Supporting Information

Palladium-Catalyzed C(sp^3)−H Nitrooxylation of Masked Alcohols

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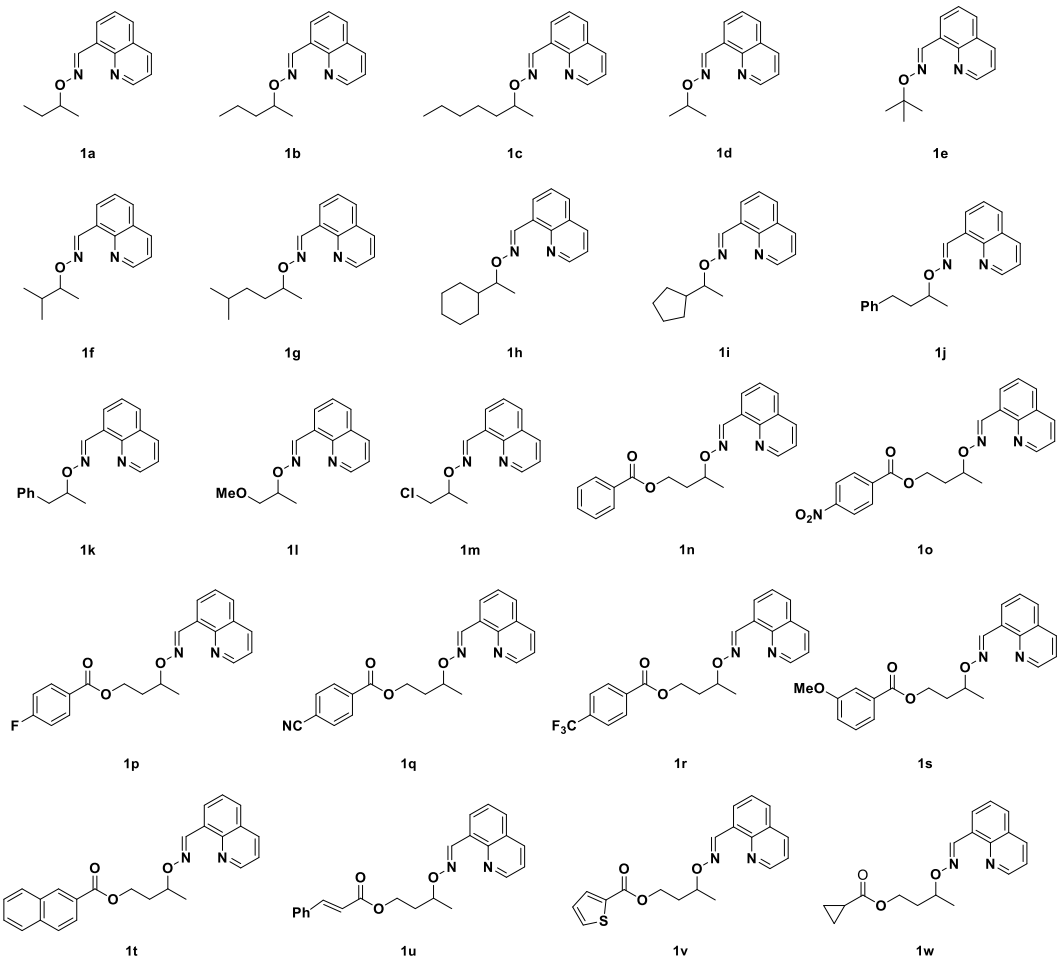
General Information

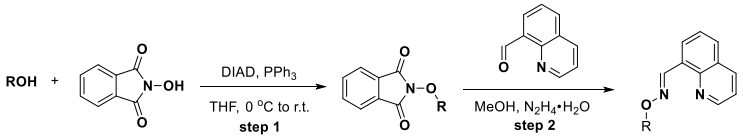
Alcohols were obtained from the commercial sources or synthesized following literature procedures, and used to prepare the corresponding substrates. $Pd(OAc)_2$ was obtained from Heowns, $AgNO_2$ was obtained from Chem Impex Int'l. Inc. Selectfluor was obtained from Macklin, Tetrabutyl ammonium hydrogen sulfate (TBAHS) was obtained from Heowns. Solvents were obtained from Nanjing Chemical Reagent and used directly without further purification. Analytical thin layer chromatography (TLC) was performed on pre-coated silica plates. Yields of the products refer to purification by silica-gel column chromatography. Silica gel 60H (200-300 mesh) manufactured by Qingdao Haiyang Chemical Group Co. (China) was used for general chromatography. ^{1}H NMR was recorded on Bruker AV-500 instrument (500 MHz). Chemical shifts were quoted in parts per million (ppm) referenced to 0.0 ppm for tetramethylsilane. The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q =quartet, m = multiplet, br = broad. Coupling constants, J, were reported in Hertz unit (Hz). ^{13}C NMR spectra were recorded on Bruker AV-500 instrument (126 MHz) were fully decoupled by broad band proton decoupling. Chemical shifts were reported in ppm referenced to either the center line of a triplet at 77.16 ppm of $CDCl_3$ or the center line of a multiplet at 39.52 ppm of DMSO-d_6. ^{19}F NMR spectra were recorded on Bruker AVANCE AV-500 instrument (471 MHz) were fully decoupled by broad band proton decoupling. High-resolution mass spectra (HRMS) were recorded on an Agilent Mass spectrometer using ESI-TOF (electrospray ionization-time of flight).

Preparation of Substrates



All the substrates were prepared according to the following procedures.[1,2] Substrates **1a** to **1m** were prepared by Method A and **1n**-**1w** were prepared by Method B.

Method A

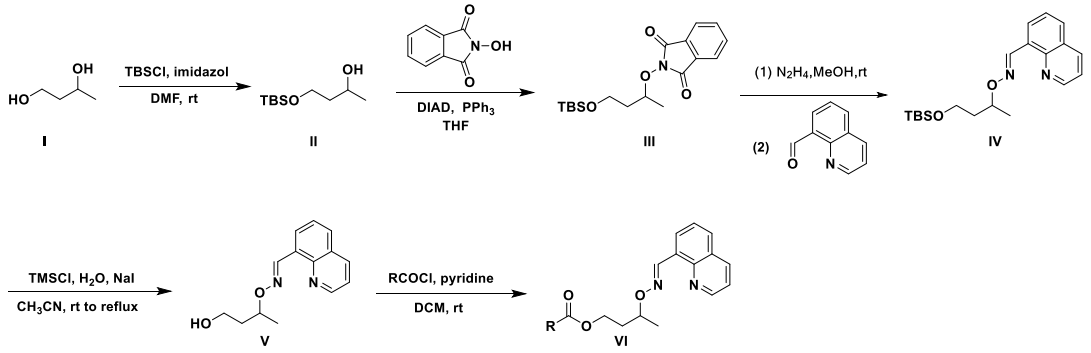


Step 1 : To a solution of the alcohol (10 mmol, 1.0 equiv), *N*-hydroxyphthalimide (1.956g, 12 mmol, 1.2 equiv) and PPh_3 (3.144 g, 12 mmol, 1.2 equiv) in 20 mL THF was added diisopropyl azodicarboxylate (2.4 mL, 12 mmol, 1.2 equiv) dropwise at 0 °C. The reaction was then allowed to warm to room temperature and stirred overnight. After the reaction is finished, hexanes were added and the precipitation was collected and directly used in the next step without further purification.

Step 2 : To a solution of *N*-alkoxyphthalimide (1.1 equiv) in MeOH (5 mL/mmol) was added hydrazine monohydrate (1.15 equiv) at room temperature. The reaction was monitored by TLC and usually completed in 30 min. The quinoline-8-carbaldehyde (1.0 equiv) was added. The reaction was monitored by TLC. After 0.5 h, the mixture was

filtered to remove precipitate if formed and then the solvent was removed under reduced pressure. The residue was purified by flash column chromatography on silica gel to give the title compounds.

Method B

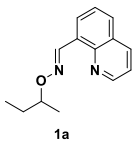


4-(*tert*-butyldimethylsilyloxy)-butan-2-ol (**II**) : Under ice-water bath conditions, add 1,3-butanediol (**I**) (1.35 g, 15 mmol, 1 equiv) and imidazole (2.25 g, 33 mmol, 2.2 equiv) to *N*,*N*-dimethylformamide (7.5 mL). Stir the reaction for 1 h. Then *tert*-butyldimethylchlorosilane (TBSCl) (2.25 g, 15 mmol, 1 equiv) was added, and the reaction mixture was gradually warmed to room temperature to react for 18 h. The reaction mixture was then quenched with water and extracted with ethyl acetate. The combined organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure to remove the solvent. The residue was purified by silica gel column chromatography to afford **II** as a colorless liquid (2.85 g, 93%).

O-(4-((*tert*-butyldimethylsilyl)oxy)but-2-yl)oxime (**IV**) was prepared by Method A in 13 mmol scale (2.84 g, 61%).

(E)-quinoline-8-carbaldehyde O-(4-hydroxybutan-2-yl) oxime (**V**) : **IV** (1.36 g, 3.8 mmol, 1.0 equiv) was dissolved in acetonitrile (MeCN) (4 mL), add trimethylchlorosilane (TMSCl) (96.4 μL, 0.76 mmol, 0.2 equiv), water (68.4 μL, 3.8 mmol, 1 equiv) and sodium iodide (NaI) (60 mg, 0.38 mmol, 0.1 equiv) The reactant was stirred at room temperature for 1 h. Then it was heated to reflux and reacted overnight. After the completion of the reaction, the solvent was concentrated by vacuum concentration and the residue was directly purified by silica gel column chromatography to afford **V** as a colorless liquid (0.88 g, 95%).

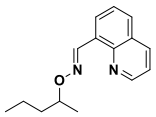
(E)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl thiophene-2-carboxylate (**VI**) : The Acyl chloride (1.33 equiv) was slowly added into a DCM solution (7 mL) of quinoline-8-carbaldehyde O-(4-hydroxybutan-2-yl) oxime (**V**) (1 equiv, 0.7 mmol, 170 mg) and pyridine (2 equiv, 111 mg) at room temperature. The mixture was stirred for 1 h, and then diluted with 1M NaOH (10 mL), extracted with DCM and washed with water and brine. The combined organic extracts were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel to give the title compounds.



1a

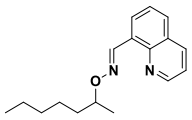
(E)-quinoline-8-carbaldehyde O-(sec-butyl) oxime (**1a**) : Colorless oil (1.50 g, 66%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.39 (s, 1H), 8.92 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.28 (dd, *J* = 7.3, 1.0 Hz, 1H), 8.15 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.83 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.42 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.33 (h, *J* = 6.3 Hz, 1H), 1.84–1.74 (m,

1H), 1.61 (ddd, *J* = 14.6, 10.5, 6.9 Hz, 1H), 1.33 (d, *J* = 6.3 Hz, 3H), 1.00 (t, *J* = 7.5 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



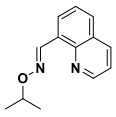
1b

(E)-quinoline-8-carbaldehyde O-pentan-2-yl oxime (**1b**) : Colorless oil (1.40 g, 58%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.39 (s, 1H), 8.92 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.28 (dd, *J* = 7.3, 1.3 Hz, 1H), 8.14 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.83 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.42 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.45–4.35 (m, 1H), 1.83–1.71 (m, 1H), 1.59–1.40 (m, 3H), 1.34 (d, *J* = 6.3 Hz, 3H), 0.96 (t, *J* = 7.2 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



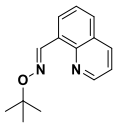
1c

(E)-quinoline-8-carbaldehyde O-heptan-2-yl oxime (**1c**) : Colorless oil (1.30 g, 48%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.39 (s, 1H), 8.93 (dd, *J* = 4.1, 1.6 Hz, 1H), 8.28 (d, *J* = 7.2 Hz, 1H), 8.15 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.83 (d, *J* = 7.5 Hz, 1H), 7.56 (t, *J* = 7.7 Hz, 1H), 7.42 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.38 (h, *J* = 6.3 Hz, 1H), 1.82–1.72 (m, 1H), 1.55 (ddd, *J* = 18.9, 10.3, 5.1 Hz, 1H), 1.51–1.39 (m, 2H), 1.34 (d, *J* = 6.3 Hz, 7H), 0.90 (t, *J* = 6.8 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



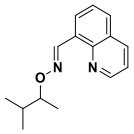
1d

(E)-quinoline-8-carbaldehyde O-isopropyl oxime (**1d**) : Colorless oil (1.35 g, 63%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.38 (s, 1H), 8.92 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.27 (dd, *J* = 7.3, 1.2 Hz, 1H), 8.15 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.83 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.42 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.57–4.50 (m, 1H), 1.35 (d, *J* = 6.3 Hz, 6H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



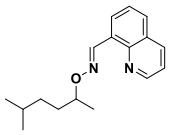
1e

(E)-quinoline-8-carbaldehyde O-(*tert*-butyl) oxime (**1e**) : Colorless oil (0.93 g, 41%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.37 (s, 1H), 8.92 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.30 (dd, *J* = 7.3, 1.5 Hz, 1H), 8.15 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.83 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.56 (t, *J* = 7.7 Hz, 1H), 7.42 (dd, *J* = 8.3, 4.2 Hz, 1H), 1.41 (s, 9H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



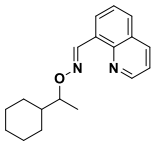
1f

(E)-quinoline-8-carbaldehyde O-(3-methylbutan-2-yl) oxime (**1f**) : Colorless oil (1.57 g, 65%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.40 (s, 1H), 8.93 (dd, *J* = 4.1, 1.6 Hz, 1H), 8.29 (dd, *J* = 7.3, 0.8 Hz, 1H), 8.15 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.83 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.56 (t, *J* = 7.7 Hz, 1H), 7.42 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.20 (p, *J* = 6.3 Hz, 1H), 2.01 (dq, *J* = 13.6, 6.8 Hz, 1H), 1.28 (d, *J* = 6.4 Hz, 3H), 1.00 (d, *J* = 6.8 Hz, 3H), 0.97 (d, *J* = 6.9 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



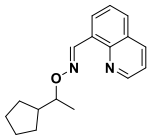
1g

(E)-quinoline-8-carbaldehyde O-(5-methylhexan-2-yl) oxime (**1g**) : Colorless oil (1.78 g, 66%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.39 (s, 1H), 8.92 (d, *J* = 3.9 Hz, 1H), 8.28 (d, *J* = 7.3 Hz, 1H), 8.14 (d, *J* = 8.3 Hz, 1H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.41 (dd, *J* = 8.2, 4.1 Hz, 1H), 4.36 (dt, *J* = 12.4, 6.2 Hz, 1H), 1.83–1.73 (m, 1H), 1.57 (tt, *J* = 12.7, 6.3 Hz, 2H), 1.41–1.36 (m, 1H), 1.34 (d, *J* = 6.1 Hz, 3H), 1.30–1.26 (m, 1H), 0.91 (d, *J* = 6.6 Hz, 6H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



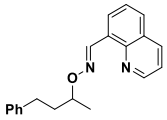
1h

(E)-quinoline-8-carbaldehyde O-(1-cyclohexylethyl) oxime (**1h**) : Colorless oil (1.38 g, 49%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.39 (s, 1H), 8.92 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.28 (dd, *J* = 7.4, 1.4 Hz, 1H), 8.14 (dt, *J* = 8.2, 1.8 Hz, 1H), 7.82 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.41 (ddd, *J* = 8.5, 4.3, 1.5 Hz, 1H), 4.19 (p, *J* = 6.3 Hz, 1H), 1.95–1.88 (m, 1H), 1.81–1.73 (m, 4H), 1.66 (dddd, *J* = 14.7, 11.7, 5.5, 2.6 Hz, 2H), 1.29 (d, *J* = 6.5 Hz, 4H), 1.20–1.04 (m, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



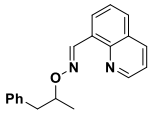
1i

(E)-quinoline-8-carbaldehyde O-(1-cyclopentylethyl) oxime (**1i**) : Colorless oil (1.13 g, 42%).^{1}H NMR (500 MHz, $CDCl_3$) δ 9.38 (s, 1H), 8.92 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.28 (dd, *J* = 7.3, 1.4 Hz, 1H), 8.14 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.82 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.41 (dd, *J* = 8.3, 4.1 Hz, 1H), 4.22 (dq, *J* = 8.1, 6.3 Hz, 1H), 2.11 (dd, *J* = 16.6, 8.3 Hz, 1H), 1.90–1.82 (m, 1H), 1.80–1.70 (m, 2H), 1.68–1.61 (m, 2H), 1.56 (dddd, *J* = 9.0, 7.4, 4.3, 2.8 Hz, 2H), 1.50–1.43 (m, 1H), 1.35 (d, *J* = 6.3 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



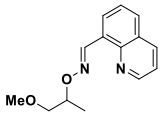
1j

(E)-quinoline-8-carbaldehyde O-(4-phenylbutan-2-yl) oxime (**1j**) : Colorless oil (2.07 g, 68%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.43 (s, 1H), 8.94 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.33–8.25 (m, 1H), 8.17 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.85 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.27 (dt, *J* = 13.9, 7.3 Hz, 4H), 7.19 (d, *J* = 7.2 Hz, 1H), 4.47–4.39 (m, 1H), 2.80 (dddd, *J* = 30.3, 13.8, 10.2, 5.9 Hz, 2H), 2.11 (dddd, *J* = 13.2, 10.2, 7.3, 5.7 Hz, 1H), 1.95–1.84 (m, 1H), 1.39 (d, *J* = 6.3 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



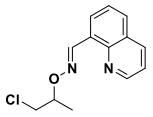
1k

(E)-quinoline-8-carbaldehyde O-(1-phenylpropan-2-yl) oxime (**1k**) : Colorless oil (1.80 g, 62%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.42 (s, 1H), 8.94 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.29 (dd, *J* = 7.3, 1.0 Hz, 1H), 8.16 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.85 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.33–7.27 (m, 4H), 7.22 (ddd, *J* = 8.6, 5.3, 2.1 Hz, 1H), 4.69–4.59 (m, 1H), 3.20 (dd, *J* = 13.6, 5.8 Hz, 1H), 2.86 (dd, *J* = 13.6, 7.1 Hz, 1H), 1.32 (d, *J* = 6.3 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



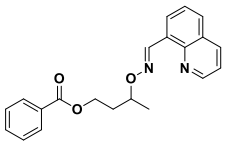
1l

(E)-quinoline-8-carbaldehyde O-(1-methoxypropan-2-yl) oxime (**1l**) : Colorless oil (1.15 g, 47%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.43 (s, 1H), 8.92 (dd, *J* = 4.1, 1.6 Hz, 1H), 8.27 (d, *J* = 7.3 Hz, 1H), 8.15 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.84 (d, *J* = 8.1 Hz, 1H), 7.56 (t, *J* = 7.7 Hz, 1H), 7.42 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.62–4.51 (m, 1H), 3.65 (dd, *J* = 10.4, 5.8 Hz, 1H), 3.55 (dd, *J* = 10.4, 4.4 Hz, 1H), 3.43 (s, 3H), 1.36 (d, *J* = 6.5 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



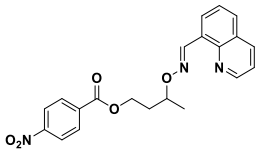
1m

(E)-quinoline-8-carbaldehyde O-(1-chloropropan-2-yl) oxime (**1m**) : Colorless oil (1.41 g, 57%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.44 (s, 1H), 8.92 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.26 (dd, *J* = 7.3, 1.2 Hz, 1H), 8.16 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.86 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.56 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.59 (td, *J* = 6.3, 4.5 Hz, 1H), 3.82 (dd, *J* = 11.1, 4.4 Hz, 1H), 3.70 (dd, *J* = 11.1, 6.2 Hz, 1H), 1.45 (d, *J* = 6.4 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



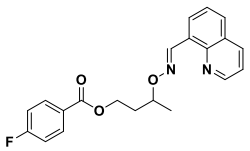
1n

(E)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl benzoate (**1n**) : Colorless oil (187.6 mg, 77%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.41 (s, 1H), 8.97–8.88 (m, 1H), 8.27 (d, *J* = 7.3 Hz, 1H), 8.16 (d, *J* = 8.2 Hz, 1H), 8.07 (d, *J* = 7.7 Hz, 2H), 7.85 (d, *J* = 8.1 Hz, 1H), 7.61–7.49 (m, 2H), 7.46–7.39 (m, 3H), 4.69–4.58 (m, 1H), 4.57–4.46 (m, 2H), 2.24 (td, *J* = 13.8, 6.4 Hz, 1H), 2.15–2.04 (m, 1H), 1.44 (d, *J* = 6.4 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



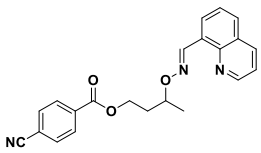
1o

(E)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 4-nitrobenzoate (**1o**) : White solid (217.3 mg, 79%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.37 (s, 1H), 8.92 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.28–8.18 (m, 5H), 8.16 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.85 (dd, *J* = 8.1, 1.0 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.61 (ddd, *J* = 20.3, 9.6, 5.5 Hz, 3H), 2.24 (td, *J* = 14.1, 6.1 Hz, 1H), 2.13 (dtd, *J* = 11.3, 6.9, 4.6 Hz, 1H), 1.45 (d, *J* = 6.4 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



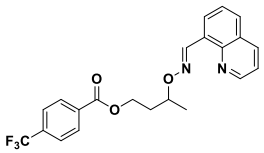
1p

(E)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 4-fluorobenzoate (**1p**) : Colorless oil (192.2 mg, 75%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.39 (s, 1H), 8.94 – 8.91 (m, 1H), 8.26 (d, *J* = 7.2 Hz, 1H), 8.15 (d, *J* = 8.3 Hz, 1H), 8.07 (dd, *J* = 8.8, 5.5 Hz, 2H), 7.84 (d, *J* = 8.1 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.1 Hz, 1H), 7.08 (t, *J* = 8.7 Hz, 2H), 4.61 (dd, *J* = 12.4, 6.4 Hz, 1H), 4.51 (td, *J* = 6.7, 2.2 Hz, 2H), 2.22 (dt, *J* = 13.8, 7.0 Hz, 1H), 2.14 – 2.03 (m, 1H), 1.44 (d, *J* = 6.4 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



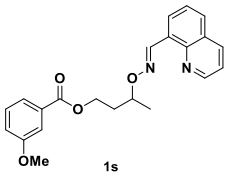
1q

(E)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 4-cyanobenzoate (**1q**) : Colorless oil (174.9 mg, 67%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.36 (s, 1H), 8.92 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.24 (dd, *J* = 7.3, 1.0 Hz, 1H), 8.21–8.09 (m, 3H), 7.84 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.68 (d, *J* = 8.5 Hz, 2H), 7.55 (d, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.64–4.59 (m, 1H), 4.55 (dd, *J* = 9.7, 3.6 Hz, 2H), 2.27–2.17 (m, 1H), 2.15–2.07 (m, 1H), 1.43 (d, *J* = 6.4 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



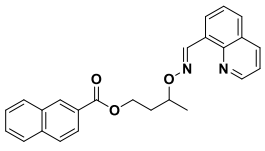
1r

(E)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 4-(trifluoromethyl)benzoate (**1r**) : Colorless oil (233.0 mg, 80%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.39 (s, 1H), 8.92 (dd, *J* = 4.1, 1.6 Hz, 1H), 8.25 (dd, *J* = 7.3, 1.3 Hz, 1H), 8.16 (dd, *J* = 8.5, 3.0 Hz, 3H), 7.84 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.67 (d, *J* = 8.1 Hz, 2H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.1 Hz, 1H), 4.66–4.59 (m, 1H), 4.56 (t, *J* = 6.2 Hz, 2H), 2.24 (dq, *J* = 13.9, 6.4 Hz, 1H), 2.12 (dtd, *J* = 14.3, 7.0, 4.6 Hz, 1H), 1.44 (d, *J* = 6.4 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



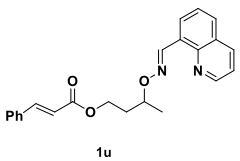
1s

(E)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 3-methoxybenzoate (**1s**) : Colorless oil (203.7 mg, 77%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.40 (s, 1H), 8.93 (dd, *J* = 3.9, 1.8 Hz, 1H), 8.26 (d, *J* = 7.3 Hz, 1H), 8.15 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.84 (d, *J* = 8.1 Hz, 1H), 7.66 (d, *J* = 7.6 Hz, 1H), 7.60–7.53 (m, 2H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.33 (t, *J* = 7.9 Hz, 1H), 7.12–7.06 (m, 1H), 4.63 (p, *J* = 6.4 Hz, 1H), 4.52 (h, *J* = 4.7 Hz, 2H), 3.84 (d, *J* = 1.3 Hz, 3H), 2.24 (dq, *J* = 13.8, 6.7 Hz, 1H), 2.16–2.04 (m, 1H), 1.44 (dd, *J* = 6.5, 1.2 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



1t

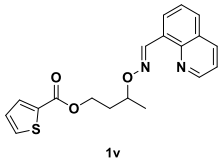
(E)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 2-naphthoate (**1t**) : Colorless oil (214.5 mg, 77%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.42 (s, 1H), 8.91 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.62 (d, *J* = 1.6 Hz, 1H), 8.27 (dd, *J* = 7.3, 1.4 Hz, 1H), 8.14 (dd, *J* = 8.3, 1.8 Hz, 1H), 8.08 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.89–7.81 (m, 3H), 7.61–7.50 (m, 3H), 7.42 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.68 (ddd, *J* = 7.9, 6.4, 4.9 Hz, 1H), 4.64–4.56 (m, 2H), 2.29 (ddt, *J* = 14.0, 7.8, 6.2 Hz, 1H), 2.16 (dtd, *J* = 14.3, 7.0, 4.8 Hz, 1H), 1.47 (d, *J* = 6.4 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



1u

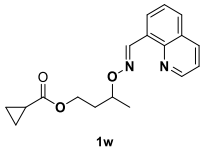
3-((((E)-quinolin-8-ylmethylene)amino)oxy)butyl cinnamate (**1u**) : Colorless oil (191.1 mg, 73%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.40 (s, 1H), 8.91 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.28 (dd, *J* = 7.3, 1.5 Hz, 1H), 8.15 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.84 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.69 (d, *J* = 16.0 Hz, 1H), 7.57–7.53 (m, 1H), 7.52–7.48 (m, 2H), 7.42 (dd, *J* =

8.3, 4.2 Hz, 1H), 7.38–7.36 (m, 3H), 6.45 (d, *J* = 16.1 Hz, 1H), 4.58 (ddd, *J* = 7.8, 6.3, 4.9 Hz, 1H), 4.41 (td, *J* = 6.6, 3.1 Hz, 2H), 2.24–2.14 (m, 1H), 2.08–1.99 (m, 1H), 1.42 (d, *J* = 6.3 Hz, 3H). The spectral data of the product was in accordance with that reported in the literature.[1,2]



1v

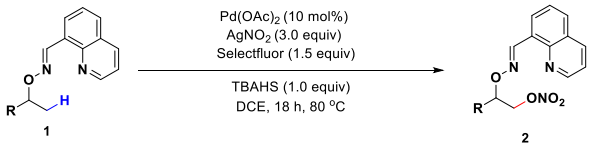
(E)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl thiophene-2-carboxylate (**1v**) : Colorless oil (161.1 mg, 65%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.39 (s, 1H), 8.92 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.26 (dd, *J* = 7.3, 1.4 Hz, 1H), 8.15 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.90–7.75 (m, 2H), 7.61–7.51 (m, 2H), 7.42 (dd, *J* = 8.2, 4.1 Hz, 1H), 7.08 (dd, *J* = 4.9, 3.7 Hz, 1H), 4.64–4.56 (m, 1H), 4.54–4.45 (m, 2H), 2.27–2.17 (m, 1H), 2.12–2.02 (m, 1H), 1.43 (d, *J* = 6.4 Hz, 3H). The The spectral data of the product was in accordance with that reported in the literature.[1,2]



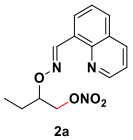
1w

(E)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl cyclopropanecarboxylate (**1w**) : Colorless oil (157.2 mg, 72%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.38 (s, 1H), 8.92 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.27 (dd, *J* = 7.3, 1.4 Hz, 1H), 8.15 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.84 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.56 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.57–4.49 (m, 1H), 4.30–4.22 (m, 2H), 2.10 (ddt, *J* = 14.0, 7.8, 6.3 Hz, 1H), 1.94 (dtd, *J* = 14.3, 7.1, 4.9 Hz, 1H), 1.62 (tt, *J* = 8.0, 4.6 Hz, 1H), 1.39 (d, *J* = 6.4 Hz, 3H), 0.99 (dt, *J* = 4.6, 3.2 Hz, 2H), 0.84 (dq, *J* = 6.8, 3.8 Hz, 2H). The spectral data of the product was in accordance with that reported in the literature.[1,2]

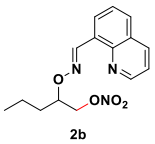
Scope of C(sp^3)–H Nitrooxylation



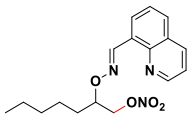
General Procedure for the Nitrooxylation of Alcohols : An oven dried 25 mL Schlenk tube, equipped with a stir bar, was charged with substrates **1a**-**1w** (0.1 mmol), $Pd(OAc)_2$ (10 mol %), $AgNO_2$ (3.0 equiv), Selectfluor (1.5 equiv), TBAHS (1.0 equiv) and DCE (2 mL). The tube was capped except for special requirement. Then the reaction mixture was stirred at 80 °C for 18 h. Upon completion, EtOAc was added to dilute the mixture and then filtered through a pad of silica gel. The solvents were evaporated under reduced pressure and then purified by preparative thin-layer chromatography.



(E)-2-(((quinolin-8-ylmethylene)amino)oxy)butyl nitrate (**2a**) : Substrate **1a** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2a** was obtained as colorless oil (19.6 mg, 68%). For 1 mmol reaction, **1a** (1 mmol, 228 mg), $Pd(OAc)_2$ (10 mol %, 22.4 mg), $AgNO_2$ (3 equiv, 450 mg), Selectfluor (1.5 equiv, 530 mg) and TBAHS (1.0 equiv, 340 mg) were used and followed the general procedure to afford 144.4 mg (50%) of **2a**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.44 (s, 1H), 8.93 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.26 (dd, *J* = 7.3, 1.2 Hz, 1H), 8.17 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.87 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.71 (dd, *J* = 5.1, 1.2 Hz, 2H), 4.47 (dd, *J* = 7.7, 4.9 Hz, 1H), 1.84 (dd, *J* = 14.7, 7.2 Hz, 1H), 1.80–1.71 (m, 1H), 1.07 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 147.1, 146.0, 136.3, 129.9, 129.8, 128.4, 126.5, 126.4, 121.6, 80.7, 73.6, 24.0, 9.8. HRMS (ESI-TOF) *m/z* calcd for $C_{14}H_{16}N_3O_4$ $[M+H]^+$ 290.1141 found 290.1137.

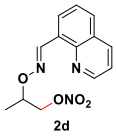


(E)-2-(((quinolin-8-ylmethylene)amino)oxy)pentyl nitrate (**2b**) : Substrate **1b** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2b** was obtained as colorless oil (26.4 mg, 87%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.43 (s, 1H), 8.93 (dd, *J* = 4.1, 1.8 Hz, 1H), 8.26 (dd, *J* = 7.3, 1.2 Hz, 1H), 8.17 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.87 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.75–4.66 (m, 2H), 4.54 (dt, *J* = 13.2, 5.0 Hz, 1H), 1.87–1.77 (m, 1H), 1.71–1.58 (m, 2H), 1.54–1.44 (m, 1H), 0.99 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 147.1, 146.1, 136.3, 130.0, 129.8, 128.4, 126.5, 126.4, 121.6, 79.3, 74.0, 32.9, 18.7, 14.1. HRMS (ESI-TOF) *m/z* calcd for $C_{15}H_{18}N_3O_4$ $[M+H]^+$ 304.1297, found 304.1291.



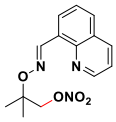
2c

(E)-2-(((quinolin-8-ylmethylene)amino)oxy)heptyl nitrate **(2c)** : Substrate **1c** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2c** was obtained as colorless oil (24.2 mg, 73%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.43 (s, 1H), 8.93 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.26 (dd, *J* = 7.3, 0.8 Hz, 1H), 8.16 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.86 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.75–4.65 (m, 2H), 4.53 (td, *J* = 9.8, 5.2 Hz, 1H), 1.88–1.76 (m, 1H), 1.73–1.63 (m, 1H), 1.60–1.50 (m, 1H), 1.50–1.41 (m, 1H), 1.39–1.29 (m, 4H), 0.90 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 147.0, 146.0, 136.4, 129.9, 129.8, 128.4, 126.5, 126.4, 121.6, 79.5, 74.0, 31.8, 30.8, 25.1, 22.6, 14.1. HRMS (ESI-TOF) *m/z* calcd for $C_{17}H_{22}N_3O_4$ $[M+H]^+$ 332.1610, found 332.1606.



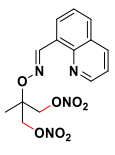
2d

(E)-2-(((quinolin-8-ylmethylene)amino)oxy)propyl nitrate (**2d**) : Substrate **1d** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2d** was obtained as colorless oil (18.7 mg, 68%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.42 (s, 1H), 8.93 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.29–8.22 (m, 1H), 8.17 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.87 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.73–4.62 (m, 3H), 1.43 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 147.3, 146.0, 136.3, 129.9, 128.4, 126.5, 126.4, 121.6, 75.6, 74.7, 16.6. HRMS (ESI-TOF) *m/z* calcd for $C_{13}H_{14}N_3O_4$ $[M+H]^+$ 276.0984, found 276.0975.



2e

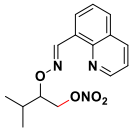
(E)-2-methyl-2-(((quinolin-8-ylmethylene)amino)oxy)propyl nitrate (**2e**) : Substrate **1e** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2e** was obtained as yellow oil (16.2 mg, 56%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.41 (s, 1H), 8.93 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.26 (dd, *J* = 7.3, 1.4 Hz, 1H), 8.17 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.87 (dt, *J* = 8.3, 2.3 Hz, 1H), 7.59 (dt, *J* = 12.5, 7.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.69 (s, 2H), 1.47 (d, *J* = 6.1 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 146.7, 146.0, 136.3, 130.3, 129.7, 128.4, 126.5, 126.3, 121.6, 78.7, 76.8, 23.5. HRMS (ESI-TOF) *m/z* calcd for $C_{14}H_{16}N_3O_4$ $[M+H]^+$ 290.1141, found 290.1135.



