Supporting Information

Palladium/copper-catalyzed multicomponent reactions of propargylic amides, halohydrocarbons and CO_2 toward functionalized oxazolidine-2,4-diones

Cong Zhou, Yaqun Dong, Jin-Tao Yu, Song Sun* and Jiang Cheng*

School of Petrochemical Engineering, Jiangsu Key Laboratory of Advanced Catalytic Materials & Technology, Jiangsu Province Key Laboratory of Fine Petrochemical Engineering, Changzhou University, Changzhou 213164, P. R. China
Email: sunsong@cczu.edu.cn; jiangcheng@cczu.edu.cn

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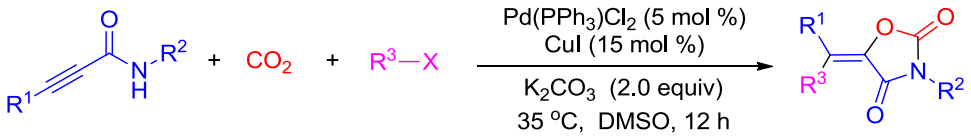
1. General experimental details

All of the manipulations were performed under N_2 atmosphere, using standard Schlenk techniques. Chemicals were used as received without special purification unless stated otherwise. Propargylic amides were prepared according to the published procedure.[1] ^{1}H and ^{13}C NMR were recorded at ambient temperature on a 400 or 300 MHz NMR spectrometer (100 or 75 MHz for ^{13}C NMR). NMR experiments are reported in δ units, parts per million (ppm), and were referenced to $CDCl_3$ (δ 7.26 or 77.0 ppm) or DMSO-d_6 (δ 2.50 or 39.5 ppm) as the internal standard. NMR analysis was carried out at 298 K unless noted otherwise. HRMS was obtained on an ESI-LC-MS/MS spectrometer.

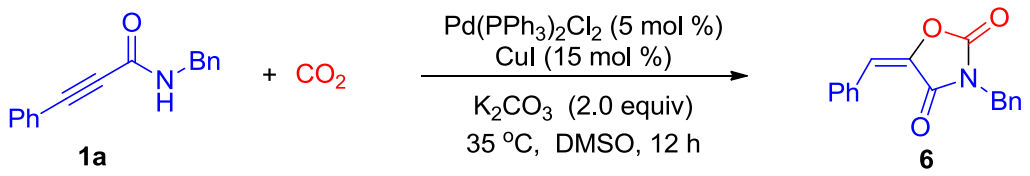
1.1 The reaction of *N*-benzyl-3-phenylpropiolamide, CO_2 and aryl halides:



1 **2** **3**

Propargylic amides **1** (0.2 mmol), aryl halides **2** (0.24 mmol), $Pd(PPh_3)_2Cl_2$ (0.01 mmol, 7.0 mg), CuI (0.03 mmol, 5.7 mg), K_2CO_3 (0.4 mmol, 55.2 mg) and DMSO (2.0 mL) was added into a 20 mL Schlenk tube equipped with a Teflon cap. The reaction vessel was evacuated to about 0.1 MPa (last 30 seconds per time) and backfilled with CO_2 (1 atm) in three times. The sealed Schlenk tube was stirred at oil bath under 35 ^{o}C for 12 h. After the reaction mixture was cooled to room temperature, saturated brine water (3.0 mL) was added to terminate the reaction. The reaction mixture was diluted with EtOAc and washed with saturated brine water. The organic layer was dried over anhydrous $MgSO_4$ and concentrated in vacuum. The residue was purified by flash column chromatography on silica gel with petroleum ether-ethyl acetate as eluent to give the desired product.

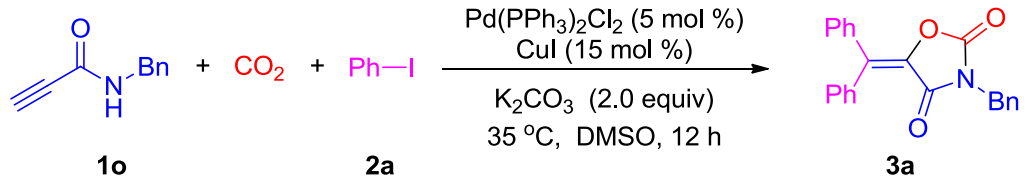
1.2 The reaction of *N*-benzyl-3-phenylpropiolamide and CO_2



1a **6**

Propargylic amides **1a** (0.2 mmol, 47.0 mg), $Pd(PPh_3)_2Cl_2$ (0.01 mmol, 7.0 mg), CuI (0.03 mmol, 5.7 mg), K_2CO_3 (0.4 mmol, 55.2 mg) and DMSO (2.0 mL) was added into a 20 mL Schlenk tube equipped with a Teflon cap. The reaction vessel was evacuated to about 0.1 MPa (last 30 seconds per time) and backfilled with CO_2 (1 atm) in three times. The sealed Schlenk tube was stirred at oil bath under 35 ^oC for 12 h. After the reaction mixture was cooled to room temperature, saturated brine water (3.0 mL) was added to terminate the reaction. The reaction mixture was diluted with EtOAc and washed with saturated brine water. The organic layer was dried over anhydrous $MgSO_4$ and concentrated in vacuum. The residue was purified by flash column chromatography on silica gel with petroleum ether-ethyl acetate as eluent to give the protonation product **6** in 45 % yield.

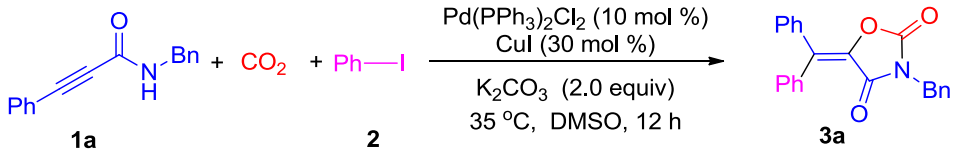
1.3 The reaction of *N*-benzylpropiolamide, CO_2, and iodobenzene



1o **2a** **3a**

Propargylic amides **1o** (0.2 mmol, 31.8 mg), iodobenzene **2a** (0.48 mmol, 97.9 mg), $Pd(PPh_3)_2Cl_2$ (0.01 mmol, 7.0 mg), CuI (0.03 mmol, 5.7 mg), K_2CO_3 (0.4 mmol, 55.2 mg) and DMSO (2.0 mL) was added into a 20 mL Schlenk tube equipped with a Teflon cap. The reaction vessel was evacuated to about 0.1 MPa (last 30 seconds per time) and backfilled with CO_2 (1 atm) in three times. The sealed Schlenk tube was stirred at oil bath under 35 ^oC for 12 h. After the reaction mixture was cooled to room temperature, saturated brine water (3.0 mL) was added to terminate the reaction. The reaction mixture was diluted with EtOAc and washed with saturated brine water. The organic layer was dried over anhydrous $MgSO_4$ and concentrated in vacuum. The residue was purified by flash column chromatography on silica gel with petroleum ether-ethyl acetate as eluent to give the desired product **3** in 53% yield.

1.4 1 mmol scale reaction of compound 1a, CO_2 and iodobenzene



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

Propargylic amides **1** (1.0 mmol, 235 mg), iodobenzene **2a** (1.2 mmol, 245 mg), $Pd(PPh_3)_2Cl_2$ (0.1 mmol, 70 mg), CuI (0.3 mmol, 57 mg), K_2CO_3 (2 mmol, 552 mg) and DMSO (10 mL) was added into a 100 mL Schlenk tube equipped with a Teflon cap. The reaction vessel was evacuated to about 0.1 MPa (last 30 seconds per time) and backfilled with CO_2 (1 atm) in three times. The sealed Schlenk tube was stirred at Oil bath under 35 ^oC for 12 h. After the reaction mixture was cooled to room temperature, saturated brine water (30 mL) was added to terminate the reaction. The reaction mixture was diluted with EtOAc and washed with saturated brine water. The organic layer was dried over anhydrous $MgSO_4$ and concentrated in vacuum. The residue was purified by flash column chromatography on silica gel with petroleum ether-ethyl acetate as eluent to give the desired product **3a** in 55% yield (195 mg).

2. ORTEP diagram of compound 3j.

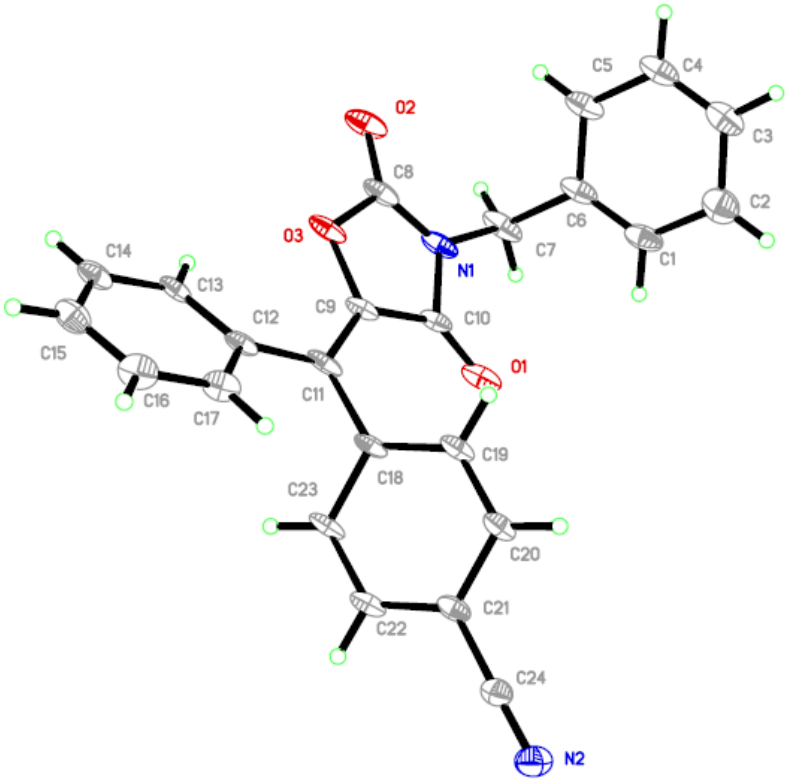
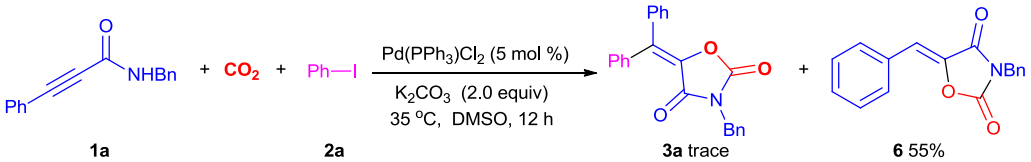


Figure S1

3. Mechanism study

3.1 Pd-catalyzed the reaction of 1a, CO_2 and iodobenzene



Propargylic amides **1a** (0.2 mmol, 47.0 mg), iodobenzene **2a** (0.24 mmol, 48.9 mg), $Pd(PPh_3)_2Cl_2$ (0.01 mmol, 7.0 mg), K_2CO_3 (0.4 mmol, 55.2 mg) and DMSO (2 mL) was added into a 20 mL Schlenk tube equipped with a Teflon cap. The reaction vessel was evacuated to about 0.1 MPa (last 30 seconds per time) and backfilled with CO_2 (1 atm) in three times. The sealed Schlenk tube was stirred at oil bath under 35 ^oC for 12 h. After the reaction mixture was cooled to room temperature, the reaction mixture was analyzed by GC-MS, the corresponding results was shown in Figure S2.

Then, saturated brine water (3.0 mL) was added to terminate the reaction. The reaction mixture was diluted with EtOAc and washed with saturated brine water. The organic layer was dried over anhydrous $MgSO_4$ and concentrated in vacuum. The residue was purified by flash column chromatography on silica gel with petroleum ether-ethyl acetate as eluent to give protonation product **6** in 55 % yield, while trace amount of **3a** only could be detected by GC-MS (Figure S2).

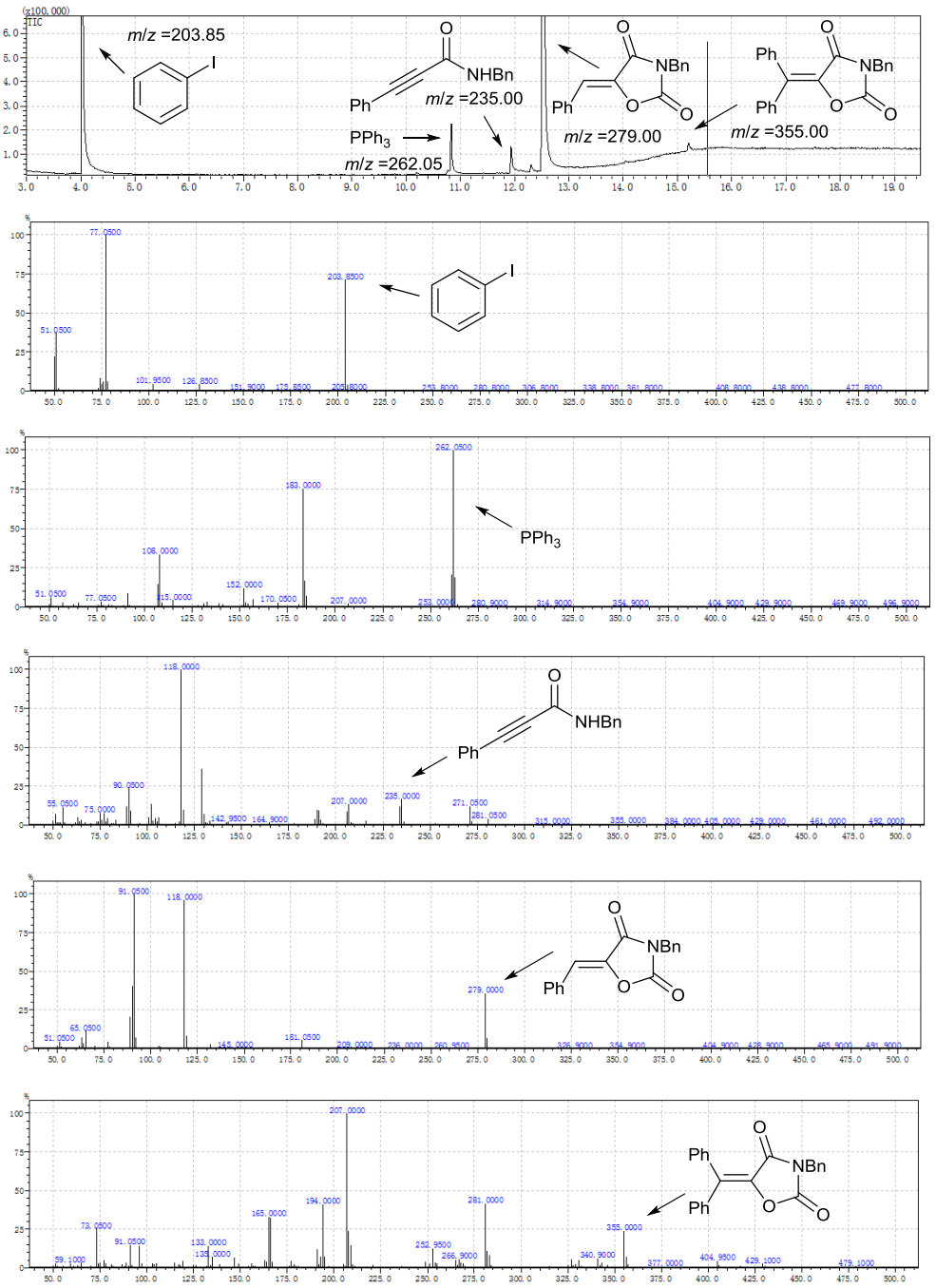
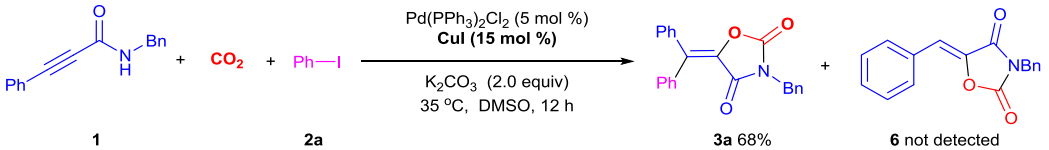


Figure S2

3.2 Pd/Cu-catalyzed the reaction of 1a, CO_2, and iodobenzene



1 **2a** **3a** 68% **6** not detected

Propargylic amides **1a** (0.2 mmol, 47.0 mg), iodobenzene **2a** (0.24 mmol, 48.9 mg), $Pd(PPh_3)_2Cl_2$ (0.01 mmol, 7.0 mg), CuI (0.03 mmol, 5.7 mg), K_2CO_3 (0.4 mmol, 55.2 mg) and DMSO (2 mL) was added into a 20 mL Schlenk tube equipped with a Teflon

cap. The reaction vessel was evacuated to about 0.1 MPa (last 30 seconds per time) and backfilled with CO_2 (1 atm) in three times. The sealed Schlenk tube was stirred at oil bath under 35 ^{o}C for 12 h. After the reaction mixture was cooled to room temperature, the reaction mixture was anlysed by GC-MS, the corresponding results was shown in Figure S3.

Then, saturated brine water (3.0 mL) was added to terminate the reaction. The reaction mixture was diluted with EtOAc and washed with saturated brine water. The organic layer was dried over anhydrous $MgSO_4$ and concentrated in vacuum. The residue was purified by flash column chromatography on silica gel with petroleum ether-ethyl acetate as eluent to give the desired product **3a** in 68% yield, while protonation product **6** was not detected by GC-MS (Figure S3).

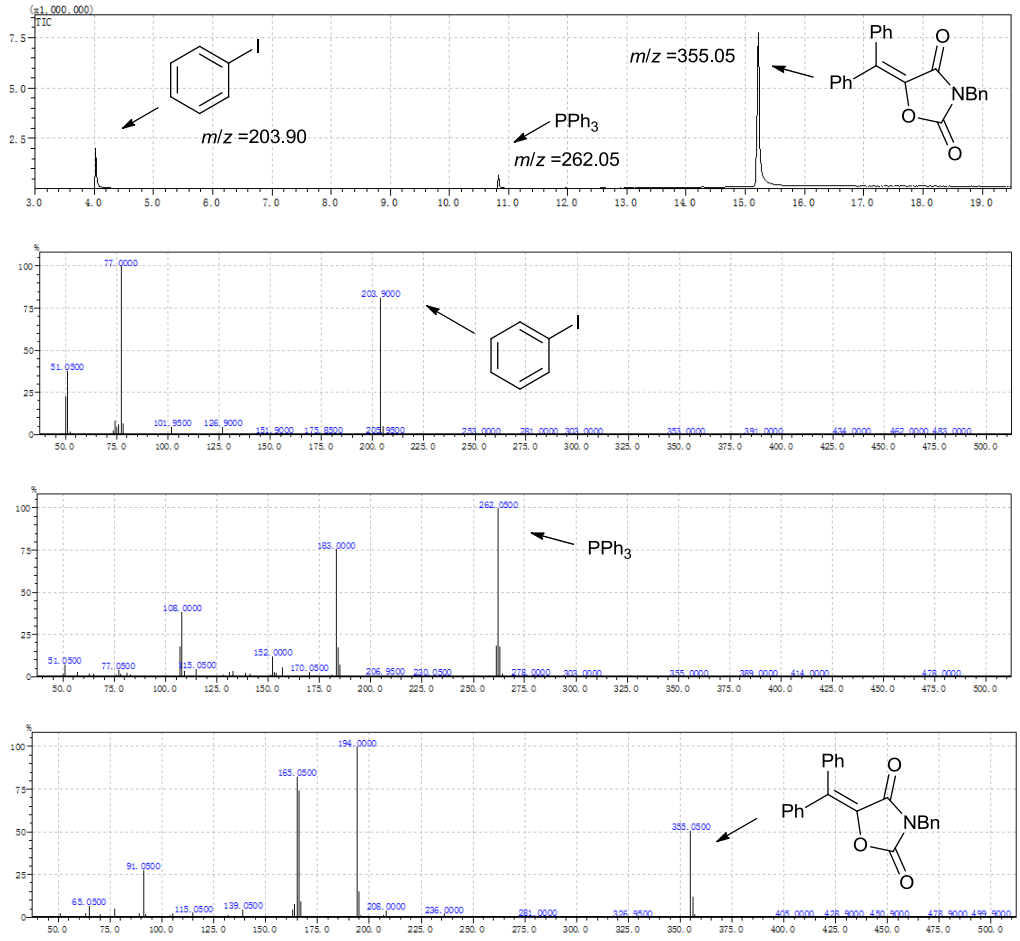
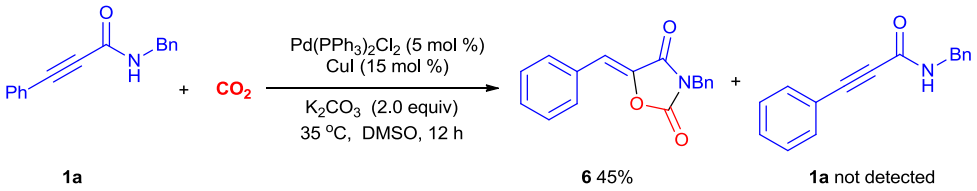


Figure S3

3.3 Pd/Cu-catalyzed the reaction of 1a, and CO_2



1a **6** 45% **1a** not detected

Propargylic amides **1a** (0.2 mmol, 47 mg), $Pd(PPh_3)_2Cl_2$ (0.01 mmol, 7.0 mg), CuI (0.03 mmol, 5.7 mg), K_2CO_3 (0.4 mmol, 55.2 mg) and DMSO (2 mL) was added into a 20 mL Schlenk tube equipped with a Teflon cap. The reaction vessel was evacuated to about 0.1 MPa (last 30 seconds per time) and backfilled with CO_2 (1 atm) in three times. The sealed Schlenk tube was stirred at oil bath under 35 ^oC for 12 h. After the reaction mixture was cooled to room temperature, the reaction mixture was anlysed by GC-MS, the corresponding results was shown in Figure S4.

Then, saturated brine water (3.0 mL) was added to terminate the reaction. The reaction mixture was diluted with EtOAc and washed with saturated brine water. The organic layer was dried over anhydrous $MgSO_4$ and concentrated in vacuum. The residue was purified by flash column chromatography on silica gel with petroleum ether-ethyl acetate as eluent to give the desired product **6** in 45% yield, while starting metaril **1a** was not detected.

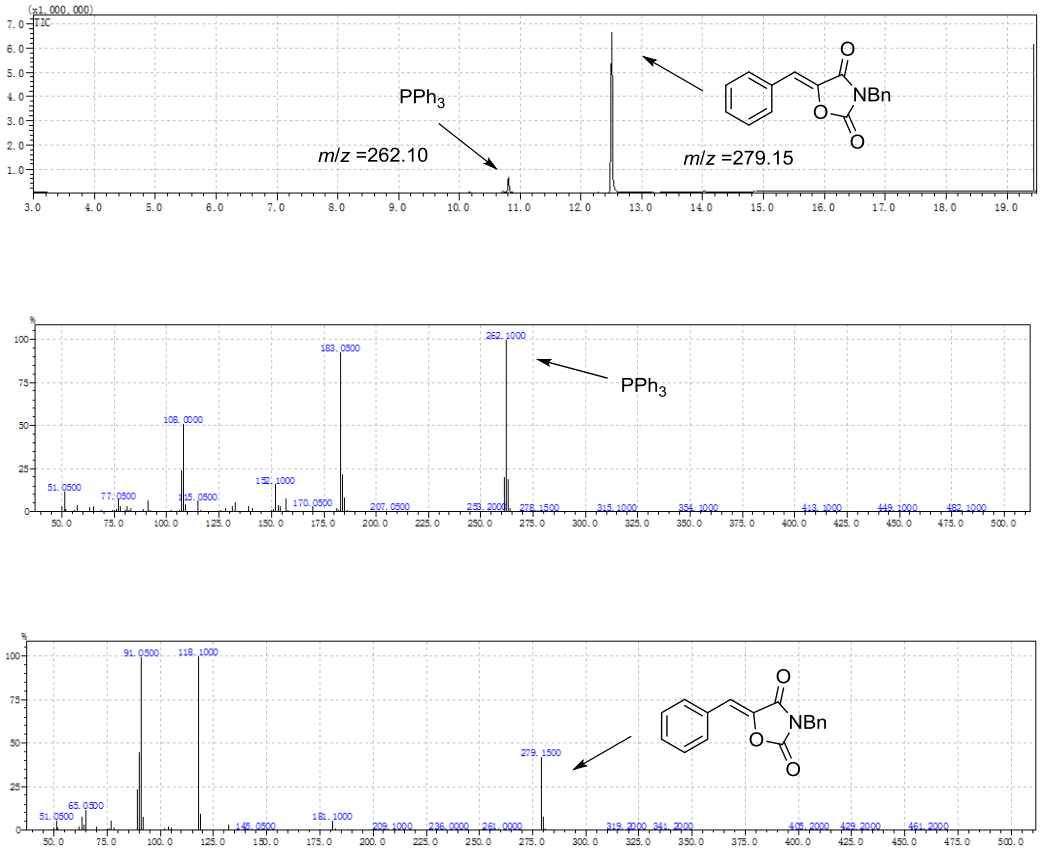
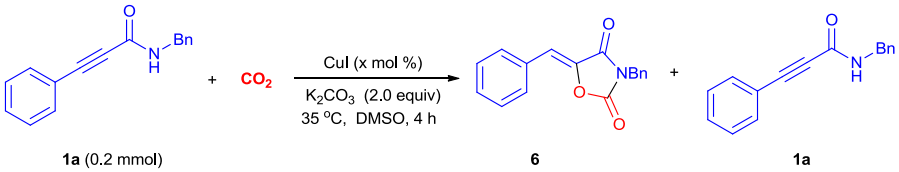


Figure S4

3.4 Control experiment results of the reaction of 1a with CO_2 promoted by CuI

Table 1. Control experiment results



1a (0.2 mmol) **6** **1a**

entry	CuI	yield of **6** (%)	recovered **1a** (%)
1	0	70%	14%
2	15 mol%	42%	43%
3	1.0 equiv	26%	62%

Reaction conditions: *N*-benzyl-3-phenylpropiolamide **1a** (0.2 mmol), CuI (indicated amount),CO_2 (0.1 MPa), K_2CO_3 (0.4 mmol), DMSO (2.0 mL), 4 h, in a sealed Schlenk tube under 35 °C.

