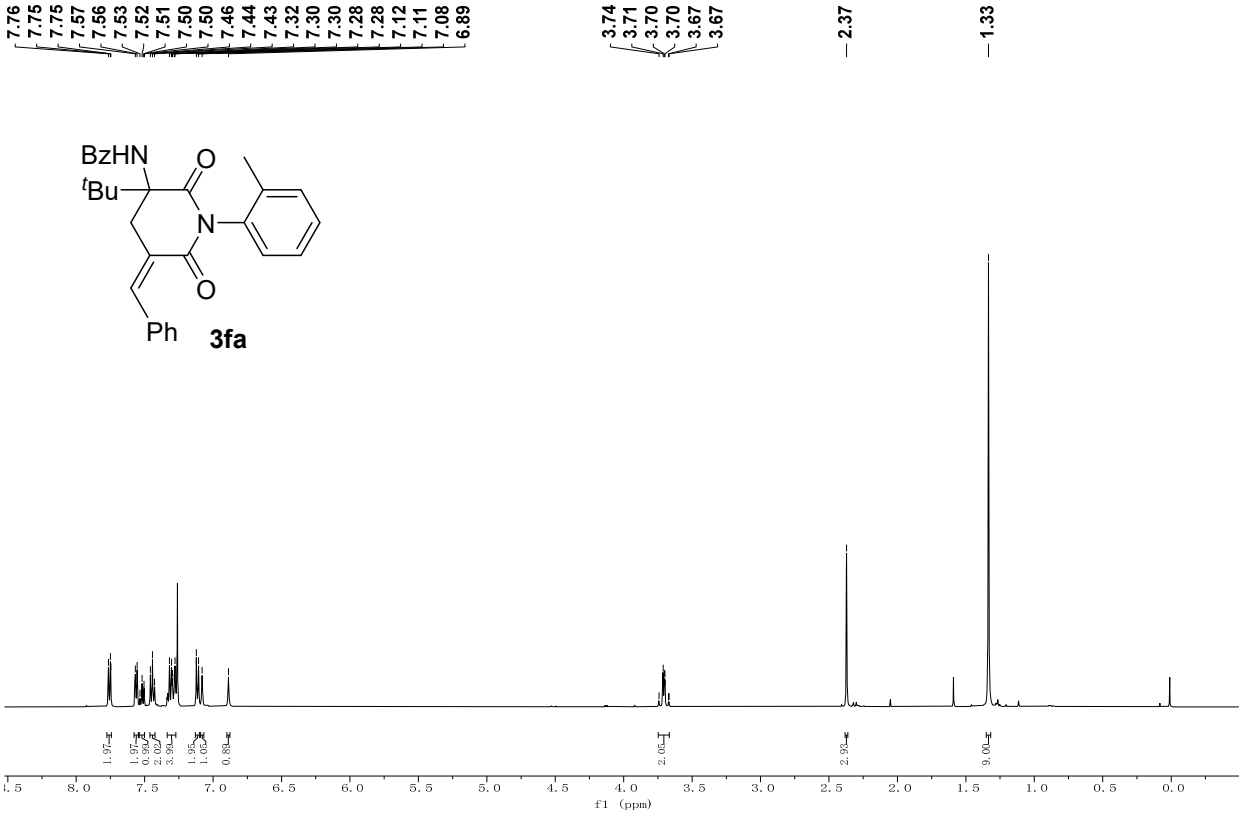
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tBu
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O
Ph
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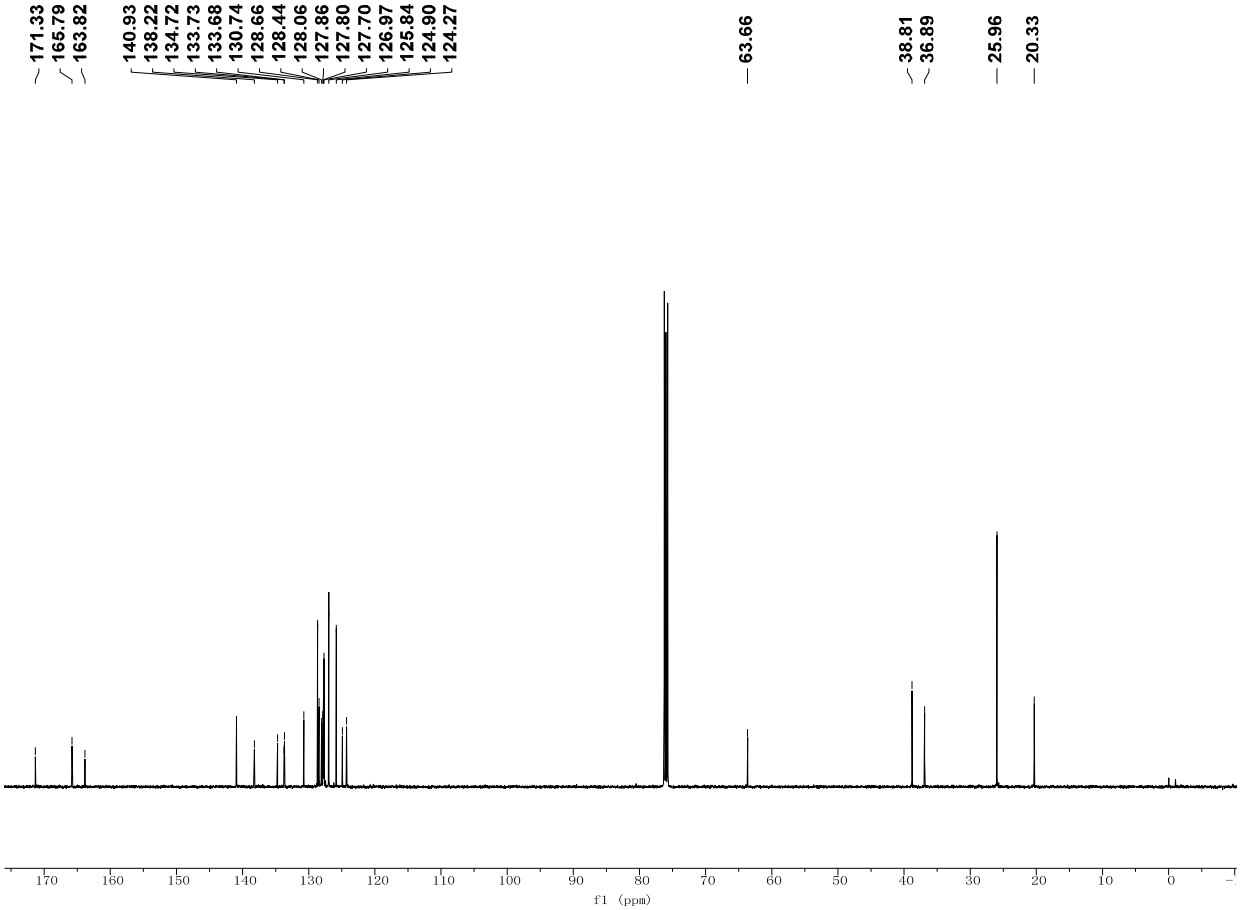
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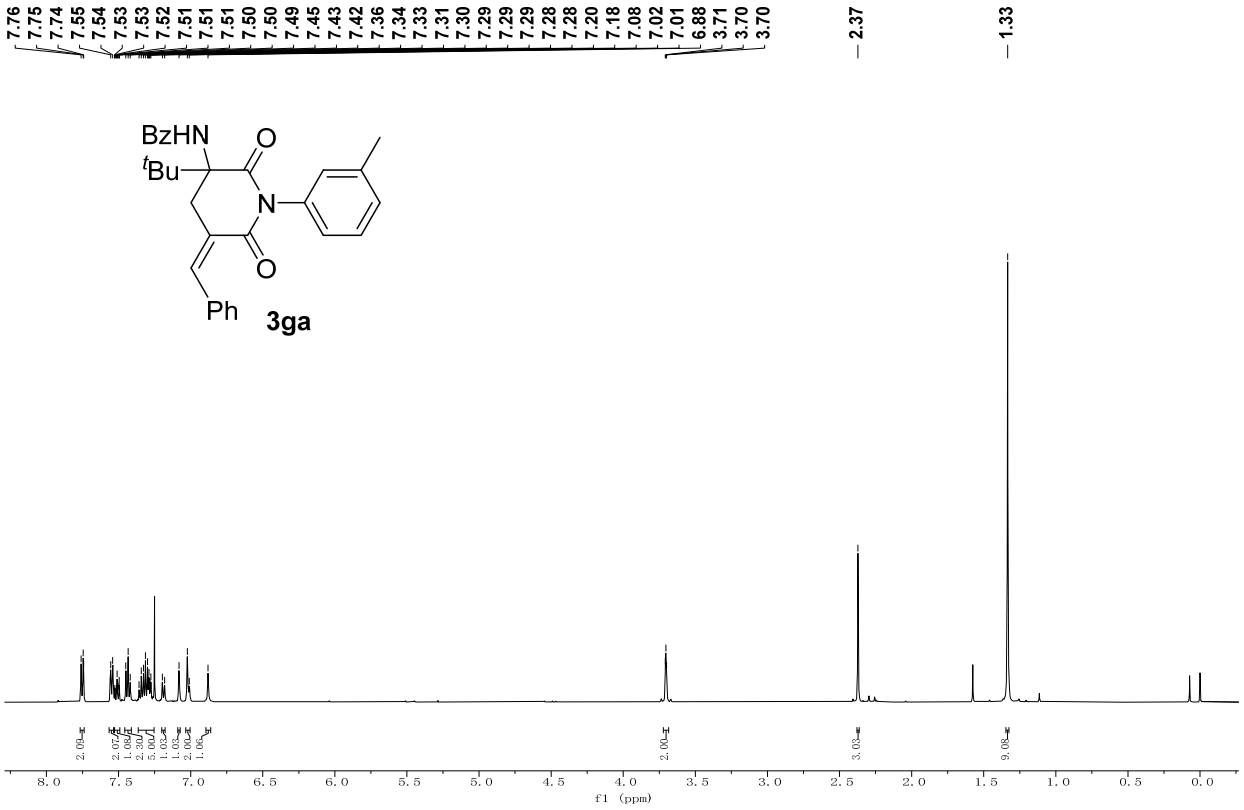
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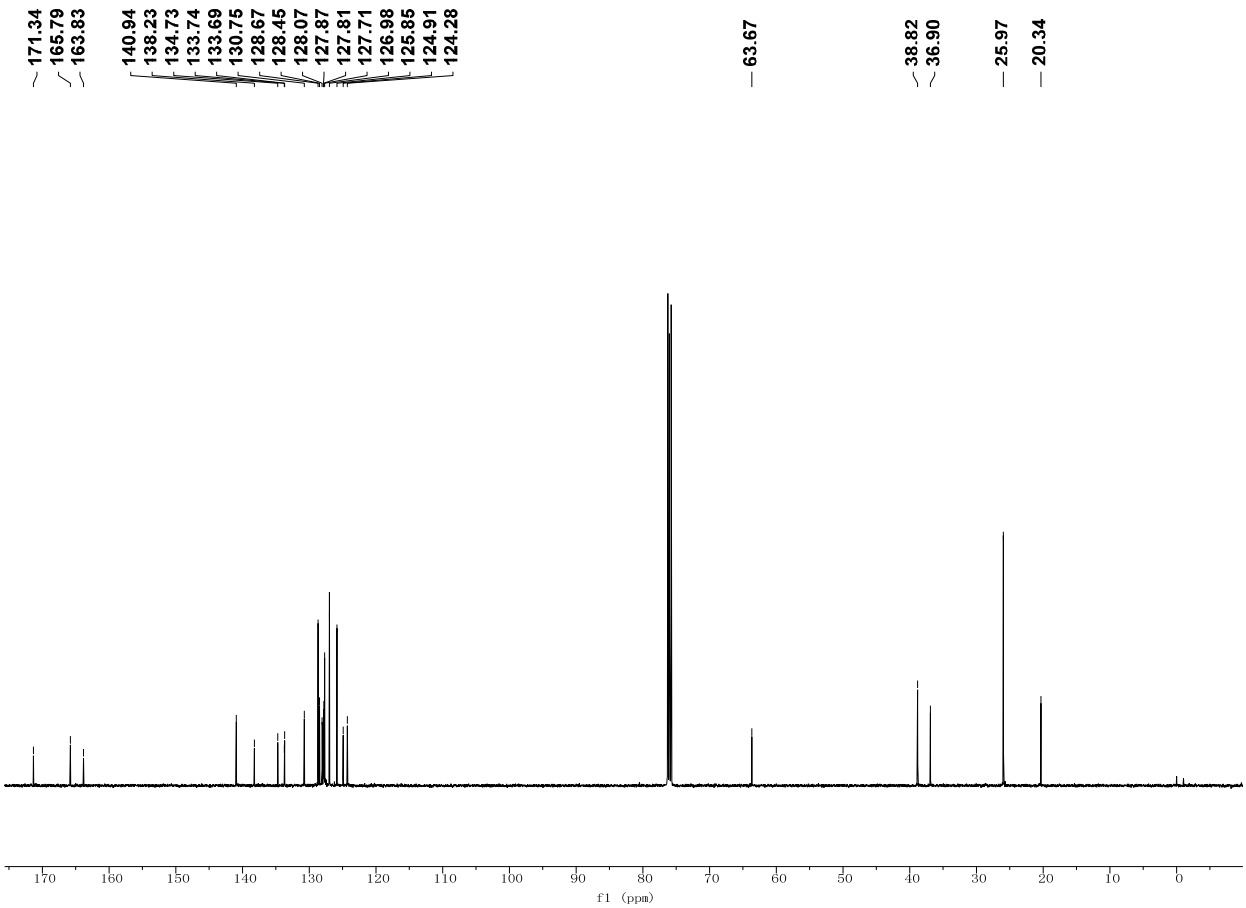
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20.34
f1 (ppm)


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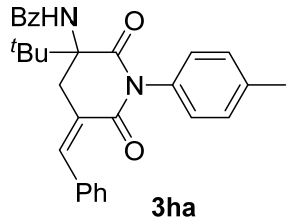
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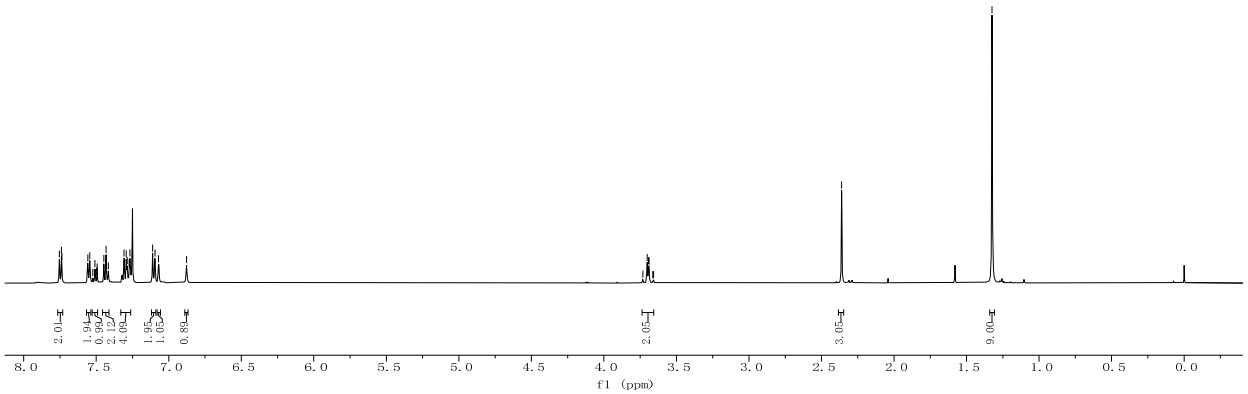
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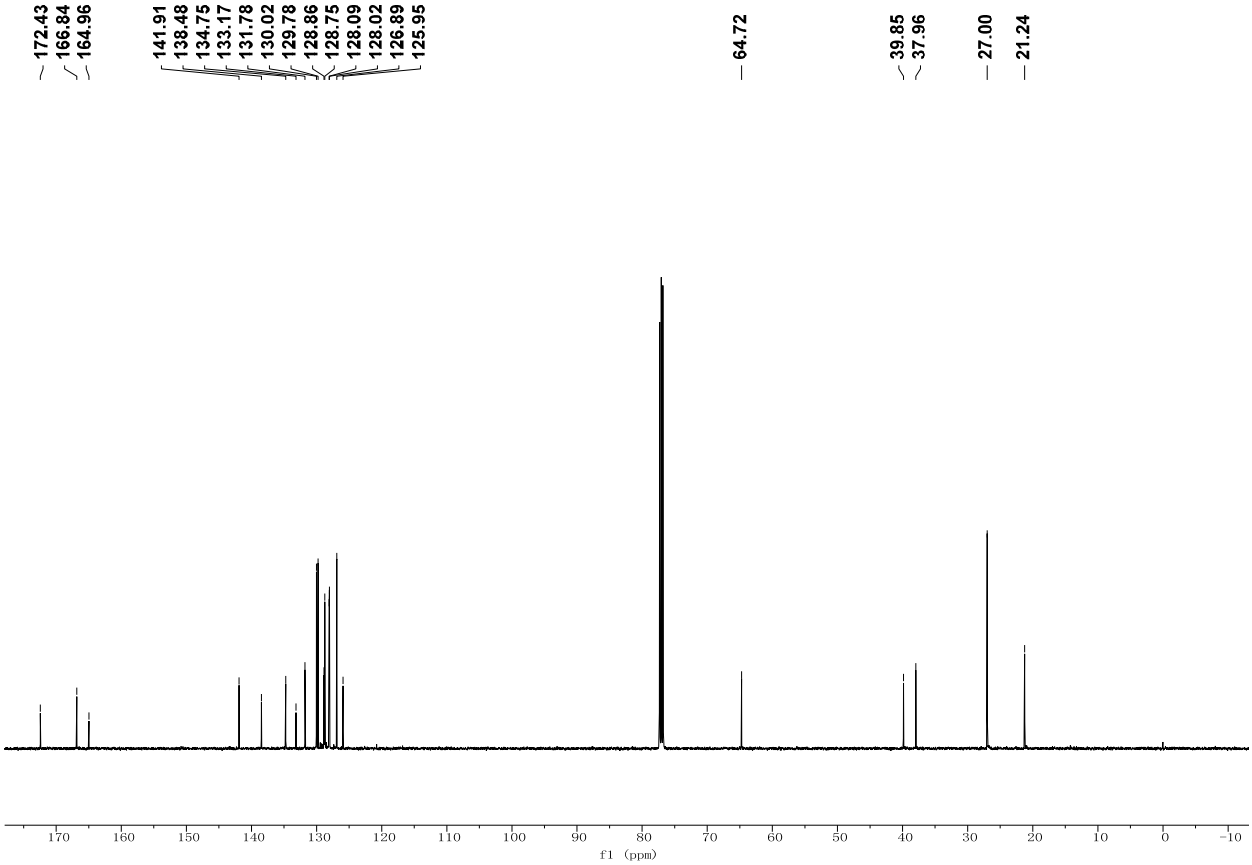
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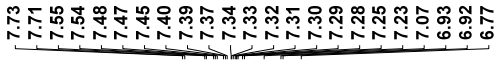
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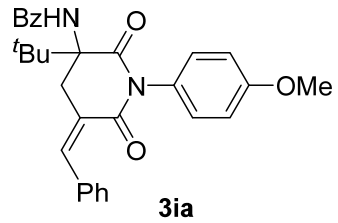
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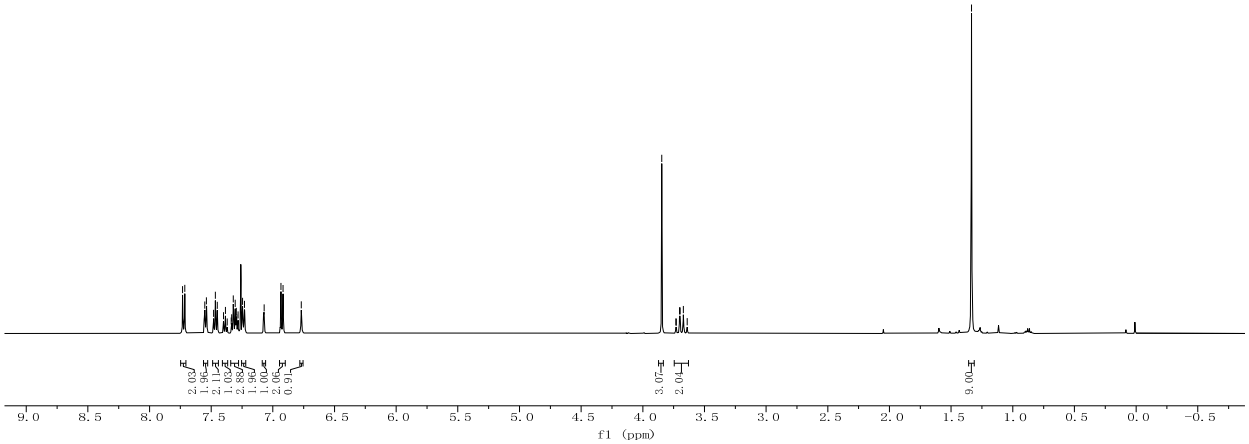
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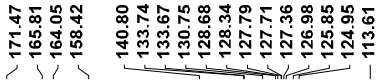
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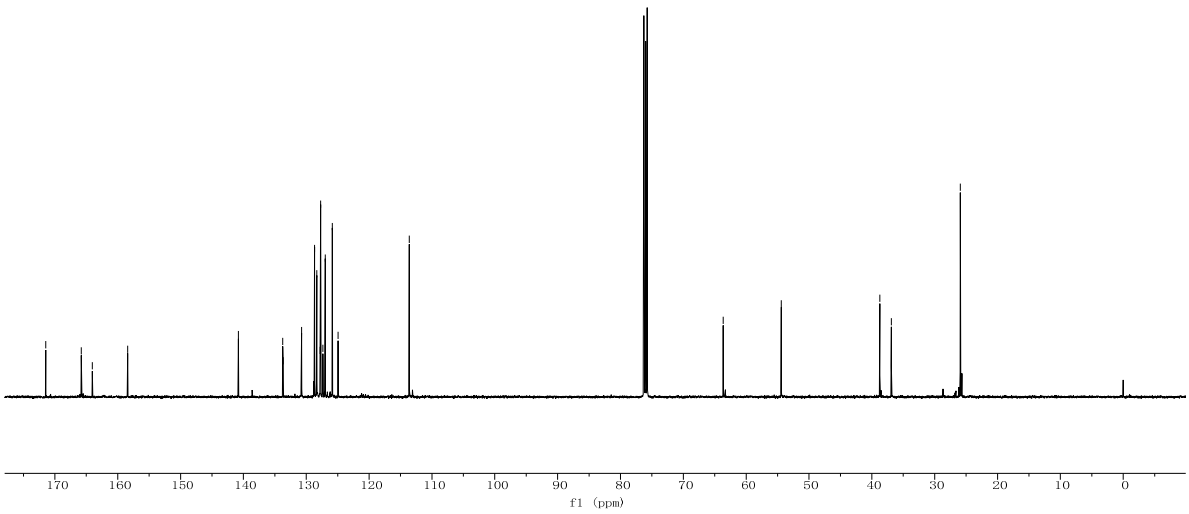
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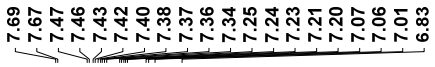
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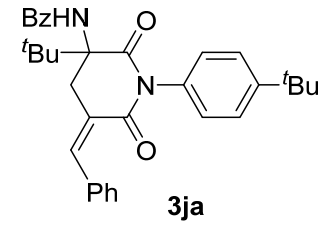
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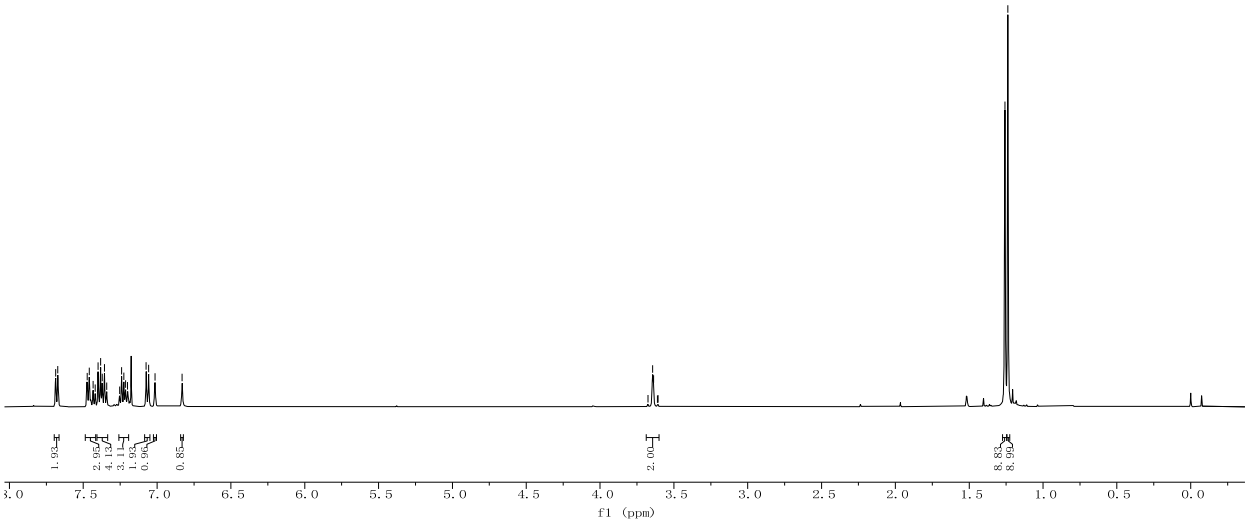
N
tBu
O
Ph

3ja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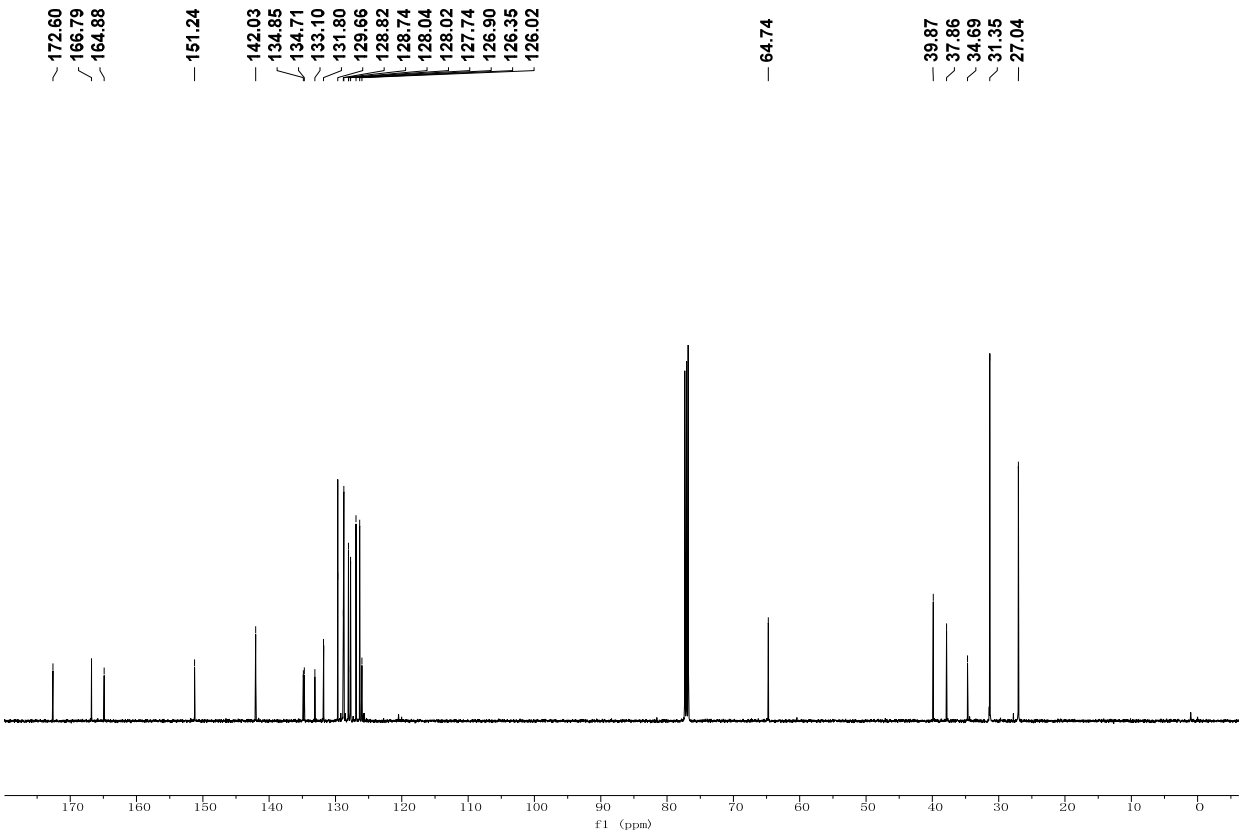
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128.04
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34.69
31.35
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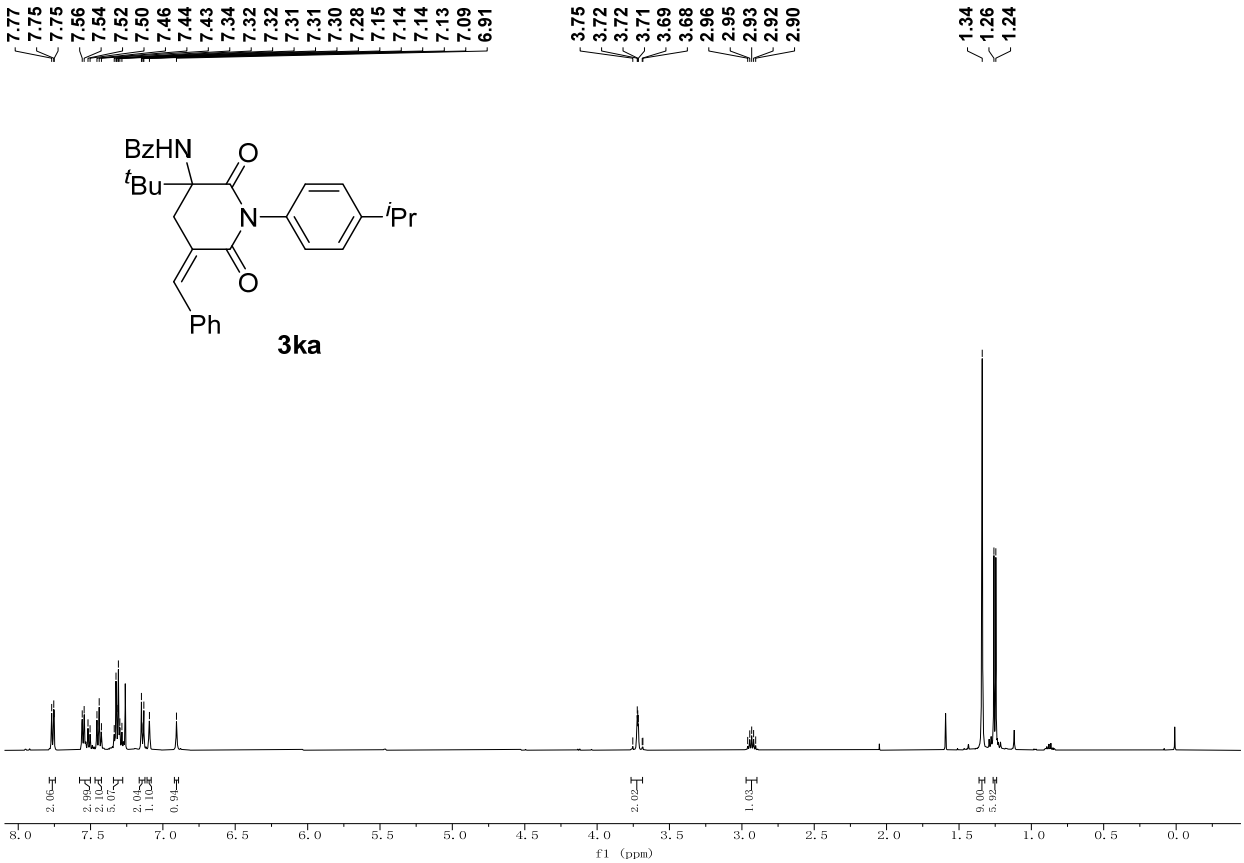
f1 (ppm)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7.14
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7.13
7.09
6.91
3.75
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3.69
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2.93
2.92
2.90
1.34
1.26
1.24
BzHN
tBu
O
N
O
iPr
Ph
3ka
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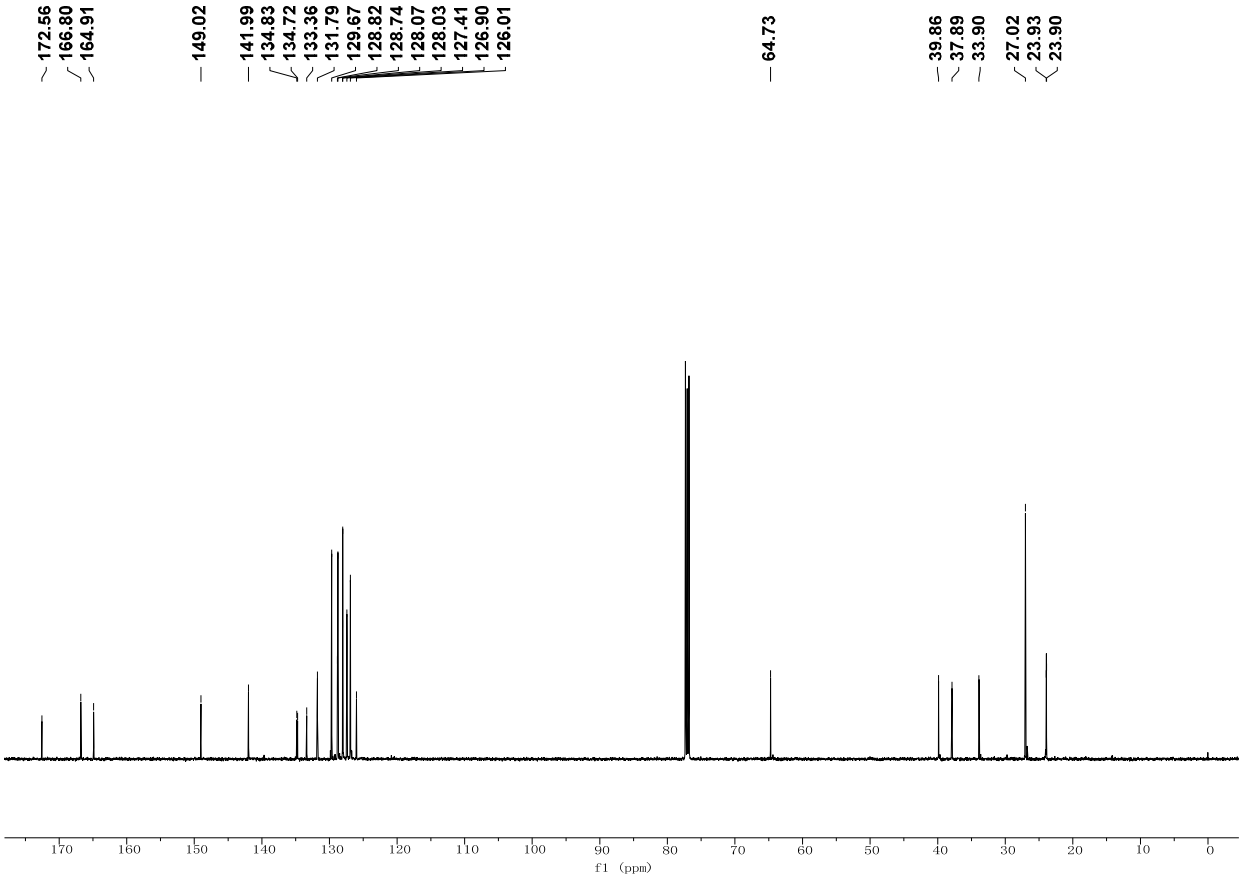
f1 (ppm)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33.90
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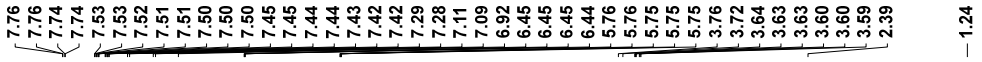
23.90
f1 (ppm)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7.45
7.44
7.44
7.43
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7.42
7.29
7.28
7.11
7.09
6.92
6.45
6.45
6.45
6.44
5.76
5.76
5.75
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3.63
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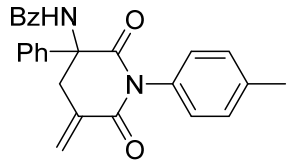
1.24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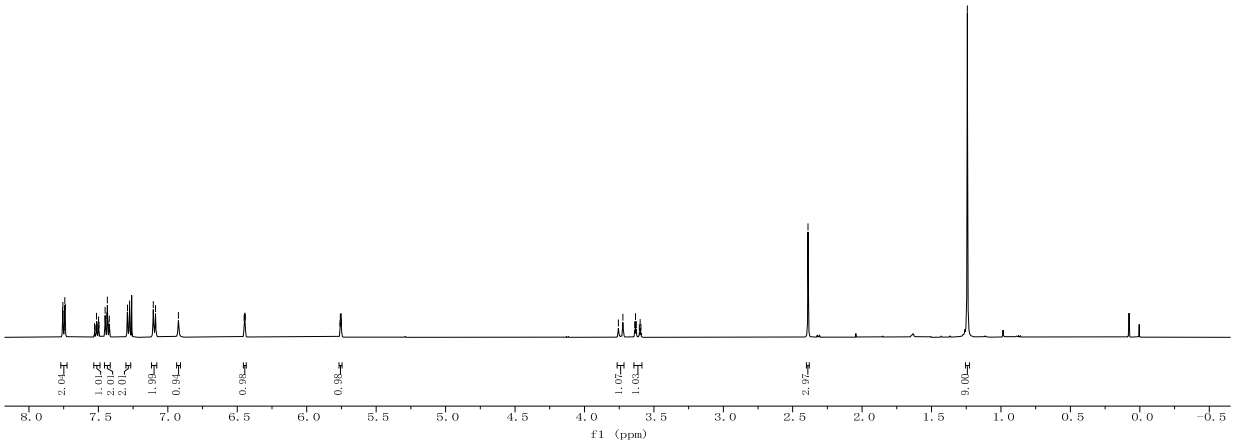
Ph
O
N
O