2e'

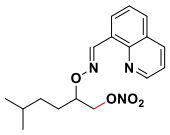
(E)-2-methyl-2-(((quinolin-8-ylmethylene)amino)oxy)propane-1,3-diyl dinitrate (**2e'**) : Substrate **1e** was nitrooxylated twice when $AgNO_2$ was added excessively. After purification by preparative thin-layer

chromatography (Hexane : EtOAc = 6:1 as eluent), **2e'** was obtained as yellow oil (8.0 mg, 23%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.45 (s, 1H), 8.98–8.93 (m, 1H), 8.22 (dd, *J* = 7.3, 1.4 Hz, 1H), 8.19 (ddd, *J* = 8.3, 4.7, 1.8 Hz, 1H), 7.91 (dt, *J* = 8.0, 1.7 Hz, 1H), 7.60 (dt, *J* = 11.0, 7.7 Hz, 1H), 7.47 (ddd, *J* = 8.3, 5.6, 4.2 Hz, 1H), 4.82 (dd, *J* = 11.5, 8.8 Hz, 2H), 4.75 (dd, *J* = 11.4, 6.9 Hz, 2H), 1.55 (d, *J* = 3.8 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.4, 148.6, 146.0, 136.4, 130.3, 129.4, 128.4, 126.5, 126.4, 121.7, 79.0, 73.2, 29.8, 19.3. HRMS (ESI-TOF) *m/z* calcd for $C_{14}H_{15}N_4O_7$ $[M+H]^+$ 351.0940, found 351.0935.



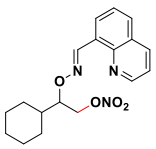
2f

(E)-3-methyl-2-(((quinolin-8-ylmethylene)amino)oxy)butyl nitrate (**2f**) : Substrate **1f** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2f** was obtained as colorless oil (21.8 mg, 72%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.44 (s, 1H), 8.92 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.26 (dd, *J* = 7.3, 0.8 Hz, 1H), 8.15 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.85 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.56 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.79 (dd, *J* = 11.9, 3.5 Hz, 1H), 4.70 (dd, *J* = 11.9, 6.5 Hz, 1H), 4.32 (td, *J* = 6.4, 3.6 Hz, 1H), 2.15 (dq, *J* = 13.6, 6.8 Hz, 1H), 1.07 (dd, *J* = 11.0, 6.9 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.1, 146.8, 146.0, 136.4, 129.9, 129.8, 128.4, 126.5, 126.3, 121.6, 84.2, 72.7, 29.5, 18.6, 18.3. HRMS (ESI-TOF) *m/z* calcd for $C_{15}H_{18}N_3O_4$ $[M+H]^+$ 304.1297, found 304.1290.



2g

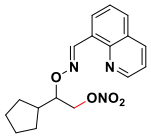
(E)-5-methyl-2-(((quinolin-8-ylmethylene)amino)oxy)hexyl nitrate (**2g**) : Substrate *1g* was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2g** was obtained as colorless oil (26.8 mg, 81%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.43 (s, 1H), 8.93 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.26 (dd, *J* = 7.3, 1.0 Hz, 1H), 8.16 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.86 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.71 (qd, *J* = 11.8, 5.0 Hz, 2H), 4.51 (dt, *J* = 9.7, 5.4 Hz, 1H), 1.87–1.76 (m, 1H), 1.69 (ddt, *J* = 13.9, 10.8, 5.3 Hz, 1H), 1.60 (dq, *J* = 13.3, 6.6 Hz, 1H), 1.45 (dddd, *J* = 16.1, 11.5, 6.8, 5.0 Hz, 1H), 1.32 (ddd, *J* = 18.2, 12.2, 5.9 Hz, 1H), 0.92 (dd, *J* = 6.6, 1.2 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 147.0, 146.0, 136.4, 129.9, 129.8, 128.4, 126.5, 126.3, 121.6, 79.8, 74.0, 34.4, 28.7, 28.1, 22.7, 22.6. HRMS (ESI-TOF) *m/z* calcd for $C_{17}H_{22}N_3O_4$ $[M+H]^+$ 332.1610, found 332.1603.



2h

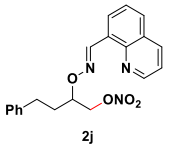
(E)-2-cyclohexyl-2-(((quinolin-8-ylmethylene)amino)oxy)ethyl nitrate (**2h**) : Substrate **1h** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2h** was obtained as yellow oil (21.6 mg, 63%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.42 (s, 1H), 8.94 (dd, *J* = 4.1, 1.8 Hz, 1H), 8.26 (dd, *J* = 7.3, 1.3 Hz, 1H), 8.17 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.86 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.81 (dd, *J* = 11.9, 3.6 Hz, 1H), 4.70 (dd, *J* = 11.8, 6.4 Hz, 1H), 4.33 (td, *J* = 6.5, 3.5 Hz, 1H), 1.96 (d, *J* = 13.0 Hz, 1H), 1.79 (dt, *J* = 12.3, 3.7 Hz, 4H), 1.70 (d, *J* =

12.7 Hz, 2H), 1.20 (ddd, *J* = 11.8, 5.4, 2.8 Hz, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.1, 146.7, 145.9, 136.4, 129.9, 129.8, 128.4, 126.5, 126.4, 121.6, 83.7, 72.6, 39.1, 29.0, 28.7, 26.4, 26.2, 26.1. HRMS (ESI-TOF) *m/z* calcd for $C_{18}H_{22}N_3O_4$ $[M+H]^+$ 344.1610, found 344.1603.



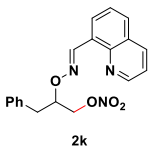
2i

(E)-2-cyclopentyl-2-(((quinolin-8-ylmethylene)amino)oxy)ethyl nitrate (**2i**) : Substrate **1i** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2i** was obtained as yellow oil (19.7 mg, 60%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.42 (s, 1H), 8.93 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.25 (dd, *J* = 7.3, 1.4 Hz, 1H), 8.16 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.86 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.82 (dd, *J* = 11.9, 3.3 Hz, 1H), 4.73–4.66 (m, 1H), 4.37 (ddd, *J* = 8.4, 6.1, 3.3 Hz, 1H), 2.25 (dt, *J* = 8.0, 6.6 Hz, 1H), 1.93–1.87 (m, 1H), 1.81 (ddd, *J* = 12.1, 7.5, 3.5 Hz, 1H), 1.72–1.65 (m, 2H), 1.62–1.53 (m, 3H), 1.44–1.37 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 146.7, 136.4, 130.0, 129.8, 128.4, 126.5, 126.4, 121.6, 83.5, 73.8, 40.7, 29.8, 29.2, 29.2, 25.7, 25.5. HRMS (ESI-TOF) *m/z* calcd for $C_{17}H_{20}N_3O_4$ $[M+H]^+$ 330.1454, found 330.1451.



2j

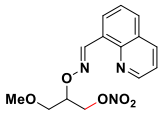
(E)-4-phenyl-2-(((quinolin-8-ylmethylene)amino)oxy)butyl nitrate **(2j)** : Substrate **1j** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2j** was obtained as colorless oil (24.5 mg, 67%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.50 (s, 1H), 8.95 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.31–8.24 (m, 1H), 8.17 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.88 (dd, *J* = 8.1, 0.8 Hz, 1H), 7.59 (t, *J* = 7.7 Hz, 1H), 7.45 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.23 (dd, *J* = 18.2, 7.2 Hz, 3H), 4.77–4.67 (m, 2H), 4.56 (dq, *J* = 9.3, 4.7 Hz, 1H), 2.97–2.86 (m, 1H), 2.85–2.73 (m, 1H), 2.26–2.12 (m, 1H), 2.00 (dddd, J = 11.4, 9.7, 7.0, 4.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 147.3, 146.0, 141.3, 136.4, 129.9, 129.8, 128.6, 128.4, 126.5, 126.4, 126.2, 121.6, 78.6, 73.8, 32.5, 31.6. HRMS (ESI-TOF) *m/z* calcd for $C_{20}H_{20}N_3O_4$ $[M+H]^+$ 366.1454, found 366.1448.



2k

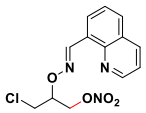
(E)-3-phenyl-2-(((quinolin-8-ylmethylene)amino)oxy)propyl nitrate (**2k**) : Substrate **1k** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2k** was obtained as colorless oil (18.3 mg, 52%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.46 (s, 1H), 8.94 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.27 (dd, *J* = 7.3, 0.9 Hz, 1H), 8.16 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.88 (dd, *J* = 8.1, 1.0 Hz, 1H), 7.58 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.37–7.27 (m, 4H), 7.28–7.24 (m, 1H), 4.80 (dd, *J* = 6.1, 3.8 Hz, 1H), 4.73 (dd, *J* = 11.7, 3.7 Hz, 1H), 4.63 (dd, *J* = 11.7, 5.9 Hz, 1H), 3.23 (dd, *J* = 14.1, 6.7 Hz, 1H), 3.05 (dd, *J* = 14.1, 7.0 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 147.5, 146.0, 136.8, 136.4, 130.0, 129.8,

129.6, 128.8, 128.4, 126.9, 126.5, 126.4, 121.6, 80.2, 72.7, 37.1. HRMS (ESI-TOF) *m/z* calcd for $C_{19}H_{18}N_3O_4$ [M+H]+ 352.1297, found 352.1291.



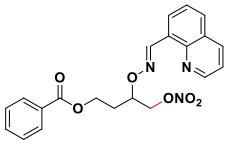
2l

(E)-3-methoxy-2-(((quinolin-8-ylmethylene)amino)oxy)propyl nitrate (**2l**) : Substrate **1l** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2l** was obtained as colorless oil (16.8 mg, 55%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.46 (s, 1H), 8.95 (s, 1H), 8.24 (d, *J* = 7.2 Hz, 1H), 8.18 (d, *J* = 8.1 Hz, 1H), 7.88 (d, *J* = 8.1 Hz, 1H), 7.58 (t, *J* = 7.7 Hz, 1H), 7.45 (dd, *J* = 8.2, 4.1 Hz, 1H), 4.82 (ddd, *J* = 17.9, 11.8, 5.1 Hz, 2H), 4.76–4.63 (m, 1H), 3.85–3.64 (m, 2H), 3.44 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.3, 148.0, 146.0, 136.3, 130.0, 129.7, 128.4, 126.5, 126.4, 121.6, 78.5, 71.6, 70.9, 59.7. HRMS (ESI-TOF) *m/z* calcd for $C_{14}H_{16}N_3O_5$ $[M+H]^+$ 306.1090, found 306.1085.



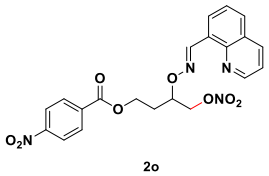
2m

(E)-3-chloro-2-(((quinolin-8-ylmethylene)amino)oxy)propyl nitrate (**2m**) : Substrate **1m** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2m** was obtained as colorless oil (15.5 mg, 50%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.48 (s, 1H), 8.93 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.24 (dd, *J* = 7.3, 1.1 Hz, 1H), 8.17 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.89 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.58 (t, *J* = 7.7 Hz, 1H), 7.45 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.87 (ddd, *J* = 17.8, 12.0, 4.9 Hz, 2H), 4.79–4.72 (m, 1H), 3.87 (qd, *J* = 11.6, 5.7 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.4, 148.7, 146.0, 136.4, 130.3, 129.3, 128.4, 126.6, 126.4, 121.7, 78.8, 70.8, 41.9. HRMS (ESI-TOF) *m/z* calcd for $C_{13}H_{13}ClN_3O_4$ $[M+H]^+$ 310.0594, found 310.0590.



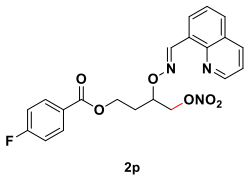
2n

(E)-4-(nitrooxy)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl benzoate(**2n**) : Substrate **1n** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2n** was obtained as colorless oil (25.8 mg, 63%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.46 (s, 1H), 8.92 (dd, *J* = 4.1, 1.6 Hz, 1H), 8.25–8.20 (m, 1H), 8.15 (dd, *J* = 8.3, 1.4 Hz, 1H), 8.08–8.01 (m, 2H), 7.86 (d, *J* = 8.1 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 2H), 7.44 (dd, *J* = 10.4, 4.5 Hz, 3H), 4.81 (dt, *J* = 14.9, 7.5 Hz, 3H), 4.62–4.47 (m, 2H), 2.36–2.26 (m, 1H), 2.25–2.14 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.5, 150.2, 147.7, 145.9, 136.3, 133.2, 130.1, 130.0, 129.7, 129.5, 128.5, 128.3, 126.4, 121.6, 76.6, 73.5, 61.2, 30.2. HRMS (ESI-TOF) *m/z* calcd for $C_{21}H_{20}N_3O_6$ $[M+H]^+$ 410.1352, found 410.1347.



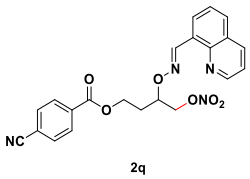
2o

(E)-4-(nitrooxy)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 4-nitrobenzoate (**2o**) : Substrate **1o** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2o** was obtained as white solid (19.1 mg, 42%), m.p. 93 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.40 (s, 1H), 8.90 (dd, *J* = 4.1, 1.6 Hz, 1H), 8.18 (ddd, *J* = 20.1, 9.9, 4.1 Hz, 6H), 7.87 (d, *J* = 7.4 Hz, 1H), 7.54 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.78 (dt, *J* = 14.1, 4.3 Hz, 3H), 4.61 (tt, *J* = 5.9, 3.9 Hz, 2H), 2.40–2.29 (m, 1H), 2.26–2.16 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.6, 150.6, 150.3, 147.8, 145.9, 136.4, 135.5, 130.8, 130.2, 129.4, 128.4, 126.4, 126.3, 123.6, 121.7, 76.7, 73.4, 62.3, 30.1. HRMS (ESI-TOF) *m/z* calcd for $C_{21}H_{19}N_4O_8$ $[M+H]^+$ 455.1203, found 455.1199.



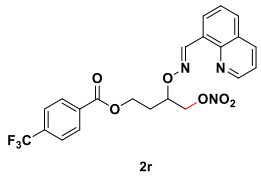
2p

(E)-4-(nitrooxy)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 4-fluorobenzoate (**2p**) : Substrate **1p** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2p** was obtained as yellow oil (21.8 mg, 51%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.44 (s, 1H), 8.98–8.92 (m, 1H), 8.22 (dd, *J* = 7.3, 1.3 Hz, 1H), 8.16 (dd, *J* = 8.3, 1.8 Hz, 1H), 8.11–8.03 (m, 2H), 7.87 (d, *J* = 8.2 Hz, 1H), 7.55 (t, *J* = 7.9 Hz, 1H), 7.47–7.42 (m, 1H), 7.14–7.06 (m, 2H), 4.89–4.79 (m, 3H), 4.62–4.49 (m, 2H), 2.39–2.27 (m, 1H), 2.20 (dddd, *J* = 11.3, 7.5, 5.9, 3.7 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.9 (d, *J* = 254.0 Hz), 165.5, 150.2, 147.7, 145.9, 136.3, 132.2 (d, *J* = 9.3 Hz), 130.0, 129.5, 128.3, 126.4 (d, *J* = 3.1 Hz), 126.4, 126.4, 121.6, 115.6 (d, *J* = 22.0 Hz), 76.6, 73.5, 61.3, 30.2. ^{19}F NMR (471 MHz, $CDCl_3$) δ -105.53 (dq, *J* = 8.3, 5.4 Hz, 1F). HRMS (ESI-TOF) *m/z* calcd for $C_{21}H_{19}FN_3O_6$ $[M+H]^+$ 428.1258, found 428.1248.



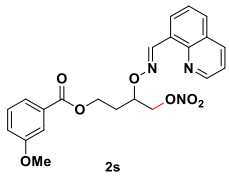
2q

(E)-4-(nitrooxy)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 4-cyanobenzoate (**2q**) : Substrate **1q** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2q** was obtained as yellow oil (26.9 mg, 62%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.41 (s, 1H), 8.92 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.20 (dd, *J* = 7.4, 1.3 Hz, 1H), 8.17 (dd, *J* = 8.4, 1.7 Hz, 1H), 8.12 (d, *J* = 8.1 Hz, 2H), 7.90–7.85 (m, 1H), 7.69 (d, *J* = 8.1 Hz, 2H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.45 (dd, *J* = 8.4, 4.2 Hz, 1H), 4.76 (dq, *J* = 18.4, 4.5 Hz, 3H), 4.66–4.54 (m, 2H), 2.31 (dt, *J* = 8.9, 5.7 Hz, 1H), 2.25–2.17 (m, 1H). ^{13}C NMR (126 MHz, CDCl3) δ 164.9, 150.3, 147.8, 145.9, 136.4, 134.0, 132.3, 130.2, 130.2, 129.4, 128.4, 126.4, 126.4, 121.7, 118.1, 116.5, 76.6, 73.4, 62.1, 30.1. HRMS (ESI-TOF) *m/z* calcd for $C_{22}H_{19}N_4O_6$ $[M+H]^+$ 435.1304, found 435.1296.



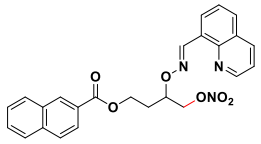
2r

(E)-4-(nitrooxy)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 4-(trifluoromethyl)benzoate (**2r**) : Substrate **1r** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2r** was obtained as yellow oil (32.9 mg, 69%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.43 (s, 1H), 8.93 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.22 (d, *J* = 7.3 Hz, 1H), 8.20–8.13 (m, 3H), 7.92–7.86 (m, 1H), 7.68 (d, *J* = 8.1 Hz, 2H), 7.56 (t, *J* = 7.7 Hz, 1H), 7.45 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.84–4.76 (m, 3H), 4.65–4.54 (m, 2H), 2.32 (ddd, *J* = 13.6, 8.4, 4.5 Hz, 1H), 2.23 (tdd, *J* = 14.0, 7.8, 4.9 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.3, 150.3, 147.9, 146.0, 136.4, 134.6 (q, *J* = 32.7 Hz), 133.4, 130.1, 129.5, 128.4, 126.4, 126.4, 125.5 (q, *J* = 3.9 Hz), 123.7 (q, *J* = 272.7 Hz), 121.7, 76.6, 73.4, 61.9, 30.2. ^{19}F NMR (471 MHz, $CDCl_3$) δ -63.12 (s, 3F). HRMS (ESI-TOF) *m/z* calcd for $C_{22}H_{19}F_3N_3O_6$ $[M+H]^+$ 478.1226, found 478.1220.



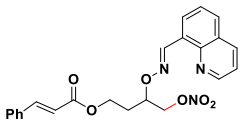
2s

(E)-4-(nitrooxy)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 3-methoxybenzoate (**2s**) : Substrate **1s** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2s** was obtained as yellow solid (31.2 mg, 71%), m.p. 84 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.45 (s, 1H), 8.94 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.23 (dd, *J* = 7.4, 1.3 Hz, 1H), 8.17 (dd, *J* = 8.4, 1.7 Hz, 1H), 7.88 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.60–7.52 (m, 2H), 7.45 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.34 (t, *J* = 7.9 Hz, 1H), 7.09 (dd, *J* = 8.3, 2.7 Hz, 1H), 4.85–4.75 (m, 3H), 4.61–4.49 (m, 2H), 3.84 (s, 3H), 2.37–2.27 (m, 1H), 2.23 (dtd, *J* = 8.1, 5.2, 4.5, 3.1 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.4, 159.7, 150.2, 147.8, 145.9, 136.4, 131.4, 130.0, 129.6, 128.4, 126.5, 126.4, 122.1, 121.6, 119.7, 114.1, 76.6, 73.5, 61.3, 55.5, 30.2. HRMS (ESI-TOF) *m/z* calcd for $C_{22}H_{22}N_3O_7$ $[M+H]^+$ 440.1458, found 440.1450.



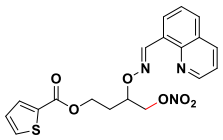
2t

(E)-4-(nitrooxy)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl 2-naphthoate (**2t**) : Substrate **1t** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2t** was obtained as yellow oil (20.2 mg, 44%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.46 (s, 1H), 8.94–8.89 (m, 1H), 8.61 (d, *J* = 1.5 Hz, 1H), 8.24 (dd, *J* = 7.3, 1.4 Hz, 1H), 8.16 (dd, *J* = 8.3, 1.7 Hz, 1H), 8.06 (dd, *J* = 8.7, 1.7 Hz, 1H), 7.94 (d, *J* = 8.2 Hz, 1H), 7.87 (dd, *J* = 8.3, 1.8 Hz, 3H), 7.63–7.50 (m, 3H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.91–4.77 (m, 3H), 4.71–4.56 (m, 2H), 2.36 (dp, *J* = 14.0, 4.9, 4.4 Hz, 1H), 2.31–2.21 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.7, 150.2, 147.7, 145.9, 136.3, 135.6, 132.5, 131.2, 130.0, 129.5, 129.5, 128.4, 128.3, 127.8, 127.3, 126.7, 126.4, 125.2, 121.5, 76.7, 73.5, 61.3, 30.2. HRMS (ESI-TOF) *m/z* calcd for $C_{25}H_{22}N_3O_6$ $[M+H]^+$ 460.1508, found 460.1502.



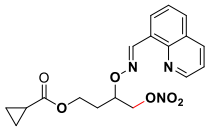
2u

4-(nitrooxy)-3-((((E)-quinolin-8-ylmethylene)amino)oxy)butyl cinnamate (**1u**) : Substrate **1u** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2u** was obtained as yellow oil (23.9 mg, 55%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.46 (d, *J* = 2.7 Hz, 1H), 8.93 (dd, *J* = 4.6, 2.4 Hz, 1H), 8.28–8.21 (m, 1H), 8.22–8.13 (m, 1H), 7.93–7.84 (m, 1H), 7.70 (dd, *J* = 15.8, 2.7 Hz, 1H), 7.59–7.54 (m, 1H), 7.52 (dd, *J* = 7.2, 3.2 Hz, 2H), 7.47–7.42 (m, 1H), 7.40–7.35 (m, 3H), 6.50–6.39 (m, 1H), 4.84–4.70 (m, 3H), 4.44 (dtdd, *J* = 15.2, 12.3, 6.3, 3.2 Hz, 2H), 2.24 (ddd, *J* = 15.0, 8.0, 4.4 Hz, 1H), 2.20–2.11 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.9, 150.2, 147.8, 145.9, 145.3, 136.4, 134.4, 130.5, 130.0, 129.6, 129.0, 128.4, 128.2, 126.5, 126.5, 121.6, 117.9, 76.6, 73.5, 60.8, 30.2. HRMS (ESI-TOF) *m/z* calcd for $C_{23}H_{22}N_3O_6$ $[M+H]^+$ 436.1508, found 436.1501.



2v

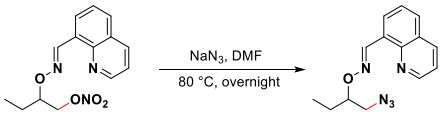
(E)-4-(nitrooxy)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl thiophene-2-carboxylate (**2v**) : Substrate **1v** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2v** was obtained as yellow oil (25.7 mg, 62%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.44 (s, 1H), 8.93 (dd, *J* = 4.1, 1.8 Hz, 1H), 8.23 (dd, *J* = 7.3, 1.5 Hz, 1H), 8.17 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.87 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.81 (dd, *J* = 3.8, 1.3 Hz, 1H), 7.59–7.53 (m, 2H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.09 (dd, *J* = 5.0, 3.7 Hz, 1H), 4.84–4.73 (m, 3H), 4.58–4.46 (m, 2H), 2.34–2.24 (m, 1H), 2.24–2.17 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.2, 150.2, 147.8, 145.9, 136.4, 133.7, 133.7, 132.7, 130.0, 129.6, 128.4, 127.9, 126.5, 126.5, 121.6, 76.6, 73.5, 61.4, 30.2. HRMS (ESI-TOF) *m/z* calcd for $C_{19}H_{18}N_3O_6S$ $[M+H]^+$ 416. 0916, found 416. 0907.



2w

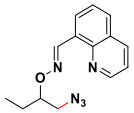
(E)-4-(nitrooxy)-3-(((quinolin-8-ylmethylene)amino)oxy)butyl cyclopropanecarboxylate (**2w**) : Substrate **1w** was nitrooxylated following the general nitrooxylation procedure. After purification by preparative thin-layer chromatography (Hexane : EtOAc = 6:1 as eluent), **2w** was obtained as yellow oil (28.3 mg, 76%).^{1}H NMR (500 MHz, $CDCl_3$) δ 9.43 (s, 1H), 8.92 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.24 (dd, *J* = 7.3, 1.3 Hz, 1H), 8.16 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.87 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.56 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.81–4.72 (m, 2H), 4.69 (dt, *J* = 14.0, 4.6 Hz, 1H), 4.32 (dd, *J* = 11.4, 5.7 Hz, 1H), 4.26 (ddd, *J* = 11.3, 8.0, 5.3 Hz, 1H), 2.15 (dt, *J* = 8.5, 5.7 Hz, 1H), 2.12–1.97 (m, 1H), 1.66–1.59 (m, 1H), 1.00 (dt, *J* = 6.8, 3.5 Hz, 2H), 0.86 (dt, *J* = 7.5, 3.9 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.9, 150.2, 147.7, 146.0, 136.3, 130.0, 129.6, 128.4, 126.4, 126.4, 121.6, 76.5, 73.5, 60.7, 30.1, 13.0, 8.7. HRMS (ESI-TOF) *m/z* calcd for $C_{18}H_{20}N_3O_6$ $[M+H]^+$ 374.1352, found 374.1348.

Removal of the Directing Group and Derivatization



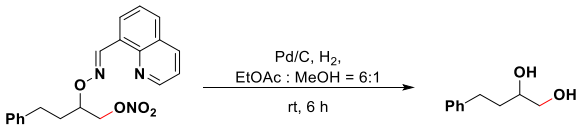
2a 3a

General Procedure : Take a 25 mL dry pressure tube into a magnetic stir bar, add **2a** (0.14 mmol, 40 mg, 1.0 equiv), NaN_3 (0.56 mmol, 35 mg, 4.0 equiv) and DMF (1.5 mL) solvents in turn, and react at 80°C overnight. Add EtOAc (20 mL) to dissolve the reaction solution, transfer to a separatory funnel, wash twice with water (20 mL), then with saturated brine, then dry with anhydrous sodium sulfate, evaporate the solvent in vacuo, and use a silica gel column for the residue Chromatographic purification gave product **3a** (29.3 mg, 79%).



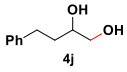
3a

(E)-quinoline-8-carbaldehyde O-(1-azidobutan-2-yl) oxime (**3a**) : Colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.47 (s, 1H), 8.93 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.29 (dd, *J* = 7.3, 1.1 Hz, 1H), 8.16 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.86 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.35 (tt, *J* = 7.4, 5.6 Hz, 1H), 3.54 (ddd, *J* = 18.7, 13.0, 4.9 Hz, 2H), 1.84 (td, *J* = 14.6, 7.4 Hz, 1H), 1.78–1.67 (m, 1H), 1.04 (t, J = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 146.9, 146.0, 136.3, 130.2, 129.7, 128.4, 126.5, 126.3, 121.6, 83.6, 53.3, 24.6, 10.0. HRMS (ESI-TOF) m/z calcd for $C_{14}H_{16}N_5O$ $[M+H]^+$ 270.1355, found 270.1349.



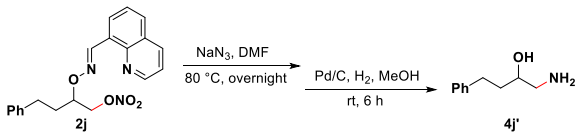
2j 4j

General Procedure : Take a 25 mL dry round bottom flask and add a magnetic stir bar. Add **2j** (0.074 mmol, 27 mg, 1.0 equiv), EtOAc (3.6 mL), MeOH (0.6 mL) and Pd/C (5% w/w, 250 mg) in sequence, connect the reaction flask to a hydrogen balloon, flush hydrogen under the vacuum pump three times, and react overnight at room temperature on a stirrer. After the reaction was completed, the solid insoluble matter was removed by filtration on diatomaceous earth, washed with EA several times, the filtrate was collected, and rotary evaporated in vacuo. The residue was purified by silica gel column chromatography to obtain the product **4j** (11.4 mg, 93%).

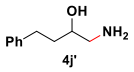


4j

4-Phenylbutane-1,2-diol (**4j**) : ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.5 Hz, 2H), 7.22–7.18 (m, 3H), 3.74 (dt, *J* = 7.8, 6.3 Hz, 1H), 3.67 (dd, *J* = 11.0, 3.0 Hz, 1H), 3.48 (dd, *J* = 11.0, 7.6 Hz, 1H), 2.85–2.78 (m, 1H), 2.75–2.66 (m, 1H), 1.81–1.80 (m, 2H), 1.79–1.75 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.8, 128.6, 128.6, 126.1, 71.7, 66.9, 34.8, 31.9.

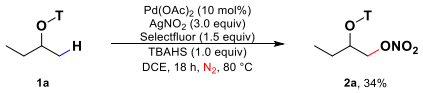


General Procedure for the Synthesis of *β*-Amino Alcohol (4j') : Take a 25 mL dry pressure tube into a magnetic stir bar, add **2j** (0.5 mmol, 18.3 mg, 1.0 equiv), NaN_3 (2 mmol, 125 mg, 4.0 equiv) and DMF (1.5 mL) solvents in turn, and react at 80°C overnight. Add EtOAc (20 mL) to dissolve the reaction solution, transfer to a separatory funnel, wash twice with water (20 mL), then with saturated brine, then dry with anhydrous sodium sulfate, evaporate the solvent in vacuo, and use a silica gel column for the residue. Chromatographic purification gave the azide product (14.1 mg, 82%).Then take another 25 mL dry round bottom flask and add a magnetic stir bar. Add azide product (0.05 mmol, 17.3 mg, 1.0 equiv), MeOH (0.6 mL) and Pd/C (5% w/w, 169 mg) in sequence, connect the reaction flask to a hydrogen balloon, flush hydrogen under the vacuum pump three times, and react overnight at room temperature on a stirrer. After the reaction was completed, the solid insoluble matter was removed by filtration on diatomaceous earth, washed with EA several times, the filtrate was collected, and rotary evaporated in vacuo. The residue was purified by silica gel column chromatography to obtain the product **4j'** (7.3 mg, 73% over the two steps).



1-Amino-4-phenylbutan-2-ol (**4j'**) : ^{1}H NMR (500 MHz, DMSO) δ 7.69 (br, 2H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.22 (d, *J* = 7.3 Hz, 2H), 7.17 (t, *J* = 7.1 Hz, 1H), 5.32 (br, 1H), 3.64 (d, *J* = 3.9 Hz, 1H), 2.84 (dd, *J* = 12.7, 3.1 Hz, 1H), 2.75–2.67 (m, 1H), 2.61 (ddd, *J* = 13.9, 11.3, 7.9 Hz, 2H), 1.75–1.59 (m, 2H). ^{13}C NMR (126 MHz, DMSO) δ 142.2, 128.3, 128.3, 125.6, 68.3, 45.9, 36.3, 31.2.

Control Experiment



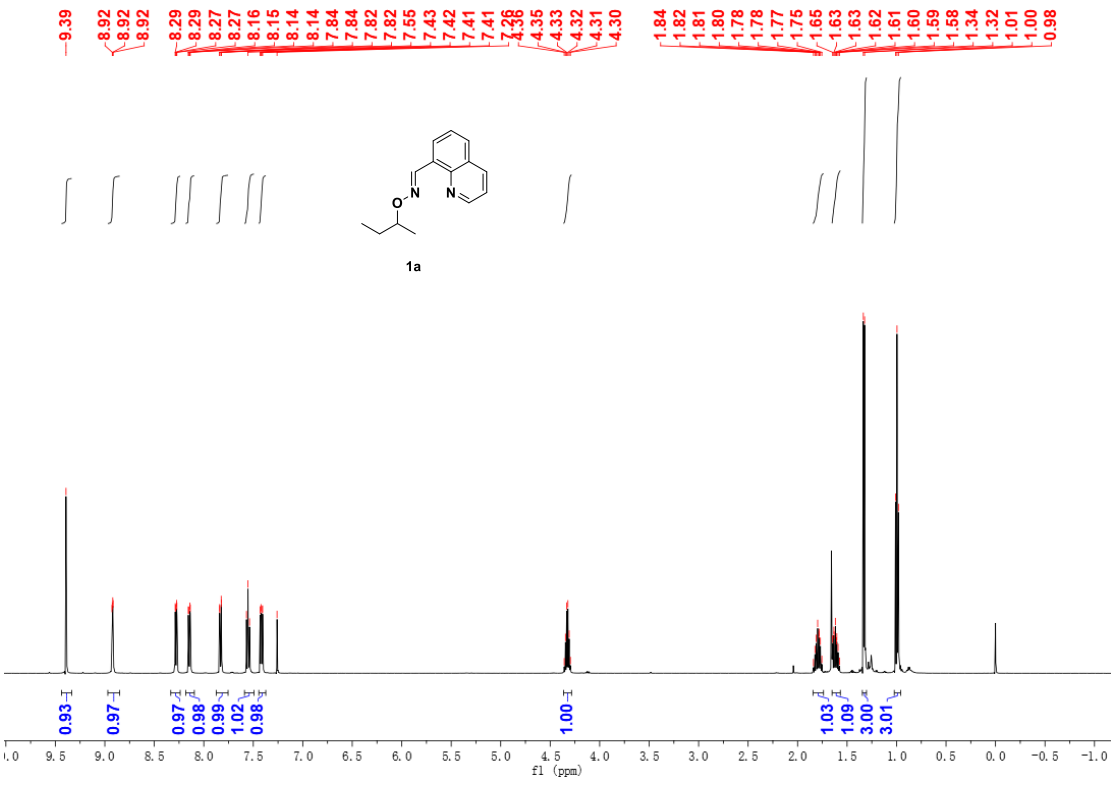
General Procedure for the control experiment : An oven dried 25 mL Schlenk tube, equipped with a stir bar, was charged with **1a** (0.1 mmol), $Pd(OAc)_2$ (10 mol %), $AgNO_2$ (3.0 equiv), Selectfluor (1.5 equiv), TBAHS (1.0 equiv) and DCE (2 mL). The tube was connectted to a N_2 balloon, flush N_2 under the vacuum pump three times. Then the reaction mixture was stirred under N_2 at 80 °C for 18 h. Upon completion, EtOAc was added to dilute the mixture and then filtered through a pad of silica gel. The solvents were evaporated under reduced pressure and then purified by preparative thin-layer chromatography. The desired product **2a** was obtained in a yield of 34%.