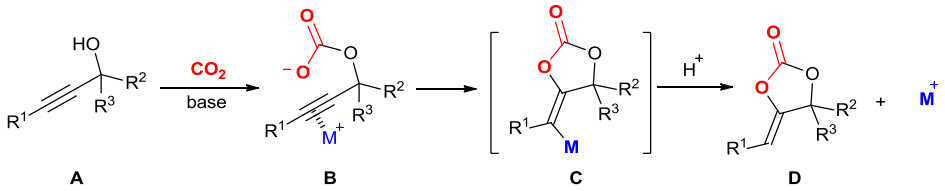
As shown in Table 1, when the reaction of compound **1a** with CO_2 was conducted in the absence of CuI, the protonation product **6** was isolated in 70% yield, along with 14% of **1a** was recovered (Table 1 entry 1). However, in the presence of 15 mol % of

CuI, compound **6** was isolated in 42% yield and 43% of **1a** was recovered (Table 1 entry 2). Moreover, increasing the amount of CuI to 1 equivalent, the yield of compound **6** sharply decreased to 26% and 62% of **1a** was recovered (Table 1 entry 3). Thus, these results strongly indicated CuI acted as inhibitor for the generation of compound **6**.

3.5 The investigation of the special role of CuI

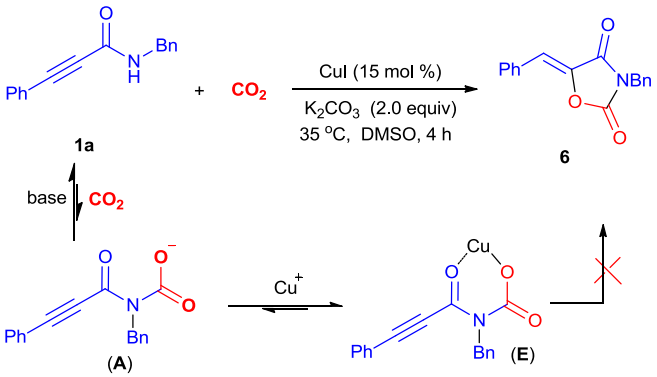
We are very interested in the specific role of CuI in this reaction. In the previous relative works about the reaction of propynylamine, *o*-alkynylaniline or propargylic alcohol with CO_2, Lewis acid such as Cu^+, Ag^+, Au^+ all served as promoter for the aforementioned reaction. ((a) *Adv. Synth. Catal.* 2015, **357**, 2556-2565. (b) *ACS Catal.* 2015, **5**, 5135-5140. (c) *Bull. Chem. Soc. Jpn.* 2011, **84**, 698-717.)

Taking propargylic alcohol **A** as example (Scheme S1), firstly, the M^+ coordinated with the carbon-carbon triple bond, followed by *trans*-oxometalation to form intermediate **C** bearing vinyl-M bond. Afterward, the protonation of **C** afforded **D** with the regeneration of Lewis acid catalyst M^+.



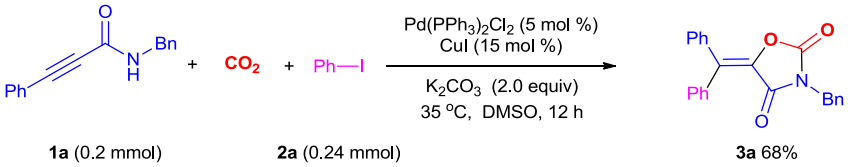
Scheme S1

However, in our current reaction, CuI acted as inhibitor for the reaction of propargylic amide **1a** with CO_2 towards compound **6**. So *trans*-oxocupration of carbon-carbon triple bond to yield vinyl-Cu bond may be not involved in this reaction (Scheme S2). We deduced the formation of a six-membered copper adduct **E** [like $Cu(acac)_2$] during the reaction, which could hamper the further *trans*-oxocupration leading to vinyl-Cu bond. Thus, in the presence of 15 mol % of CuI, the formation of compound **6** was inhibited. We have tried our best to characterize the proposed intermediate **E** by ESI-MS, but no positive results were obtained.



Scheme S2

Furthermore, in our newly developed Pd/Cu catalyzed three component reaction of propargylic amide **1a**, CO_2 and iodobenzene towards functionalized oxazolidine-2,4-diones (Scheme S3). With the addition of 15 mol % CuI, the formation of the protonation product **6** was almost inhibited. So CuI would act as promotor for the formation of the desired 3-benzyl-5-(diphenylmethylene)oxazolidine-2,4-dione **3a**.



Scheme S3

We also tested the CV of the reaction mixture (Figure S5. According to the CV results, the oxidation-reduction potential of Cu(I) indeed changed after the reaction (Figure 1, CV curves A vs B), indicating the formation of copper adduct during the reaction. But the exact structure of the Cu adduct kept unknown at current stage.

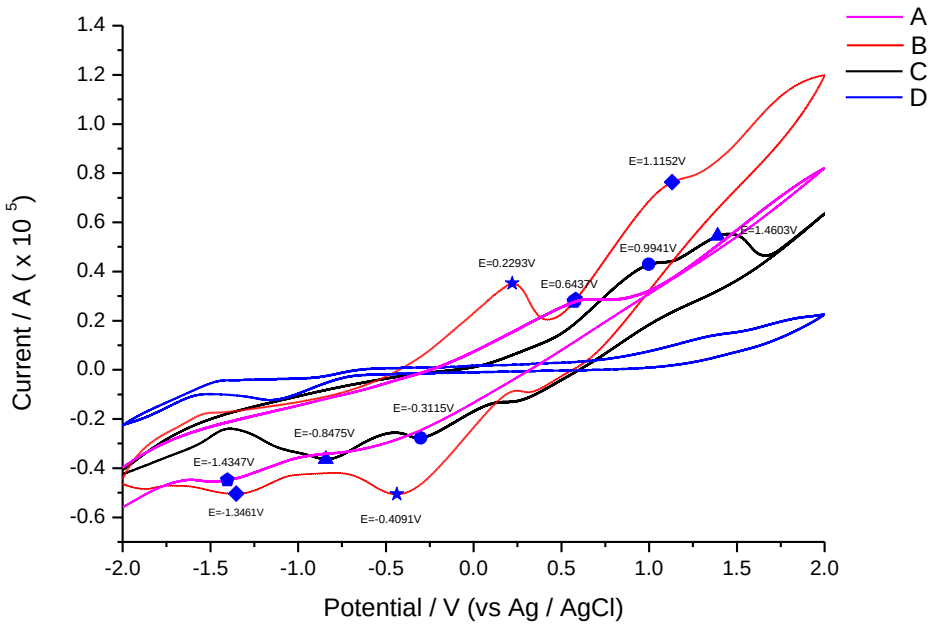
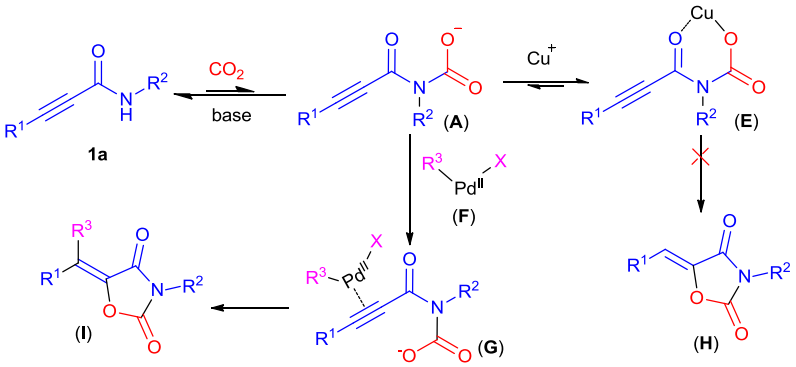


Figure S5: A): *N*-Benzyl-3-phenylpropiolamide (0.1 mmol), CuI (0.1 mmol), K_2CO_3 (0.2 mmol), DMSO (1.5 mL) CO_2 (1 atm) was mixed and stirred in a sealed Schlenk tube, then it was diluted with DMSO into 30 mL for CV test immediately. **B)**: *N*-Benzyl-3-phenylpropiolamide (0.1 mmol), CuI (0.1 mmol), K_2CO_3 (0.2 mmol), DMSO (1.5 mL) CO_2 (1atm) in a sealed Schlenk tube was stirred under 35 ^oC for 5 h, then it was diluted with DMSO into 30 mL for CV test. **C)**: CuI (0.1 mmol), K_2CO_3 (0.2 mmol), DMSO (1.5 mL) CO_2 (1atm) in a sealed Schlenk tube was stirred under 35 ^oC for 5 h, then it was diluted with DMSO into 30 mL for CV test. **D)**: *N*-benzyl-3-phenylpropiolamide (0.1 mmol), K_2CO_3 (0.2 mmol), DMSO (1.5 mL) CO_2 (1atm) in a sealed Schlenk tube was stirred under 35 ^oC for 5 h, then it was diluted with DMSO into 30 mL for CV test.

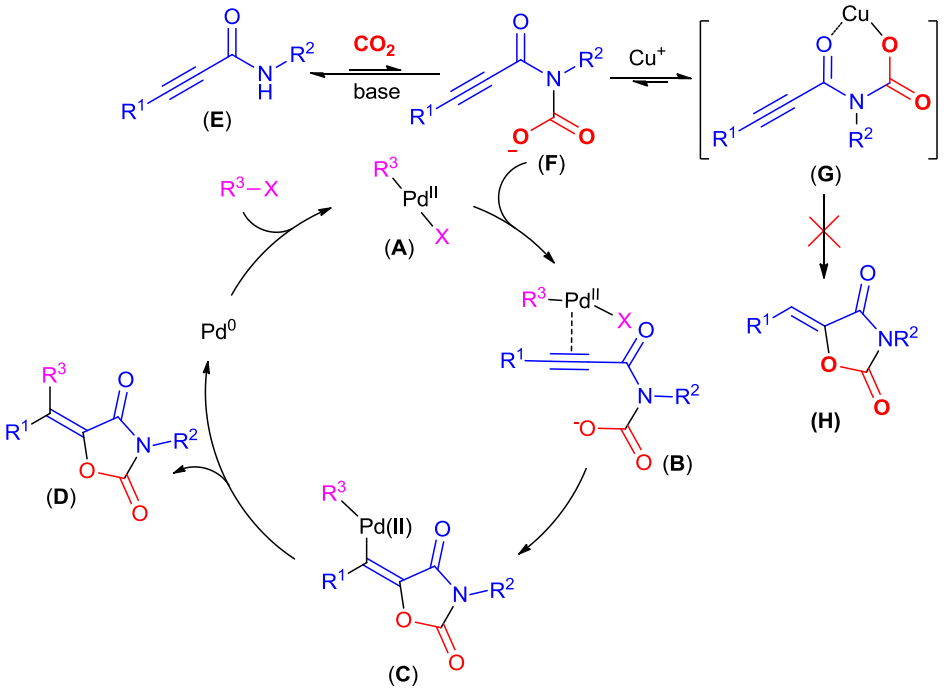
Based on the experiment results obtained, the special role of CuI in these reactions may be explained as follows:

1. According to the theory of hard and soft acids and bases, Cu^+ is hard acid, and it preferred to coordinate with hard base, such as O^-, N^- to form intermediate **E**. which hampered the formation of the protonation product **H** (Scheme S4).
2. However, in the presence of R^3PdX, Pd^{2+} was soft acid, which like to coordinate with soft base, such as the carbon-carbon triple bond. Thus, palladium adduct (**F**) is like to coordinate with carbon-carbon triple bond along with the dissociation of the copper ions in **E** leading to intermediate **G**. Then *trans*-oxopalladation of triple bond in **G** followed by the reductive elimination to generate the desired product **I**. Thus, the ArPdX species was also crucial for the formation of the final product **I**.



Scheme S4

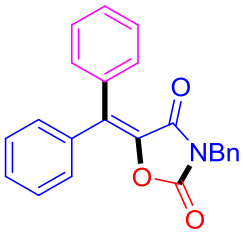
4. Proposed mechanism



Scheme S5

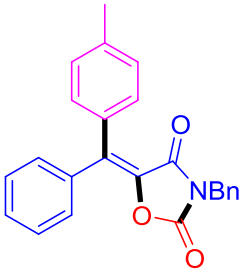
5. Characterization data for the products

3-benzyl-5-(diphenylmethylene)oxazolidine-2,4-dione (3a)



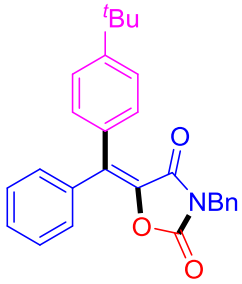
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (48 mg, 68% yield) as a white solid, mp 166.9-168.6 ^oC. The compound is unknown. ^1H NMR ($CDCl_3$ 400 MHz): 7.48-7.41 (m, 7H), 7.31 (s, 1H), 7.40-7.33 (m, 6H), 7.31-7.28 (m, 3H), 4.72 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.7, 152.0, 136.0, 134.9, 134.9, 134.8, 133.3, 130.9, 130.4, 130.0, 129.5, 129.3, 129.0, 128.6, 128.5, 128.4, 43.8. HRMS (ESI-TOF) m/z [M + H]$^+$: calcd. for $[C_{19}H_{22}NO_2]^+$ 356.1281, found 356.1285.

(*E*)-3-benzyl-5-(phenyl(*p*-tolyl)methylene)oxazolidine-2,4-dione (3b)



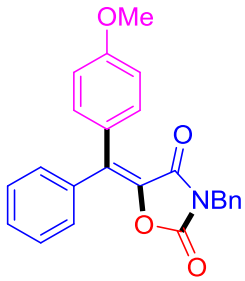
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (52 mg, 70% yield) a white solid, mp 120.0-121.4 ^oC. The compound is unknown. ^1H NMR ($CDCl_3$ 400 MHz): 7.44-7.37 (m, 4H), 7.36-7.28 (m, 6H), 7.22-7.20 (m, 2H), 4.69 (s, 2H), 2.40 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 152.1, 139.7, 136.2, 134.8, 133.8, 132.0, 130.9, 130.5, 129.9, 129.7, 129.3, 129.1, 129.1, 129.0, 129.0, 128.6, 128.6, 128.4, 43.8, 43.7, 21.7. HRMS (ESI-TOF) *m/z* [M + H]$^+$: calcd. for $[C_{24}H_{20}NO_3]^+$ 370.1438, found 370.1446.

(*E*)-3-benzyl-5-((4-(*tert*-butyl)phenyl)(phenyl)methylene)oxazolidine-2,4-dione (3c)



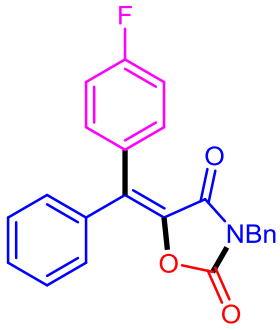
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (60 mg, 73% yield) a white solid, mp 155.9-158.3 ^oC. The compound is unknown. ^1H NMR ($CDCl_3$ 300 MHz): 7.37-7.33 (m, 3H), 7.31 (s, 1H), 7.30-7.26 (m, 5H), 7.26-7.21 (m, 3H), 7.17-7.12 (m, 2H), 4.64 (s, 2H), 1.27 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.7, 152.6, 151.9, 136.3, 134.7, 134.6, 134.1, 131.7, 130.8, 130.3, 129.7, 129.0, 128.9, 128.5, 128.3, 125.1, 43.7, 34.9, 31.3. HRMS (ESI-TOF) *m/z* $[M + H]^+$: calcd. for $[C_{27}H_{26}NO_3]^+$ 412.1907, found 412.1918.

(*E*)-3-benzyl-5-((4-methoxyphenyl)(phenyl)methylene)oxazolidine-2,4-dione (3d)



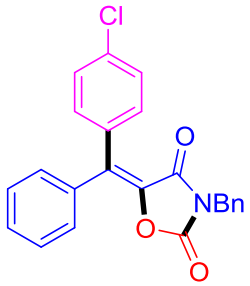
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.5) give the product (56 mg, 72% yield) as a white solid, mp 137.4-139.1 ^oC. The compound is unknown. ^1H NMR ($CDCl_3$ 300 MHz): 7.46-7.41 (m, 2H), 7.38-7.35 (m, 5H), 7.34-7.29 (m, 3H), 7.25-7.23 (m, 1H), 7.22-7.19 (m, 1H), 4.71 (s, 2H), 3.84 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.7, 160.7, 151.9, 136.4, 134.8, 134.4, 133.9, 132.2, 130.8, 129.8, 129.1, 128.9, 128.5, 128.3, 126.8, 113.6, 55.3, 43.7. HRMS (ESI-TOF) *m/z* $[M + H]^+$: calcd. for $[C_{24}H_{20}NO_4]^+$ 386.1387, found 386.1392.

(*E*)-3-benzyl-5-((4-fluorophenyl)(phenyl)methylene)oxazolidine-2,4-dione (3e)



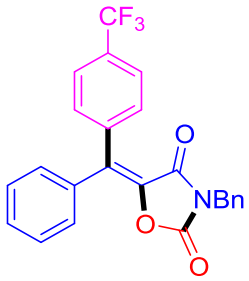
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (50.7 mg, 68% yield) as a colorless white solid, mp 127.5-129.7 ^{o}C. The compound is unknown. ^{1}H NMR ($CDCl_3$ 300 MHz): δ 7.45-7.40 (m, 2H), 7.40-7.36 (m, 5H), 7.36-7.33 (m, 3H), 7.30-7.25 (m, 1H), 7.26-7.22 (m, 1H), 4.71 (s, 2H). ^{13}C NMR ($CDCl_3$ 75 MHz): δ 163.4 (d, $J_{C\text{-}F}$ = 248.2 Hz), 160.6, 151.8, 135.8, 134.8, 134.6, 132.4 (d, $J_{C\text{-}F}$ = 21.7 Hz), 132.2, 130.7, 130.6 (d, $J_{C\text{-}F}$ = 7.9 Hz), 130.0, 129.1, 128.9, 128.5, 128.4, 115.5 (d, $J_{C\text{-}F}$ = 3.4 Hz), 43.8. HRMS (ESI-TOF) *m*/*z* $[M + H]^+$: calcd. for $[C_{23}H_{17}FNO_3]^+$ 374.1187, found 374.1200.

(*E*)-3-benzyl-5-(phenyl(p-tolyl)methylene)oxazolidine-2,4-dione (3f)



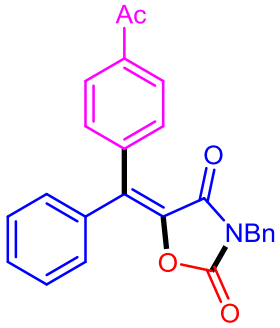
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (50.8 mg, 66% yield) as a white solid, mp 125.2-127.8 ^{o}C. The compound is unknown. ^{1}H NMR ($CDCl_3$ 300 MHz): 7.45-7.39 (m, 3H), 7.39-7.36 (m, 6H), 7.35-7.31 (m, 3H), 4.71 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.5, 151.7, 135.5, 135.5, 134.9, 134.5, 134.4, 133.2, 131.8, 131.7, 130.7, 130.1, 130.0, 129.1, 128.9, 128.6, 128.6, 128.5, 127.6, 43.8. HRMS (ESI-TOF) *m*/*z* $[M + H]^+$: calcd. for $[C_{23}H_{17}ClNO_3]^+$ 390.0891, found 390.0902.

(*E*)-3-benzyl-5-(phenyl(4-(trifluoromethyl)phenyl)methylene)oxazolidine-2,4-dione (3g)



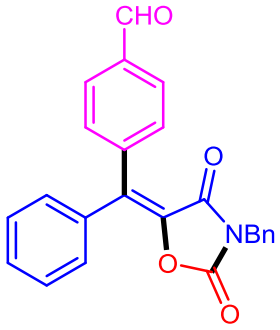
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (51 mg, 60% yield) as a white solid, mp 138.0-139.8 ^{o}C. The compound is unknown. ^{1}H NMR ($CDCl_3$ 300 MHz): 7.70-7.66 (m, 2H), 7.44-7.36 (m, 9H), 7.37-7.30 (m, 3H), 4.71 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.5, 151.6, 138.5, 135.1, 134.4, 131.4, 131.1, 130.9, 130.7, 130.6, 130.2, 128.9,128.8 (q, J_{C-F} = 40.8 Hz), 128.6, 125.8, 125.3 (q, J_{C-F} = 3.7 Hz), 124.0 (q, J_{C-F} = 270.6 Hz), 43.8. HRMS (ESI-TOF) *m*/*z* [M + H]$^{+}$: calcd. for $[C_{24}H_{17}F_3NO_3]^{+}$ 424.1155, found 424.1172.

(*E*)-5-((4-acetylphenyl)(phenyl)methylene)-3-benzyloxazolidine-2,4-dione (3h)



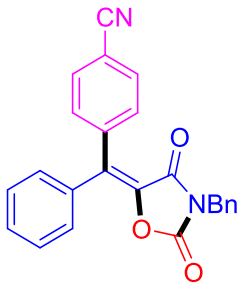
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (54 mg, 68% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): 8.15 (d, *J* = 8.3, 2H), 7.43-7.37 (m, 9H), 7.35-7.32 (m, 3H), 4.71 (s, 2H), 2.64 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.6, 160.6, 151.8, 139.8, 137.5, 135.2, 134.6, 131.6, 130.8, 130.7, 130.3, 129.3, 129.0, 128.7, 128.7, 128.4, 44.0, 26.9. HRMS (ESI-TOF) *m*/*z* [M + H]$^{+}$: calcd. for $[C_{25}H_{20}NO_4]^{+}$ 398.1387, found 398.1401.

(*E*)-4-((3-benzyl-2,4-dioxooxazolidin-5-ylidene)(phenyl)methyl)benzaldehyde (3i)



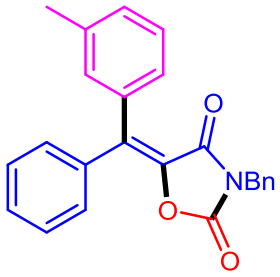
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (49 mg, 64% yield) as a yellow oil. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): 8.32-8.21 (m, 2H), 7.50-7.47 (m, 2H), 7.44-7.36 (m, 10H), 4.73 (s, 2H), 2.64 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.7, 160.5, 151.6, 141.1, 136.5, 135.1, 134.9, 134.4, 131.1, 131.0, 130.6, 130.2, 129.6, 129.1, 128.9, 128.6, 128.6, 43.9. HRMS (ESI-TOF) *m/z* [M + Na]$^+$: calcd. for $[C_{24}H_{17}NNaO_4]^+$ 406.1050, found 406.1047.

(*E*)-4-((3-benzyl-2,4-dioxooxazolidin-5-ylidene)(phenyl)methyl)benzonitrile(3j)



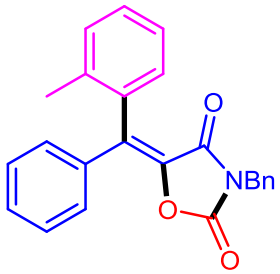
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.3) give the product (46.3 mg, 61% yield) as a brown solid, mp 175.2-177.8 ^{o}C. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): 7.73-7.70 (m, 2H), 7.44-7.32 (m, 13H), 4.71 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.6, 151.7, 139.8, 135.3, 134.8, 134.5, 132.2, 131.3, 130.7, 130.5, 130.5, 129.3, 129.1, 128.8, 118.6, 113.1, 44.0. HRMS (ESI-TOF) *m/z* [M + Na]$^+$: calcd. for $[C_{24}H_{16}N_2O_3Na]^+$ 403.1053, found 403.1052.

(*E*)-3-benzyl-5-(phenyl(*m*-tolyl)methylene)oxazolidine-2,4-dione (3k)



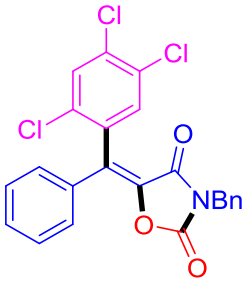
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (47.2 mg, 64% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$ 300 MHz): 7.37-7.11 (m, 4H), 7.30-7.25 (m, 3H), 7.25-7.15 (m, 5H), 7.04-6.95 (m, 2H), 4.62 (s, 2H), 2.27 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.5, 152.0, 138.0, 136.0, 134.7, 134.7, 133. 3, 130.8, 130.7, 130.1, 129.8, 129.1, 128.9, 128.5, 128.3, 128.1, 127.4, 43.7, 21.5. HRMS (ESI-TOF) *m*/*z* [M + H]$^+$: calcd. for $[C_{24}H_{20}NO_3]^+$ 370.1438, found 370.1446.

(*E*)-3-benzyl-5-(phenyl(*o*-tolyl)methylene)oxazolidine-2,4-dione (3l)



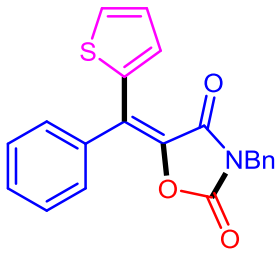
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (42.1 mg, 57% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$ 300 MHz): 7.46-7.38 (m, 2H), 7.35-7.15 (m, 11H), 7.09-7.05 (m, 1H), 4.60 (s, 2H). 1.97 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.5, 152.0, 136.6, 134.9, 134.7, 134.6, 134.3, 131.1, 130.5, 130.4, 123.0, 129.9, 129.1, 129.0, 128.9, 128.6, 128.5, 126.0, 43.7, 19.7. HRMS (ESI-TOF) *m*/*z* [M + H]$^+$calcd. for $[C_{24}H_{20}NO_3]^+$: 370.1438, found 370.1445.

(*E*)-3-benzyl-5-(phenyl(3,4,5-trichlorophenyl)methylene)oxazolidine-2,4-dione (3m)



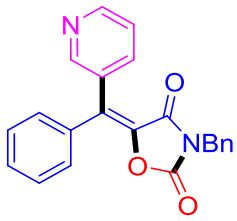
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:3, R_f = 0.43) give the product (42 mg, 46% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): 7.64-7.62 (m 1H), 7.51-7.47 (m, 2H), 7.44-7.40 (m, 5H), 7.37-7.32 (m, 4H), 4.72 (d, *J* = 3.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.4, 151.6, 136.2, 134.5, 134.4, 134.1, 133.9, 133.3, 133.1, 132.6, 131.7, 131.4, 130.6, 130.2, 129.2, 129.1, 129.0, 128.8, 127.8, 125.6, 44.1. HRMS (ESI-TOF) *m*/*z* $[M + Na]^+$: calcd. for $[C_{23}H_{14}Cl_3NO_3Na]^+$ 479.9931, found 479.9925.

(*E*)-3-benzyl-5-(phenyl(thiophen-2-yl)methylene)oxazolidine-2,4-dione (3n)



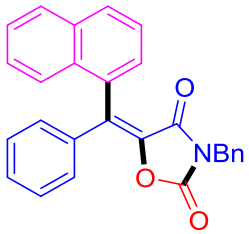
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:3, R_f = 0.6) give the product (42.5 mg, 59% yield) as a brown solid, mp 152.7-153.3^{o}C. The compound is unknown. ^{1}H NMR ($CDCl_3$ 300 MHz): 7.53-7.49 (m, 2H), 7.48-7.44 (m, 2H), 7.43-7.37 (m, 5H), 7.37-7.24 (m, 4H), 4.75 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.3, 151.5, 136.5, 136.1, 134.6, 134.4, 134.4, 132.7, 130.3, 130.03, 129.99, 129.7, 129.1, 128.9, 128.5, 128.3, 127.8, 127.6, 127.3, 43.8. HRMS (ESI-TOF) *m*/*z* $[M + H]^+$: calcd. for $[C_{21}H_{16}NO_3S]^+$ 362.0845, found 362.0854.

(*E*)-3-benzyl-5-(phenyl(pyridin-3-yl)methylene)oxazolidine-2,4-dione (3o)



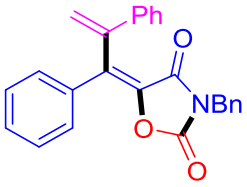
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (36.3 mg, 51% yield) as a yellow oil. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): 8.69-8.52 (m, 2H), 7.62-7.58 (m, 1H), 7.44-7.38 (m, 7H), 7.37-7.31 (m, 4H), 4.71 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.7, 151.7, 150.8, 150.3, 137.9, 135.5, 135.2, 134.5, 131.1, 130.8, 130.4, 129.3, 129.2, 129.1, 128.7, 123.2, 44.0. HRMS (ESI-TOF) *m*/*z* [M + H]$^+$: calcd. for $[C_{22}H_{17}N_2O_3]^+$ 357.1234, found 357.1241.

(*E*)-3-benzyl-5-(naphthalen-1-yl(phenyl)methylene)oxazolidine-2,4-dione (3p)



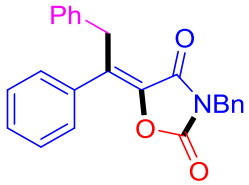
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (44.6 mg, 55% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): 7.86 (d, *J* = 8.3 Hz, 1H), 7.78 (d, *J* = 8.2 Hz, 1H), 7.52 (d, *J* = 8.3 Hz, 1H), 7.47-7.42 (m, 3H), 7.36-7.29 (m, 2H), 7.26-7.15 (m, 9H), 4.51 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.4, 152.1, 135.9, 135.3, 134.7, 133.9, 132.2, 130.5, 130.2, 130.2, 129.6, 129.2, 129.0, 128.8, 128.7, 128.6, 128.2, 126.9, 126.2, 125.5, 125.1, 43.8. HRMS (ESI-TOF) *m*/*z* [M + H]$^+$: calcd. for $[C_{27}H_{20}NO_3]^+$ 406.1447, found 406.1438.

(*E*)-3-benzyl-5-(1,2-diphenylallylidene)oxazolidine-2,4-dione (3q)



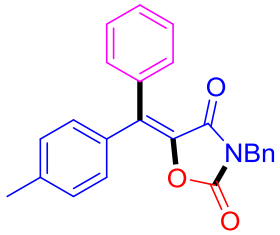
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (40 mg, 52% yield) as a yellow oil. The compound is unknown. ^{1}H NMR (DMSO 400 MHz): 7.65-7.62 (m, 2H), 7.51-7.48 (m, 2H), 7.42-7.34 (m, 8H), 7.29-7.22 (m, 3H), 6.07 (s, 1H) , 5.54 (s, 1H) , 4.67 (s, 2H). ^{13}C NMR (126 MHz, DMSO) δ 160.5, 151.8, 140.7, 137.5, 136.5, 135.1, 133.8, 129.4, 129.4, 128.7, 128.6, 128.4, 128.4, 127.9, 127.8, 127.8, 126.4, 118.6, 42.9.HRMS (ESI-TOF) m/z [M + Na]$^+$: calcd. for $[C_{25}H_{19}NO_3Na]^+$ 404.1257, found 404.1255.

(*Z*)-3-benzyl-5-(1,2-diphenylethylidene)oxazolidine-2,4-dione (3r)



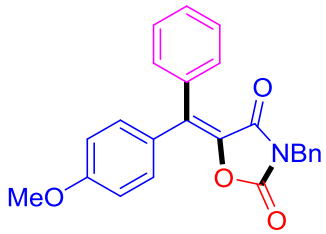
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.6) give the product (22.1 mg, 30% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): 7.50-7.46 (m, 2H), 7.42-7.32 (m, 8H), 7.23-7.10 (m, 5H), 4.80 (s, 2H) , 4.50 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.0, 151.8, 137.7, 135.6, 134.9, 134.8, 133.5, 129.5, 129.1, 129.1, 129.0, 128.8, 128.7, 128.6, 126.8, 43.8, 35.5. HRMS (ESI-TOF) *m/z* [M + Na]$^+$:calcd. for $[C_{24}H_{19}NO_3Na]^+$ 392.1257, found 392.1265.

(*Z*)-3-benzyl-5-(phenyl(*p*-tolyl)methylene)oxazolidine-2,4-dione (4a)



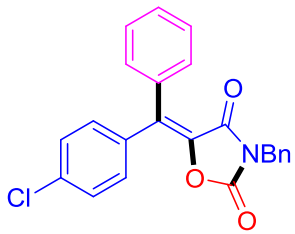
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.5) give the product (51.7 mg, 70% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$, 400 MHz): 7.50-7.43 (m, 5H), 7.39-7.29 (m, 7H), 7.22-7.19 (m, 2H), 4.73 (s, 2H), 2.40 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 152.1, 140.5, 135.1, 134.9, 134.5, 133.3, 133.2, 131.0, 130.5, 129.4, 129.3, 129.0, 128.6, 128.4, 43.8, 21.6. HRMS (ESI-TOF) m/z [M + H]$^+$: calcd. for $[C_{24}H_{20}NO_3]^+$ 370.1438, found 370.1447.

(*Z*)-3-benzyl-5-((4-methoxyphenyl)(phenyl)methylene)oxazolidine-2,4-dione (4b)



Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:3, R_f = 0.6) give the product (32.3 mg, 42% yield) as a orange oil. The compound is unknown. ^{1}H NMR ($CDCl_3$, 400 MHz): *δ* 7.46-7.38 (m, 7H), 7.32-7.29 (m, 3H), 7.26-7.23 (m, 2H), 4.68 (s, 2H), 3.81 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.0, 160.8, 152.2, 135.2, 134.9, 133.8, 132.9, 130.4, 129.3, 129.2, 129.0, 128.6, 128.4, 128.2, 113.9, 55.5, 43.7. HRMS (ESI-TOF) *m/z* [M + H]$^+$: calcd. for $[C_{24}H_{20}NO_4]^+$ 386.1387, found 386.1398.

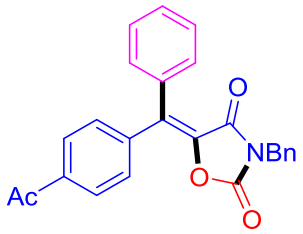
(*Z*)-3-benzyl-5-((4-chlorophenyl)(phenyl)methylene)oxazolidine-2,4-dione (4c)



Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:3, R_f = 0.6) give the product (47.5 mg, 61% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$, 400 MHz): 7.48-7.41 (m, 5H), 7.39-7.31 (m, 7H), 7.29-7.26 (m, 1H), 7.26-7.25 (m, 1H), 4.71 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ

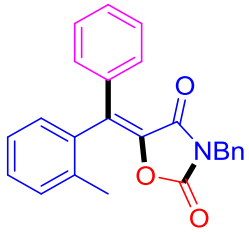
160.5, 151.8, 136.2, 135.0, 134.7, 134.5, 134.4, 132.2, 131.7, 130.4, 129.7, 129.3, 129.0, 128.8, 128.8, 128.5, 43.9. HRMS (ESI-TOF) *m/z* [M + Na]$^+$calcd. for [$C_{23}H_{16}ClNO_3Na$]$^+$ 412.0711, found 412.0715.

(*Z*)-5-((4-acetylphenyl)(phenyl)methylene)-3-benzyloxazolidine-2,4-dione (4d)



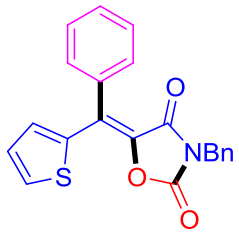
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:2, R_f = 0.43) give the product (60.3 mg, 76% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$, 300 MHz): 7.95-7.92 (m, 2H), 7.53-7.42 (m, 8H), 7.36-7.31 (m, 3H), 7.29-7.26 (m, 2H), 4.71 (s, 2H), 2.60 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.5, 160.4, 151.7, 140.5, 137.5, 135.7, 134.6, 134.3, 131.7, 131.0, 130.4, 129.8, 129.3, 129.0, 128.7, 128.6, 128.3, 44.0, 26.9. HRMS (ESI-TOF) *m/z* [M + Na]$^+$calcd. for [$C_{25}H_{19}NO_4Na$]$^+$ 420.1206, found 420.1199.

(*Z*)-3-benzyl-5-(phenyl(*o*-tolyl)methylene)oxazolidine-2,4-dione (4e)



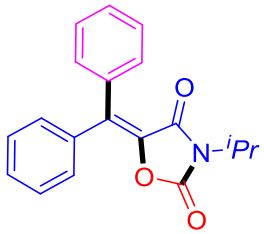
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:3, R_f = 0.6) give the product (48 mg, 65% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$, 400 MHz): 7.49-7.46 (m, 2H), 7.43-7.30 (m, 9H), 7.29-7.20 (m, 3H), 7.15-7.12 (m, 1H), 4.76 (s, 2H), 2.15 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.4 151.8, 136.3, 136.2, 135.5, 135.1, 134.8, 134.2, 130.9, 130.3, 129.9, 129.5, 129.3, 129.1, 128.7, 128.1, 126.0, 43.9, 20.2. HRMS (ESI-TOF) m/z [M + H]$^+$: calcd. for [$C_{24}H_{20}NO_3$]$^+$ 370.1438, found 370.1448.

(*Z*)-3-benzyl-5-(phenyl(thiophen-2-yl)methylene)oxazolidine-2,4-dione (4f)



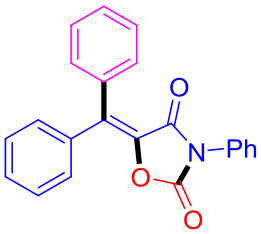
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:15, R_f = 0.45) give the product (38 mg, 53% yield) as a brown solid, mp 145.6-147.8 ^{o}C. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 7.63-7.61 (m, 1H), 7.53-7.46 (m, 3H), 7.43-7.40 (m, 2H), 7.37-7.30 (m, 5H), 7.26-7.23 (m, 1H), 7.10-7.07 (m, 1H), 4.70 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.3, 151.7, 139.1, 134.8, 133.8, 133.8, 132.6, 132.4, 129.9, 129.4, 129.2, 129.0, 128.6, 128.5, 127.9, 126.0, 43.8. HRMS (ESI-TOF) *m/z* [M + Na]$^{+}$calcd. for $[C_{21}H_{15}NO_3SNa]^{+}$ 384.0665, found 384.0661.

5-(diphenylmethylene)-3-isopropyloxazolidine-2,4-dione (4g)



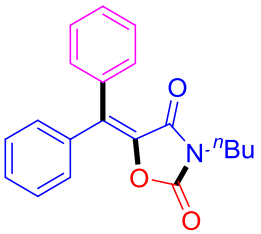
Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:3, R_f = 0.6) give the product (40 mg, 65% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): 7.47-7.41 (m, 5H), 7.40-7.35 (m, 3H), 7.32-7.28 (m, 2H), 4.40-4.32 (m, 1H), 1.45 (d, *J* = 7.0 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.0, 151.5, 136.1, 135.1, 134.6, 132.2, 130.8, 130.3, 129.8, 129.3, 128.5, 128.4, 45.2, 19.5. HRMS (ESI-TOF) *m/z* [M + H]$^{+}$: calcd. for $[C_{19}H_{18}NO_3]^{+}$ 308.1281, found 308.1291.

5-(diphenylmethylene)-3-phenyloxazolidine-2,4-dione (4h)



Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:3, R_f = 0.49) give the product (21.1 mg, 31% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$, 400 MHz): 7.53-7.49 (m, 2H), 7.47-7.45 (m, 4H), 7.45-7.37 (m, 7H), 7.35-7.32 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) 159.9, 150.9, 136.0, 134.9, 134.4, 134. 0, 131. 0, 130.8, 130.4, 130.2, 129.5, 129.4, 129.0, 128.6, 128.5, 125.7. HRMS (ESI-TOF) *m/z* [M + H]$^+$calcd. for $[C_{22}H_{16}NO_3]^+$ 342.1125, found 342.1136.

3-butyl-5-(diphenylmethylene)oxazolidine-2,4-dione (4i)

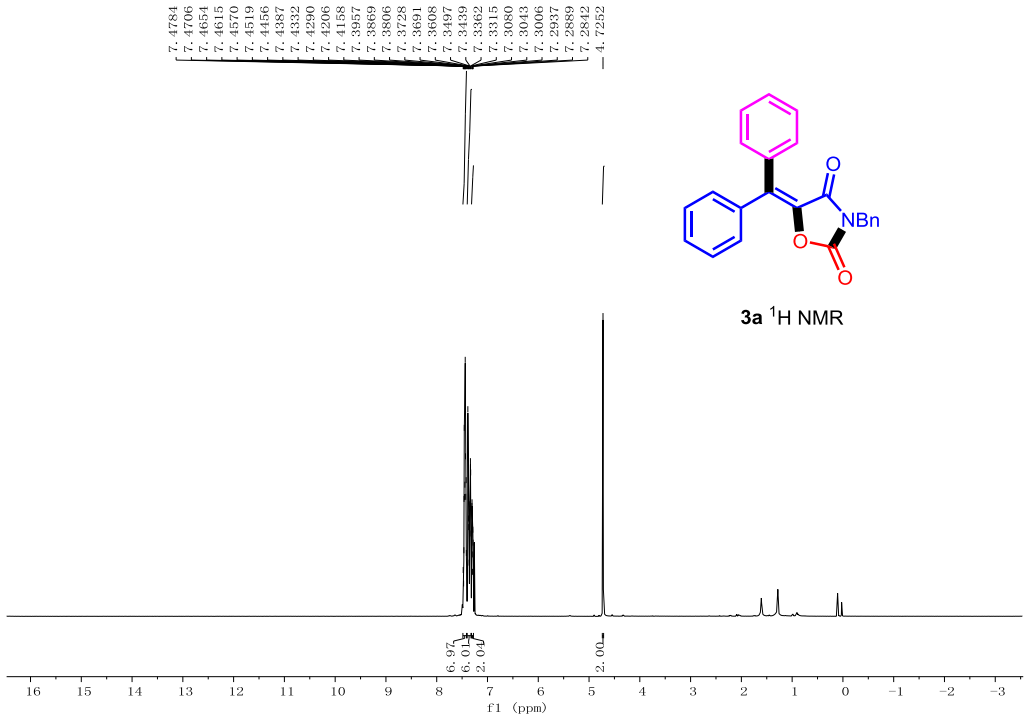


Flash column chromatography on a silica gel (ethyl acetate-petroleum ether, 1:3, R_f = 0.46) give the product (43 mg, 67% yield) as a colorless oil. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): δ7.49-7.41 (m, 5H), 7.40-7.36 (m, 3H), 7.32-7.28 (m, 2H), 3.58 (t, *J* = 7.4 Hz, 2H), 1.70-1.63 (m, 2H), 1.40-1.32 (m, 2H), 0.93 (t, *J* = 7.4, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.1, 152.3, 136.0, 135.0, 134.9, 132.7, 130.9, 130.4, 129.9, 129.4, 128.5, 128.4, 40.1, 29.8 20.1 13.7. HRMS (ESI-TOF) *m/z* [M + Na]$^+$calcd. for $[C_{20}H_{19}NO_3Na]^+$ 344.1257, found 344.1253.

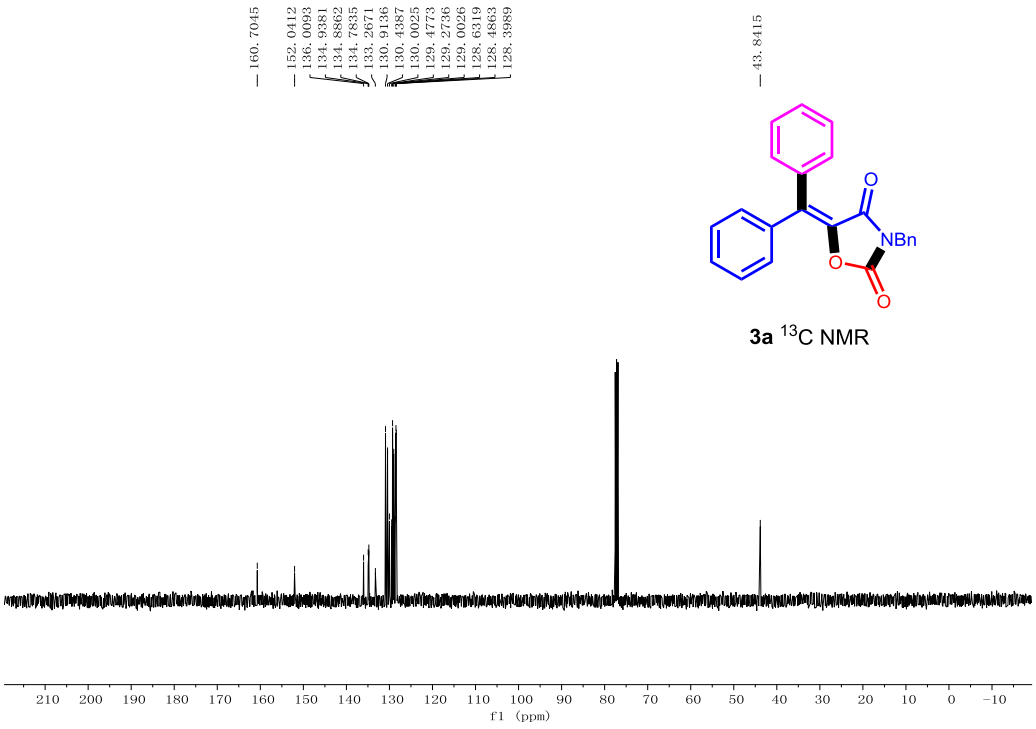
References

[1] Y. Zhou, X. Zhang, Y. Zhang, L. Ruan, J. Zhang, D. Zhang-Negrerie and Y. Du. *Org. Lett.*, 2017**, 19**, 150-153.

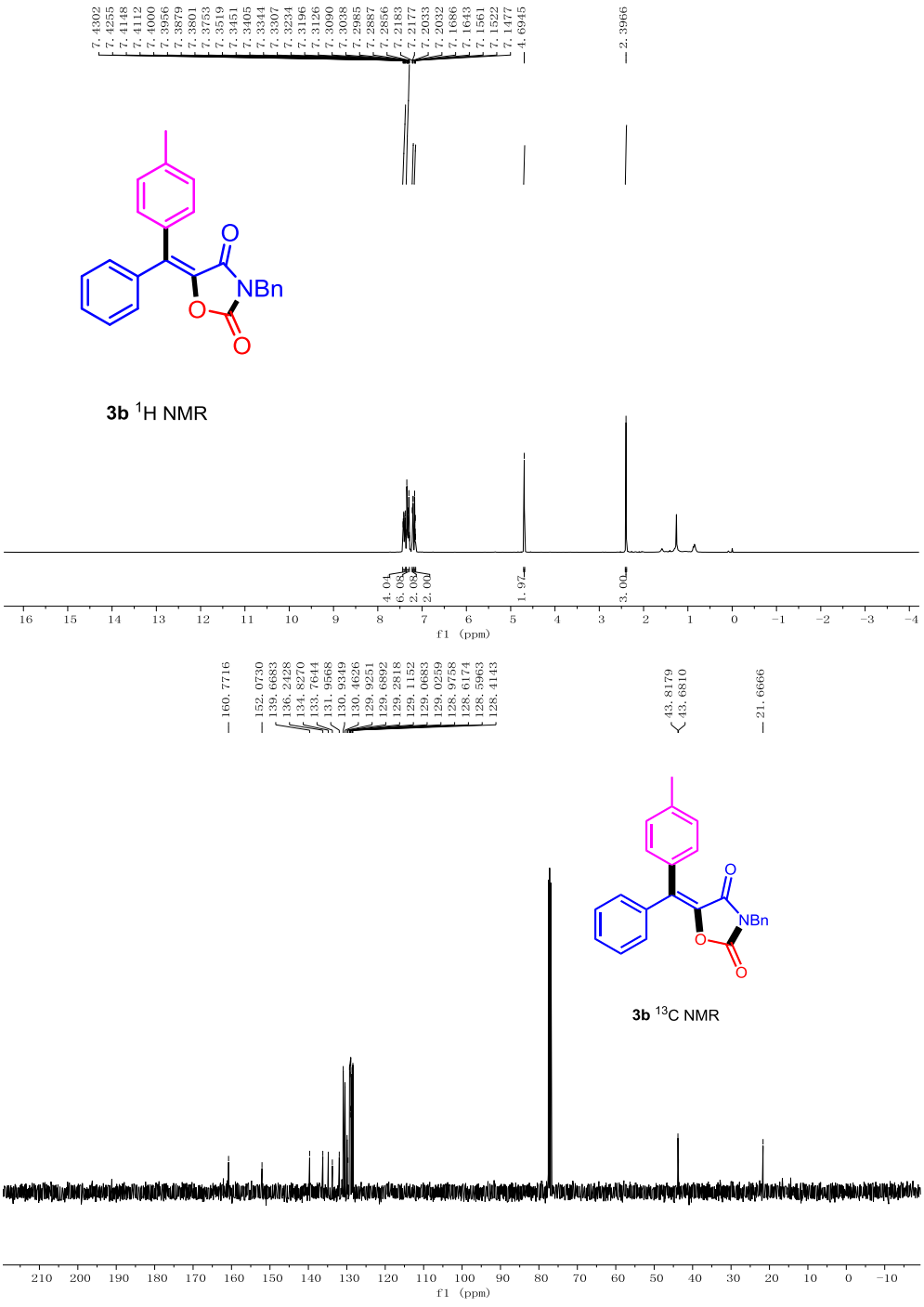
6. Copies of ^{1}H NMR and ^{13}C NMR spectra of 3a-r, 4a-i



3a ^{1}H NMR



3a ^{13}C NMR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7.3519
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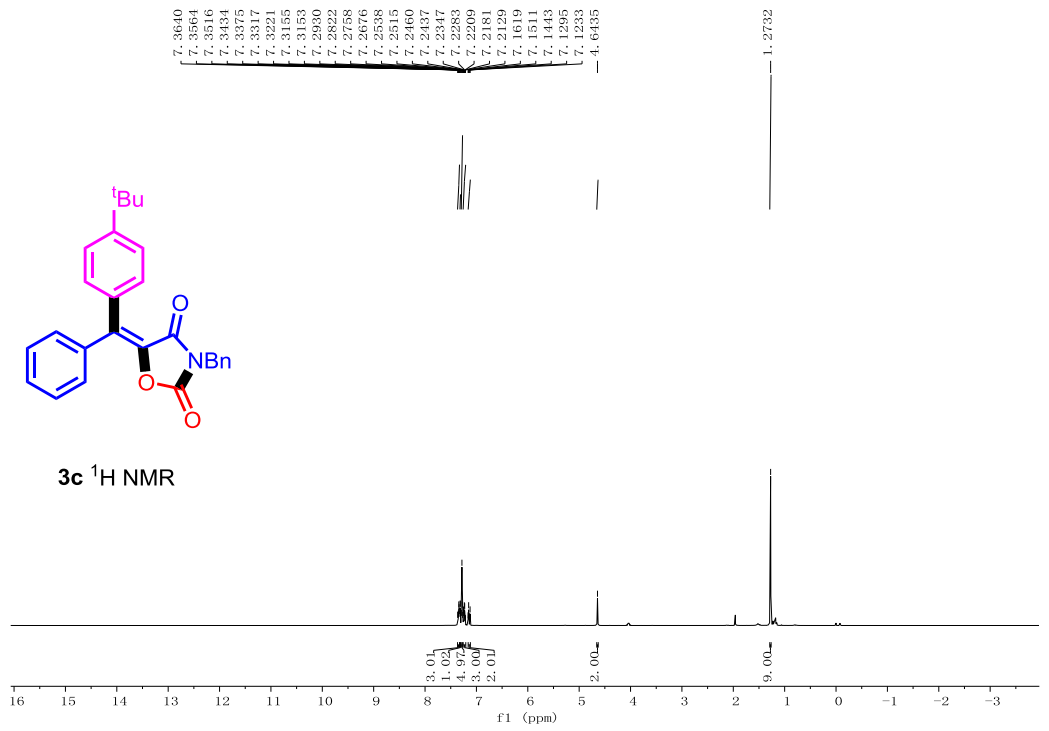
7.3344
7.3307
7.3234
7.3196
7.3126
7.3090
7.3038
7.2985
7.2887
7.2856
7.2183
7.2177
7.2033
7.2032
7.1686
7.1643
7.1561
7.1522
7.1477
4.6945
2.3966
O
NBn
O
O
3b 1H NMR
4.04
6.08
2.08
2.00
1.97
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3 −4
f1 (ppm)
160.7716
152.0730
139.6683
136.2428
134.8270
133.7644
131.9568
130.9349
130.4626
129.9251
129.6892
129.2818
129.1152
129.0683
129.0259
128.9758
128.6174
128.5963
128.4143
43.8179
43.6810
21.6666
O
NBn
O
O
3b 13C NMR
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)



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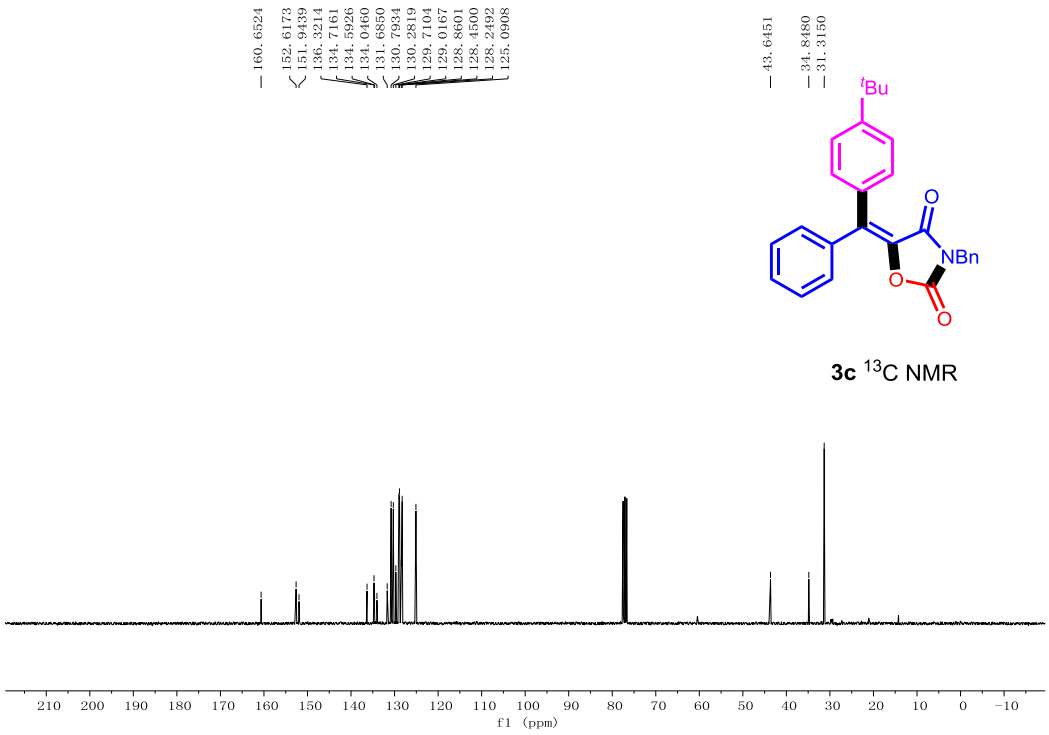
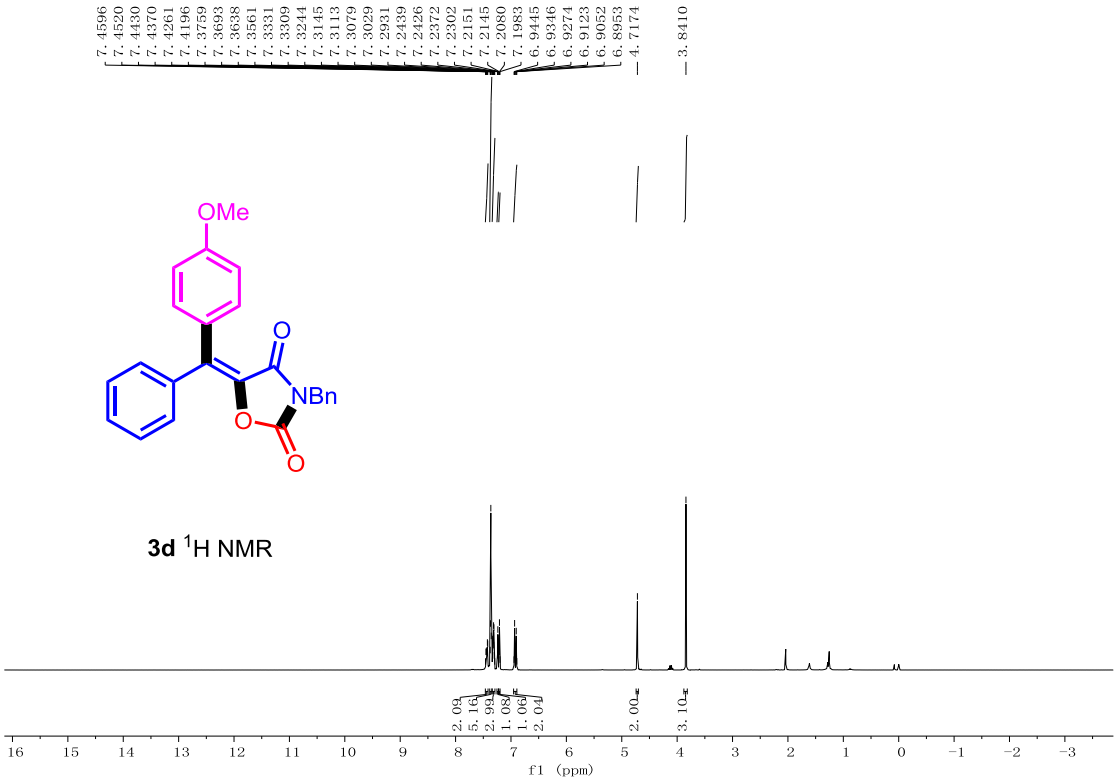


3c ^{13}C NMR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7.3244
7.3145
7.3113
7.3079
7.3029
7.2931
7.2439
7.2426
7.2372
7.2302
7.2151
7.2145
7.2080
7.1983
6.9445
6.9346
6.9274
6.9123
6.9052
6.8953
4.7174
3.8410
OMe
O
NBn
O
O
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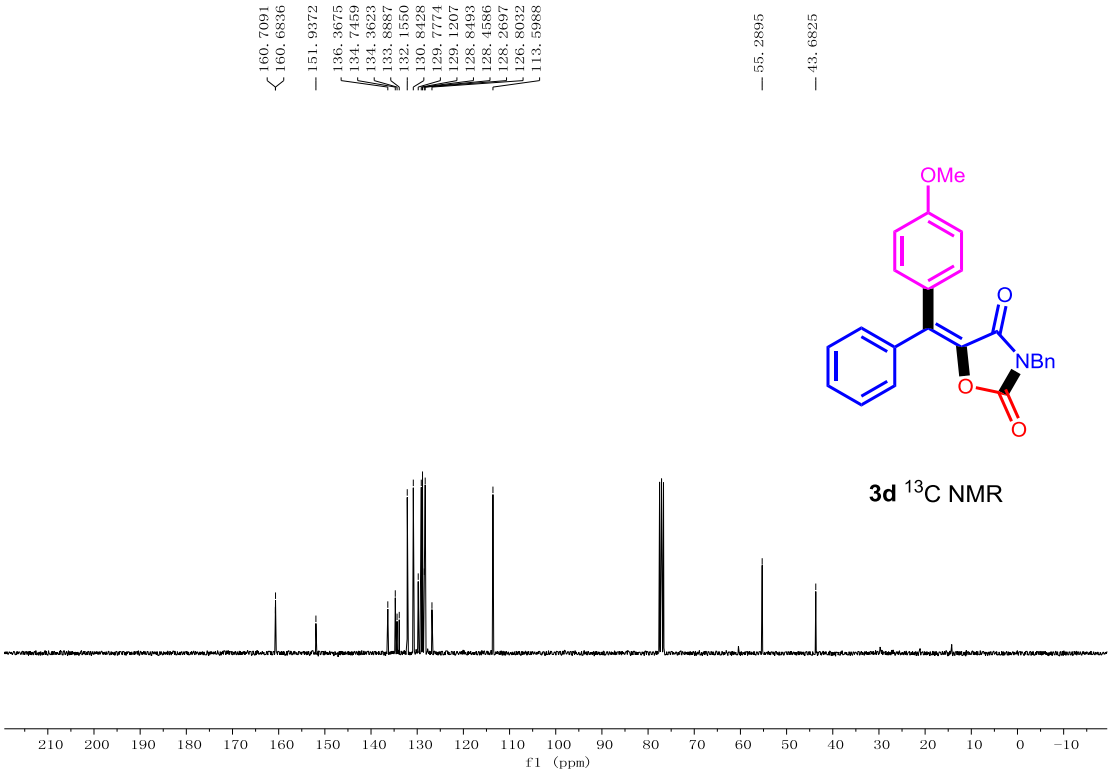
5.16
2.99
1.08
1.06
2.04
2.00
3.10
f1 (ppm)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133.8887
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129.1207
128.8493
128.4586
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113.5988
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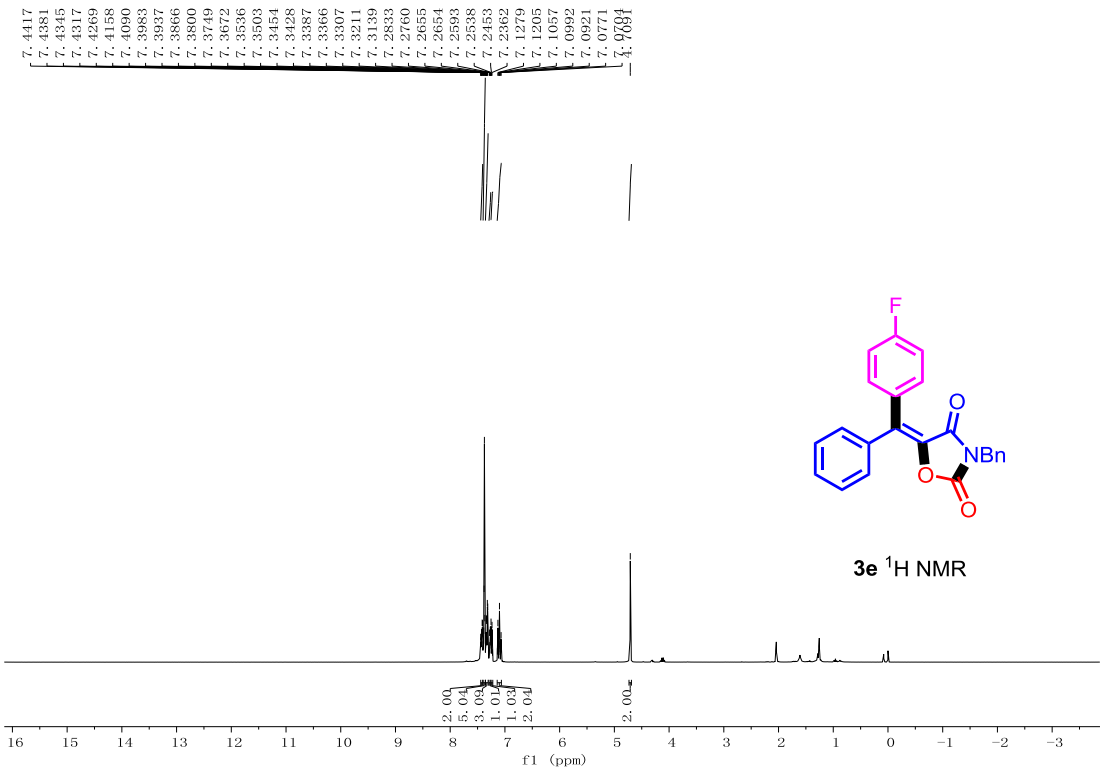
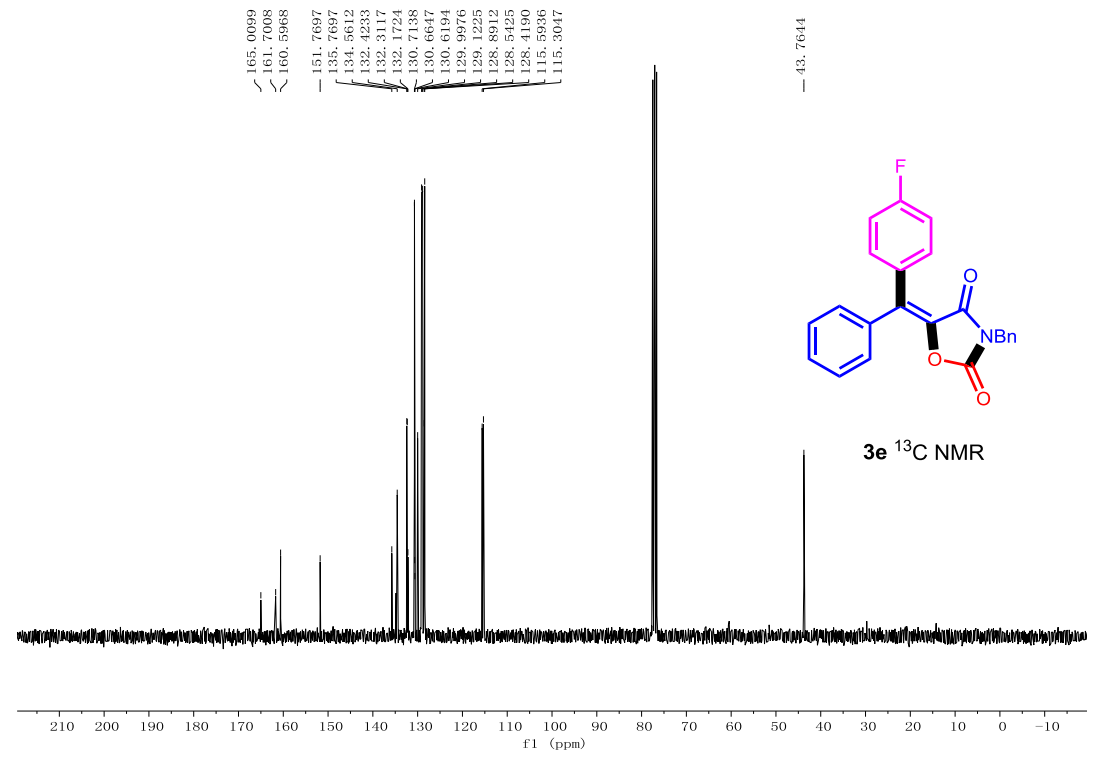
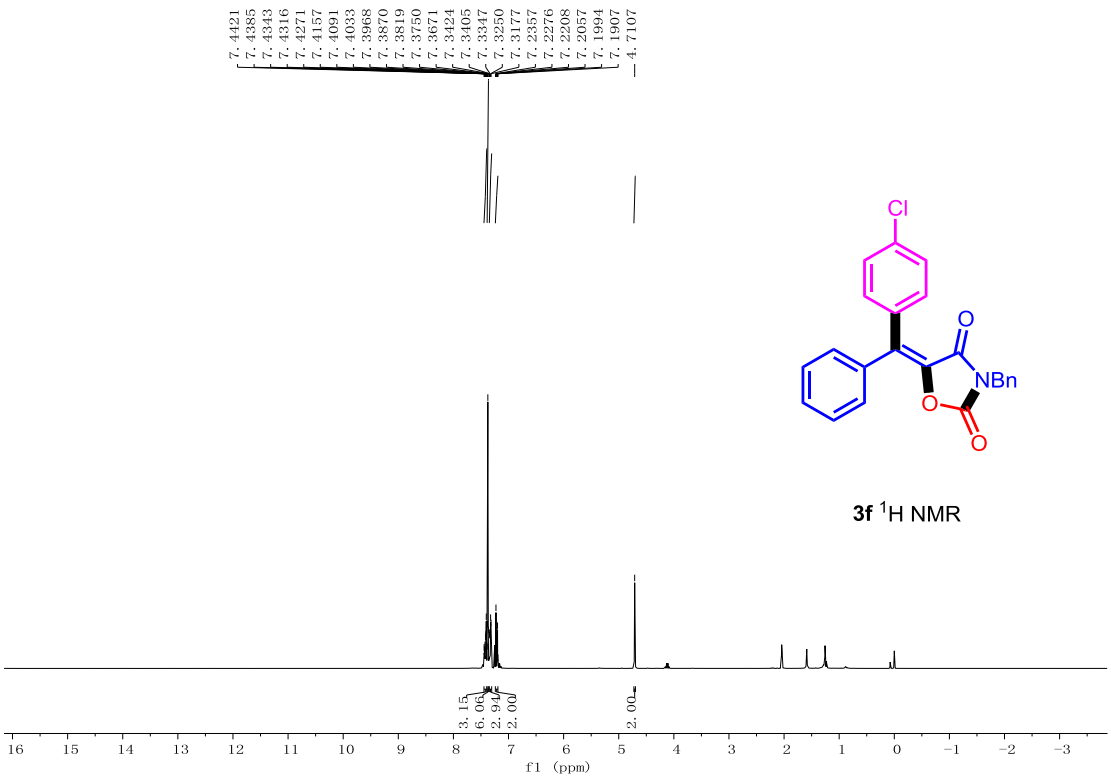
OMe
O
NBn
O
O
3d 13C NMR
f1 (ppm)



3e ^{1}H NMR



3e ^{13}C NMR



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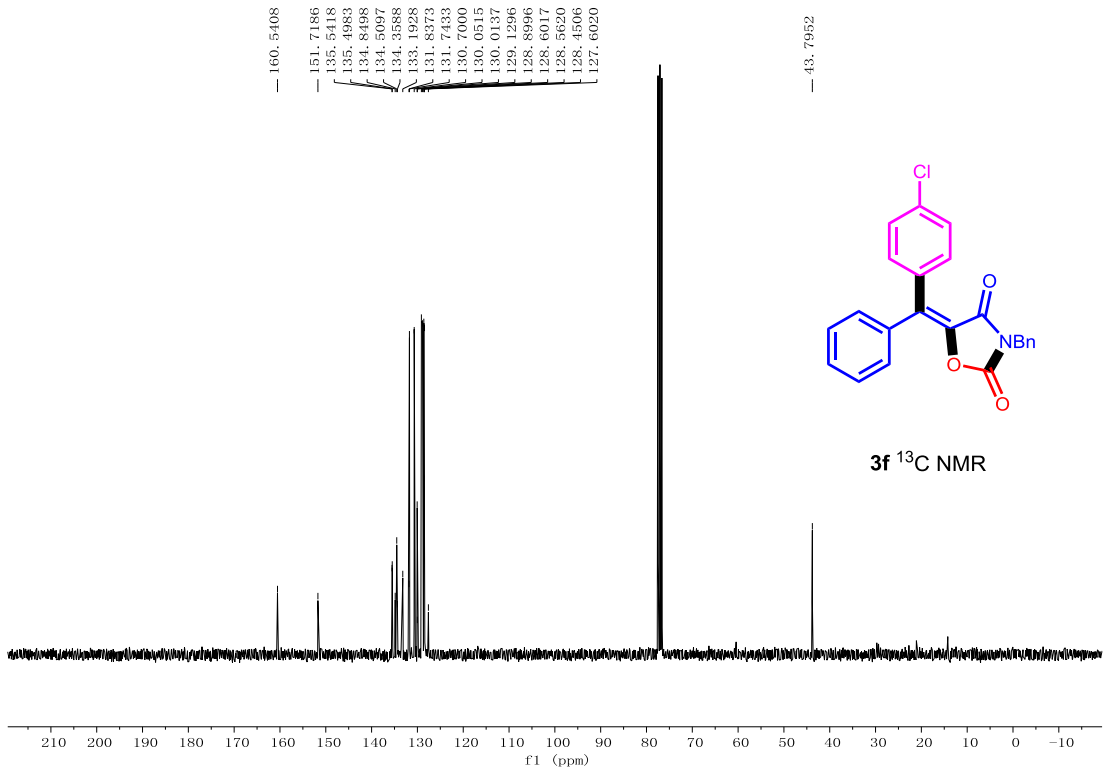
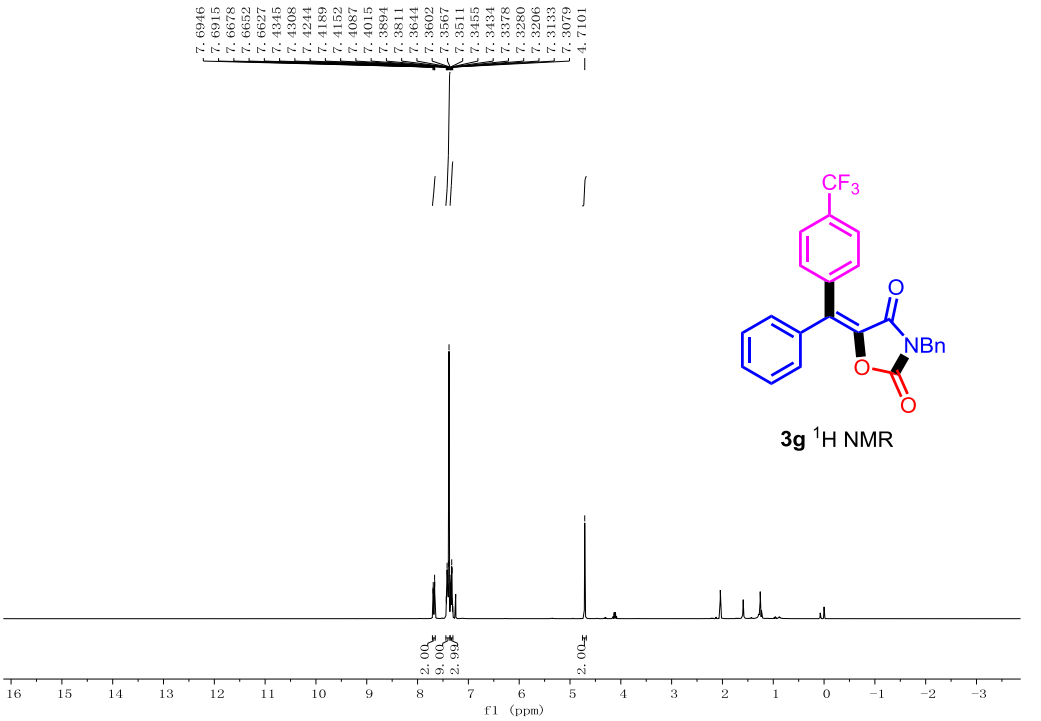


3f ^{13}C NMR



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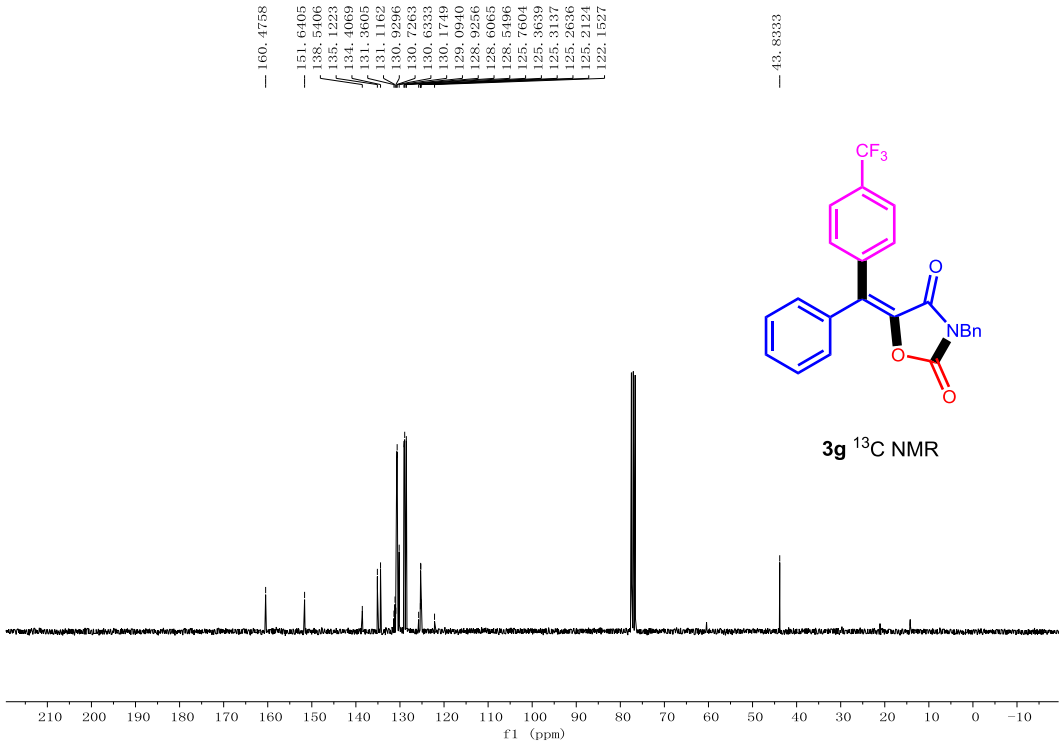

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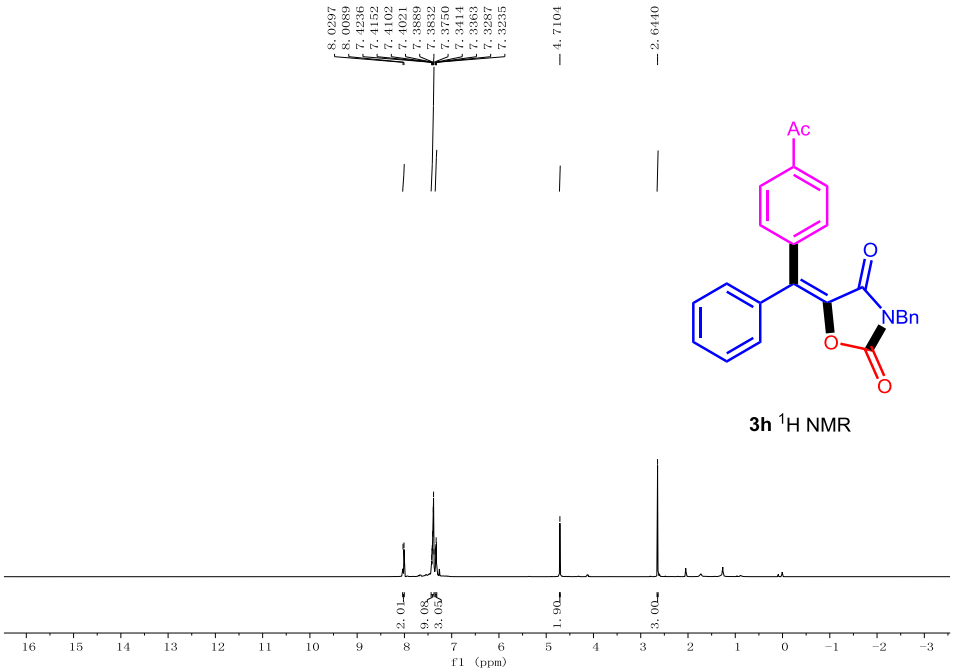

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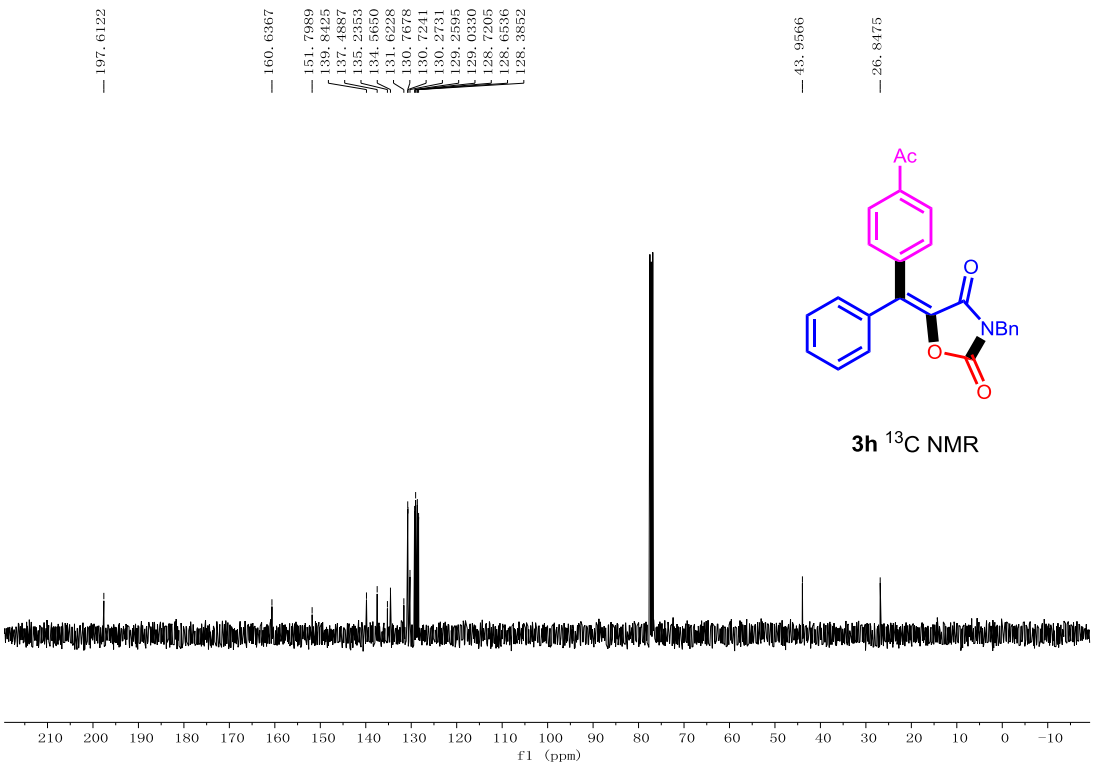

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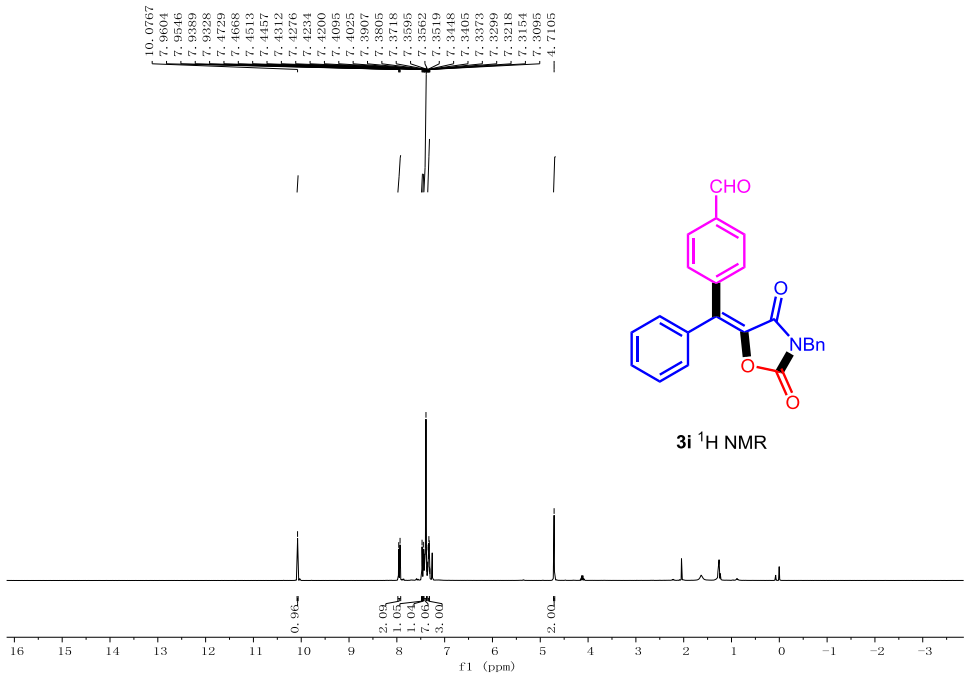
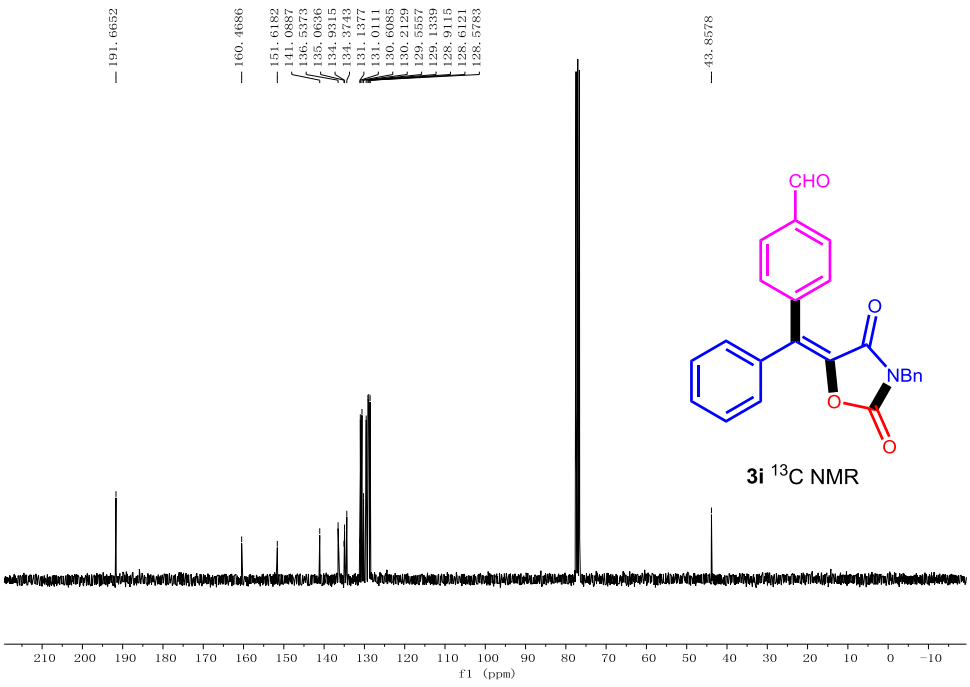


3i ^{1}H NMR



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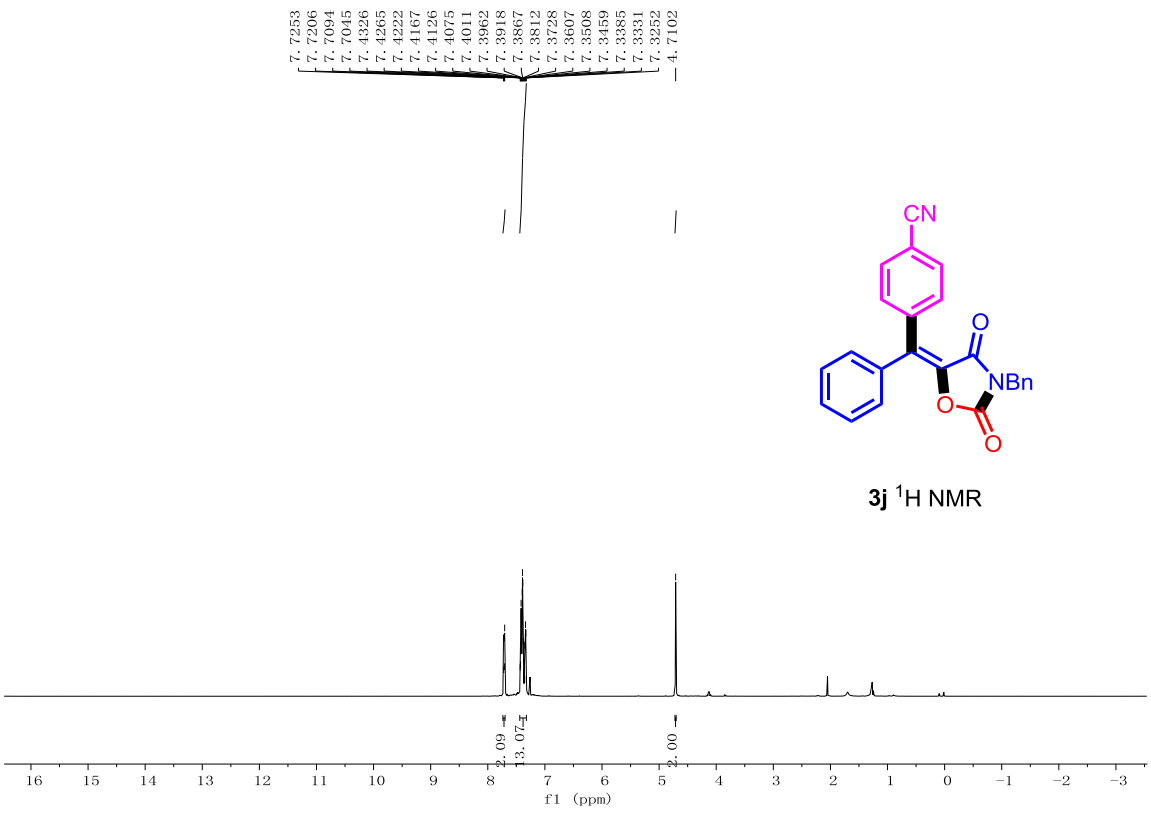

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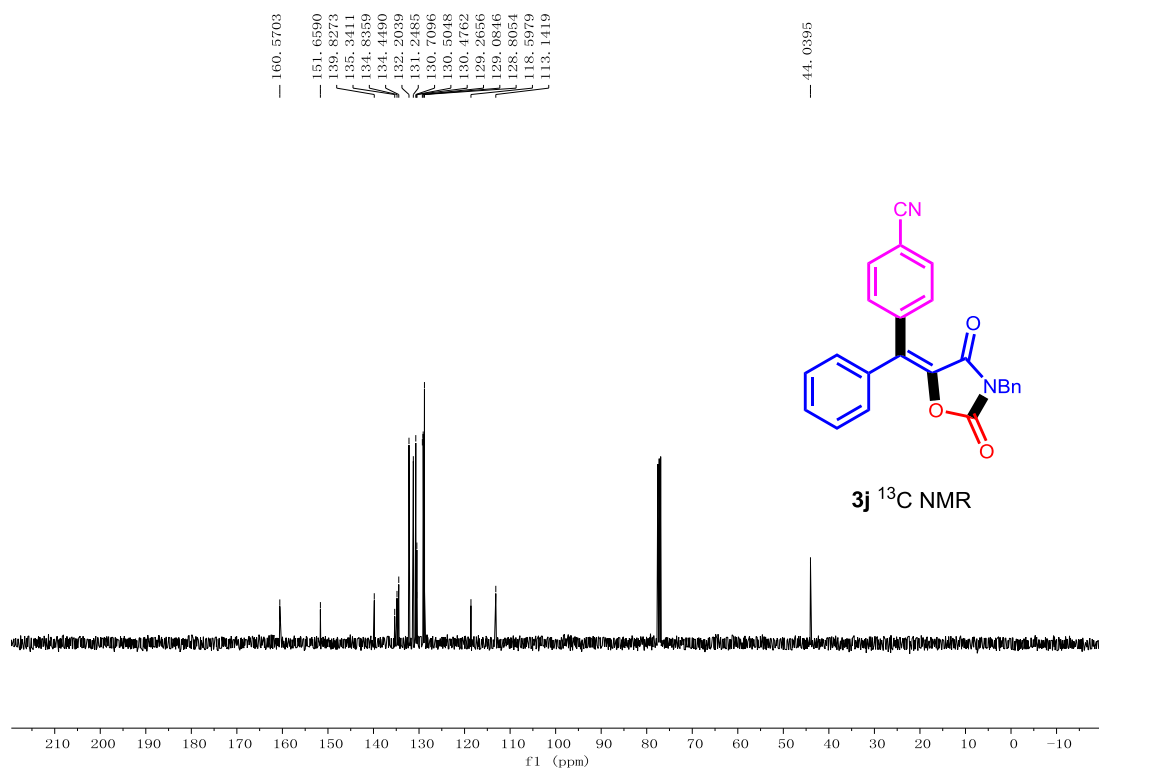

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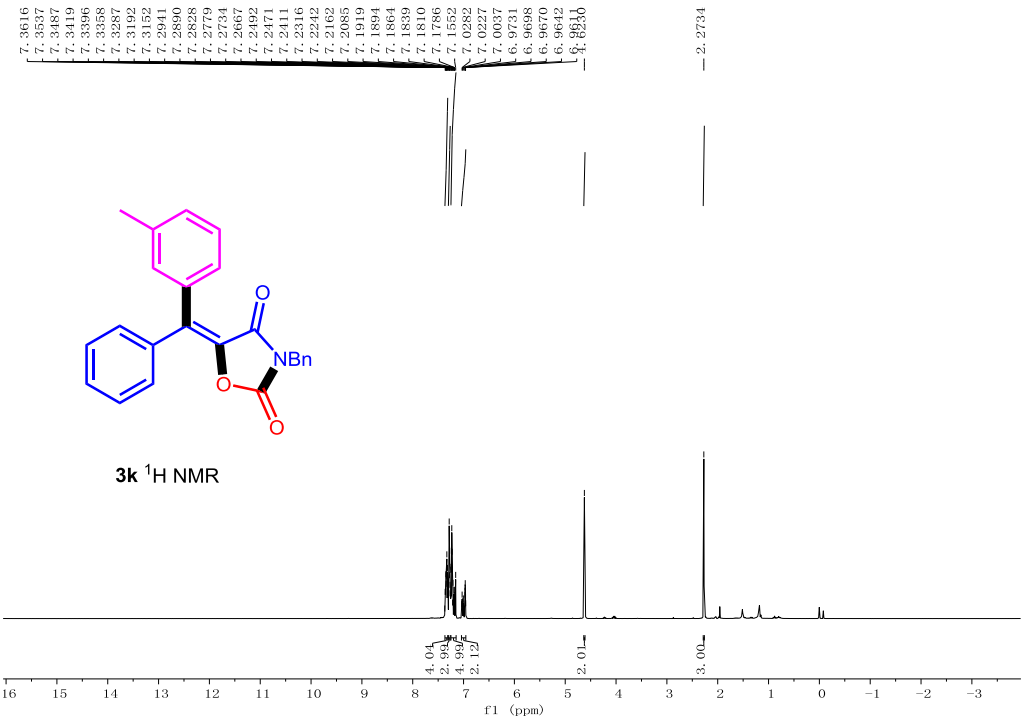

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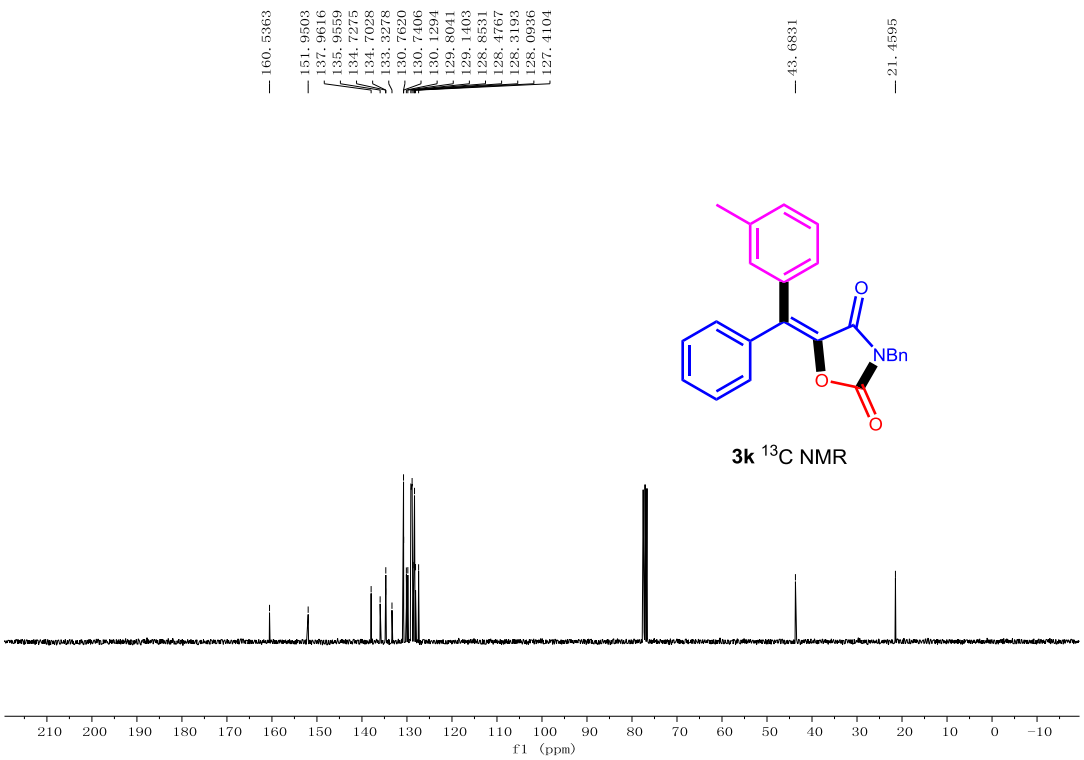

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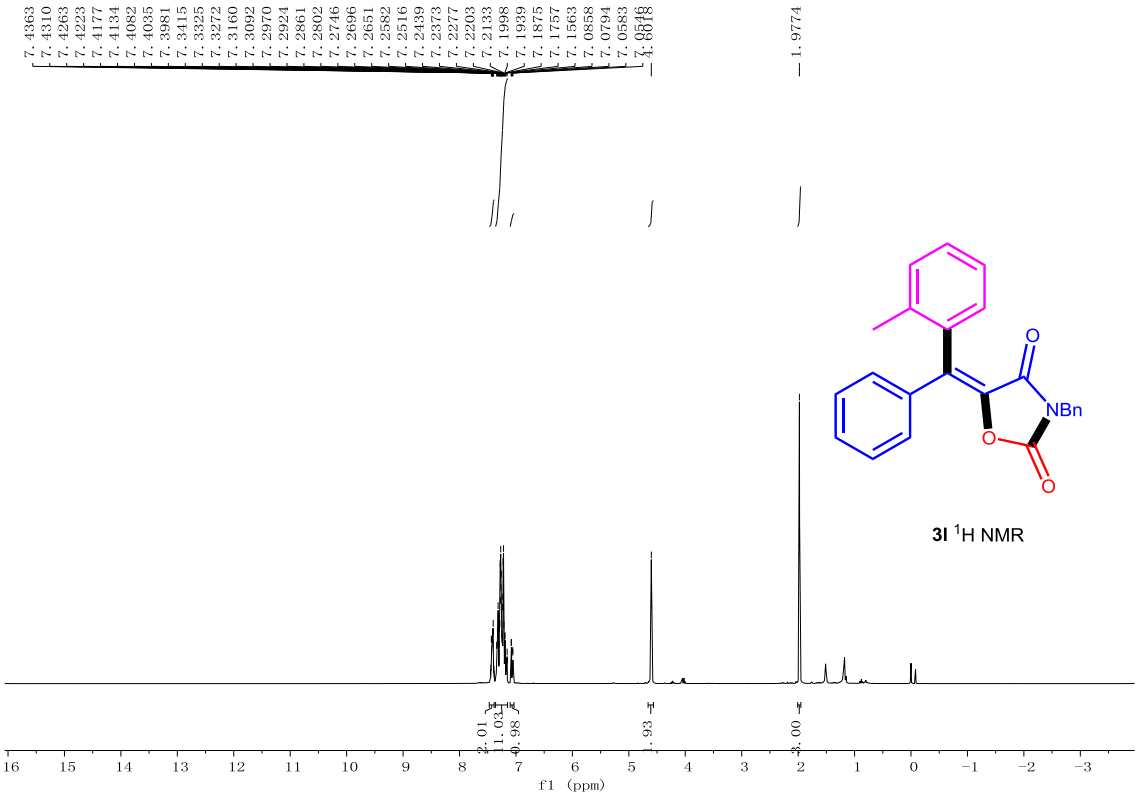

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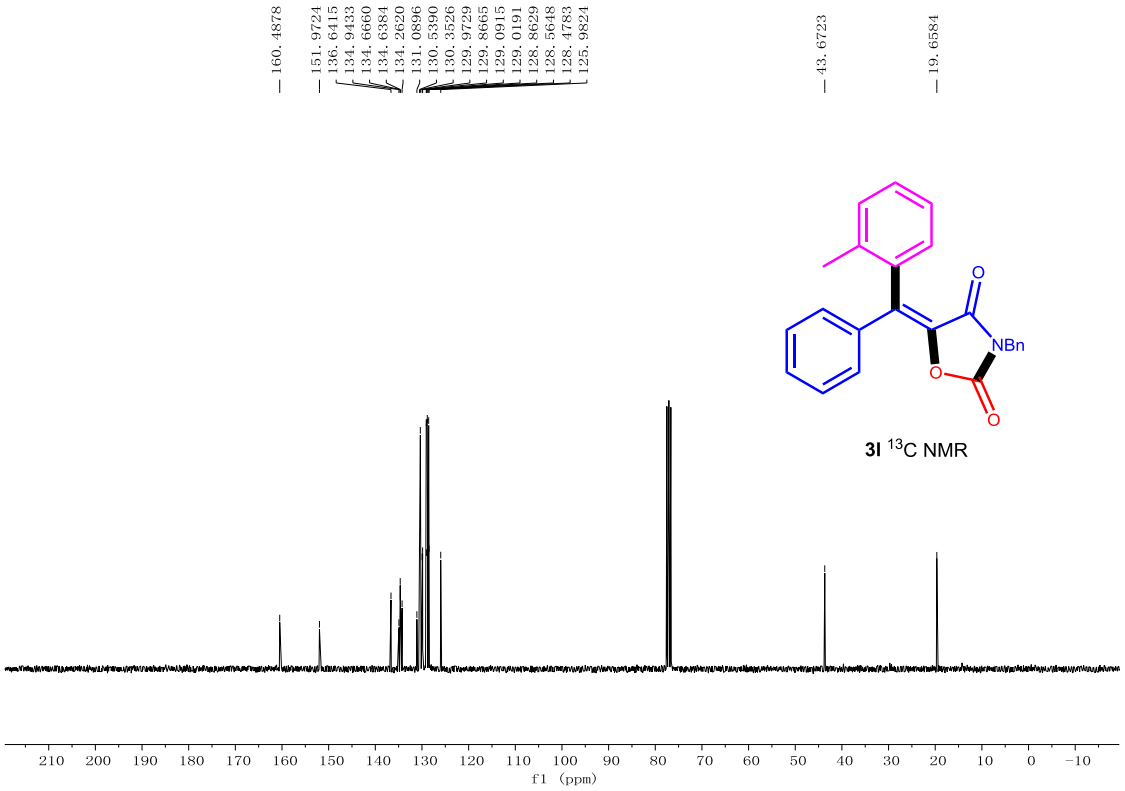

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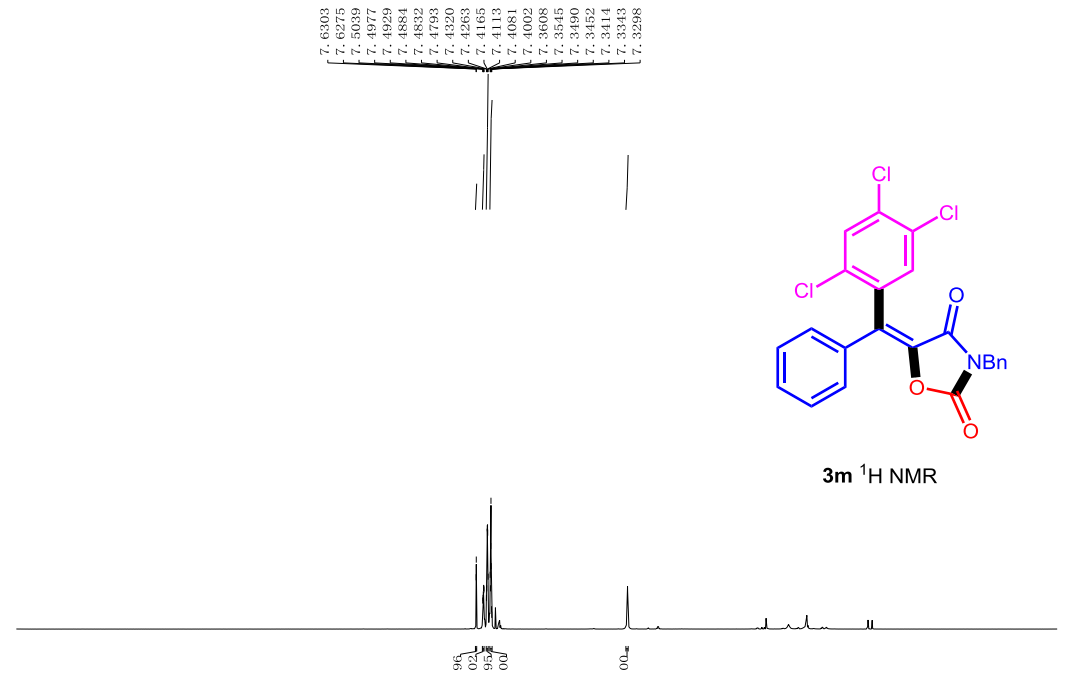
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7.4002
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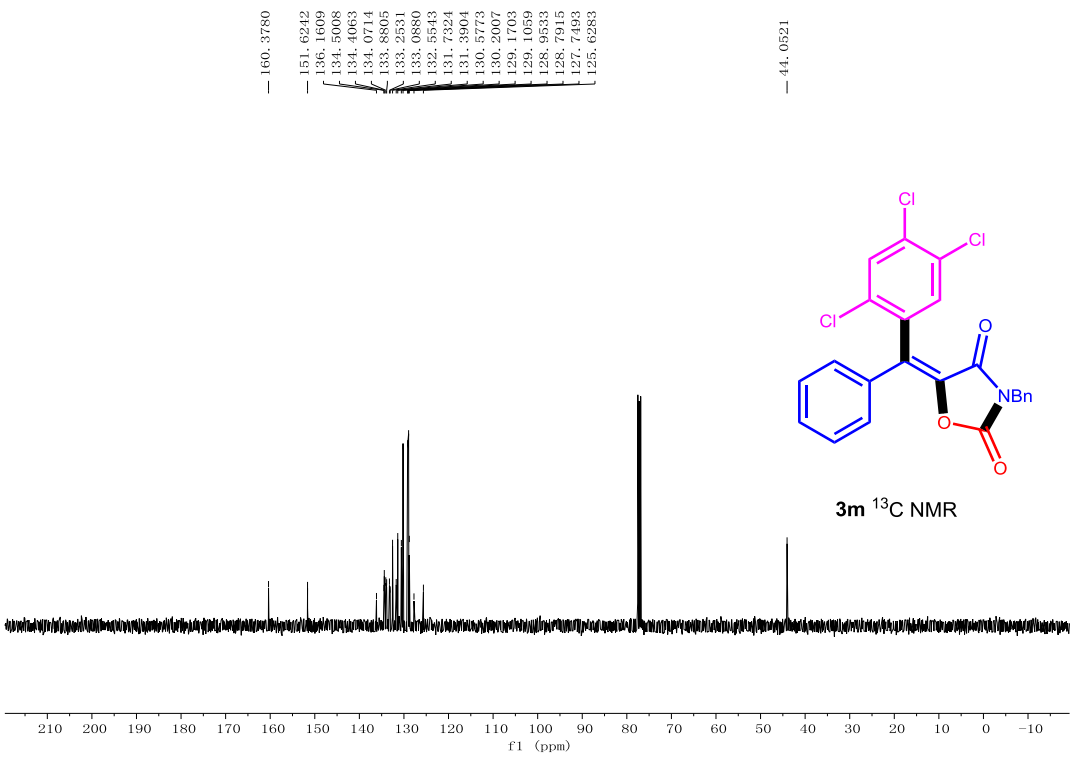
02
95
00
00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125.6283
44.0521
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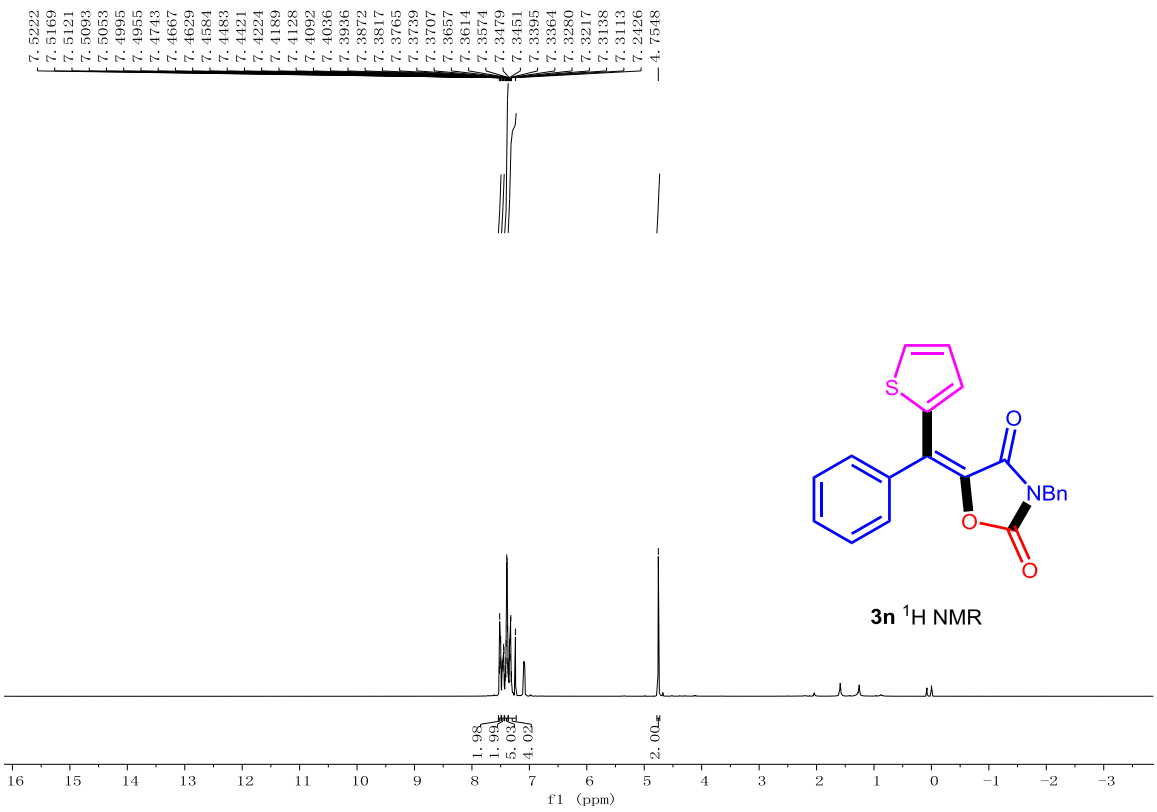
f1 (ppm)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O
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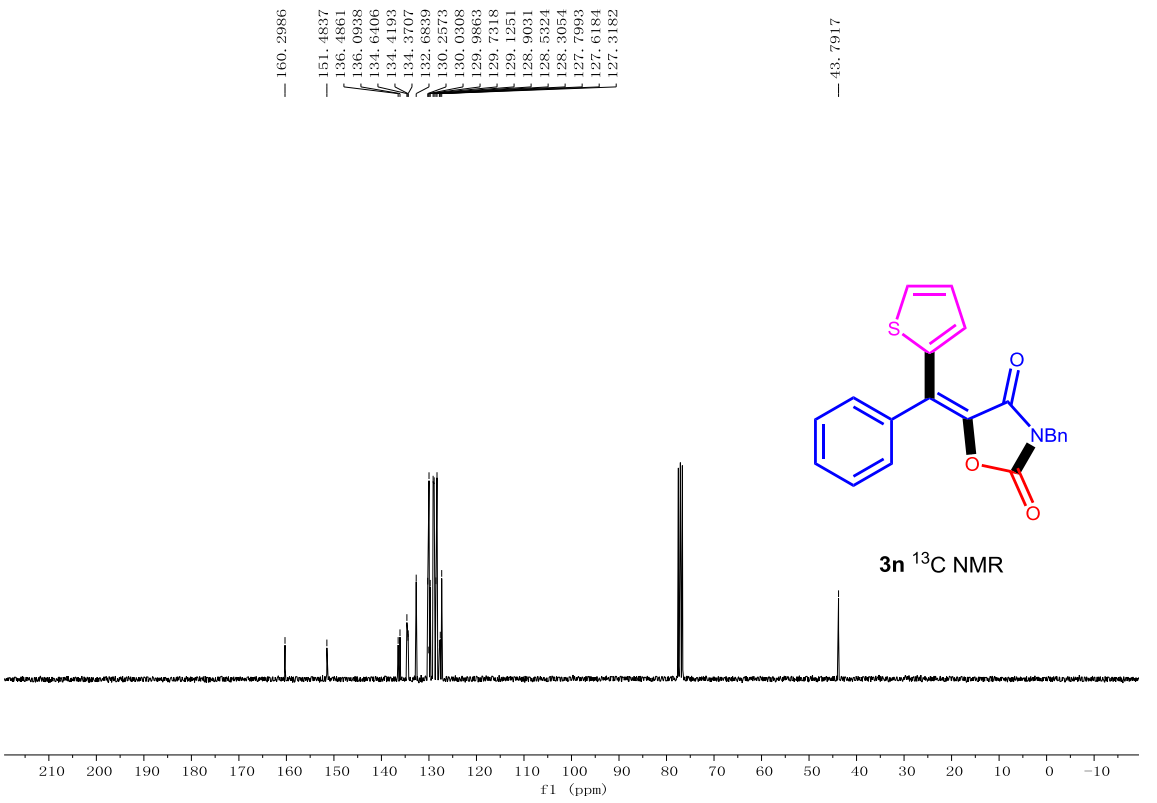
f1 (ppm)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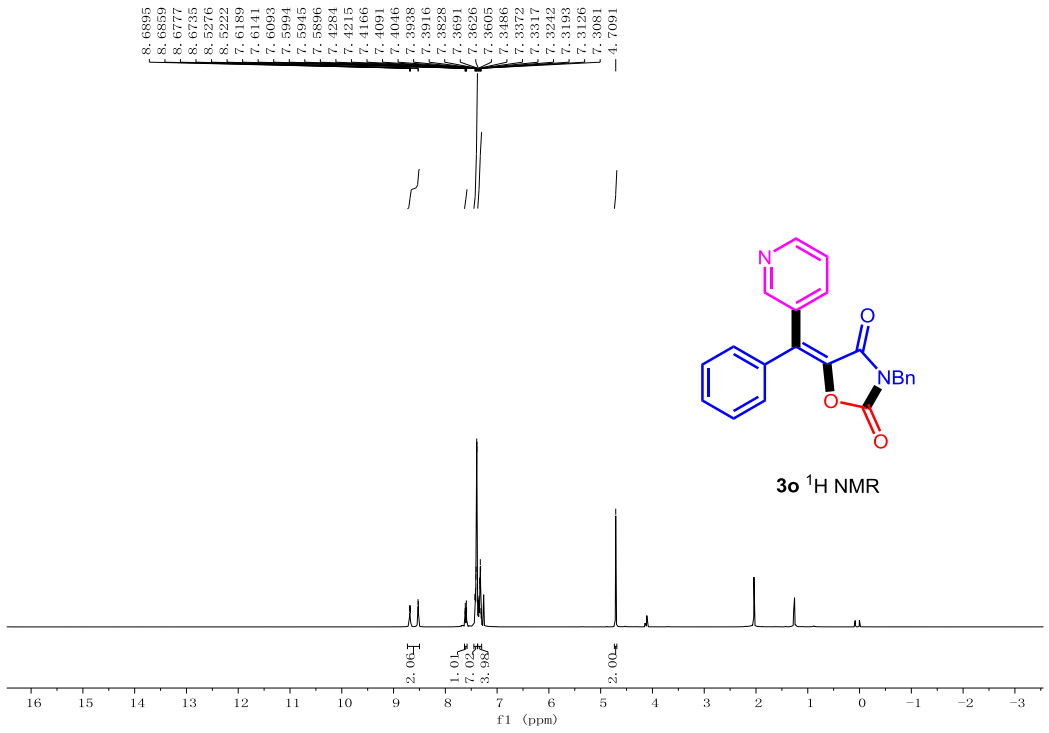
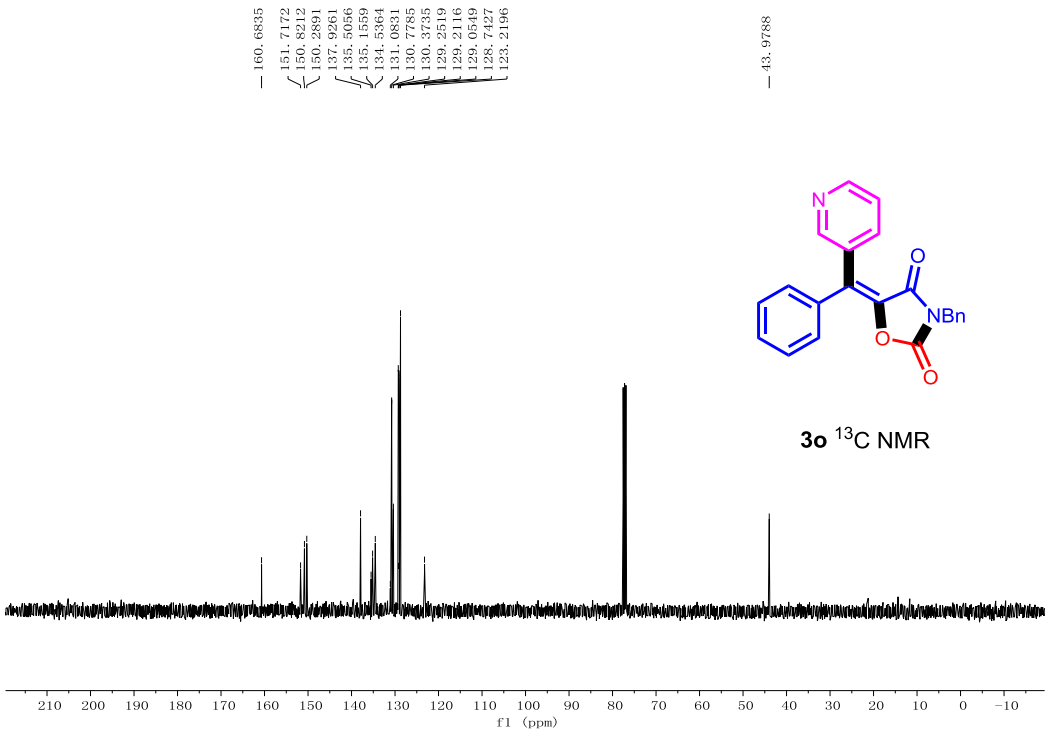
129.9863
129.7318
129.1251
128.9031
128.5324
128.3054
127.7993
127.6184
127.3182
43.7917
S
O
NBn
O
O
3n ^{13}C NMR
f1 (ppm)

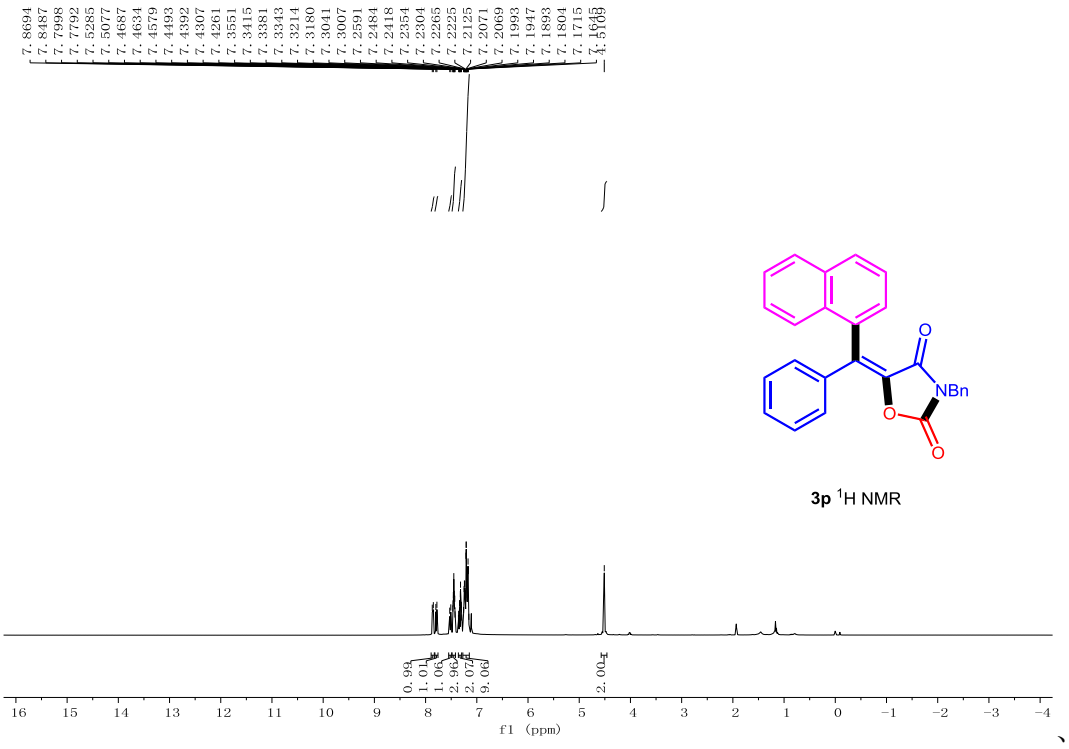


3o ^{1}H NMR

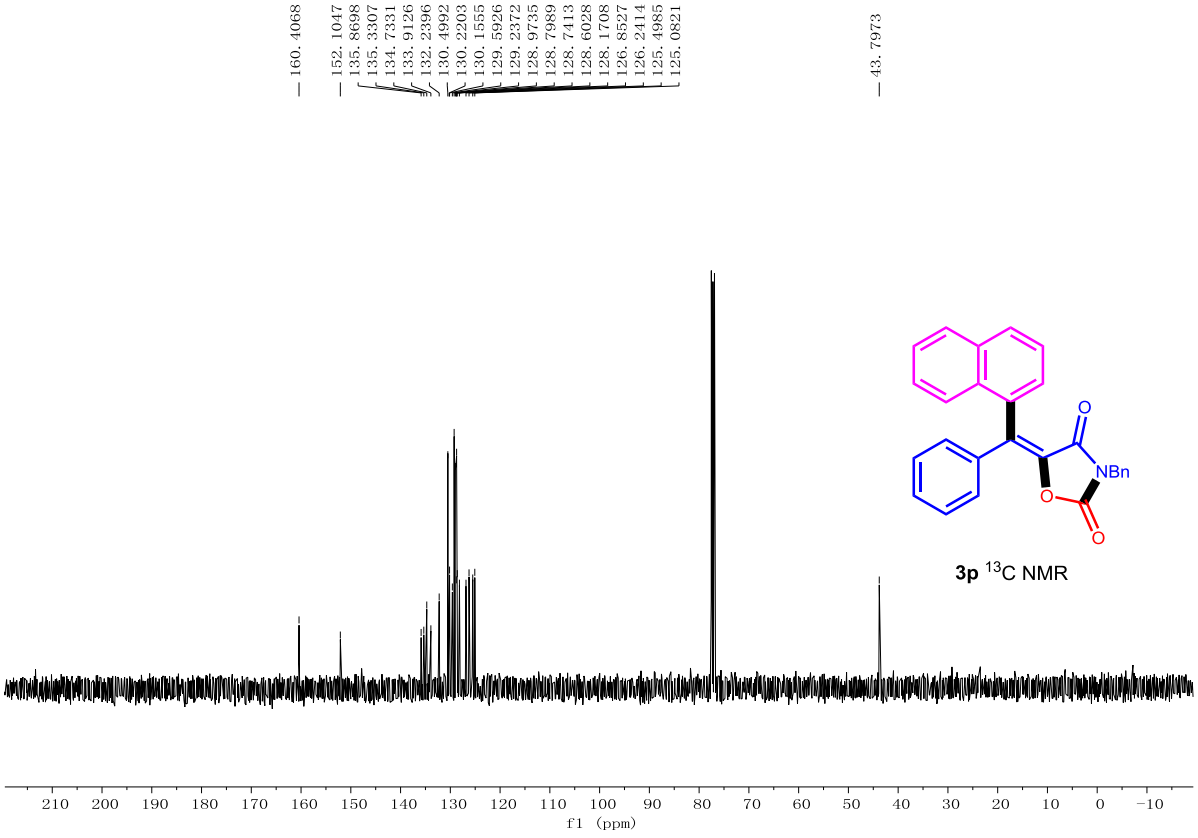


3o ^{13}C NMR

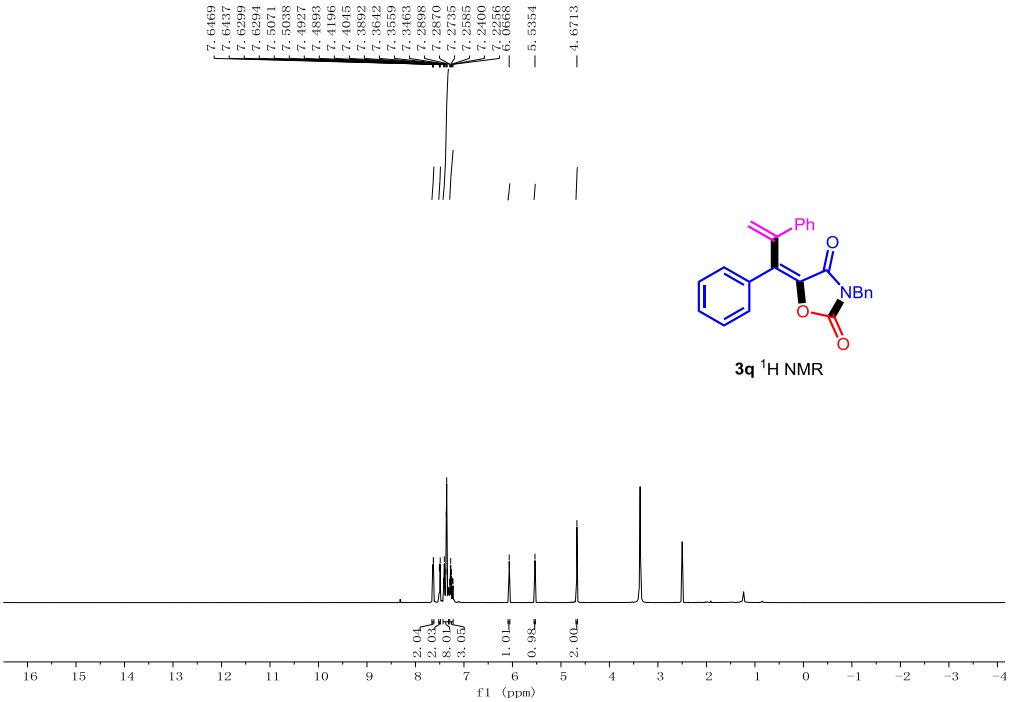
-+521



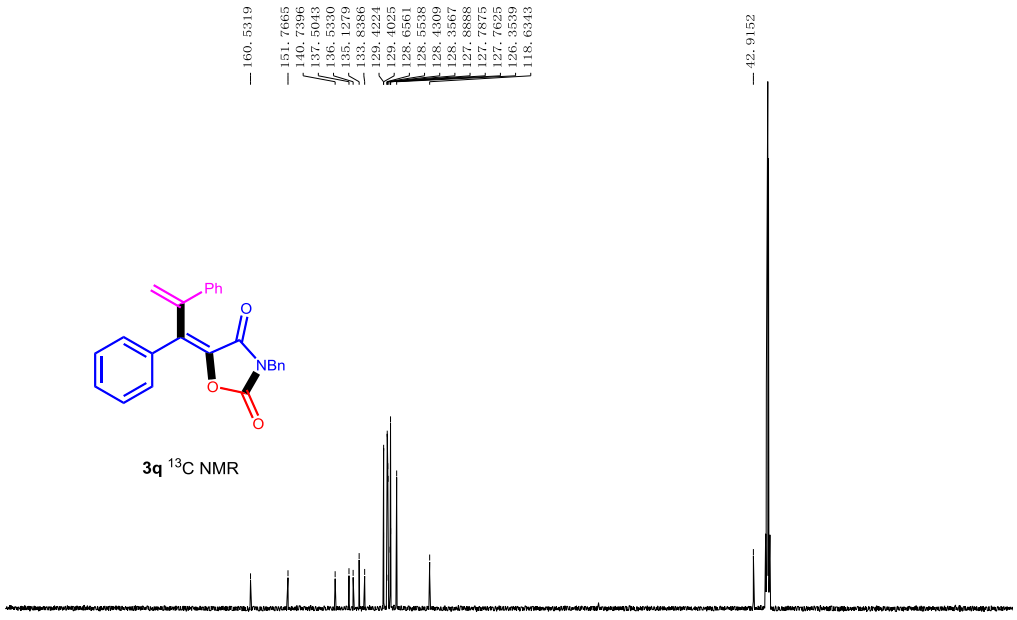
3p ^{1}H NMR



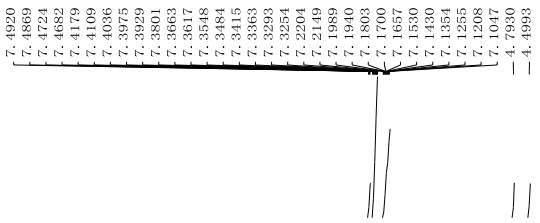
3p ^{13}C NMR

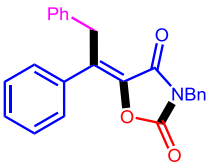


3q ^{1}H NMR

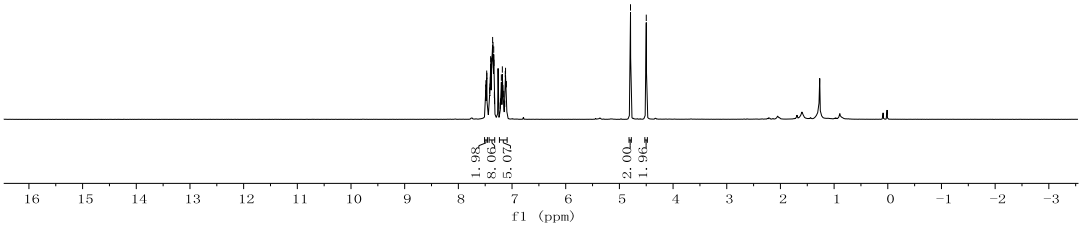


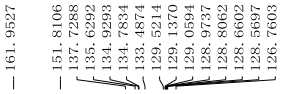
3q ^{13}C NMR

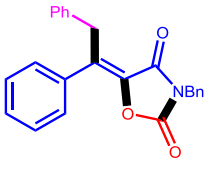




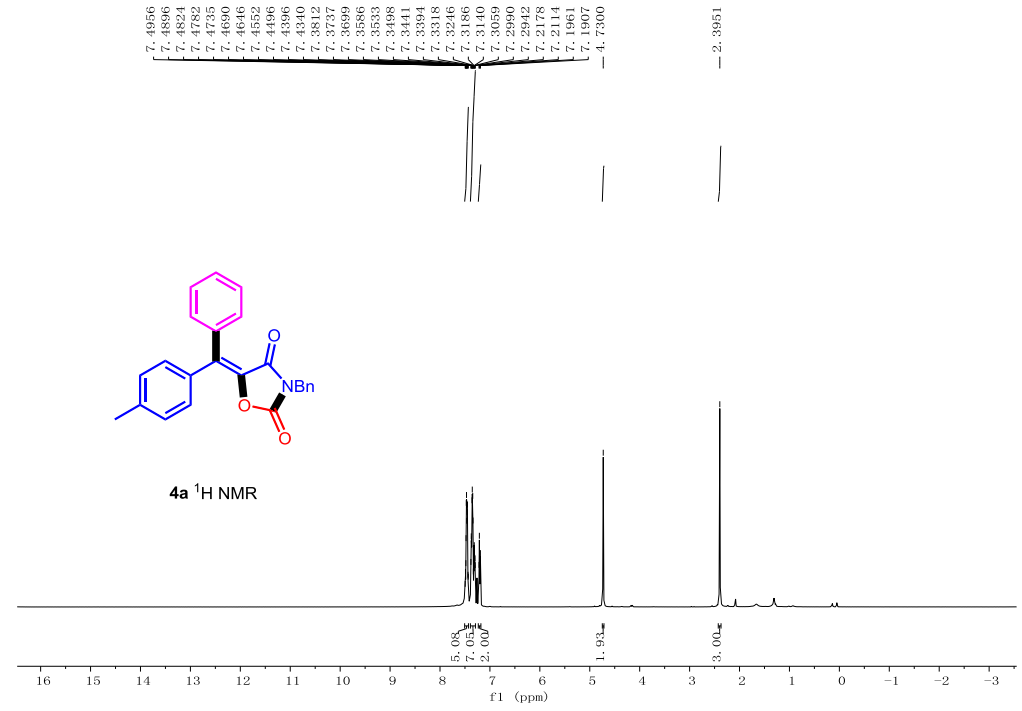
3r ^{1}H NMR



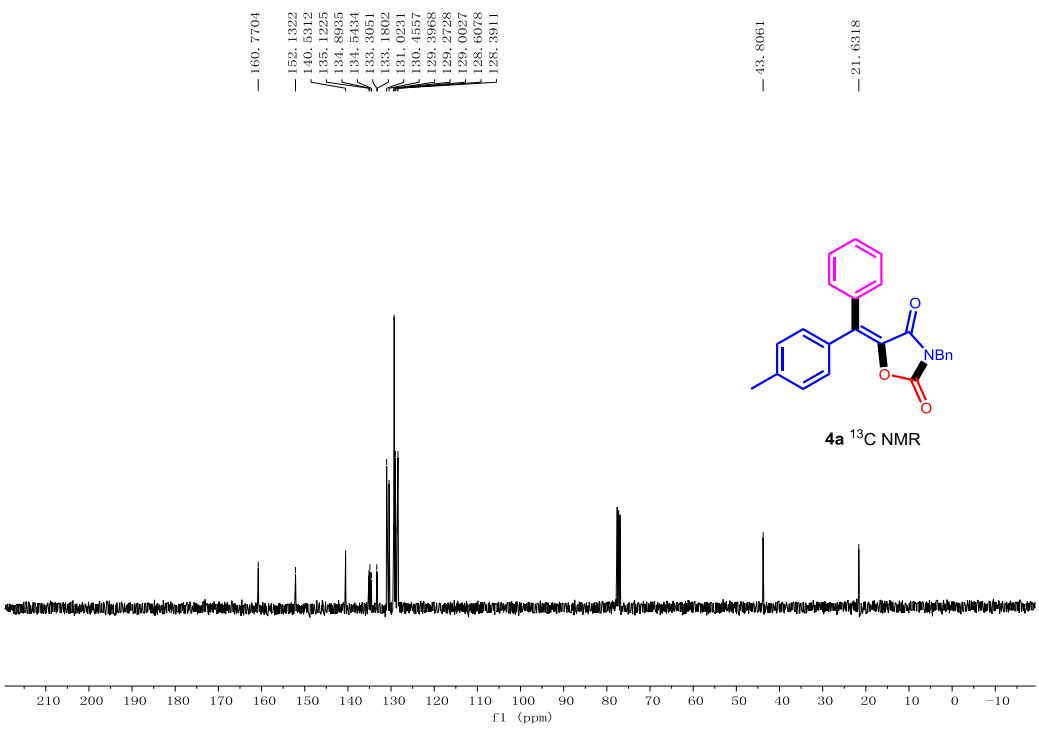




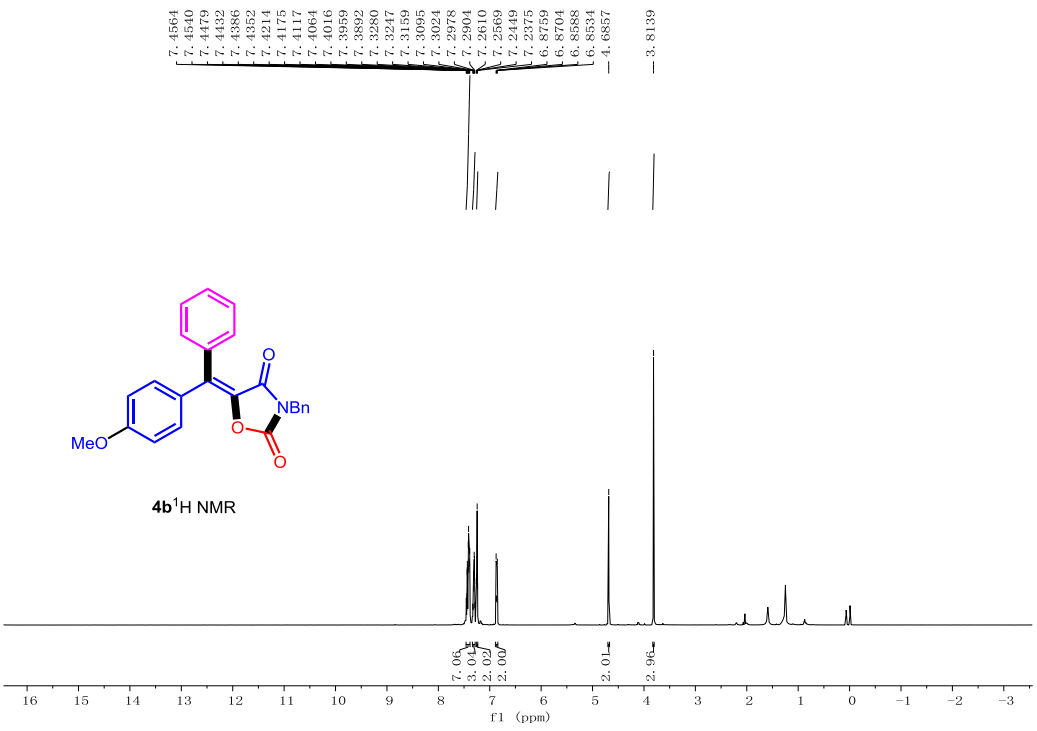
3r ^{13}C NMR



4a ^{1}H NMR



4a ^{13}C NMR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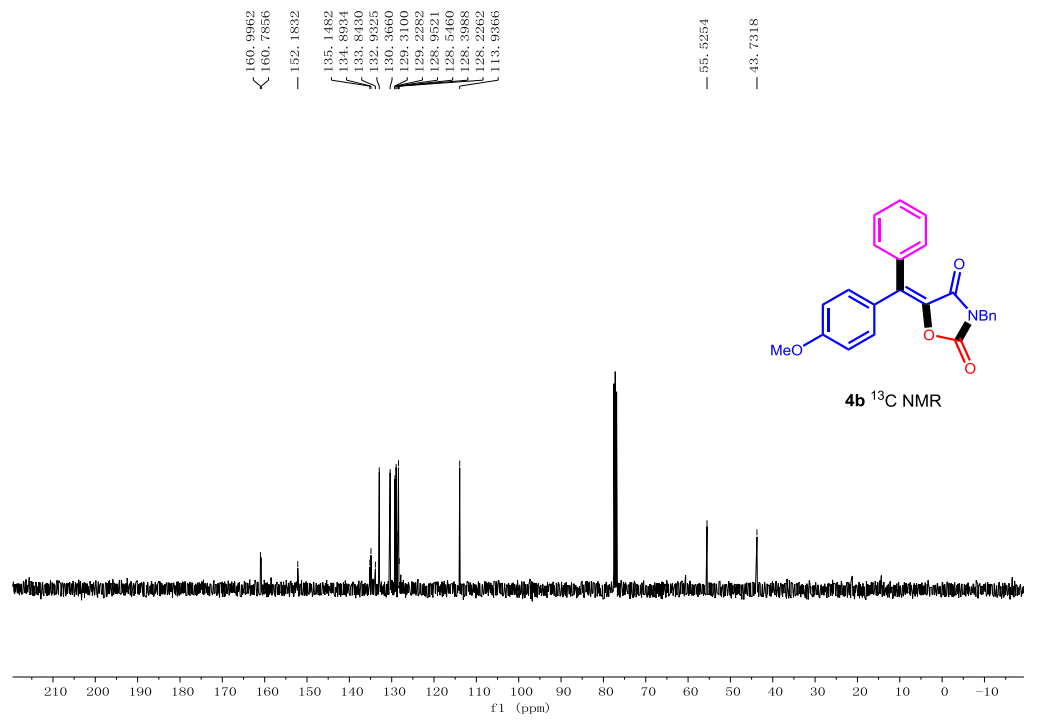
7.4479
7.4432
7.4386
7.4352
7.4214
7.4175
7.4117
7.4064
7.4016
7.3959
7.3892
7.3280
7.3247
7.3159
7.3095
7.3024
7.2978
7.2904
7.2610
7.2569
7.2449
7.2375
6.8759
6.8704
6.8588
6.8534
4.6857
3.8139
O
NBn
O
O
MeO
4b 1H NMR
7.06
3.04
2.02
2.00
2.01
2.96
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)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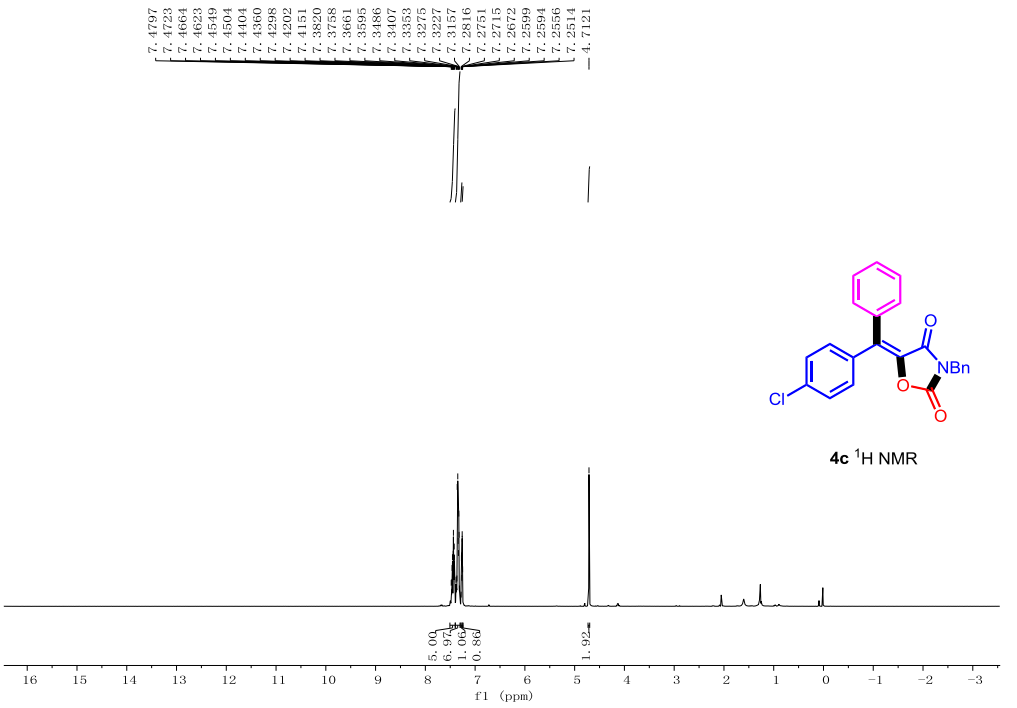
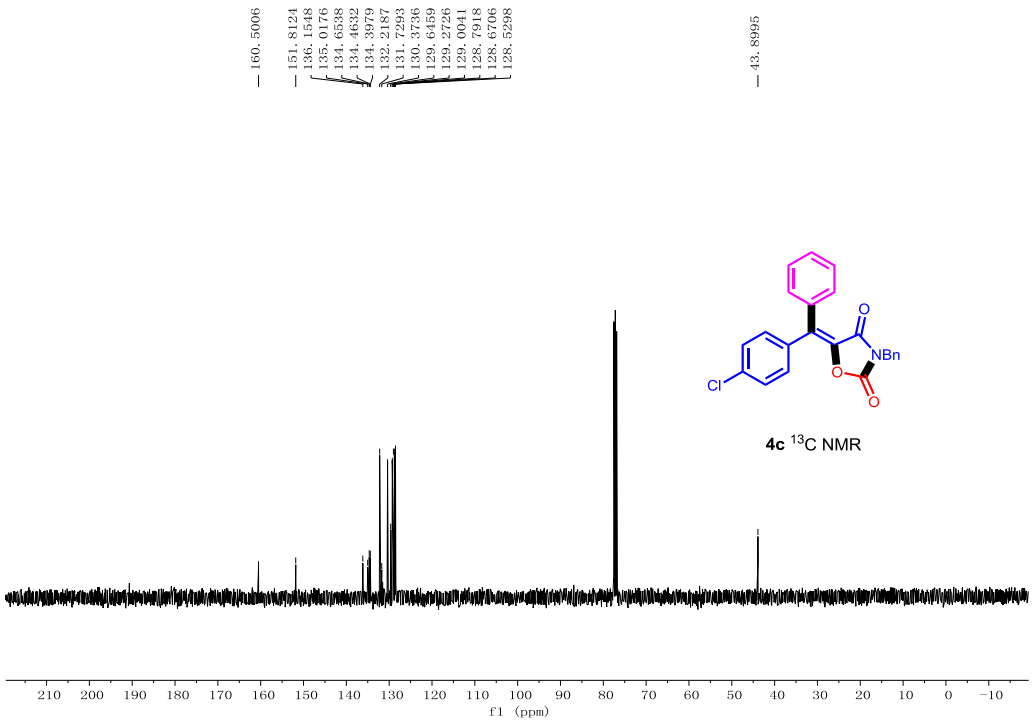
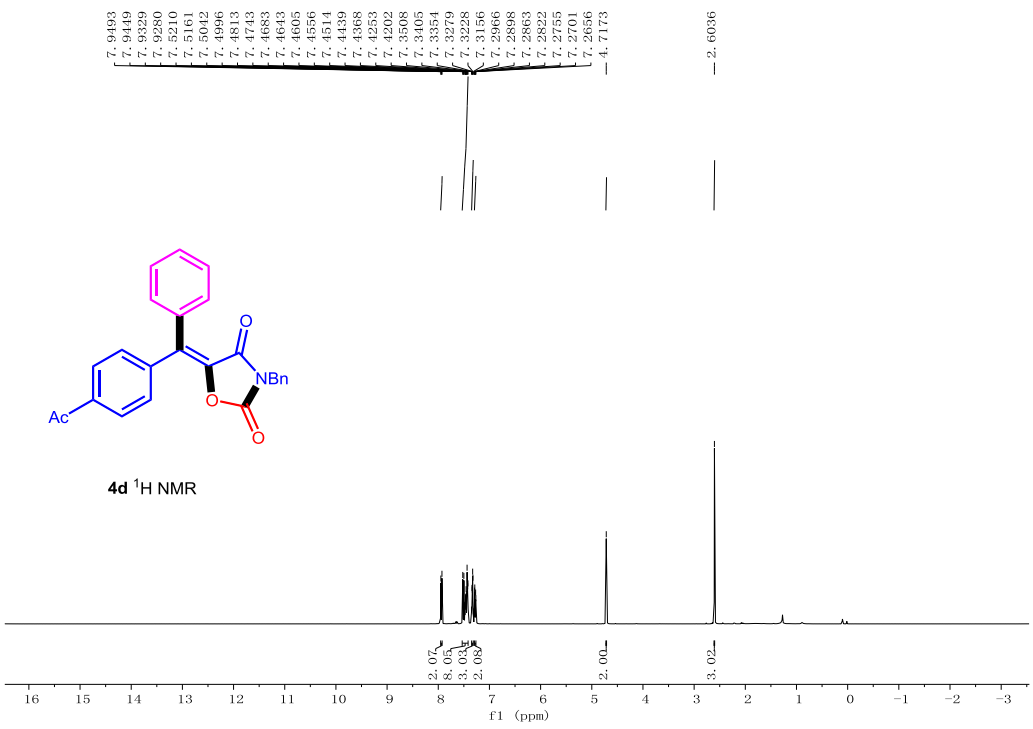
160.7856
152.1832
135.1482
134.8934
133.8430
132.9325
130.3660
129.3100
129.2282
128.9521
128.5460
128.3988
128.2262
113.9366
55.5254
43.7318
O
NBn
O
O
MeO
4b 13C NMR
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)



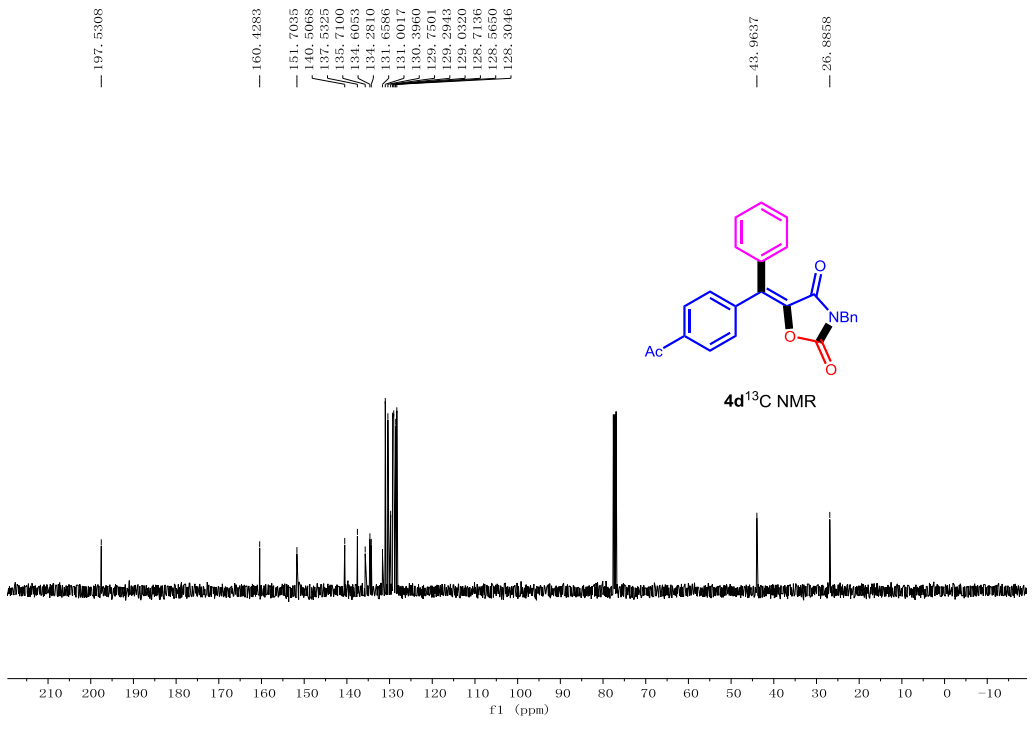
4c ^{1}H NMR



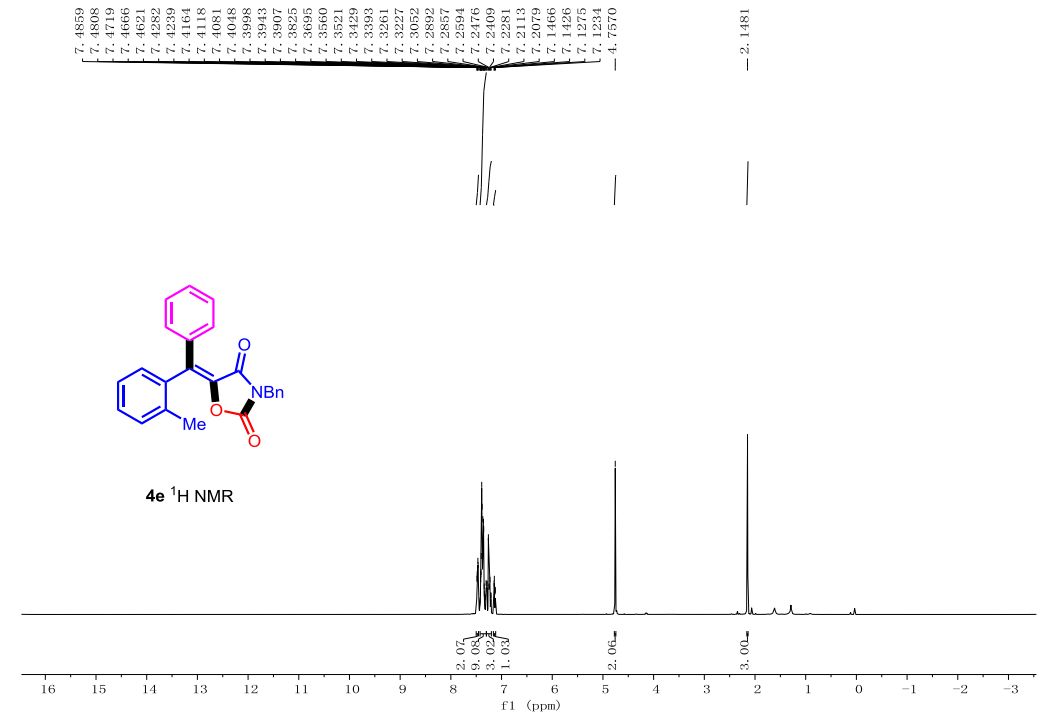
4c ^{13}C NMR



4d ^{1}H NMR



4d^{13}C NMR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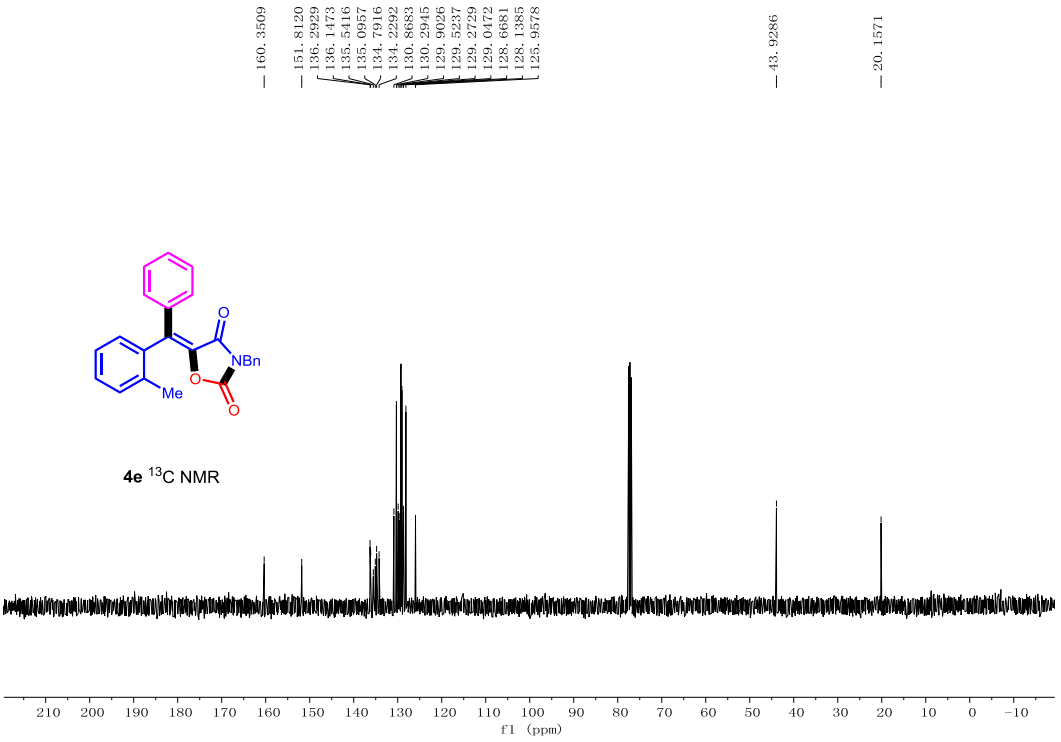
7.4666
7.4621
7.4282
7.4239
7.4164
7.4118
7.4081
7.4048
7.3998
7.3943
7.3907
7.3825
7.3695
7.3560
7.3521
7.3429
7.3393
7.3261
7.3227
7.3052
7.2892
7.2857
7.2594
7.2476
7.2409
7.2281
7.2113
7.2079
7.1466
7.1426
7.1275
7.1234
4.7570
2.1481
O
NBn
O
Me
O
4e 1H NMR
2.07
9.08
3.02
1.03
2.06
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)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136.1473
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130.8683
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129.5237
129.2729
129.0472
128.6681
128.1385
125.9578
43.9286
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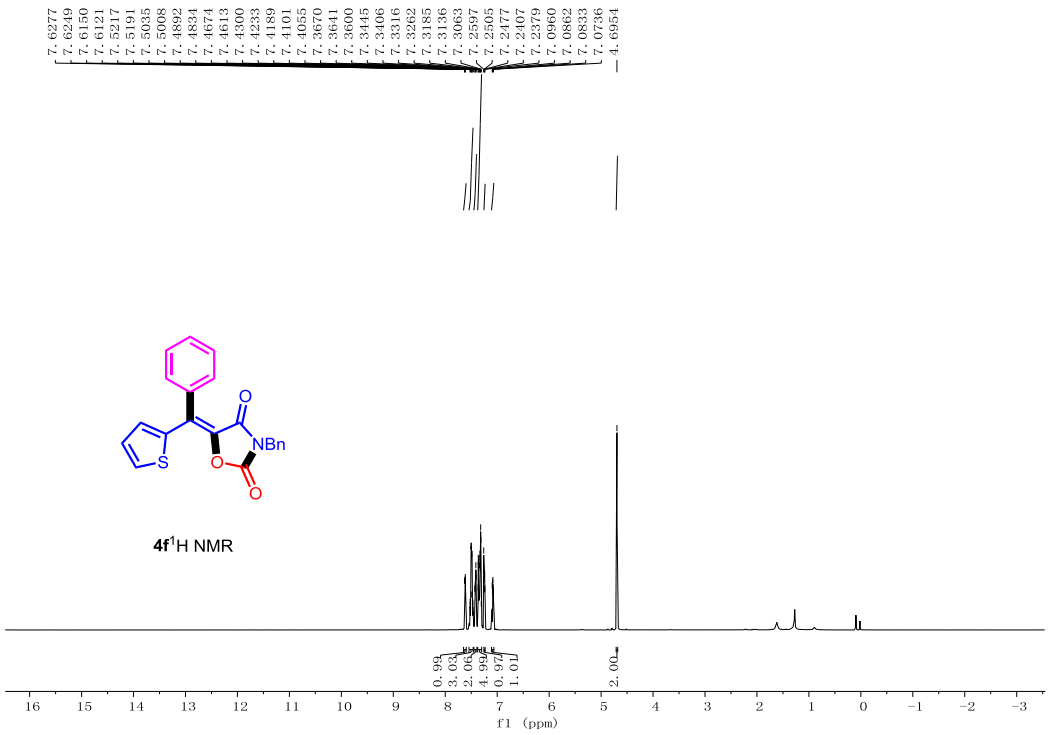
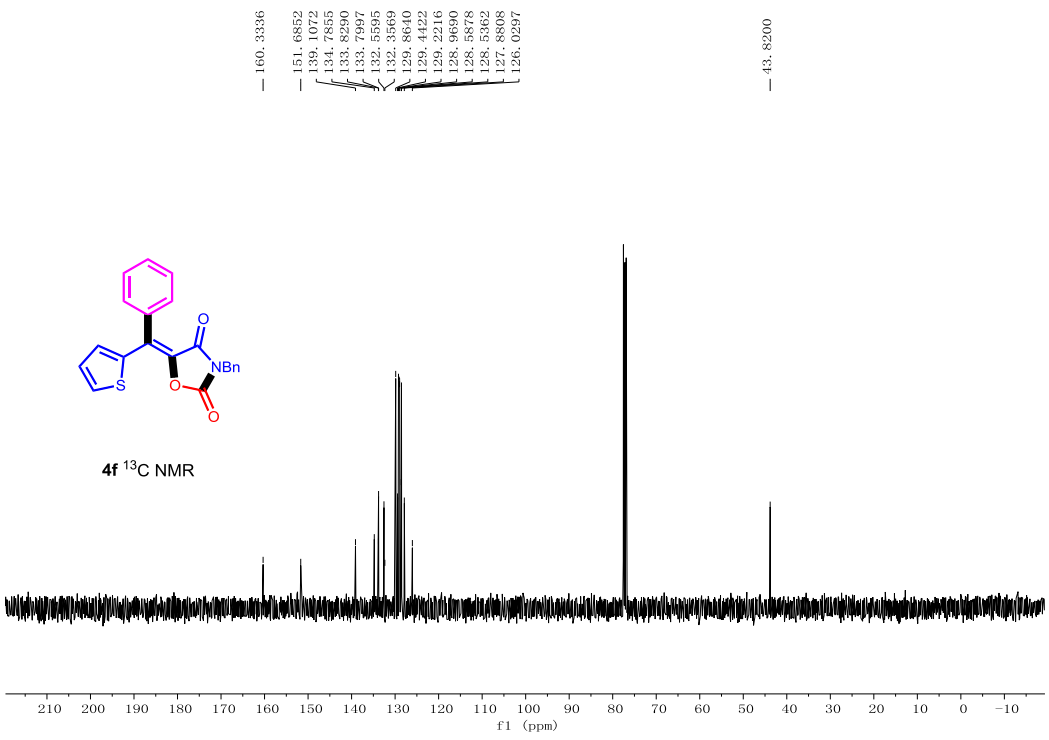
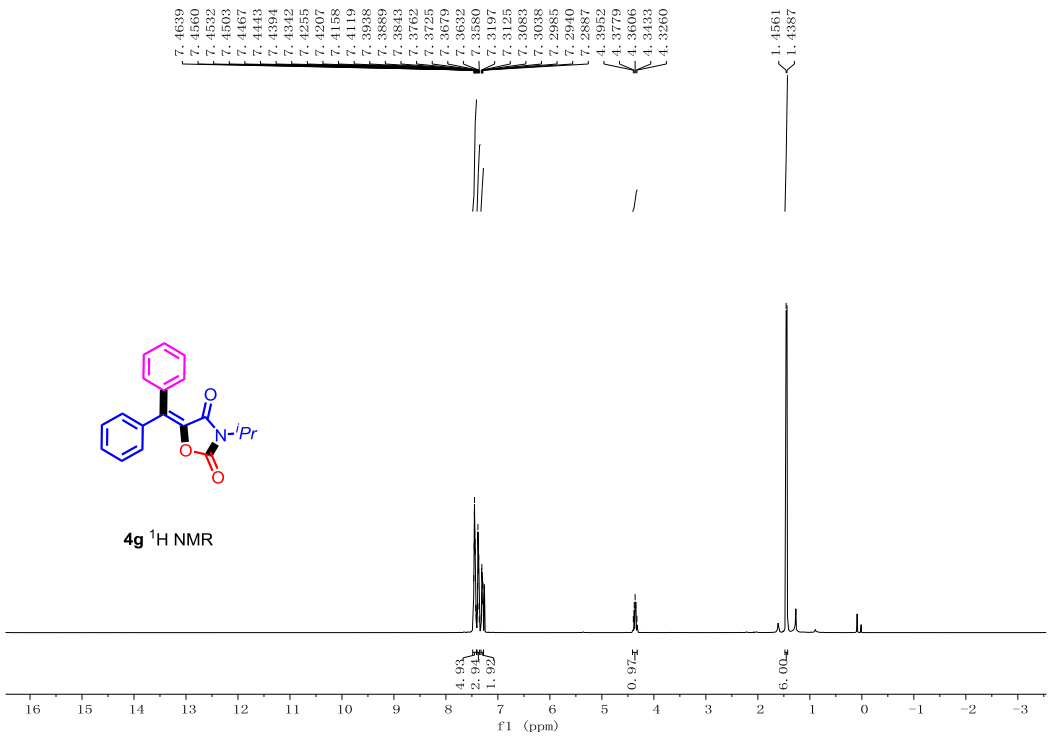
O
NBn
O
Me
O
4e 13C NMR
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)



4f ^{1}H NMR



4f ^{13}C NMR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7.4467
7.4443
7.4394
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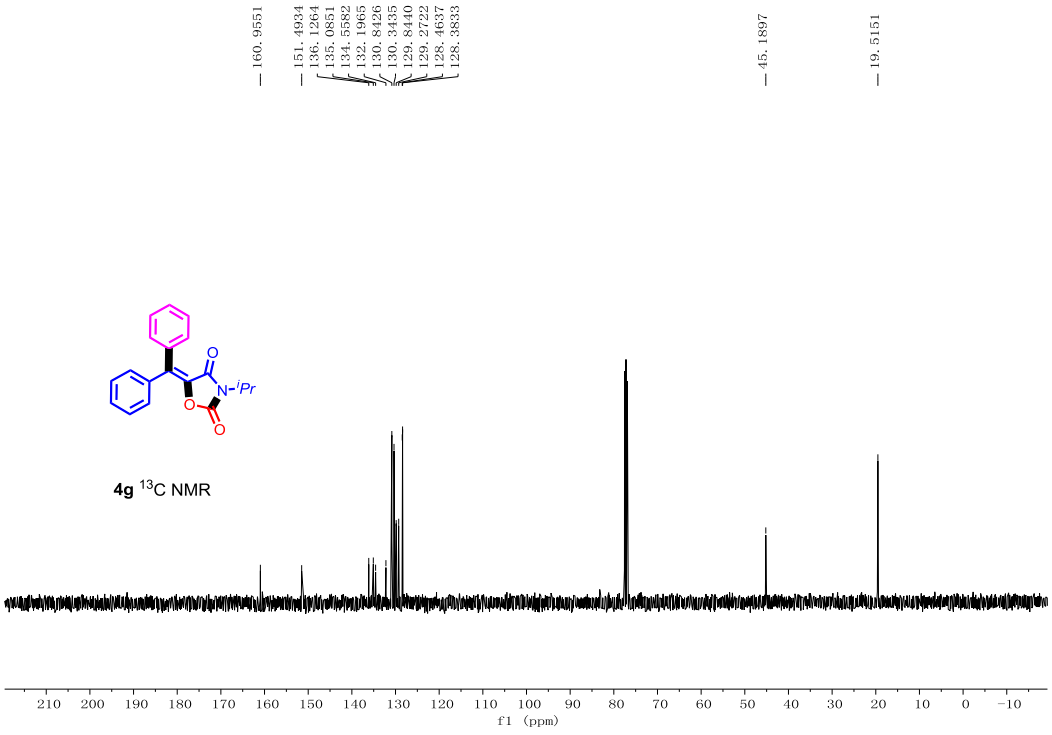
7.4255
7.4207
7.4158
7.4119
7.3938
7.3889
7.3843
7.3762
7.3725
7.3679
7.3632
7.3580
7.3197
7.3125
7.3083
7.3038
7.2985
7.2940
7.2887
4.3952
4.3779
4.3606
4.3433
4.3260
1.4561
1.4387
O
N-iPr
O
O
4g 1H NMR
4.93
2.94
1.92
0.97
6.00
f1 (ppm)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132.1965
130.8426
130.3435
129.8440
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128.3833
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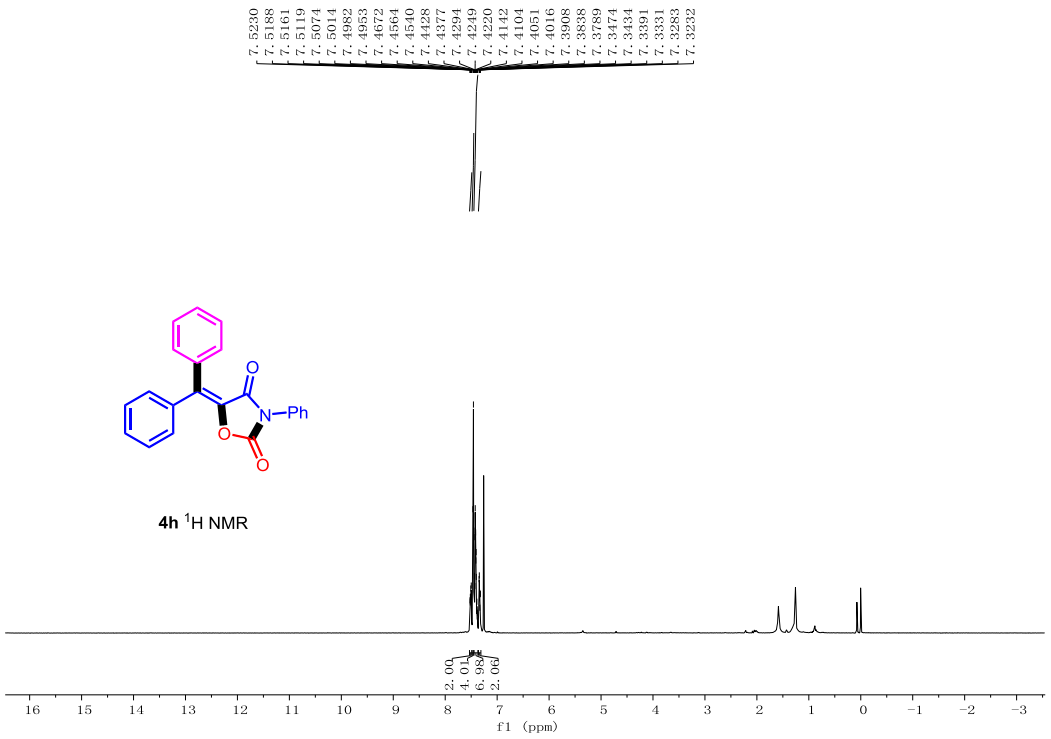
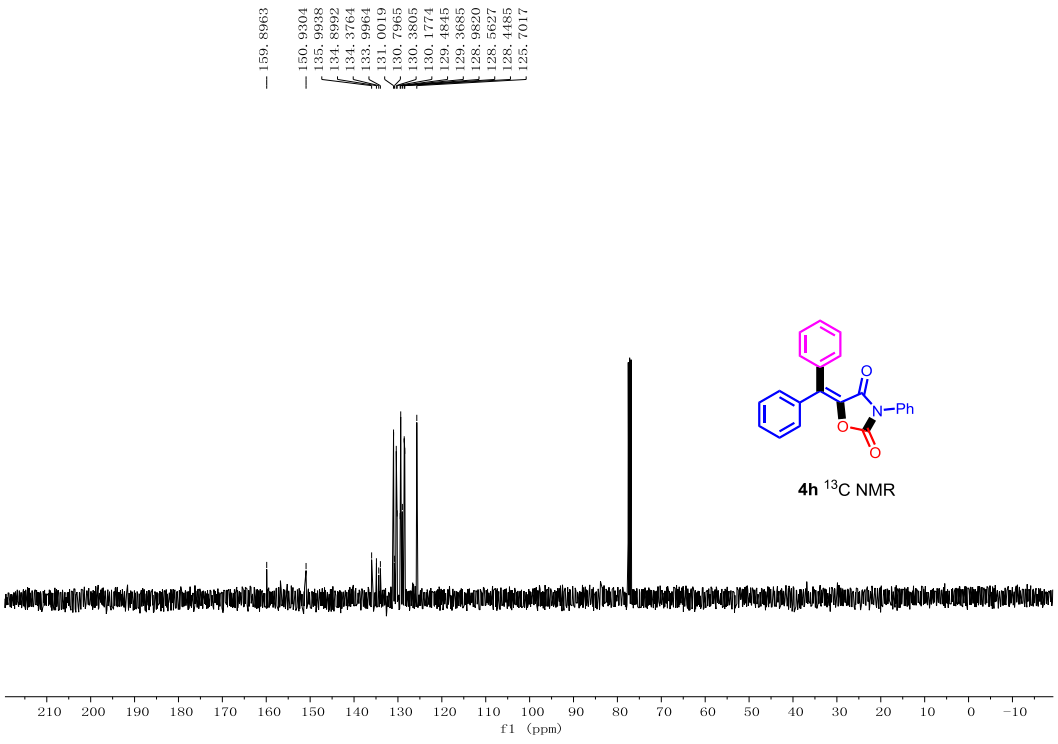
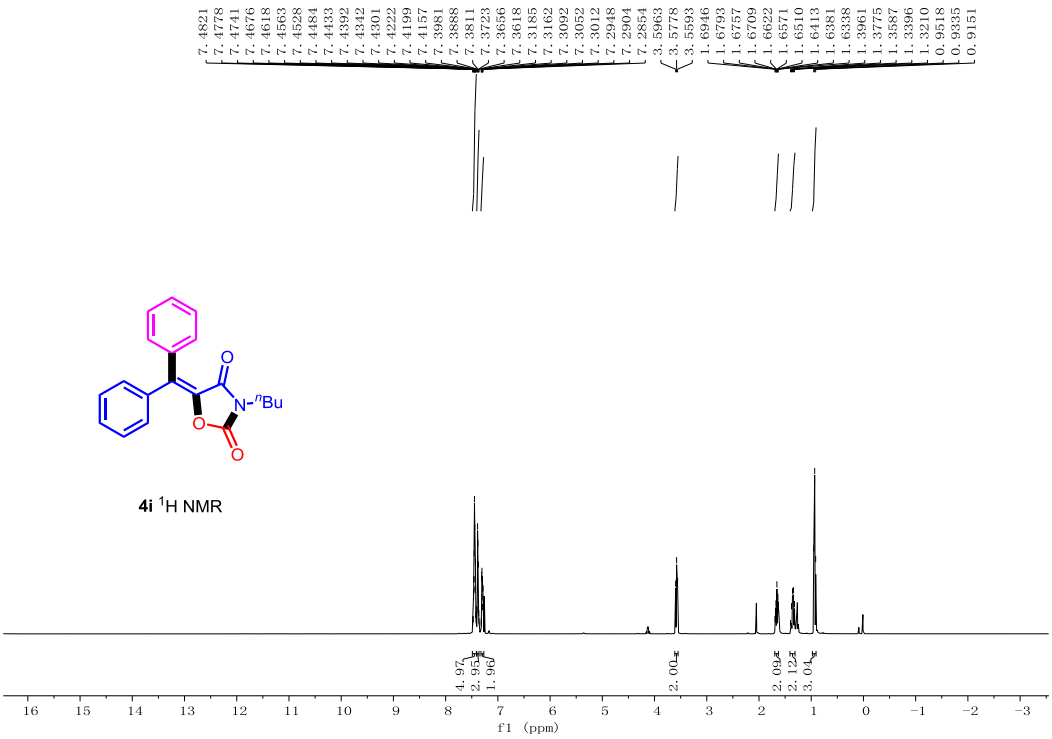
O
N-iPr
O
O
4g 13C NMR
f1 (ppm)



4h ^{1}H NMR



4h ^{13}C NMR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7.4342
7.4301
7.4222
7.4199
7.4157
7.3981
7.3888
7.3811
7.3723
7.3656
7.3618
7.3185
7.3162
7.3092
7.3052
7.3012
7.2948
7.2904
7.2854
3.5963
3.5778
3.5593
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1.6793
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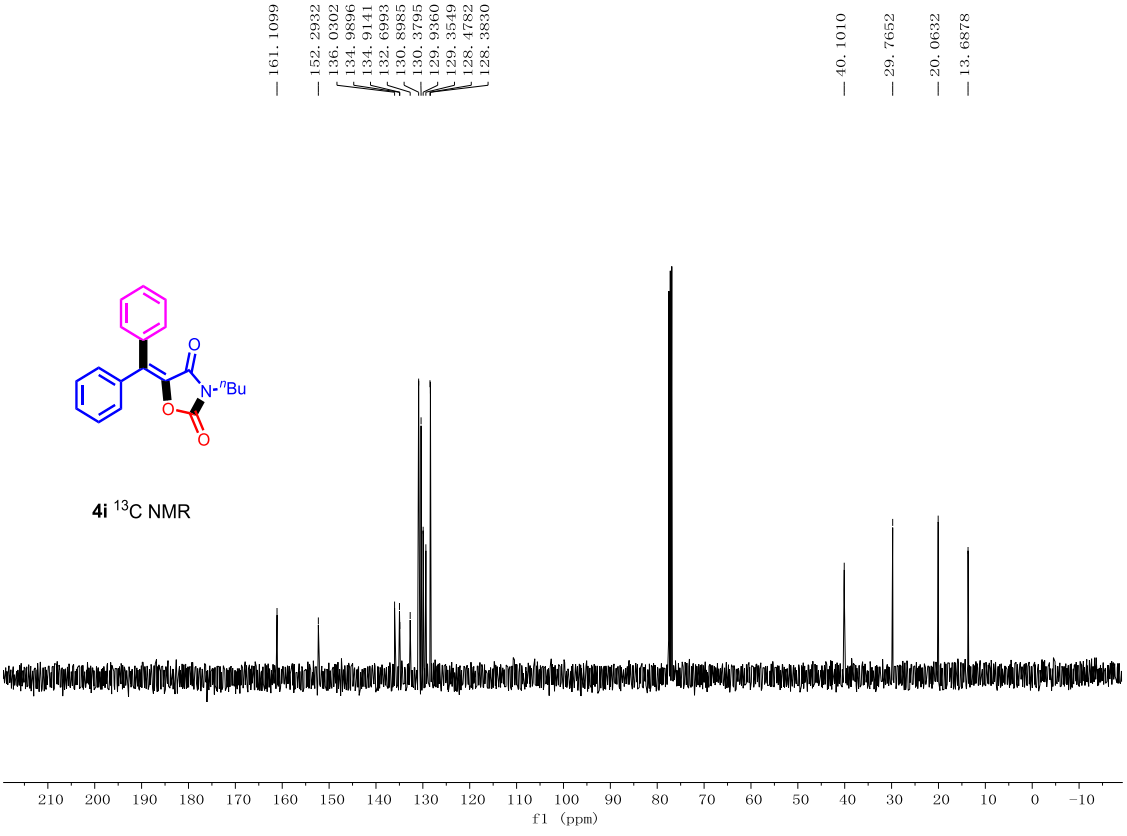
1.6571
1.6510
1.6413
1.6381
1.6338
1.3961
1.3775
1.3587
1.3396
1.3210
0.9518
0.9335
0.9151
O
N-nBu
O
O
4i 1H NMR
4.97
2.95
1.96
2.00
2.09
2.12
3.04
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)


161.1099
152.2932
136.0302
134.9896
134.9141
132.6993
130.8985
130.3795
129.9360
129.3549
128.4782
128.3830
40.1010
29.7652
20.0632
13.6878
O
N-nBu
O
O
4i 13C NMR
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)