Supporting Information

Cu-Catalyzed Radical Cascade Annulations of Alkyne-Tethered *N*-Alkoxyamides with Air: Facile Access to Isoxazolidine/1,2-Oxazinane-Fused Isoquinolin-1(2*H*)-ones

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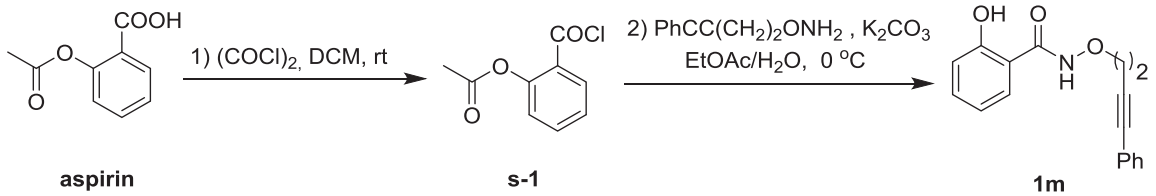
General:

All reagents were purchased from commercial suppliers and used without further purification. Flash chromatography was carried out with silica gel (200-300 mesh). Analytical TLC was performed with silica gel GF254 plates, and the products were visualized by UV detection. ^{1}H NMR （400 MHz or 300 MHz），^{13}C NMR (100.6 MHz or 75.4 MHz) and ^{19}F NMR (376 MHz) spectra were recorded in $CDCl_3$ or DMSO–d_6. Chemical shifts (δ) are reported in ppm using TMS as internal standard and spin-spin coupling constants (J) are given in Hz. The high-resolution mass spectra (HRMS) were measured on a Bruker Daltonics APEX II 47e spectrometer by ESI. Electron paramagnetic resonance (EPR) data were measured on a Bruker A300 EPR spectrometer (X-band). The calculated g values are obtained by using 2,2-diphenyl-1- picrylhydrazyl (DPPH) as a standard. The EPR spectrum of radical $\mathbf{R_N}$ was simulated using WinEPR SimFonia. Data collections for crystal structure were performed at room temperature (293 K) using MoKα radiation on a Bruker APEXII diffractometer. Integration of the frames and data reduction was carried out using SAINT. The structure was solved by direct methods using SHELXS-97.

Substrates preparation:

Substrates **1a**–**l**, **1n**–**ad** and **3a**–**n** were synthesized according to the literature.[1]

Procedure for the synthesis of substrate 1m:

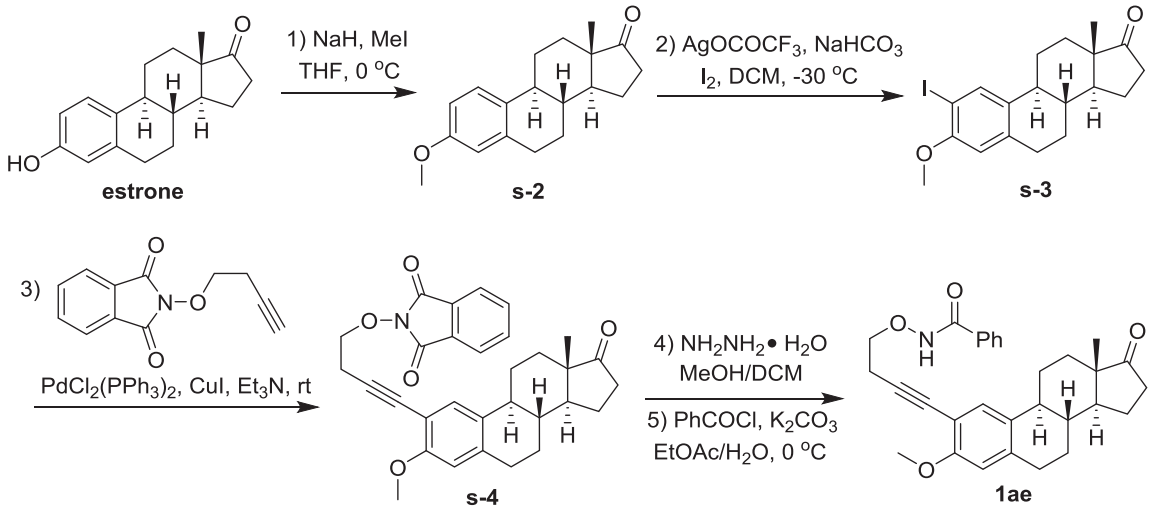


1) Aspirin (1.0 equiv) was suspended in dichloromethane (DCM) (0.5 M) and stirred at room temperature. Oxalyl chloride (2.0 equiv) was added to this stirred solution. After stirring for 0.5 h, a drop of dimethylformamide (DMF) was added to this solution and the reaction was stirred for additional 6 h at room temperature. The corresponding acyl chloride **s-1** was obtained quantitatively.[2]

2) $PhCC(CH_2)_2ONH_2$ (2.0 equiv) which was synthesized according to the literature[1] and K_2CO_3 (1.2 equiv) were suspended in a 2:1 mixture of EtOAc:H_2O (0.2 M). The resulting

solution was cooled to 0 °C followed by dropwise addition of the acyl chloride **s-1** which was obtained in the previous step. The reaction was allowed to stir at same temperature for 2 h. Upon completion (indicated by TLC), the phases were separated, and the aqueous phase was extracted three times with EtOAc. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under reduced pressure. The residue was purified by flash column chromatography on silica gel (gradient eluent of EtOAc/petroleum ether: 1/5 to 1/3) to give compound **1m**.

Procedure for the synthesis of substrate 1ae:



1) NaH (60% dispersion in oil) (1.2 equiv) was added to a solution of estrone (1.0 equiv) in dry THF (0.2 M) at 0 °C, the mixture was stirred until no gas evolution was observed and then iodomethane (3.3 equiv) was added. After stirring for 8 h at rt under an Argon atmosphere, more iodomethane (6.6 equiv) was added. The resulting mixture was stirred for next 12 h, and then reaction mixture was poured into a brine solution and extracted with ethyl acetate. The combined organic layers were dried over $MgSO_4$ and concentrated to yield compound **s-2** as white solid.[3]

2) Compound **s-2** (1.0 equiv), silver trifluoroacetate (1.1 equiv) and $NaHCO_3$ (5.0 equiv) were mixed in DCM (0.25 M) at −30 °C under Ar. I_2 (1.05 equiv) was added and the resulting mixture was stirred vigorously for 1 h during which the red colour completely disappeared. The reaction mixture was then filtered and washed with DCM and concentrated. The residue was purified by flash column chromatography on silica gel (gradient eluent of EtOAc/petroleum ether: 1/4 to 1/3) to give compound **s-3**.[4]

3) To a 100 mL three-necked round-bottom flask, under Ar, was added $PdCl_2(PPh_3)_2$ (0.01 equiv), CuI (0.02 equiv), terminal alkyne (1.0 equiv) which was synthesized according to the literature,[1] compound **s-3** (1.2 equiv) and anhydrous Et_3N (0.5 M). The mixture was stirred at room temperature for 24 h. Upon completion (indicated by TLC), the reaction is filtered over celite, washing with dichloromethane. The solvent was removed, and the residue purified by flash column chromatography on silica gel (gradient eluent of EtOAc/petroleum ether: 1/10 to 1/5) to give compound **s-4**.[1]

4) In a 100 mL round-bottom flask was charged compound **s-4** (1.0 equiv), MeOH/DCM (ratio 1:2) (0.2 M), and then slowly added Hydrazine monohydrate (1.05 equiv), then stirred at room temperature for 2 h. Upon completion (indicated by TLC), the solvent was then removed under reduce pressure. The residue was washed with DCM and filtered, collect the DCM part and removed the solvent to give the crude *O*-alkoxylamine, which was used in next step without further purification.[1]

5) The crude *O*-alkoxylamine (1.0 equiv) which was obtained in the previous step was added to a biphasic mixture of K_2CO_3 (1.2 equiv) in a 2:1 mixture of EtOAc:H_2O (0.2 M). The resulting solution was cooled to 0 ^oC followed by dropwise addition of benzoyl chloride. The reaction was allowed to stir at same temperature for 2 h. Upon completion (indicated by TLC), the phases were separated, and the aqueous phase was extracted twice with EtOAc. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under reduced pressure. The residue was purified by flash column chromatography on silica gel (gradient eluent of EtOAc/petroleum ether: 1/5 to 1/3) to give compound **1ae**.[1]

General experimental procedure:

Typical experimental procedure for the cascade annulations:

Substrates **1** or **3** (0.40 mmol, 1.00 equiv), CuCl (0.02 mmol, 0.05 equiv) and MeCN or DMF (4 mL) were added to a 25 mL round bottom flask which was equiped with a condenser pipe. The mixture was stired at 80 or 120 ^oC open to air until the starting material was completely consumed. The solvent was then removed under vacuo. When the solvent is DMF, the mixture was diluted with EtOAc. Then, organic layer was washed

with saturated brine solution five times and dried over anhydrous $MgSO_4$. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/3 to 1/1) to give the corresponding product. The identity and purity of the products were confirmed by ^1H, ^{13}C and ^{19}F NMR spectroscopic analysises.

Procedure for the follow-up transformations of 2a:

Procedure for the transformation of 2a to product 5:

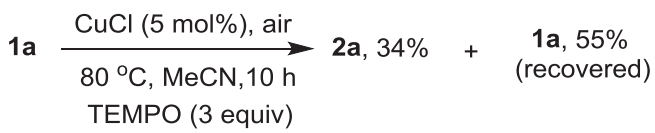
The mixture of **2a** (0.3 mmol), $CuCl_2$ (5.0 equiv) and MeCN (4 mL) were added to a 50 mL schlenk tube. The tube was evacuated and backfilled with argon three times. The mixture was stired at 80 ^oC for 48 h. The solvent was then removed under vacuo. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/3 to 1/1) to give the corresponding product **5** in 82% yield.

Procedure for the transformation of 2a to product 6:

The mixture of **2a** (0.3 mmol), Zn (11.0 equiv), In (20 mol%) and EtOH/saturated NH_4Cl(aq) (v/v = 2:1)(2 mL) were added to a 10 mL round bottom flask which was equiped with a condenser pipe. The mixture was stired at 80 ^oC for 24 h. Upon completion of the reaction, the mixture was diluted with EtOAc. Organic layer was washed with saturated brine solution and dried over anhydrous $MgSO_4$. The solvent was then removed under vacuo. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/1 to 2/1) to give the corresponding product **6** in 92% yield.[5]

Control experiments:

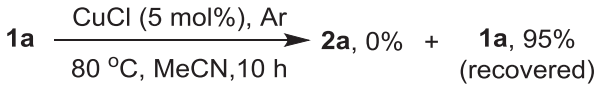
TEMPO inhibiting experiment:



1a (0.30 mmol, 1.0 equiv), CuCl (5 mol%), TEMPO (3.0 equiv) and MeCN (3 mL) were added to a 25 mL round bottom flask which was equiped with a condenser pipe. The

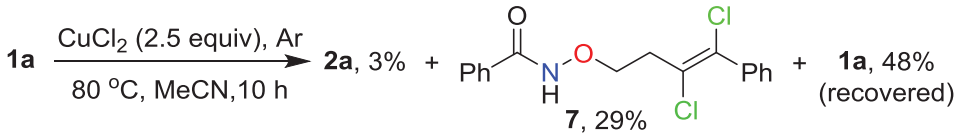
mixture was stired at 80 °C open to air for 10 h. The solvent was then removed under vacuo. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/3 to 1/1) to give the corresponding product **2a** in 34% yield, with 55% of **1a** being recovered.

Control experiment under argon atmosphere:



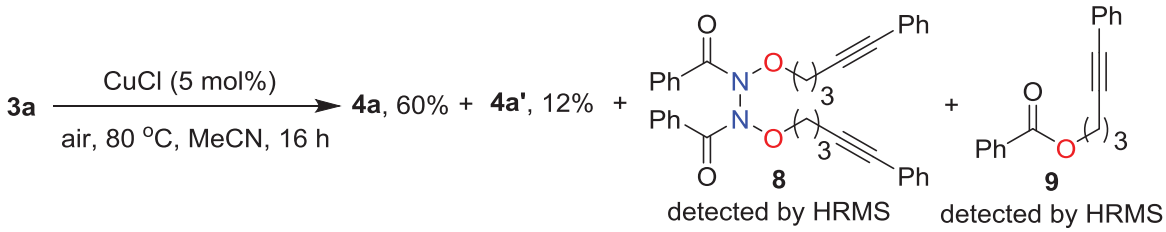
1a (0.30 mmol, 1.0 equiv), CuCl (5 mol%) and MeCN (3 mL) were added to a 50 mL schlenk tube. The tube was evacuated and backfilled with argon three times. The mixture was stired at 80 °C for 10 h. The solvent was then removed under vacuo. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/3 to 1/1) to give the corresponding product **2a** in 0% yield, with 95% of **1a** being recovered.

Control experiment with superstoichiometric $CuCl_2$ as oxidant:



1a (0.30 mmol, 1.0 equiv), $CuCl_2$ (2.5 equiv) and MeCN (3 mL) were added to a 50 mL schlenk tube. The tube was evacuated and backfilled with argon three times. The mixture was stired at 80 °C for 10 h. The solvent was then removed under vacuo. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/3 to 1/1) to give the corresponding product **2a** in 3% yield and compound **7** in 29% yield, with 48% of **1a** being recovered.

Control experiment by using HRMS to detect the reaction system:



3a (0.40 mmol, 1.0 equiv), CuCl (5 mol%) and MeCN (4 mL) were added to a 25 mL round

bottom flask which were equiped with a condenser pipe. The mixture was stired at 80 °C open to air for 16 h. The solvent was removed, and the residue purified by flash column chromatography on silica gel. Before separation by using flash column chromatography, the reaction mixture was detected by high resolution mass spectrometry (HRMS) to confirm compound **8** and **9**. The result is outlined below (Figure S1 and Figure S2).

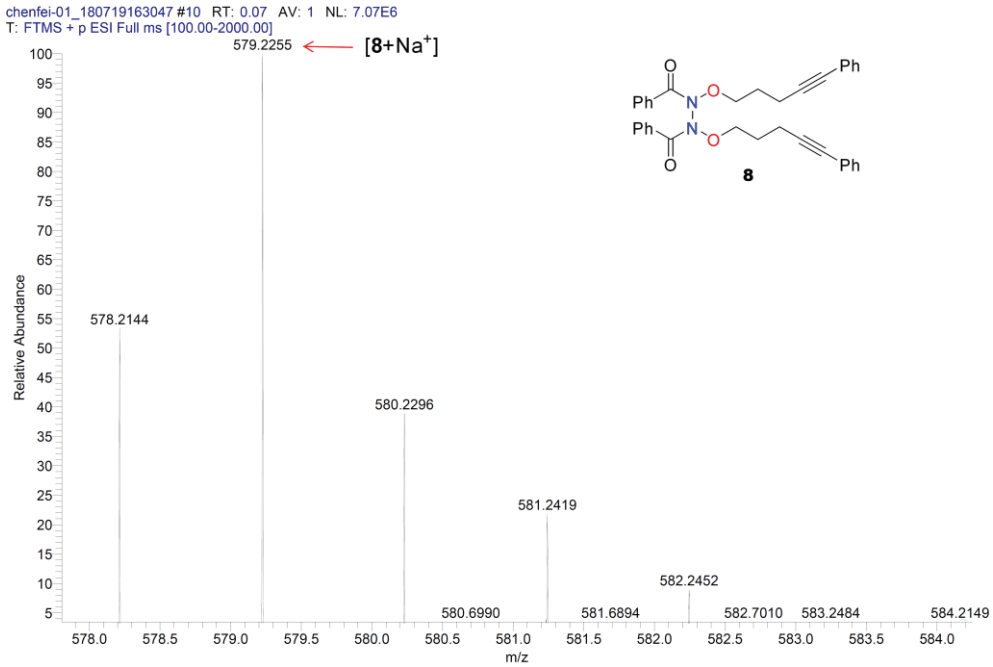


Figure S1. The HRMS spectrum of compound **8**.

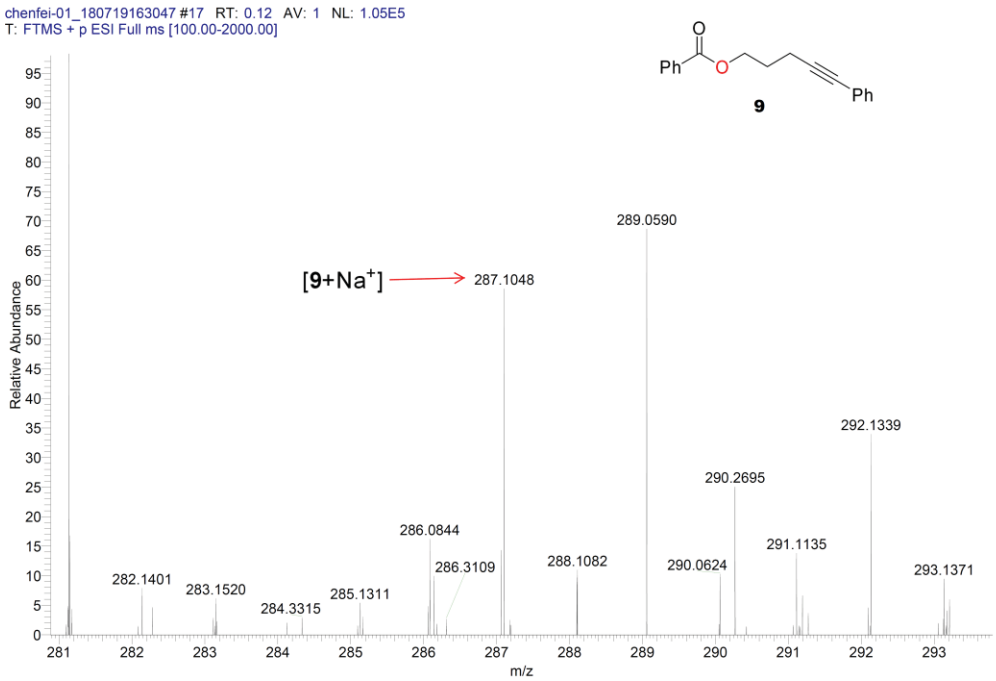
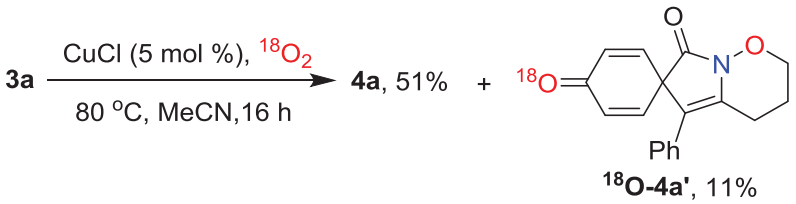


Figure S2. The HRMS spectrum of compound **9**.

The ^{18}O-labeling experiment:

$^{18}O_2$ -labeling experiment:



3a (0.30 mmol, 1.0 equiv), CuCl (5 mol%) and MeCN (3 mL) were added to a 50 mL schlenk tube. The tube was evacuated and backfilled with argon five times. The tube is then evacuated again and filled with $^{18}O_2$. The tube was closed and the reaction mixture was stired at 80 °C for 16 h. The solvent was removed, and the residue purified by flash column chromatography on silica gel. Before separation by using flash column chromatography, the reaction mixture was detected by high resolution mass spectrometry. The result is outlined below (Figure S3).

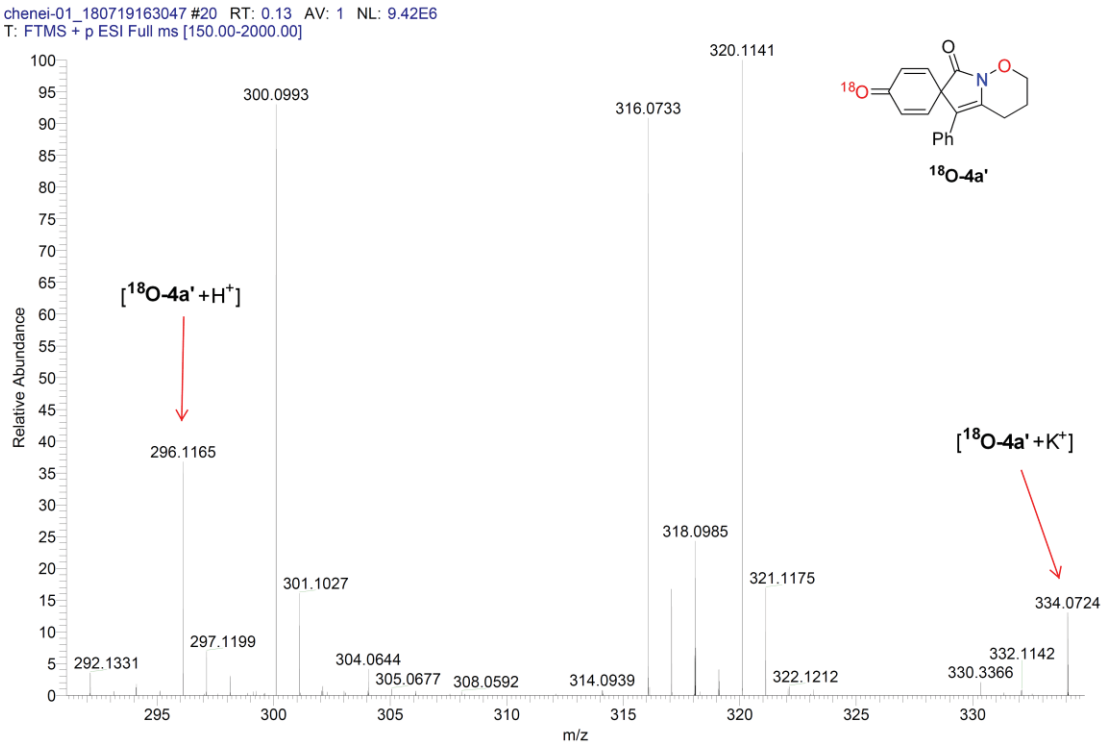
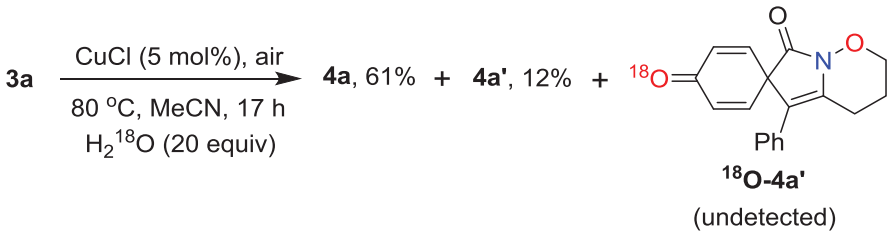


Figure S3. The HRMS spectrum of the reaction under $^{18}O_2$.

$H_2{}^{18}O$ -labeling experiment:



3a (0.20 mmol, 1.0 equiv), $H_2{}^{18}O$ (20.0 equiv), CuCl (5 mol%) and MeCN (2 mL) were added to a 10 mL round bottom flask which were equiped with a condenser pipe and a drying pipe. The mixture was stired at 80 °C open to air for 17 h. The solvent was removed, and the residue purified by flash column chromatography on silica gel. Before separation by using flash column chromatography, the reaction mixture was detected by high resolution mass spectrometry. The result is outlined below (Figure S4).

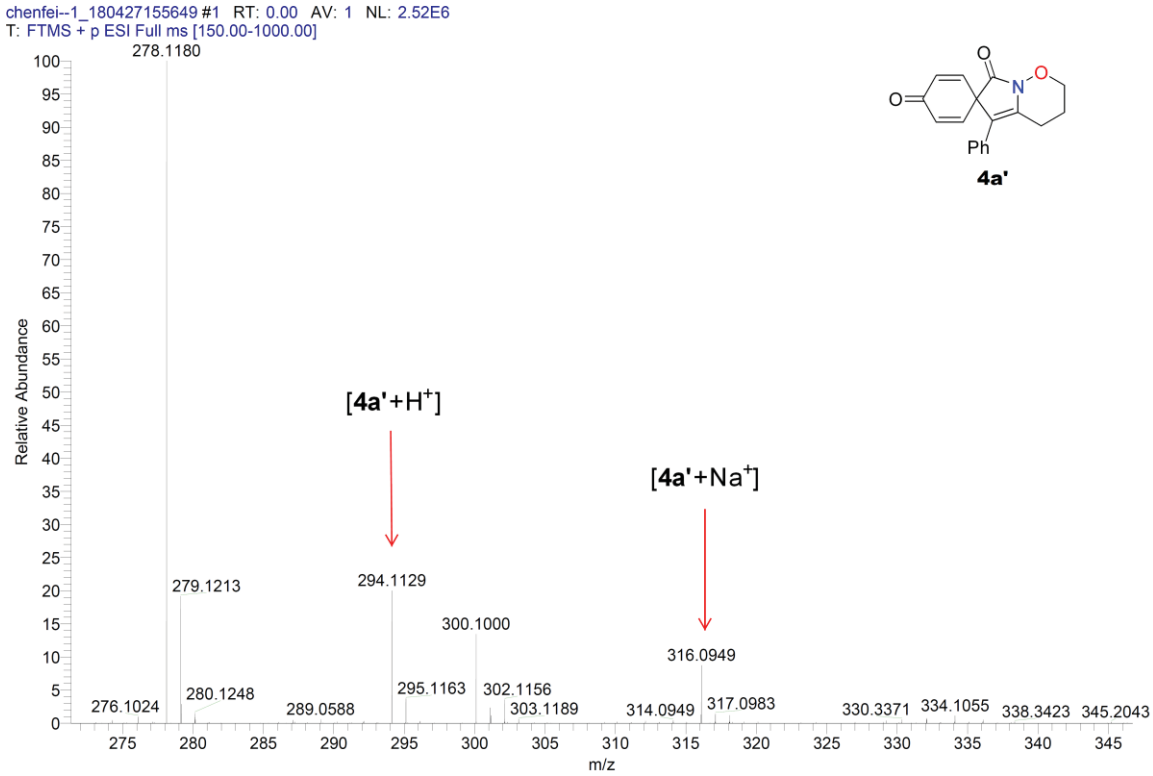
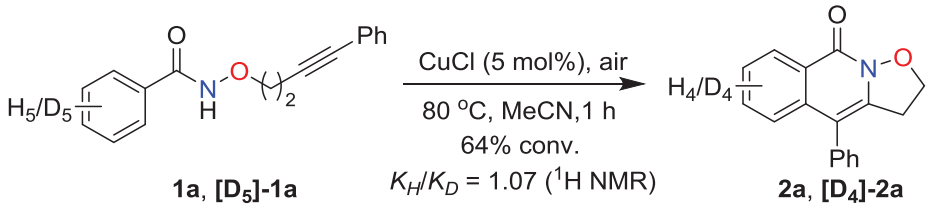


Figure S4. The HRMS spectrum of the reaction with $H_2{}^{18}O$.

The kinetic isotope effect experiment:



1a (0.20 mmol, 0.5 equiv), **[D$_5$]-1a** (0.20 mmol, 0.5 equiv), CuCl (0.02 mmol, 0.05 equiv) and MeCN (4 mL) were added to a 25 mL round bottom flask which was equiped with a condenser pipe. The mixture was stired at 80 °C open to air for 1 h. The solvent was removed, and the residue purified by flash column chromatography on silica gel. The ratio of **2a**/**[D$_4$]-2a** in the crude mixture of products was determined to be 1.07 by ^{1}H NMR (Figure S5).

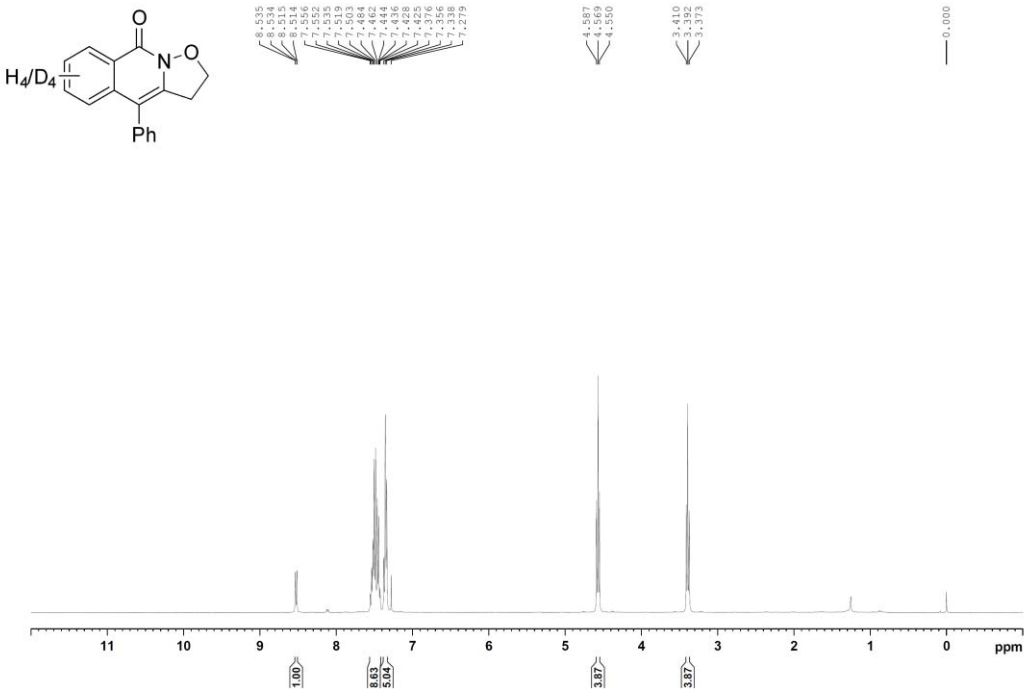
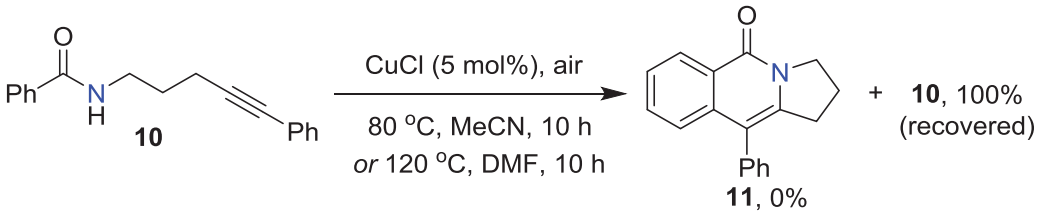


Figure S5. The ^{1}H NMR spectrum of the kinetic isotope effect experiment.

Control experiment to study the necessity of the N-O linkage in the substrates:



Compound **10** (0.30 mmol, 1.0 equiv) which was synthesized according to the literature,[6] CuCl (5 mol%) and MeCN (3 mL) were added to a 25 mL round bottom flask which was equiped with a condenser pipe. The mixture was stired at 80 °C open to air for 10 h. The solvent was then removed under vacuo. After removing the solvent under vacuo, the residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/5 to 1/3) to give the corresponding product **11** in 0% yield, with 100% of **10** being recovered. Furthermore, when the reaction was conducted at 120 °C in DMF, the same result was obtained.

General procedure of the EPR experiments:

A) and B): 2,2-dimethyl-3,4-dihydro-2*H*-pyrrole 1-oxide (DMPO) (0.20 mmol, 1.0 equiv), CuCl (0.05 equiv) and MeCN (2 mL) were added to a 10 mL round bottom flask which was equiped with a condenser pipe. The mixture was stired at 80 °C open to air. The examples for ESR detection were collected once per hour during the reaction. The EPR spectrum is shown in Figure S6A. The enlargement of the triple signal in box of A is shown in Figure S6B. This triplet signal (a_N = 13.98 G and g = 2.0056) was attributed to a nitroxyl radical which was derived from the decomposition of the adducts of CuOO· and DMPO.[7]

C): 1a (0.20 mmol, 1.0 equiv), DMPO (1.0 equiv), CuCl (0.05 equiv) and MeCN (2 mL) were added to a 10 mL round bottom flask which was equiped with a condenser pipe. The mixture was stired at 80 °C open to air. The examples for ESR detection were collected once per hour during the reaction. The EPR spectrum is shown in Figure S6C. Data analysis suggests that an in situ generated N-centered radical is promptly trapped by DMPO to produce the metastable radical $\mathbf{R_N}$ (a_{N1} = 13.73 G, a_H = 16.59 G, a_{N2} = 2.86 G, and g = 2.0058), which is consistent with the previous report.[8] The examples for ESR detection were also detected by HRMS and the results are shown in Figure S7 and Figure S8.

D): The simulated EPR spectrum of radical $\mathbf{R_N}$ is shown in Figure S6D.

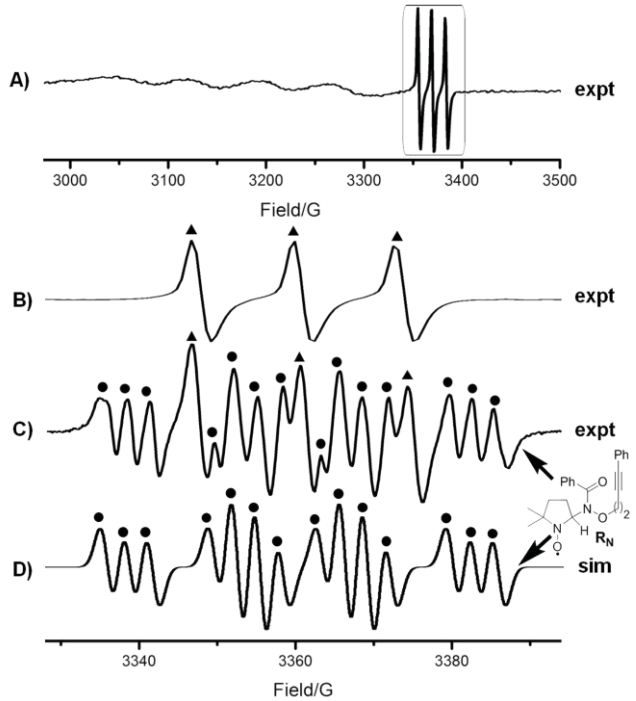


Figure S6. Electron paramagnetic resonance (EPR) spectra (X band, 9.4 GHz, rt) of (A) reaction mixture of CuCl and DMPO in MeCN under air at 80 °C; (B) the enlargement of the triple signal in box of A); (C) reaction mixture of **1a**, CuCl, and DMPO in MeCN under air at 80 °C; (D) simulated spectrum of trapping the in situ formed N-centered radical.

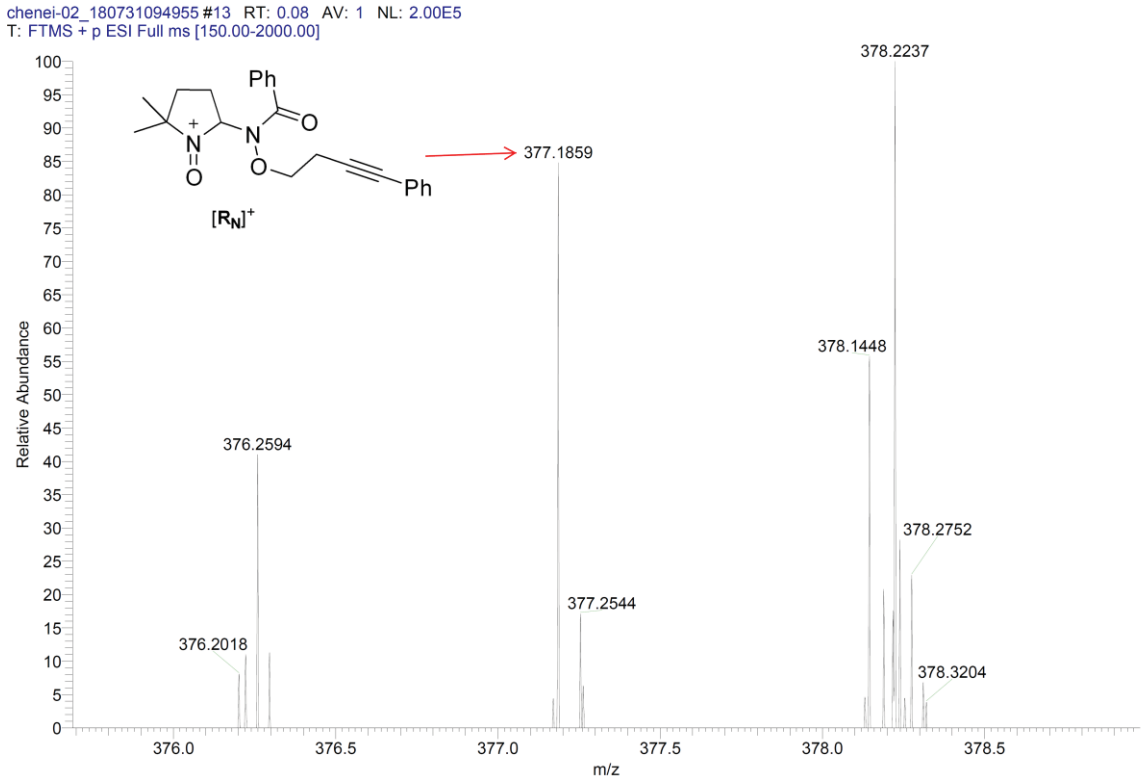


Figure S7. The HRMS spectrum of radical trapping experiments.

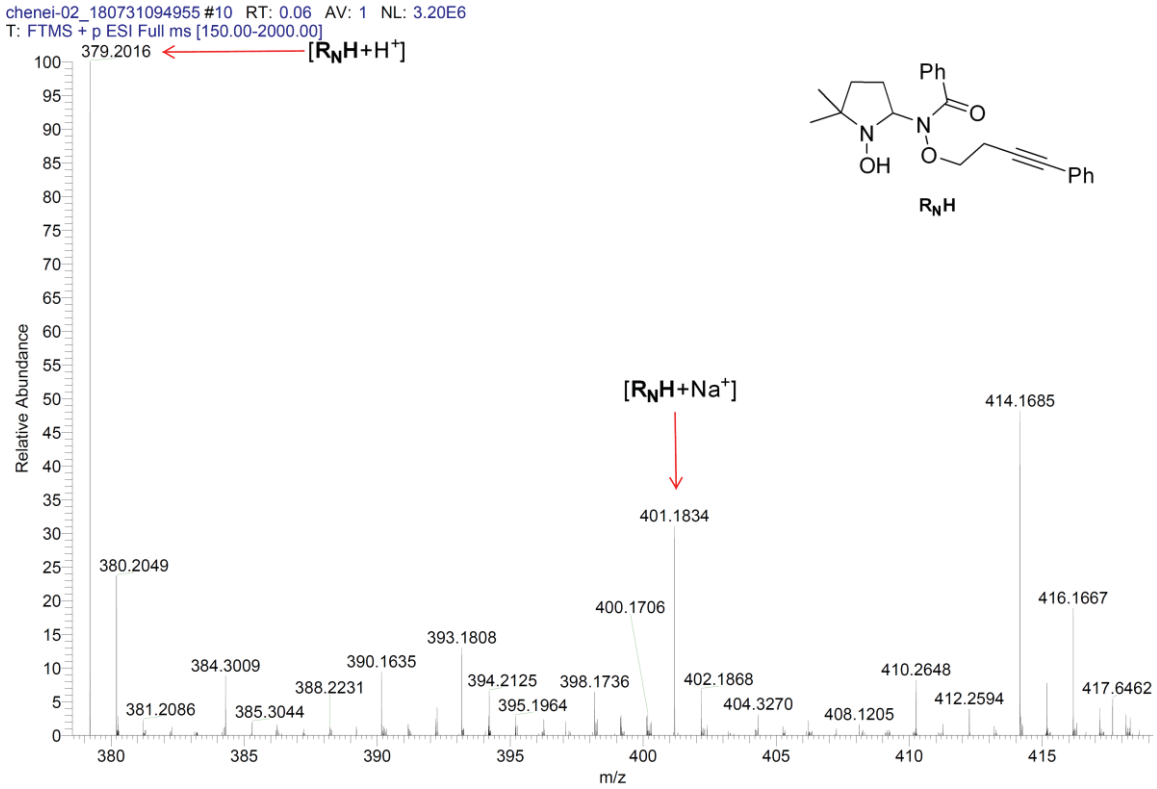
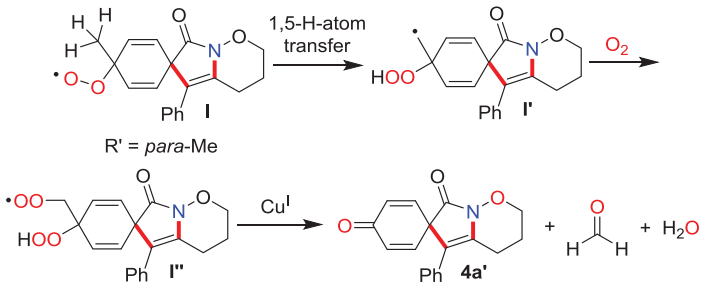


Figure S8. The HRMS spectrum of radical trapping experiments.

A plausible mechanism for the generation of 4a' from 3b:

The aerobic demethylation has been reported by literature,[9,10] however, the exact mechanism is still unclear. Herein, we provide a plausible mechanism for the generation of **4a'** from **3b** as shown below.



References:

(1) Xu, X.; Liu, Y.; Park, C.-M. Rhodium(III)-catalyzed intramolecular annulation through C–H activation: total synthesis of (±)-antofine, (±)-septicine, (±)-tylophorine, and rosettacin. *Angew. Chem., Int. Ed.* **2012**, *51*, 9372–9376.

(2) Pathak, R. K.; Dhar, S. A Nanoparticle cocktail: temporal release of predefined drug

combinations. *J. Am. Chem. Soc.* **2015**, *137*, 8324–8327.

(3) Arora, A.; Weaver, J. D. Photocatalytic generation of 2-azolyl radicals: intermediates for the azolylation of arenes and heteroarenes via C−H functionalization. *Org. Lett.* **2016**, *18*, 3996–3999.

(4) Labrie, F.; Belanger, A.; Gauthier, S.; Luu-The, V.; Merand, Y.; Poirier, D.; Provencher, L.; Singh, S. M. Inhibitors of type 5 and type 3 17*β*-hydroxysteroid dehydrogenase and methods for their use. *PCT Int. Appl.* WO 9946279A2 990916.

(5) Ashoorzadeh, A.; Archibald, G.; Caprio, V. Synthetic evaluation of an enantiopure tetrahydropyridine N-oxide. Synthesis of (+)-febrifugine. *Tetrahedron* **2009**, *65*, 4671–4680.

(6) Quiñones, N.; Seoane, A.; García-Fandiño, R.; Mascareñas, J. L.; Gulías, M. Rhodium(III)-catalyzed intramolecular annulations involving amide-directed C−H activations: synthetic scope and mechanistic studies. *Chem. Sci.* **2013**, *4*, 2874–2879.

(7) (a) Hamilton, D. E.; Drago, R. S.; Telser, J. Spin trapping of a cobalt-dioxygen complex. *J. Am. Chem. Soc.* **1984**, *106*, 5353−5355. (b) Liu, W.; Zhang, L.; Liu, X.; Liu, X.; Yang, X.; Miao, S.; Wang, W.; Wang, A.; Zhang, T. Discriminating catalytically active FeN_x species of atomically dispersed Fe−N−C catalyst for selective oxidation of the C−H Bond. *J. Am. Chem. Soc.* **2017**, *139*, 10790−10798.

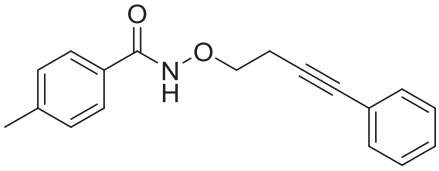
(8) Zhou, L.; Yi, H.; Zhu, L.; Qi, X.; Jiang, H.; Liu, C.; Feng, Y.; Lan, Y.; Lei, A. Tuning the reactivity of radical through a triplet diradical Cu(II) intermediate in radical oxidative cross-coupling. *Sci. Rep.* **2015**, *5*, 15934.

(9) Gao, P.; Zhang, W.; Zhang, Z. Copper-catalyzed oxidative *ipso*-annulation of activated alkynes with silanes: an approach to 3-silyl azaspiro[4,5]trienones. *Org. Lett.* **2016**, *18*, 5820–5823.

(10) Wu, L.-J.; Tan, F.-L.; Li, M.; Song, R.-J.; Li, J.-H. Fe-catalyzed oxidative spirocyclization of *N*-arylpropiolamides with silanes and TBHP involving the formation of C−Si bonds. *Org. Chem. Front.* **2017**, *4*, 350–353.

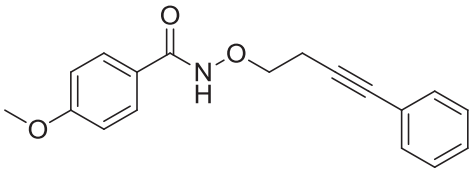
Analytical data for new substrates:

4-methyl-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1b)



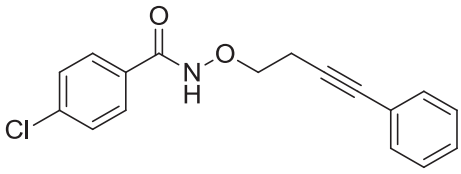
Pale yellow solid; mp 68−70 °C; R_f = 0.13 (5:1 petroleum ether / ethyl acetate); 1297 mg, 93% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.85 (brs, 1H), 7.66 (d, J = 8.0 Hz, 2H), 7.35−7.33 (m, 2H), 7.28−7.21 (m, 3H), 7.14 (d, J = 8.0 Hz, 2H), 4.17 (t, J = 6.8 Hz, 2H), 2.81 (t, J = 6.8 Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.7, 142.5, 131.5, 129.2, 128.7, 128.1, 127.8, 127.1, 123.2, 85.9, 81.9, 74.3, 21.4, 19.4; ESI-HRMS: Calcd for $C_{18}H_{17}NO_2$[M]: 279.1259, found 279.1258.

4-methoxy-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1c)



White solid; mp 82−84 °C; R_f = 0.13 (3:1 petroleum ether / ethyl acetate); 1239 mg, 84% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.35 (brs, 1H), 7.77 (d, J = 8.8 Hz, 2H), 7.34−7.32 (m, 2H), 7.26−7.19 (m, 3H), 6.80 (d, J = 8.8 Hz, 2H), 4.16 (t, J = 7.2 Hz, 2H), 3.72 (s, 3H), 2.80 (t, J = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.3, 162.4, 131.4, 129.0, 128.0, 127.7, 123.7, 123.2, 113.6, 85.9, 81.8, 74.1, 55.1, 19.3; ESI-HRMS: Calcd for $C_{18}H_{17}NO_3$[M]: 295.1208, found 295.1210.

4-chloro-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1d)

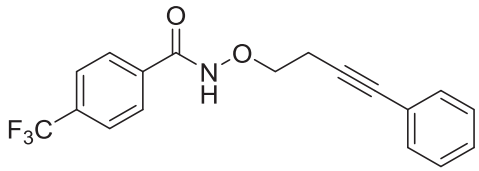


(the ratio of rotamers is 10:1)

White solid; mp 72−74 °C; R_f = 0.25 (5:1 petroleum ether / ethyl acetate); 1061 mg, 71% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.39 (brs, 1H), 9.37 (brs, 1H), 7.77 (d, J = 8.8 Hz, 2H), 7.68 (d, J = 8.8 Hz, 2H), 7.42−7.39 (m, 2H), 7.36−7.32 (m, 6H), 7.29−7.25 (m, 6H), 4.31 (t, J = 6.4 Hz, 2H), 4.21 (t, J = 6.4 Hz, 2H), 2.84 (t, J = 6.8 Hz, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 165.7, 155.5, 138.4, 136.5, 131.6, 130.1, 130.0, 129.1, 128.9, 128.6, 128.2, 128.0, 127.9, 127.4, 127.1, 123.3, 123.0, 86.0, 85.9, 82.2, 81.9, 74.6, 71.9, 20.3, 19.6;

ESI-HRMS: Calcd for $C_{17}H_{14}ClNO_2$[M]: 299.0713, found 299.0716.

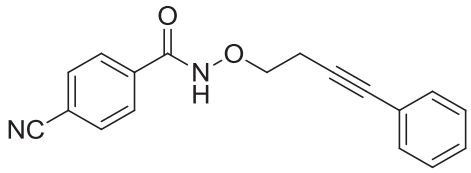
N-((4-phenylbut-3-yn-1-yl)oxy)-4-(trifluoromethyl)benzamide (1e)



(the ratio of rotamers is 9.5:1)

White solid; mp 105−107 °C; R_f = 0.19 (5:1 petroleum ether / ethyl acetate); 1132 mg, 68% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.23 (brs, 2H), 7.95 (d, J = 8.0 Hz, 2H), 7.84 (d, J = 8.0 Hz, 2H), 7.63 (d, J = 8.0 Hz, 2H), 7.57 (d, J = 8.0 Hz, 2H), 7.41−7.39 (m, 2H), 7.32−7.20 (m, 8H), 4.33 (t, J = 6.8 Hz, 2H), 4.21 (t, J = 6.4 Hz, 2H), 2.88−2.81 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 165.3, 154.9, 134.8, 133.6 (q, $^2J_{C,F}$ = 33.2 Hz), 132.4, 132.0 (q, $^2J_{C,F}$ = 32.2 Hz), 131.6, 131.5, 128.2, 128.0, 127.9, 127.7, 126.1, 125.5 (q, $^3J_{C,F}$ = 3.0 Hz), 125.2 (q, $^3J_{C,F}$ = 3.0 Hz), 123.8 (q, $^1J_{C,F}$ = 272.8 Hz), 123.4 (q, $^1J_{C,F}$ = 272.6 Hz), 123.3, 123.0, 86.0, 85.8, 82.2, 81.9, 74.5, 72.0, 20.2, 19.6; ^{19}F NMR (376 MHz, $CDCl_3$): δ -62.82, -63.06; ESI-HRMS: Calcd for $C_{18}H_{14}F_3NO_2$[M]: 333.0977, found 333.0976.

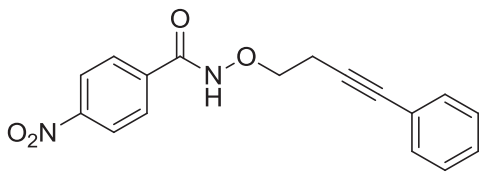
4-cyano-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1f)



(the ratio of rotamers is 3.7:1)

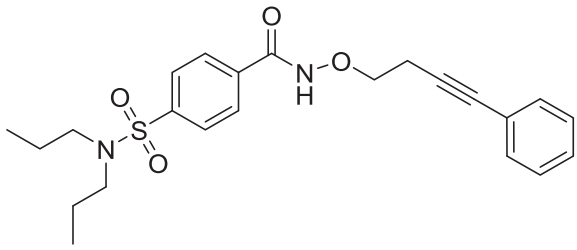
White solid; mp 113−115 °C; R_f = 0.15 (3:1 petroleum ether / ethyl acetate); 798 mg, 55% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.78 (brs, 1H), 7.94 (d, J = 8.4 Hz, 2H), 7.85 (d, J = 8.4 Hz, 2H), 7.76 (s, 1H), 7.67−7.62 (m, 4H), 7.41−7.38 (m, 2H), 7.34−7.31 (m, 2H), 7.30−7.24 (m, 6H), 4.35 (t, J = 6.8 Hz, 2H), 4.22 (t, J = 6.4 Hz, 2H), 2.88−2.83 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 164.4, 154.0, 135.4, 133.4, 132.2, 131.9, 131.4, 131.3, 128.1, 127.93, 127.88, 127.8, 126.2, 123.1, 122.9, 118.4, 117.7, 115.2, 113.2, 85.8, 85.6, 82.1, 81.8, 74.4, 72.0, 20.1, 19.4; ESI-HRMS: Calcd for $C_{18}H_{14}N_2O_2$[M]: 290.1055, found 290.1059.

4-nitro-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1g)



White solid; mp 134−136 °C; R_f = 0.10 (5:1 petroleum ether / ethyl acetate); 682 mg, 44% yield; ^{1}H NMR (400 MHz, DMSO–d_6): δ 12.18 (brs, 1H), 8.30 (d, J = 8.4 Hz, 2H), 8.01 (d, J = 8.4 Hz, 2H), 7.39−7.33 (m, 5H), 4.10 (t, J = 6.4 Hz, 2H), 2.85 (t, J = 5.6 Hz, 2H); ^{13}C NMR (100.6 MHz, DMSO–d_6): δ 162.8, 149.3, 138.0, 131.4, 128.8, 128.6, 128.2, 123.7, 123.0, 87.5, 81.4, 73.4, 19.1; ESI-HRMS: Calcd for $C_{17}H_{14}N_2O_4$[M]: 310.0954, found 310.0951.

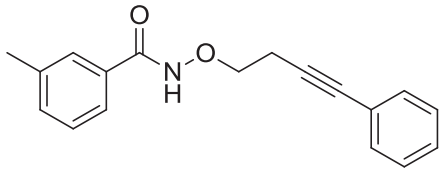
4-(N,N-dipropylsulfamoyl)-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1h)



(the ratio of rotamers is 12.5:1)

White solid; mp 104−106 °C; R_f = 0.18 (3:1 petroleum ether / ethyl acetate); 1819 mg, 85% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ10.51 (brs, 1H), 10.41 (brs, 1H), 7.96 (d, J = 8.4 Hz, 2H), 7.87 (d, J = 8.8 Hz, 2H), 7.79 (d, J = 8.4 Hz, 2H), 7.70 (d, J = 8.4 Hz, 2H), 7.41−7.38 (m, 2H), 7.36−7.33 (m, 2H), 7.29−7.22 (m, 6H), 4.32 (t, J = 6.8 Hz, 2H), 4.22 (t, J = 6.8 Hz, 2H), 3.08−3.01 (m, 8H), 2.85 (t, J = 6.8 Hz, 4H), 1.55−1.45 (m, 8H), 0.87−0.82 (m, 12H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 164.9, 154.4, 142.8, 141.2, 135.2, 132.8, 131.4, 128.1, 128.0, 127.8 126.9, 126.7, 126.2, 123.2, 123.0, 85.8, 85.6, 81.9, 81.8, 74.3, 71.9, 49.8, 21.7, 20.1, 19.4, 10.9; ESI-HRMS: Calcd for $C_{23}H_{28}N_2O_4S$[M]: 428.1770, found 428.1764.

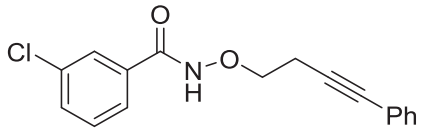
3-methyl-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1i)



Pale yellow solid; mp 63−65 °C; R_f = 0.17 (5:1 petroleum ether / ethyl acetate); 1088 mg, 78% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.74 (brs, 1H), 7.56−7.52 (m, 2H), 7.37−7.34 (m,

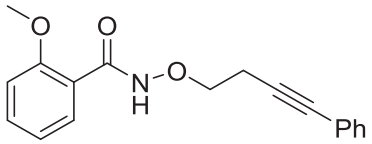
2H), 7.28−7.21 (m, 5H), 4.19 (t, *J* = 6.8 Hz, 2H), 2.82 (t, *J* = 6.8 Hz, 2H), 2.29 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.8, 138.4, 132.7, 131.6, 131.5, 128.4, 128.1, 127.82, 127.75, 124.1, 123.2, 85.9, 82.0, 74.3, 21.1, 19.5; ESI-HRMS: Calcd for $C_{18}H_{17}NO_2$[M]: 279.1259, found 279.1261.

3-chloro-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1j)



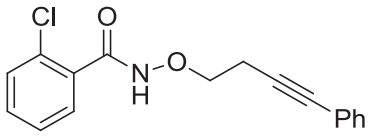
White solid; mp 81−83 °C; R_f = 0.16 (5:1 petroleum ether / ethyl acetate); 1121 mg, 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.76 (brs, 1H), 7.75−7.74 (m, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.46−7.43 (m, 1H), 7.37−7.34 (m, 2H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.28−7.25 (m, 3H), 4.21 (t, *J* = 6.8 Hz, 2H), 2.84 (t, *J* = 6.4 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 165.3, 134.7, 133.2, 132.1, 131.5, 129.9, 128.2, 128.0, 127.4, 125.3, 123.0, 85.8, 82.1, 74.5, 19.6; ESI-HRMS: Calcd for $C_{17}H_{14}ClNO_2$[M]: 299.0713, found 299.0714.

2-methoxy-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1k)



White solid; mp 54−56 °C; R_f = 0.18 (3:1 petroleum ether / ethyl acetate); 560 mg, 38% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.34 (brs, 1H), 8.20 (dd, *J* = 7.6 Hz, *J* = 1.6 Hz, 1H), 7.49−7.45 (m, 1H), 7.40−7.36 (m, 2H), 7.29−7.26 (m, 3H), 7.12−7.08 (m, 1H), 6.95 (d, *J* = 8.4 Hz, 1H), 4.25 (t, *J* = 6.8 Hz, 2H), 3.90 (s, 3H), 2.88 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 164.3, 156.9, 133.3, 132.1, 131.5, 128.1, 127.8, 123.3, 121.4, 119.3, 111.1, 85.7, 81.8, 74.1, 55.9, 19.4; ESI-HRMS: Calcd for $C_{18}H_{17}NO_3$[M]: 295.1208, found 295.1204.

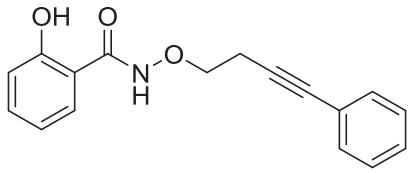
2-chloro-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1l)



Pale yellow oil; R_f = 0.09 (5:1 petroleum ether / ethyl acetate); 688 mg, 46% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.47 (brs, 1H), 7.47 (d, *J* = 7.2 Hz, 1H), 7.38−7.30 (m, 4H),

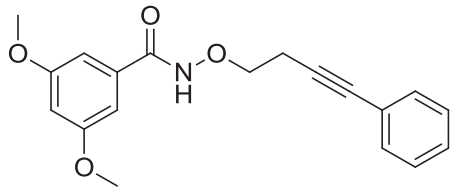
7.26−7.21 (m, 4H), 4.19 (t, *J* = 6.8 Hz, 2H), 2.83 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 164.7, 132.1, 131.7, 131.5, 131.2, 130.0, 129.9, 128.1, 127.8, 126.8, 123.2, 85.5, 82.0, 74.3, 19.3; ESI-HRMS: Calcd for $C_{17}H_{14}ClNO_2$[M]: 299.0713, found 299.0711.

2-hydroxy-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1m)



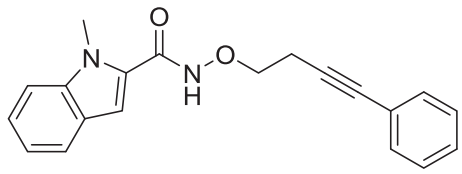
White solid; mp 68−70 °C; R_f = 0.26 (5:1 petroleum ether / ethyl acetate); 984 mg, 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.45 (brs, 1H), 9.65 (brs, 1H), 7.39−7.35 (m, 4H), 7.31−7.25 (m, 3H), 6.99 (d, *J* = 8.0 Hz, 1H), 6.77−6.73 (m, 1H), 4.21 (t, *J* = 6.8 Hz, 2H), 2.84 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 168.9, 160.8, 134.8, 131.6, 128.2, 128.0, 125.6, 123.0, 119.1, 118.5, 112.2, 85.7, 82.3, 74.8, 19.6; ESI-HRMS: Calcd for $C_{17}H_{15}NO_3$[M]: 281.1052, found 281.1061.

3,5-dimethoxy-N-((4-phenylbut-3-yn-1-yl)oxy)benzamide (1n)



White solid; mp 83−85 °C; R_f = 0.19 (3:1 petroleum ether / ethyl acetate); 942 mg, 58% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.17 (brs, 1H), 7.34−7.32 (m, 2H), 7.26−7.22 (m, 3H), 6.90 (d, *J* = 1.2 Hz, 2H), 6.52 (s, 1H), 4.18 (t, *J* = 6.8 Hz, 2H), 3.71 (s, 6H), 2.81 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.3, 160.6, 133.4, 131.4, 128.1, 127.7, 123.1, 104.8, 104.2, 85.7, 81.9, 74.2, 55.3, 19.3; ESI-HRMS: Calcd for $C_{19}H_{19}NO_4$[M]: 325.1314, found 325.1315.

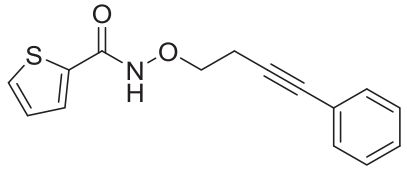
1-methyl-N-((4-phenylbut-3-yn-1-yl)oxy)-1H-indole-2-carboxamide (1o)



Pale yellow solid; mp 101−103 °C; R_f = 0.14 (5:1 petroleum ether / ethyl acetate); 874 mg, 55% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.67 (brs, 1H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.34 (d, *J* =

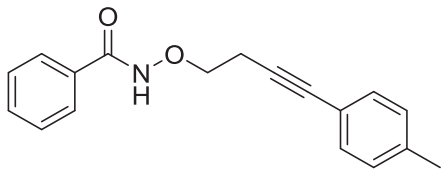
6.8 Hz, 2H), 7.30−7.27 (m, 2H), 7.24−7.18 (m, 3H), 7.11−7.08 (m, 1H), 6.81 (s, 1H), 4.19 (t, *J* = 6.8 Hz, 2H), 3.89 (s, 3H), 2.82 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 162.0, 139.0, 131.5, 128.1, 127.8, 125.7, 124.3, 123.1, 121.8, 120.4, 110.0, 105.0, 85.8, 82.0, 74.5, 31.2, 19.4; ESI-HRMS: Calcd for $C_{20}H_{18}N_2O_2$[M]: 318.1368, found 318.1363.

N-((4-phenylbut-3-yn-1-yl)oxy)thiophene-2-carboxamide (1p)



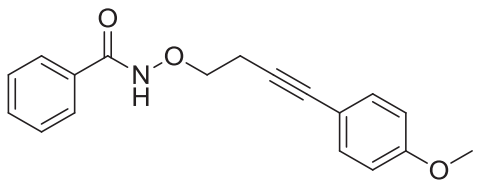
Pale yellow solid; mp 79−81 °C; R_f = 0.24 (3:1 petroleum ether / ethyl acetate); 678 mg, 50% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.19 (brs, 1H), 7.75 (s, 1H), 7.46 (d, *J* = 4.8 Hz, 1H), 7.36−7.33 (m, 2H), 7.27−7.20 (m, 3H), 7.01 (dd, *J* = 5.2 Hz, *J* = 4.0 Hz, 1H), 4.18 (t, *J* = 7.2 Hz, 2H), 2.82 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 162.0, 134.6, 131.5, 131.1, 128.1, 127.8, 127.6, 123.1, 85.7, 82.0, 74.5, 19.3; ESI-HRMS: Calcd for $C_{15}H_{13}NO_2S$[M]: 271.0667, found 271.0665.

N-((4-(p-tolyl)but-3-yn-1-yl)oxy)benzamide (1q)



White solid; mp 93−95 °C; R_f = 0.13 (5:1 petroleum ether / ethyl acetate); 1242 mg, 89% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.95 (brs, 1H), 7.76−7.74 (m, 2H), 7.48−7.44 (m, 1H), 7.36−7.33 (m, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 4.17 (t, *J* = 6.8 Hz, 2H), 2.81 (t, *J* = 6.8 Hz, 2H), 2.31 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.6, 137.8, 132.0, 131.5, 131.4, 128.9, 128.5, 127.1, 120.0, 85.0, 82.0, 74.4, 21.3, 19.4; ESI-HRMS: Calcd for $C_{18}H_{17}NO_2$[M]: 279.1259, found 279.1255.

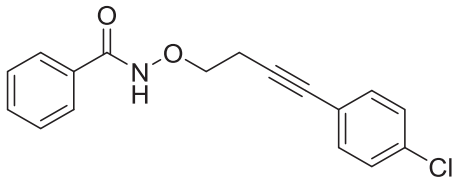
N-((4-(4-methoxyphenyl)but-3-yn-1-yl)oxy)benzamide (1r)



Pale yellow solid; mp 53−55 °C; R_f = 0.22 (3:1 petroleum ether / ethyl acetate); 1224 mg,

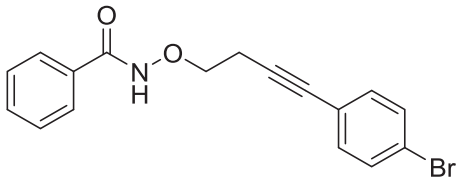
83% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.65 (brs, 1H), 7.74 (d, J = 7.2 Hz, 2H), 7.50–7.46 (m, 1H), 7.38–7.33 (m, 2H), 7.29 (d, J = 8.8 Hz, 2H), 6.78 (d, J = 8.8 Hz, 2H), 4.19 (t, J = 6.8 Hz, 2H), 3.77 (s, 3H), 2.82 (t, J = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.6, 159.2, 132.9, 132.0, 131.6, 128.6, 127.1, 115.3, 113.8, 84.3, 81.8, 74.5, 55.2, 19.5; ESI-HRMS: Calcd for $C_{18}H_{17}NO_3$[M]: 295.1208, found 295.1209.

N-((4-(4-chlorophenyl)but-3-yn-1-yl)oxy)benzamide (1s)



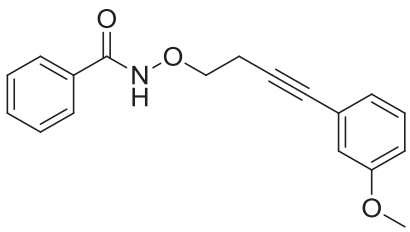
White solid; mp 99–101 °C; R_f = 0.13 (5:1 petroleum ether / ethyl acetate); 1151 mg, 77% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.16 (brs, 1H), 7.76 (d, J = 7.2 Hz, 2H), 7.48–7.42 (m, 1H), 7.36–7.32 (m, 2H), 7.24 (d, J = 8.4 Hz, 2H), 7.19 (d, J = 8.4 Hz, 2H), 4.16 (t, J = 6.8 Hz, 2H), 2.80 (t, J = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.6, 133.7, 132.7, 132.0, 131.5, 128.5, 128.4, 127.1, 121.6, 86.9, 80.8, 74.1, 19.4; ESI-HRMS: Calcd for $C_{17}H_{14}ClNO_2$[M]: 299.0713, found 299.0720.

N-((4-(4-bromophenyl)but-3-yn-1-yl)oxy)benzamide (1t)



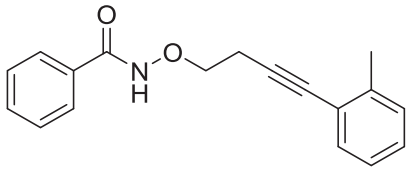
Pale yellow solid; mp 114–116 °C; R_f = 0.11 (5:1 petroleum ether / ethyl acetate); 1252 mg, 73% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.61 (brs, 1H), 7.76–7.74 (m, 2H), 7.52–7.48 (m, 1H), 7.40–7.36 (m, 4H), 7.19 (d, J = 8.4 Hz, 2H), 4.19 (t, J = 6.8 Hz, 2H), 2.82 (t, J = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.7, 133.0, 132.1, 131.5, 131.4, 128.6, 127.1, 122.1, 122.0, 87.2, 81.0, 74.2, 19.5; ESI-HRMS: Calcd for $C_{17}H_{14}BrNO_2$[M]: 343.0208, found 343.0205.

N-((4-(3-methoxyphenyl)but-3-yn-1-yl)oxy)benzamide (1u)



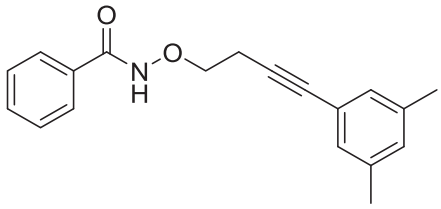
Pale yellow oil; R_f = 0.18 (3:1 petroleum ether / ethyl acetate); 1195 mg, 81% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.54 (brs, 1H), 7.74 (d, J = 7.2 Hz, 2H), 7.51−7.47 (m, 1H), 7.39−7.35 (m, 2H), 7.19−7.15 (m, 1H), 6.96 (d, J = 7.6 Hz, 1H), 6.90 (s, 1H), 6.84 (dd, J = 8.4 Hz, J = 2.4 Hz, 1H), 4.21 (t, J = 6.8 Hz, 2H), 3.75 (s, 3H), 2.84 (t, J = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.6, 159.1, 132.1, 131.6, 129.2, 128.6, 127.1, 124.14, 124.07, 116.3, 114.5, 85.8, 82.0, 74.4, 55.2, 19.5; ESI-HRMS: Calcd for $C_{18}H_{17}NO_3$[M]: 295.1208, found 295.1213.

N-((4-(o-tolyl)but-3-yn-1-yl)oxy)benzamide (1v)



White solid; mp 49−51 ^{o}C; R_f = 0.15 (5:1 petroleum ether / ethyl acetate); 1200 mg, 86% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.38 (brs, 1H), 7.74−7.72 (m, 2H), 7.51−7.47 (m, 1H), 7.39−7.32 (m, 3H), 7.20−7.15 (m, 2H), 7.10−7.06 (m, 1H), 4.22 (t, J = 6.8 Hz, 2H), 2.89 (t, J = 6.8 Hz, 2H), 2.38 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.7, 140.0, 132.1, 131.8, 131.6, 129.3, 128.6, 127.9, 127.1, 125.4, 122.9, 89.6, 81.0, 74.6, 20.6, 19.7; ESI-HRMS: Calcd for $C_{18}H_{17}NO_2$[M]: 279.1259, found 279.1253.

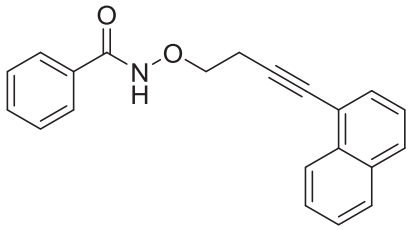
N-((4-(3,5-dimethylphenyl)but-3-yn-1-yl)oxy)benzamide (1w)



White solid; mp 113−115 ^{o}C; R_f = 0.16 (5:1 petroleum ether / ethyl acetate); 1304 mg, 89% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.26 (brs, 1H), 7.74 (d, J = 7.2 Hz, 2H), 7.52−7.48 (m, 1H), 7.40−7.36 (m, 2H), 6.99 (s, 2H), 6.92 (s, 1H), 4.22 (t, J = 6.8 Hz, 2H), 2.84 (t, J =

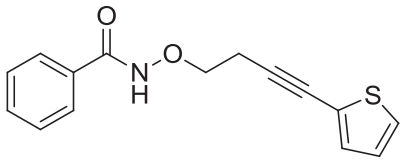
6.4 Hz, 2H), 2.25 (s, 6H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.6, 137.8, 132.1, 131.7, 129.9, 129.3, 128.6, 127.1, 122.7, 85.2, 82.4, 74.6, 21.0, 19.7; ESI-HRMS: Calcd for $C_{19}H_{19}NO_2$[M]: 293.1416, found 293.1418.

N-((4-(naphthalen-1-yl)but-3-yn-1-yl)oxy)benzamide (1x)



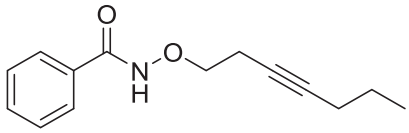
Pale yellow solid; mp 95−96 °C; R_f = 0.14 (5:1 petroleum ether / ethyl acetate); 1292 mg, 82% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.48 (brs, 1H), 8.30 (d, J = 8.0 Hz, 1H), 7.83−7.77 (m, 2H), 7.73−7.71 (m, 2H), 7.58 (d, J = 6.4 Hz, 1H), 7.54−7.43 (m, 3H), 7.38−7.29 (m, 3H), 4.30 (t, J = 6.8 Hz, 2H), 2.99 (t, J = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.8, 133.4, 133.1, 132.2, 131.6, 130.3, 128.7, 128.5, 128.3, 127.2, 126.8, 126.4, 126.1, 125.2, 120.9, 91.0, 80.2, 74.7, 20.0; ESI-HRMS: Calcd for $C_{21}H_{17}NO_2$[M]: 315.1259, found 315.1254.

N-((4-(thiophen-2-yl)but-3-yn-1-yl)oxy)benzamide (1y)



Pale yellow solid; mp 104−106 °C ; R_f = 0.12 (5:1 petroleum ether / ethyl acetate); 650 mg, 48% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.57 (brs, 1H), 7.76−7.74 (m, 2H), 7.52−7.48 (m, 1H), 7.40−7.36 (m, 2H), 7.18 (dd, J = 5.2 Hz, J = 1.2 Hz, 1H), 7.11 (d, J = 3.2 Hz, 1H), 6.92 (dd, J = 5.2 Hz, J = 3.6 Hz, 1H), 4.19 (t, J = 6.8 Hz, 2H), 2.86 (t, J = 6.8 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.7, 132.1, 131.6, 128.6, 127.1, 126.8, 126.4, 123.2, 90.0, 75.2, 74.1, 19.8; ESI-HRMS: Calcd for $C_{15}H_{13}NO_2S$[M]: 271.0667, found 271.0663.

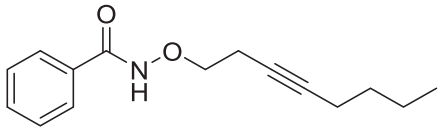
N-(hept-3-yn-1-yloxy)benzamide (1z)



Colourless oil; R_f = 0.22 (5:1 petroleum ether / ethyl acetate); 1097 mg, 95% yield; ^{1}H

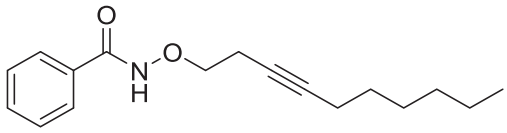
NMR (400 MHz, $CDCl_3$): δ 9.52 (brs, 1H), 7.76−7.74 (m, 2H), 7.53−7.49 (m, 1H), 7.43−7.39 (m, 2H), 4.09 (t, J = 6.8 Hz, 2H), 2.62−2.57 (m, 2H), 2.12−2.07 (m, 2H), 1.52−1.42 (m, 2H), 0.94 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.5, 132.0, 131.7, 128.6, 127.1, 82.0, 75.9, 74.8, 22.2, 20.6, 18.8, 13.4; ESI-HRMS: Calcd for $C_{14}H_{17}NO_2$[M]: 231.1259, found 231.1254.

N-(oct-3-yn-1-yloxy)benzamide (1aa)



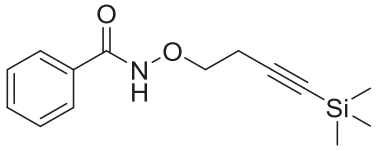
Colourless oil; R_f = 0.29 (5:1 petroleum ether / ethyl acetate); 1090 mg, 89% yield; ^1H NMR (400 MHz, $CDCl_3$): δ 9.52 (brs, 1H), 7.76−7.74 (m, 2H), 7.53−7.49 (m, 1H), 7.43−7.39 (m, 2H), 4.09 (t, J = 6.8 Hz, 2H), 2.61−2.56 (m, 2H), 2.14−2.10 (m, 2H), 1.47−1.32 (m, 4H), 0.88 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.5, 132.0, 131.7, 128.6, 127.1, 82.1, 75.7, 74.9, 30.9, 21.9, 18.8, 18.3, 13.6; ESI-HRMS: Calcd for $C_{15}H_{19}NO_2$[M]: 245.1416, found 245.1415.

N-(dec-3-yn-1-yloxy)benzamide (1ab)



Colourless oil; R_f = 0.26 (5:1 petroleum ether / ethyl acetate); 1242 mg, 91% yield; ^1H NMR (400 MHz, $CDCl_3$): δ 9.44 (brs, 1H), 7.76−7.74 (m, 2H), 7.53−7.49 (m, 1H), 7.43−7.39 (m, 2H), 4.09 (t, J = 7.2 Hz, 2H), 2.62−2.57 (m, 2H), 2.14−2.09 (m, 2H), 1.48−1.41 (m, 2H), 1.37−1.21 (m, 6H), 0.88 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.5, 132.0 131.7, 128.6, 127.1, 82.2, 75.8, 74.9, 31.3, 28.8, 28.5, 22.5, 18.8, 18.7, 14.0; ESI-HRMS: Calcd for $C_{17}H_{23}NO_2$[M]: 273.1729, found 273.1727.

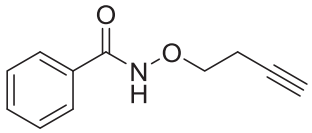
N-((4-(trimethylsilyl)but-3-yn-1-yl)oxy)benzamide (1ac)



White solid; mp 75−77 °C; R_f = 0.47 (3:1 petroleum ether / ethyl acetate); 1096 mg, 84% yield; ^1H NMR (400 MHz, $CDCl_3$): δ 9.82 (brs, 1H), 7.78−7.76 (m, 2H), 7.55−7.50 (m, 1H),

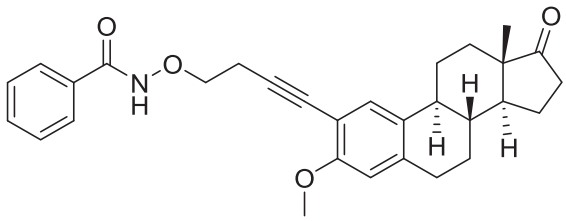
7.44−7.40 (m, 2H), 4.13 (t, *J* = 7.2 Hz, 2H), 2.69 (t, *J* = 6.8 Hz, 2H), 0.15 (s, 9H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.6, 132.0, 131.7, 128.6, 127.1, 102.7, 86.5, 74.3, 20.0, -0.07; ESI-HRMS: Calcd for $C_{14}H_{19}NO_2Si$[M]: 261.1185, found 261.1195.

N-(but-3-yn-1-yloxy)benzamide (1ad)



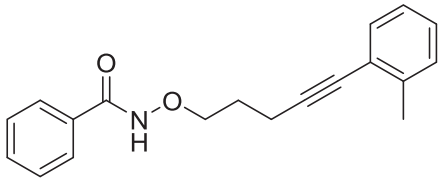
White solid; mp 61−63 °C; R_f = 0.12 (5:1 petroleum ether / ethyl acetate); 737 mg, 78% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.60 (brs, 1H), 7.76−7.74 (m, 2H), 7.54−7.50 (m, 1H), 7.43−7.39 (m, 2H), 4.13 (t, *J* = 6.8 Hz, 2H), 2.62 (td, *J* = 6.8 Hz, *J* = 2.8 Hz, 2H), 2.03 (t, *J* = 2.4 Hz, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.8, 132.1, 131.5, 128.6, 127.1, 80.4, 74.1, 70.0, 18.5; ESI-HRMS: Calcd for $C_{11}H_{11}NO_2$[M]: 189.0790, found 189.0788.

N-((4-((8R,9S,13S,14S)-3-methoxy-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-2-yl)but-3-yn-1-yl)oxy)benzamide (1ae)



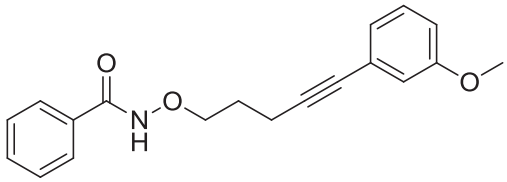
White solid; mp 144−146 °C; R_f = 0.33 (1:1 petroleum ether / ethyl acetate); 1554 mg, 66% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.04 (brs, 1H), 7.75−7.73 (m, 2H), 7.49−7.45 (m, 1H), 7.36−7.32 (m, 2H), 7.26 (s, 1H), 6.53 (s, 1H), 4.24 (t, *J* = 6.4 Hz, 2H), 3.66 (s, 3H), 2.90−2.86 (m, 4H), 2.52−2.45 (m, 1H), 2.32−2.29 (m, 1H), 2.18−2.08 (m, 2H), 2.05−1.97 (m, 2H), 1.92−1.89 (m, 1H), 1.63−1.49 (m, 2H), 1.48−1.35 (m, 4H), 0.88 (s, 3H); ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 220.8, 166.2, 157.6, 138.1, 131.94, 131.86, 131.7, 130.4, 128.4, 127.1, 110.8, 109.5, 89.4, 78.7, 74.7, 55.4, 50.2, 47.8, 43.5, 38.0, 35.7, 31.4, 29.7, 26.3, 25.7, 21.4, 20.2, 13.7; ESI-HRMS: Calcd for $C_{30}H_{33}NO_4$[M]: 471.2410, found 471.2412.

N-((5-(o-tolyl)pent-4-yn-1-yl)oxy)benzamide (3e)



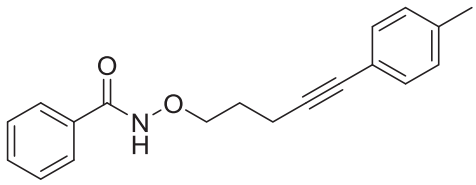
White solid; mp 62−64 °C; R_f = 0.13 (5:1 petroleum ether / ethyl acetate); 1143 mg, 78% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.89 (brs, 1H), 7.74 (d, J = 7.2 Hz, 2H), 7.46−7.42 (m, 1H), 7.36−7.32 (m, 3H), 7.17−7.13 (m, 2H), 7.10−7.05 (m, 1H), 4.14 (t, J = 6.4 Hz, 2H), 2.57 (t, J = 7.2 Hz, 2H), 2.37 (s, 3H), 1.99−1.93 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.5, 139.8, 131.8, 131.7, 129.2, 128.4, 127.6, 127.1, 125.3, 123.3, 93.0, 79.9, 75.2, 27.3, 20.6, 16.1; ESI-HRMS: Calcd for $C_{19}H_{19}NO_2$[M]: 293.1416, found 293.1412.

N-((5-(3-methoxyphenyl)pent-4-yn-1-yl)oxy)benzamide (3f)



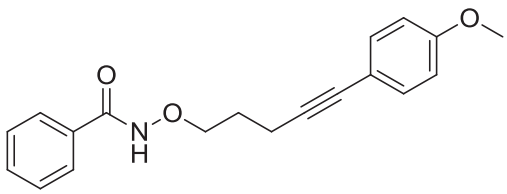
Pale yellow oil; R_f = 0.10 (5:1 petroleum ether / ethyl acetate); 1267 mg, 82% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.34 (brs, 1H), 7.74−7.72 (m, 2H), 7.51−7.47 (m, 1H), 7.40−7.36 (m, 2H), 7.20−7.16 (m, 1H), 6.98 (d, J = 7.6 Hz, 1H), 6.92 (d, J = 2.4 Hz, 1H), 6.83 (dd, J = 8.4 Hz, J = 2.8 Hz, 1H), 4.16 (t, J = 6.4 Hz, 2H), 3.76 (s, 3H), 2.57 (t, J = 7.2 Hz, 2H), 2.02−1.95 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.6, 159.2, 132.0, 131.8, 129.2, 128.6, 127.0, 124.6, 124.0, 116.3, 114.2, 89.0, 81.0, 75.4, 55.2, 27.1, 16.0; ESI-HRMS: Calcd for $C_{19}H_{19}NO_3$[M]: 309.1365, found 309.1369.

N-((5-(p-tolyl)pent-4-yn-1-yl)oxy)benzamide (3g)



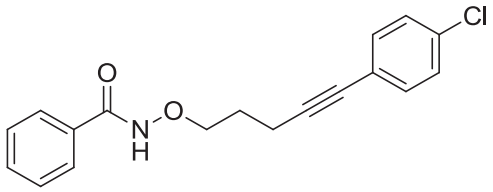
White solid; mp 82−84 °C; R_f = 0.12 (5:1 petroleum ether / ethyl acetate); 1348 mg, 92% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.71 (brs, 1H), 7.74−7.72 (m, 2H), 7.48−7.44 (m, 1H), 7.37−7.34 (m, 2H), 7.26 (d, J = 8.4 Hz, 2H), 7.06 (d, J = 8.0 Hz, 2H), 4.13 (t, J = 6.0 Hz, 2H), 2.53 (t, J = 7.2 Hz, 2H), 2.31 (s, 3H), 1.98−1.91 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.5, 137.6, 131.9, 131.7, 131.3, 128.9, 128.5, 127.1, 120.4, 88.3, 81.1, 75.3, 27.2, 21.3, 16.0; ESI-HRMS: Calcd for $C_{19}H_{19}NO_2$[M]: 293.1416, found 293.1415.

N-((5-(4-methoxyphenyl)pent-4-yn-1-yl)oxy)benzamide (3h)



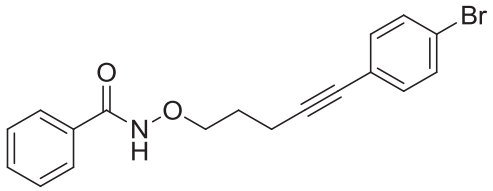
White solid; mp 84−86 °C; R_f = 0.08 (5:1 petroleum ether / ethyl acetate); 1390 mg, 90% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.35 (brs, 1H), 7.73 (d, J = 7.2 Hz, 2H), 7.51−7.47 (m, 1H), 7.40−7.36 (m, 2H), 7.31 (d, J = 8.4 Hz, 2H), 6.79 (d, J = 8.4 Hz, 2H), 4.16 (t, J = 6.0 Hz, 2H), 3.78 (s, 3H), 2.55 (t, J = 6.8 Hz, 2H), 2.01−1.94 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.6, 159.0, 132.8, 132.0, 131.8, 128.6, 127.0, 115.6, 113.8, 87.5, 80.8, 75.4, 55.2, 27.2, 16.0; ESI-HRMS: Calcd for $C_{19}H_{19}NO_3$[M]: 309.1365, found 309.1363.

N-((5-(4-chlorophenyl)pent-4-yn-1-yl)oxy)benzamide (3i)



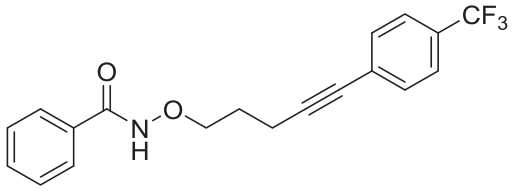
White solid; mp 124−126 °C; R_f = 0.12 (5:1 petroleum ether / ethyl acetate); 1299 mg, 83% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.35 (brs, 1H), 7.75−7.72 (m, 2H), 7.52−7.48 (m, 1H), 7.41−7.37 (m, 2H), 7.31−7.27 (m, 2H), 7.24−7.22 (m, 2H), 4.16 (t, J = 6.4 Hz, 2H), 2.57 (t, J = 6.8 Hz, 2H), 2.01−1.95 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.6, 133.5, 132.7, 132.0, 131.7, 128.6, 128.4, 127.0, 122.0, 90.1, 80.0, 75.2, 27.0, 16.0; ESI-HRMS: Calcd for $C_{18}H_{16}ClNO_2$[M]: 313.0870, found 313.0876.

N-((5-(4-bromophenyl)pent-4-yn-1-yl)oxy)benzamide (3j)



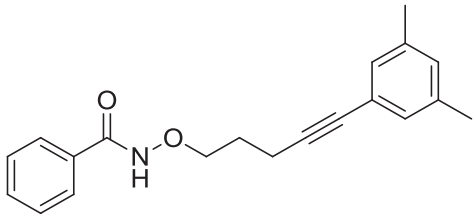
White solid; mp 140−142 °C; R_f = 0.11 (5:1 petroleum ether / ethyl acetate); 1428 mg, 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.10 (brs, 1H), 7.74−7.72 (m, 2H), 7.53−7.50 (m, 1H), 7.43−7.39 (m, 4H), 7.27−7.22 (m, 2H), 4.17 (t, J = 6.0 Hz, 2H), 2.58 (t, J = 7.2 Hz, 2H), 2.03−1.96 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.7, 133.0, 132.1, 131.8, 131.4, 128.7, 127.0, 122.5, 121.8, 90.4, 80.2, 75.4, 27.0. 16.1; ESI-HRMS: Calcd for $C_{18}H_{16}BrNO_2$[M]: 357.0364, found 357.0354.

N-((5-(4-(trifluoromethyl)phenyl)pent-4-yn-1-yl)oxy)benzamide (3k)



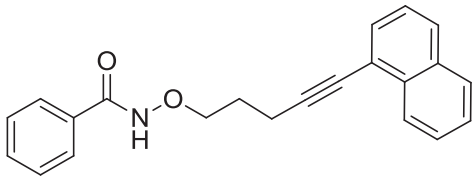
White solid; mp 144−145 °C; R_f = 0.10 (5:1 petroleum ether / ethyl acetate); 1336 mg, 77% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.12 (brs, 1H), 7.74 (d, J = 7.2 Hz, 2H), 7.53−7.46 (m, 5H), 7.43−7.39 (m, 2H), 4.18 (t, J = 6.0 Hz, 2H), 2.62 (t, J = 6.8 Hz, 2H), 2.05−1.98 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.8, 132.1, 131.8, 129.4 (q, $^2J_{C,F}$ = 32.2 Hz), 128.7, 127.5, 127.0, 125.1 (q, $^3J_{C,F}$ = 3.0 Hz), 123.9 (q, $^1J_{C,F}$ = 271.6 Hz), 91.9, 80.0, 75.3, 27.0, 16.1; ^{19}F NMR (376 MHz, $CDCl_3$): δ -62.70; ESI-HRMS: Calcd for $C_{19}H_{16}F_3NO_2$[M]: 347.1133, found 347.1135.

N-((5-(3,5-dimethylphenyl)pent-4-yn-1-yl)oxy)benzamide (3l)



Pale yellow oil; R_f = 0.14 (5:1 petroleum ether / ethyl acetate); 1397 mg, 91% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.31 (brs, 1H), 7.73 (d, J = 7.6 Hz, 2H), 7.51−7.47 (m, 1H), 7.40−7.36 (m, 2H), 7.01 (s, 2H), 6.90 (s, 1H), 4.16 (t, J = 6.4 Hz, 2H), 2.56 (t, J = 6.8 Hz, 2H), 2.25 (s, 6H), 2.01−1.94 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.6, 137.7, 132.0, 131.8, 129.6, 129.2, 128.6, 127.1, 123.1, 88.4, 81.4, 75.4, 27.2, 21.0, 16.0; ESI-HRMS: Calcd for $C_{20}H_{21}NO_2$[M]: 307.1572, found 307.1575.

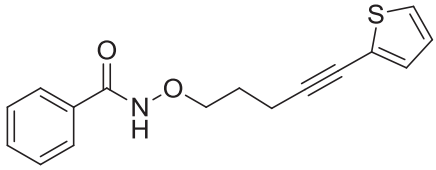
N-((5-(naphthalen-1-yl)pent-4-yn-1-yl)oxy)benzamide (3m)



White solid; mp 84−86 °C; R_f = 0.14 (5:1 petroleum ether / ethyl acetate); 1217 mg, 74% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.41 (brs, 1H), 8.31 (d, J = 8.4 Hz, 1H), 7.81 (d, J = 7.6 Hz, 1H), 7.76 (d, J = 8.0 Hz, 1H), 7.70 (d, J = 8.0 Hz, 2H), 7.60 (d, J = 7.2 Hz, 1H), 7.54−7.42

(m, 3H), 7.38−7.31 (m, 3H), 4.22 (t, *J* = 6.4 Hz, 2H), 2.72 (t, *J* = 7.2 Hz, 2H), 2.10−2.04 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): *δ* 166.6, 133.3, 133.1, 132.0, 131.7, 130.1, 128.5, 128.2, 128.1, 127.0, 126.6, 126.2, 126.1, 125.2, 121.2, 94.2, 79.1, 75.4, 27.4, 16.4; ESI-HRMS: Calcd for $C_{22}H_{19}NO_2$[M]: 329.1416, found 329.1413.

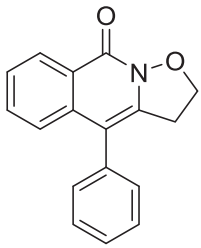
N-((5-(thiophen-2-yl)pent-4-yn-1-yl)oxy)benzamide (3n)



Pale yellow solid; mp 95−97 °C; R_f = 0.12 (5:1 petroleum ether / ethyl acetate); 741 mg, 52% yield; ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 9.48 (brs, 1H), 7.75−7.73 (m, 2H), 7.51−7.47 (m, 1H), 7.40−7.36 (m, 2H), 7.16 (dd, *J* = 5.2 Hz, *J* = 0.8 Hz, 1H), 7.11 (d, *J* = 2.8 Hz, 1H), 6.92 (dd, *J* = 5.2 Hz, *J* = 3.6 Hz, 1H), 4.13 (t, *J* = 6.0 Hz, 2H), 2.58 (t, *J* = 6.8 Hz, 2H), 2.00−1.93 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl3): δ 166.6, 132.0, 131.7, 131.1, 128.6, 127.1, 126.8, 126.1, 123.6, 93.2, 75.2, 74.2, 27.0, 16.3; ESI-HRMS: Calcd for $C_{16}H_{15}NO_2S$[M]: 285.0823, found 285.0829.

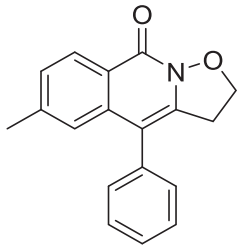
Analytical data for products:

4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2a)



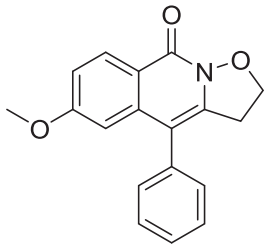
White solid; mp 210−212 °C; R_f = 0.29 (1:2 petroleum ether / ethyl acetate); 102 mg, 97% yield; ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 8.51 (d, *J* = 8.0 Hz, 1H), 7.54−7.43 (m, 5H), 7.37−7.29 (m, 3H), 4.56 (t, *J* = 7.6 Hz, 2H), 3.38 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): *δ* 153.7, 136.0, 134.8, 132.4, 131.5, 130.1, 128.8, 127.9, 127.2, 126.10, 126.06, 124.6, 112.9, 69.4, 32.3; ESI-HRMS: Calcd for $C_{17}H_{13}NO_2$[M]: 263.0946, found 263.0945.

6-methyl-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2b)



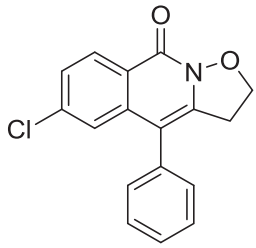
Pale yellow solid; mp 216−218 °C; R_f = 0.30 (1:2 petroleum ether / ethyl acetate); 100 mg, 90% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.41 (d, *J* = 8.4 Hz, 1H), 7.53−7.44 (m, 3H), 7.34 (d, *J* = 6.8 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 1H) 7.13 (s, 1H), 4.56 (t, *J* = 7.2 Hz, 2H), 3.37 (t, *J* = 7.6 Hz, 2H), 2.36 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.8, 142.1, 136.2, 135.0, 132.4, 130.2, 128.8, 127.9, 127.8, 127.2, 124.2, 123.9, 112.7, 69.5, 32.4, 21.8; ESI-HRMS: Calcd for $C_{18}H_{15}NO_2$[M]: 277.1103, found 277.1100.

6-methoxy-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2c)



Pale yellow solid; mp 218−220 °C; R_f = 0.21 (1:2 petroleum ether / ethyl acetate); 82 mg, 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.44 (d, *J* = 8.8 Hz, 1H), 7.52−7.42 (m, 3H), 7.36 (d, *J* = 7.2 Hz, 2H), 7.05 (dd, *J* = 8.8 Hz, *J* = 2.4 Hz, 1H), 6.72 (d, *J* = 2.4 Hz, 1H), 4.56 (t, *J* = 7.6 Hz, 2H), 3.74 (s, 3H), 3.37 (t, *J* = 7.2 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 162.2, 153.7, 138.2, 135.0, 133.1, 130.1, 129.3, 128.9, 128.0, 119.9, 115.0, 112.4, 106.4, 69.4, 55.2, 32.5; ESI-HRMS: Calcd for $C_{18}H_{15}NO_3$[M]: 293.1052, found 293.1059.

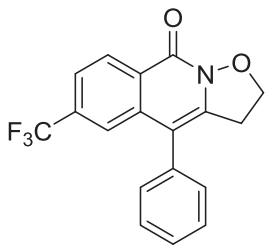
6-chloro-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2d)



Pale yellow solid; mp 248−250 °C; R_f = 0.32 (1:2 petroleum ether / ethyl acetate); 94 mg, 79% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (d, *J* = 8.4 Hz, 1H), 7.55−7.46 (m, 3H),

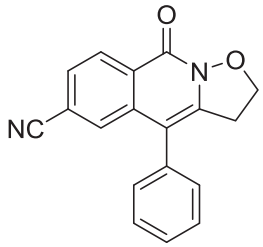
7.43−7.39 (m, 1H), 7.36−7.31 (m, 3H), 4.60 (t, J = 7.6 Hz, 2H), 3.41 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.3, 138.3, 137.4, 134.1, 134.0, 130.1, 129.1, 129.0, 128.3, 126.7, 124.4, 124.0, 112.1, 69.6, 32.5; ESI-HRMS: Calcd for $C_{17}H_{12}ClNO_2$[M]: 297.0557, found 297.0565.

4-phenyl-6-(trifluoromethyl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2e)



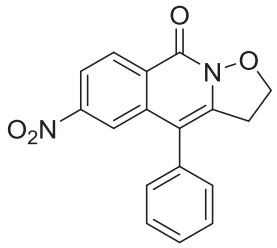
Pale yellow solid; mp 228−230 °C; R_f = 0.35 (1:2 petroleum ether / ethyl acetate); 118 mg, 89% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.62 (d, J = 8.4 Hz, 1H), 7.67−7.64 (m, 2H), 7.57−7.48 (m, 3H), 7.38−7.36 (m, 2H), 4.63 (t, J = 7.6 Hz, 2H), 3.46 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.0, 136.0, 134.2, 133.8, 133.2 (q, $^2J_{C,F}$ = 32.2 Hz), 130.0, 129.2, 128.5, 128.4, 128.1, 123.6 (q, $^1J_{C,F}$ = 272.6 Hz), 122.2 (q, $^3J_{C,F}$ = 3.0 Hz), 121.9 (q, $^3J_{C,F}$ = 4.0 Hz), 112.9, 69.7, 32.4; ^{19}F NMR (376 MHz, $CDCl_3$): δ -62.82; ESI-HRMS: Calcd for $C_{18}H_{12}F_3NO_2$[M]: 331.0820, found 331.0813.

9-oxo-4-phenyl-3,9-dihydro-2H-isoxazolo[2,3-b]isoquinoline-6-carbonitrile (2f)



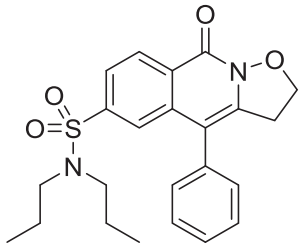
Pale yellow solid; mp 214−216 °C; R_f = 0.30 (1:2 petroleum ether / ethyl acetate); 79 mg, 69% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.59 (d, J = 8.4 Hz, 1H), 7.69−7.64 (m, 2H), 7.58−7.50 (m, 3H), 7.35−7.33 (m, 2H), 4.64 (t, J = 7.6 Hz, 2H), 3.46 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 152.7, 136.1, 134.8, 133.4, 130.0, 129.7, 129.3, 128.7, 128.5, 128.4, 127.7, 118.1, 115.2, 112.2, 69.8, 32.4; ESI-HRMS: Calcd for $C_{18}H_{12}N_2O_2$[M]: 288.0899, found 288.0891.

6-nitro-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2g)



Yellow solid; mp 303−305 °C; R_f = 0.28 (1:2 petroleum ether / ethyl acetate); 71 mg, 58% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.70 (d, J = 8.4 Hz, 1H), 8.24−8.22 (m, 2H), 7.59−7.50 (m, 3H), 7.37−7.35 (m, 2H), 4.65 (t, J = 8.0 Hz, 2H), 3.47 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 152.6, 150.0, 136.8, 135.3, 133.5, 130.1, 129.8, 129.6, 129.4, 128.9, 120.4, 119.9, 113.1, 69.9, 32.6; ESI-HRMS: Calcd for $C_{17}H_{12}N_2O_4$[M]: 308.0797, found 308.0787.

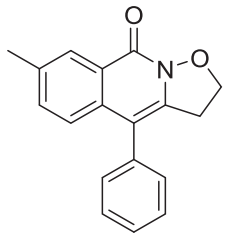
9-oxo-4-phenyl-N,N-dipropyl-3,9-dihydro-2H-isoxazolo[2,3-b]isoquinoline-6-sulfonamide (2h)



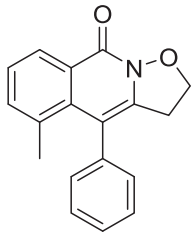
Pale yellow solid; mp 160−162 °C; R_f = 0.30 (1:2 petroleum ether / ethyl acetate); 90 mg, 53% yield; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.64 (d, J = 9.0 Hz, 1H), 7.84−7.82 (m, 2H), 7.53−7.48 (m, 3H), 7.35−7.32 (m, 2H), 4.63 (t, J = 7.8 Hz, 2H), 3.45 (t, J = 7.8 Hz, 2H), 3.04 (t, J = 7.5 Hz, 4H), 1.54−1.42 (m, 4H), 0.82 (t, J = 7.5 Hz, 6H); ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 152.9, 143.2, 136.3, 134.4, 133.7, 129.9, 129.2, 128.7, 128.6, 128.1, 123.7, 123.4, 113.0, 69.8, 49.6, 32.4, 21.6, 11.0; ESI-HRMS: Calcd for $C_{23}H_{26}N_2O_4S$[M]: 426.1613, found 426.1610.

7-methyl-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2i)

5-methyl-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2i')



2i, 28 mg, 25% yield

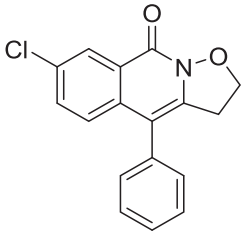


2i', 71 mg, 64% yield

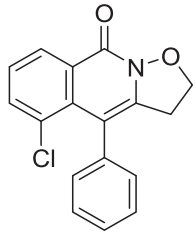
White solid; R_f = 0.31 (1:2 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.46 (dd, J = 7.2 Hz, J = 2.0 Hz, 1H), 8.32 (s, 1H), 7.51−7.48 (m, 2H), 7.46−7.40 (m, 4H), 7.37−7.29 (m, 8H), 4.56 (t, J = 7.6 Hz, 2H), 4.52 (t, J = 7.6 Hz, 2H), 3.38 (t, J = 7.6 Hz, 2H), 3.22 (t, J = 7.6 Hz, 2H), 2.47 (s, 3H), 1.85 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.9, 153.7, 139.0, 136.3, 135.4, 134.9, 134.6, 134.1, 133.7, 133.3, 133.0, 131.4, 130.1, 130.0, 128.7, 128.4, 127.8, 127.7, 127.2, 126.7, 125.92, 125.88, 124.5, 112.92, 112.89, 69.5, 69.3, 32.9, 32.1, 23.8, 21.1; ESI-HRMS: Calcd for $C_{18}H_{15}NO_2$[M]: 277.1103, found 277.1095.

7-chloro-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2j)

5-chloro-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2j')



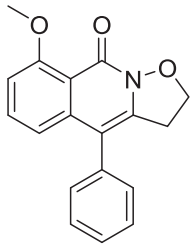
2j, 30 mg, 25% yield



2j', 72 mg, 61% yield

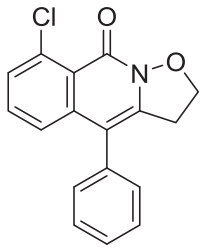
White solid; R_f = 0.40 (1:2 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (dd, J = 8.4 Hz, J = 1.2 Hz, 1H), 8.45 (d, J = 2.0 Hz, 1H), 7.56−7.48 (m, 2H), 7.46−7.43 (m, 2H), 7.41−7.39 (m, 4H), 7.36−7.34 (m, 2H), 7.32−7.28 (m, 4H), 4.62−4.54 (m, 4H), 3.42 (t, J = 7.6 Hz, 2H), 3.26 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 152.9, 152.6, 137.4, 135.1, 134.9, 134.3, 134.2, 132.8, 132.29, 132.26, 131.9, 130.3, 130.02, 130.01, 128.9, 128.4, 128.14, 128.11, 127.6, 127.0, 126.7, 126.4, 126.3, 126.2, 112.5, 111.4, 69.7, 69.5, 33.0, 32.2; ESI-HRMS: Calcd for $C_{17}H_{12}ClNO_2$[M]: 297.0557, found 297.0550.

8-methoxy-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2k)



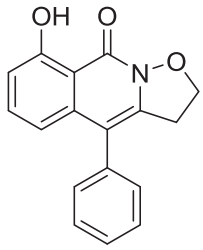
Pale yellow solid; mp 213−215 °C; R_f = 0.13 (ethyl acetate); 48 mg, 41% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51−7.47 (m, 2H), 7.45−7.40 (m, 2H), 7.33−7.28 (m, 2H), 6.90−6.86 (m, 2H), 4.53 (t, *J* = 8.0 Hz, 2H), 4.02 (s, 3H), 3.34 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 161.1, 153.0, 139.4, 135.5, 133.2, 132.1, 130.3, 128.8, 127.9, 117.0, 115.7, 111.9, 107.7, 69.0 56.4, 32.6; ESI-HRMS: Calcd for $C_{18}H_{15}NO_3$[M]: 293.1052, found 293.1043.

8-chloro-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2l)



White solid; mp 247−249 °C; R_f = 0.35 (1:2 petroleum ether / ethyl acetate); 42 mg, 35% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53−7.44 (m, 4H), 7.37−7.31 (m, 3H), 7.23 (dd, *J* = 8.4 Hz, *J* = 1.2 Hz, 1H), 4.56 (t, *J* = 7.6 Hz, 2H), 3.36 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 152.5, 139.3, 135.4, 134.9, 133.7, 131.1, 130.3, 129.5, 129.0, 128.2, 123.9, 122.5, 111.9, 69.2, 32.6; ESI-HRMS: Calcd for $C_{17}H_{12}ClNO_2$[M]: 297.0557, found 297.0551.

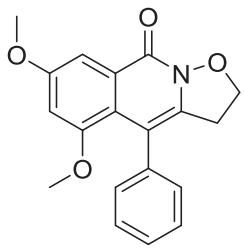
8-hydroxy-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2m)



White solid; mp 220−222 °C; R_f = 0.41 (1:1 petroleum ether / ethyl acetate); 71 mg, 64% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.53 (brs, 1H), 7.52−7.42 (m, 3H), 7.41−7.37 (m, 1H),

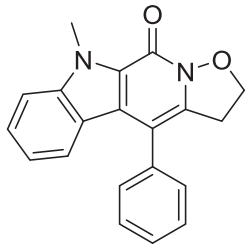
7.34−7.32 (m, 2H), 6.86 (d, *J* = 7.6 Hz, 1H), 6.75 (d, *J* = 8.0 Hz, 1H), 4.58 (t, *J* = 7.6 Hz, 2H), 3.36 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 161.3, 156.7, 137.4, 134.7, 133.6, 131.9, 130.0, 128.9, 128.2, 114.8, 112.7, 112.0, 69.9, 32.1; ESI-HRMS: Calcd for $C_{17}H_{13}NO_3$[M]: 279.0895, found 279.0898.

5,7-dimethoxy-4-phenyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2n)



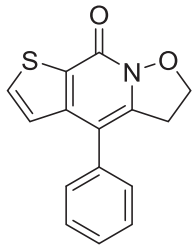
Pale yellow solid; mp 145−147 °C; R_f = 0.22 (1:2 petroleum ether / ethyl acetate); 125 mg, 97% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (d, *J* = 2.4 Hz, 1H), 7.38−7.29 (m, 3H), 7.23−7.22 (m, 2H), 6.58 (d, *J* = 2.4 Hz, 1H), 4.53 (t, *J* = 7.6 Hz, 2H), 3.93 (s, 3H), 3.36 (s, 3H), 3.23 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 158.8, 157.0, 152.9, 139.3, 130.2, 129.0, 128.8, 127.4, 126.5, 120.9, 111.5, 103.8, 99.2, 69.6, 55.7, 55.3, 32.4; ESI-HRMS: Calcd for $C_{19}H_{17}NO_4$[M]: 323.1158, found 323.1152.

9-methyl-4-phenyl-3,9-dihydroisoxazolo[2',3':1,6]pyrido[3,4-b]indol-10(2H)-one (2o)



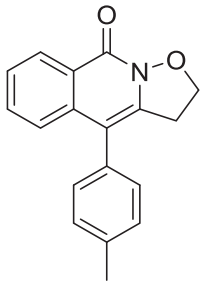
Pale yellow solid; mp 205−207 °C; R_f = 0.21 (1:2 petroleum ether / ethyl acetate); 102 mg, 81% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54−7.44 (m, 5H), 7.41−7.36 (m, 2H), 7.08 (d, *J* = 8.0 Hz, 1H), 6.97−6.93 (m, 1H), 4.62 (t, *J* = 7.6 Hz, 2H), 4.32 (s, 3H), 3.39 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 148.5, 141.2, 135.6, 129.7, 128.7, 128.1, 128.0, 126.7, 126.1, 122.8, 122.4, 120.9, 119.5, 110.6, 109.7, 70.5, 31.6, 31.2; ESI-HRMS: Calcd for $C_{20}H_{16}N_2O_2$[M]: 316.1212, found 316.1208.

4-phenyl-5,6-dihydro-9H-isoxazolo[2,3-a]thieno[3,2-d]pyridin-9-one (2p)



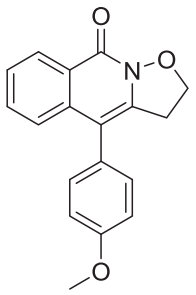
Pale yellow solid; mp 235−237 °C; R_f = 0.24 (1:2 petroleum ether / ethyl acetate); 80 mg, 74% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, J = 5.2 Hz, 1H), 7.51−7.48 (m, 2H), 7.44−7.39 (m, 3H), 7.11 (d, J = 5.6 Hz, 1H), 4.65 (t, J = 7.6 Hz, 2H), 3.52 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 150.5, 144.3, 135.2, 133.8, 132.8, 129.3, 129.2, 128.8, 128.0, 123.6, 111.6, 70.6, 32.1; ESI-HRMS: Calcd for $C_{15}H_{11}NO_2S$[M]: 269.0510, found 269.0501.

4-(p-tolyl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2q)



Pale yellow solid; mp 230−232 °C; R_f = 0.32 (1:2 petroleum ether / ethyl acetate); 104 mg, 94% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52 (d, J = 8.0 Hz, 1H), 7.55−7.52 (m, 1H), 7.48−7.44 (m, 1H), 7.39 (d, J = 8.0 Hz, 1H), 7.31 (d, J = 7.6 Hz, 2H), 7.23 (d, J = 8.0 Hz, 2H), 4.57 (t, J = 7.6 Hz, 2H), 3.40 (t, J = 7.6 Hz, 2H), 2.45 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.8, 137.8, 136.2, 132.3, 131.7, 131.5, 130.0, 129.5, 127.2, 126.11, 126.06, 124.7, 112.9, 69.5, 32.3, 21.2; ESI-HRMS: Calcd for $C_{18}H_{15}NO_2$[M]: 277.1103, found 277.1107.

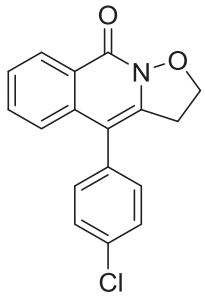
4-(4-methoxyphenyl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2r)



Pale yellow solid; mp 241−243 °C; R_f = 0.24 (1:2 petroleum ether / ethyl acetate); 86 mg,

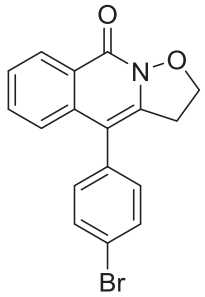
73% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52 (dd, J = 8.0 Hz, J = 1.2 Hz, 1H), 7.57−7.52 (m, 1H), 7.49−7.45 (m, 1H), 7.39 (d, J = 8.0 Hz, 1H), 7.28−7.25 (m, 2H), 7.05−7.01 (m, 2H), 4.58 (t, J = 7.6 Hz, 2H), 3.89 (s, 3H), 3.40 (t, J = 7,6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 159.2, 153.8, 136.4, 132.4, 131.5, 131.3, 127.3, 126.9, 126.12, 126.06, 124.7, 114.2, 112.6, 69.5, 55.3, 32.3; ESI-HRMS: Calcd for $C_{18}H_{15}NO_3$[M]: 293.1052, found 293.1053.