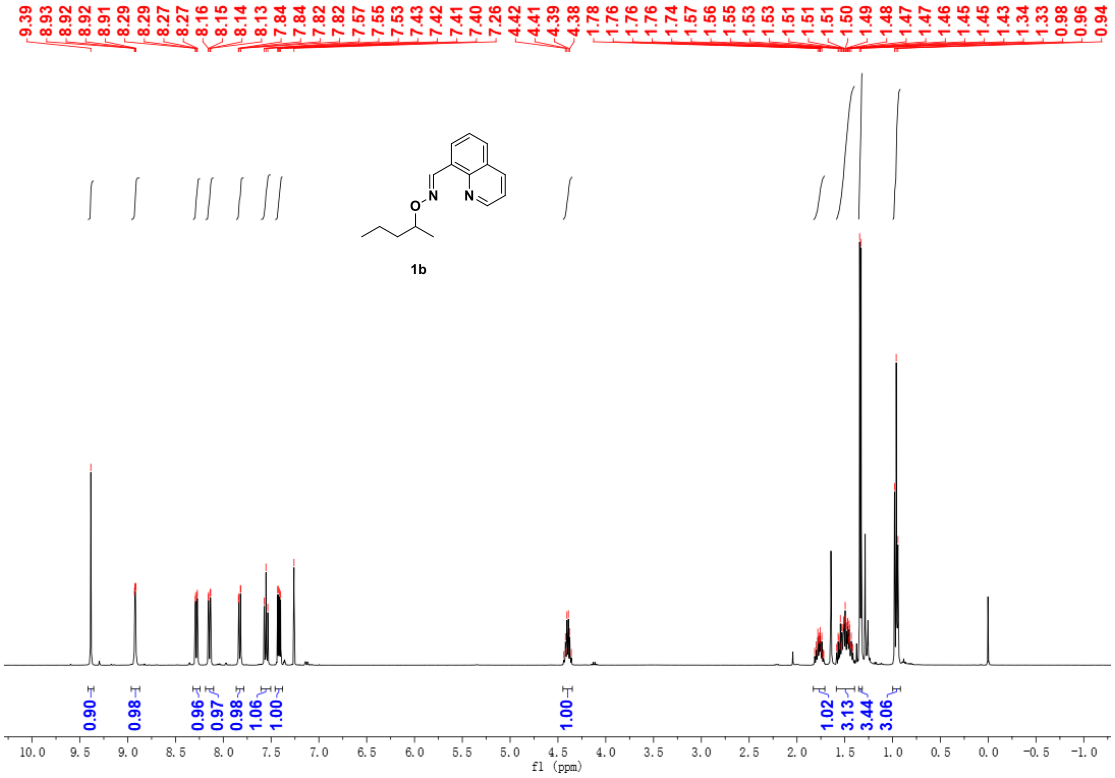
References

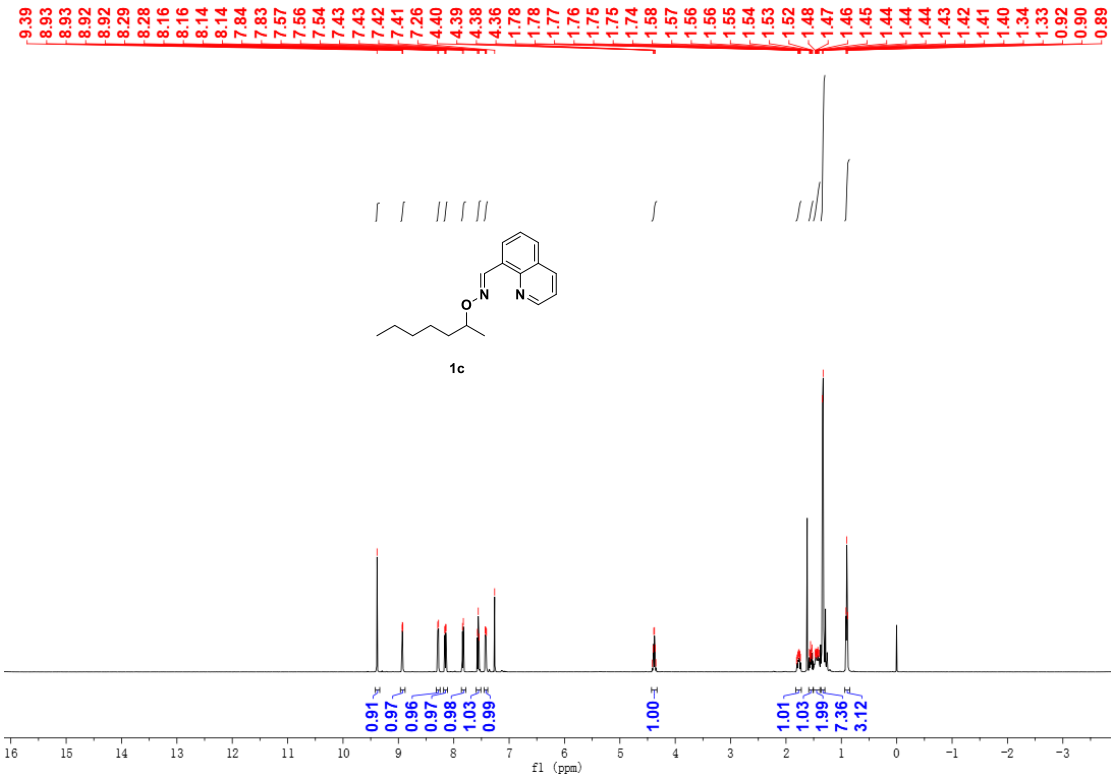
1. Xu, Y.; Yan, G.; Ren, Z.; Dong, G., Diverse sp^3 C-H functionalization through alcohol beta-sulfonyloxylation. *Nat. Chem.*, **2015**, *7*, 829-34.

2. (a) Dong, Y.; Liu, G., Auxiliary-Assisted Palladium-Catalyzed Direct C(sp^3)-H Sulfonamidation To Afford 1,2-Amino Alcohol Derivatives. *J. Org. Chem.*, **2017**, *82*, 3864-3872; (b) Jin, L.; Zeng, X.; Li, S.; Hong, X.; Qiu, G.; Liu, P., Palladium-catalyzed intermolecular amination of unactivated C(sp^3)-H bonds via a cleavable directing group. *Chem. Commun.*, **2017**, *53*, 3986-3989.

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



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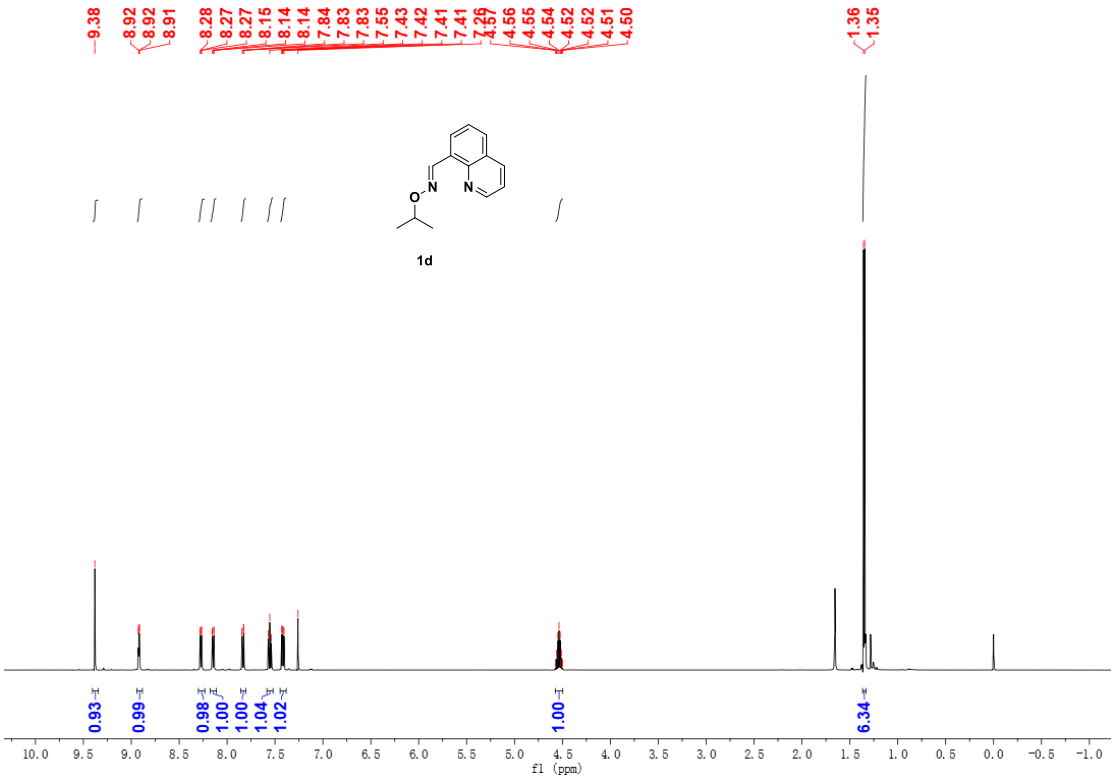
f1 (ppm)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1d
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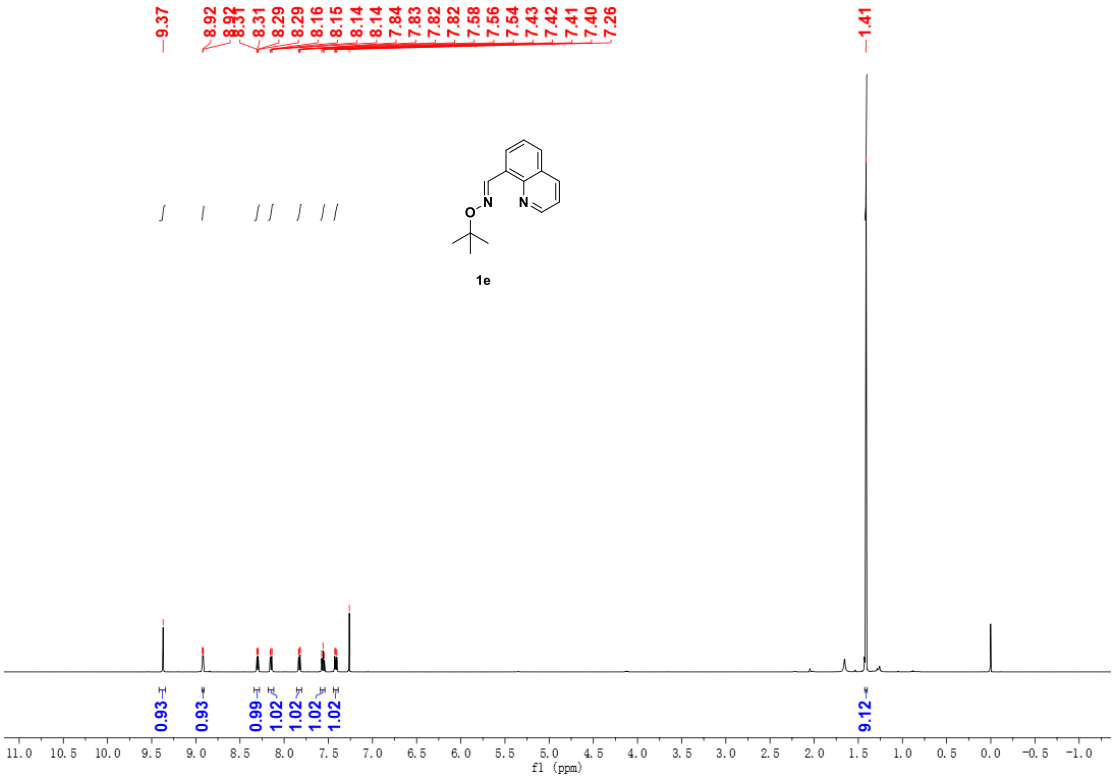
f1 (ppm)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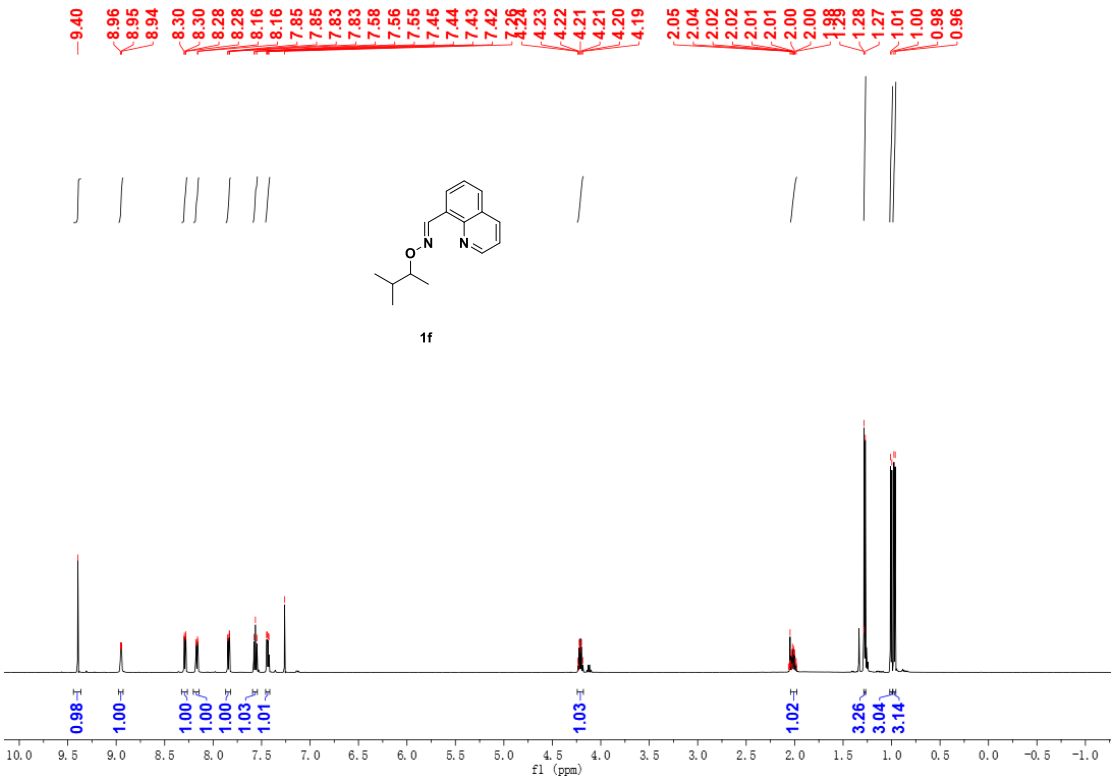
f1 (ppm)

1e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2.02
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1.00
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1.03
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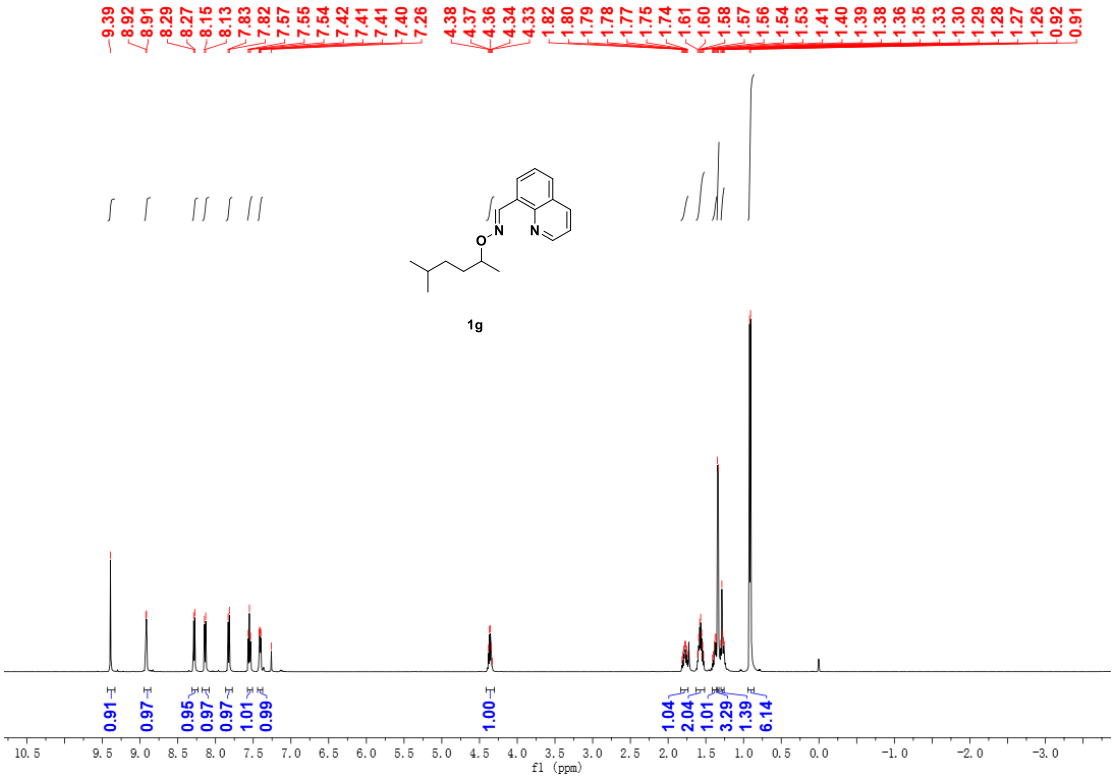
f1 (ppm)

1f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4.38 4.37 4.36 4.34 4.33
1.82 1.80 1.79 1.78 1.77 1.75 1.74 1.61 1.60 1.58 1.57 1.56 1.54 1.53 1.41 1.40 1.39 1.38 1.36 1.35 1.33 1.30 1.29 1.28 1.27 1.26 0.92 0.91
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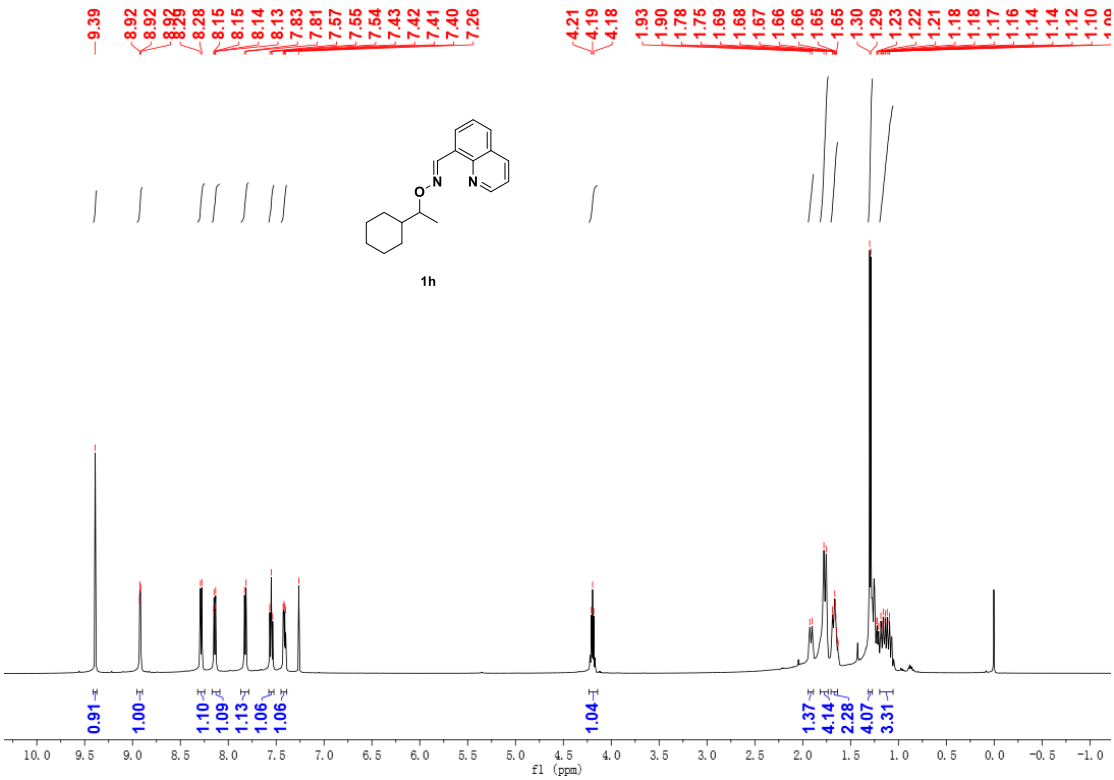
f1 (ppm)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4.21 4.19 4.18
1.93 1.90 1.78 1.75 1.69 1.68 1.67 1.66 1.66 1.65 1.65 1.30 1.29 1.23 1.22 1.21 1.18 1.18 1.17 1.16 1.14 1.14 1.12 1.10
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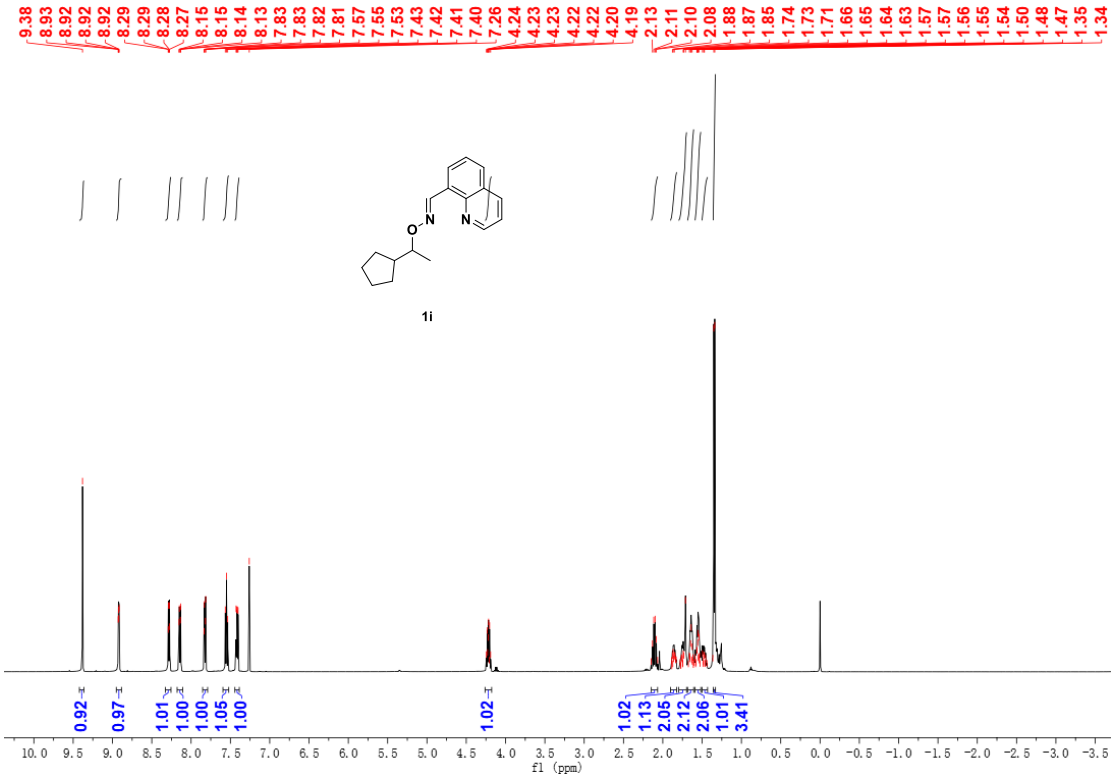
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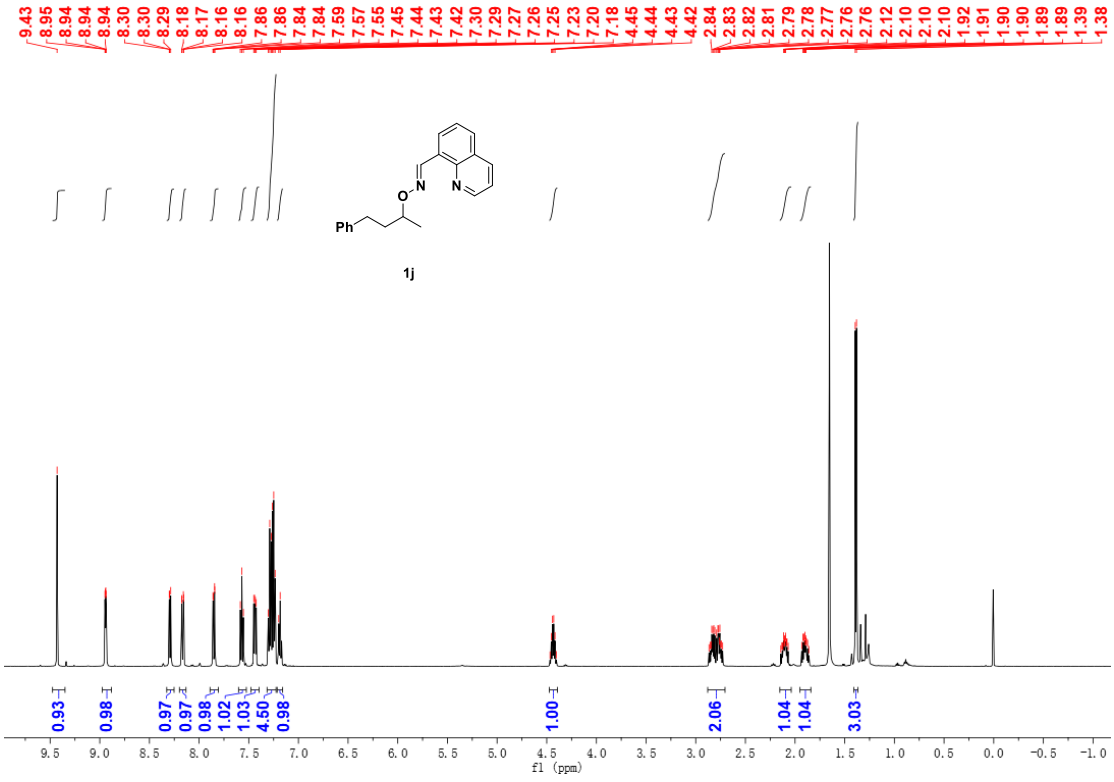
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Ph
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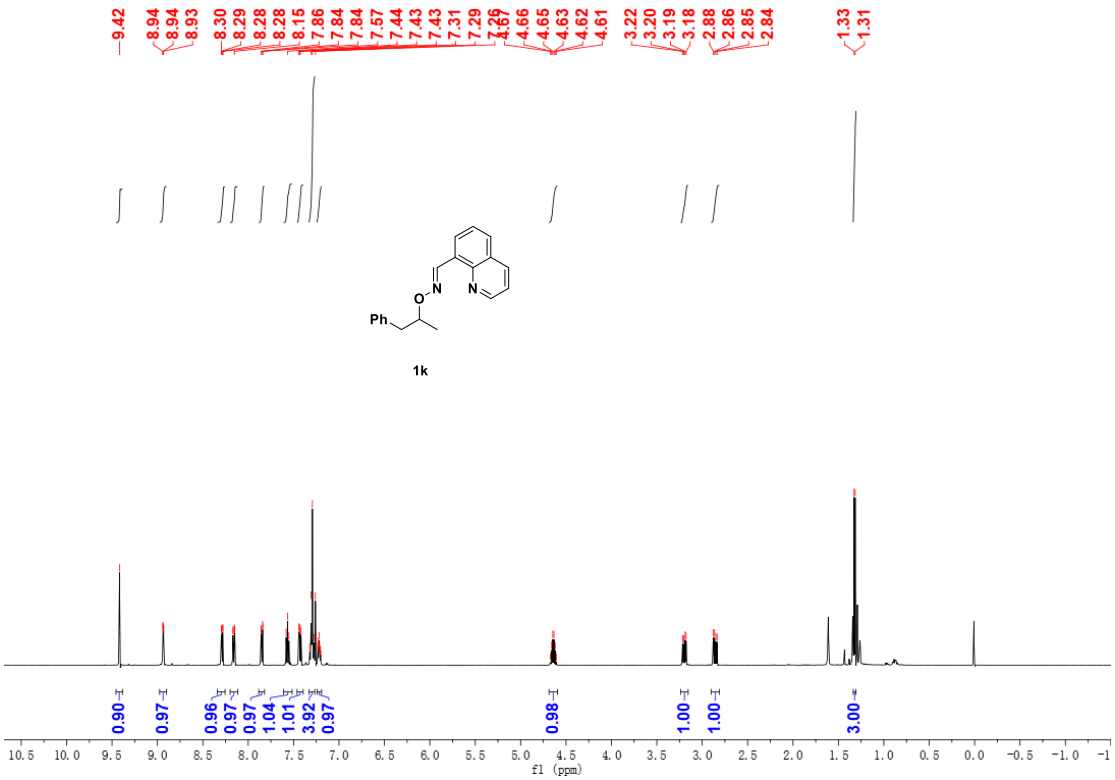
f1 (ppm)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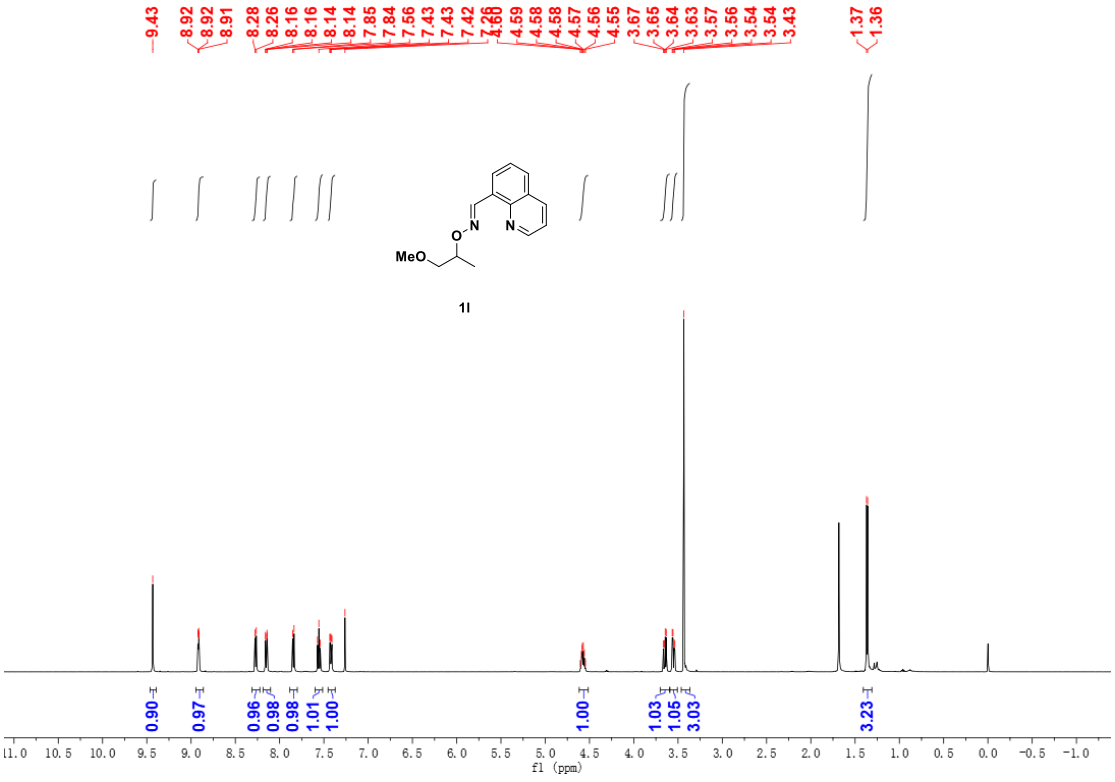
3.00
f1 (ppm)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7.43
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MeO
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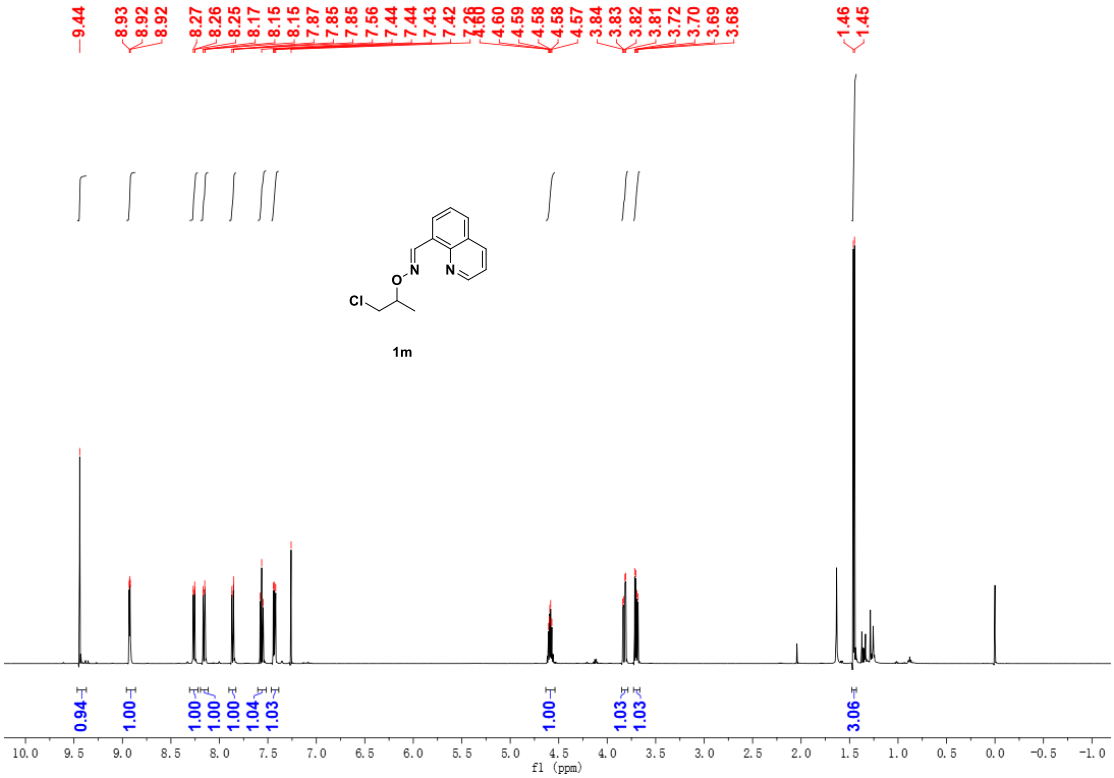
f1 (ppm)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4.57
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3.83
3.82
3.81
3.72
3.70
3.69
3.68
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1.45
Cl
N
O
N
1m
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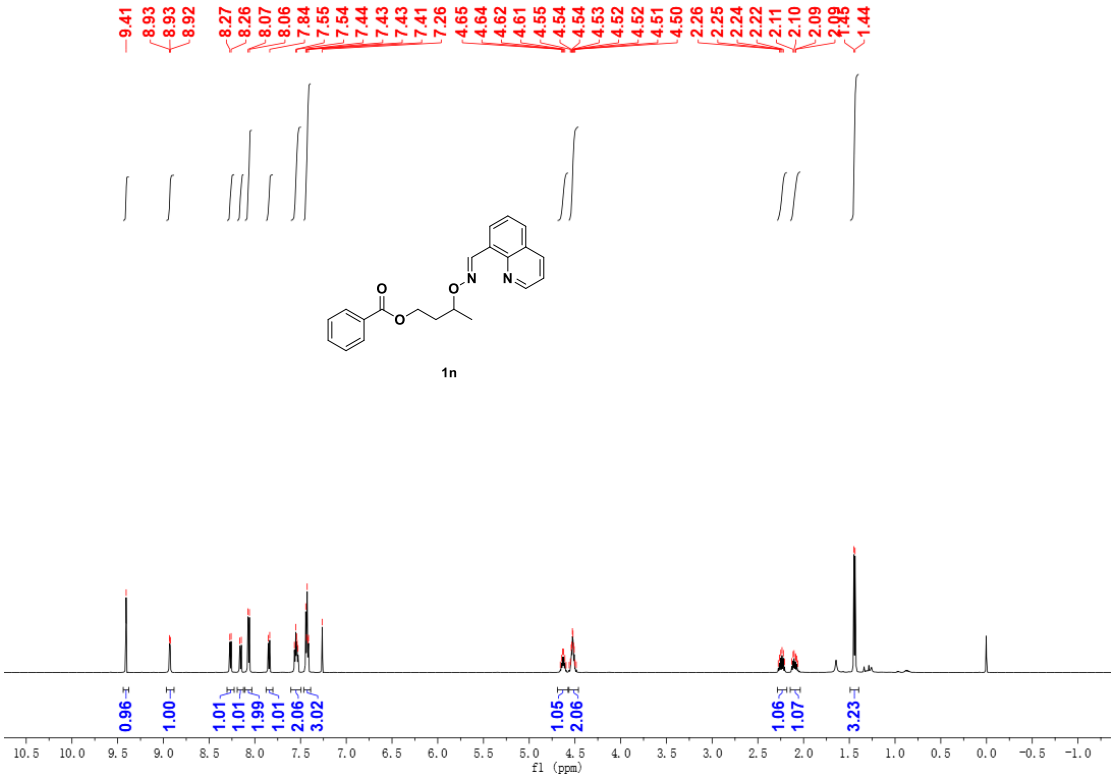
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2.25
2.24
2.22
2.11
2.10
2.09
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1.44
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N
O
O
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0.96
1.00
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2.06
1.06
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3.23
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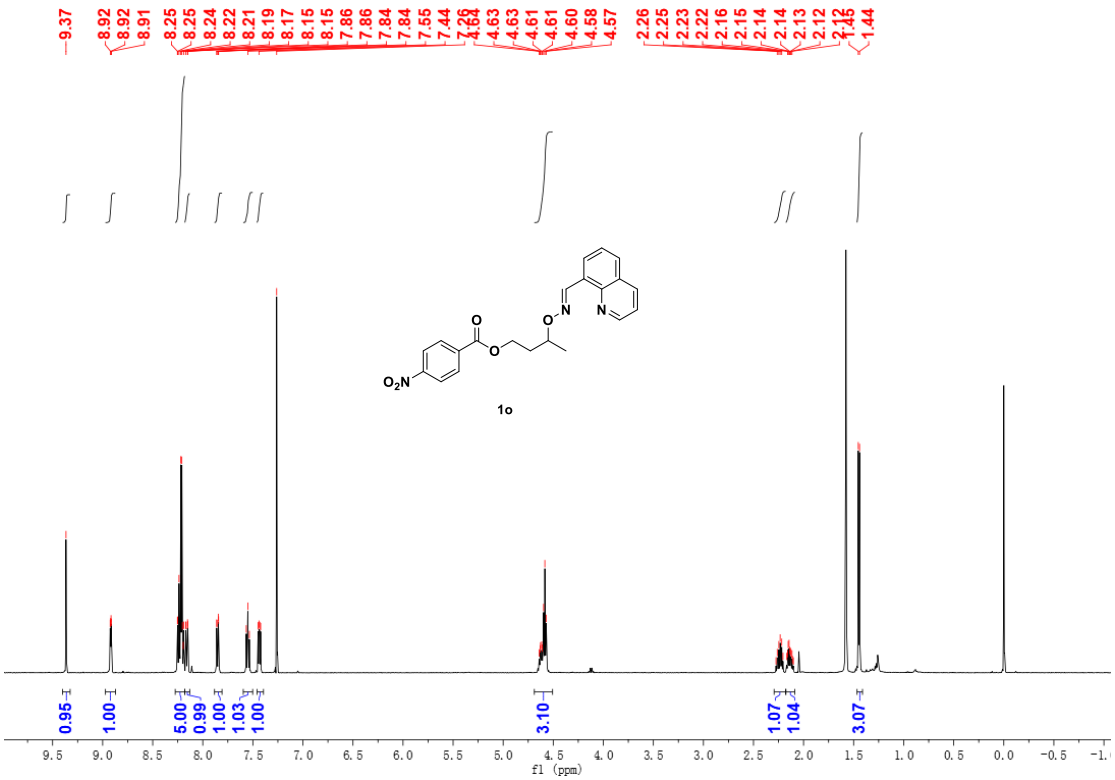
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4.61
4.61
4.60
4.58
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2.16
2.15
2.14
2.14
2.13
2.12
1.44
O2N
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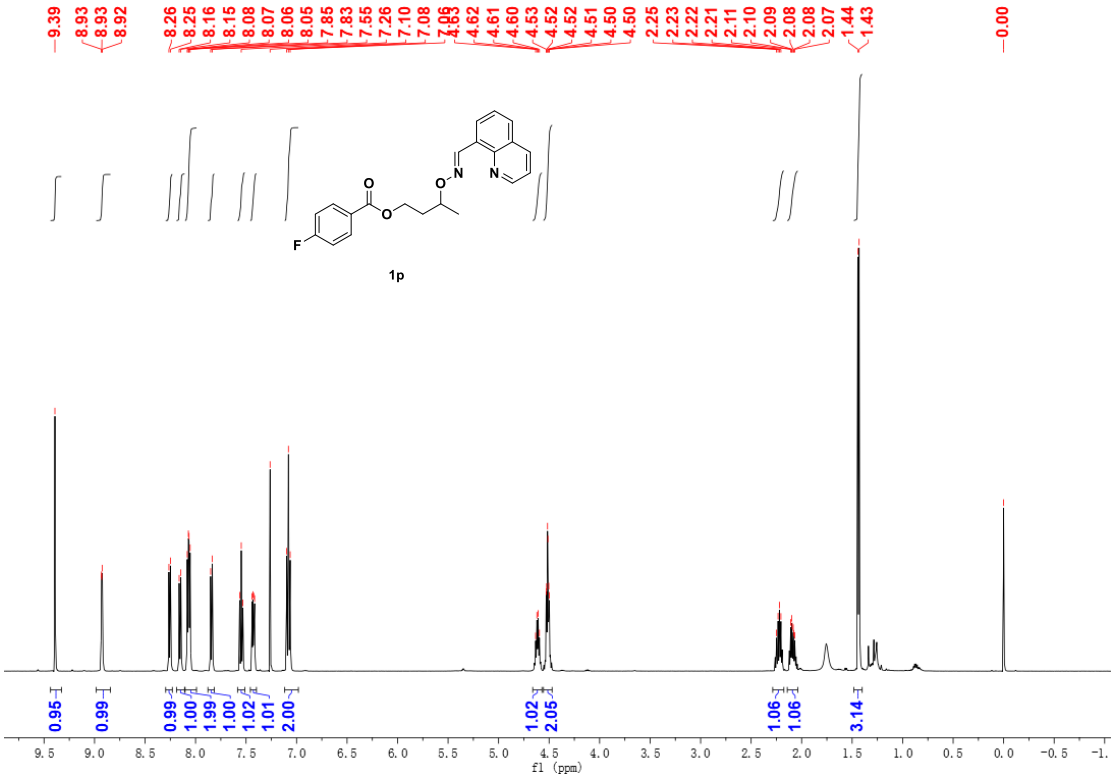
3.07
f1 (ppm)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2.08
2.07
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1.43
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1p
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0.99
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1.01
2.00
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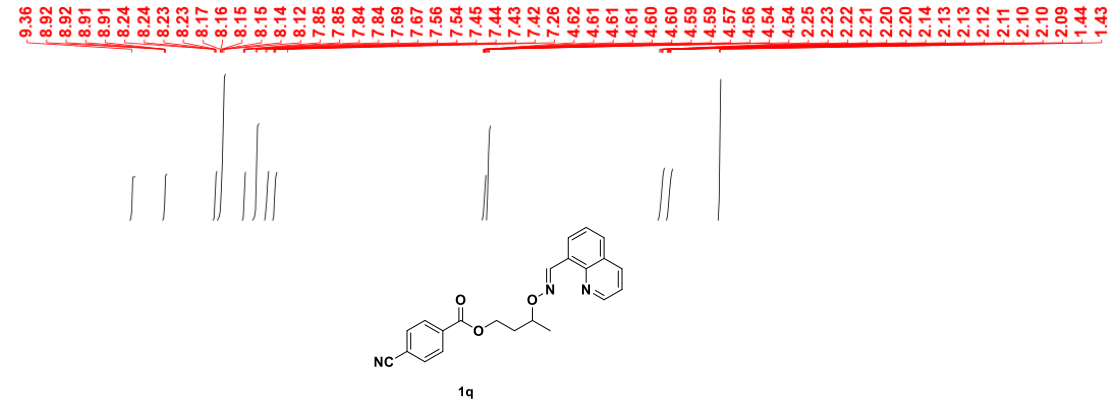
1.06
1.06
3.14
f1 (ppm)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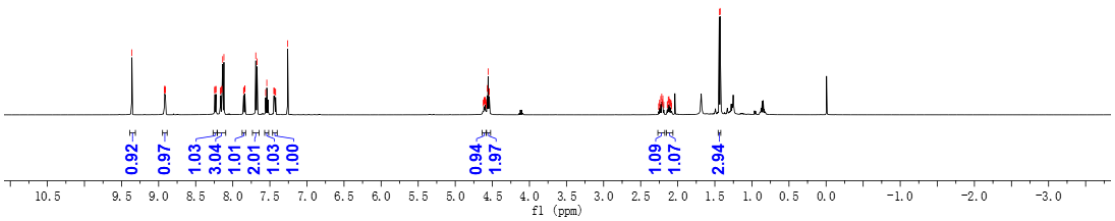
O
N
NC