3la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1.03
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7.0
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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
-0.5
f1 (ppm)


171.29
165.81
164.07
137.52
134.26
133.63
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126.86
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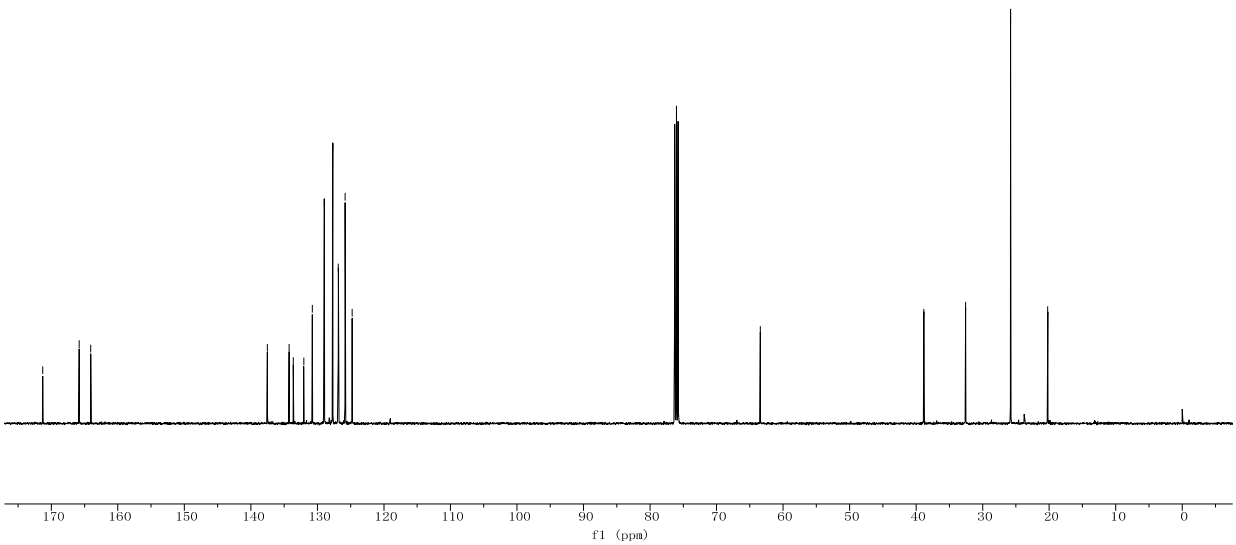
63.43
38.85
32.57
25.80
20.21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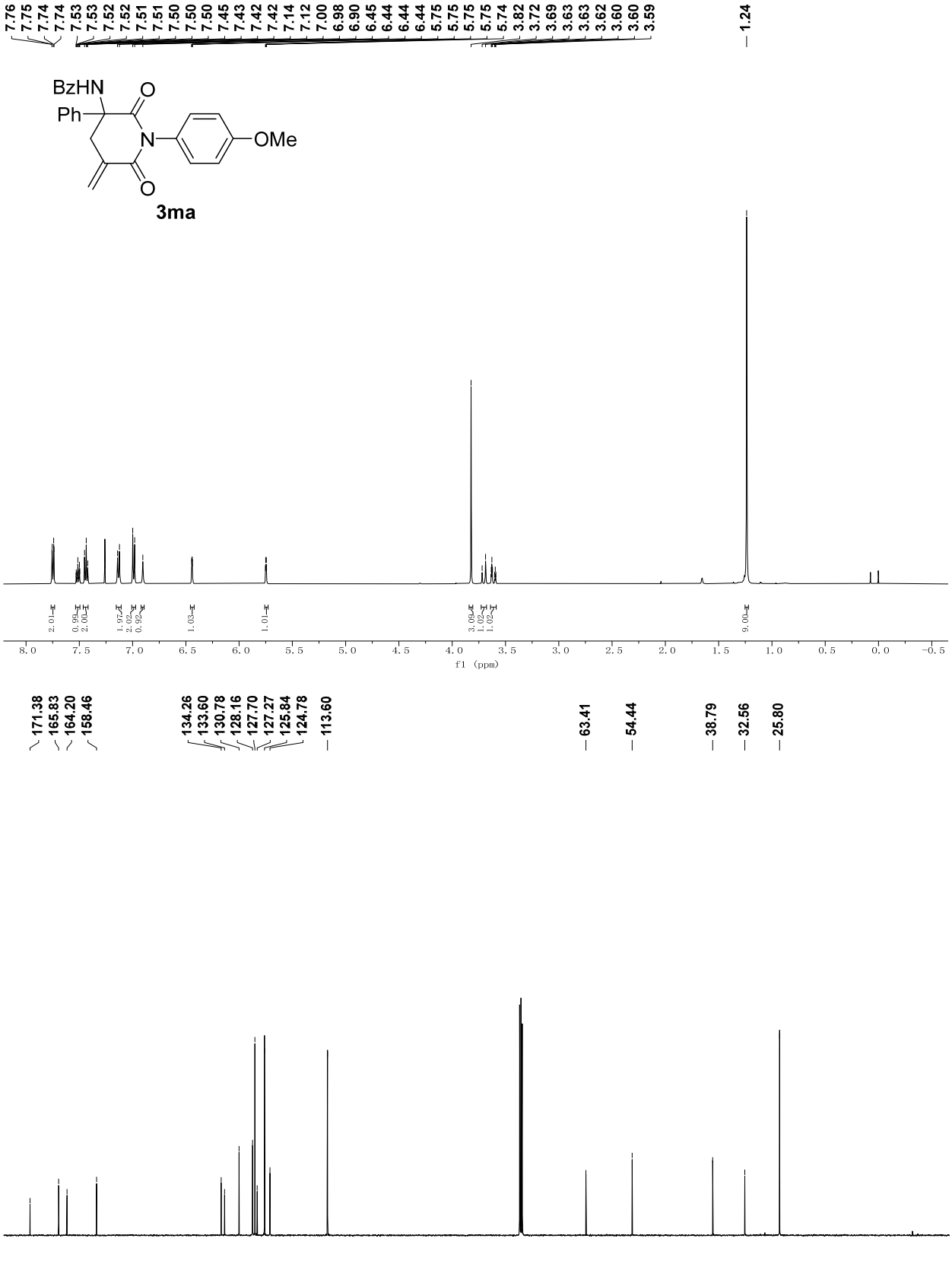
10
0
f1 (ppm)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7.14
7.12
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6.98
6.90
6.45
6.44
6.44
6.44
5.75
5.75
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3.69
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3.63
3.62
3.60
3.60
3.59
1.24
BzHN
Ph
O
N
OMe
O
3ma
2.01
0.96
2.00
1.97
2.02
0.92
1.03
1.01
3.09
1.02
1.02
9.00
f1 (ppm)
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158.46
134.26
133.60
130.78
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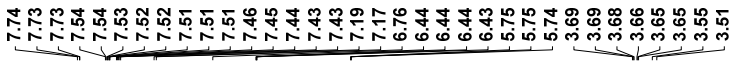
25.80
f1 (ppm)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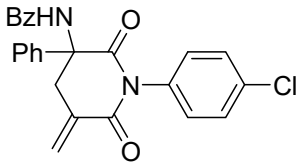
3.55
3.51
1.24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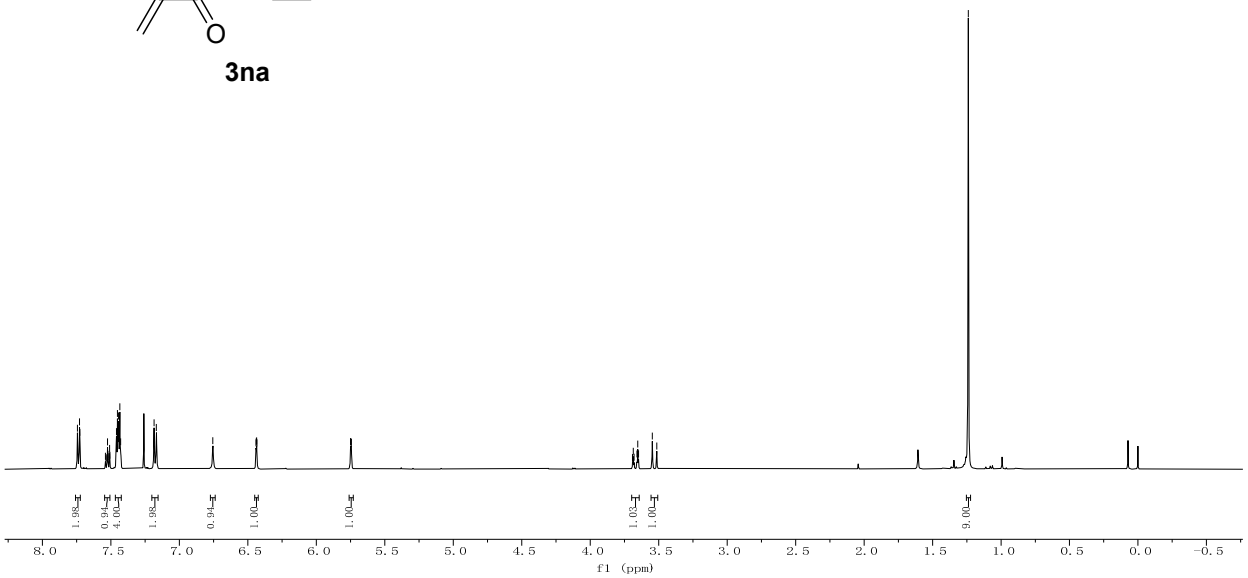
Ph
N
Cl
O

3na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1.00
1.00
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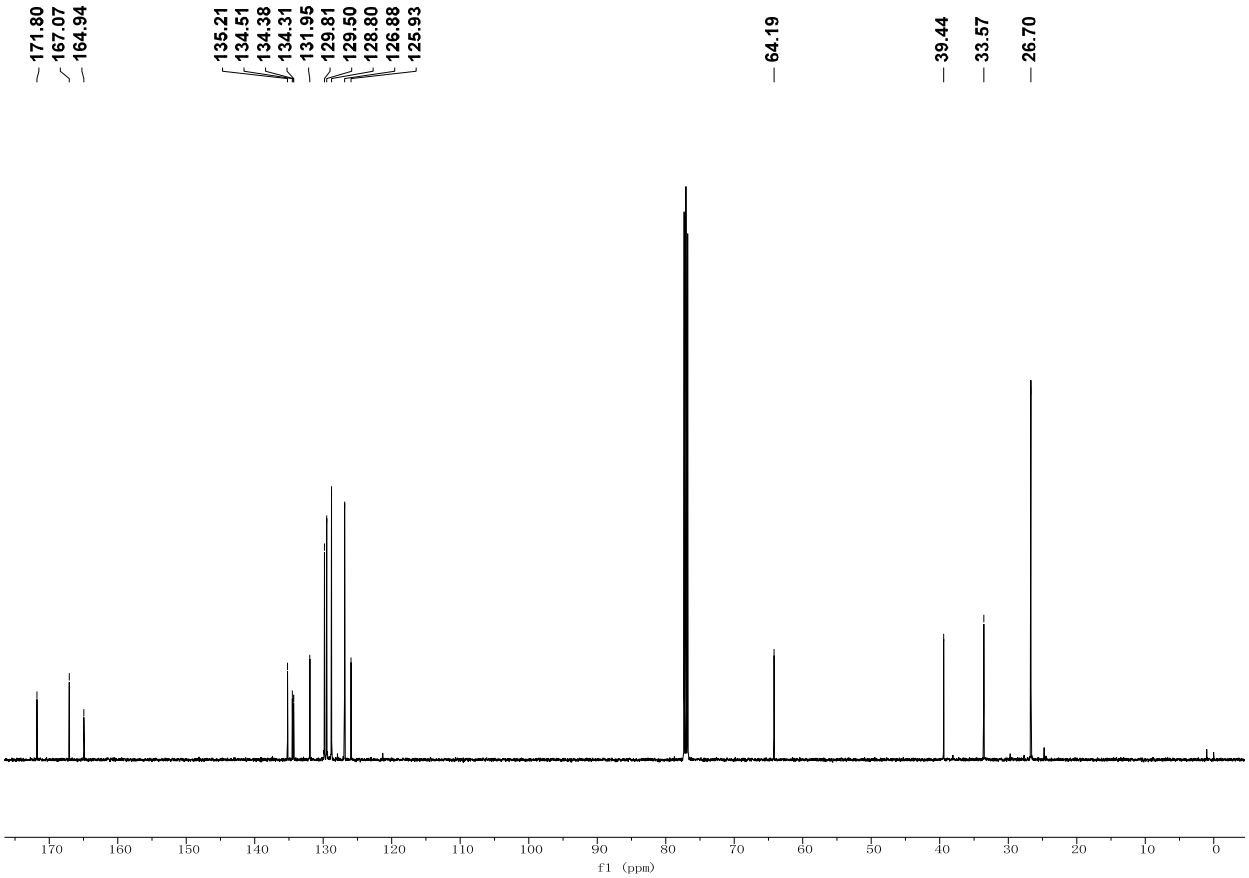
1.00
9.00
f1 (ppm)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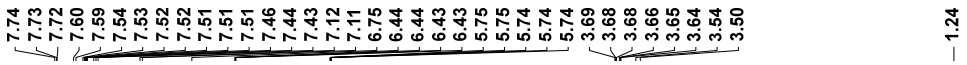
33.57
26.70
f1 (ppm)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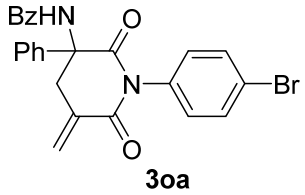
3.50
1.24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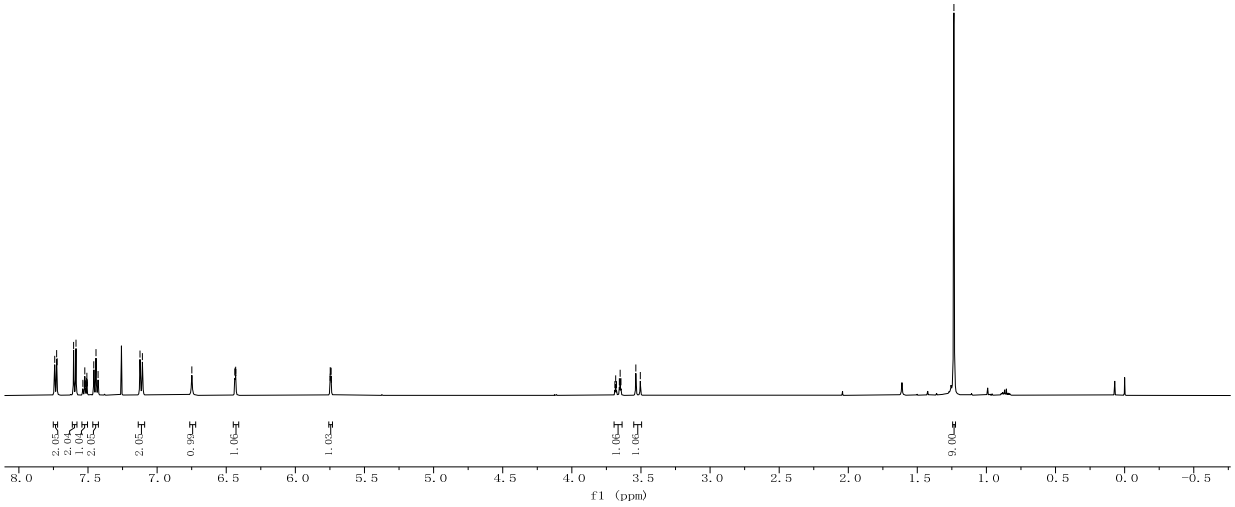
Ph
O
N
Br
O

3oa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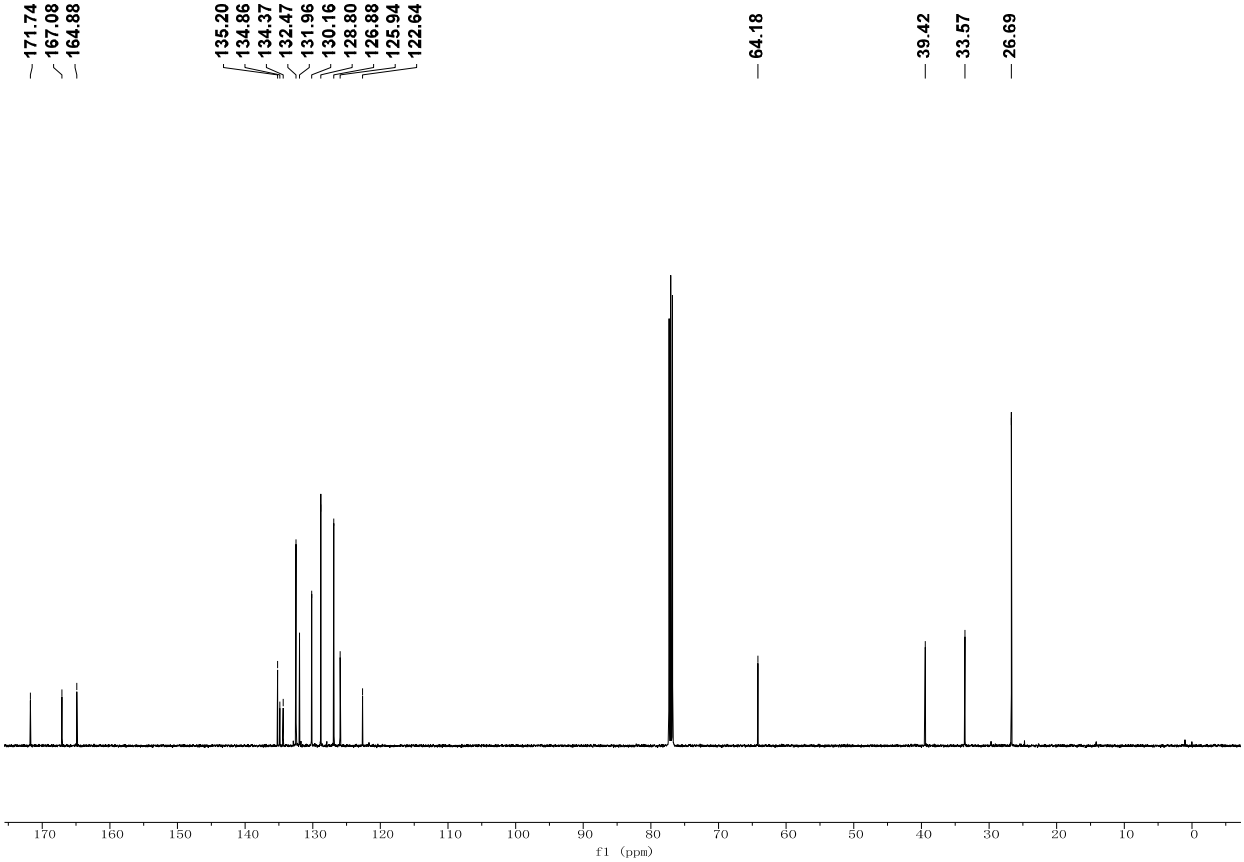
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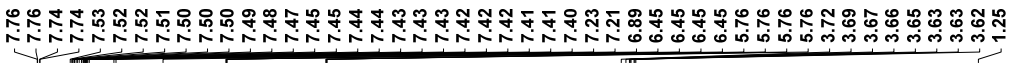
26.69
f1 (ppm)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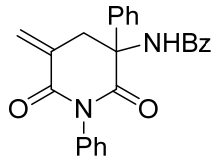
1.25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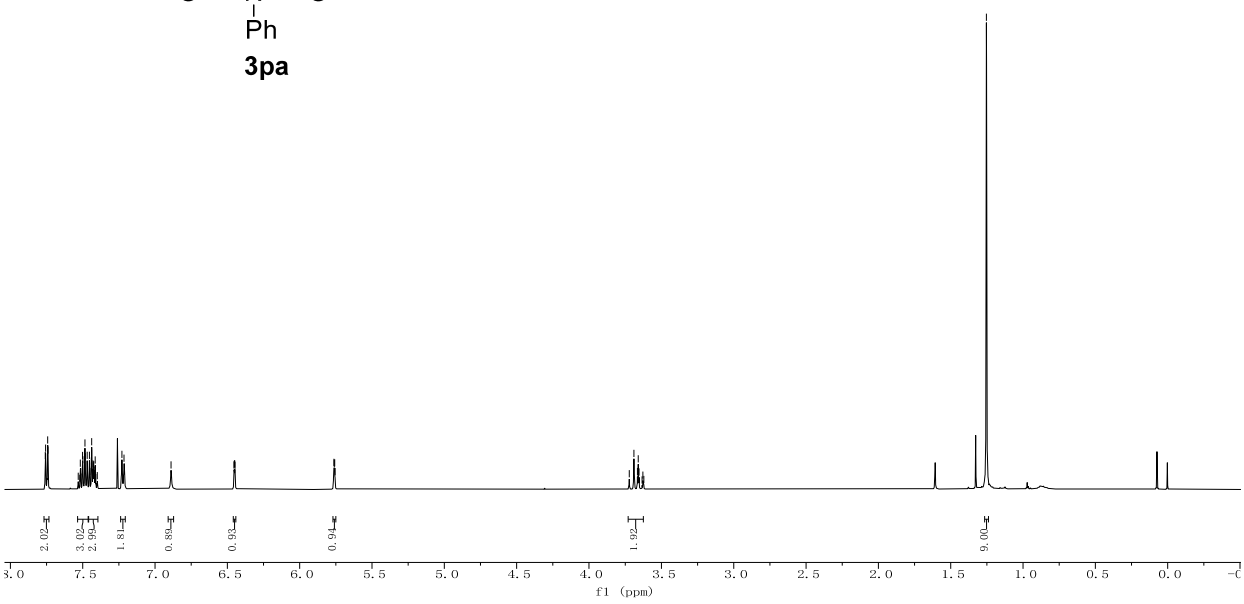
NHBz
O
N
O
Ph

3pa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0.93
0.94
1.92
9.00
f1 (ppm)