4-(4-chlorophenyl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2s)



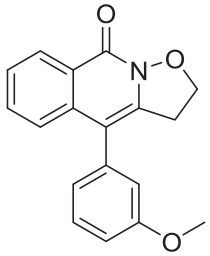
Pale yellow solid; mp 245−247 °C; R_f = 0.26 (1:2 petroleum ether / ethyl acetate); 106 mg, 89% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.50 (dd, J = 8.0 Hz, J = 1.2 Hz, 1H), 7.57−7.53 (m, 1H), 7.50−7.45 (m, 3H), 7.33−7.29 (m, 3H), 4.58 (t, J = 7.6 Hz, 2H), 3.39 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.7, 135.7, 134.0, 133.3, 132.6, 131.7, 131.6, 129.1, 127.4, 126.3, 126.0, 124.3, 111.6, 69.5, 32.3; ESI-HRMS: Calcd for $C_{17}H_{12}ClNO_2$[M]: 297.0557, found 297.0562.

4-(4-bromophenyl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2t)



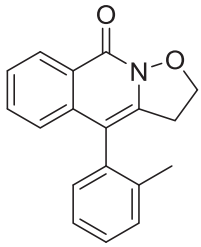
White solid; mp 247−249 °C; R_f = 0.29 (1:2 petroleum ether / ethyl acetate); 131 mg, 96% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (d, J = 7.6 Hz, 1H), 7.64 (d, J = 8.4 Hz, 2H), 7.56−7.52 (m, 1H), 7.48−7.44 (m, 1H), 7.32 (d, J = 8.0 Hz, 1H), 7.25 (d, J = 8.4 Hz, 2H), 4.58 (t, J = 7.6 Hz, 2H), 3.40 (t, J = 7.6 Hz, 2H); ^{13}CNMR (100.6 MHz, $CDCl_3$): δ 153.7, 135.6, 133.7, 132.6, 132.0, 131.9, 131.7, 127.3, 126.3, 126.0, 124.3, 122.2, 111.6, 69.5, 32.3; ESI-HRMS: Calcd for $C_{17}H_{12}BrNO_2$[M]: 341.0051, found 341.0040.

4-(3-methoxyphenyl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2u)



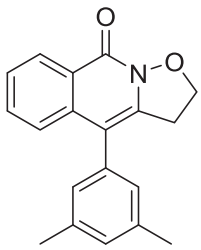
Pale yellow solid; mp 206−208 °C; R_f = 0.27 (1:2 petroleum ether / ethyl acetate); 104 mg, 89% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52 (d, *J* = 7.2 Hz, 1H), 7.57−7.53 (m, 1H), 7.49−7.40 (m, 3H), 6.99 (dd, *J* = 8.4 Hz, *J* = 2.4 Hz, 1H), 6.94−6.90 (m, 2H), 4.58 (t, *J* = 7.6 Hz, 2H), 3.86 (s, 3H), 3.42 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 159.7, 153.8, 136.1, 136.0, 132.4, 131.6, 129.9, 127.2, 126.2, 126.0, 124.7, 122.5, 115.9, 113.2, 112.8, 69.5, 55.2, 32.3; ESI-HRMS: Calcd for $C_{18}H_{15}NO_3$[M]: 293.1052, found 293.1047.

4-(o-tolyl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2v)



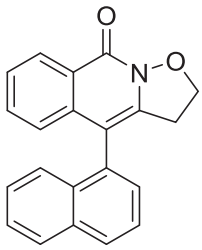
Pale yellow solid; mp 140−142 °C; R_f = 0.40 (1:2 petroleum ether / ethyl acetate); 104 mg, 94% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (dd, *J* = 8.0 Hz, *J* = 1.2 Hz, 1H), 7.54−7.45 (m, 2H), 7.41−7.35 (m, 2H), 7.33−7.29 (m, 1H), 7.21 (d, *J* = 7.6 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 1H), 4.59 (t, *J* = 7.6 Hz, 2H), 3.38−3.31 (m, 1H), 3.22−3.14 (m, 1H), 2.07 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.9, 137.5, 136.0, 133.9, 132.3, 131.6, 130.5, 130.4, 128.5, 127.3, 126.4, 126.2, 126.0, 124.4, 112.2, 69.4, 32.0, 19.6; ESI-HRMS: Calcd for $C_{18}H_{15}NO_2$[M]: 277.1103, found 277.1107.

4-(3,5-dimethylphenyl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2w)



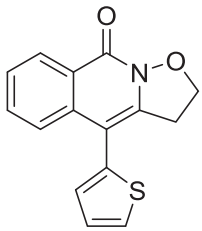
Pale yellow solid; mp 235−237 °C; R_f = 0.39 (1:2 petroleum ether / ethyl acetate); 100 mg, 86% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52 (dd, J = 8.0 Hz, J = 0.8 Hz, 1H), 7.56−7.52 (m, 1H), 7.48−7.44 (m, 1H), 7.39 (d, J = 8.4 Hz, 1H), 7.08 (s, 1H), 6.95 (s, 2H), 4.57 (t, J = 8.0 Hz, 2H), 3.41 (t, J = 7.6 Hz, 2H), 2.38 (s, 6H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.8, 138.3, 136.2, 134.6, 132.2, 131.4, 129.6, 127.8, 127.2, 126.1, 126.0, 124.8, 113.2, 69.5, 32.3, 21.3; ESI-HRMS: Calcd for $C_{19}H_{17}NO_2$[M]: 291.1259, found 291.1260.

4-(naphthalen-1-yl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2x)



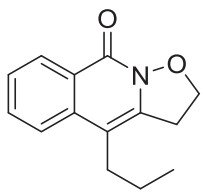
Pale yellow solid; mp 138−140 °C; R_f = 0.32 (1:2 petroleum ether / ethyl acetate); 120 mg, 96% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.58−8.56 (m, 1H), 8.00−7.96 (m, 2H), 7.62−7.58 (m, 1H), 7.54−7.37 (m, 6H), 7.01 (d, J = 7.6 Hz, 1H), 4.62−4.52 (m, 2H), 3.35−3.27 (m, 1H), 3.14−3.06 (m, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 154.0, 136.7, 133.8, 133.4, 132.2, 132.1, 131.6, 128.9, 128.6, 128.5, 127.2, 126.7, 126.25, 126.20, 126.0, 125.6, 125.1, 125.0, 110.8, 69.5, 32.1; ESI-HRMS: Calcd for $C_{21}H_{15}NO_2$[M]: 313.1103, found 313.1104.

4-(thiophen-2-yl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2y)



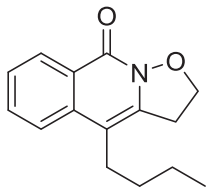
Pale yellow solid; mp 204−206 °C; R_f = 0.36 (1:2 petroleum ether / ethyl acetate); 100 mg, 93% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.50 (d, J = 8.0 Hz, 1H), 7.62−7.55 (m, 2H), 7.51−7.47 (m, 2H), 7.19 (dd, J = 5.2 Hz, J = 3.6 Hz, 1H), 7.10 (dd, J = 3.6 Hz, J = 1.2 Hz, 1H), 4.60 (t, J = 7.6 Hz, 2H), 3.51 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.9, 136.5, 135.0, 134.4, 131.8, 128.9, 127.4, 127.2, 126.8, 126.4, 125.8, 124.5, 105.4, 69.4, 32.8; ESI-HRMS: Calcd for $C_{15}H_{11}NO_2S$[M]: 269.0510, found 269.0503.

4-propyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2z)



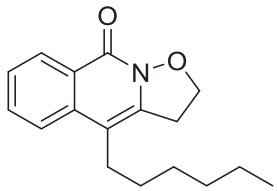
White solid; mp 104−106 °C; R_f = 0.16 (1:2 petroleum ether / ethyl acetate); 62 mg, 68% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.50 (d, J = 8.0 Hz, 1H), 7.66−7.65 (m, 2H), 7.50−7.44 (m, 1H), 4.59 (t, J = 7.6 Hz, 2H), 3.53 (t, J = 7.6 Hz, 2H), 2.69 (t, J = 7.2 Hz, 2H), 1.70−1.61 (m, 2H), 1.00 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.6, 135.6, 131.6, 131.4, 127.6, 126.6, 125.9, 122.8, 110.2, 69.1, 31.6, 30.1, 22.8, 14.0; ESI-HRMS: Calcd for $C_{14}H_{15}NO_2$[M]: 229.1103, found 229.1098.

4-butyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2aa)



White solid; mp 74−76 °C; R_f = 0.27 (1:2 petroleum ether / ethyl acetate); 58 mg, 60% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.51 (d, J = 8.0 Hz, 1H), 7.67−7.66 (m, 2H), 7.51−7.45 (m, 1H), 4.60 (t, J = 7.6 Hz, 2H), 3.53 (t, J = 7.6 Hz, 2H), 2.71 (t, J = 7.6 Hz, 2H), 1.63−1.56 (m, 2H), 1.47−1.38 (m, 2H), 0.96 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.7, 135.6, 131.5, 131.4, 127.7, 126.6, 125.9, 122.8, 110.6, 69.2, 31.8, 31.5, 28.0, 22.7, 13.9; ESI-HRMS: Calcd for $C_{15}H_{17}NO_2$[M]: 243.1259, found 243.1262.

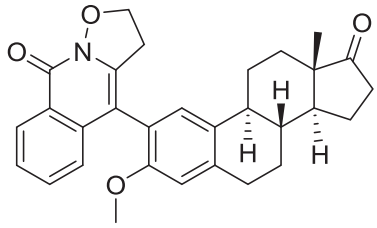
4-hexyl-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2ab)



White solid; mp 80−82 °C; R_f = 0.30 (1:2 petroleum ether / ethyl acetate); 76 mg, 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.51 (d, J = 8.0 Hz, 1H), 7.69−7.66 (m, 2H), 7.50−7.46 (m, 1H), 4.60 (t, J = 7.2 Hz, 2H), 3.52 (t, J = 7.2 Hz, 2H), 2.70 (t, J = 7.6 Hz, 2H), 1.64−1.56 (m, 2H), 1.43−1.36 (m, 2H), 1.32−1.31 (m, 4H), 0.89 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.6, 135.5, 131.45, 131.38, 127.6, 126.5, 125.9, 122.7, 110.6, 69.2, 31.6, 31.5, 29.6, 29.3, 28.2, 22.6, 14.0; ESI-HRMS: Calcd for $C_{17}H_{21}NO_2$[M]: 271.1572, found

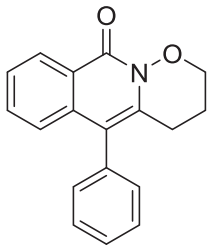
271.1570.

4-((8R,9S,13S,14S)-3-methoxy-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-2-yl)-2,3-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (2ae)



White solid; dr = 1:1 (a nonseparable mixture of two diastereomers); R_f = 0.12 (1:2 petroleum ether / ethyl acetate); 171 mg, 91% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.51−8.49 (m, 2H), 7.55−7.49 (m, 2H), 7.45−7.41 (m, 2H), 7.31−7.23 (m, 2H), 7.15 (s, 2H), 6.78 (s, 2H), 4.61−4.54 (m, 4H), 3.71 (s, 6H), 3.41−3.28 (m, 4H), 3.03−3.01 (m, 4H), 2.55−2.48 (m, 2H), 2.34−2.28 (m, 4H), 2.20−2.08 (m, 6H), 1.96−1.90 (m, 2H), 1.72−1.62 (m, 4H), 1.58−1.41 (m, 8H), 0.94 (s, 3H), 0.93 (s, 3H); ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 220.56, 220.55, 155.4, 153.9, 138.1, 136.5, 136.4, 133.1, 132.1, 132.0, 131.25, 131.23, 129.1, 129.0, 127.14, 127.12, 126.05, 126.04, 125.8, 124.71, 124.69, 120.5, 111.61, 111.58, 109.38, 109.36, 69.4, 55.50, 55.47, 50.3, 50.2, 47.9, 47.8, 43.9, 43.8, 38.2, 35.7, 32.33, 32.26, 31.4, 29.73, 29.70, 26.43, 26.40, 26.0, 25.9, 21.5, 13.80, 13.78; ESI-HRMS: Calcd for $C_{30}H_{31}NO_4$[M]: 469.2253, found 469.2252.

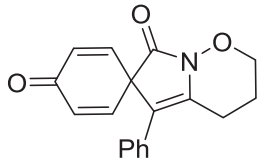
5-phenyl-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4a)



White solid; mp 213−215 ^{o}C; R_f = 0.23 (1:1 petroleum ether / ethyl acetate); 66 mg, 60% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (d, J = 7.6 Hz, 1H), 7.54−7.43 (m, 5H), 7.30−7.28 (m, 2H), 7.18 (d, J = 8.0 Hz, 1H), 4.44 (t, J = 8.0 Hz, 2H), 2.71 (t, J = 6.8 Hz, 2H), 2.11−2.04 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 157.3, 138.4, 136.0, 134.9, 131.9, 131.0, 128.8, 127.9, 127.7, 125.9, 125.3, 125.0, 114.7, 70.8, 21.7, 21.3; ESI-HRMS: Calcd for

$C_{18}H_{15}NO_2$[M]: 277.1103, found 277.1107.

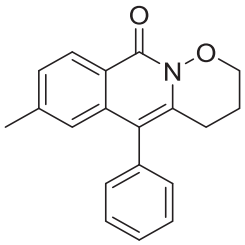
5'-phenyl-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4a')



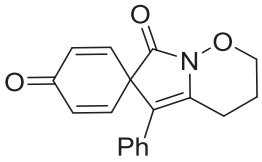
Pale yellow oil; R_f = 0.18 (1:1 petroleum ether / ethyl acetate); 14 mg, 12% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32−7.25 (m, 3H), 7.20−7.18 (m, 2H), 6.71 (d, J = 10.4 Hz, 2H), 6.57 (d, J = 10.0 Hz, 2H), 4.34 (t, J = 5.2 Hz, 2H), 3.02 (t, J = 6.8 Hz, 2H), 2.08−2.02 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 185.0, 166.0, 143.7, 136.0, 132.9, 131.6, 128.8, 127.8, 127.3, 114.1, 70.9, 57.6, 21.2, 20.9; ESI-HRMS: Calcd for $C_{18}H_{15}NO_3$[M]: 293.1052, found 293.1056.

7-methyl-5-phenyl-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4b)

5'-phenyl-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4a')



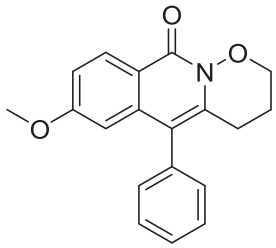
4b, 62 mg, 53% yield



4a', 2 mg, 2% yield

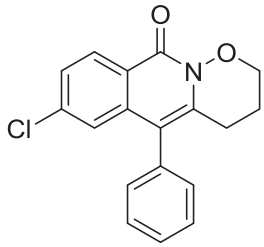
White solid; R_f = 0.22 (1:1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.41 (d, J = 8.4 Hz, 1H), 7.52−7.41 (m, 5H), 7.29−7.24 (m, 3H), 7.22−7.19 (m, 3H), 6.93 (s, 1H), 6.69 (d, J = 10 Hz, 2H), 6.54 (d, J = 10 Hz, 2H), 4.42 (t, J = 7.6 Hz, 2H), 4.34 (t, J = 5.6 Hz, 2H), 3.01 (t, J = 6.8 Hz, 2H), 2.68 (t, J = 6.8 Hz, 2H), 2.33 (s, 3H), 2.09−2.02 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 184.8, 165.7, 157.2, 143.7, 142.4, 138.3, 136.1, 135.9, 135.1, 132.7, 131.5, 131.0, 128.7, 128.6, 127.7, 127.6, 127.5, 127.1, 124.6, 123.1, 114.4, 113.8, 70.8, 70.7, 57.4, 21.8, 21.7, 21.3, 21.0, 20.7; ESI-HRMS: Calcd for $C_{19}H_{17}NO_2$[M] (**4b**): 291.1259, found 291.1258; $C_{18}H_{15}NO_3$[M] (**4a'**): 293.1052, found 293.1050.

7-methoxy-5-phenyl-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4c)



Pale yellow solid; mp 166−168 °C; R_f = 0.13 (1:1 petroleum ether / ethyl acetate); 70 mg, 57% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.44 (d, *J* = 8.8 Hz, 1H), 7.51−7.42 (m, 3H), 7.29 (d, *J* = 6.4 Hz, 2H), 7.02 (dd, *J* = 8.8 Hz, *J* = 2.0 Hz, 1H), 6.52 (d, *J* = 2.0 Hz, 1H), 4.42 (t, *J* = 7.6 Hz, 2H), 3.70 (s, 3H), 2.68 (t, *J* = 6.8 Hz, 2H), 2.09−2.02 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 162.4, 157.1, 139.0, 138.1, 135.1, 131.0, 129.8, 128.8, 127.9, 119.2, 114.7, 114.2, 106.8, 70.7, 55.2, 21.7, 21.4; ESI-HRMS: Calcd for $C_{19}H_{17}NO_3$[M]: 307.1208, found 307.1207.

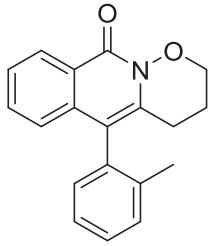
7-chloro-5-phenyl-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4d)



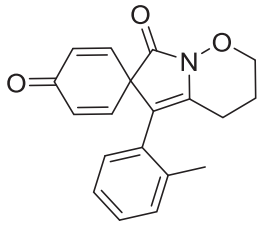
Pale yellow solid; mp 185−187 °C; R_f = 0.21 (1:1 petroleum ether / ethyl acetate); 53 mg, 43% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.44 (d, *J* = 8.4 Hz, 1H), 7.54−7.45 (m, 3H), 7.38−7.36 (m, 1H), 7.28−7.26 (m, 2H), 7.12 (d, *J* = 1.6 Hz, 1H), 4.44 (t, *J* = 7.6 Hz, 2H), 2.70 (t, *J* = 6.8 Hz, 2H), 2.11−2.04 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 156.7, 139.9, 138.6, 137.3, 134.2, 130.9, 129.5, 129.0, 128.2, 126.4, 124.3, 123.7, 113.8, 70.8, 21.6, 21.4; ESI-HRMS: Calcd for $C_{18}H_{14}ClNO_2$[M]: 311.0713, found 311.0714.

5-(o-tolyl)-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4e)

5'-(o-tolyl)-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4e')



4e, 79 mg, 68% yield

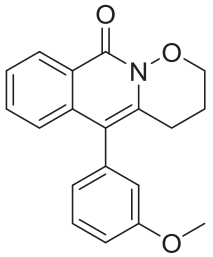


4e', 10 mg, 8% yield

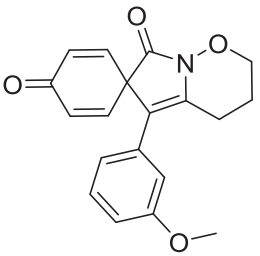
White solid; R_f = 0.32 (1:1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (dd, J = 7.6, J = 1.2 Hz, 1H), 7.52−7.47 (m, 1H), 7.45−7.41 (m, 1H), 7.39−7.35 (m, 2H), 7.32−7.28 (m, 2H), 7.20−7.19 (m, 2H), 7.15 (d, J = 7.2 Hz, 1H), 7.10−7.06 (m, 1H), 6.96 (d, J = 7.6 Hz, 1H), 6.74 (d, J = 9.6 Hz, 2H), 6.42 (d, J = 10.4 Hz, 2H), 4.50−4.39 (m, 2H), 4.33−4.30 (m, 2H), 2.67−2.53 (m, 4H), 2.28 (s, 3H), 2.11−2.04 (m, 5H), 2.01−1.96 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 184.7, 168.0, 157.2, 142.0, 138.0, 137.9, 137.0, 136.6, 135.6, 134.1, 132.5, 131.9, 131.1, 130.34, 130.29, 130.2, 129.0, 128.6, 128.2, 127.6, 126.2, 125.8, 125.5, 125.4, 124.6, 113.4, 113.2, 70.72, 70.66, 58.6, 21.5, 21.0, 20.6, 20.4, 20.0, 19.5; ESI-HRMS: Calcd for $C_{19}H_{17}NO_2$[M] (**4e**): 291.1259, found 291.1260; $C_{19}H_{17}NO_3$[M] (**4e'**): 307.1208, found 307.1214.

5-(3-methoxyphenyl)-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4f)

5'-(3-methoxyphenyl)-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4f')



4f, 71 mg, 58% yield

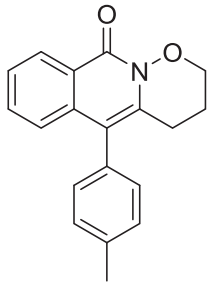


4f', 14 mg, 11% yield

White solid; R_f = 0.19 (1:1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.51 (d, J = 7.6 Hz, 1H), 7.53−7.49 (m, 1H), 7.44−7.39 (m, 2H), 7.22−7.18 (m, 2H), 6.99 (dd, J = 8.0 Hz, J = 2.0 Hz, 1H), 6.88−6.84 (m, 2H), 6.80−6.77 (m, 2H), 6.74 (s, 1H), 6.70 (d, J = 10.0 Hz, 2H), 6.54 (d, J = 10.0 Hz, 2H), 4.43 (t, J = 7.6 Hz, 2H), 4.33 (t, J = 5.6 Hz, 2H), 3.84 (s, 3H), 3.73 (s, 3H), 3.02 (t, J = 6.8 Hz, 2H), 2.72 (t, J = 6.8 Hz, 2H), 2.11−2.02 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 184.8, 165.5, 159.7, 159.4,

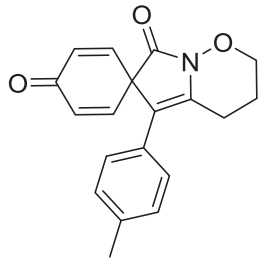
157.1, 143.8, 138.2, 136.2, 136.1, 135.9, 129.7, 129.6, 127.5, 125.7, 125.2, 124.9, 123.2, 119.4, 116.6, 114.3, 113.5, 113.1, 113.0, 112.6, 70.7, 57.3, 55.1, 55.0, 21.6, 21.2, 21.1, 20.6; ESI-HRMS: Calcd for $C_{19}H_{17}NO_3$[M] (**4f**): 307.1208, found 307.1215; $C_{19}H_{17}NO_4$[M] (**4f'**): 323.1158, found 323.1166.

5-(p-tolyl)-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4g)



White solid; mp 176−178 °C; R_f = 0.23 (1:1 petroleum ether / ethyl acetate); 72 mg, 62% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52 (d, J = 8.0 Hz, 1H), 7.52−7.49 (m, 1H), 7.44−7.41 (m, 1H), 7.30 (d, J = 8.0 Hz, 2H), 7.21−7.16 (m, 3H), 4.43 (t, J = 7.6 Hz, 2H), 2.71 (t, J = 6.8 Hz, 2H), 2.45 (s, 3H), 2.10−2.02 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 157.2, 138.3, 137.6, 136.1, 131.8, 131.7, 130.8, 129.4, 127.6, 125.8, 125.3, 125.0, 114.5, 70.8, 21.6, 21.25, 21.18; ESI-HRMS: Calcd for $C_{19}H_{17}NO_2$[M]: 291.1259, found 291.1264.

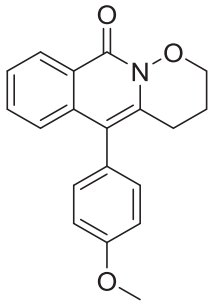
5'-(p-tolyl)-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4g')



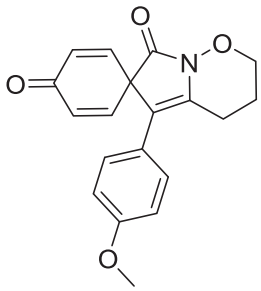
Pale yellow oil; R_f = 0.23 (1:1 petroleum ether / ethyl acetate); 14 mg, 11% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.11−7.06 (m, 4H), 6.69 (d, J = 10.0 Hz, 2H), 6.54 (d, J = 10.0 Hz, 2H), 4.32 (t, J = 5.2 Hz, 2H), 2.98 (t, J = 6.8 Hz, 2H), 2.31 (s, 3H), 2.06−2.00 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 185.0, 166.0, 143.9 ,137.8, 135.4, 132.8, 129.4, 128.6, 127.2, 114.2, 70.8, 57.6, 21.2, 21.1, 20.9; ESI-HRMS: Calcd for $C_{19}H_{17}NO_3$[M]: 307.1208, found 307.1212.

5-(4-methoxyphenyl)-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4h)

5'-(4-methoxyphenyl)-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4h')



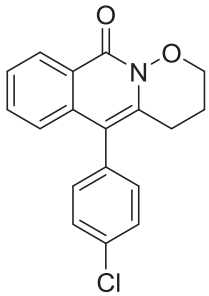
4h, 55 mg, 45% yield



4h', 8 mg, 6% yield

White solid; R_f = 0.17 (1:1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.51 (dd, *J* = 8.0 Hz, *J* = 0.8 Hz, 1H), 7.53−7.49 (m, 1H), 7.44−7.40 (m, 1H), 7.22−7.19 (m, 3H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.03 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.8 Hz, 2H), 6.69 (d, *J* = 10.0 Hz, 2H), 6.53 (d, *J* = 10.0 Hz, 2H), 4.42 (t, *J* = 7.6 Hz, 2H), 4.32 (t, *J* = 5.6 Hz, 2H), 3.88 (s, 3H), 3.77 (s, 3H), 2.97 (t, *J* = 6.4 Hz, 2H), 2.72 (t, *J* = 7.2 Hz, 2H), 2.10−2.00 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 184.8, 165.8, 159.1, 158.9, 157.1, 138.4, 136.3, 134.6, 132.0, 131.9, 131.8, 131.7, 128.4, 128.3, 127.5, 126.8, 125.7 ,125.3, 124.9, 123.7, 114.13, 114.06, 113.6, 70.7, 57.5, 55.2, 55.1, 21.6, 21.2, 21.0, 20.8; ESI-HRMS: Calcd for $C_{19}H_{17}NO_3$[M] (**4h**): 307.1208, found 307.1211; $C_{19}H_{17}NO_4$[M] (**4h'**): 323.1158, found 323.1163.

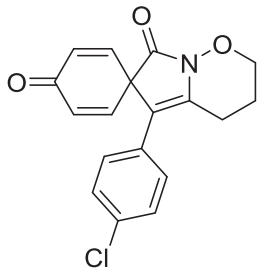
5-(4-chlorophenyl)-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4i)



White solid; mp 186−188 °C; R_f = 0.21 (1:1 petroleum ether / ethyl acetate); 77 mg, 62% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (d, *J* = 8.0 Hz, 1H), 7.56−7.52 (m, 1H), 7.50−7.44 (m, 3H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 1H), 4.44 (t, *J* = 8.0 Hz, 2H), 2.70 (t, *J* = 6.8 Hz, 2H), 2.12−2.04 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 157.1, 138.6, 135.7, 133.9, 133.4, 132.4, 132.0, 129.0, 127.7, 126.0, 125.4, 124.6, 113.3, 70.8, 21.6, 21.3; ESI-HRMS:

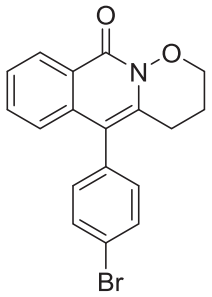
Calcd for $C_{18}H_{14}ClNO_2$[M]: 311.0713, found 311.0715.

5'-(4-chlorophenyl)-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4i')



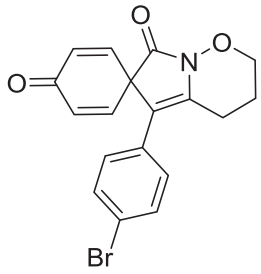
Pale yellow oil; R_f = 0.16 (1:1 petroleum ether / ethyl acetate); 20 mg, 15% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.8 Hz, 2H), 6.67 (d, *J* = 10.0 Hz, 2H), 6.56 (d, *J* = 10.0 Hz, 2H), 4.34 (t, *J* = 5.6 Hz, 2H), 2.98 (t, *J* = 6.4 Hz, 2H), 2.09−2.03 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 184.8, 165.6, 143.5, 136.4, 133.6, 133.1, 130.0, 129.0, 128.4, 112.8, 70.8, 57.3, 21.2, 20.7; ESI-HRMS: Calcd for $C_{18}H_{14}ClNO_3$[M]: 327.0662, found 327.0666.

5-(4-bromophenyl)-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4j)



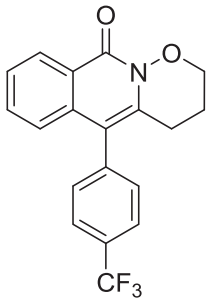
White solid; mp 196−198 ^{o}C; R_f = 0.24 (1:1 petroleum ether / ethyl acetate); 84 mg, 59% yield; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.52 (d, *J* = 8.1 Hz, 1H), 7.64 (d, *J* = 10.8 Hz, 2H), 7.57−7.51 (m, 1H), 7.48−7.43 (m, 1H), 7.20−7.13 (m, 3H), 4.44 (t, *J* = 7.8 Hz, 2H), 2.70 (t, *J* = 6.9 Hz, 2H), 2.13−2.04 (m, 2H); ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 157.2, 138.5, 135.6, 133.9, 132.7, 132.0, 131.6, 127.8, 126.1, 125.3, 124.7, 122.1, 113.3, 70.8, 21.6, 21.3; ESI-HRMS: Calcd for $C_{18}H_{14}BrNO_2$[M]: 355.0208, found 355.0209.

5'-(4-bromophenyl)-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4j')



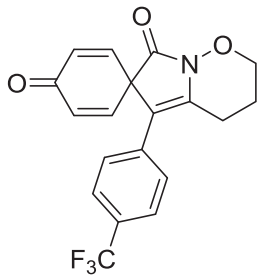
Colourless oil; R_f = 0.17 (1:1 petroleum ether / ethyl acetate); 15 mg, 10% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, J = 8.4 Hz, 2H), 7.07 (d, J = 8.4 Hz, 2H), 6.68 (d, J = 10.0 Hz, 2H), 6.57 (d, J = 10.0 Hz, 2H), 4.34 (t, J = 5.2 Hz, 2H), 2.99 (t, J = 6.8 Hz, 2H), 2.10−2.04 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 184.8, 165.6, 143.5, 136.5, 133.1, 131.9, 130.5, 128.6, 121.8, 112.8, 70.8, 57.2, 21.2, 20.7; ESI-HRMS: Calcd for $C_{18}H_{14}BrNO_3$[M]: 371.0157, found 371.0159.

5-(4-(trifluoromethyl)phenyl)-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4k)



White solid; mp 212−213 ^{o}C; R_f = 0.24 (1:1 petroleum ether / ethyl acetate); 73 mg, 53% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.54 (dd, J = 8.0 Hz, J = 0.8 Hz, 1H), 7.78 (d, J = 8.0 Hz, 2H), 7.56−7.52 (m, 1H), 7.48−7.44 (m, 3H), 7.10 (d, J = 8.0 Hz, 1H), 4.45 (t, J = 7.6 Hz, 2H), 2.70 (t, J = 6.8 Hz, 2H), 2.13−2.06 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 156.9, 138.8, 138.7, 135.3, 131.9, 131.5, 129.9 (q, $^2J_{C,F}$ = 32.2 Hz), 127.6, 125.9, 125.6, 125.2, 124.4 (q, $^3J_{C,F}$ = 3.0 Hz), 123.9 (q, $^1J_{C,F}$ = 271.6 Hz), 113.0, 70.7, 21.4, 21.2; ^{19}F NMR (376 MHz, $CDCl_3$): δ -62.56; ESI-HRMS: Calcd for $C_{19}H_{14}F_3NO_2$[M]: 345.0977, found 345.0987.

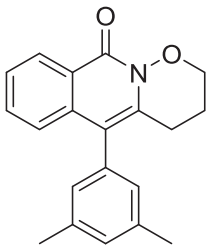
5'-(4-(trifluoromethyl)phenyl)-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4k')



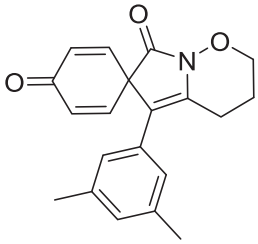
Pale yellow oil; R_f = 0.16 (1:1 petroleum ether / ethyl acetate); 19 mg, 13% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55 (d, J = 8.4 Hz, 2H), 7.32 (d, J = 8.4 Hz, 2H), 6.70 (d, J = 10.0 Hz, 2H), 6.59 (d, J = 10.0 Hz, 2H), 4.36 (t, J = 5.6 Hz, 2H), 3.05 (t, J = 6.4 Hz, 2H), 2.12−2.06 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 184.7, 165.4, 143.4, 137.9, 135.3, 133.2, 129.5 (q, $^2J_{C,F}$ = 33.2 Hz), 127.2, 125.7 (q, $^3J_{C,F}$ = 4.0 Hz), 123.8 (q, $^1J_{C,F}$ = 271.6 Hz), 112.4, 70.8, 57.2, 21.2, 20.6; ^{19}F NMR (376 MHz, $CDCl_3$): δ -62.74; ESI-HRMS: Calcd for $C_{19}H_{14}F_3NO_3$[M]: 361.0926, found 361.0923.

5-(3,5-dimethylphenyl)-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4l)

5'-(3,5-dimethylphenyl)-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4l')



4l, 63 mg, 52% yield

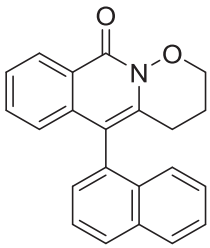


4l', 6 mg, 5% yield

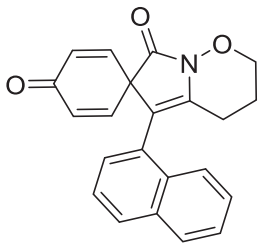
White solid; R_f = 0.27 (1:1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52 (d, J = 8.0 Hz, 1H), 7.53−7.49 (m, 1H), 7.44−7.40 (m, 1H), 7.19 (d, J = 8.0, Hz, 1H), 7.08 (s, 1H), 6.92−6.89 (m, 3H), 6.80 (s, 2H), 6.69 (d, J = 10.0 Hz, 2H), 6.53 (d, J = 10.0 Hz, 2H), 4.43 (t, J = 8.0 Hz, 2H), 4.32 (t, J = 5.2 Hz, 2H), 2.98 (t, J = 6.8 Hz, 2H), 2.72 (t, J = 6.8 Hz, 2H), 2.38 (s, 6H), 2.25 (s, 6H), 2.11−2.00 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 184.9, 166.0, 157.2, 143.8, 138.2, 138.14, 138.06, 136.2, 135.6, 134.7, 132.6, 131.7, 131.3, 129.5, 129.4, 128.6, 127.5, 125.7, 125.3, 125.1, 114.8, 114.2, 70.8, 57.6, 21.7, 21.3, 21.2, 21.0, 20.8; ESI-HRMS: Calcd for $C_{20}H_{19}NO_2$[M] (**4l**): 305.1416, found 305.1415; $C_{20}H_{19}NO_3$[M] (**4l'**): 321.1365, found 321.1361.

5-(naphthalen-1-yl)-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4m)

5'-(naphthalen-1-yl)-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4m')



4m, 88 mg, 67% yield

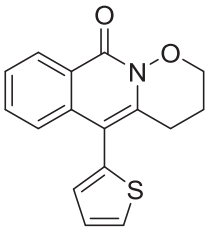


4m', 7 mg, 5% yield

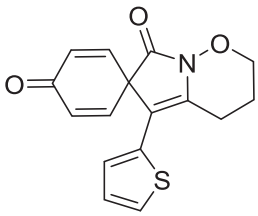
White solid; R_f = 0.24 (1:1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.56 (d, J = 7.6 Hz, 1H), 7.98−7.93 (m, 2H), 7.87−7.83 (m, 2H), 7.79 (d, J = 8.0 Hz, 1H), 7.60−7.54 (m, 2H), 7.51−7.47 (m, 2H), 7.42−7.33 (m, 6H), 7.23 (d, J = 7.2 Hz, 1H), 6.92 (d, J = 6.0 Hz, 1H), 6.85 (d, J = 7.6 Hz, 1H), .6.69 (d, J = 8.0 Hz, 1H), 6.55 (d, J = 8.4 Hz, 1H), 6.16 (d, J = 8.0 Hz, 1H), 4.50−4.40 (m, 2H), 4.37−4.33 (m, 2H), 2.60−2.53 (m, 2H), 2.49−2.42 (m, 2H), 2.05−1.92 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 184.5, 167.6, 157.2, 139.1, 137.8, 136.3, 133.64, 133.55, 132.9, 132.2, 131.8, 131.6, 129.0, 128.92, 128.87, 128.5, 128.3, 128.1, 127.5, 127.3, 126.7, 126.5, 126.0, 125.9, 125.8, 125.5, 125.3, 125.1, 125.0, 124.8, 124.1, 112.0, 111.4, 70.8, 70.7, 58.6, 21.5, 21.2, 20.4; ESI-HRMS: Calcd for $C_{22}H_{17}NO_2$[M] (**4m**): 327.1259, found 327.1265; $C_{22}H_{17}NO_3$[M] (**4m'**): 343.1208, found 343.1217.

5-(thiophen-2-yl)-3,4-dihydro-[1,2]oxazino[2,3-b]isoquinolin-10(2H)-one (4n)

5'-(thiophen-2-yl)-3',4'-dihydro-2'H,7'H-spiro[cyclohexane-1,6'-pyrrolo[1,2-b][1,2]oxazine]-2,5-diene-4,7'-dione (4n')



4n, 66 mg, 58% yield

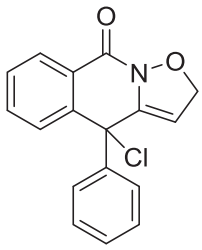


4n', 5 mg, 4% yield

White solid; R_f = 0.28 (1:1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.48 (dd, J = 8.0 Hz, J = 0.8 Hz, 1H), 7.57−7.53 (m, 1H), 7.49−7.48 (m, 1H), 7.45−7.41 (m, 1H), 7.33−7.30 (m, 1H), 7.21−7.20 (m, 1H), 7.19−7.17 (m, 1H), 7.03−7.02 (m, 1H), 6.94−6.91 (m, 1H), 6.81 (d, J = 3.2 Hz, 1H), 6.67 (d, J = 10.0 Hz, 2H), 6.60 (d, J = 10.0 Hz,

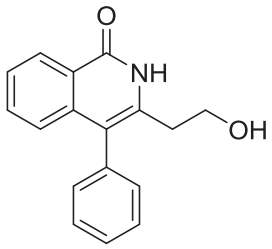
2H), 4.42 (t, *J* = 7.6 Hz, 2H), 4.31 (t, *J* = 5.6 Hz, 2H), 3.07 (t, *J* = 6.8 Hz, 2H), 2.80 (t, *J* = 6.8, 2H), 2.13−2.06 (m, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 184.7, 164.2, 157.1, 143.7, 140.7, 136.6, 135.2, 133.82, 133.79, 133.0, 132.0, 129.4, 127.4, 127.3, 127.1, 127.0, 126.0, 124.9, 124.7, 124.5, 123.8, 109.0, 106.5, 70.8, 70.6, 56.8, 21.45, 21.44, 21.1, 20.3; ESI-HRMS: Calcd for $C_{16}H_{13}NO_2S$[M] (**4n**): 283.0667, found 283.0673; $C_{16}H_{13}NO_3S$[M] (**4n'**): 299.0616, found 299.0625.

4-chloro-4-phenyl-2,4-dihydro-9H-isoxazolo[2,3-b]isoquinolin-9-one (5)



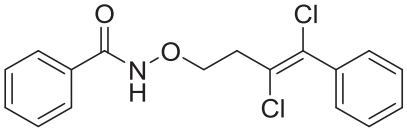
White solid; mp 203−205 °C; R_f = 0.25 (1:1 petroleum ether / ethyl acetate); 73 mg, 82% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.59−8.57 (m, 1H), 7.61−7.51 (m, 6H), 7.38−7.32 (m, 2H), 5.32 (d, *J* = 3.6 Hz, 1H), 4.74 (d, *J* = 10.0 Hz, 1H), 4.69 (dd, *J* = 10.0 Hz, *J* = 4.0 Hz, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 153.4, 136.0, 133.1, 132.1, 131.9, 130.9, 129.9, 128.85, 128.78, 128.6, 127.6, 127.4, 127.1, 125.8, 115.5, 77.6, 56.5; ESI-HRMS: Calcd for $C_{17}H_{12}ClNO_2$[M]: 297.0557, found 297.0560.

3-(2-hydroxyethyl)-4-phenylisoquinolin-1(2H)-one (6)



White solid; mp 209−211 °C; R_f = 0.17 (1:2 petroleum ether / ethyl acetate); 73 mg, 92% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.58 (s, 1H), 8.33 (d, *J* = 8.0 Hz, 1H), 7.48−7.40 (m, 4H), 7.35−7.32 (m, 1H), 7.26−7.24 (m, 2H), 7.04 (d, *J* = 8.4 Hz, 1H), 4.82 (brs, 1H), 3.93 (d, *J* = 5.6 Hz, 2H), 2.73 (d, *J* = 5.6 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 163.7, 139.0, 137.2, 135.9, 132.4, 131.1, 128.7, 127.7, 126.9, 125.9, 125.2, 124.0, 117.9, 61.5, 33.7; ESI-HRMS: Calcd for $C_{17}H_{15}NO_2$[M]: 265.1103, found 265.1108.

(E)-N-((3,4-dichloro-4-phenylbut-3-en-1-yl)oxy)benzamide (7)



Colourless oil; R_f = 0.23 (5:1 petroleum ether / ethyl acetate); 29 mg, 29% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.98 (brs, 1H), 7.78−7.76 (m, 2H), 7.54−7.50 (m, 1H), 7.46−7.41 (m, 4H), 7.39−7.34 (m, 3H), 4.36 (t, J = 6.4 Hz, 2H), 3.19 (t, J = 6.4 Hz, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 166.8, 136.7, 132.2, 131.7, 129.1, 129.0, 128.8, 128.7, 128.1, 127.2, 127.1, 72.7, 35.8; ESI-HRMS: Calcd for $C_{17}H_{15}Cl_2NO_2$[M]: 335.0480, found 335.0483.

ORTEP plot, crystal data and refinement results for compound 2g:

ORTEP plot for compound 2g:

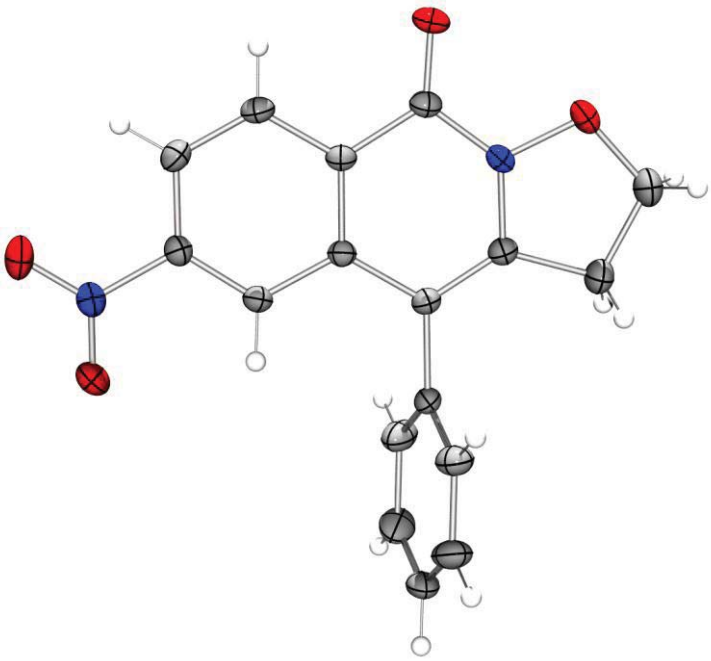
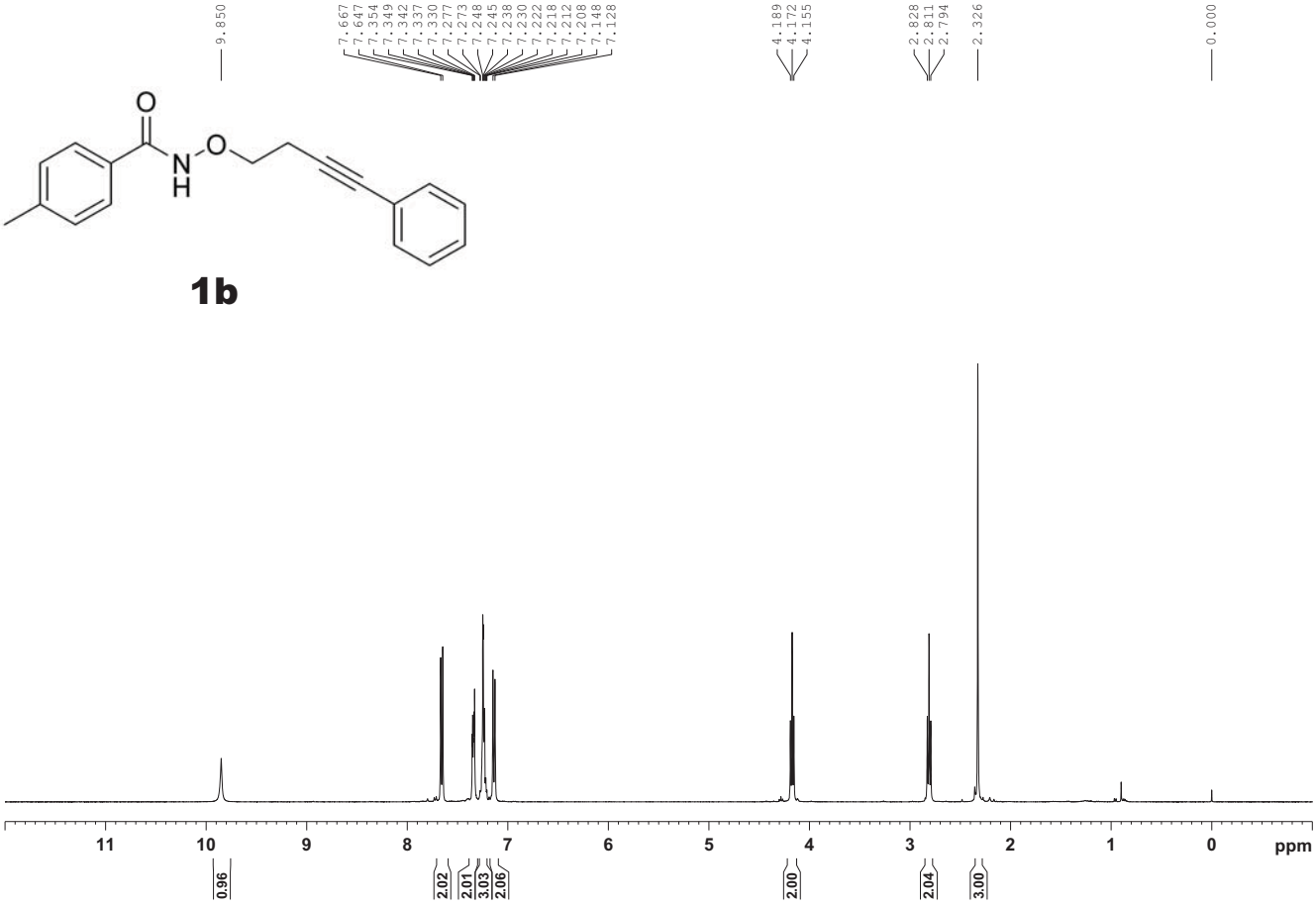


Figure S9. Compound **2g**, thermal ellipsoids are drawn at 30% probability level.

Crystal Data and Refinement Results for Compound 2g:

Table S1. The crystal data and refinement results of compound **2g**

Compound number	**2g**
Formula	$C_{17}H_{12}N_2O_4$
Fw	308.29
Temp	290.04(10)
Crystal system	Monoclinic
Space group	P 21/c
a Å	6.8554 (3)
b Å	18.0992 (7)
c Å	11.1588 (4)
α°	90.000
β°	92.472 (4)
γ°	90.000
V $Å^3$	1383.27 (10)
Z	4
Density(calcd) $g \cdot cm^{-3}$	1.480
Absorb.coeff. mm^{-1}	0.896
F(000)	640
Index ranges	$-6 \le h \le 8$ $-21 \le k \le 21$ $-9 \le l \le 13$
refln./restr./param.	2524/0/208
GOF	0.849
[$I > 2\sigma(I)$]	R_1=0.0505 wR_2=0.1364
CCDC numbers	1838258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7.330
7.277
7.273
7.248
7.245
7.238
7.230
7.222
7.218
7.212
7.208
7.148
7.128
4.189
4.172
4.155
2.828
2.811
2.794
2.326
0.000
1b
11
10
9
8
7
6
5
4
3
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1
0
ppm
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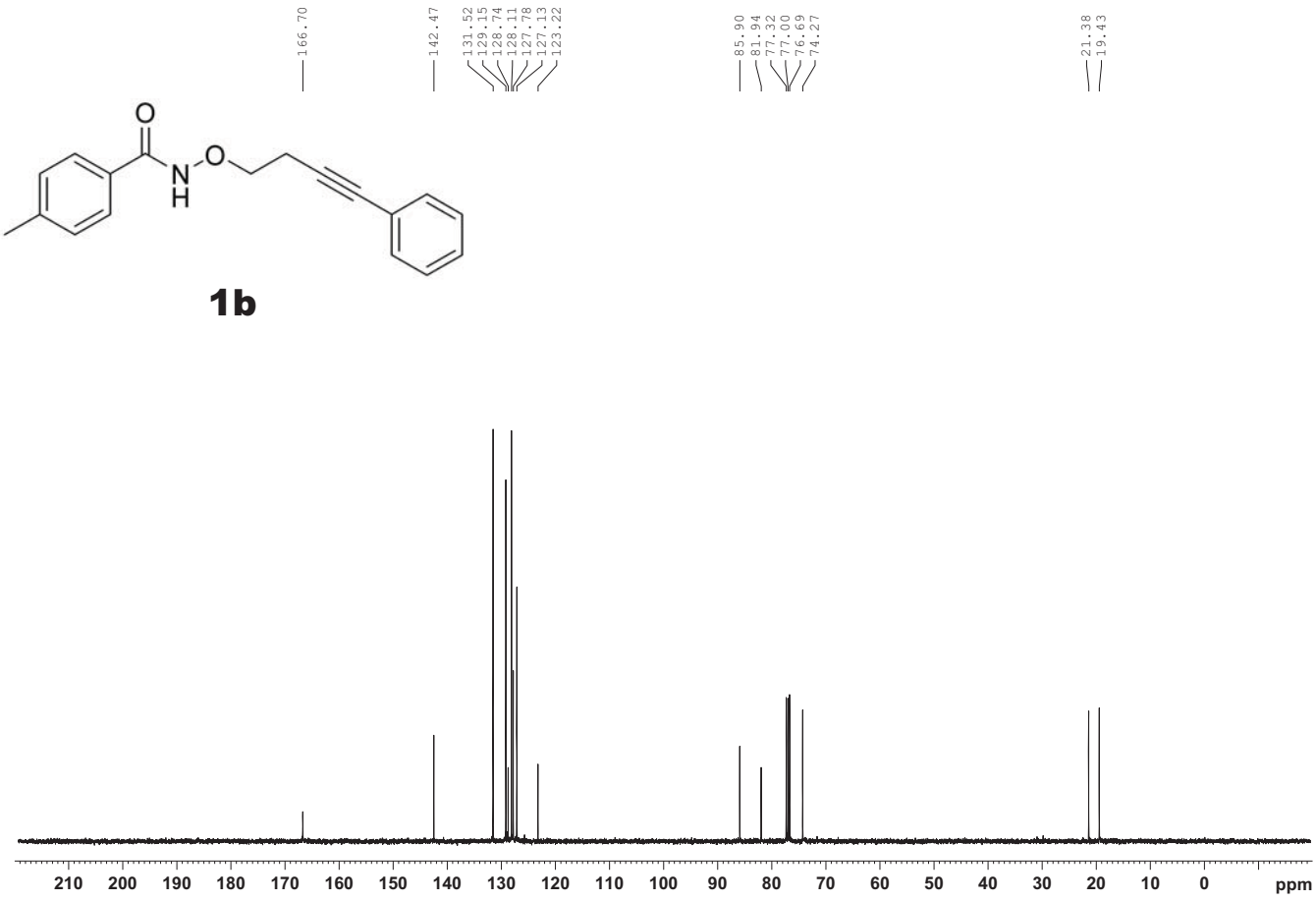
2.01
3.03
2.06
2.00
2.04
3.00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127.13
123.22
85.90
81.94
77.32
77.00
76.69
74.27
21.38
19.43
1b
210
200
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130
120
110
100
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60
50
40
30
20
10
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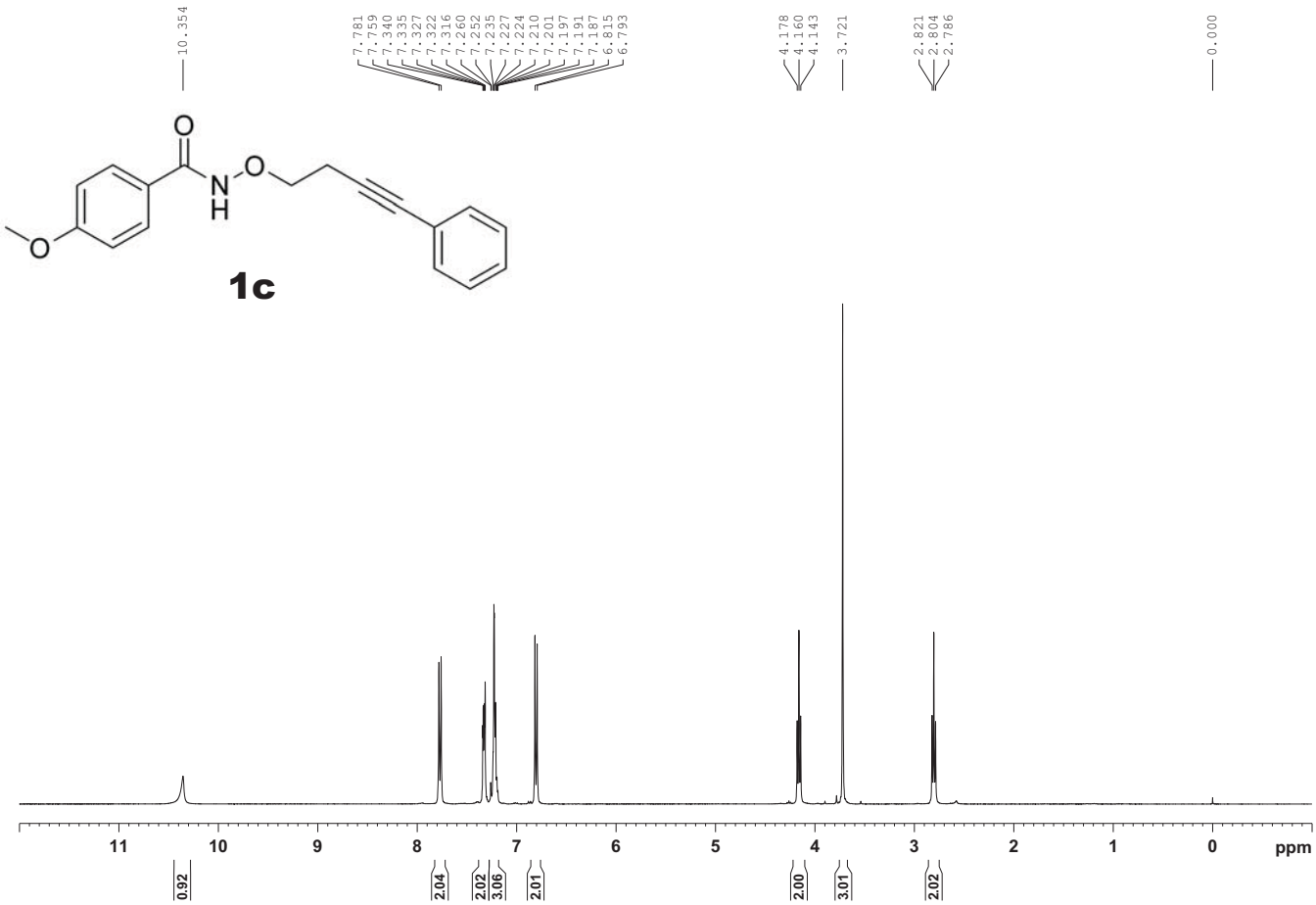
ppm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7.235
7.227
7.224
7.210
7.201
7.197
7.191
7.187
6.815
6.793
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4.160
4.143
3.721
2.821
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0.000
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11
10
9
8
7
6
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3.06
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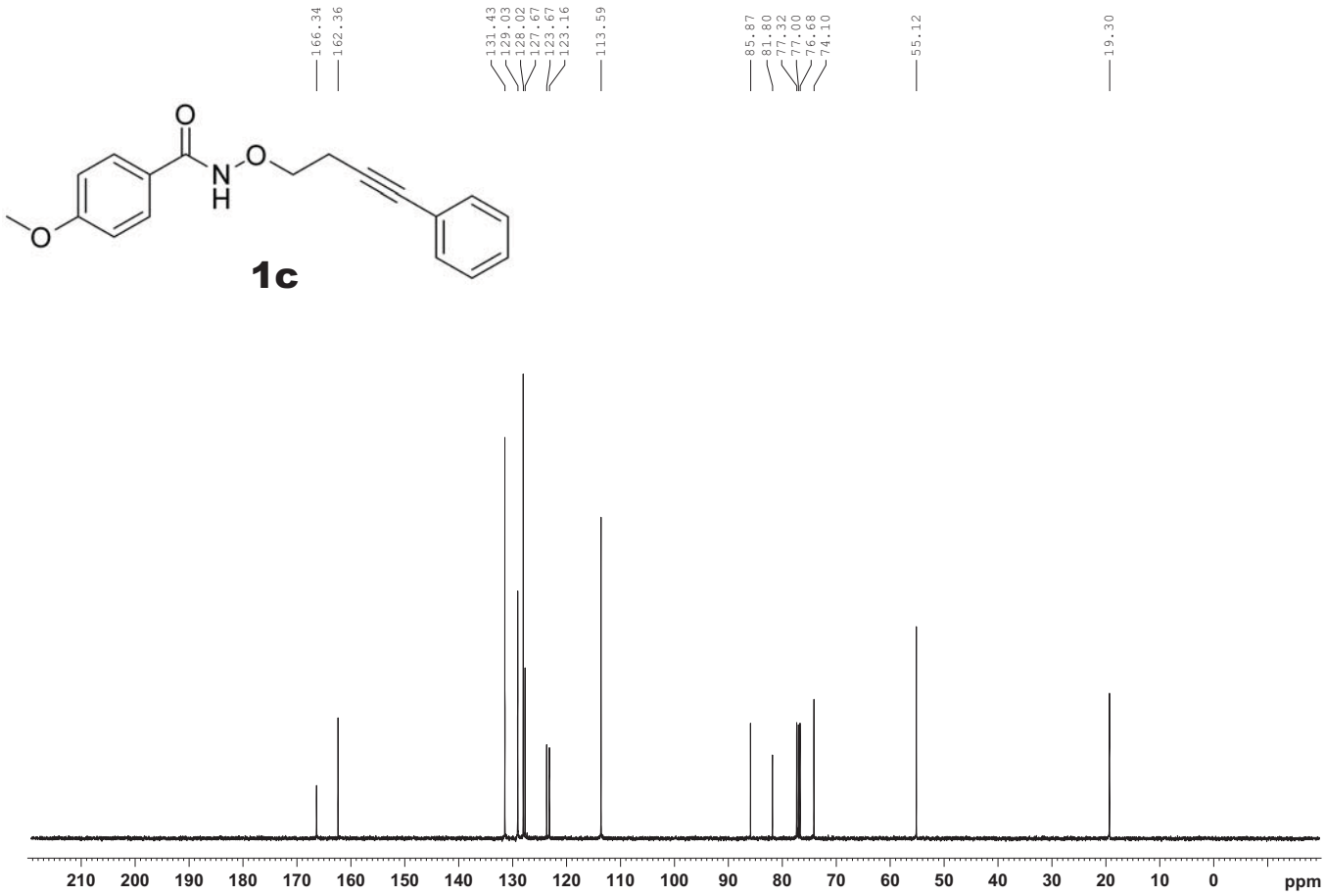
3.01
2.02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74.10
55.12
19.30
1c
210
200
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130
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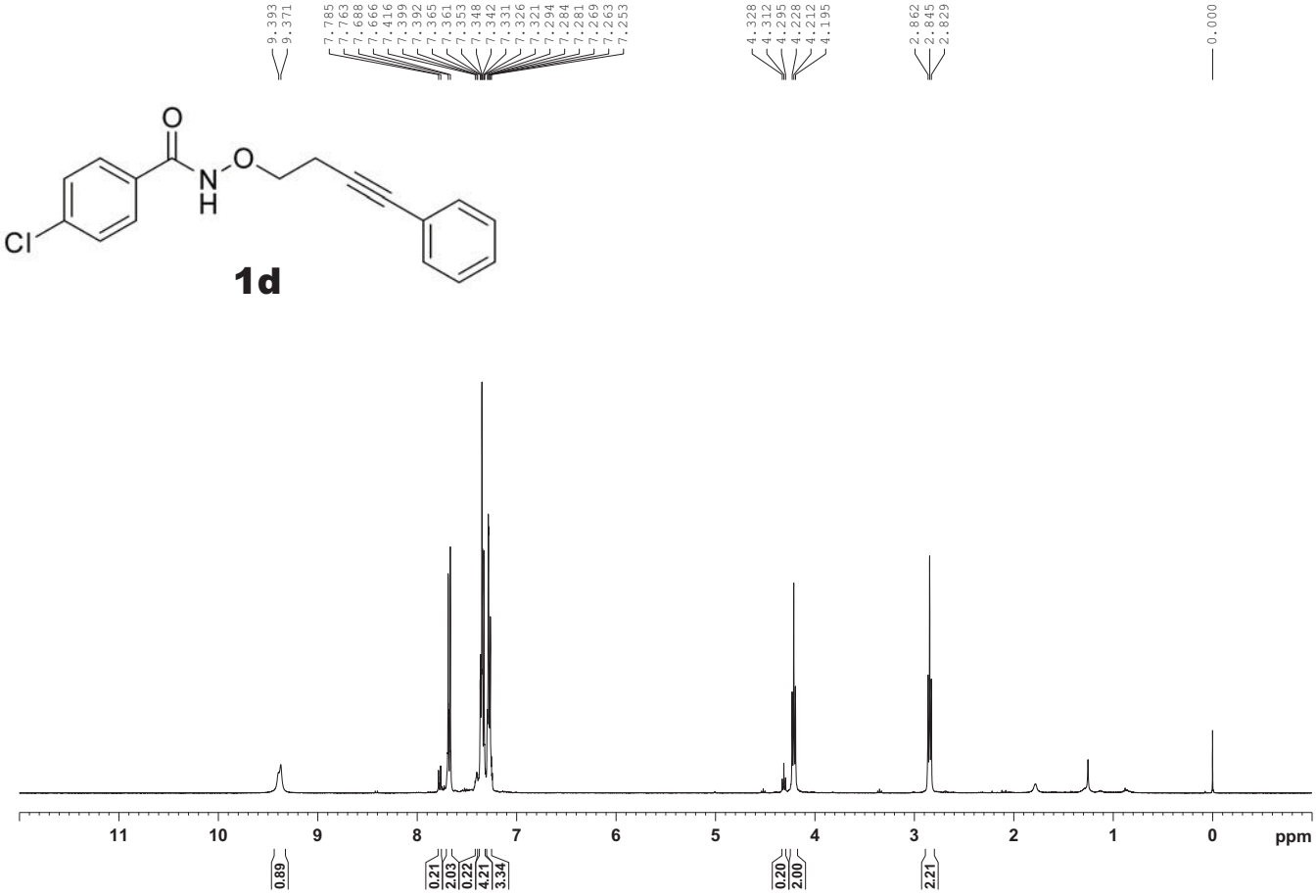
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7.399
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7.365
7.361
7.353
7.348
7.342
7.331
7.326
7.321
7.294
7.284
7.281
7.269
7.263
7.253
4.328
4.312
4.295
4.228
4.212
4.195
2.862
2.845
2.829
0.000
O
N
H
O
Cl
1d
11
10
9
8
7
6
5
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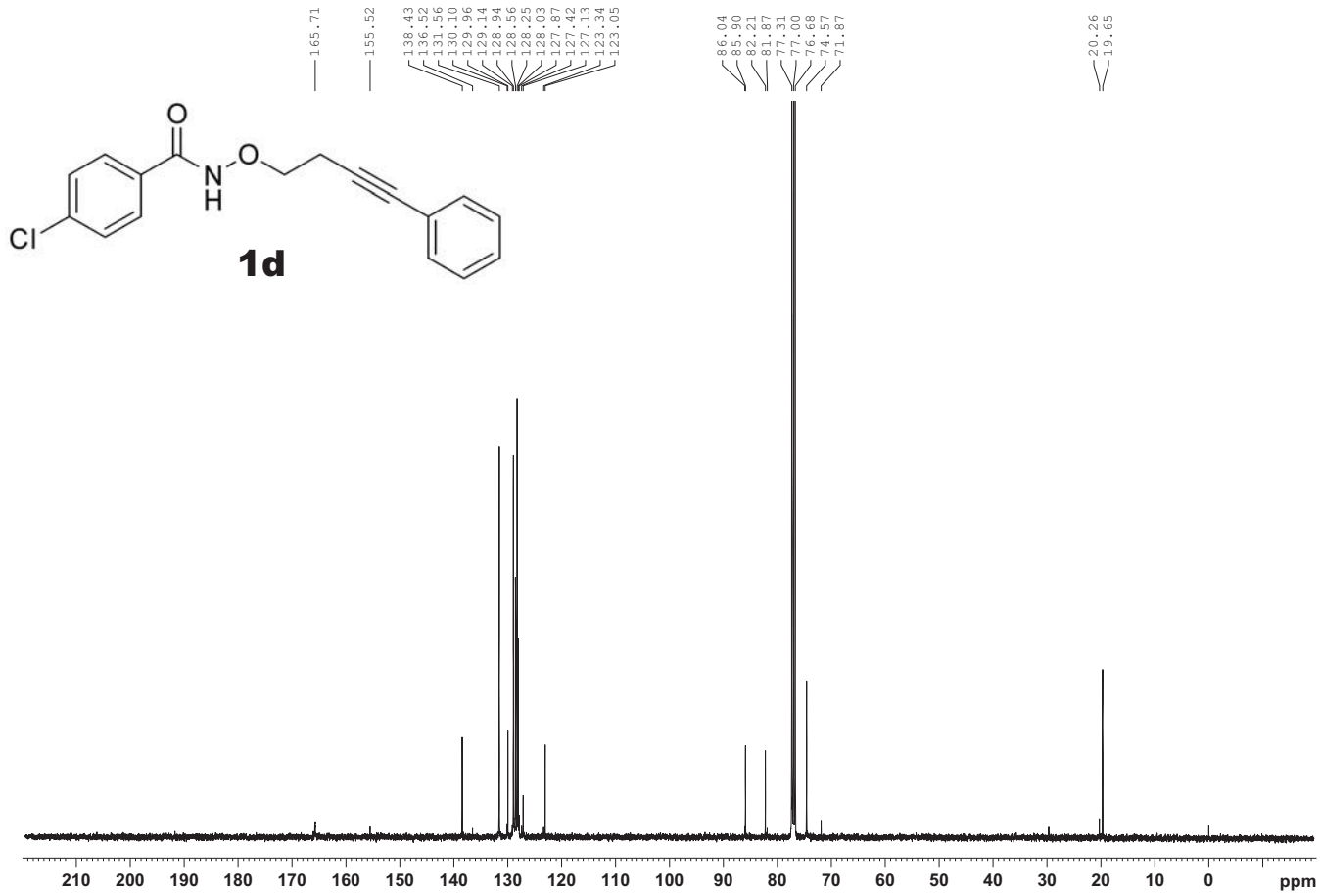
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127.42
127.13
123.34
123.05
86.04
85.90
82.21
81.87
77.31
77.00
76.68
74.57
71.87
20.26
19.65
O
N
H
O
Cl
1d
210
200
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140
130
120
110
100
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60
50
40
30
20
10
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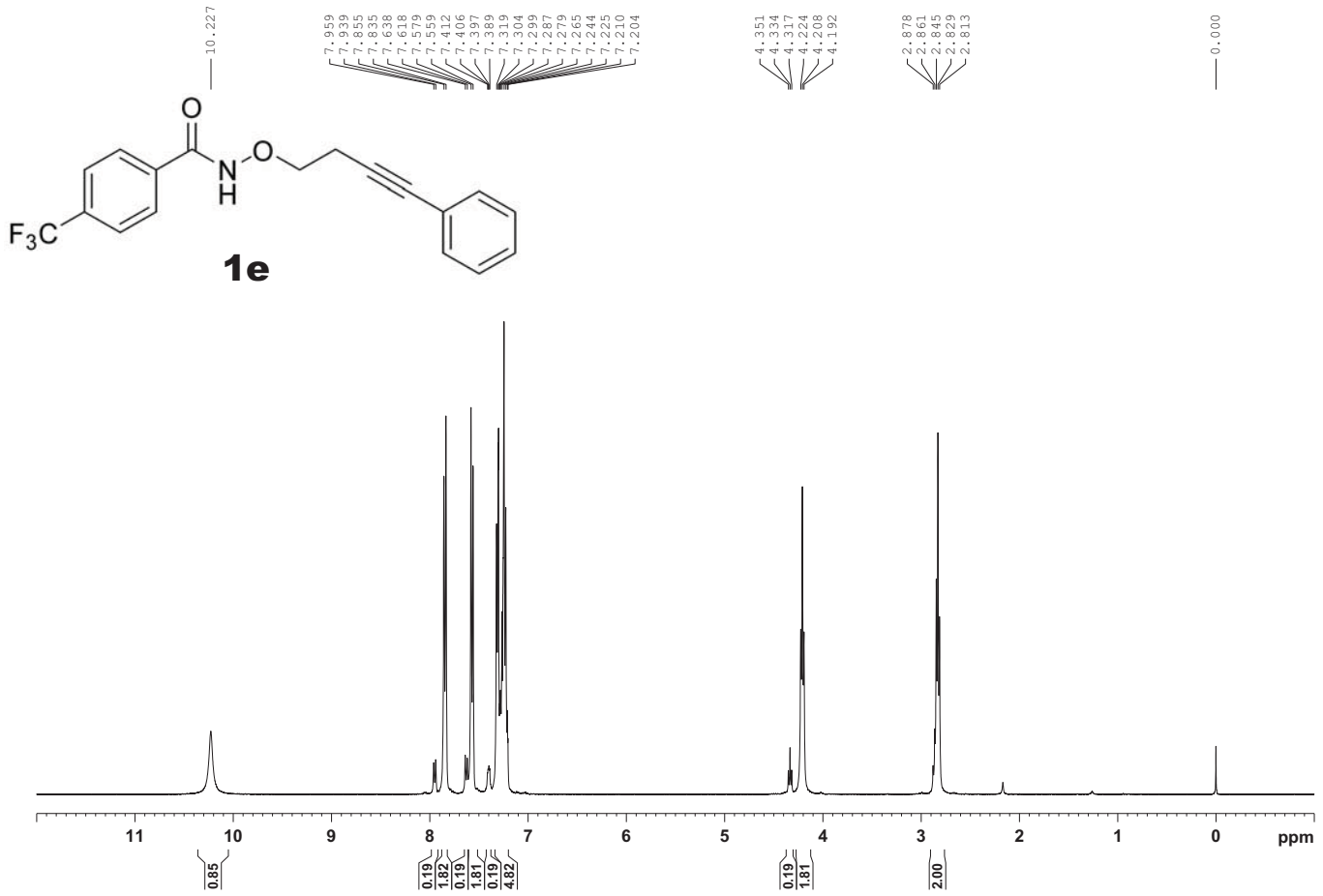
ppm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7.579
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7.319
7.304
7.299
7.287
7.279
7.265
7.244
7.225
7.210
7.204
4.351
4.334
4.317
4.224
4.208
4.192
2.878
2.861
2.845
2.829
2.813
0.000
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N
H
O
F3C
1e
11
10
9
8
7
6
5
4
3
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0.19
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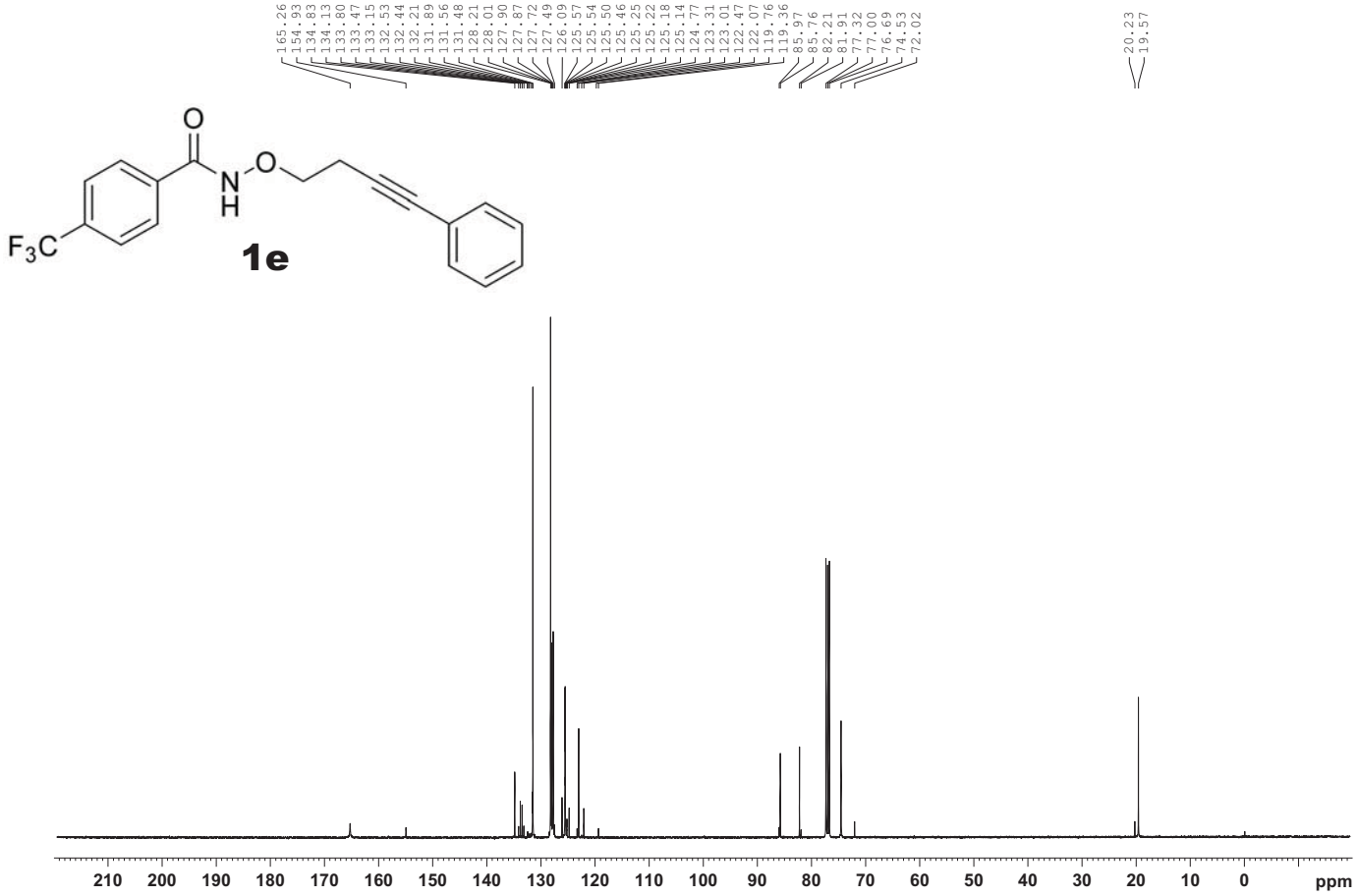
1.81
2.00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128.01
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127.87
127.72
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125.54
125.50
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H
O
F3C
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200
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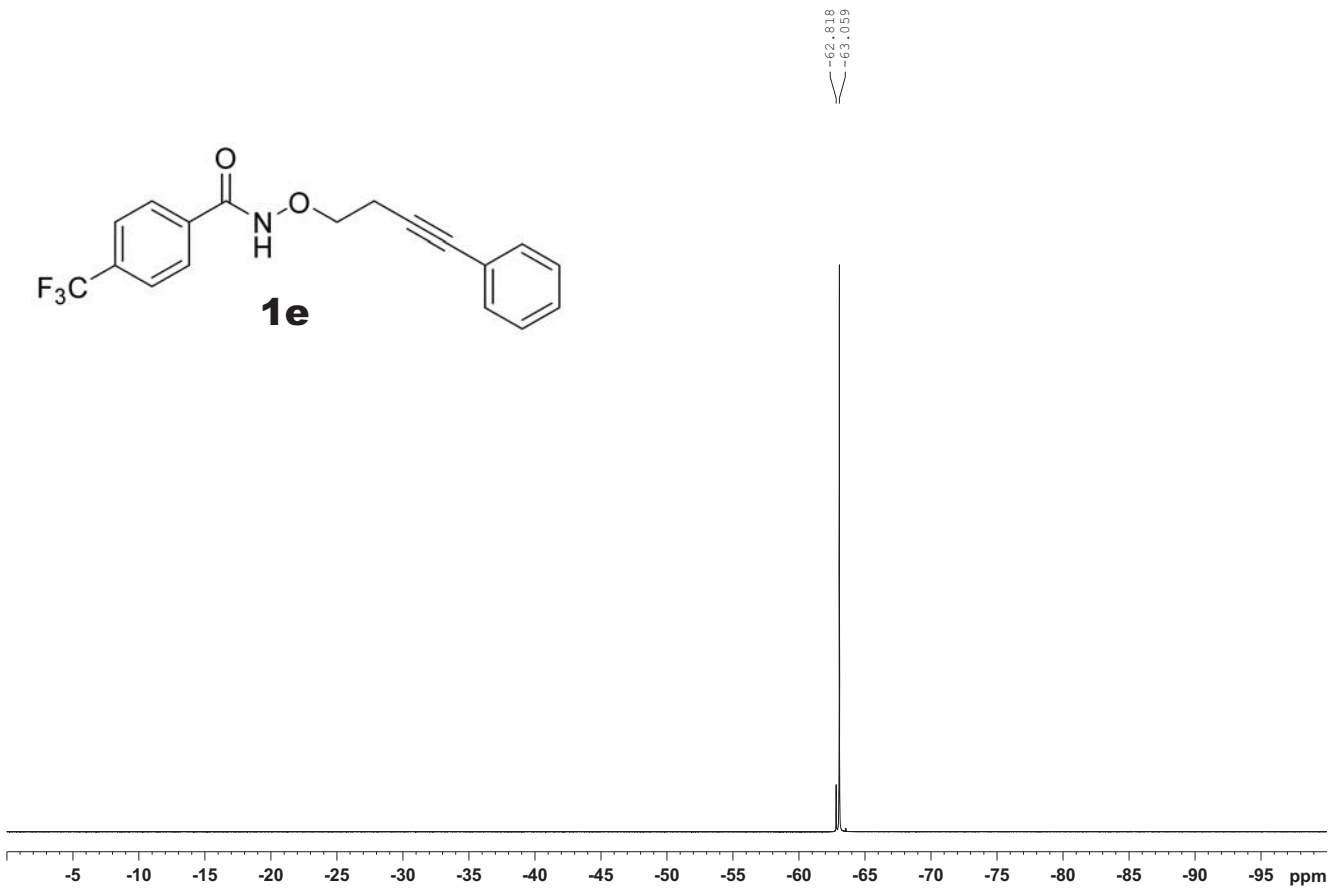
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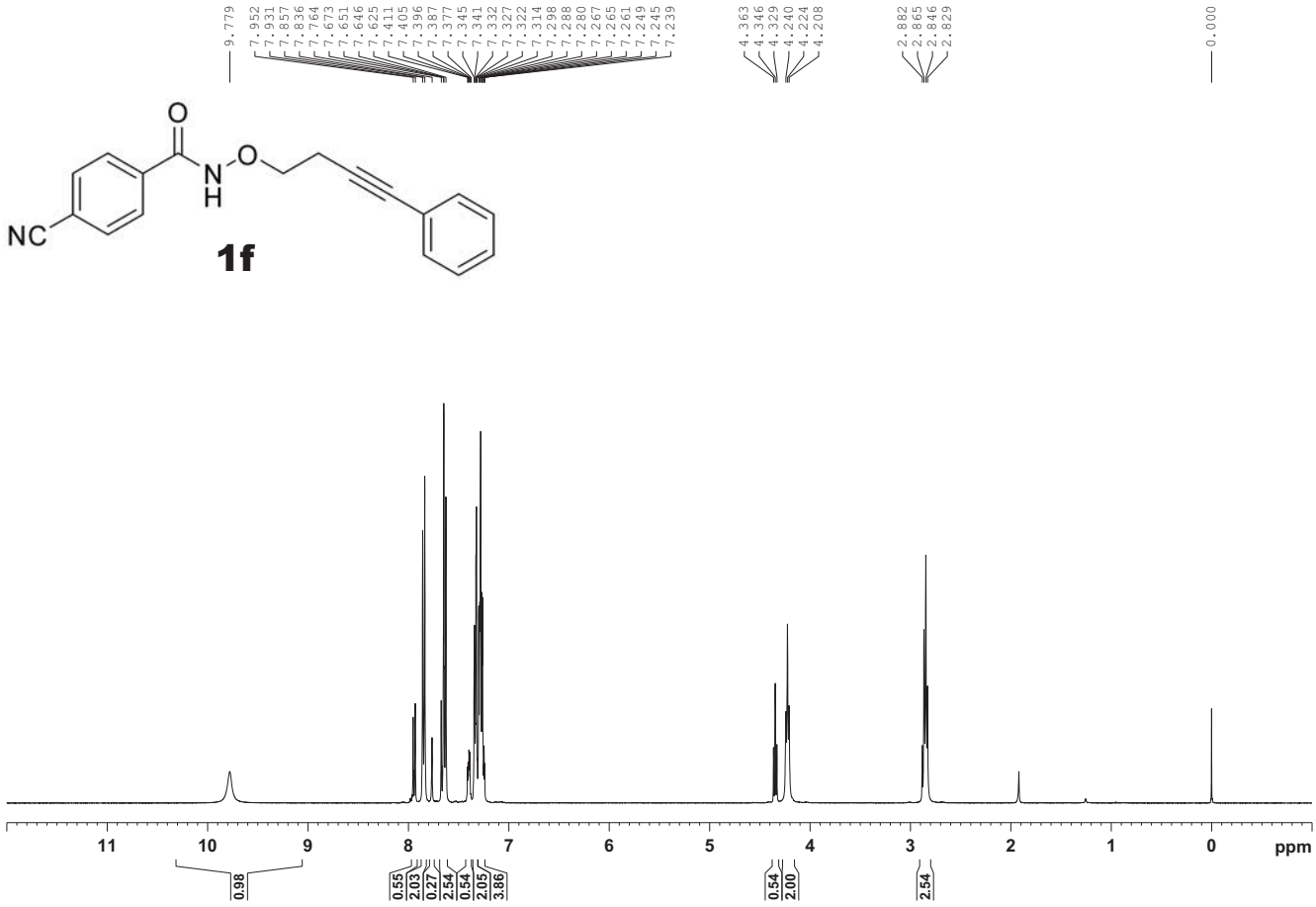
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7.327
7.322
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H
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7
6
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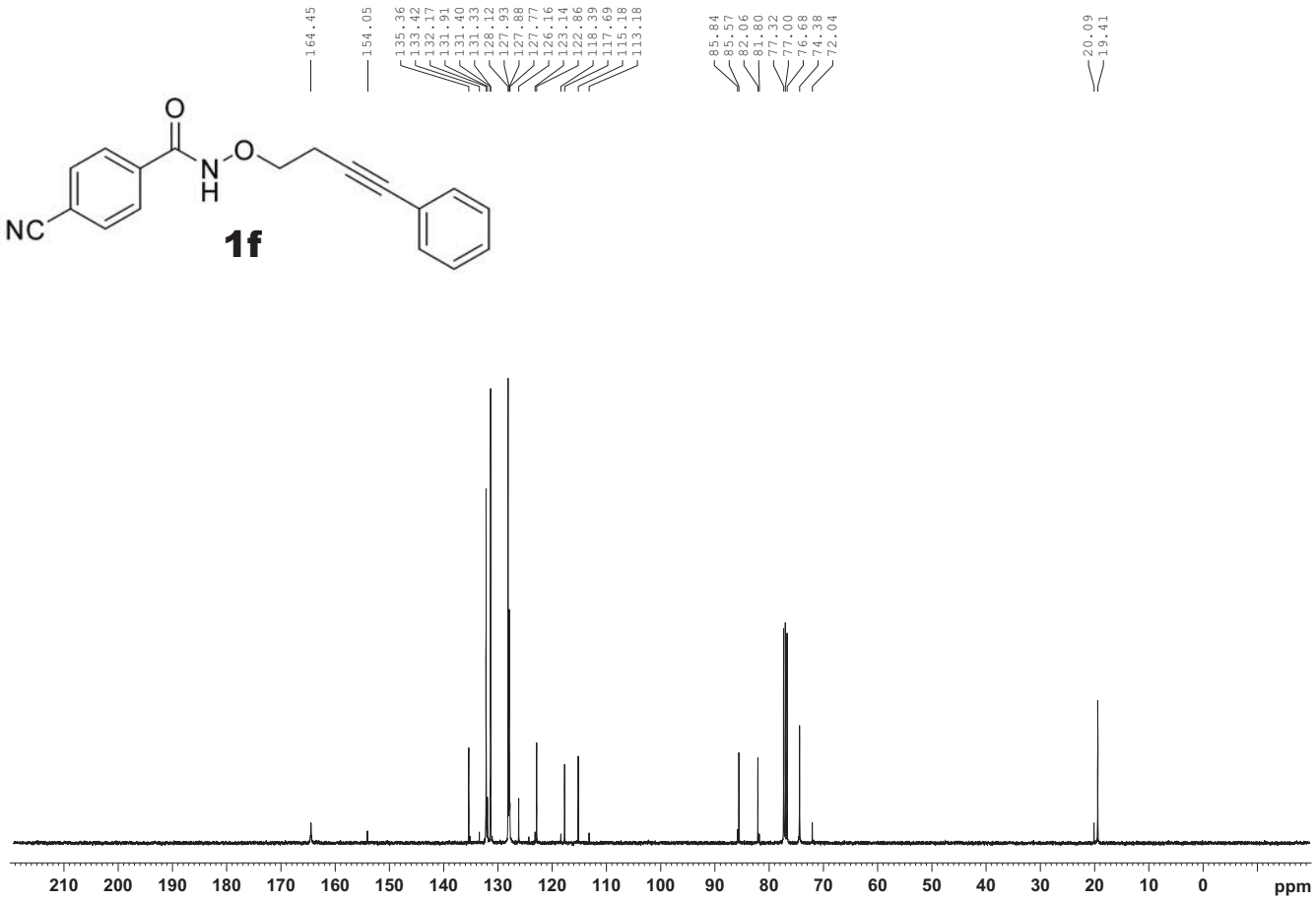
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113.18
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85.57
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77.32
77.00
76.68
74.38
72.04
20.09
19.41
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H
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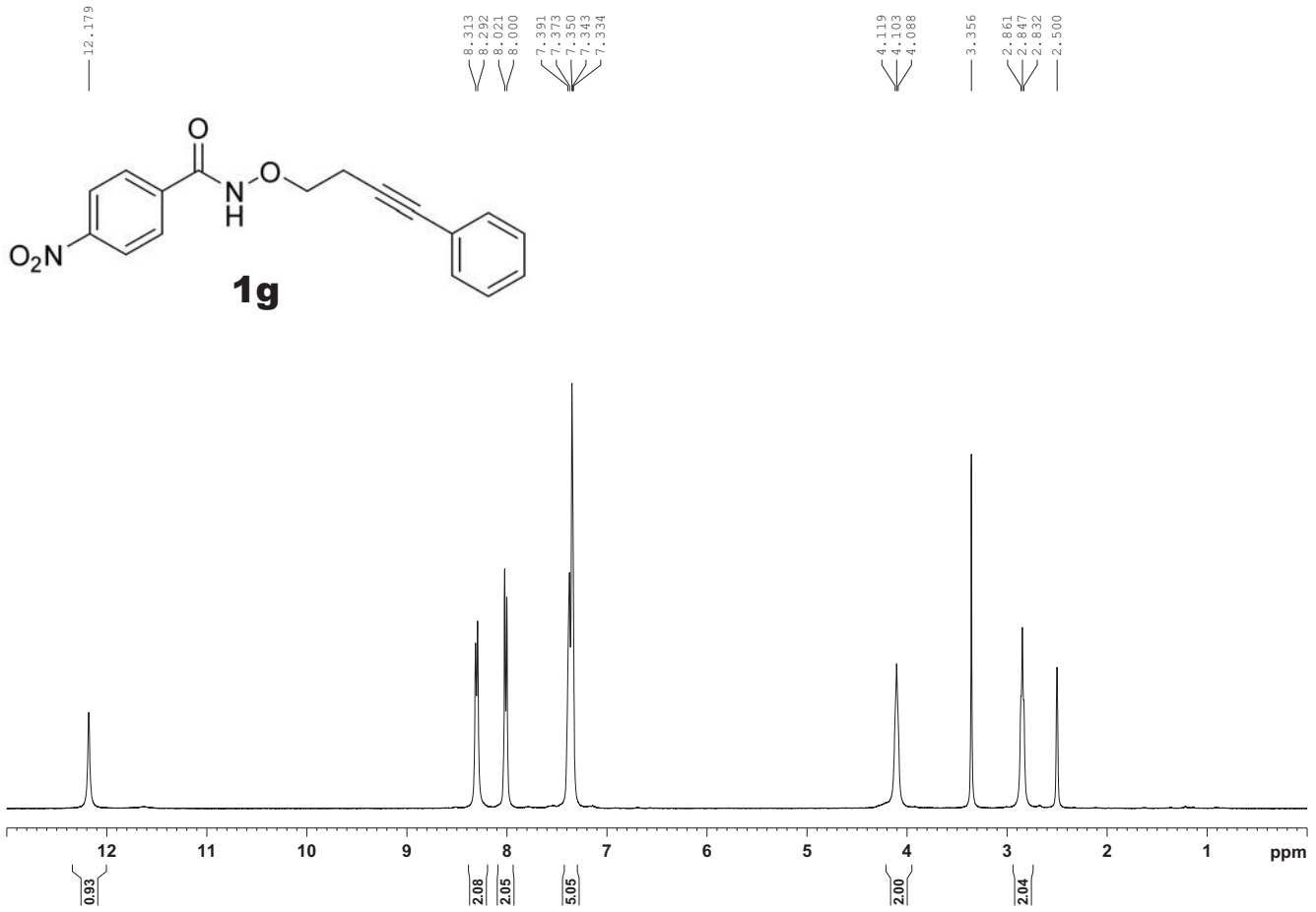
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3.356
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2.847
2.832
2.500
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N
H
O
O_2N
1g
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4
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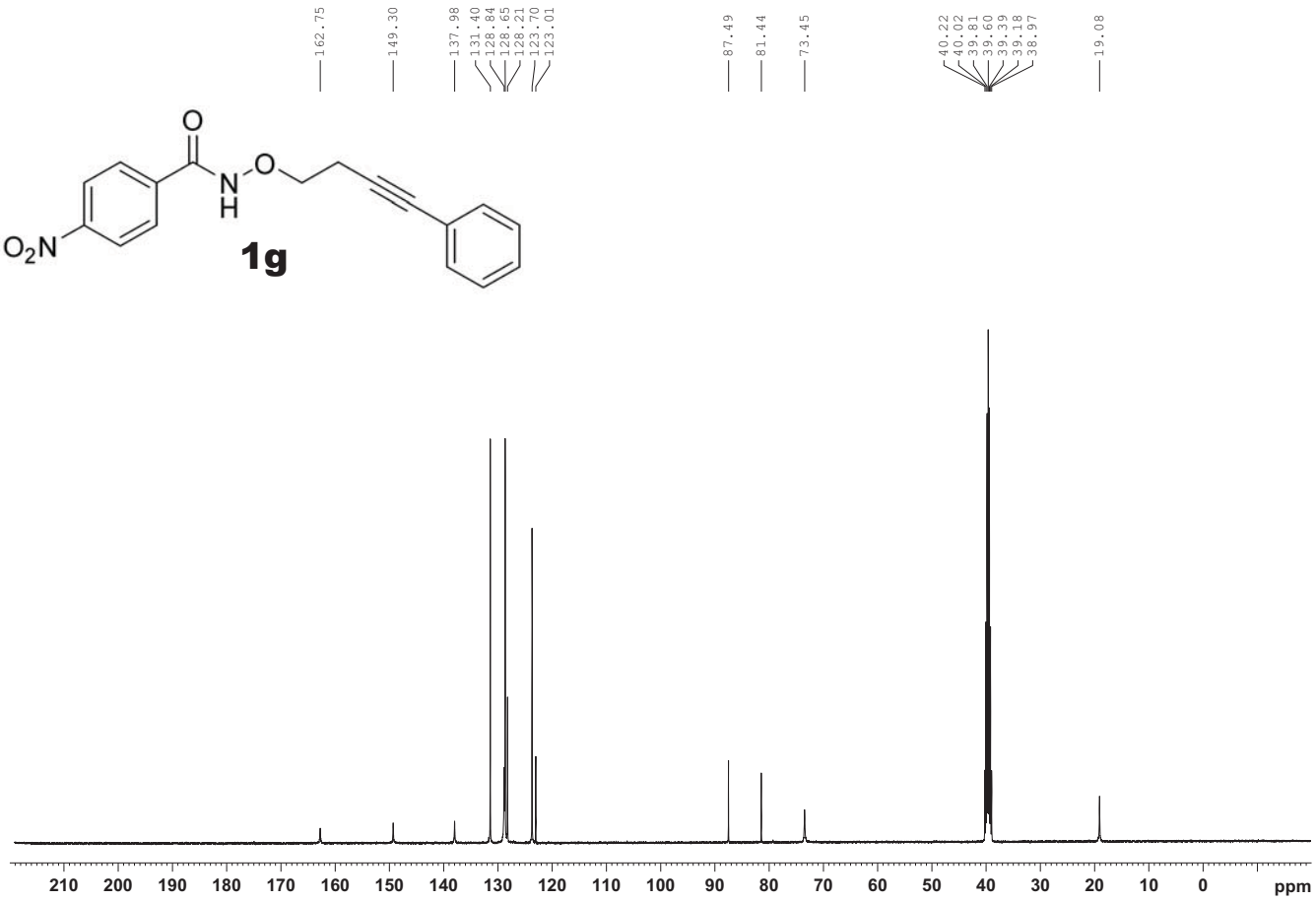
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40.02
39.81
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O
N
H
O
O_2N
1g
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200
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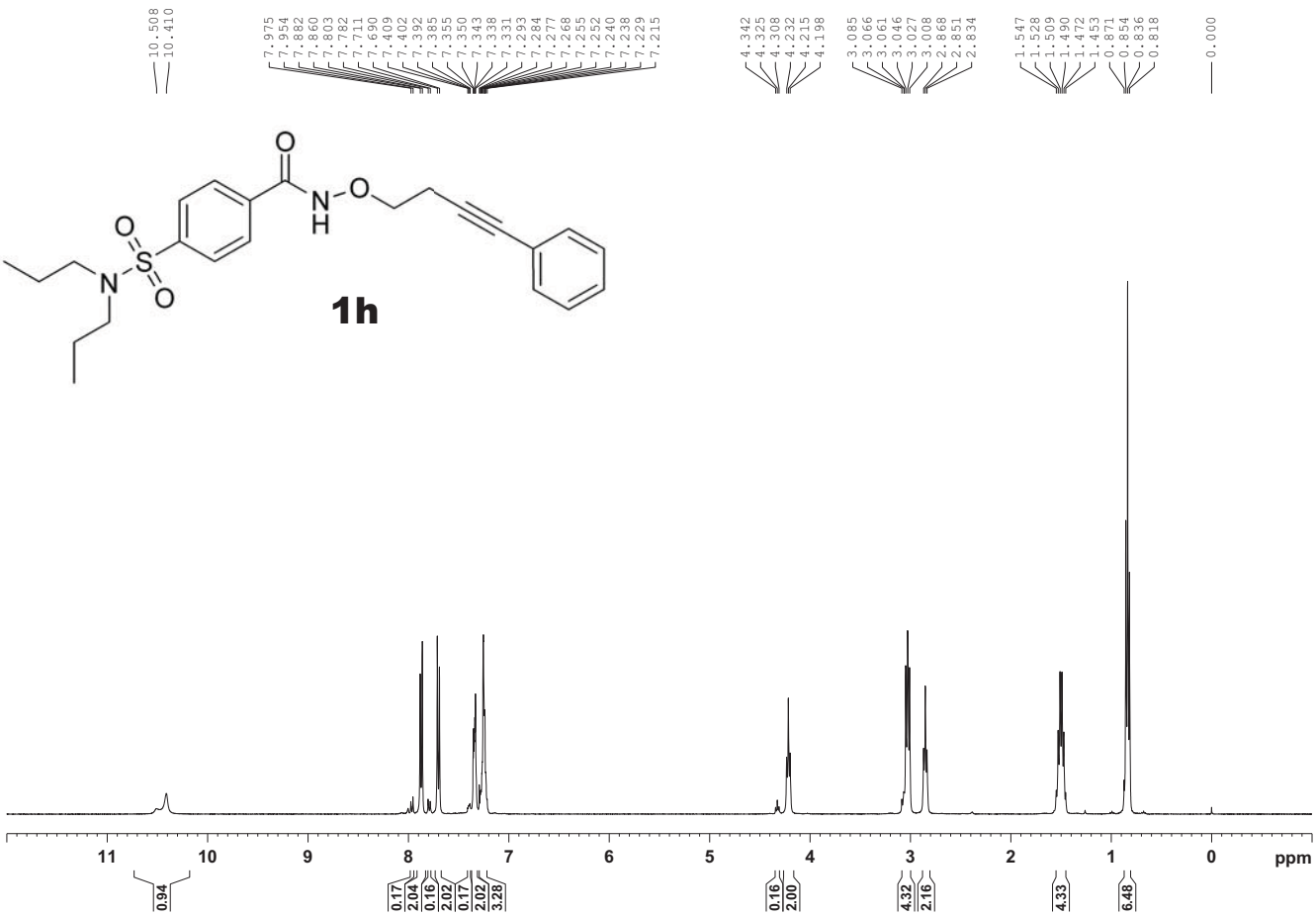
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7.277
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7.255
7.252
7.240
7.238
7.229
7.215
4.342
4.325
4.308
4.232
4.215
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3.085
3.066
3.061
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6
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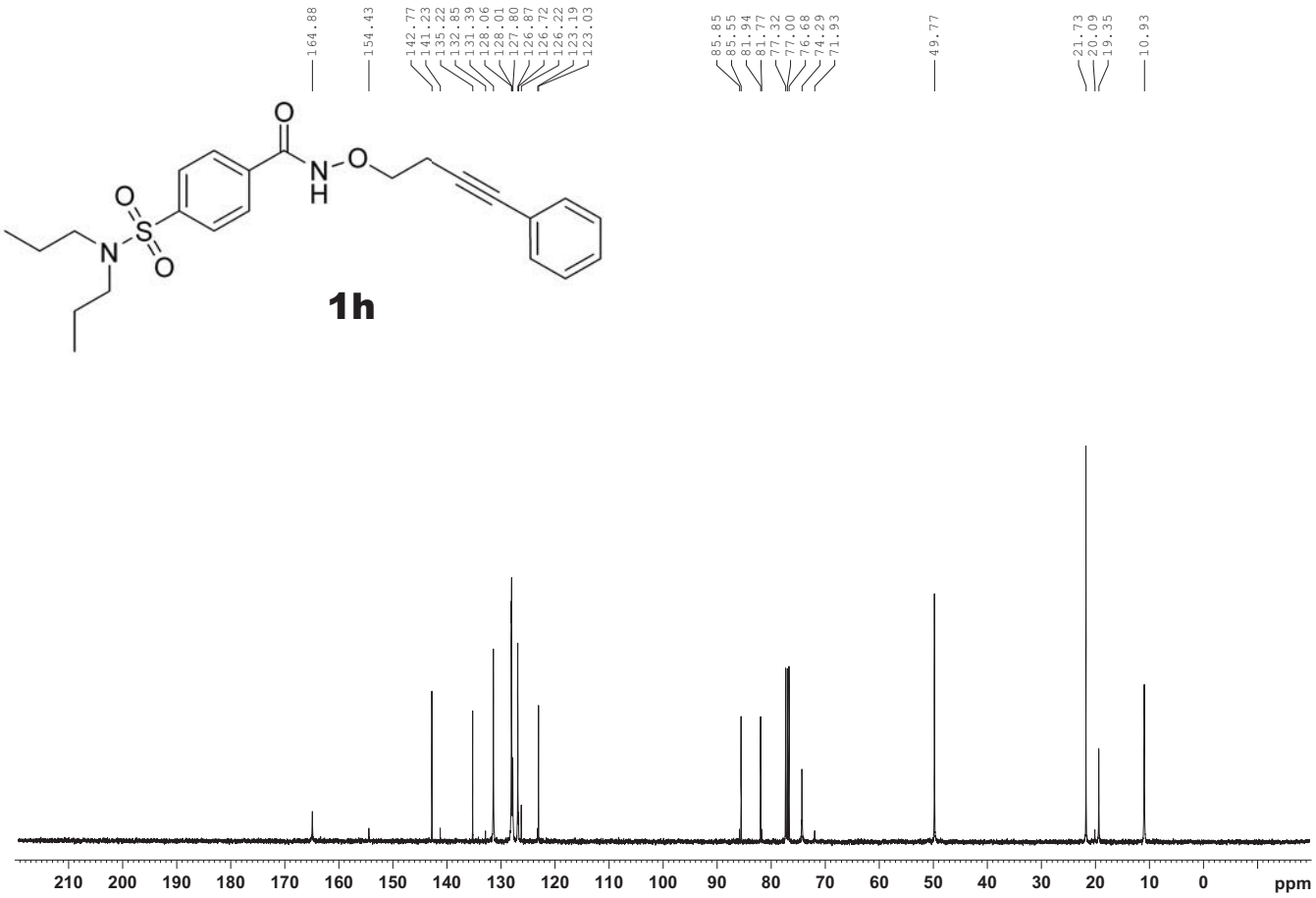
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123.19
123.03
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85.55
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81.77
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77.00
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71.93
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21.73
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19.35
10.93
1h
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200
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140
130
120
110
100
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60
50
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20
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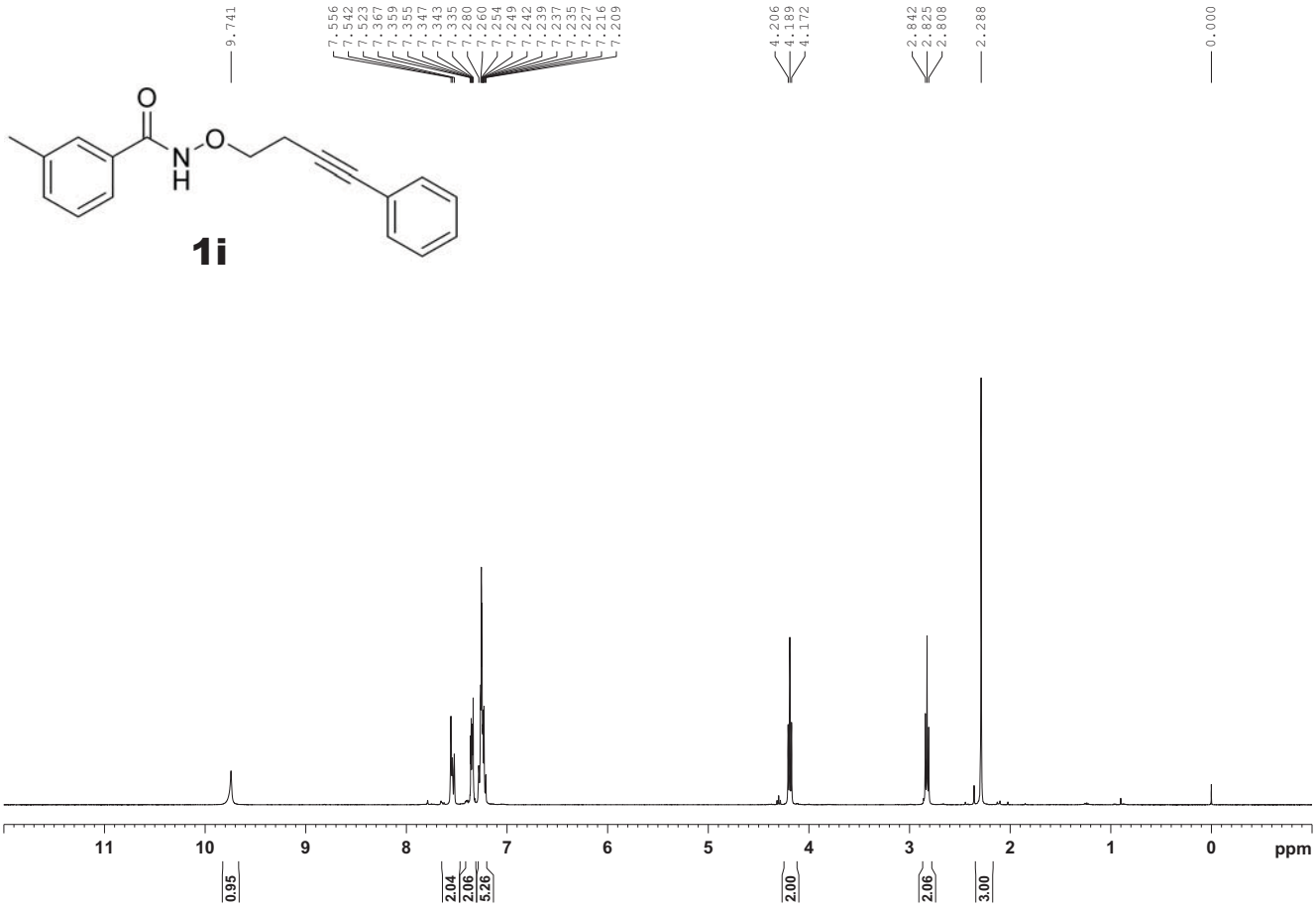
0
ppm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7.260
7.254
7.249
7.242
7.239
7.237
7.235
7.227
7.216
7.209
4.206
4.189
4.172
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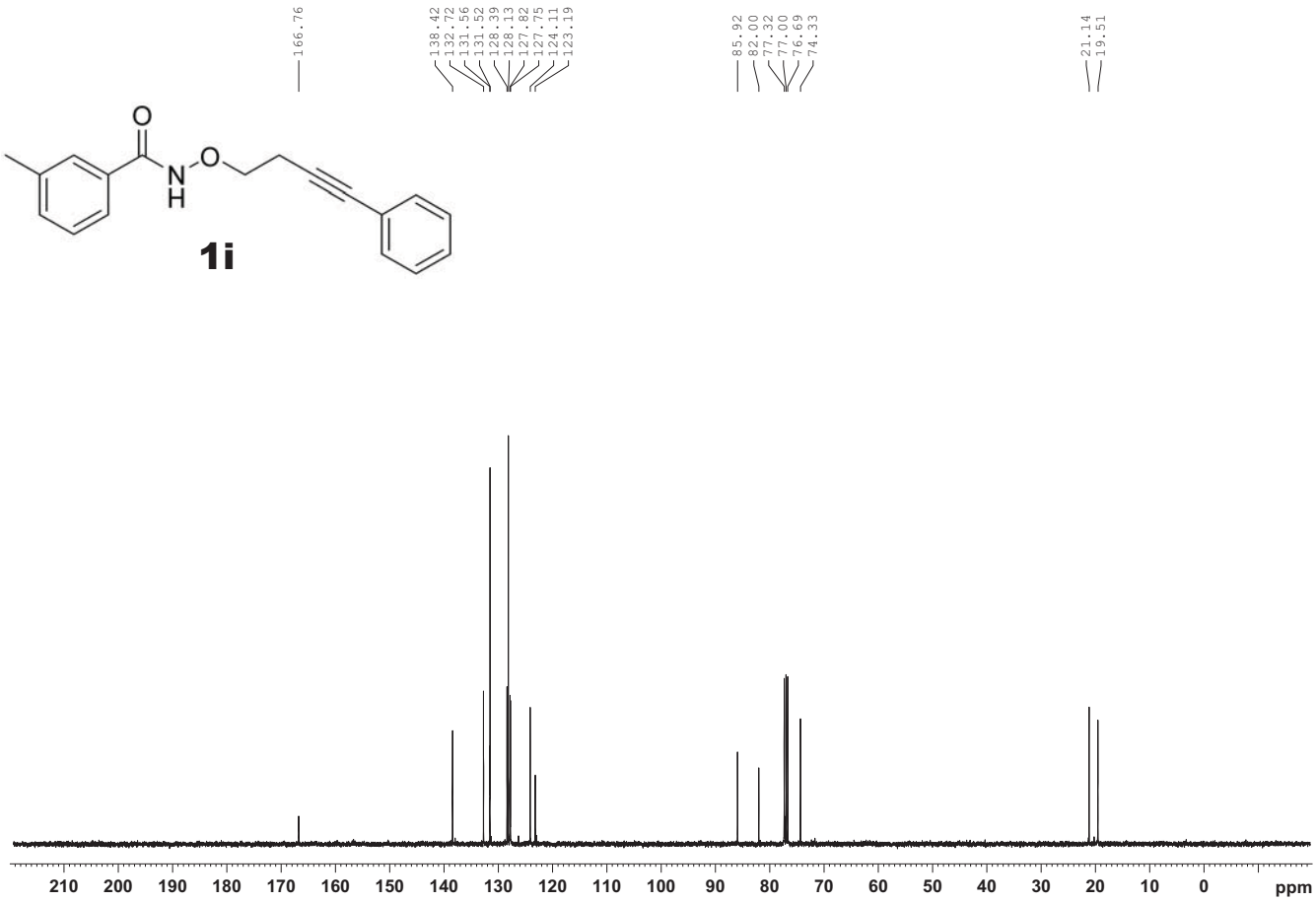
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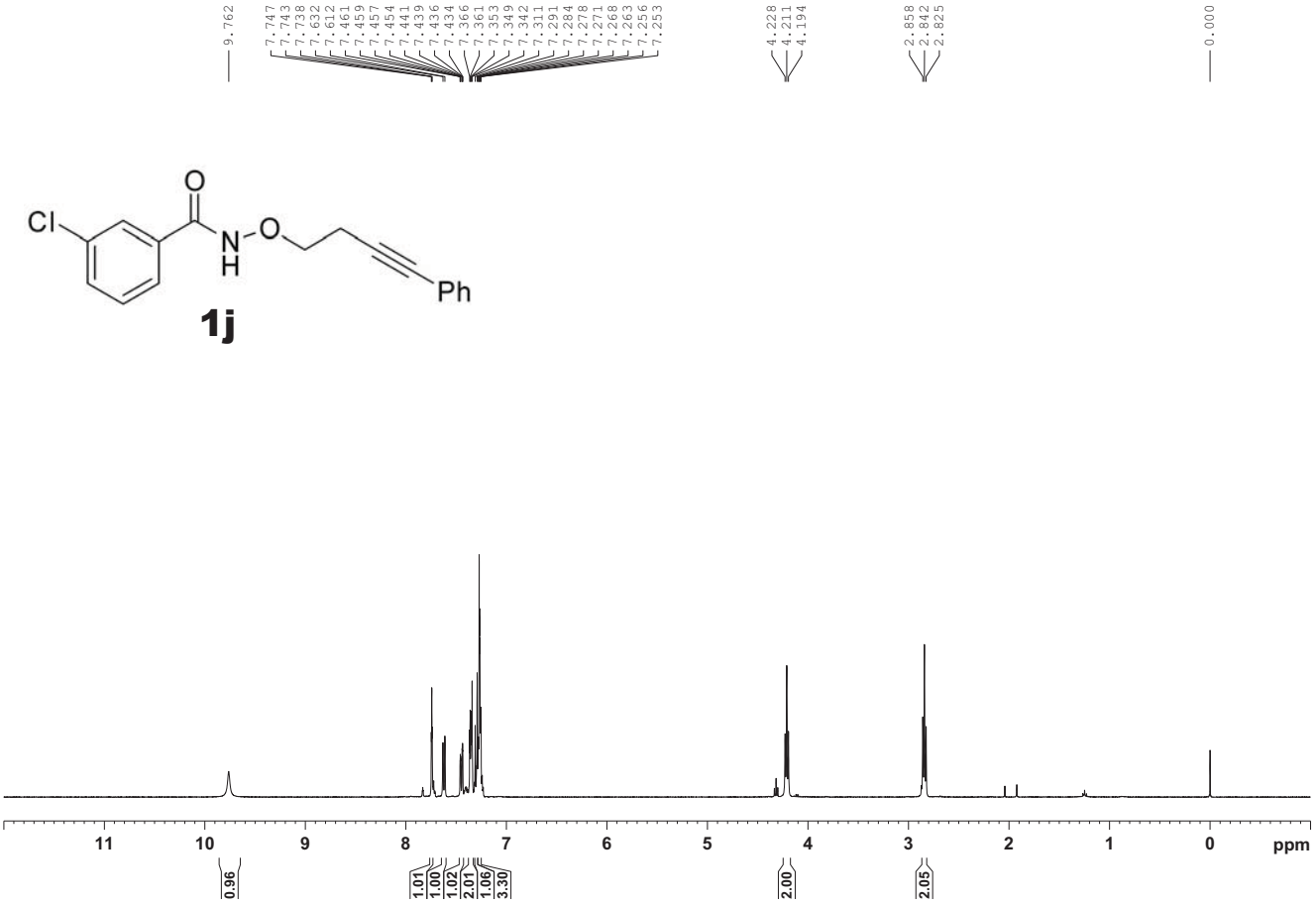
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7.291
7.284
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7.268
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N
H
O
Ph
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9
8
7
6
5
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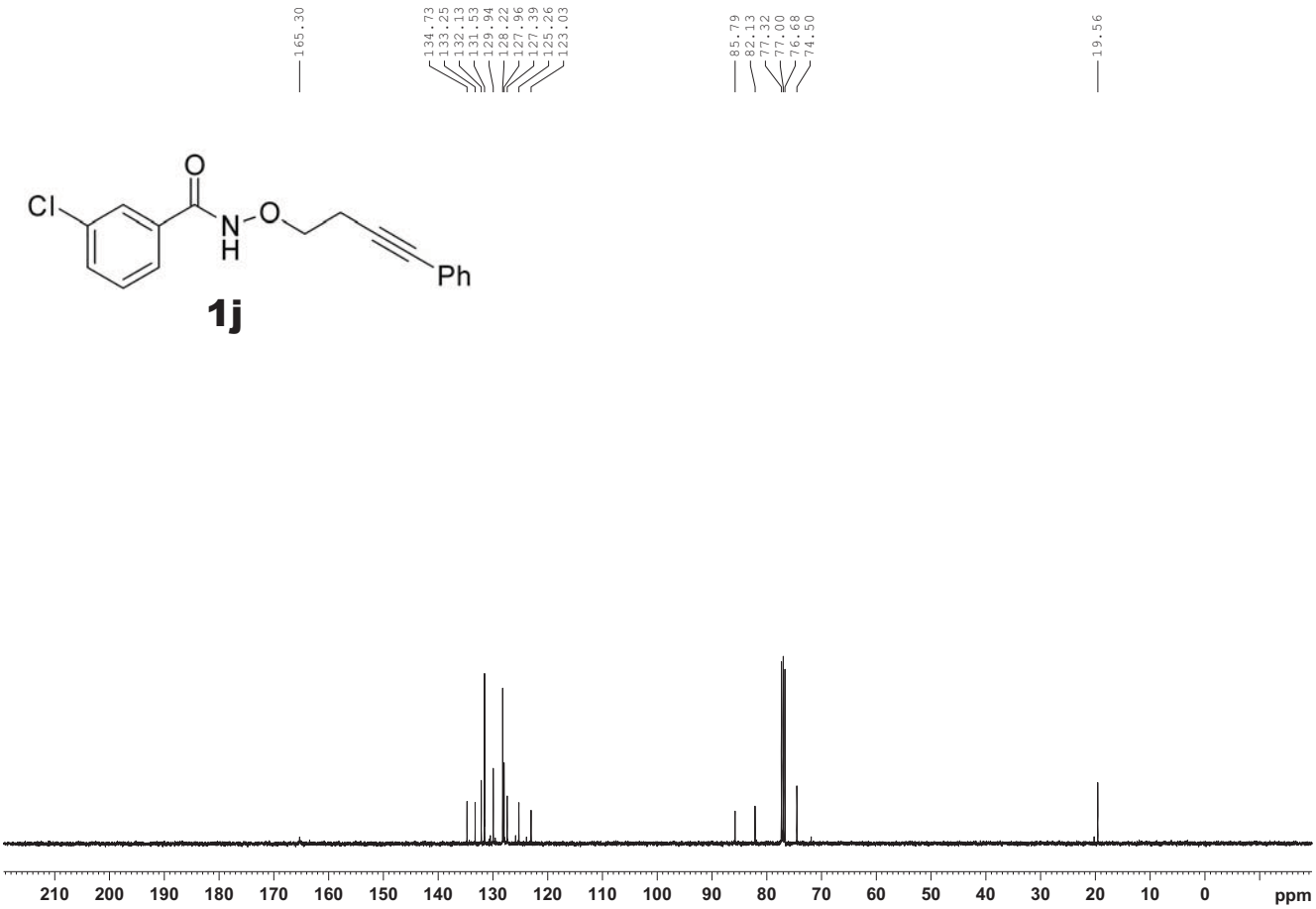
2.05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128.22
127.96
127.39
125.26
123.03
85.79
82.13
77.32
77.00
76.68
74.50
19.56
Cl
O
N
H
O
Ph
1j
210
200
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60
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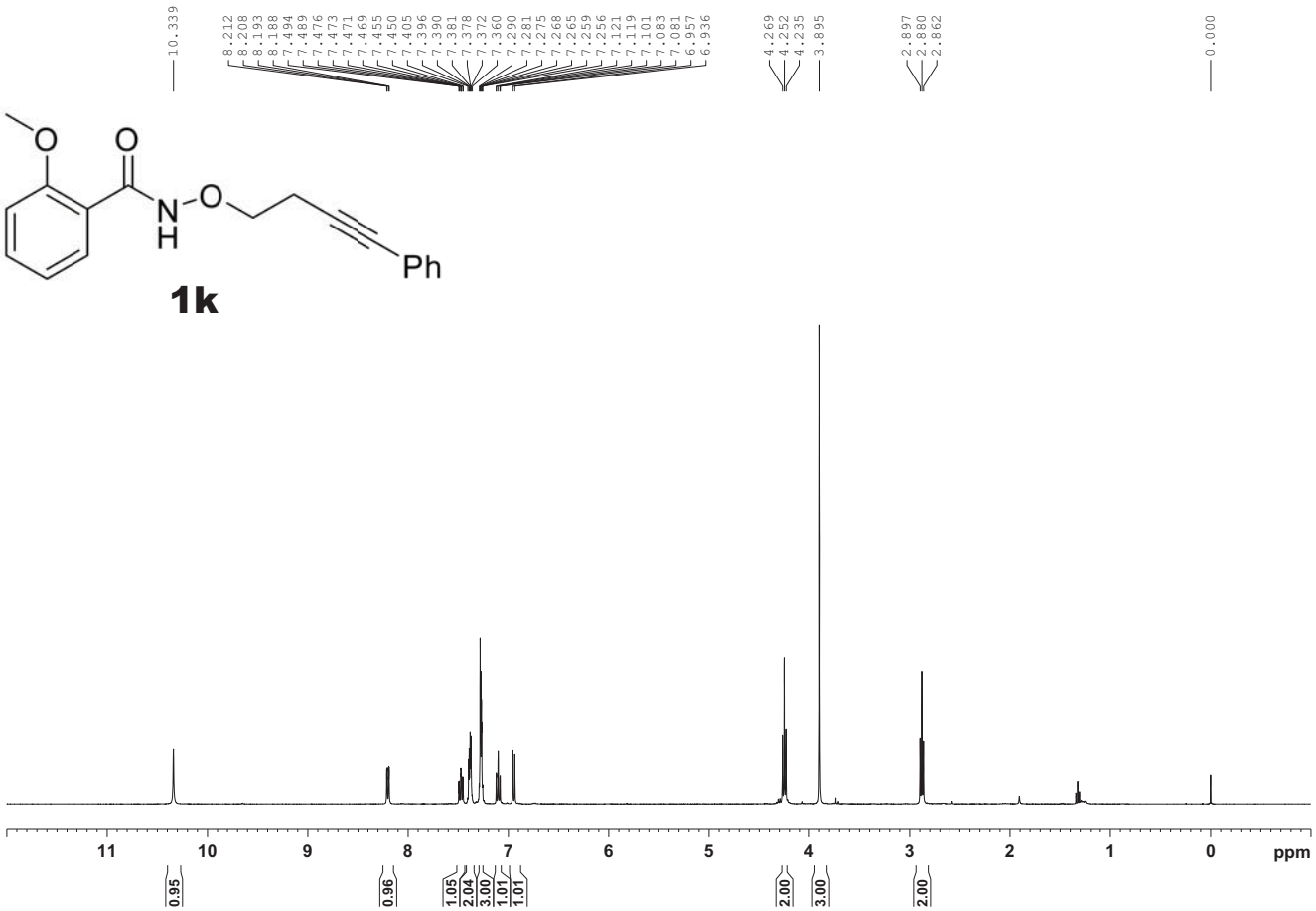
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7.469
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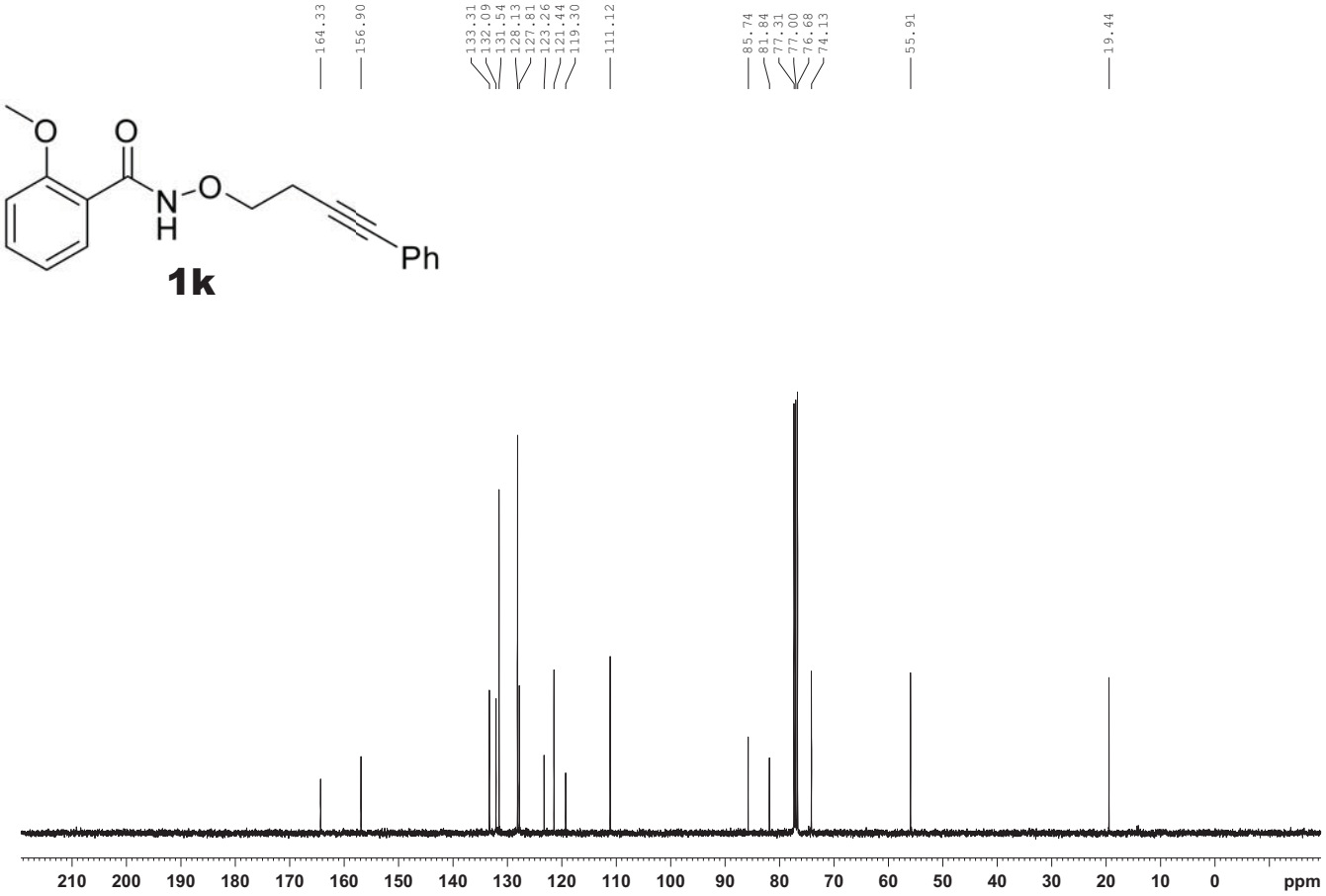
7.405
7.396
7.390
7.381
7.378
7.372
7.360
7.290
7.281
7.275
7.268
7.265
7.259
7.256
7.121
7.119
7.101
7.083
7.081
6.957
6.936
4.269
4.252
4.235
3.895
2.897
2.880
2.862
0.000
O
O
N
O
H
Ph
1k
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
0.96
1.05
2.04
3.00
1.01
1.01
2.00
3.00
2.00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77.00
76.68
74.13
55.91
19.44
O
O
N
O
H
Ph
1k
210
200
190
180
170
160
150
140
130
120
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Cl
O
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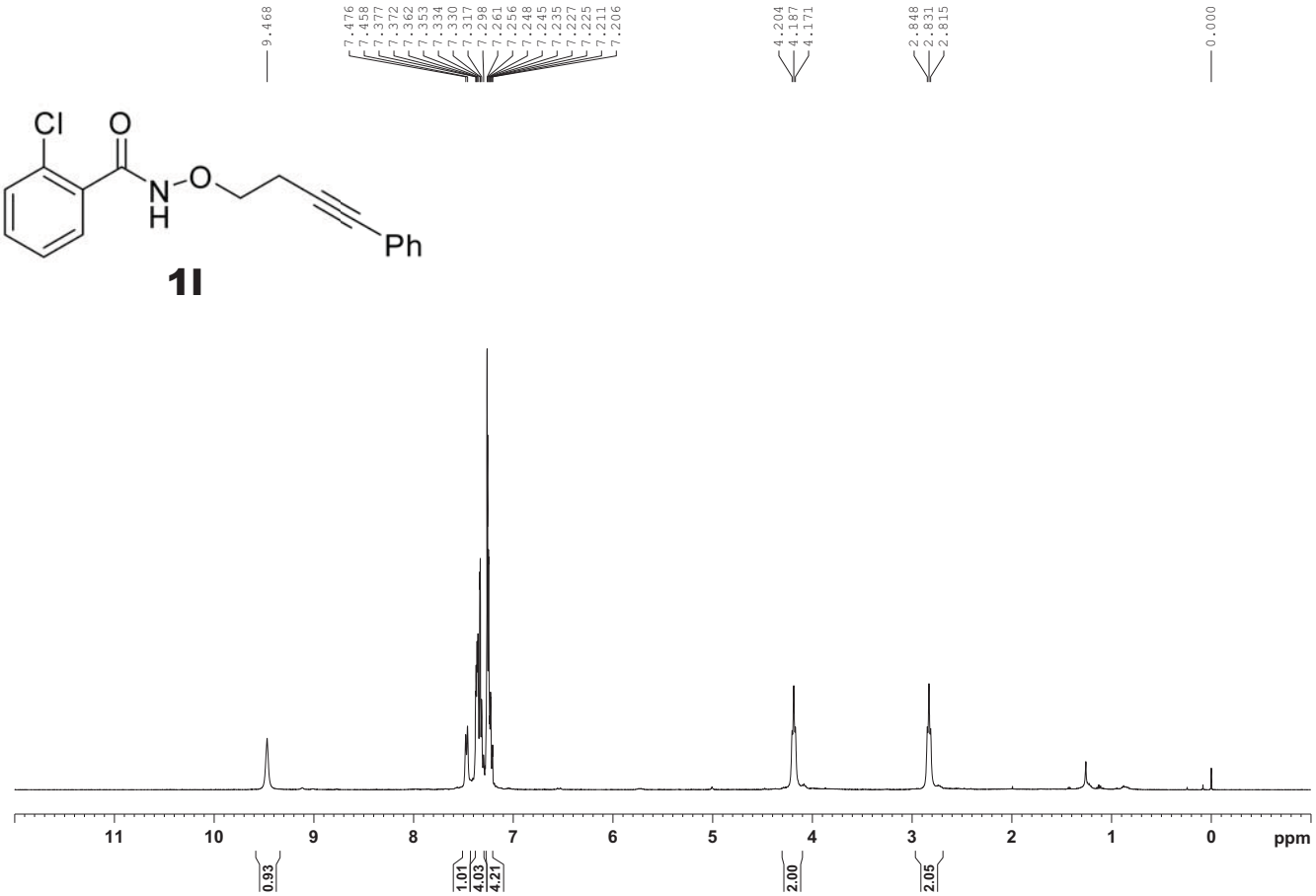
O
H
Ph