1q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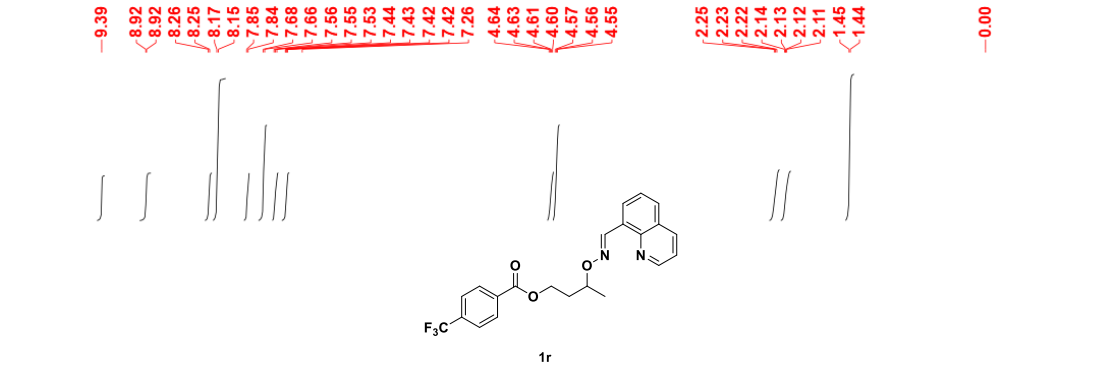
f1 (ppm)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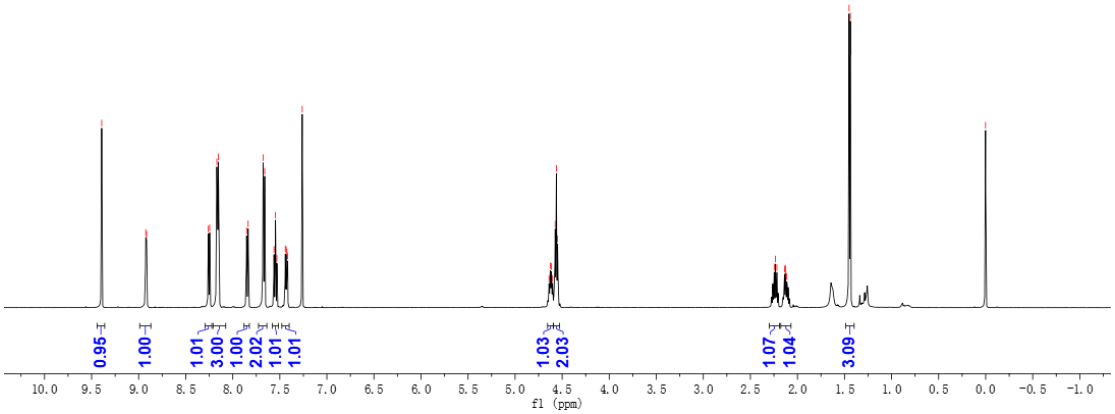
N
F3C

1r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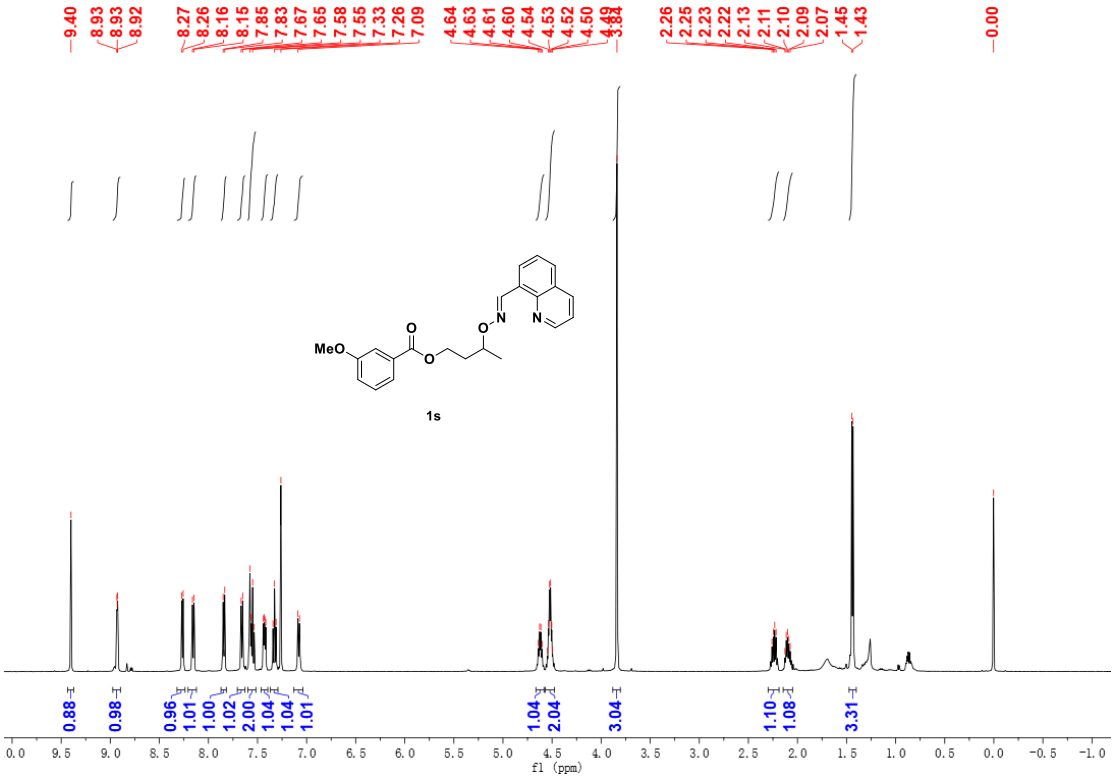
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2.11
2.10
2.09
2.07
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MeO
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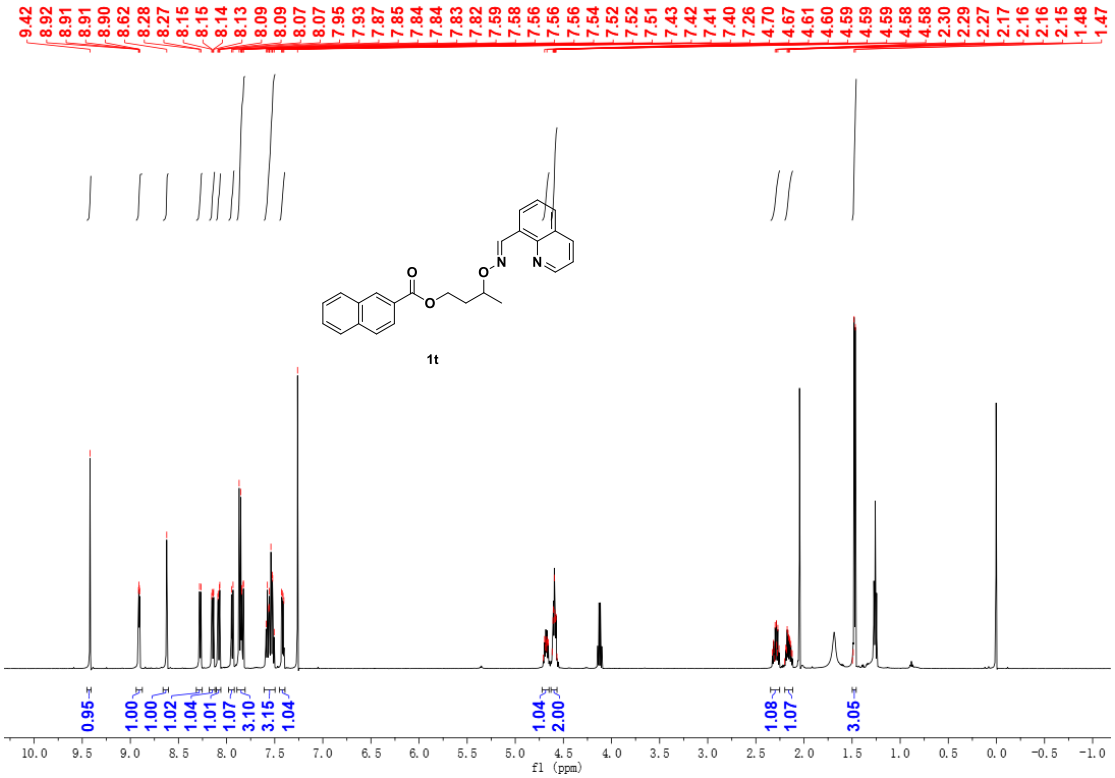
f1 (ppm)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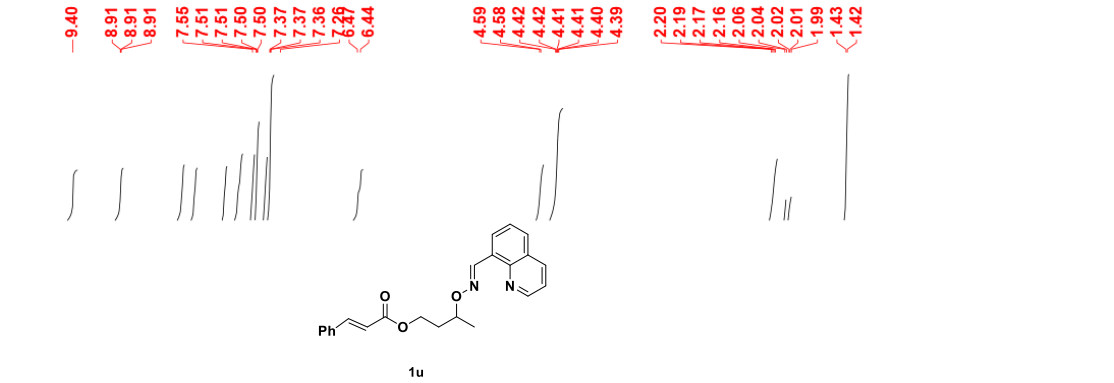
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O
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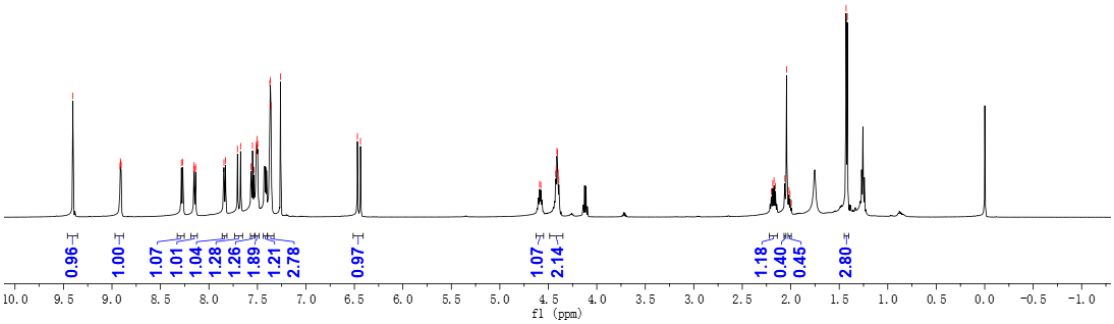
1u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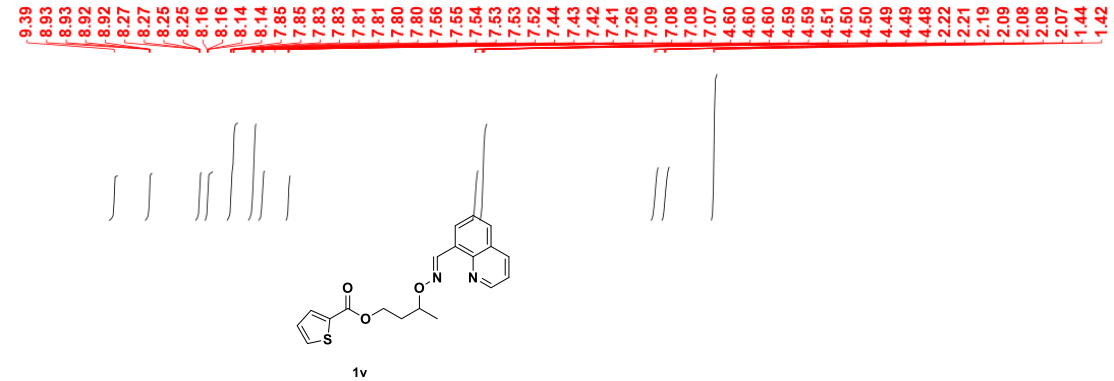
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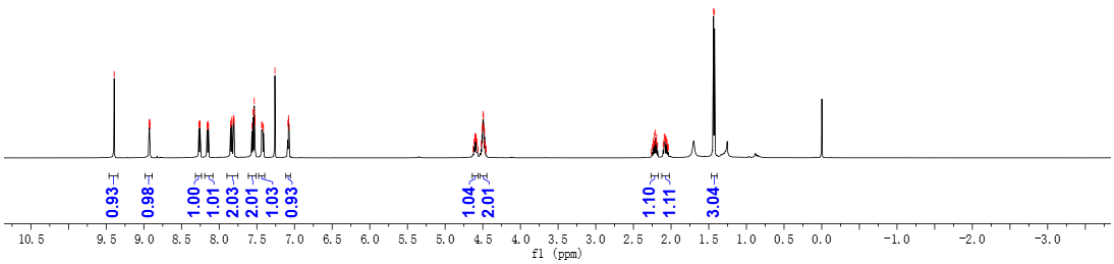
1v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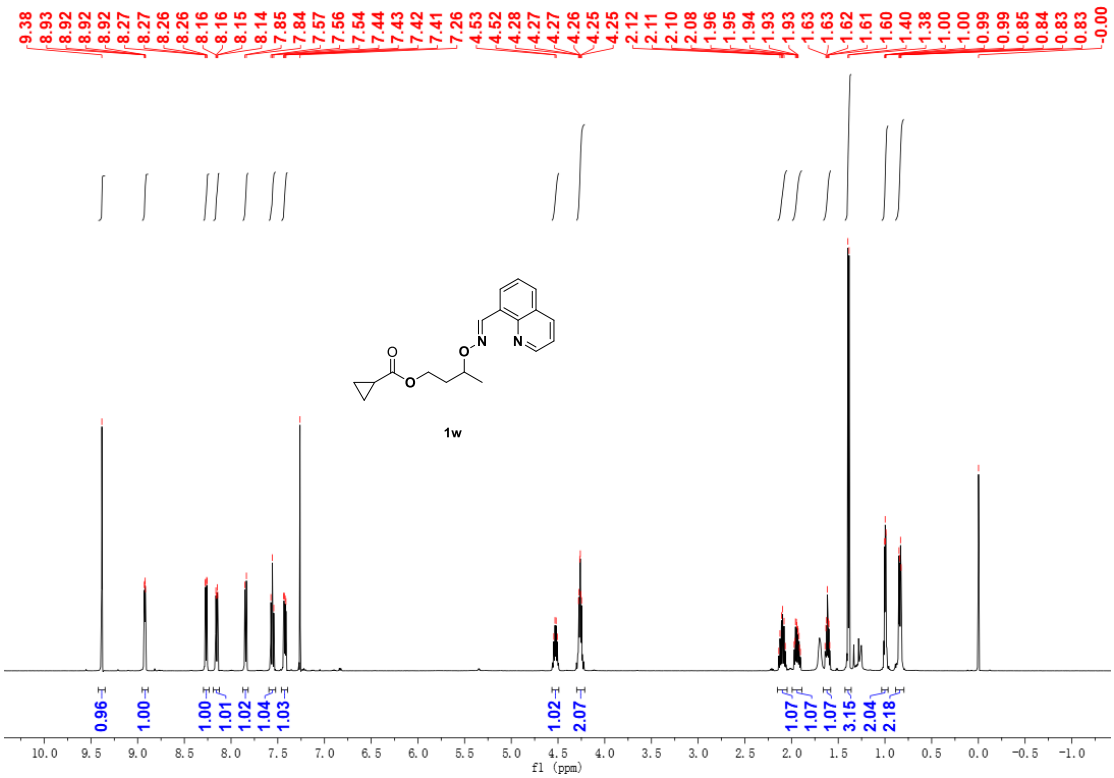
f1 (ppm)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0.83
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1.07
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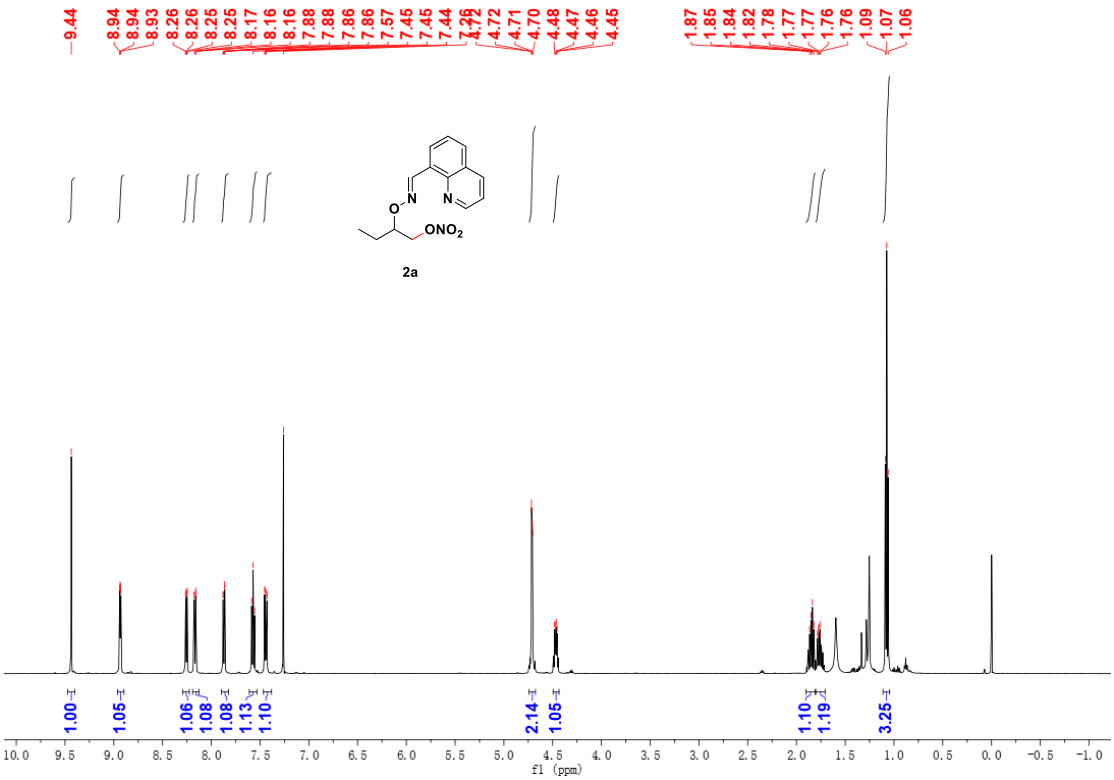
f1 (ppm)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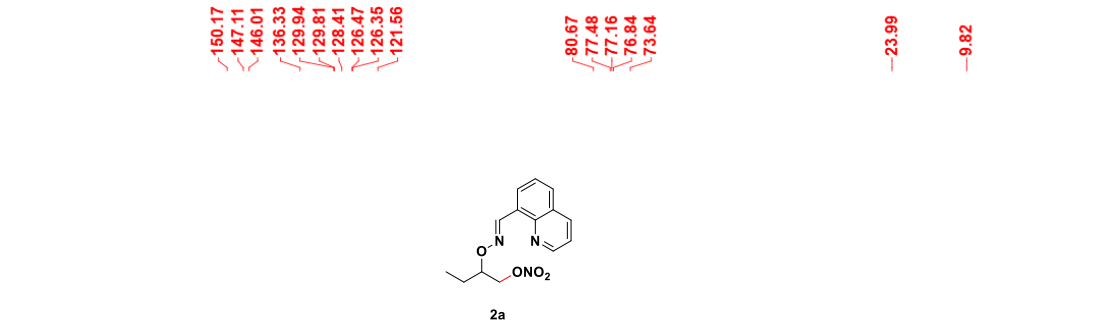
3.25
f1 (ppm)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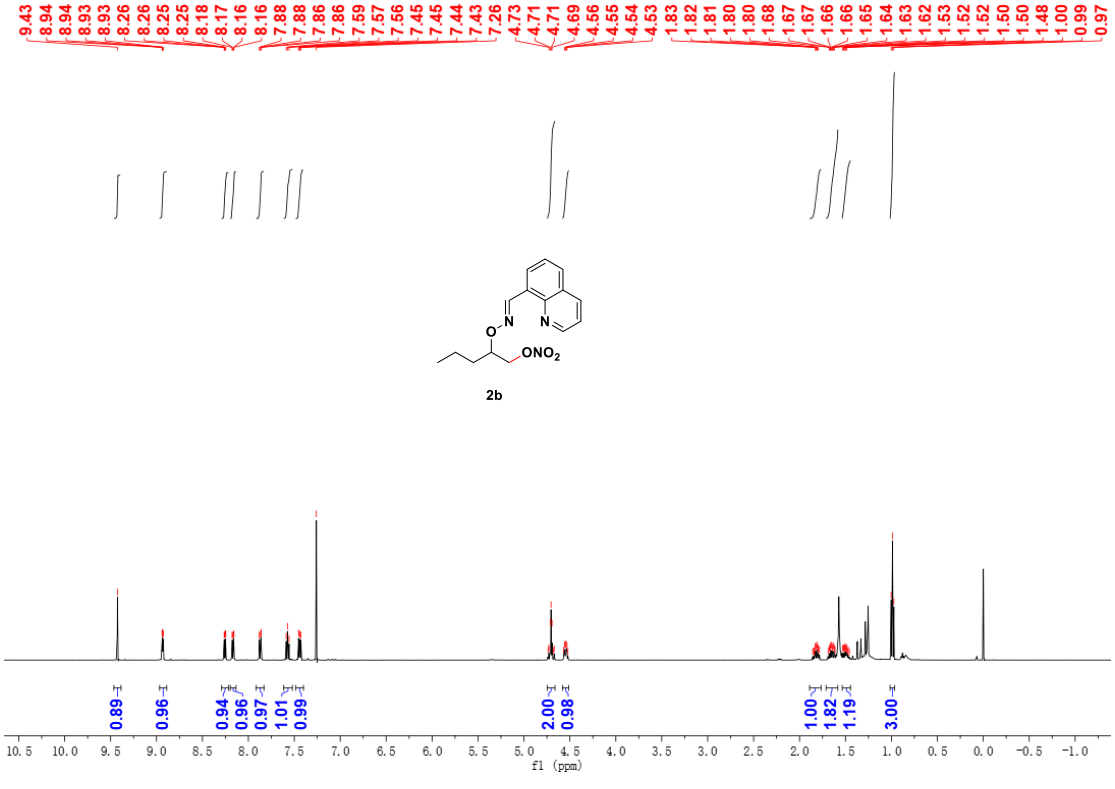
129.81
128.41
126.47
126.35
121.56
80.67
77.48
77.16
76.84
73.64
23.99
9.82
N
O
N
ONO2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