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134.79
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127.60
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125.85
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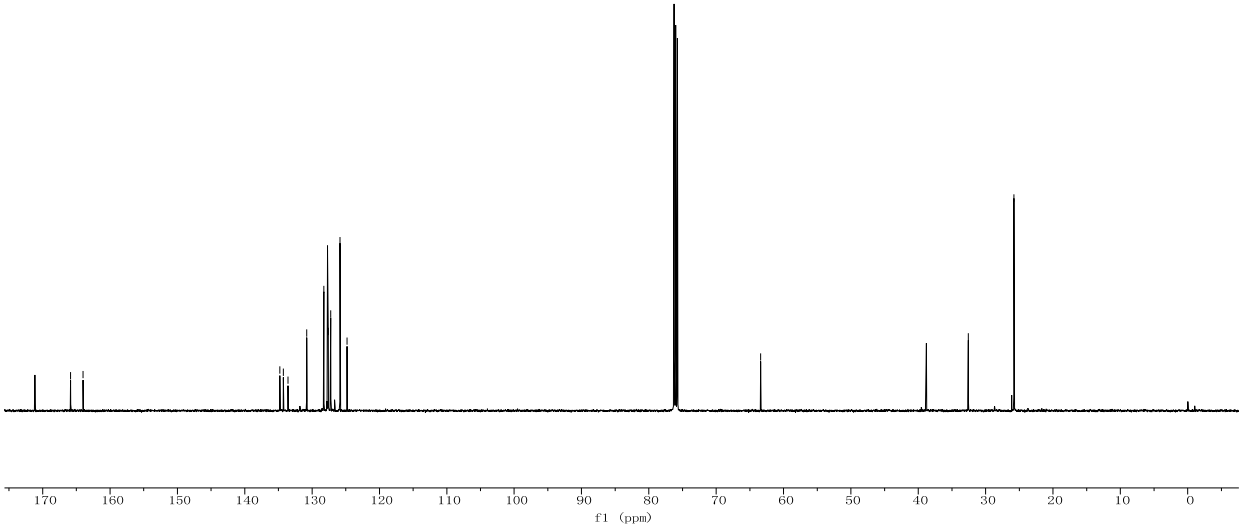
63.41
32.58
25.80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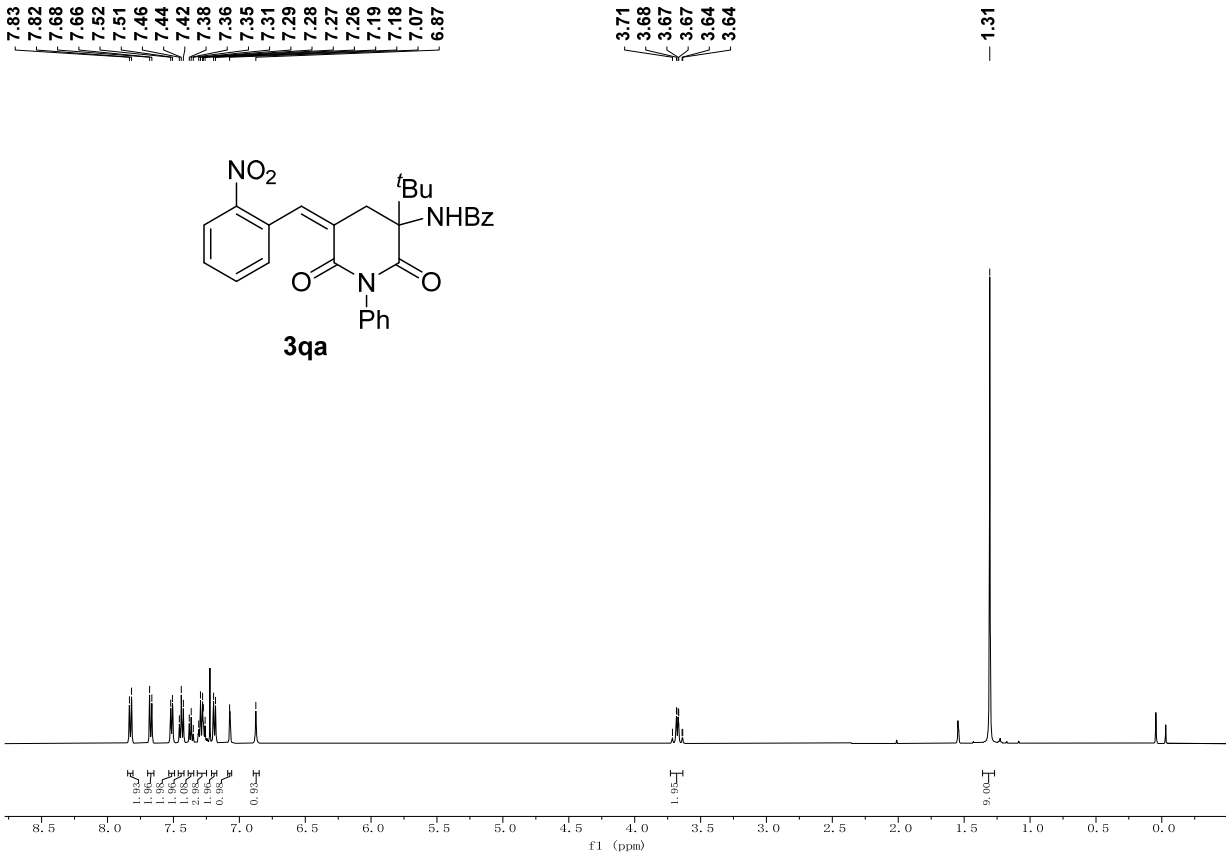
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7.31
7.29
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7.27
7.26
7.19
7.18
7.07
6.87
3.71
3.68
3.67
3.67
3.64
3.64
1.31
NO2
tBu
NHBz
O
N
O
Ph
3qa
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1.96
1.98
1.96
1.08
2.98
1.96
0.98
0.93
1.95
9.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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3.0
2.5
2.0
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1.0
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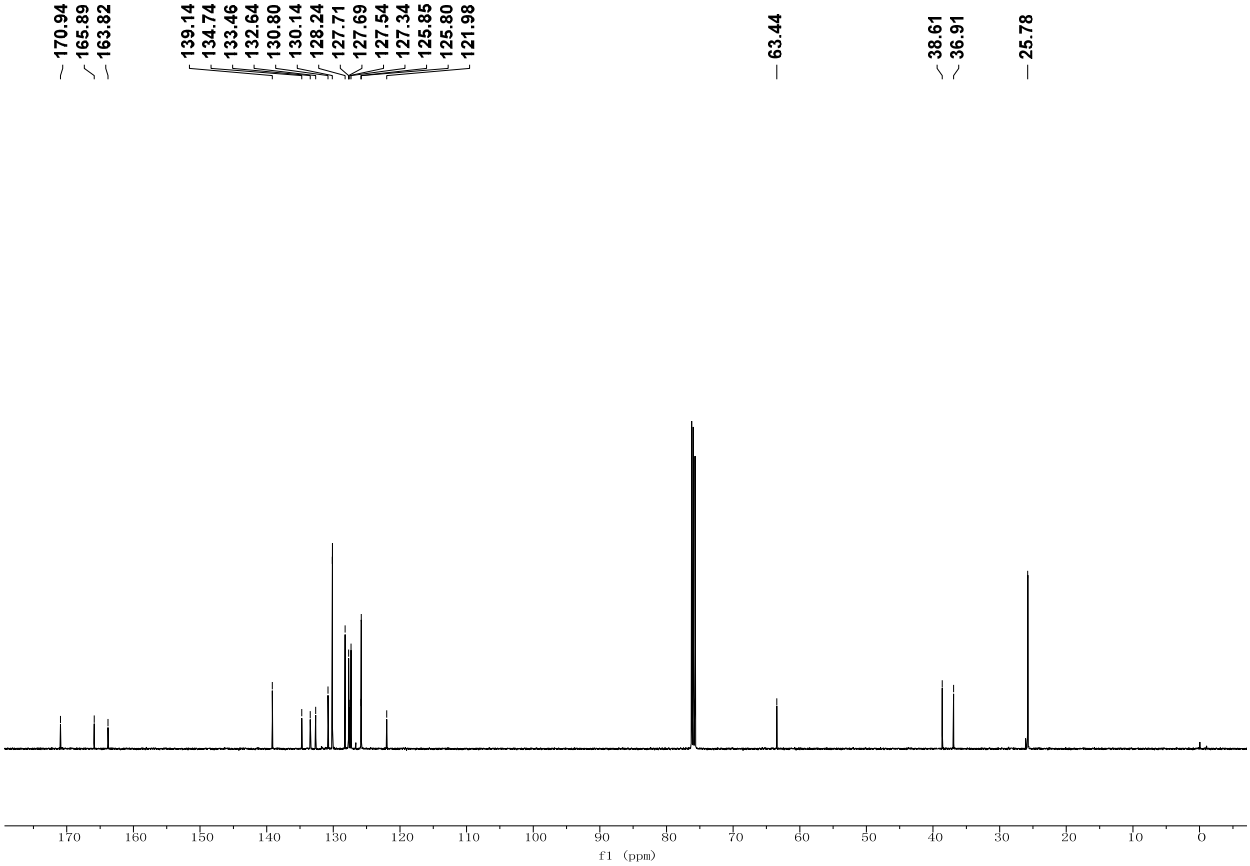
0.0
f1 (ppm)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127.71
127.69
127.54
127.34
125.85
125.80
121.98
63.44
38.61
36.91
25.78
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130
120
110
100
90
80
70
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50
40
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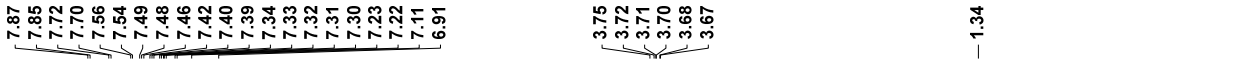
0
f1 (ppm)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7.33
7.32
7.31
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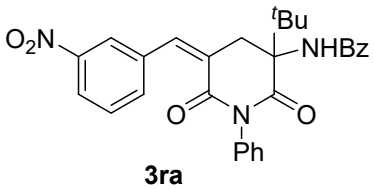
3.75
3.72
3.71
3.70
3.68
3.67
1.34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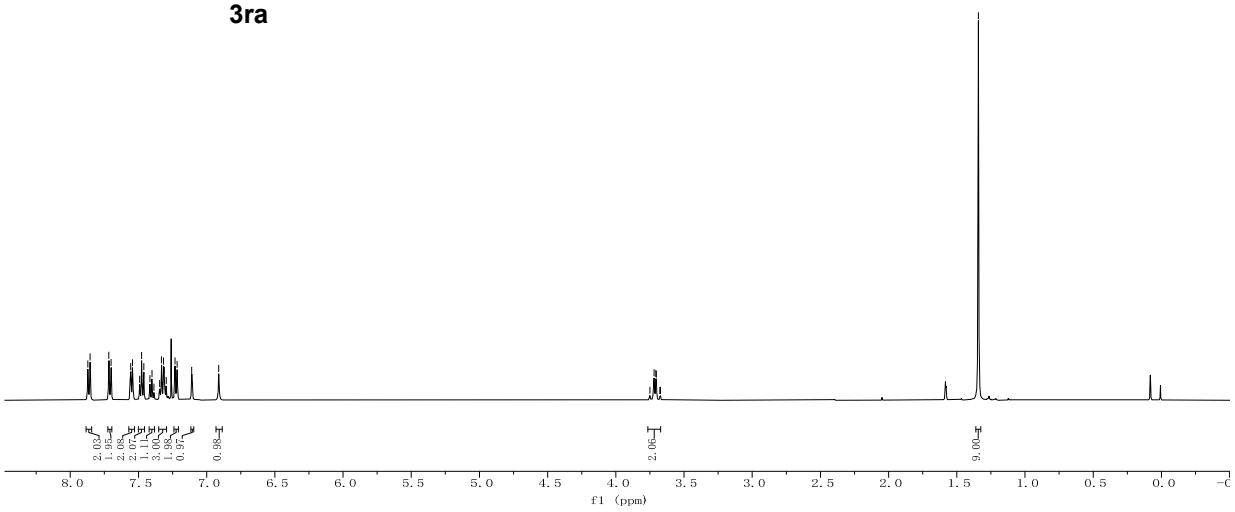
O
Ph

3ra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1.11
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1.98
0.97
0.98
2.06
9.00
f1 (ppm)


170.76
166.00
163.74
140.88
133.97
133.62
133.39
131.42
130.87
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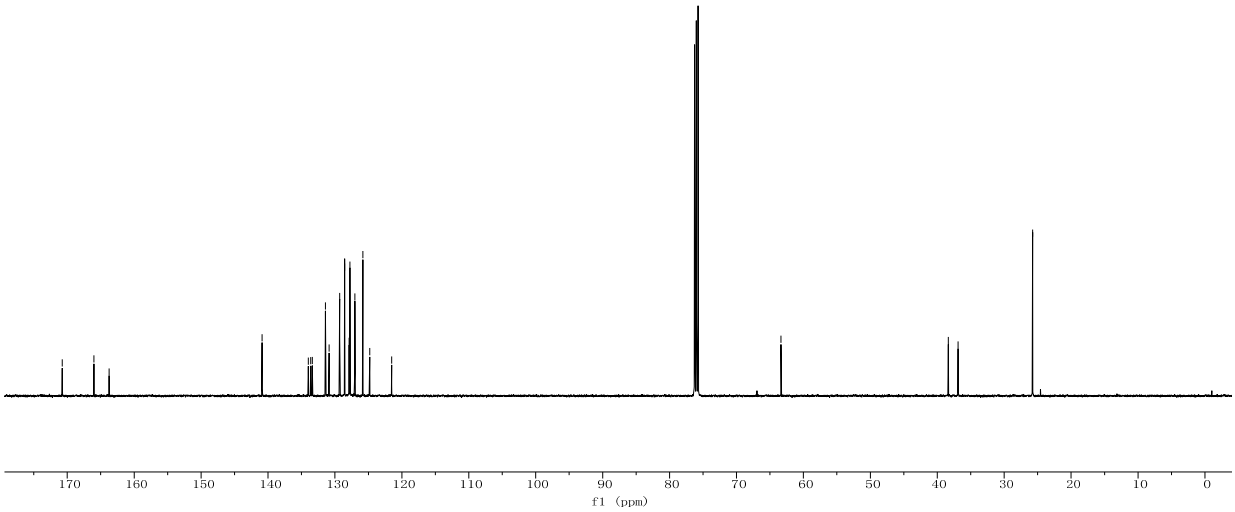
38.34
36.89
25.76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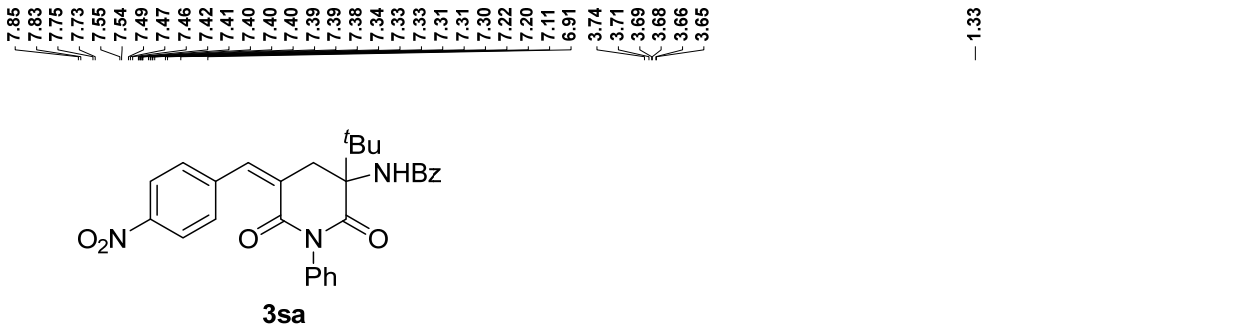
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7.11
6.91
3.74
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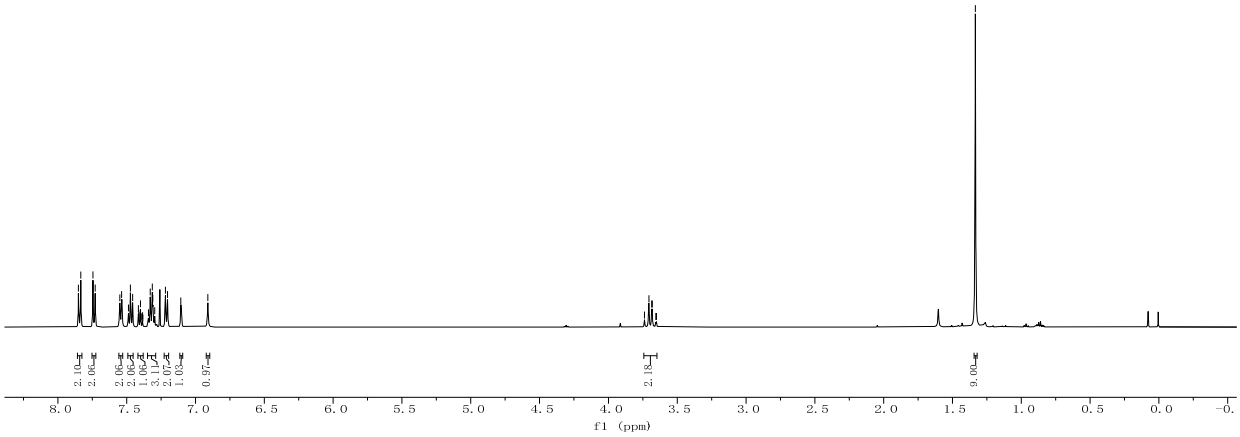
1.33

tBu
NHBz
O2N
O
N
O
Ph

3sa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2.06
2.06
1.06
3.11
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6.5
6.0
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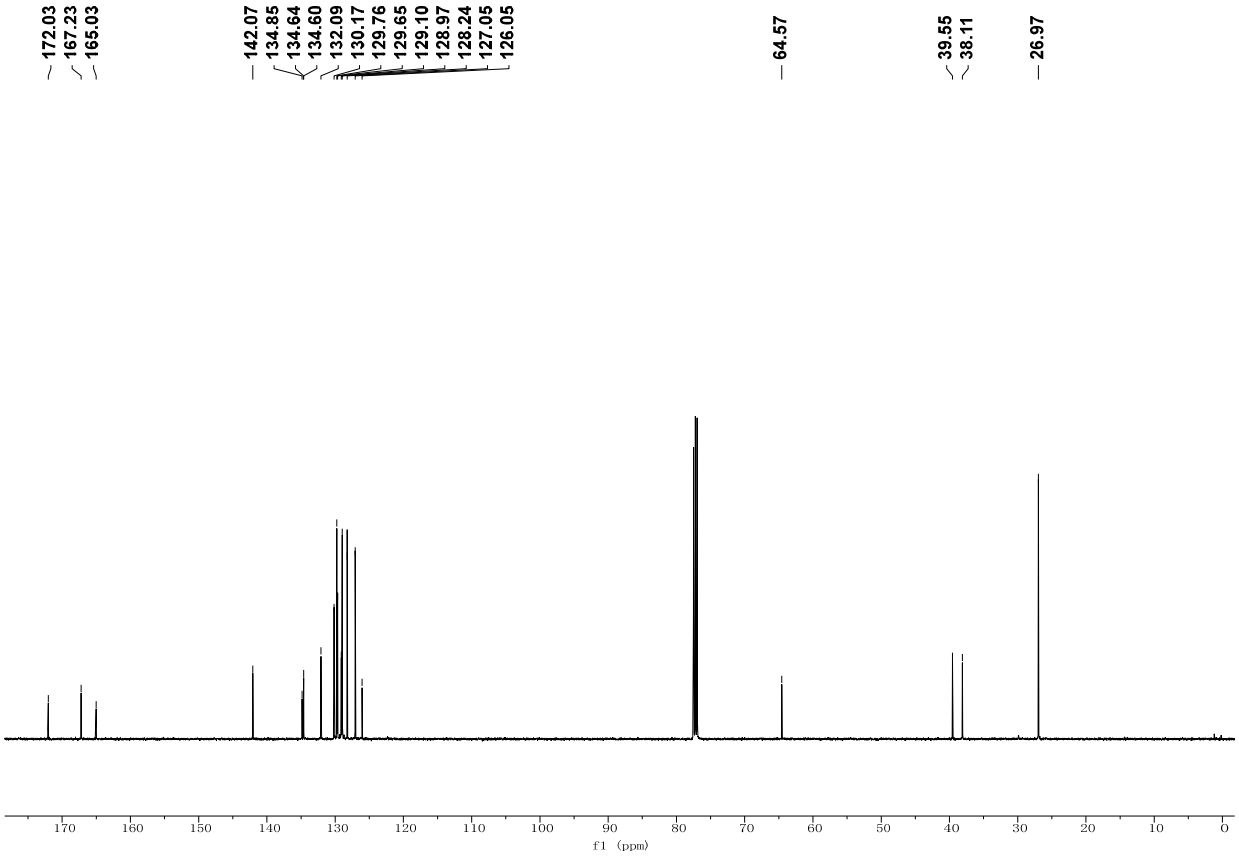
0.0
f1 (ppm)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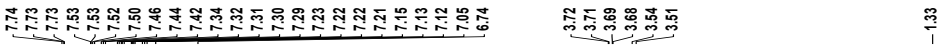
f1 (ppm)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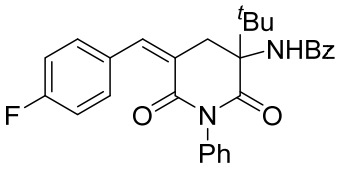
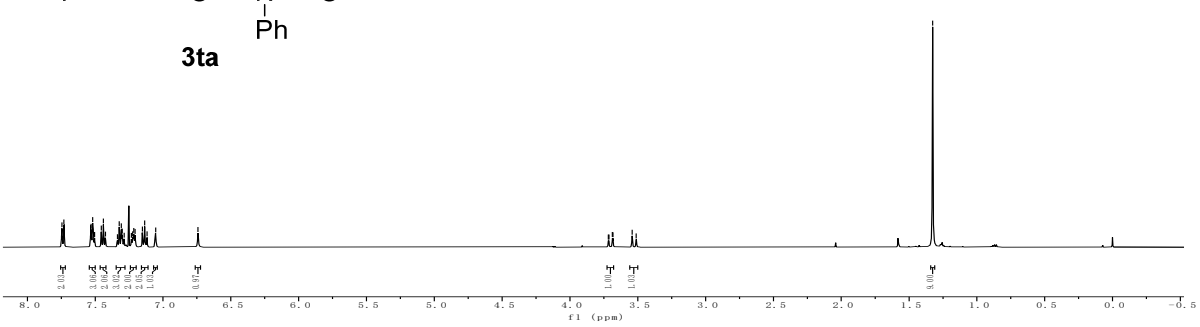
3.51
1.33


tBu
NHBz
F
O
N
O
Ph

3ta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2.06
3.02
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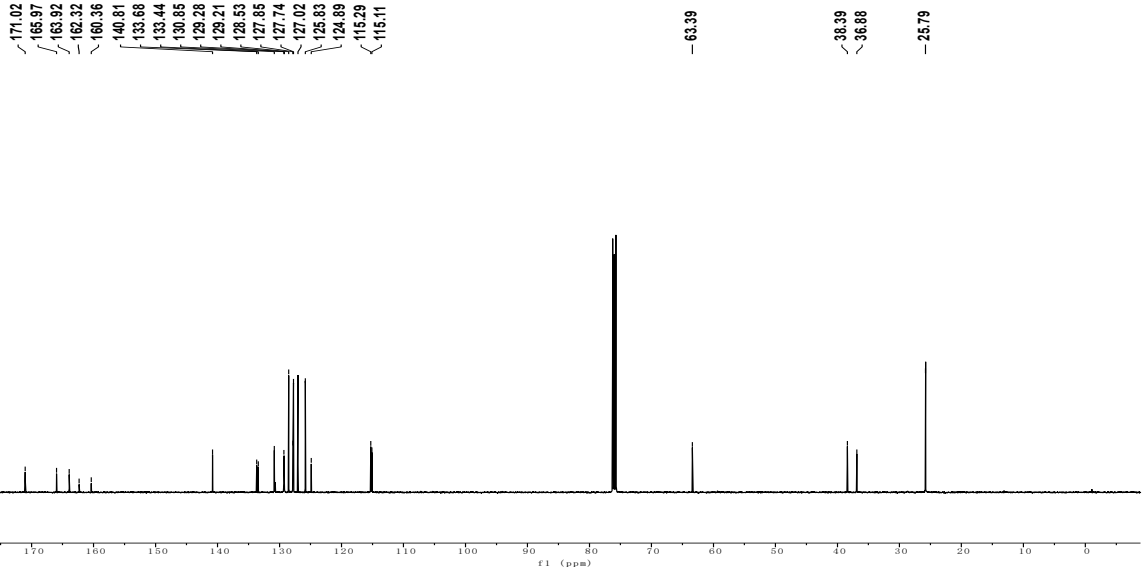
2.03
1.03
0.97
1.00
1.03
9.00
f1 (ppm)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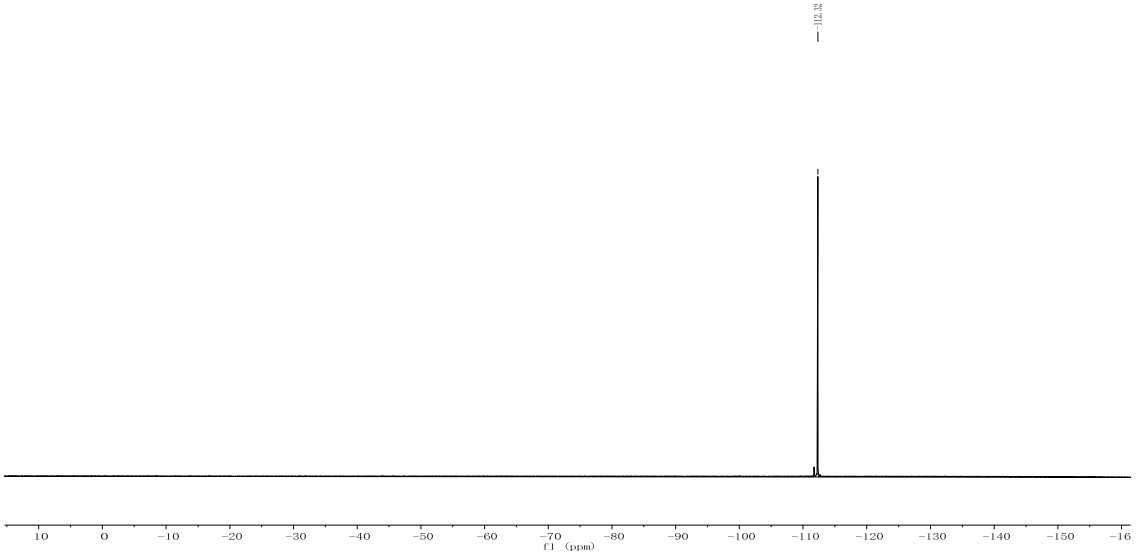
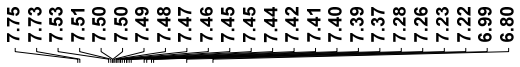
160.36
140.81
133.68
133.44
130.85
129.28
129.21
128.53
127.85
127.74
127.02
125.83
124.89
115.29
115.11
63.39
38.39
36.88
25.79
f1 (ppm)


-112.32
f1 (ppm)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7.73
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7.51
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7.49
7.48
7.47
7.46
7.45
7.45
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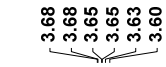
7.28
7.26
7.23
7.22
6.99
6.80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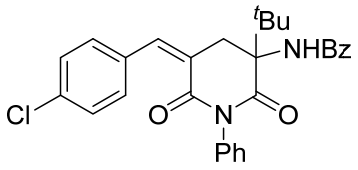
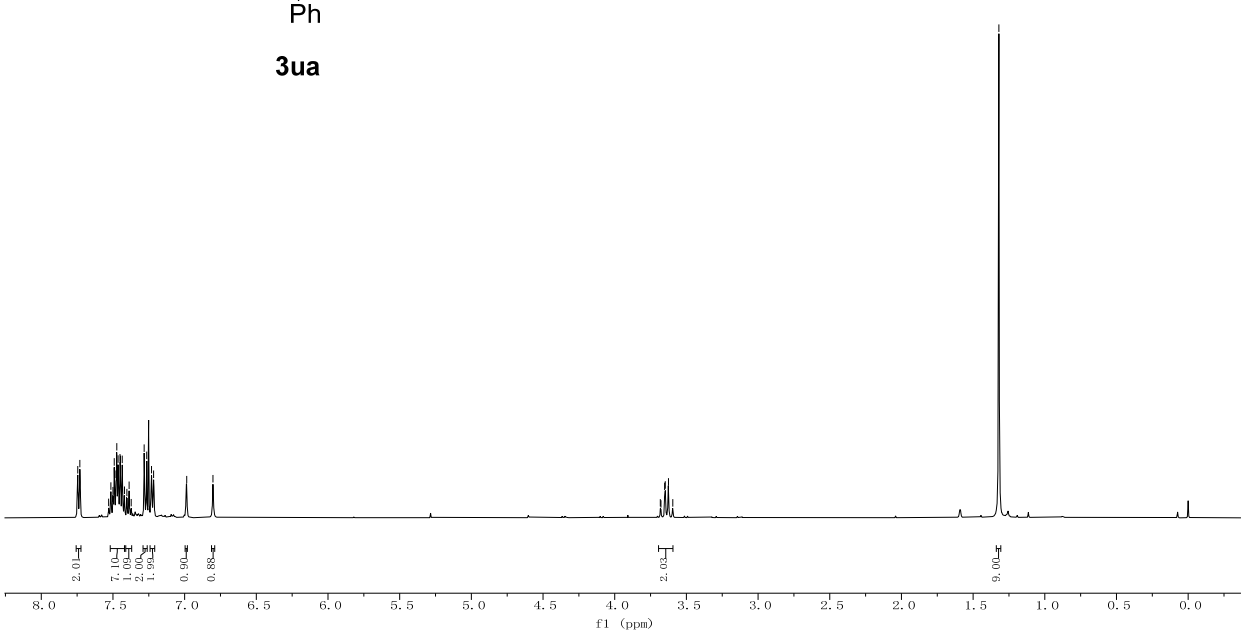
3.68
3.65
3.65
3.63
3.60


1.32


tBu
NHBz
Cl
O
N
O
Ph

3ua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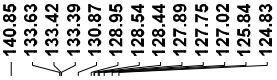
7.10
1.00
2.00
1.99
0.90
0.88
2.03
9.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


170.81
166.01
163.81


140.85
133.63
133.42
133.39
130.87
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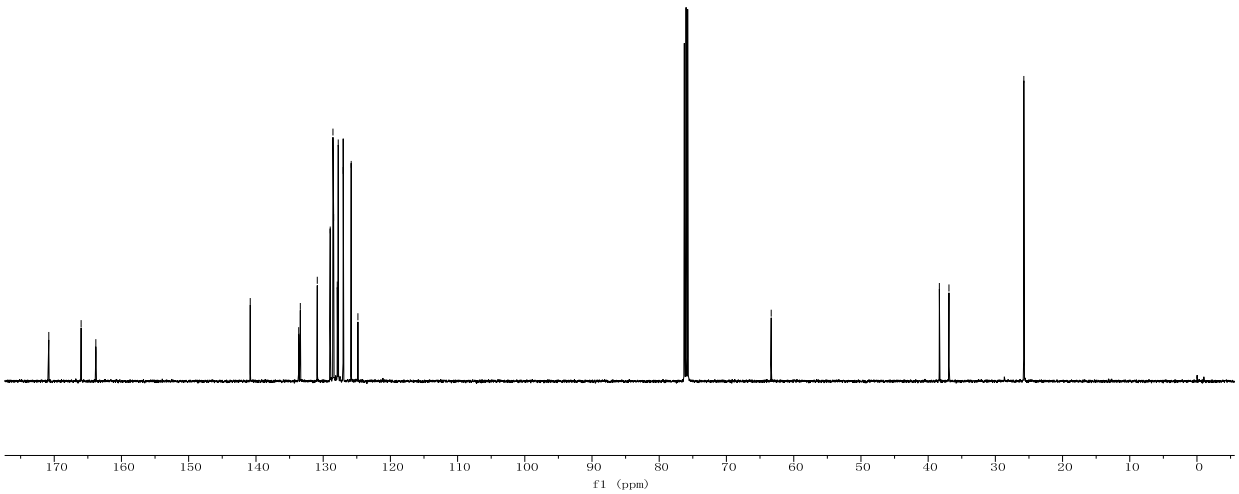
128.44
127.89
127.75
127.02
125.84
124.83


63.35


38.33
36.89


25.75


170
160
150
140
130
120
110
100
90
80
70
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50
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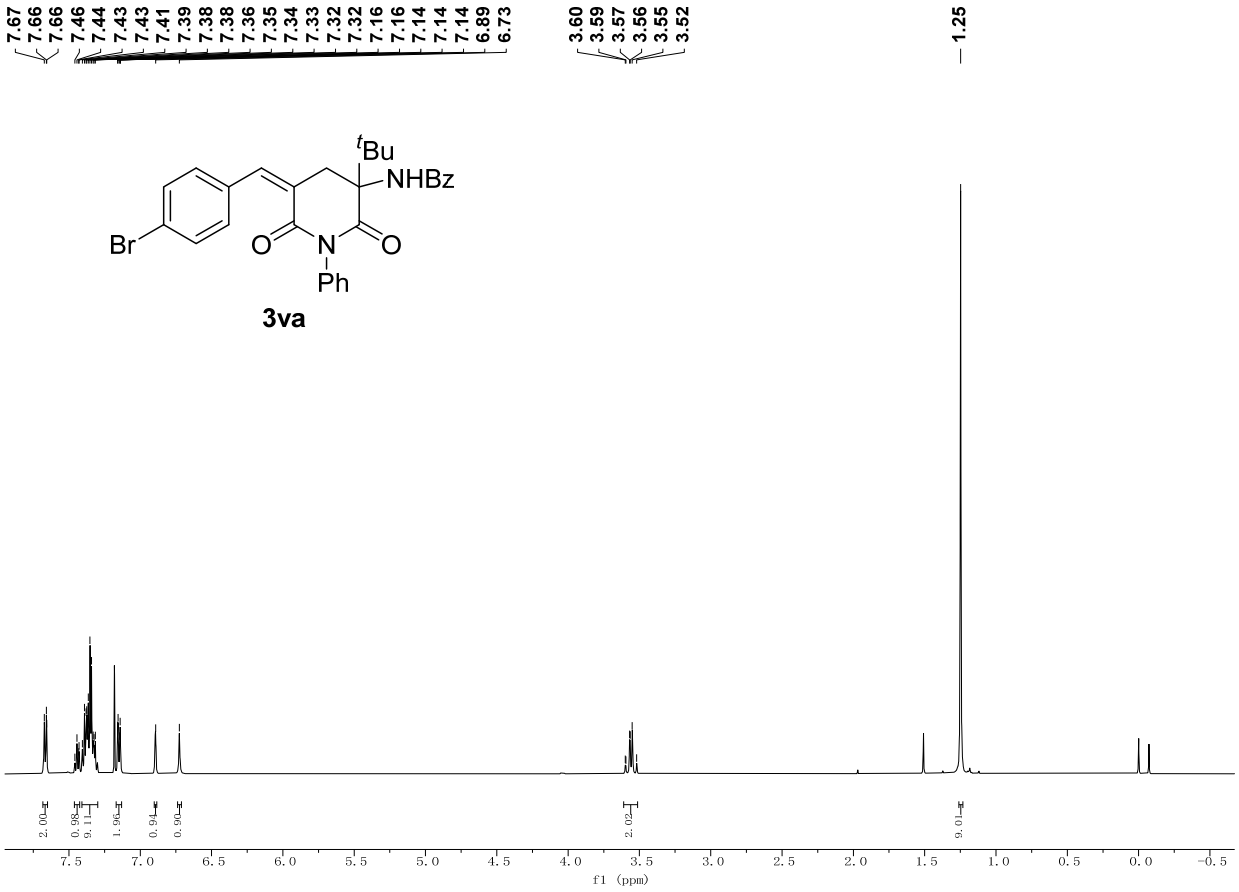
30
20
10
0
f1 (ppm)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7.66
7.66
7.46
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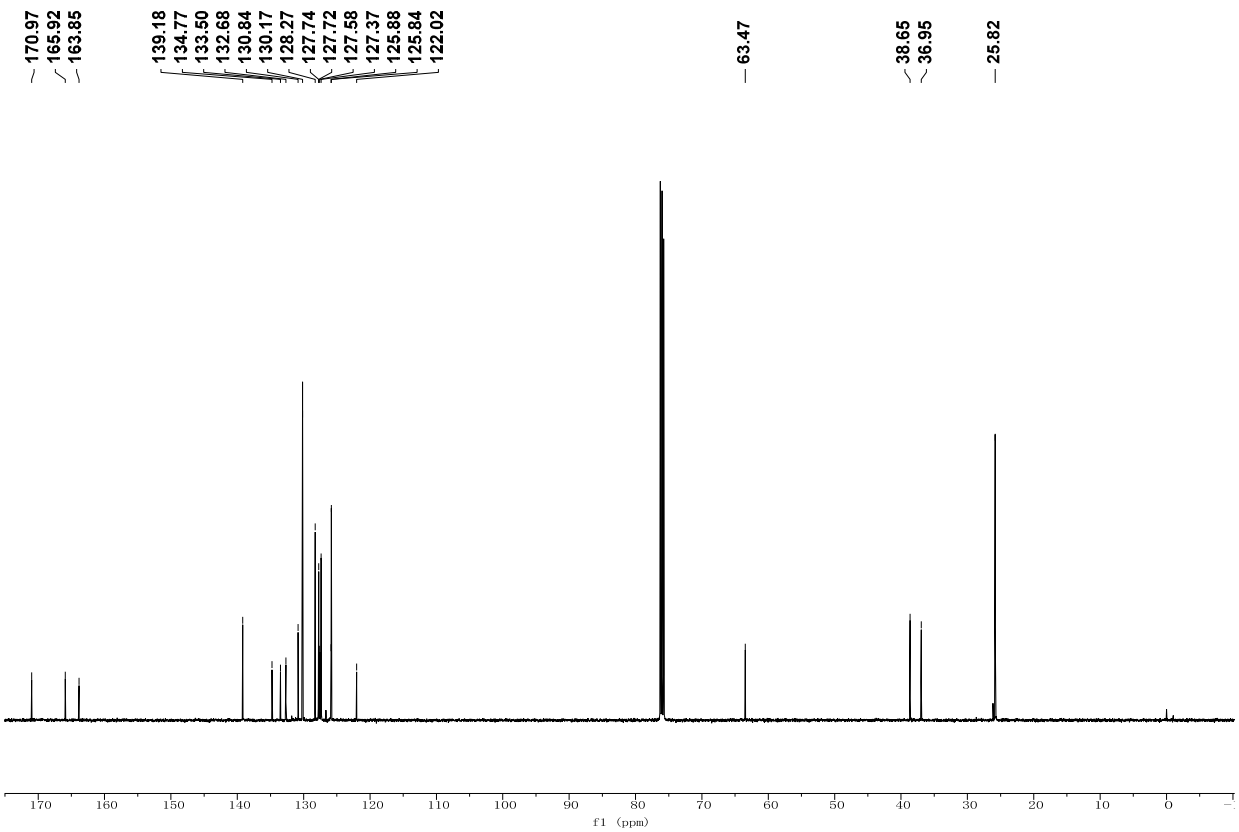
7.43
7.43
7.41
7.39
7.38
7.38
7.36
7.35
7.34
7.33
7.32
7.32
7.16
7.16
7.14
7.14
7.14
6.89
6.73
3.60
3.59
3.57
3.56
3.55
3.52
1.25
tBu
NHBz
Br
O
N
O
Ph
2.00
0.98
9.11
1.96
0.94
0.90
2.02
9.01
f1 (ppm)