1l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7.245
7.235
7.227
7.225
7.211
7.206
4.204
4.187
4.171
2.848
2.831
2.815
0.000
11
10
9
8
7
6
5
4
3
2
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4.21
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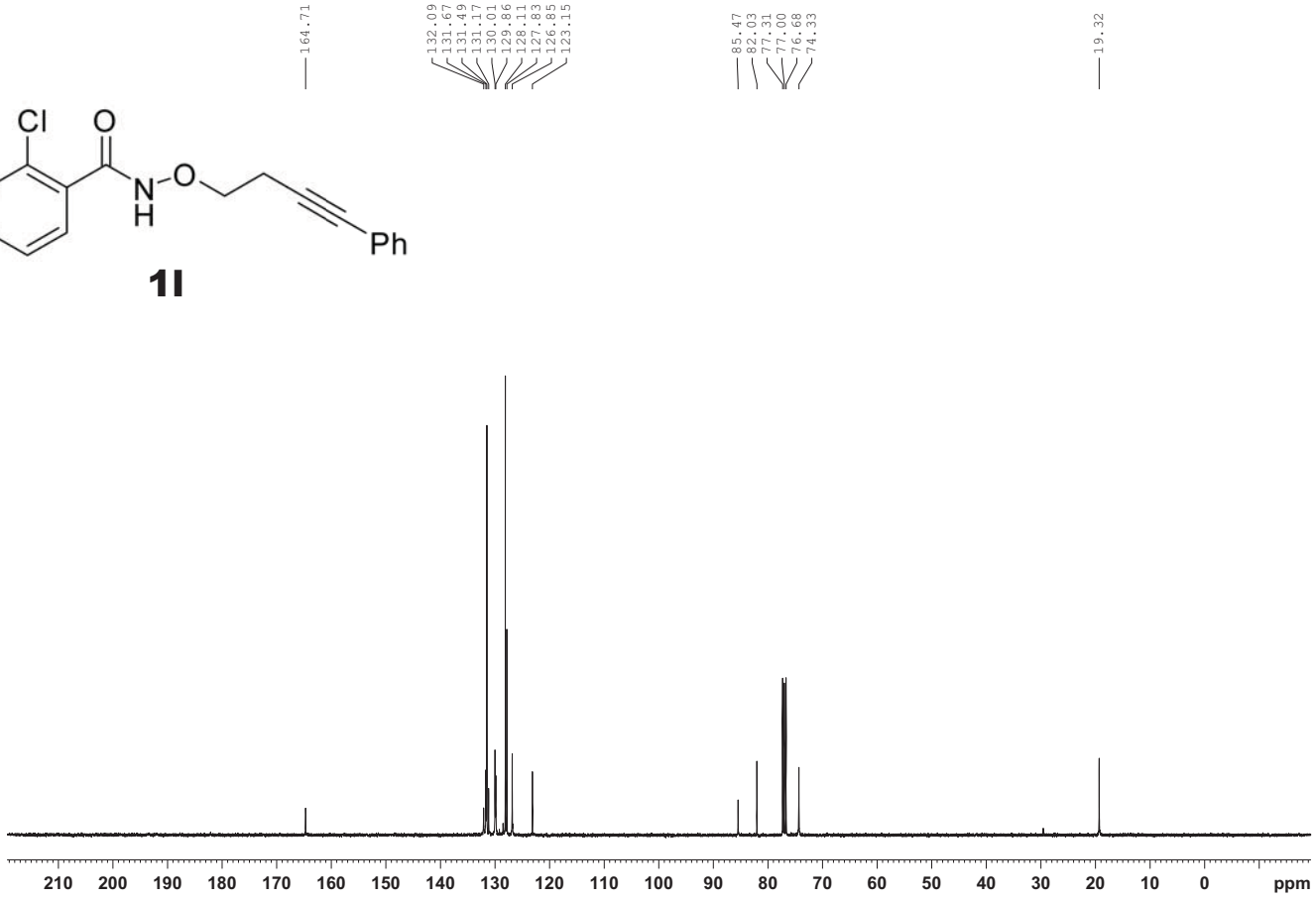
2.05

Cl
O
N
O
H
Ph

1l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76.68
74.33
19.32
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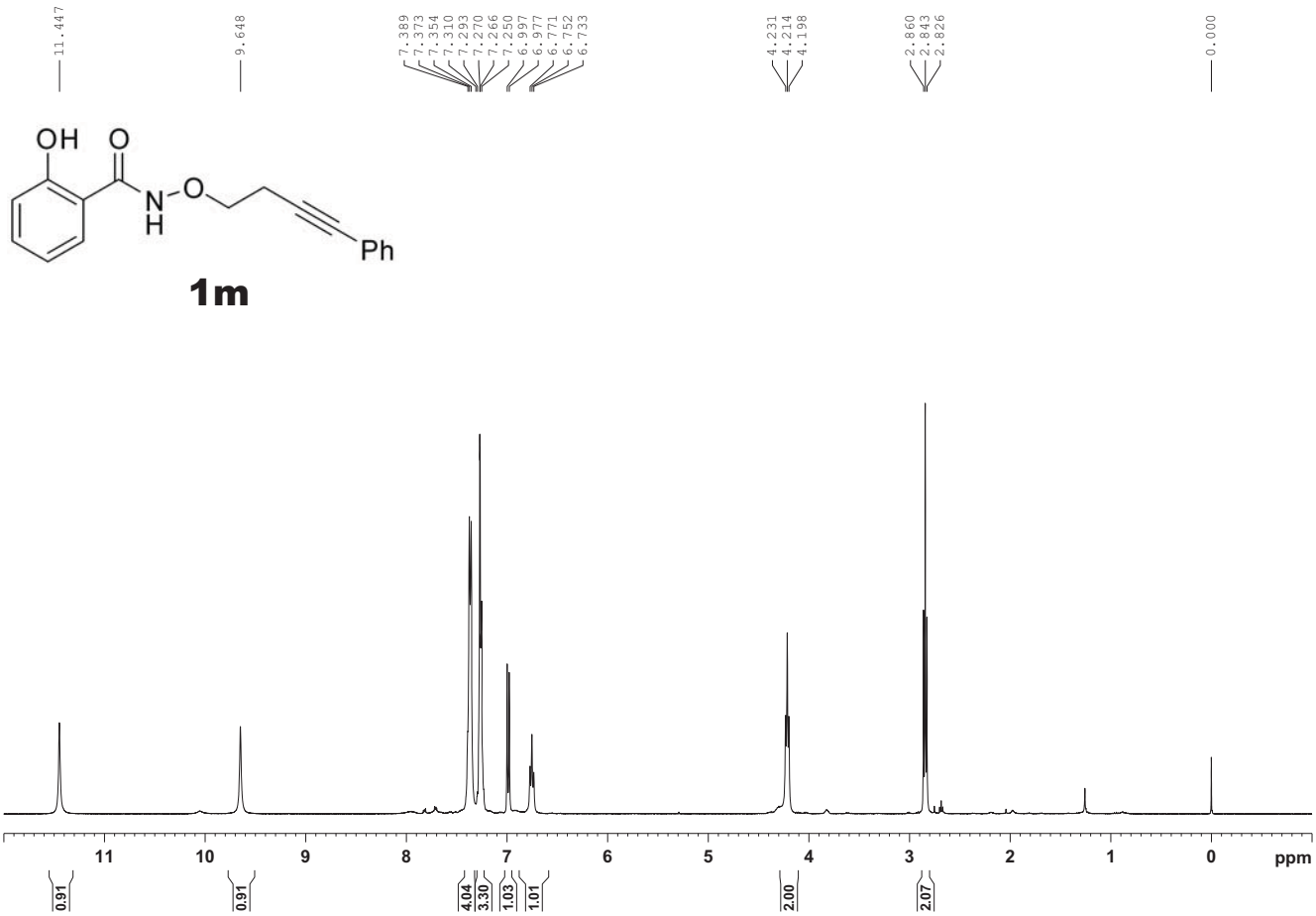
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6.752
6.733
4.231
4.214
4.198
2.860
2.843
2.826
0.000
OH
O
N
O
H
Ph
1m
11
10
9
8
7
6
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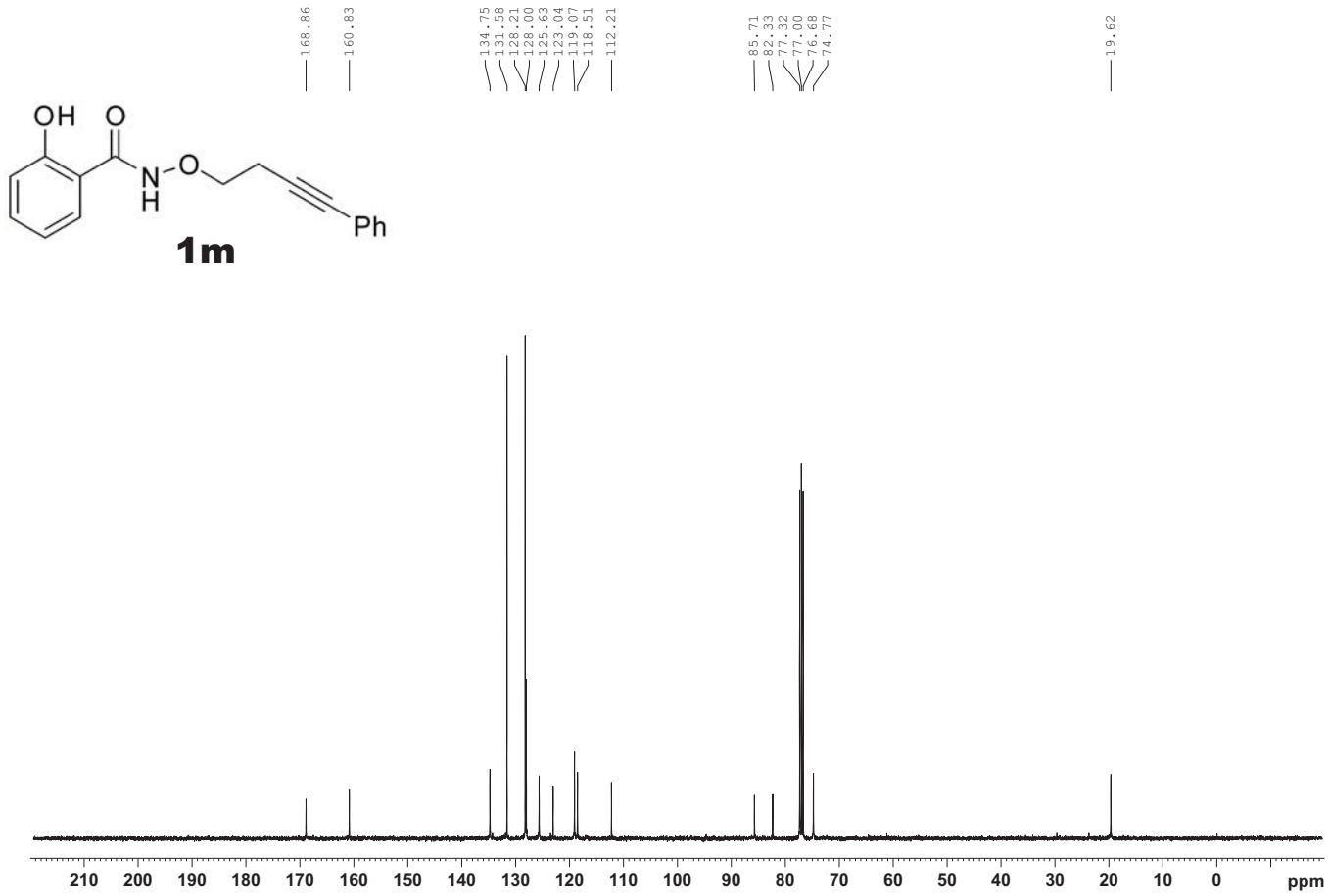
2.07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82.33
77.32
77.00
76.68
74.77
19.62
OH
O
N
O
H
Ph
1m
210
200
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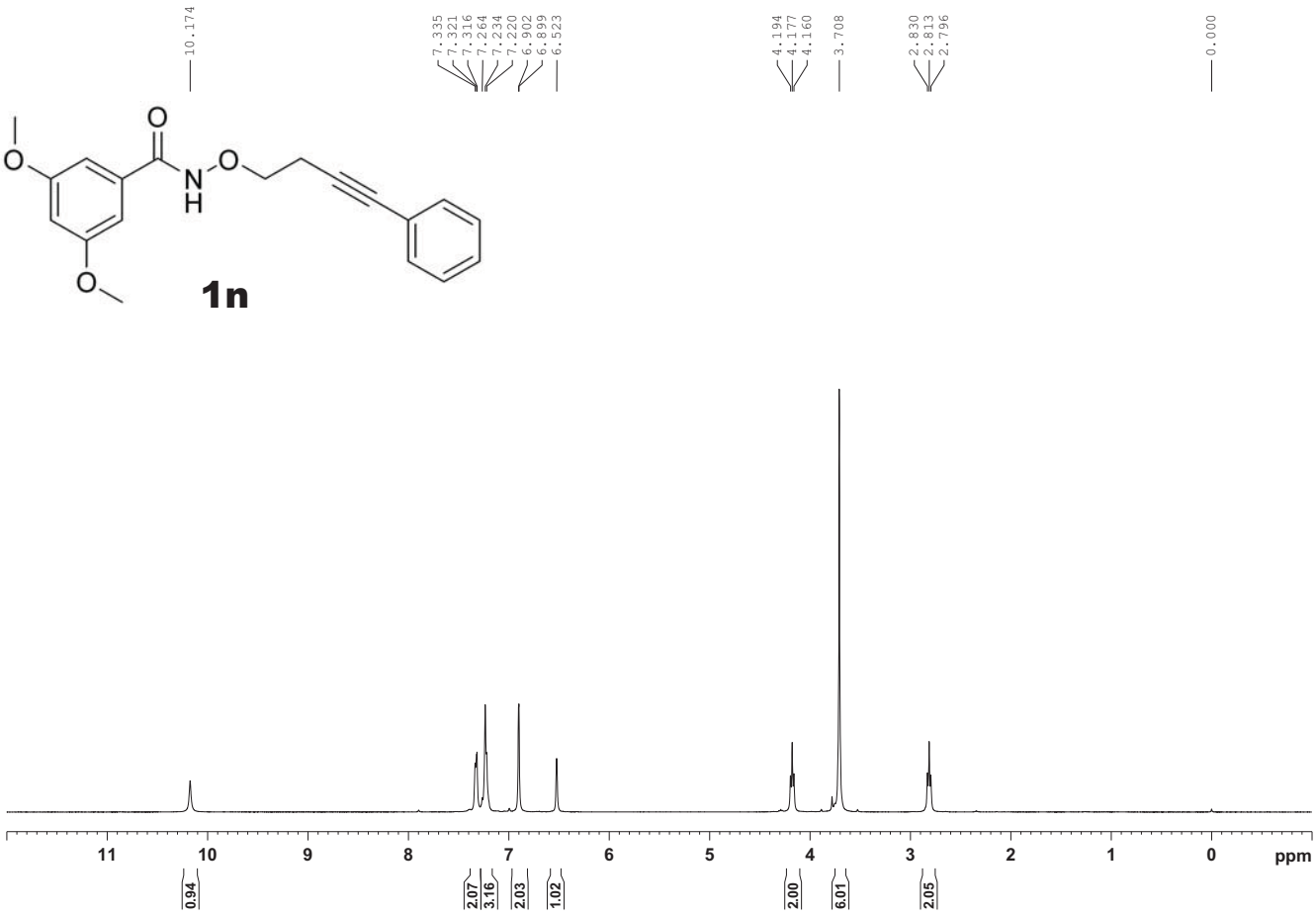
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2.830
2.813
2.796
0.000
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11
10
9
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6
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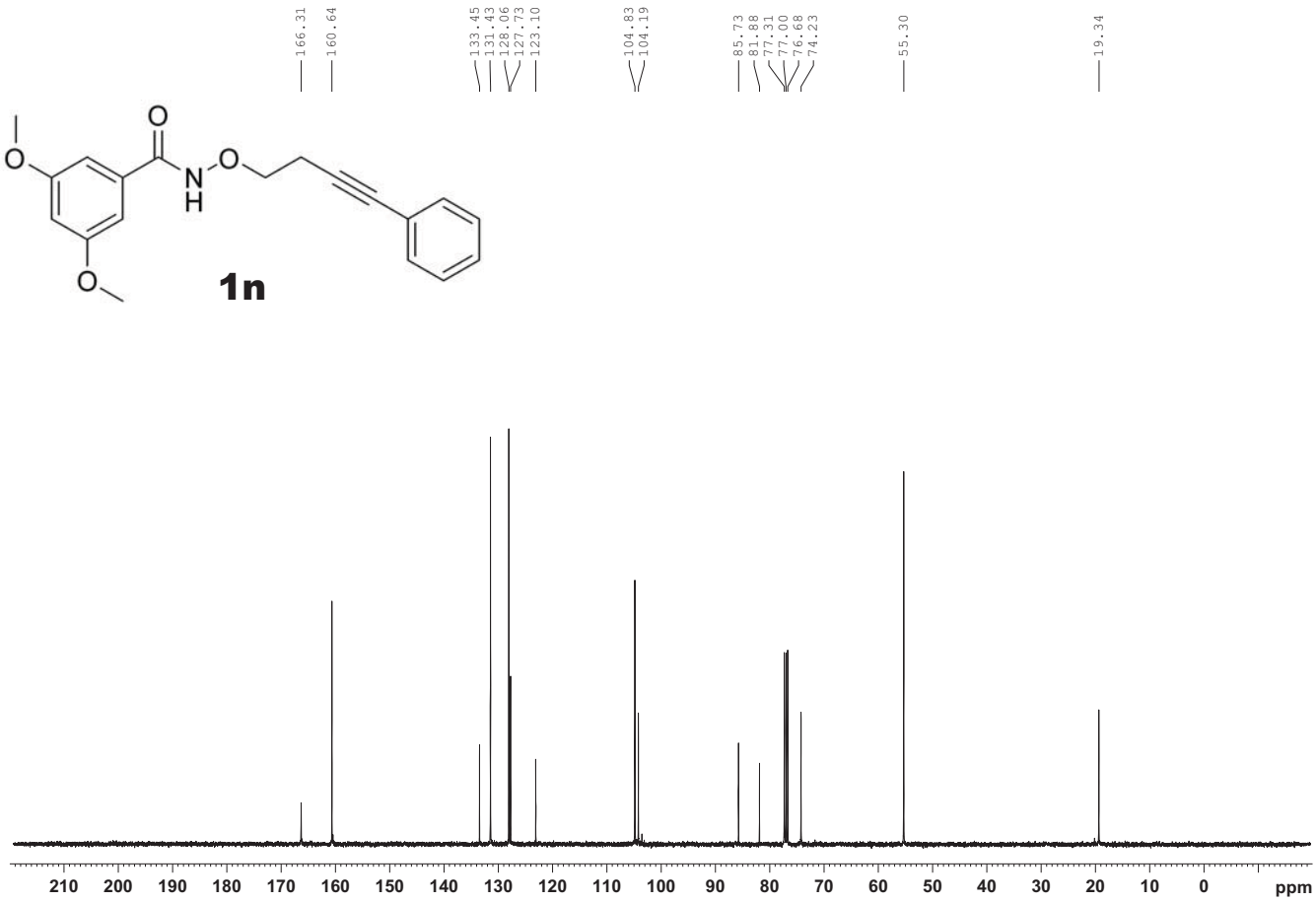
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104.83
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77.00
76.68
74.23
55.30
19.34
1n
210
200
190
180
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10
0
ppm

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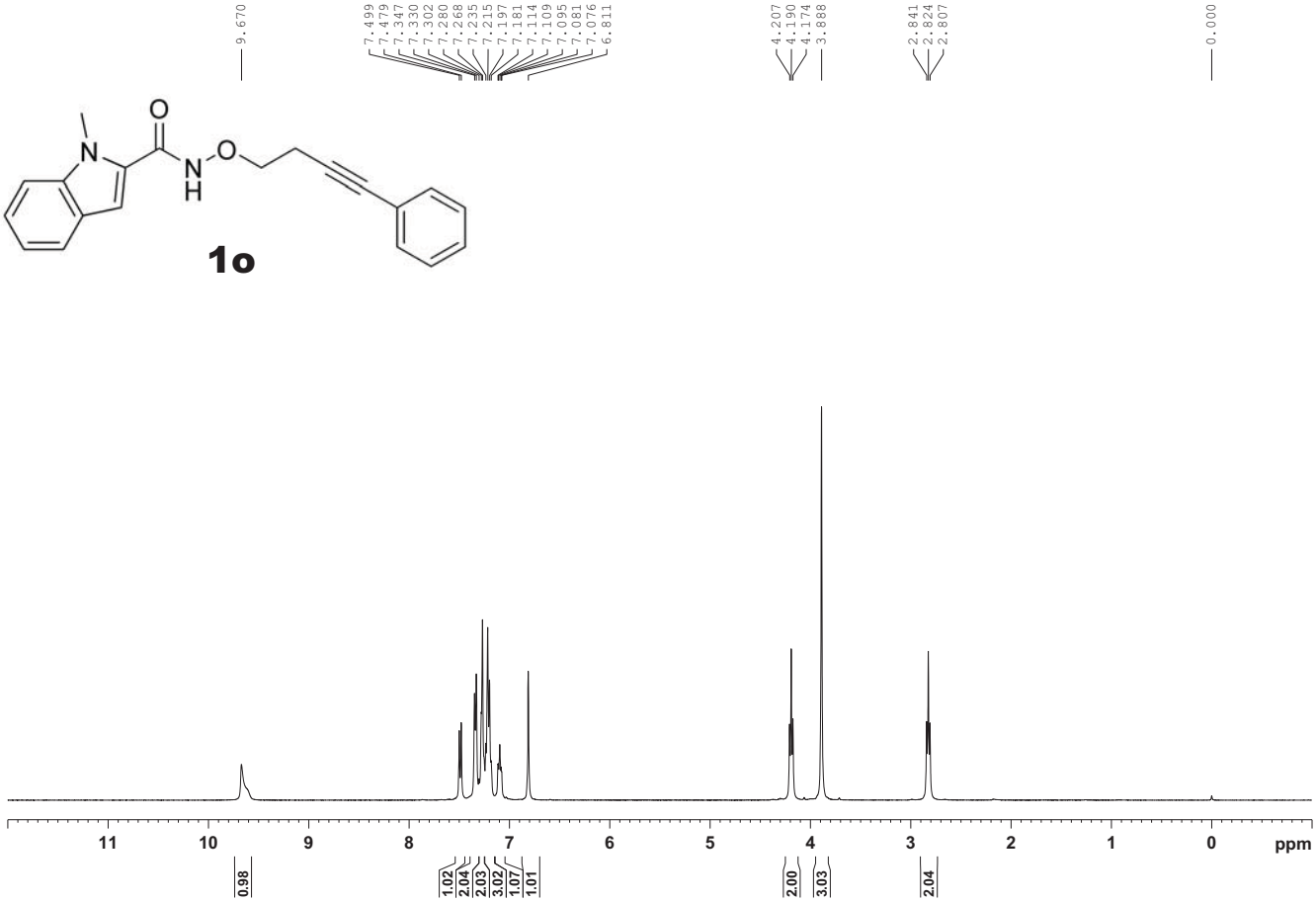
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7.114
7.109
7.095
7.081
7.076
6.811
4.207
4.190
4.174
3.888
2.841
2.824
2.807
0.000
11
10
9
8
7
6
5
4
3
2
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3.02
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1.01
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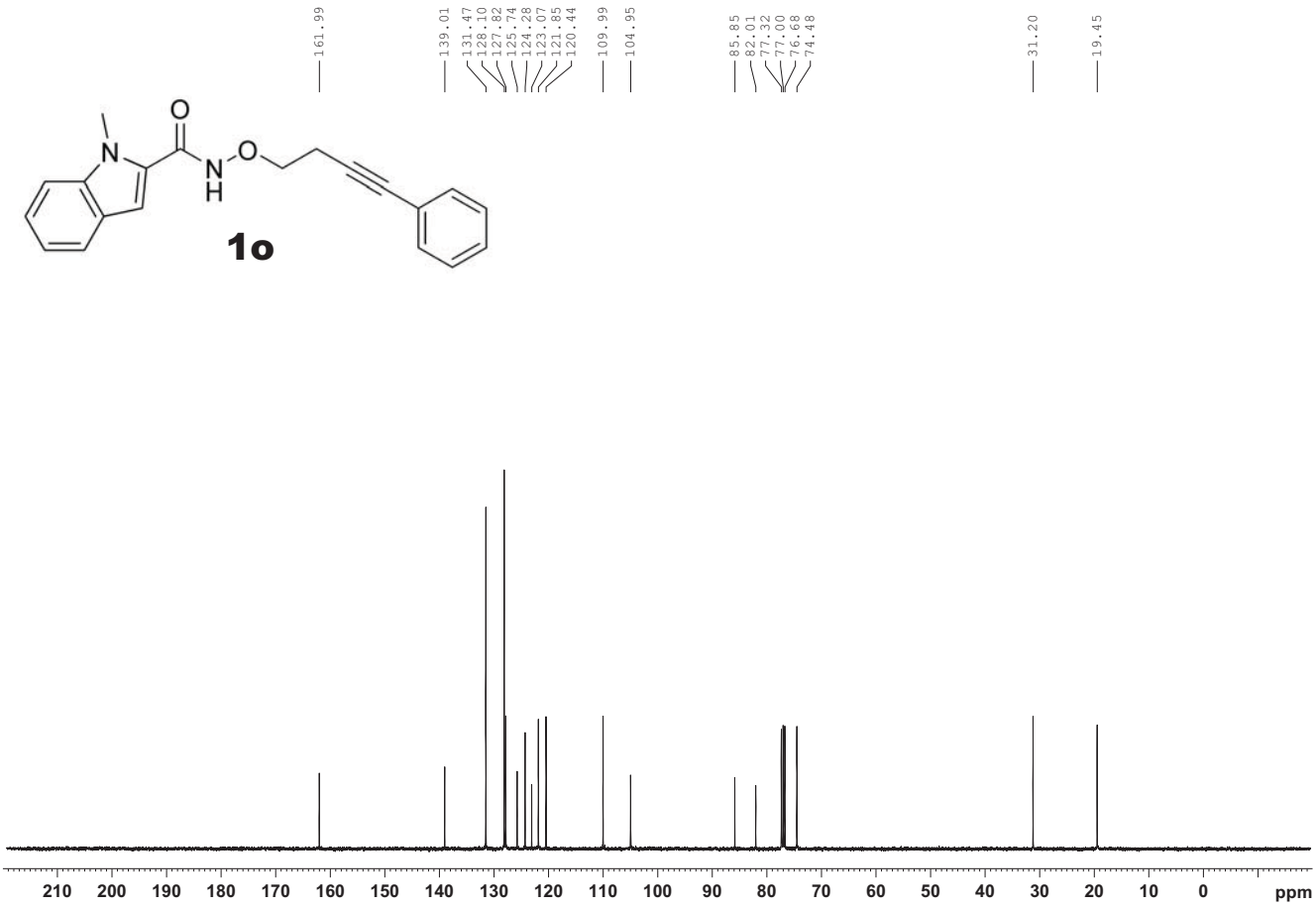
2.04

1o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82.01
77.32
77.00
76.68
74.48
31.20
19.45
210
200
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140
130
120
110
100
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60
50
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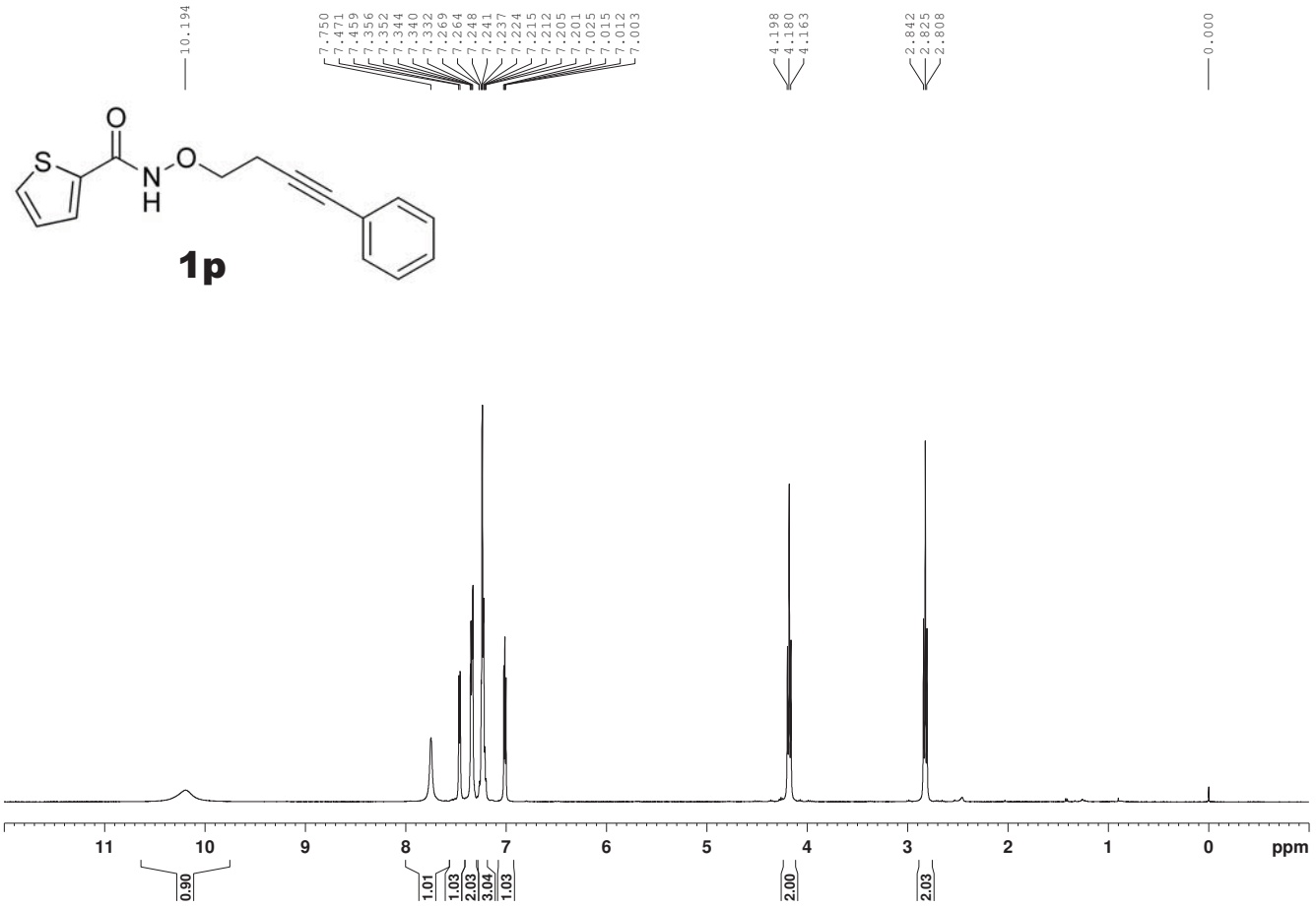
30
20
10
0
ppm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7.340
7.332
7.269
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7.241
7.237
7.224
7.215
7.212
7.205
7.201
7.025
7.015
7.012
7.003
4.198
4.180
4.163
2.842
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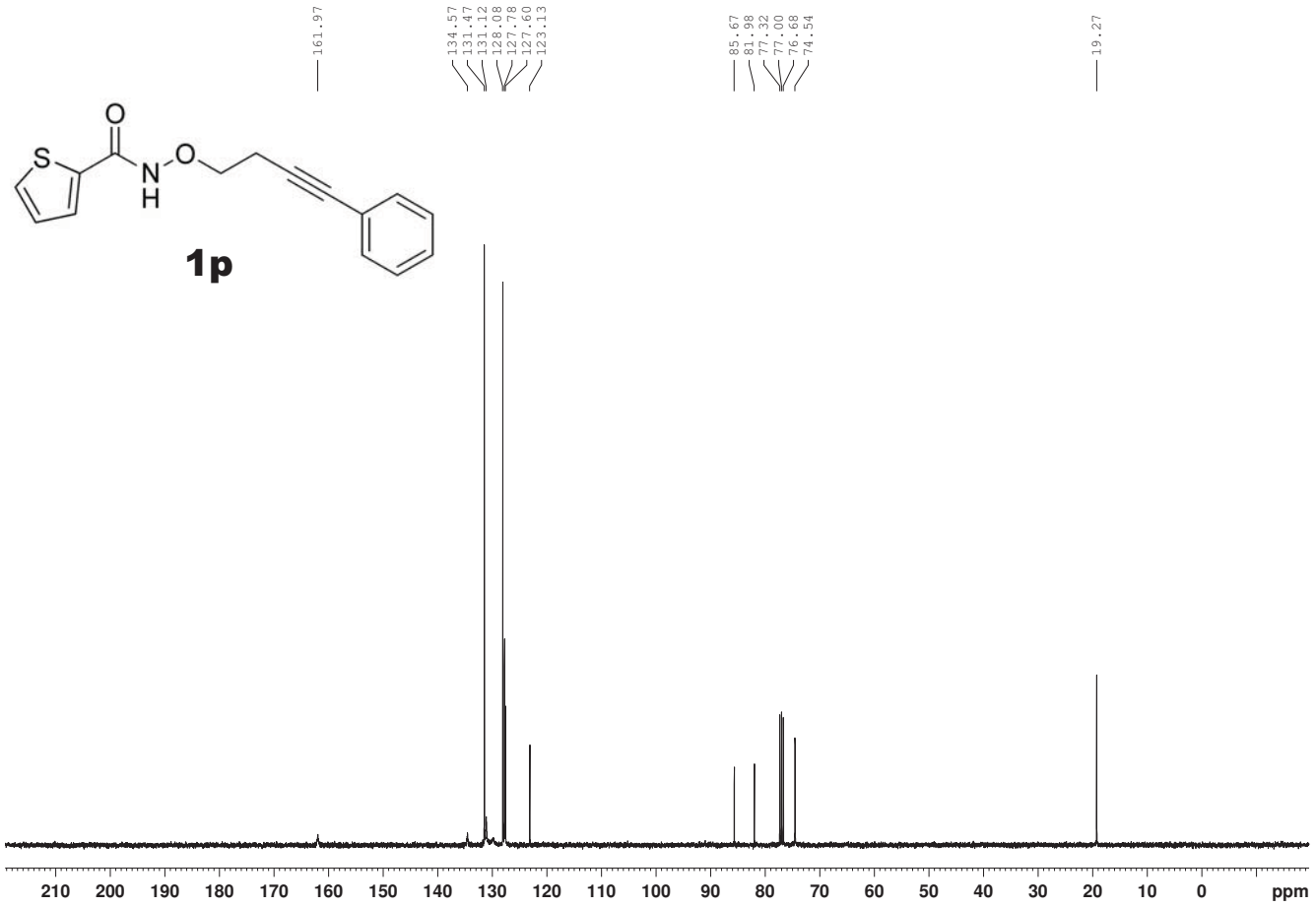
3.04
1.03
2.00
2.03
ppm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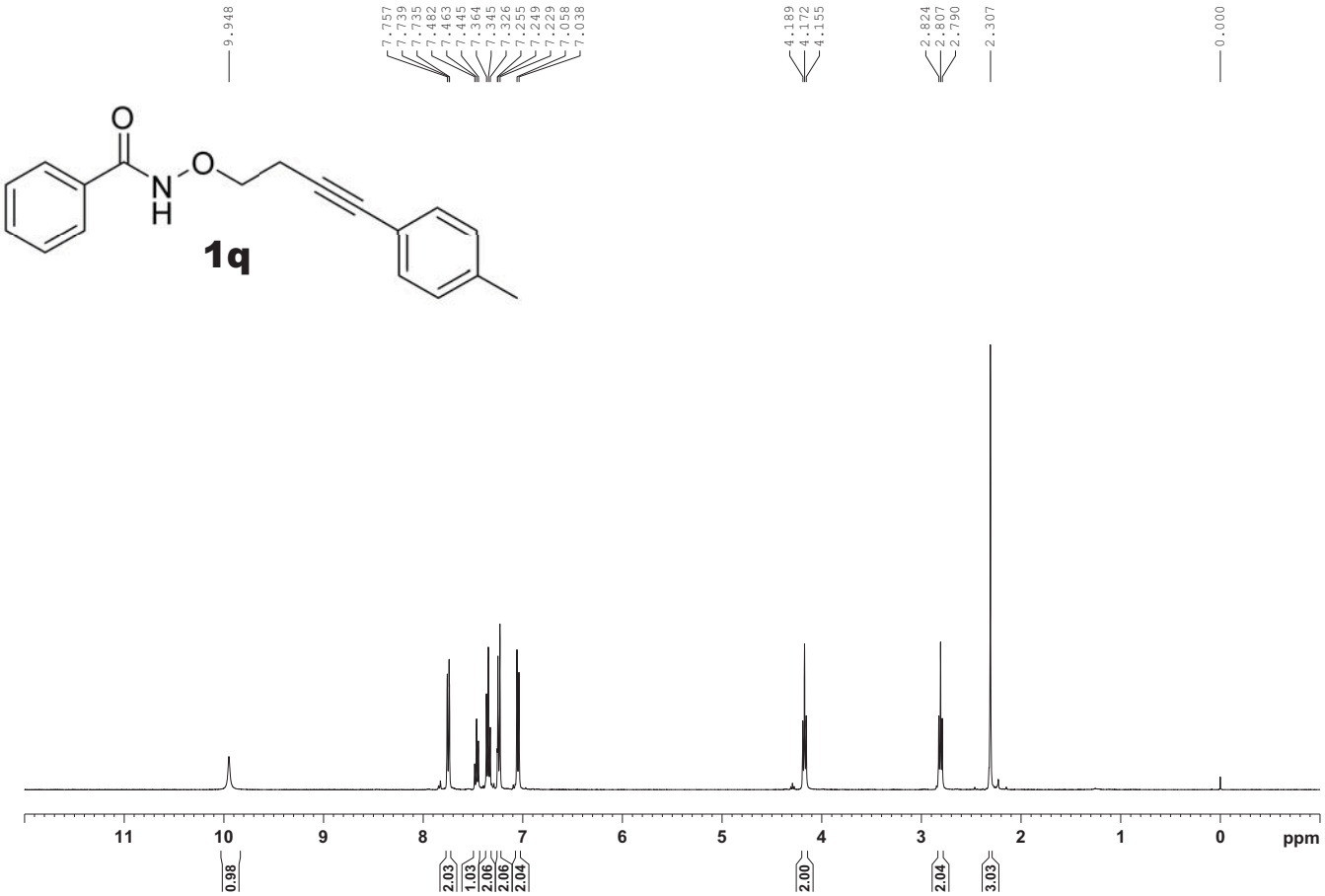
1p
ppm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7.229
7.058
7.038
4.189
4.172
4.155
2.824
2.807
2.790
2.307
0.000
1q
11
10
9
8
7
6
5
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ppm
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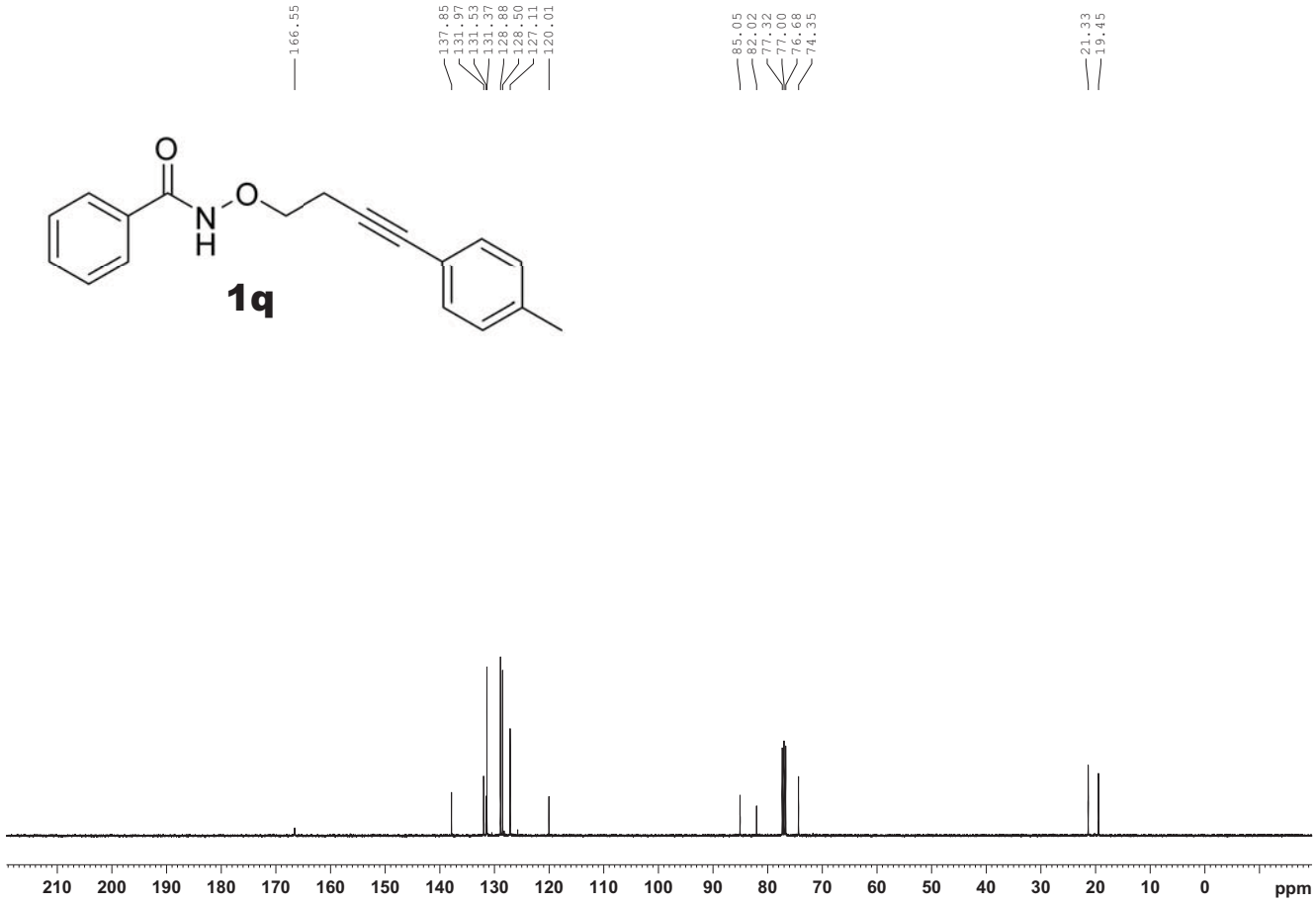
1.03
2.06
2.06
2.04
2.00
2.04
3.03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76.68
74.35
21.33
19.45
1q
210
200
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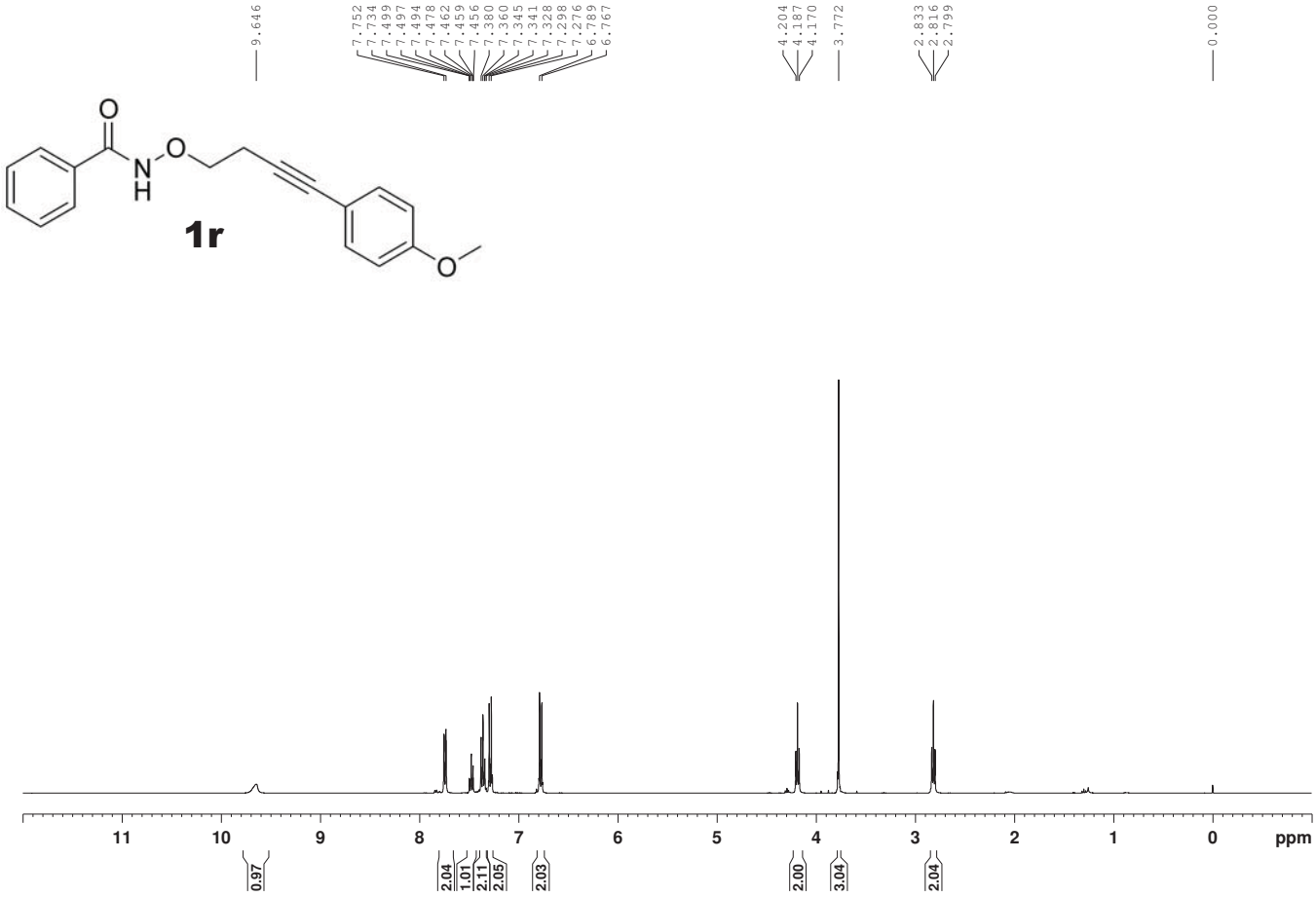
ppm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7.345
7.341
7.328
7.298
7.276
6.789
6.767
4.204
4.187
4.170
3.772
2.833
2.816
2.799
0.000
1r
11
10
9
8
7
6
5
4
3
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ppm
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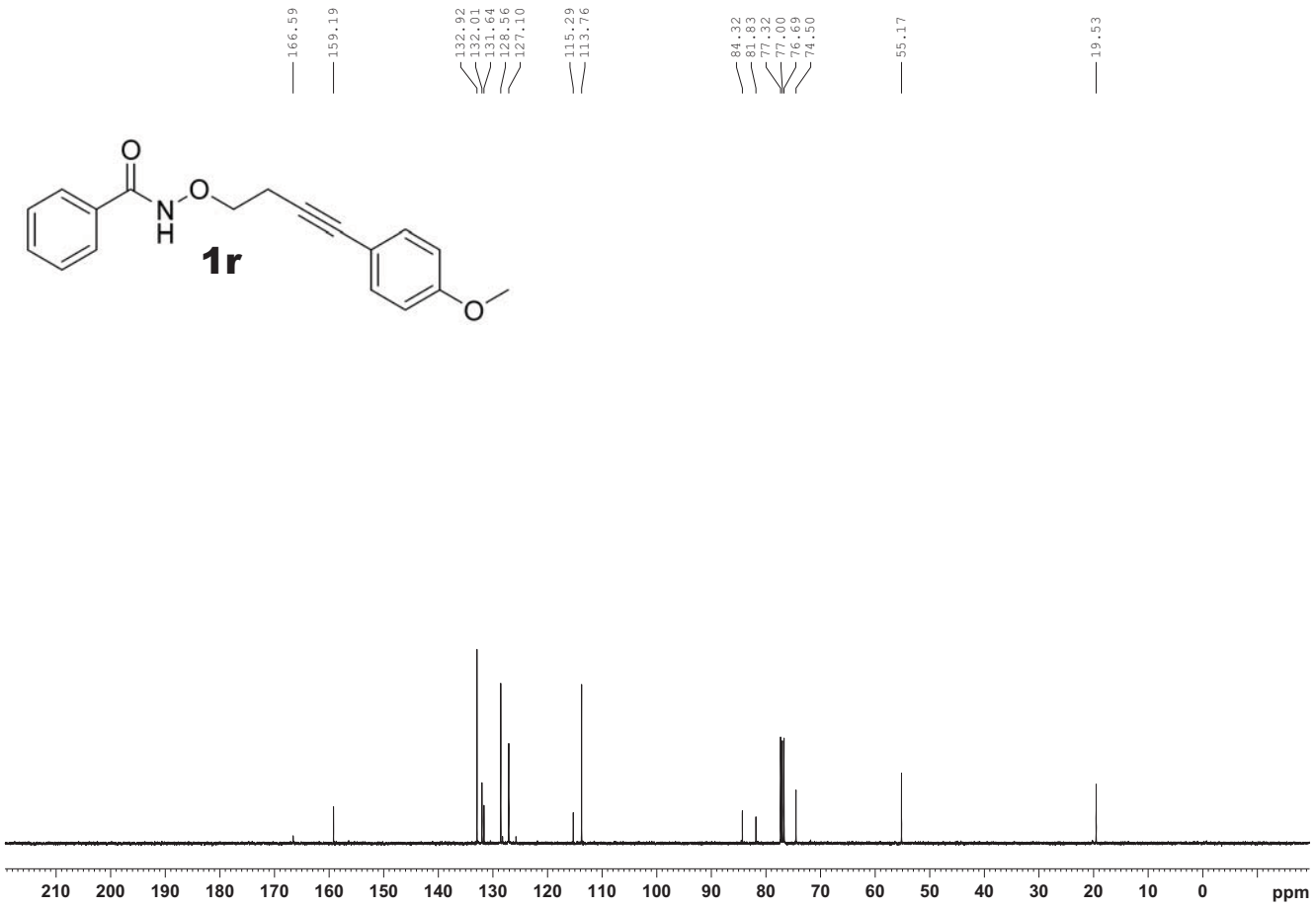
2.00
3.04
2.04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77.00
76.69
74.50
55.17
19.53
1r
210
200
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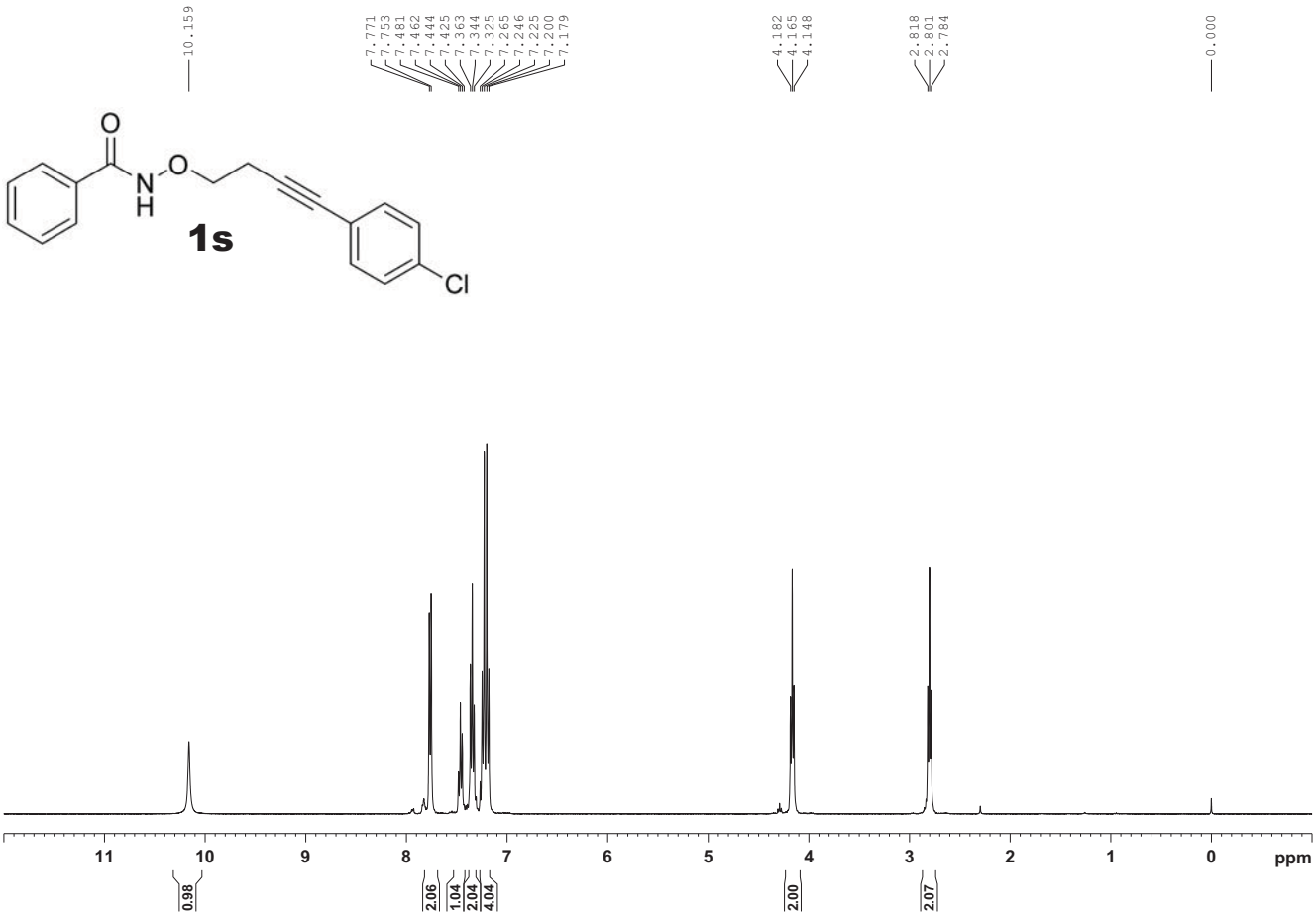
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7.200
7.179
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4.165
4.148
2.818
2.801
2.784
0.000
1s
Cl
11
10
9
8
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2
1
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ppm
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1.04
2.04
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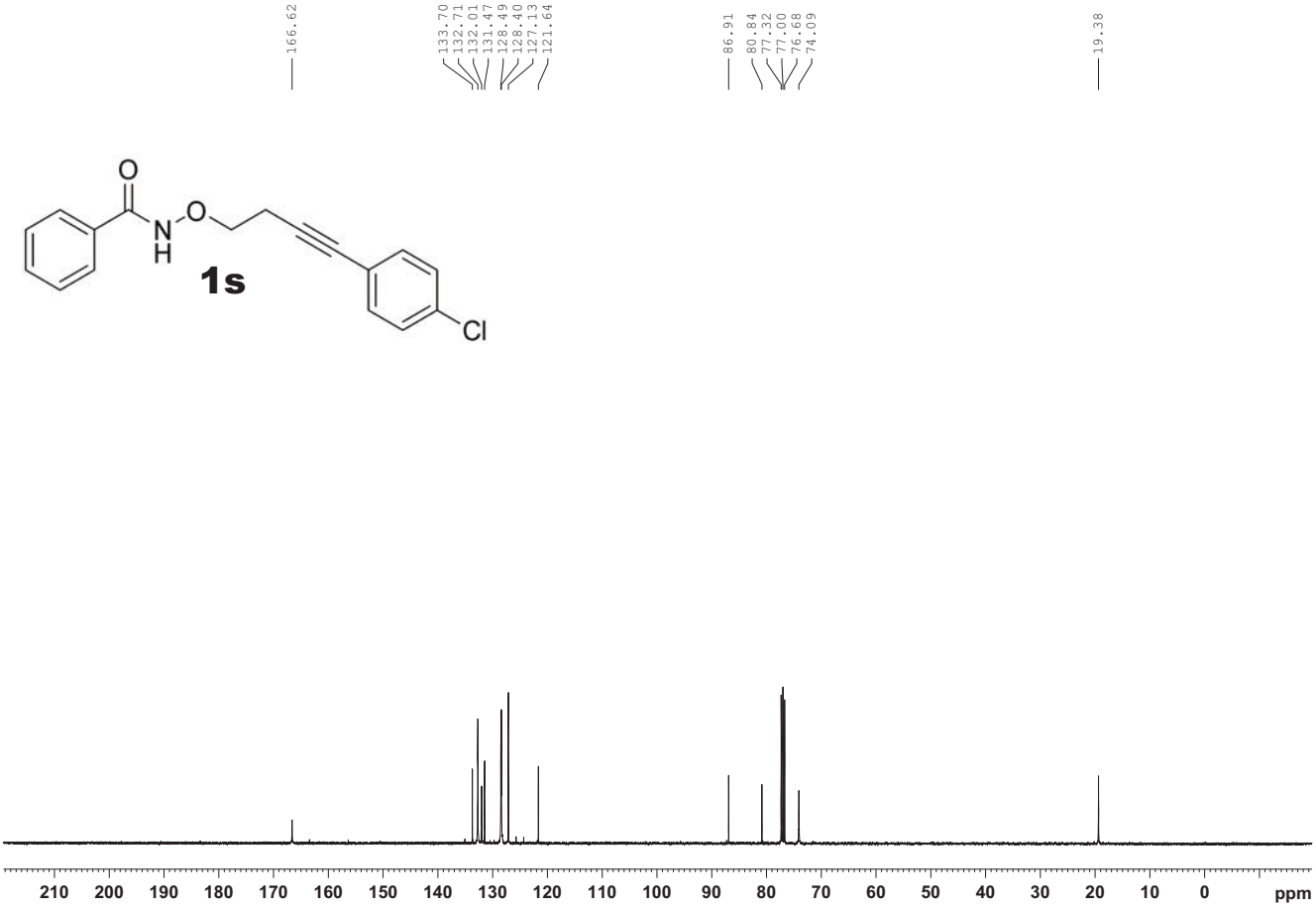
2.07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77.00
76.68
74.09
19.38
1s
Cl
210
200
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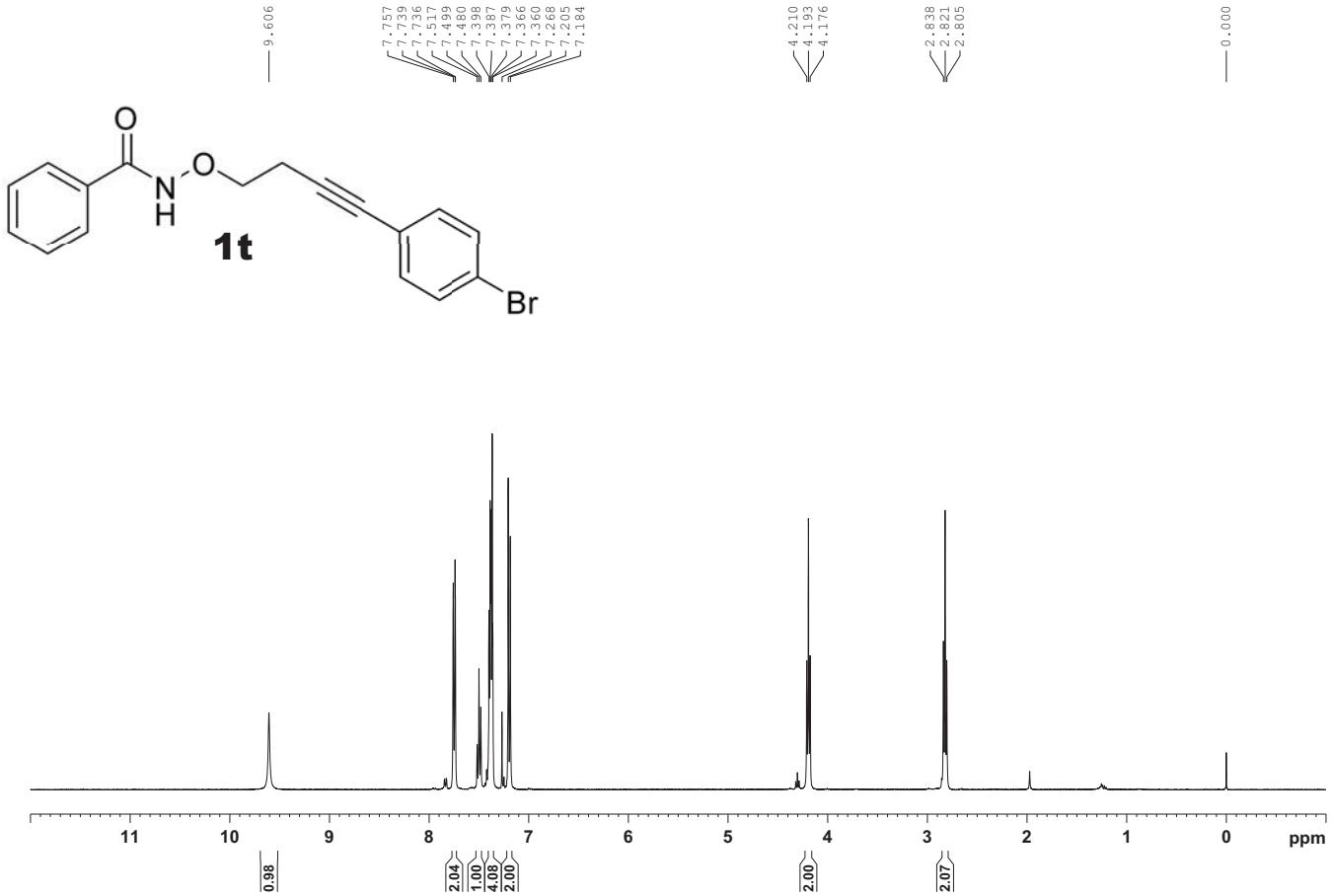
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7.360
7.268
7.205
7.184
4.210
4.193
4.176
2.838
2.821
2.805
0.000
O
N
H
O
1t
Br
11
10
9
8
7
6
5
4
3
2
1
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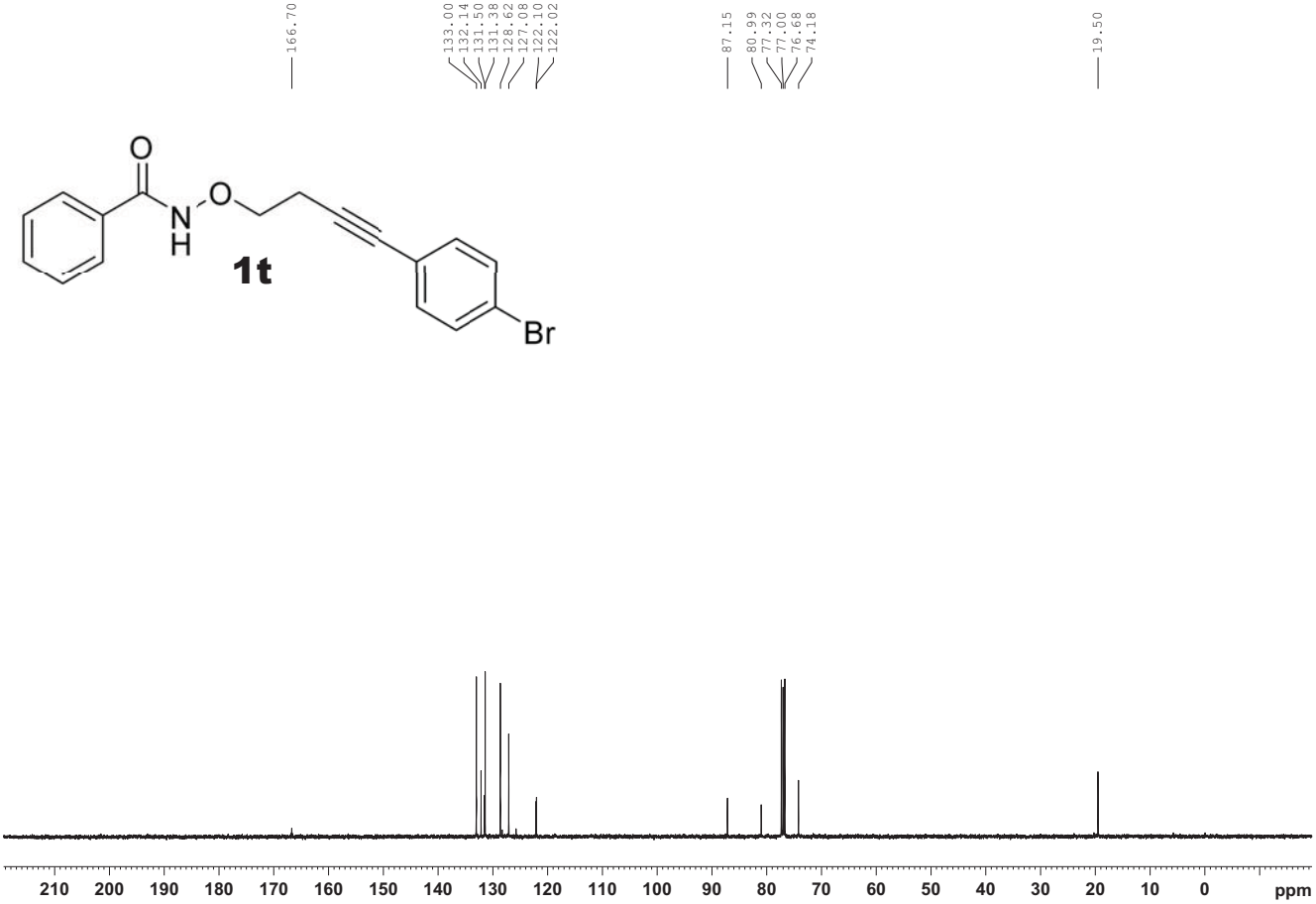
ppm
0.98
2.04
1.00
4.08
2.00
2.00
2.07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77.32
77.00
76.68
74.18
19.50
O
N
H
O
1t
Br
210
200
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60
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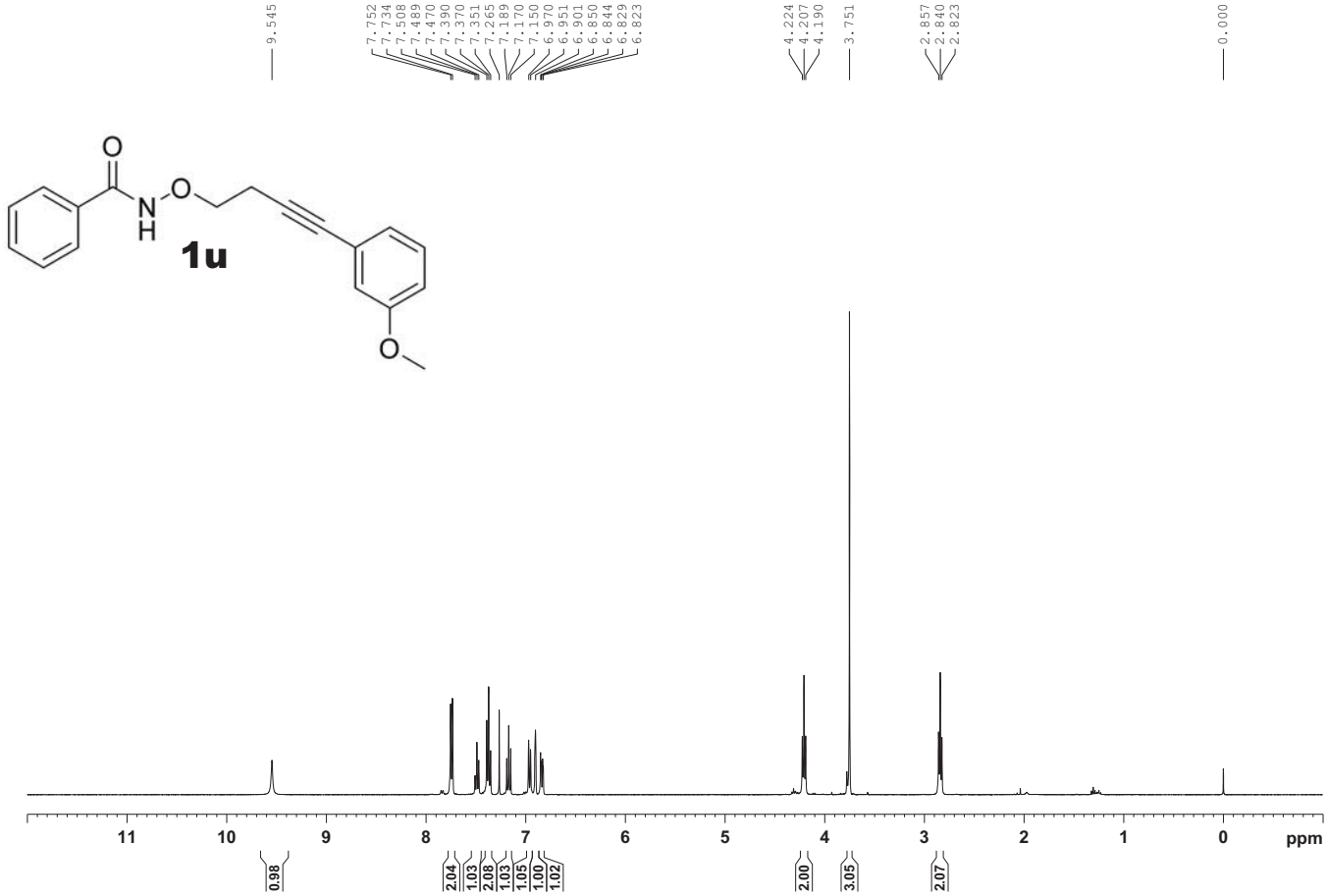
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6.901
6.850
6.844
6.829
6.823
4.224
4.207
4.190
3.751
2.857
2.840
2.823
0.000
1u
11 10 9 8 7 6 5 4 3 2 1 0 ppm
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1.03
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1.05
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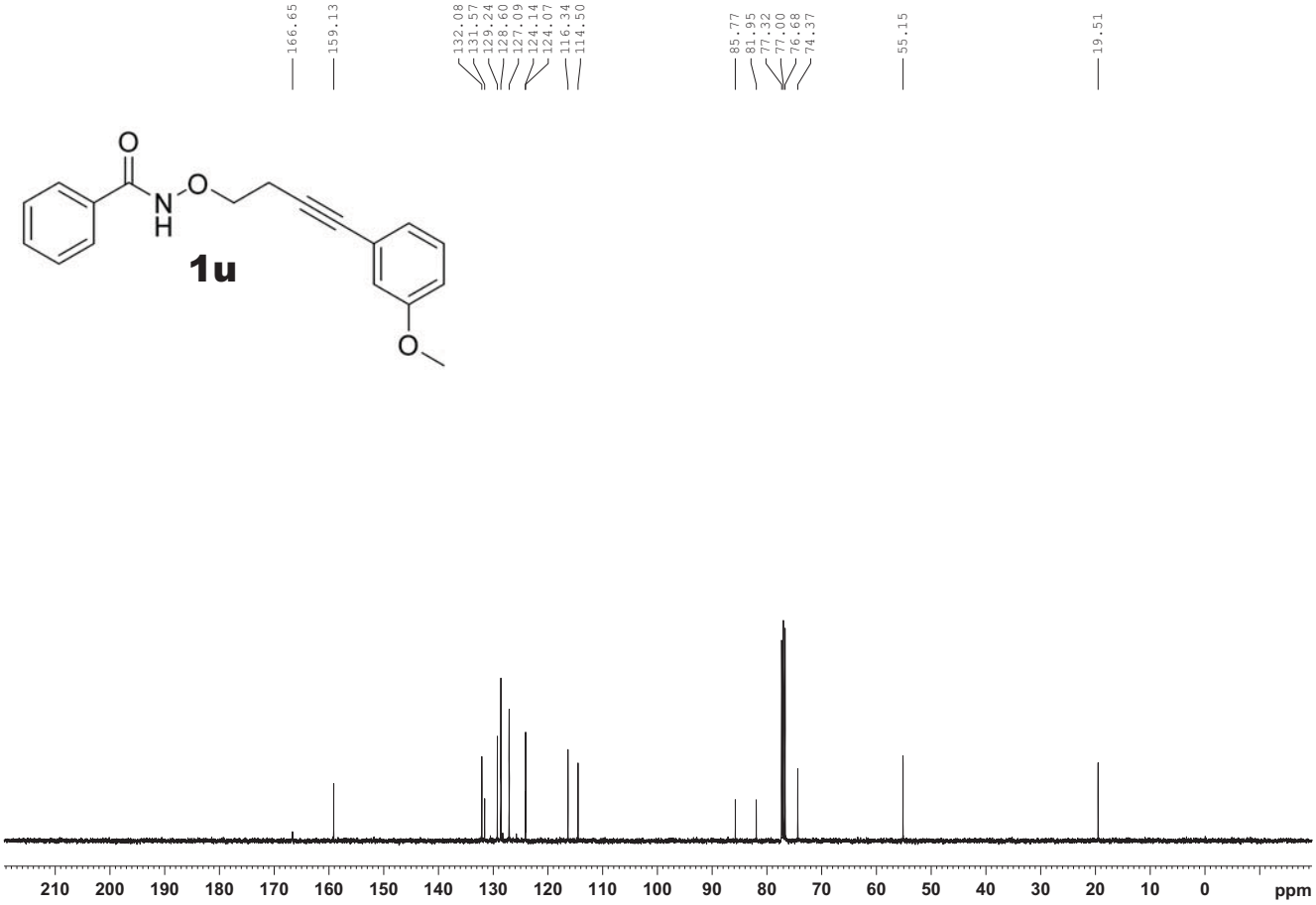
2.00
3.05
2.07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81.95
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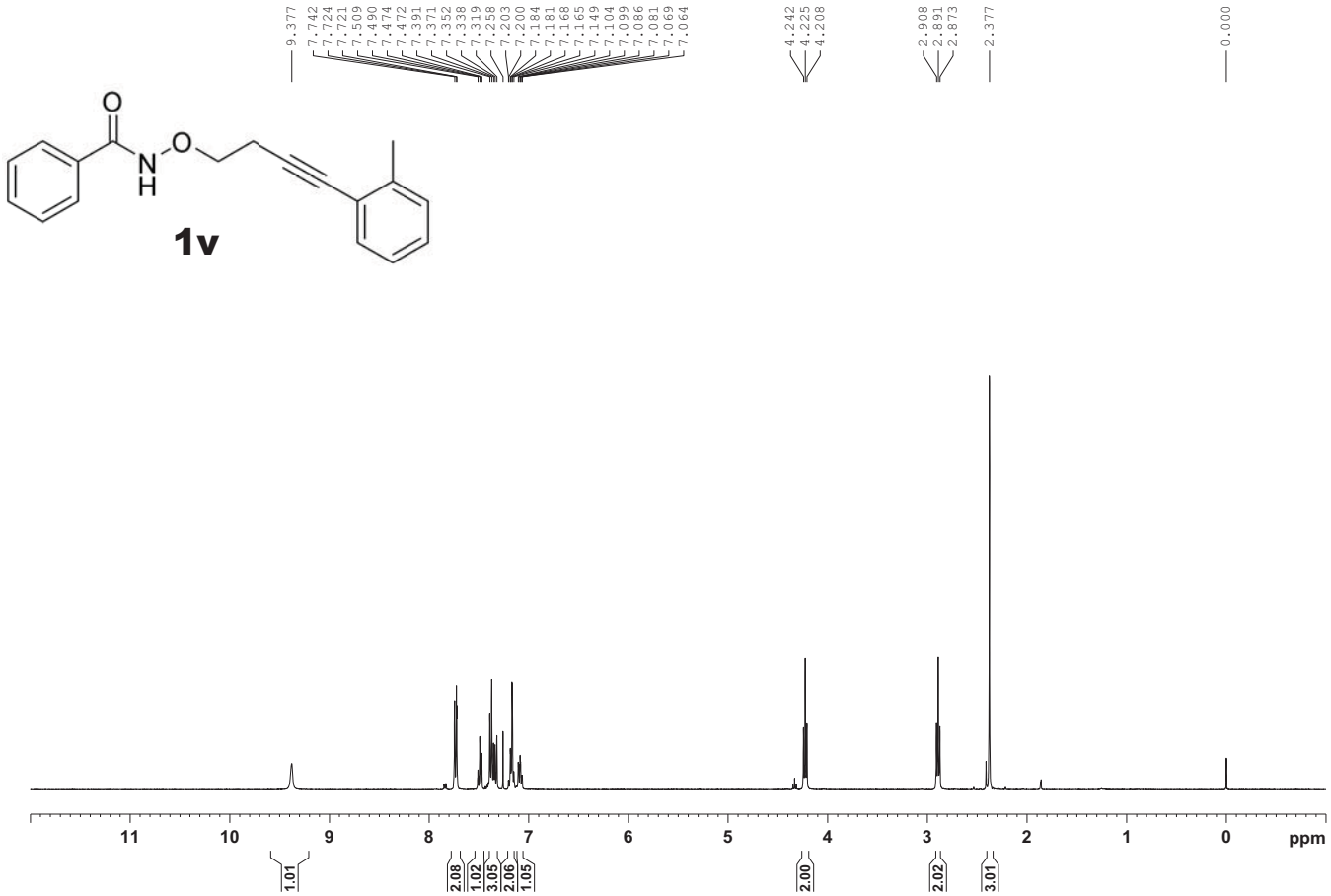
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7.258
7.203
7.200
7.184
7.181
7.168
7.165
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7.099
7.086
7.081
7.069
7.064
4.242
4.225
4.208
2.908
2.891
2.873
2.377
0.000
1v
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1.02
3.05
2.06
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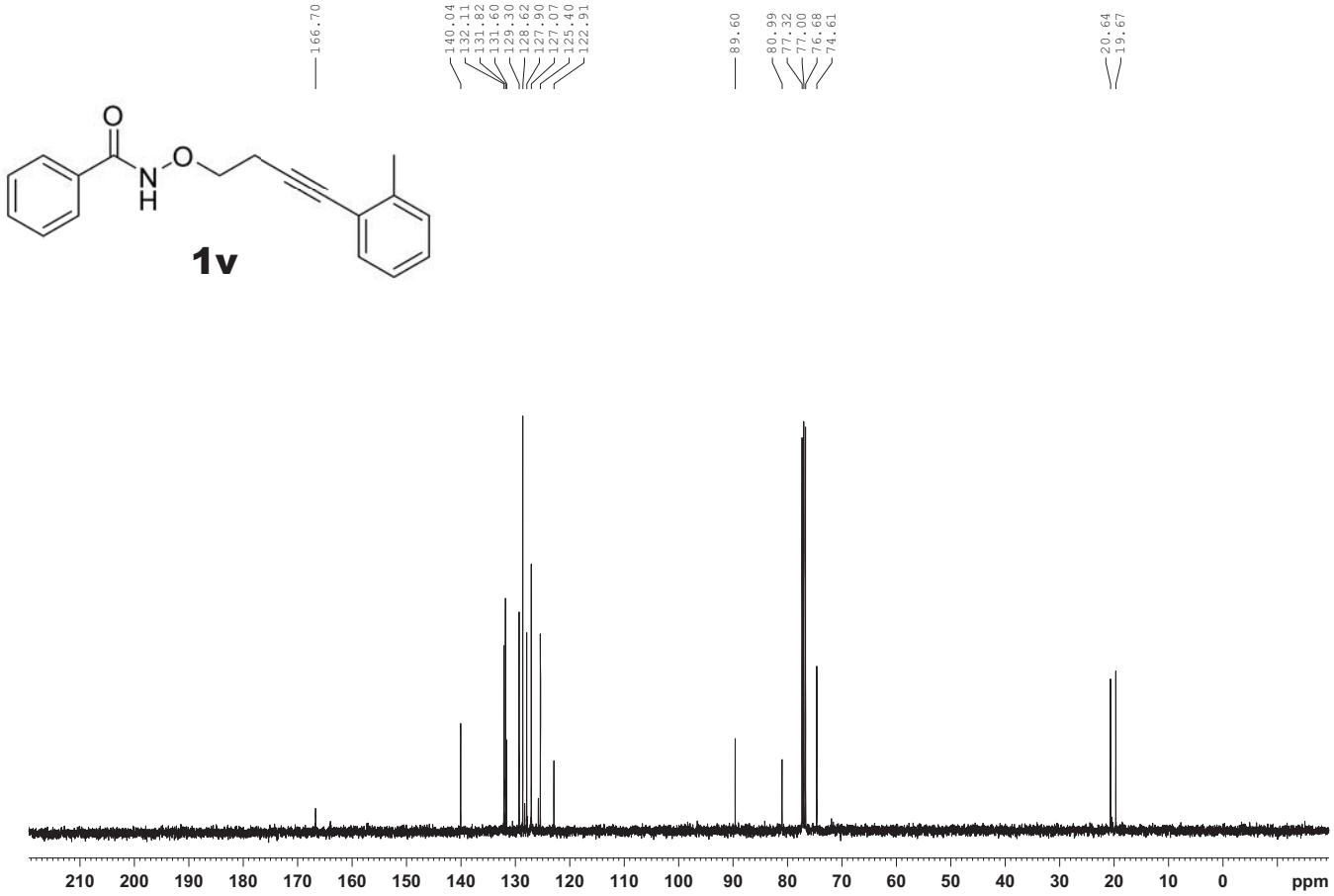
2.02
3.01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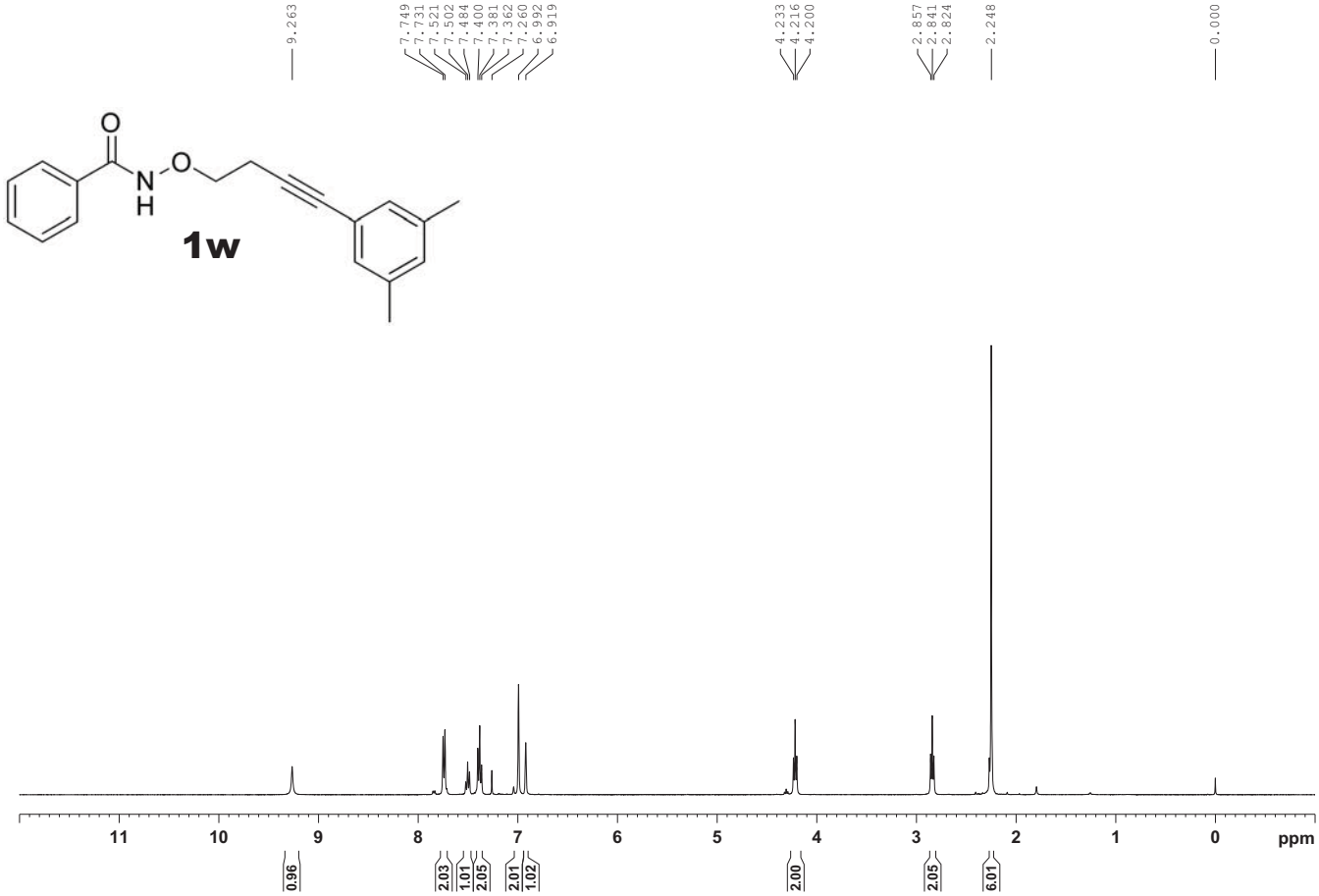
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4.233
4.216
4.200
2.857
2.841
2.824
2.248
0.000
1w
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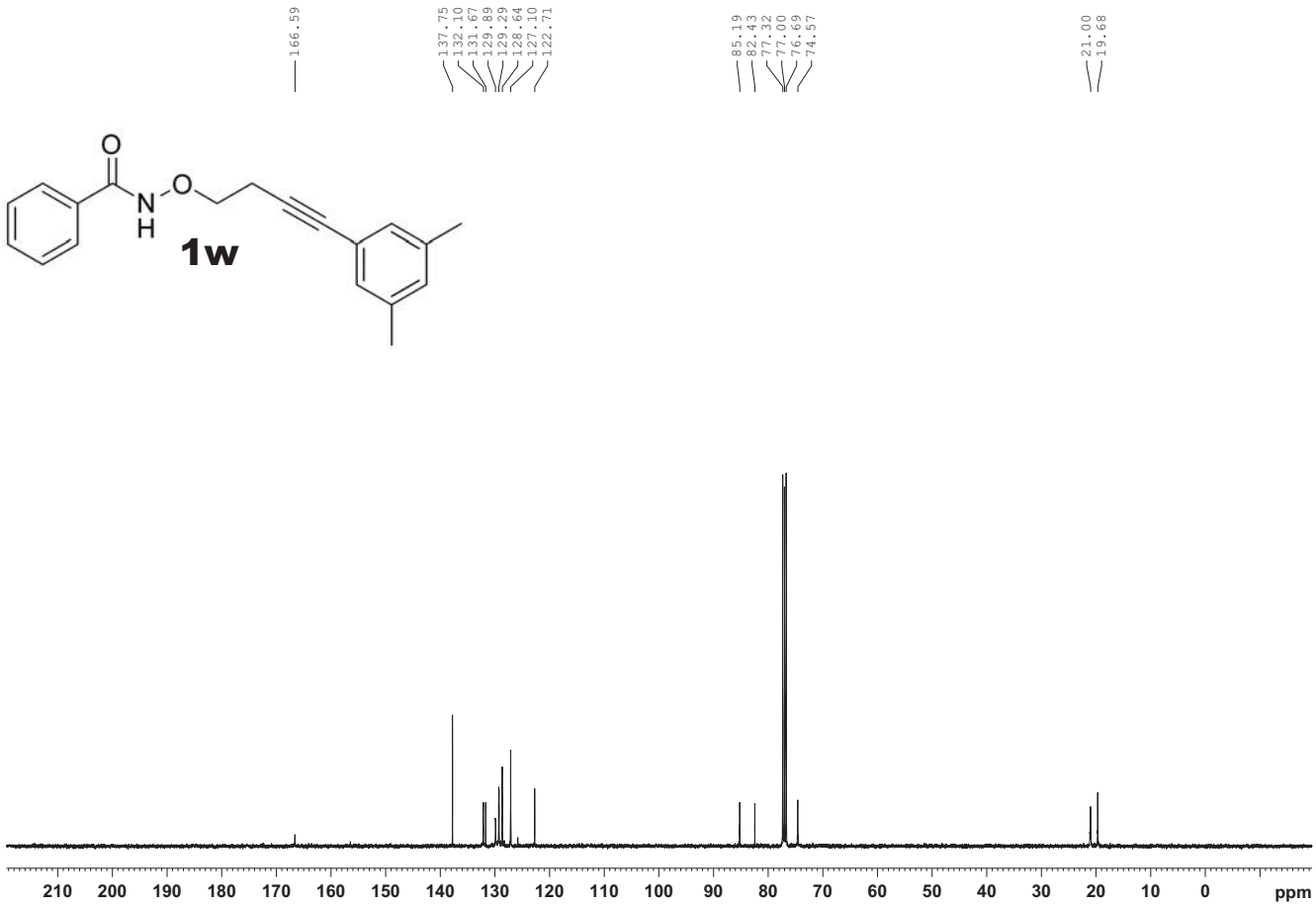
1.01
2.05
2.01
1.02
2.00
2.05
6.01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19.68
1w
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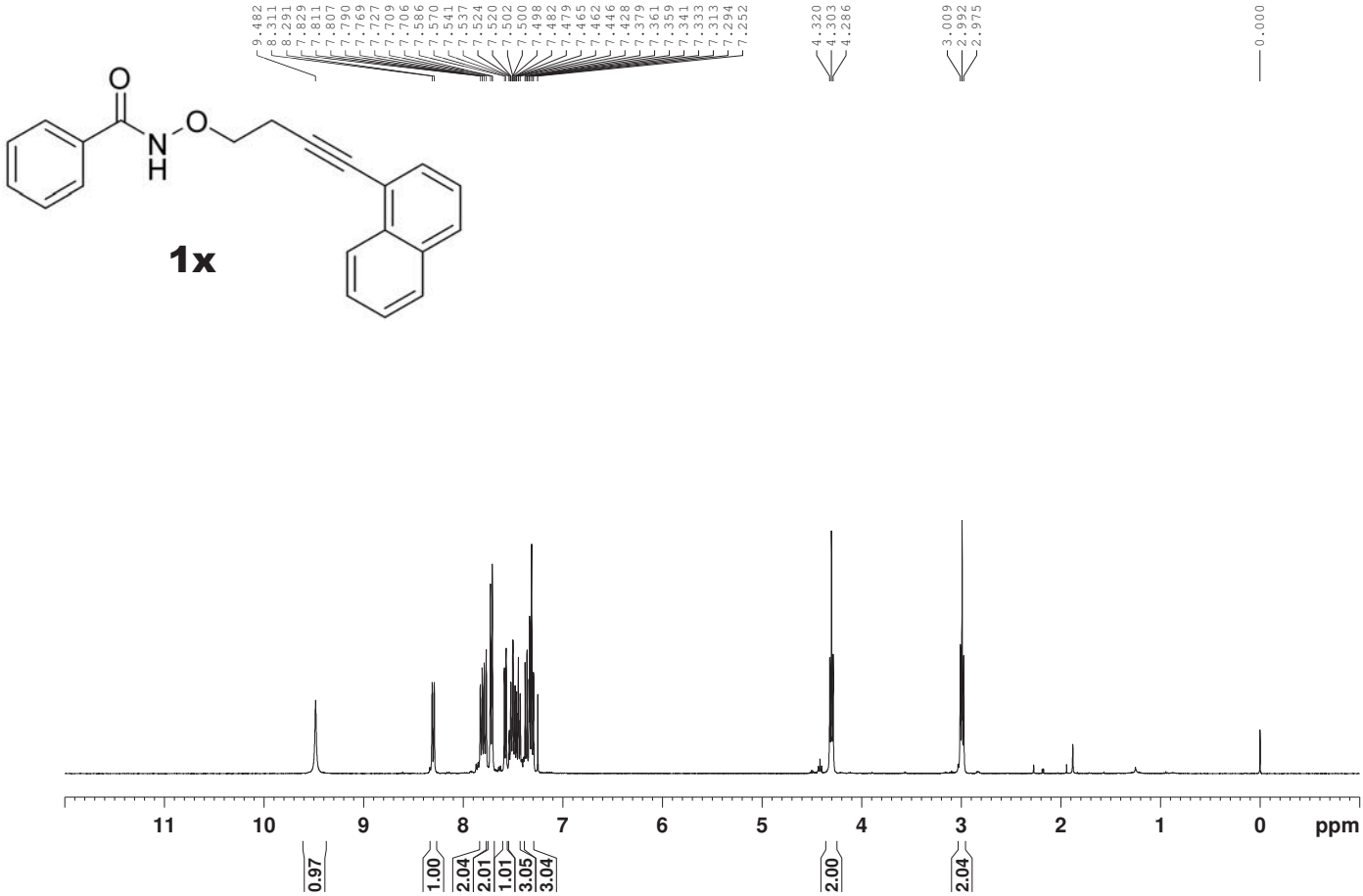
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H
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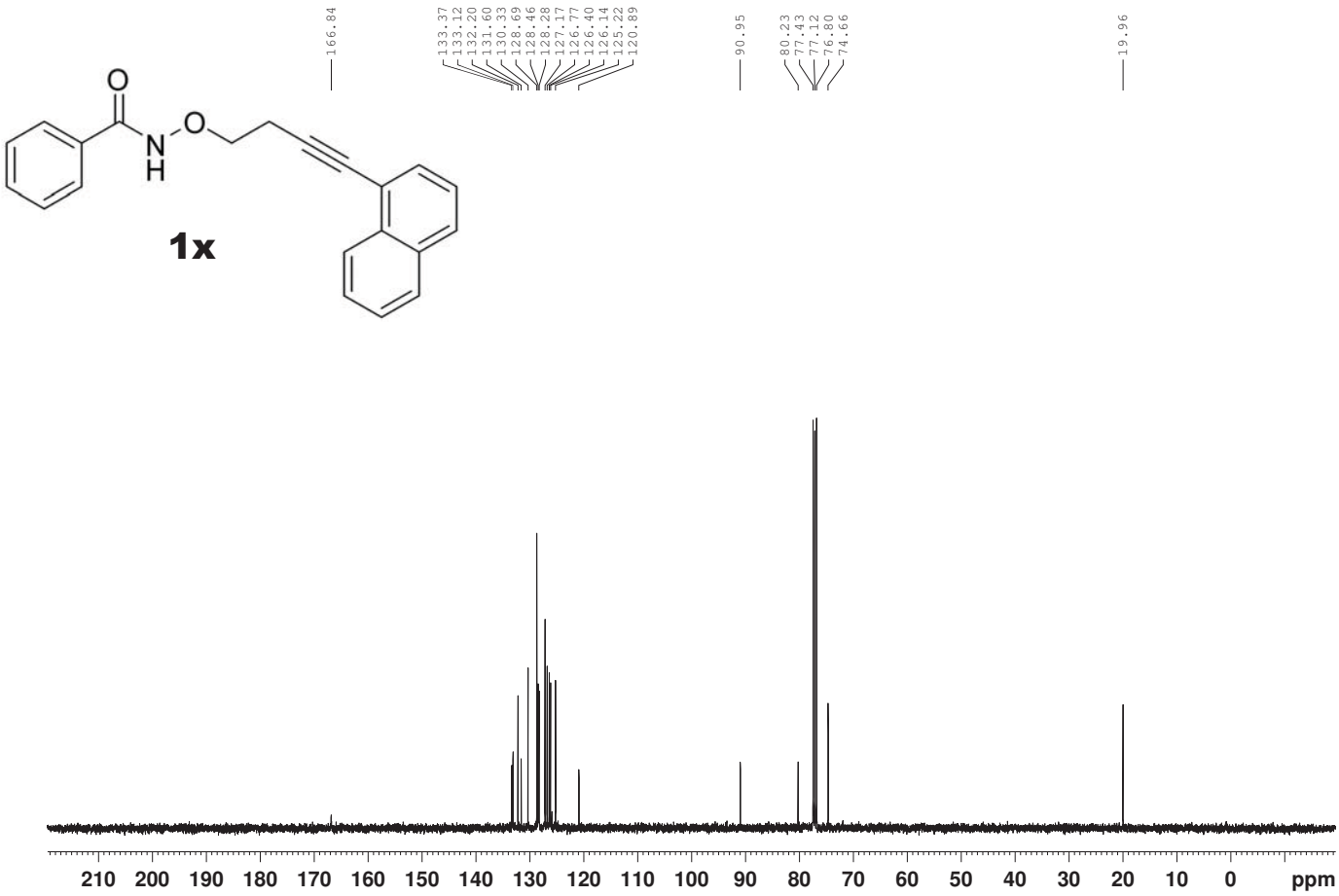
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76.80
74.66
19.96
O
N
H
O
1x
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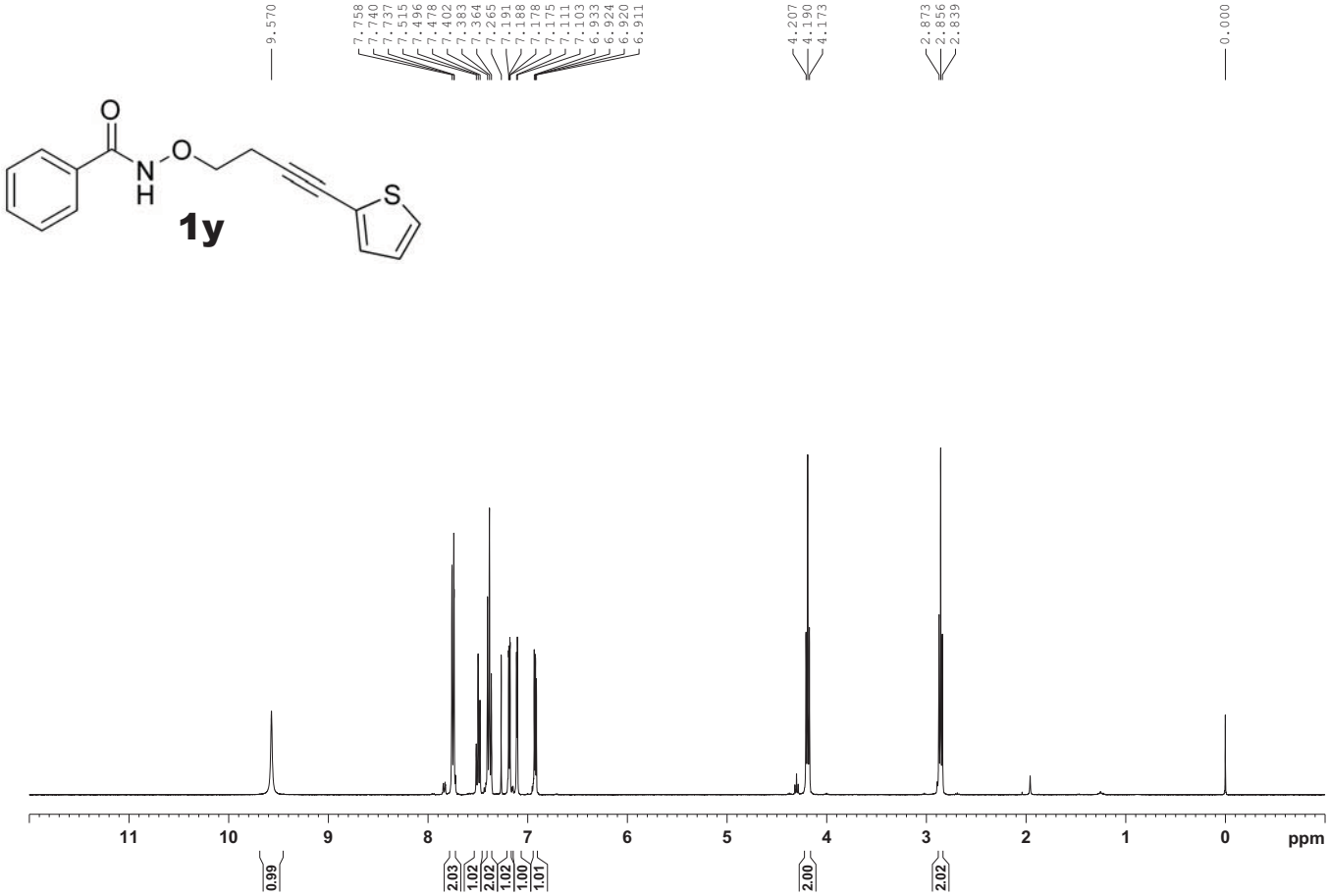
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6.924
6.920
6.911
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4.190
4.173
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2.856
2.839
0.000
1y
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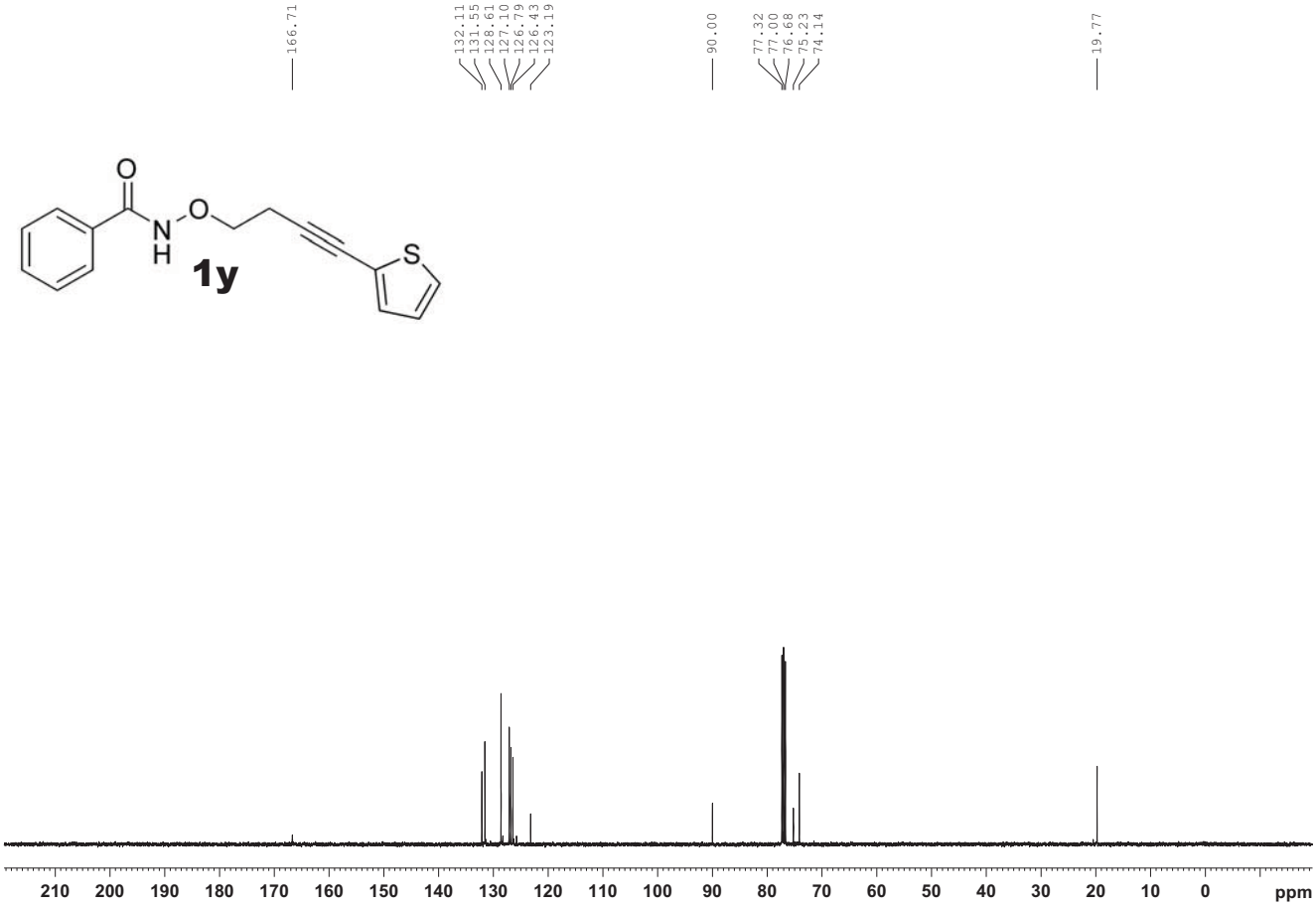
2.02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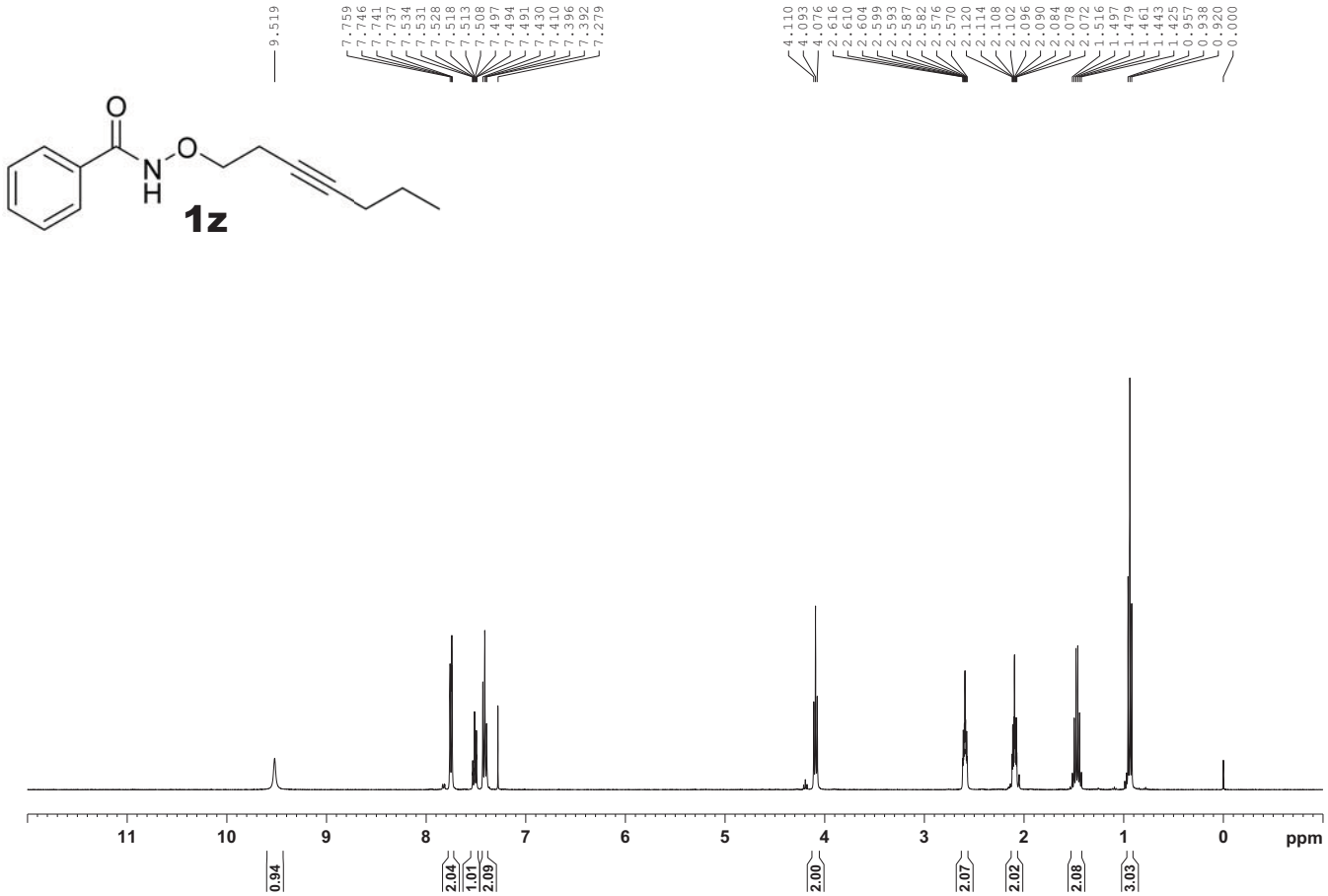
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7.491
7.430
7.410
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7.392
7.279
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4.093
4.076
2.616
2.610
2.604
2.599
2.593
2.587
2.582
2.576
2.570
2.120
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2.108
2.102
2.096
2.090
2.084
2.078
2.072
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1.479
1.461
1.443
1.425
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0.938
0.920
0.000
O
N
H
O
1z
11
10
9
8
7
6
5
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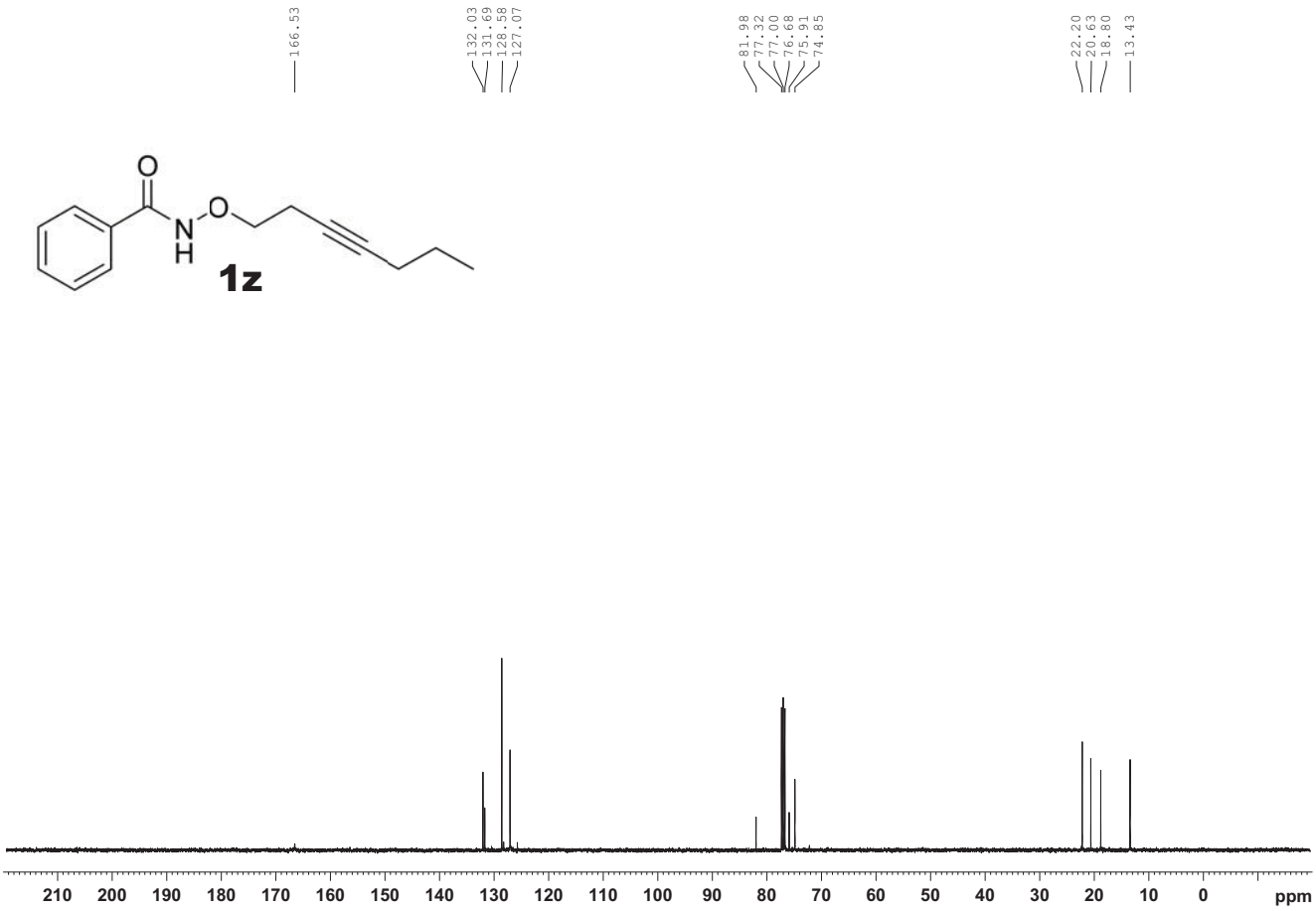
3.03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18.80
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O
N
H
O
1z
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200
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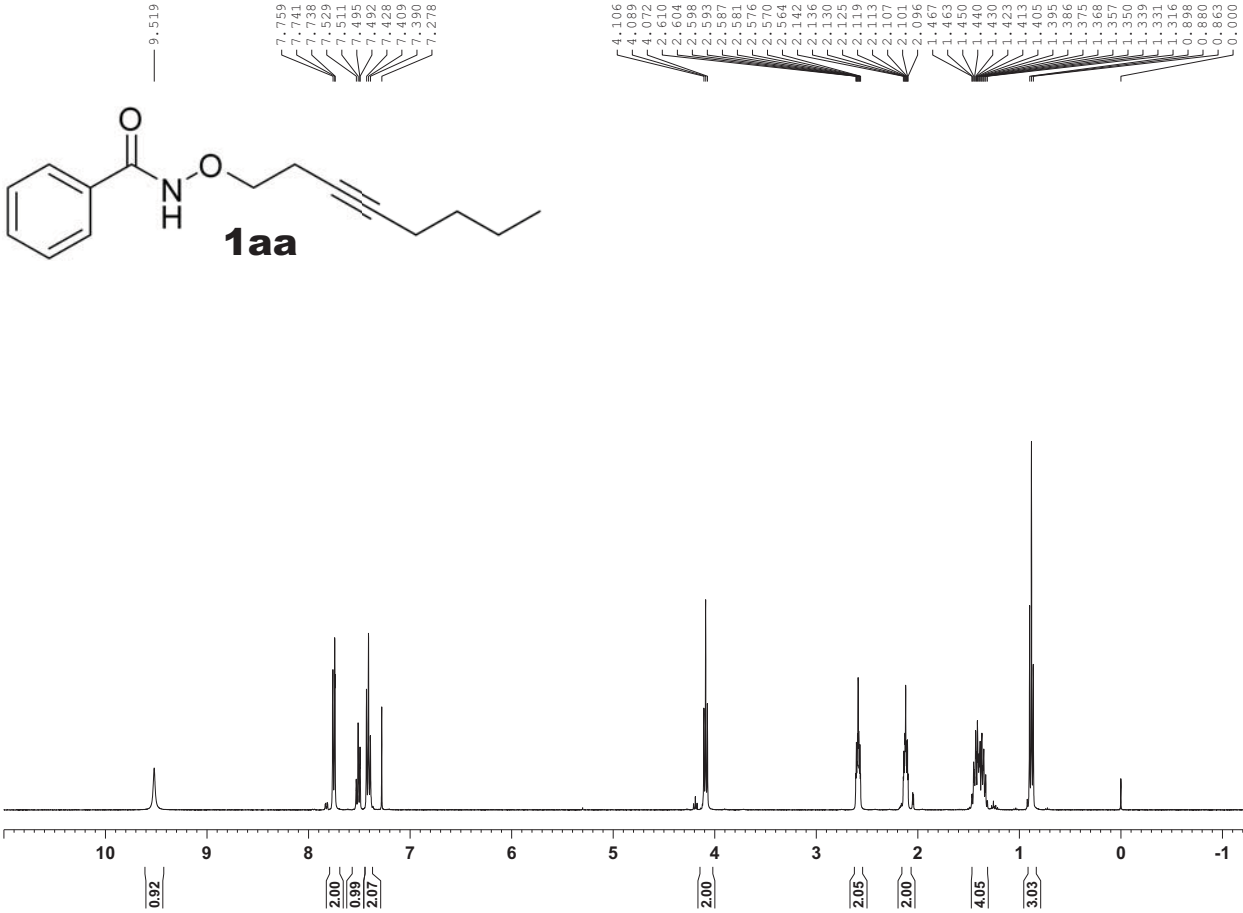
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2.581
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2.136
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2.113
2.107
2.101
2.096
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1.463
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1.440
1.430
1.423
1.413
1.405
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6
5
4
3
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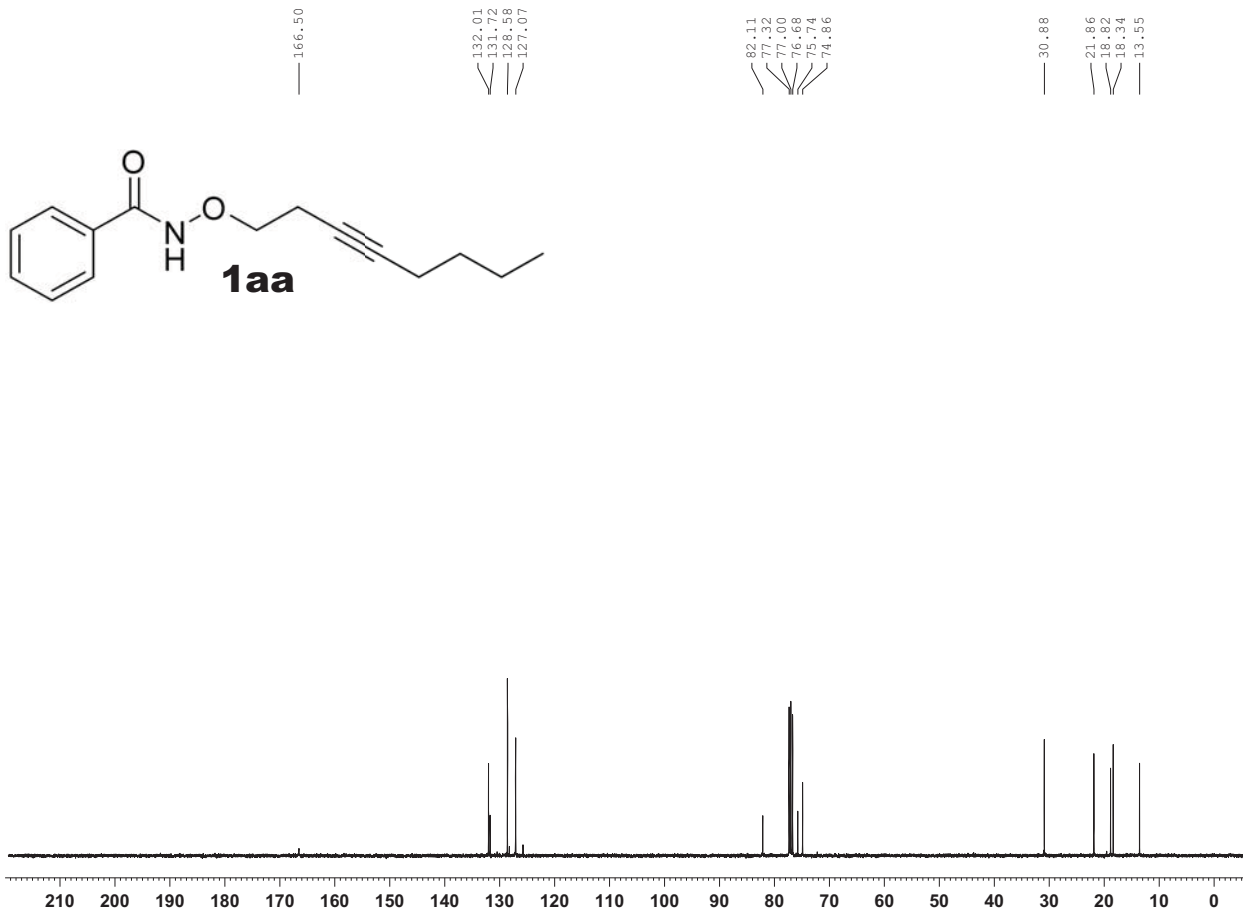
4.05
3.03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18.82
18.34
13.55
O
N
H
O
1aa
210
200
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20
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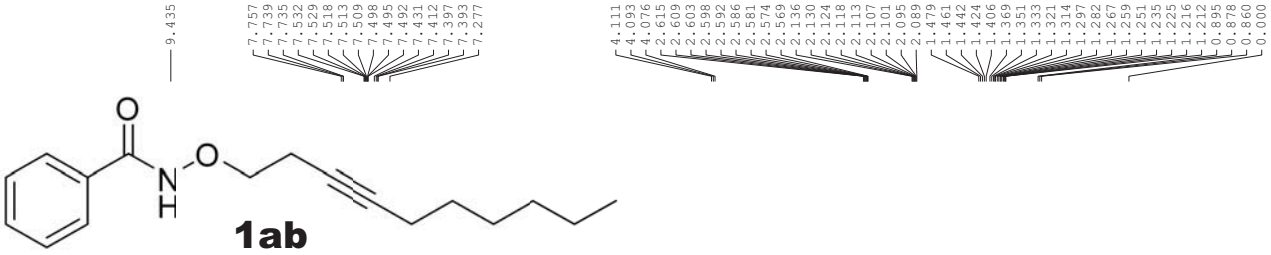
ppm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7.431
7.412
7.397
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O
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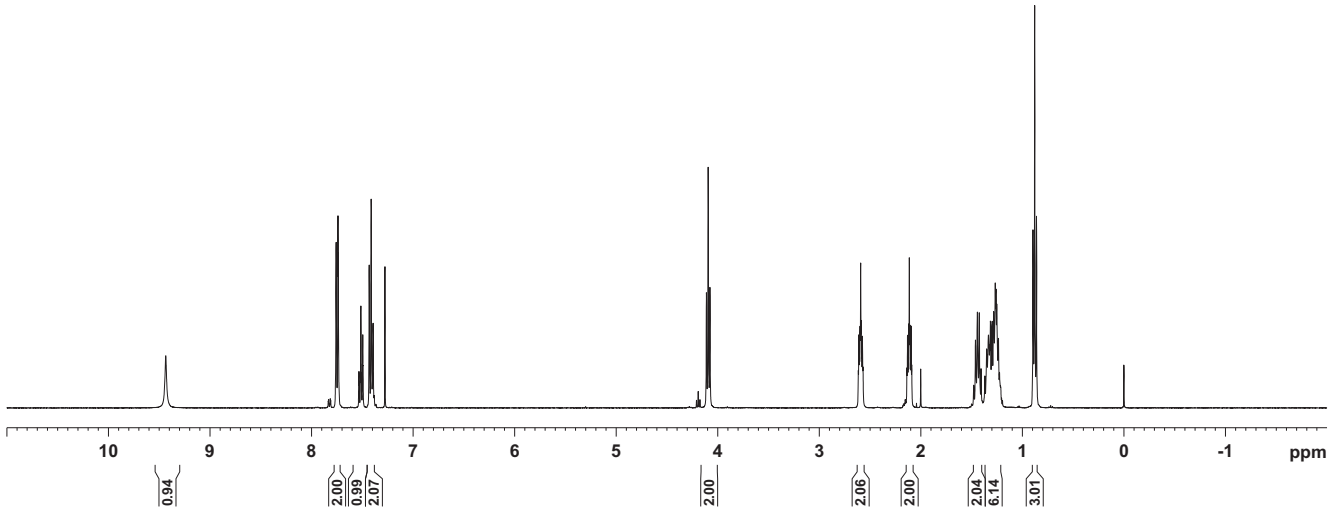
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
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0.99
2.07
2.00
2.06
2.00
2.04
6.14
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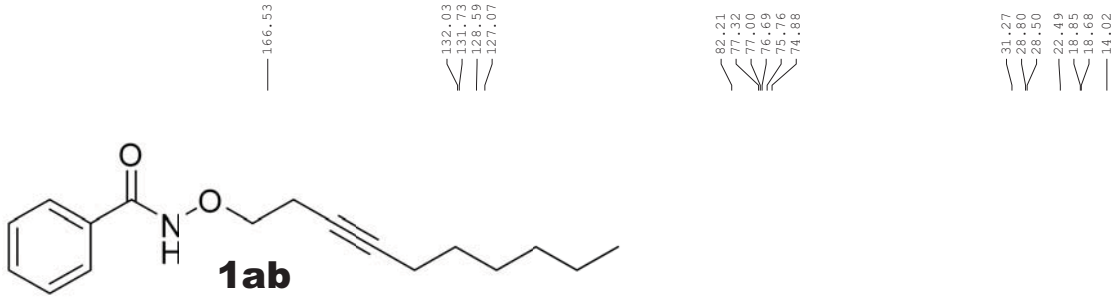
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H
O
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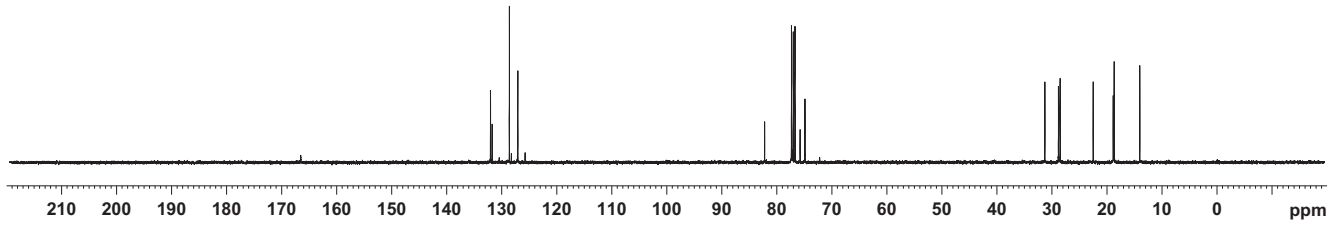
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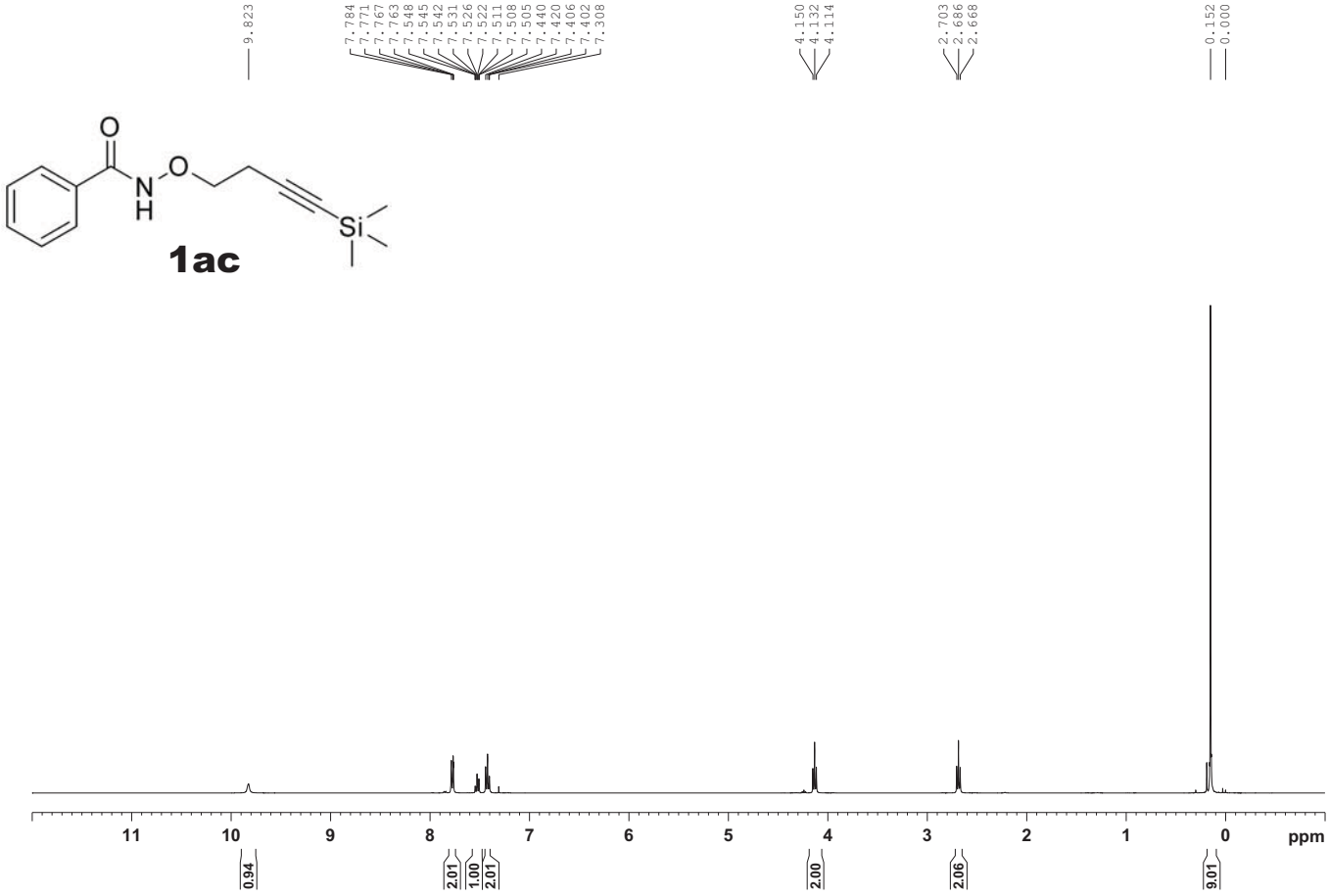
0
ppm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O
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H
O
Si
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10
9
8
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1.00
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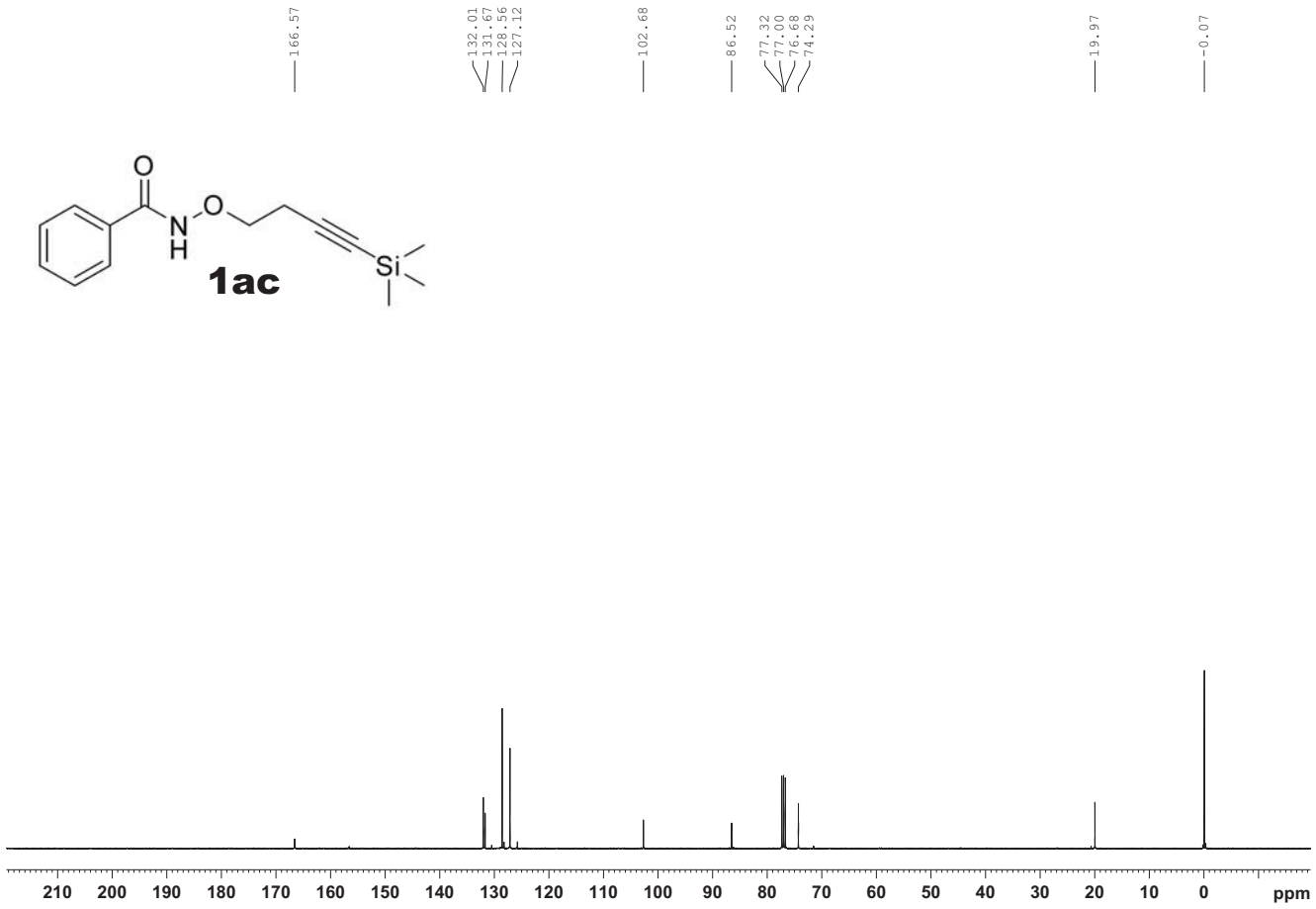
2.00
2.06
9.01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N
H
O
Si
1ac
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200
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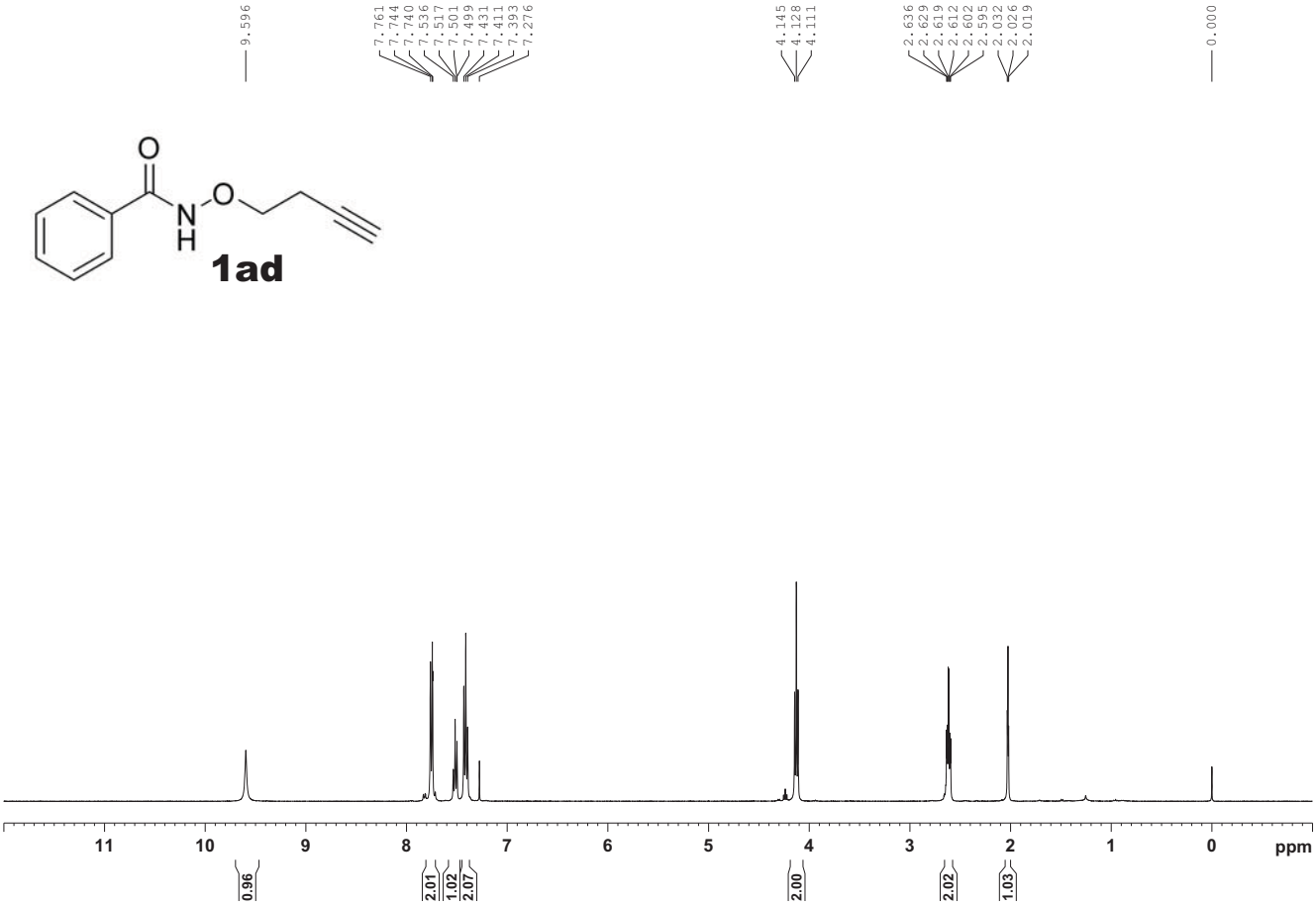
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4.128
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2.619
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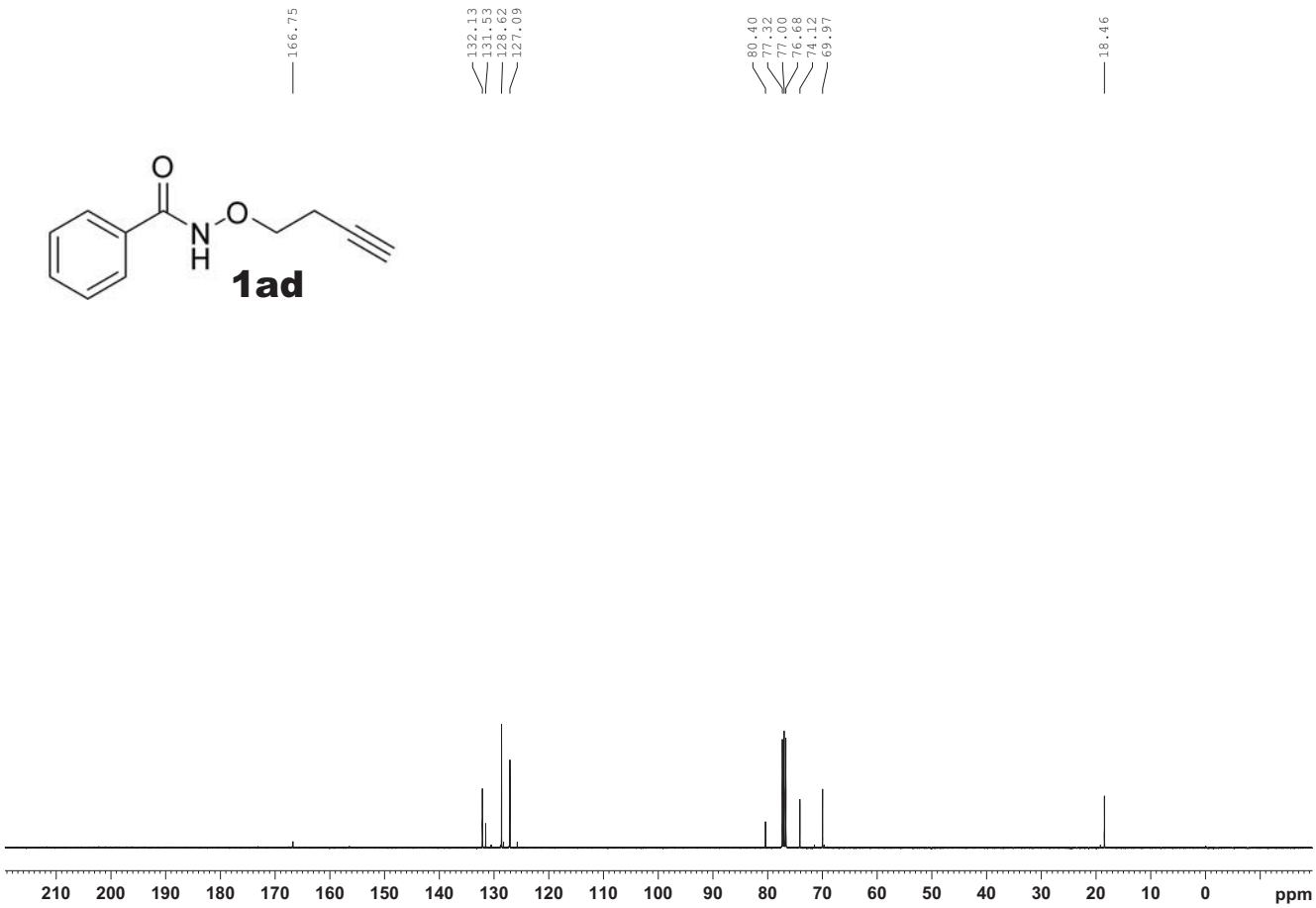
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200
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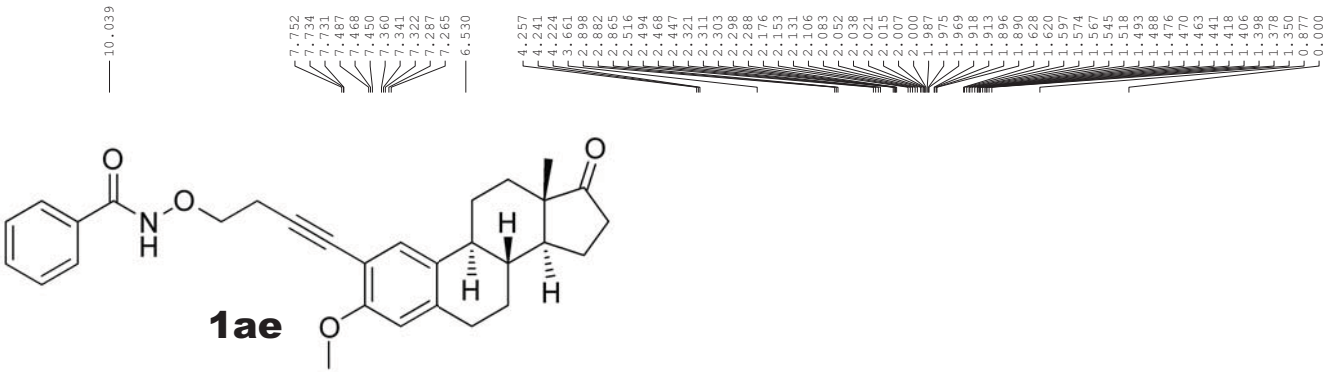
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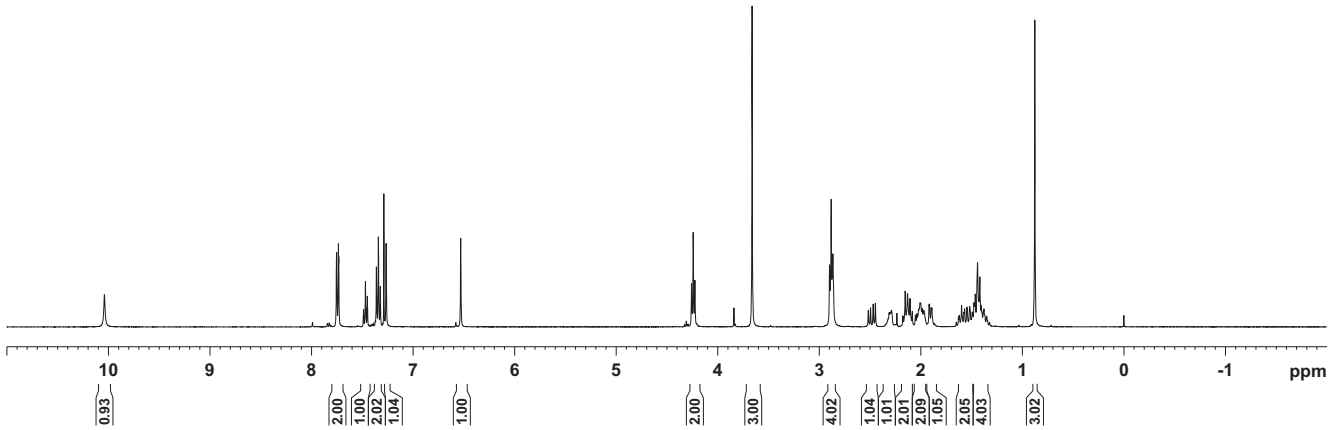