2a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8.94
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8.25
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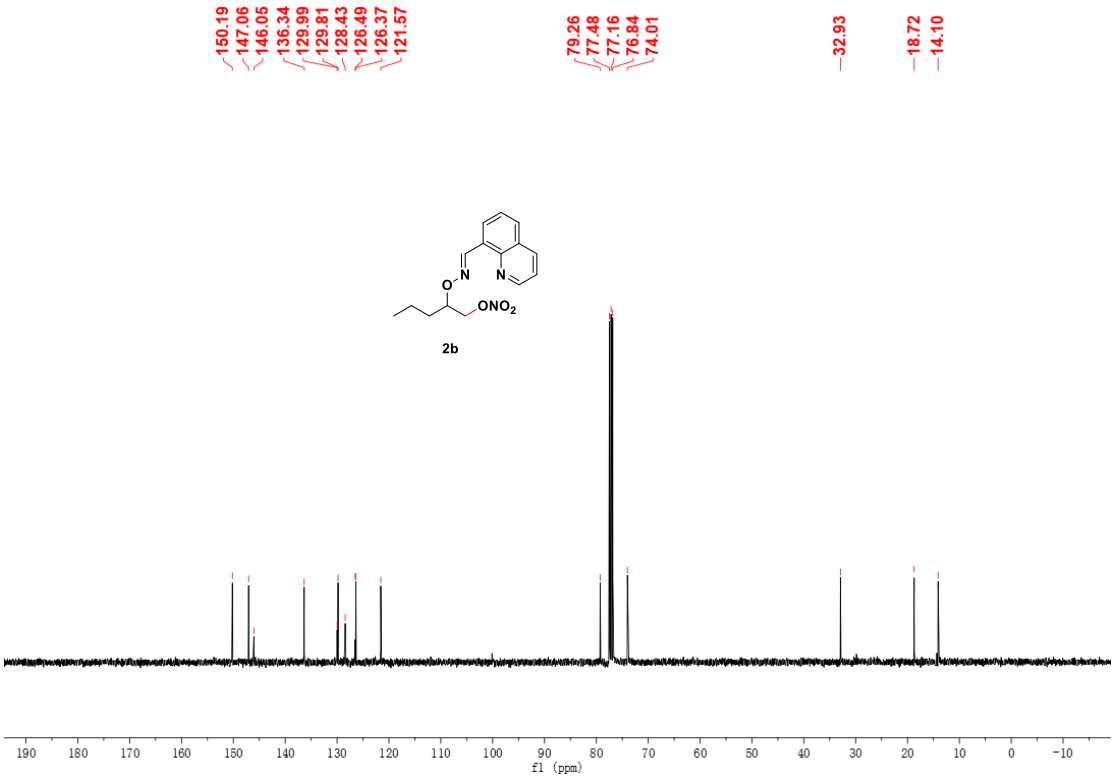
8.18
8.17
8.16
8.16
7.88
7.88
7.86
7.86
7.59
7.57
7.56
7.45
7.45
7.44
7.43
7.26
4.73
4.71
4.71
4.69
4.56
4.55
4.54
4.53
1.83
1.82
1.81
1.80
1.80
1.68
1.67
1.67
1.66
1.66
1.65
1.64
1.63
1.62
1.53
1.52
1.52
1.50
1.50
1.48
1.00
0.99
0.97
N
O
N
ONO2
0.89
0.96
0.94
0.96
0.97
1.01
0.99
2.00
0.98
1.00
1.82
1.19
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

2b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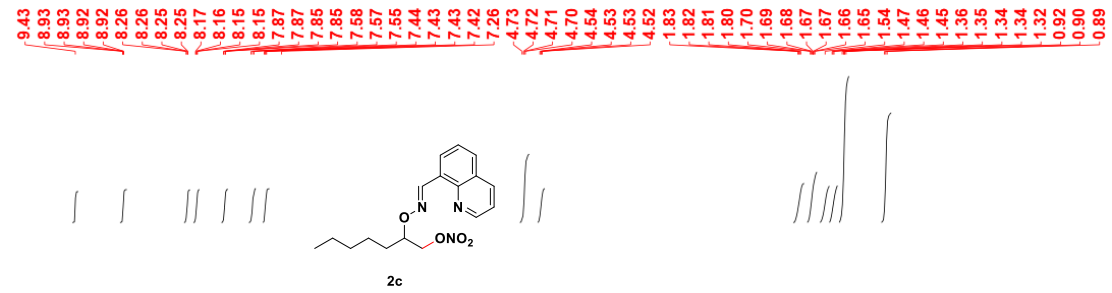
128.43
126.49
126.37
121.57
79.26
77.48
77.16
76.84
74.01
32.93
18.72
14.10
N
O
N
ONO2
2b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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8.93
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8.25
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8.17
8.16
8.15
8.15
7.87
7.87
7.85
7.85
7.58
7.57
7.55
7.44
7.43
7.43
7.42
7.26
4.73
4.72
4.71
4.70
4.54
4.53
4.53
4.52
1.83
1.82
1.81
1.80
1.70
1.69
1.68
1.67
1.67
1.66
1.65
1.54
1.47
1.46
1.45
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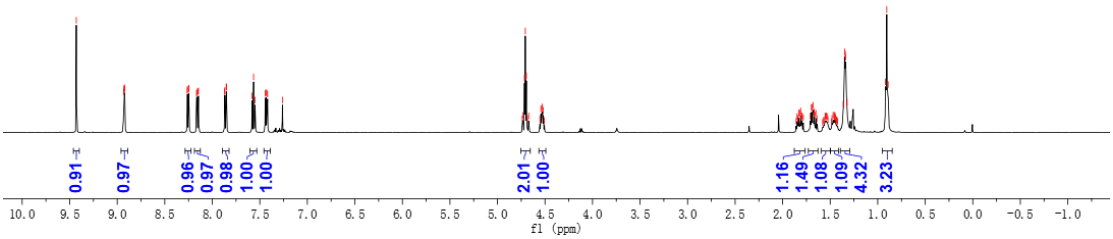
1.34
1.34
1.32
0.92
0.90
0.89
N
N
O
ONO2

2c


0.91
0.97
0.96
0.97
0.98
1.00
1.00
2.01
1.00
1.16
1.49
1.08
1.09
4.32
3.23
f1 (ppm)


150.17
147.02
145.99
136.35
129.93
129.81
128.40
126.48
126.35
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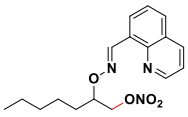
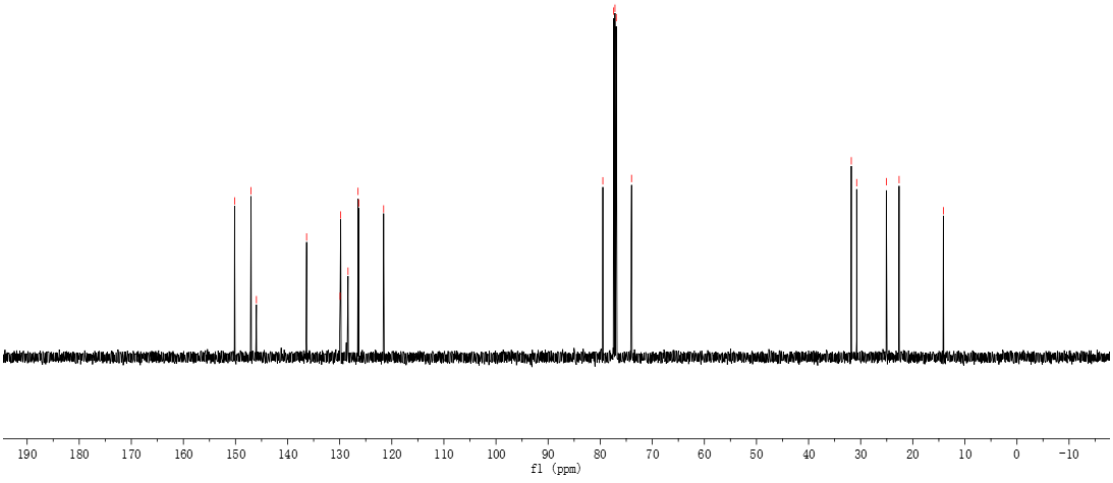
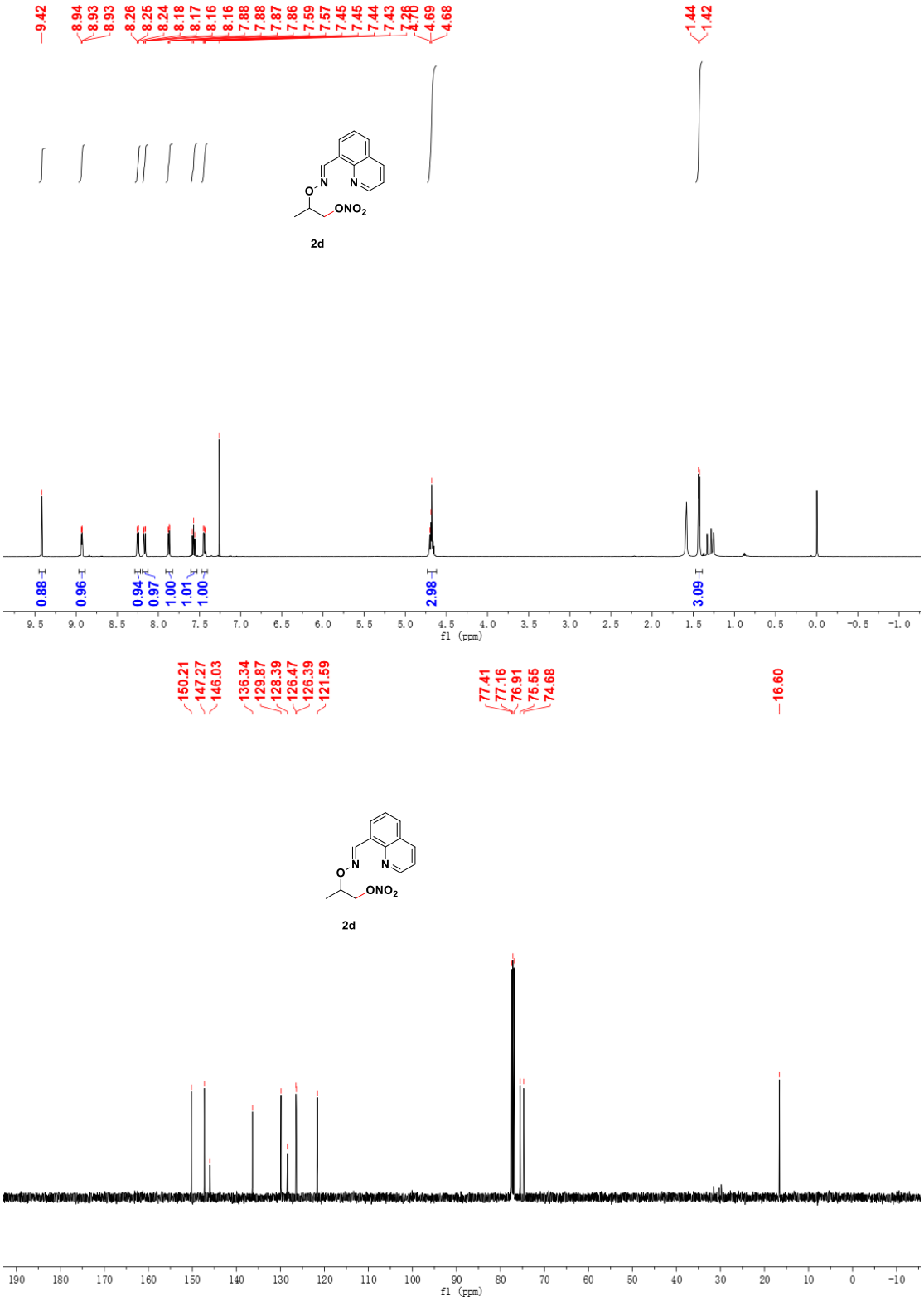
79.49
77.41
77.16
76.91
73.98
31.80
30.77
25.07
22.63
14.13


N
N
O
ONO2

2c


f1 (ppm)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8.93
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8.25
8.24
8.18
8.17
8.16
8.16
7.88
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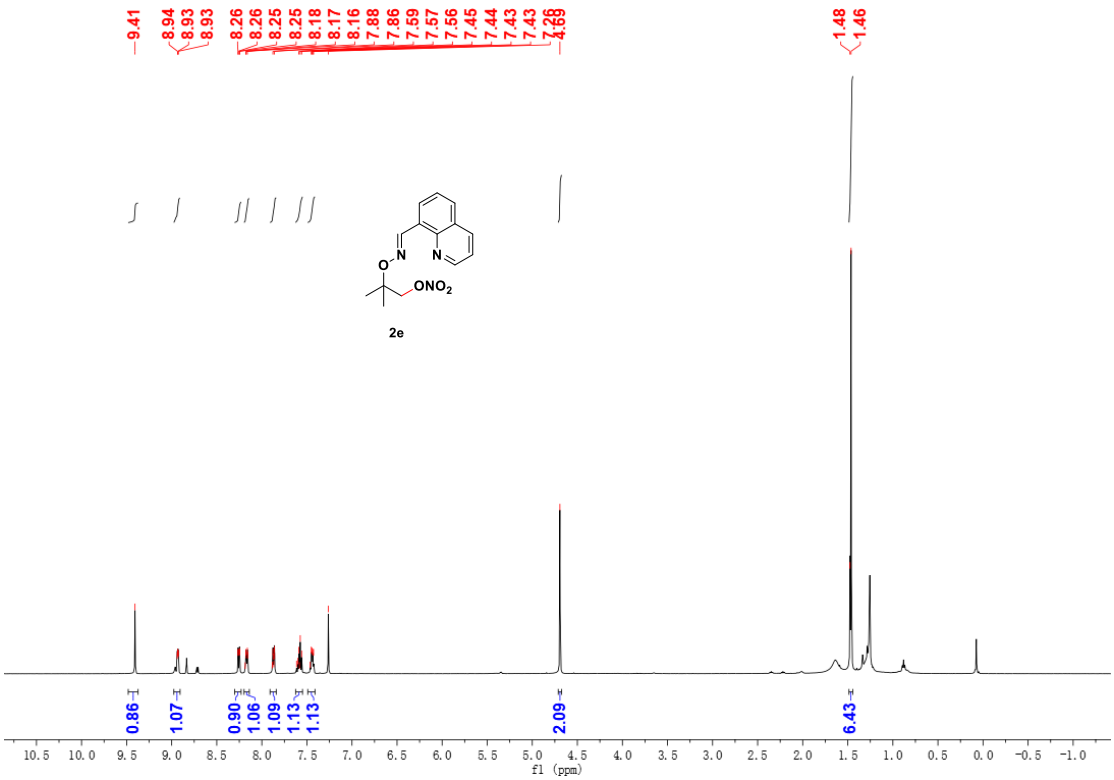
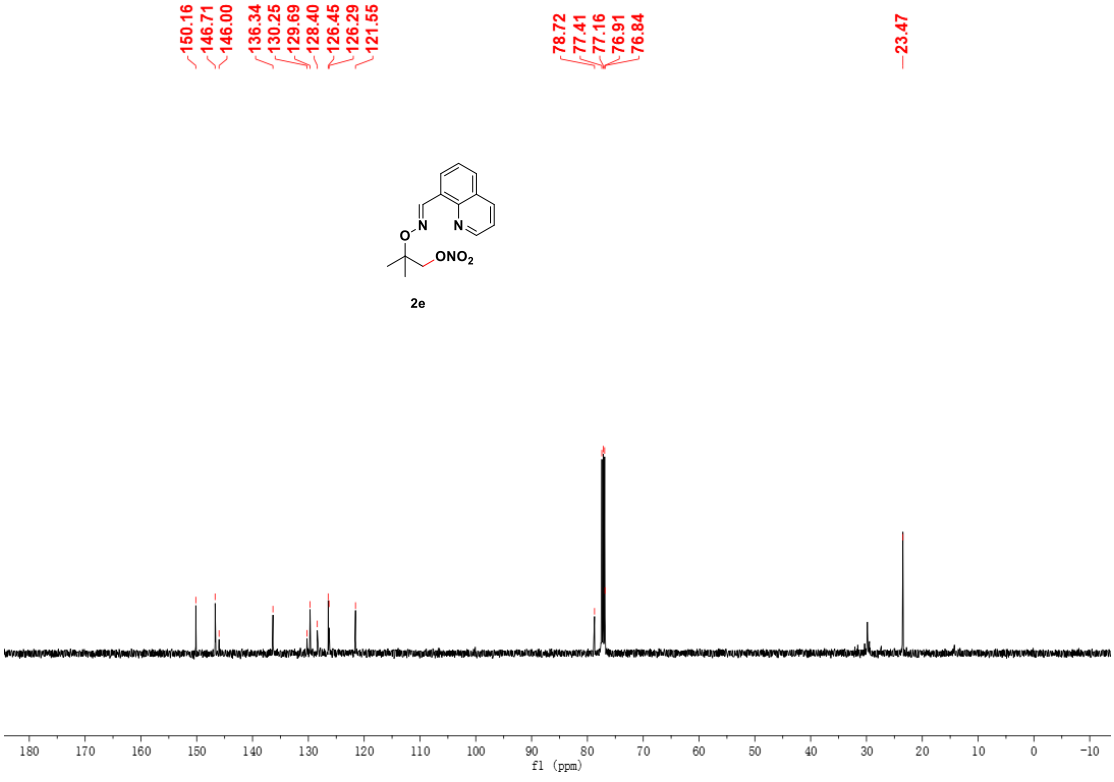
7.87
7.86
7.59
7.57
7.45
7.45
7.44
7.43
7.26
4.70
4.69
4.68
1.44
1.42
ONO2
2d
0.88
0.96
0.94
0.97
1.00
1.01
1.00
2.98
3.09
f1 (ppm)
150.21
147.27
146.03
136.34
129.87
128.39
126.47
126.39
121.59
77.41
77.16
76.91
75.55
74.68
16.60
ONO2
2d
f1 (ppm)


9.41
8.94
8.93
8.93
8.26
8.26
8.25
8.25
8.18
8.17
8.16
7.88
7.86
7.59
7.57
7.56
7.45
7.44
7.43
7.43
7.26
4.69
1.48
1.46
ONO2
2e
0.86
1.07
0.90
1.06
1.09
1.13
1.13
2.09
6.43
f1 (ppm)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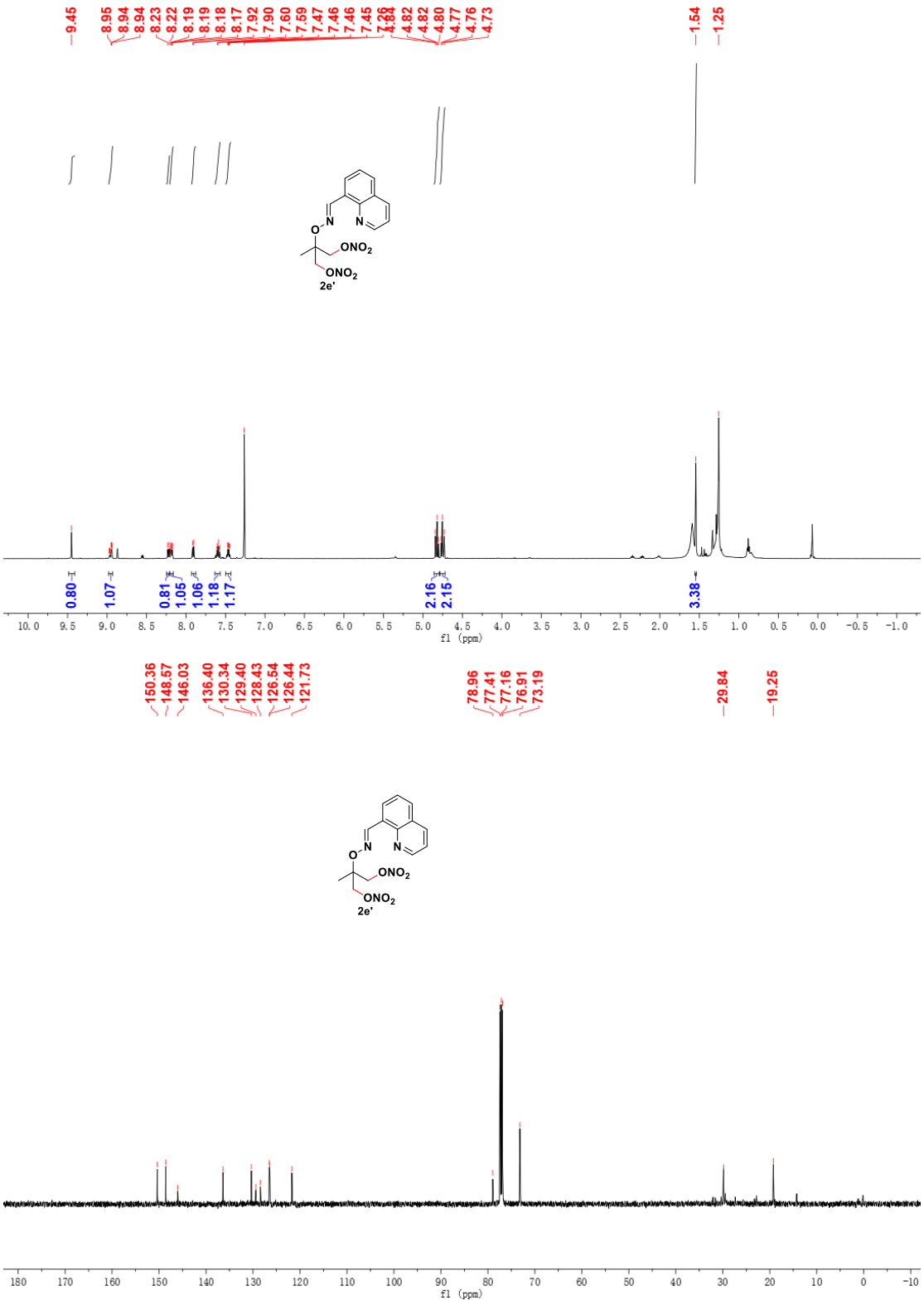
130.25
129.69
128.40
126.45
126.29
121.55
78.72
77.41
77.16
76.91
76.84
23.47
ONO2
2e
f1 (ppm)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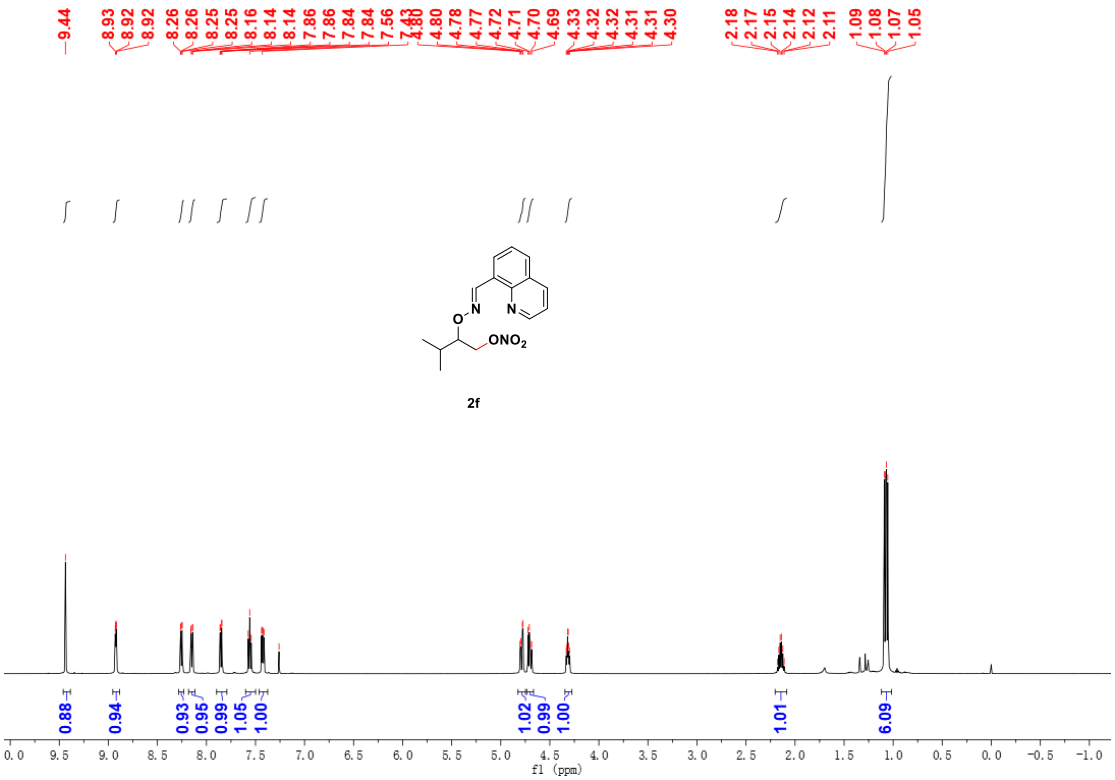
8.95
8.94
8.94
8.23
8.22
8.19
8.19
8.18
8.17
7.92
7.90
7.60
7.59
7.47
7.46
7.46
7.45
7.26
4.82
4.82
4.80
4.77
4.76
4.73
1.54
1.25
ONO2
ONO2
2e'
0.80
1.07
0.81
1.05
1.06
1.18
1.17
2.16
2.15
3.38
f1 (ppm)
150.36
148.57
146.03
136.40
130.34
129.40
128.43
126.54
126.44
121.73
78.96
77.41
77.16
76.91
73.19
29.84
19.25
ONO2
ONO2
2e'
f1 (ppm)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8.93
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8.92
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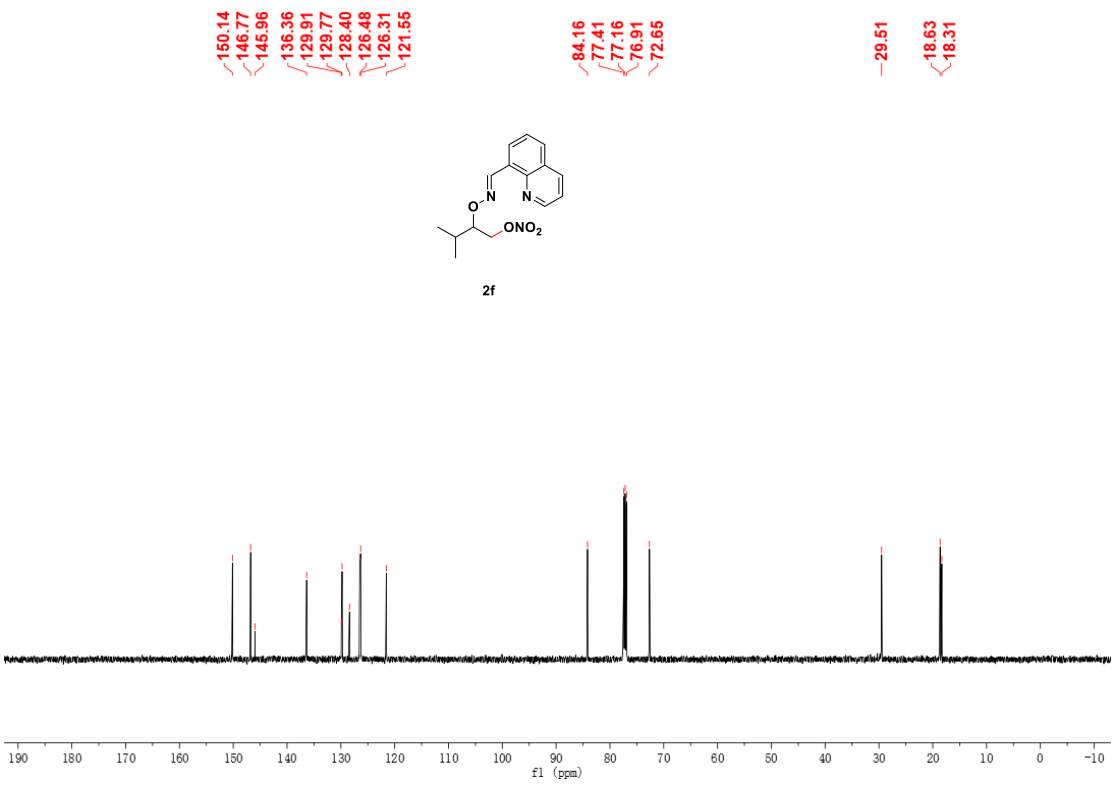
8.26
8.25
8.25
8.16
8.14
8.14
7.86
7.86
7.84
7.84
7.56
4.80
4.78
4.77
4.72
4.71
4.70
4.69
4.33
4.32
4.32
4.31
4.31
4.30
2.18
2.17
2.15
2.14
2.12
2.11
1.09
1.08
1.07
1.05
ONO2
2f
0.88
0.94
0.93
0.95
0.99
1.05
1.00
1.02
0.99
1.00
1.01
6.09
f1 (ppm)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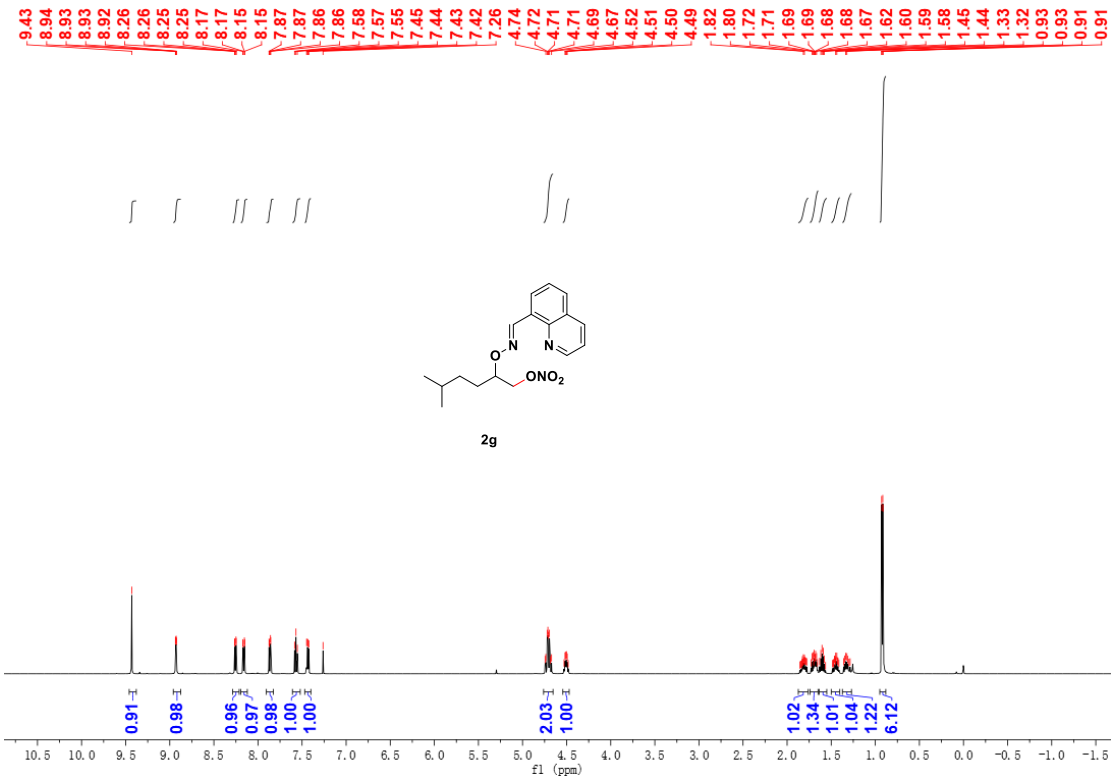
129.77
128.40
126.48
126.31
121.55
84.16
77.41
77.16
76.91
72.65
29.51
18.63
18.31
ONO2
2f
f1 (ppm)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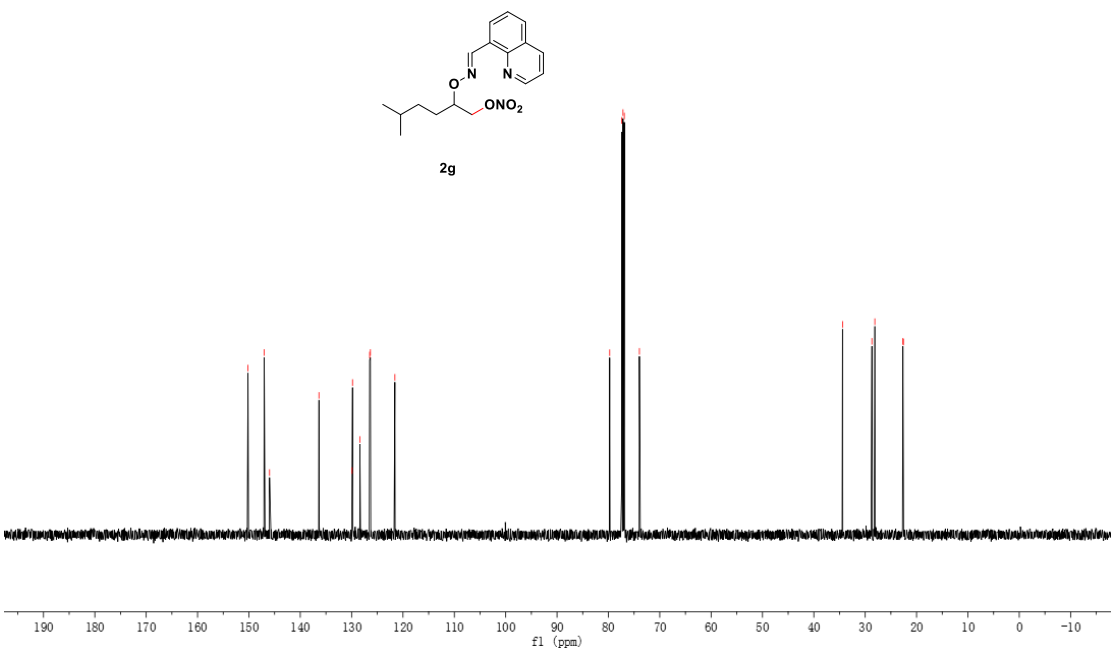
8.94
8.93
8.93
8.92
8.26
8.26
8.25
8.25
8.17
8.17
8.15
8.15
7.87
7.87
7.86
7.86
7.58
7.57
7.55
7.45
7.44
7.43
7.42
7.26
4.74
4.72
4.71
4.71
4.69
4.67
4.52
4.51
4.50
4.49
1.82
1.80
1.72
1.71
1.69
1.69
1.68
1.68
1.67
1.62
1.60
1.59
1.58
1.45
1.44
1.33
1.32
0.93
0.93
0.91
0.91
N
O
N
ONO2
2g
0.91
0.98
0.96
0.97
0.98
1.00
1.00
2.03
1.00
1.02
1.34
1.01
1.04
1.22
6.12
f1 (ppm)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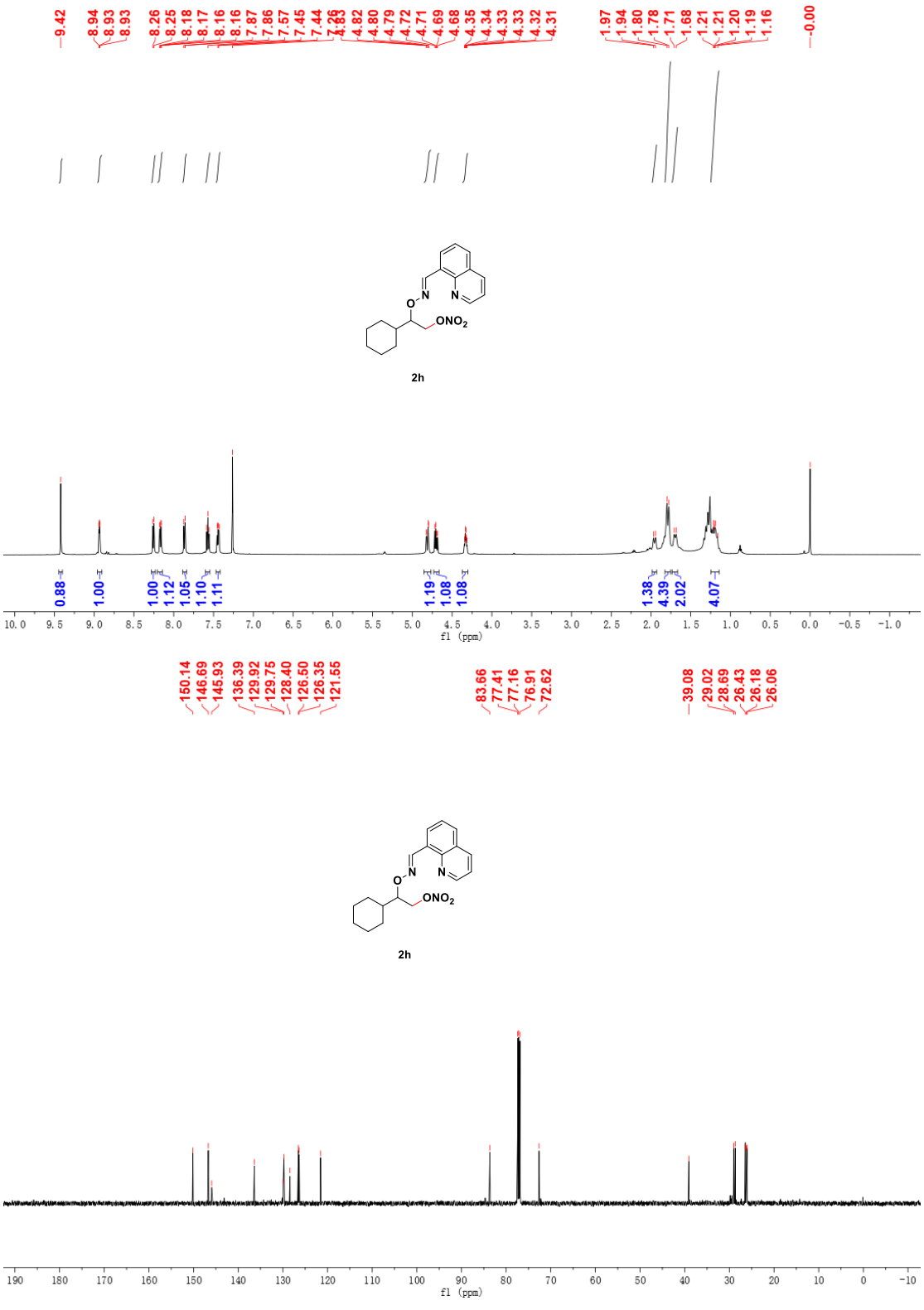
147.02
145.98
136.36
129.91
129.82
128.39
126.49
126.34
121.58
79.75
77.41
77.16
76.91
73.96
34.42
28.69
28.12
22.66
22.57
N
O
N
ONO2
2g
f1 (ppm)