3va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165.92
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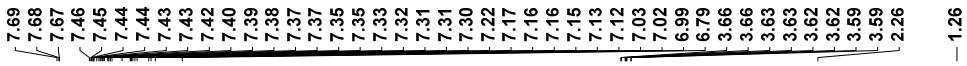
139.18
134.77
133.50
132.68
130.84
130.17
128.27
127.74
127.72
127.58
127.37
125.88
125.84
122.02
63.47
38.65
36.95
25.82
f1 (ppm)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7.67
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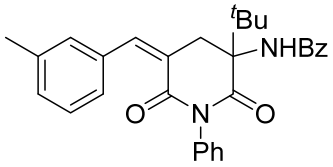
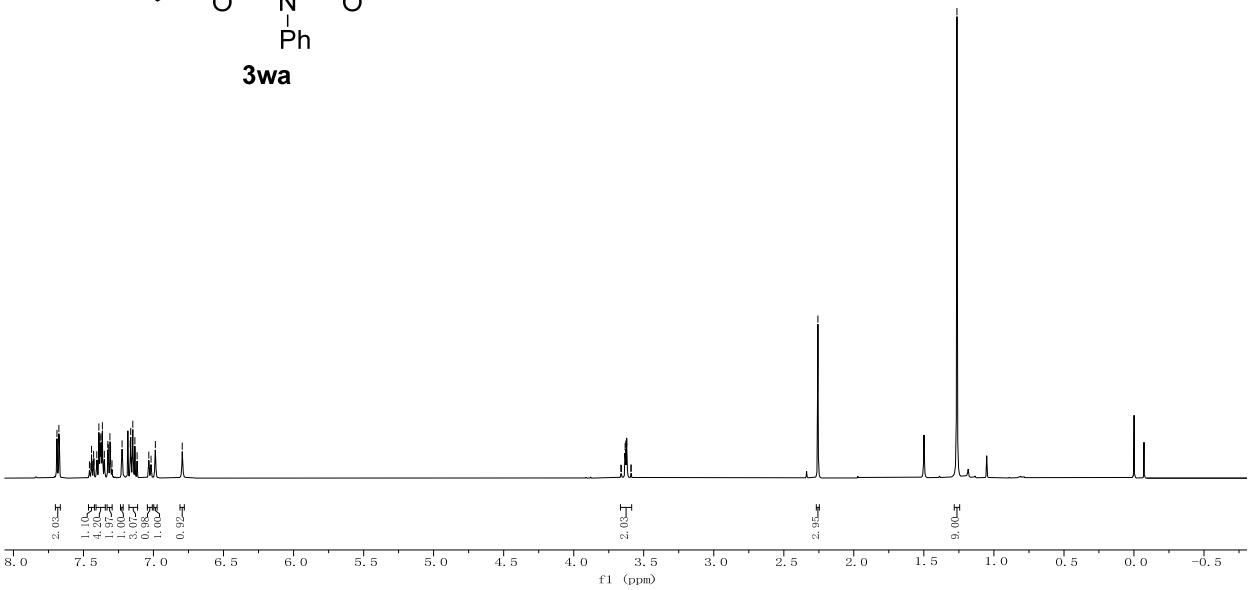
7.45
7.44
7.44
7.43
7.43
7.42
7.40
7.39
7.38
7.37
7.37
7.35
7.35
7.33
7.32
7.31
7.31
7.30
7.22
7.17
7.16
7.16
7.15
7.13
7.12
7.03
7.02
6.99
6.79
3.66
3.66
3.63
3.63
3.62
3.62
3.59
3.59
2.26
1.26


tBu
NHBz
O
N
O
Ph

3wa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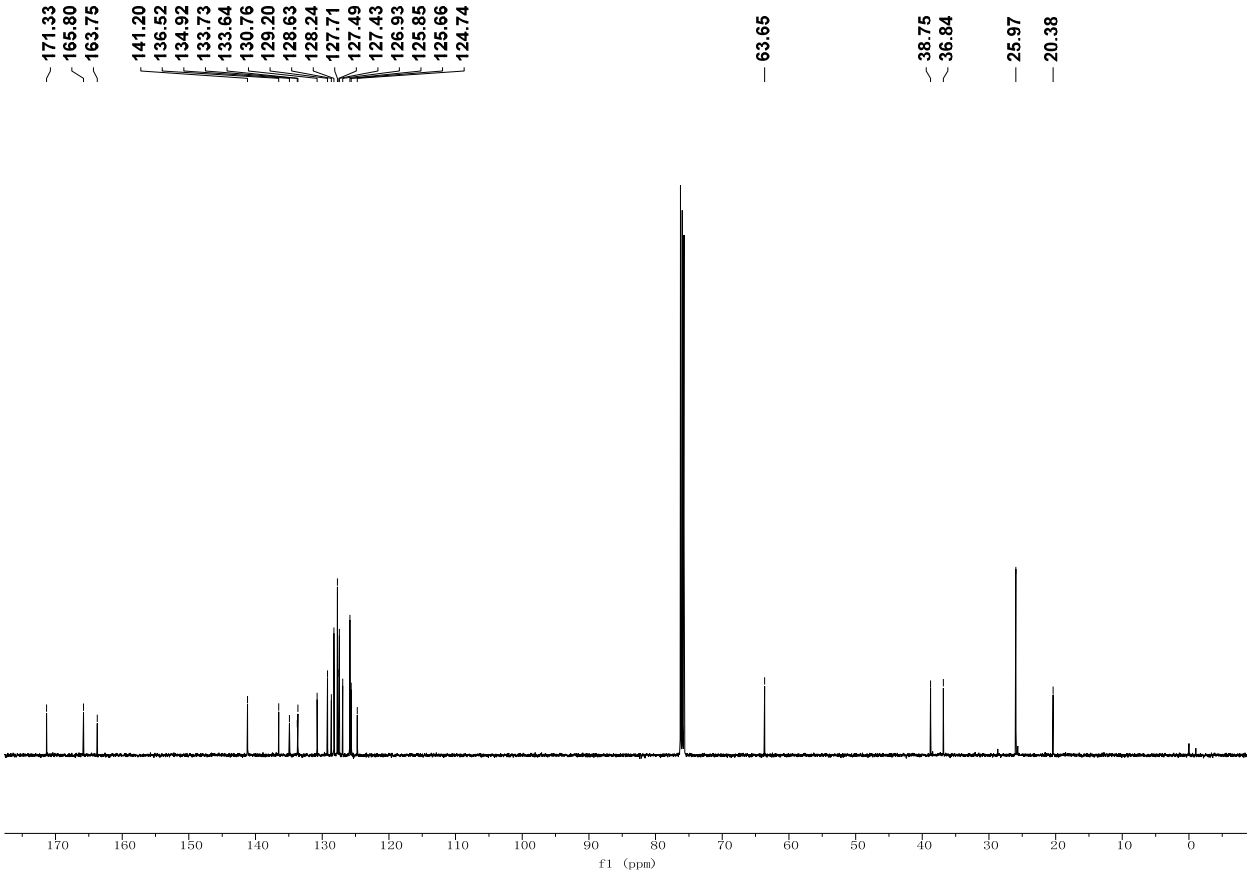
1.10
4.20
1.97
1.00
3.07
0.98
1.00
0.92
2.03
2.95
9.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)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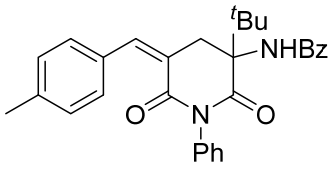
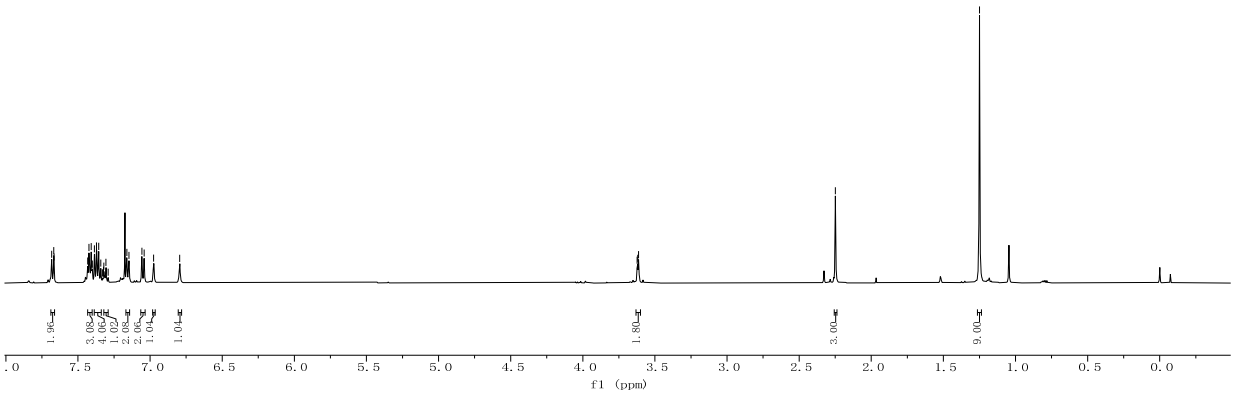
134.92
133.73
133.64
130.76
129.20
128.63
128.24
127.71
127.49
127.43
126.93
125.85
125.66
124.74
63.65
38.75
36.84
25.97
20.38
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.68
7.67
7.67
7.43
7.42
7.41
7.40
7.39
7.37
7.36
7.34
7.33
7.32
7.32
7.31
7.29
7.16
7.15
7.06
7.04
6.98
6.79
3.62
3.62
3.61
2.25
1.25


tBu
NHBz
O
N
O
Ph

3xa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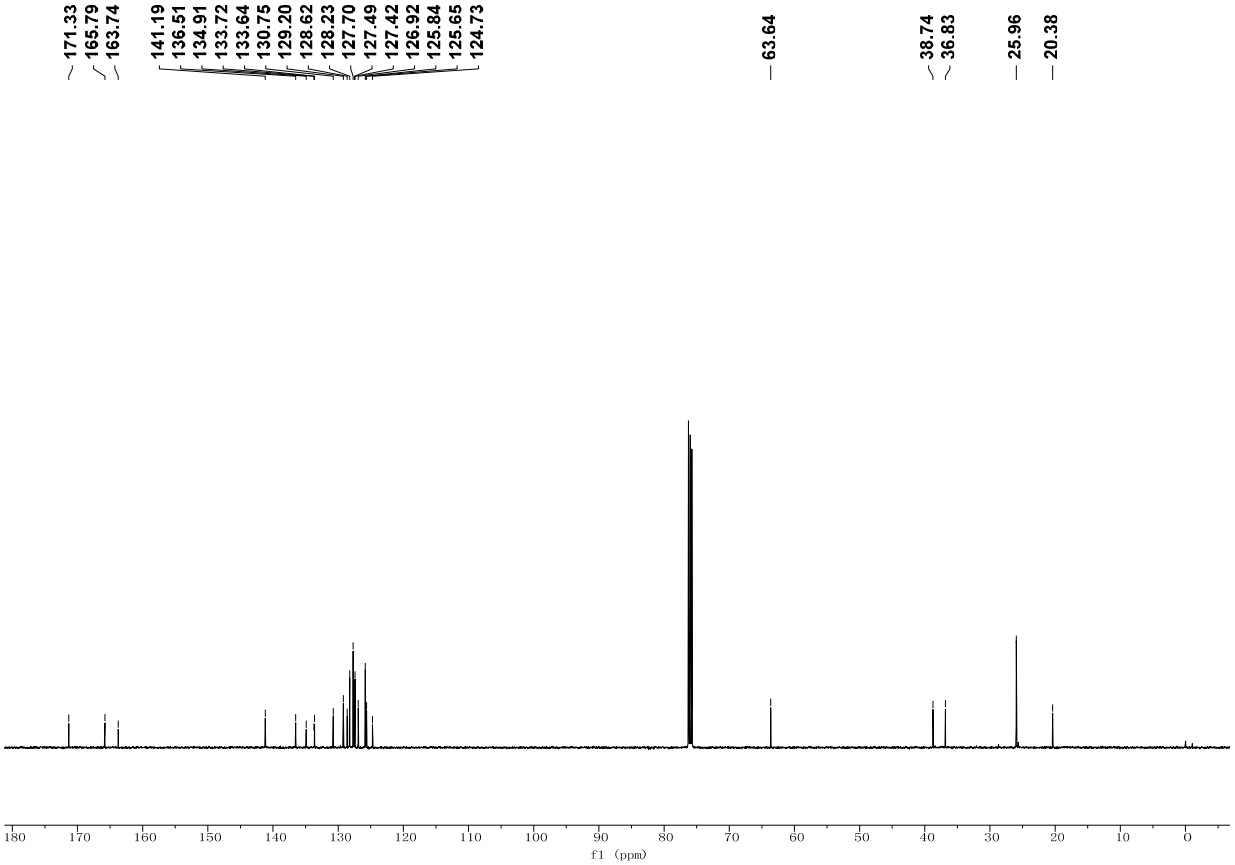
3.08
4.06
1.02
2.08
2.06
1.04
1.04
1.80
3.00
9.00
f1 (ppm)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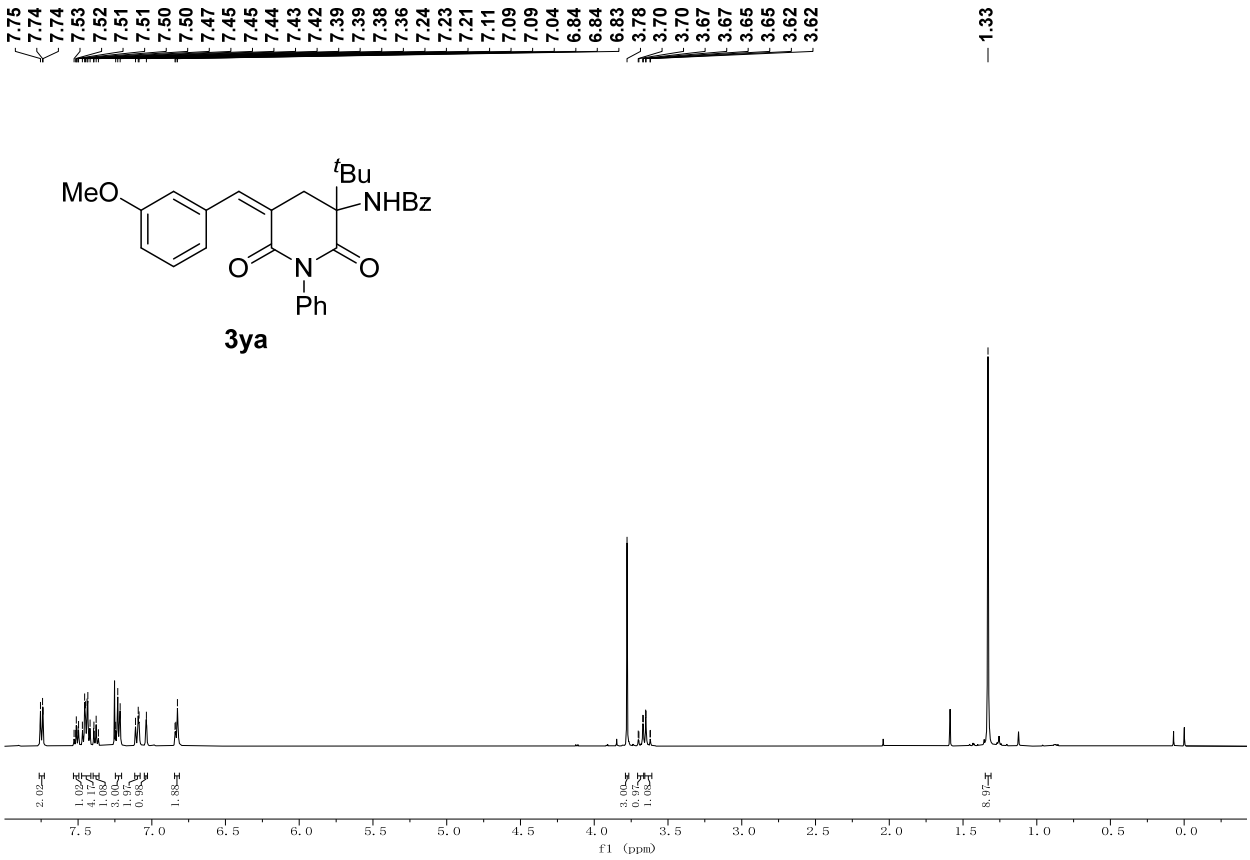
134.91
133.72
133.64
130.75
129.20
128.62
128.23
127.70
127.49
127.42
126.92
125.84
125.65
124.73
63.64
38.74
36.83
25.96
20.38
f1 (ppm)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7.50
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7.45
7.45
7.44
7.43
7.42
7.39
7.39
7.38
7.36
7.24
7.23
7.21
7.11
7.09
7.09
7.04
6.84
6.84
6.83
3.78
3.70
3.70
3.67
3.67
3.65
3.65
3.62
3.62
1.33
MeO
tBu
NHBz
O
N
O
Ph
3ya
2.02
1.02
4.17
1.08
3.00
1.97
0.98
1.88
3.00
0.97
1.08
8.97
f1 (ppm)
172.24
166.87
164.82
159.18
141.44
136.20
135.96
134.63
131.82
129.27
129.06
128.76
128.52
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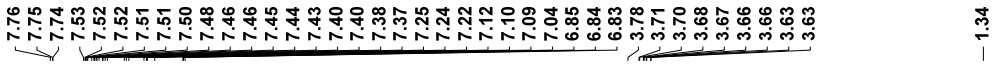
126.45
121.99
114.99
114.35
64.65
55.26
39.73
37.93
26.94
f1 (ppm)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7.51
7.50
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7.46
7.45
7.44
7.43
7.40
7.40
7.38
7.37
7.25
7.24
7.22
7.12
7.10
7.09
7.04
6.85
6.84
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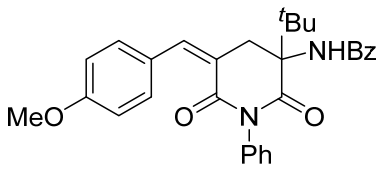
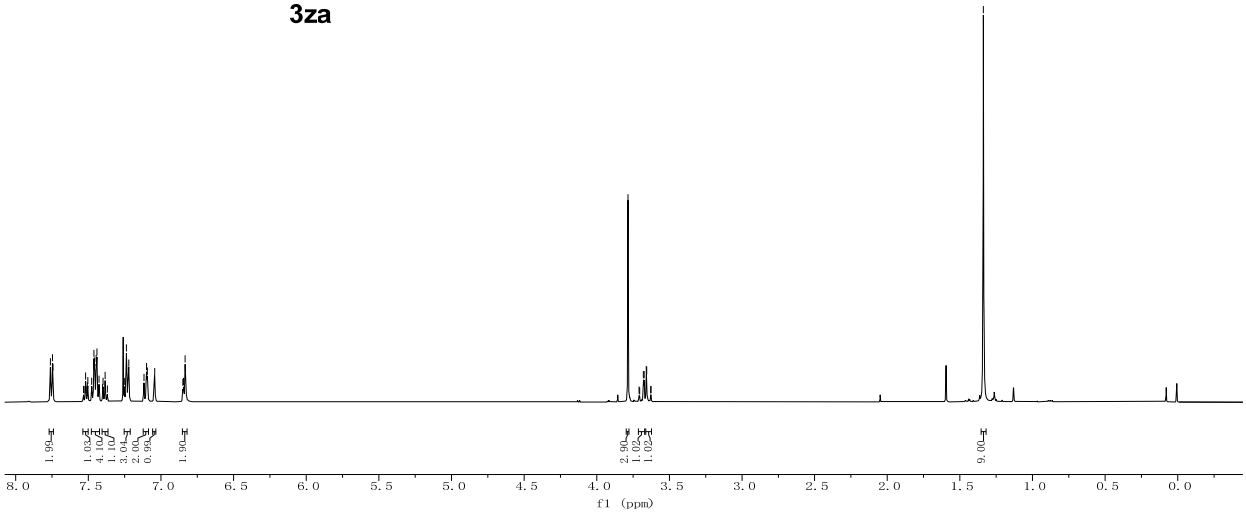
3.67
3.66
3.66
3.63
3.63
1.34


tBu
NHBz
MeO
O
N
O
Ph

3za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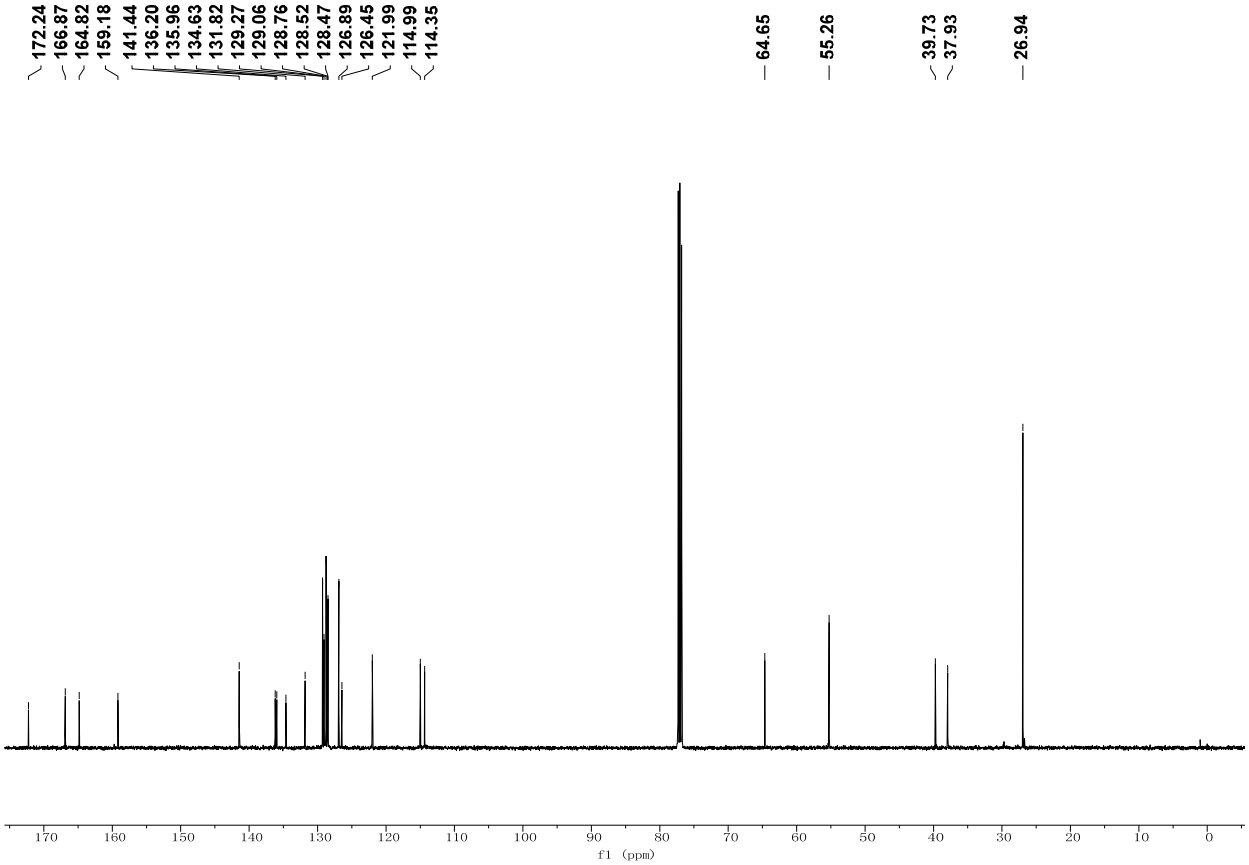
4.10
1.10
3.04
2.00
0.95
1.90
2.90
1.02
1.02
9.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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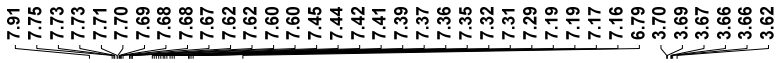
164.82
159.18
141.44
136.20
135.96
134.63
131.82
129.27
129.06
128.76
128.52
128.47
126.89
126.45
121.99
114.99
114.35
64.65
55.26
39.73
37.93
26.94
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.60
7.45
7.44
7.42
7.41
7.39
7.37
7.36
7.35
7.32
7.31
7.29
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7.19
7.17
7.16
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3.69
3.67
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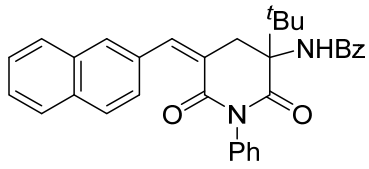
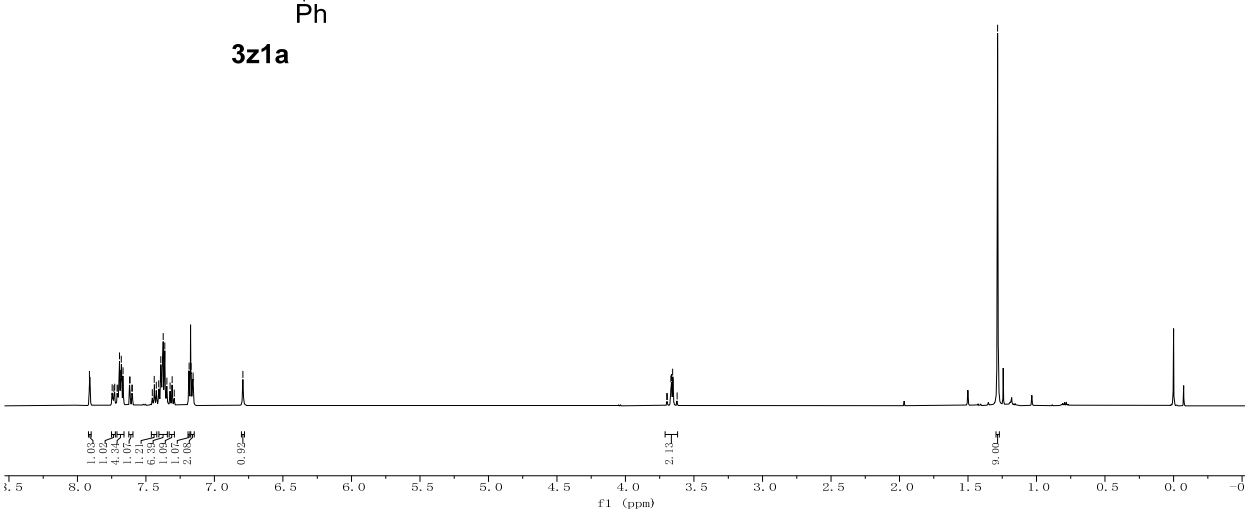
3.66
3.62
1.28


tBu
NHBz
O
N
O
Ph

3z1a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1.02
4.34
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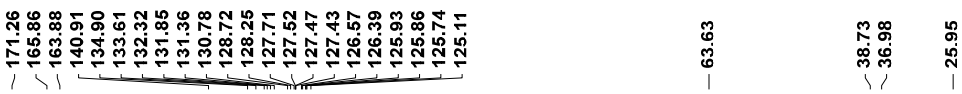
1.21
6.84
1.00
1.07
2.08
0.92
2.13
9.00
f1 (ppm)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133.61
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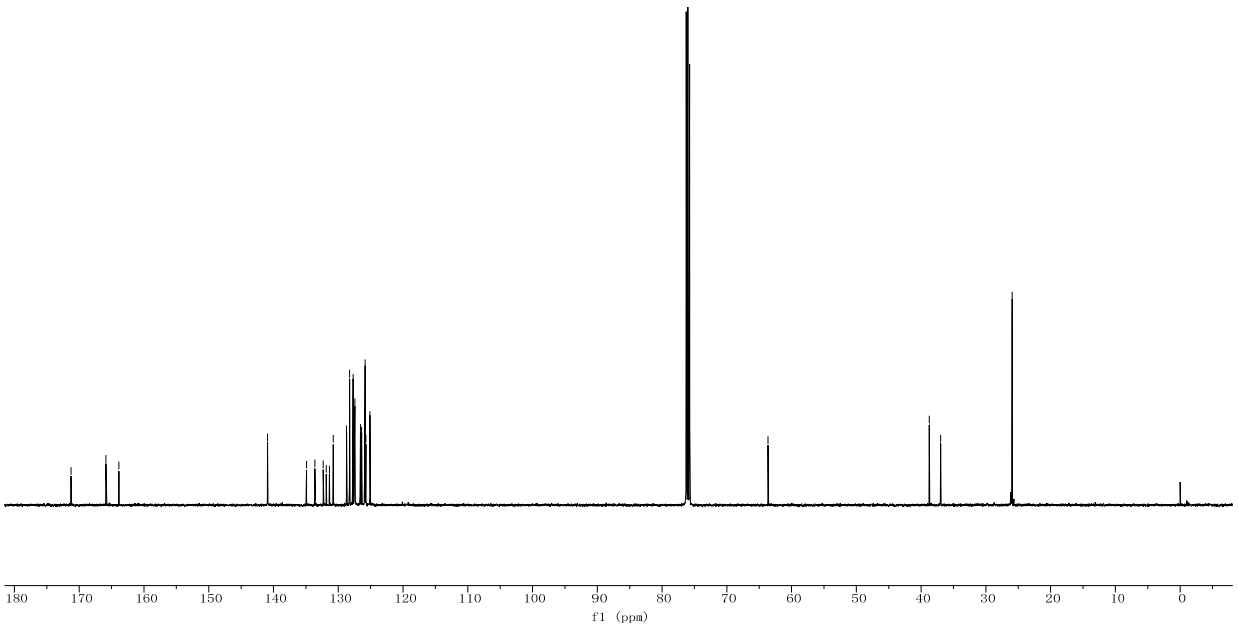
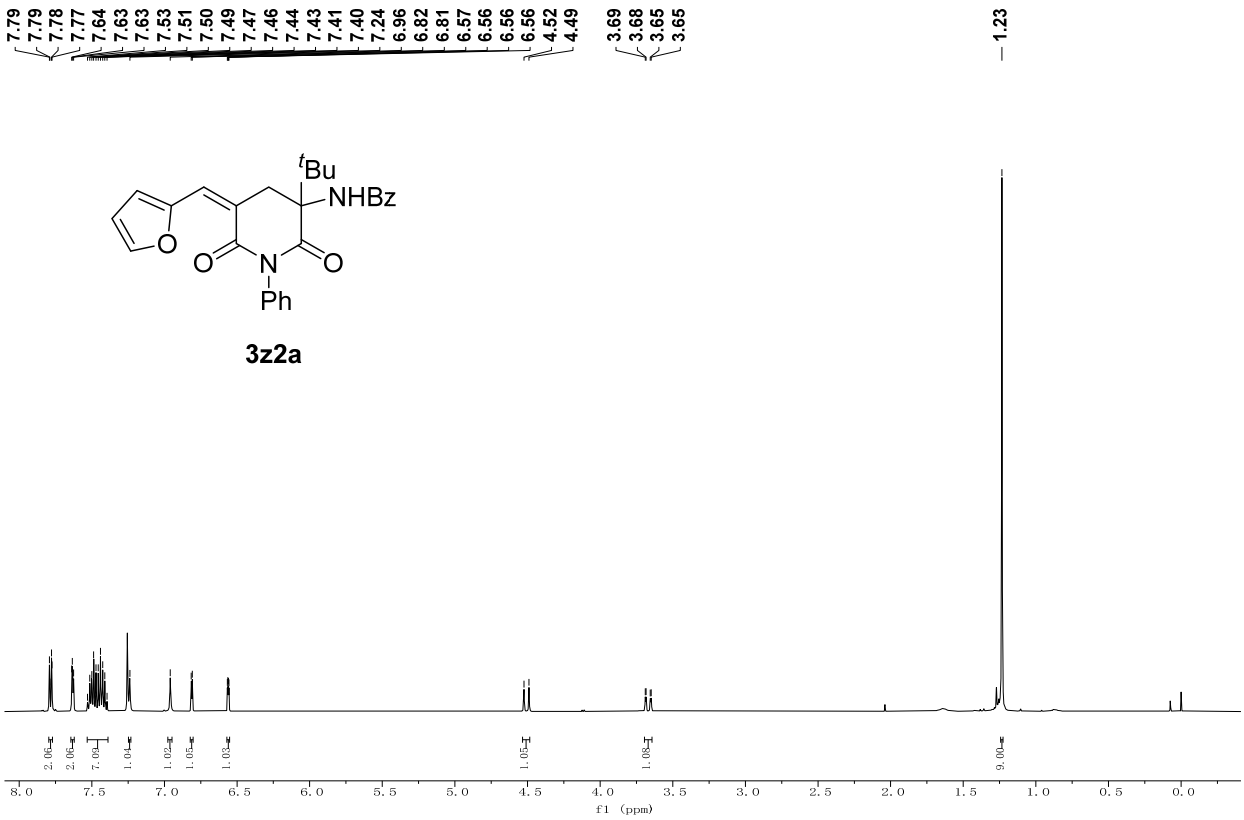
131.85
131.36
130.78
128.72
128.25
127.71
127.52
127.47
127.43
126.57
126.39
125.93
125.86
125.74
125.11
63.63
38.73
36.98
25.95