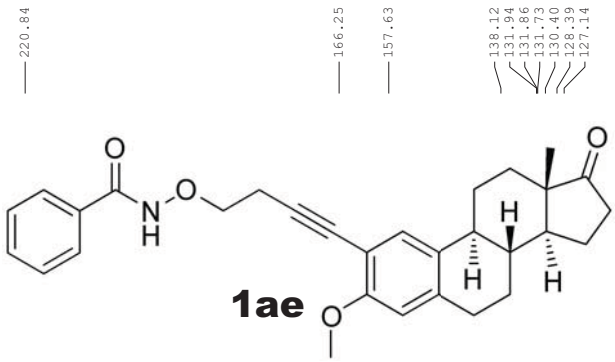
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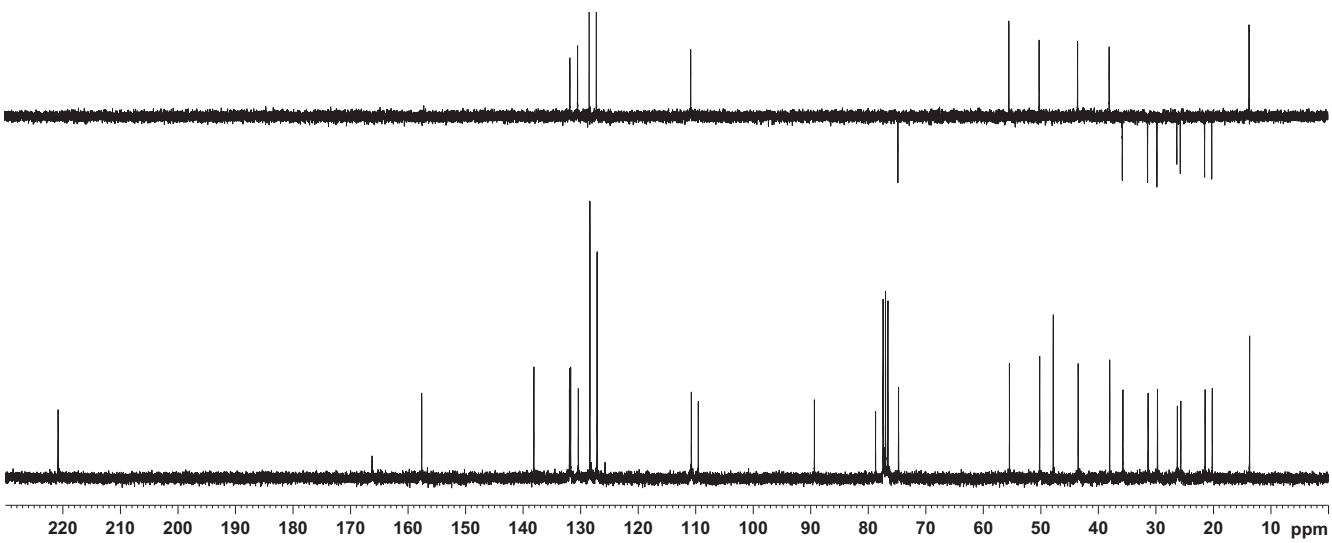

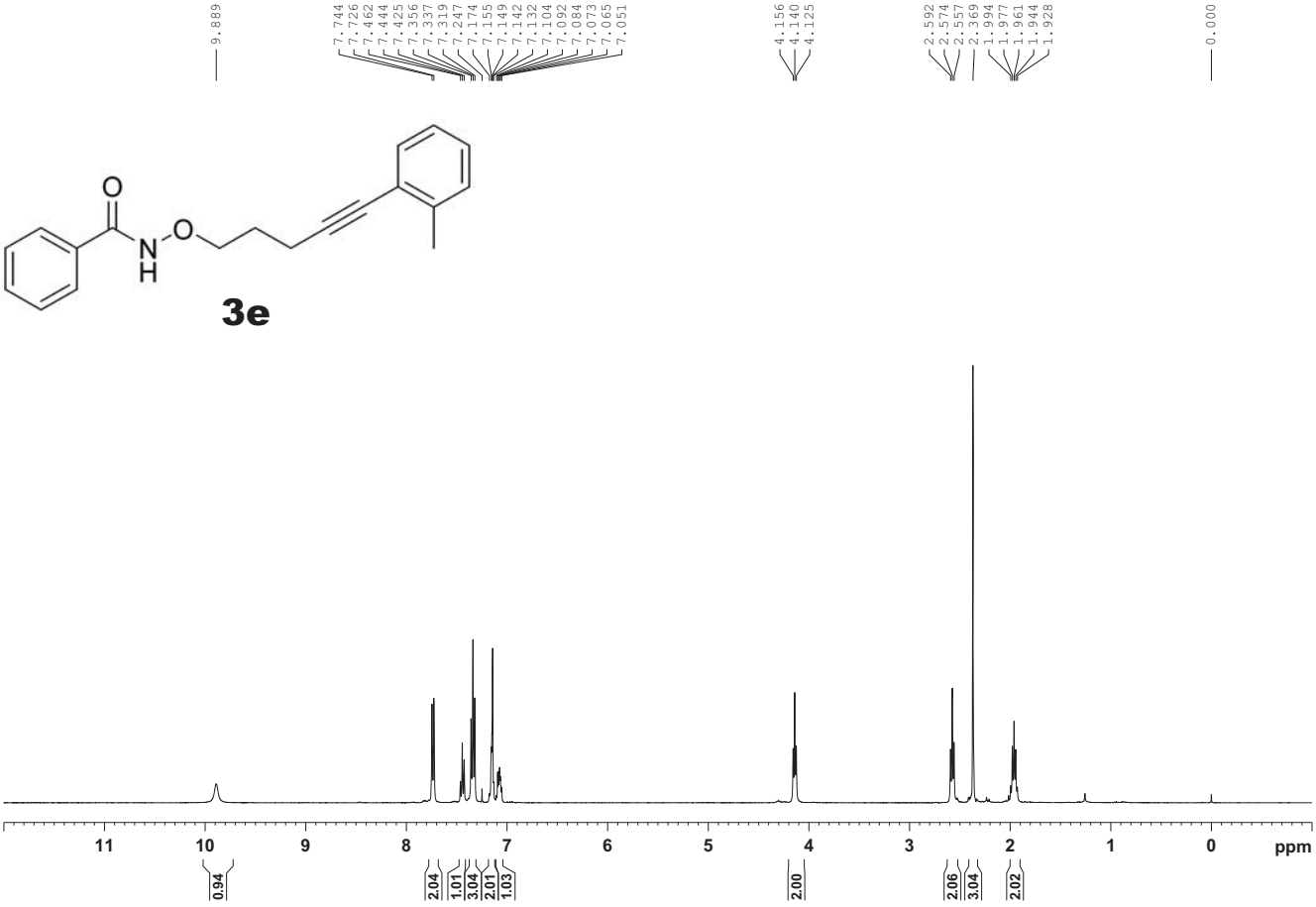
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O
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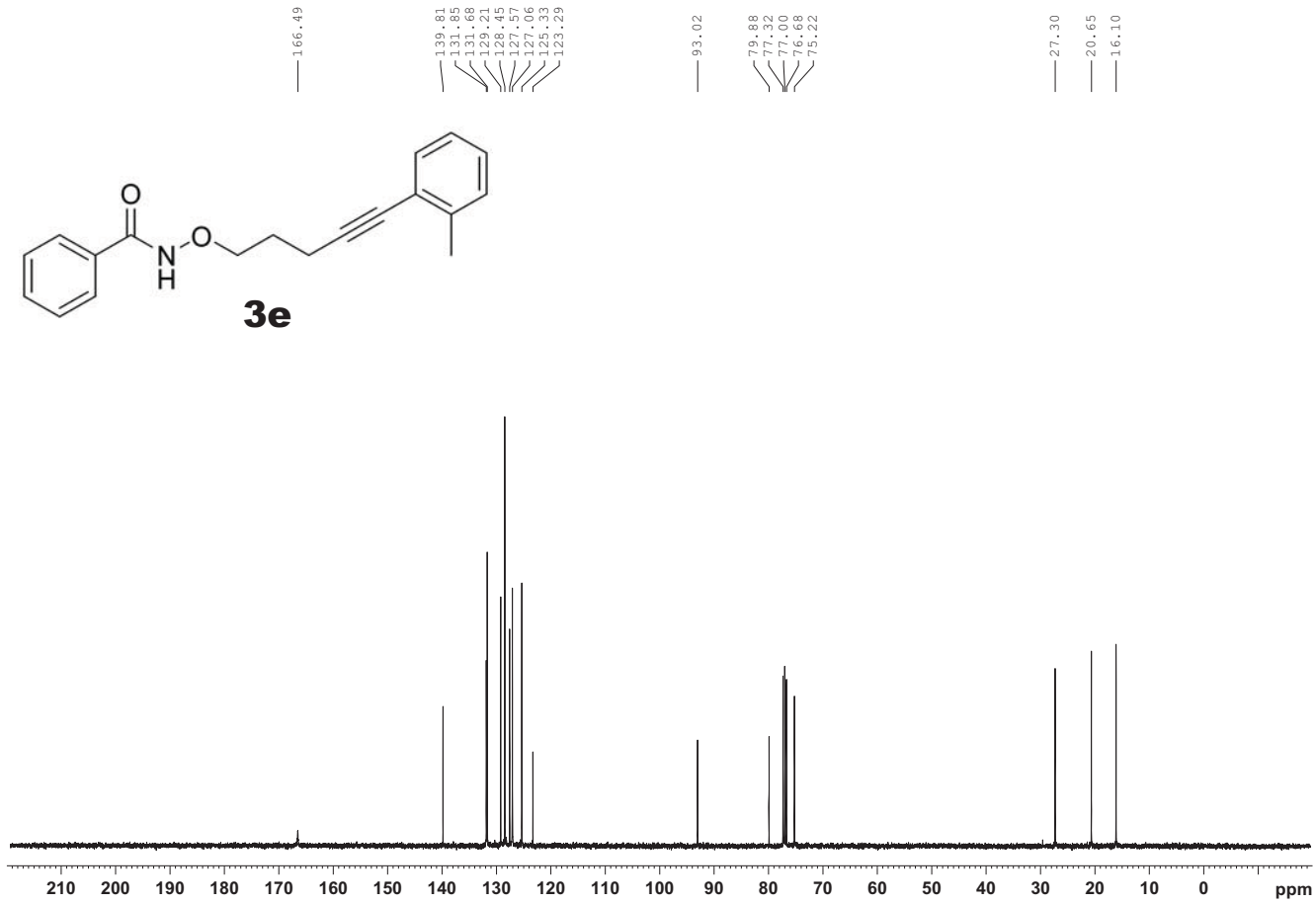
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27.30
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O
H
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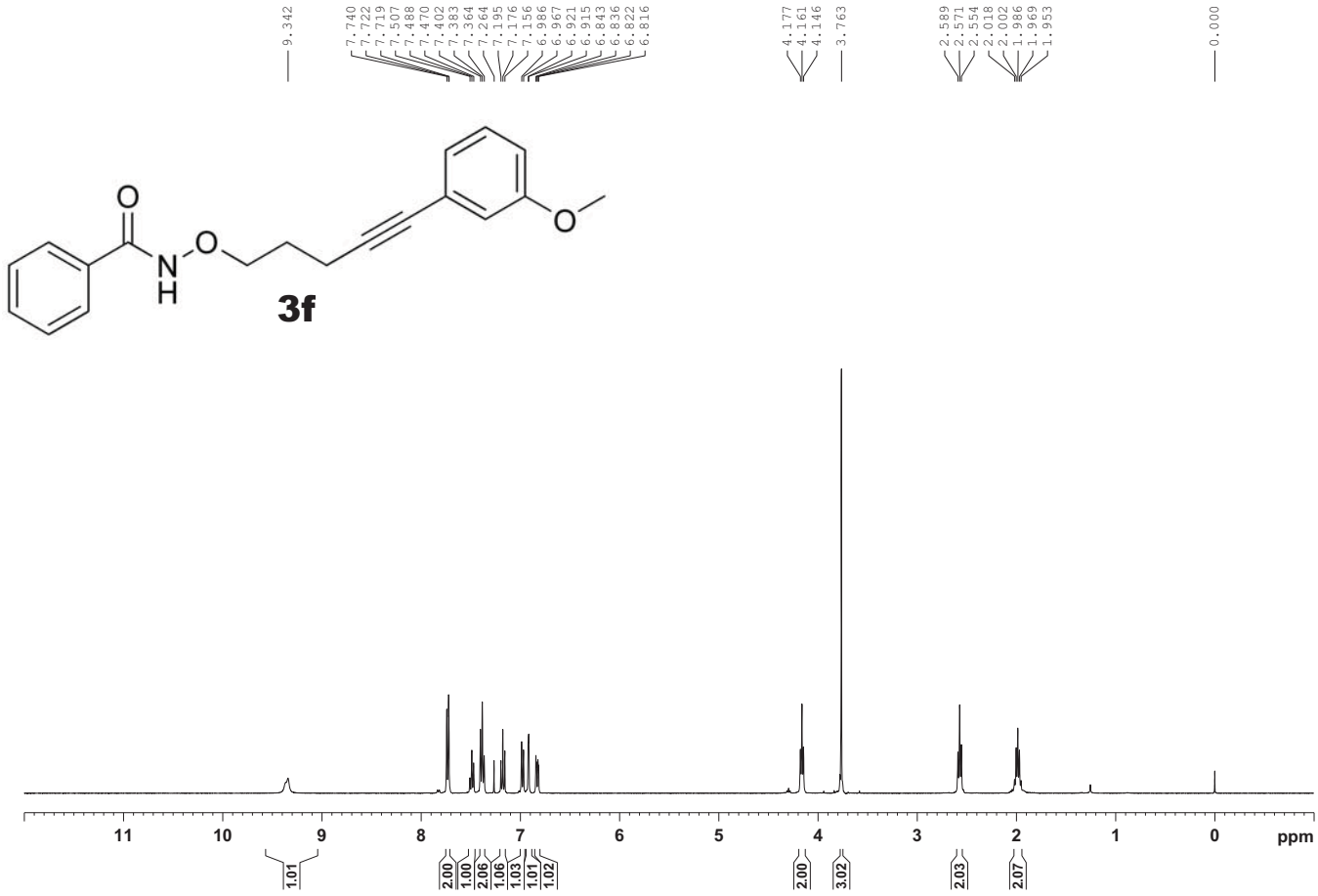
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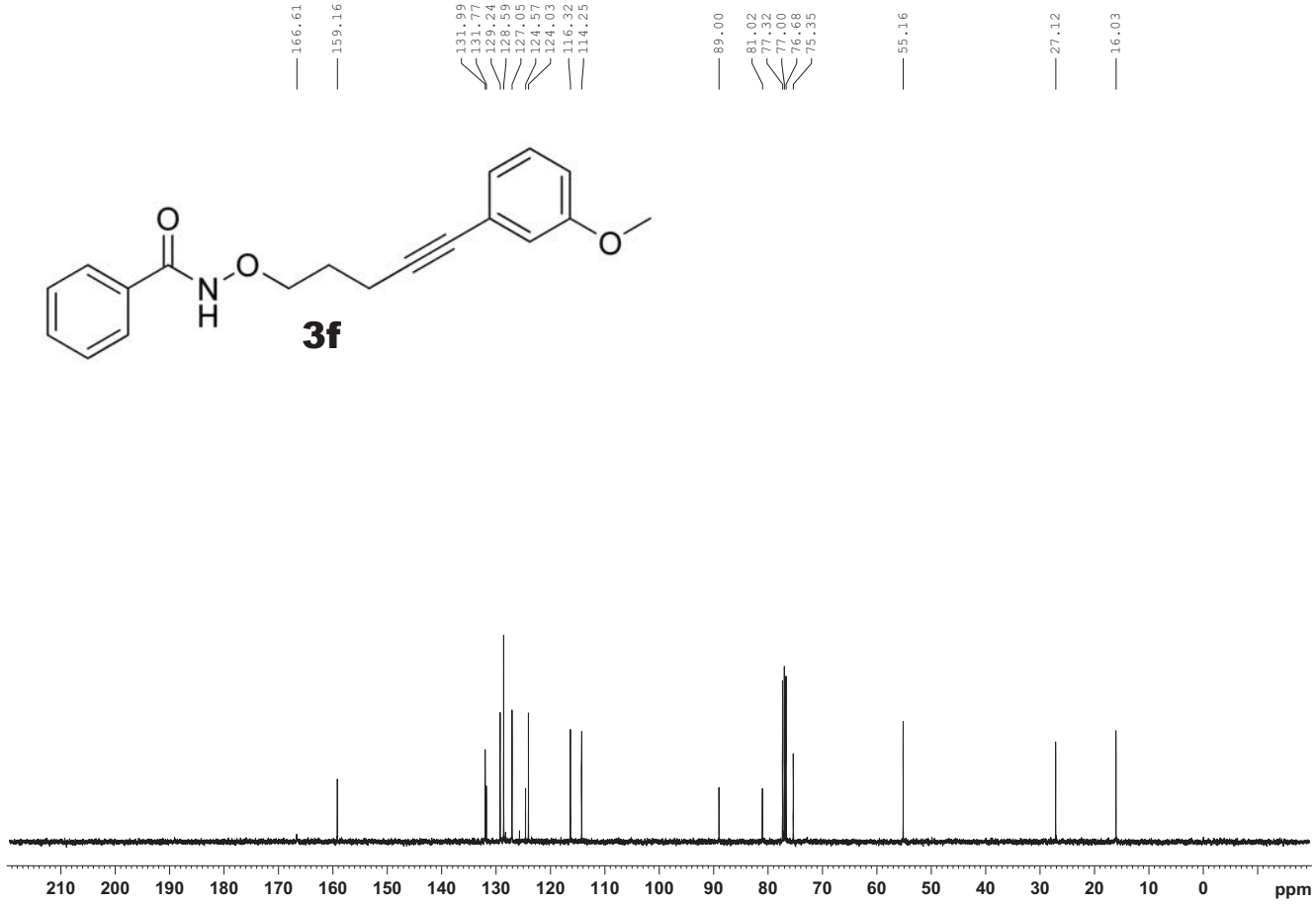
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77.32
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76.68
75.35
55.16
27.12
16.03
O
N
H
O
O
3f
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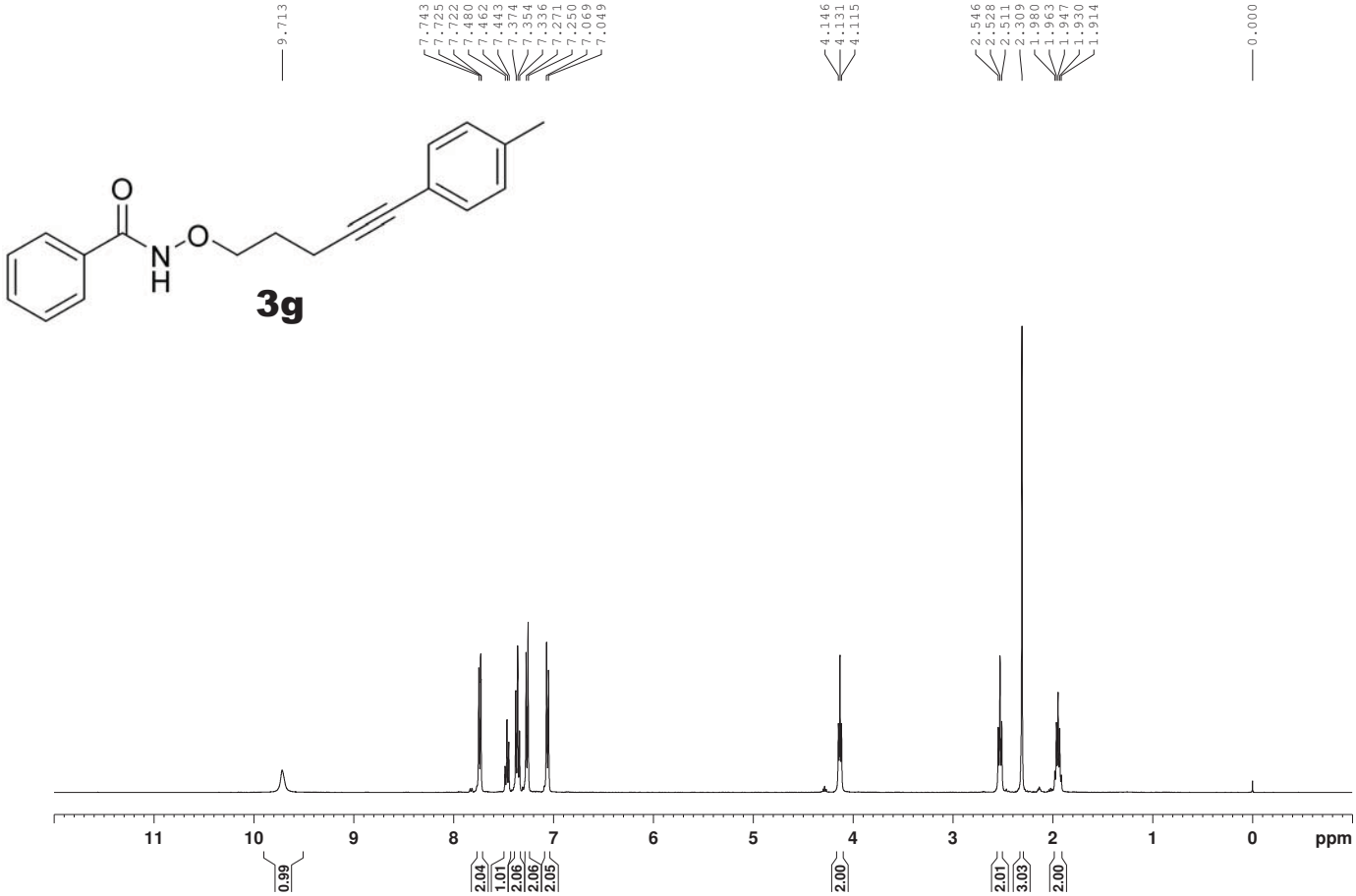
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7.069
7.049
4.146
4.131
4.115
2.546
2.528
2.511
2.309
1.980
1.963
1.947
1.930
1.914
0.000
O
N
H
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5
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2.05
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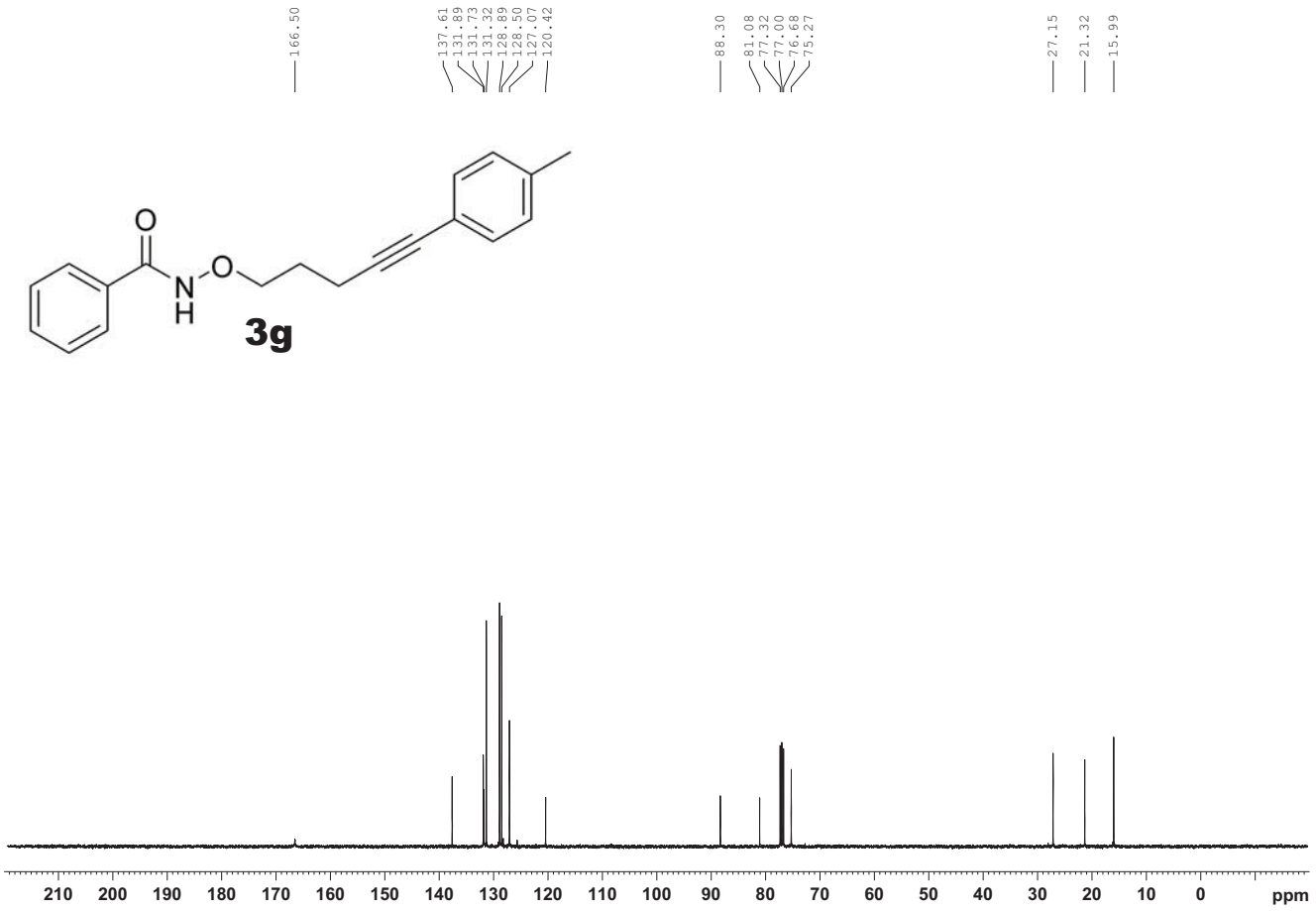
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77.00
76.68
75.27
27.15
21.32
15.99
O
N
H
O
3g
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200
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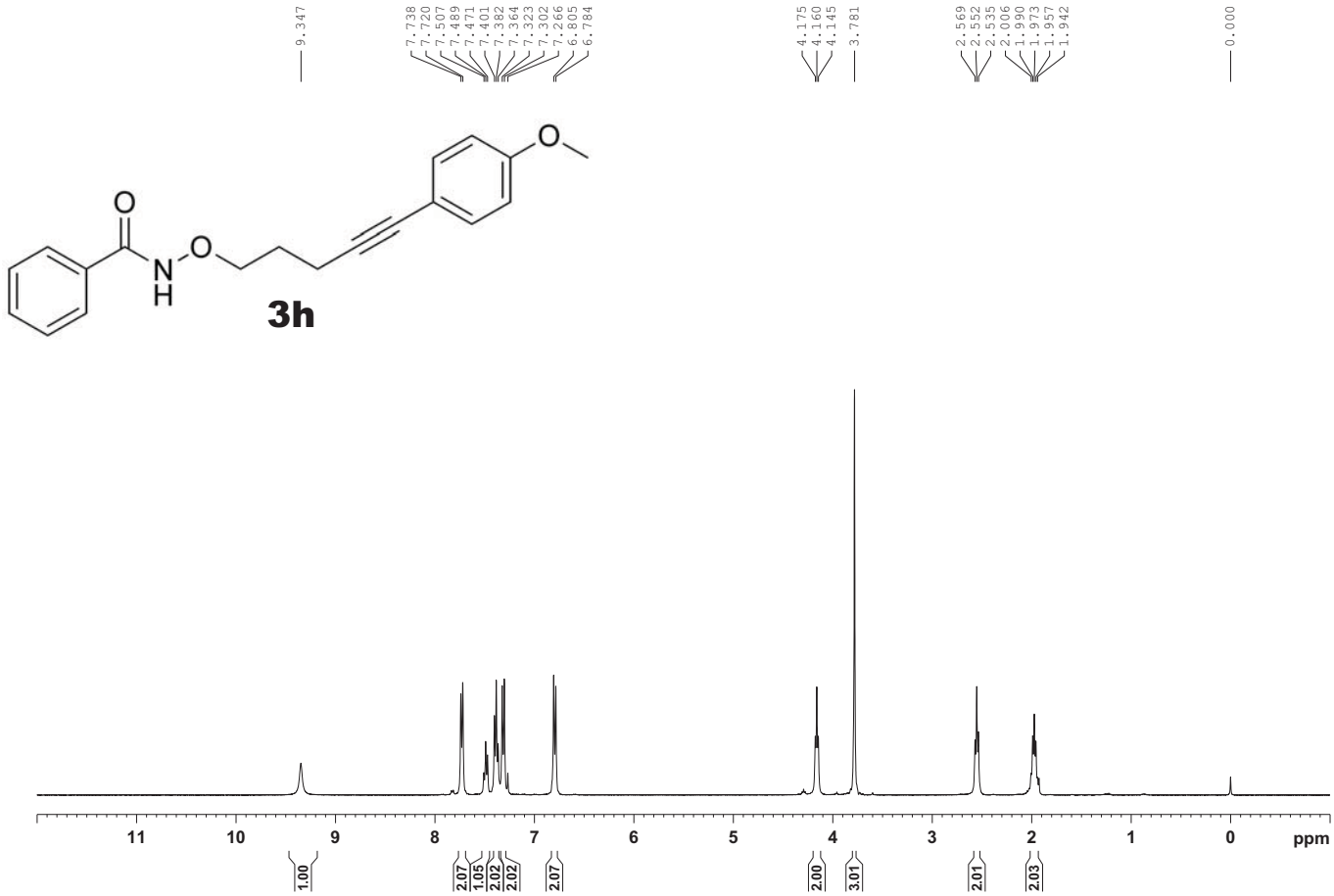
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7.507
7.489
7.471
7.401
7.382
7.364
7.323
7.302
7.266
6.805
6.784
4.175
4.160
4.145
3.781
2.569
2.552
2.535
2.006
1.990
1.973
1.957
1.942
0.000
3h
11
10
9
8
7
6
5
4
3
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2.07
1.05
2.02
2.02
2.07
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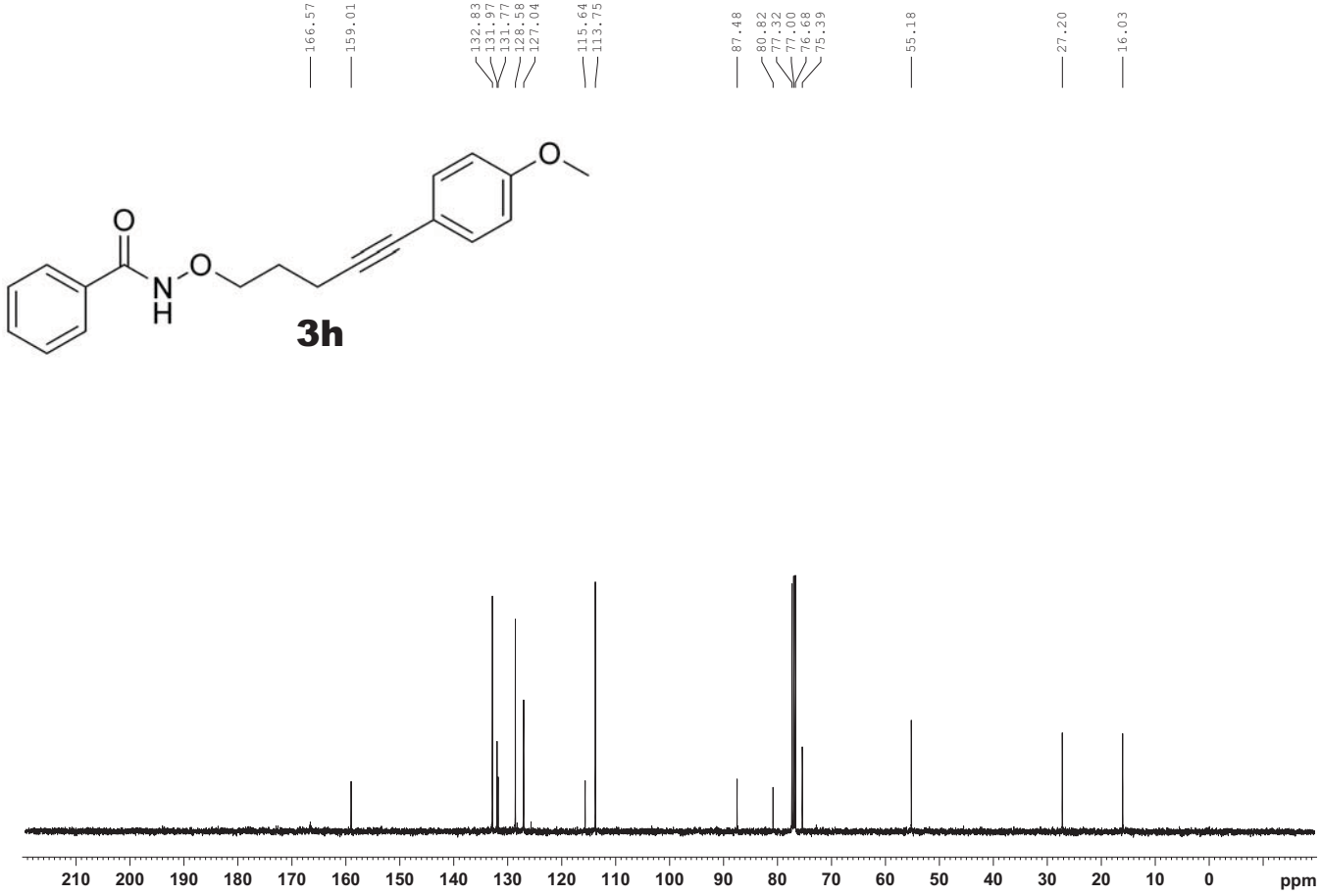
3.01
2.01
2.03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127.04
115.64
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80.82
77.32
77.00
76.68
75.39
55.18
27.20
16.03
3h
210
200
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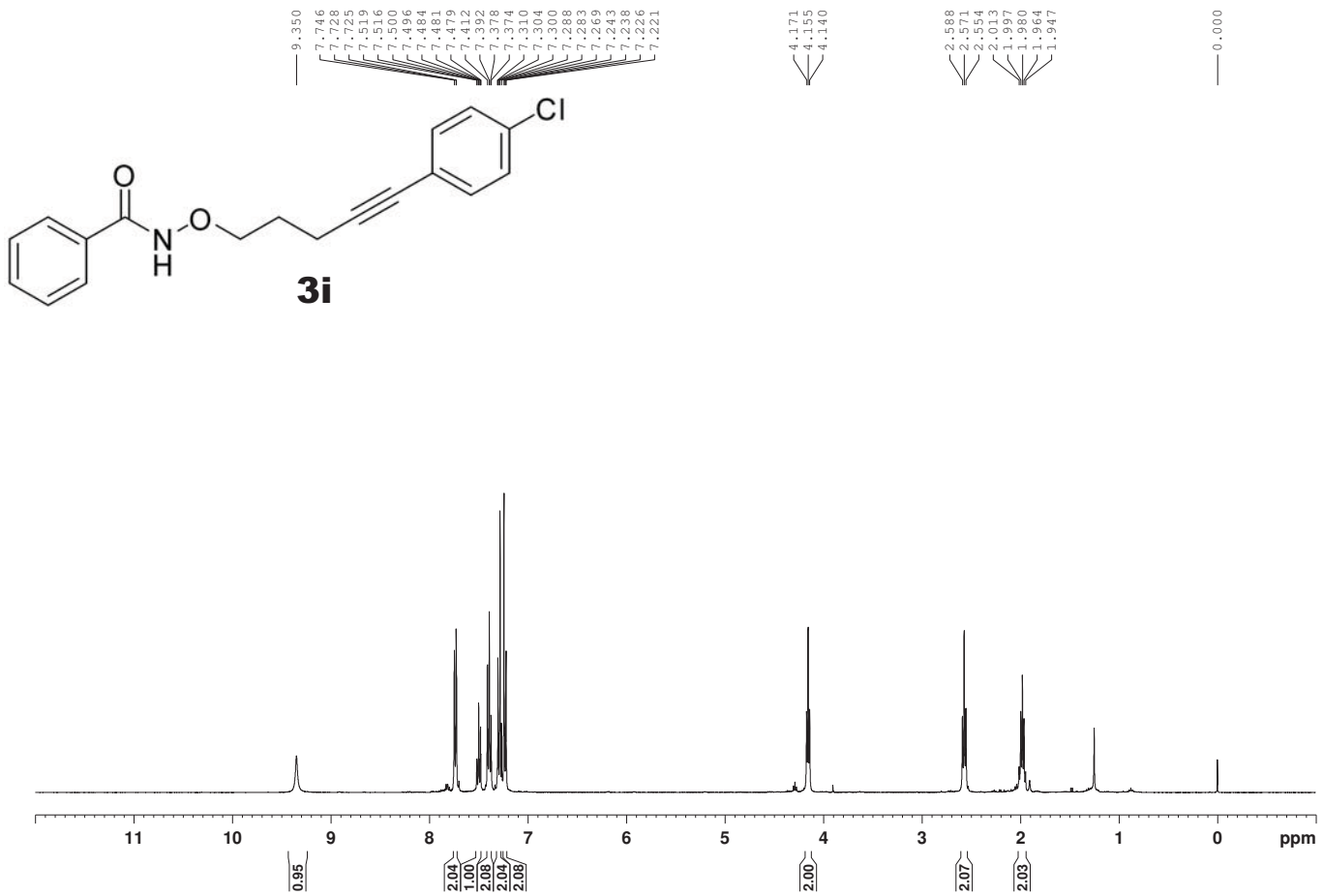
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7.725
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7.500
7.496
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7.479
7.412
7.392
7.378
7.374
7.310
7.304
7.300
7.288
7.283
7.269
7.243
7.238
7.226
7.221
4.171
4.155
4.140
2.588
2.571
2.554
2.013
1.997
1.980
1.964
1.947
0.000
Cl
O
N
H
O
3i
11
10
9
8
7
6
5
4
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2.04
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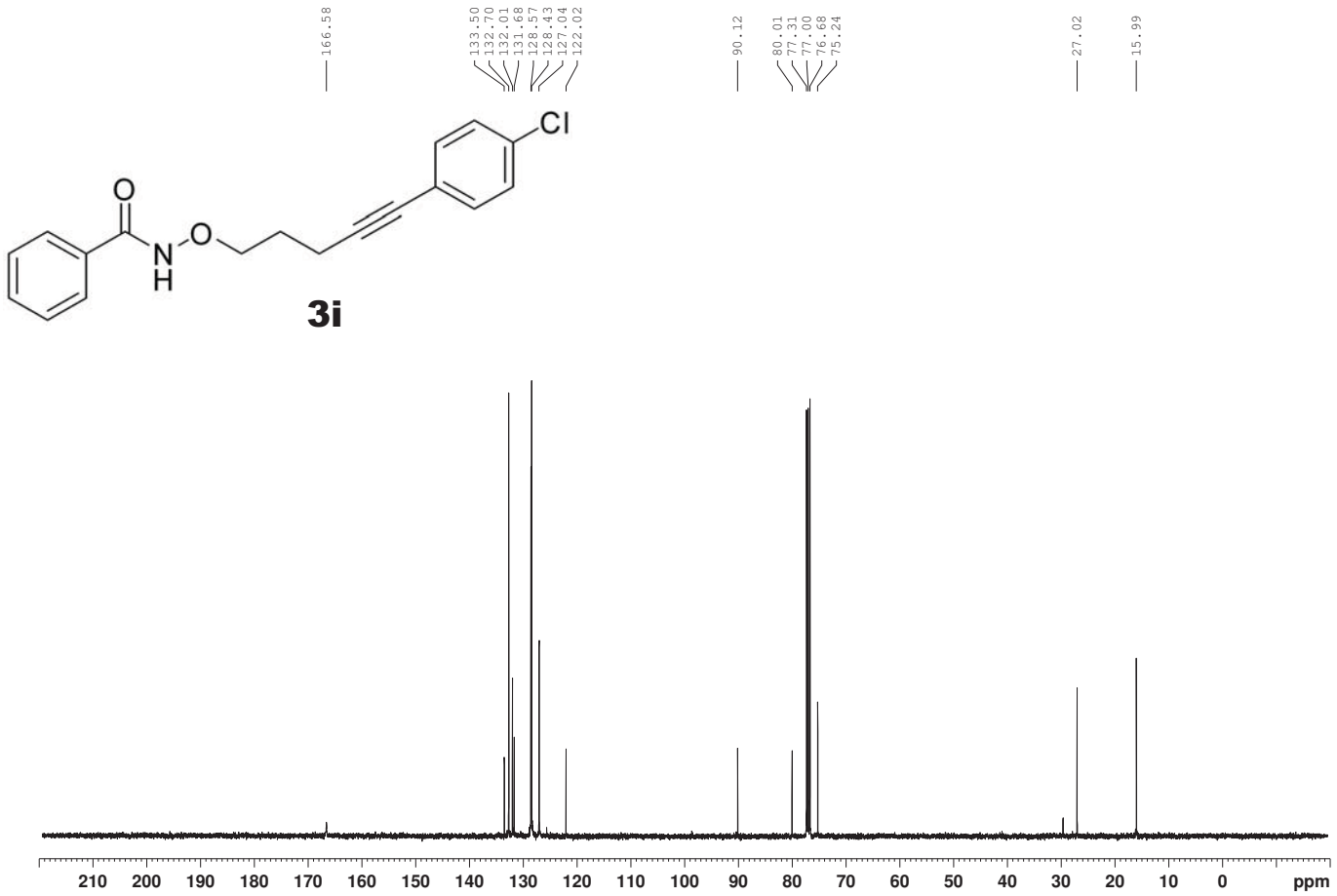
2.07
2.03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77.00
76.68
75.24
27.02
15.99
Cl
O
N
H
O
3i
210
200
190
180
170
160
150
140
130
120
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100
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80
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60
50
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30
20
10
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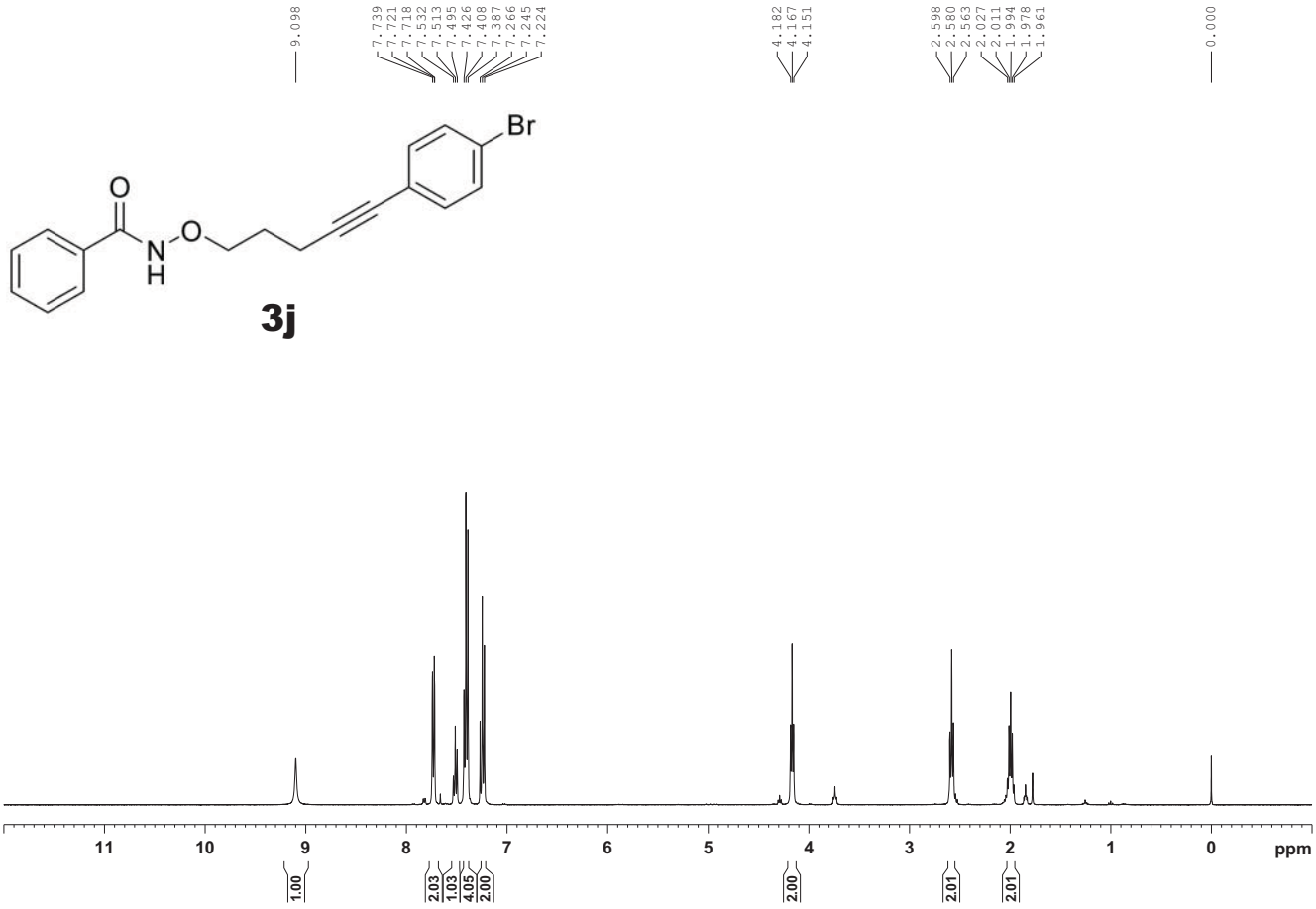
ppm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7.721
7.718
7.532
7.513
7.495
7.426
7.408
7.387
7.266
7.245
7.224
4.182
4.167
4.151
2.598
2.580
2.563
2.027
2.011
1.994
1.978
1.961
0.000
Br
O
N
O
H
3j
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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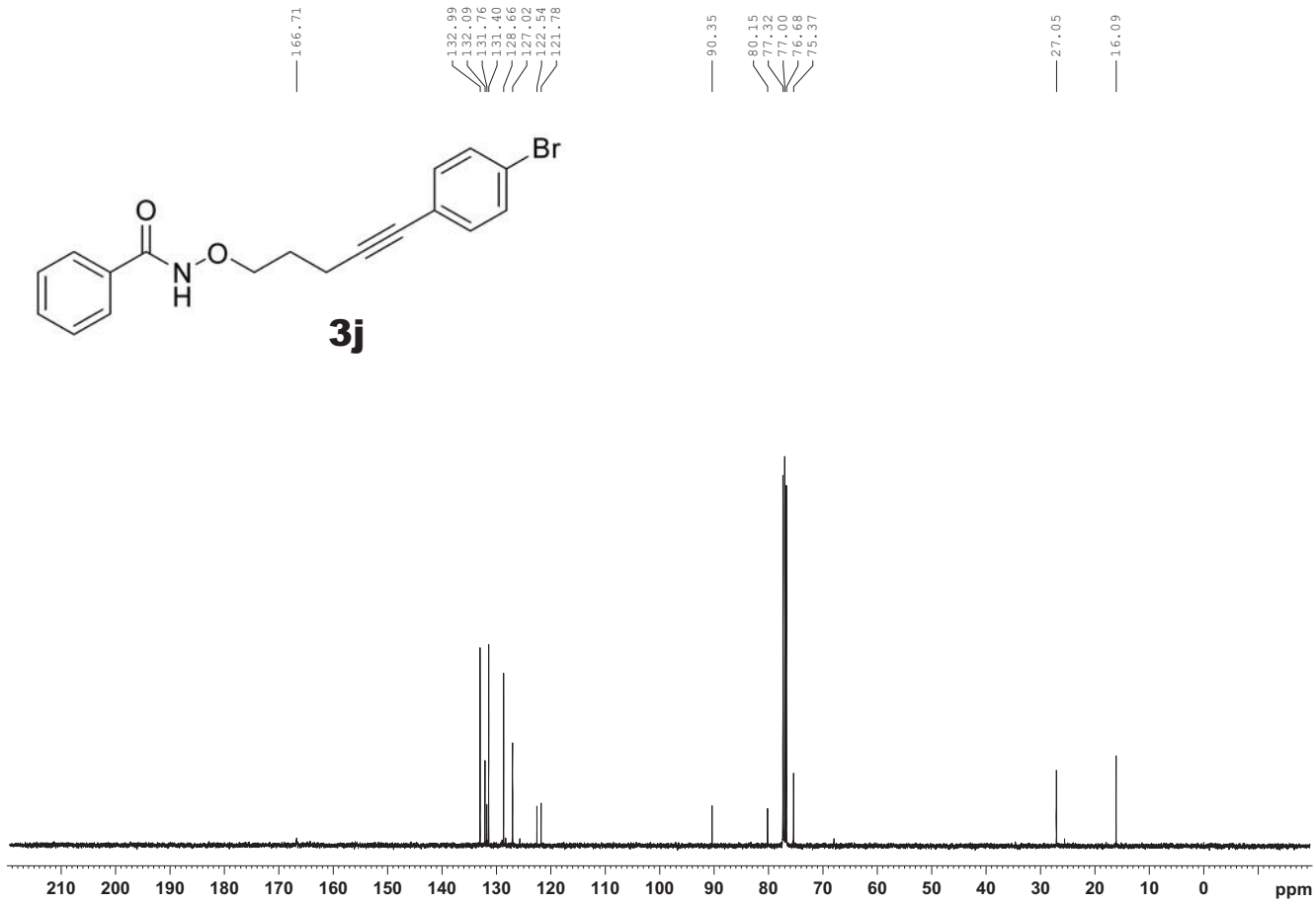
2.03
1.03
4.05
2.00
2.00
2.01
2.01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131.76
131.40
128.66
127.02
122.54
121.78
90.35
80.15
77.32
77.00
76.68
75.37
27.05
16.09
Br
O
N
O
H
3j
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
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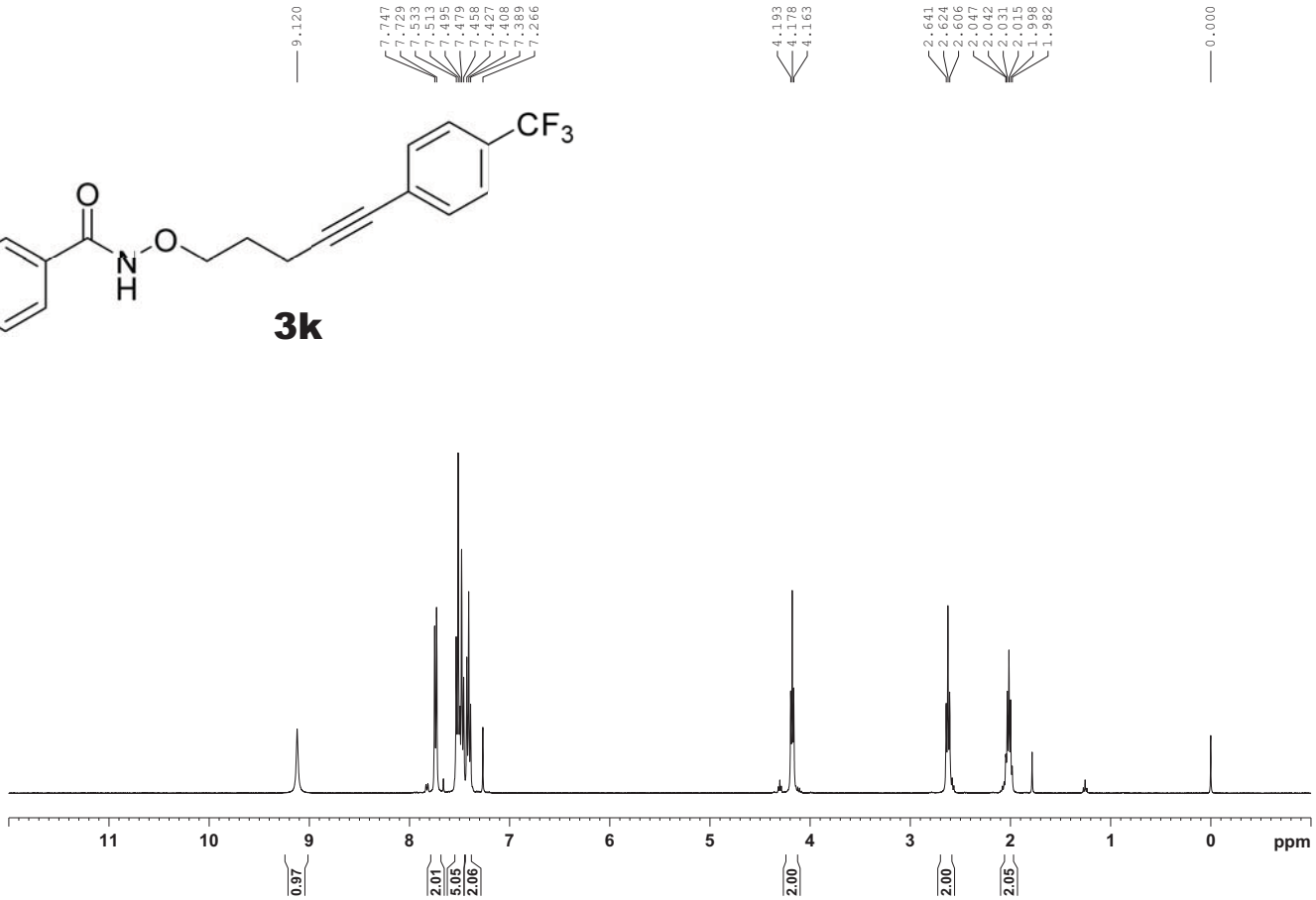
30
20
10
0
ppm

CF3
O
N
H
O

3k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7.533
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7.495
7.479
7.458
7.427
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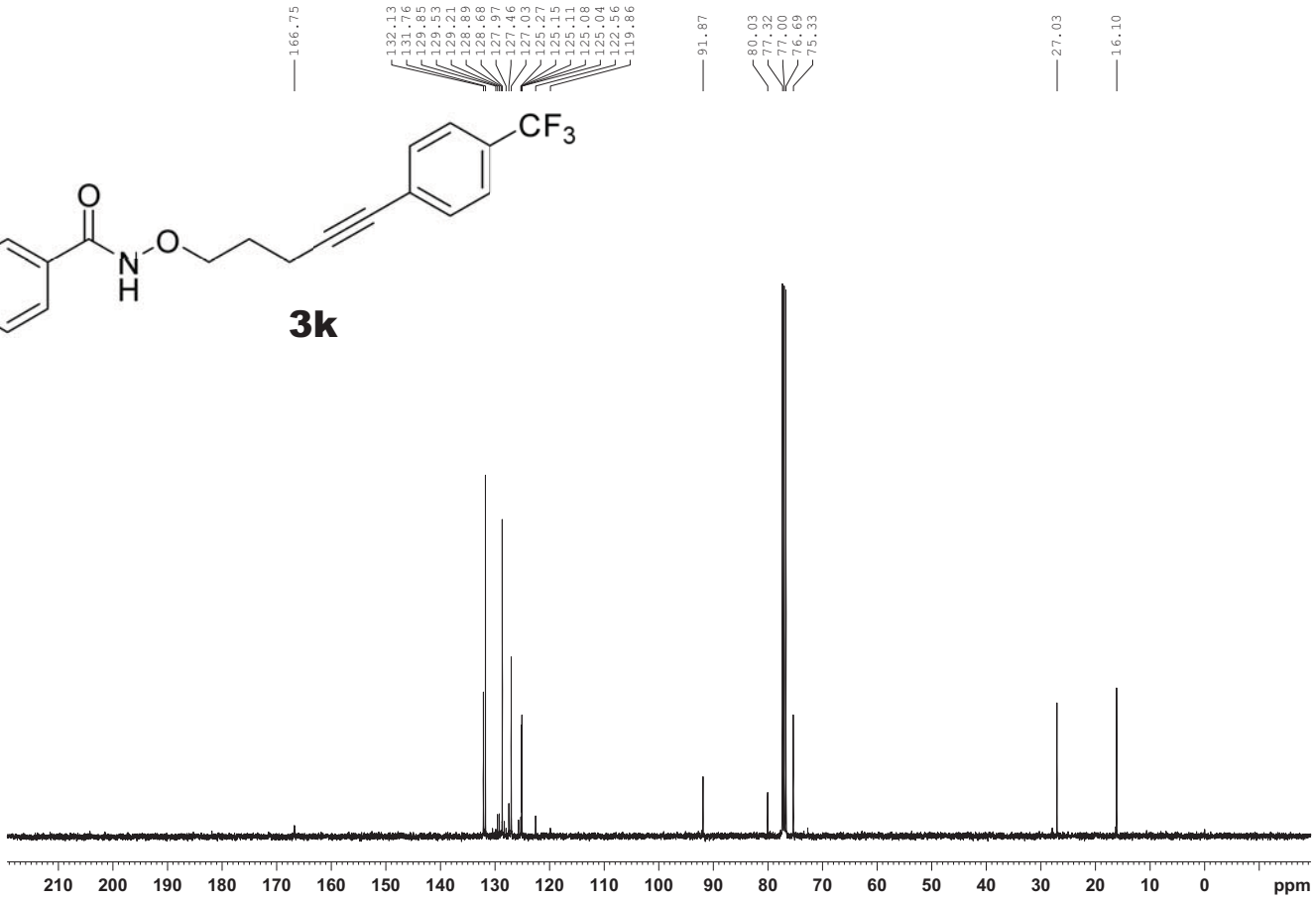
7.389
7.266
4.193
4.178
4.163
2.641
2.624
2.606
2.047
2.042
2.031
2.015
1.998
1.982
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.97
2.01
5.05
2.06
2.00
2.00
2.05

CF3
O
N
H
O

3k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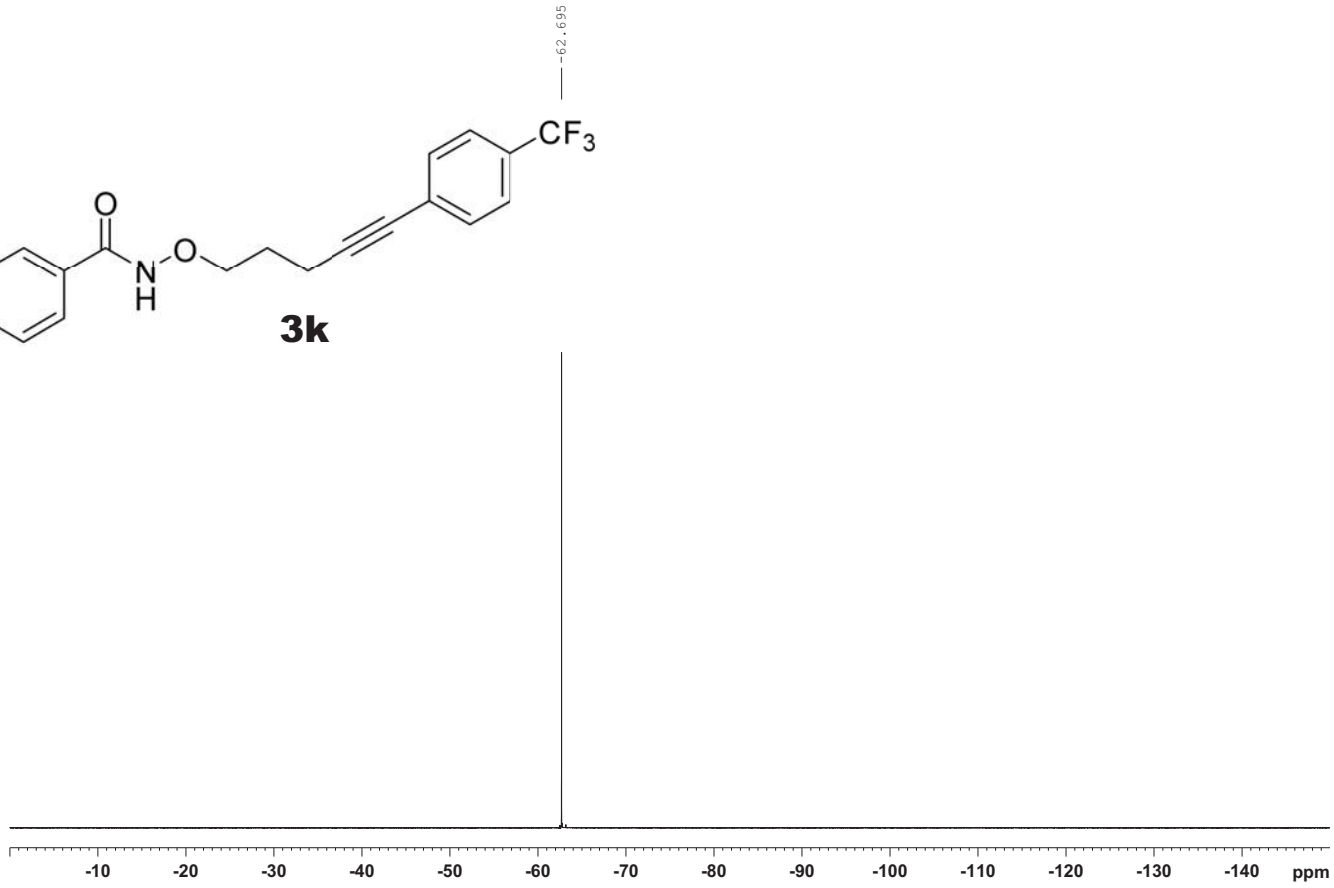
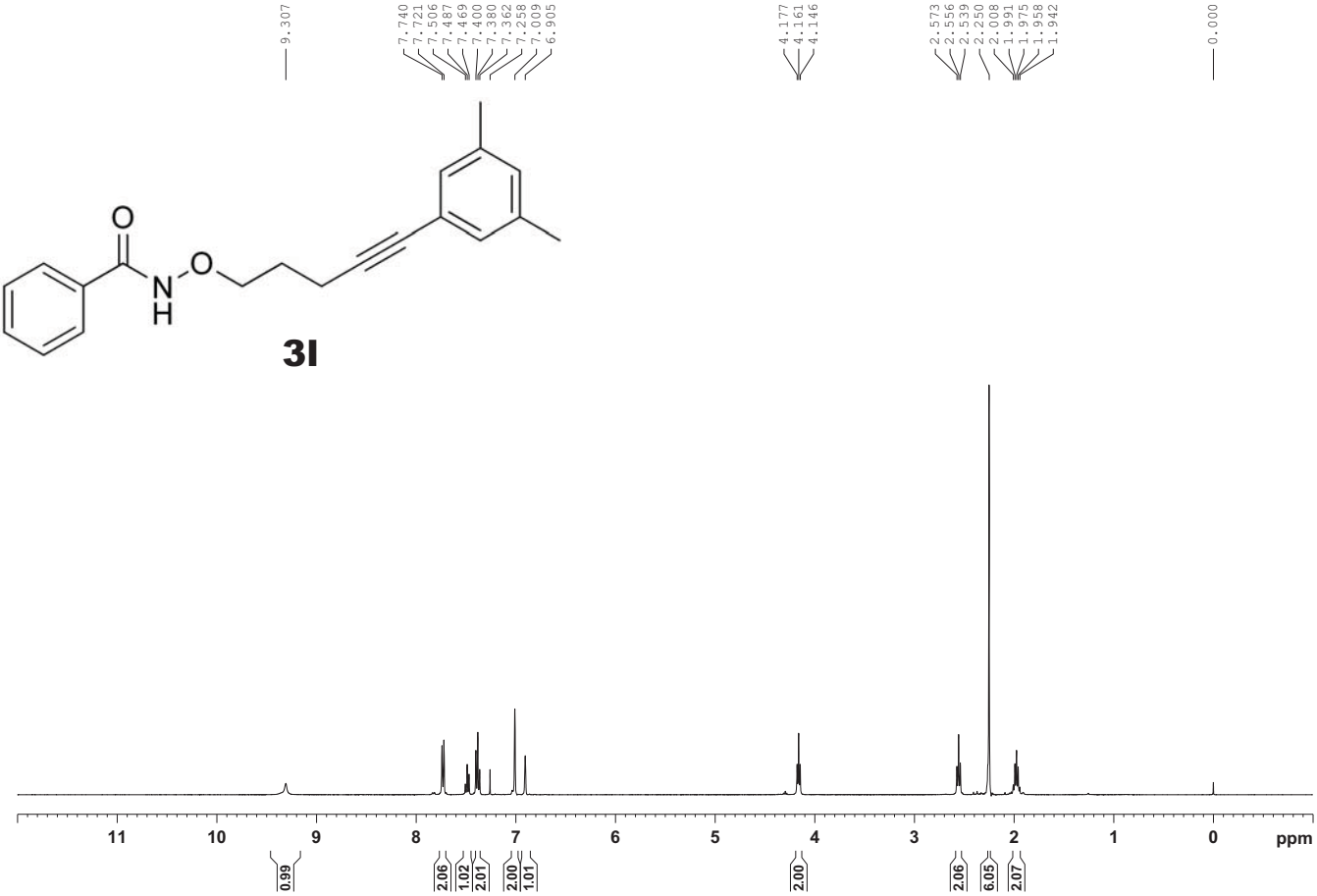
132.13
131.76
129.85
129.53
129.21
128.89
128.68
127.97
127.46
127.03
125.27
125.15
125.11
125.08
125.04
122.56
119.86
91.87
80.03
77.32
77.00
76.69
75.33
27.03
16.10
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3k