9.42
8.94
8.93
8.93
8.26
8.25
8.18
8.17
8.16
8.16
7.87
7.86
7.57
7.45
7.44
4.82
4.80
4.79
4.72
4.71
4.69
4.68
4.35
4.34
4.33
4.33
4.32
4.31
1.97
1.94
1.80
1.78
1.71
1.68
1.21
1.21
1.20
1.19
1.16
0.00
ONO2
2h
0.88
1.00
1.00
1.12
1.05
1.10
1.11
1.19
1.08
1.08
1.38
4.39
2.02
4.07
f1 (ppm)
150.14
146.69
145.93
136.39
129.92
129.75
128.40
126.50
126.35
121.55
83.66
77.41
77.16
76.91
72.62
39.08
29.02
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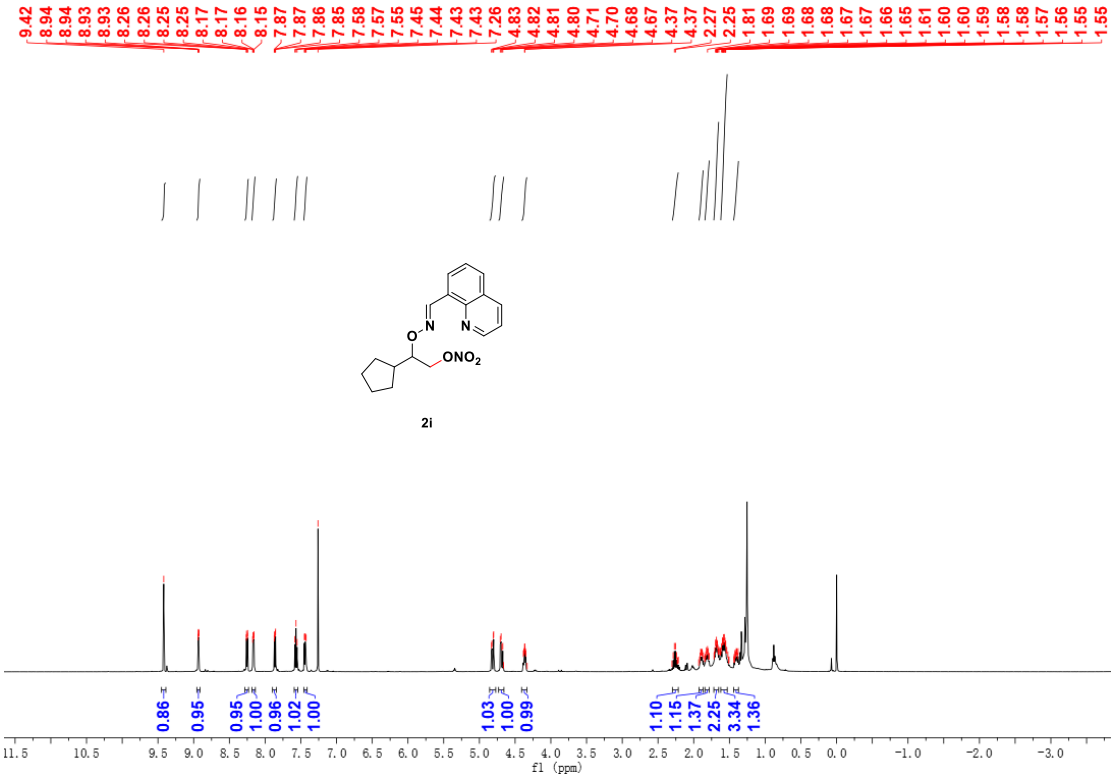
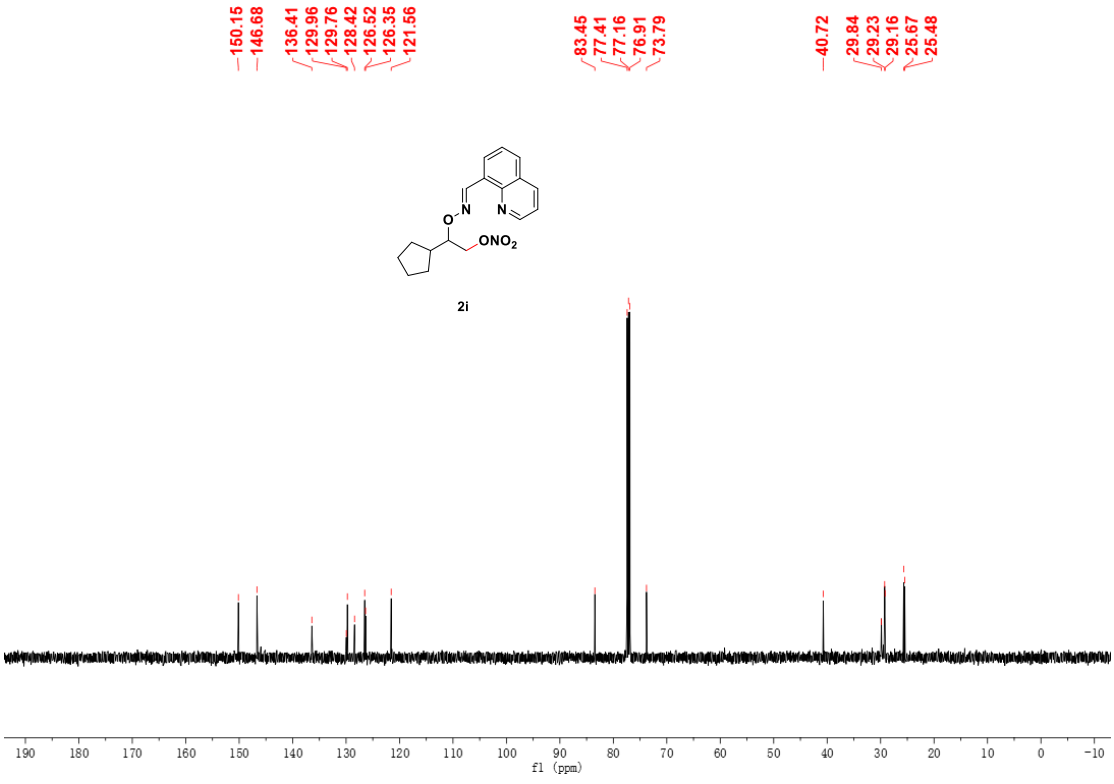
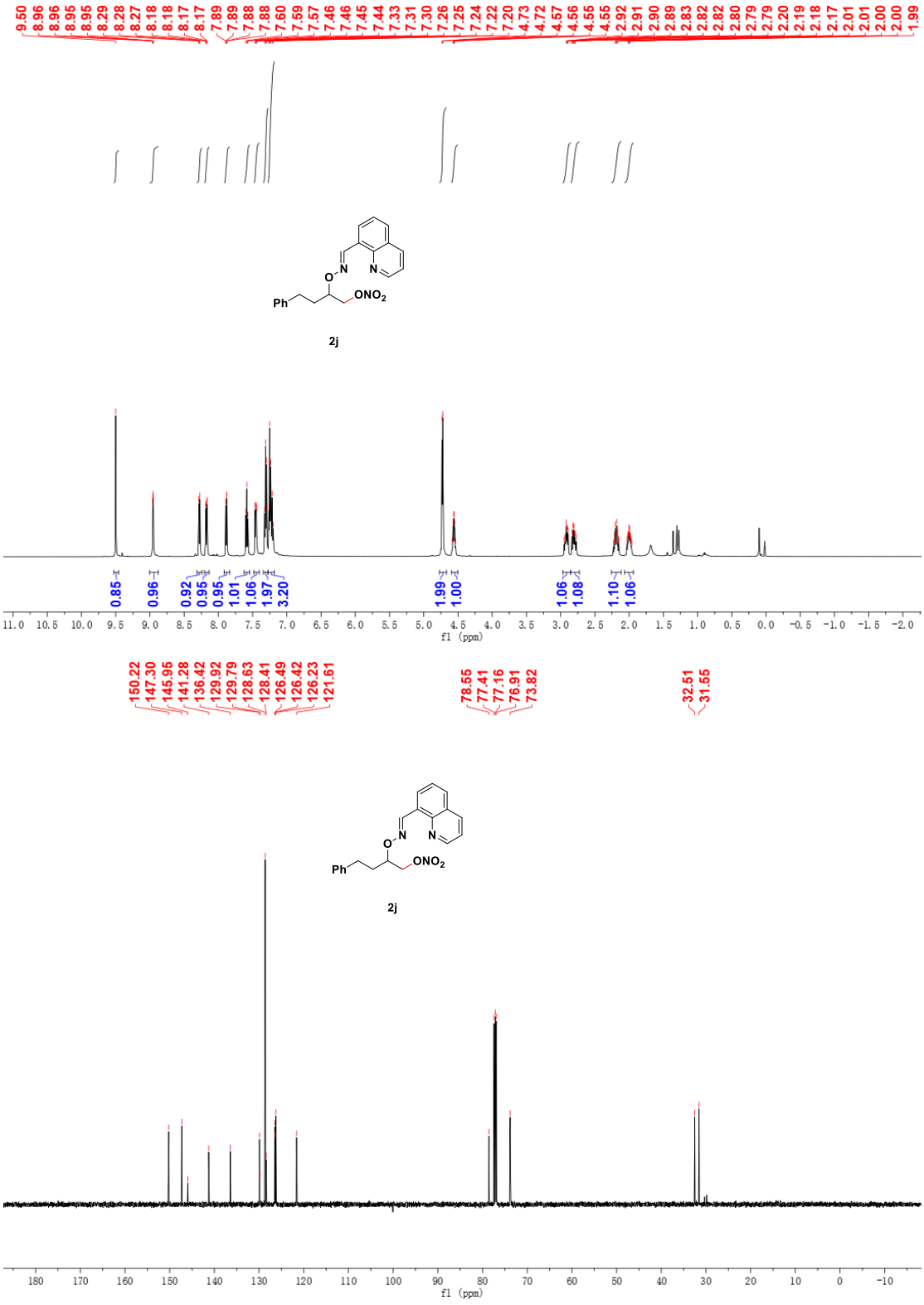
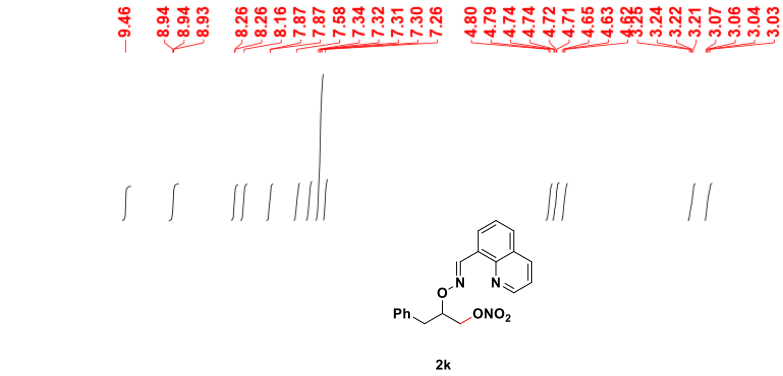
26.18
26.06
ONO2
2h
f1 (ppm)


9.42 8.94 8.94 8.93 8.93 8.26 8.26 8.25 8.25 8.17 8.17 8.16 8.15 7.87 7.87 7.86 7.85 7.58 7.57 7.55 7.45 7.44 7.43 7.43 7.26 4.83 4.82 4.81 4.80 4.71 4.70 4.68 4.67 4.37 4.37 2.27 2.25 1.81 1.69 1.69 1.68 1.68 1.67 1.67 1.66 1.65 1.61 1.60 1.60 1.59 1.58 1.58 1.57 1.56 1.55 1.55
ONO2
2i
0.86 0.95 0.95 1.00 0.96 1.02 1.00 1.03 1.00 0.99 1.10 1.15 1.37 2.25 3.34 1.36
f1 (ppm)


150.15 146.68 136.41 129.96 129.76 128.42 126.52 126.35 121.56 83.45 77.41 77.16 76.91 73.79 40.72 29.84 29.23 29.16 25.67 25.48
ONO2
2i
f1 (ppm)


9.50 8.96 8.96 8.95 8.95 8.29 8.28 8.27 8.18 8.18 8.17 8.17 7.89 7.89 7.88 7.88 7.60 7.59 7.57 7.46 7.46 7.45 7.44 7.33 7.31 7.30 7.26 7.25 7.24 7.22 7.20 4.73 4.72 4.57 4.56 4.55 4.55 2.92 2.91 2.90 2.89 2.83 2.82 2.82 2.80 2.79 2.79 2.20 2.19 2.18 2.17 2.01 2.01 2.00 2.00 1.99
Ph
ONO2
2j
0.85 0.96 0.92 0.95 0.95 1.01 1.06 1.97 3.20 1.99 1.00 1.06 1.08 1.10 1.06
f1 (ppm)
150.22 147.30 145.95 141.28 136.42 129.92 129.79 128.63 128.41 126.49 126.42 126.23 121.61 78.55 77.41 77.16 76.91 73.82 32.51 31.55
Ph
ONO2
2j
f1 (ppm)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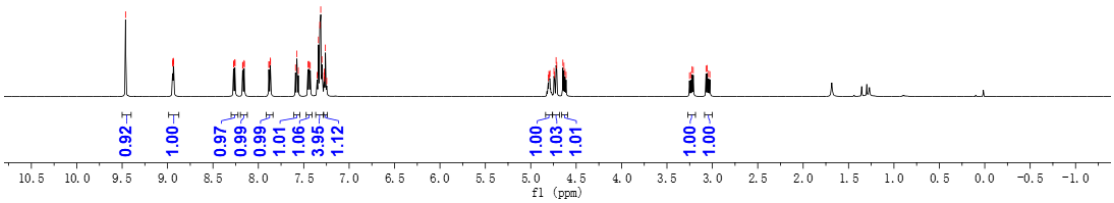
8.94
8.93
8.26
8.26
8.16
7.87
7.87
7.58
7.34
7.32
7.31
7.30
7.26
4.80
4.79
4.74
4.74
4.72
4.71
4.65
4.63
3.24
3.22
3.21
3.07
3.06
3.04
3.03
Ph
ONO2

2k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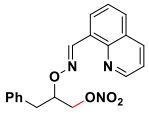
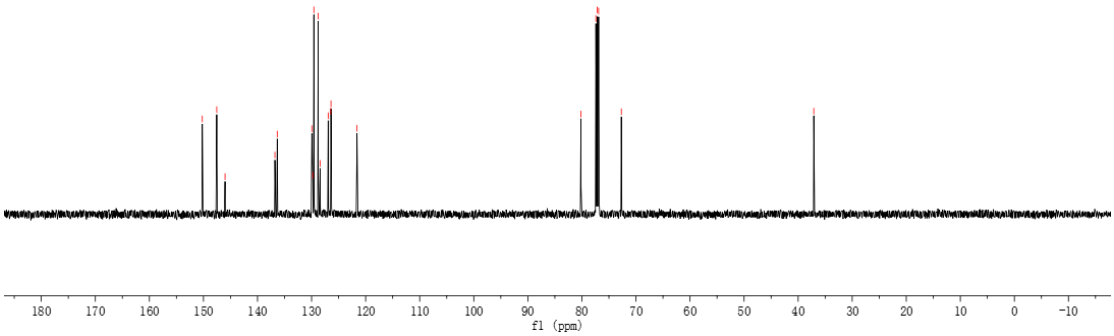
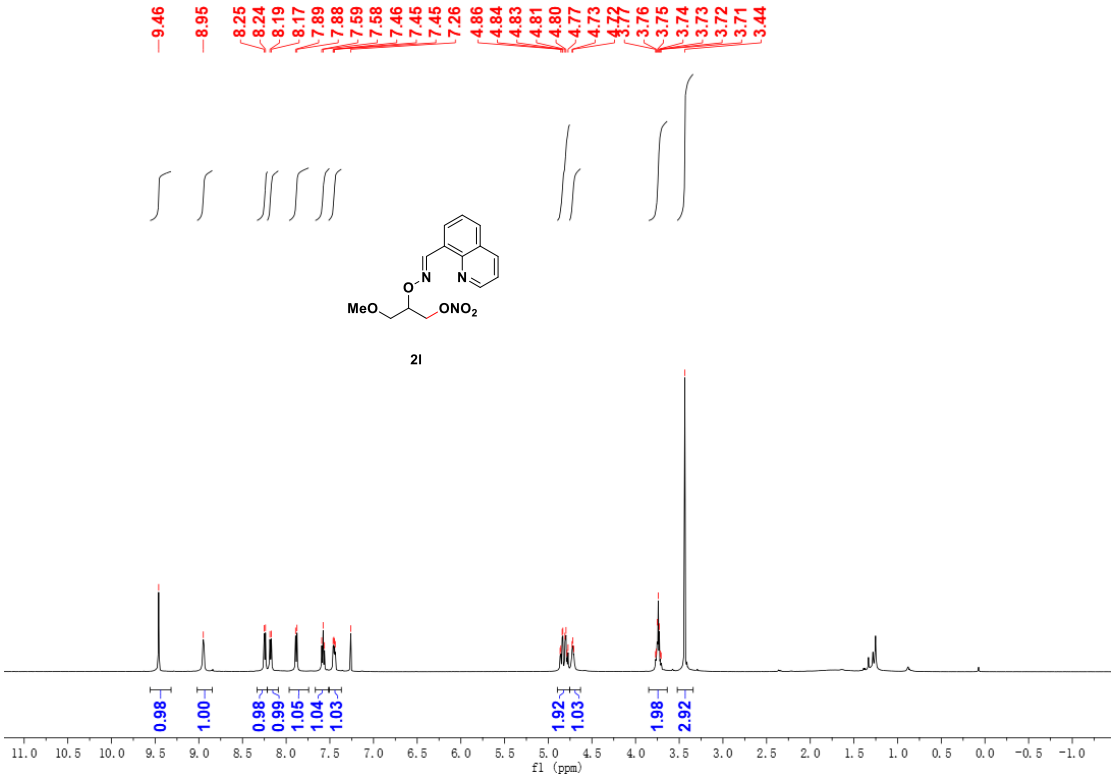
0.97
0.99
0.99
1.01
1.06
3.95
1.12
1.00
1.03
1.01
1.00
1.00
f1 (ppm)


150.22
147.54
146.01
136.78
136.35
129.95
129.78
129.58
128.77
128.41
126.93
126.45
126.41
121.60
80.17
77.41
77.16
76.91
72.71
37.11


Ph
ONO2

2k


f1 (ppm)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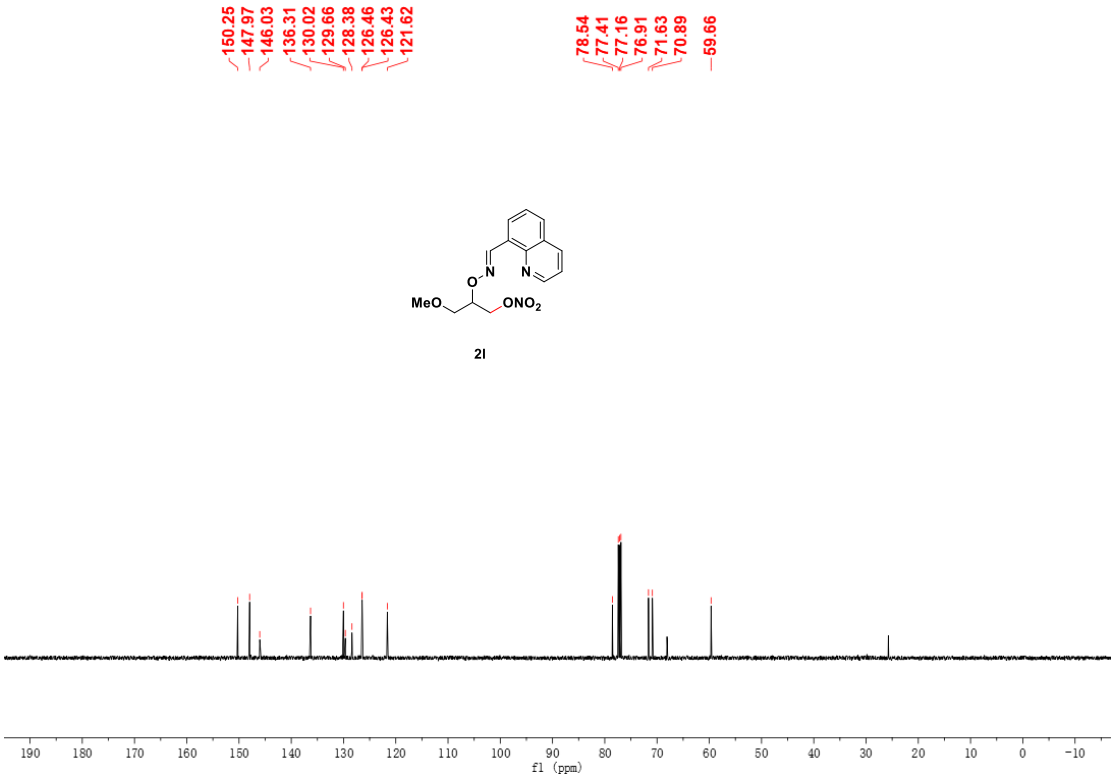
8.25
8.24
8.19
8.17
7.89
7.88
7.59
7.58
7.46
7.45
7.45
7.26
4.86
4.84
4.83
4.81
4.80
4.77
4.73
3.76
3.75
3.74
3.73
3.72
3.71
3.44
MeO
ONO2
2l
0.98
1.00
0.98
0.99
1.05
1.04
1.03
1.92
1.03
1.98
2.92
f1 (ppm)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129.66
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126.46
126.43
121.62
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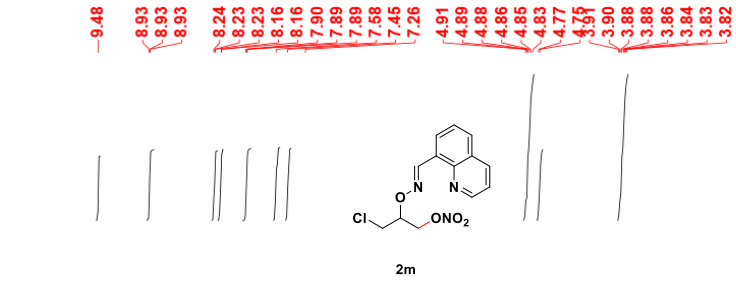
77.41
77.16
76.91
71.63
70.89
59.66
MeO
ONO2
2l
f1 (ppm)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8.23
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8.16
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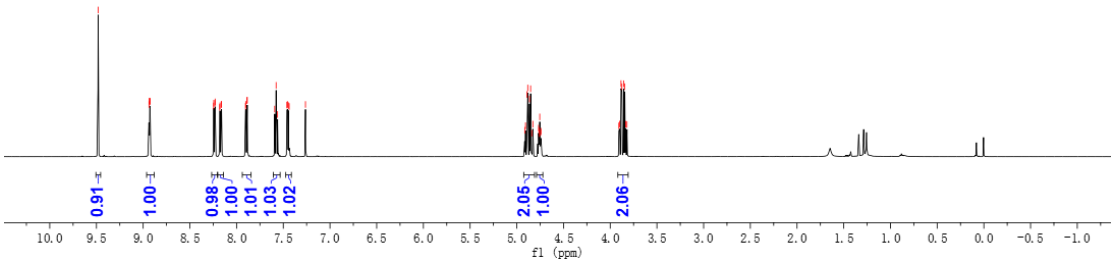
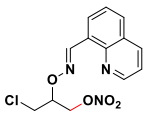
7.89
7.89
7.58
7.45
7.26
4.91
4.89
4.88
4.86
4.85
4.83
4.77
3.91
3.90
3.88
3.88
3.86
3.84
3.83
3.82
N
O
N
Cl
ONO2

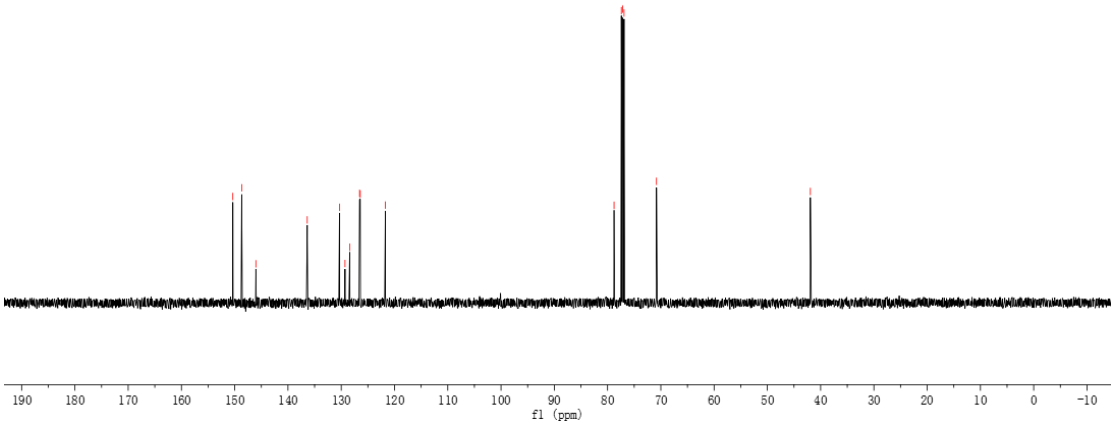
2m

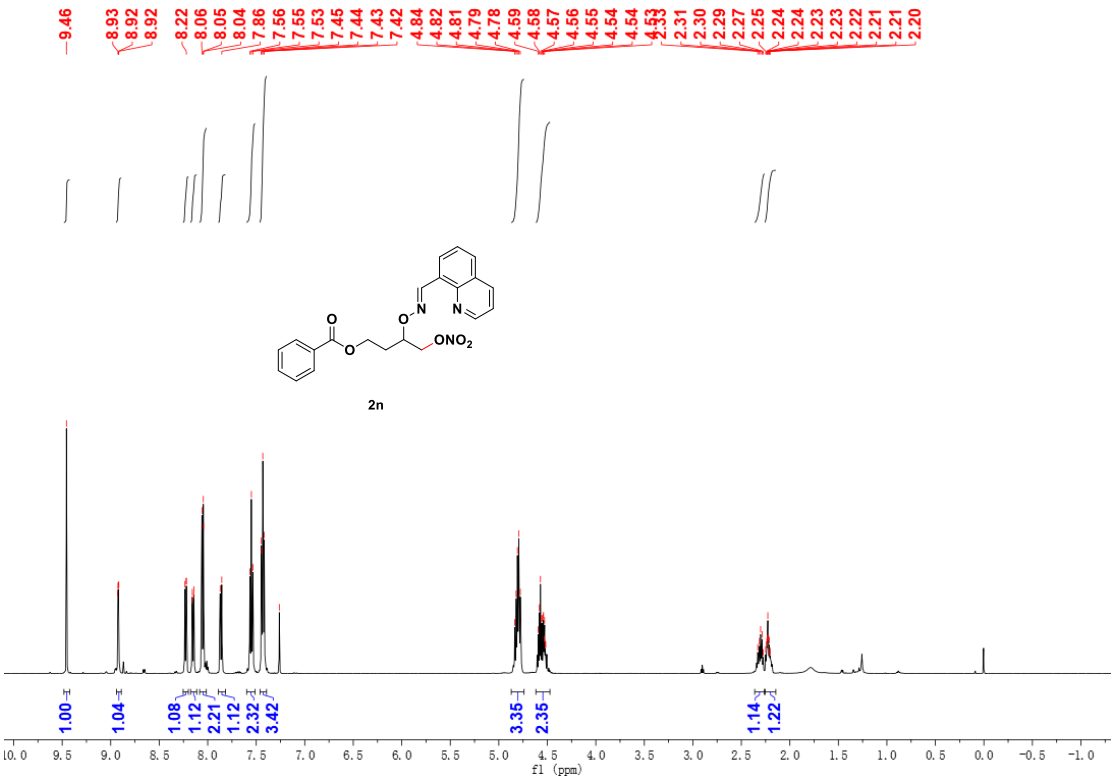

0.91
1.00
0.98
1.00
1.01
1.03
1.02
2.05
1.00
2.06
f1 (ppm)

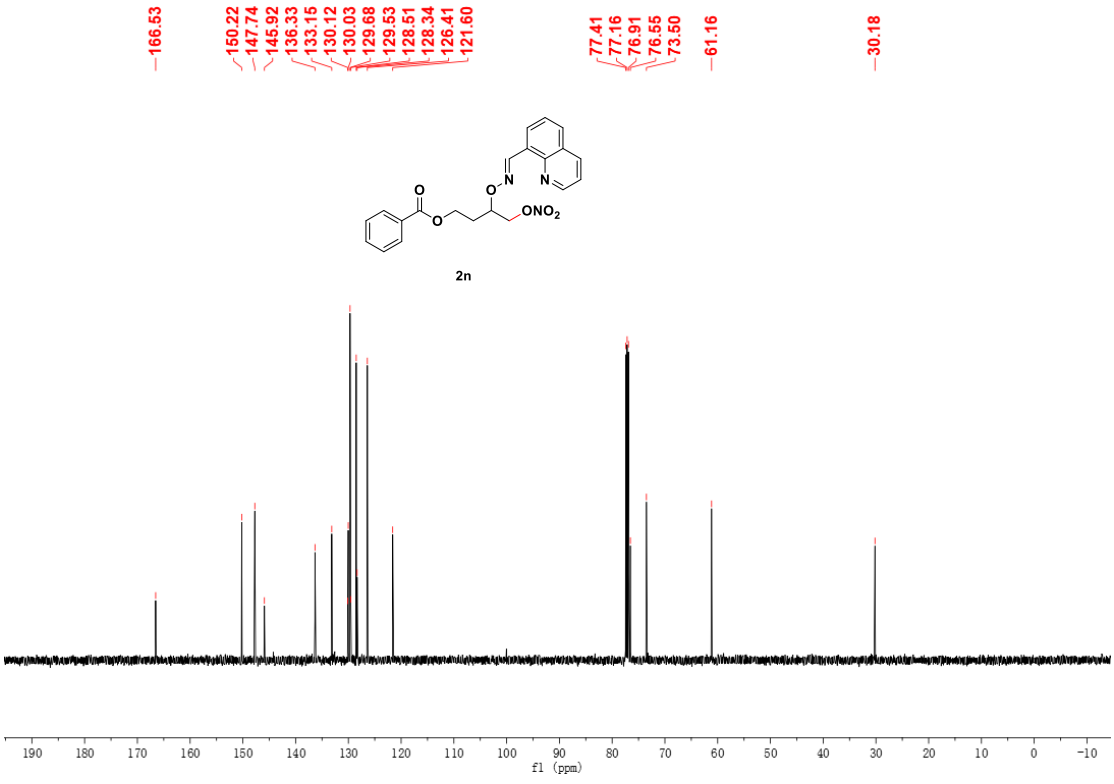

150.35
148.69
146.03
136.39
130.33
129.29
128.41
126.56
126.43
121.72
78.78
77.41
77.16
76.91
70.82
41.93