f1 (ppm)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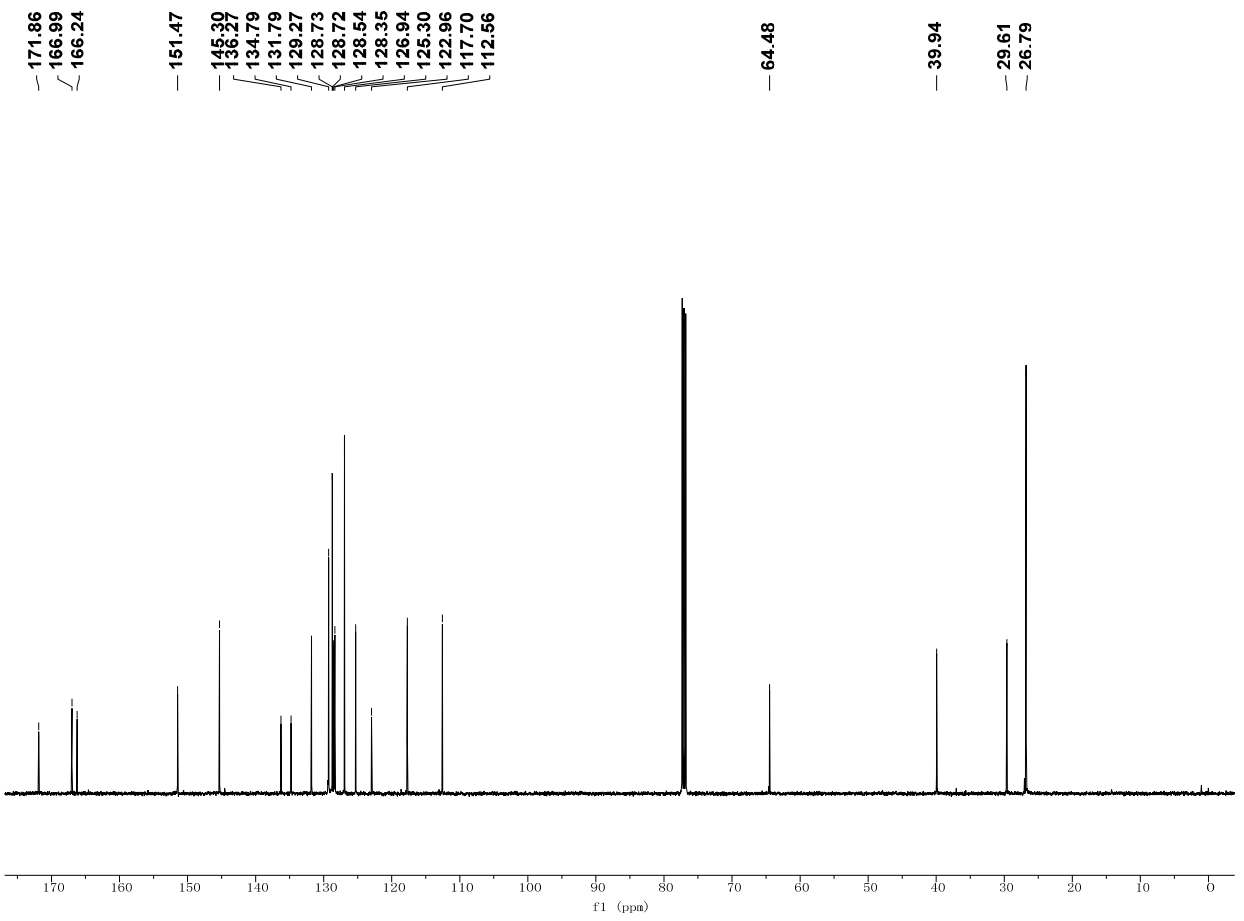
7.51
7.50
7.49
7.47
7.46
7.44
7.43
7.41
7.40
7.24
6.96
6.82
6.81
6.57
6.56
6.56
6.56
4.52
4.49
3.69
3.68
3.65
3.65
1.23
tBu
NHBz
O
O
N
O
Ph
2.06
2.06
7.69
1.04
1.02
1.05
1.03
1.05
1.08
9.00
f1 (ppm)

3z2a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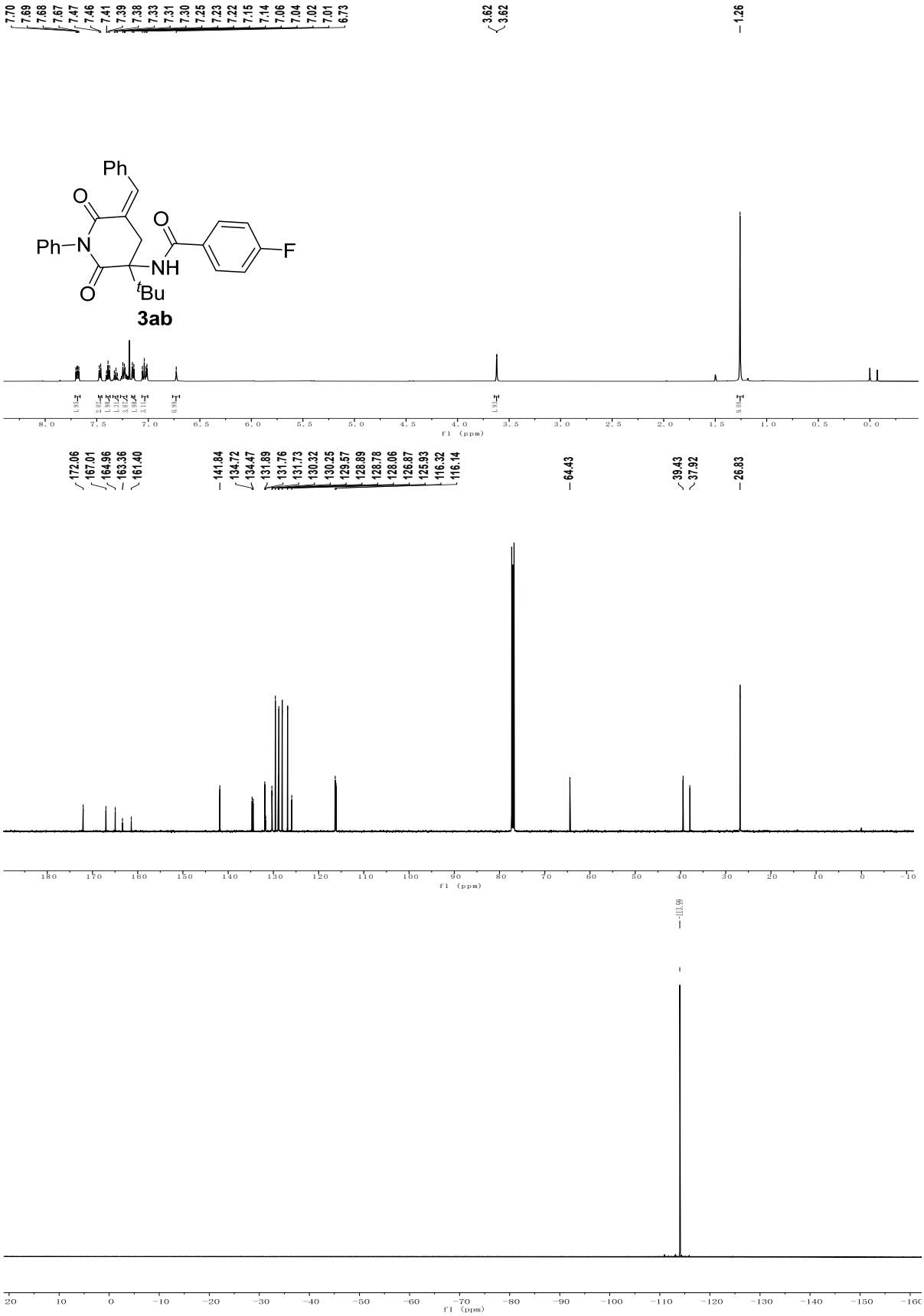
166.24
151.47
145.30
136.27
134.79
131.79
129.27
128.73
128.72
128.54
128.35
126.94
125.30
122.96
117.70
112.56
64.48
39.94
29.61
26.79
f1 (ppm)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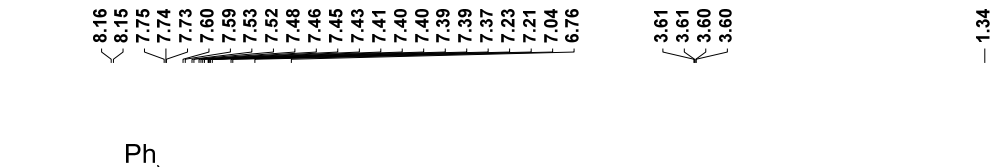
7.38
7.33
7.31
7.30
7.25
7.23
7.22
7.15
7.14
7.06
7.04
7.02
7.01
6.73
3.62
3.62
1.26
Ph
O
Ph–N
O
NH
O
tBu
O
F
3ab
f1 (ppm)
172.06
167.01
164.96
163.36
161.40
141.84
134.72
134.47
131.89
131.76
131.73
130.32
130.25
129.57
128.89
128.78
128.06
126.87
125.93
116.32
116.14
64.43
39.43
37.92
26.83
f1 (ppm)
-113.99
f1 (ppm)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7.41
7.40
7.40
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7.21
7.04
6.76
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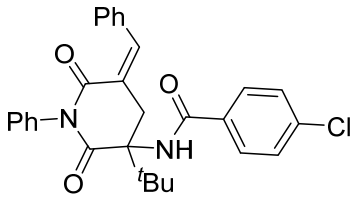
3.61
3.60
3.60
1.34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Ph–N
O
NH
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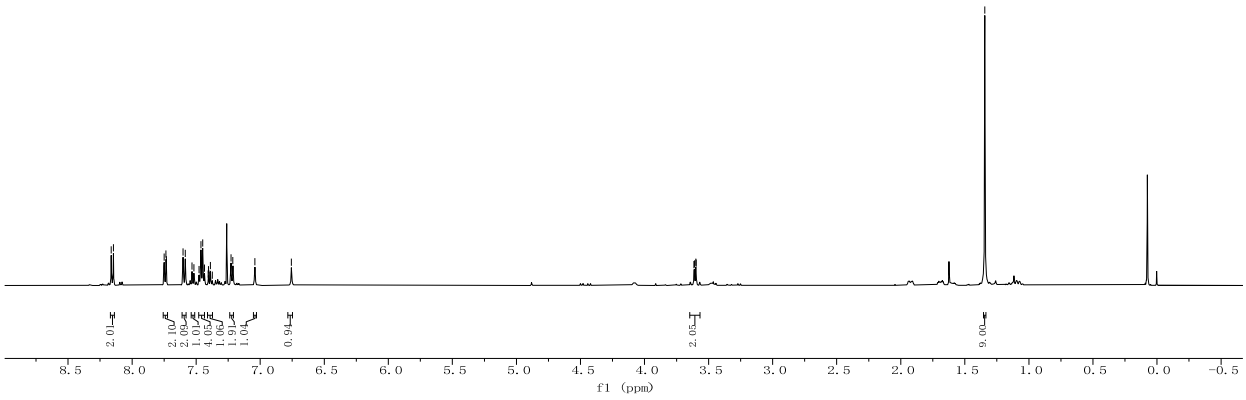
Cl
tBu
O

3ac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1.01
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1.91
1.04
0.94
2.05
9.00
8.5
8.0
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7.0
6.5
6.0
5.5
5.0
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2.0
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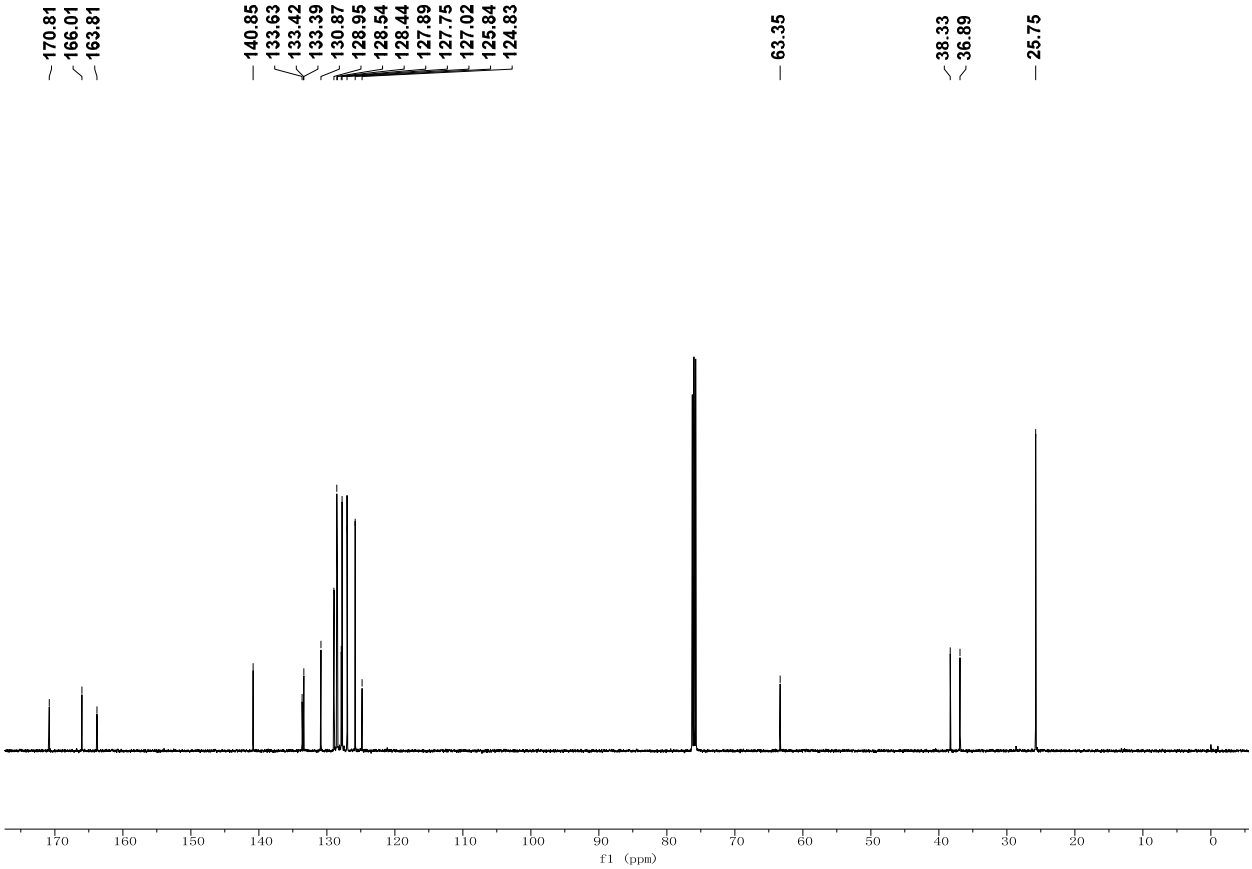
f1 (ppm)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125.84
124.83
63.35
38.33
36.89
25.75
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100
90
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50
40
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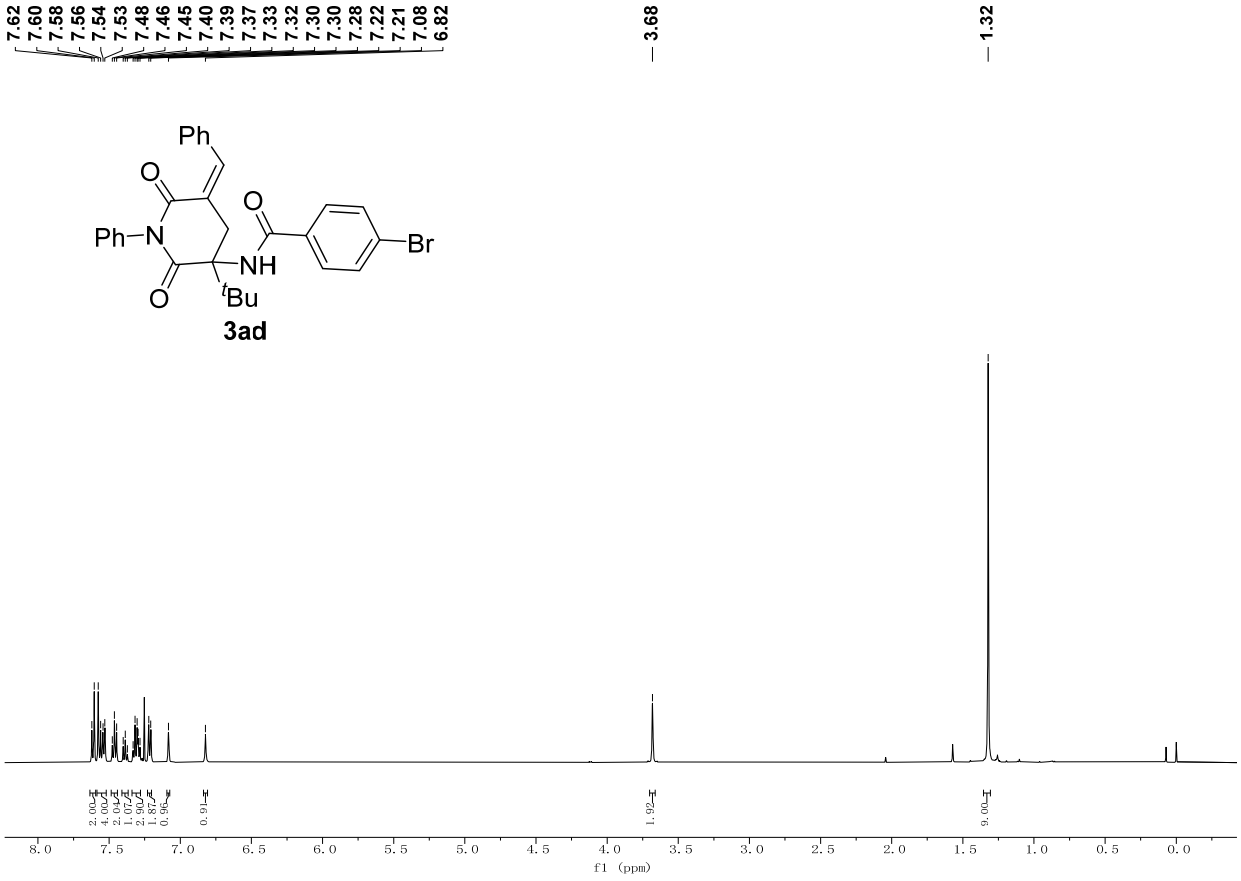
20
10
0
f1 (ppm)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7.39
7.37
7.33
7.32
7.30
7.28
7.22
7.21
7.08
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3.68
1.32
Ph
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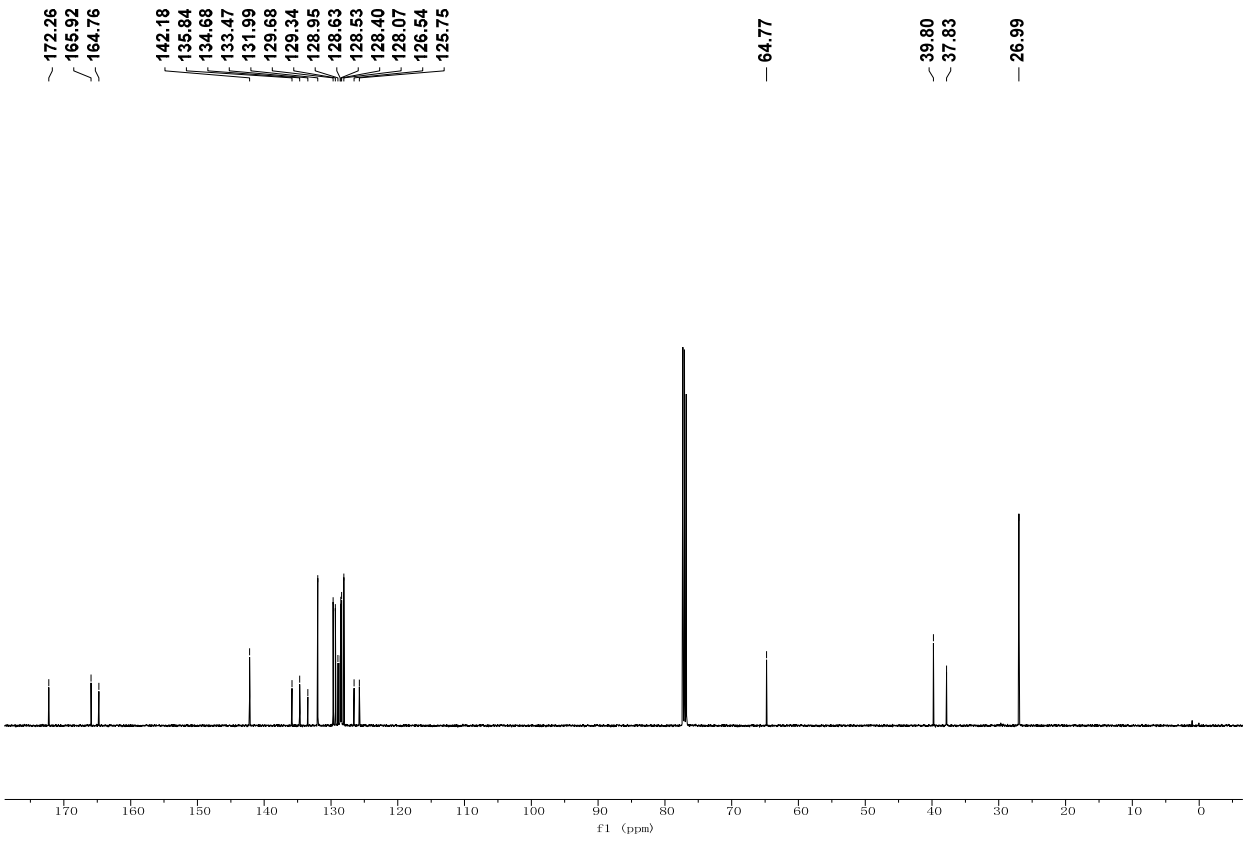
Ph–N
O
O
NH
tBu
Br
3ad
f1 (ppm)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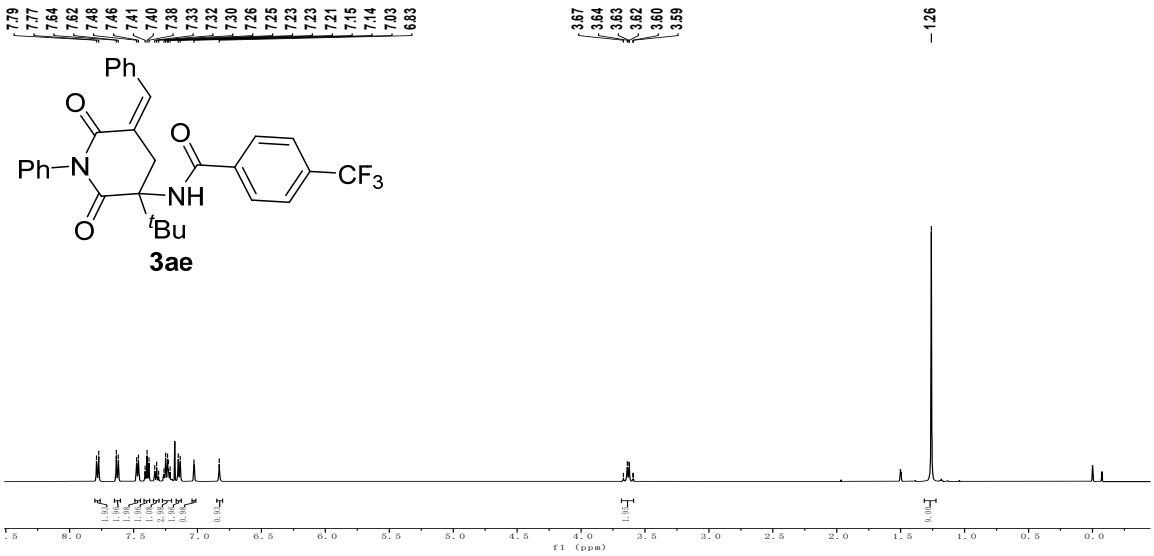
64.77
39.80
37.83
26.99
f1 (ppm)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7.23
7.23
7.21
7.15
7.14
7.03
6.83
3.67
3.64
3.63
3.62
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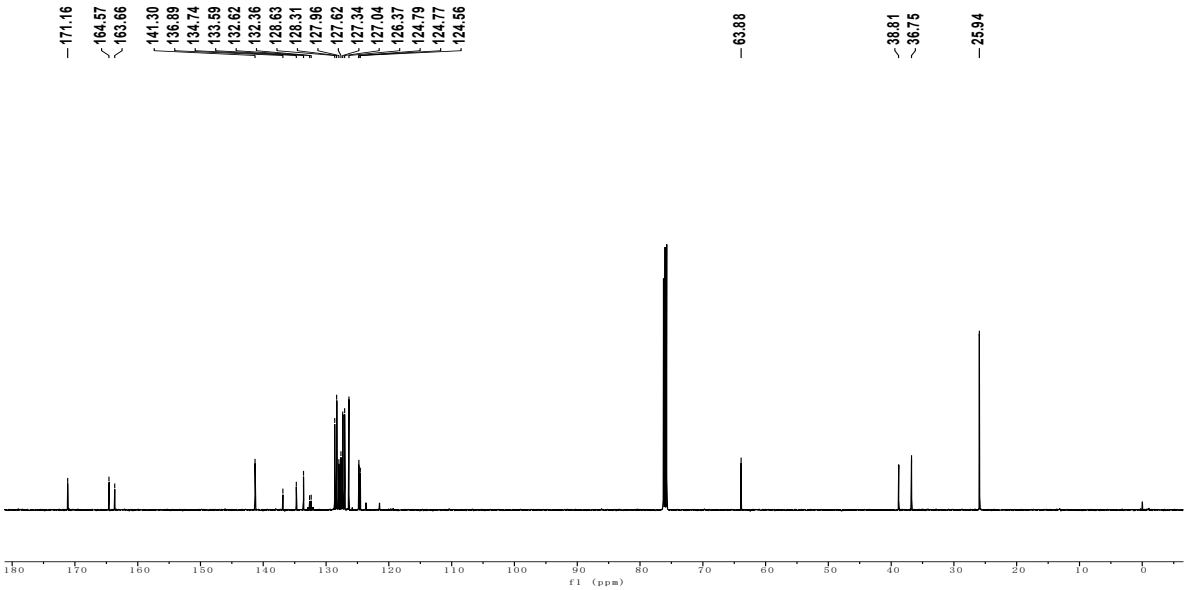
3.59
1.26
Ph
O
Ph–N
O
O
NH
CF3
tBu
3ae
f1 (ppm)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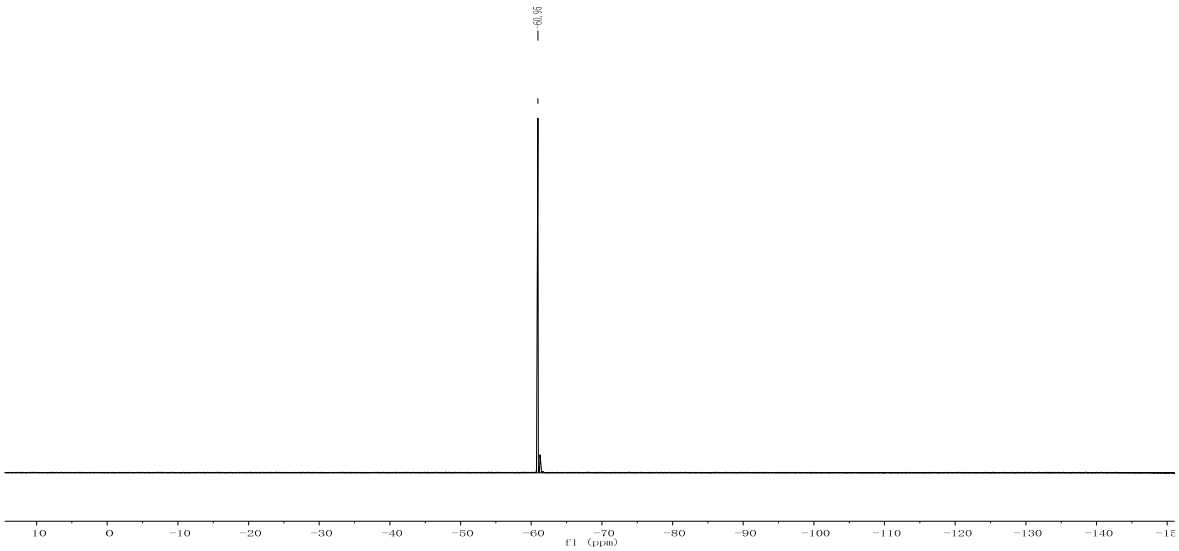
63.88
38.81
36.75
25.94
f1 (ppm)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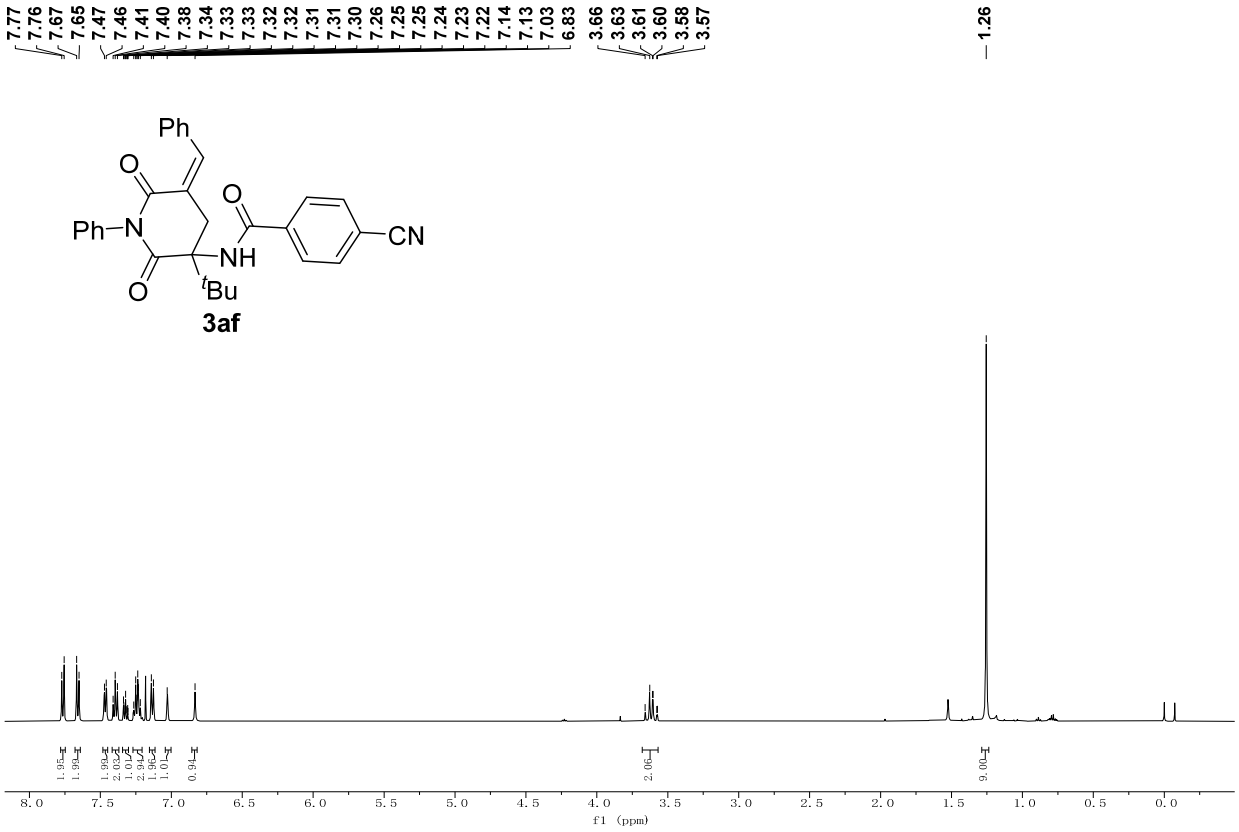
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7.31
7.31
7.30
7.26
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7.14
7.13
7.03
6.83
3.66
3.63
3.61
3.60
3.58
3.57
1.26
Ph
O
Ph–N
O
O
NH
CN
tBu
3af
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1.99
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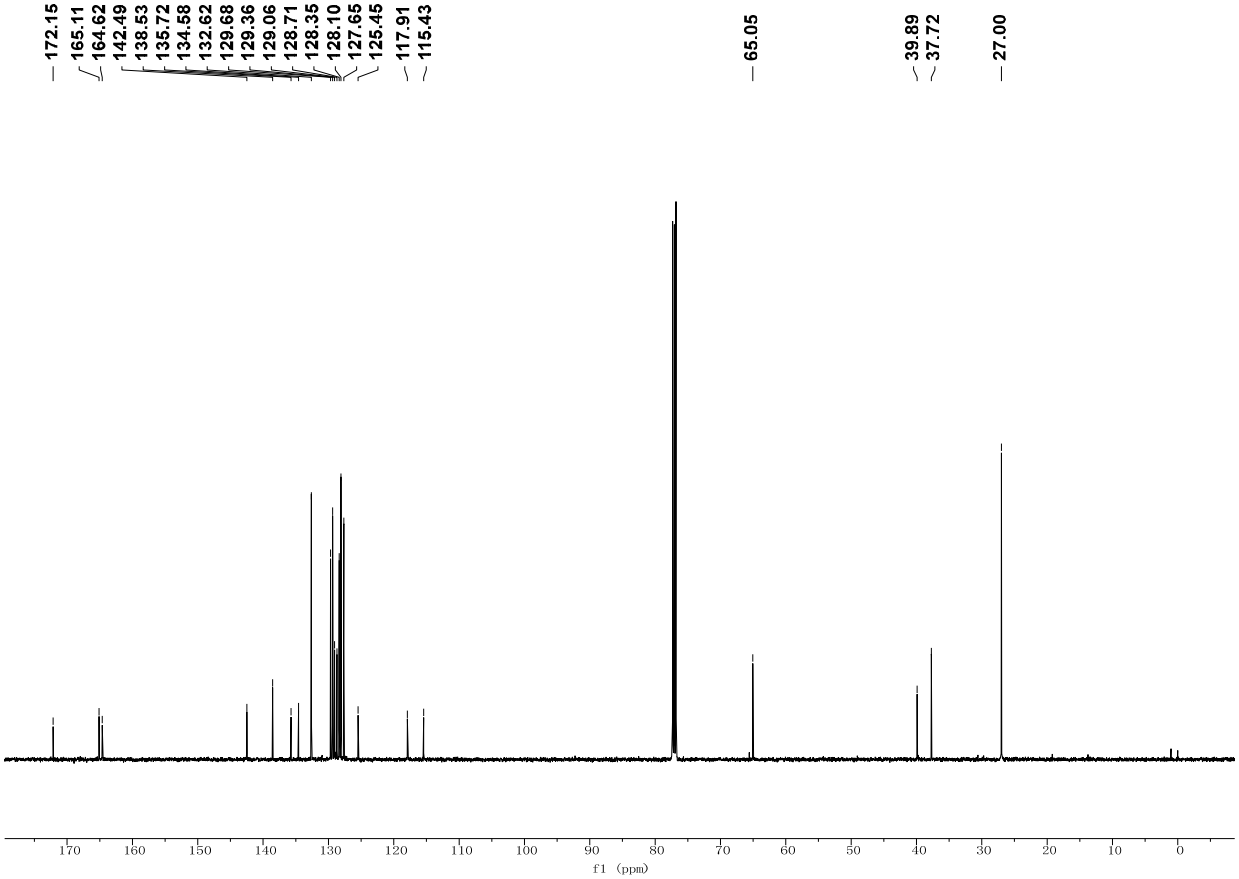
2.03
1.04
2.94
1.96
1.01
0.94
2.06
9.00
f1 (ppm)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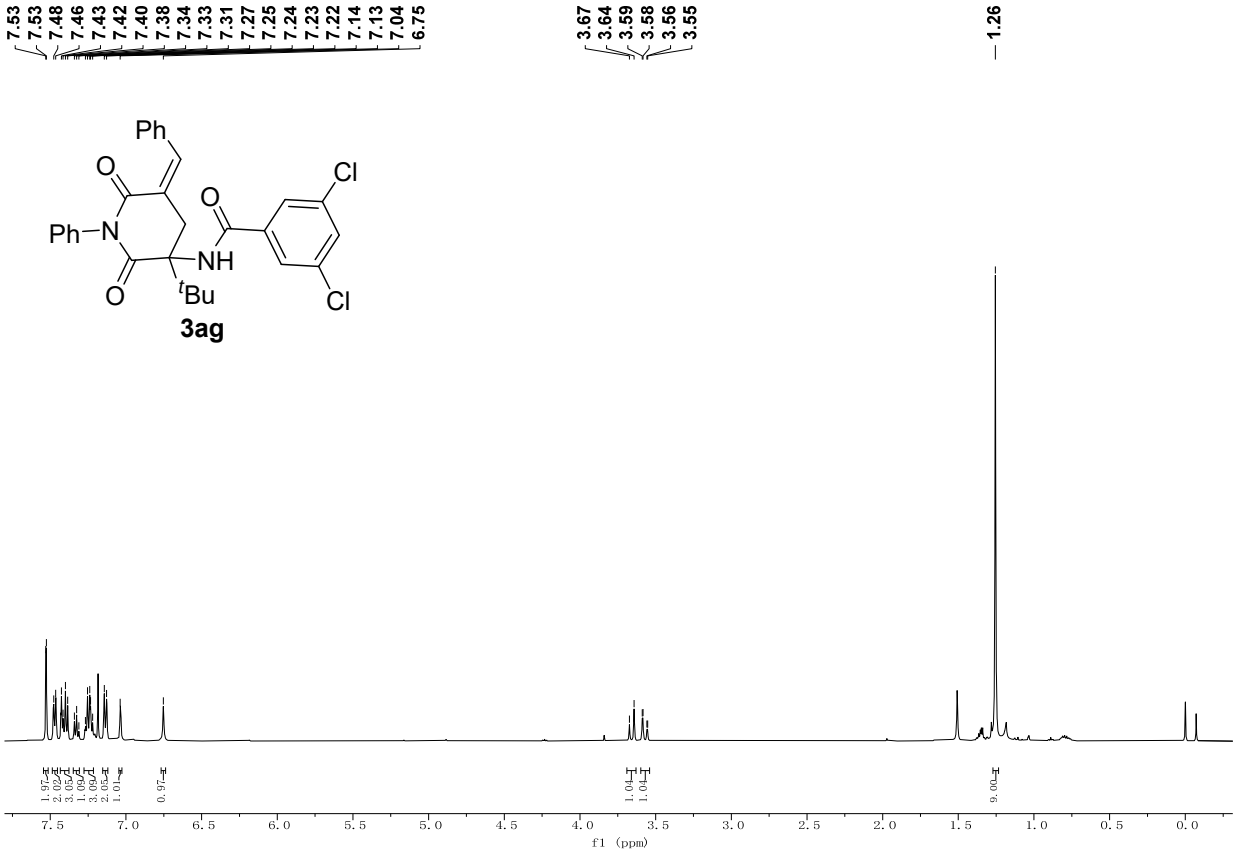
39.89
37.72
27.00
f1 (ppm)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7.27
7.25
7.24
7.23
7.22
7.14
7.13
7.04
6.75
3.67
3.64
3.59
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3.56
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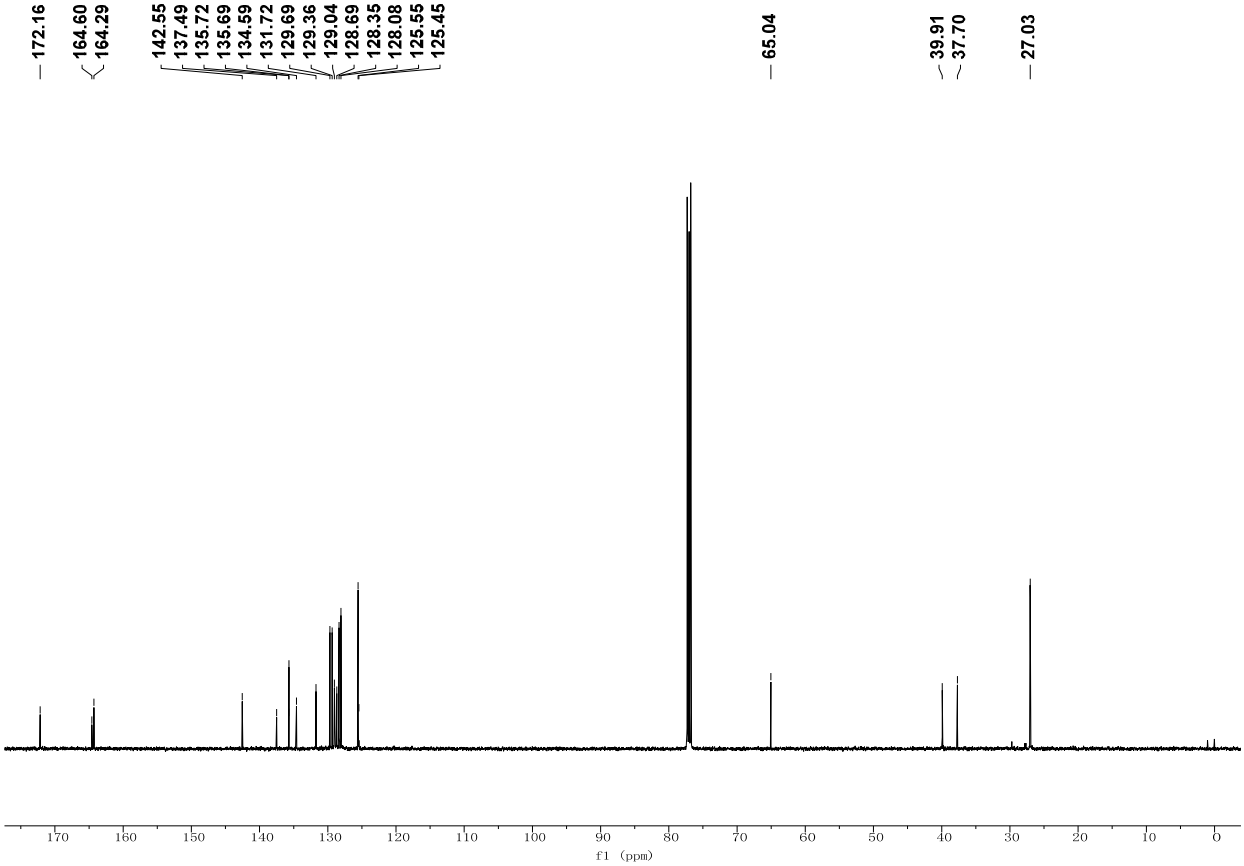
1.26
Ph
O
Cl
O
Ph–N
NH
O
tBu
Cl
3ag
f1 (ppm)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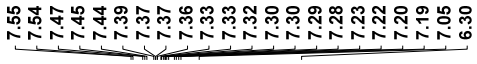
65.04
39.91
37.70
27.03
f1 (ppm)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7.30
7.30
7.29
7.28
7.23
7.22
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6.30
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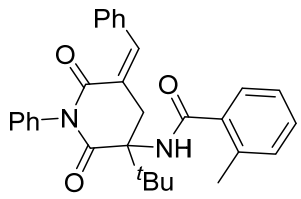
3.77
3.77
3.47
3.44
2.43
1.31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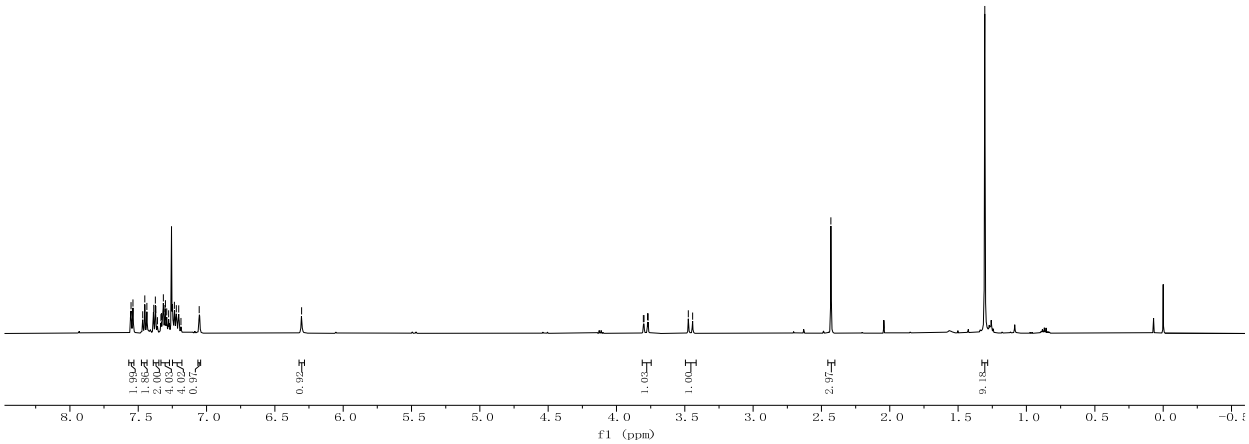
Ph–N
O
NH
O
tBu

3ah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0.92
1.03
1.00
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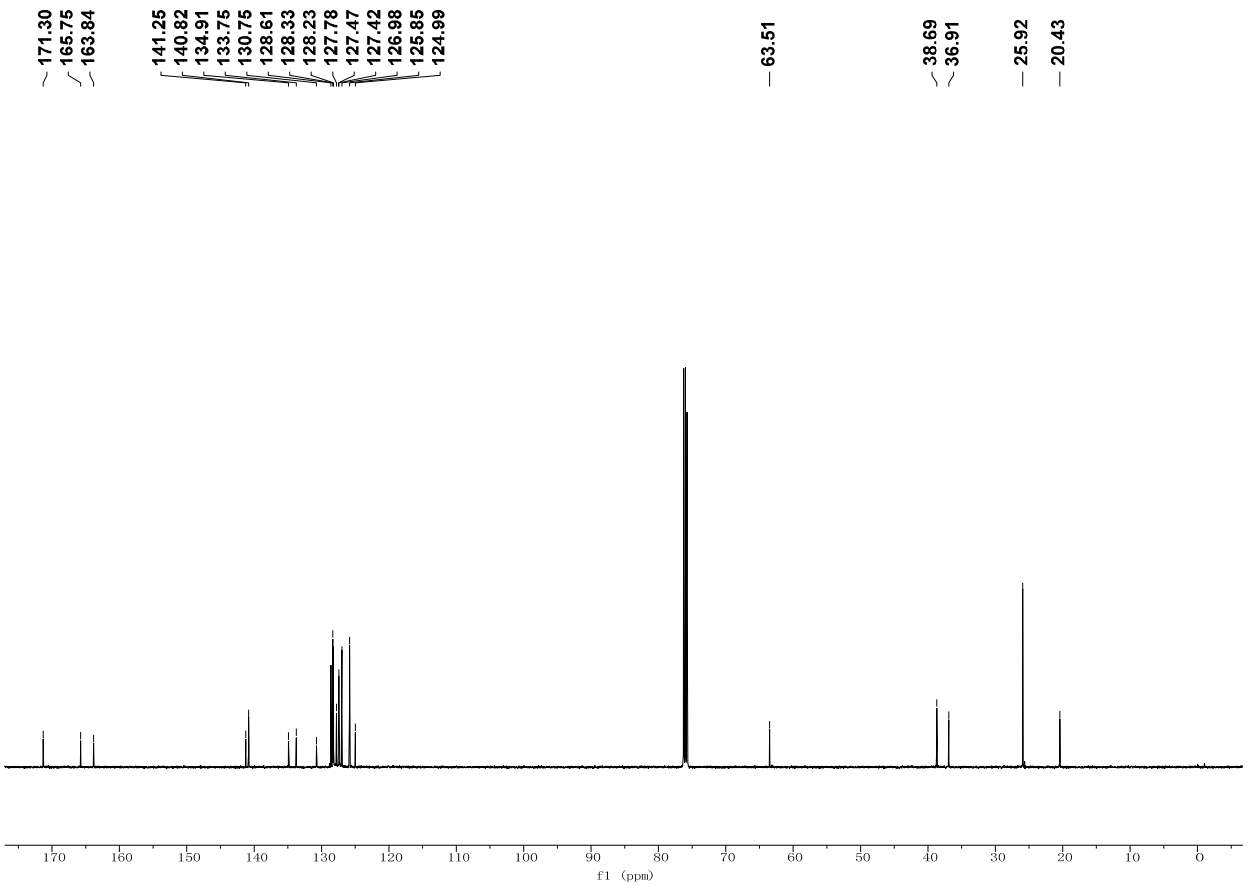
9.18
f1 (ppm)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63.51
38.69
36.91
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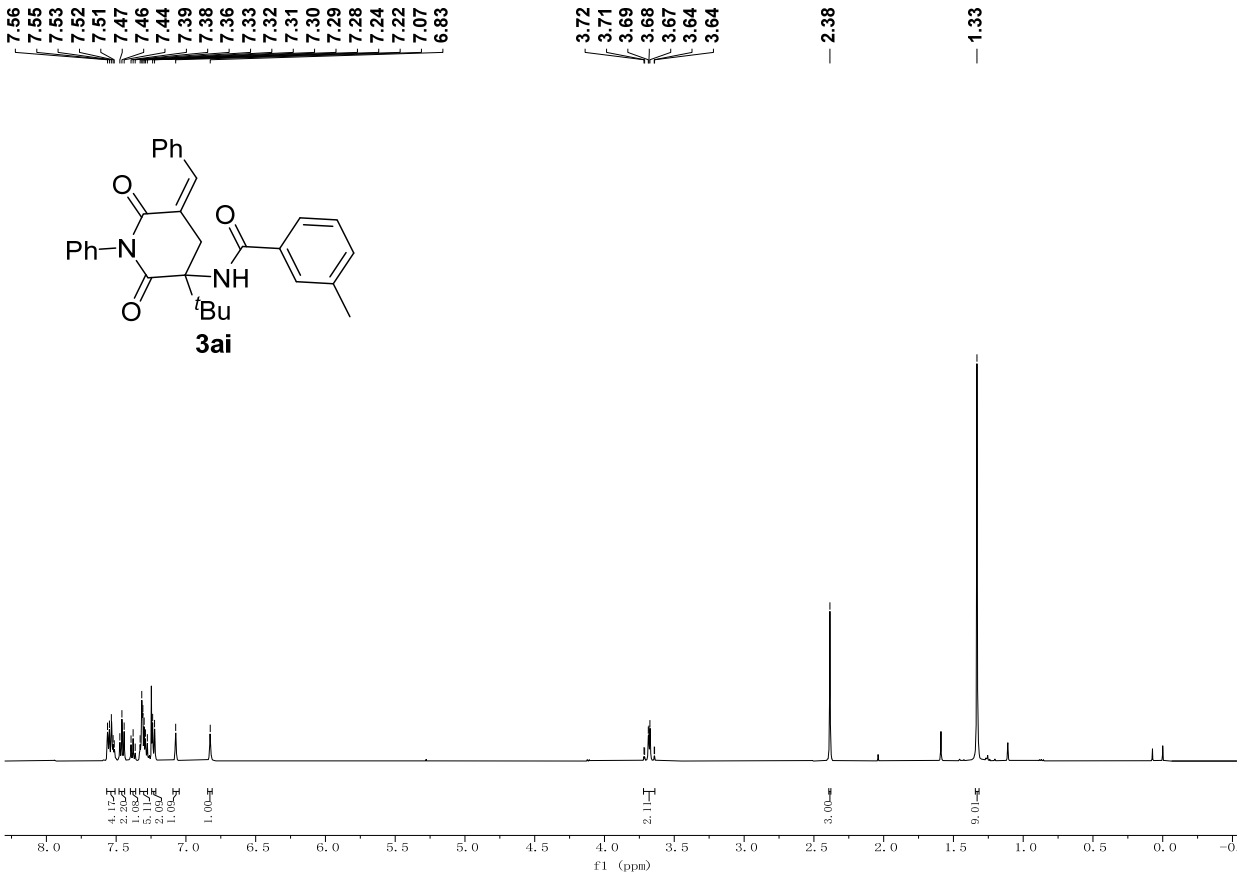
20.43
f1 (ppm)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7.31
7.30
7.29
7.28
7.24
7.22
7.07
6.83
3.72
3.71
3.69
3.68
3.67
3.64
3.64
2.38
1.33
Ph
O
Ph–N
O
NH
O
tBu
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1.06
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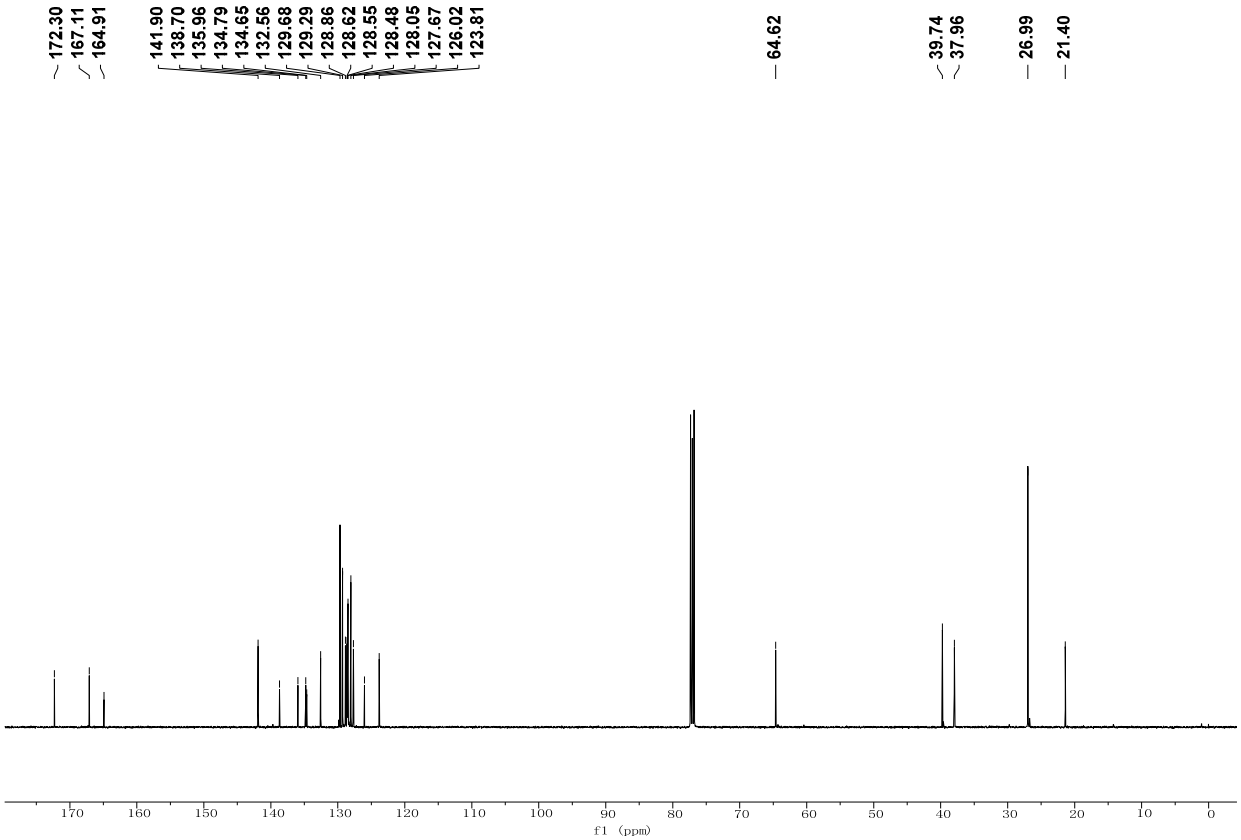
2.09
1.05
1.00
2.11
3.00
9.01
f1 (ppm)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64.62
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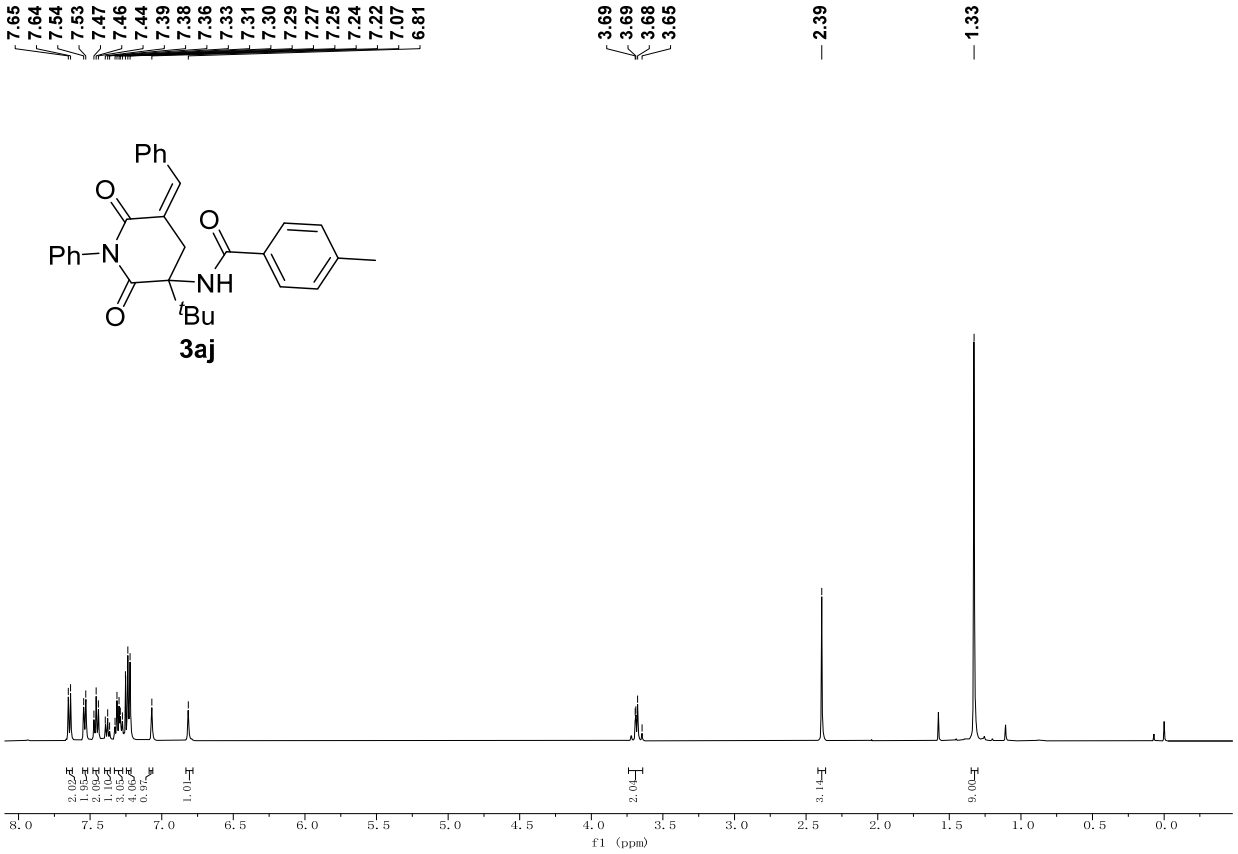
37.96
26.99
21.40
f1 (ppm)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7.31
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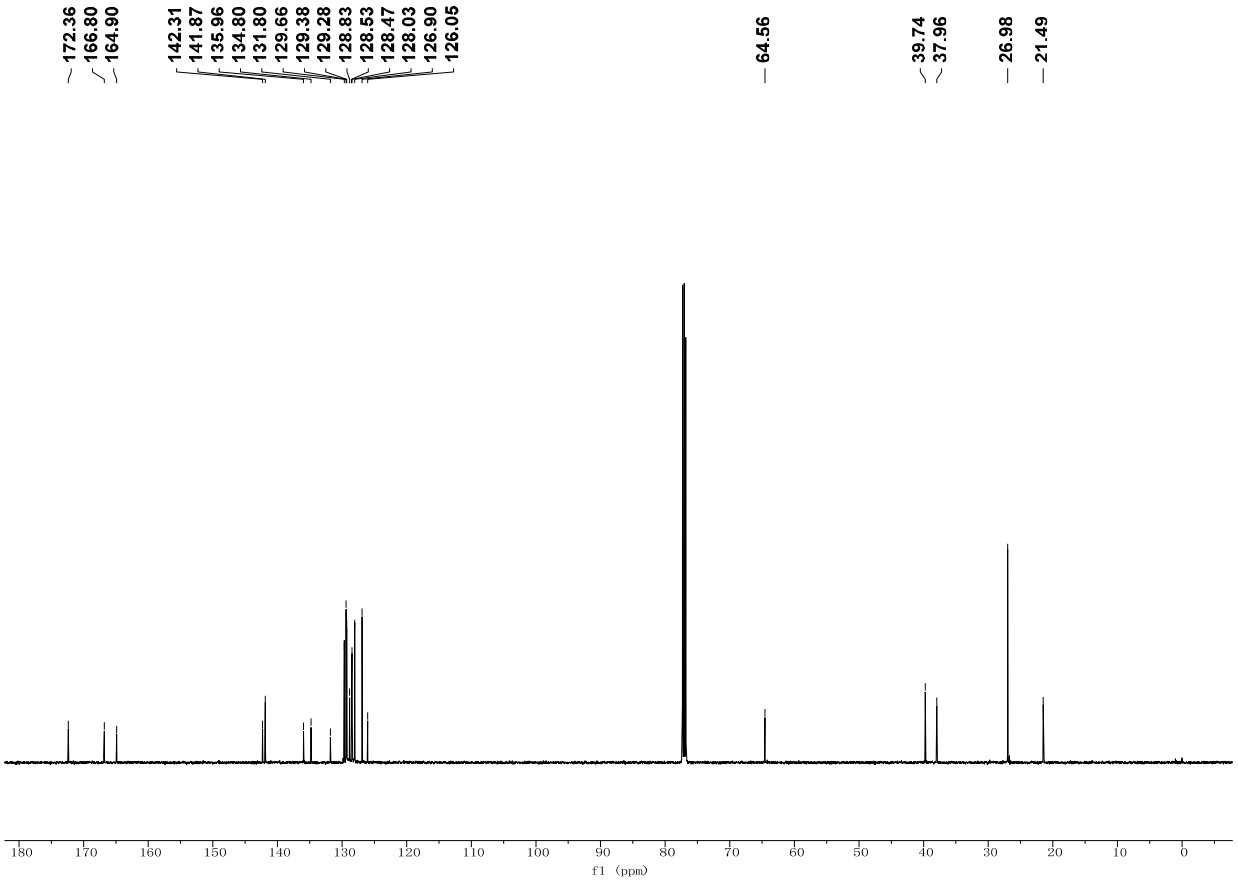
7.29
7.27
7.25
7.24
7.22
7.07
6.81
3.69
3.69
3.68
3.65
2.39
1.33
Ph
O
Ph–N
O
NH
O
tBu
3aj
f1 (ppm)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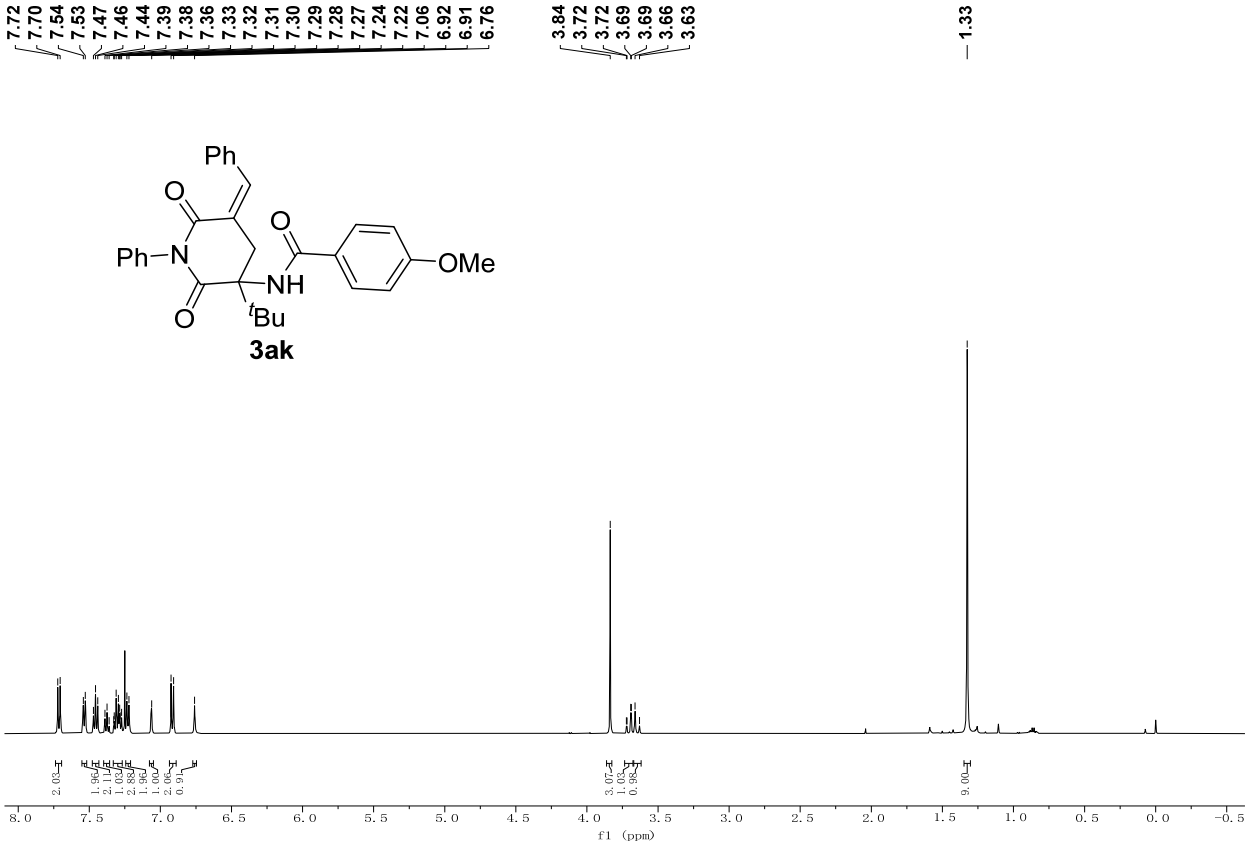
64.56
39.74
37.96
26.98
21.49
f1 (ppm)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7.30
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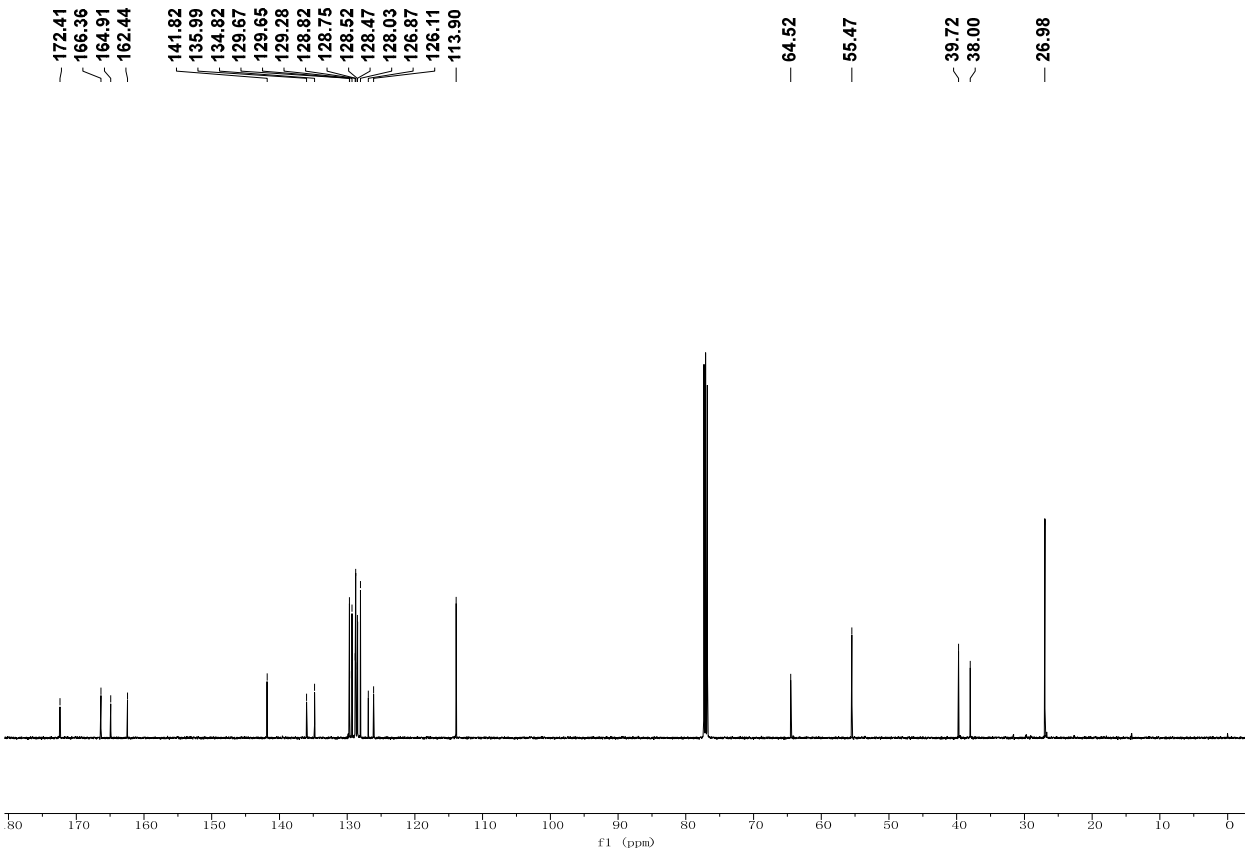
7.24
7.22
7.06
6.92
6.91
6.76
3.84
3.72
3.72
3.69
3.69
3.66
3.63
1.33
Ph
O
Ph–N
O
O
NH
OMe
tBu
3ak
f1 (ppm)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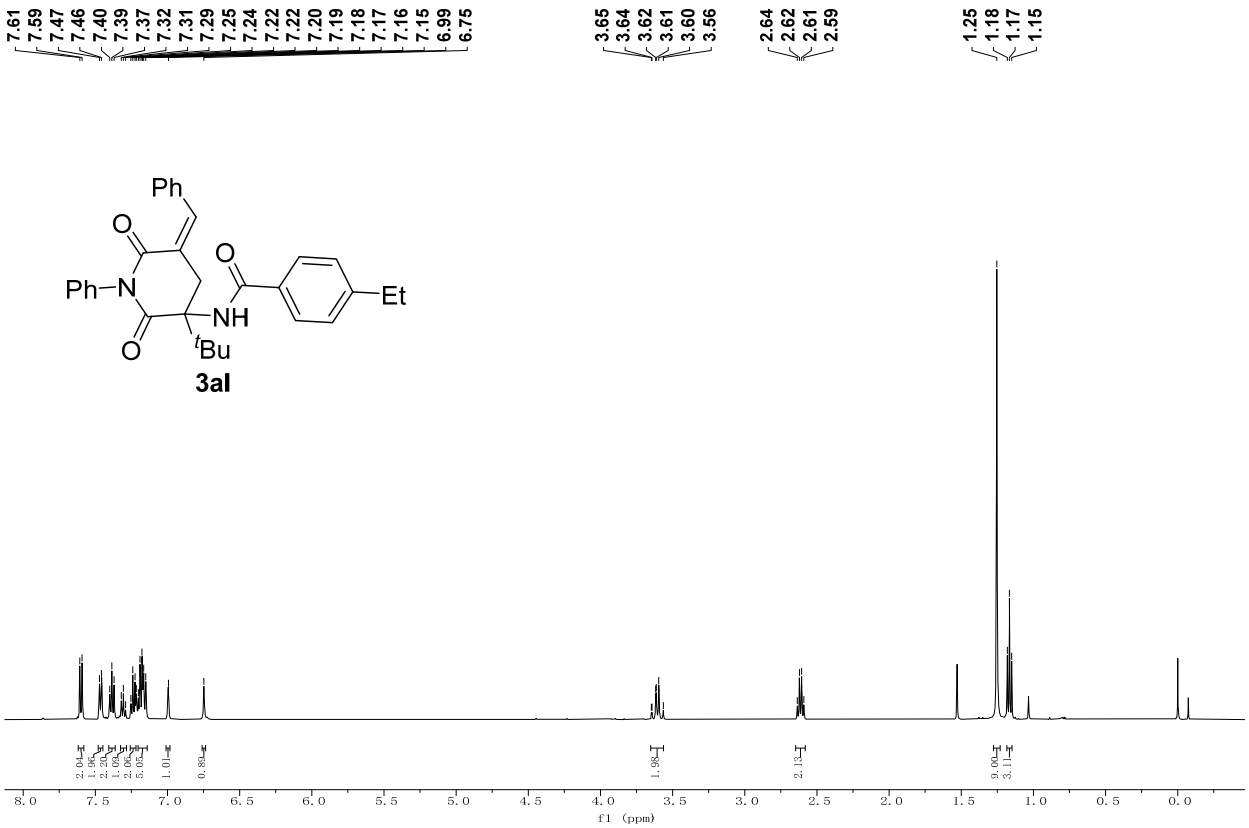
126.87
126.11
113.90
64.52
55.47
39.72
38.00
26.98
f1 (ppm)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7.29
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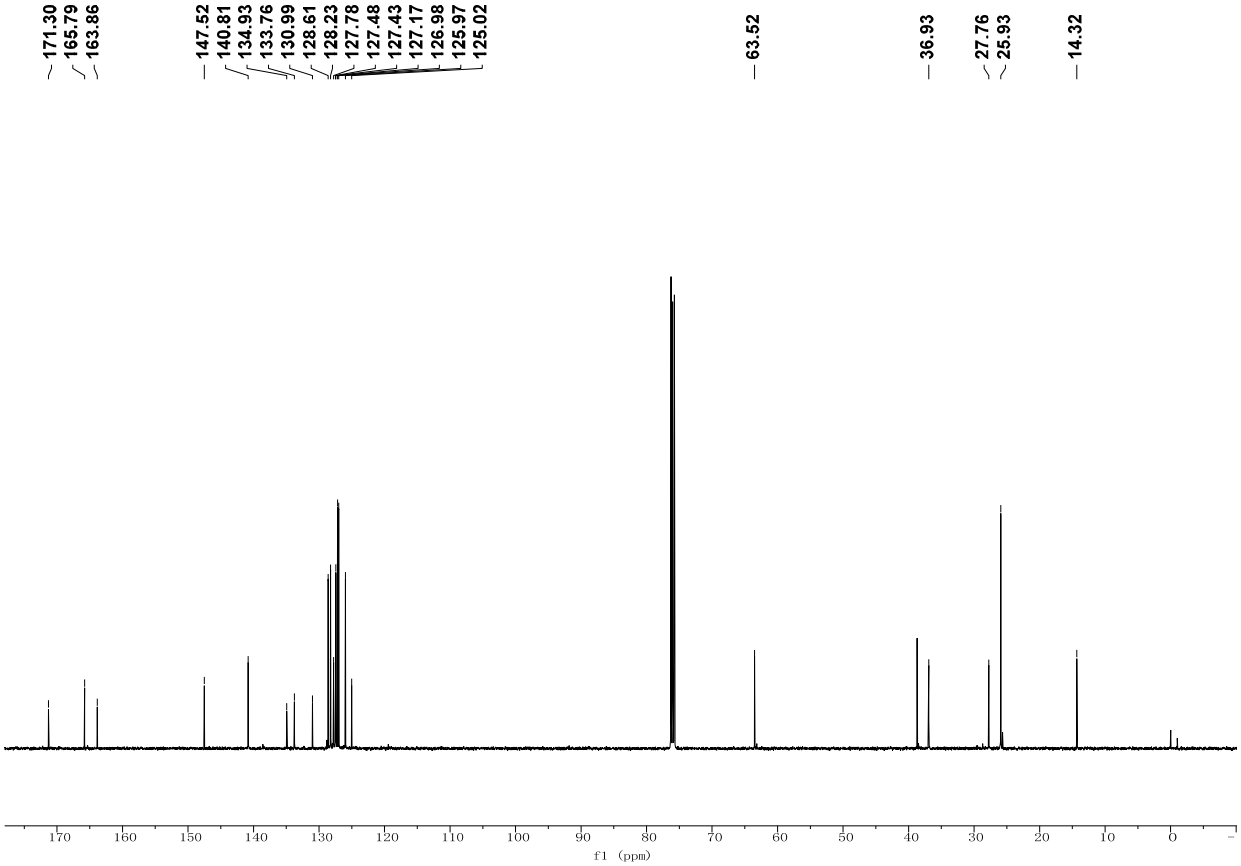
7.24
7.22
7.22
7.20
7.19
7.18
7.17
7.16
7.15
6.99
6.75
3.65
3.64
3.62
3.61
3.60
3.56
2.64
2.62
2.61
2.59
1.25
1.18
1.17
1.15
Ph
O
Ph–N
O
NH
O
tBu
O
Et
3aI
f1 (ppm)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125.02
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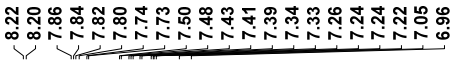
27.76
25.93
14.32
f1 (ppm)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7.43
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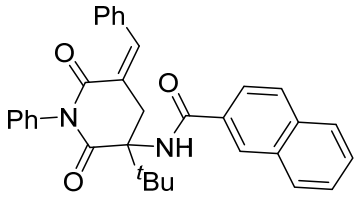
7.34
7.33
7.26
7.24
7.24
7.22
7.05
6.96
3.69
1.31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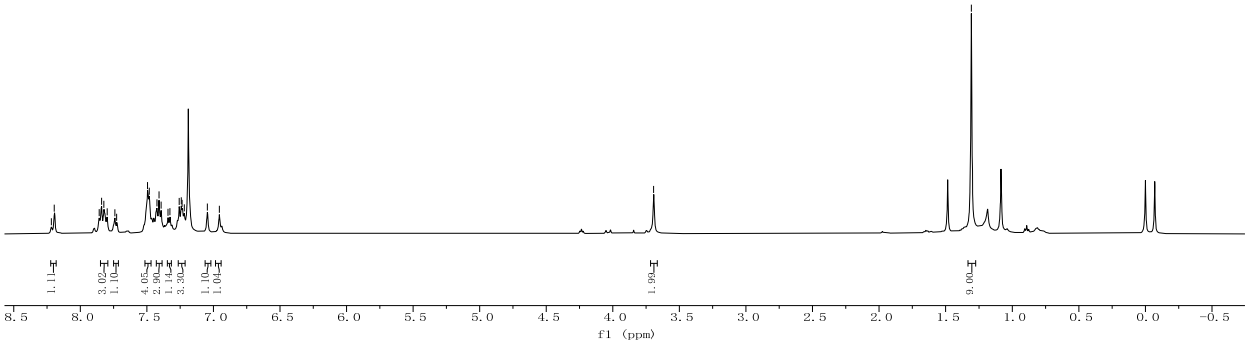
Ph–N
O
NH
O
tBu

3am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3.02
1.10
4.05
2.90
1.14
3.20
1.10
1.04
1.99
9.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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3.0
2.5
2.0
1.5
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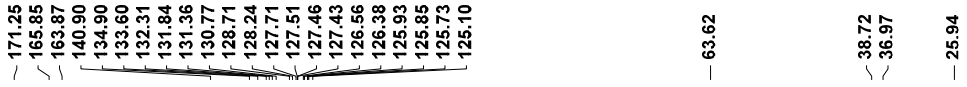
0.5
0.0
−0.5
f1 (ppm)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127.71
127.51
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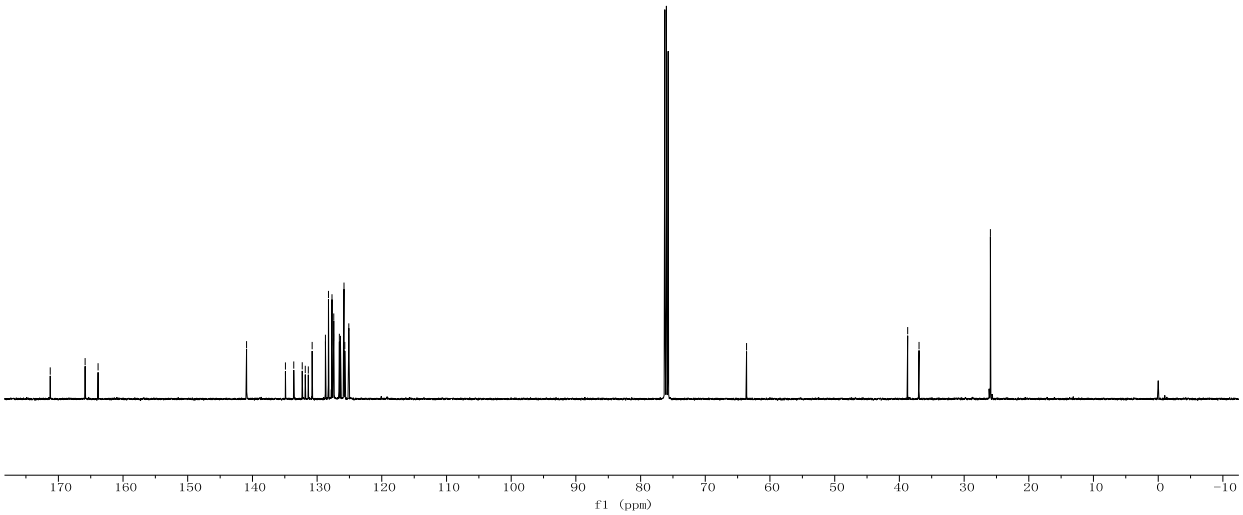
125.73
125.10
63.62
38.72
36.97
25.94


170
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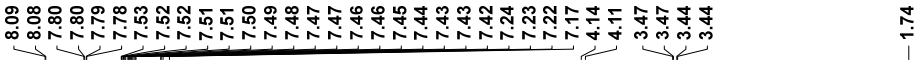
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)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7.47
7.47
7.46
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7.44
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7.43
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7.17
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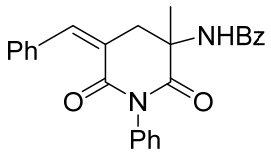
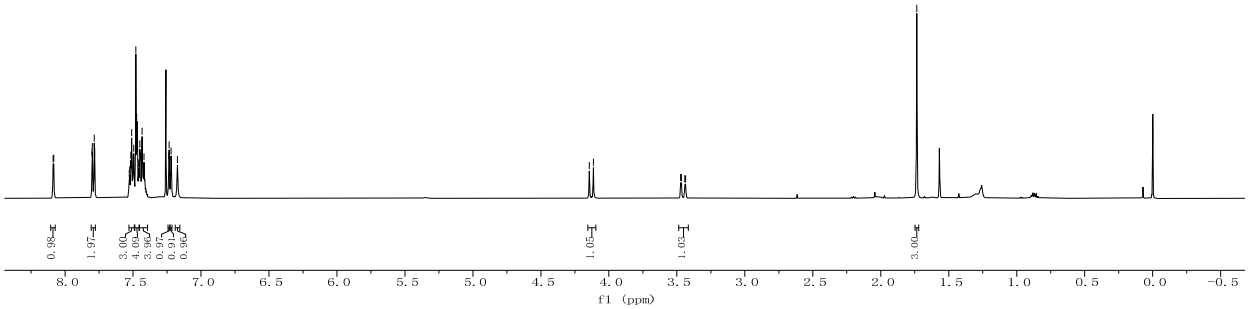
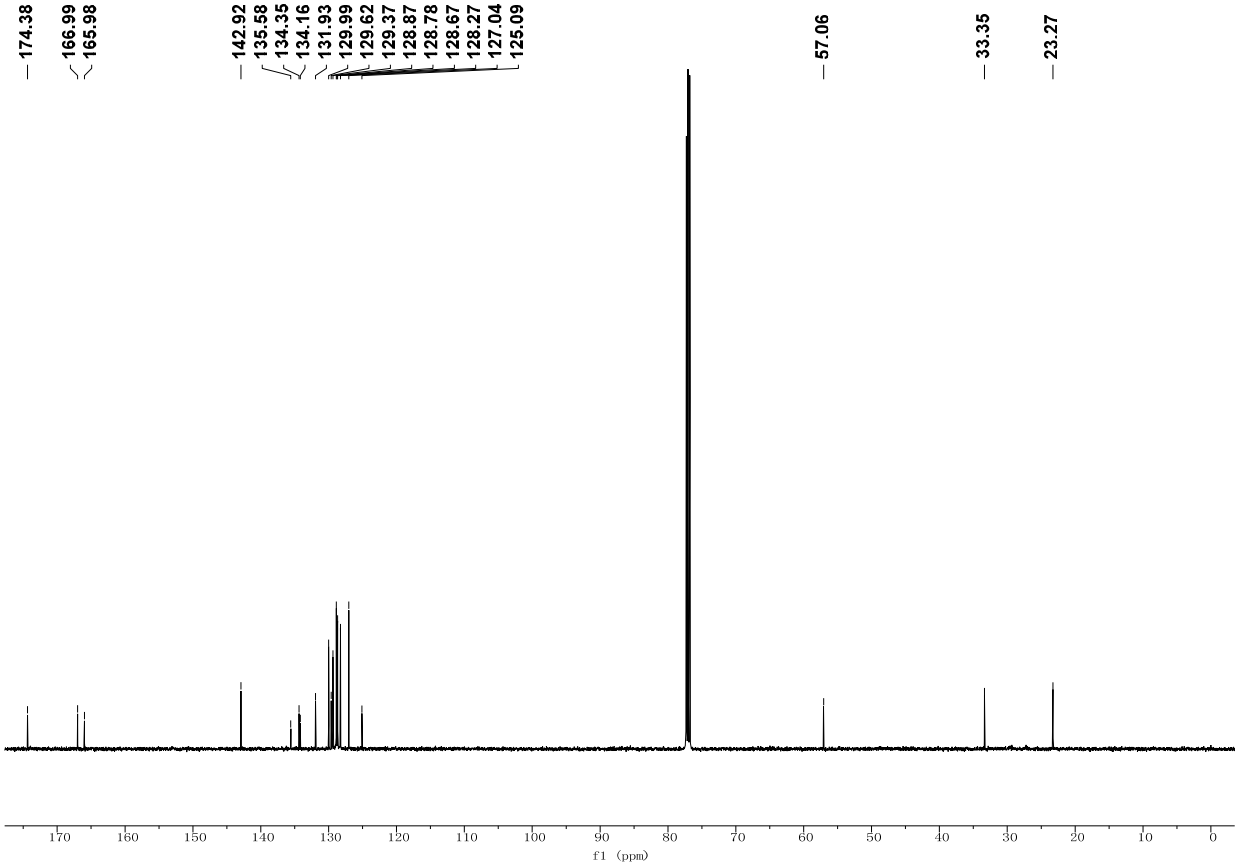
1.74


Ph
NHBz
O
N
O
Ph

3an


0.98
1.97
3.00
4.08
3.96
0.97
0.91
0.96
1.05
1.03
3.00
f1 (ppm)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166.99
165.98
142.92
135.58
134.35
134.16
131.93
129.99
129.62
129.37
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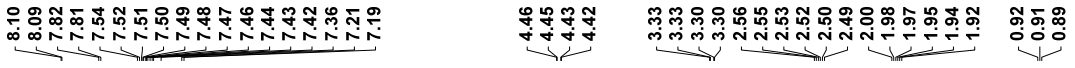
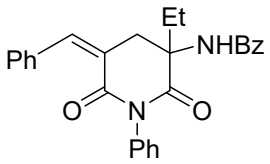
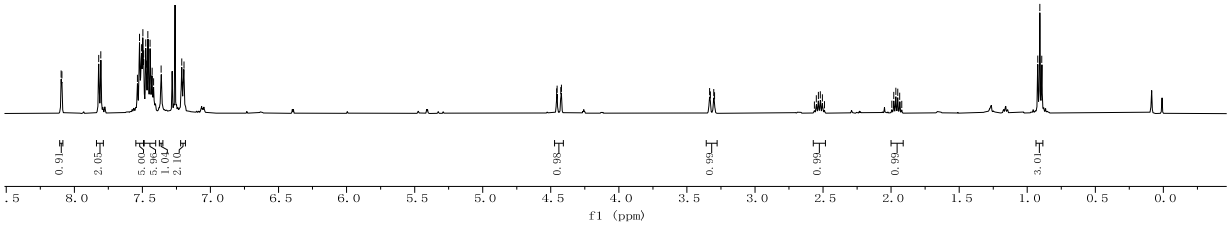
128.78
128.67
128.27
127.04
125.09
57.06
33.35
23.27
f1 (ppm)

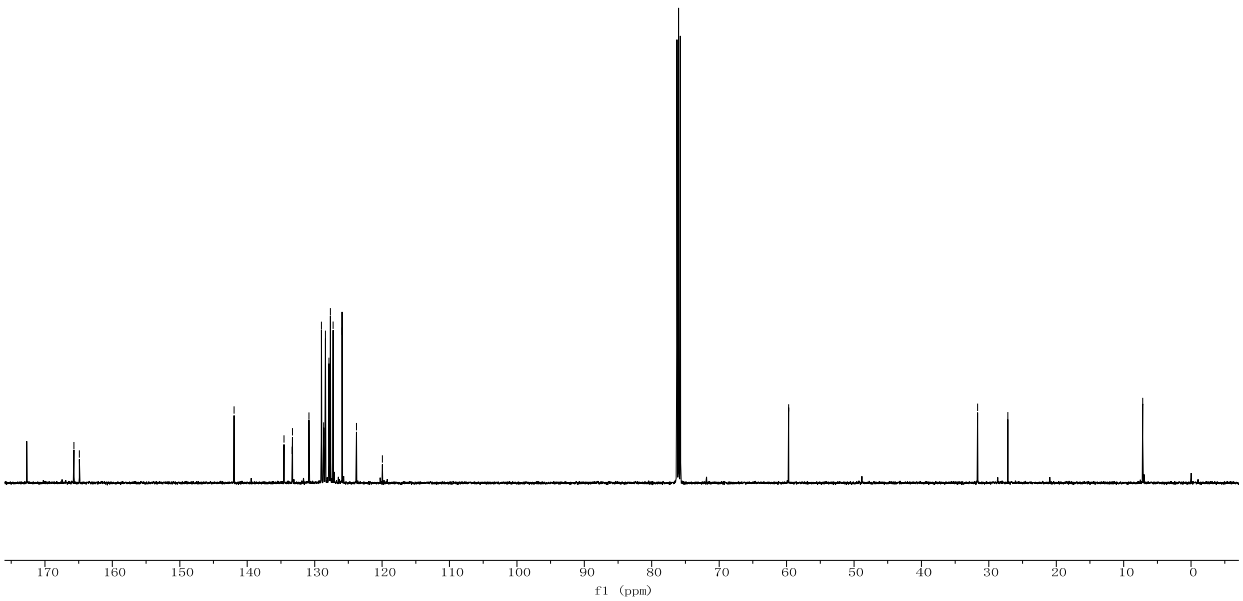

8.10
8.09
7.82
7.81
7.54
7.52
7.51
7.50
7.49
7.48
7.47
7.46
7.44
7.43
7.42
7.36
7.21
7.19
4.46
4.45
4.43
4.42
3.33
3.33
3.30
3.30
2.56
2.55
2.53
2.52
2.50
2.49
2.00
1.98
1.97
1.95
1.94
1.92
0.92
0.91
0.89


Et
NHBz
Ph
O
N
O
Ph

3ao


0.91
2.05
5.00
5.96
1.00
2.10
0.98
0.99
0.99
0.99
3.01
f1 (ppm)


165.70
164.89
141.95
134.55
133.34
133.28
130.85
129.00
128.67
128.41
127.87
127.80
127.67
127.27
125.94
123.80
119.96
59.71
31.67
27.19
7.18


f1 (ppm)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7.79
7.60
7.59
7.52
7.51
7.48
7.47
7.46
7.45
7.45
7.43
7.41
7.40
7.35
7.33
7.32
7.30
7.21
7.18
7.17
7.00
3.93
3.90
3.63
3.63
3.60
3.60
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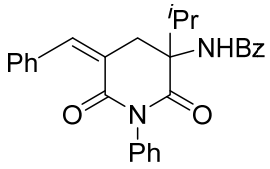
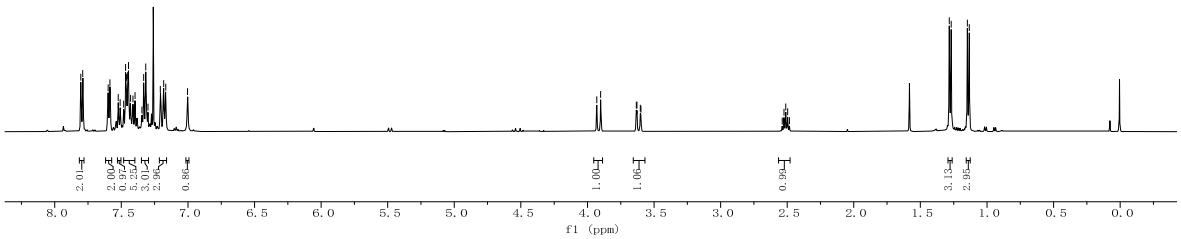
2.52
2.51
2.50
2.48
1.28
1.27
1.15
1.13


iPr
NHBz
Ph
O
N
O
Ph

3ap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0.86
1.00
1.06
0.99
3.13
2.95
f1 (ppm)