3l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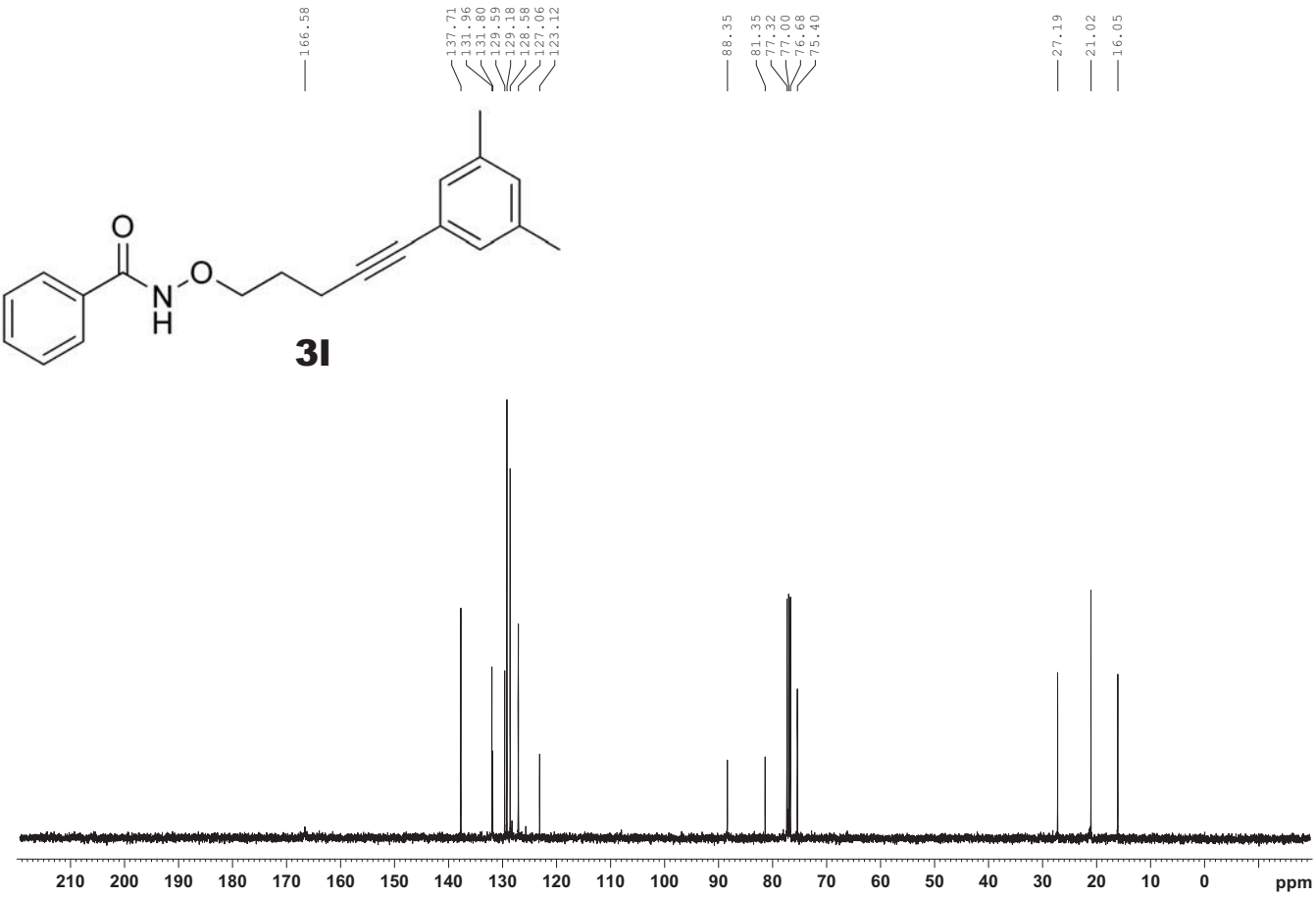
7.721
7.506
7.487
7.469
7.400
7.380
7.362
7.258
7.009
6.905
4.177
4.161
4.146
2.573
2.556
2.539
2.250
2.008
1.991
1.975
1.958
1.942
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.99
2.06
1.02
2.01
2.00
1.01
2.00
2.06
6.05
2.07

3l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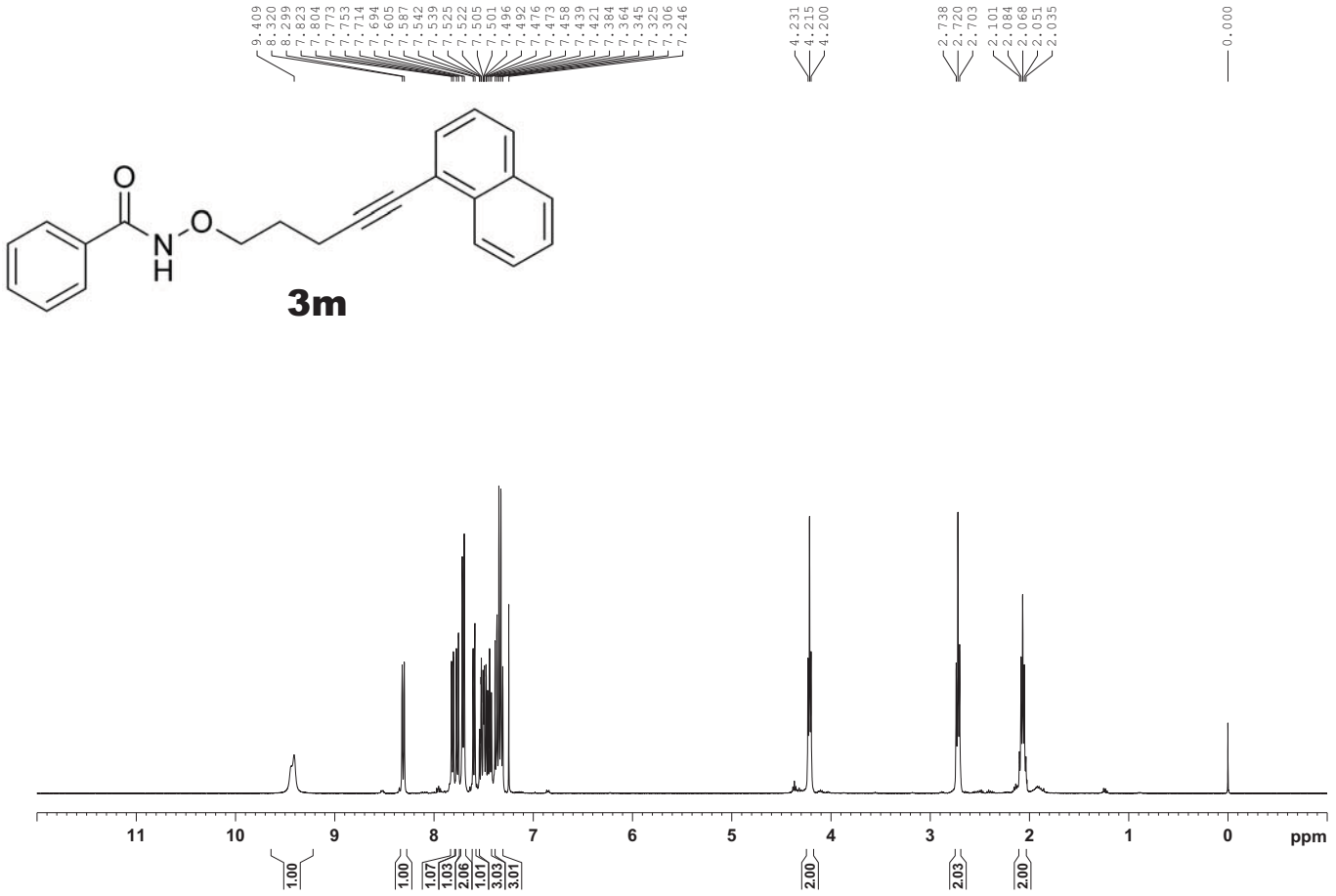
131.80
129.59
129.18
128.58
127.06
123.12
88.35
81.35
77.32
77.00
76.68
75.40
27.19
21.02
16.05
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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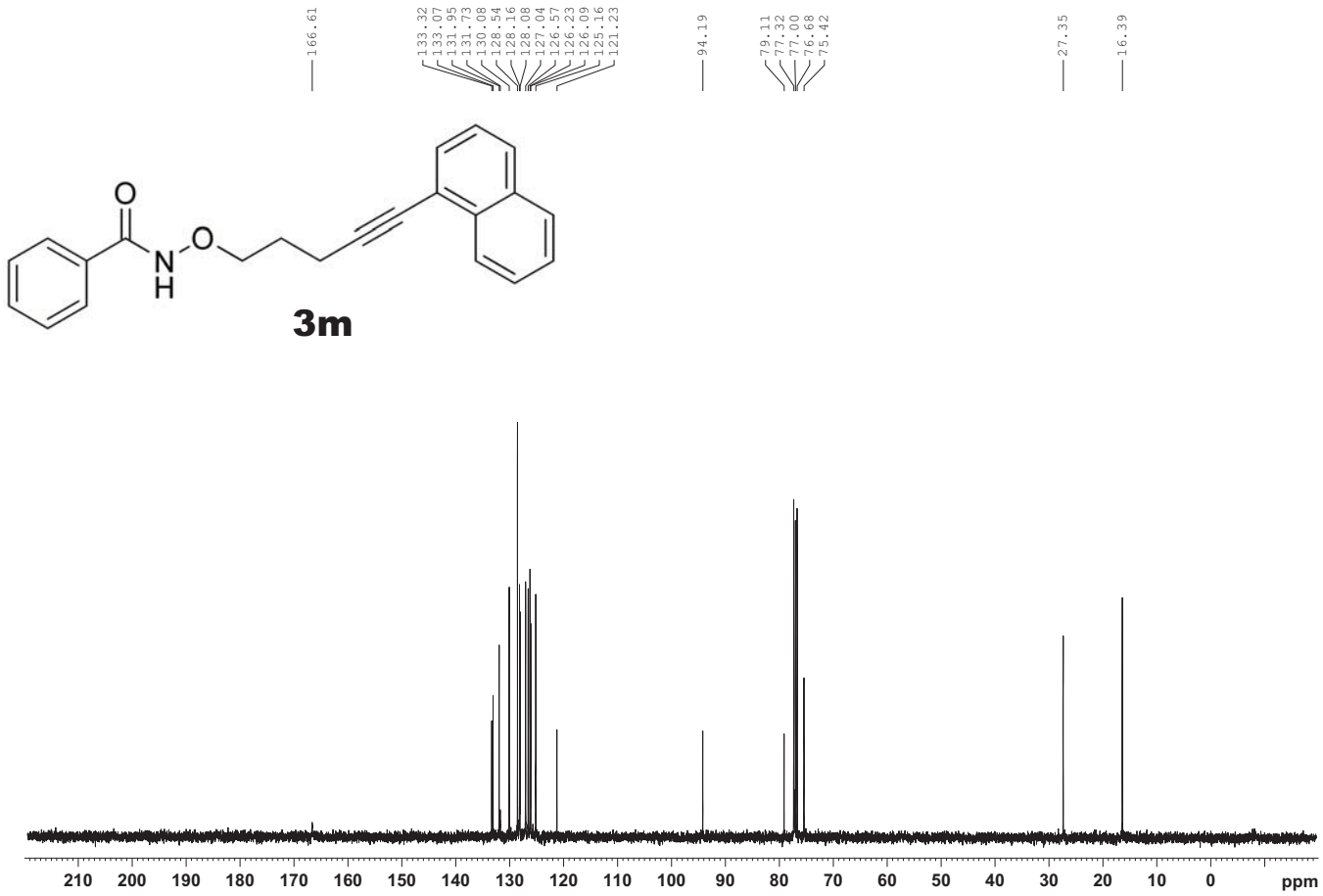
8.299
7.823
7.804
7.773
7.753
7.714
7.694
7.605
7.587
7.542
7.539
7.525
7.522
7.505
7.501
7.496
7.492
7.476
7.473
7.458
7.439
7.421
7.384
7.364
7.345
7.325
7.306
7.246
4.231
4.215
4.200
2.738
2.720
2.703
2.101
2.084
2.068
2.051
2.035
0.000
O
N
H
O
3m
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.00
1.07
1.03
2.06
1.01
3.03
3.01
2.00
2.03
2.00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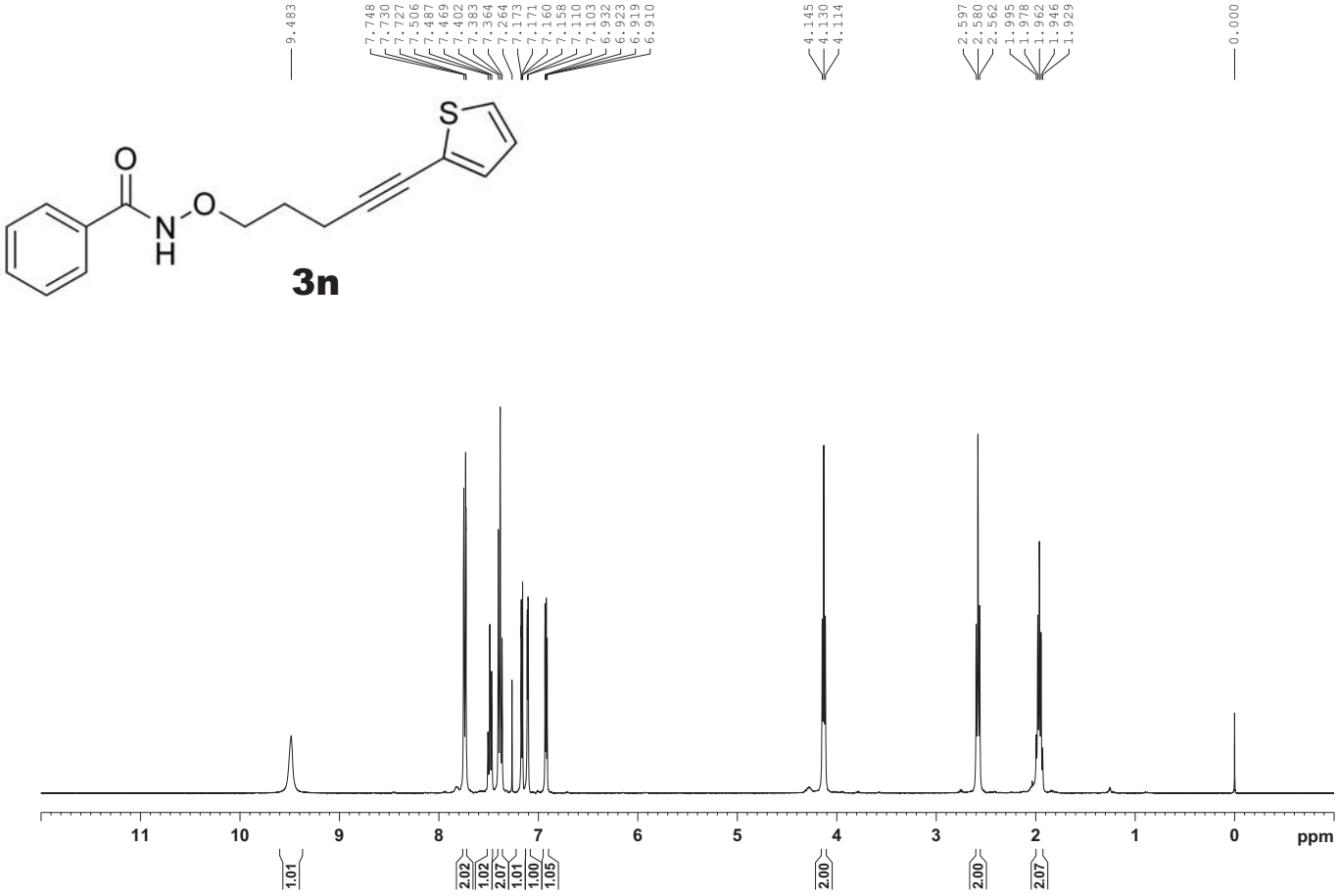
133.07
131.95
131.73
130.08
128.54
128.16
128.08
127.04
126.57
126.23
126.09
125.16
121.23
94.19
79.11
77.32
77.00
76.68
75.42
27.35
16.39
O
N
H
O
3m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.727
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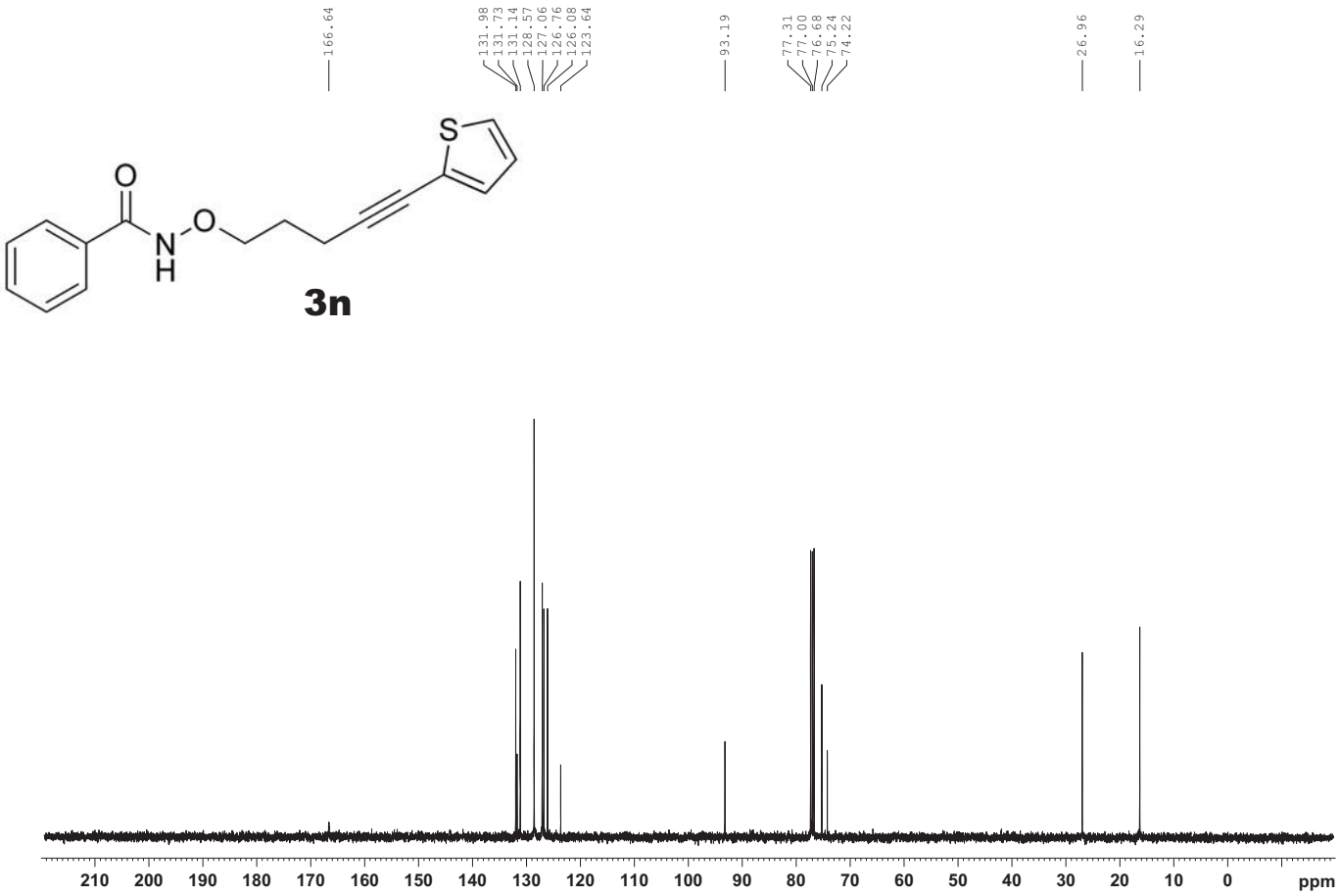
7.487
7.469
7.402
7.383
7.364
7.264
7.173
7.171
7.160
7.158
7.110
7.103
6.932
6.923
6.919
6.910
4.145
4.130
4.114
2.597
2.580
2.562
1.995
1.978
1.962
1.946
1.929
0.000
S
O
N
O
H
3n
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
2.02
1.02
2.07
1.01
1.00
1.05
2.00
2.00
2.07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131.73
131.14
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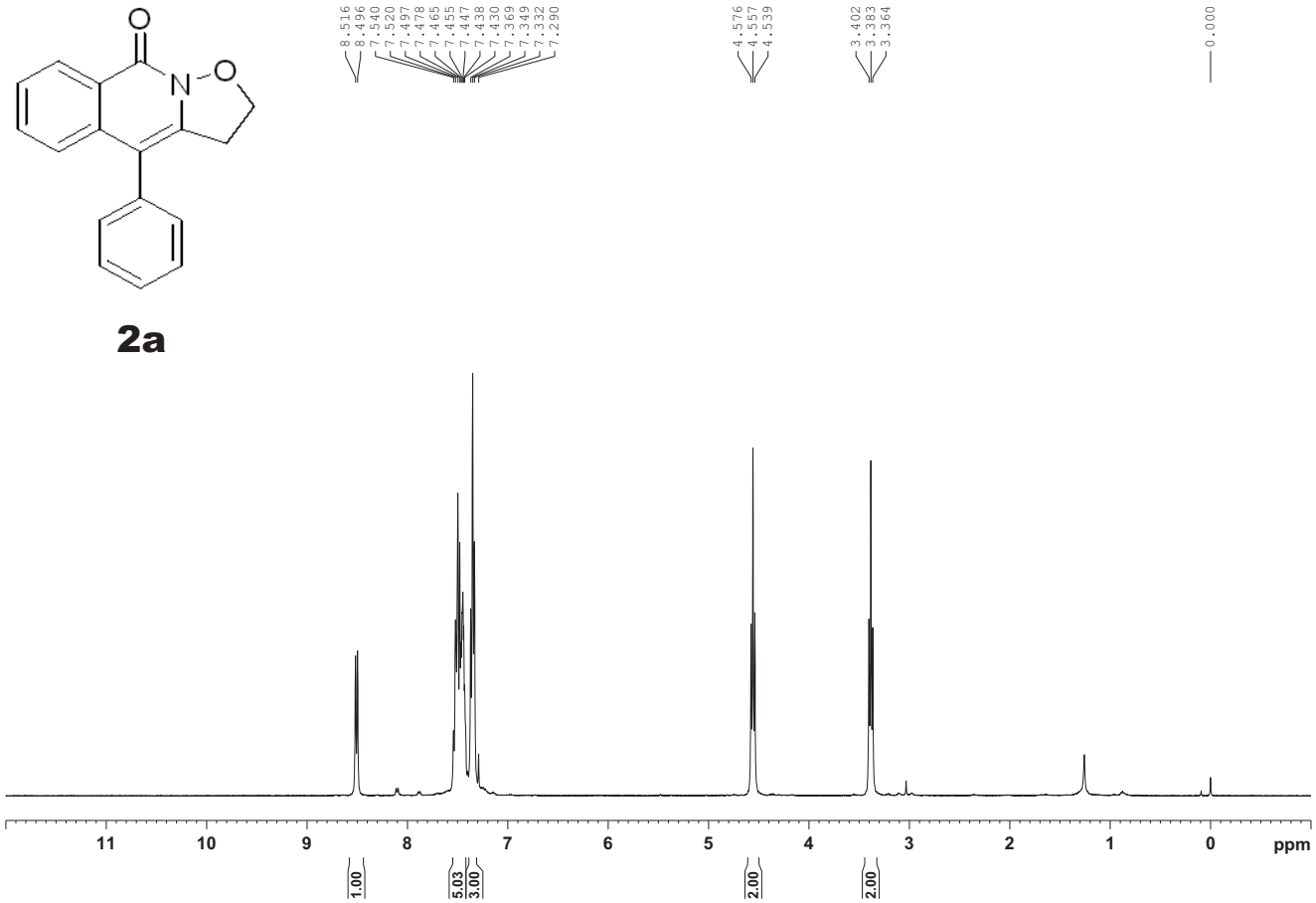
127.06
126.76
126.08
123.64
93.19
77.31
77.00
76.68
75.24
74.22
26.96
16.29
S
O
N
O
H
3n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

2a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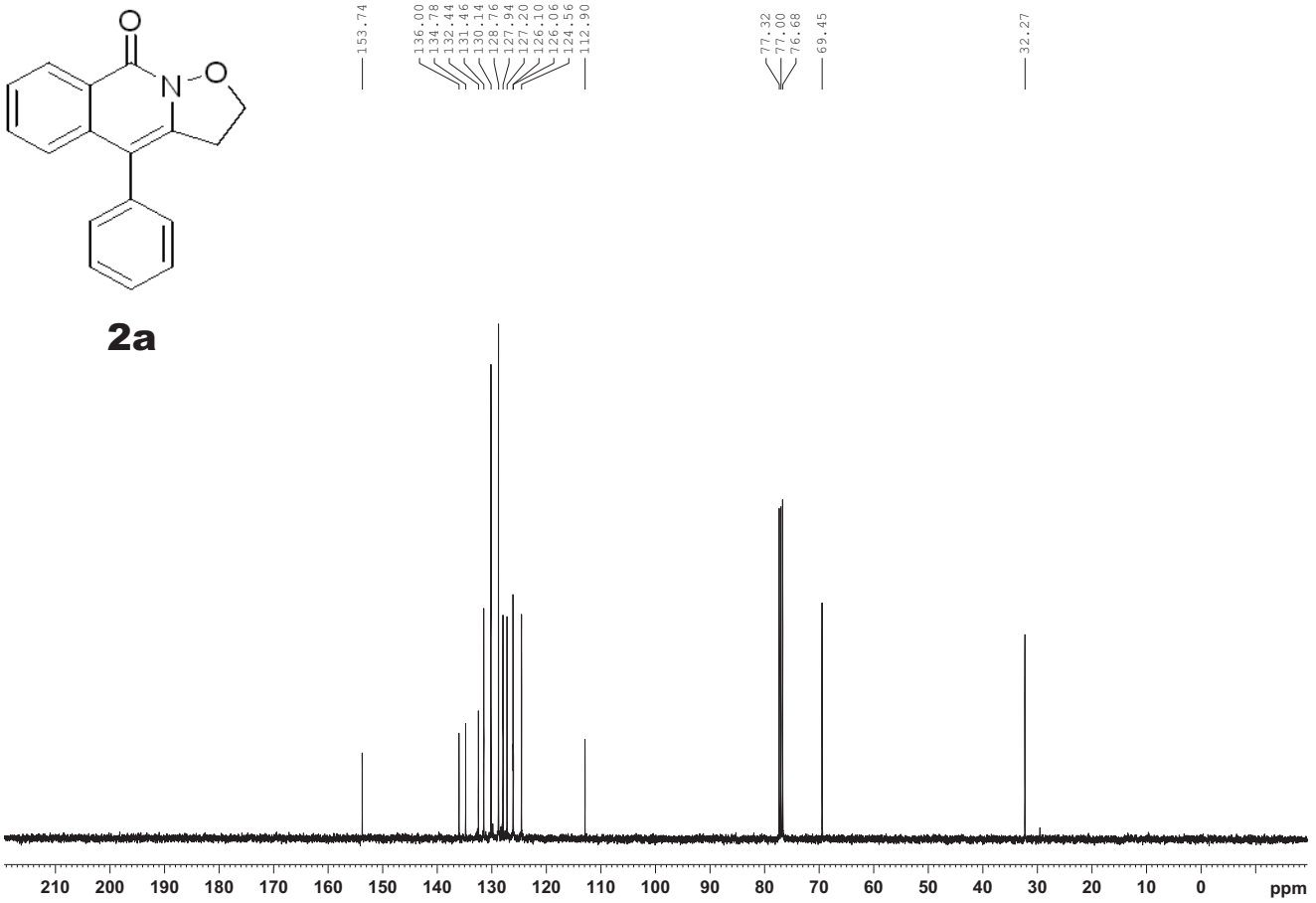
7.540
7.520
7.497
7.478
7.465
7.455
7.447
7.438
7.430
7.369
7.349
7.332
7.290
4.576
4.557
4.539
3.402
3.383
3.364
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
5.03
3.00
2.00
2.00

2a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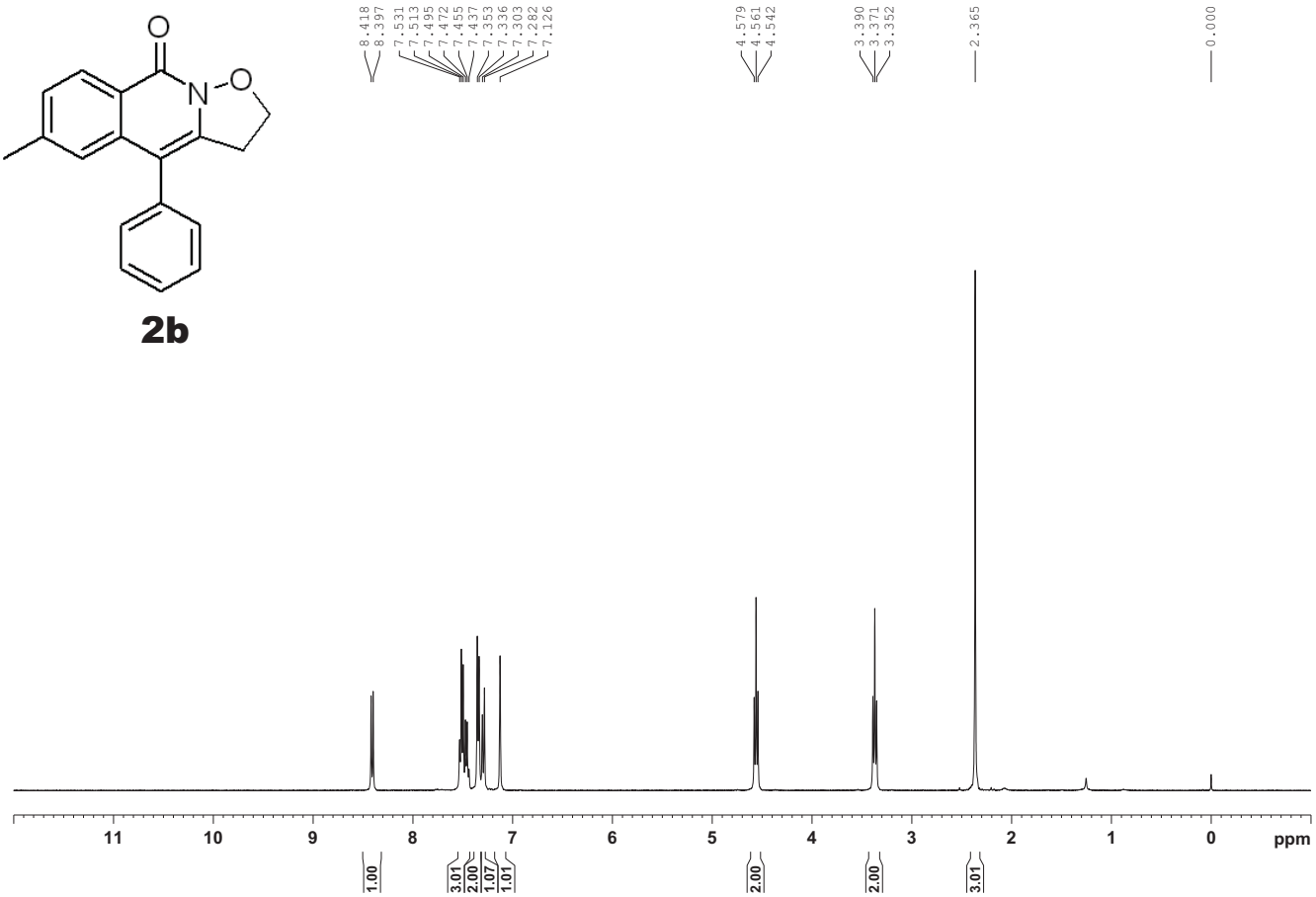
136.00
134.78
132.44
131.46
130.14
128.76
127.94
127.20
126.10
126.06
124.56
112.90
77.32
77.00
76.68
69.45
32.27
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

2b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8.397
7.531
7.513
7.495
7.472
7.455
7.437
7.353
7.336
7.303
7.282
7.126
4.579
4.561
4.542
3.390
3.371
3.352
2.365
0.000
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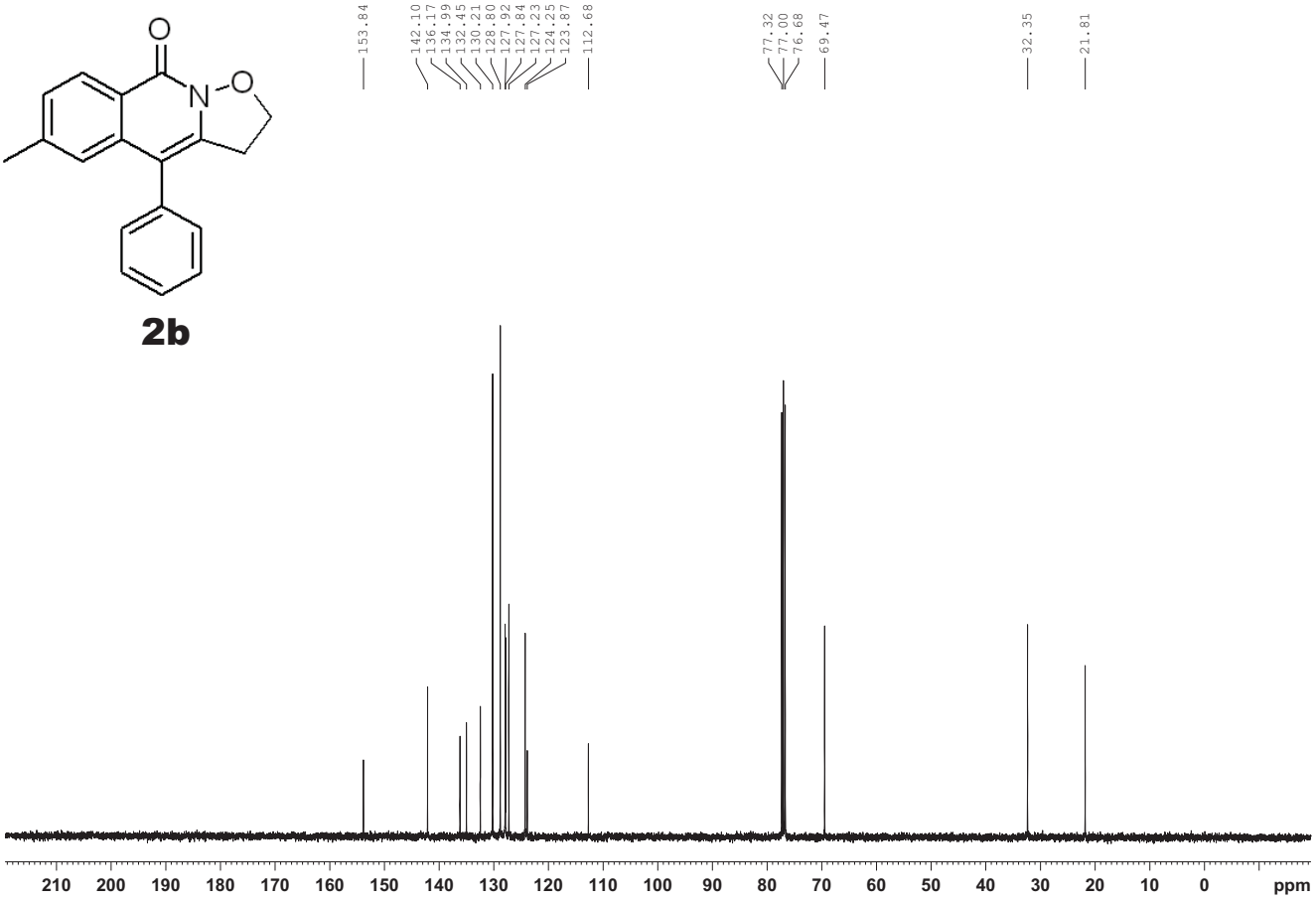
1.00
3.01
2.00
1.07
1.01
2.00
2.00
3.01

2b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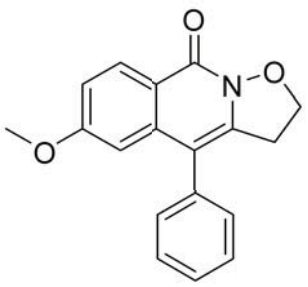
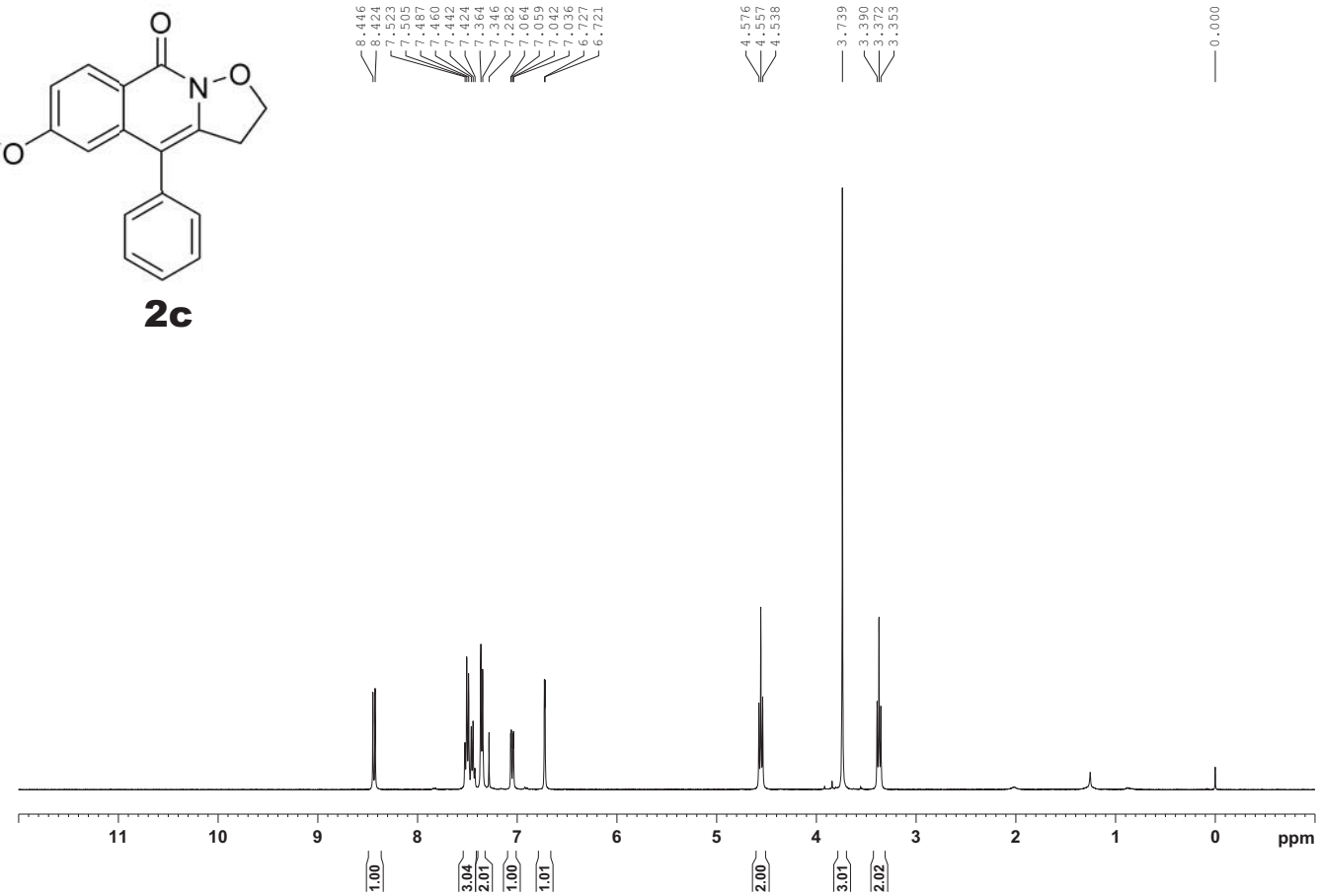
142.10
136.17
134.99
132.45
130.21
128.80
127.92
127.84
127.23
124.25
123.87
112.68
77.32
77.00
76.68
69.47
32.35
21.81
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



2c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8.424
7.523
7.505
7.487
7.460
7.442
7.424
7.364
7.346
7.282
7.064
7.059
7.042
7.036
6.727
6.721
4.576
4.557
4.538
3.739
3.390
3.372
3.353
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
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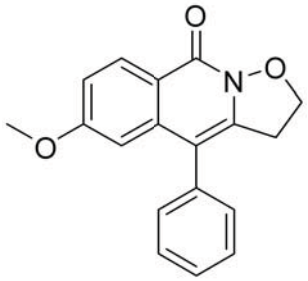
3.04
2.01
1.00
1.01
2.00
3.01
2.02



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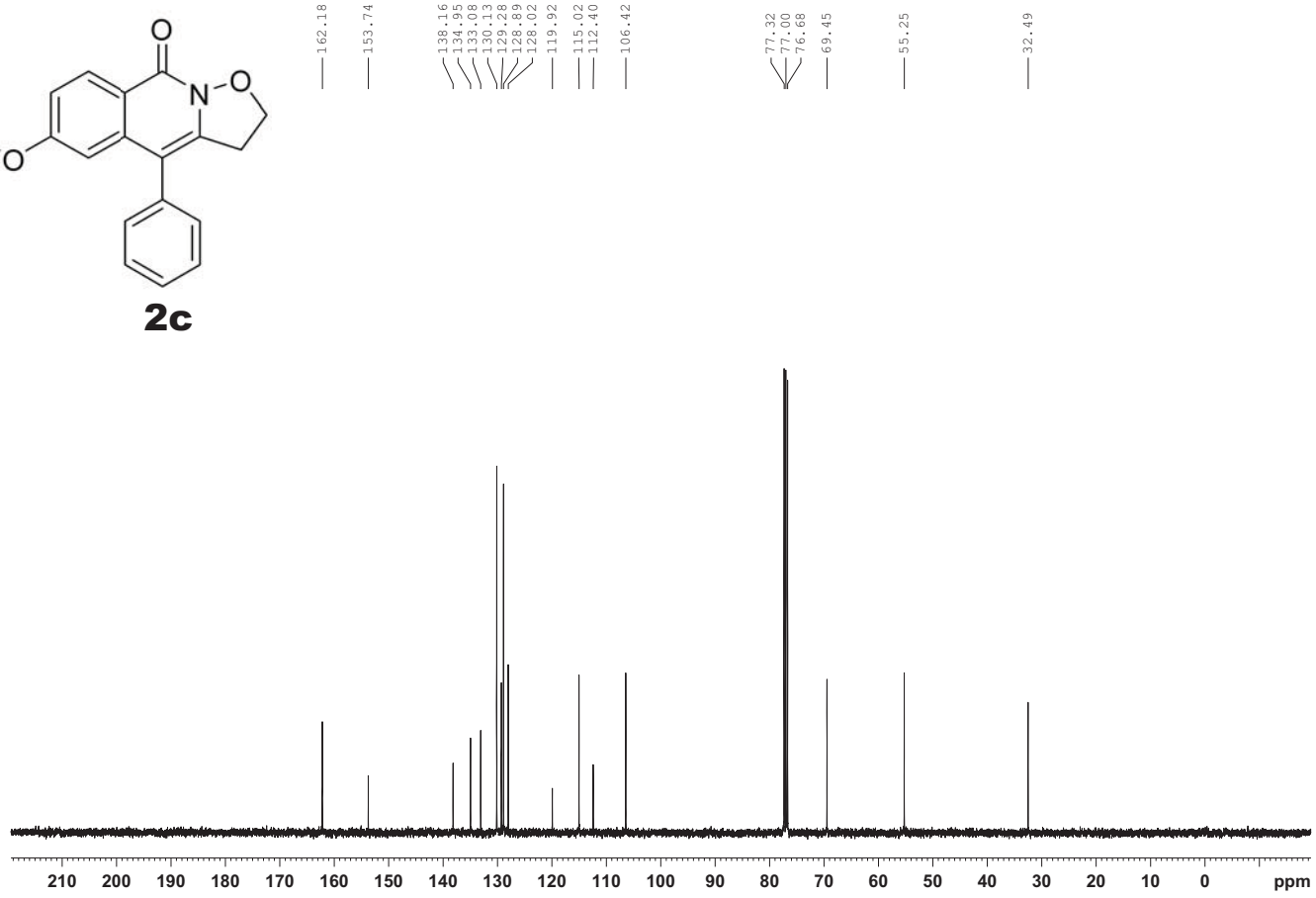
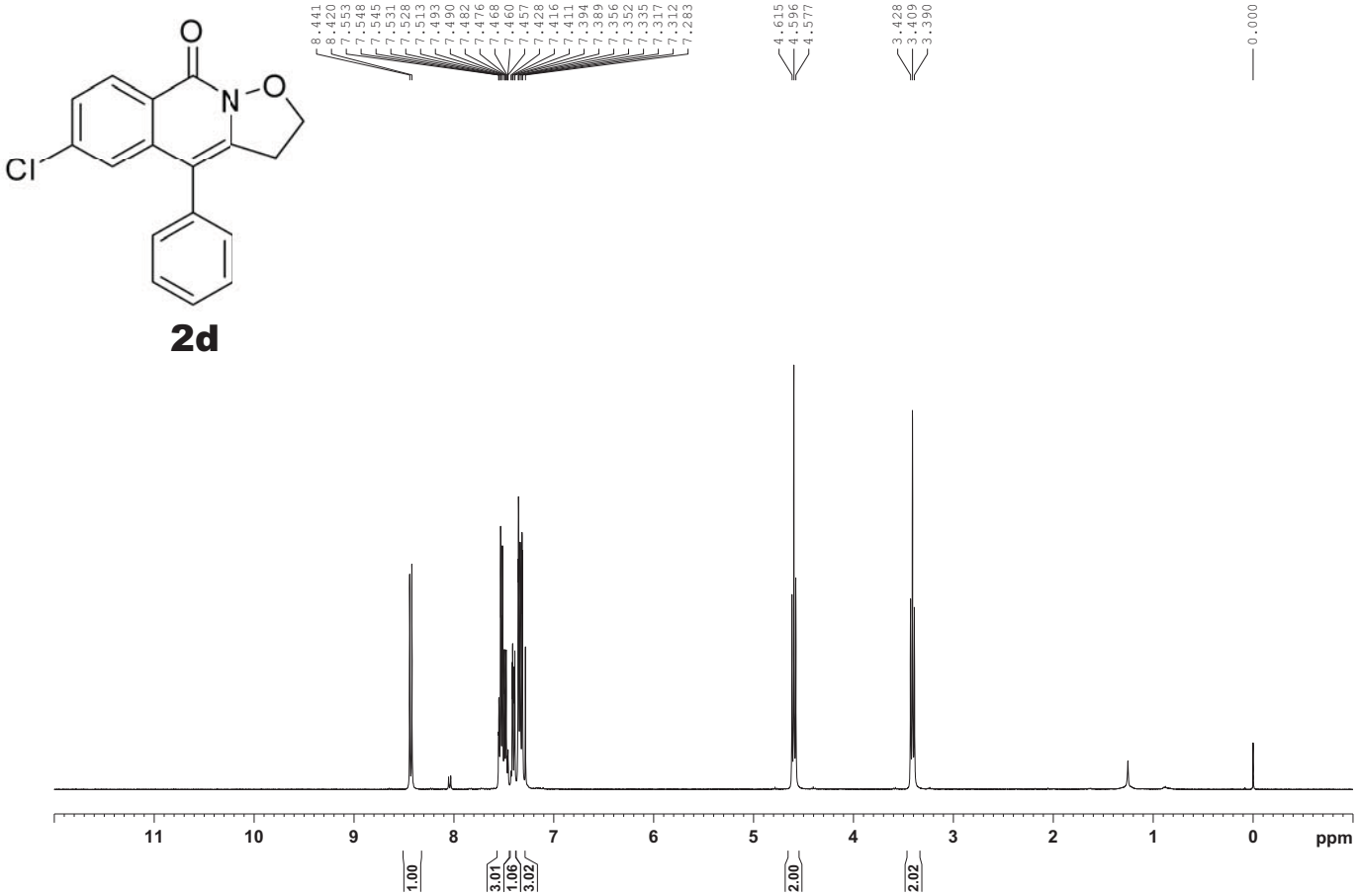

162.18
153.74
138.16
134.95
133.08
130.13
129.28
128.89
128.02
119.92
115.02
112.40
106.42
77.32
77.00
76.68
69.45
55.25
32.49
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.441
8.420
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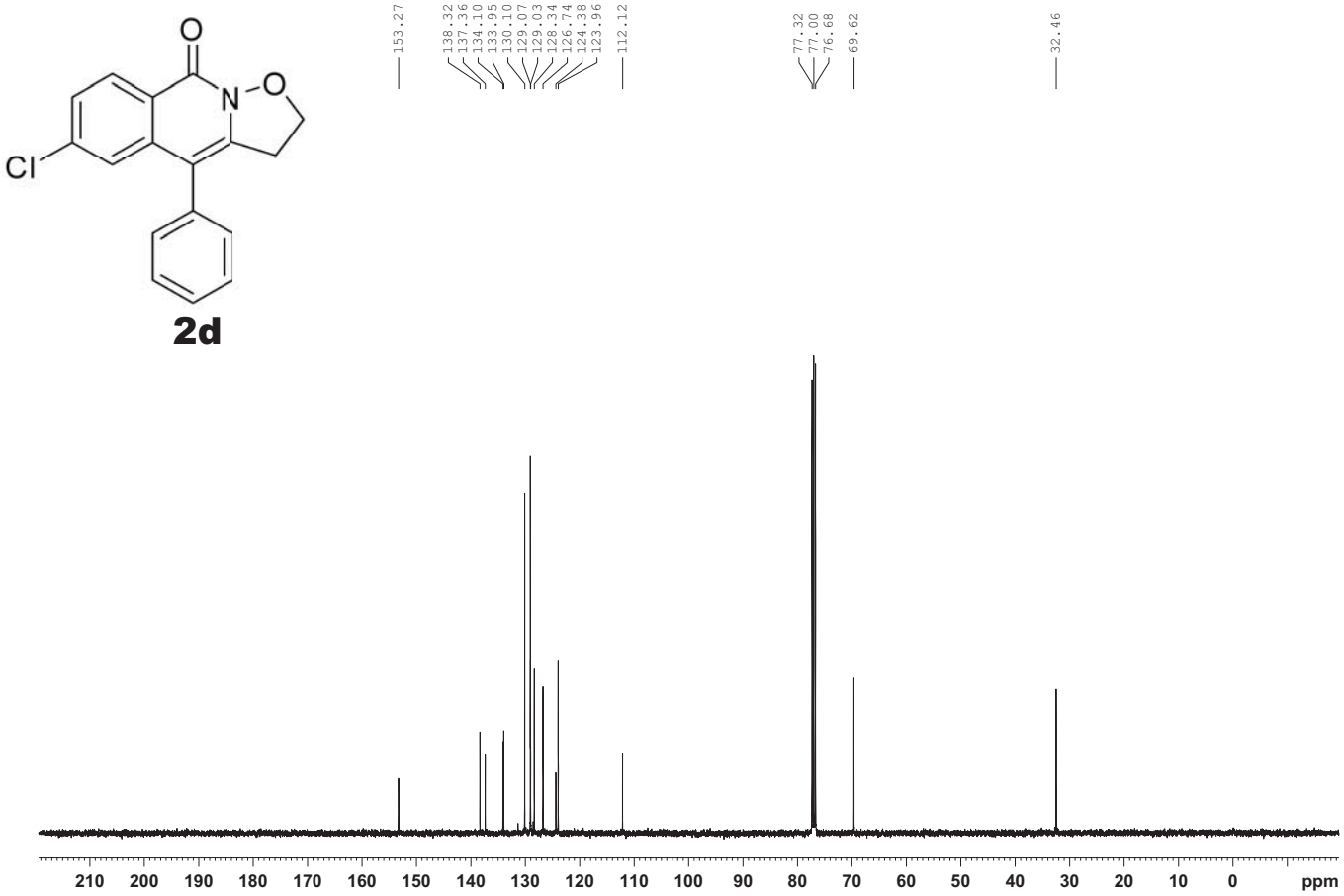
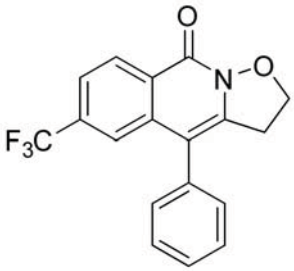
7.548
7.545
7.531
7.528
7.513
7.493
7.490
7.482
7.476
7.468
7.460
7.457
7.428
7.416
7.411
7.394
7.389
7.356
7.352
7.335
7.317
7.312
7.283
4.615
4.596
4.577
3.428
3.409
3.390
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
3.01
1.06
3.02
2.00
2.02

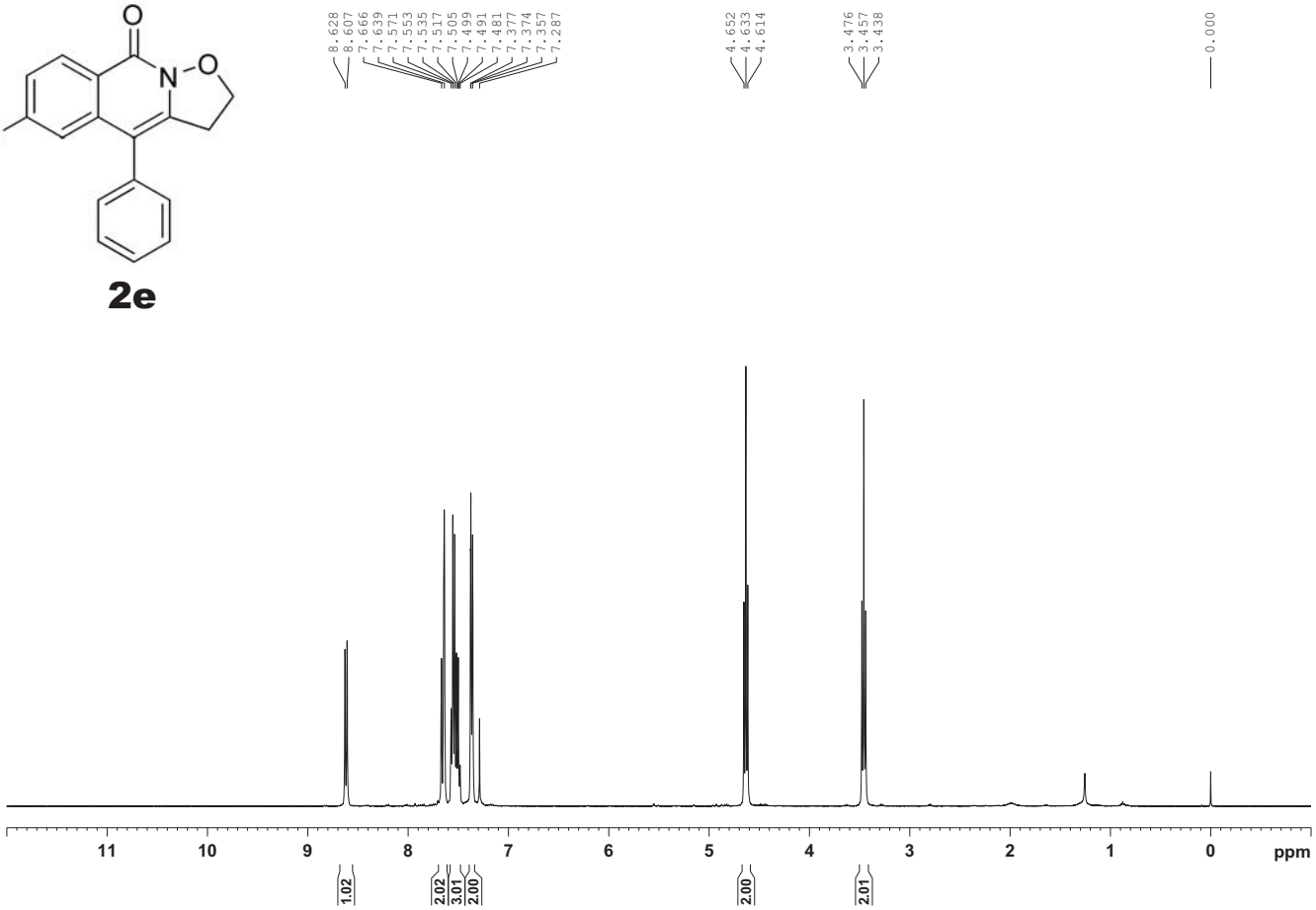
2d

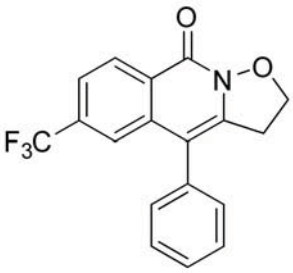

153.27
138.32
137.36
134.10
133.95
130.10
129.07
129.03
128.34
126.74
124.38
123.96
112.12
77.32
77.00
76.68
69.62
32.46
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

2d

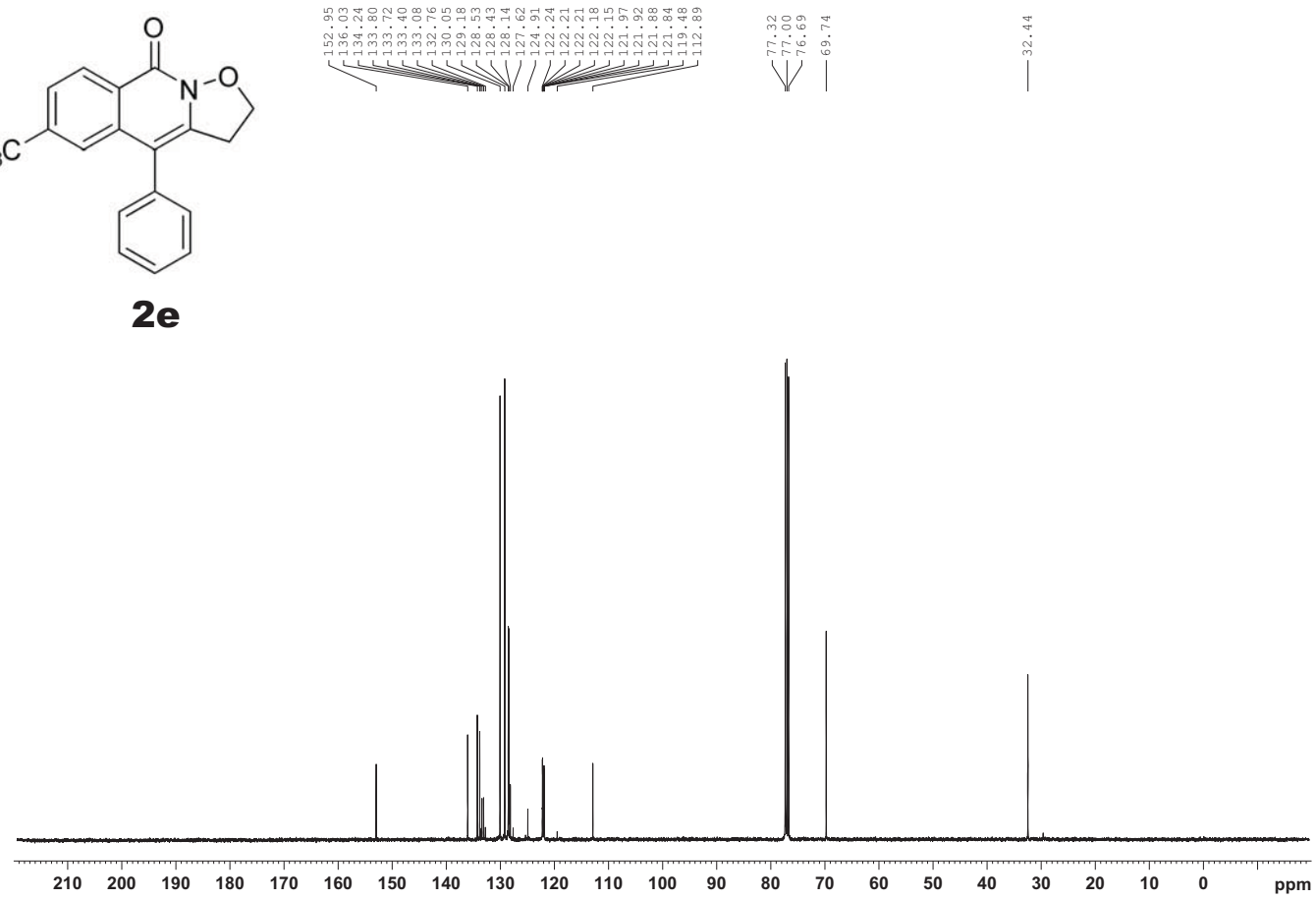


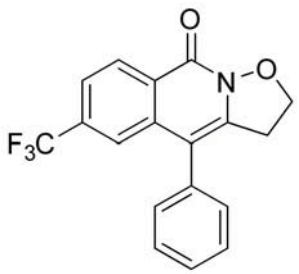
2e


8.628
8.607
7.666
7.639
7.571
7.553
7.535
7.517
7.505
7.499
7.491
7.481
7.377
7.374
7.357
7.287
4.652
4.633
4.614
3.476
3.457
3.438
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.02
2.02
3.01
2.00
2.00
2.01

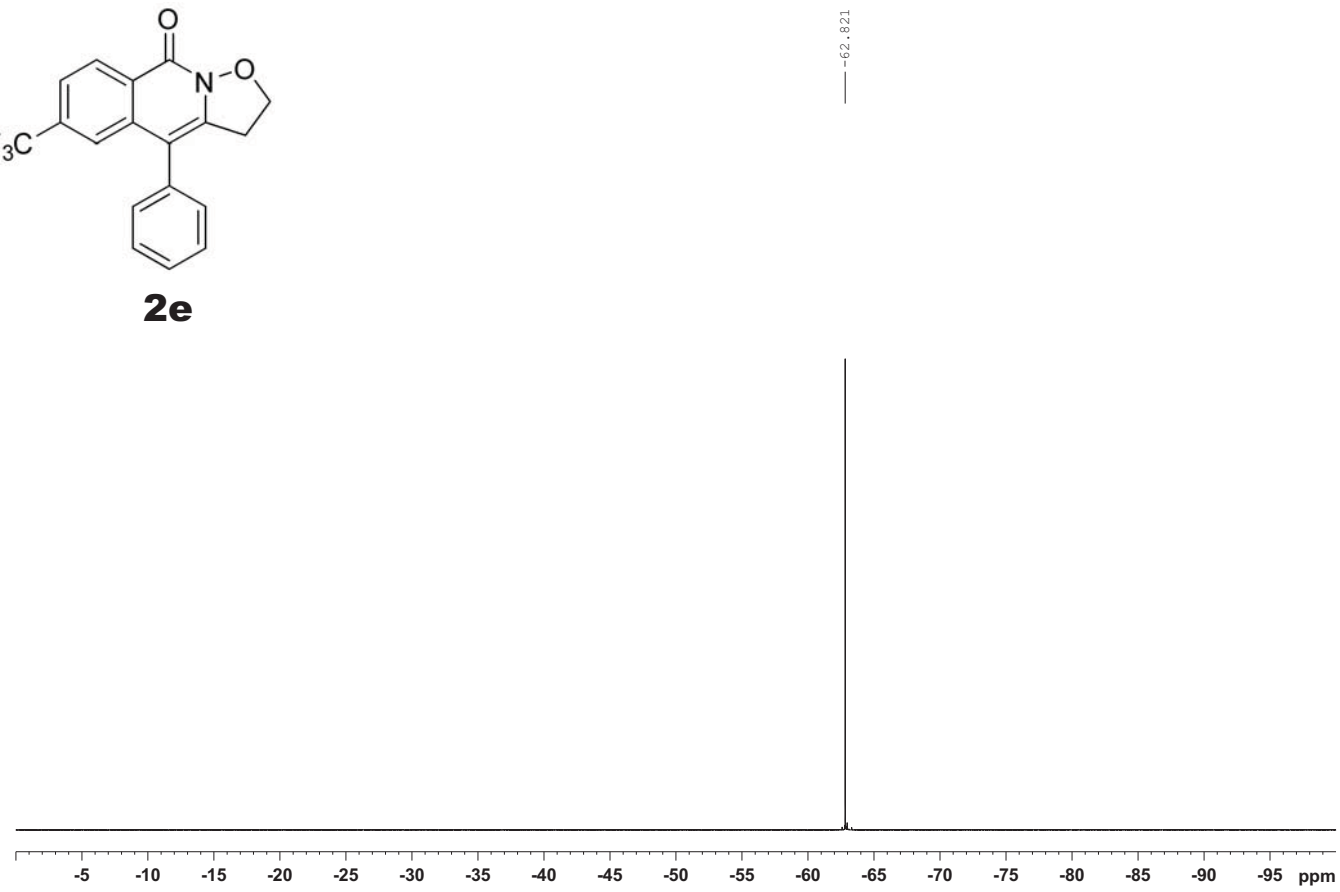


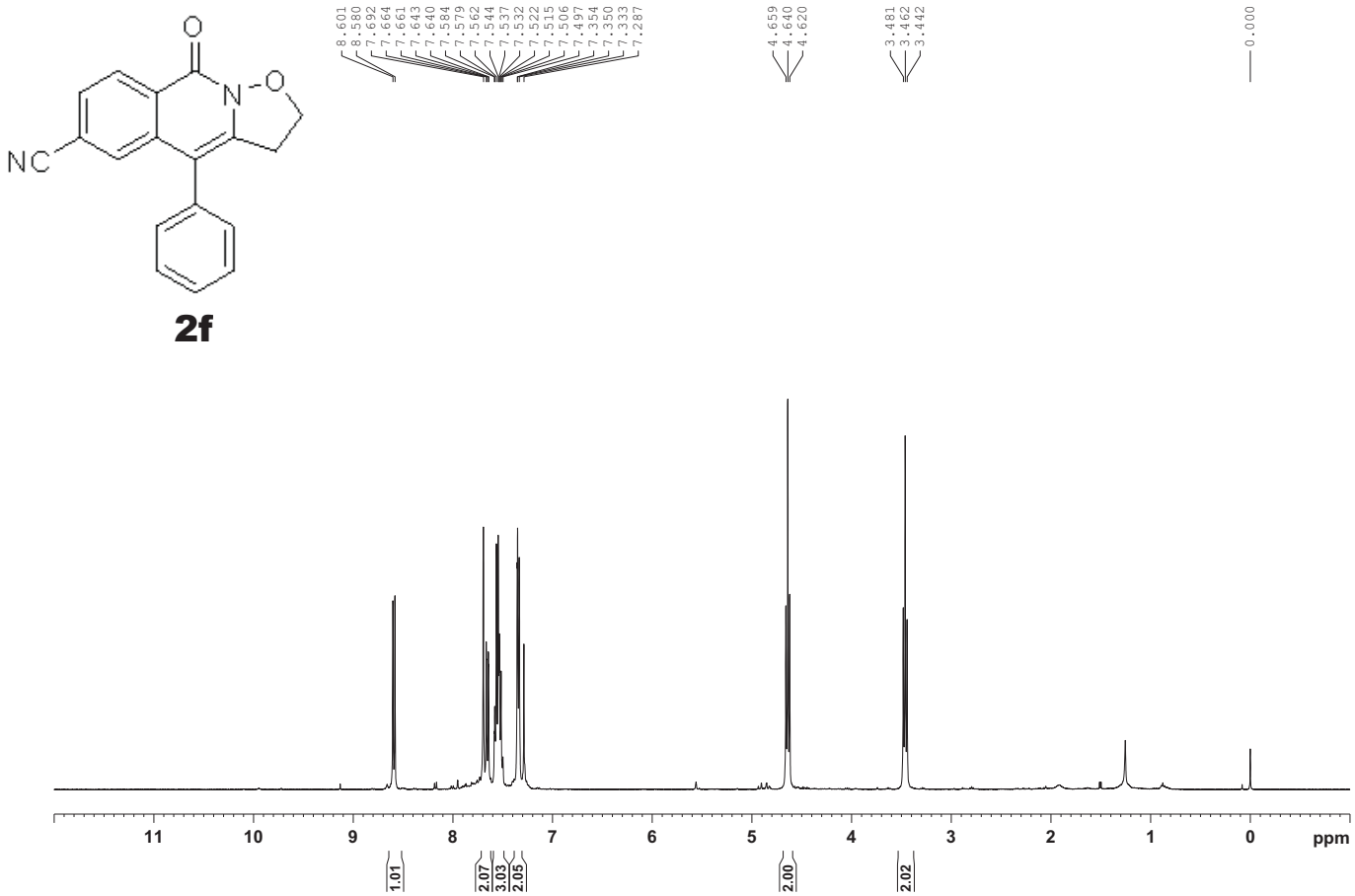
2e

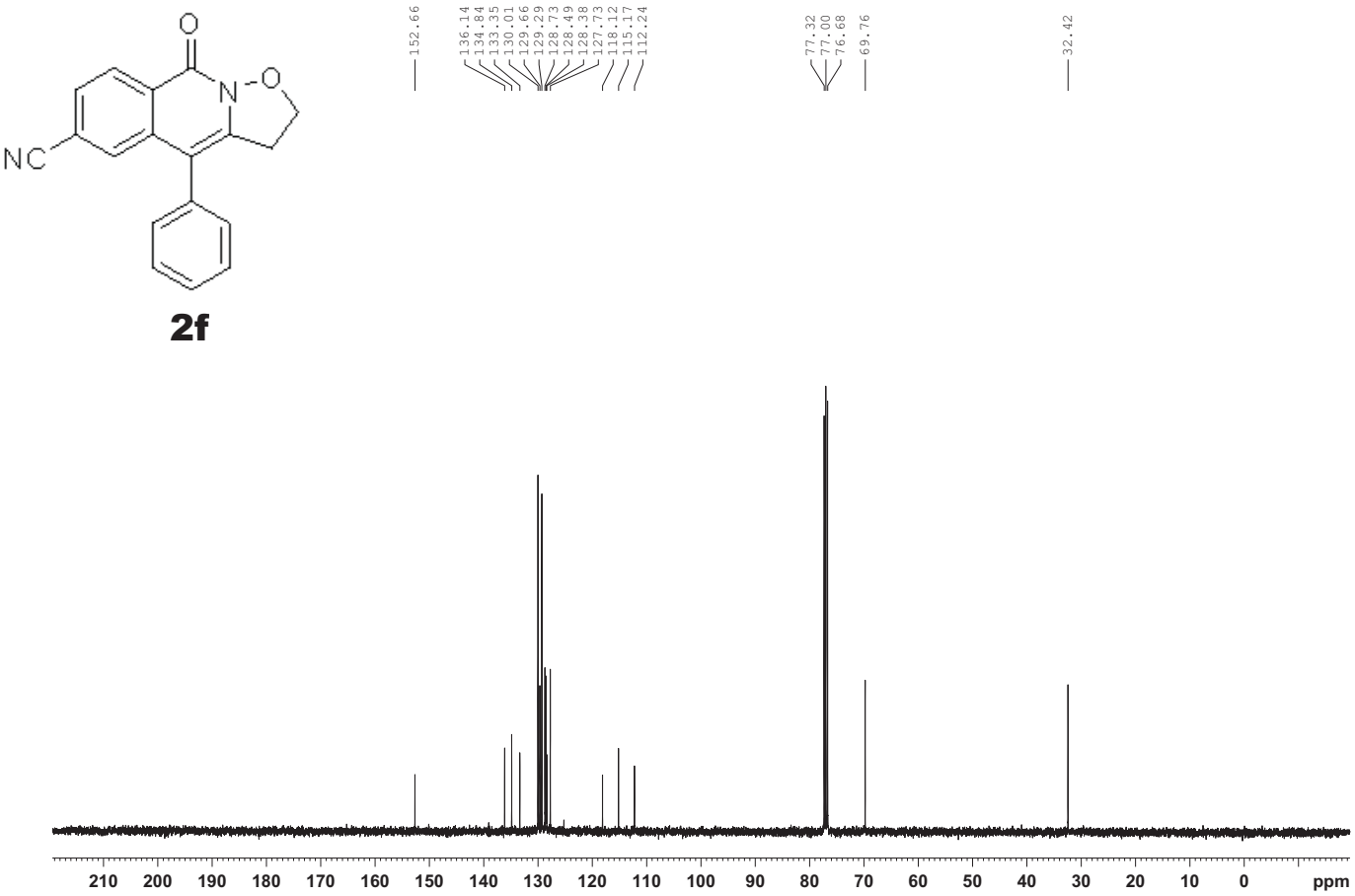

152.95
136.03
134.24
133.80
133.72
133.40
133.08
132.76
130.05
129.18
128.53
128.43
128.14
127.62
124.91
122.24
122.21
122.21
122.18
122.15
121.97
121.92
121.88
121.84
119.48
112.89
77.32
77.00
76.69
69.74
32.44
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

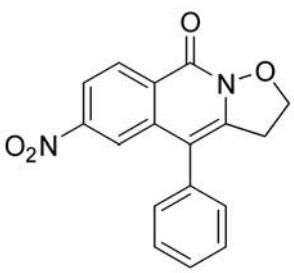

O
N
O
F3C

2e

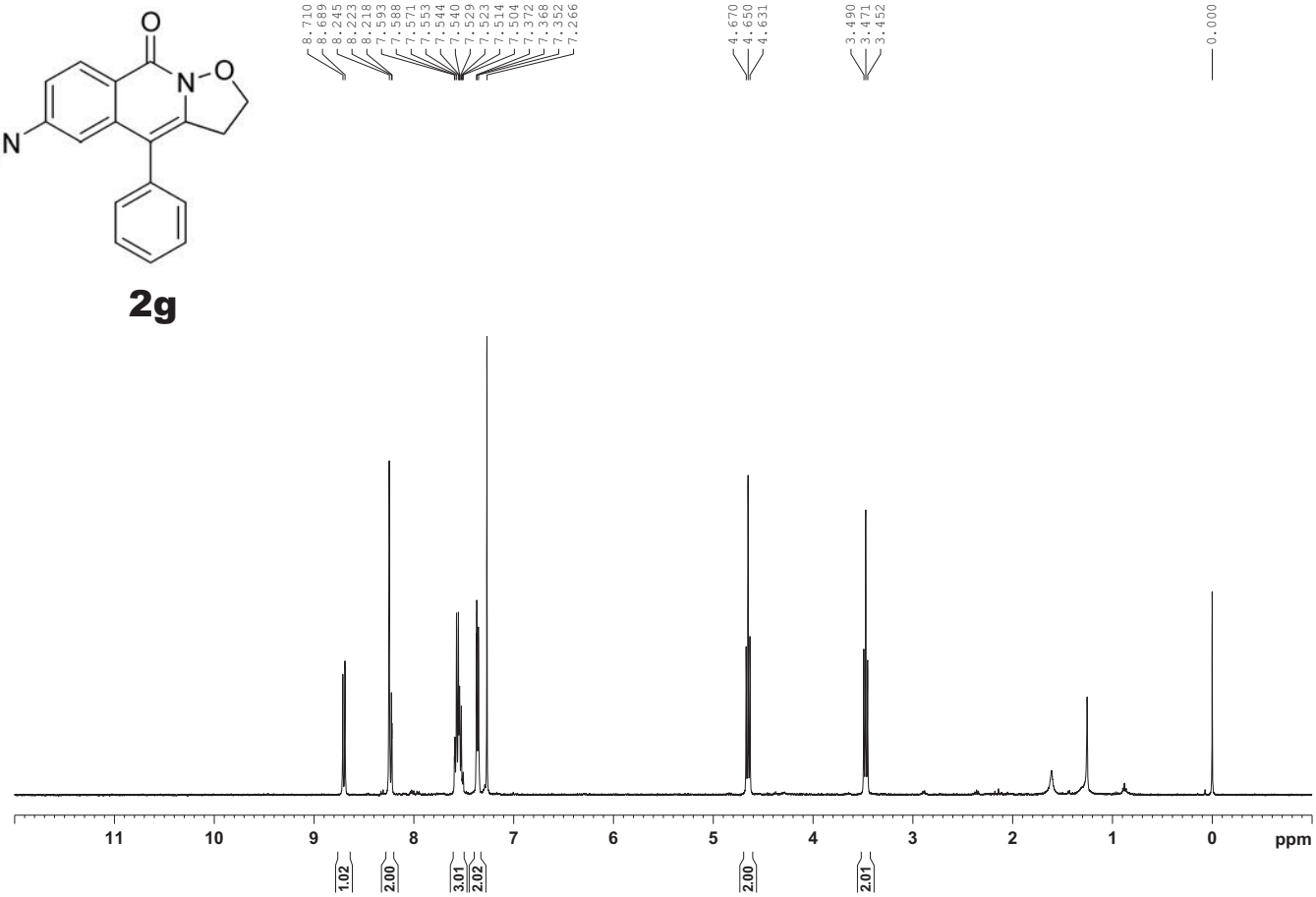

-62.821
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
ppm

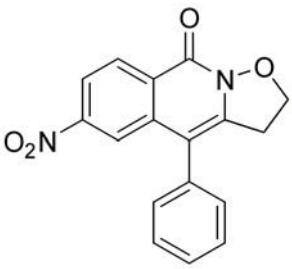

8.601
8.580
7.692
7.664
7.661
7.643
7.640
7.584
7.579
7.562
7.544
7.537
7.532
7.522
7.515
7.506
7.497
7.354
7.350
7.333
7.287
4.659
4.640
4.620
3.481
3.462
3.442
0.000
NC
O
N
O
2f
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
2.07
3.03
2.05
2.00
2.02


152.66
136.14
134.84
133.35
130.01
129.66
129.29
128.73
128.49
128.38
127.73
118.12
115.17
112.24
77.32
77.00
76.68
69.76
32.42
NC
O
N
O
2f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



2g


8.710
8.689
8.245
8.223
8.218
7.593
7.588
7.571
7.553
7.544
7.540
7.529
7.523
7.514
7.504
7.372
7.368
7.352
7.266
4.670
4.650
4.631
3.490
3.471
3.452
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.02
2.00
3.01
2.02
2.00
2.01



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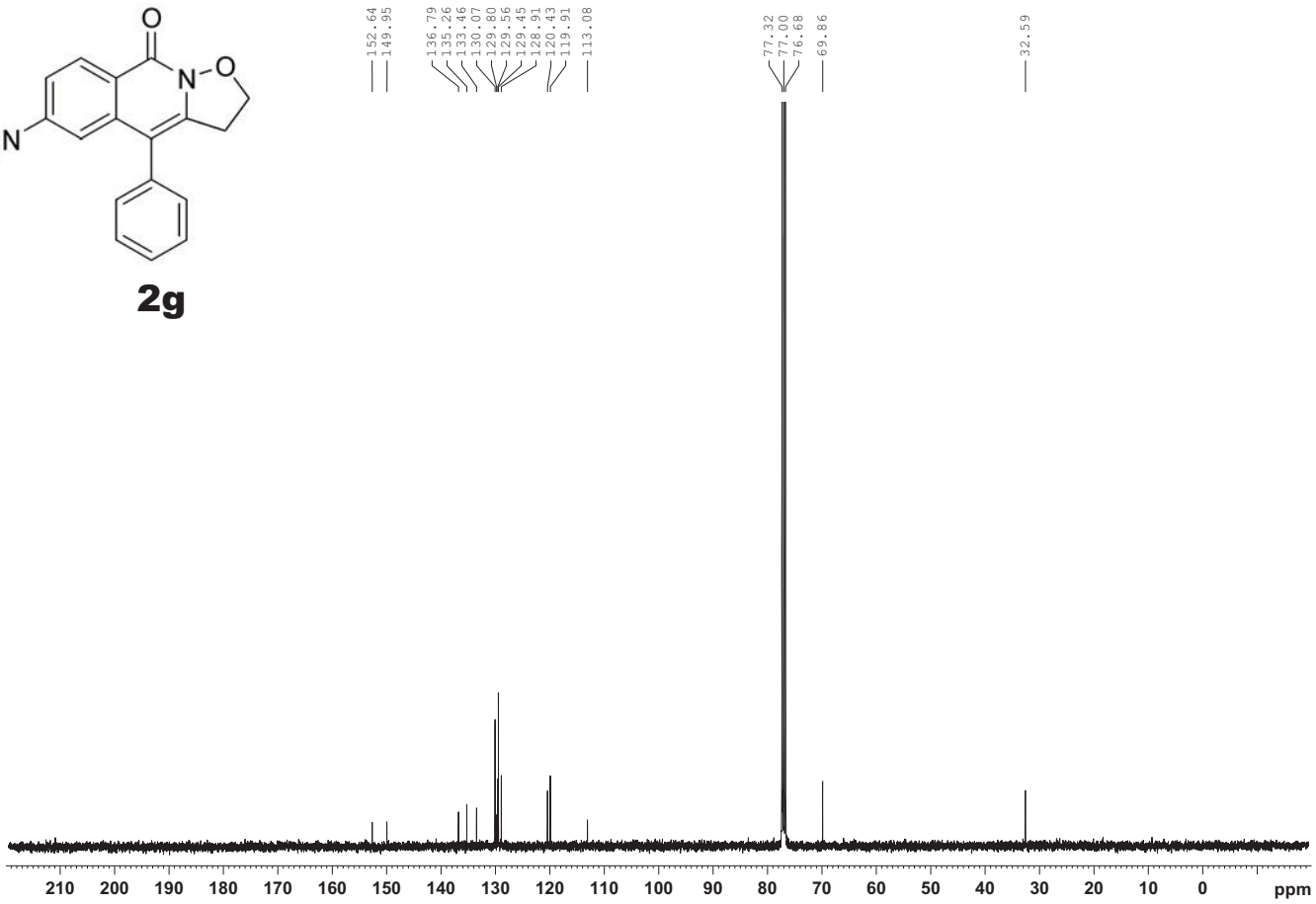
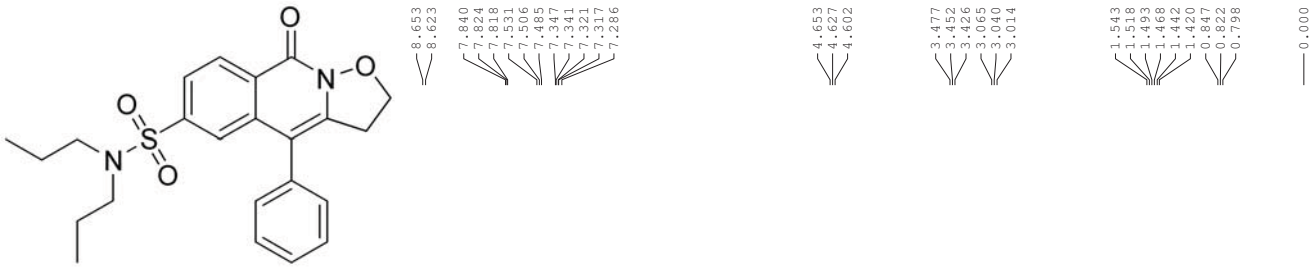
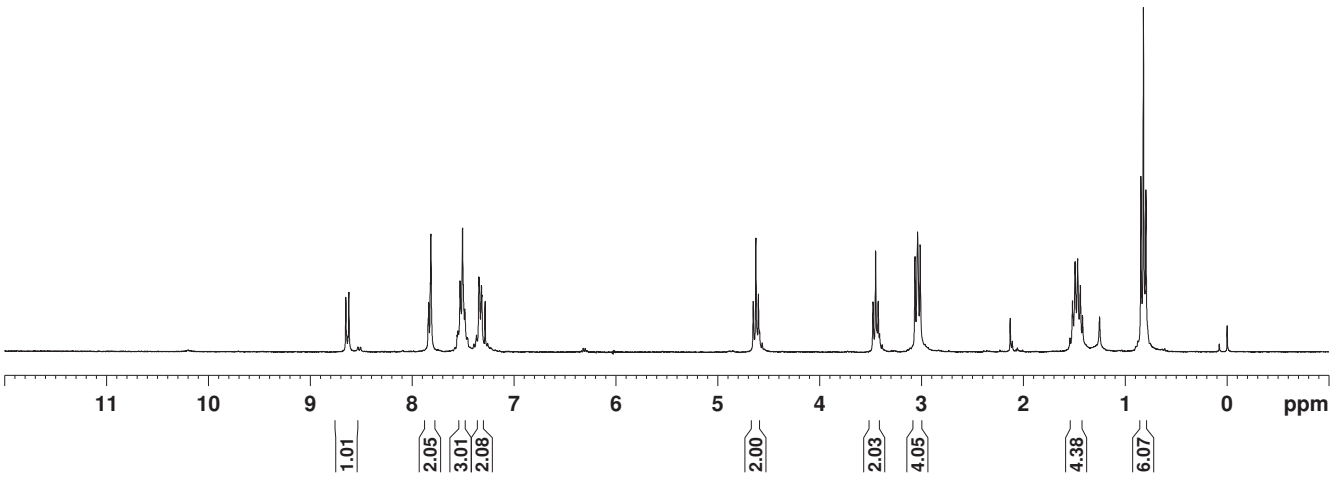
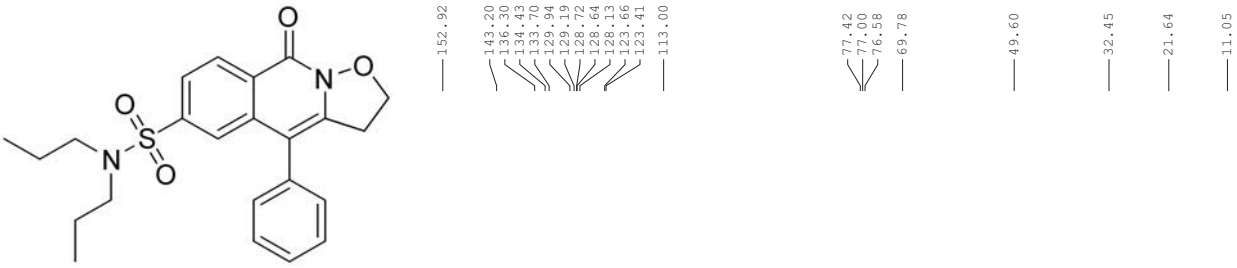

152.64
149.95
136.79
135.26
133.46
130.07
129.80
129.56
129.45
128.91
120.43
119.91
113.08
77.32
77.00
76.68
69.86
32.59
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

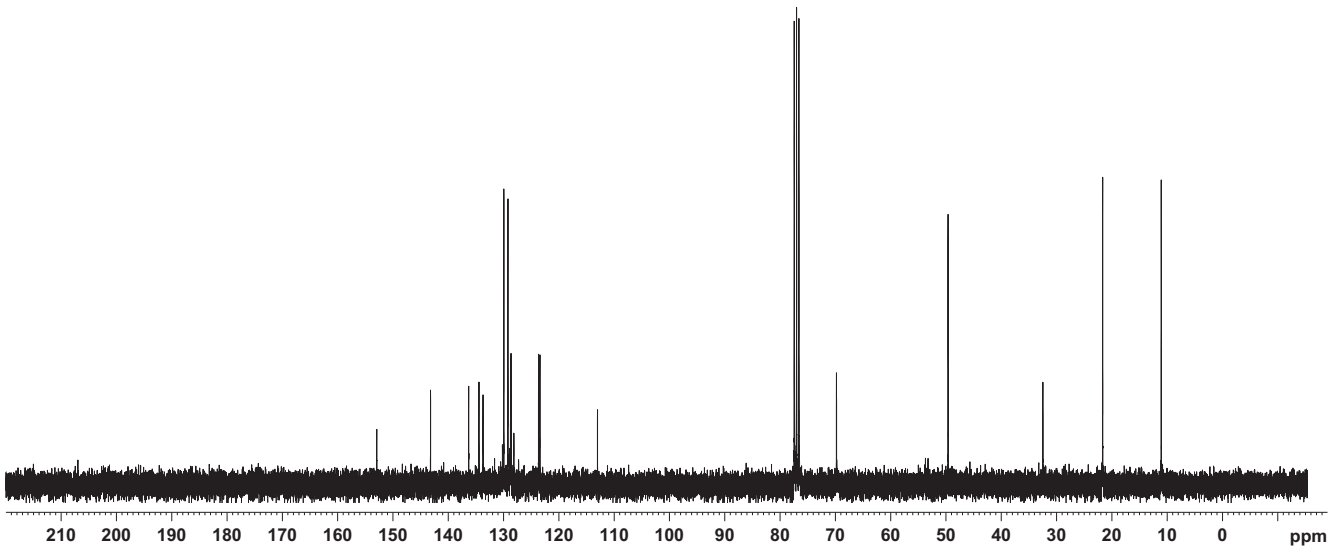

8.653
8.623
7.840
7.824
7.818
7.531
7.506
7.485
7.347
7.341
7.321
7.317
7.286
4.653
4.627
4.602
3.477
3.452
3.426
3.065
3.040
3.014
1.543
1.518
1.493
1.468
1.442
1.420
0.847
0.822
0.798
0.000

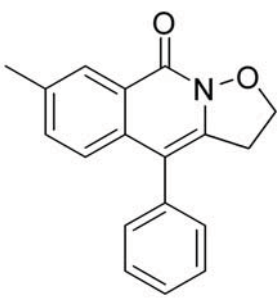
2h


11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
2.05
3.01
2.08
2.00
2.03
4.05
4.38
6.07


152.92
143.20
136.30
134.43
133.70
129.94
129.19
128.72
128.64
128.13
123.66
123.41
113.00
77.42
77.00
76.58
69.78
49.60
32.45
21.64
11.05

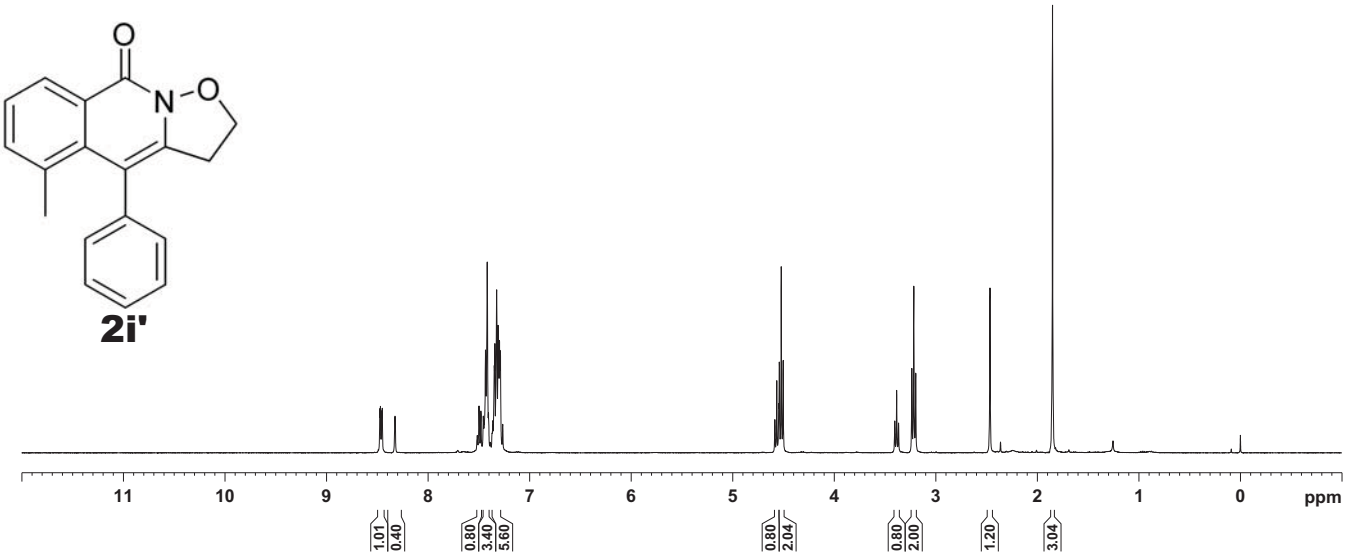
2h

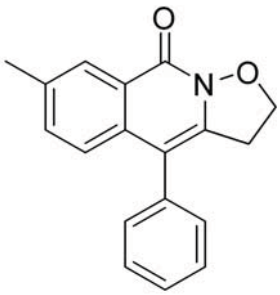

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



2i

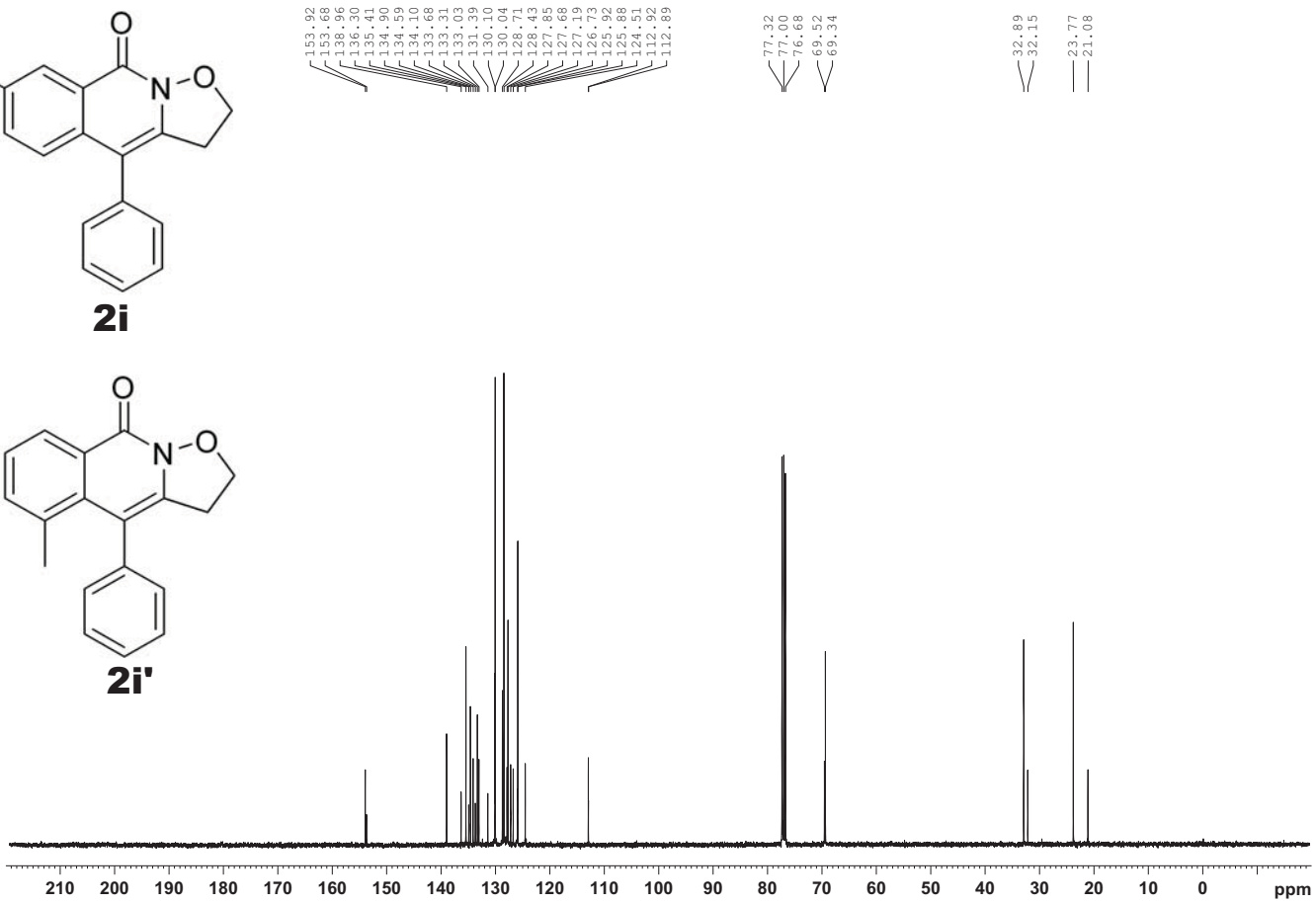
2i'

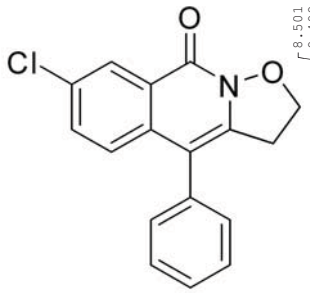

8.473
8.468
8.455
8.450
8.325
7.514
7.497
7.479
7.455
7.452
7.446
7.433
7.416
7.404
7.369
7.365
7.359
7.347
7.341
7.323
7.310
7.305
7.295
7.291
7.286
7.264
4.584
4.565
4.546
4.540
4.521
4.502
3.404
3.385
3.366
3.235
3.216
3.197
2.466
1.850
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.01
0.40
0.80
3.40
5.60
0.80
2.04
0.80
2.00
1.20
3.04



2i

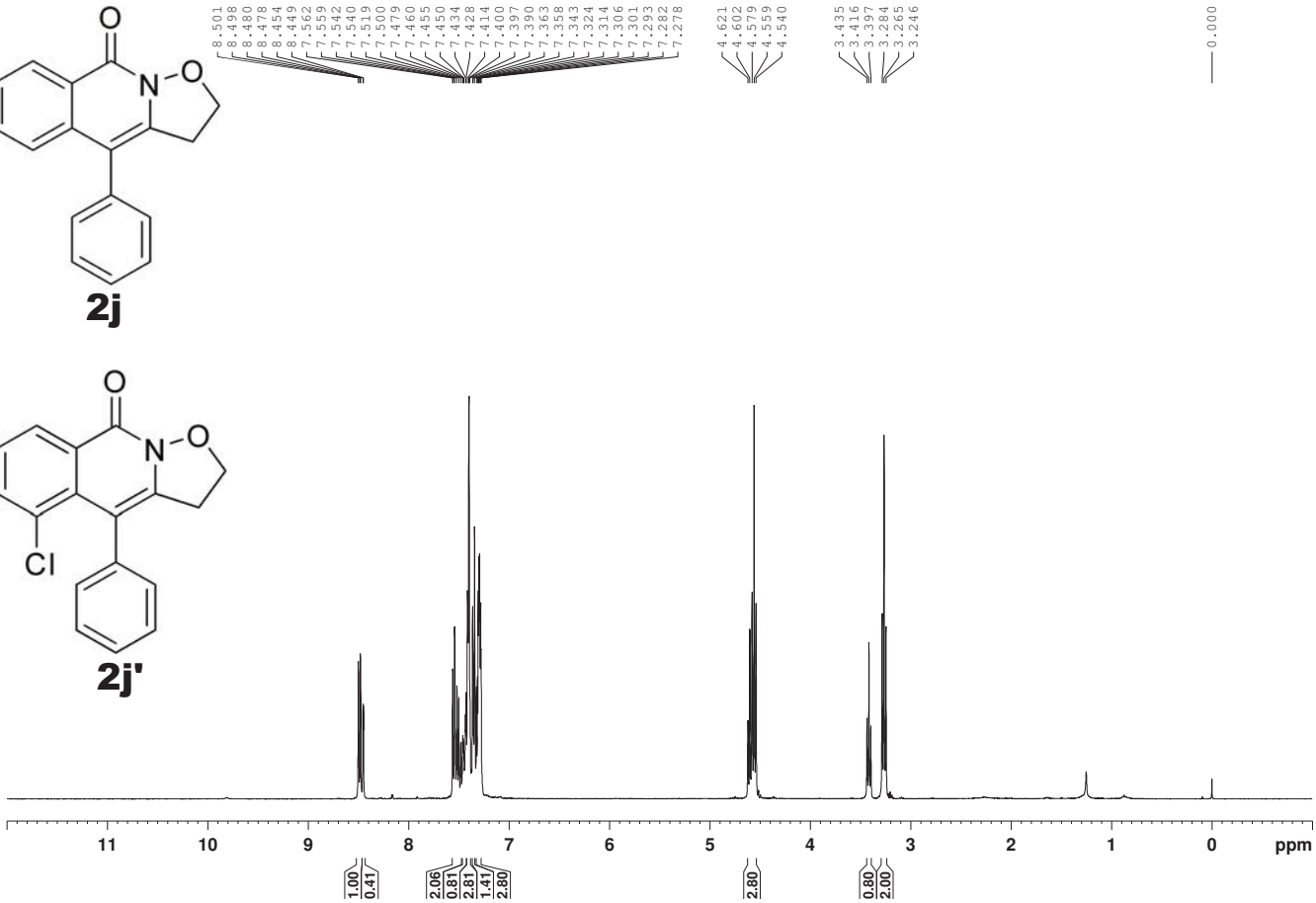
2i'

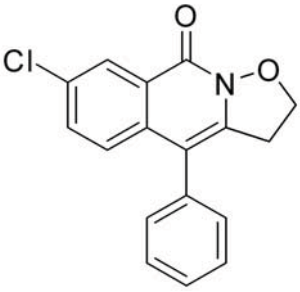

153.92
153.68
138.96
136.30
135.41
134.90
134.59
134.10
133.68
133.31
133.03
131.39
130.10
130.04
128.71
128.43
127.85
127.68
127.19
126.73
125.92
125.88
124.51
112.92
112.89
77.32
77.00
76.68
69.52
69.34
32.89
32.15
23.77
21.08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



2j

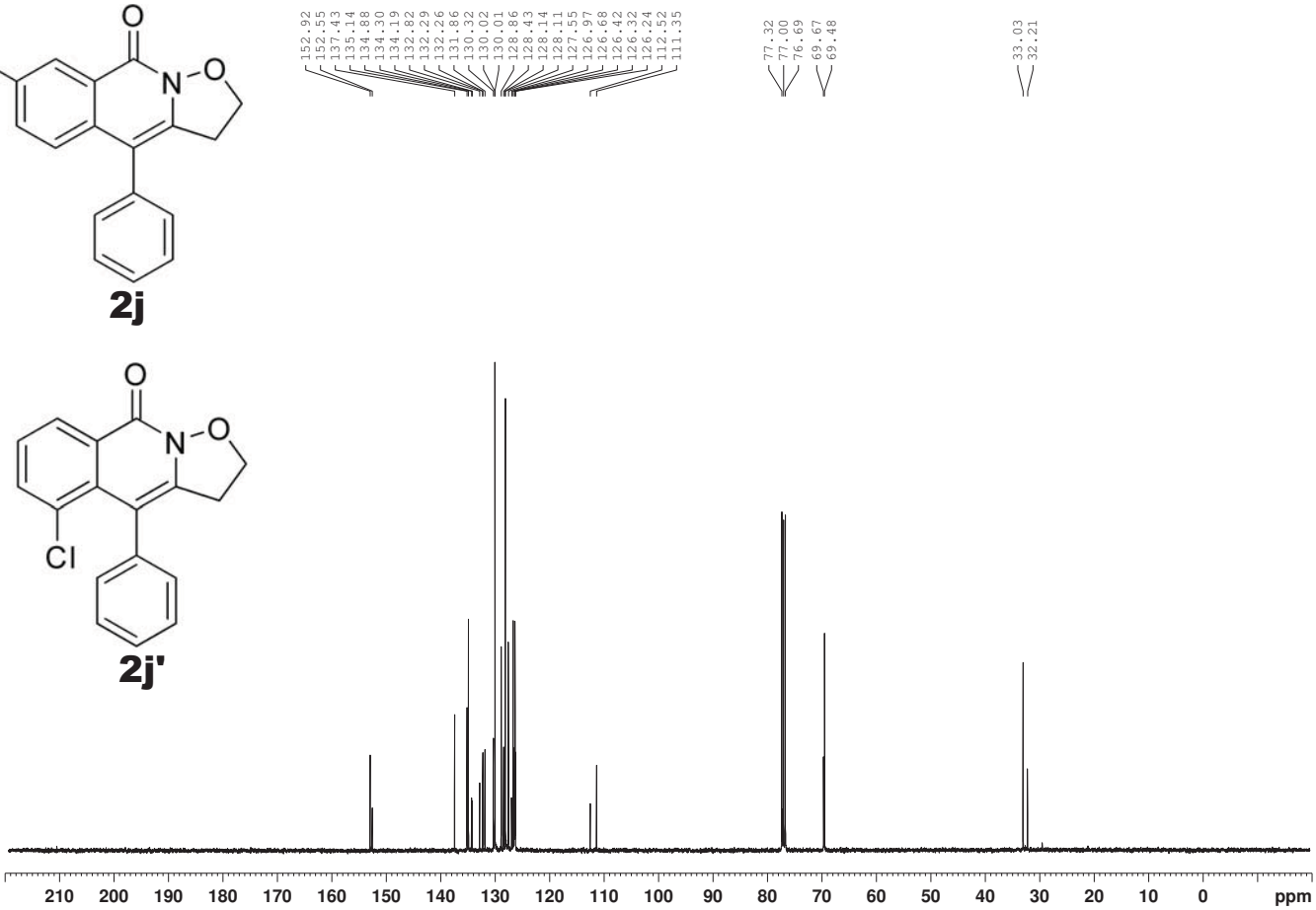
2j'

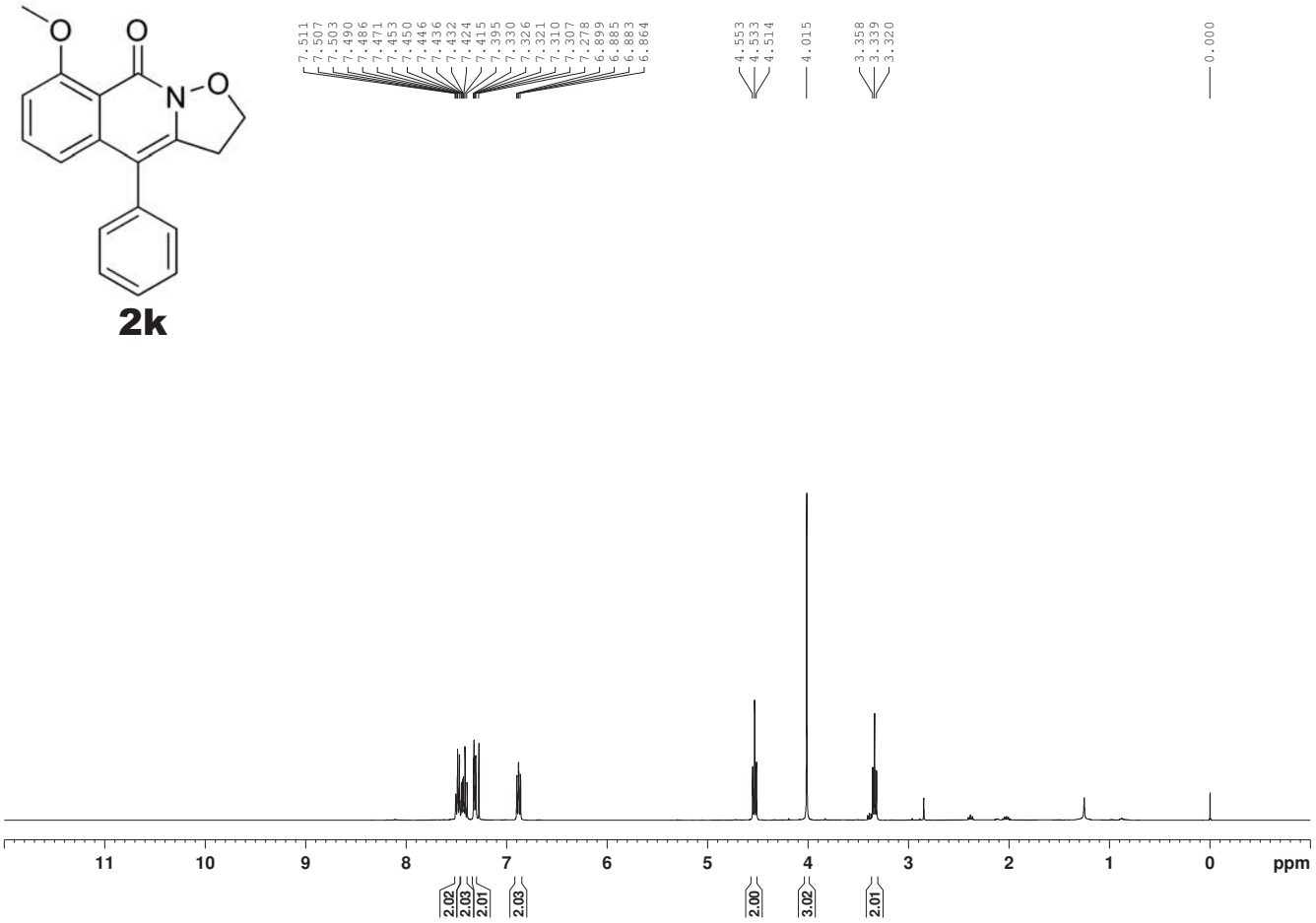

8.501
8.498
8.480
8.478
8.454
8.449
7.562
7.559
7.542
7.540
7.519
7.500
7.479
7.460
7.455
7.450
7.434
7.428
7.414
7.400
7.397
7.390
7.363
7.358
7.343
7.324
7.314
7.306
7.301
7.293
7.282
7.278
4.621
4.602
4.579
4.559
4.540
3.435
3.416
3.397
3.284
3.265
3.246
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
0.41
2.06
0.81
2.81
1.41
2.80
2.80
0.80
2.00