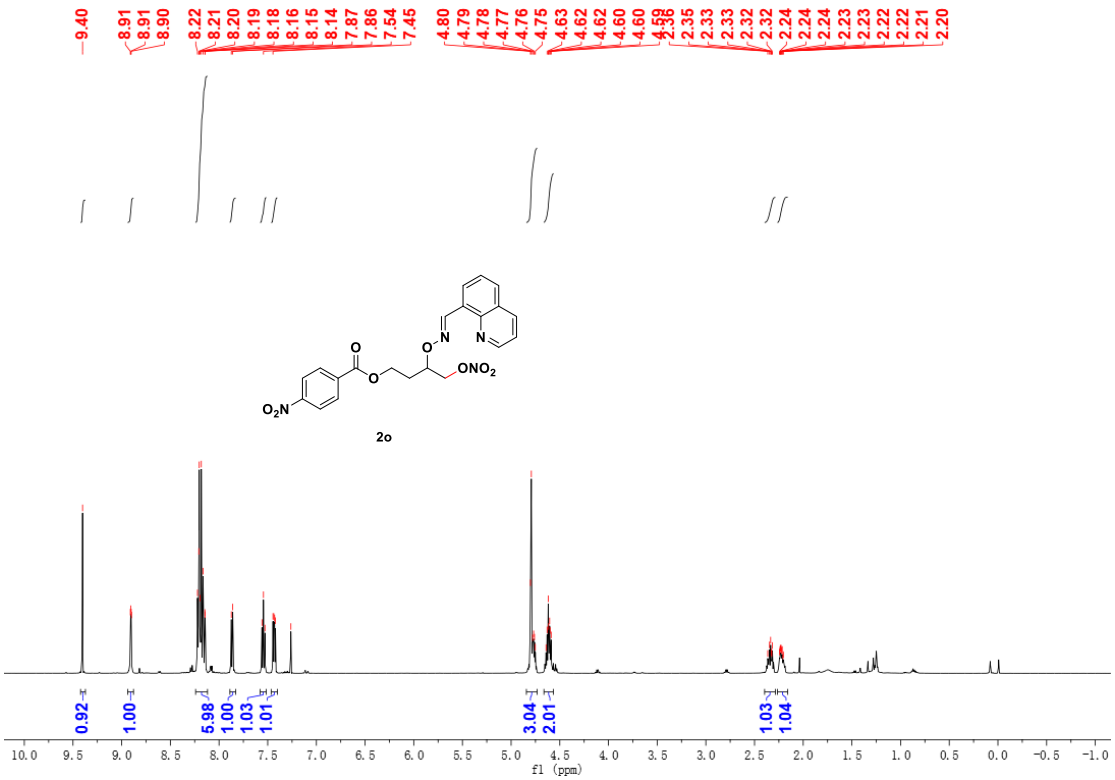

N
O
N
Cl
ONO2

2m

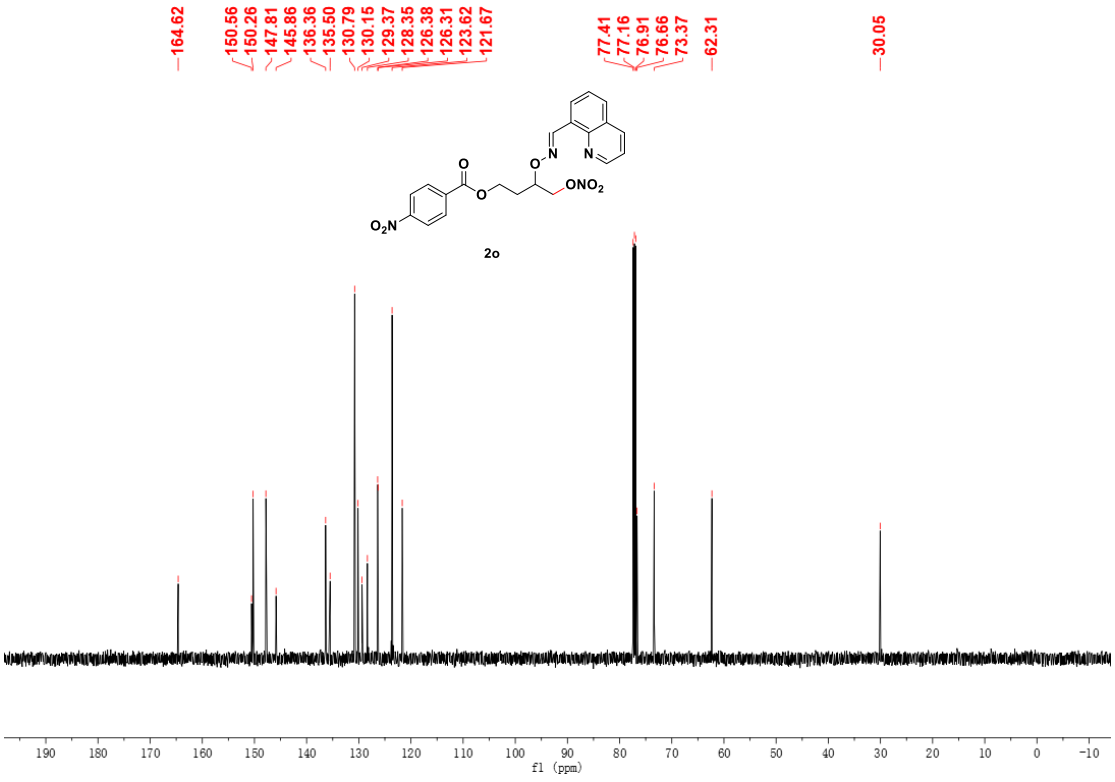

f1 (ppm)


9.46
8.93
8.92
8.92
8.22
8.06
8.05
8.04
7.86
7.56
7.55
7.53
7.45
7.44
7.43
7.42
4.84
4.82
4.81
4.79
4.78
4.59
4.58
4.57
4.56
4.55
4.54
4.54
4.53
2.31
2.30
2.29
2.27
2.25
2.24
2.24
2.23
2.23
2.22
2.21
2.21
2.20
O
O
O-N
N
ONO2
2n
1.00
1.04
1.08
1.12
2.21
1.12
2.32
3.42
3.35
2.35
1.14
1.22
f1 (ppm)


166.53
150.22
147.74
145.92
136.33
133.15
130.12
130.03
129.68
129.53
128.51
128.34
126.41
121.60
77.41
77.16
76.91
76.55
73.50
61.16
30.18
O
O
O-N
N
ONO2
2n
f1 (ppm)


9.40
8.91
8.91
8.90
8.22
8.21
8.20
8.19
8.18
8.16
8.15
8.14
7.87
7.86
7.54
7.45
4.80
4.79
4.78
4.77
4.76
4.75
4.63
4.62
4.62
4.60
4.60
4.59
4.58
2.35
2.33
2.33
2.32
2.32
2.24
2.24
2.24
2.23
2.23
2.22
2.22
2.21
2.20
0.92
1.00
5.98
1.00
1.03
1.01
3.04
2.01
1.03
1.04
f1 (ppm)

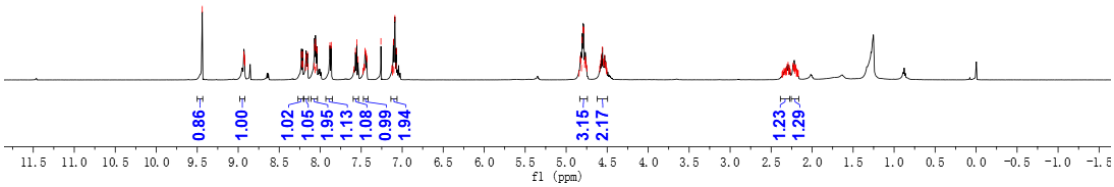
2o


164.62
150.56
150.26
147.81
145.86
136.36
135.50
130.79
130.15
129.37
128.35
126.38
126.31
123.62
121.67
77.41
77.16
76.91
76.66
73.37
62.31
30.05
f1 (ppm)

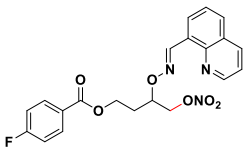
2o


9.44 8.93 8.93 8.92 8.92 8.23 8.23 8.21 8.21 8.17 8.17 8.16 8.15 8.08 8.07 8.06 8.05 8.04 7.88 7.87 7.57 7.55 7.54 7.47 7.46 7.45 7.44 7.44 7.43 7.26 7.11 7.09 7.08 7.07 4.83 4.82 4.80 4.80 4.78 4.77 4.77 4.76 4.76 4.58 4.57 4.56 4.55 4.54 4.53 4.53 4.52 4.51 2.31 2.29 2.23 2.23
O
N
O
N
O
ONO2
O
F

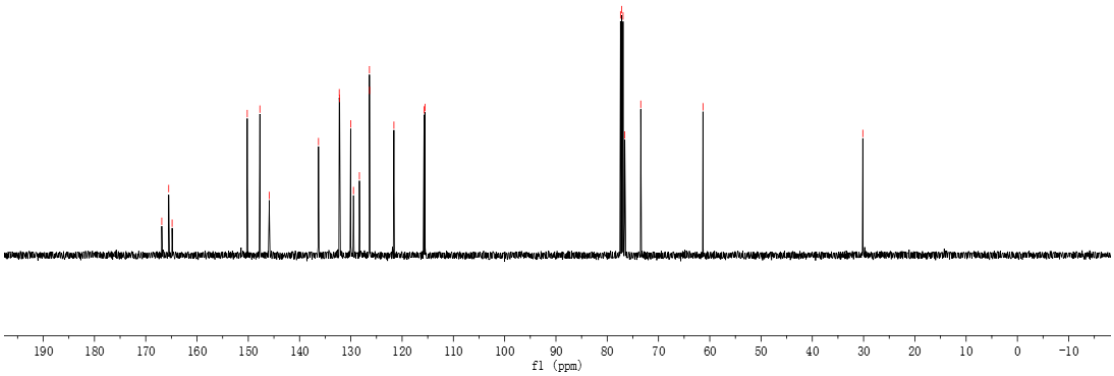
2p

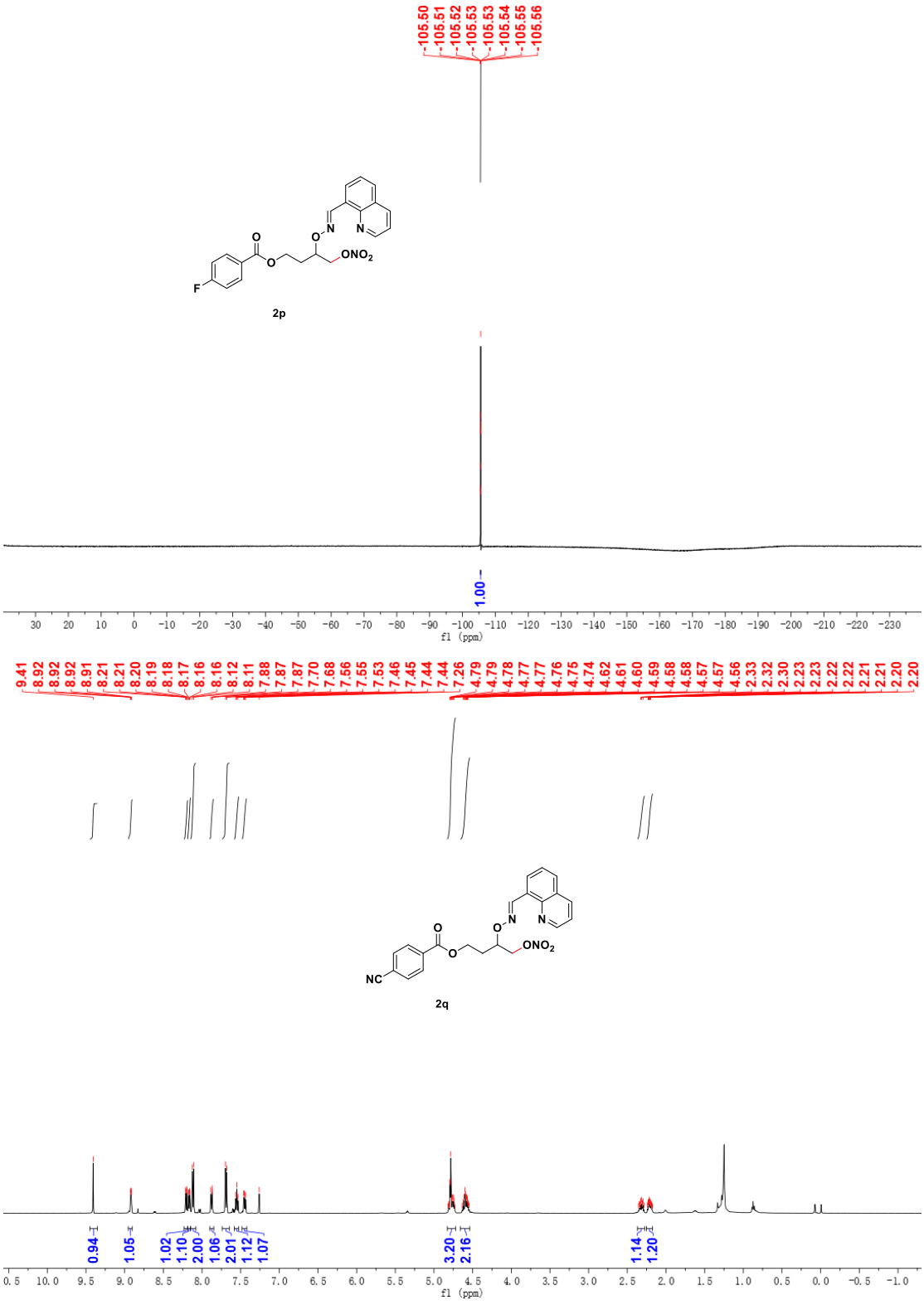

0.86 1.00 1.02 1.05 1.95 1.13 1.08 0.99 1.94 3.15 2.17 1.23 1.29
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

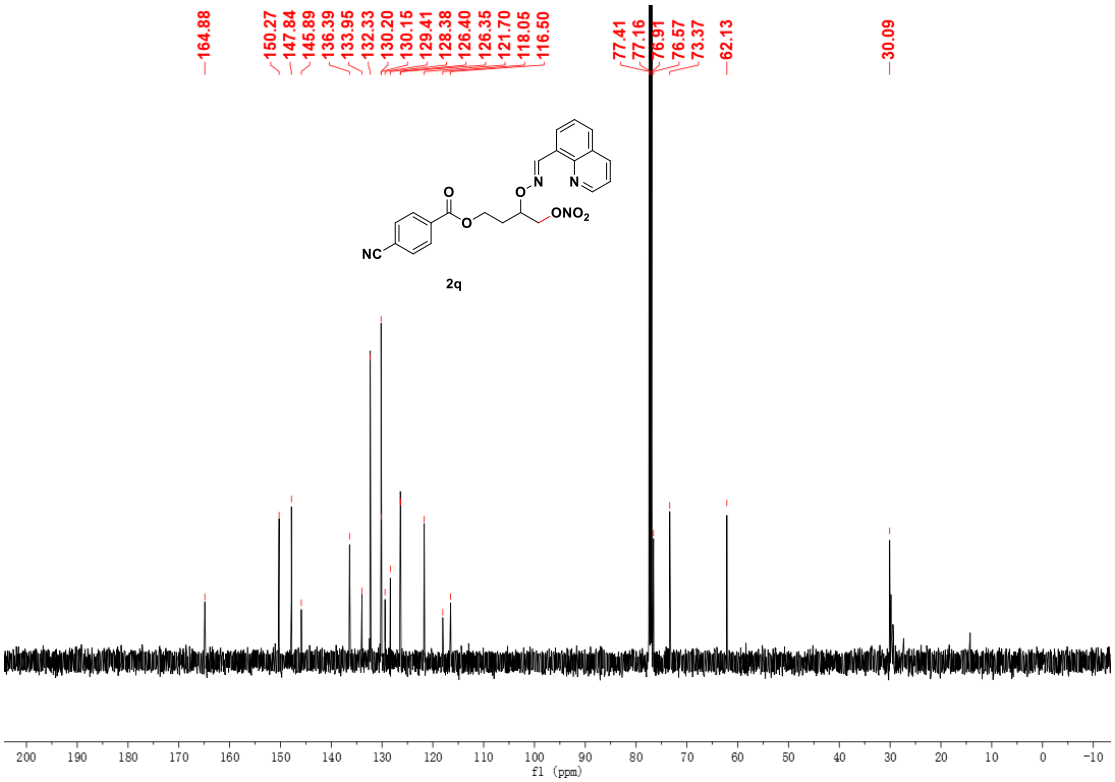

166.88 165.52 164.86 150.20 147.74 145.90 136.31 132.26 132.18 130.04 129.49 128.33 126.37 126.35 121.60 115.72 115.54 77.41 77.16 76.91 76.57 73.46 61.33 30.15


O
N
O
N
O
ONO2
O
F

2p


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-105.50
-105.51
-105.52
-105.53
-105.53
-105.54
-105.55
-105.56
F
O
O
N
O
N
ONO2
2p
1.00
f1 (ppm)
9.41
8.92
8.92
8.92
8.91
8.21
8.21
8.20
8.19
8.18
8.17
8.16
8.16
8.12
8.11
7.88
7.87
7.87
7.70
7.68
7.56
7.55
7.53
7.46
7.45
7.44
7.44
7.26
4.79
4.79
4.78
4.77
4.77
4.76
4.75
4.74
4.62
4.61
4.60
4.59
4.58
4.58
4.57
4.57
4.56
2.33
2.32
2.30
2.23
2.23
2.22
2.22
2.21
2.21
2.20
2.20
NC
O
O
N
O
N
ONO2
2q
0.94
1.05
1.02
1.10
2.00
1.06
2.01
1.12
1.07
3.20
2.16
1.14
1.20
f1 (ppm)


164.88
150.27
147.84
145.89
136.39
133.95
132.33
130.20
130.15
129.41
128.38
126.40
126.35
121.70
118.05
116.50
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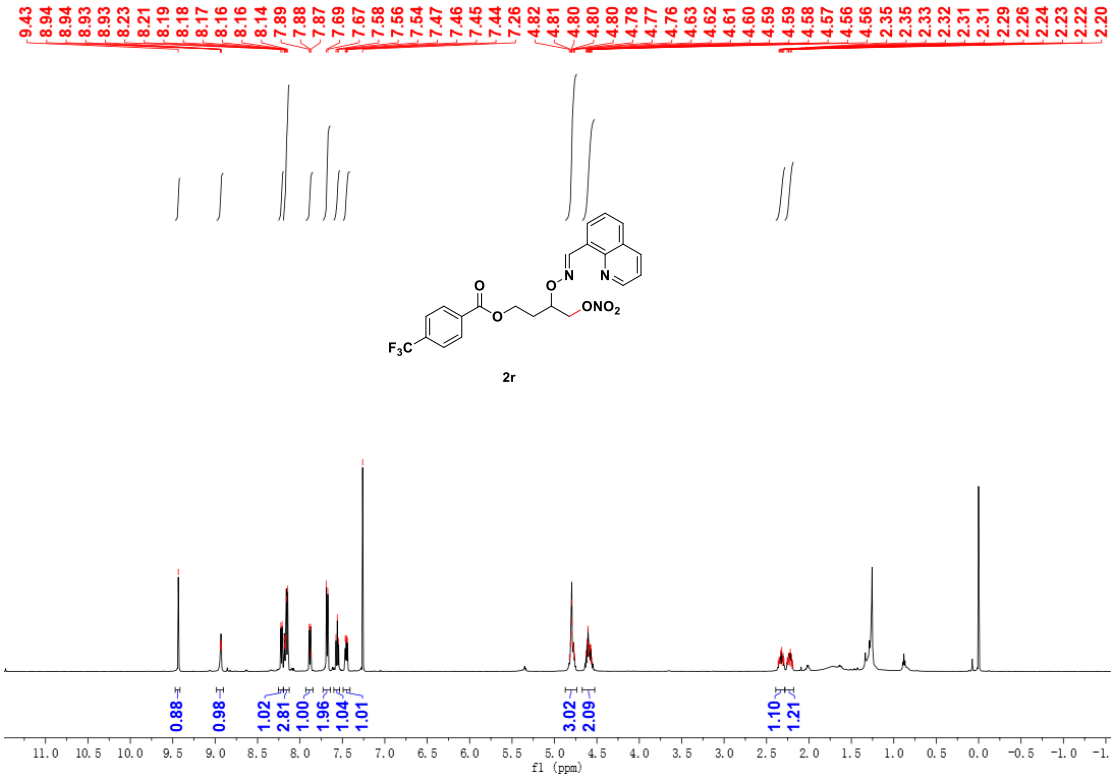
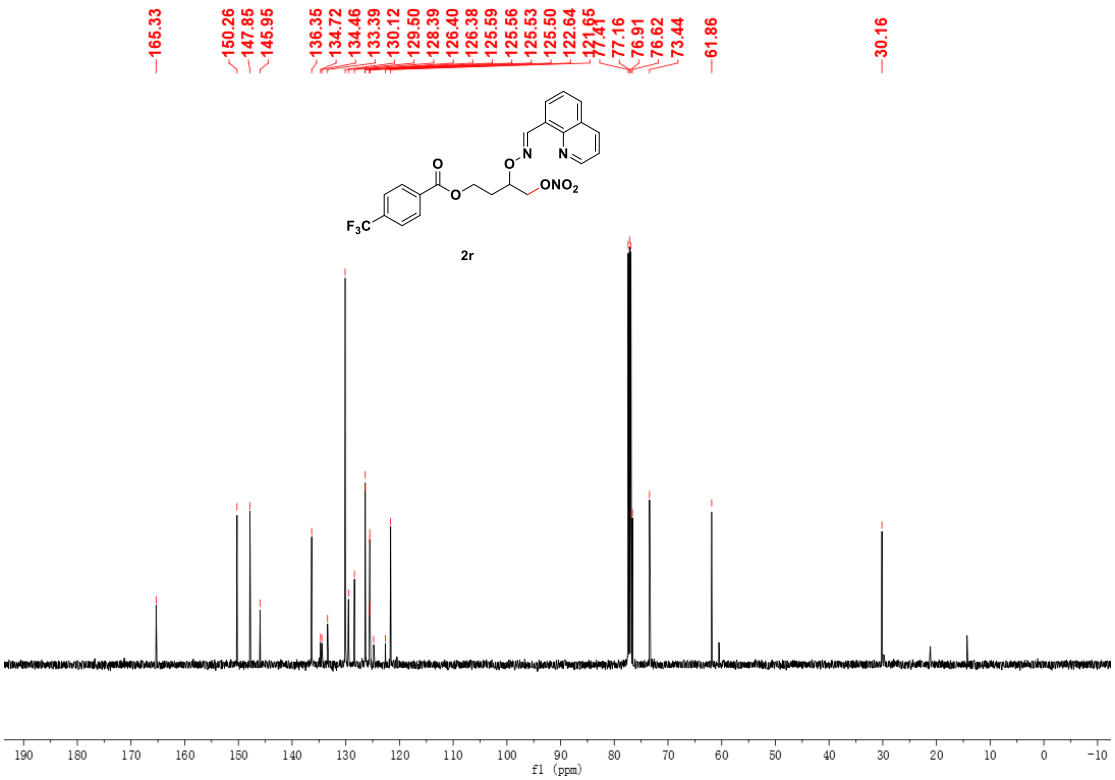
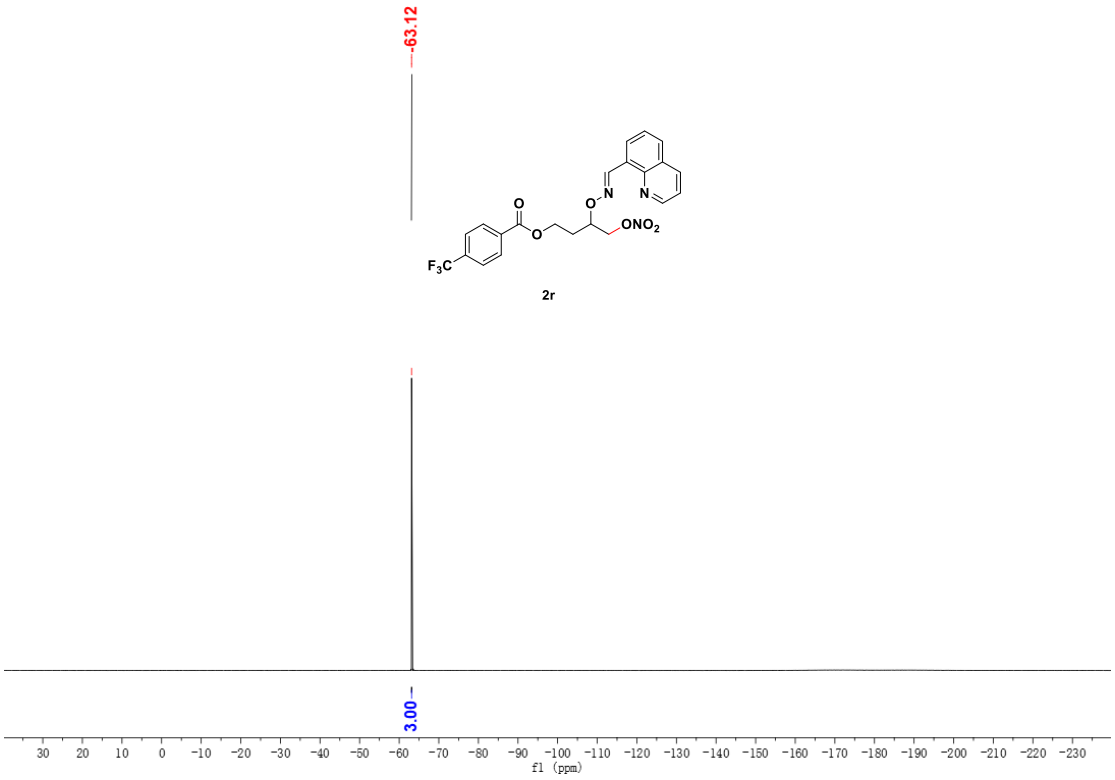
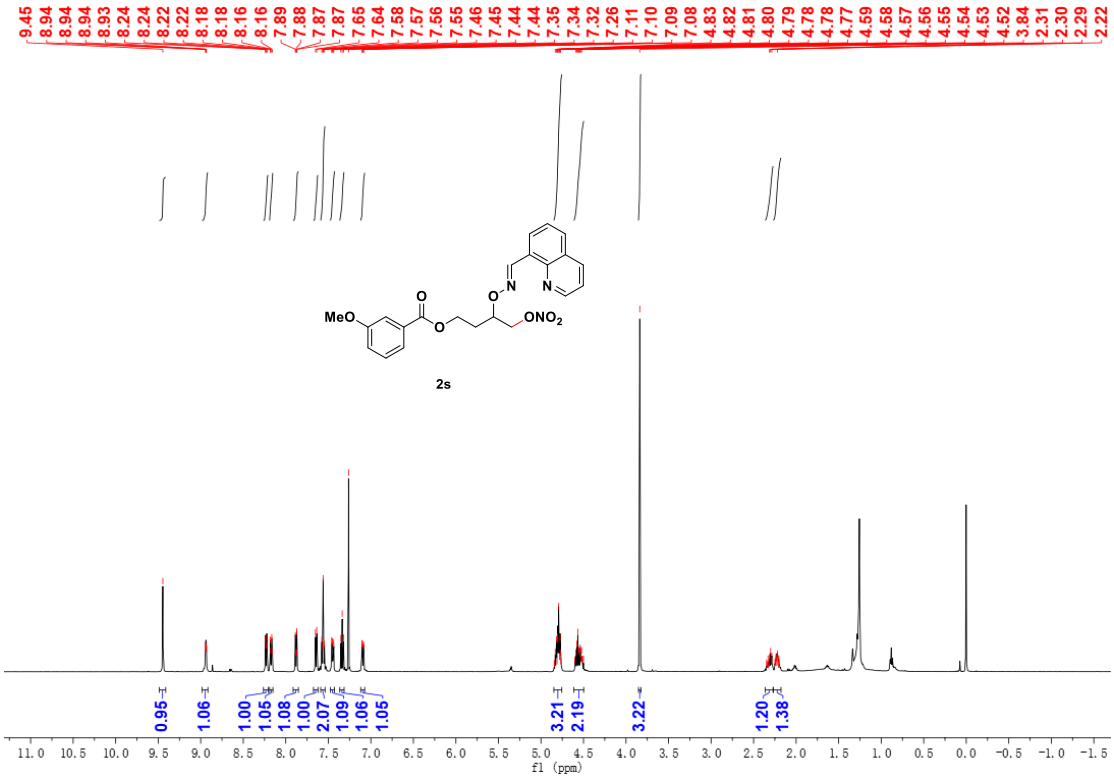
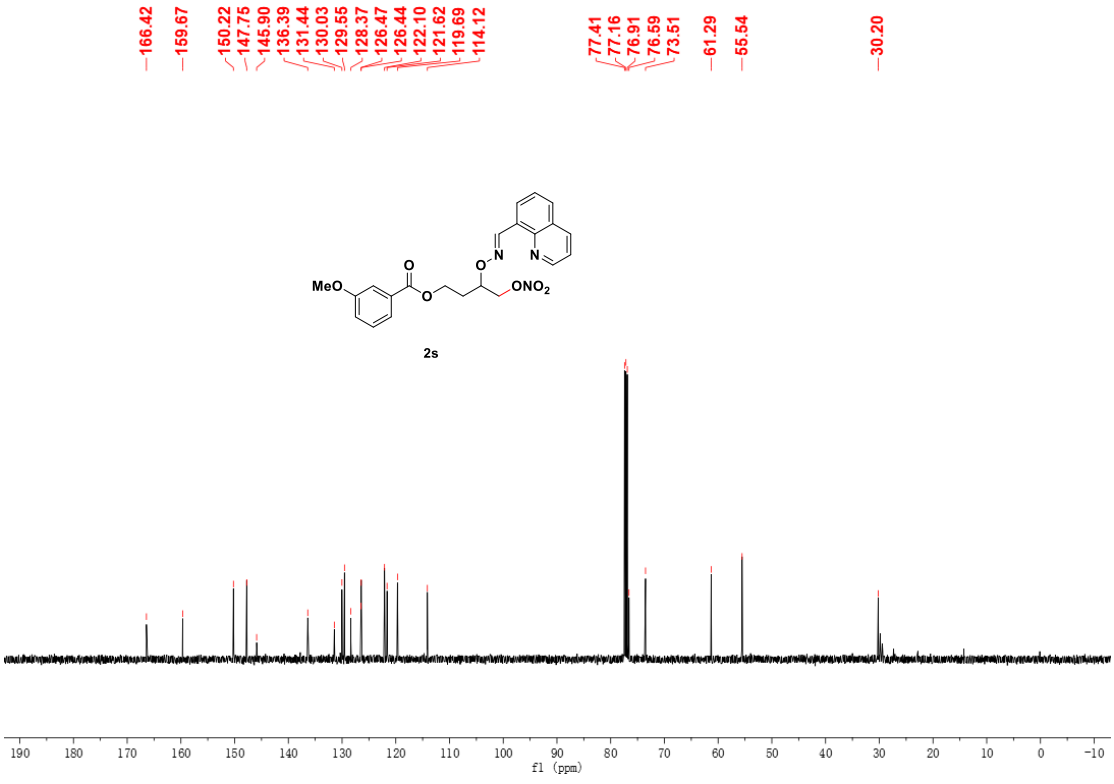
76.91
76.57
73.37
62.13
30.09
NC
ONO2
2q
f1 (ppm)

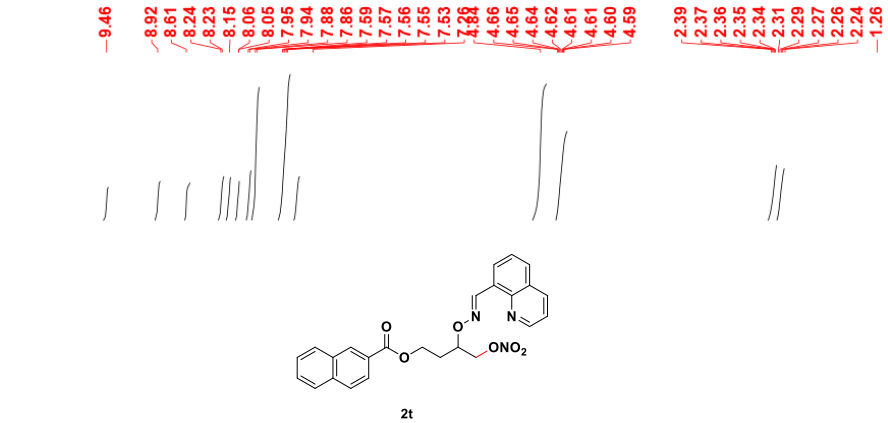

9.43
8.94
8.94
8.93
8.93
8.23
8.21
8.19
8.18
8.17
8.16
8.16
8.14
7.89
7.88
7.87
7.69
7.67
7.58
7.56
7.54
7.47
7.46
7.45
7.44
7.26
4.82
4.81
4.80
4.80
4.80
4.78
4.77
4.76
4.63
4.62
4.61
4.60
4.59
4.59
4.58
4.57
4.56
4.56
2.35
2.35
2.33
2.32
2.31
2.31
2.29
2.26
2.24
2.23
2.22
2.20
F3C
ONO2
2r
0.88
0.98
1.02
2.81
1.00
1.96
1.04
1.01
3.02
2.09
1.10
1.21
f1 (ppm)


165.33
150.26
147.85
145.95
136.35
134.72
134.46
133.39
130.12
129.50
128.39
126.40
126.38
125.59
125.56
125.53
125.50
122.64
121.65
77.41
77.16
76.91
76.62
73.44
61.86
30.16
O
N
O
N
O
ONO2
F3C
2r
f1 (ppm)

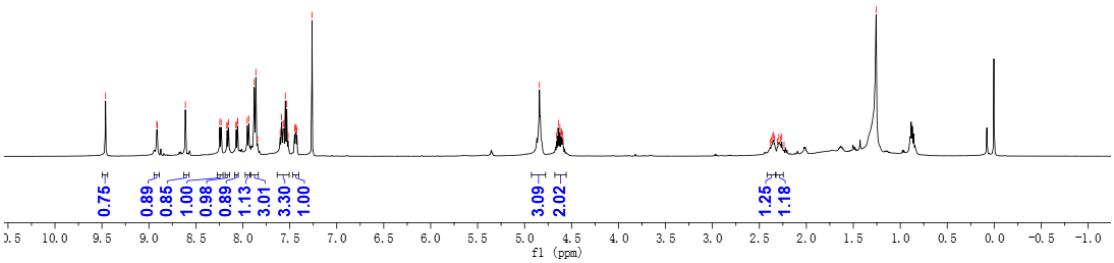

-63.12
O
N
O
N
O
ONO2
F3C
2r
3.00
f1 (ppm)


9.45 8.94 8.94 8.94 8.93 8.24 8.24 8.22 8.22 8.18 8.18 8.16 8.16 7.89 7.88 7.87 7.87 7.65 7.64 7.58 7.57 7.56 7.55 7.46 7.45 7.44 7.44 7.35 7.34 7.32 7.26 7.11 7.10 7.09 7.08 4.83 4.82 4.81 4.80 4.79 4.78 4.78 4.77 4.59 4.58 4.57 4.56 4.55 4.54 4.53 4.52 3.84 2.31 2.30 2.29 2.22
MeO
O
O
N
O
N
ONO2
2s
0.95 1.06 1.00 1.05 1.08 1.00 2.07 1.09 1.06 1.05 3.21 2.19 3.22 1.20 1.38
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

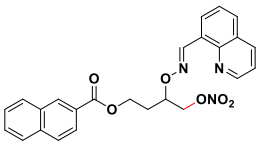

166.42 159.67 150.22 147.75 145.90 136.39 131.44 130.03 129.55 128.37 126.47 126.44 122.10 121.62 119.69 114.12
77.41 77.16 76.91 76.59 73.51 61.29 55.54 30.20
MeO
O
O
N
O
N
ONO2
2s
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


9.46
8.92
8.61
8.24
8.23
8.15
8.06
8.05
7.95
7.94
7.88
7.86
7.59
7.57
7.56
7.55
7.53
4.66
4.65
4.64
4.62
4.61
4.61
4.60
4.59
2.39
2.37
2.36
2.35
2.34
2.31
2.29
2.27
2.26
2.24
1.26
O
O
N
O
N
ONO2

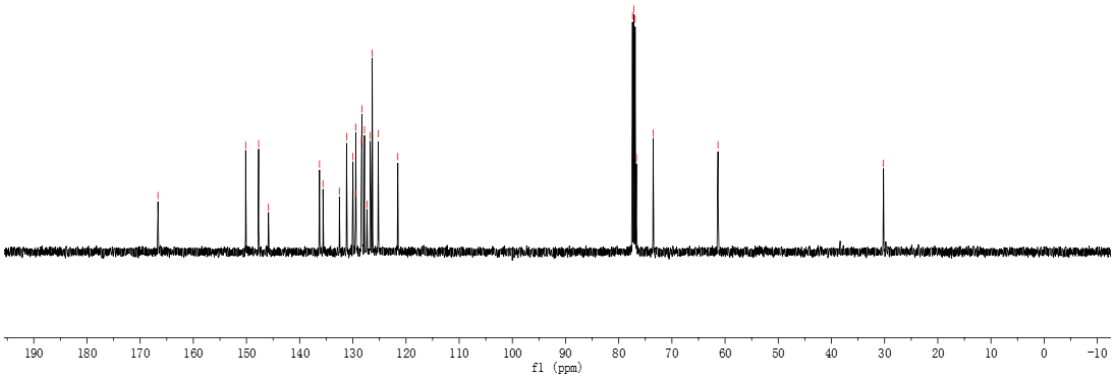
2t

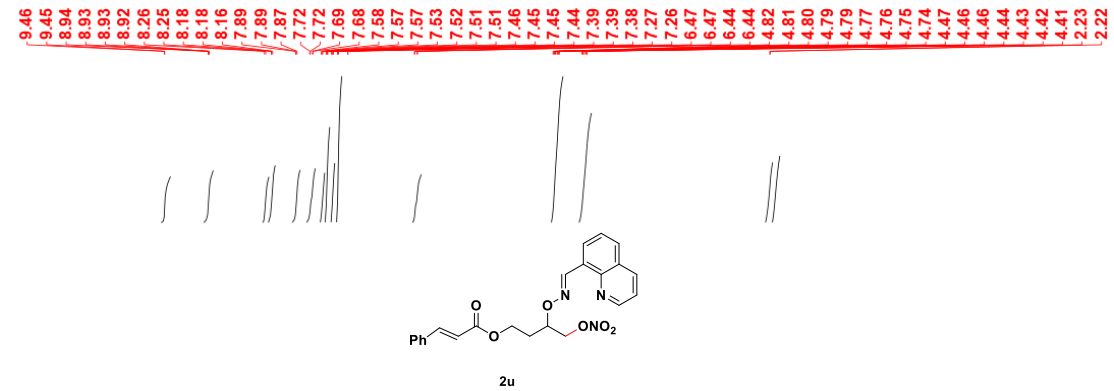

0.75
0.89
0.85
1.00
0.98
0.89
1.13
3.01
3.30
1.00
3.09
2.02
1.25
1.18
f1 (ppm)


166.65
150.16
147.72
145.87
136.26
135.61
132.53
131.17
129.98
129.49
129.45
128.36
128.28
127.81
127.34
126.73
126.36
125.23
121.54
77.41
77.16
76.90
76.67
73.53
61.33
30.21

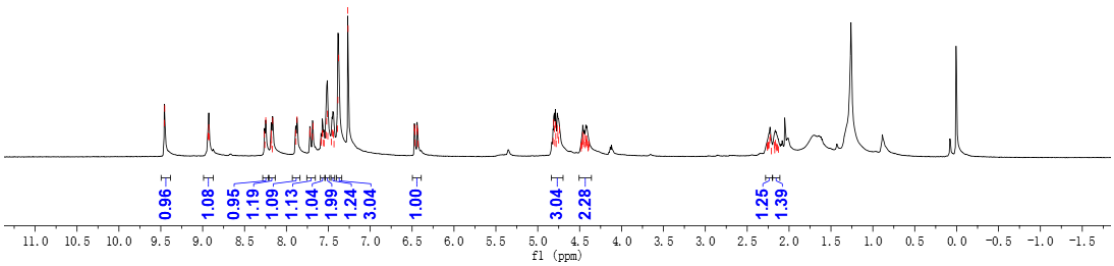

O
O
N
O
N
ONO2

2t

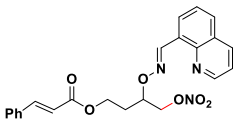

f1 (ppm)


9.46
9.45
8.94
8.93
8.93
8.92
8.26
8.25
8.18
8.18
8.16
7.89
7.89
7.87
7.72
7.72
7.69
7.68
7.58
7.57
7.57
7.53
7.52
7.51
7.51
7.46
7.45
7.45
7.44
7.39
7.39
7.38
7.27
7.26
6.47
6.47
6.44
6.44
4.82
4.81
4.80
4.79
4.79
4.77
4.76
4.75
4.74
4.47
4.46
4.46
4.44
4.43
4.42
4.41
2.23
2.22
O
N
O
N
O
Ph
O
ONO2

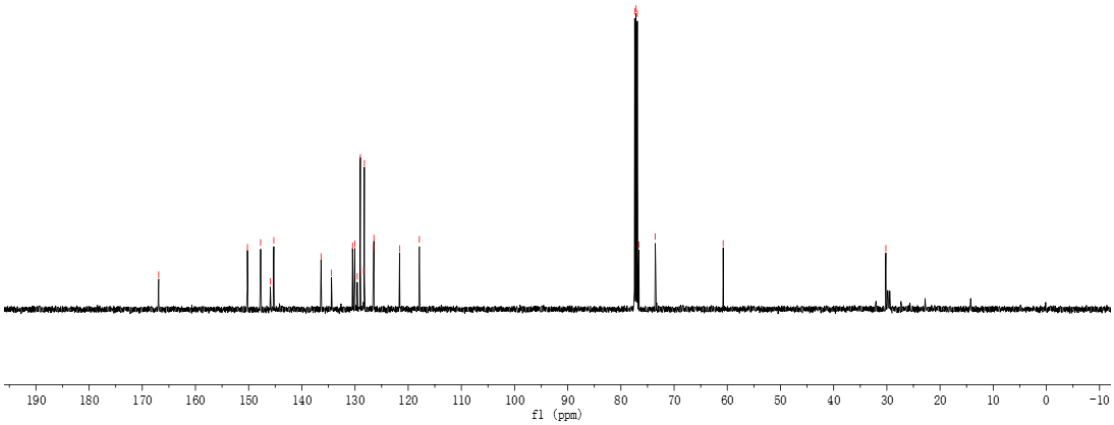
2u

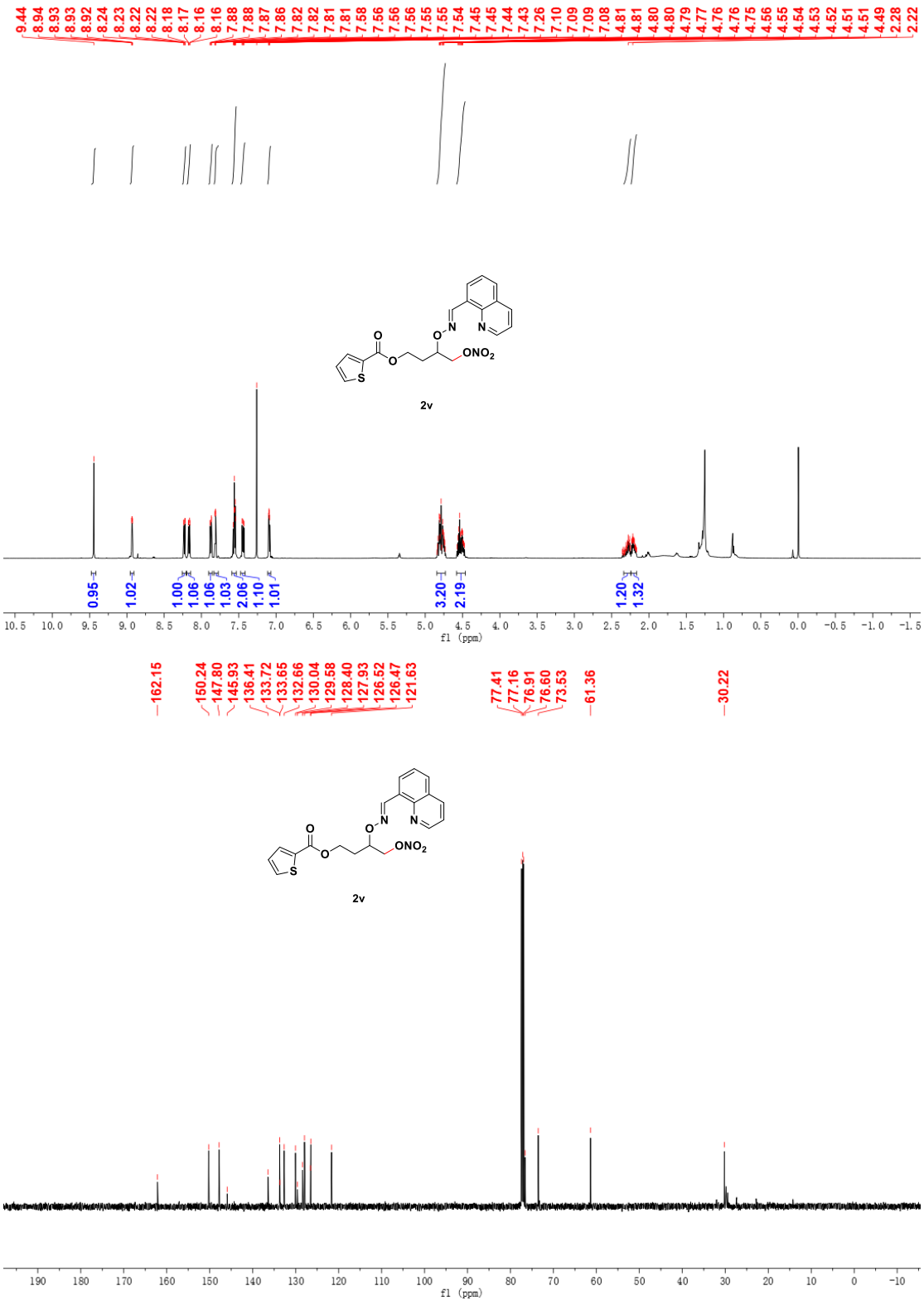

0.96
1.08
0.95
1.19
1.09
1.13
1.04
1.99
1.24
3.04
1.00
3.04
2.28
1.25
1.39
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

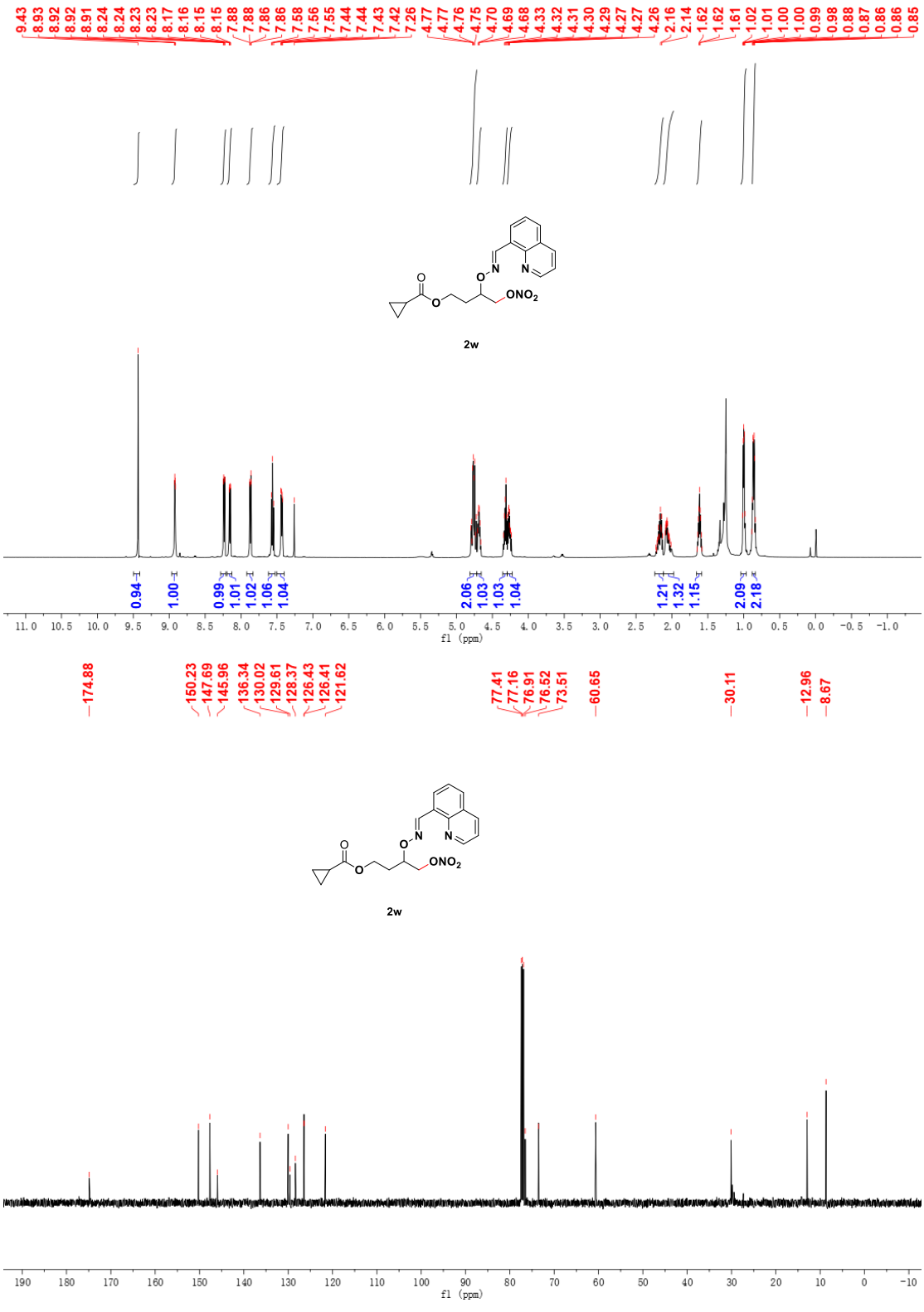

166.92
150.23
147.75
145.92
145.27
136.39
134.43
130.47
130.03
129.58
129.00
128.39
128.24
126.50
126.45
121.62
117.91
77.41
77.16
76.91
76.62
73.54
60.77
30.18

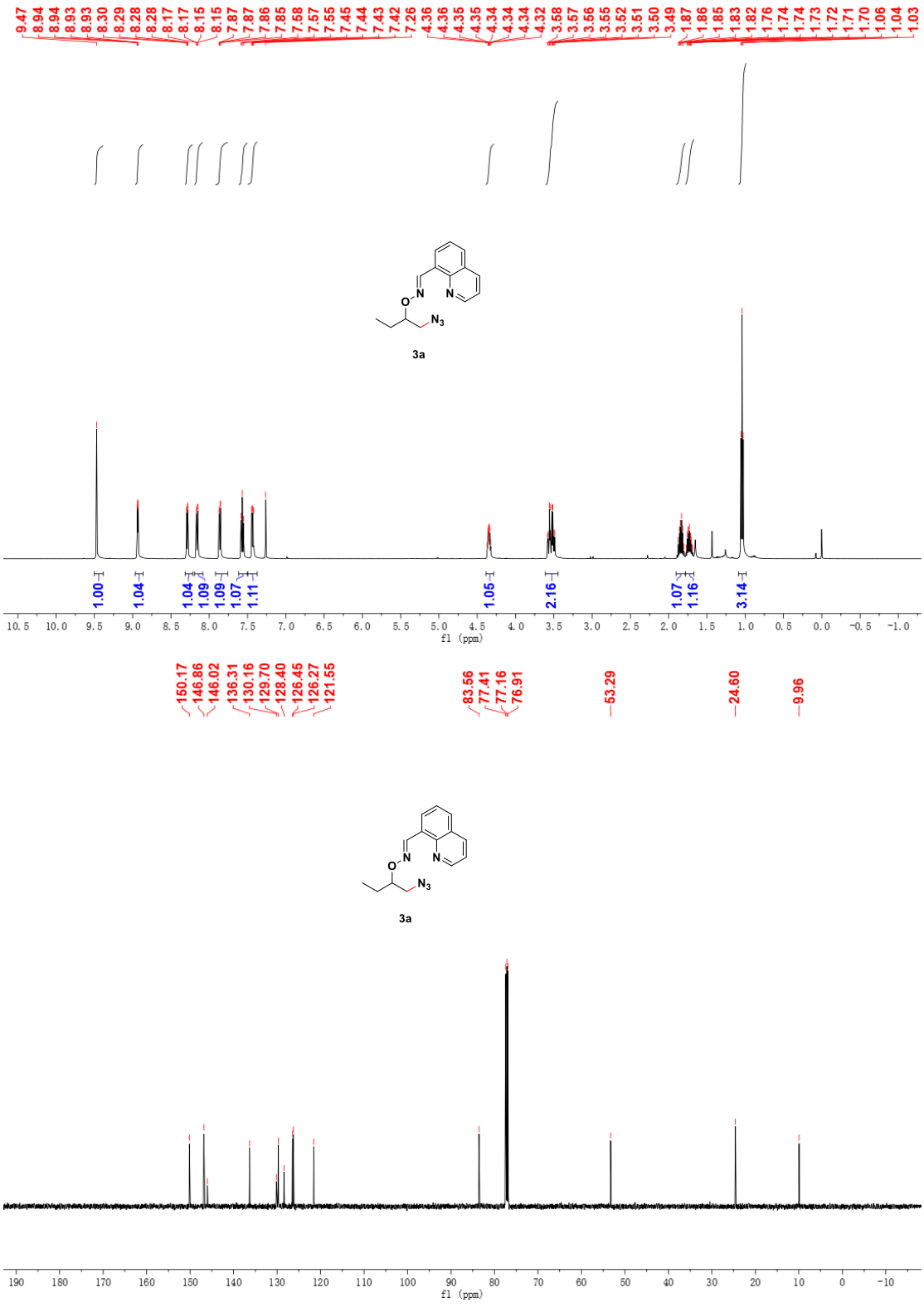

O
N
O
N
O
Ph
O
ONO2

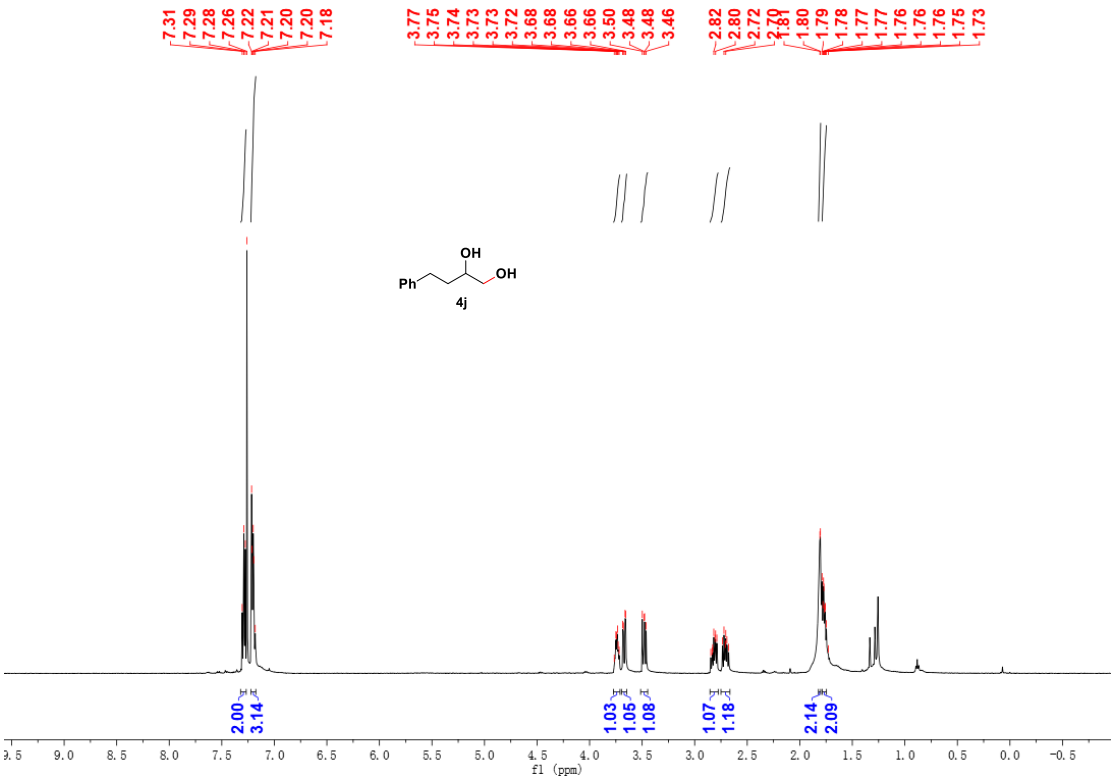
2u


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


9.44 8.94 8.93 8.93 8.92 8.24 8.23 8.22 8.22 8.18 8.17 8.16 8.16 7.88 7.88 7.87 7.86 7.82 7.82 7.81 7.81 7.58 7.56 7.56 7.56 7.55 7.55 7.54 7.45 7.45 7.44 7.43 7.26 7.10 7.09 7.09 7.08 4.81 4.81 4.80 4.80 4.79 4.77 4.76 4.76 4.75 4.56 4.55 4.54 4.53 4.52 4.51 4.51 4.49 2.28 2.22
2v
0.95 1.02 1.00 1.06 1.06 1.03 2.06 1.10 1.01 3.20 2.19 1.20 1.32
f1 (ppm)
162.15 150.24 147.80 145.93 136.41 133.72 133.65 132.66 130.04 129.58 128.40 127.93 126.52 126.47 121.63 77.41 77.16 76.91 76.60 73.53 61.36 30.22
2v
f1 (ppm)


9.43 8.93 8.92 8.92 8.91 8.24 8.24 8.23 8.23 8.17 8.16 8.15 8.15 7.88 7.88 7.86 7.86 7.58 7.56 7.55 7.44 7.44 7.43 7.42 7.26 4.77 4.77 4.76 4.75 4.70 4.69 4.68 4.33 4.32 4.31 4.30 4.29 4.27 4.27 4.26 2.16 2.14 1.62 1.62 1.61 1.02 1.01 1.00 1.00 0.99 0.98 0.88 0.87 0.86 0.86 0.85
2w
0.94 1.00 0.99 1.01 1.02 1.06 1.04 2.06 1.03 1.03 1.04 1.21 1.32 1.15 2.09 2.18
f1 (ppm)
174.88 150.23 147.69 145.96 136.34 130.02 129.61 128.37 126.43 126.41 121.62 77.41 77.16 76.91 76.52 73.51 60.65 30.11 12.96 8.67
2w
f1 (ppm)


9.47
8.94
8.94
8.93
8.93
8.30
8.29
8.28
8.28
8.17
8.17
8.15
8.15
7.87
7.87
7.86
7.85
7.58
7.57
7.55
7.45
7.44
7.43
7.42
7.26
4.36
4.36
4.35
4.35
4.34
4.34
4.34
4.32
3.58
3.57
3.56
3.55
3.52
3.51
3.50
3.49
1.87
1.86
1.85
1.83
1.82
1.76
1.74
1.74
1.73
1.72
1.71
1.70
1.06
1.04
1.03
3a
1.00
1.04
1.04
1.09
1.09
1.07
1.11
1.05
2.16
1.07
1.16
3.14
f1 (ppm)
150.17
146.86
146.02
136.31
130.16
129.70
128.40
126.45
126.27
121.55
83.56
77.41
77.16
76.91
53.29
24.60
9.96
3a
f1 (ppm)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7.29
7.28
7.26
7.22
7.21
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7.20
7.18
3.77
3.75
3.74
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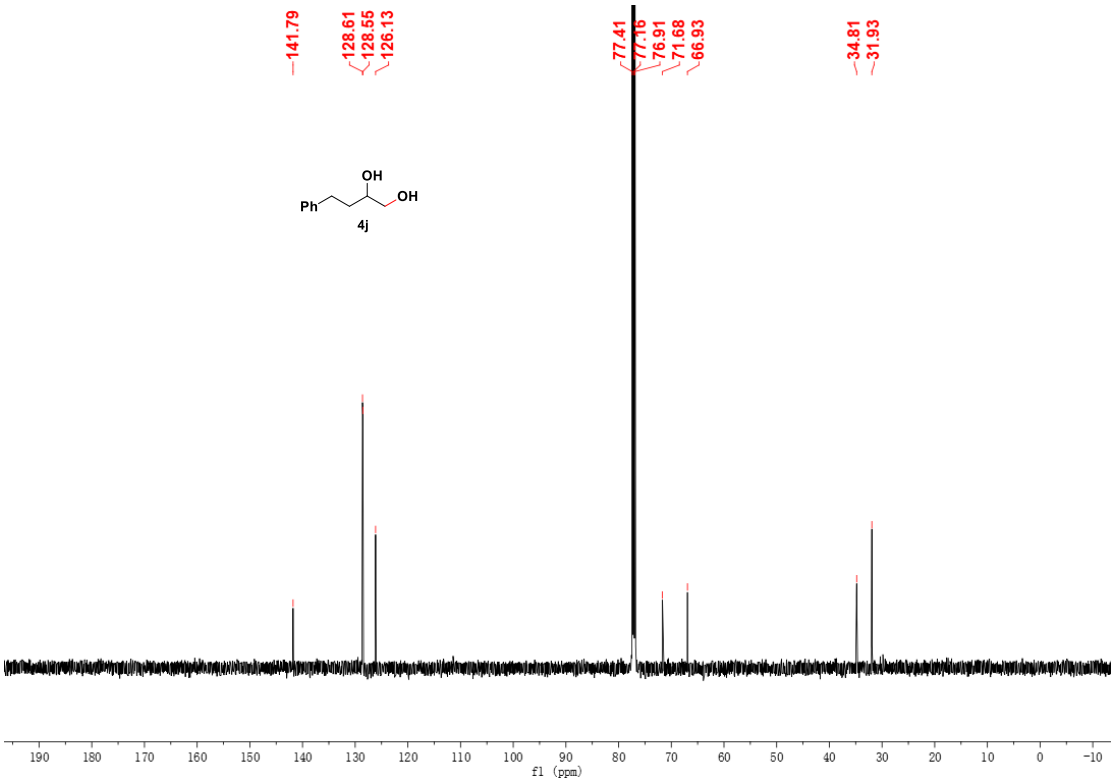
3.73
3.72
3.68
3.68
3.66
3.66
3.50
3.48
3.48
3.46
2.82
2.80
2.72
2.70
1.81
1.80
1.79
1.78
1.77
1.77
1.76
1.76
1.76
1.75
1.73
OH
OH
Ph
4j
2.00
3.14
1.03
1.05
1.08
1.07
1.18
2.14
2.09
f1 (ppm)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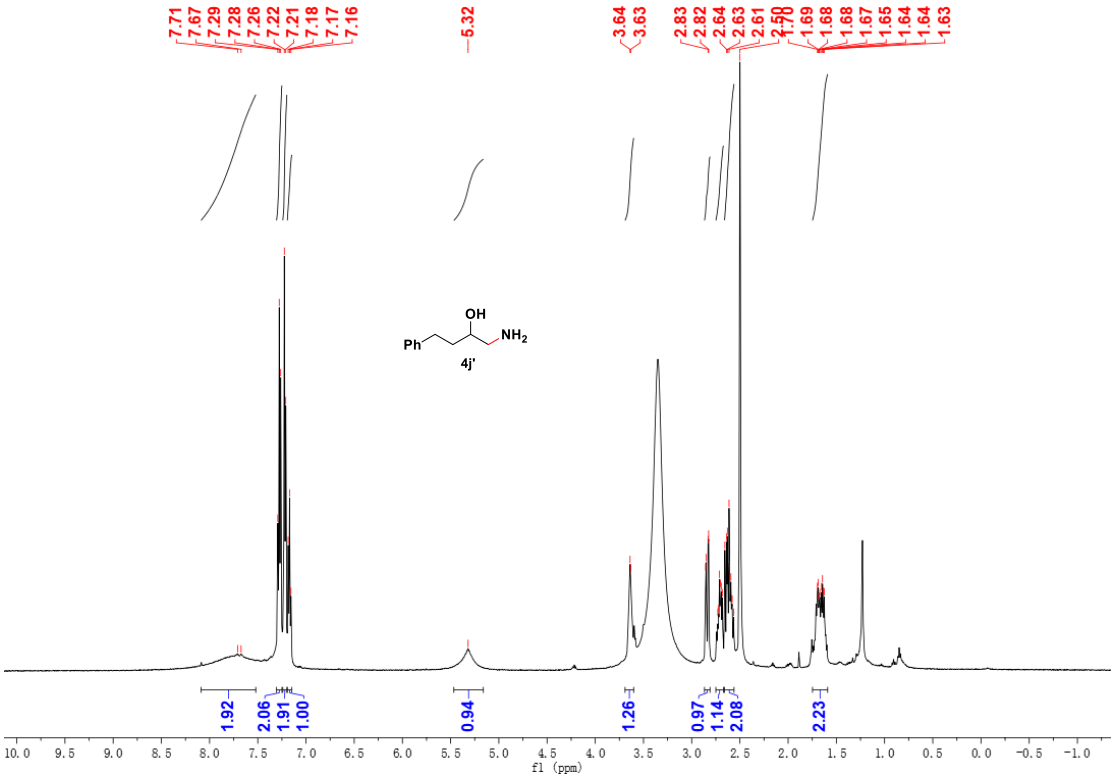
128.61
128.55
126.13
77.41
77.16
76.91
71.68
66.93
34.81
31.93
OH
OH
Ph
4j
f1 (ppm)


7.71
7.67
7.29
7.28
7.26
7.22
7.21
7.18
7.17
7.16
5.32
3.64
3.63
2.83
2.82
2.64
2.63
2.61
2.50
1.70
1.69
1.68
1.68
1.67
1.65
1.64
1.64
1.63
OH
NH2
Ph
4j'
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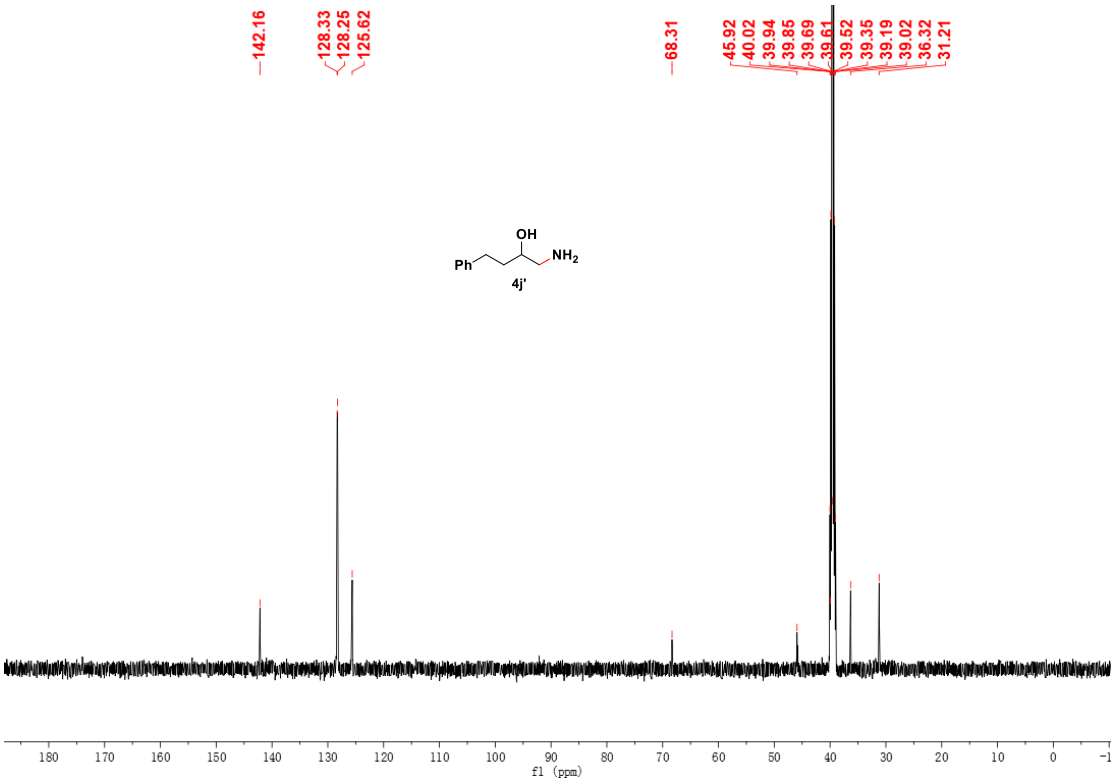
2.06
1.91
1.00
0.94
1.26
0.97
1.14
2.08
2.23
f1 (ppm)


142.16
128.33
128.25
125.62
68.31
45.92
40.02
39.94
39.85
39.69
39.61
39.52
39.35
39.19
39.02
36.32
31.21
OH
NH2
Ph
4j'
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)