173.54
167.27
164.38
144.01
135.53
134.69
134.57
131.84
129.94
129.36
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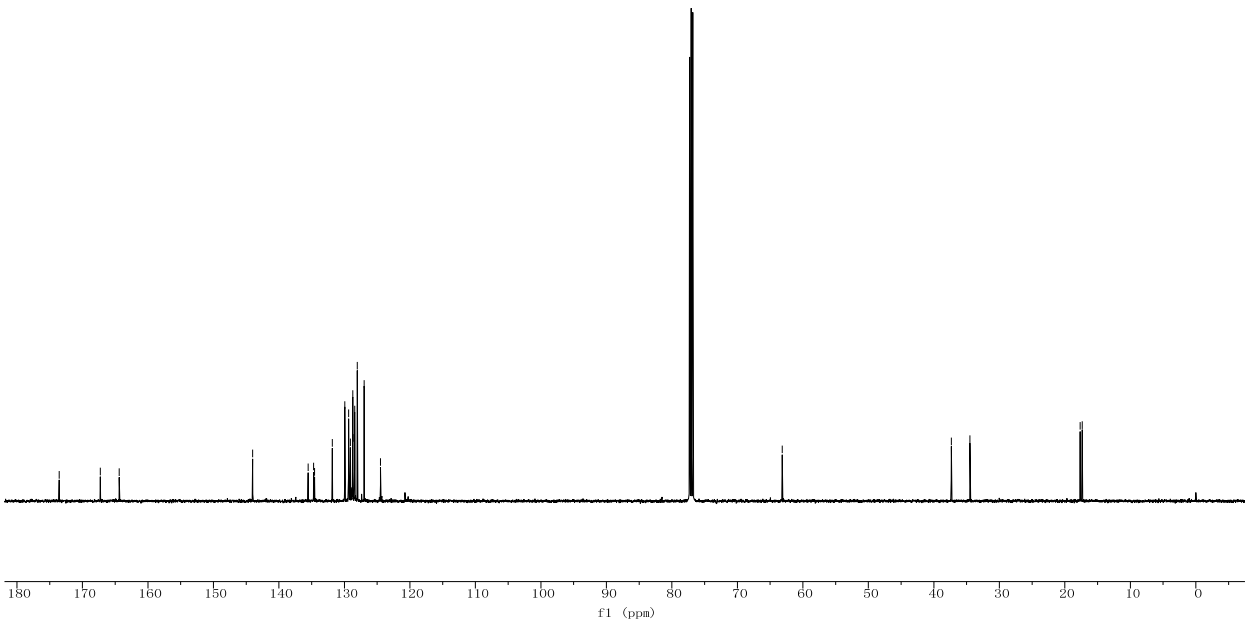
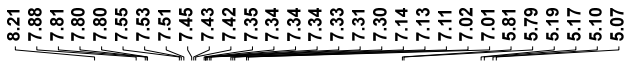
128.03
126.98
124.49
63.15
37.33
34.48
17.66
17.34


f1 (ppm)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7.45
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7.35
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7.34
7.33
7.31
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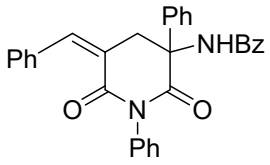
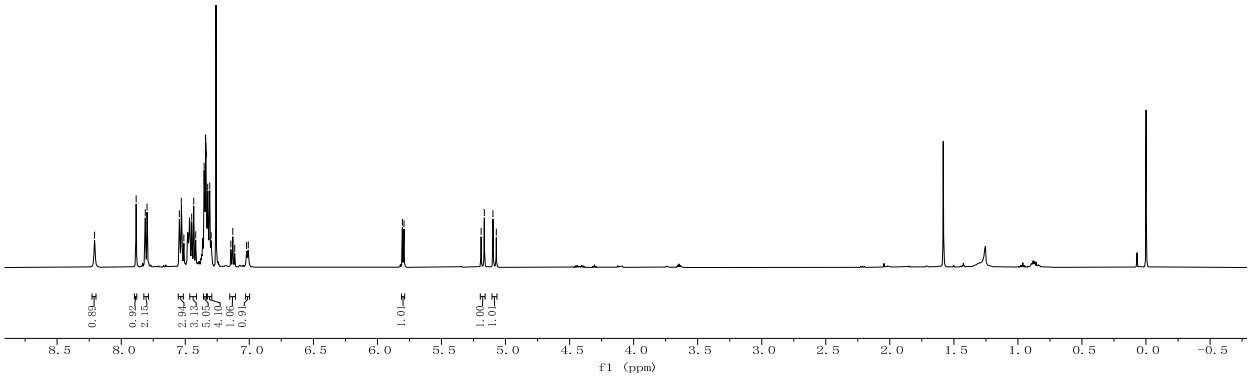
5.81
5.79
5.19
5.17
5.10
5.07


Ph
NHBz
Ph
O
N
O
Ph

3aq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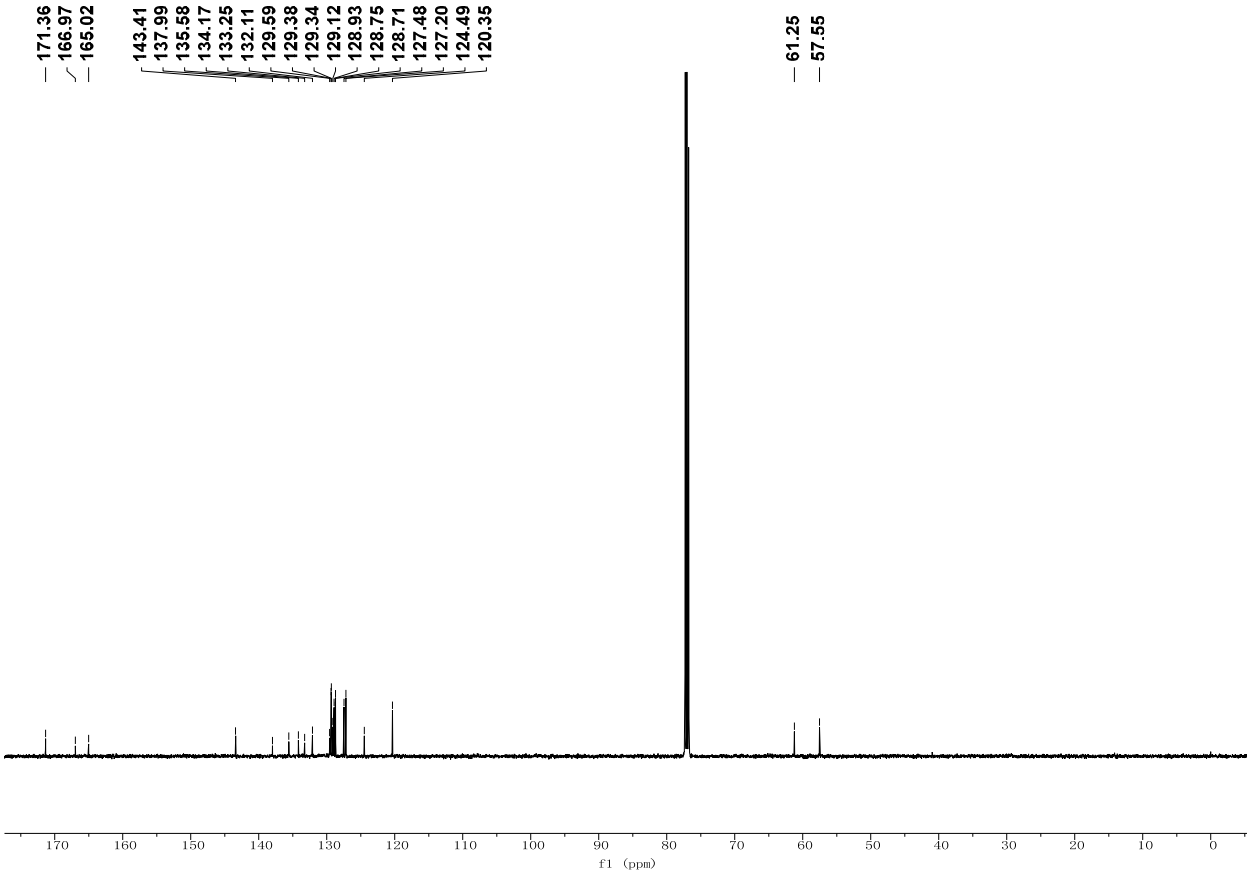
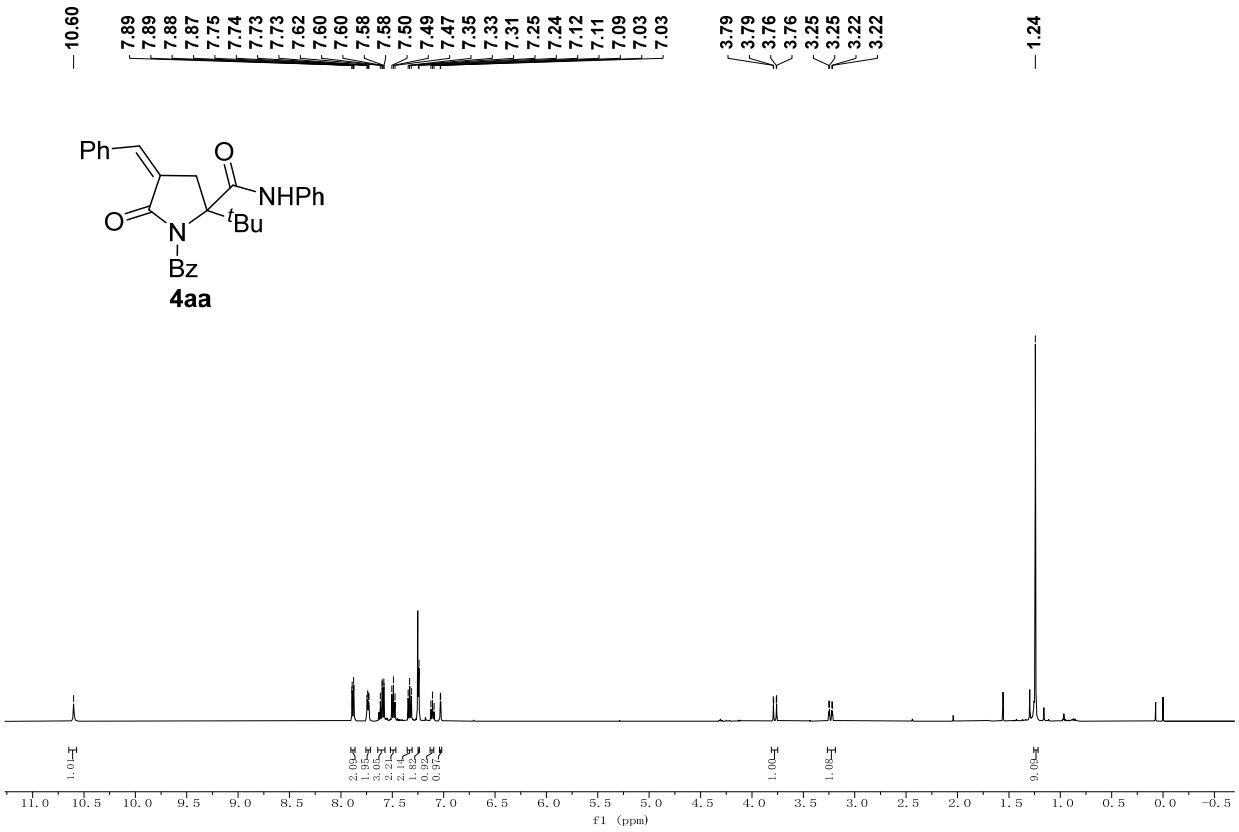
0.92
2.15
2.94
3.13
5.05
4.10
1.06
0.91
1.01
1.00
1.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)


171.36
166.97
165.02
143.41
137.99
135.58
134.17
133.25
132.11
129.59
129.38
129.34
129.12
128.93
128.75
128.71
127.48
127.20
124.49
120.35
61.25
57.55
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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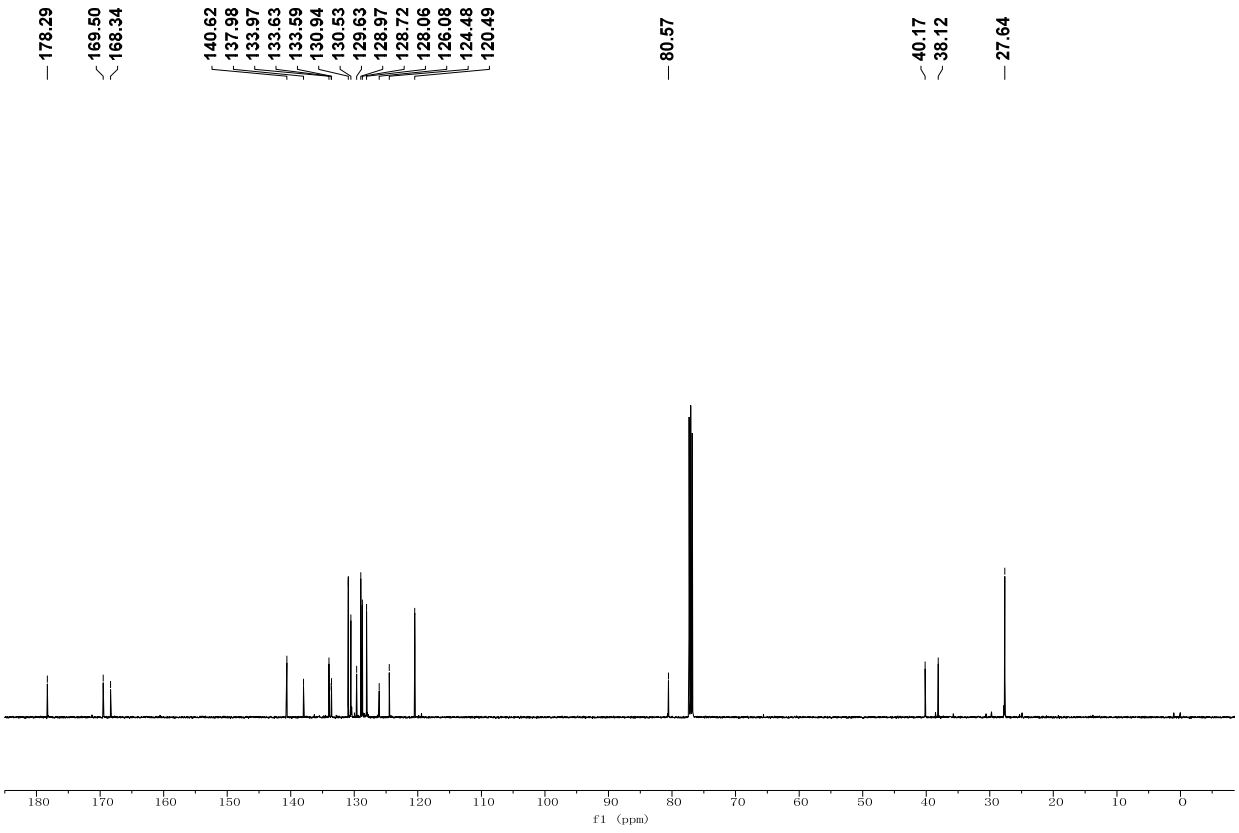
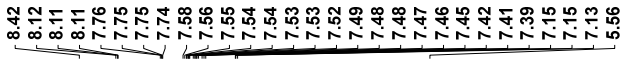
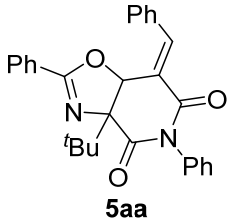
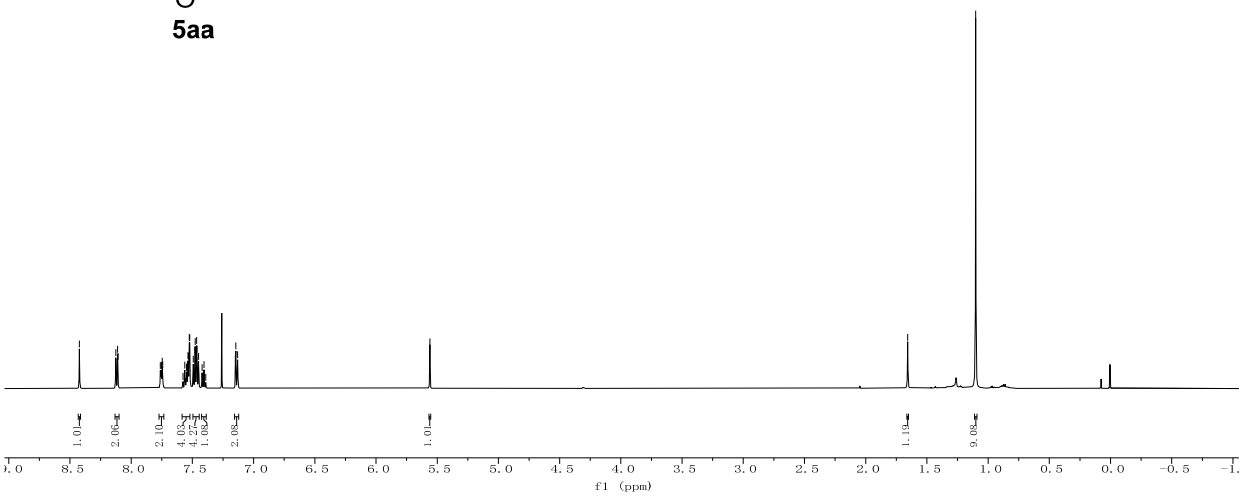
7.89
7.88
7.87
7.75
7.74
7.73
7.73
7.62
7.60
7.60
7.58
7.58
7.50
7.49
7.47
7.35
7.33
7.31
7.25
7.24
7.12
7.11
7.09
7.03
7.03
3.79
3.79
3.76
3.76
3.25
3.25
3.22
3.22
1.24
Ph
O
NHPh
O
N
tBu
Bz
4aa
1.01
2.09
1.95
3.05
2.21
4.14
1.82
0.92
0.97
1.00
1.08
9.09
f1 (ppm)

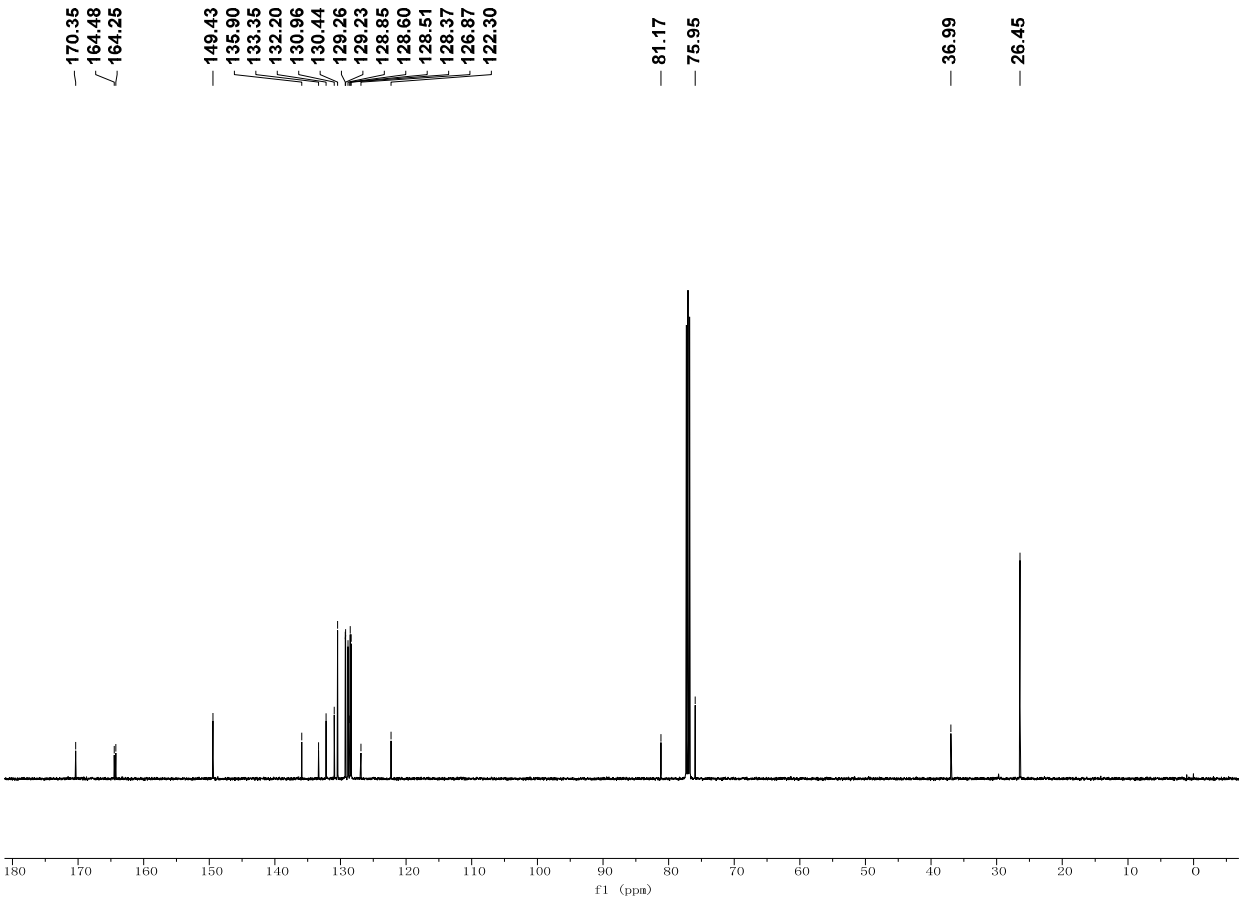

178.29
169.50
168.34
140.62
137.98
133.97
133.63
133.59
130.94
130.53
129.63
128.97
128.72
128.06
126.08
124.48
120.49
80.57
40.17
38.12
27.64
f1 (ppm)


8.42
8.12
8.11
8.11
7.76
7.75
7.75
7.74
7.58
7.56
7.55
7.54
7.54
7.53
7.53
7.52
7.49
7.48
7.48
7.47
7.46
7.45
7.42
7.41
7.39
7.15
7.15
7.13
5.56
1.66
1.10

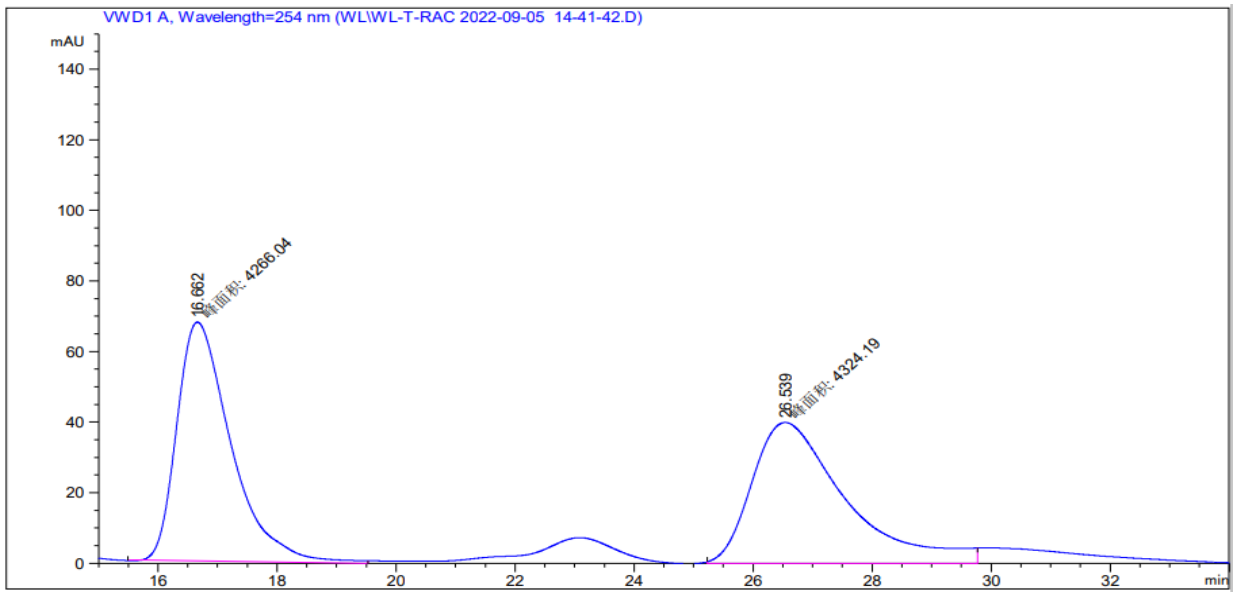

Ph
Ph
O
N
tBu
O
O
N
Ph
O

5aa


1.01
2.06
2.10
4.03
4.27
1.08
2.08
1.01
1.19
9.08
f1 (ppm)

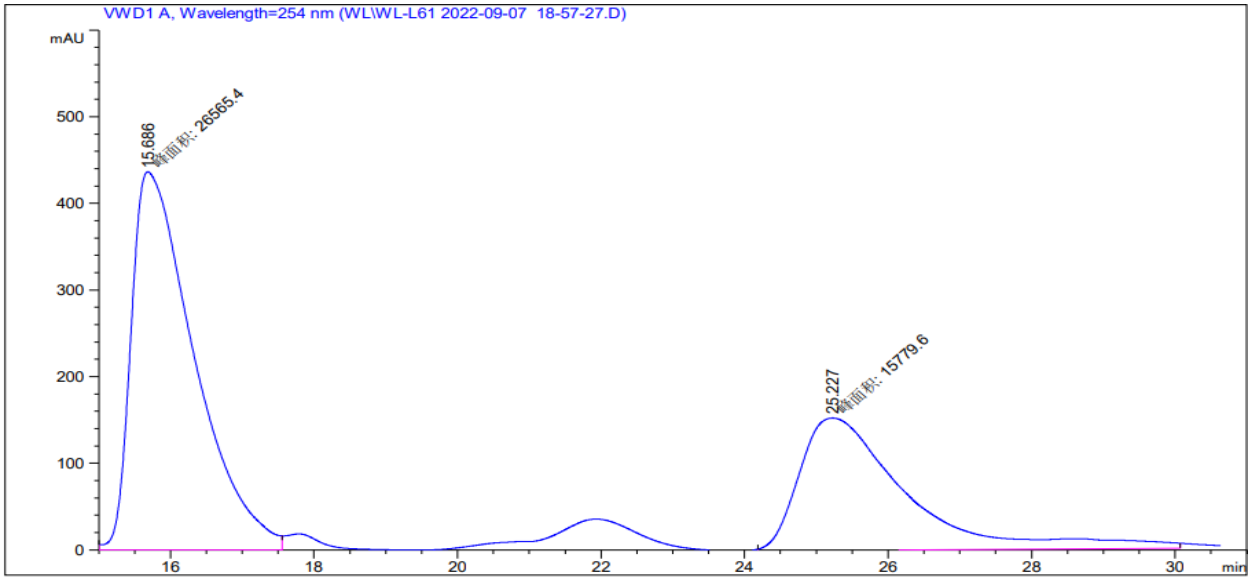

170.35
164.48
164.25
149.43
135.90
133.35
132.20
130.96
130.44
129.26
129.23
128.85
128.60
128.51
128.37
126.87
122.30
81.17
75.95
36.99
26.45
f1 (ppm)

HPLC chromatogram of racemic 3aa



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	16.662	MM	1.0510	4266.03613	67.64932	49.6615
2	26.539	MM	1.8075	4324.18701	39.87323	50.3385

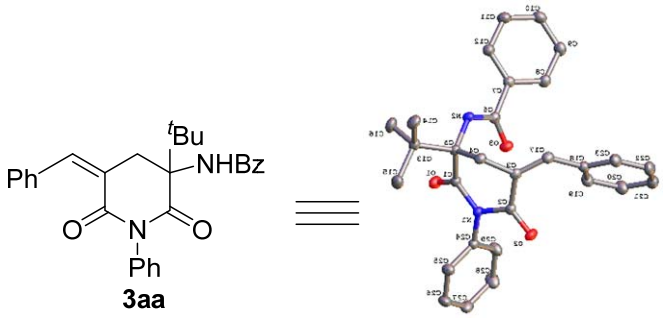
HPLC chromatogram of chiral 3aa



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.686	MM	1.0149	2.65654e4	436.26987	62.7356
2	25.227	MM	1.7226	1.57796e4	152.67024	37.2644

X-ray Crystallographic Data of Product 3aa

Crystallographic data for **3aa** has been deposited with the Cambridge Crystallographic Data Centre as deposition number CCDC 2180470. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223336033.



3aa

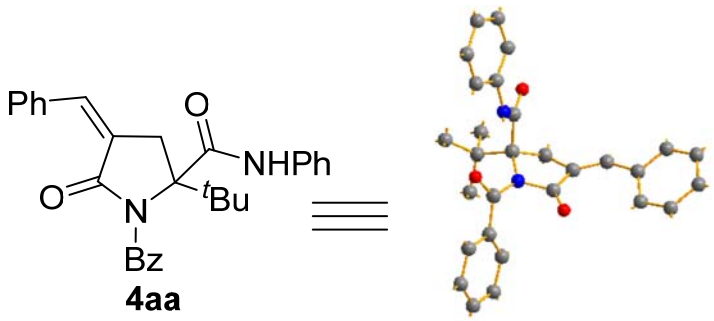
Table S2. Crystal data and structure refinement for 3aa

Identification code	2112299415_b_0m	
Empirical formula	C58 H56 N4 O6	
Formula weight	905.06	
Temperature	213.00 K	
Wavelength	1.34139 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 10.5983(2) Å	a= 90°.
	b = 42.7134(7) Å	b= 107.8230(10)°.
	c = 11.3092(2) Å	g = 90°.
Volume	4873.85(15) Å3	
Z	4	
Density (calculated)	1.233 Mg/m3	
Absorption coefficient	0.406 mm-1	
F(000)	1920	
Crystal size	0.08 x 0.07 x 0.07 mm3	

Theta range for data collection	3.683 to 54.974°.
Index ranges	-12<=h<=11, -52<=k<=50, -13<=l<=10
Reflections collected	50921
Independent reflections	9221 [R(int) = 0.0512]
Completeness to theta = 53.594°	99.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7508 and 0.6402
Refinement method	Full-matrix least-squares on F2
Data / restraints / parameters	9221 / 0 / 619
Goodness-of-fit on F2	1.012
Final R indices [I>2sigma(I)]	R1 = 0.0430, wR2 = 0.1041
R indices (all data)	R1 = 0.0660, wR2 = 0.1175
Extinction coefficient	n/a
Largest diff. peak and hole	0.203 and -0.219 e.Å-3

X-ray Crystallographic Data of Product 4aa

Crystallographic data for **4aa** has been deposited with the Cambridge Crystallographic Data Centre as deposition number CCDC 2194808. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223336033.



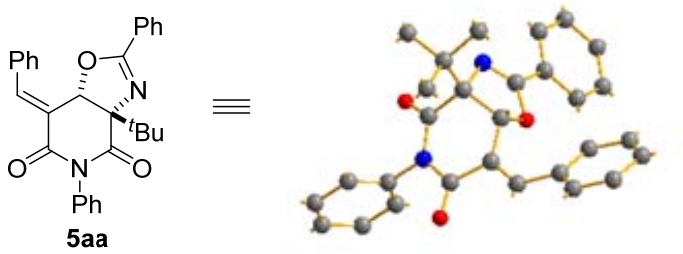
4aa

Table S3. Crystal data and structure refinement for 4aa

Identification code	exp_1755_auto
Empirical formula	C29H28N2O3
Formula weight	452.53
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	C2/c
a/Å	27.6176(2)
b/Å	9.44290(10)
c/Å	17.83660(10)
α/°	90
β/°	92.4570(10)
γ/°	90
Volume/Å3	4647.34(7)
Z	8
ρcalcg/cm3	1.294
μ/mm-1	0.669
F(000)	1920.0
Crystal size/mm3	0.2 × 0.15 × 0.1
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	9.9 to 153.24
Index ranges	-34 ≤ h ≤ 34, -11 ≤ k ≤ 11, -22 ≤ l ≤ 22
Reflections collected	38329
Independent reflections	4720 [Rint = 0.0240, Rsigma = 0.0114]
Data/restraints/parameters	4720/0/311
Goodness-of-fit on F2	1.056
Final R indexes [I>=2σ (I)]	R1 = 0.0367, wR2 = 0.0901
Final R indexes [all data]	R1 = 0.0374, wR2 = 0.0906
Largest diff. peak/hole / e Å-3	0.41/-0.41

X-ray Crystallographic Data of Product 5aa

Crystallographic data for **5aa** has been deposited with the Cambridge Crystallographic Data Centre as deposition number CCDC 2194809. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223336033.



5aa

Table S4. Crystal data and structure refinement for 5aa

Identification code	exp_1756_auto
Empirical formula	C29H26N2O3
Formula weight	450.52
Temperature/K	100.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	9.93777(19)
b/Å	10.32738(11)
c/Å	11.6731(2)
α/°	81.4575(12)
β/°	87.3485(15)
γ/°	81.1675(12)
Volume/Å3	1170.34(3)
Z	2
ρcalcg/cm3	1.278
μ/mm-1	0.663

F(000)	476.0
Crystal size/mm3	0.15 × 0.1 × 0.07
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	7.66 to 133.194
Index ranges	-11 ≤ h ≤ 10, -12 ≤ k ≤ 12, -13 ≤ l ≤ 13
Reflections collected	38919
Independent reflections	4126 [Rint = 0.0289, Rsigma = 0.0125]
Data/restraints/parameters	4126/0/411
Goodness-of-fit on F2	1.030
Final R indexes [I>=2σ (I)]	R1 = 0.0335, wR2 = 0.0833
Final R indexes [all data]	R1 = 0.0343, wR2 = 0.0839
Largest diff. peak/hole / e Å-3	0.25/-0.25