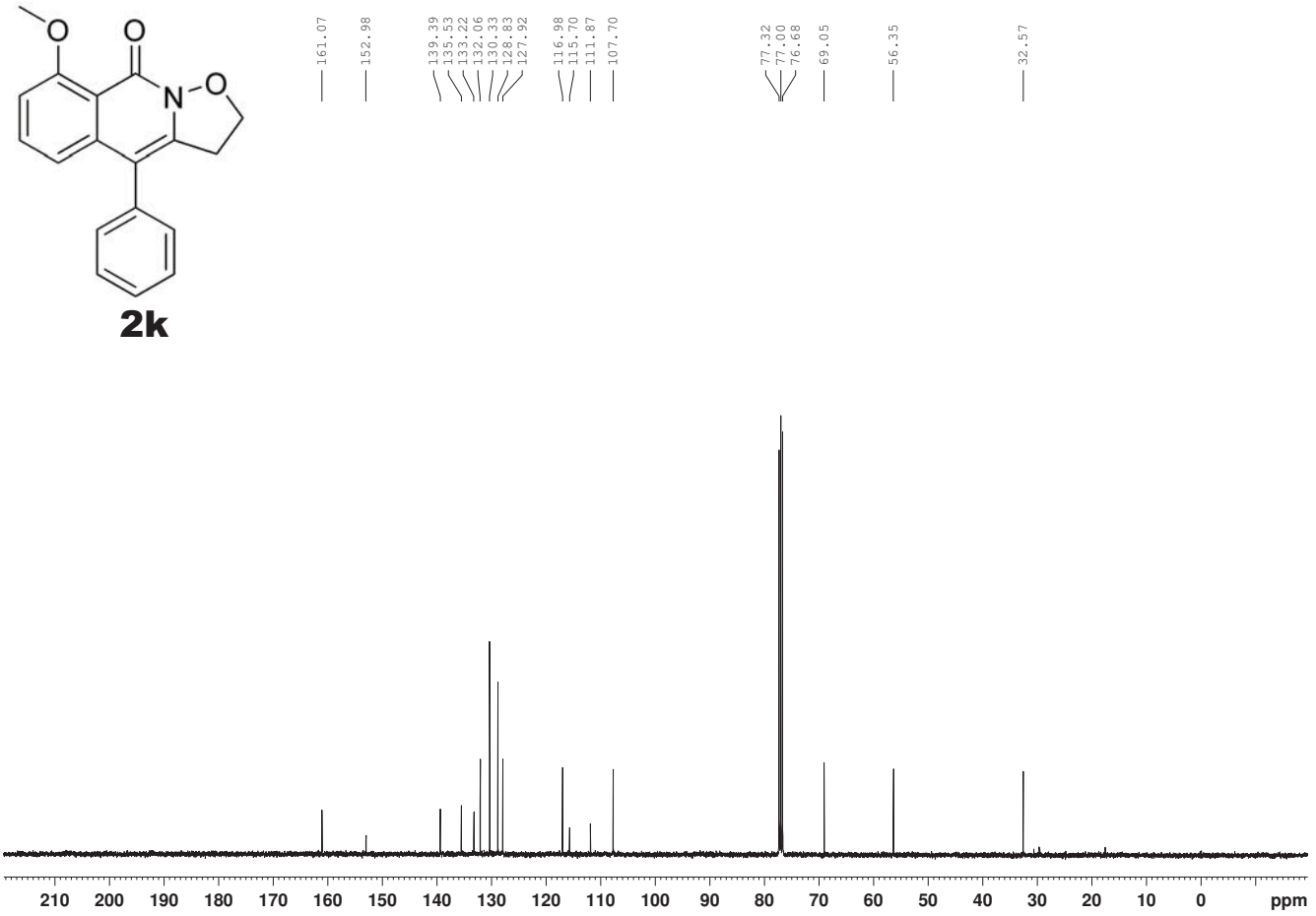


2j

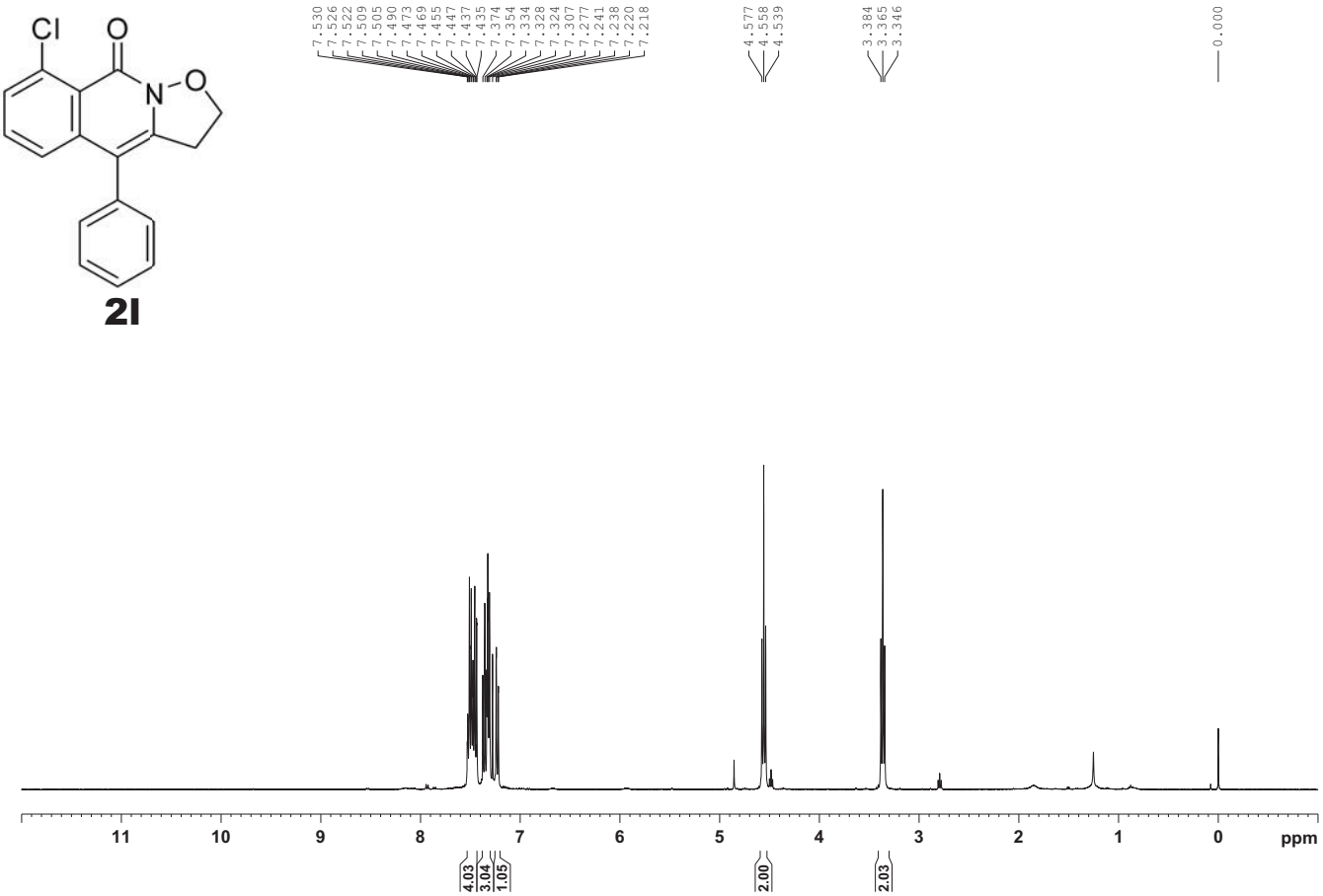
2j'


152.92
152.55
137.43
135.14
134.88
134.30
134.19
132.82
132.29
132.26
131.86
130.32
130.02
130.01
128.86
128.43
128.14
128.11
127.55
126.97
126.68
126.42
126.32
126.24
112.52
111.35
77.32
77.00
76.69
69.67
69.48
33.03
32.21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


2k
7.511
7.507
7.503
7.490
7.486
7.471
7.453
7.450
7.446
7.436
7.432
7.424
7.415
7.395
7.330
7.326
7.321
7.310
7.307
7.278
6.899
6.885
6.883
6.864
4.553
4.533
4.514
4.015
3.358
3.339
3.320
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.03
2.01
2.03
2.00
3.02
2.01


2k
161.07
152.98
139.39
135.53
133.22
132.06
130.33
128.83
127.92
116.98
115.70
111.87
107.70
77.32
77.00
76.68
69.05
56.35
32.57
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

2l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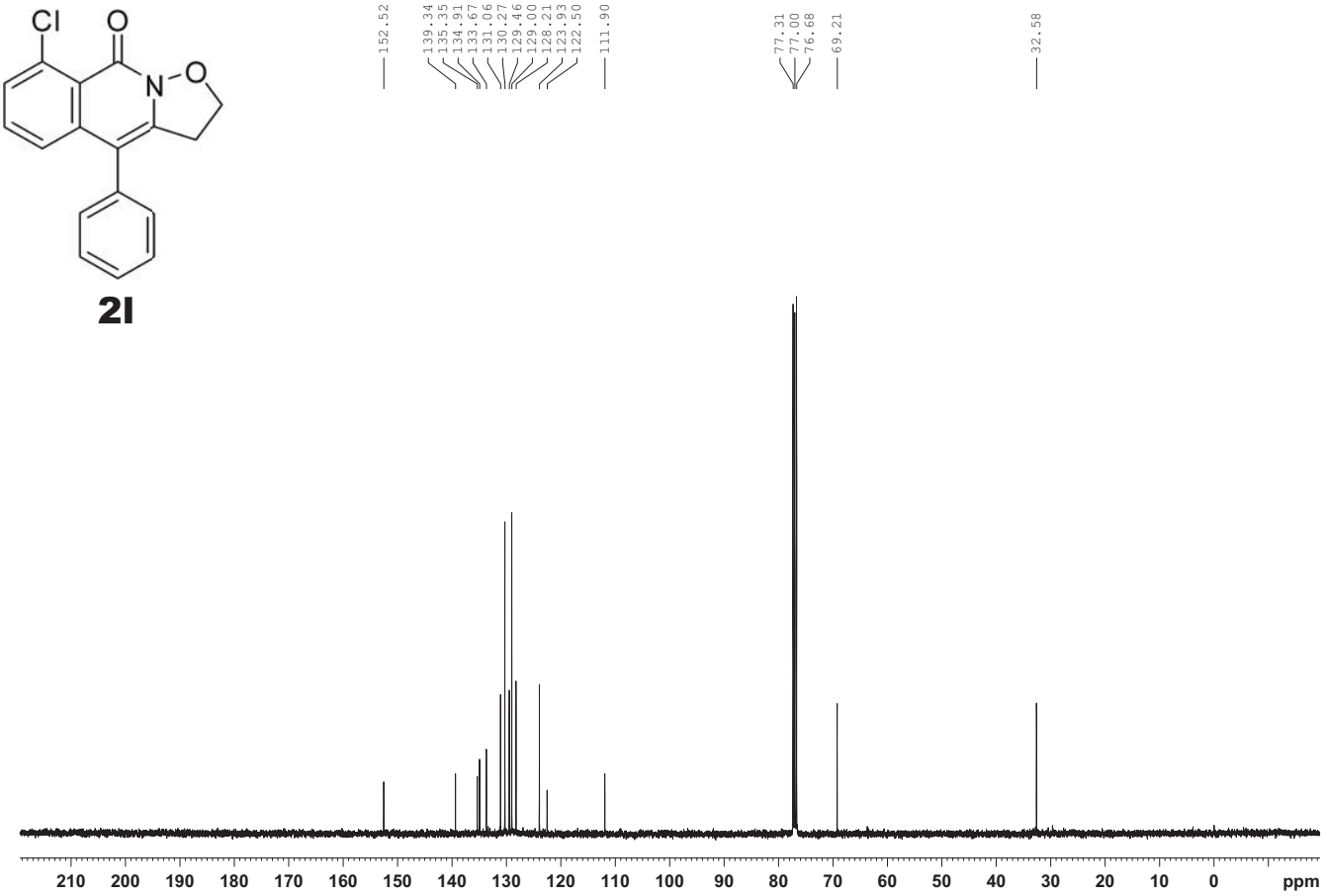
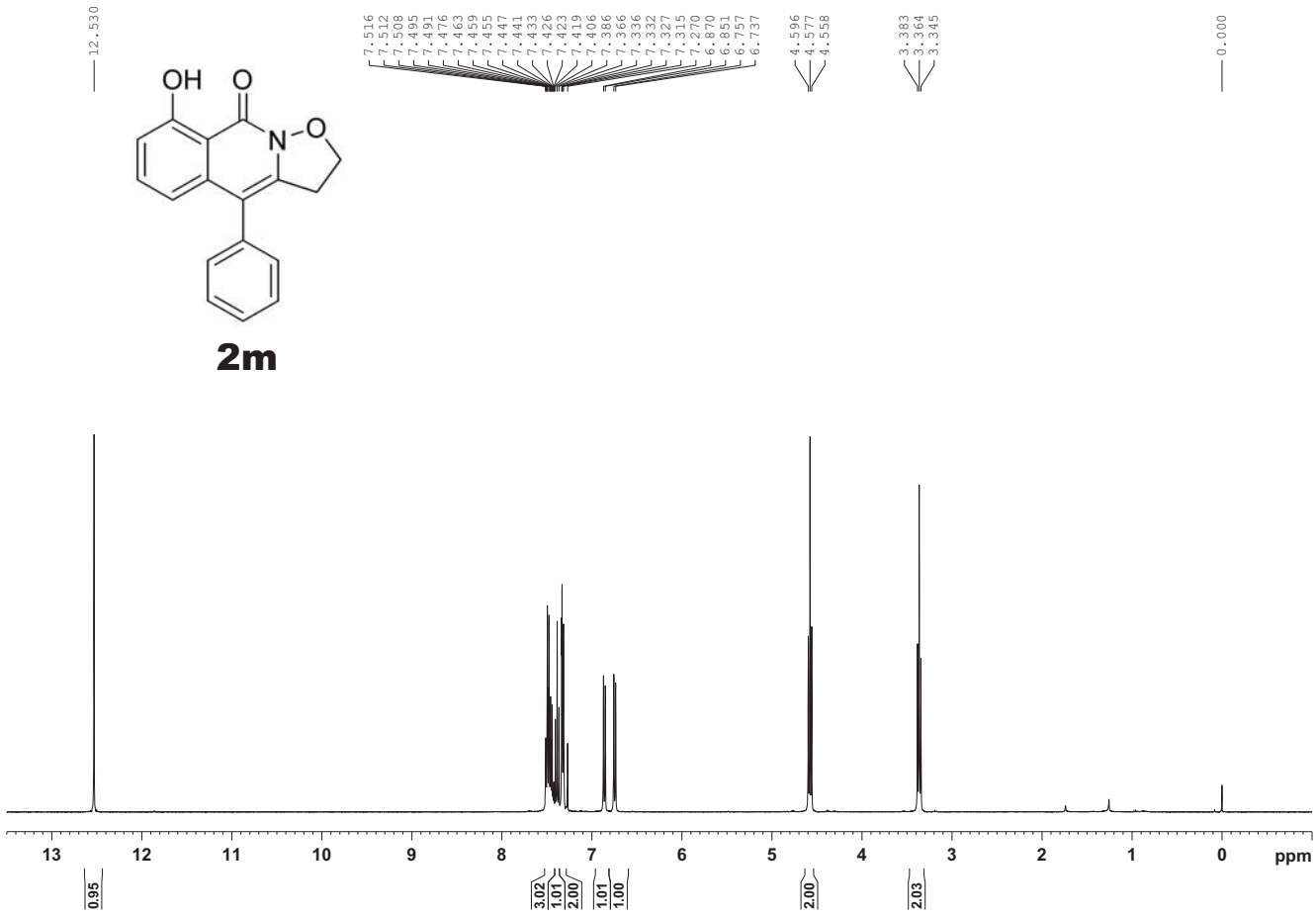
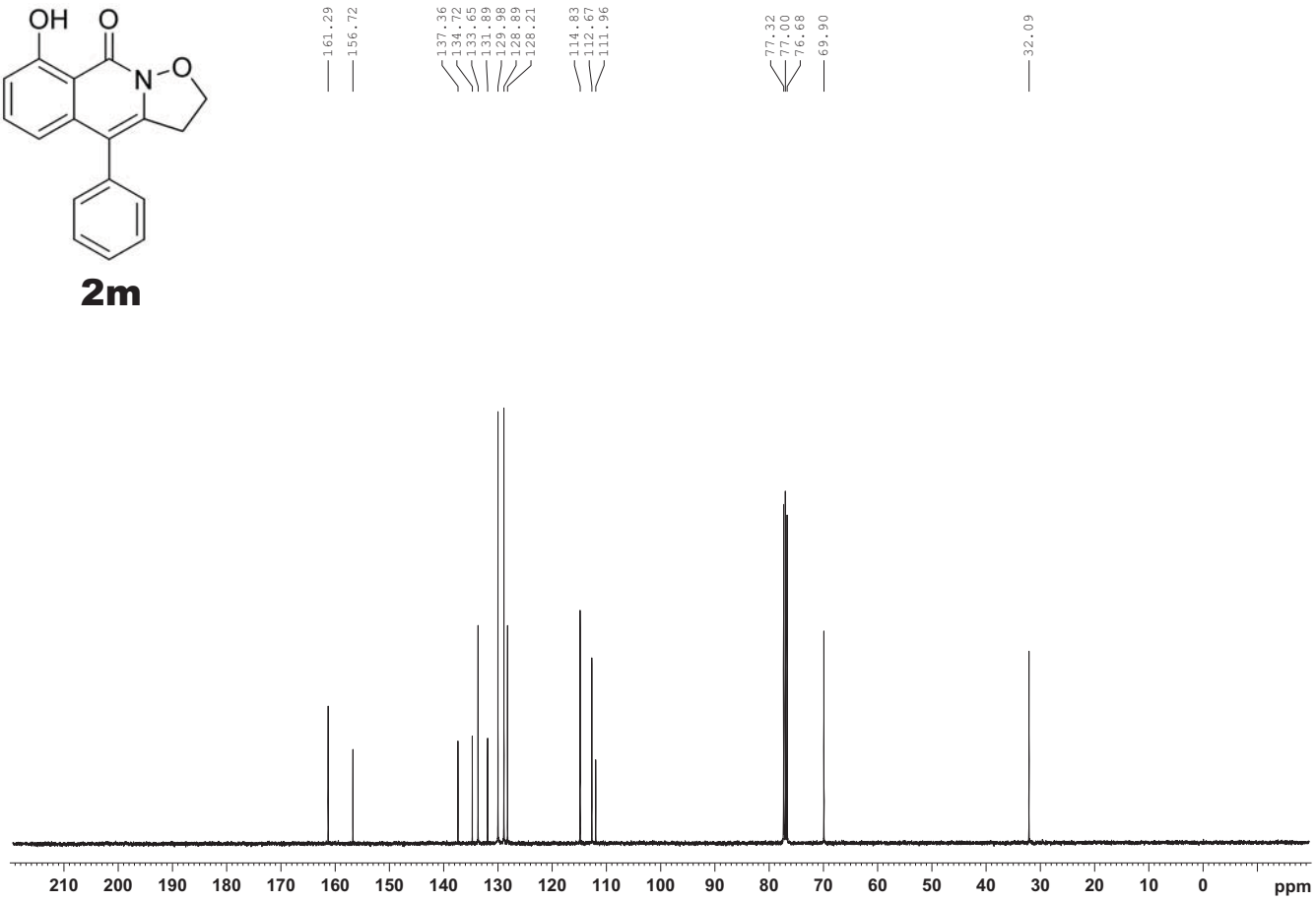
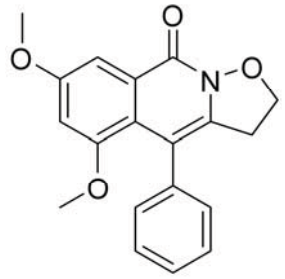
7.526
7.522
7.509
7.505
7.490
7.473
7.469
7.455
7.447
7.437
7.435
7.374
7.354
7.334
7.328
7.324
7.307
7.277
7.241
7.238
7.220
7.218
4.577
4.558
4.539
3.384
3.365
3.346
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
4.03
3.04
1.05
2.00
2.03

2l

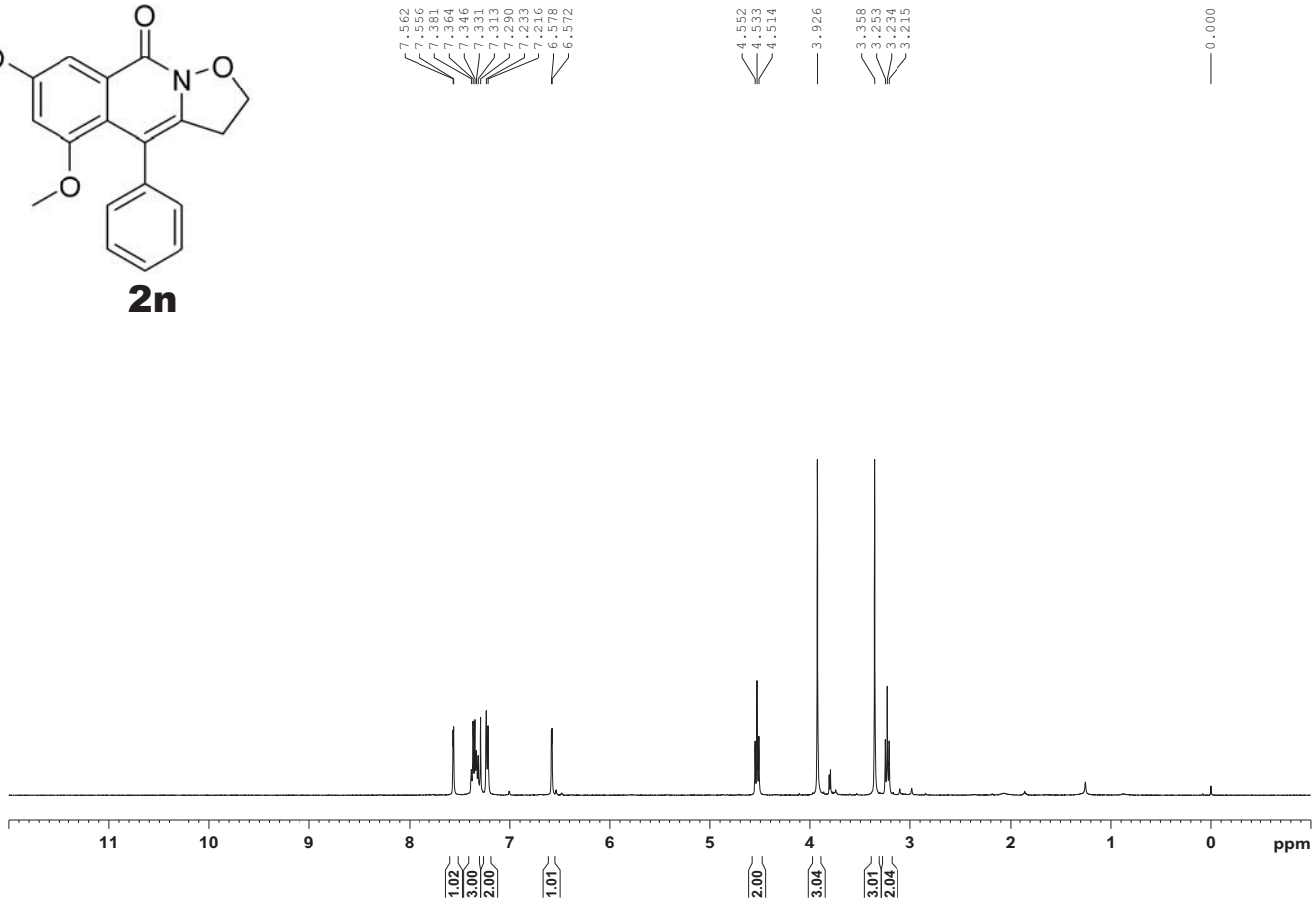

152.52
139.34
135.35
134.91
133.67
131.06
130.27
129.46
129.00
128.21
123.93
122.50
111.90
77.31
77.00
76.68
69.21
32.58
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

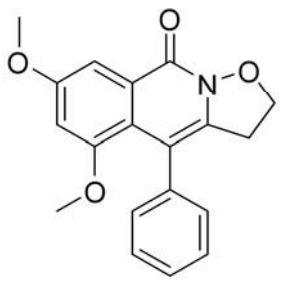

12.530
7.516
7.512
7.508
7.495
7.491
7.476
7.463
7.459
7.455
7.447
7.441
7.433
7.426
7.423
7.419
7.406
7.386
7.366
7.336
7.332
7.327
7.315
7.270
6.870
6.851
6.757
6.737
4.596
4.577
4.558
3.383
3.364
3.345
0.000
OH
O
N
O
2m
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
3.02
1.01
2.00
1.01
1.00
2.00
2.03


OH
O
N
O
2m
161.29
156.72
137.36
134.72
133.65
131.89
129.98
128.89
128.21
114.83
112.67
111.96
77.32
77.00
76.68
69.90
32.09
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

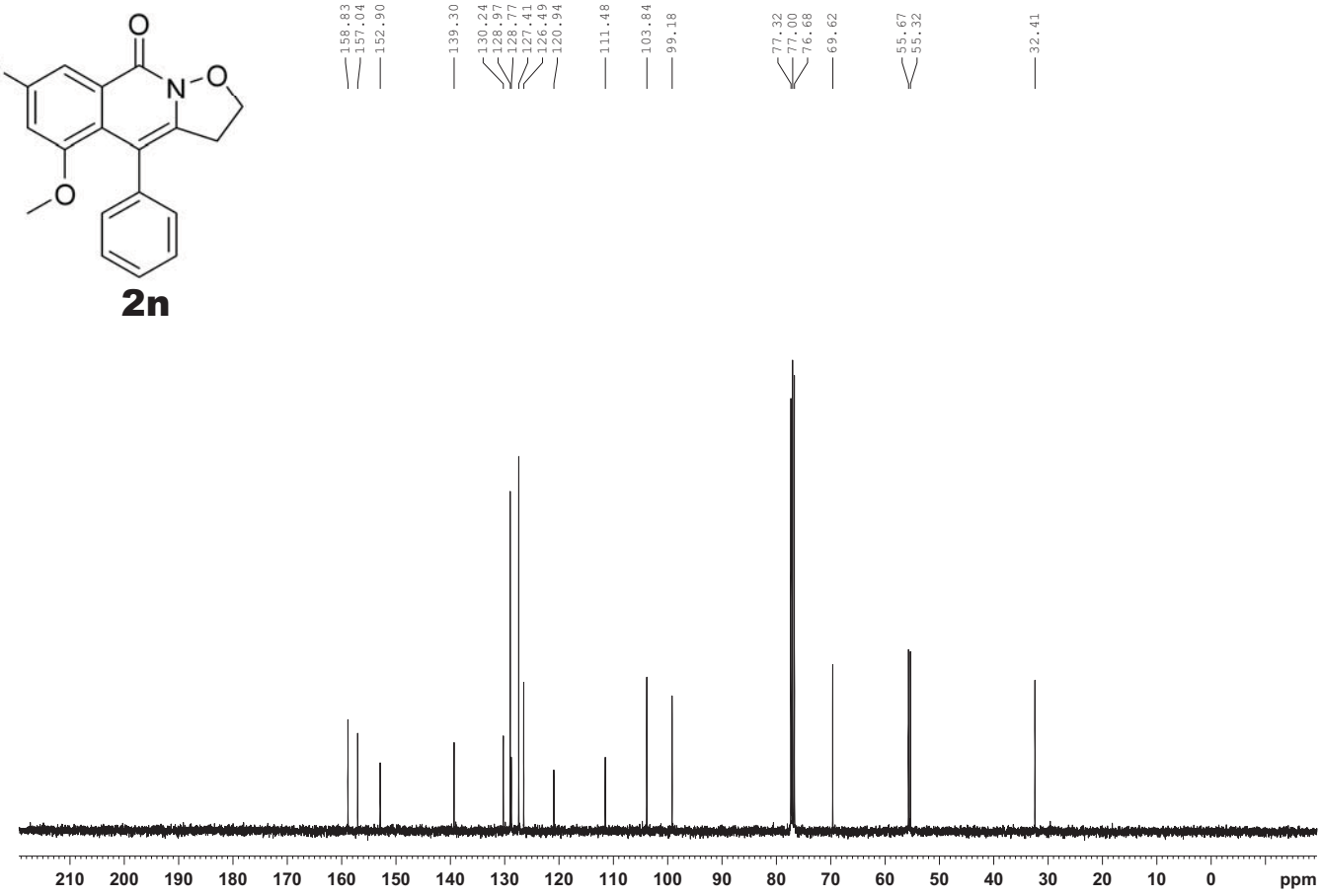


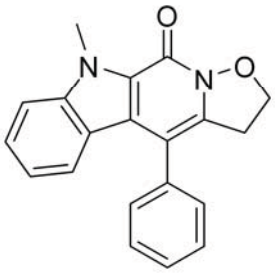
2n


7.562
7.556
7.381
7.364
7.346
7.331
7.313
7.290
7.233
7.216
6.578
6.572
4.552
4.533
4.514
3.926
3.358
3.253
3.234
3.215
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.02
3.00
2.00
1.01
2.00
3.04
3.01
2.04



2n


158.83
157.04
152.90
139.30
130.24
128.97
128.77
127.41
126.49
120.94
111.48
103.84
99.18
77.32
77.00
76.68
69.62
55.67
55.32
32.41
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



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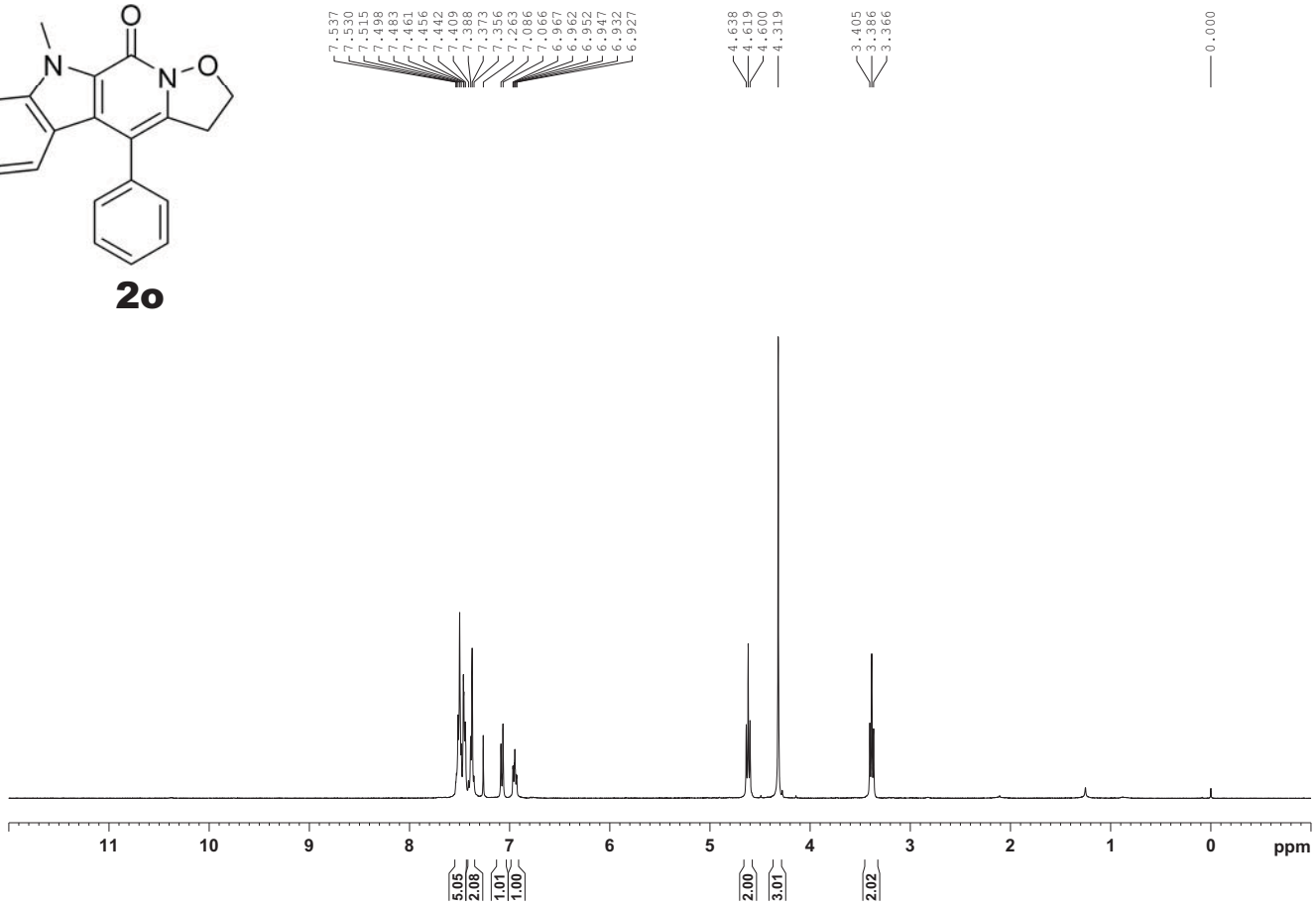
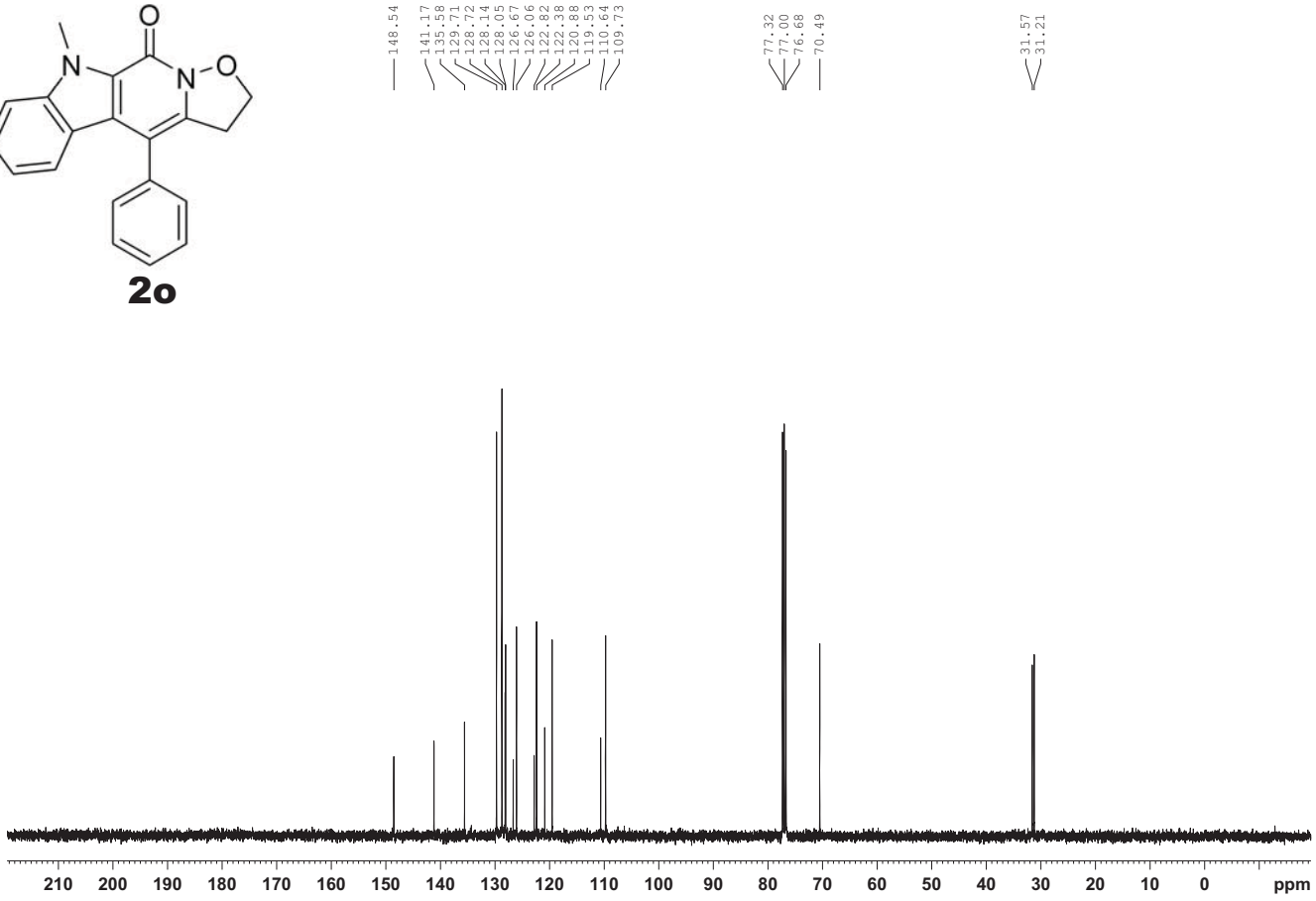
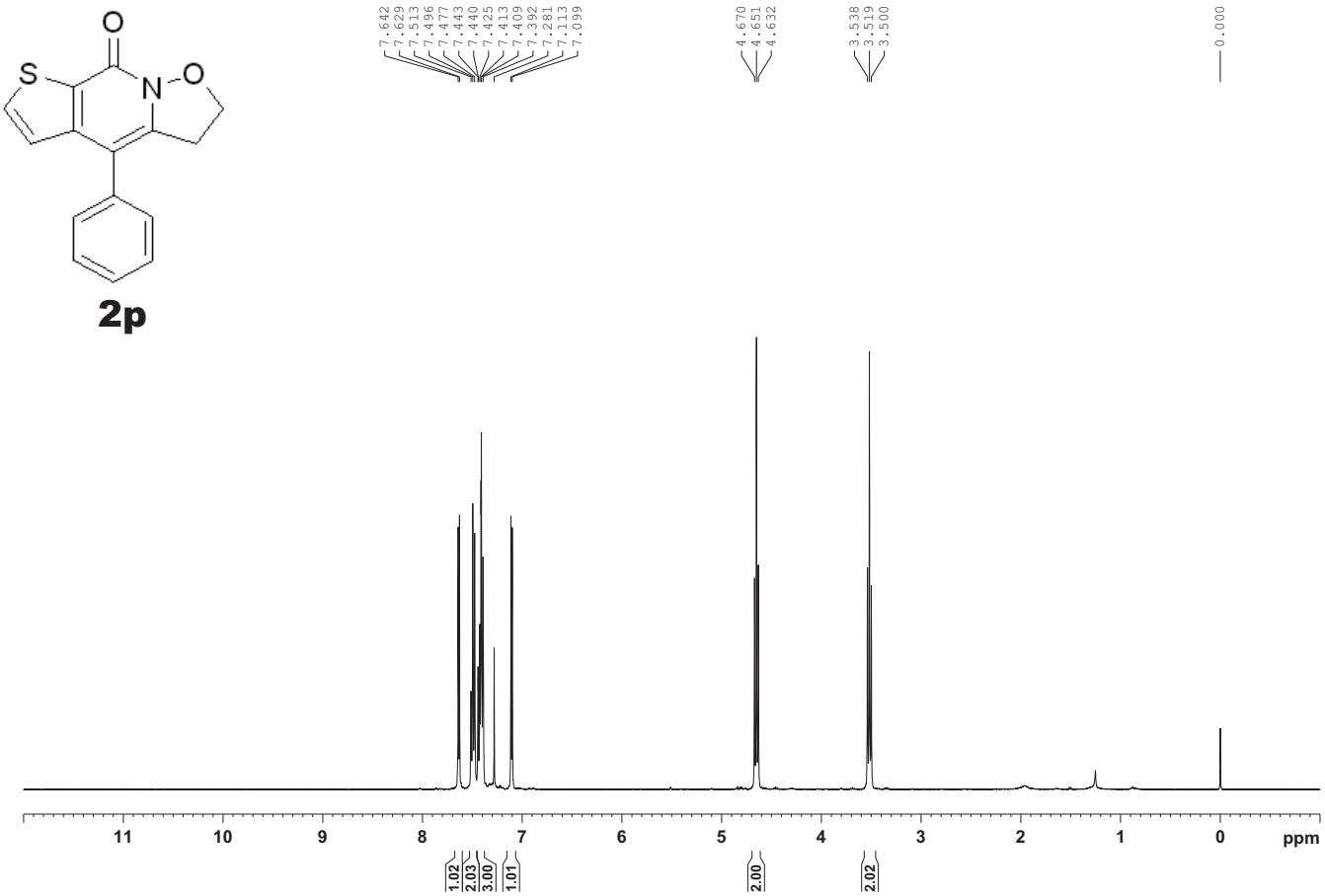

7.537
7.530
7.515
7.498
7.483
7.461
7.456
7.442
7.409
7.388
7.373
7.356
7.263
7.086
7.066
6.967
6.962
6.952
6.947
6.932
6.927
4.638
4.619
4.600
4.319
3.405
3.386
3.366
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
5.05
2.08
1.01
1.00
2.00
3.01
2.02

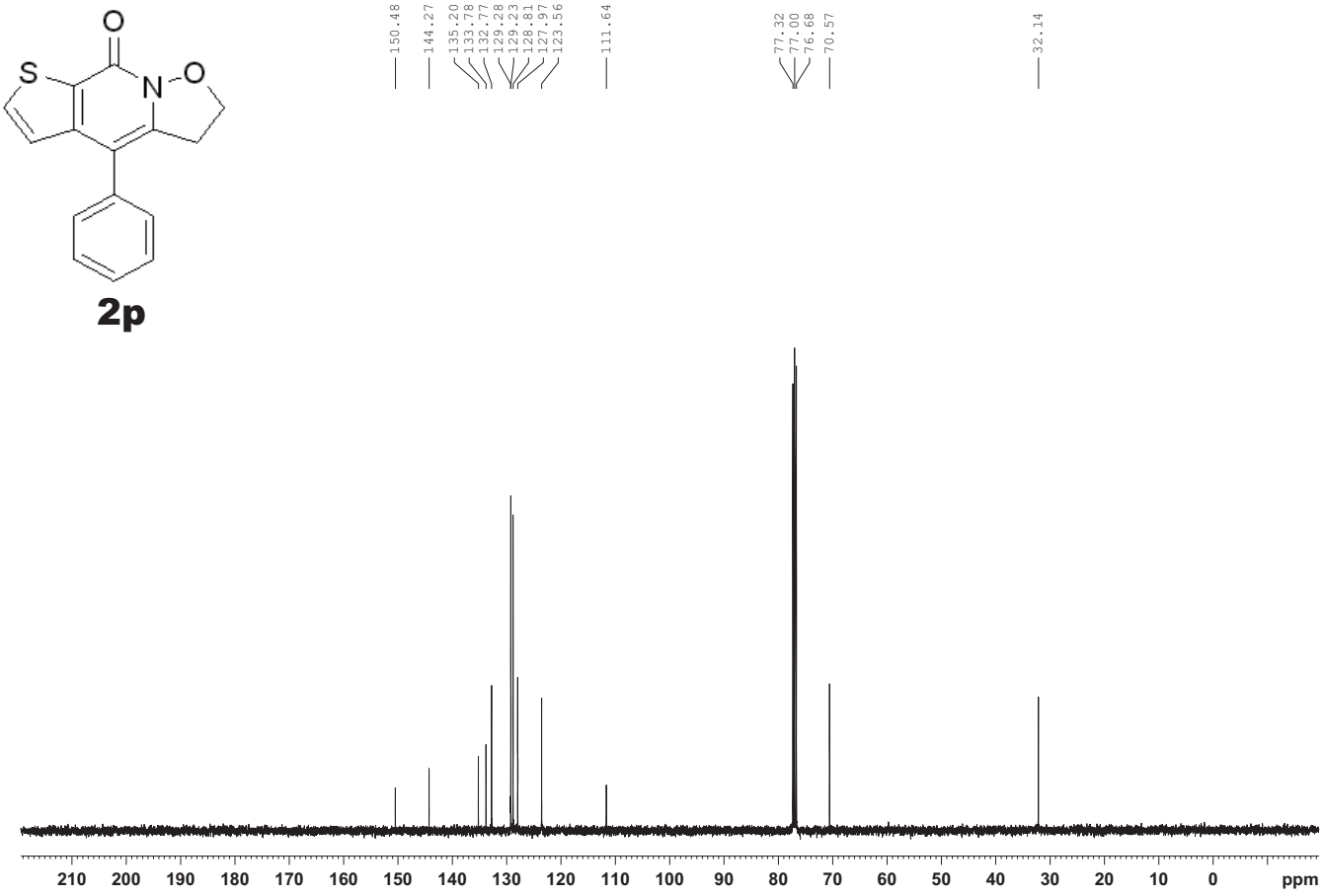
2o


148.54
141.17
135.58
129.71
128.72
128.14
128.05
126.67
126.06
122.82
122.38
120.88
119.53
110.64
109.73
77.32
77.00
76.68
70.49
31.57
31.21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

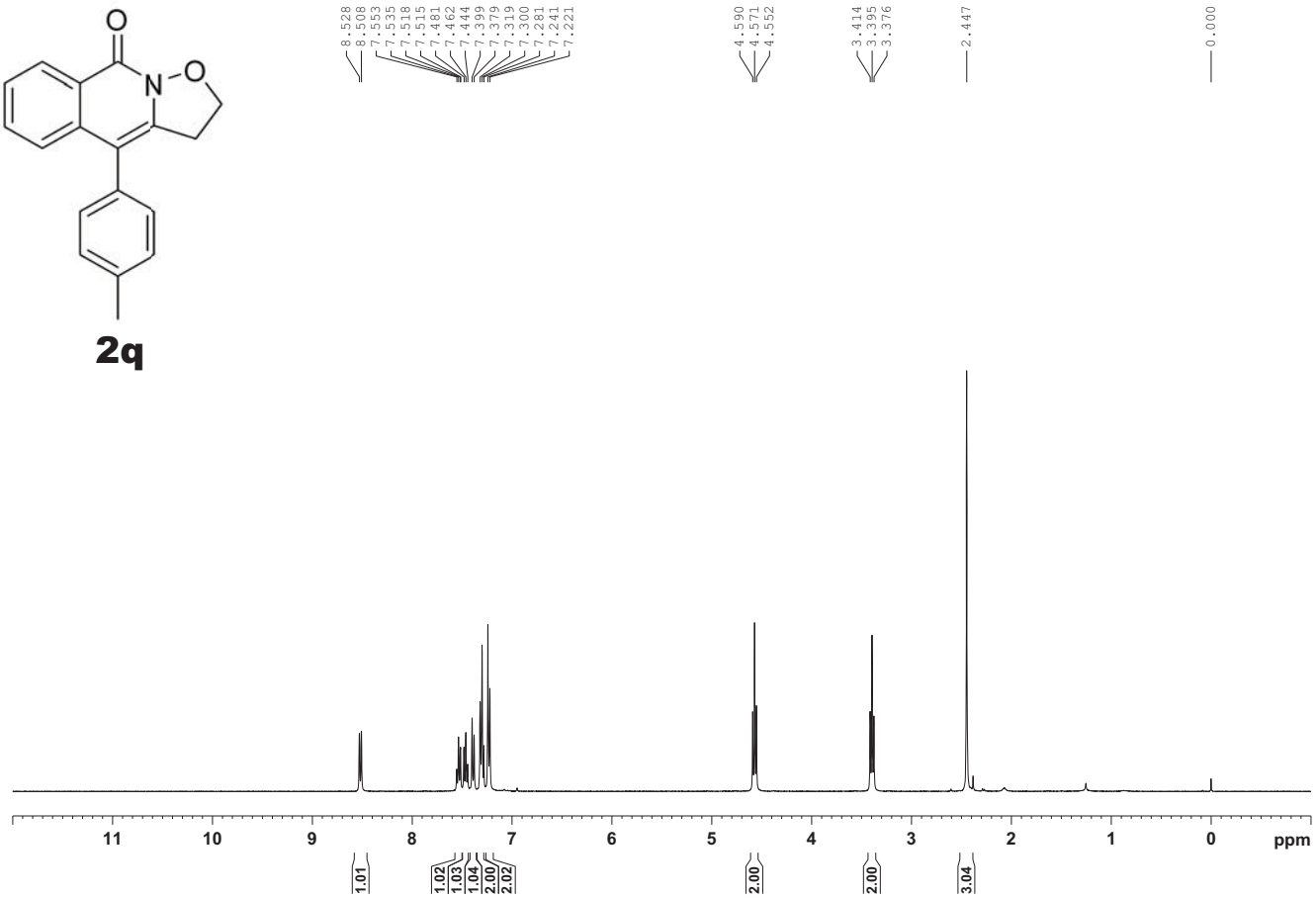
2p


7.642
7.629
7.533
7.496
7.477
7.443
7.440
7.425
7.413
7.409
7.392
7.281
7.113
7.099
4.670
4.651
4.632
3.538
3.519
3.500
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.02
2.03
3.00
1.01
2.00
2.02

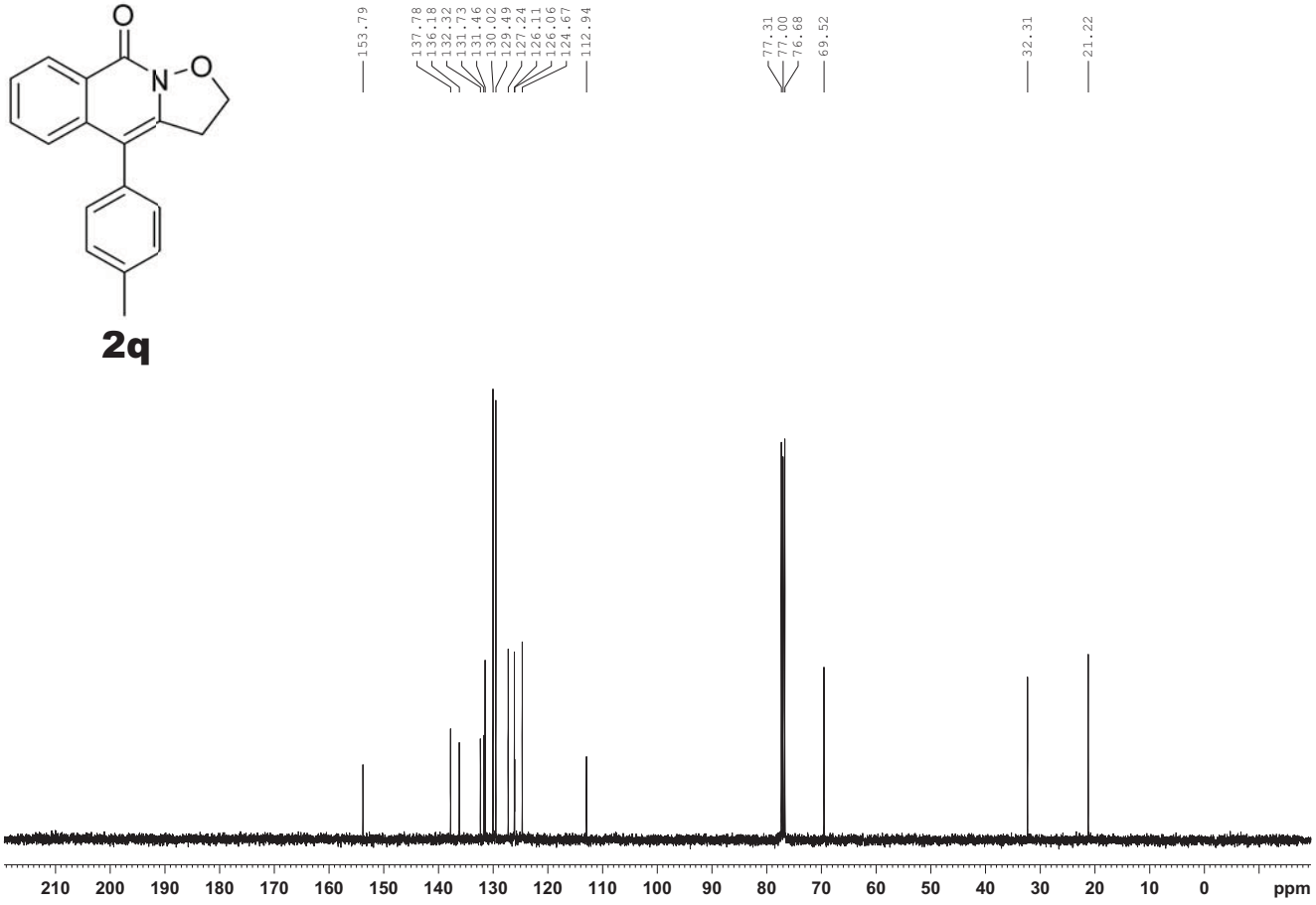
2p


150.48
144.27
135.20
133.78
132.77
129.28
129.23
128.81
127.97
123.56
111.64
77.32
77.00
76.68
70.57
32.14
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

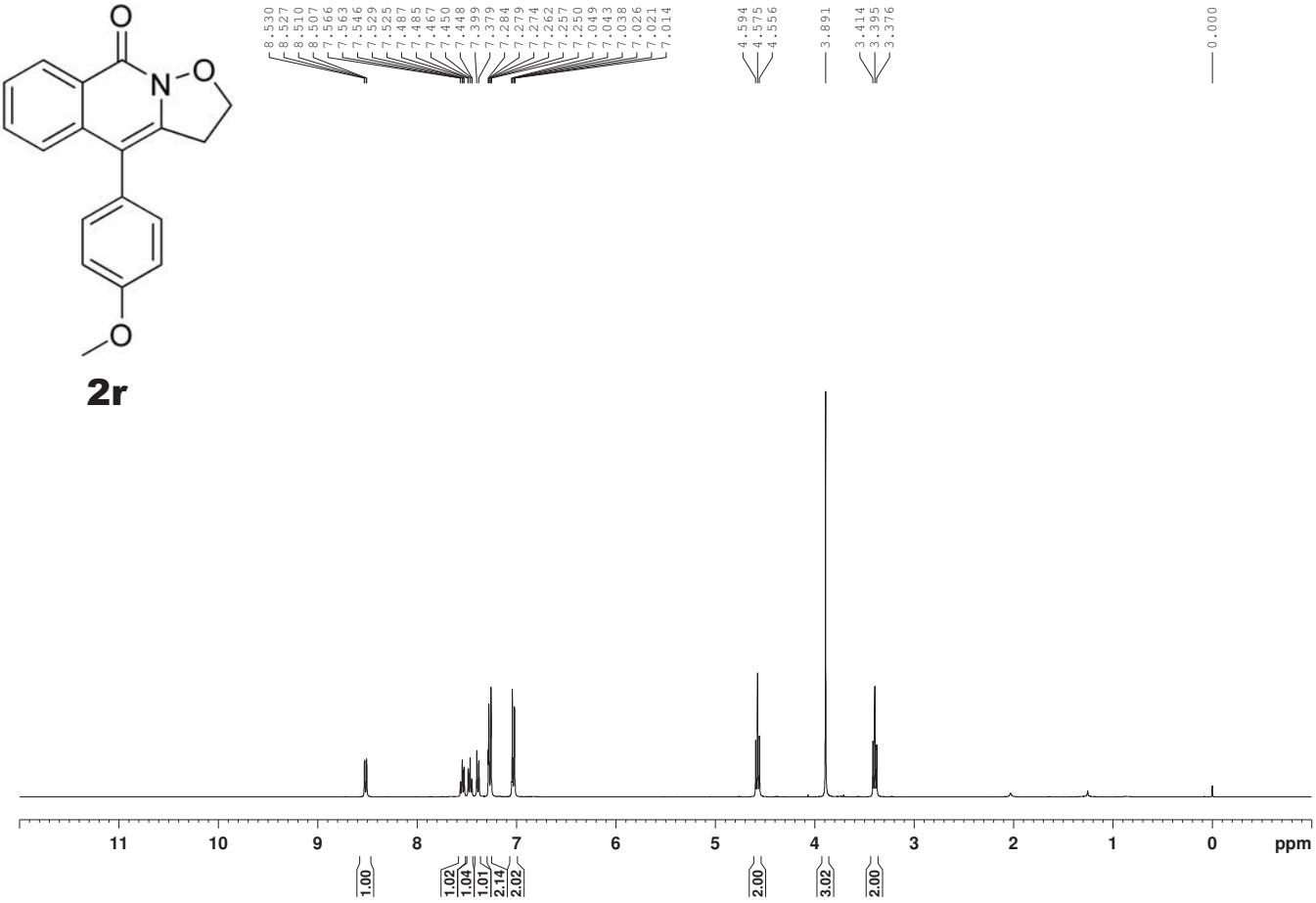
2q

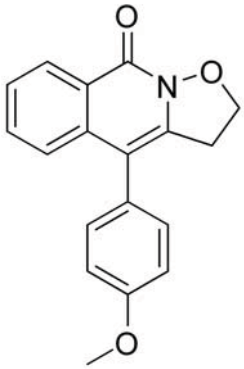

8.528
8.508
7.553
7.535
7.518
7.515
7.481
7.462
7.444
7.399
7.379
7.319
7.300
7.281
7.241
7.221
4.590
4.571
4.552
3.414
3.395
3.376
2.447
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.01
1.02
1.03
1.04
2.00
2.02
2.00
2.00
3.04

2q

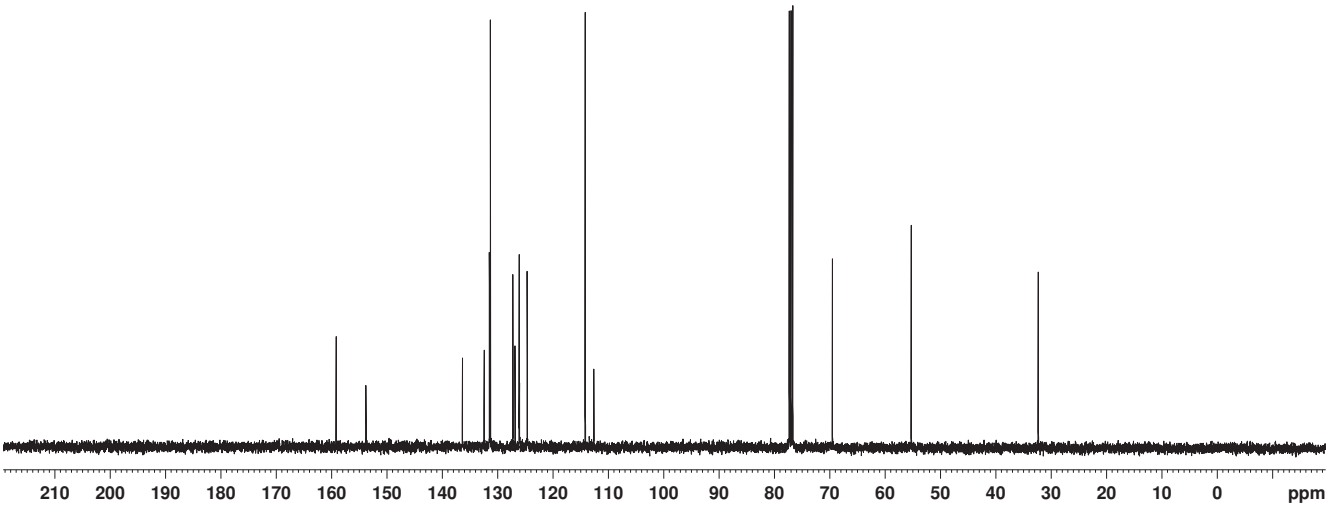

153.79
137.78
136.18
132.32
131.73
131.46
130.02
129.49
127.24
126.11
126.06
124.67
112.94
77.31
77.00
76.68
69.52
32.31
21.22
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

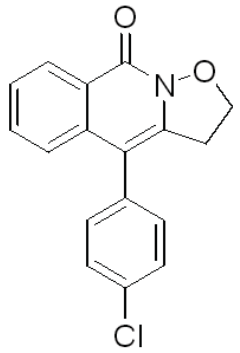
2r


8.530
8.527
8.510
8.507
7.566
7.563
7.546
7.529
7.525
7.487
7.485
7.467
7.450
7.448
7.399
7.379
7.284
7.279
7.274
7.262
7.257
7.250
7.049
7.043
7.038
7.026
7.021
7.014
4.594
4.575
4.556
3.891
3.414
3.395
3.376
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.02
1.04
1.01
2.14
2.02
2.00
3.02
2.00

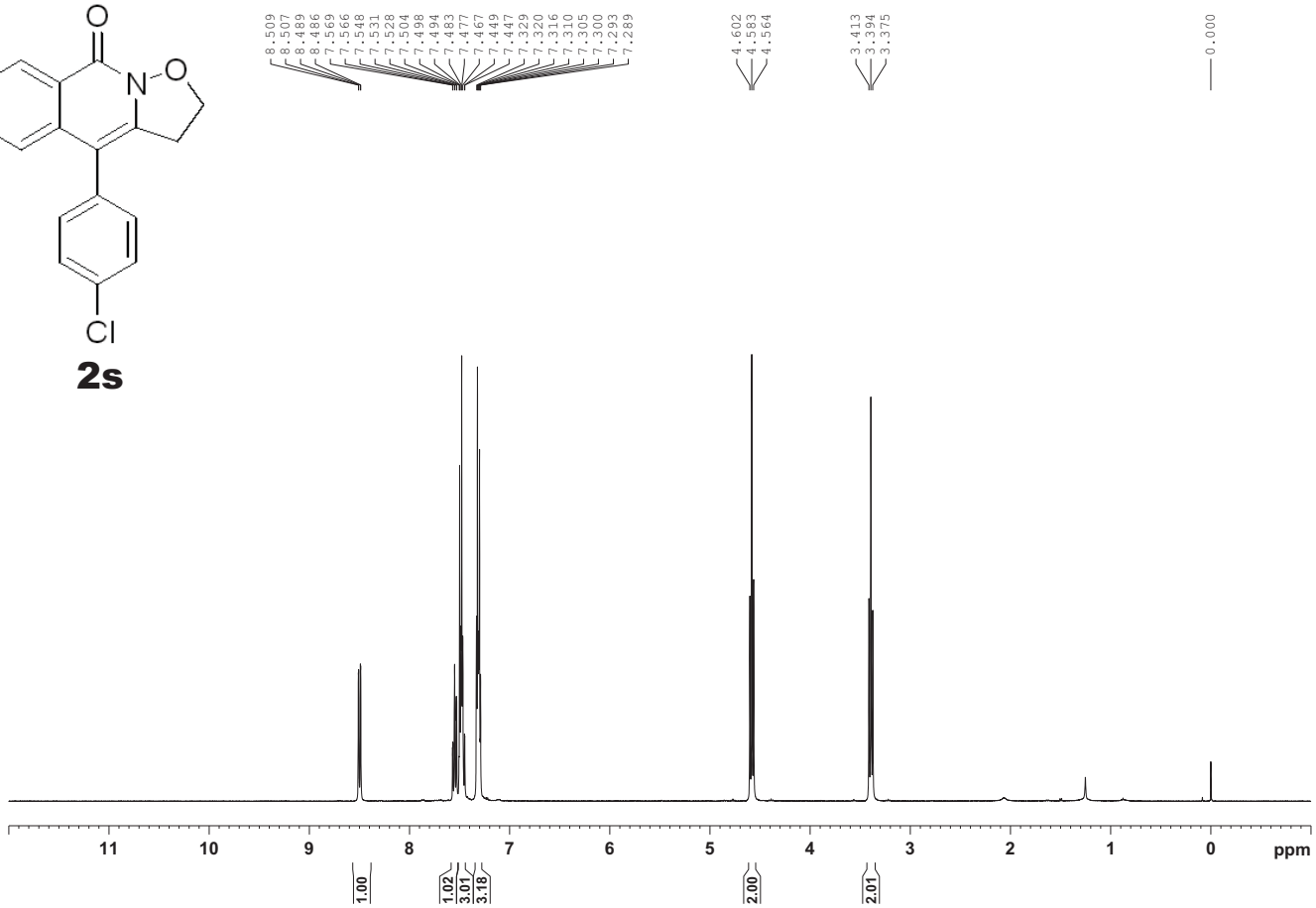


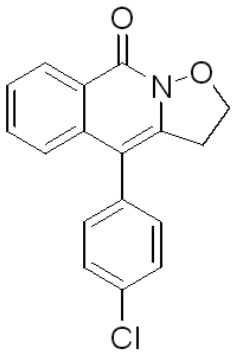
2r


159.19
153.80
136.37
132.45
131.49
131.31
127.26
126.86
126.12
126.06
124.66
114.19
112.63
77.31
77.00
76.68
69.52
55.28
32.33
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

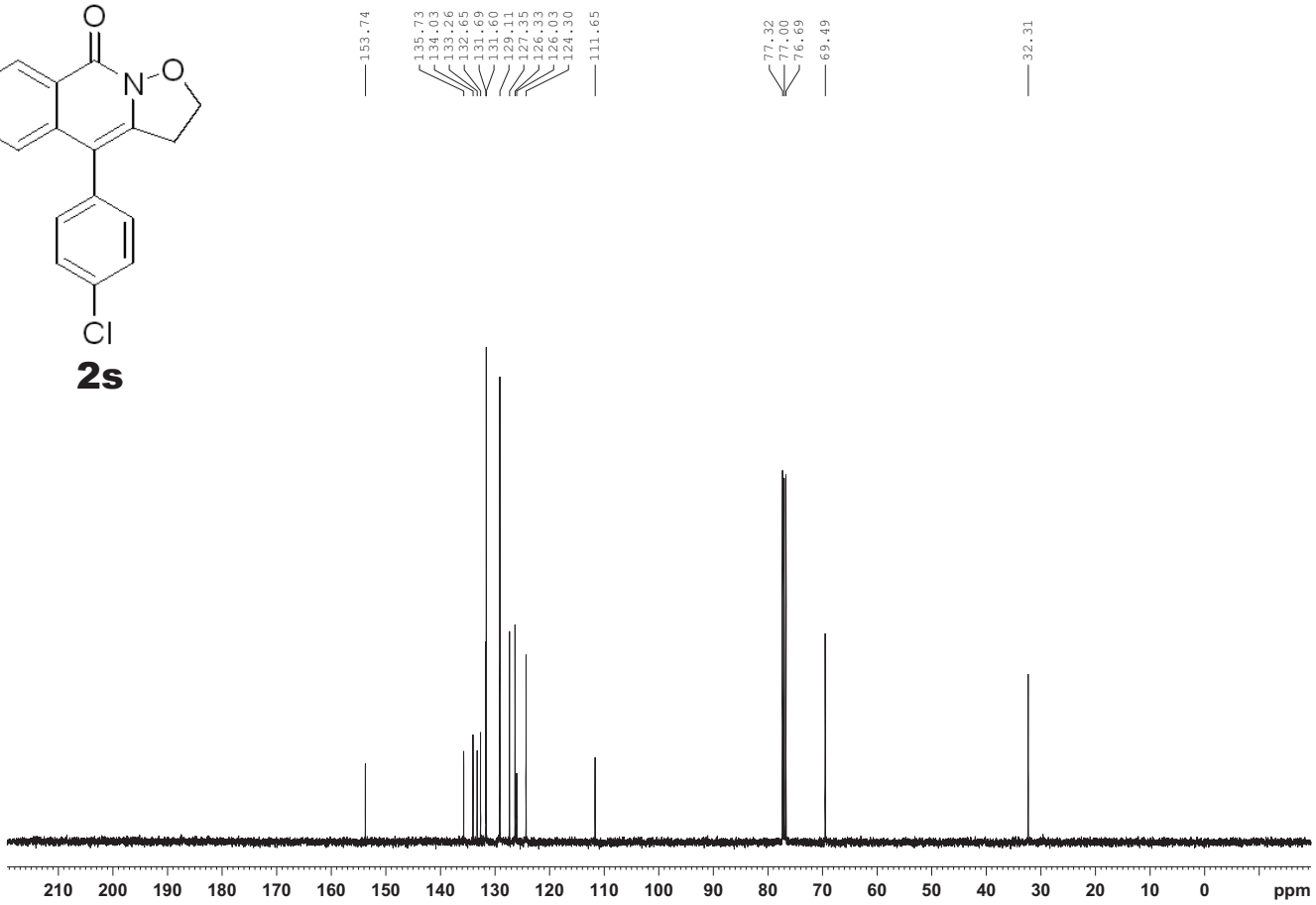


2s

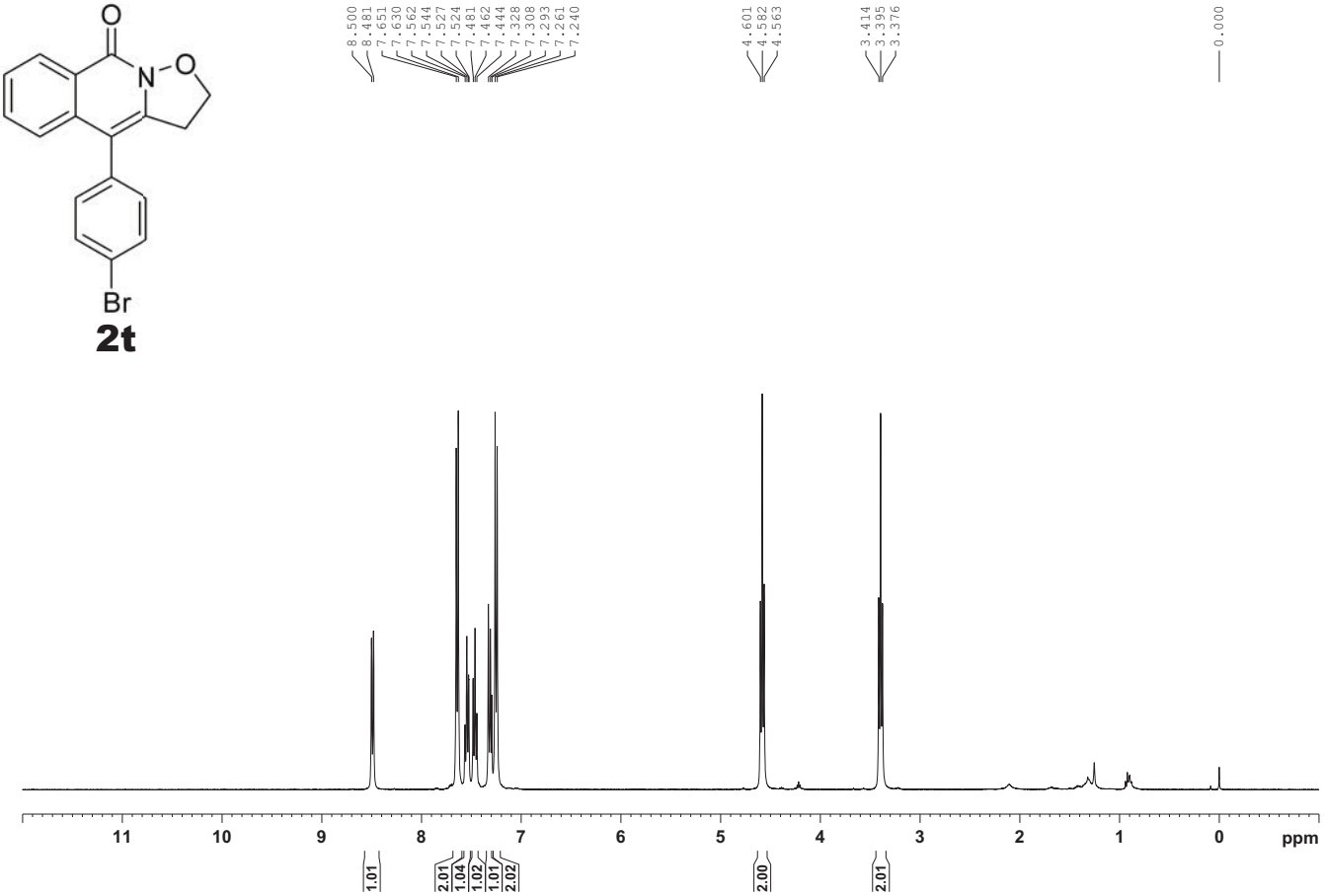

8.509
8.507
8.489
8.486
7.566
7.548
7.531
7.528
7.504
7.498
7.494
7.483
7.477
7.467
7.449
7.447
7.329
7.320
7.316
7.310
7.305
7.300
7.293
7.289
4.602
4.583
4.564
3.413
3.394
3.375
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.02
3.01
3.18
2.00
2.01



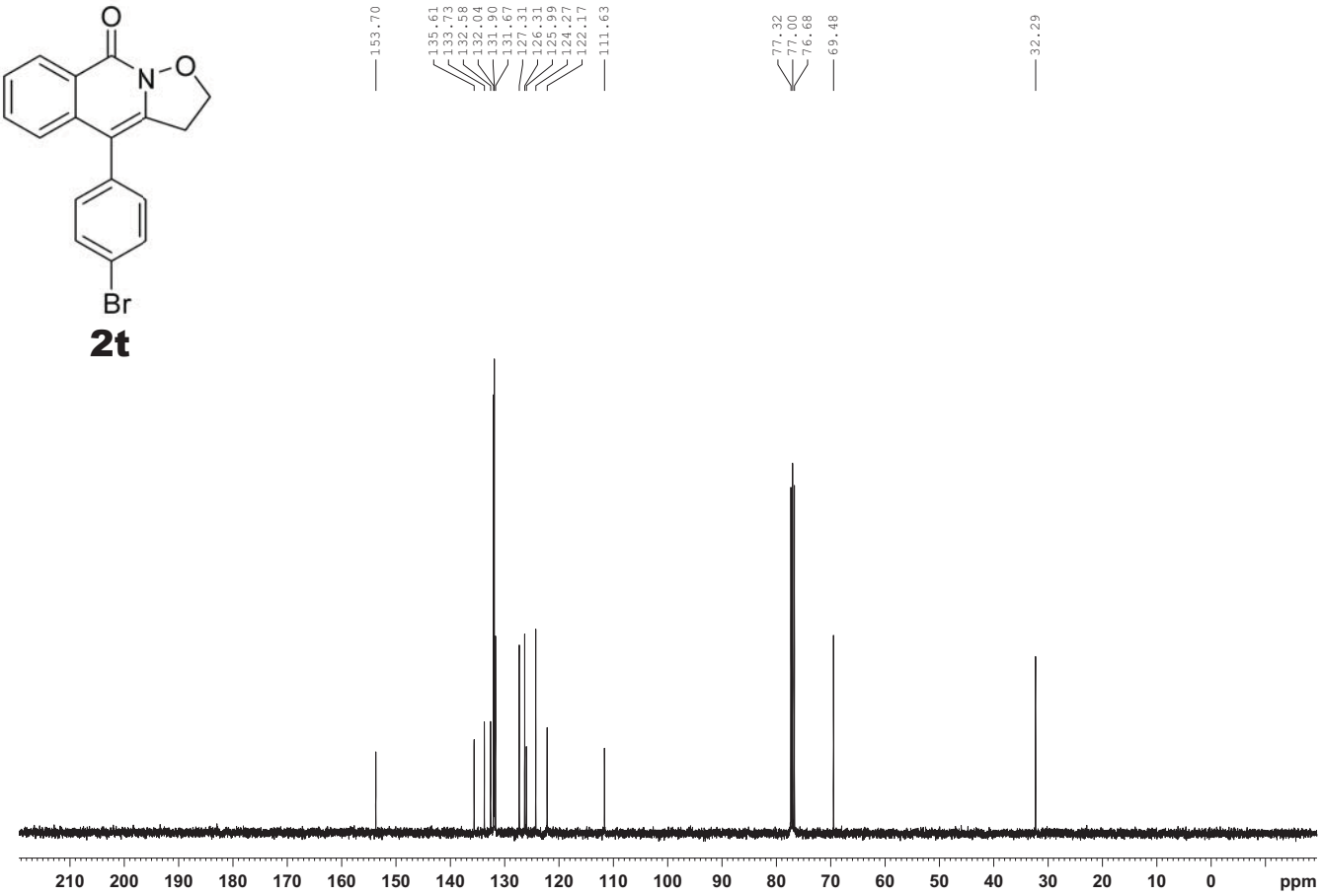
2s

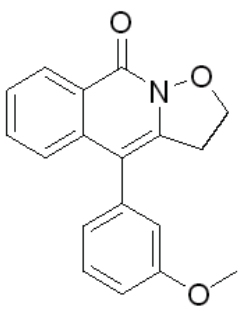

153.74
135.73
134.03
133.26
132.65
131.69
131.60
129.11
127.35
126.33
126.03
124.30
111.65
77.32
77.00
76.69
69.49
32.31
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

2t

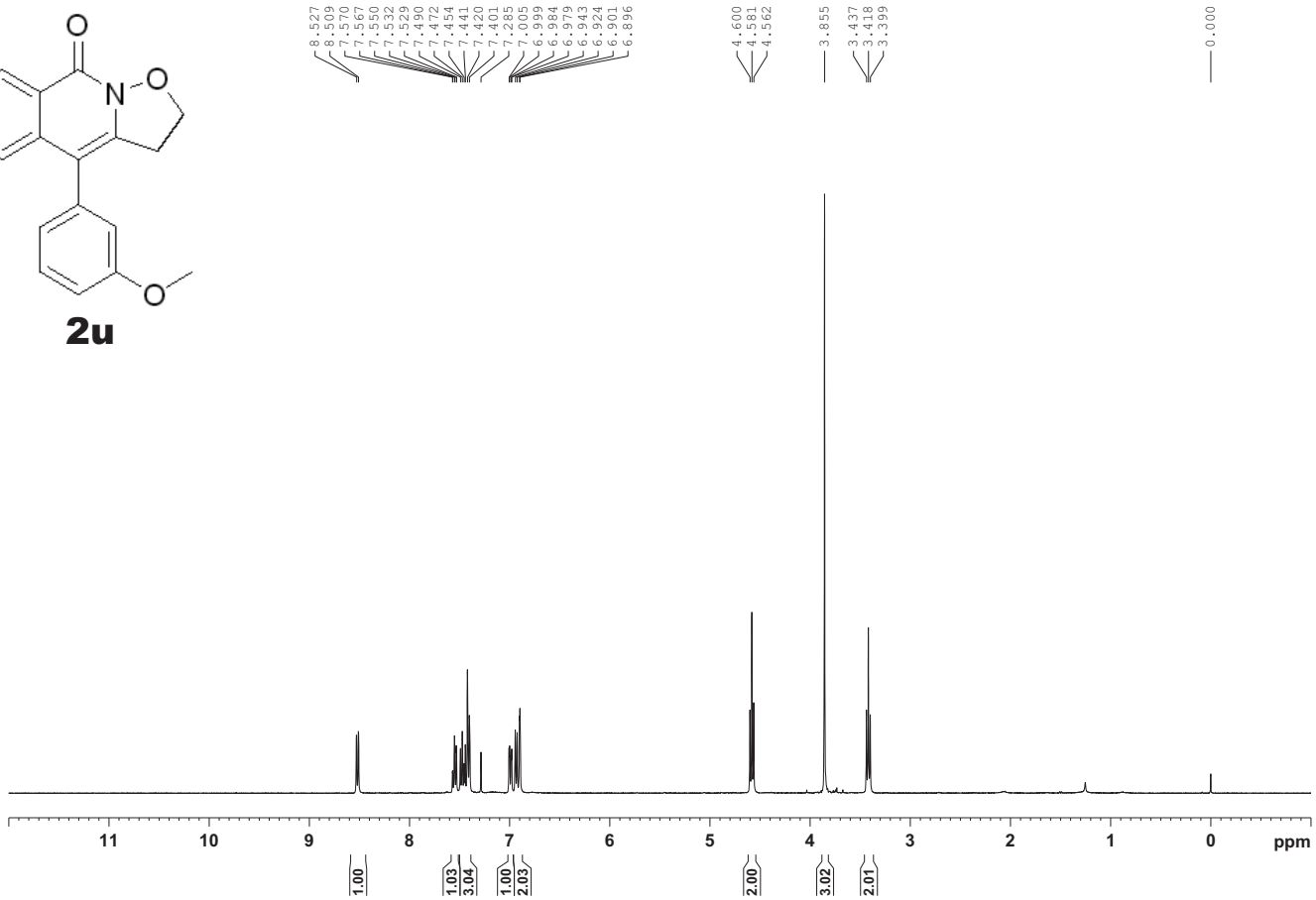

8.500
8.481
7.651
7.630
7.562
7.544
7.527
7.524
7.481
7.462
7.444
7.328
7.308
7.293
7.261
7.240
4.601
4.582
4.563
3.414
3.395
3.376
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.01
2.01
1.04
1.02
1.01
2.02
2.00
2.01

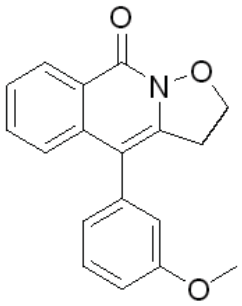
2t


153.70
135.61
133.73
132.58
132.04
131.90
131.67
127.31
126.31
125.99
124.27
122.17
111.63
77.32
77.00
76.68
69.48
32.29
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

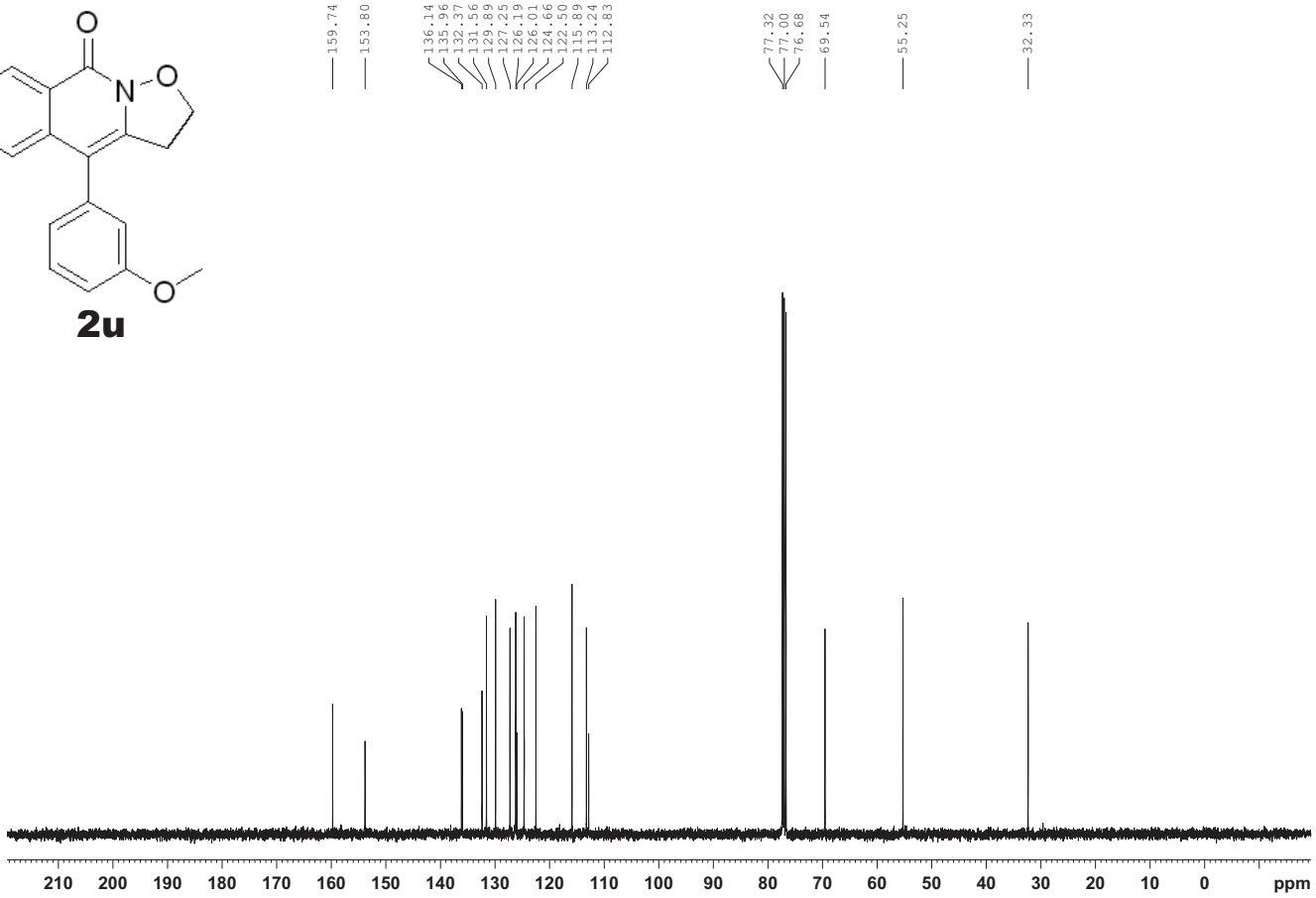


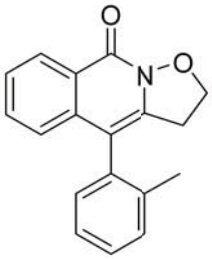
2u


8.527
8.509
7.570
7.567
7.550
7.532
7.529
7.490
7.472
7.454
7.441
7.420
7.401
7.285
7.005
6.999
6.984
6.979
6.943
6.924
6.901
6.896
4.600
4.581
4.562
3.855
3.437
3.418
3.399
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.03
3.04
1.00
2.03
2.00
3.02
2.01

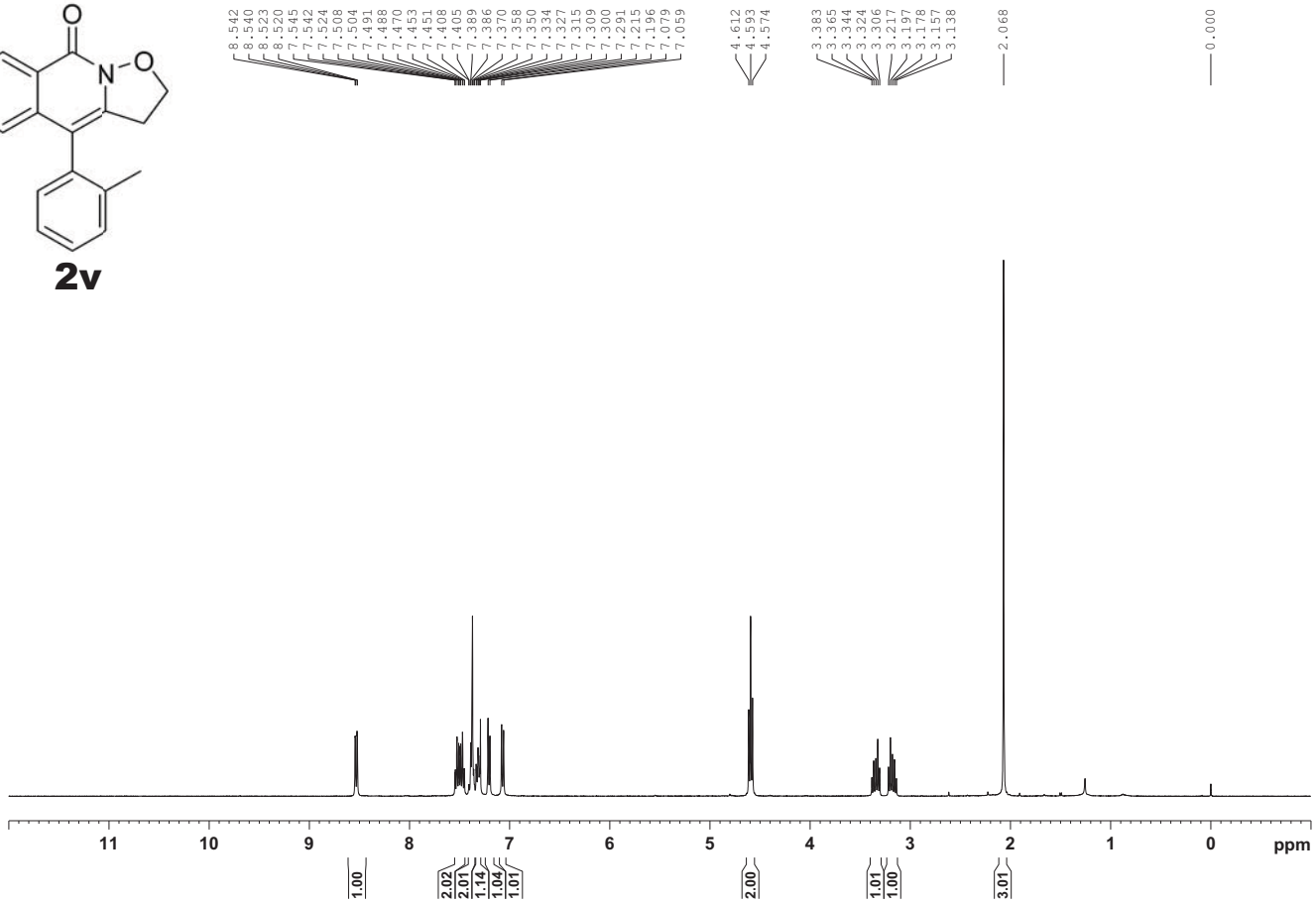


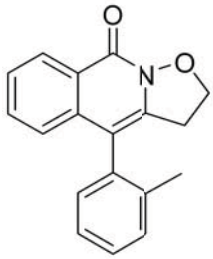
2u


159.74
153.80
136.14
135.96
132.37
131.56
129.89
127.25
126.19
126.01
124.66
122.50
115.89
113.24
112.83
77.32
77.00
76.68
69.54
55.25
32.33
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

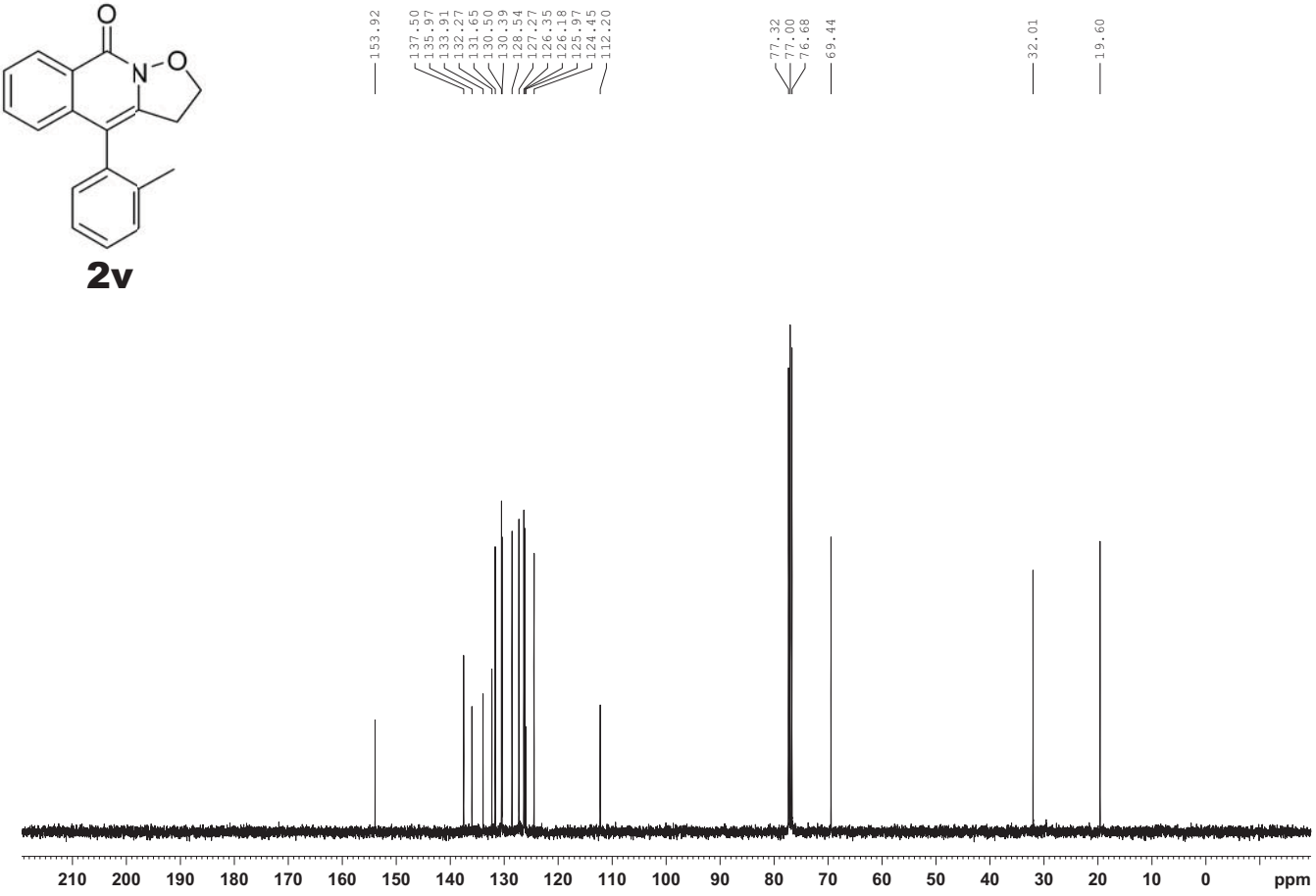


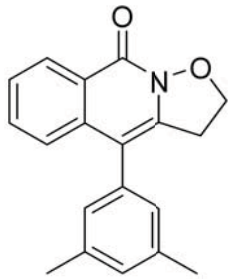
2v


8.542
8.540
8.523
8.520
7.545
7.542
7.524
7.508
7.504
7.491
7.488
7.470
7.453
7.451
7.408
7.405
7.389
7.386
7.370
7.358
7.350
7.334
7.327
7.315
7.309
7.300
7.291
7.215
7.196
7.079
7.059
4.612
4.593
4.574
3.383
3.365
3.344
3.324
3.306
3.217
3.197
3.178
3.157
3.138
2.068
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
2.02
2.01
1.14
1.04
1.01
2.00
1.01
1.00
3.01

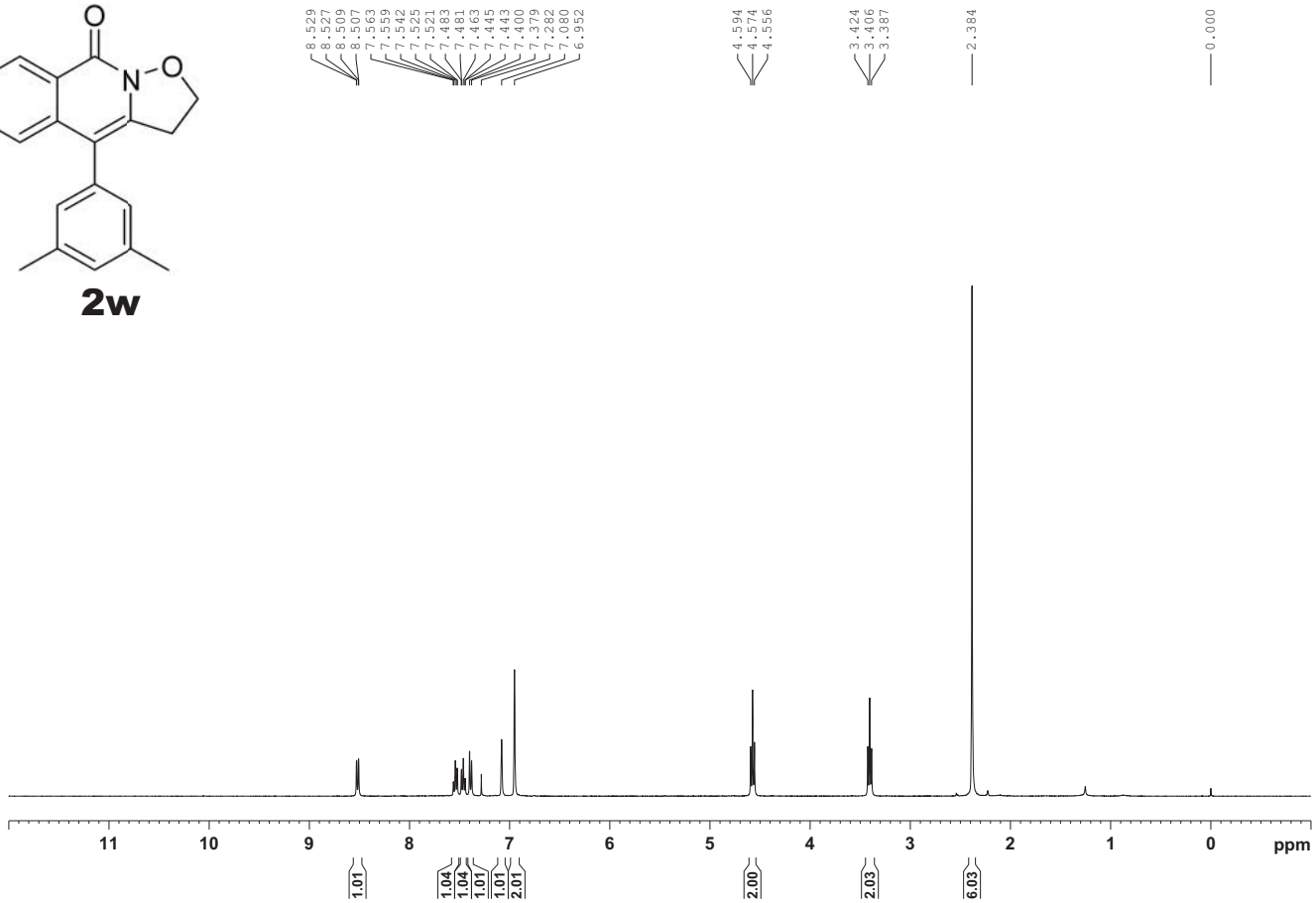


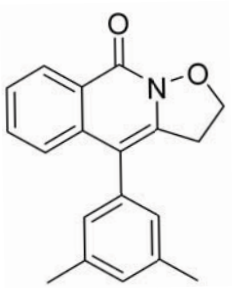
2v


153.92
137.50
135.97
133.91
132.27
131.65
130.50
130.39
128.54
127.27
126.35
126.18
125.97
124.45
112.20
77.32
77.00
76.68
69.44
32.01
19.60
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

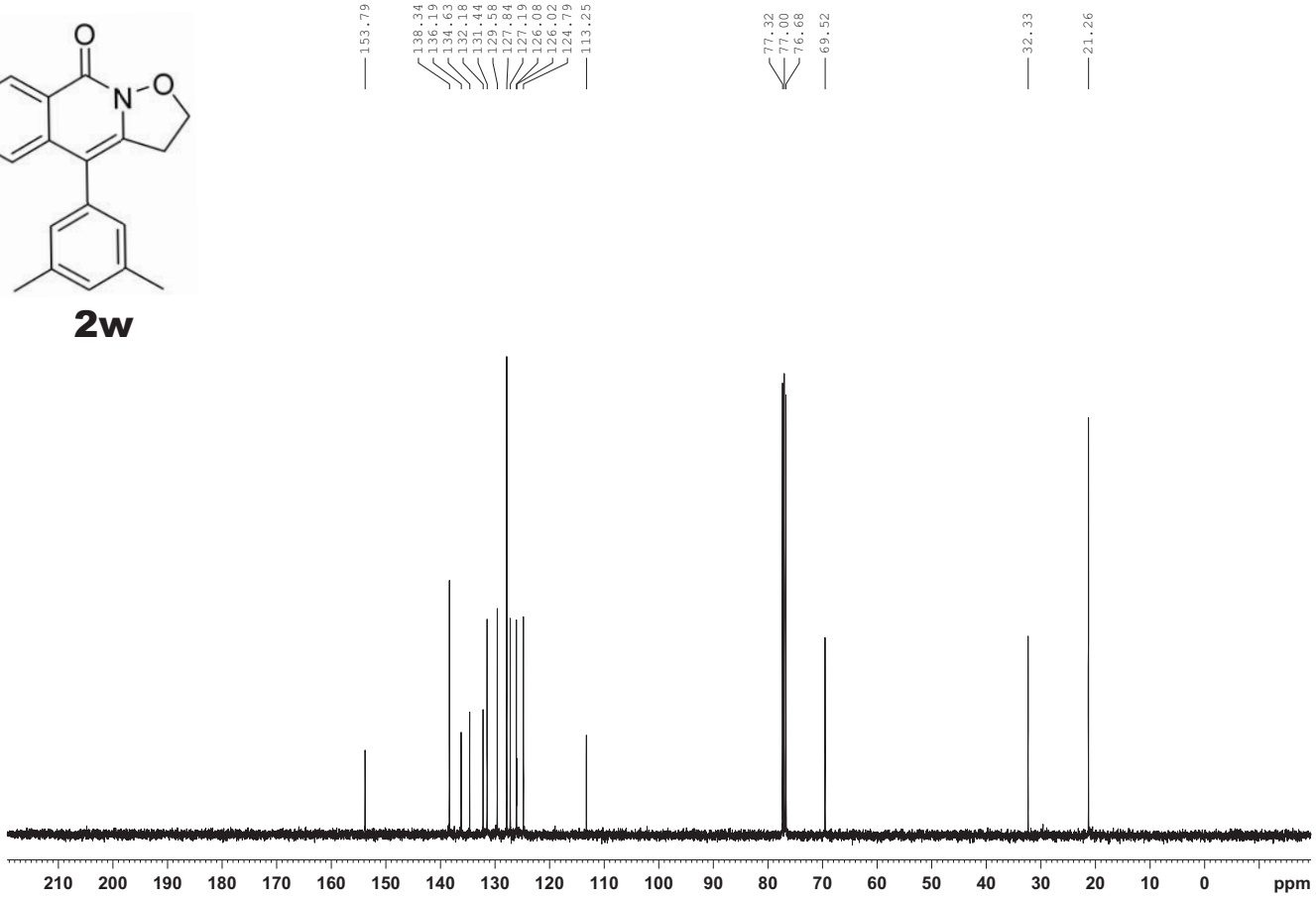


2w

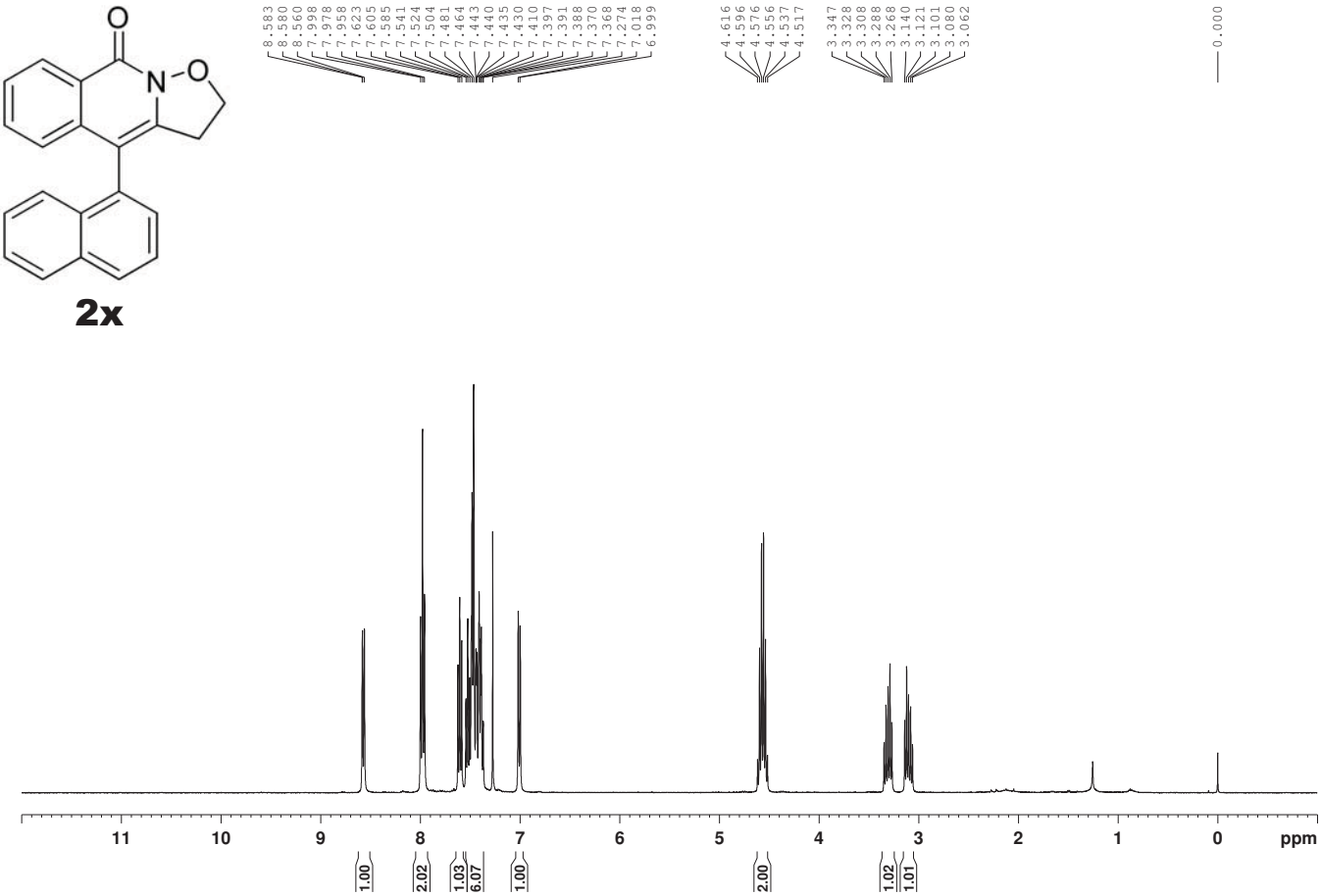

8.529
8.527
8.509
8.507
7.563
7.559
7.542
7.525
7.521
7.483
7.481
7.463
7.445
7.443
7.400
7.379
7.282
7.080
6.952
4.594
4.574
4.556
3.424
3.406
3.387
2.384
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.01
1.04
1.04
1.01
1.01
2.01
2.00
2.03
6.03



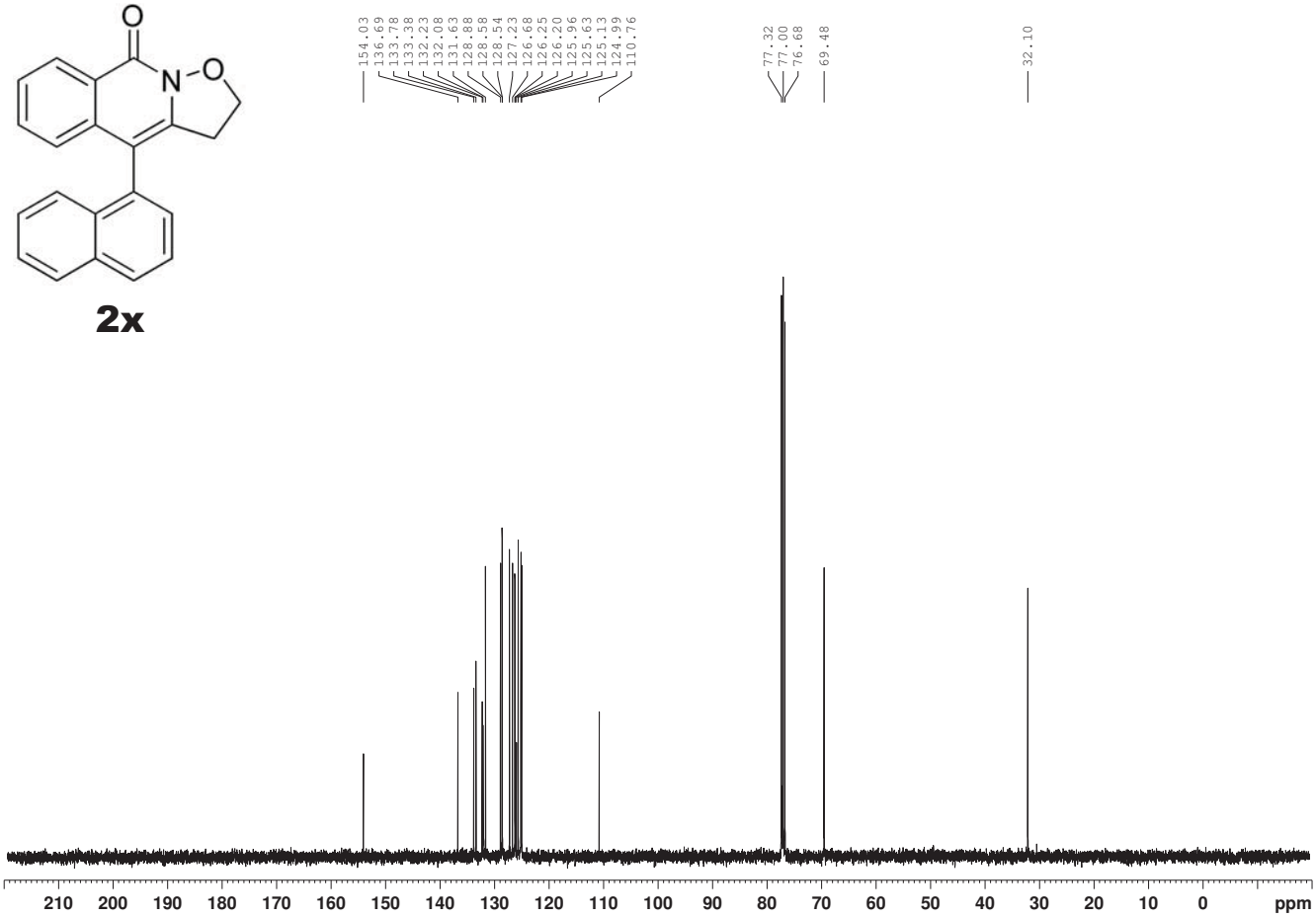
2w


153.79
138.34
136.19
134.63
132.18
131.44
129.58
127.84
127.19
126.08
126.02
124.79
113.25
77.32
77.00
76.68
69.52
32.33
21.26
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

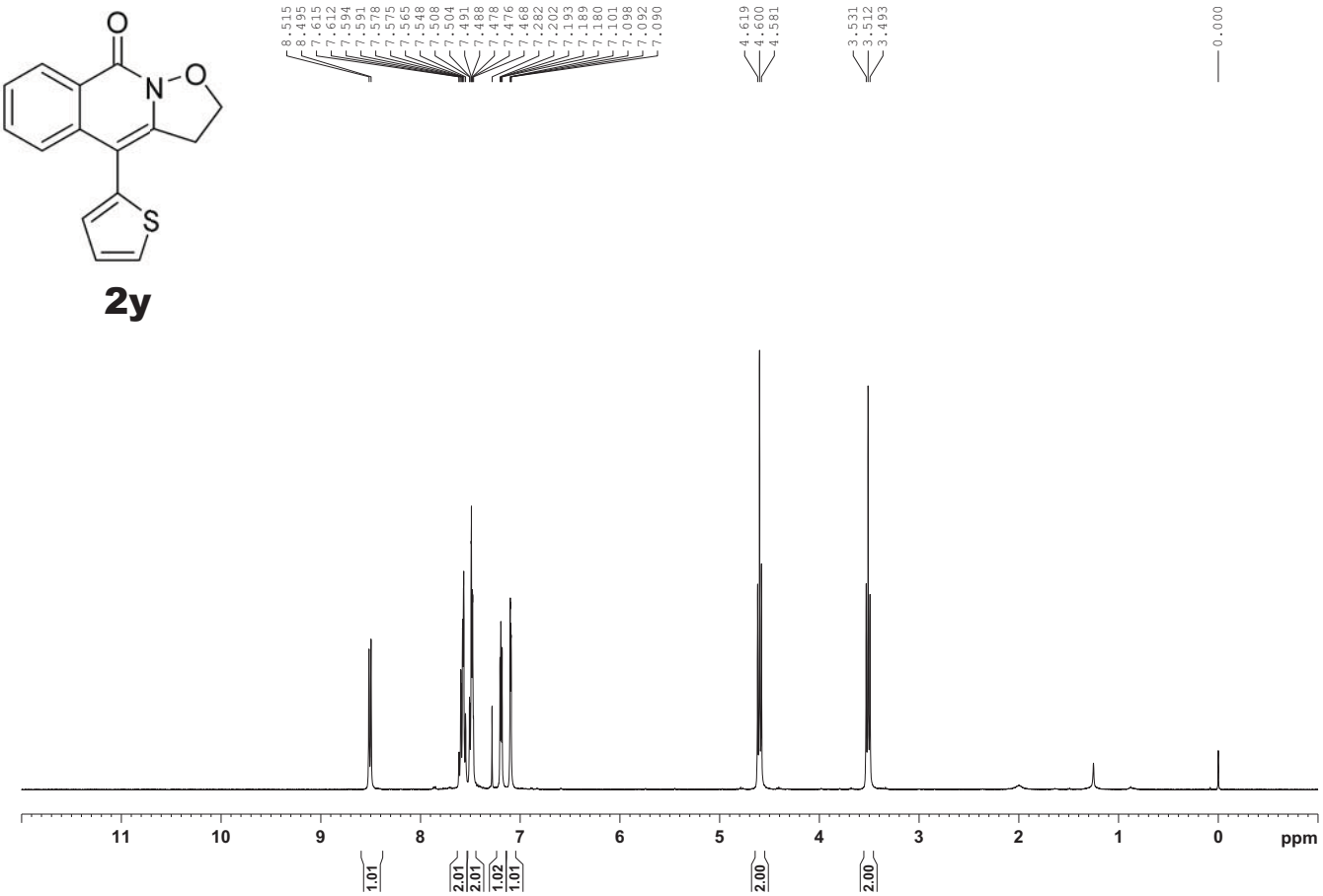
2x

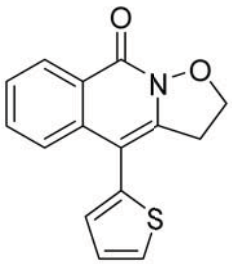

8.583
8.580
8.560
7.998
7.978
7.958
7.623
7.605
7.585
7.541
7.524
7.504
7.481
7.464
7.443
7.440
7.435
7.430
7.410
7.397
7.391
7.388
7.370
7.368
7.274
7.018
6.999
4.616
4.596
4.576
4.556
4.537
4.517
3.347
3.328
3.308
3.288
3.268
3.140
3.121
3.101
3.080
3.062
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
2.02
1.03
6.07
1.00
2.00
1.02
1.01

2x

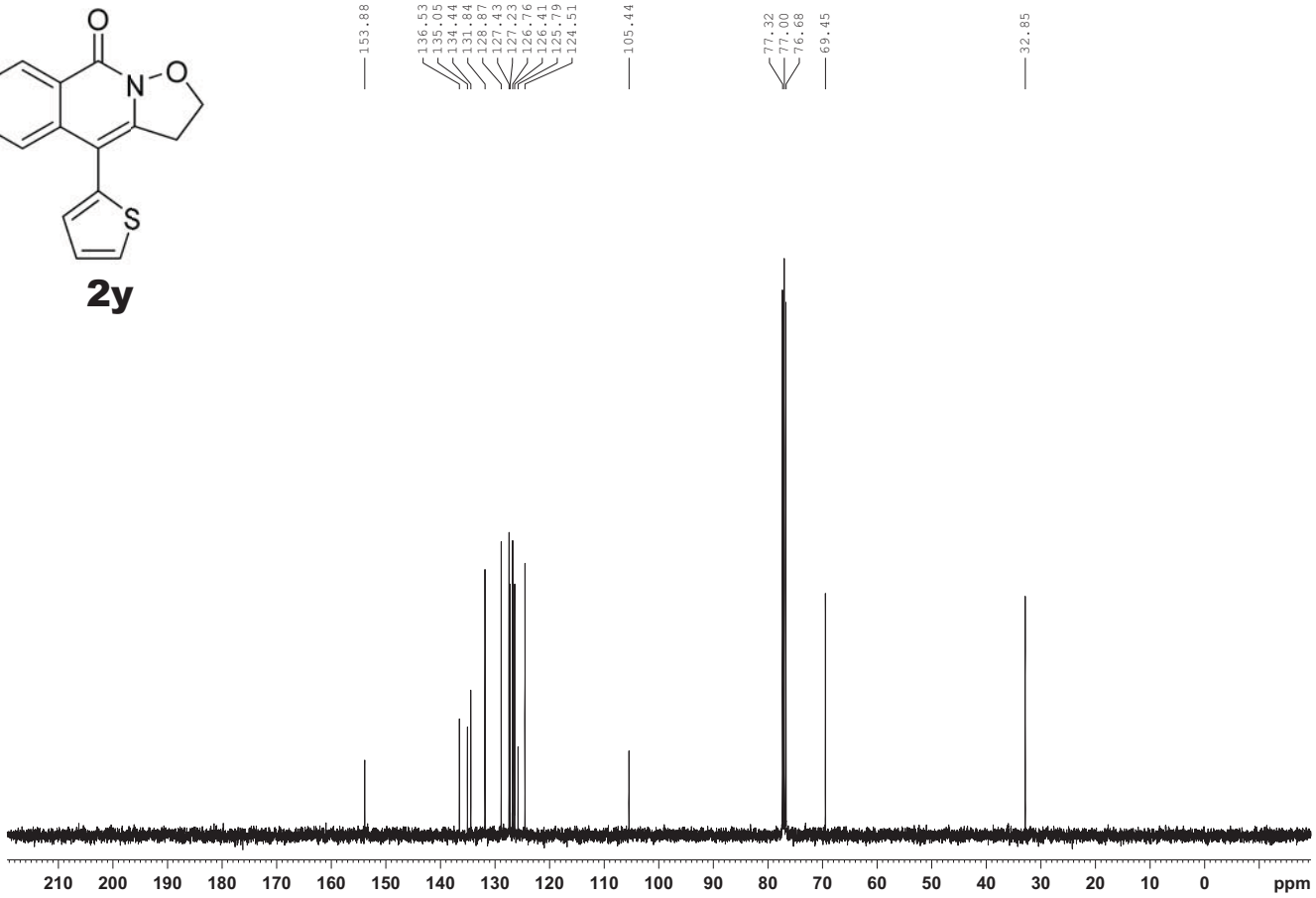

154.03
136.69
133.78
133.38
132.23
132.08
131.63
128.88
128.58
128.54
127.23
126.68
126.25
126.20
125.96
125.63
125.13
124.99
110.76
77.32
77.00
76.68
69.48
32.10
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

2y

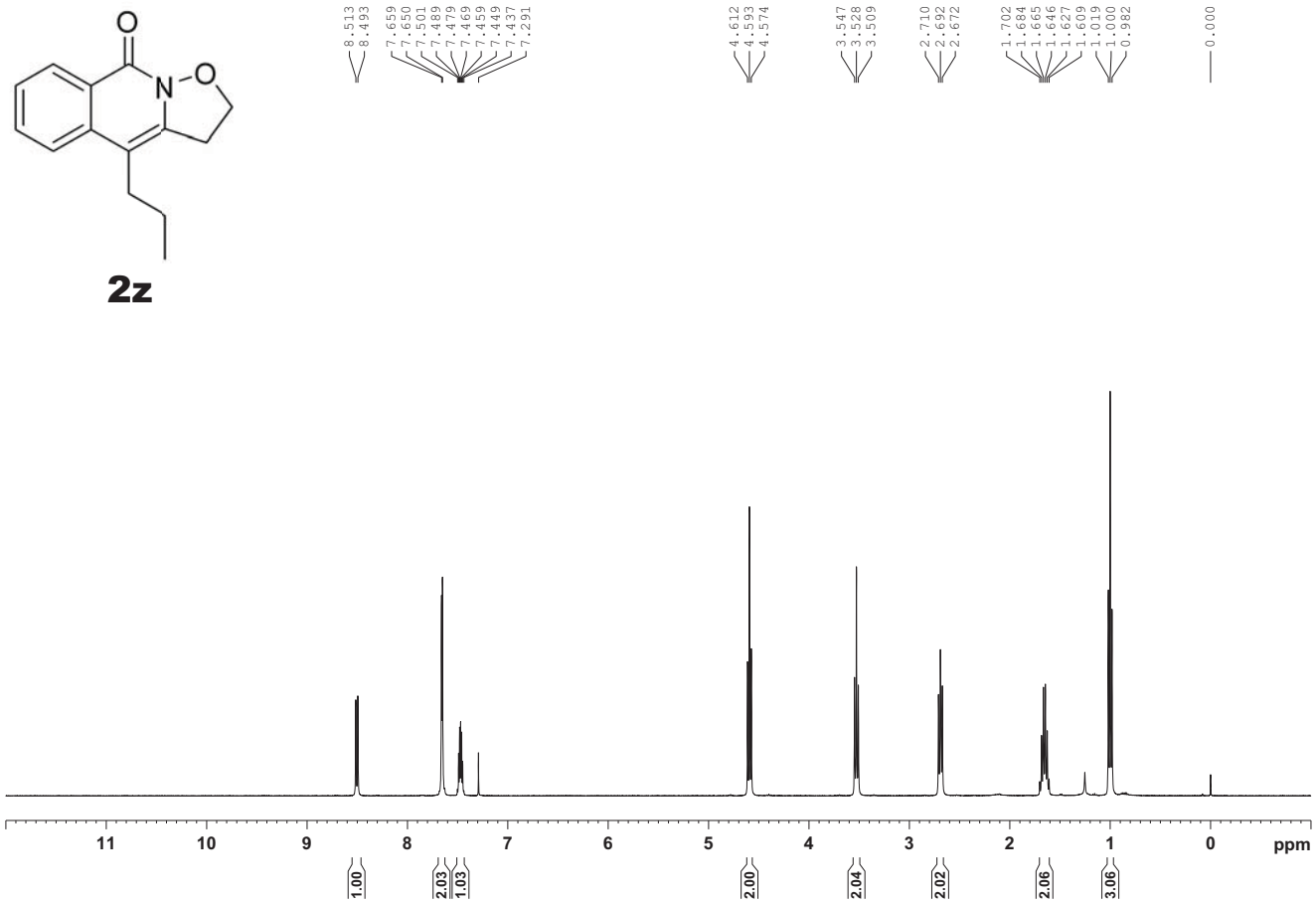

8.515
8.495
7.615
7.612
7.594
7.591
7.578
7.575
7.565
7.548
7.508
7.504
7.491
7.488
7.478
7.476
7.468
7.282
7.202
7.193
7.189
7.180
7.101
7.098
7.092
7.090
4.619
4.600
4.581
3.531
3.512
3.493
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.01
2.01
2.01
1.02
1.01
2.00
2.00



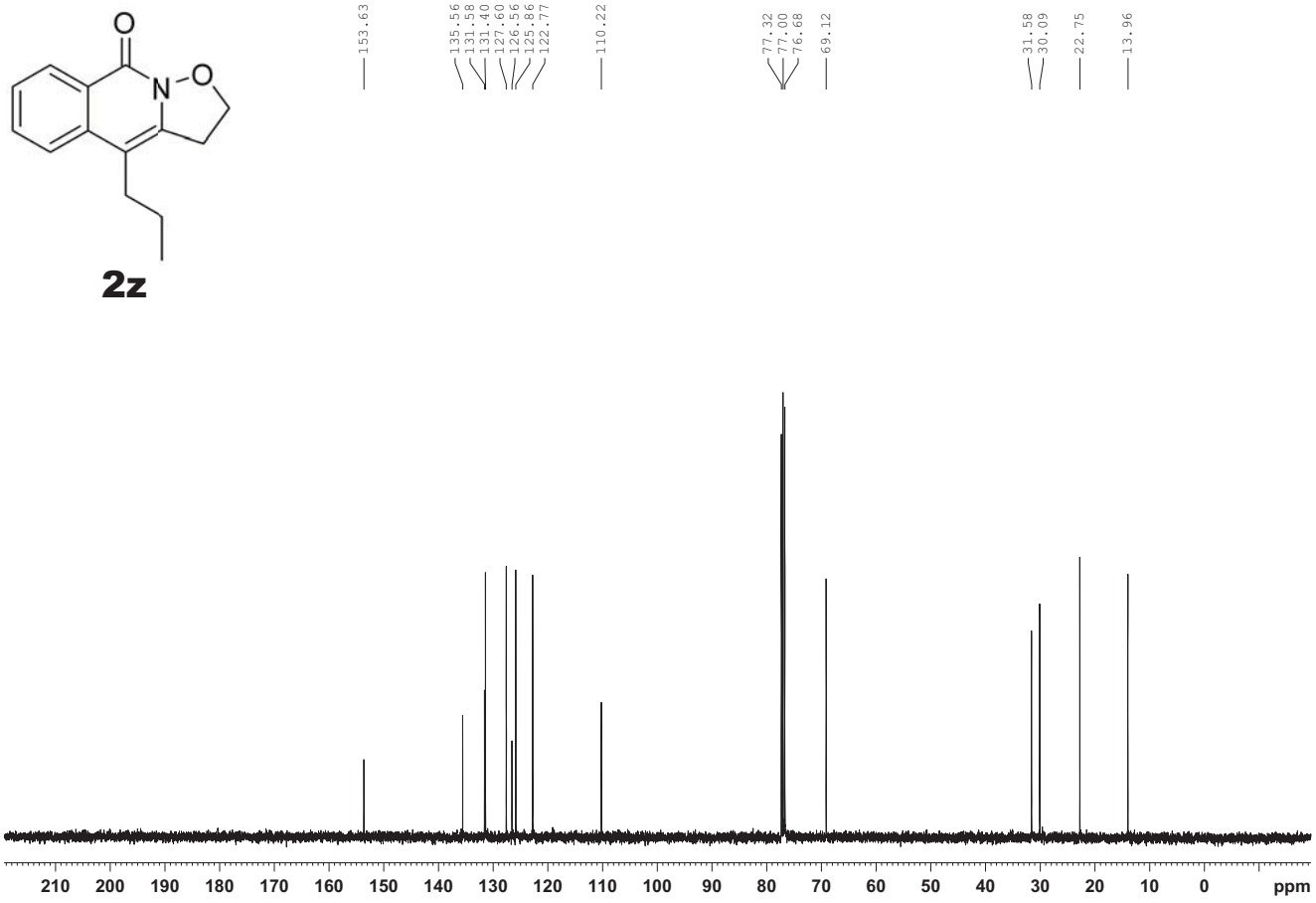
2y


153.88
136.53
135.05
134.44
131.84
128.87
127.43
127.23
126.76
126.41
125.79
124.51
105.44
77.32
77.00
76.68
69.45
32.85
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

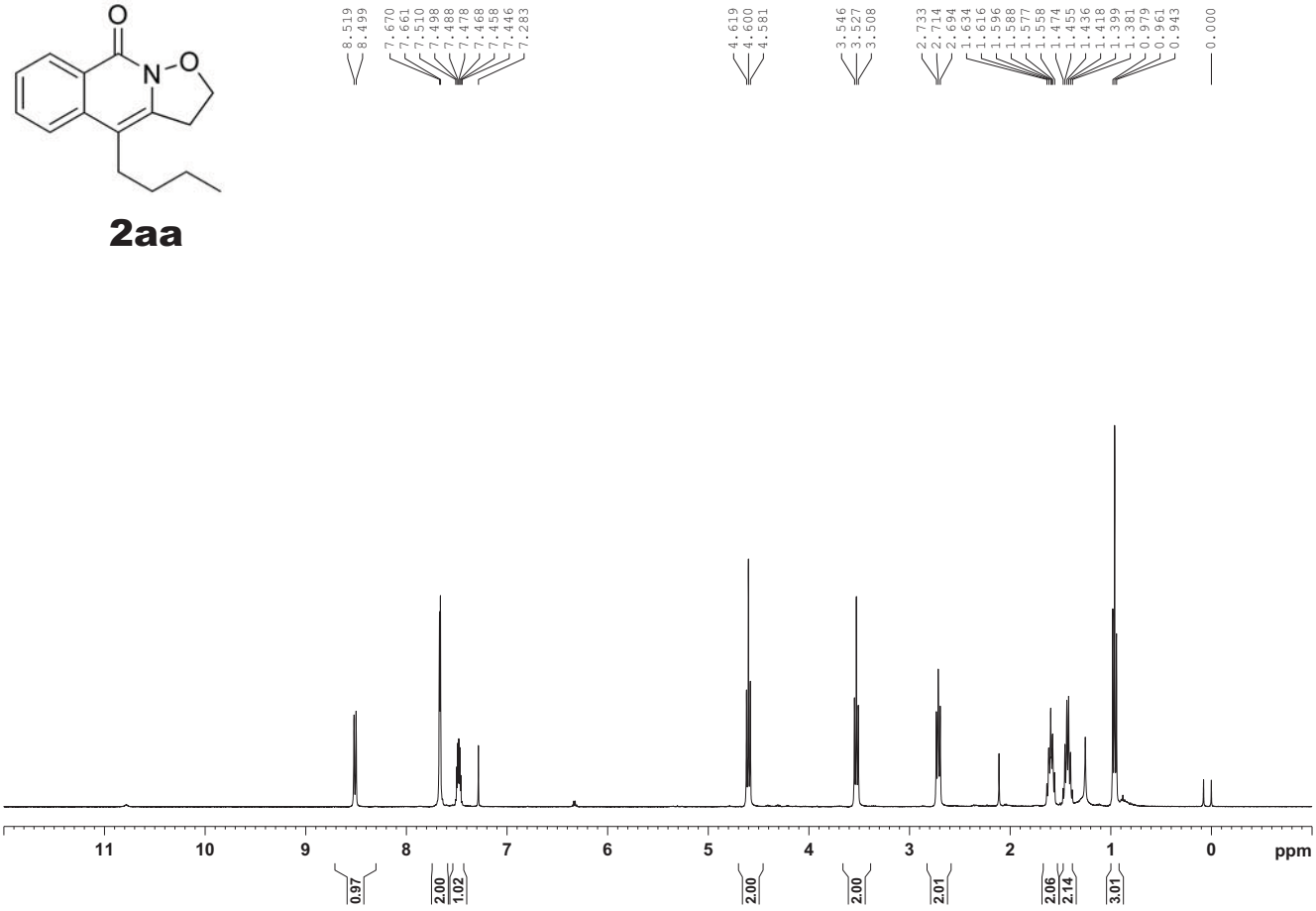
2z


8.513
8.493
7.659
7.650
7.501
7.489
7.479
7.469
7.459
7.449
7.437
7.291
4.612
4.593
4.574
3.547
3.528
3.509
2.710
2.692
2.672
1.702
1.684
1.665
1.646
1.627
1.609
1.019
1.000
0.982
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
2.03
1.03
2.00
2.04
2.02
2.06
3.06

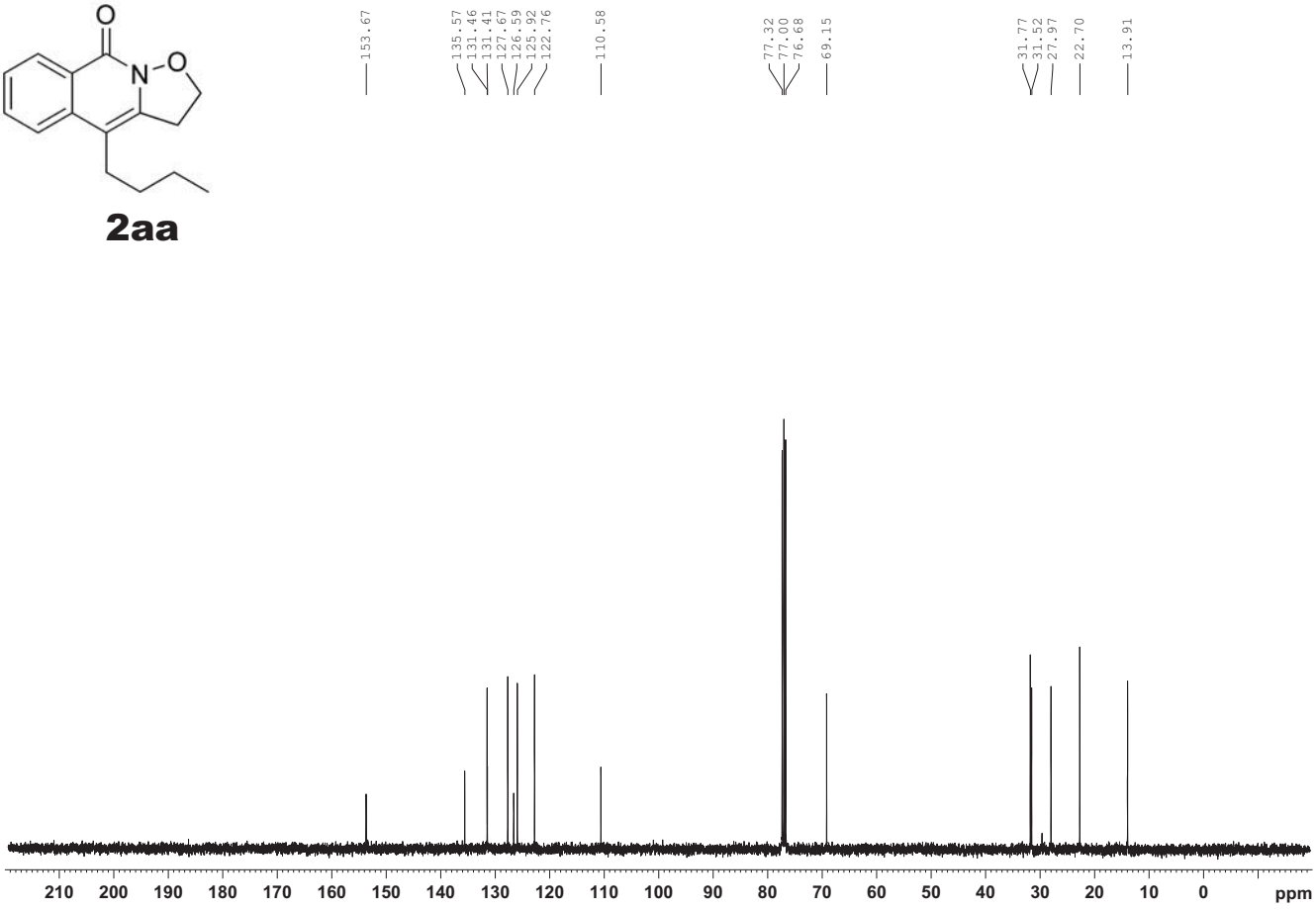
2z

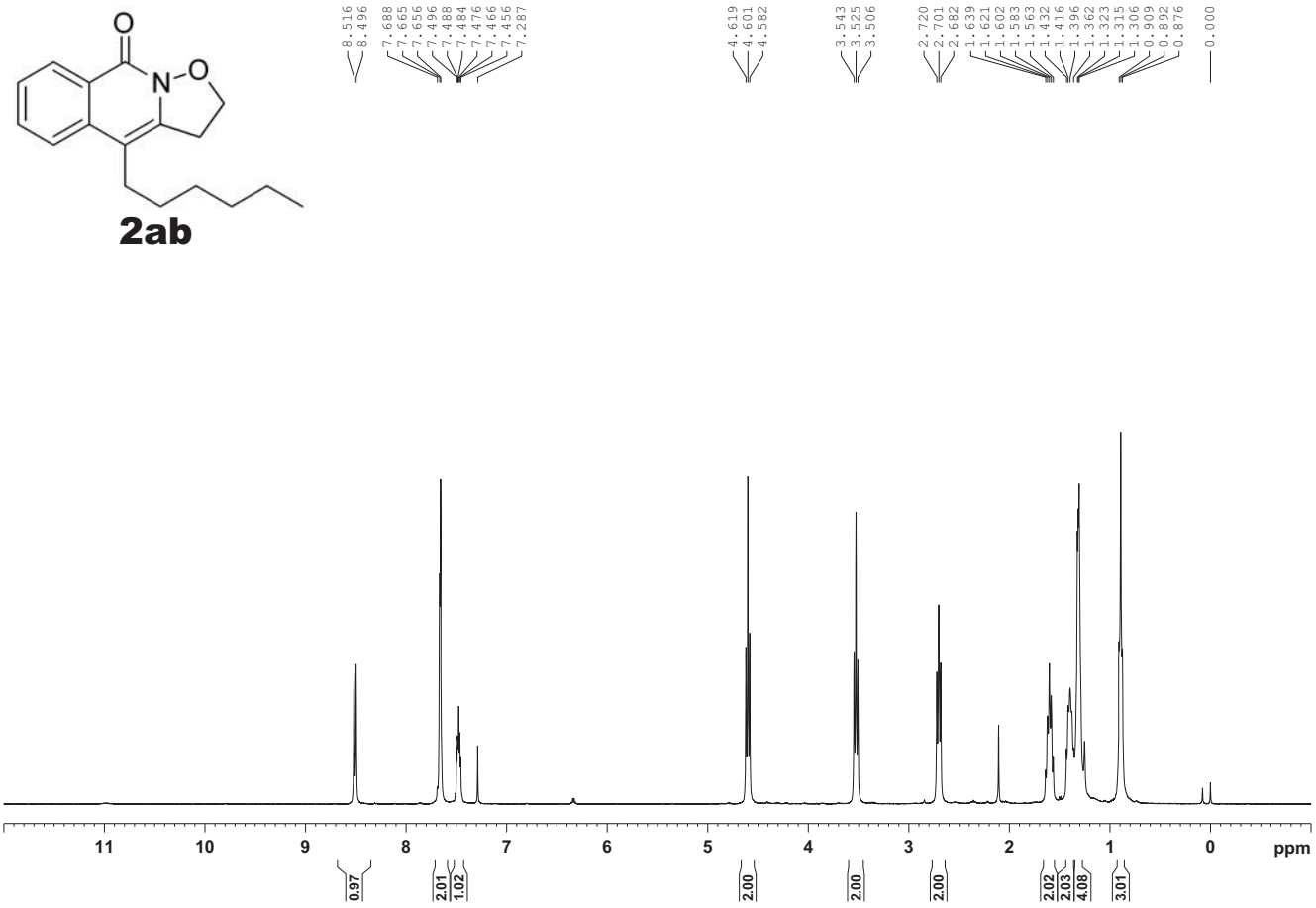

153.63
135.56
131.58
131.40
127.60
126.56
125.86
122.77
110.22
77.32
77.00
76.68
69.12
31.58
30.09
22.75
13.96
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

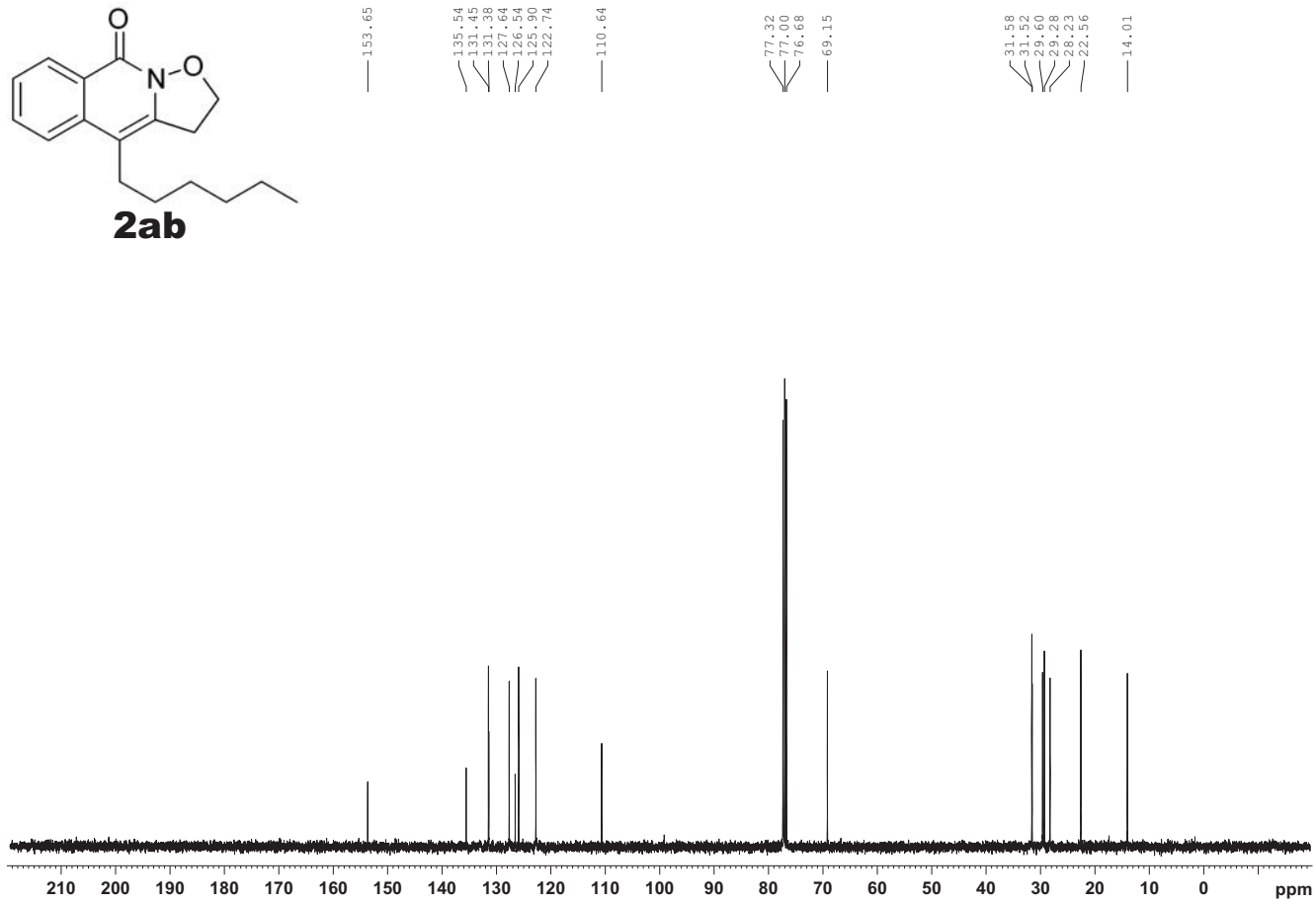
2aa

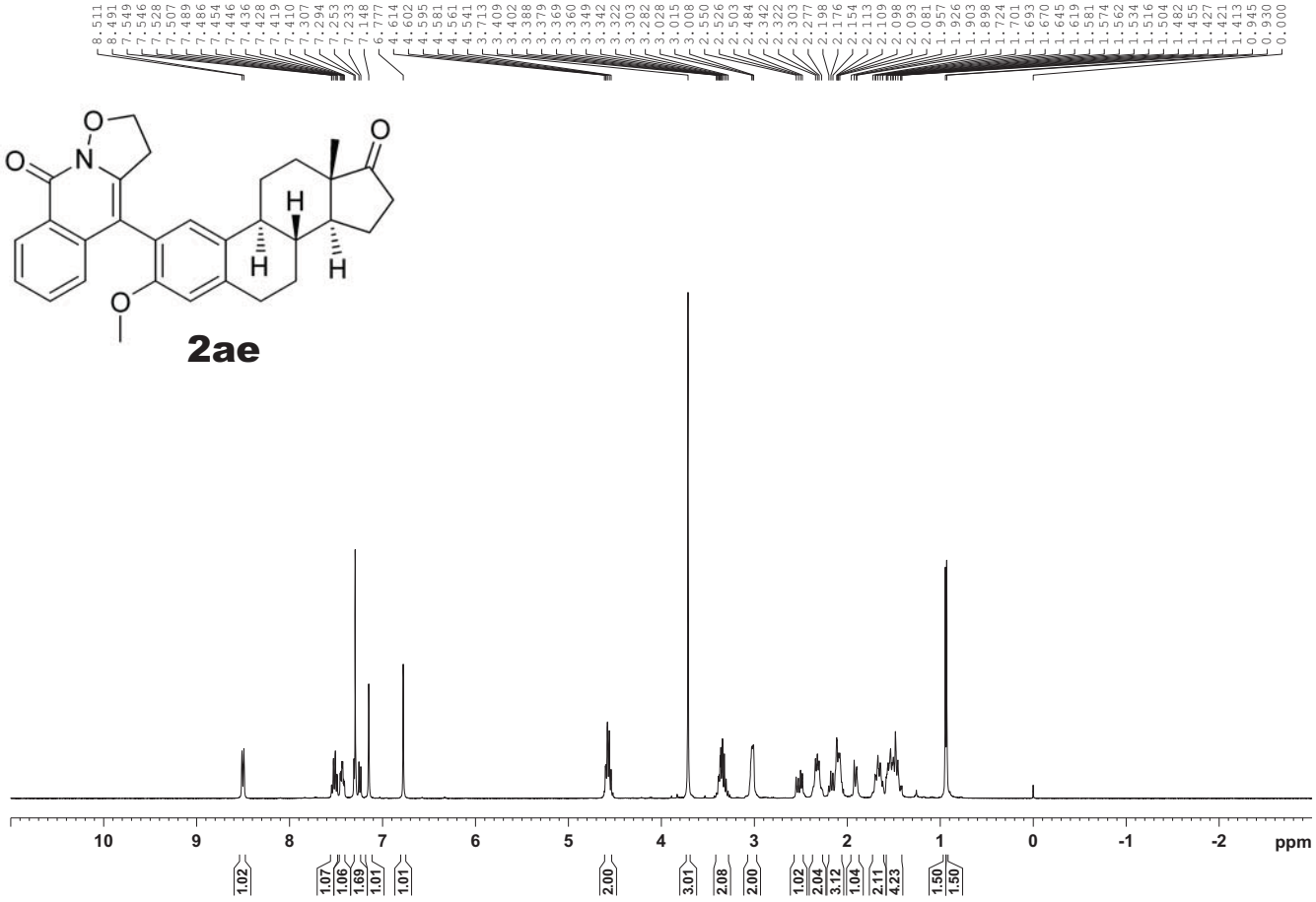

8.519
8.499
7.670
7.661
7.510
7.498
7.488
7.478
7.468
7.458
7.446
7.283
4.619
4.600
4.581
3.546
3.527
3.508
2.733
2.714
2.694
1.634
1.616
1.596
1.588
1.577
1.558
1.474
1.455
1.436
1.418
1.399
1.381
0.979
0.961
0.943
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.97
2.00
1.02
2.00
2.00
2.01
2.06
2.14
3.01

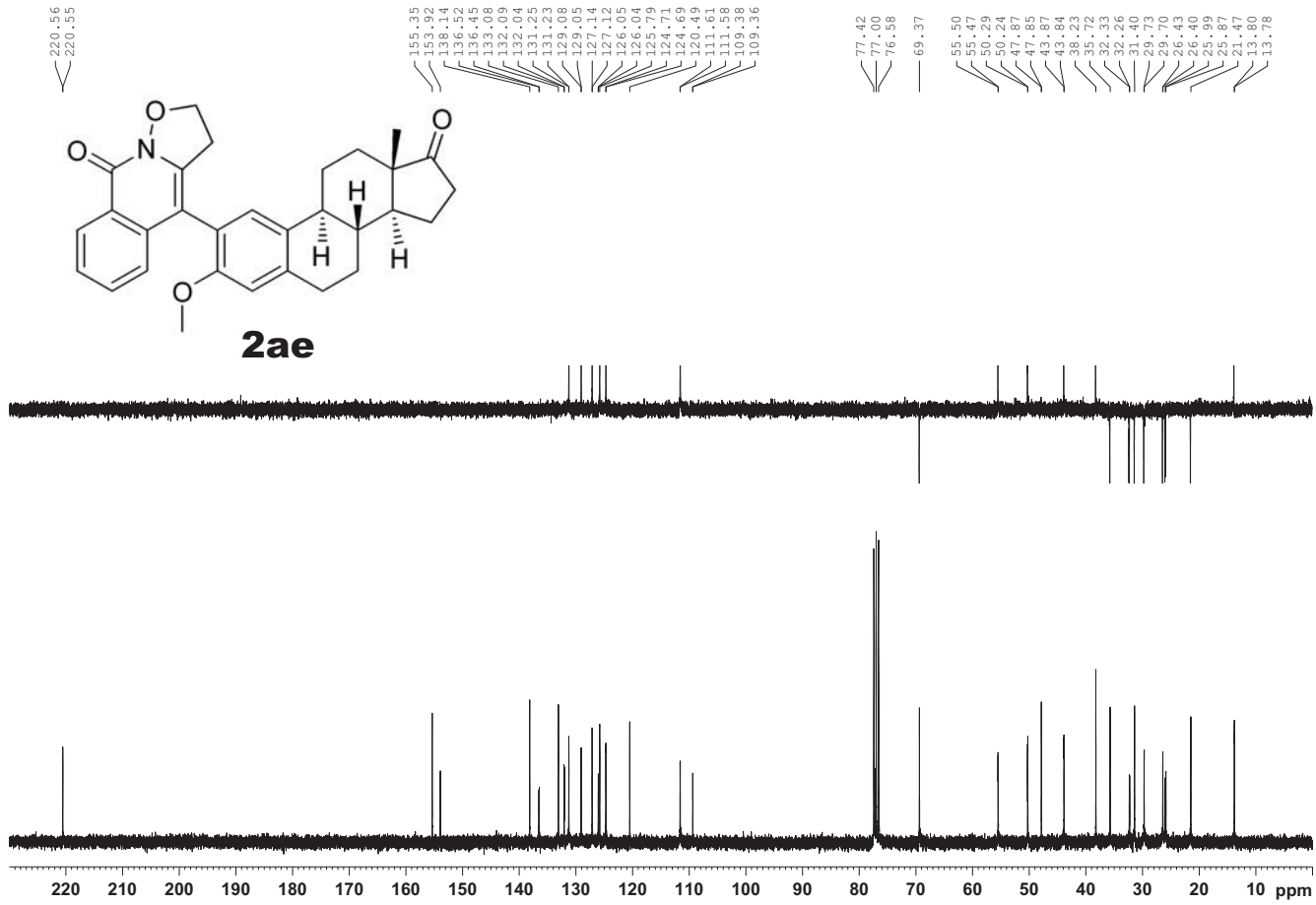
2aa


153.67
135.57
131.46
131.41
127.67
126.59
125.92
122.76
110.58
77.32
77.00
76.68
69.15
31.77
31.52
27.97
22.70
13.91
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

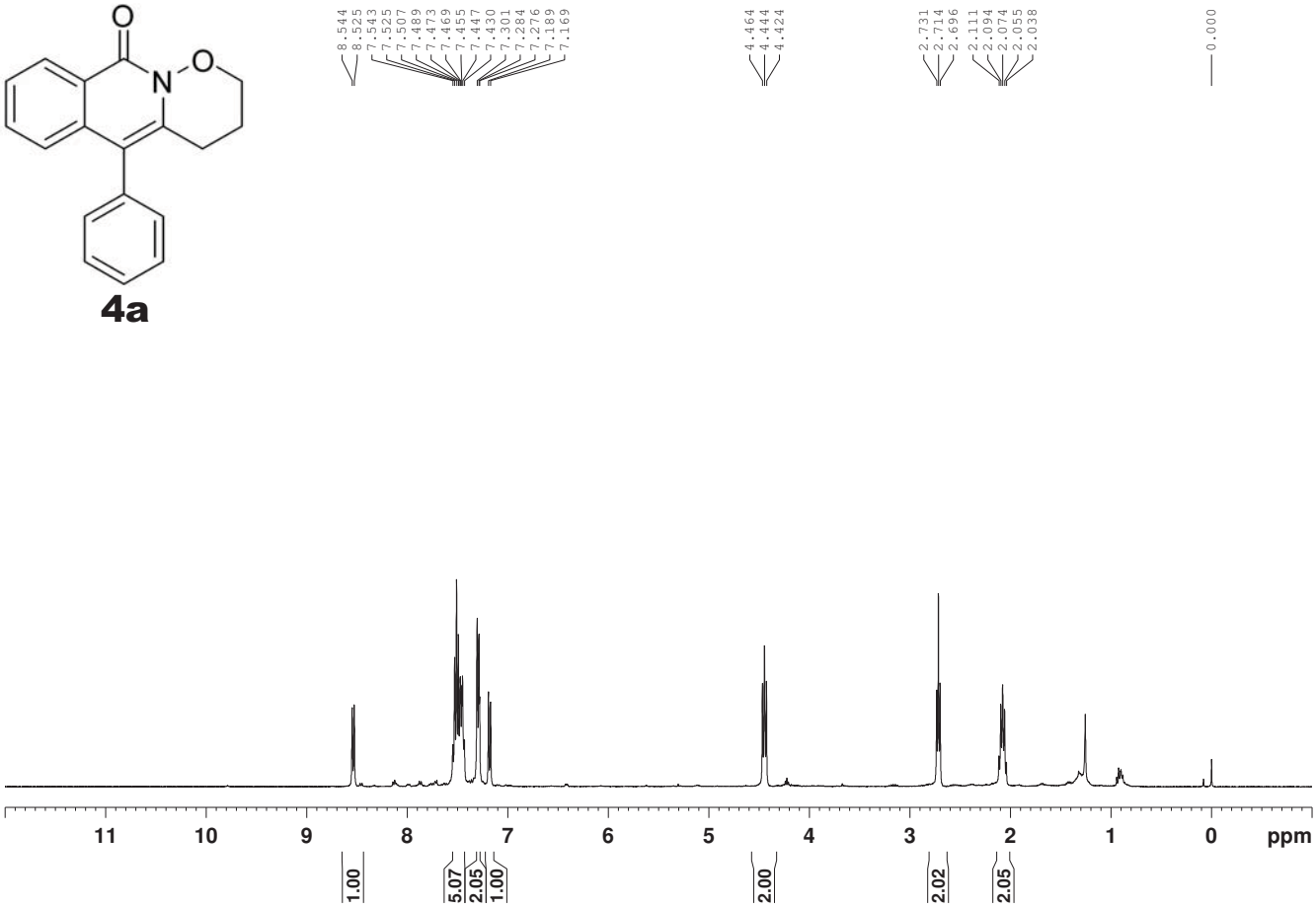

2ab
8.516
8.496
7.688
7.665
7.656
7.496
7.488
7.484
7.476
7.466
7.456
7.287
4.619
4.601
4.582
3.543
3.525
3.506
2.720
2.701
2.682
1.639
1.621
1.602
1.583
1.563
1.432
1.416
1.396
1.362
1.323
1.315
1.306
0.909
0.892
0.876
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.97
2.01
1.02
2.00
2.00
2.00
2.02
2.03
4.08
3.01


2ab
153.65
135.54
131.45
131.38
127.64
126.54
125.90
122.74
110.64
77.32
77.00
76.68
69.15
31.58
31.52
29.60
29.28
28.23
22.56
14.01
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

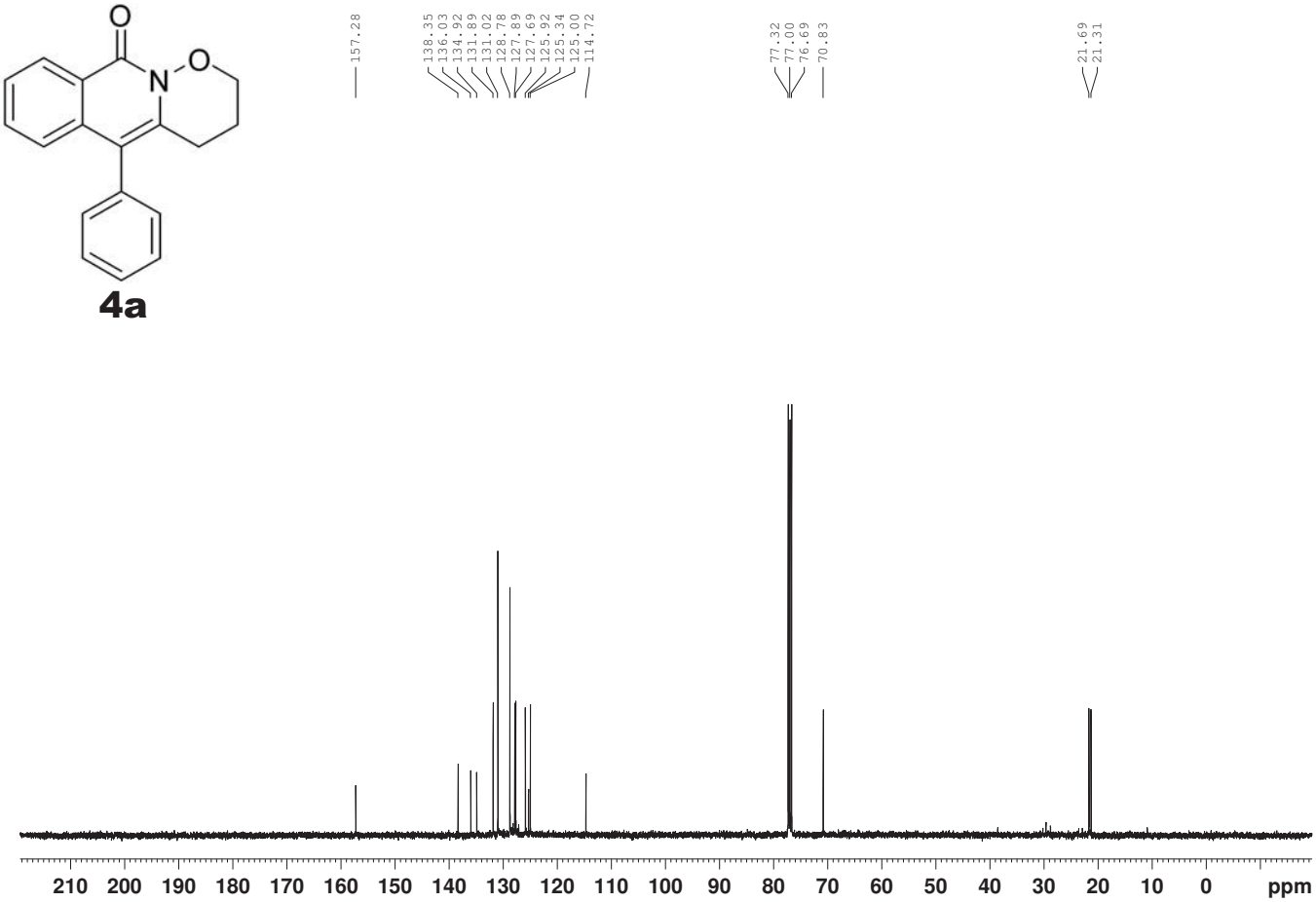

2ae
ppm

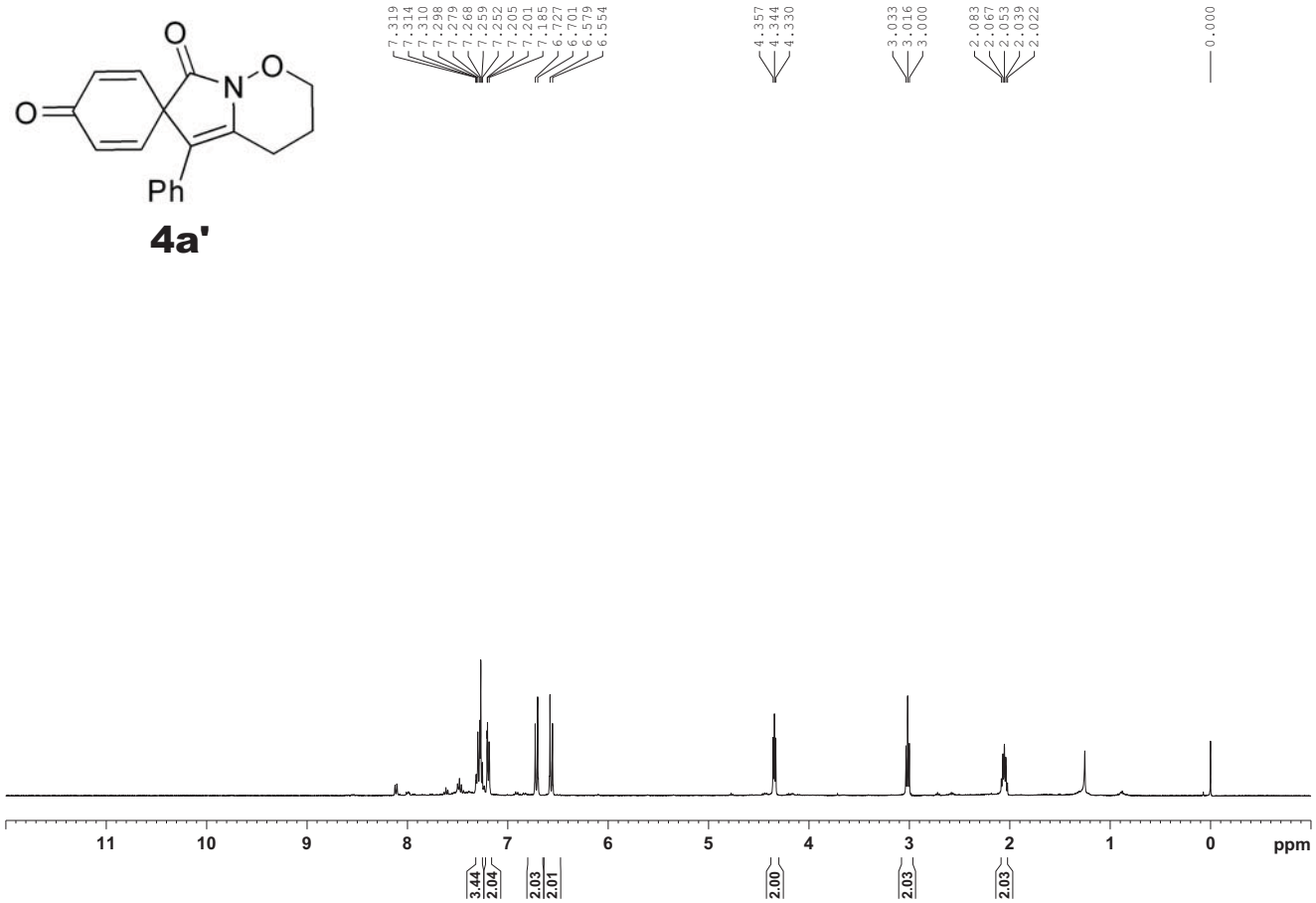

2ae
ppm

4a

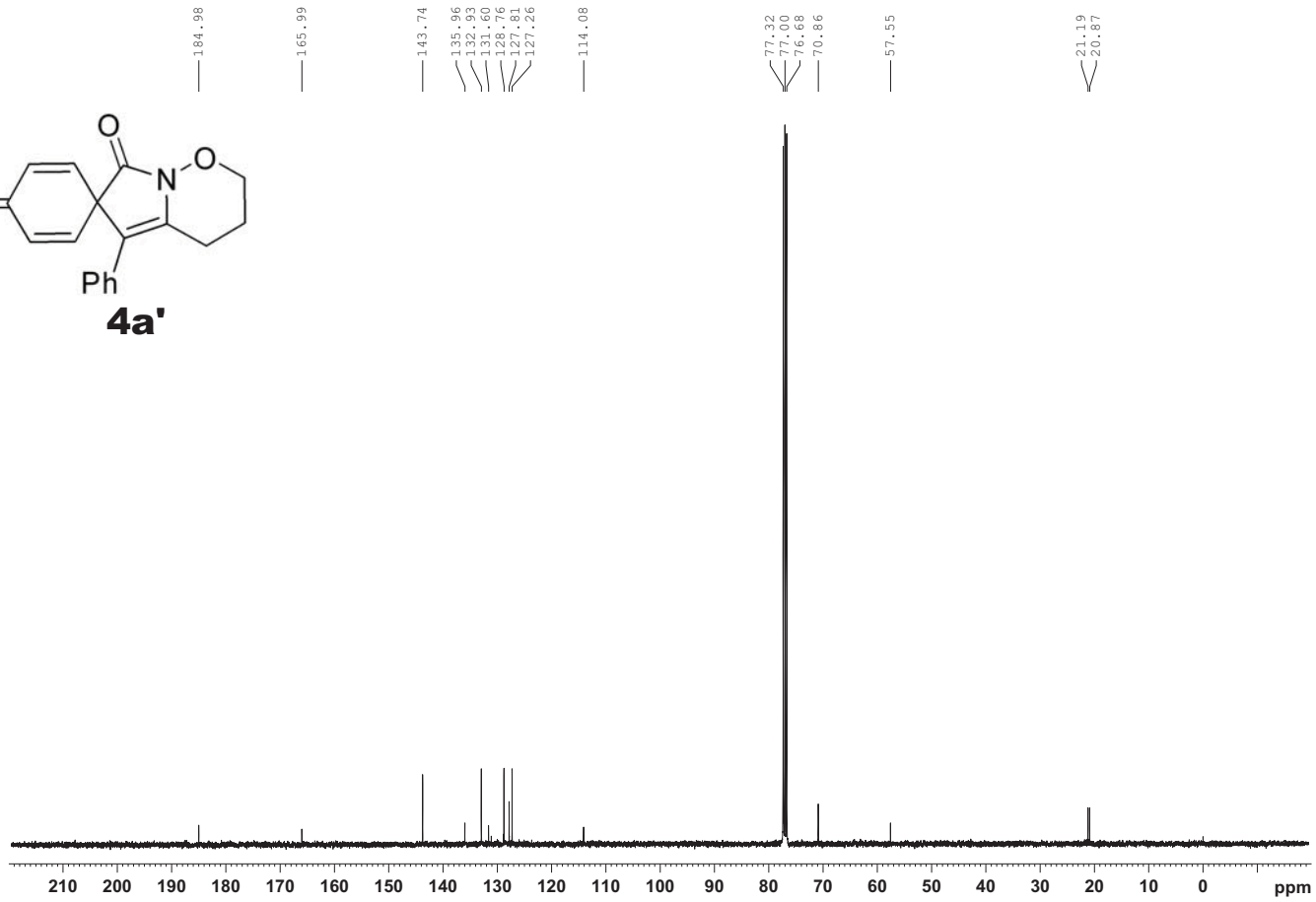

8.544
8.525
7.543
7.525
7.507
7.489
7.473
7.469
7.455
7.447
7.430
7.301
7.284
7.276
7.189
7.169
4.464
4.444
4.424
2.731
2.714
2.696
2.111
2.094
2.074
2.055
2.038
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
5.07
2.05
1.00
2.00
2.02
2.05

4a

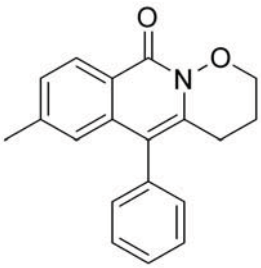

157.28
138.35
136.03
134.92
131.89
131.02
128.78
127.89
127.69
125.92
125.34
125.00
114.72
77.32
77.00
76.69
70.83
21.69
21.31
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.319
7.314
7.310
7.298
7.279
7.268
7.259
7.252
7.205
7.201
7.185
6.727
6.701
6.579
6.554
4.357
4.344
4.330
3.033
3.016
3.000
2.083
2.067
2.053
2.039
2.022
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
3.44
2.04
2.03
2.01
2.00
2.03
2.03

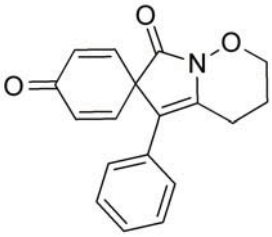
4a'


184.98
165.99
143.74
135.96
132.93
131.60
128.76
127.81
127.26
114.08
77.32
77.00
76.68
70.86
57.55
21.19
20.87
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

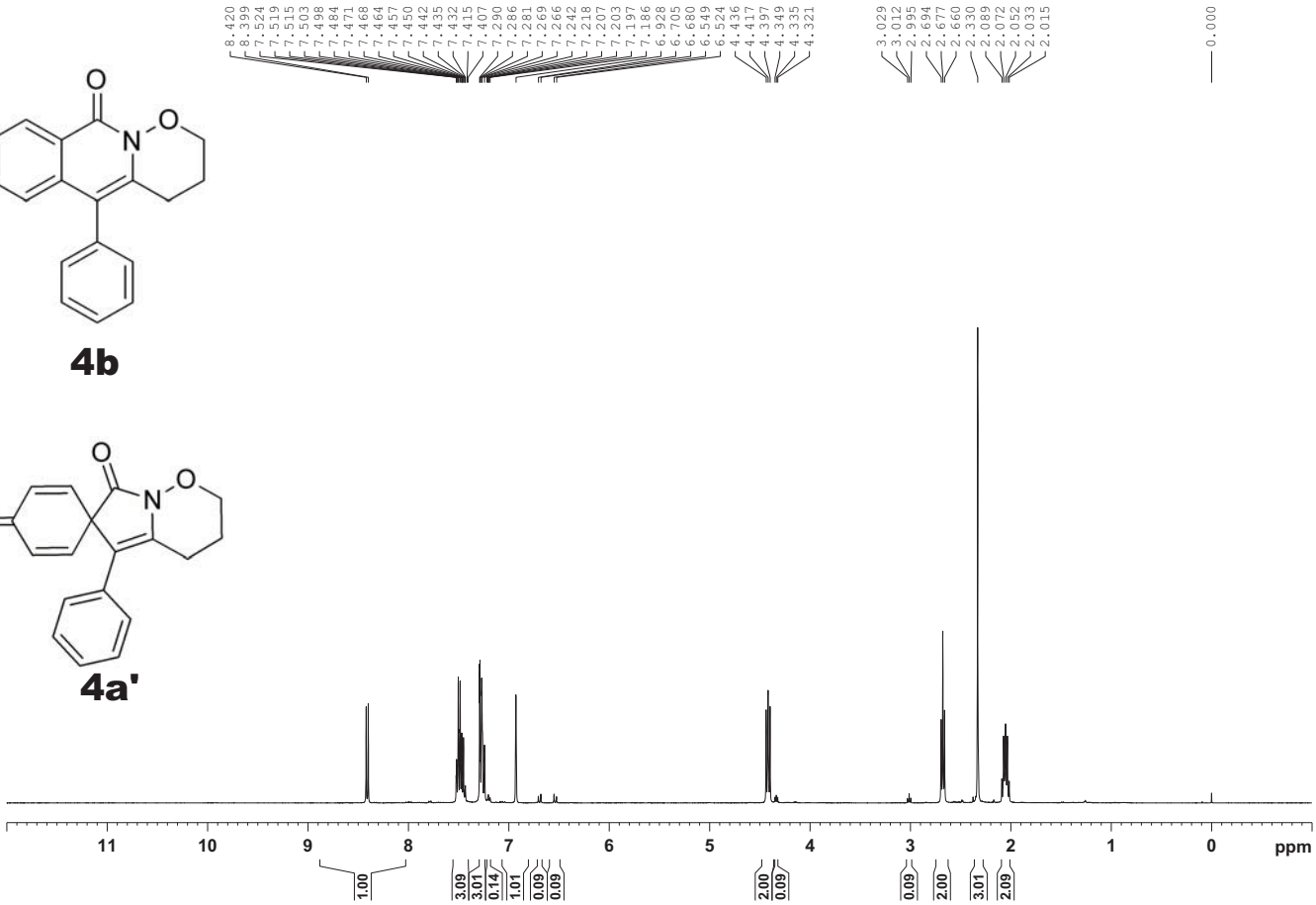
4a'

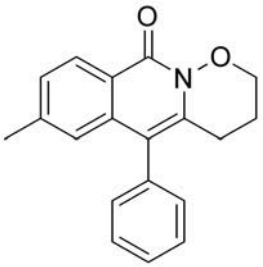


4b

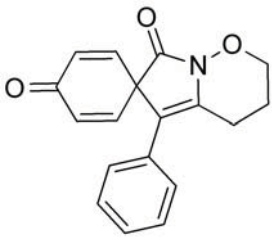


4a'

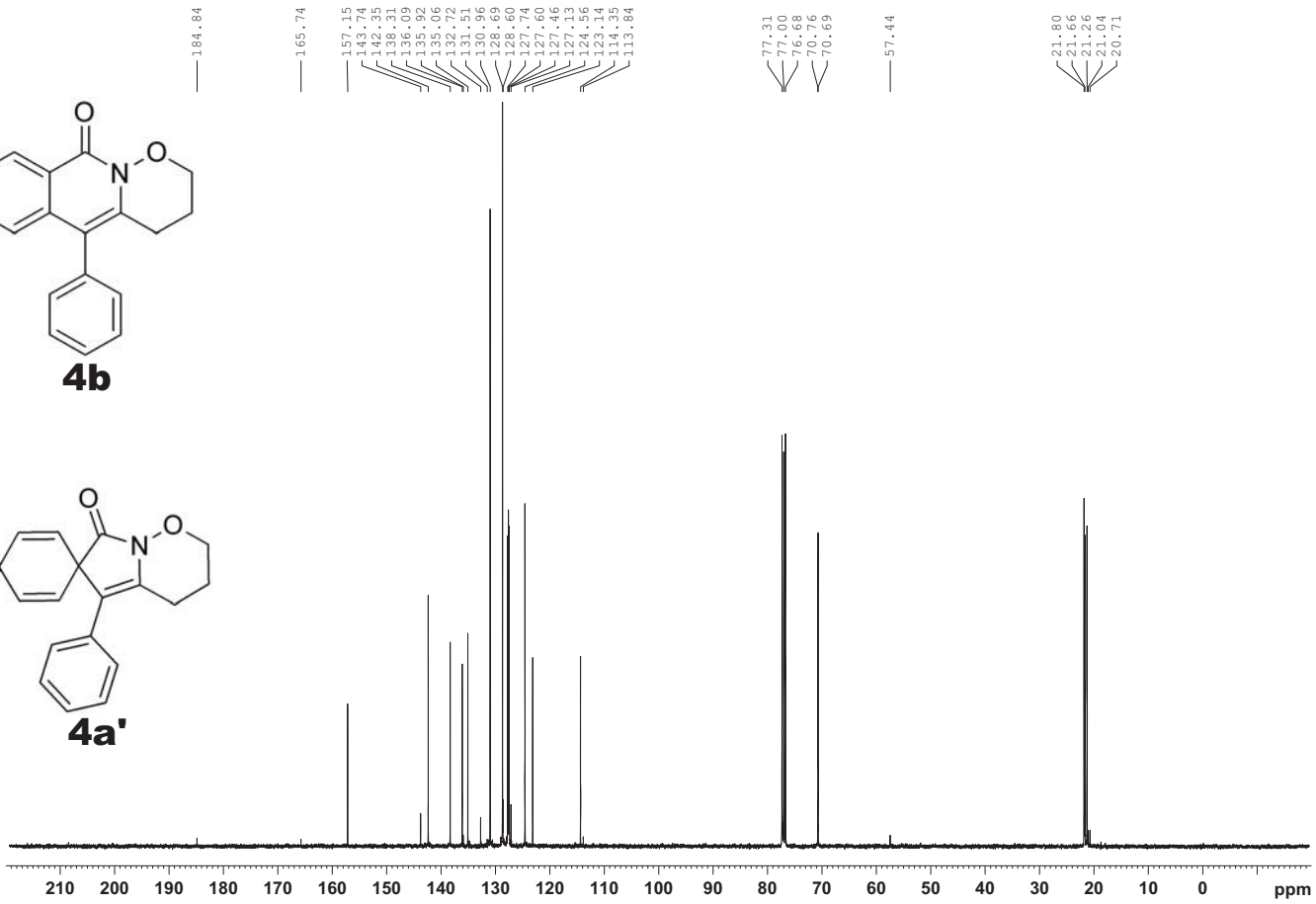

8.420
8.399
7.524
7.519
7.515
7.503
7.498
7.484
7.471
7.468
7.464
7.457
7.450
7.442
7.435
7.432
7.415
7.407
7.290
7.286
7.281
7.269
7.266
7.242
7.218
7.207
7.203
7.197
7.186
6.928
6.705
6.680
6.549
6.524
4.436
4.417
4.397
4.349
4.335
4.321
3.029
3.012
2.995
2.694
2.677
2.660
2.330
2.089
2.072
2.052
2.033
2.015
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
3.09
3.01
0.14
1.01
0.09
0.09
2.00
0.09
0.09
2.00
3.01
2.09



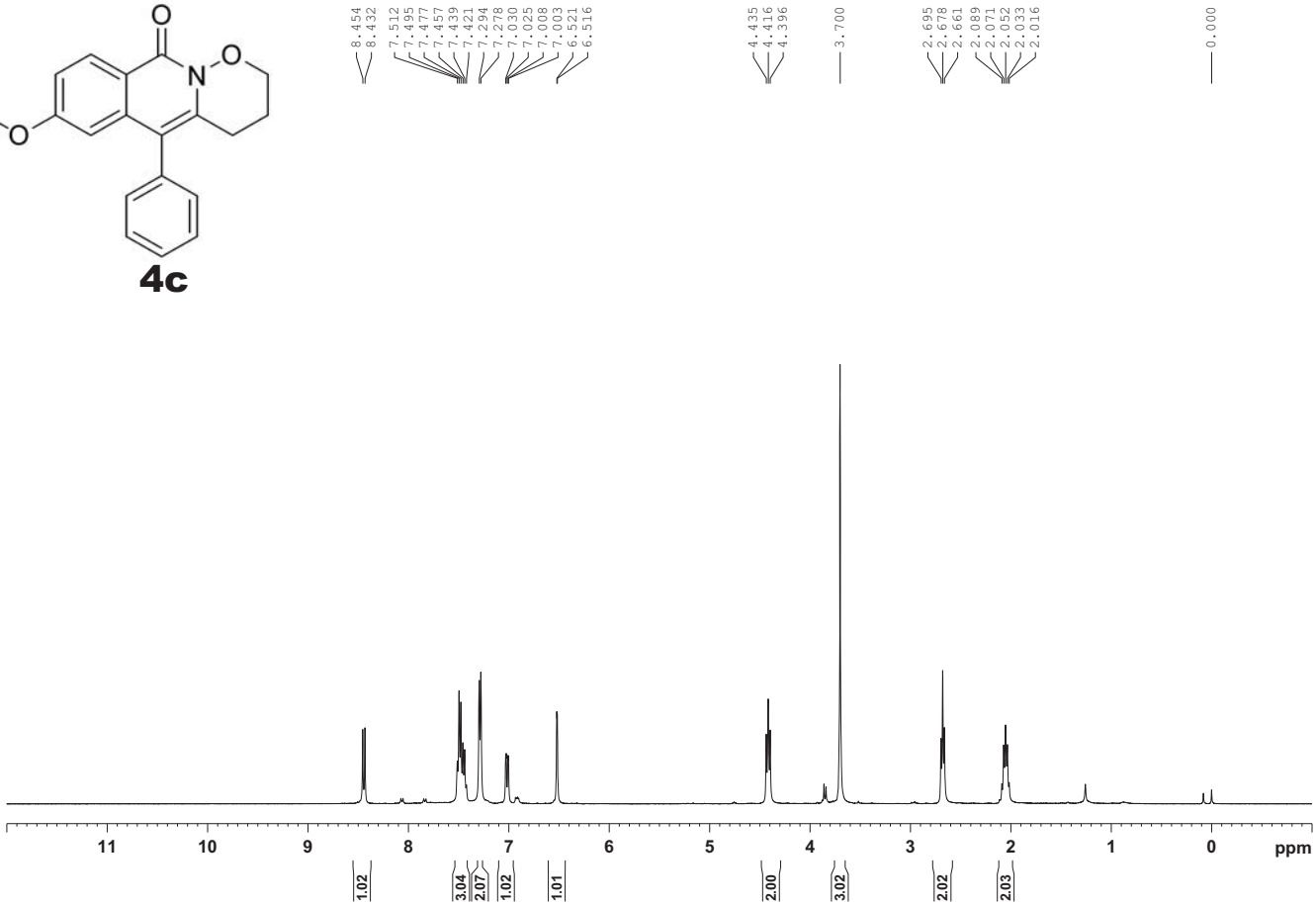
4b

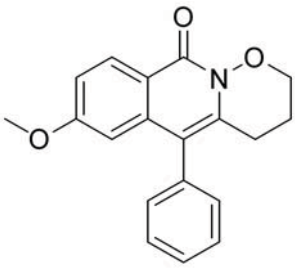


4a'

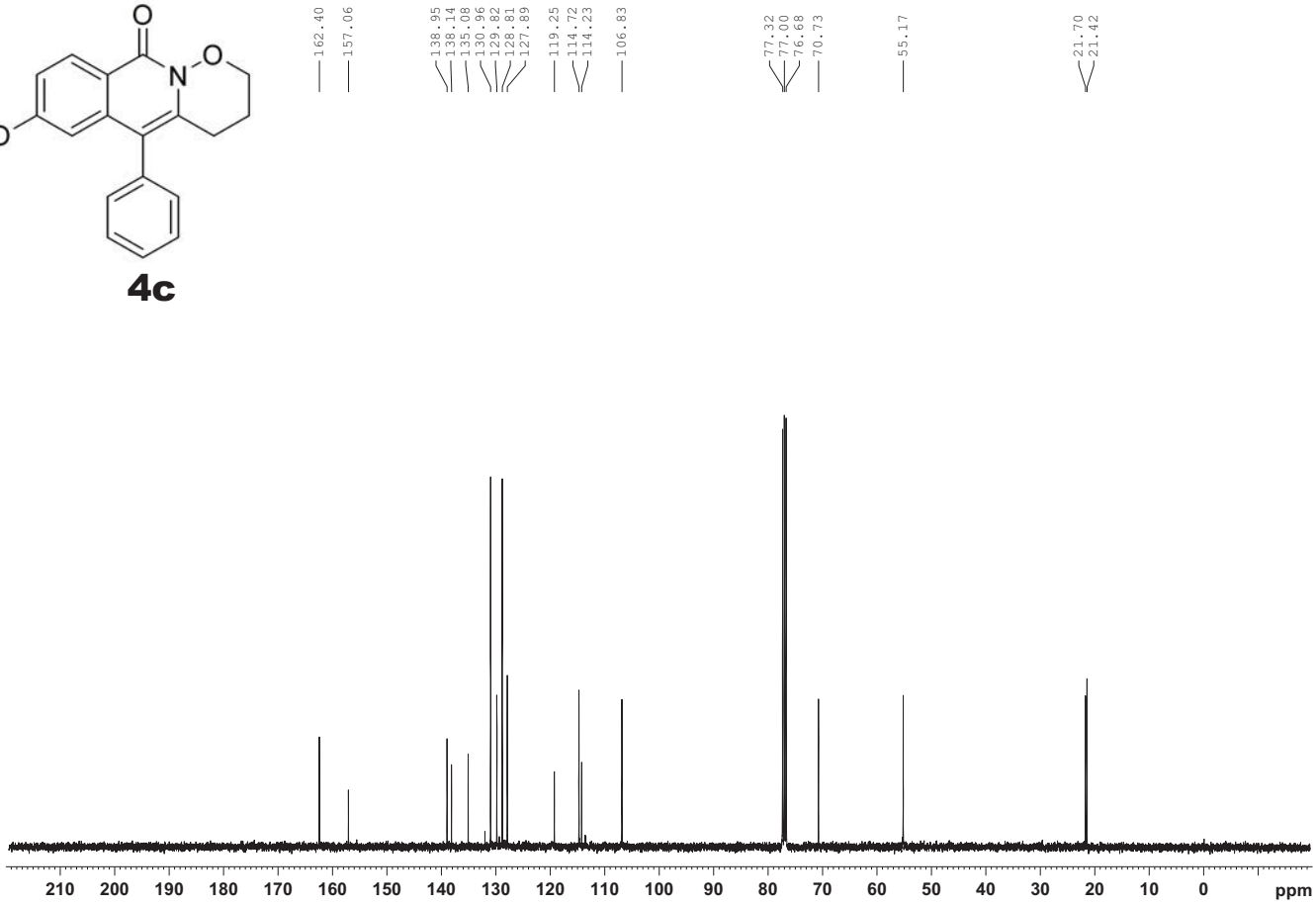

184.84
165.74
157.15
143.74
142.35
138.31
136.09
135.92
135.06
132.72
131.51
130.96
128.69
128.60
127.74
127.60
127.46
127.13
124.56
123.14
114.35
113.84
77.31
77.00
76.68
70.76
70.69
57.44
21.80
21.66
21.26
21.04
20.71
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

4c

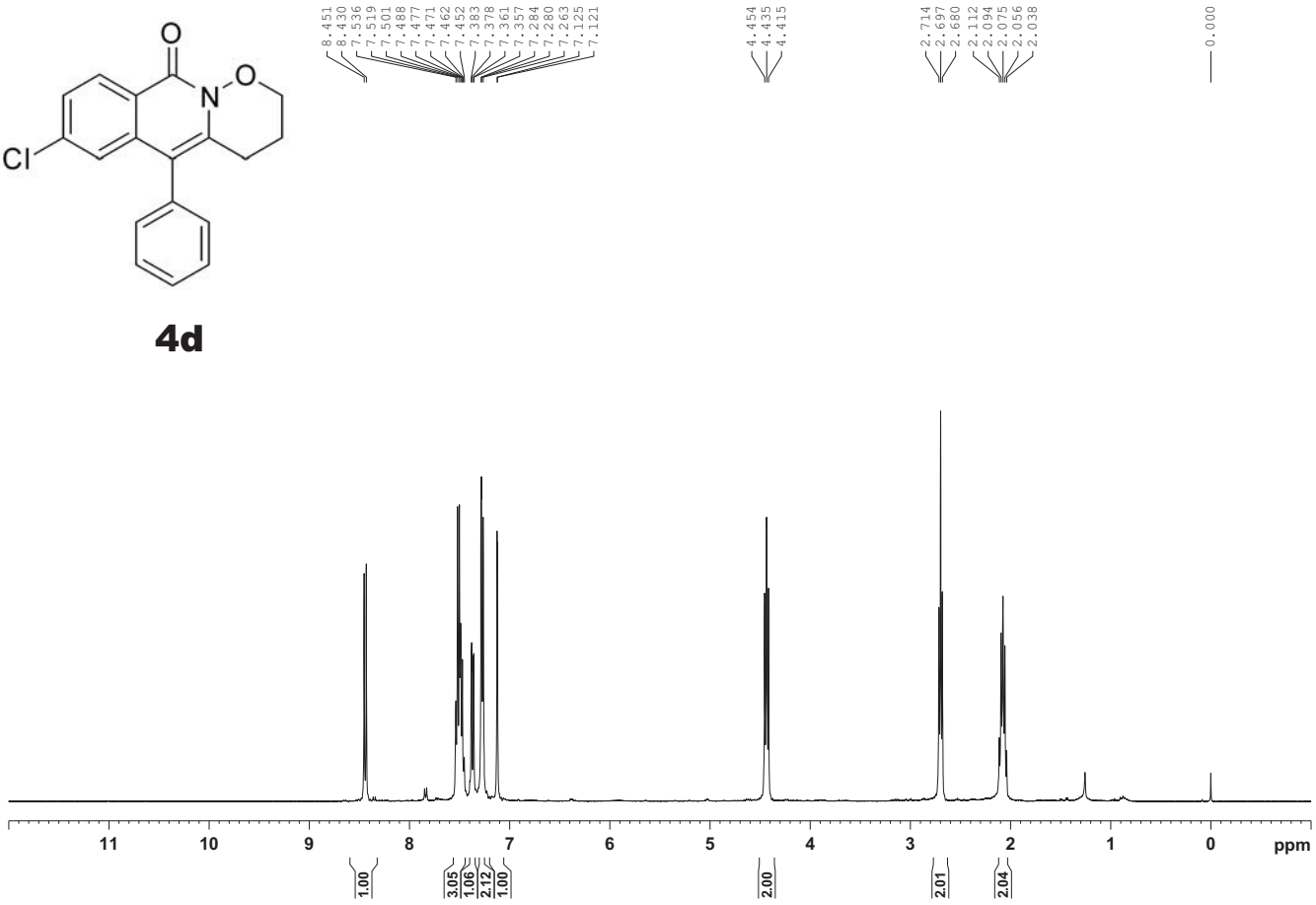

8.454
8.432
7.512
7.495
7.477
7.457
7.439
7.421
7.294
7.278
7.030
7.025
7.008
7.003
6.521
6.516
4.435
4.416
4.396
3.700
2.695
2.678
2.661
2.089
2.071
2.052
2.033
2.016
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.02
3.04
2.07
1.02
1.01
2.00
3.02
2.02
2.03



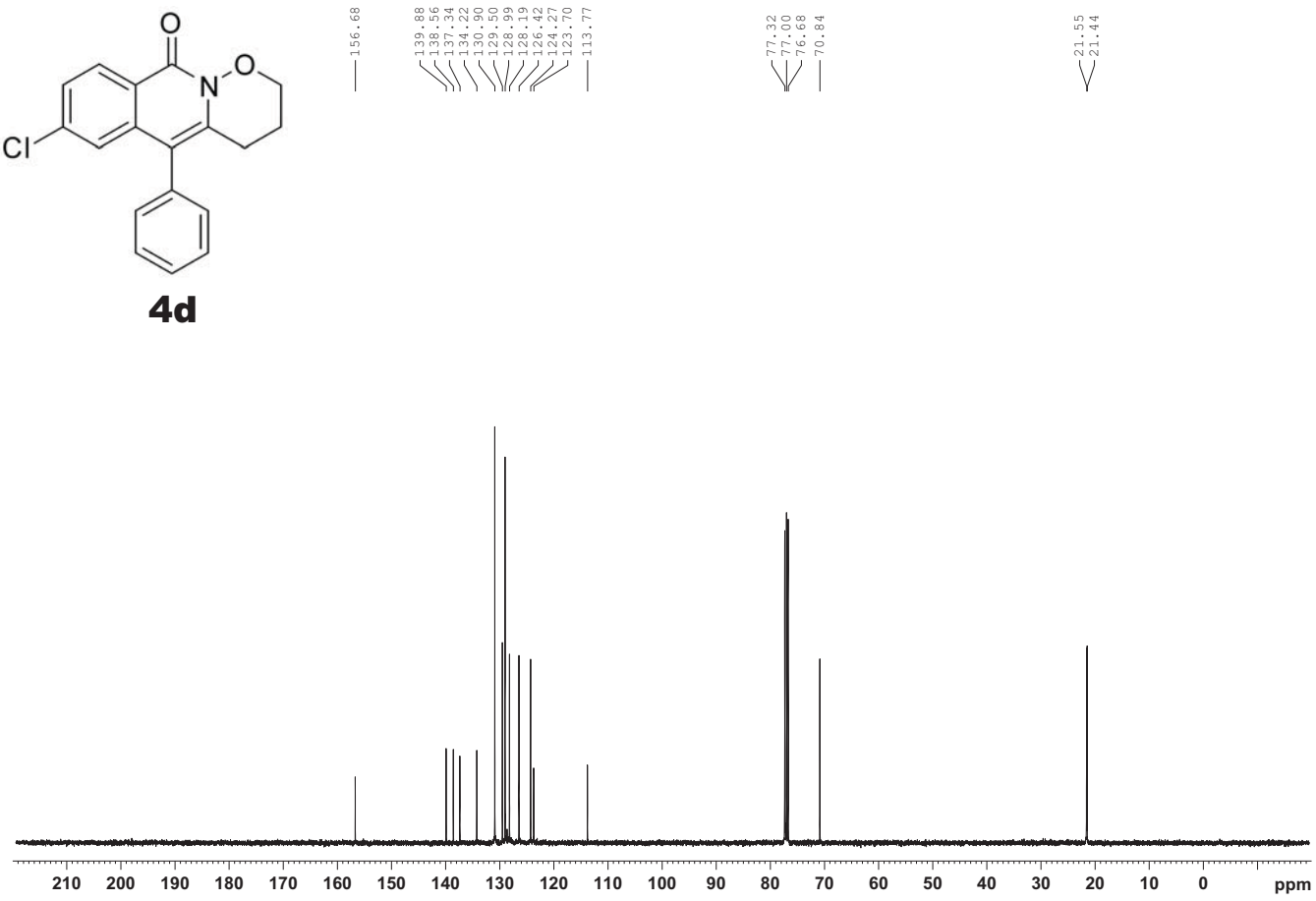
4c

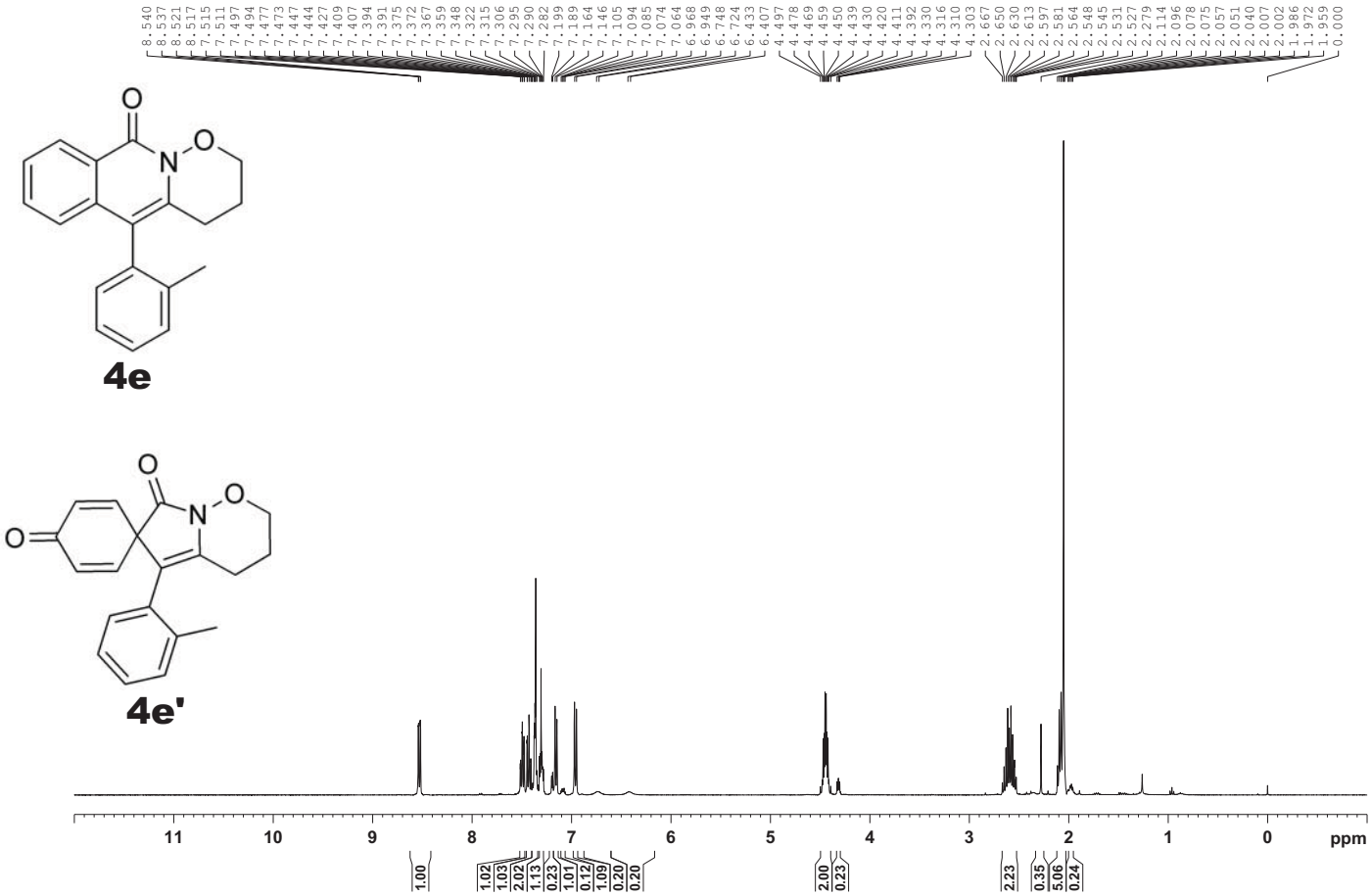

162.40
157.06
138.95
138.14
135.08
130.96
129.82
128.81
127.89
119.25
114.72
114.23
106.83
77.32
77.00
76.68
70.73
55.17
21.70
21.42
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

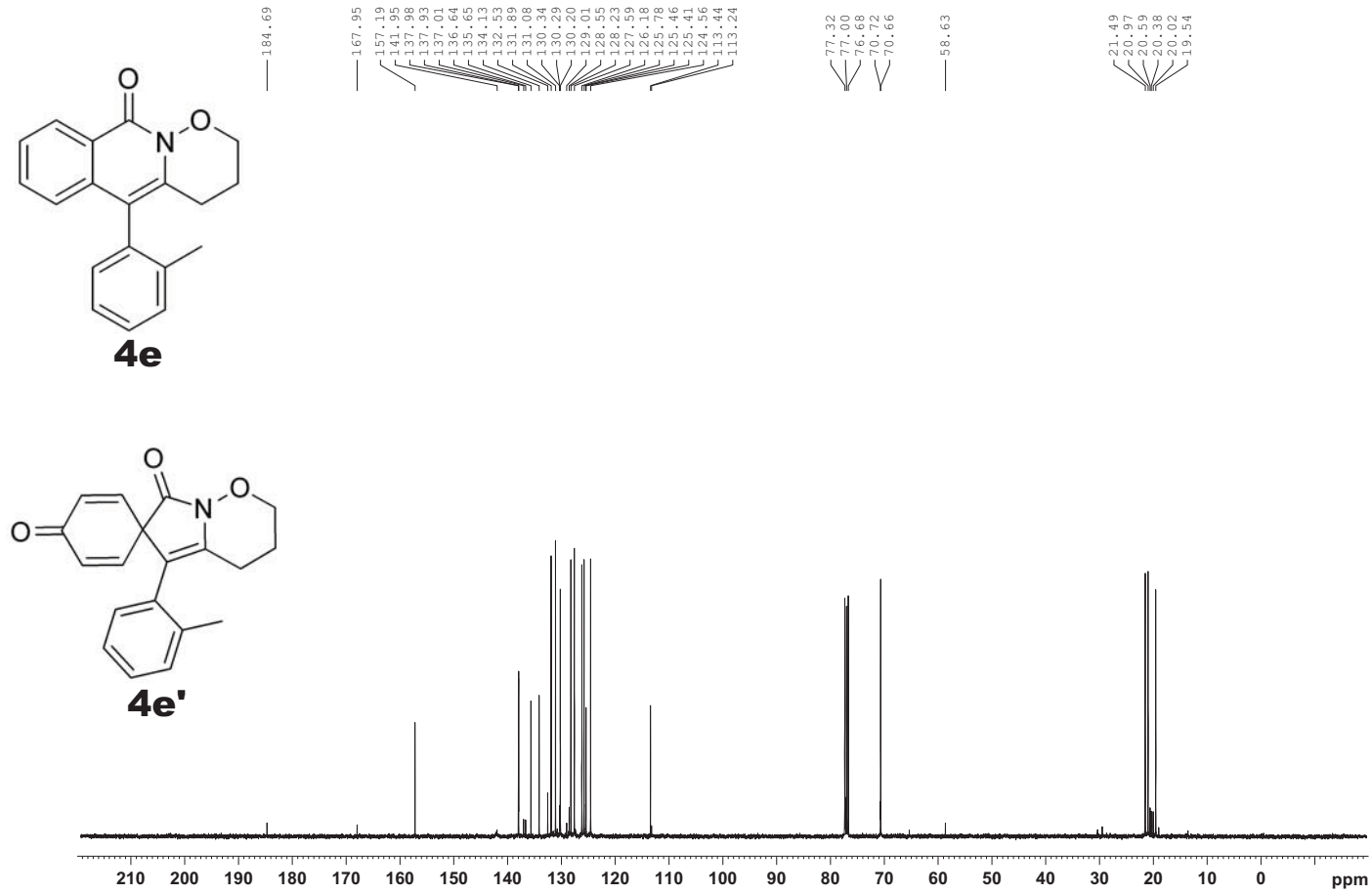
4d

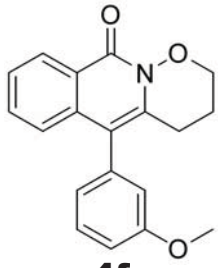

8.451
8.430
7.536
7.519
7.501
7.488
7.477
7.471
7.462
7.452
7.383
7.378
7.361
7.357
7.284
7.280
7.263
7.125
7.121
4.454
4.435
4.415
2.714
2.697
2.680
2.112
2.094
2.075
2.056
2.038
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
3.05
1.06
2.12
1.00
2.00
2.01
2.04

4d

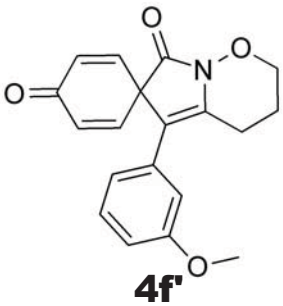

156.68
139.88
138.56
137.34
134.22
130.90
129.50
128.99
128.19
126.42
124.27
123.70
113.77
77.32
77.00
76.68
70.84
21.55
21.44
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


4e
4e'
ppm

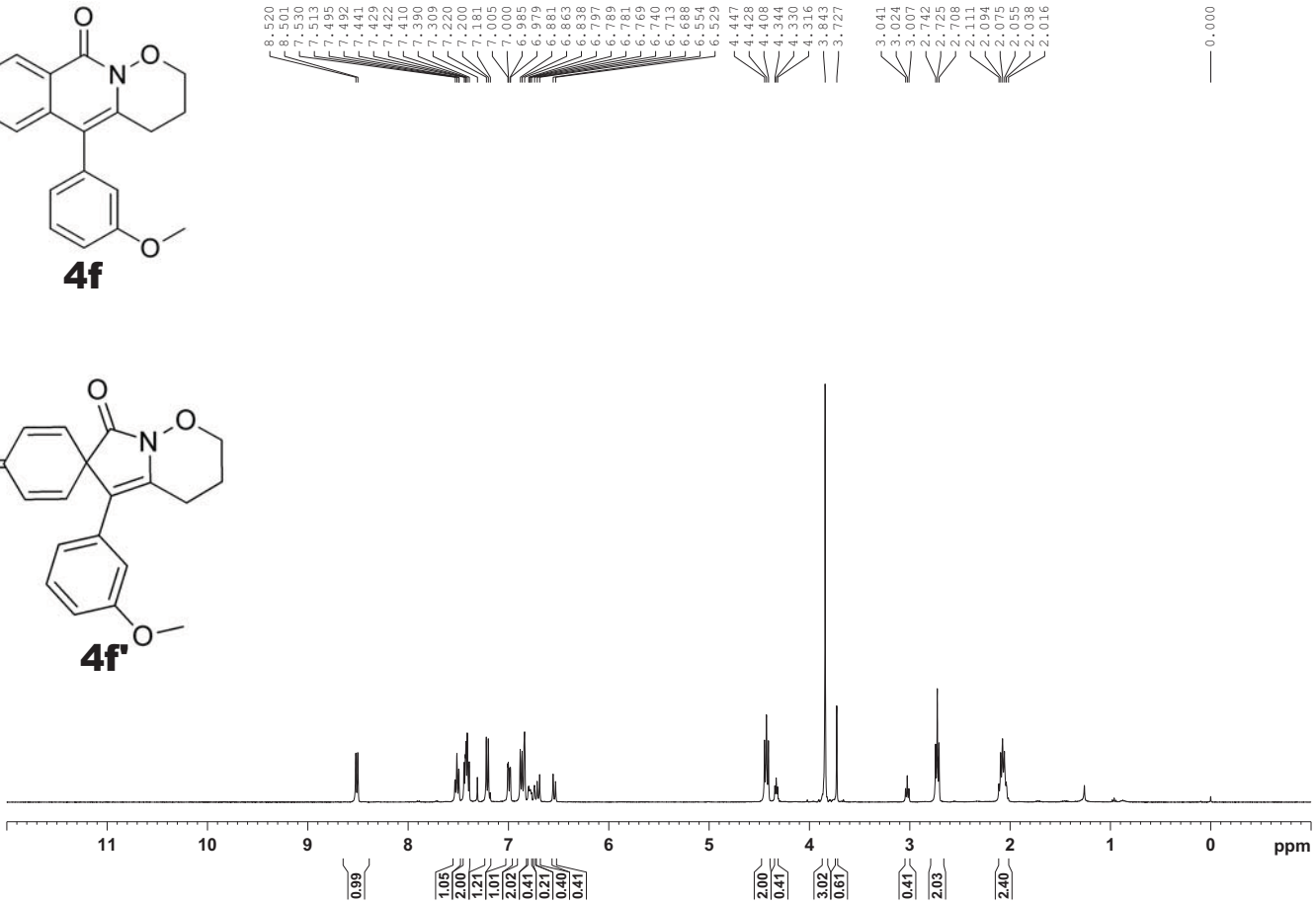

4e
4e'
ppm

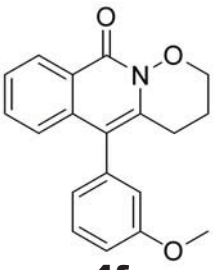


4f

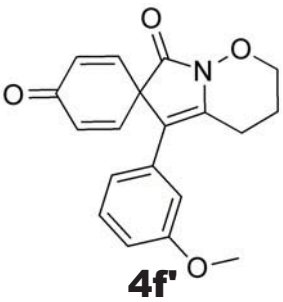


4f'

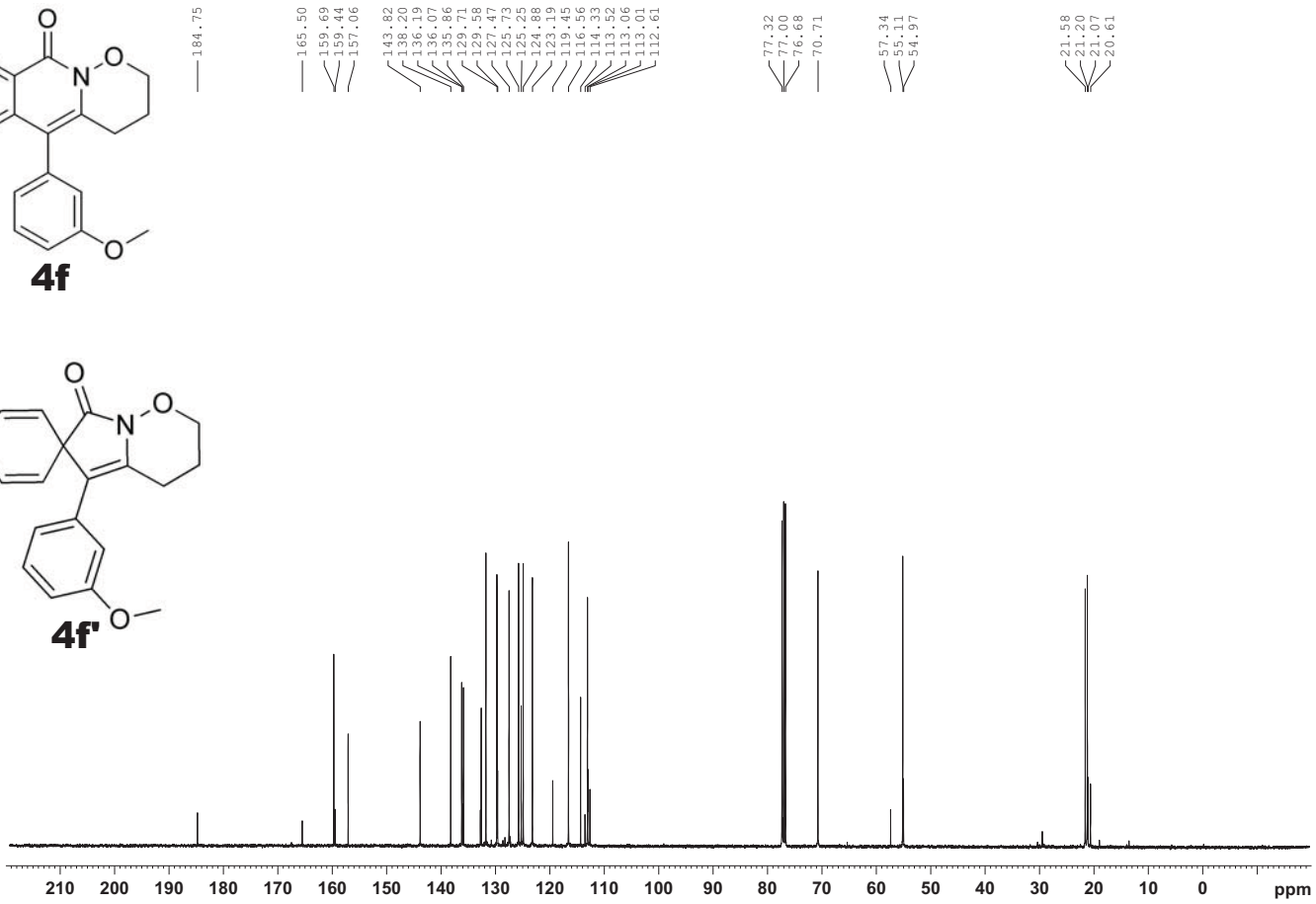

8.520
8.501
7.530
7.513
7.495
7.492
7.441
7.429
7.422
7.410
7.390
7.309
7.220
7.200
7.181
7.005
7.000
6.985
6.979
6.881
6.863
6.838
6.797
6.789
6.781
6.769
6.740
6.713
6.688
6.554
6.529
4.447
4.428
4.408
4.344
4.330
4.316
3.843
3.727
3.041
3.024
3.007
2.742
2.725
2.708
2.111
2.094
2.075
2.055
2.038
2.016
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.99
1.05
2.00
1.21
1.01
2.02
0.41
0.21
0.40
0.41
2.00
0.41
3.02
0.61
0.41
2.03
2.40

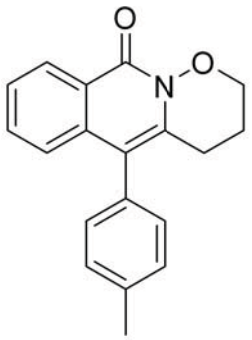


4f

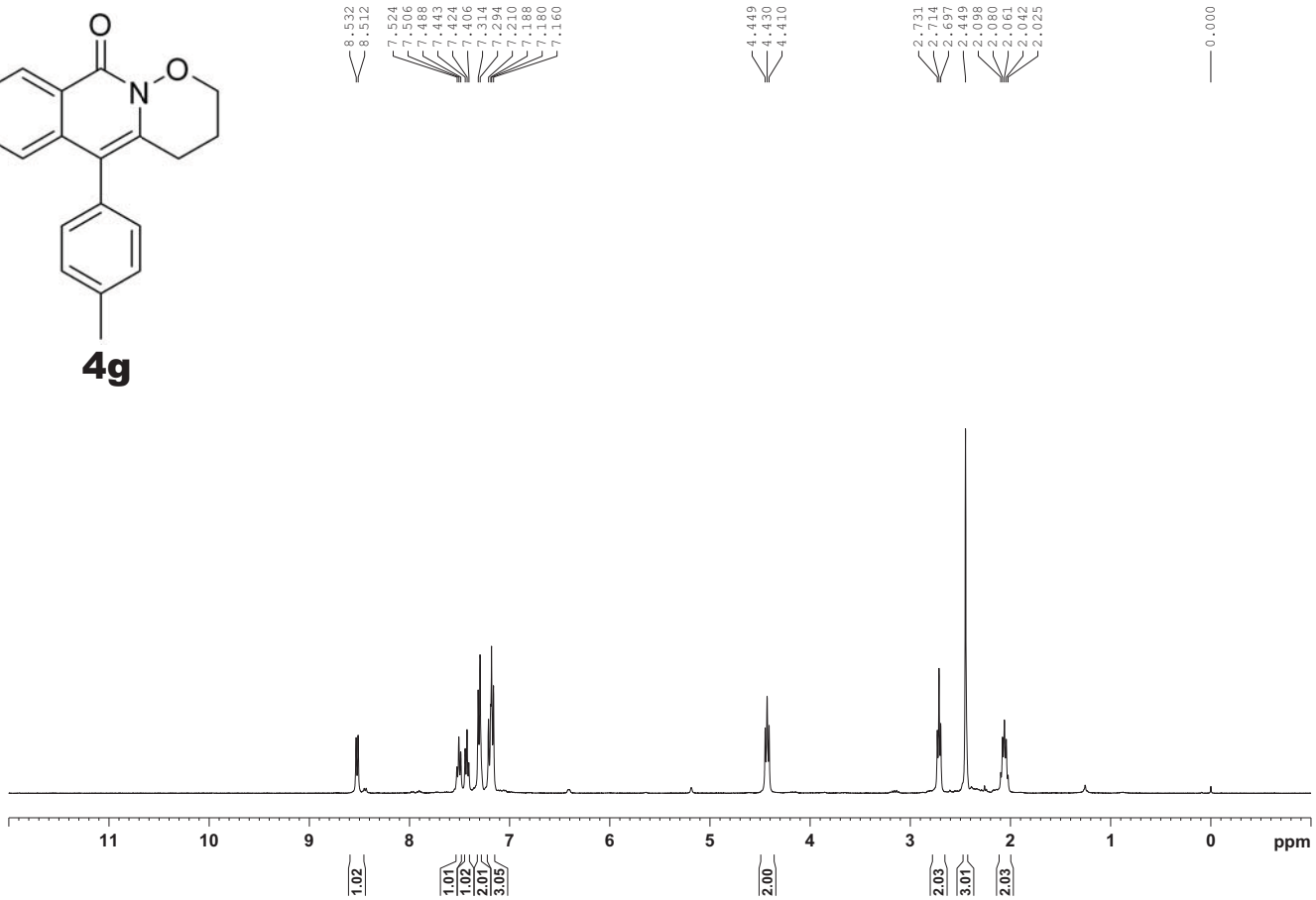


4f'

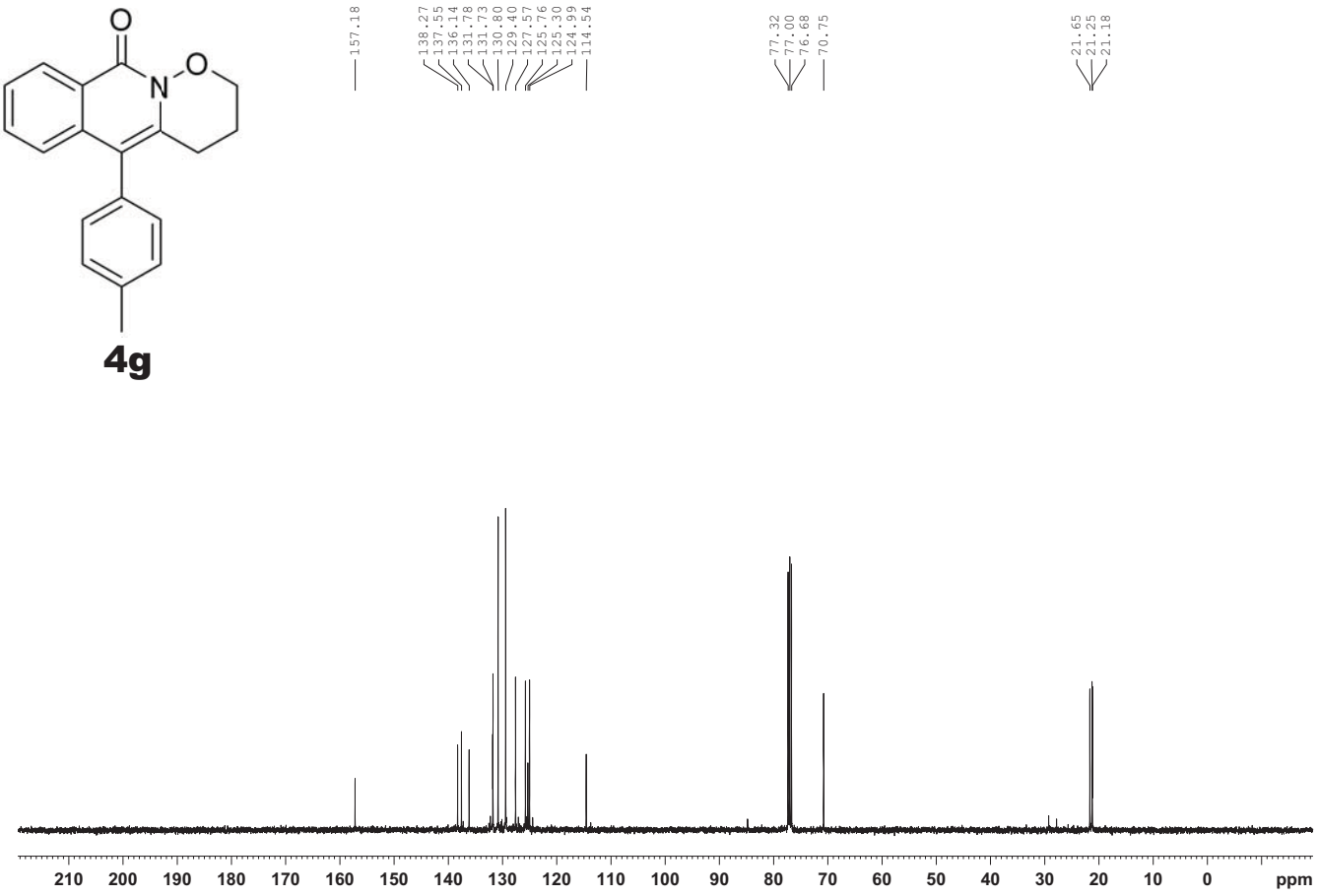

184.75
165.50
159.69
159.44
157.06
143.82
138.20
136.19
136.07
135.86
129.71
129.58
127.47
125.73
125.25
124.88
123.19
119.45
116.56
114.33
113.52
113.06
113.01
112.61
77.32
77.00
76.68
70.71
57.34
55.11
54.97
21.58
21.20
21.07
20.61
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

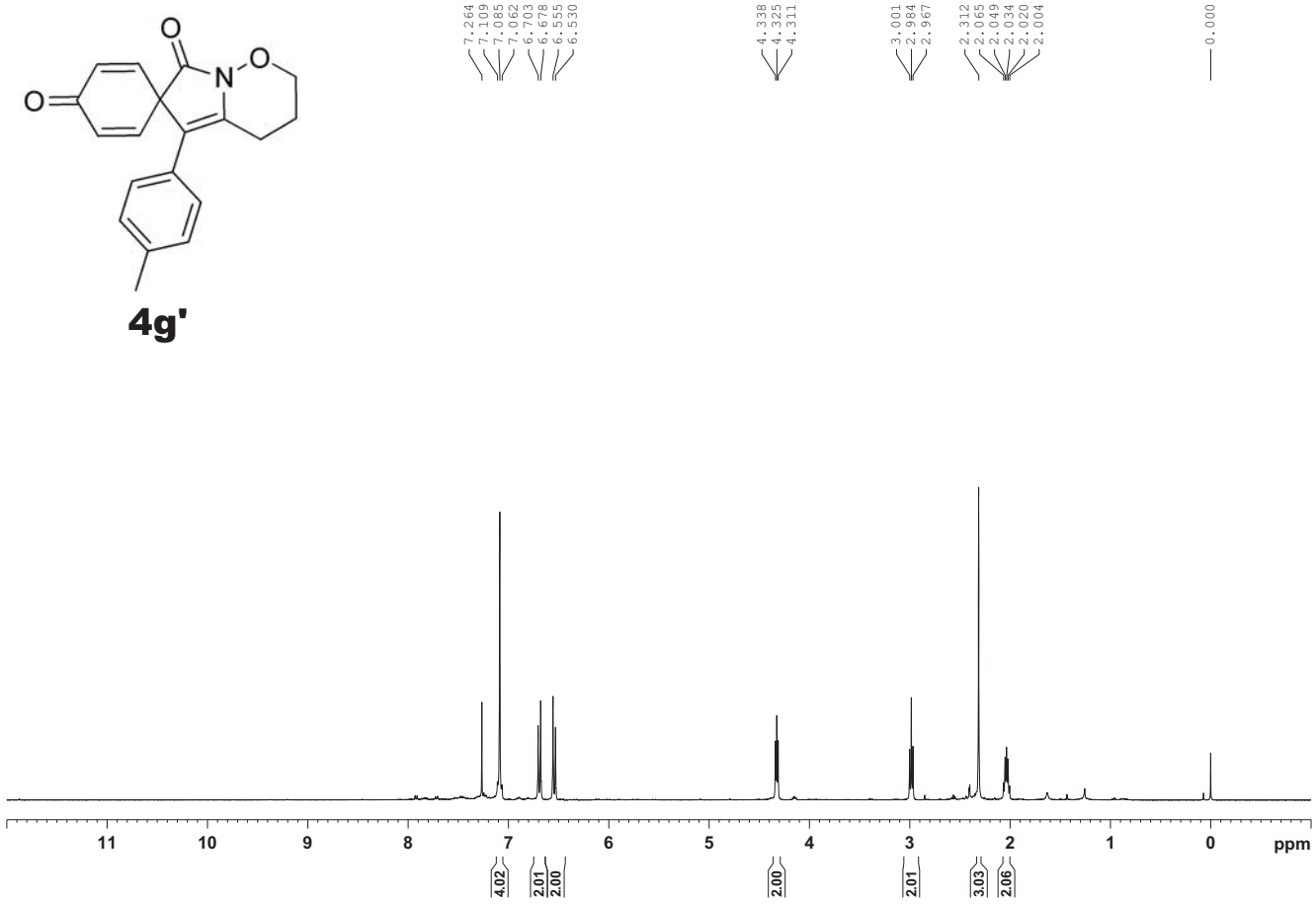


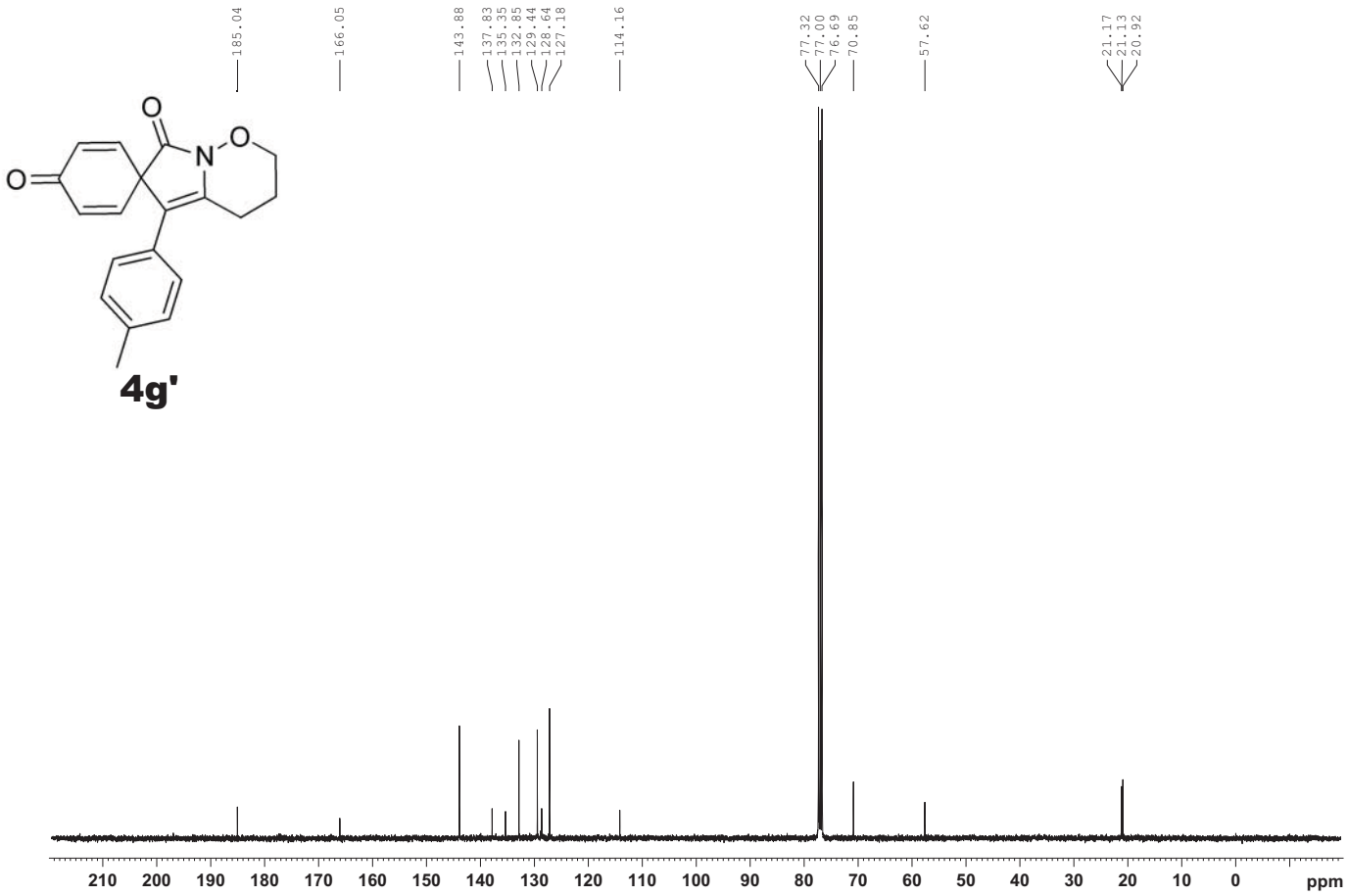
4g

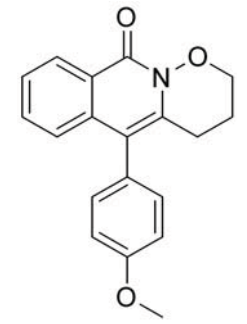

8.532
8.512
7.524
7.506
7.488
7.443
7.424
7.406
7.314
7.294
7.210
7.188
7.180
7.160
4.449
4.430
4.410
2.731
2.714
2.697
2.449
2.098
2.080
2.061
2.042
2.025
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.02
1.01
1.02
2.01
3.05
2.00
2.03
3.01
2.03

4g

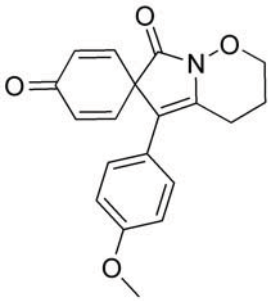

157.18
138.27
137.55
136.14
131.78
131.73
130.80
129.40
127.57
125.76
125.30
124.99
114.54
77.32
77.00
76.68
70.75
21.65
21.25
21.18
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


4g'
7.264
7.109
7.085
7.062
6.703
6.678
6.555
6.530
4.338
4.325
4.311
3.001
2.984
2.967
2.312
2.065
2.049
2.034
2.020
2.004
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
4.02
2.01
2.00
2.00
2.01
3.03
2.06

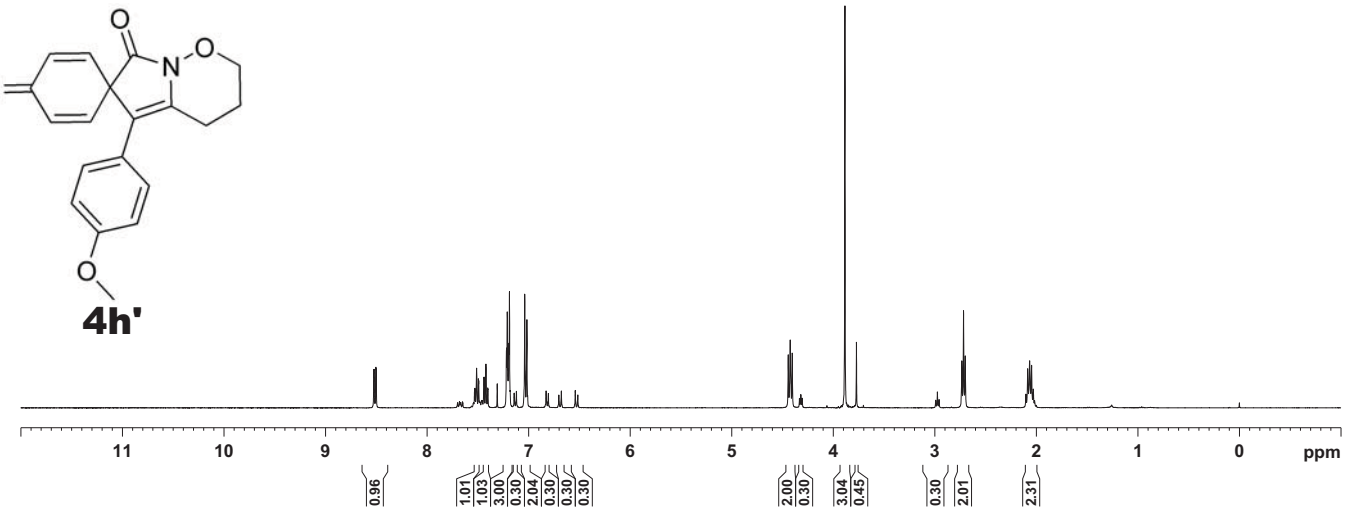

4g'
185.04
166.05
143.88
137.83
135.35
132.85
129.44
128.64
127.18
114.16
77.32
77.00
76.69
70.85
57.62
21.17
21.13
20.92
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

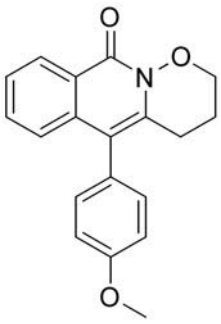


4h

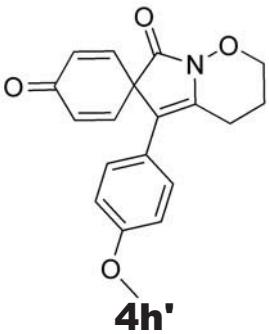


4h'

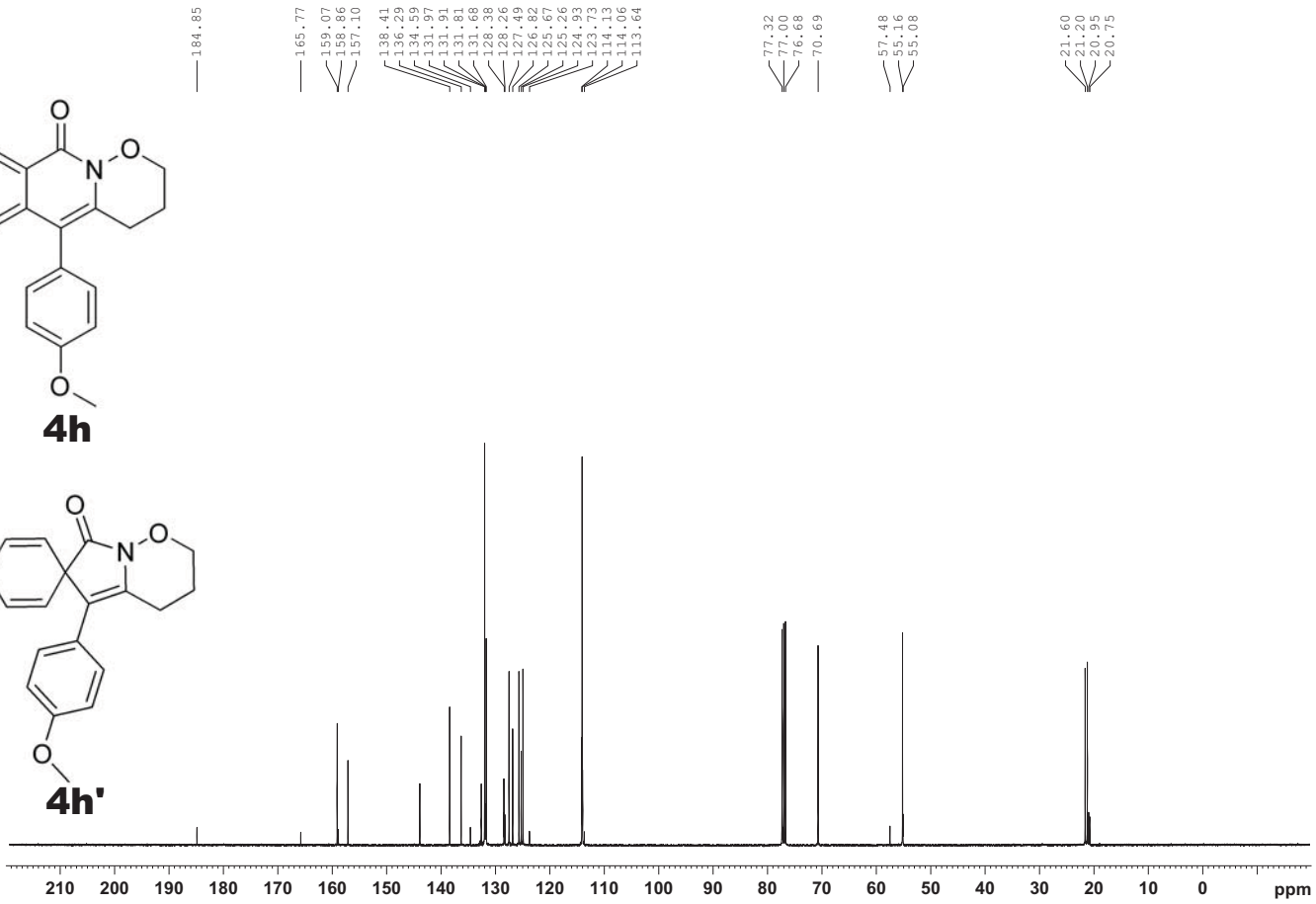

8.523
8.521
8.503
8.501
7.532
7.528
7.511
7.494
7.490
7.439
7.437
7.420
7.402
7.400
7.308
7.217
7.210
7.205
7.194
7.188
7.141
7.119
7.038
7.016
6.827
6.805
6.702
6.677
6.540
6.515
4.442
4.423
4.403
4.332
4.318
4.305
3.884
3.771
2.990
2.974
2.957
2.734
2.716
2.699
2.102
2.085
2.065
2.046
2.029
2.017
2.001
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.96
1.01
1.03
3.00
0.30
2.04
0.30
0.30
0.30
2.00
0.30
3.04
0.45
0.30
2.01
2.31



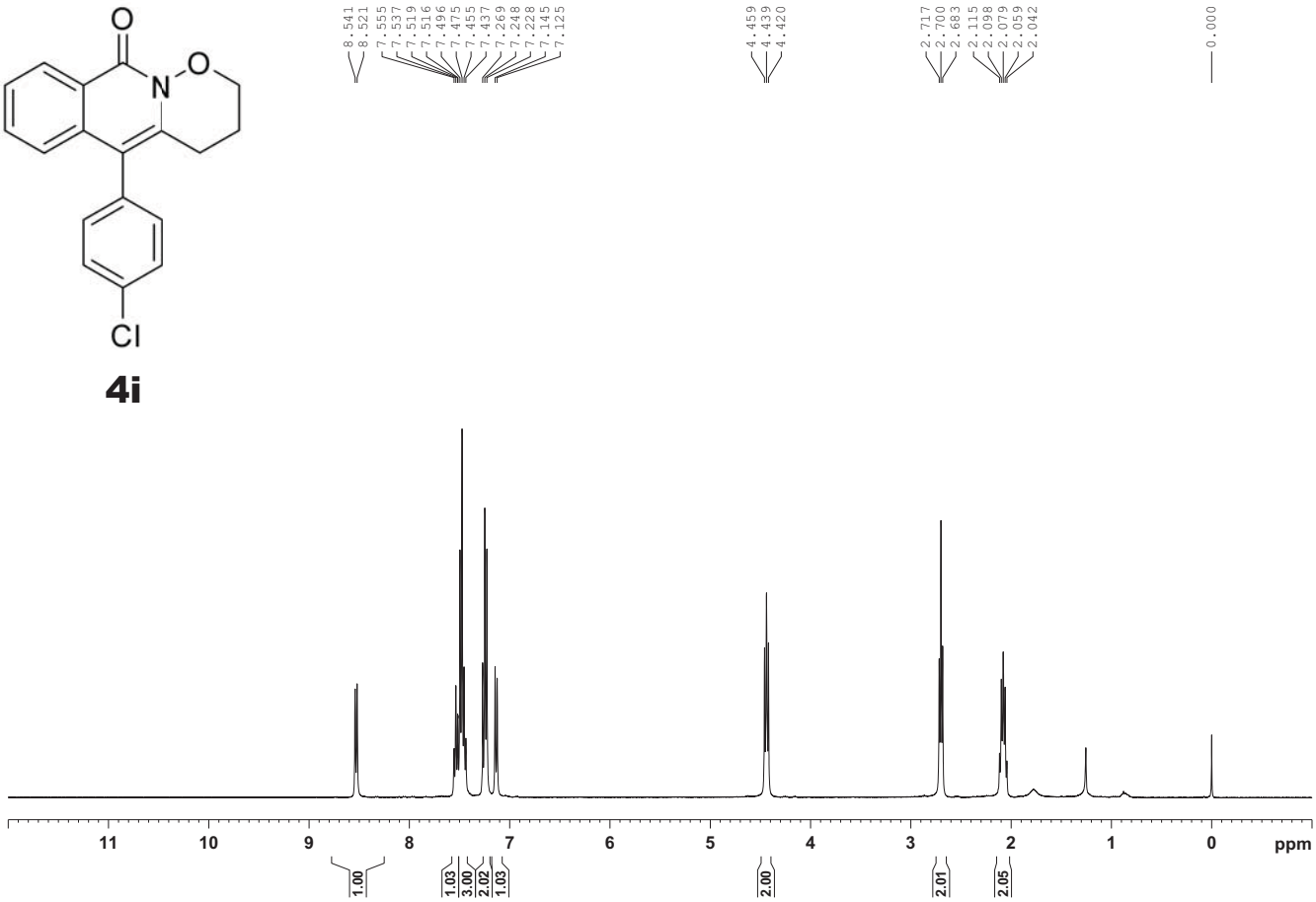
4h



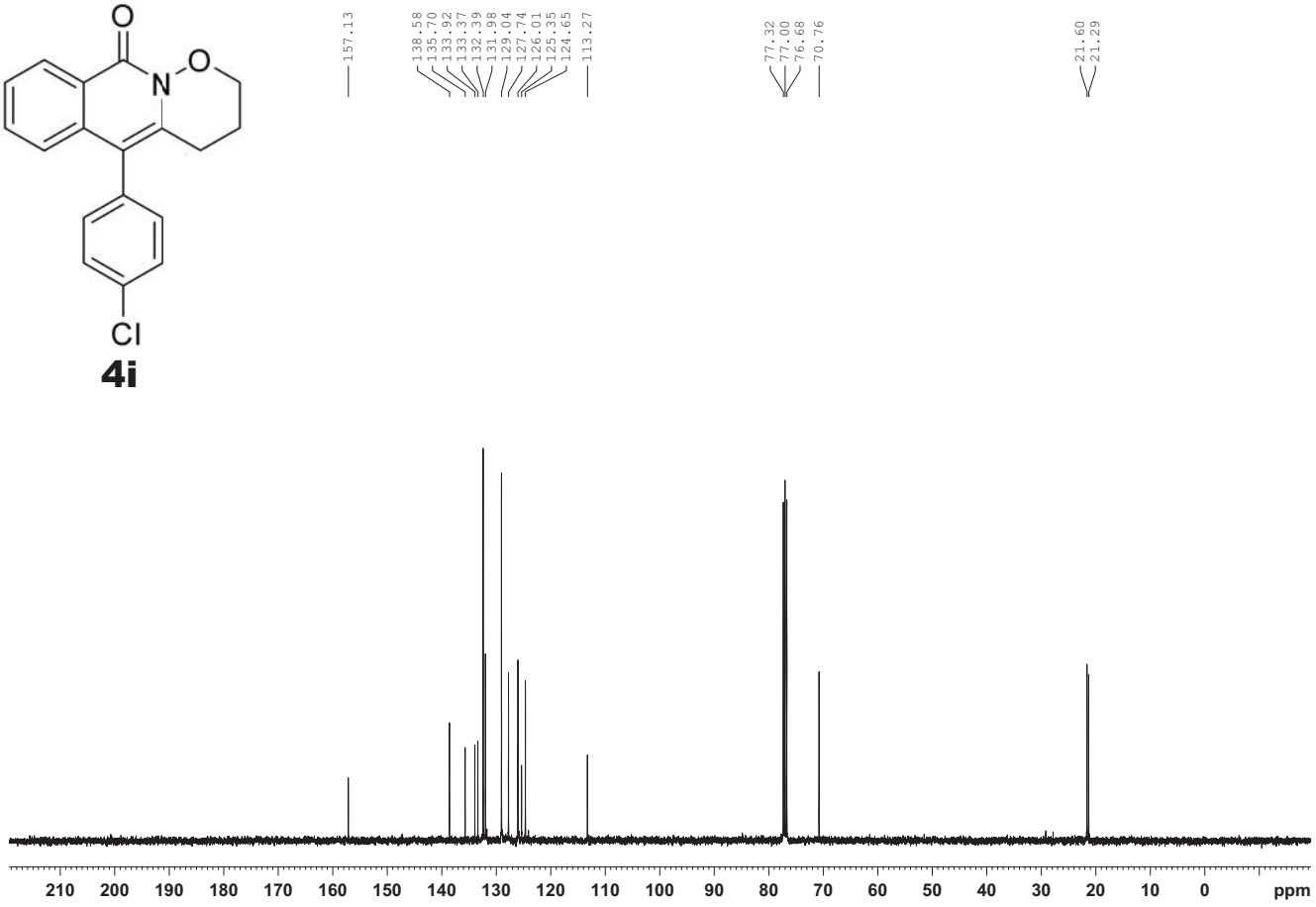
4h'

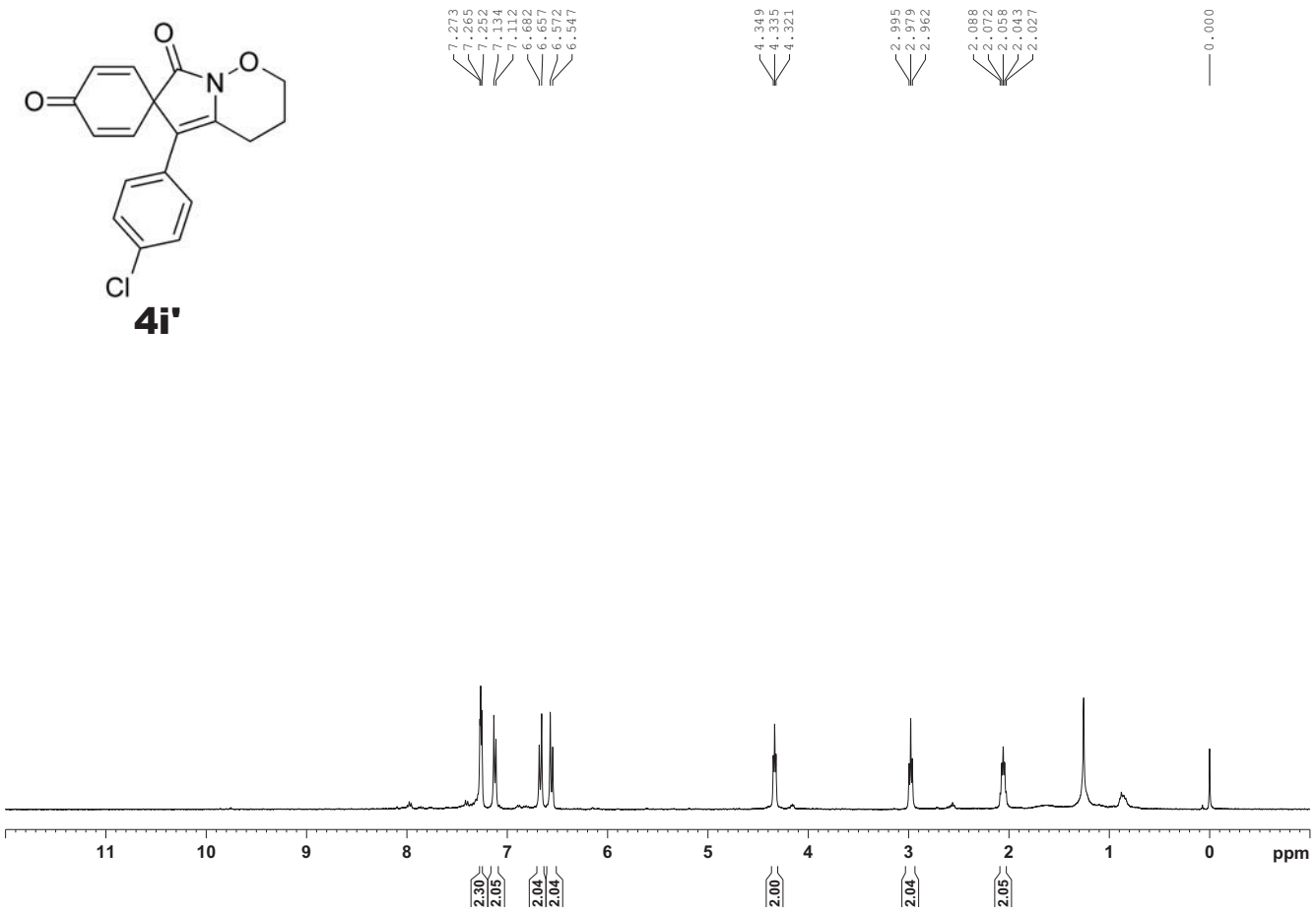

184.85
165.77
159.07
158.86
157.10
138.41
136.29
134.59
131.97
131.91
131.81
131.68
128.38
128.26
127.49
126.82
125.67
125.26
124.93
123.73
114.13
114.06
113.64
77.32
77.00
76.68
70.69
57.48
55.16
55.08
21.60
21.20
20.95
20.75
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

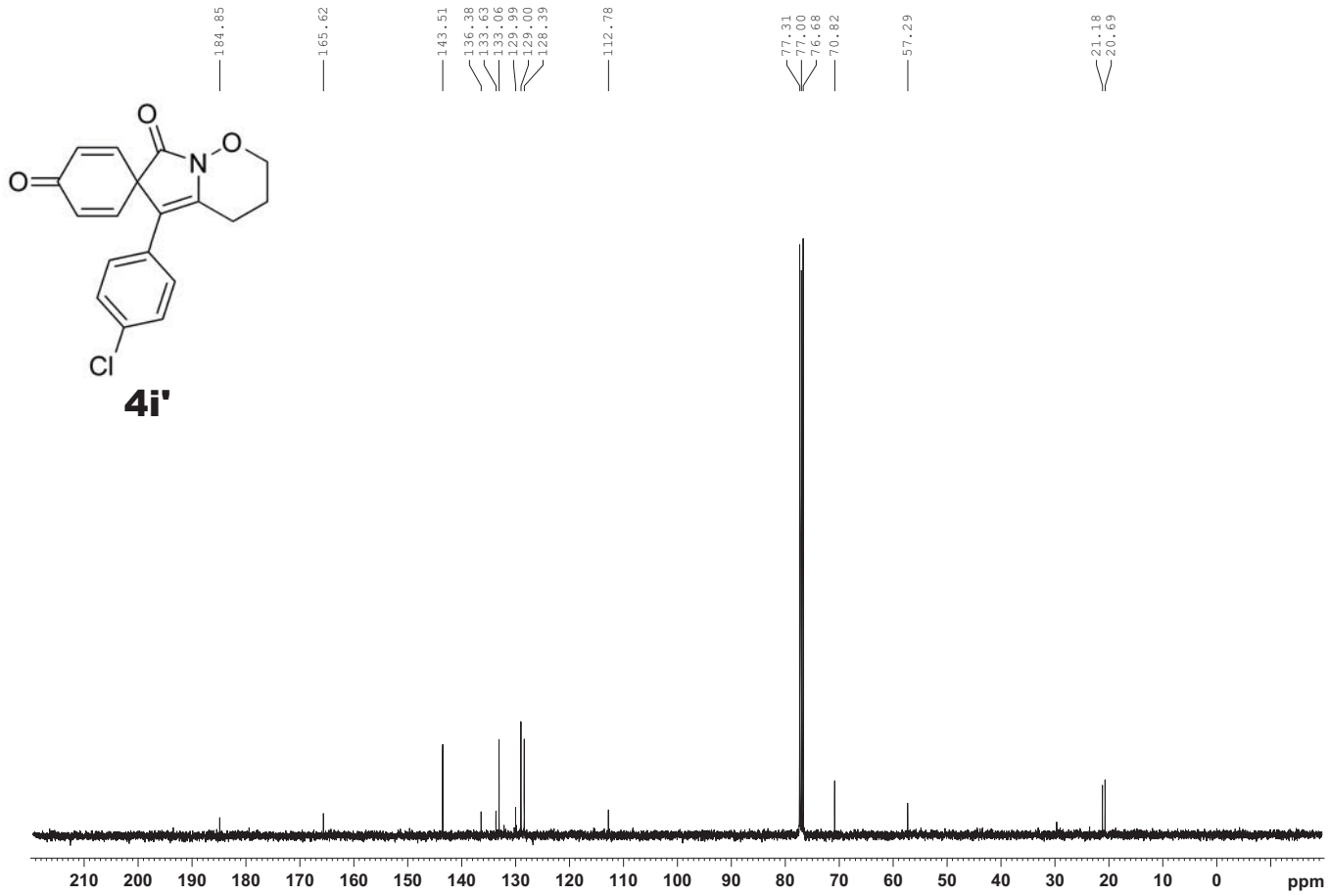
4i

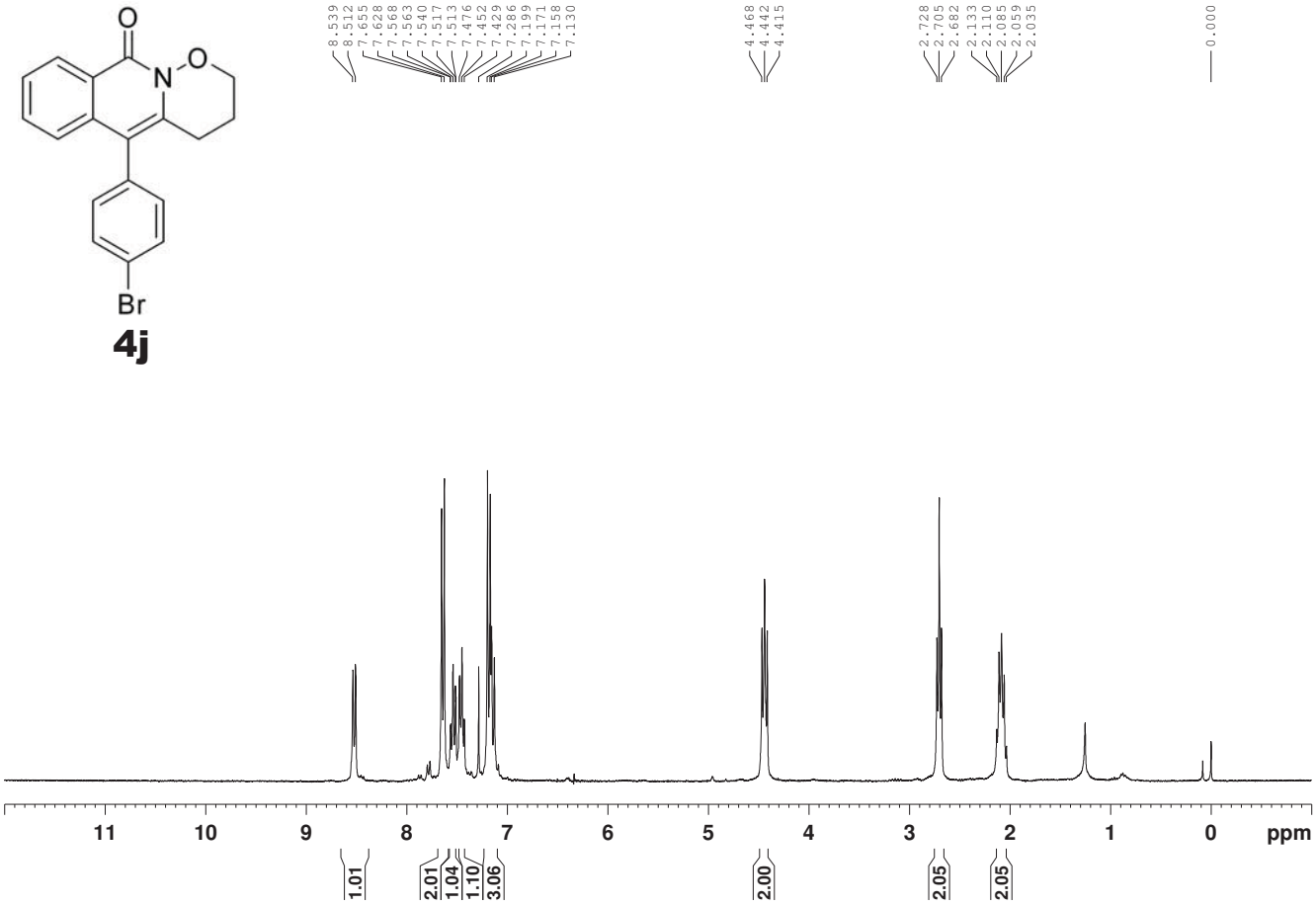

8.541
8.521
7.555
7.537
7.519
7.516
7.496
7.475
7.455
7.437
7.269
7.248
7.228
7.145
7.125
4.459
4.439
4.420
2.717
2.700
2.683
2.115
2.098
2.079
2.059
2.042
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.03
3.00
2.02
1.03
2.00
2.01
2.05

4i

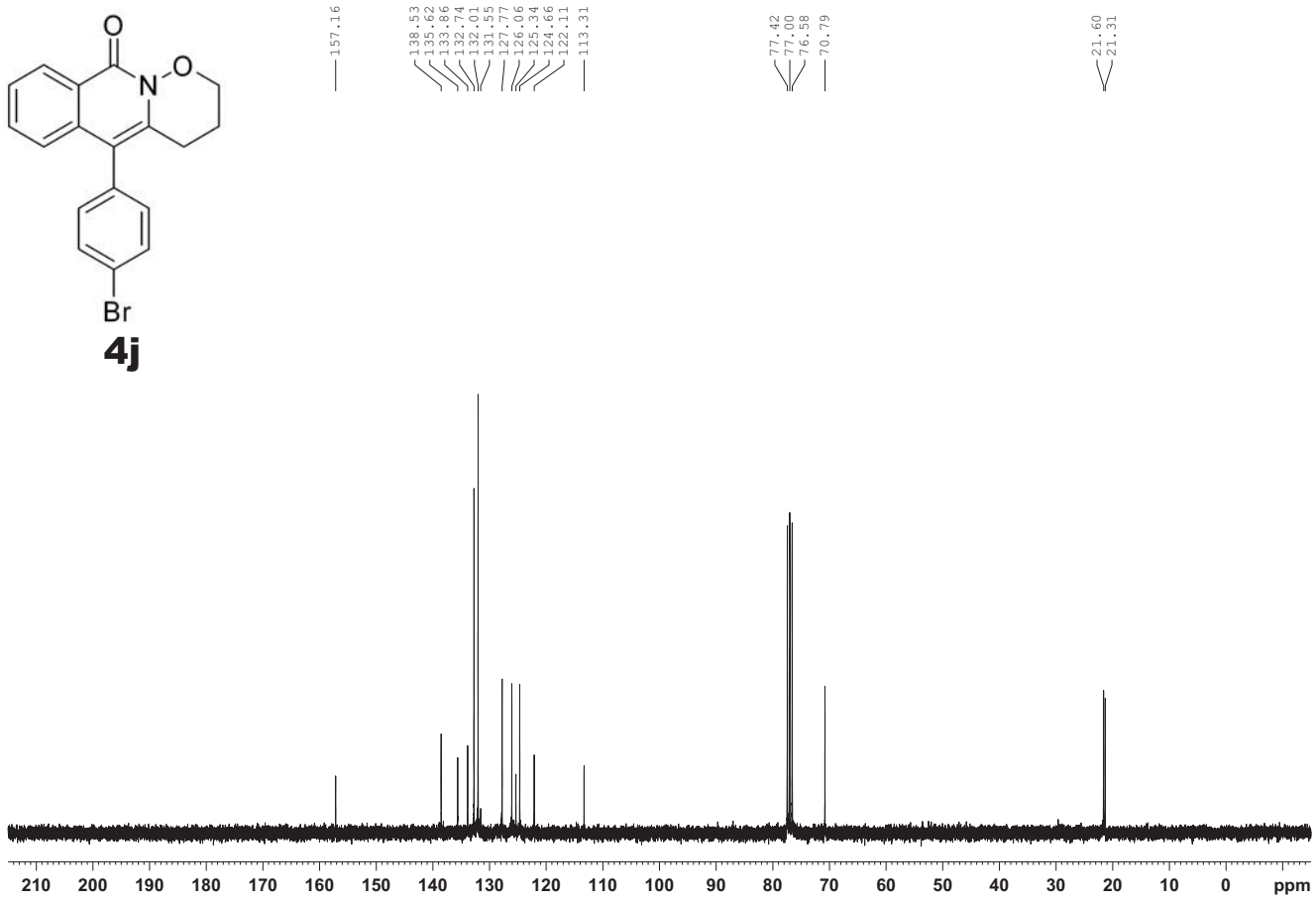

157.13
138.58
135.70
133.92
133.37
132.39
131.98
129.04
127.74
126.01
125.35
124.65
113.27
77.32
77.00
76.68
70.76
21.60
21.29
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


4i'
7.273
7.265
7.252
7.134
7.112
6.682
6.657
6.572
6.547
4.349
4.335
4.321
2.995
2.979
2.962
2.088
2.072
2.058
2.043
2.027
0.000
2.30
2.05
2.04
2.04
2.00
2.04
2.05
ppm

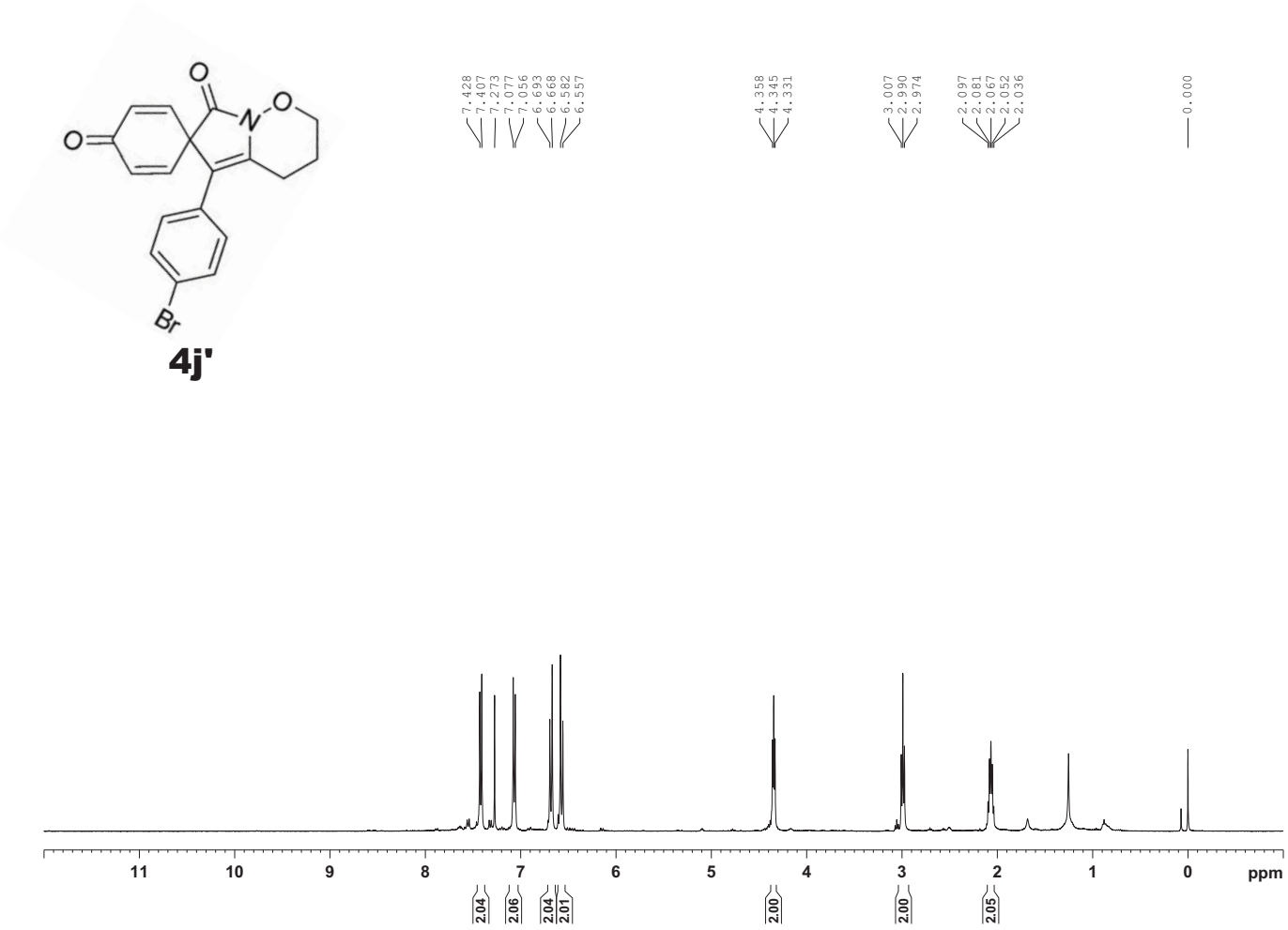

4i'
184.85
165.62
143.51
136.38
133.63
133.06
129.99
129.00
128.39
112.78
77.31
77.00
76.68
70.82
57.29
21.18
20.69
ppm

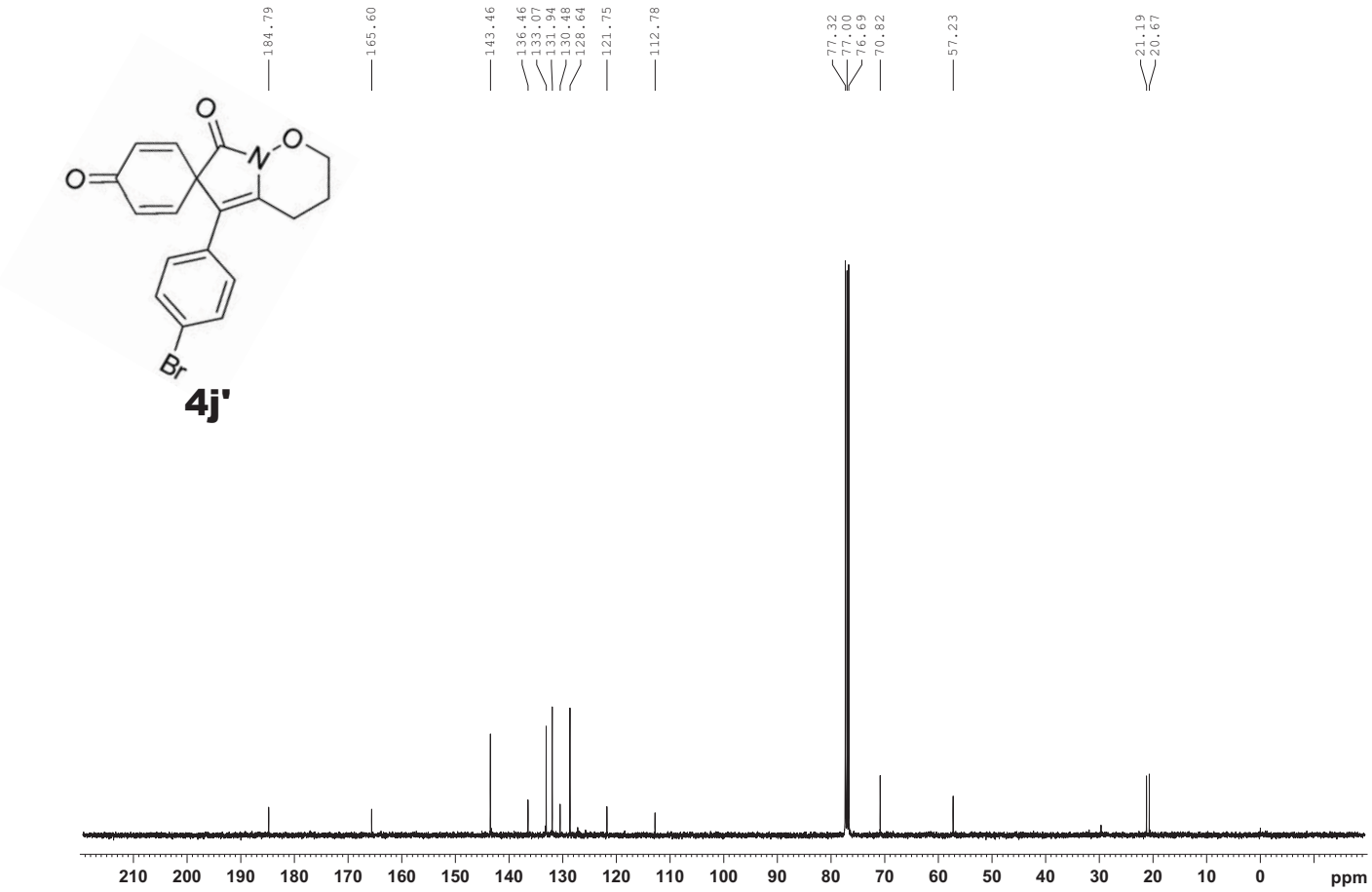

8.539
8.512
7.655
7.628
7.568
7.563
7.540
7.517
7.513
7.476
7.452
7.429
7.286
7.199
7.171
7.158
7.130
4.468
4.442
4.415
2.728
2.705
2.682
2.133
2.110
2.085
2.059
2.035
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.01
2.01
1.04
1.10
3.06
2.00
2.05
2.05

4j


157.16
138.53
135.62
133.86
132.74
132.01
131.55
127.77
126.06
125.34
124.66
122.11
113.31
77.42
77.00
76.58
70.79
21.60
21.31
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

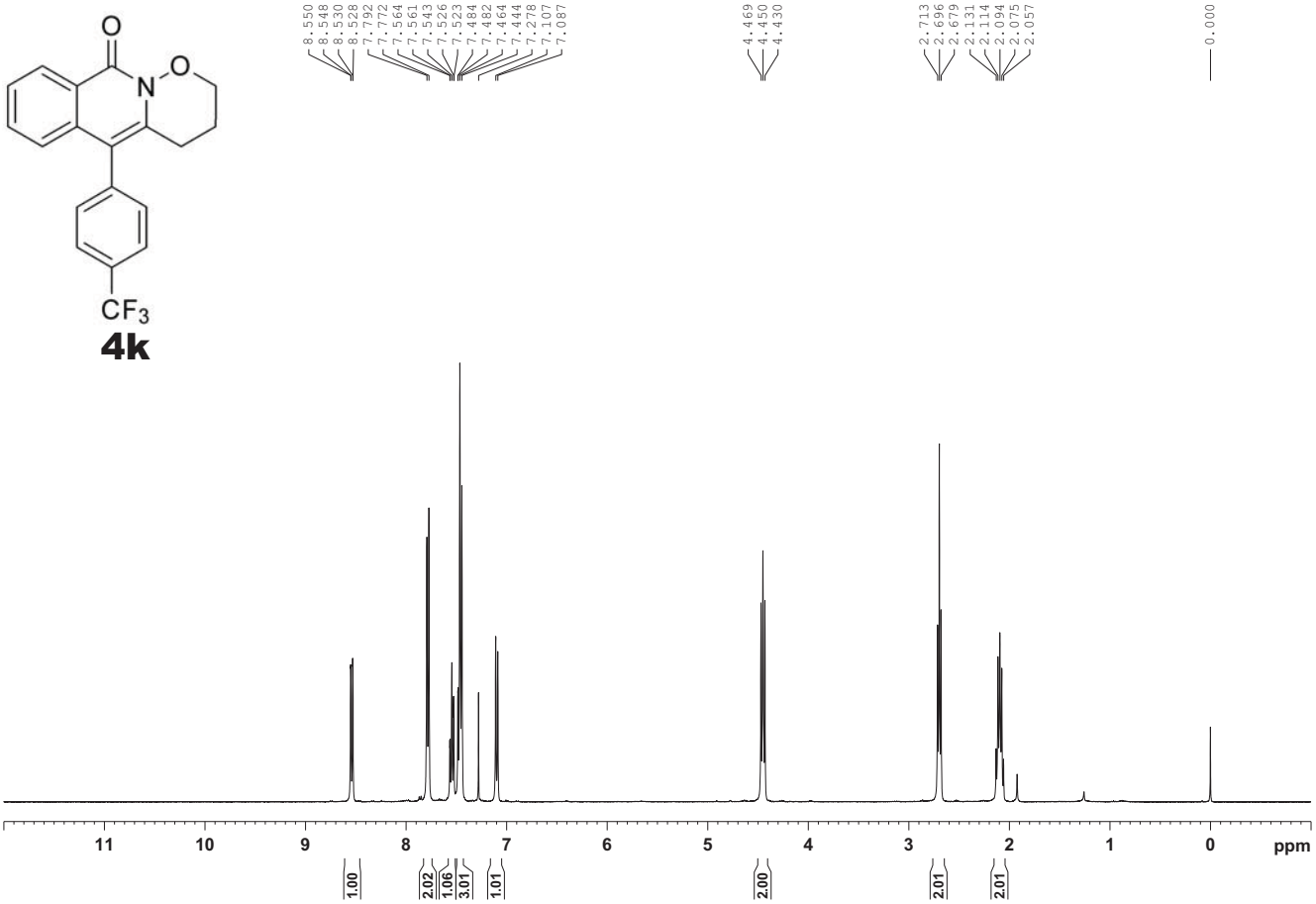
4j

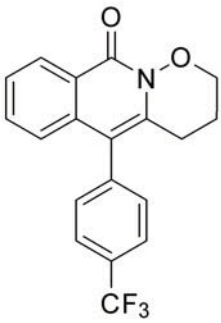

4j'
7.428
7.407
7.273
7.077
7.056
6.693
6.668
6.582
6.557
4.358
4.345
4.331
3.007
2.990
2.974
2.097
2.081
2.067
2.052
2.036
0.000
2.04
2.06
2.04
2.01
2.00
2.00
2.05
ppm


4j'
184.79
165.60
143.46
136.46
133.07
131.94
130.48
128.64
121.75
112.78
77.32
77.00
76.69
70.82
57.23
21.19
20.67
ppm

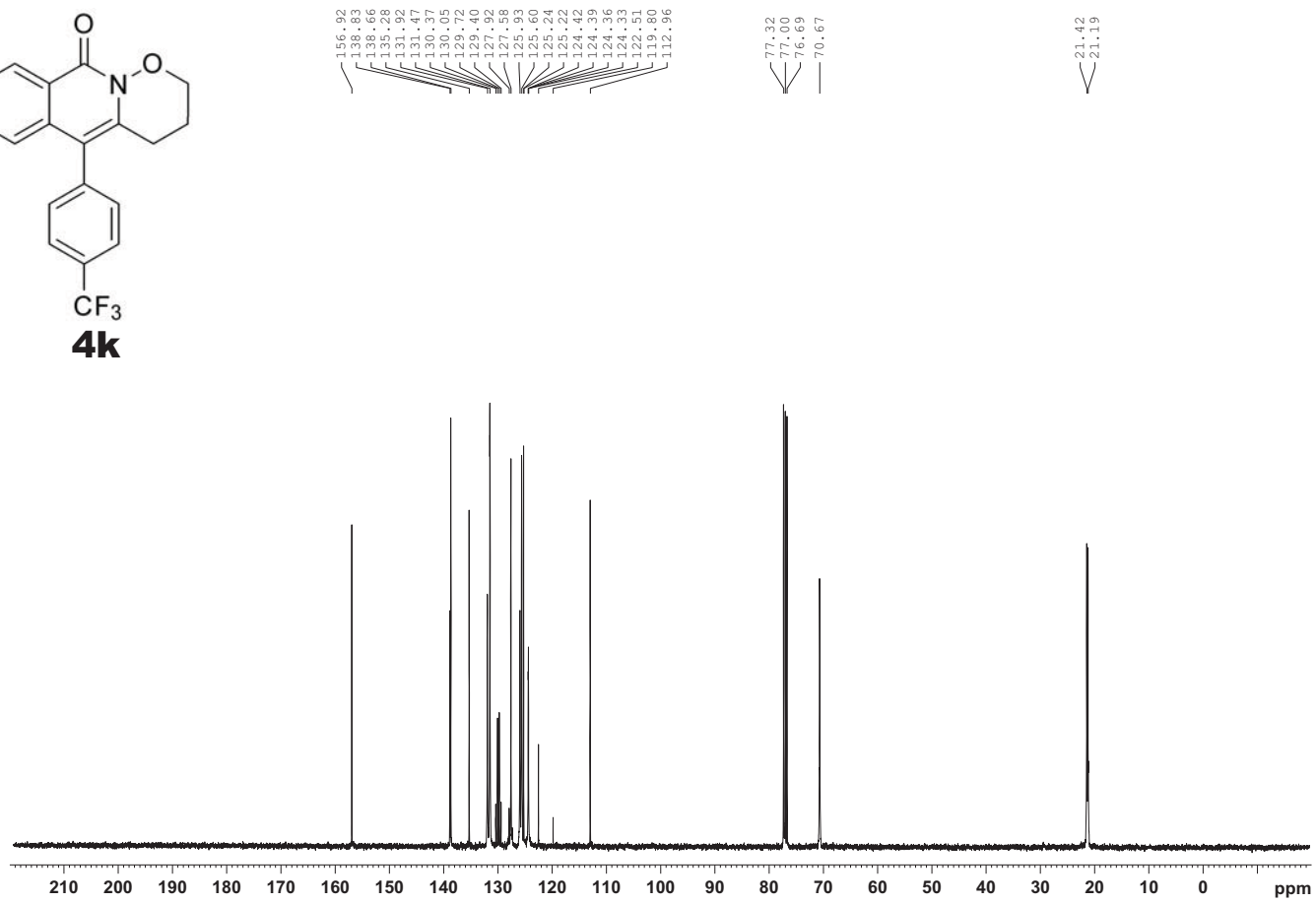
O
N
O
CF3

4k

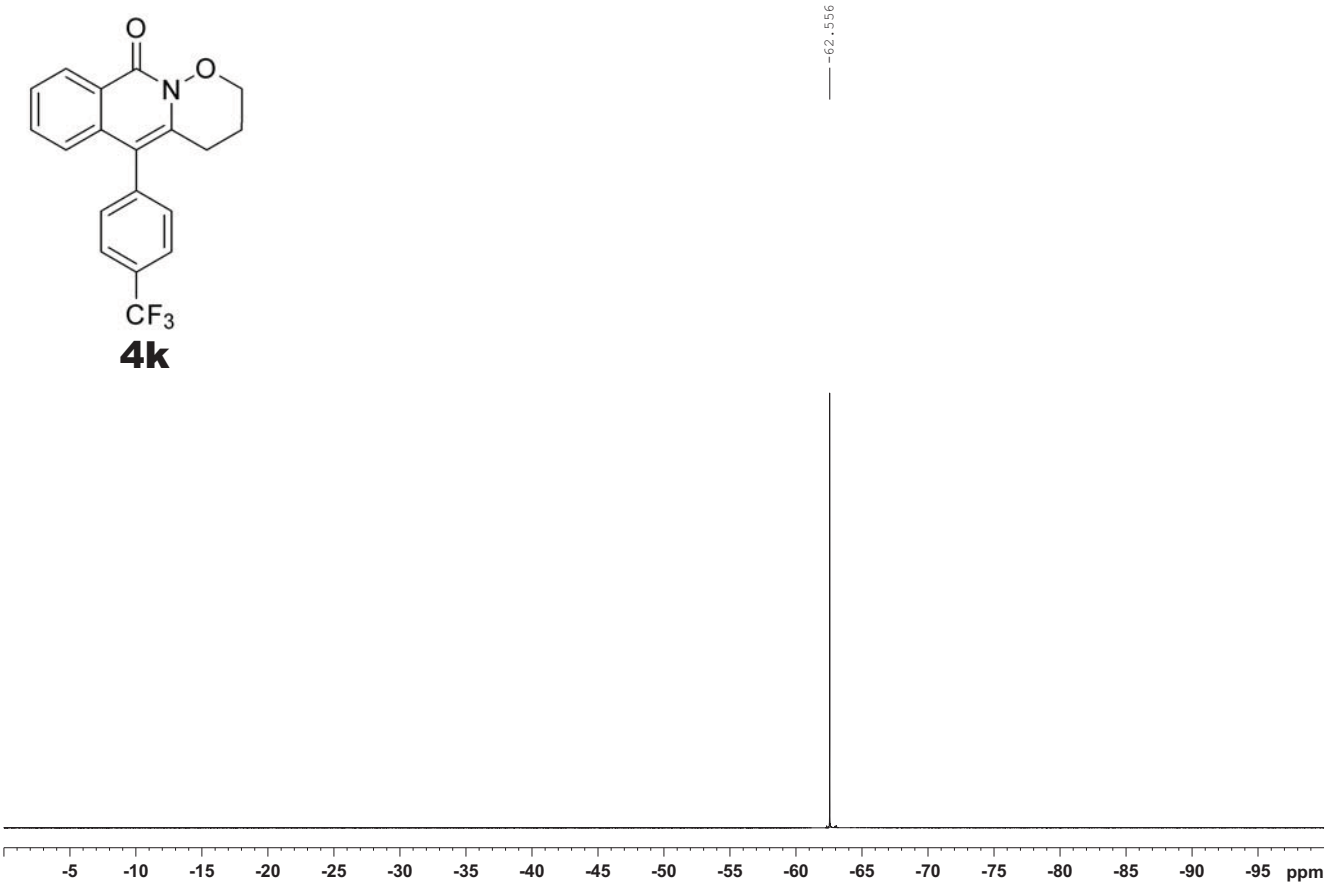

8.550
8.548
8.530
8.528
7.792
7.772
7.564
7.561
7.543
7.526
7.523
7.484
7.482
7.464
7.444
7.278
7.107
7.087
4.469
4.450
4.430
2.713
2.696
2.679
2.131
2.114
2.094
2.075
2.057
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
2.02
1.06
3.01
1.01
2.00
2.01
2.01


O
N
O
CF3

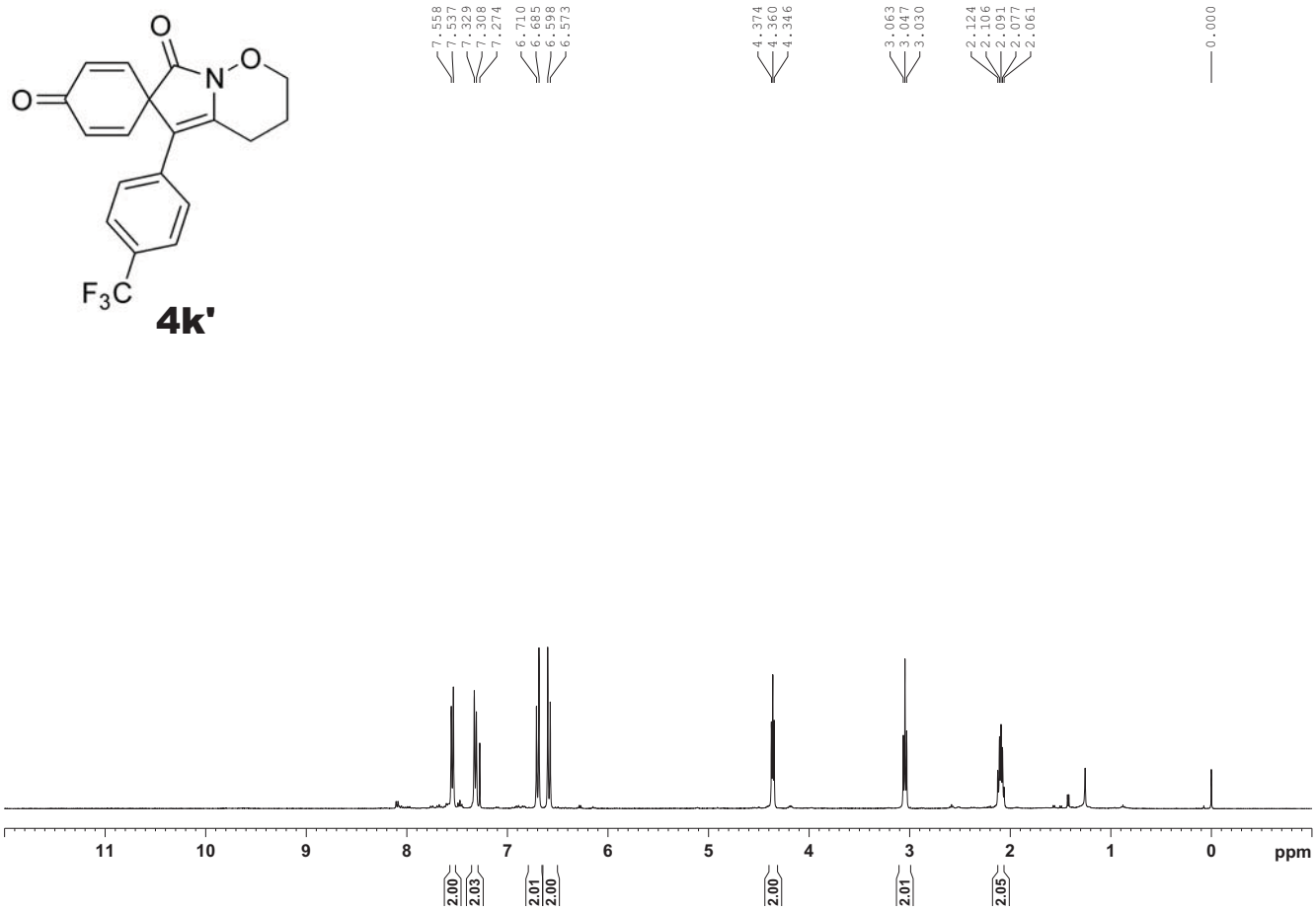
4k


156.92
138.83
138.66
135.28
131.92
131.47
130.37
130.05
129.72
129.40
127.92
127.58
125.93
125.60
125.24
125.22
124.42
124.39
124.36
124.33
122.51
119.80
112.96
77.32
77.00
76.69
70.67
21.42
21.19
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

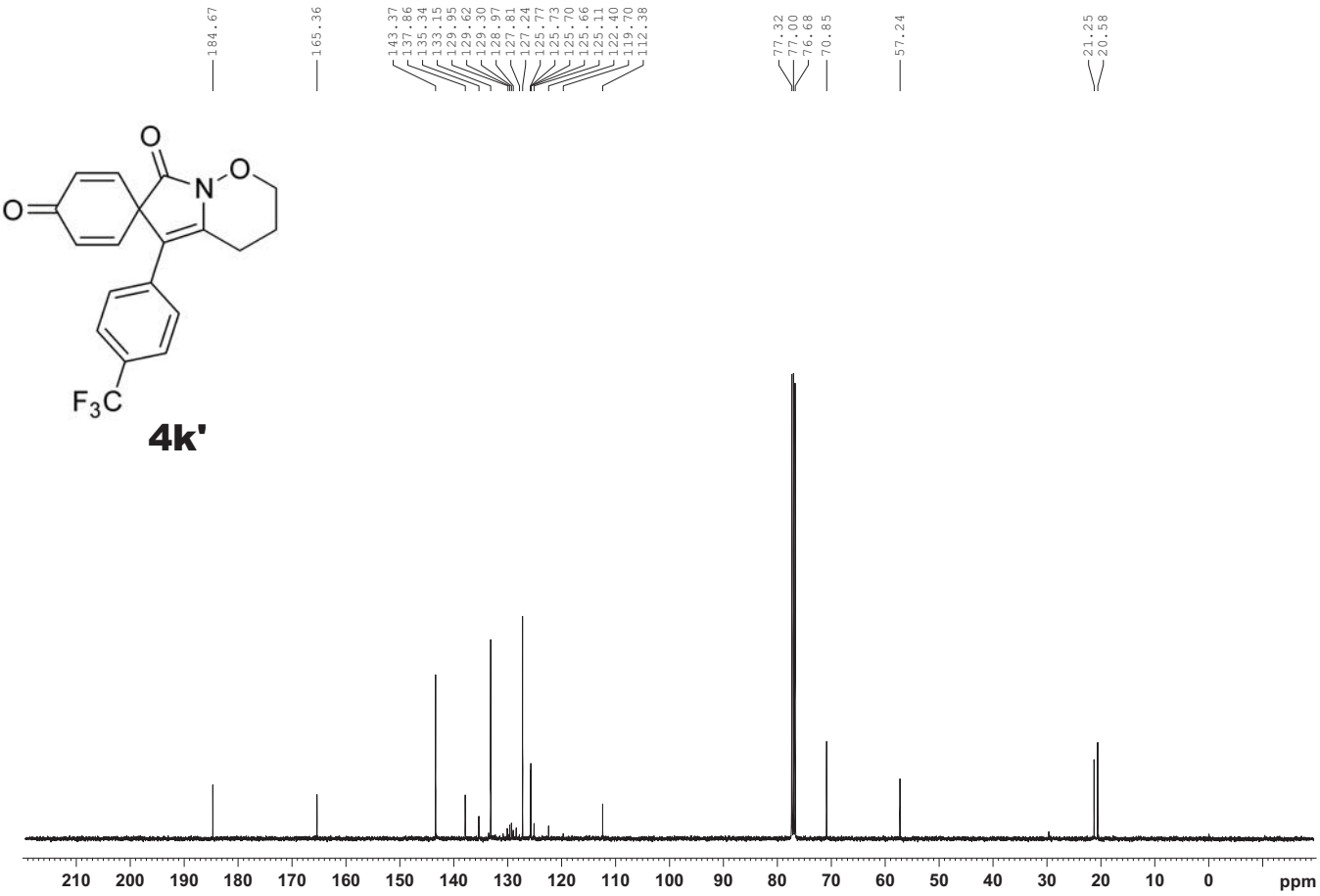
4k



4k'

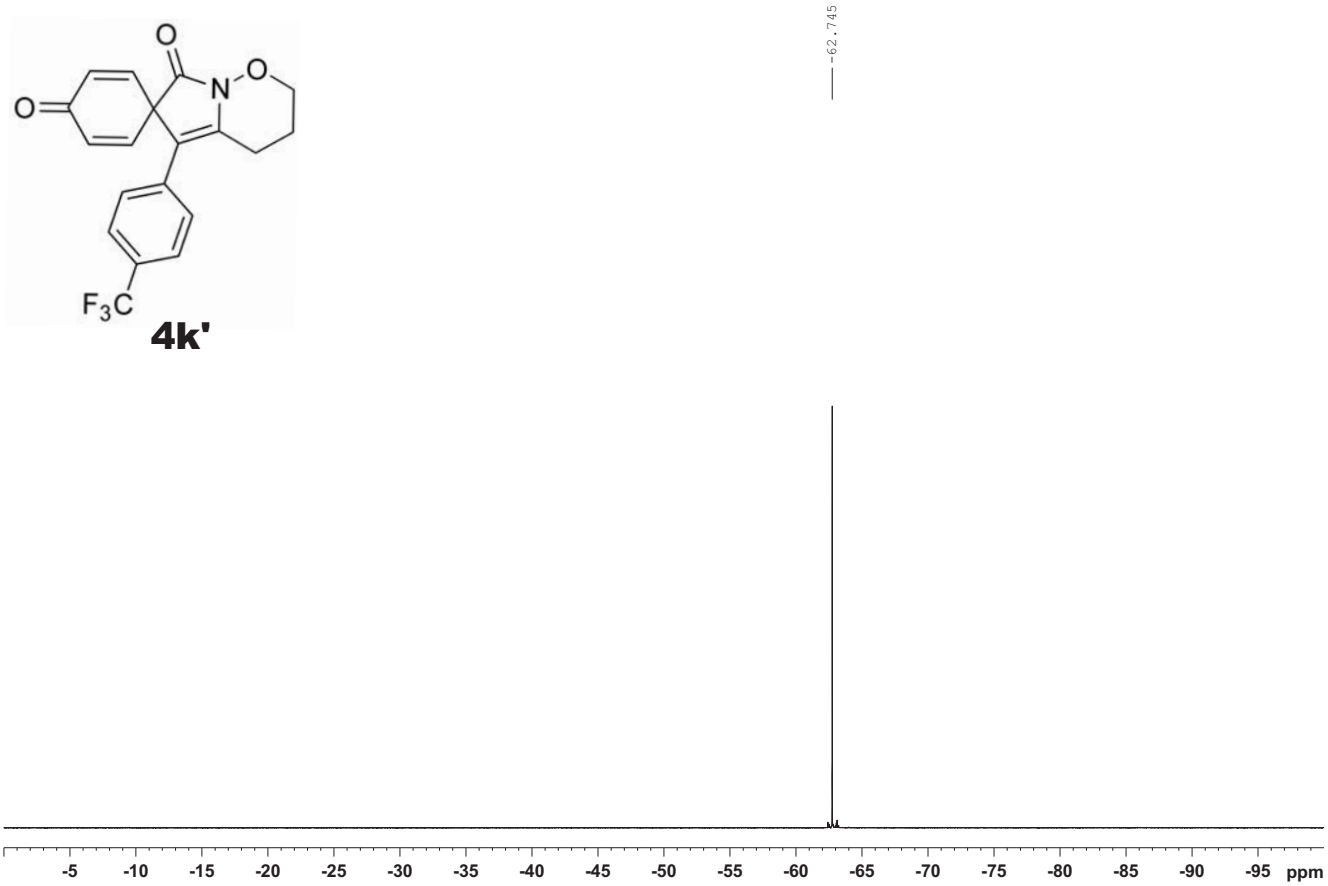

7.558
7.537
7.329
7.308
7.274
6.710
6.685
6.598
6.573
4.374
4.360
4.346
3.063
3.047
3.030
2.124
2.106
2.091
2.077
2.061
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
2.03
2.01
2.00
2.00
2.01
2.05

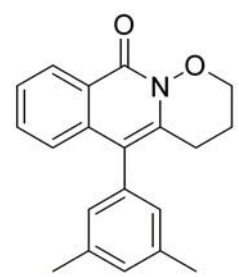
4k'


184.67
165.36
143.37
137.86
135.34
133.15
129.95
129.62
129.30
128.97
127.81
127.24
125.77
125.73
125.70
125.66
125.11
122.40
119.70
112.38
77.32
77.00
76.68
70.85
57.24
21.25
20.58
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

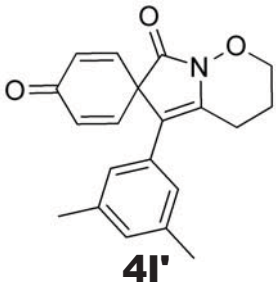
O
O
N
O
F3C

4k'

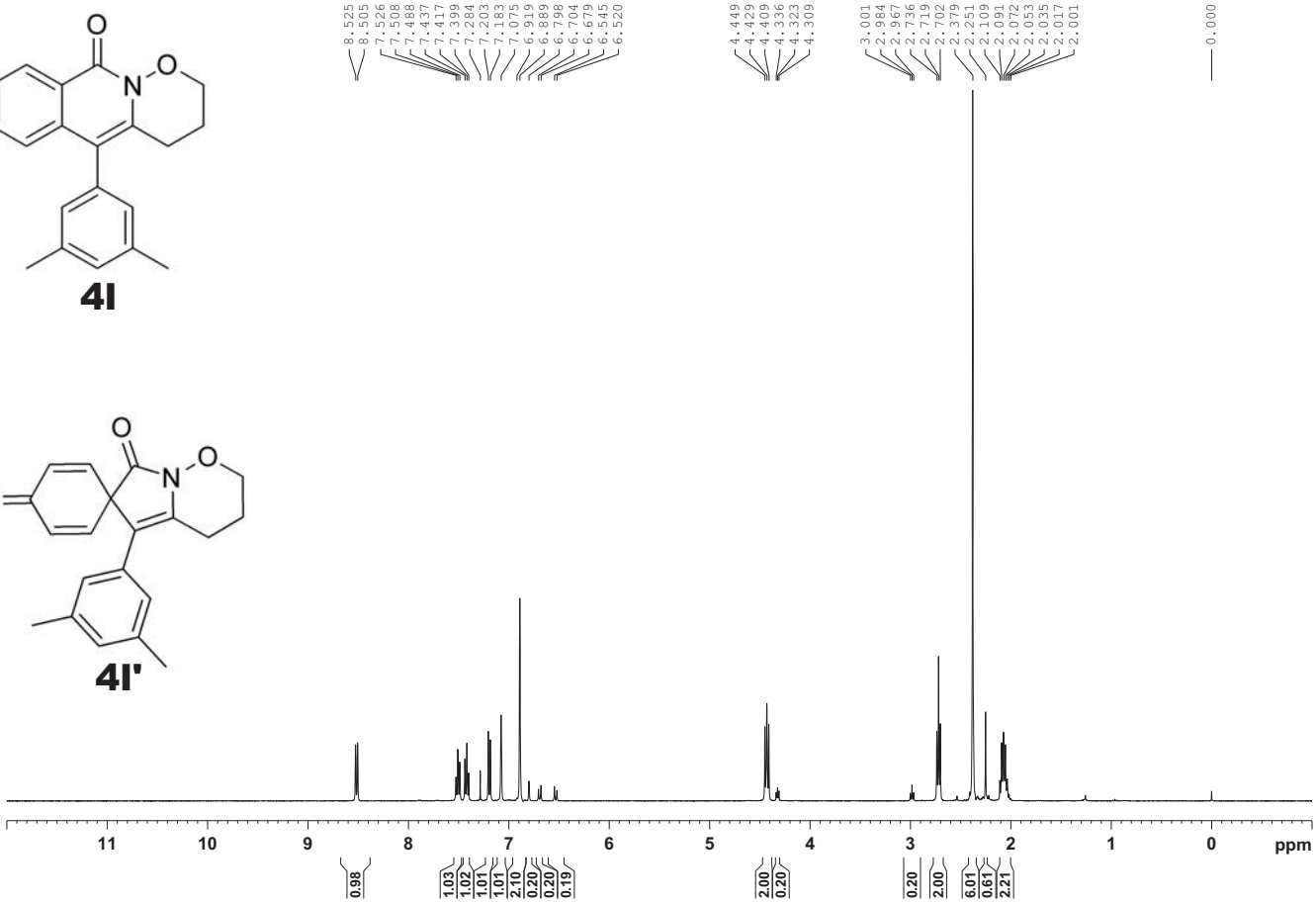

-62.745
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
ppm

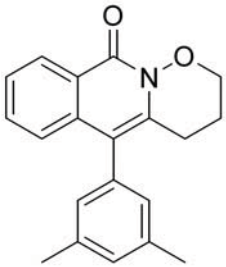


4l

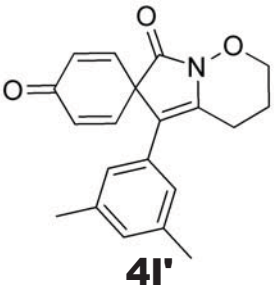


4l'

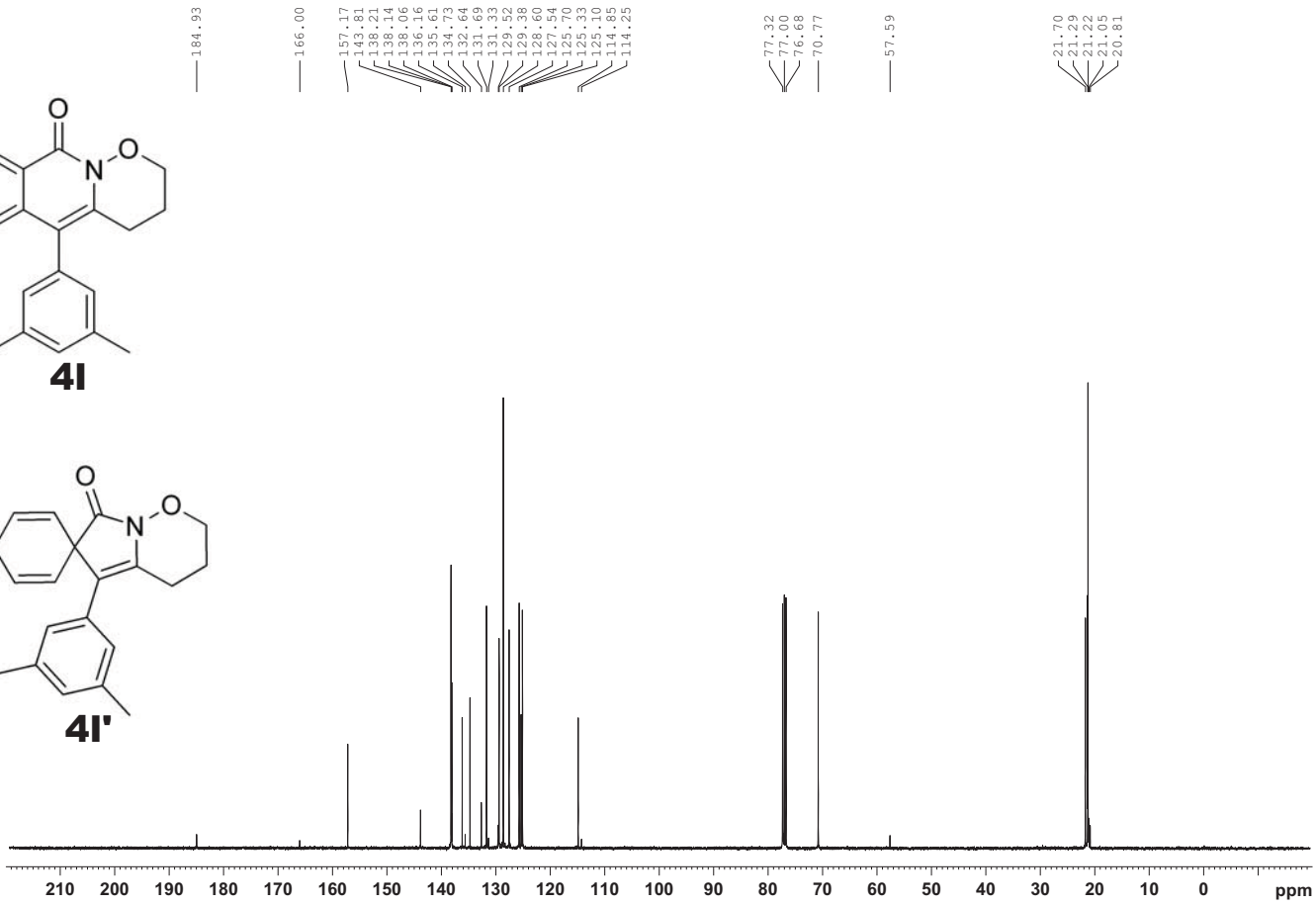

8.525
8.505
7.526
7.508
7.488
7.437
7.417
7.399
7.284
7.203
7.183
7.075
6.919
6.889
6.798
6.704
6.679
6.545
6.520
4.449
4.429
4.409
4.336
4.323
4.309
3.001
2.984
2.967
2.736
2.719
2.702
2.379
2.251
2.109
2.091
2.072
2.053
2.035
2.017
2.001
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.98
1.03
1.02
1.01
1.01
2.10
0.20
0.20
0.19
2.00
0.20
0.20
2.00
6.01
0.61
2.21

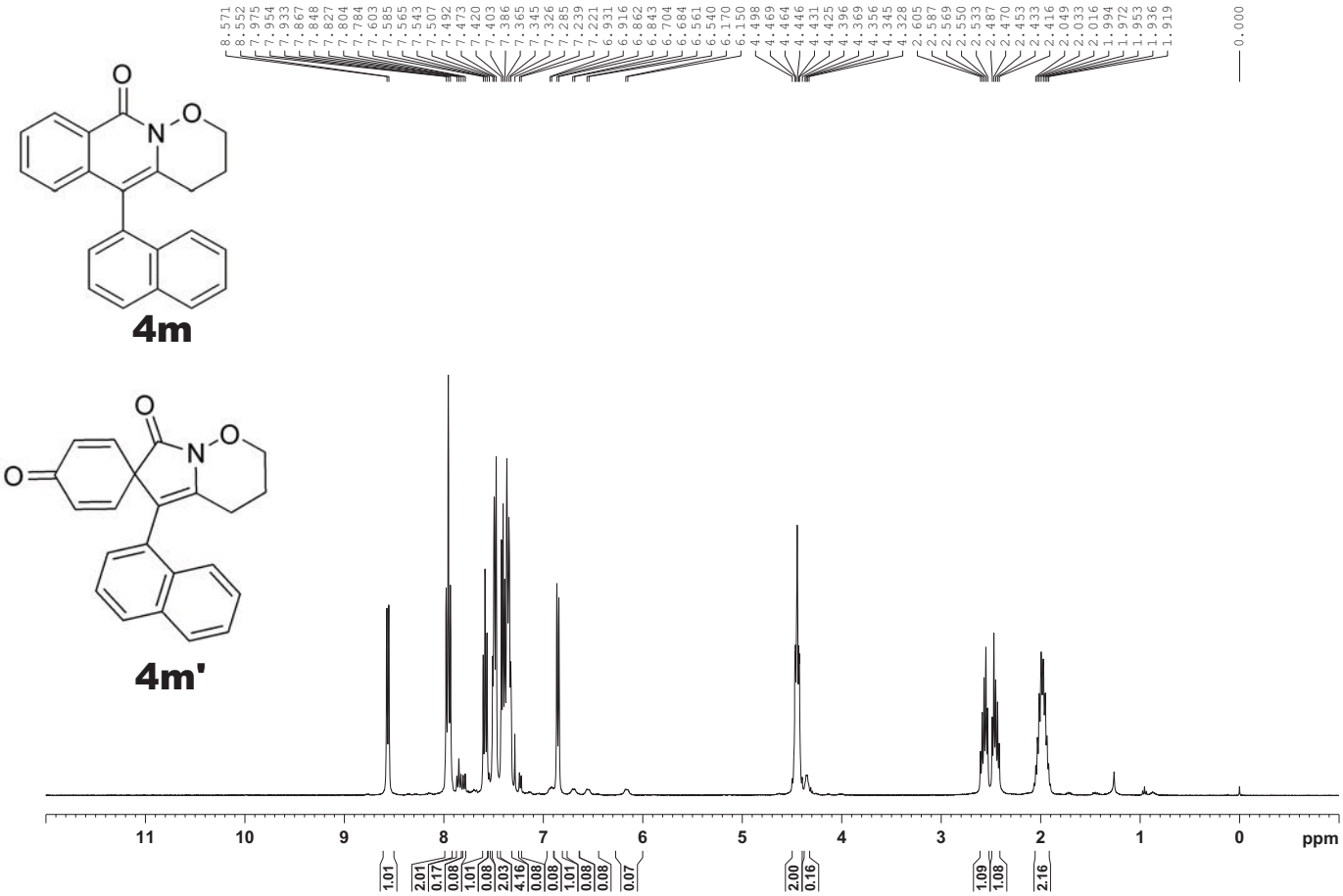


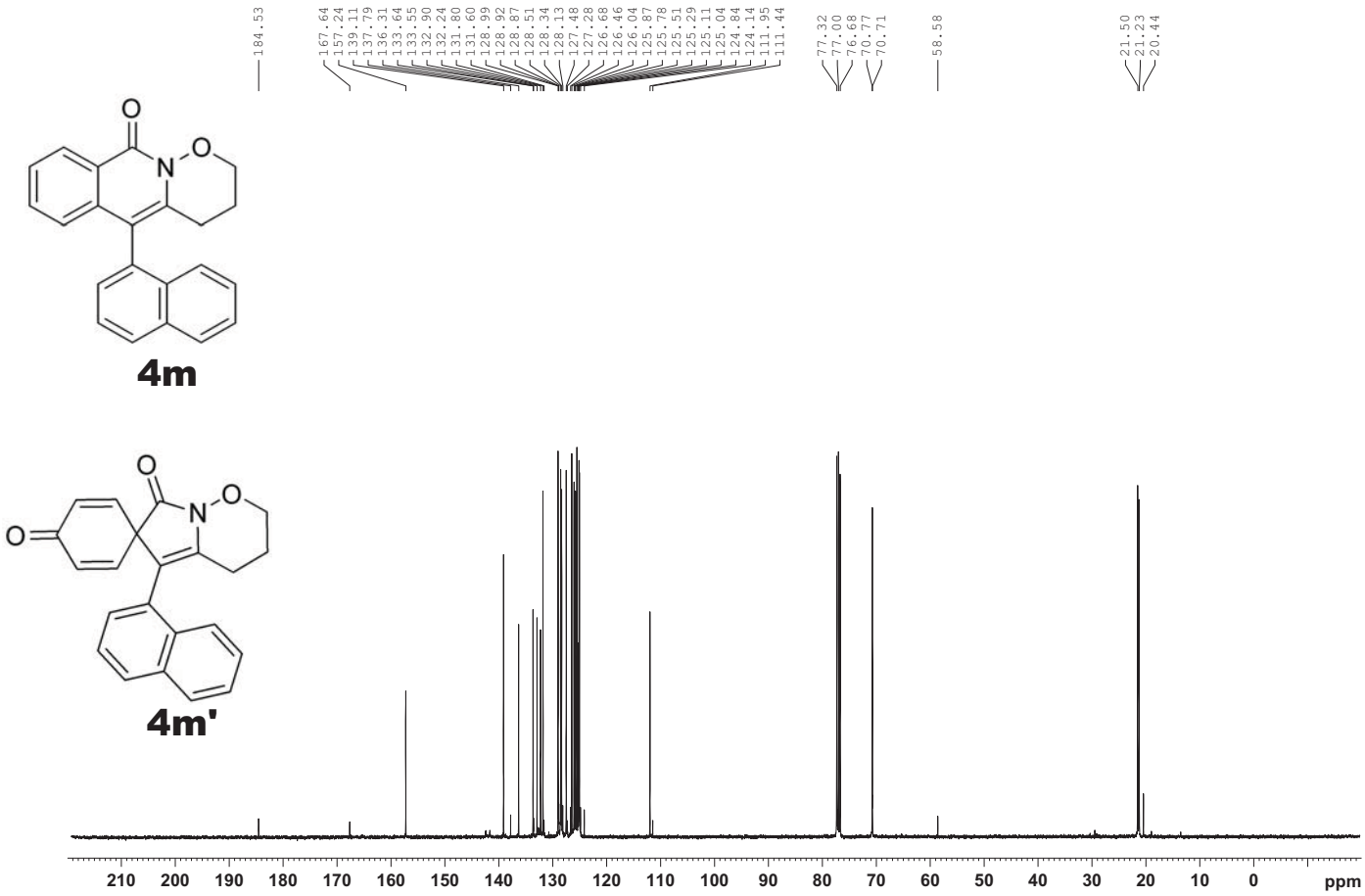
4l

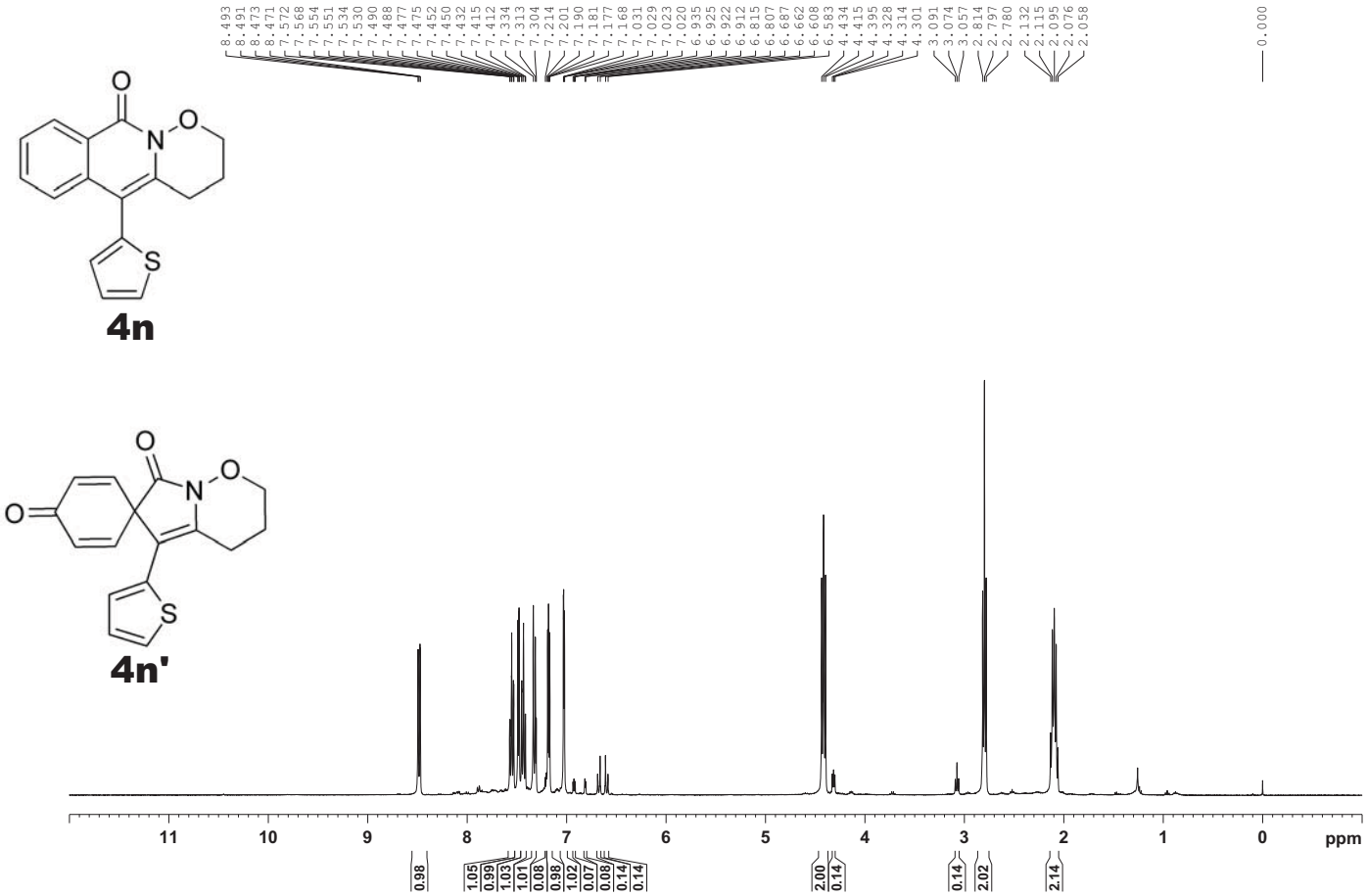


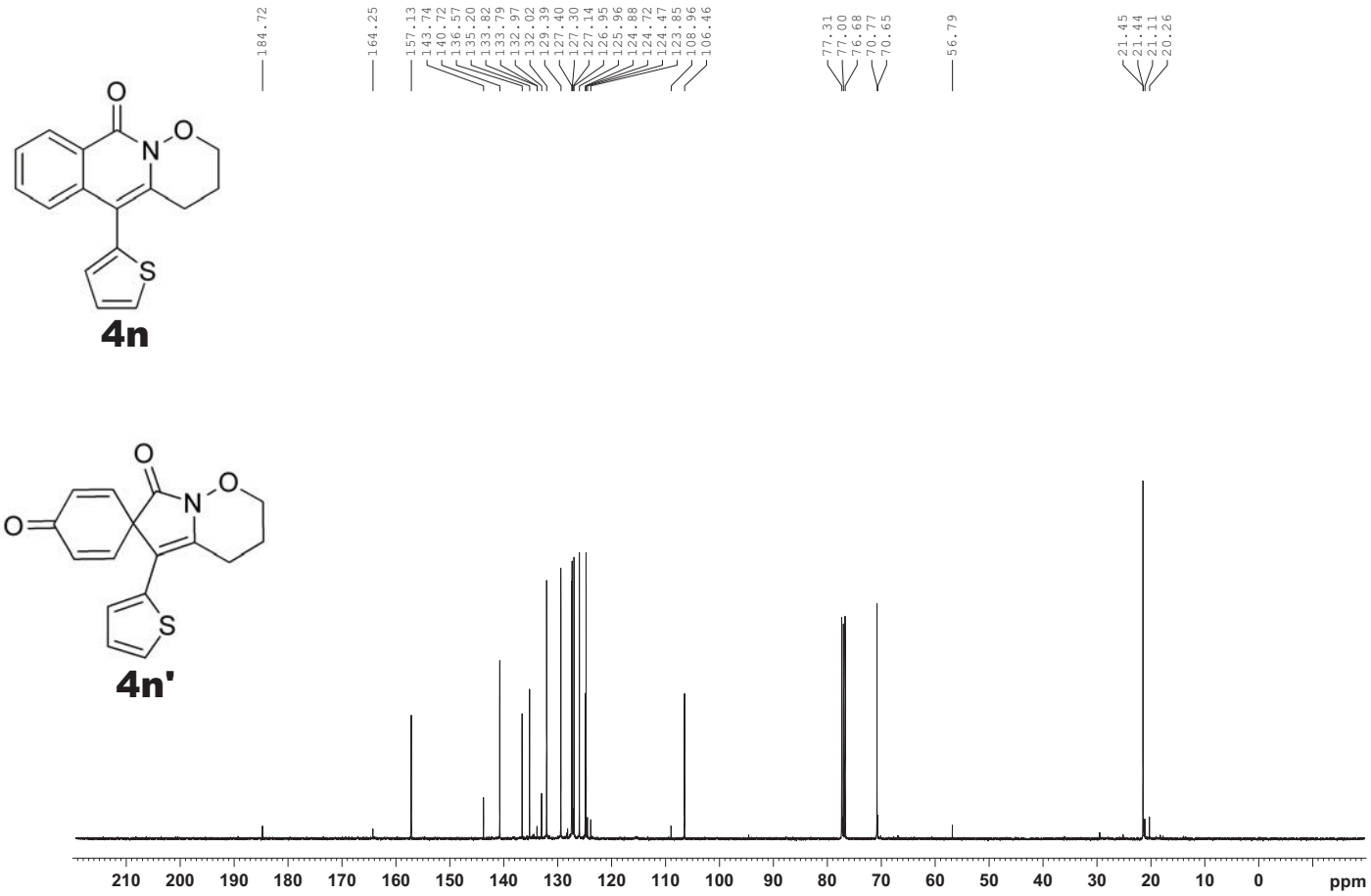
4l'


184.93
166.00
157.17
143.81
138.21
138.14
138.06
136.16
135.61
134.73
132.64
131.69
131.33
129.52
129.38
128.60
127.54
125.70
125.33
125.10
114.85
114.25
77.32
77.00
76.68
70.77
57.59
21.70
21.29
21.22
21.05
20.81
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

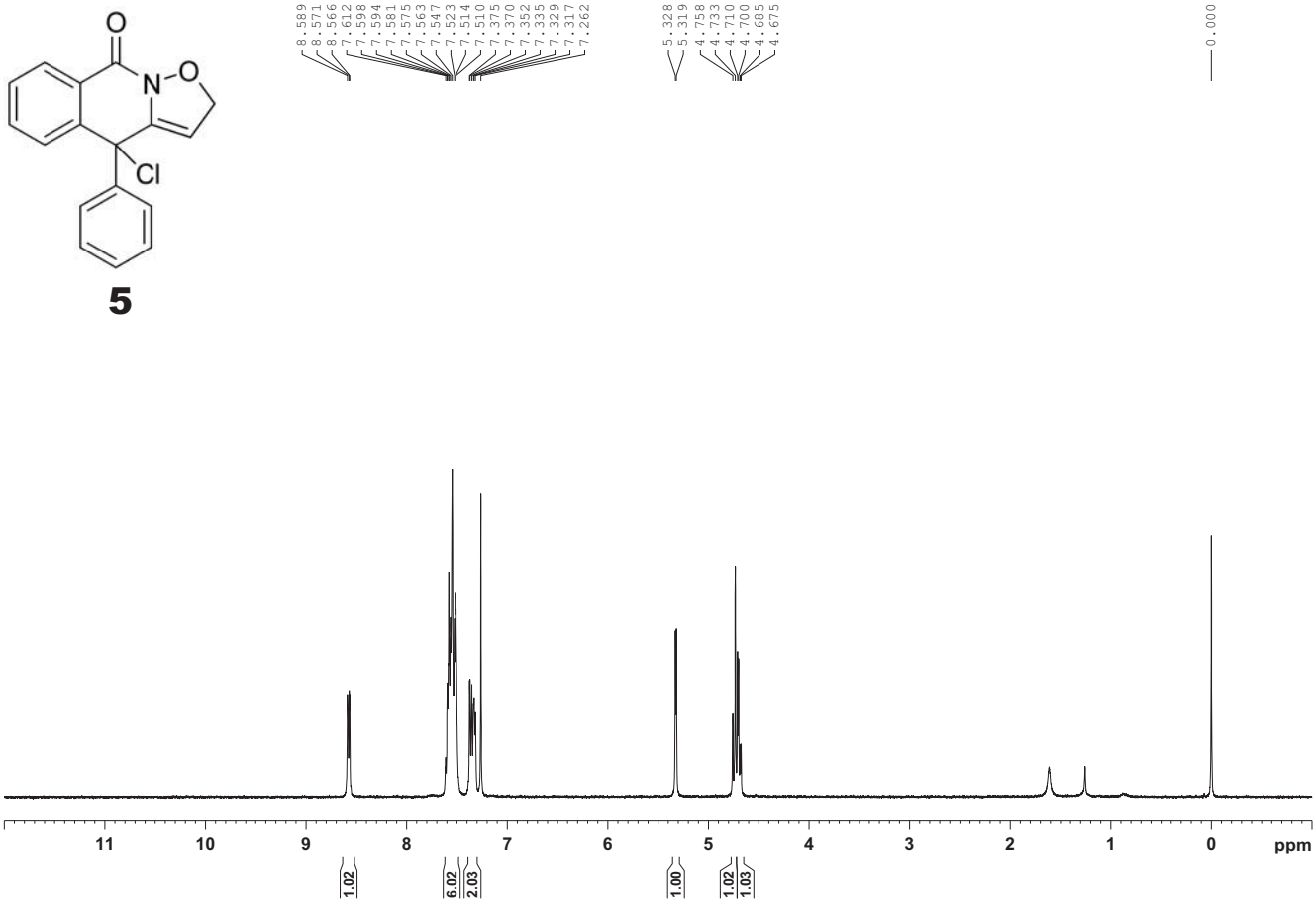

4m
4m'
ppm


4m
4m'
ppm

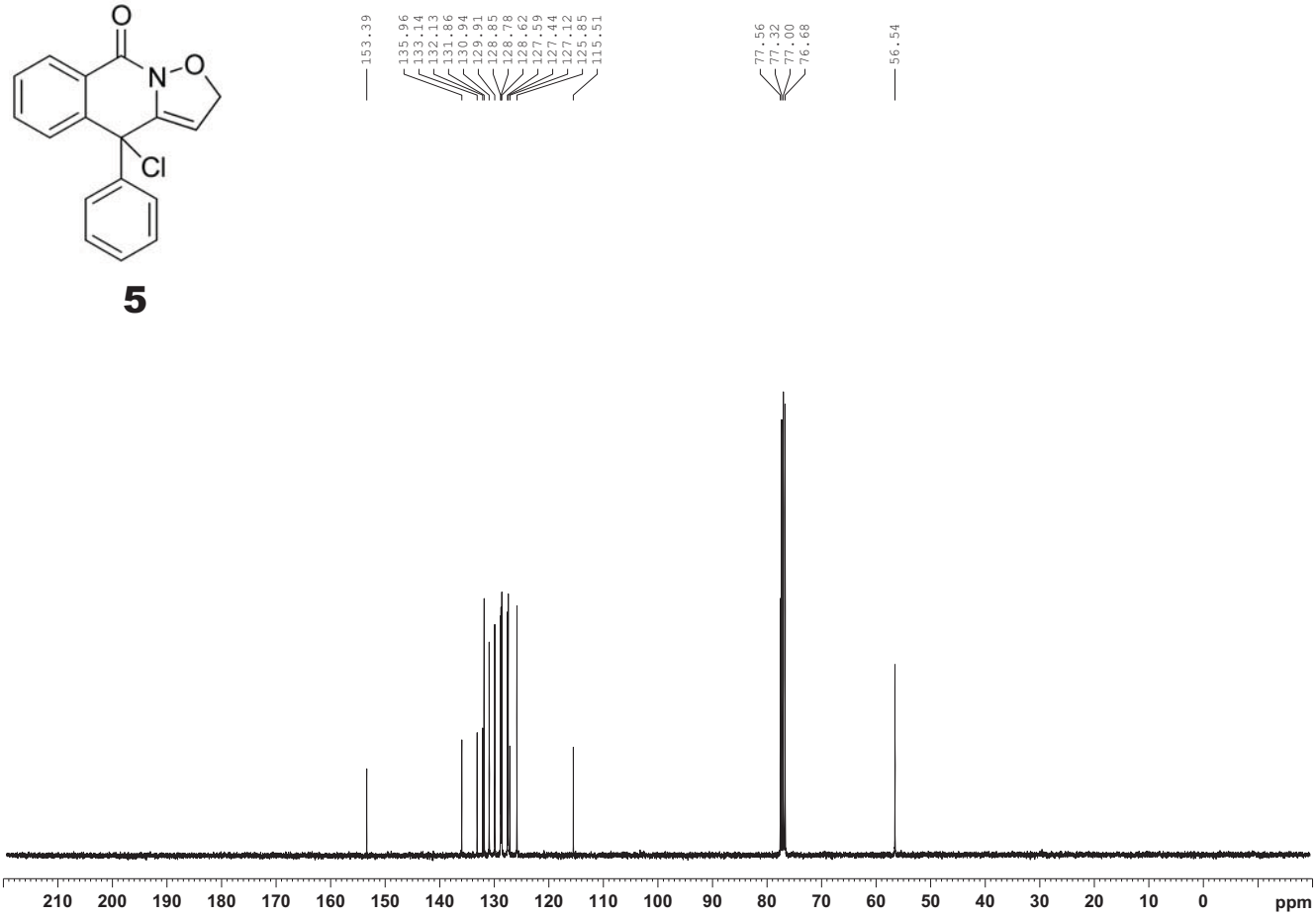

4n
4n'
ppm

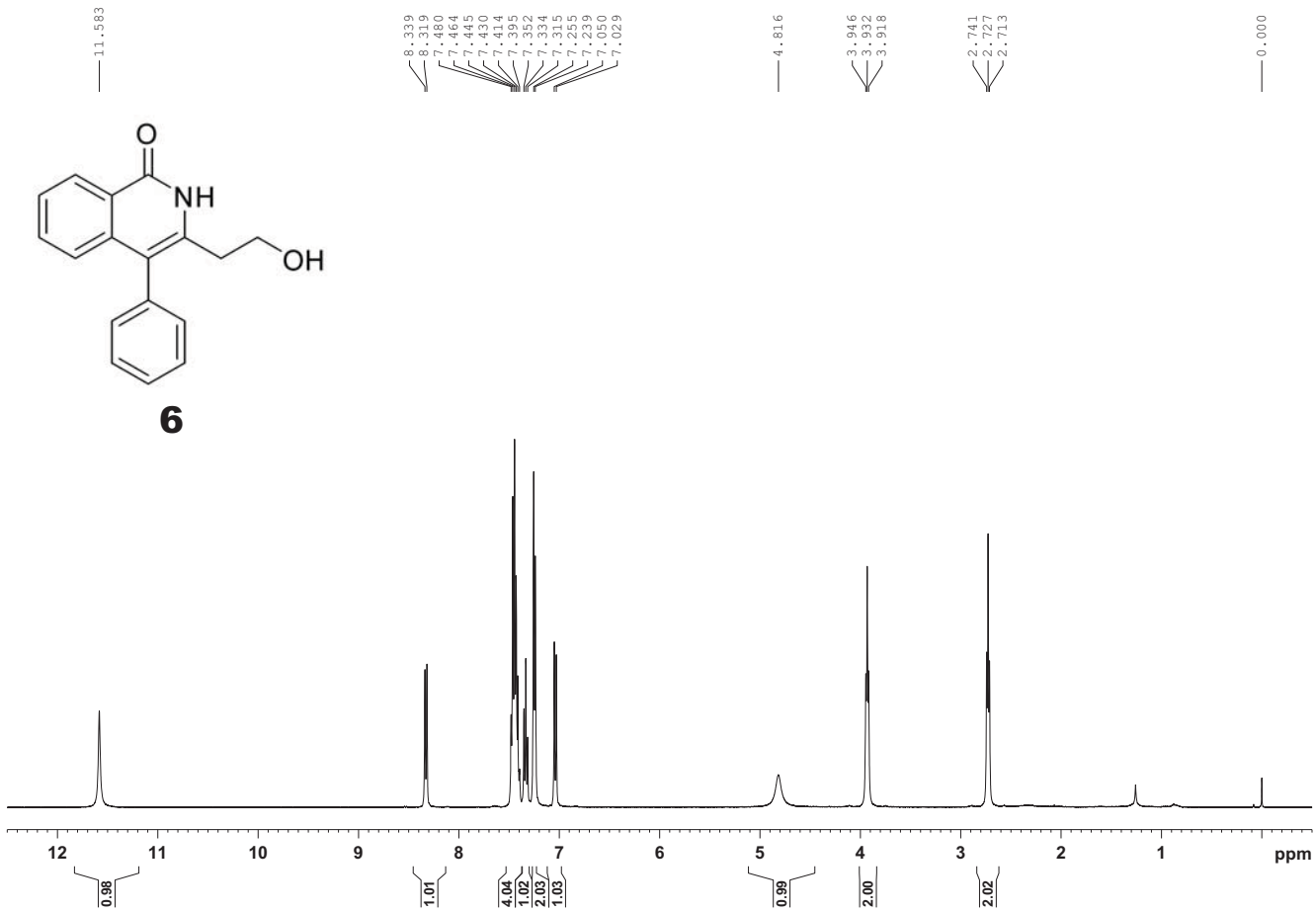

4n
4n'
ppm

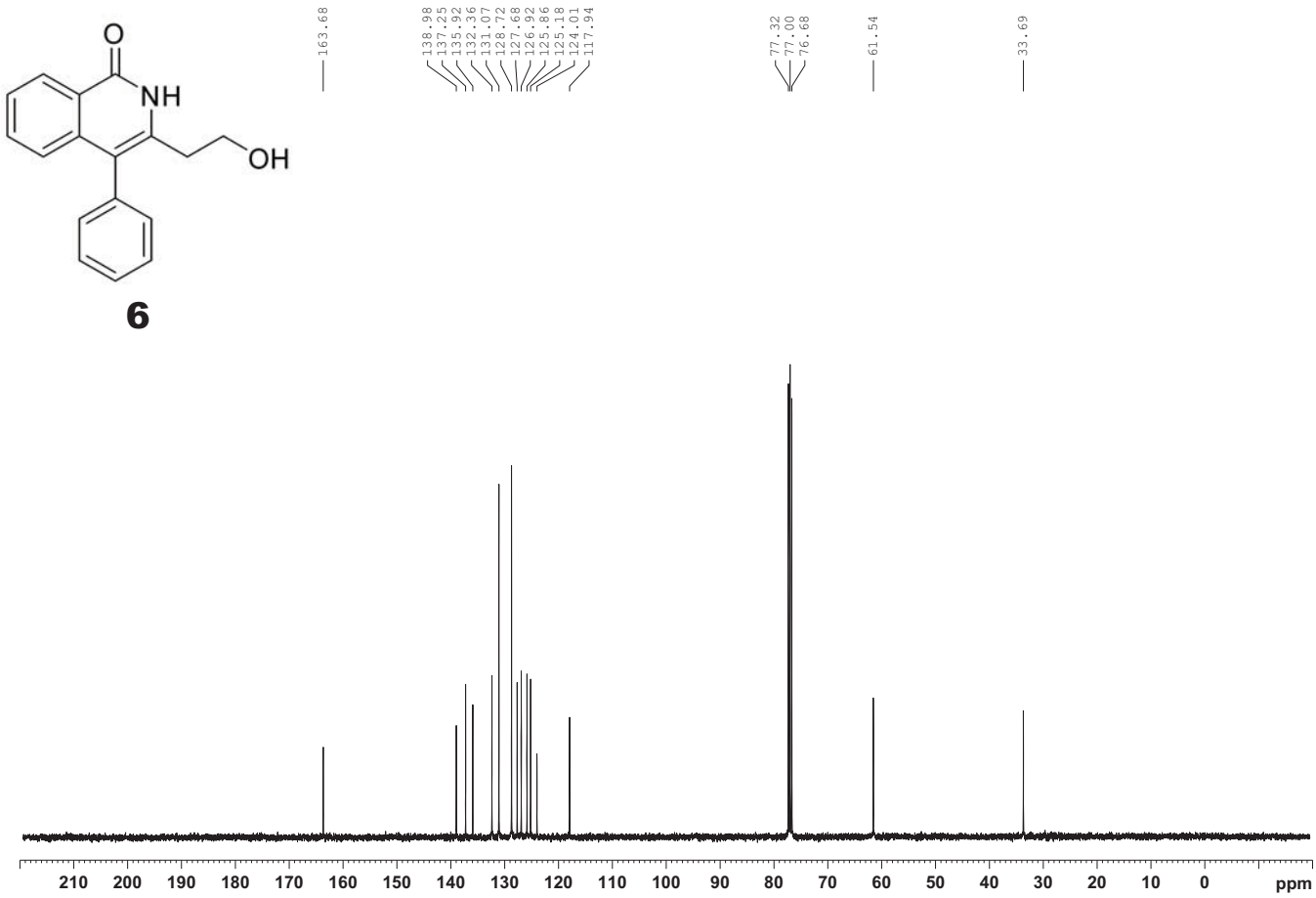
5

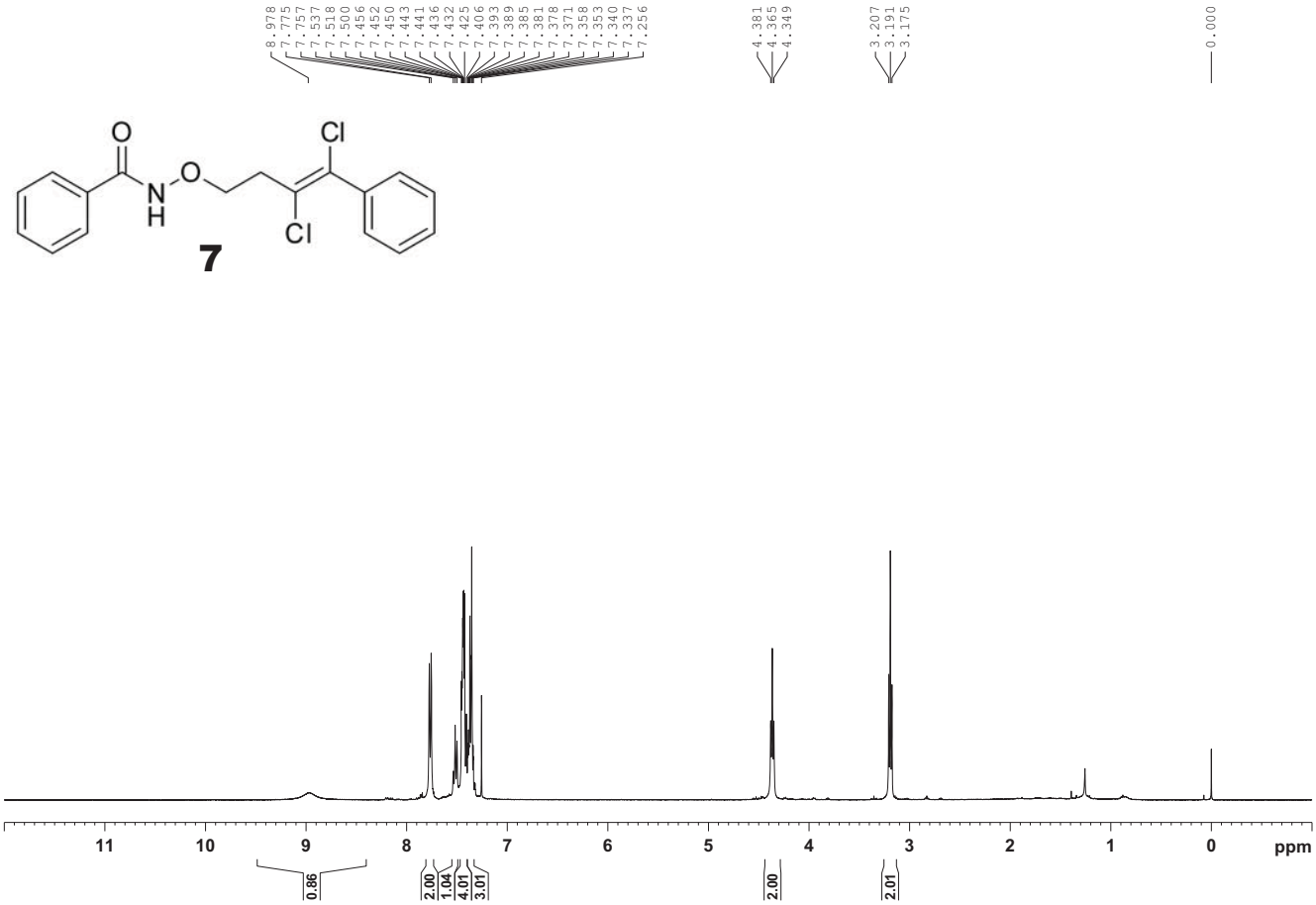

8.589
8.571
8.566
7.612
7.598
7.594
7.581
7.575
7.563
7.547
7.523
7.514
7.510
7.375
7.370
7.352
7.335
7.329
7.317
7.262
5.328
5.319
4.758
4.733
4.710
4.700
4.685
4.675
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.02
6.02
2.03
1.00
1.02
1.03

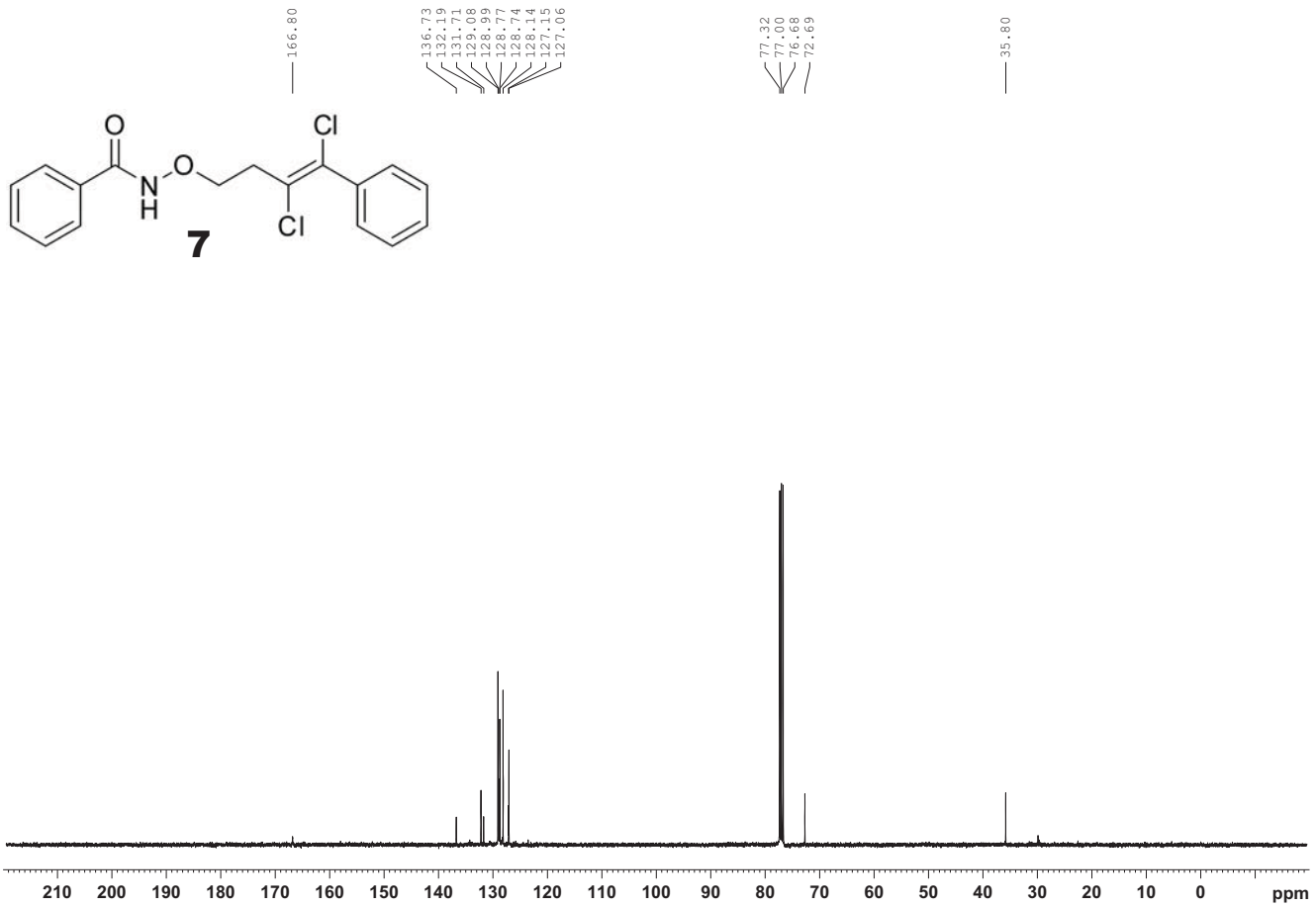
5


153.39
135.96
133.14
132.13
131.86
130.94
129.91
128.85
128.78
128.62
127.59
127.44
127.12
125.85
115.51
77.56
77.32
77.00
76.68
56.54
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


11.583
8.339
8.319
7.480
7.464
7.445
7.430
7.414
7.395
7.352
7.334
7.315
7.255
7.239
7.050
7.029
4.816
3.946
3.932
3.918
2.741
2.727
2.713
0.000
O
NH
OH
6
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.98
1.01
4.04
1.02
2.03
1.03
0.99
2.00
2.02


163.68
138.98
137.25
135.92
132.36
131.07
128.72
127.68
126.92
125.86
125.18
124.01
117.94
77.32
77.00
76.68
61.54
33.69
O
NH
OH
6
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.978
7.775
7.757
7.537
7.518
7.500
7.456
7.452
7.450
7.443
7.441
7.436
7.432
7.425
7.406
7.393
7.389
7.385
7.381
7.378
7.371
7.358
7.353
7.340
7.337
7.256
4.381
4.365
4.349
3.207
3.191
3.175
0.000
O
N
H
O
Cl
Cl
7
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.86
2.00
1.04
4.01
3.01
2.00
2.01


166.80
136.73
132.19
131.71
129.08
128.99
128.77
128.74
128.14
127.15
127.06
77.32
77.00
76.68
72.69
35.80
O
N
H
O
Cl
Cl
7
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm