Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information

The Synthesis of Sulfonated 4*H*-3,1-Benzoxazines via an Electrochemcial Radical Cascade Reaction

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1. General information

Commercial solvents and reagents were used without further purification unless otherwise noted. Anhydrous MeCN was dried with CaH_2, purify by distillation and stored with 4 Å molecular sieves.

Electrolysis reactions were conducted using a Model QJ3005T (32V) DC power supply purchased from Ningbo Jiuyuan Electronic Co., Ltd., China.

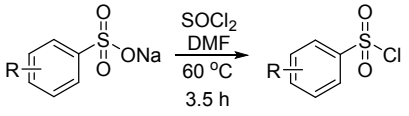
Analytical thin layer chromatography (TLC) plates and the silica gel (200 – 300 mesh) for column chromatography were phased from Qingdao Haiyang Chemical and Special Silica Gel Co, Ltd.

Proton nuclear magnetic resonance (^{1}H NMR), carbon nuclear magnetic resonance (^{13}C NMR) and fluorine nuclear magnetic resonance (^{19}F NMR) spectroscopy were performed on Bruker Advance III-400 spectrometers (400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR, 376 MHz for ^{19}F NMR) and Bruker Ascend^{TM} 500 spectrometers (500 MHz for ^{1}H NMR, 125 MHz for ^{13}C NMR, 470 MHz for ^{19}F NMR). Chemical shifts of ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were reported as in units of parts per million (ppm) downfield from TMS (δ 0.0 ppm) and relative to the signal of $CDCl_3$ (δ 7.26 ppm for ^{1}H NMR and δ 77.1 ppm for ^{13}C NMR). Multiplicities were given as: s (singlet); br s (broad singlet); d (doublet); t (triplet); q (quartet); m (multiplets), etc. The number of protons (n) for a given resonance was indicated by nH.

GC-MS analyses were obtained on a Thermo Scientific Trace GC Ultra Gas Chromatograph equipped with a TG-5MS 30 m × 0.25 mm ID × 0.25 μm capillary column (Thermo Scientific). The GC was directly interfaced to a Thermo Scientific DSQ single quadrupole mass spectrometer (EI, 70 eV). The following GC oven temperature programs were used: 80 °C hold for 2 min, ramp 25 °C/min to a final temperature of 280 °C, and hold for 3 min. Helium was used as a carrier gas, with a constant column flow of 1.0 mL/min. The injector temperature was held constant at 260 °C; HR-MS was carried out on a high-resolution mass spectrometer (LCMS-IT-TOF).

Cyclic voltammetry (CV) analysis was performed on Ingsens IGS-1030 electrochemical workstation (Ingsens Instruments (Guangzhou) Co., Ltd., China) with a conventional three-electrode cell, using a platinum electrode (d = 2 mm) as working electrode, a Pt wire as counter electrode and saturated calomel electrode (SCE) as a reference electrode. Cyclic voltammograms were recorded at 100 mV/s scan rate.

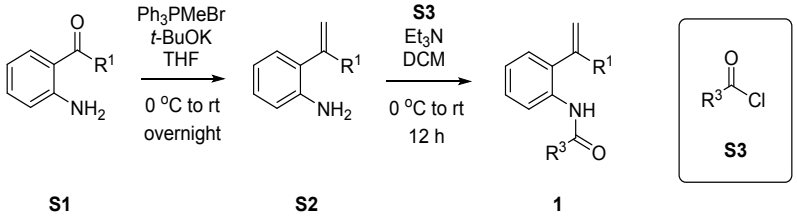
2. General procedures for the synthesis of 4-hydroxy-benzenesulfonylchloride and 4-amino-benzenesulfonylchloride [1]



R = 4-OH, 4-NH_2

Into a round bottom flask, sodium 4-hydroxy-benzenesulfonate/sodium 4-amino-benzenesulfonate (5 mmol), thionyl chloride (1.9 mL) and DMF (0.03 mL) was added. The resulting solution was stirred at 60 °C for 3.5 h. Cool the reaction mixture to room temperature. Then the reaction mixture was quenched with ice water and extracted with CH_2Cl_2 (10 mL × 3). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4. Removal of the solvent by rotary evaporation gave crude 4-hydroxy-benzenesulfonylchloride/4-amino-benzenesulfonylchloride (quant.), which was used in the subsequent reaction without further purification.

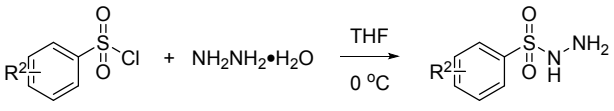
3. General procedures for the synthesis of substrates 1 and 2[2,3]



S1 **S2** **1**

To a stirred solution of Ph_3PMeBr (1.5 equiv., 7.5 mmol) in anhydrous THF (10 mL) was added *t*-BuOK (1.5 equiv., 7.5 mmol) in portions under nitrogen. After the mixture was stirred at room temperature for 0.5 h, a solution of the corresponding ketone (1.0 equiv., 5 mmol) in THF (10 mL) was added dropwisely. The reaction mixture was then stirred at room temperature under nitrogen overnight. The reaction mixture was quenched with water and extracted with EtOAc (25 mL × 2). The combined organic layers were washed with saturated $NaHCO_3$ (25 mL) and brine (25 mL), dried over anhydrous Na_2SO_4, and concentrated on rotary evaporator under vacuum and the residue was purified by column chromatography on silica gel to give 2-alkenylanilines.

Acyl chloride derivatives (1.2 equiv., 1.2 mmol) were added dropwisely to the stirred solution of the obtained alkenes (1.0 equiv., 1 mmol) and triethylamine (1.2 equiv., 1.2 mmol) in CH_2Cl_2 (2.5 mL) at 0 °C. The reaction mixture was stirred at room temperature for 12 h. After completion, the reaction was washed by 10 mol % aqueous HCl solution (8 mL), saturated aqueous $NaHCO_3$ solution (8 mL), brine (12 mL) and dried over anhydrous Na_2SO_4. The organic solvent was removed by rotary evaporator under vacuum and the residue was purified by column chromatography on silica gel to provide products **1**.



S4 **S5** **2**

Sulfonylhydrazides were prepared according to a literature procedure. The hydrazine monohydrate (15 mmol) was added dropwisely into a solution of sulfonyl chloride (5 mmol) in THF (25 mL) under nitrogen at 0 °C. Subsequently, the mixture was stirred at 0 °C for another

30 minutes. After the completion of the reaction, the solvent was removed by evaporation, the residue was extracted with dichloromethane (3 ×10 mL), and the combined organic layer was washed with brine and dried over Na_2SO_4. Purified products **2** were obtained after column chromatography on silica gel.

4. General procedure for the synthesis of products 3 and 4

Into a round bottom tube was added *N*-(2-(prop-1-en-2-yl)phenyl)benzamide **1** (0.2 mmol, 1.0 equiv.), sulfonylhydrazine **2** (0.5 mmol, 2.5 equiv.) and *n*-Bu_4NBF_4 (0.25 mmol). The tube was equipped with a carbon rod anode (= 5 mm, immersion length: 1.5 cm) and a platinum plate cathode (1 × 1.5 cm^2). The tube was flushed with nitrogen. Then anhydrous MeCN (2.5 mL) was added. The resulting solution was electrolyzed under a constant current (j_{anode} = 0.19 mA cm^{-2}) in an undivided cell at room temperature for 3.5 hours. After electrolysis, the mixture was quenched by water and extracted with ethyl acetate (3 × 5 mL). The combined organic layer was washed with brine (8 mL) and dried over Na_2SO_4. Purified product **3**/**4** was obtained after column chromatography on silica gel using a solvent mixture of petroleum ether and ethyl acetate.

5. CV experiments

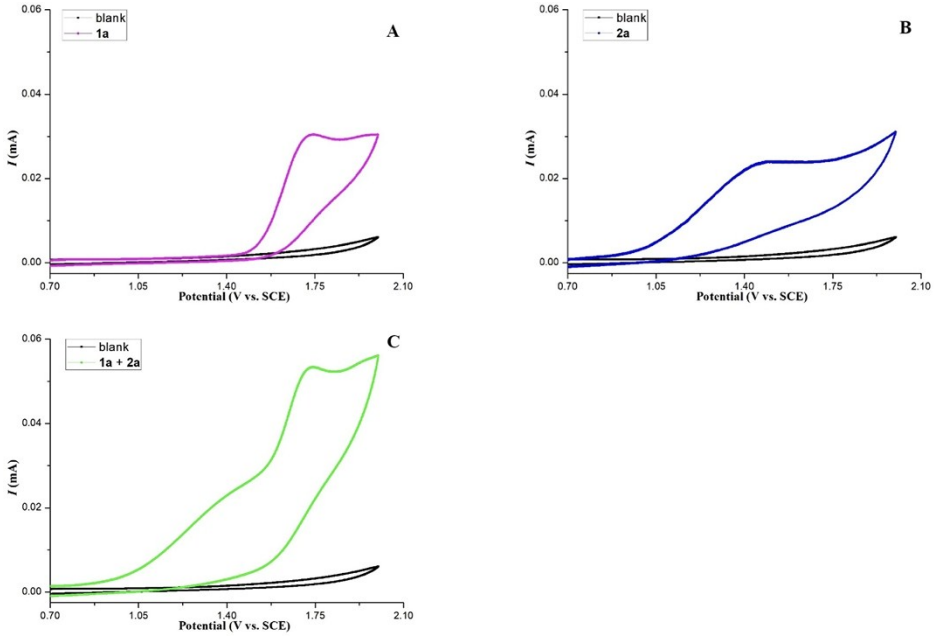
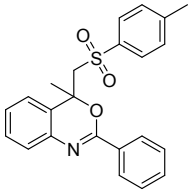


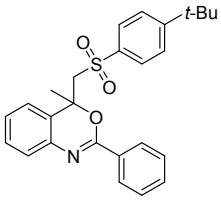
Figure S1 Cyclic voltammograms of 0.1 M *n*-Bu_4NBF_4 solution in MeCN at room temperature. (A) **1a** (1.5×10^{-6} mmol/L); (B) **2a** (4×10^{-6} mmol/L); (C) **1a** (1.5×10^{-6} mmol/L) + **2a** (4×10^{-6} mmol/L). The voltammogram was obtained with Pt wire as an auxiliary electrode and a saturated calomel electrode (SCE) as a reference electrode. The scan rate was 0.1 V/s on a platinum disk electrode (d = 2 mm).

6. Spectroscopic data of products 3 and 4



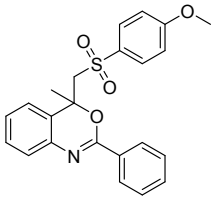
3a

4-methyl-2-phenyl-4-(tosylmethyl)-4*H*-benzo[d][1,3]oxazine (3a)[4] The crude product was purified by column chromatography on silica gel to give **3a** as colorless oil (59.5 mg, 76%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (d, *J* = 7.5 Hz, 2H), 7.58 (d, *J* = 8.1 Hz, 2H), 7.50 – 7.46 (m, 1H), 7.41 – 7.37 (m, 2H), 7.32 – 7.25 (m, 2H), 7.19 – 7.10 (m, 4H), 3.82 (d, *J* = 14.8 Hz, 1H), 3.61 (d, *J* = 14.8 Hz, 1H), 2.28 (s, 3H), 2.06 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 155.6, 144.5, 138.3, 137.7, 132.0, 131.5, 129.8, 129.4, 128.2, 128.1, 127.8, 127.0, 125.6, 123.3, 77.9, 64.1, 27.2, 21.5.



3b

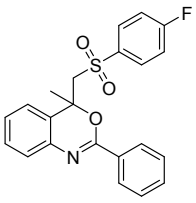
4-(((4-(tert-butyl)phenyl)sulfonyl)methyl)-4-methyl-2-phenyl-4*H*-benzo[d][1,3]oxazine (3b)[4] The crude product was purified by column chromatography on silica gel to give **3b** as colorless oil (52.0 mg, 60%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, *J* = 8.0 Hz, 2H), 7.60 (d, *J* = 8.2 Hz, 2H), 7.50 – 7.48 (m, 1H), 7.43 – 7.39 (m, 2H), 7.34 – 7.32 (m, 2H), 7.29 – 7.23 (m, 2H), 7.15 – 7.10 (m, 2H), 3.81 (d, *J* = 14.7 Hz, 1H), 3.64 (d, *J* = 14.8 Hz, 1H), 2.05 (s, 3H), 1.23 (s, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 157.4, 155.7, 138.4, 137.6, 132.2, 131.6, 129.4, 128.3, 128.2, 127.6, 126.9, 126.1, 125.5, 123.3, 78.0, 64.0, 35.1, 31.0, 27.3.



3c

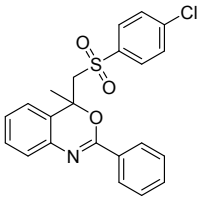
4-(((4-methoxyphenyl)sulfonyl)methyl)-4-methyl-2-phenyl-4*H*-benzo[d][1,3]oxazine (3c)[4] The crude product was purified by column chromatography on silica gel to give **3c** as colorless oil (61.1 mg, 75%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.02 – 8.00 (m, 2H), 7.62 – 7.61 (m, 2H), 7.49 – 7.46 (m, 1H), 7.41 – 7.38 (m, 2H), 7.31 – 7.25 (m, 2H), 7.18 – 7.12 (m, 2H), 6.77 – 6.75 (m, 2H), 3.82 (d, *J* = 14.9 Hz, 1H), 3.61 (d, *J* = 14.9 Hz, 1H), 2.04 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz,

$CDCl_3$): δ 163.5, 155.6, 138.3, 132.2, 132.1, 131.5, 130.0, 129.4, 128.12, 128.11, 126.98, 126.95, 125.6, 123.3, 114.3, 78.0, 64.3, 55.5, 27.3.



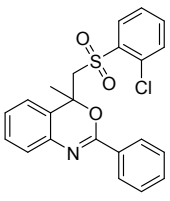
3d

4-(((4-fluorophenyl)sulfonyl)methyl)-4-methyl-2-phenyl-4*H*-benzo[d][1,3]oxazine (3d)[4] The crude product was purified by column chromatography on silica gel to give **3d** as colorless oil (49.8 mg, 63%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, *J* = 7.7 Hz, 2H), 7.70 – 7.66 (m, 2H), 7.51 – 7.47 (m, 1H), 7.42 – 7.38 (m, 2H), 7.31 – 7.24 (m, 2H), 7.17 – 7.09 (m, 2H), 6.98 – 6.94 (m, 2H), 3.84 (d, *J* = 14.9 Hz, 1H), 3.65 (d, *J* = 14.9 Hz, 1H), 2.04 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 165.6 (d, $J_{C\text{-}F}$ = 255.0 Hz), 155.5, 138.3, 136.5 (d, $J_{C\text{-}F}$ = 3.0 Hz), 132.0, 131.6, 130.7, 130.6, 129.6, 128.1 (d, $J_{C\text{-}F}$ = 15.0 Hz), 127.0, 126.6, 125.7, 123.3, 116.4 (d, $J_{C\text{-}F}$ = 23.0 Hz), 77.9, 64.3, 27.5. ^{19}F NMR (377 MHz, $CDCl_3$) δ -103.59.



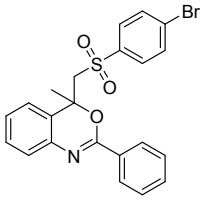
3e

4-(((4-chlorophenyl)sulfonyl)methyl)-4-methyl-2-phenyl-4*H*-benzo[d][1,3]oxazine (3e)[4] The crude product was purified by column chromatography on silica gel to give **3e** as colorless oil (45.3 mg, 55%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 – 7.96 (m, 2H), 7.61 – 7.59 (m, 2H), 7.51 – 7.48 (m, 1H), 7.42 – 7.39 (m, 2H), 7.30 – 7.24 (m, 4H), 7.18 – 7.14 (m, 1H), 7.11 – 7.09 (m, 1H), 3.85 (d, *J* = 15.0 Hz, 1H), 3.66 (d, *J* = 14.9 Hz, 1H), 2.03 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 155.4, 140.3, 138.9, 138.2, 131.9, 131.7, 129.6, 129.4, 129.3, 128.2, 128.0, 127.0, 126.5, 125.7, 123.2, 77.8, 64.3, 27.6.



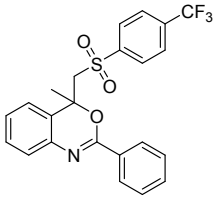
3f

4-(((2-chlorophenyl)sulfonyl)methyl)-4-methyl-2-phenyl-4*H*-benzo[d][1,3]oxazine (3f)[4] The crude product was purified by column chromatography on silica gel to give **3f** as colorless oil (42.0 mg, 51%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 – 7.99 (m, 2H), 7.89 – 7.86 (m, 1H), 7.48 – 7.44 (m, 1H), 7.39 – 7.35 (m, 2H), 7.33 – 7.20 (m, 5H), 7.11 – 7.09 (m, 2H), 4.16 (d, *J* = 15.0 Hz, 1H), 3.93 (d, *J* = 15.0 Hz, 1H), 2.05 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 155.5, 138.3, 137.8, 134.5, 132.4, 132.0, 131,7, 131.4, 131.3, 129.6, 128.10, 128.09, 127.3, 126.9, 126.5, 125.6, 123.1, 77.9, 62.3, 27.7.



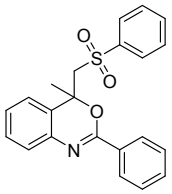
3g

4-(((4-bromophenyl)sulfonyl)methyl)-4-methyl-2-phenyl-4*H*-benzo[d][1,3]oxazine (3g)[4] The crude product was purified by column chromatography on silica gel to give **3g** as colorless oil (34.6 mg, 38%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 – 7.96 (m, 2H), 7.53 – 7.48 (m, 3H), 7.43 – 7.39 (m, 4H), 7.32 – 7.24 (m, 2H), 7.18 – 7.14 (m, 1H), 7.10 – 7.08 (m, 1H), 3.85 (d, *J* = 14.9 Hz, 1H), 3.66 (d, *J* = 14.9 Hz, 1H), 2.02 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 155.4, 139.4, 138.2, 132.4, 131.9, 131.7, 129.6, 129.3, 128.9, 128.2, 128.0, 127.0, 126.5, 125.7, 123.2, 77.8, 64.3, 27.6.



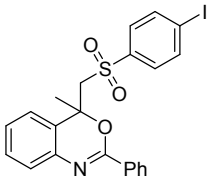
3h

4-methyl-2-phenyl-4-(((4-(trifluoromethyl)phenyl)sulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (3h) The crude product was purified by column chromatography on silica gel to give **3h** as colorless oil (45.3 mg, 51%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 – 7.95 (m, 2H), 7.79 – 7.77 (m, 2H), 7.54 – 7.46 (m, 3H), 7.41 – 7.37 (m, 2H), 7.30 – 7.22 (m, 2H), 7.14 – 7.06 (m, 2H), 3.90 (d, *J* = 15.0 Hz, 1H), 3.74 (d, *J* = 15.0 Hz, 1H), 2.01 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 155.2, 143.7, 138.2, 135.5 – 134.5 (q, $J_{C\text{-}F}$ = 33.0 Hz), 131.8, 131.7, 129.7, 128.4, 128.2, 127.9, 127.0, 126.2 – 126.1 (q, $J_{C\text{-}F}$ = 4.0 Hz), 126.1, 125.8, 123.3, 127.1-118.9 (q, $J_{C\text{-}F}$ = 271.0 Hz), 77.8, 64.2, 28.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ -63.29. HRMS (ESI) calculated for $C_{23}H_{18}F_3NNaO_3S$ $[M+Na]^+$: 468.0857; found: 468.0851.



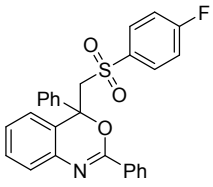
3j

4-methyl-2-phenyl-4-((phenylsulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (3j)[4] The crude product was purified by column chromatography on silica gel to give **3j** as colorless oil (57.4 mg, 76%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 – 8.04 (m, 2H), 7.72 – 7.69 (m, 2H), 7.49 – 7.40 (m, 4H), 7.35 – 7.24 (m, 4H), 7.17 – 7.10 (m, 2H), 3.83 (d, *J* = 14.8 Hz, 1H), 3.63 (d, *J* = 14.8 Hz, 1H), 2.07 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 155.7, 140.6, 138.3, 133.5, 132.0, 131.6, 129.5, 129.2, 128.22, 128.19, 127.7, 127.01, 126.95, 125.6, 123.2, 77.9, 64.0, 27.1.



3m

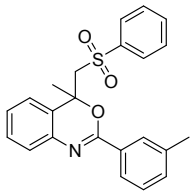
4-(((4-iodophenyl)sulfonyl)methyl)-4-methyl-2-phenyl-4*H*-benzo[d][1,3]oxazine (3m) The crude product was purified by column chromatography on silica gel to give **3m** as colorless oil (16.1 mg, 16%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 – 7.96 (m, 2H), 7.65 – 7.64 (m, 2H), 7.52 – 7.49 (m, 1H), 7.44 – 7.41 (m, 2H), 7.38 – 7.36 (m, 2H), 7.33 – 7.30 (m, 1H), 7.26 – 7.24 (m, 1H), 7.18 – 7.15 (m, 1H), 7.10 – 7.09 (m, 1H), 3.86 (d, *J* = 15.0 Hz, 1H), 3.67 (d, *J* = 15.1 Hz, 1H), 2.02 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 155.4, 140.0, 138.4, 138.2, 131.8, 131.7, 129.6, 129.1, 128.3, 128.0, 127.1, 126.4, 125.7, 123.2, 101.6, 77.8, 64.2, 27.7.



3n

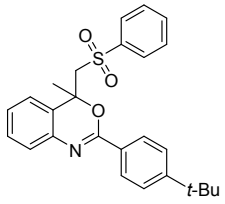
4-(((4-fluorophenyl)sulfonyl)methyl)-2,4-diphenyl-4*H*-benzo[d][1,3]oxazine (3n) The crude product was purified by column chromatography on silica gel to give **3n** as colorless oil (60.3 mg, 66%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95 – 7.94 (m, 2H), 7.66 – 7.63 (m, 2H), 7.51 – 7.48 (m, 1H), 7.41 – 7.34 (m, 3H), 7.30 – 7.23 (m, 8H), 6.91 – 6.87 (m, 2H), 4.33 (d, *J* = 15.4 Hz, 1H), 4.27 (d, *J* = 15.4 Hz, 1H). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 165.6 (d, $J_{C\text{-}F}$ = 255.0 Hz), 154.6, 141.7, 138.7, 136.6 (d, $J_{C\text{-}F}$ = 2.5 Hz), 131.8, 131.6, 131.2 (d, $J_{C\text{-}F}$ = 10.0 Hz), 129.7, 128.8, 128.7, 128.3, 127.8, 126.5, 126.1, 125.33, 125.25, 123.9, 116.1 (d, $J_{C\text{-}F}$ = 22.5 Hz), 80.9, 64.9.

^{19}F NMR (471 MHz, $CDCl_3$) δ -103.92. HRMS (ESI) calculated for $C_{27}H_{20}FNNaO_3S$ $[M+Na]^+$: 480.1046; found: 480.1052.



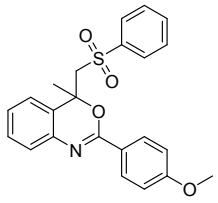
4a

4-methyl-4-((phenylsulfonyl)methyl)-2-(m-tolyl)-4*H*-benzo[d][1,3]oxazine (4a) The crude product was purified by column chromatography on silica gel to give **4a** as colorless oil (58.7 mg, 75%). ^1H NMR (500 MHz, $CDCl_3$): δ 7.87 – 7.83 (m, 2H), 7.71 – 7.69 (m, 2H), 7.45 – 7.42 (m, 1H), 7.34 – 7.24 (m, 6H), 7.16 – 7.11 (m, 2H), 3.82 (d, *J* = 14.8 Hz, 1H), 3.65 (d, *J* = 14.9 Hz, 1H), 2.40 (s, 3H), 2.07 (s, 3H). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): δ 155.8, 140.6, 138.4, 137.8, 133.4, 132.4, 132.0, 129.5, 129.1, 128.6, 128.1, 127.7, 126.93, 126.89, 125.5, 125.4, 123.3, 77.9, 63.9, 27.1, 21.4. HRMS (ESI) calculated for $C_{23}H_{21}NNaO_3S$ $[M+Na]^+$: 414.1140; found: 414.1143.



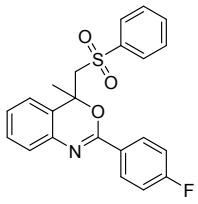
4b

2-(4-(tert-butyl)phenyl)-4-methyl-4-((phenylsulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (4b) The crude product was purified by column chromatography on silica gel to give **4b** as colorless oil (49.4 mg, 57%). ^1H NMR (500 MHz, $CDCl_3$): δ 7.99 – 7.97 (m, 2H), 7.72 – 7.70 (m, 2H), 7.46 – 7.41 (m, 3H), 7.35 – 7.32 (m, 2H), 7.29 – 7.23 (m, 2H), 7.15 – 7.11 (m, 2H), 3.81 (d, *J* = 14.8 Hz, 1H), 3.62 (d, *J* = 14.8 Hz, 1H), 2.08 (s, 3H), 1.35 (s, 9H). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): δ 155.8, 155.1, 140.7, 138.5, 133.4, 129.5, 129.2, 129.1, 128.1, 127.7, 127.0, 126.8, 125.5, 125.2, 123.2, 77.4, 63.9, 35.0, 31.2, 26.9. HRMS (ESI) calculated for $C_{26}H_{27}NNaO_3S$ $[M+Na]^+$: 456.1609; found: 456.1612



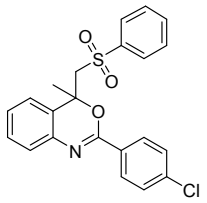
4c

2-(4-methoxyphenyl)-4-methyl-4-((phenylsulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (4c)[4] The crude product was purified by column chromatography on silica gel to give **4c** as colorless oil (59.4 mg, 73%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.04 – 8.02 (m, 2H), 7.72 – 7.71 (m, 2H), 7.48 – 7.45 (m, 1H), 7.37 – 7.33 (m, 2H), 7.28 – 7.21 (m, 2H), 7.12 – 7.11 (m, 2H), 6.92 – 6.90 (m, 2H), 3.85 (s, 3H), 3.80 (d, *J* = 14.8 Hz, 1H), 3.58 (d, *J* = 14.8 Hz, 1H), 2.08 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 162.5, 155.7, 140.7, 138.6, 133.5, 130.2, 129.5, 129.2, 127.7, 127.0, 126.6, 125.3, 124.5, 123.1, 113.6, 77.8, 63.8, 55.4, 26.7.



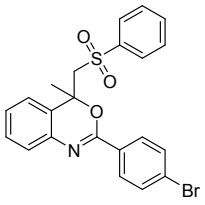
4d

2-(4-fluorophenyl)-4-methyl-4-((phenylsulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (4d) The crude product was purified by column chromatography on silica gel to give **4d** as colorless oil (46.6 mg, 59%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.11 – 8.08 (m, 2H), 7.73 – 7.72 (m, 2H), 7.50 – 7.47 (m, 1H), 7.39 – 7.36 (m, 2H), 7.31 – 7.28 (m, 1H), 7.25 – 7.23 (m, 1H), 7.17 – 7.14 (m, 1H), 7.12 – 7.07 (m, 3H), 3.81 (d, *J* = 14.9 Hz, 1H), 3.57 (d, *J* = 14.9 Hz, 1H), 2.10 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 165.0 (d, $J_{C\text{-}F}$ = 255.0 Hz), 154.9, 140.7, 138.2, 133.6, 130.6 (d, $J_{C\text{-}F}$ = 10.0 Hz), 129.6, 129.2, 128.2 (d, $J_{C\text{-}F}$ = 2.5 Hz), 127.7, 127.1, 126.9, 125.5, 123.1, 115.3 (d, $J_{C\text{-}F}$ = 22.5 Hz), 78.1, 63.9, 26.9. ^{19}F NMR (471 MHz, $CDCl_3$) δ -108.06. HRMS (ESI) calculated for $C_{22}H_{18}FNNaO_3S$ $[M+Na]^+$: 418.0889; found: 418.0883.



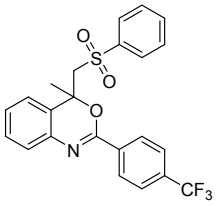
4e

2-(4-chlorophenyl)-4-methyl-4-((phenylsulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (4e) The crude product was purified by column chromatography on silica gel to give **4e** as colorless oil (44.3 mg, 54%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.03 – 8.01 (m, 2H), 7.73 – 7.71 (m, 2H), 7.51 – 7.48 (m, 1H), 7.40 – 7.36 (m, 4H), 7.31 – 7.28 (m, 1H), 7.25 – 7.24 (m, 1H), 7.18 – 7.15 (m, 1H), 7.12 – 7.10 (m, 1H), 3.80 (d, *J* = 14.9 Hz, 1H), 3.56 (d, *J* = 14.9 Hz, 1H), 2.10 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 154.8, 140.6, 138.1, 137.8, 133.6, 130.5, 129.6, 129.2, 128.5, 127.7, 127.2, 127.0, 125.6, 123.1, 78.1, 63.9, 26.9. HRMS (ESI) calculated for $C_{22}H_{18}ClNNaO_3$ $[M+Na]^+$: 434.0594; found: 434.0597.



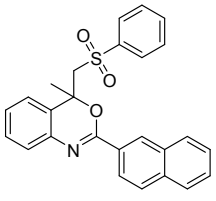
4f

2-(4-bromophenyl)-4-methyl-4-((phenylsulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (4f) The crude product was purified by column chromatography on silica gel to give **4f** as colorless oil (49.2mg, 54%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95 – 7.93 (m, 2H), 7.72 – 7.71 (m, 2H), 7.54 – 7.53 (m, 2H), 7.50 – 7.48 (m, 1H), 7.38 – 7.35 (m, 2H), 7.30 – 7.27 (m, 1H), 7.26 – 7.23 (m, 1H), 7.17 – 7.14 (m, 1H), 7.11 – 7.09 (m, 1H), 3.80 (d, *J* = 14.9 Hz, 1H), 3.56 (d, *J* = 14.9 Hz, 1H), 2.08 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 154.9, 140.6, 138.1, 133.6, 131.4, 131.0, 129.8, 129.6, 129.2, 127.7, 127.3, 127.0, 126.4, 125.7, 123.1, 78.1, 63.9, 27.0. HRMS (ESI) calculated for $C_{22}H_{18}BrNNaO_3$ [M+Na]$^{+}$: 478.0088; found: 478.0090.



4g

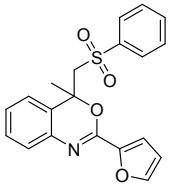
4-methyl-4-((phenylsulfonyl)methyl)-2-(4-(trifluoromethyl)phenyl)-4*H*-benzo[d][1,3]oxazine (4g) The crude product was purified by column chromatography on silica gel to give **4g** as colorless oil (55.2 mg, 62%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.20 – 8.18 (m, 2H), 7.73 – 7.71 (m, 2H), 7.67 – 7.65 (m, 2H), 7.49 – 7.46 (m, 1H), 7.38 – 7.35 (m, 2H), 7.31 – 7.29 (m, 2H), 7.19 – 7.17 (m, 1H), 7.11 – 7.10 (m, 1H), 3.81 (d, *J* = 14.9 Hz, 1H), 3.57 (d, *J* = 14.9 Hz, 1H), 2.11 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 154.3, 140.6, 137.8, 135.4, 133.6, 133.3 – 132.5 (q, $J_{C\text{-}F}$ = 32.5 Hz), 129.7, 129.23, 129.21, 128.5, 127.7, 127.6, 127.0, 125.9, 125.14 – 125.05 (q, $J_{C\text{-}F}$ = 3.8 Hz), 123.1, 127.1-118.9 (q, $J_{C\text{-}F}$ = 271.0 Hz), 78.3, 64.1, 27.2. ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.84. HRMS (ESI) calculated for $C_{23}H_{18}F_3NNaO_3S$ [M+Na]$^{+}$: 468.0857; found: 468.0851.



4h

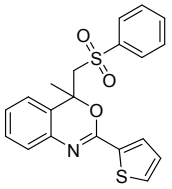
4-methyl-2-(naphthalen-2-yl)-4-((phenylsulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (4h)[4] The crude product was purified by column chromatography on silica gel to give **4h** as colorless oil (44.3 mg, 52%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.50 (m, 1H), 8.19 – 8.17 (m, 1H), 7.95 – 7.94

(m, 1H), 7.87 – 7.85 (m, 2H), 7.73 – 7.71 (m, 2H), 7.57 – 7.52 (m, 2H), 7.37 – 7.29 (m, 5H), 7.21 – 7.16 (m, 2H), 3.88 (d, *J* = 14.9 Hz, 1H), 3.67 (d, *J* = 14.9 Hz, 1H), 2.14 (s, 3H). $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ 155.7, 140.6, 138.4, 135.0, 133.4, 132.7, 129.6, 129.32, 129.26, 129.1, 128.9, 127.8, 127.72, 127.70, 127.6, 127.1, 127.0, 126.4, 125.6, 124.6, 123.2, 78.0, 63.9, 27.1.



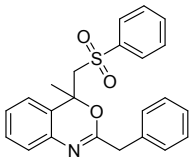
4i

2-(furan-2-yl)-4-methyl-4-((phenylsulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (4i)[4] The crude product was purified by column chromatography on silica gel to give **4i** as colorless oil (49.2 mg, 67%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74 – 7.72 (m, 2H), 7.590 – 7.585 (m, 1H), 7.53 – 7.49 (m, 1H), 7.41 – 7.38 (m, 2H), 7.31 – 7.27 (m, 2H), 7.17 – 7.14 (m, 1H), 7.12 – 7.10 (m, 1H), 7.04 – 7.03 (m, 1H), 6.51 – 6.50 (m, 1H), 3.78 (d, *J* = 14.9 Hz, 1H), 3.57 (d, *J* = 14.8 Hz, 1H), 2.09 (s, 3H). $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ 148.6, 146.0, 145.8, 140.6, 137.8, 133.5, 129.7, 129.2, 127.7, 127.1, 127.0, 125.6, 123.3, 115.9, 111.9, 78.0, 63.6, 26.7.



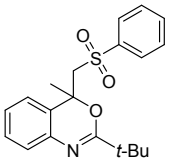
4j

4-methyl-4-((phenylsulfonyl)methyl)-2-(thiophen-2-yl)-4*H*-benzo[d][1,3]oxazine (4j)[4] The crude product was purified by column chromatography on silica gel to give **4j** as colorless oil (54.4 mg, 71%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.73 – 7.71 (m, 2H), 7.64 – 7.63 (m, 1H), 7.49 – 7.46 (m, 2H), 7.39 – 7.35 (m, 2H), 7.29 – 7.25 (m, 1H), 7.22 – 7.20 (m, 1H), 7.15 – 7.10 (m, 2H), 7.07 – 7.05 (m, 1H), 3.80 (d, *J* = 14.8 Hz, 1H), 3.60 (d, *J* = 14.8 Hz, 1H), 2.07 (s, 3H). $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ 152.3, 140.7, 138.3, 136.2, 133.5, 130.9, 130.6, 129.6, 129.2, 127.7, 126.9, 126.8, 125.3, 123.3, 78.3, 63.8, 26.9.



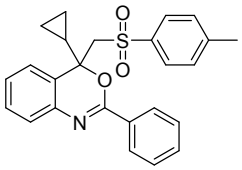
4k

2-benzyl-4-methyl-4-((phenylsulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (4k) The crude product was purified by column chromatography on silica gel to give **4k** as colorless oil (35.2 mg, 45%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.69 – 7.68 (m, 2H), 7.58 – 7.55 (m, 1H), 7.46 – 7.43 (m, 2H), 7.30 – 7.29 (m, 4H), 7.24 – 7.20 (m, 2H), 7.11 – 7.07 (m, 2H), 7.01 – 6.99 (m, 1H), 3.59 (d, *J* = 14.9 Hz, 1H), 3.51 (d, *J* = 14.3 Hz, 1H), 3.44 (d, *J* = 14.9 Hz, 1H), 3.38 (d, *J* = 14.3 Hz, 1H), 1.81 (s, 3H). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 159.8, 140.7, 137.7, 135.3, 133.5, 129.4, 129.3, 129.1, 128.4, 127.9, 126.92, 126.89, 126.3, 125.1, 123.3, 77.5, 64.0, 41.6, 27.2. HRMS (ESI) calculated for $C_{23}H_{21}NNaO_3S$ [M+Na]$^{+}$: 414.1140; found: 414.1143.



4l

(*tert*-butyl)-4-methyl-4-((phenylsulfonyl)methyl)-4*H*-benzo[d][1,3]oxazine (4l) The crude product was purified by column chromatography on silica gel to give **4l** as colorless oil (53.5 mg, 75%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.70 – 7.68 (m, 2H), 7.55 – 7.52 (m, 1H), 7.43 – 7.40 (m, 2H), 7.21 – 7.17 (m, 1H), 7.10 – 7.09 (m, 1H), 7.03 – 6.96 (m, 2H), 3.73 (d, *J* = 14.8 Hz, 1H), 3.67 (d, *J* = 14.8 Hz, 1H), 1.88 (s, 3H), 1.22 (s, 9H). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 166.7, 140.8, 138.2, 133.5, 129.21, 129.18, 127.7, 126.4, 126.2, 125.2, 123.2, 77.1, 64.1, 37.2, 27.4. HRMS (ESI) calculated for $C_{20}H_{23}NNaO_3S$ [M+Na]$^{+}$: 380.1296; found: 380.1294.



8

4-cyclopropyl-2-phenyl-4-(tosylmethyl)-4*H*-benzo[d][1,3]oxa-zine (8) The crude product was purified by column chromatography on silica gel to give **8** as colorless oil (21.6 mg, 26%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87 – 7.86 (m, 2H), 7.50 – 7.48 (m, 2H), 7.46 – 7.43 (m, 1H), 7.37 – 7.34 (m, 2H), 7.28 – 7.27 (m, 1H), 7.24 – 7.22 (m, 1H), 7.14 – 7.11 (m, 1H), 7.08 – 7.07 (m, 1H), 7.004 – 6.989 (m, 1H), 4.04 (d, *J* = 15.1 Hz, 1H), 3.95 (d, *J* = 15.1 Hz, 1H), 2.17 (s, 3H), 1.50 – 1.47 (m, 1H), 0.48 – 0.43 (m, 3H), 0.29 – 0.27 (m, 1H). ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 155.0,

144.3, 138.8, 137.6, 132.0, 131.4, 129.5, 129.3, 128.04, 127.93, 127.8, 126.4, 125.5, 124.6, 123.2, 79.5, 64.8, 22.0, 21.4, 1.3, 1.0. HRMS (ESI) calculated for $C_{20}H_{23}NNaO_3S$ $[M+Na]^+$: 417.1399 found: 417.1396.

Reference

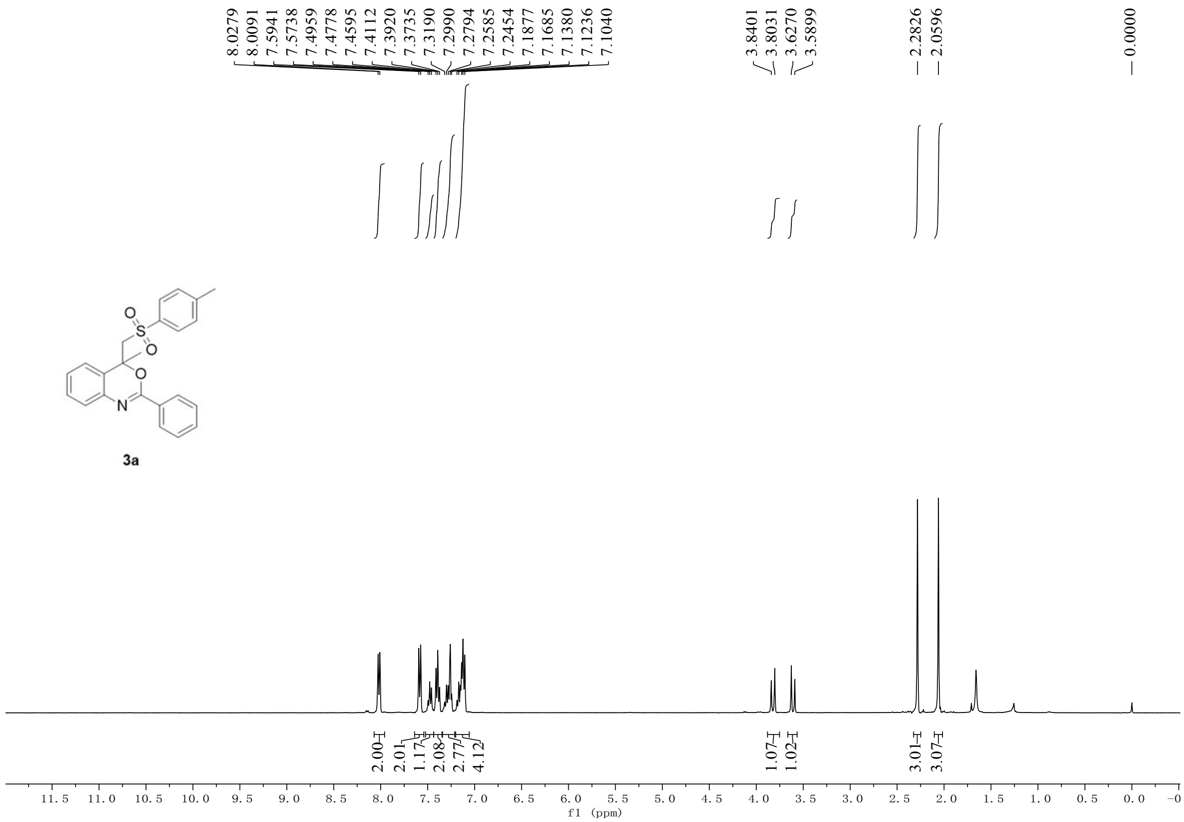
[1] Li, Y.-l.; Qi, X.-y.; Jiang, H.; Deng, X.-d.; Dong, Y.-p.; Ding, T.-b.; Zhou, Lu.; Men, P.; Chu, Y.; Wang, R.-x.; Jiang, X.-c.; Ye, D.-y. *Bioorg. Med. Chem*. **2015**, *23*, 6173–6184.

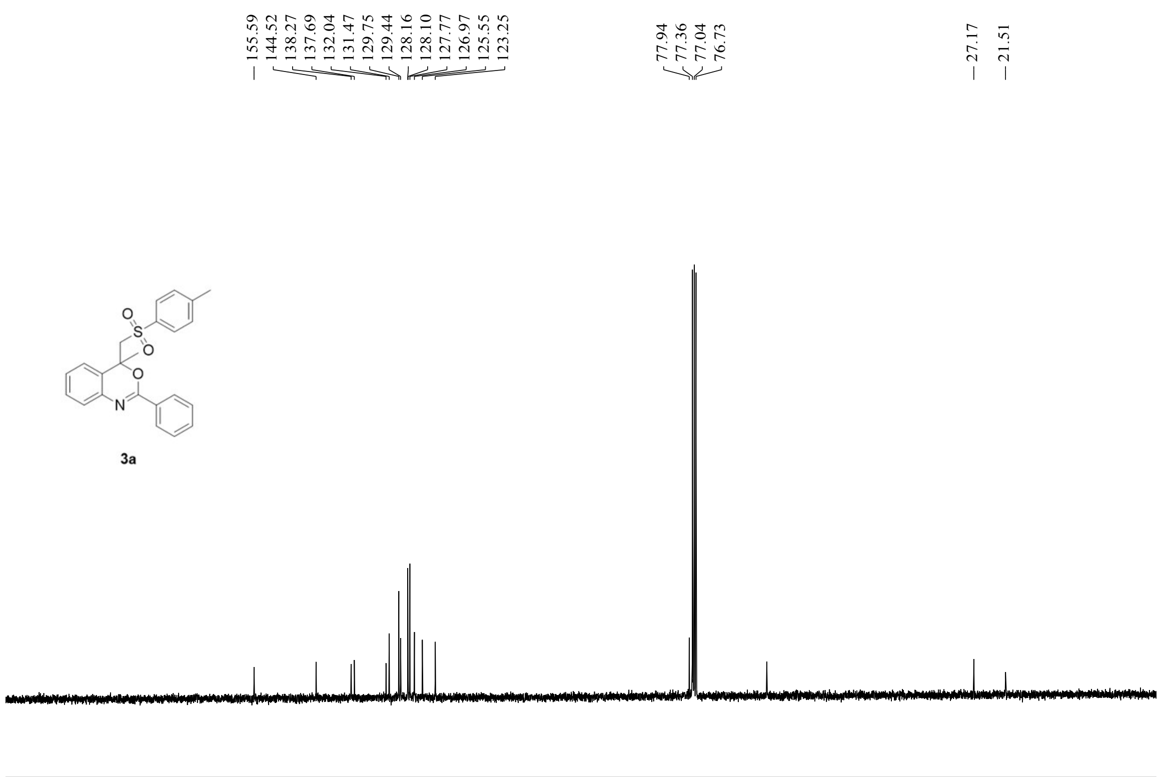
[2] Jana, S.; Ashokan, A.; Kumar, S.; Verma, A.; Kumar, S. *Org. Biomol. Chem.* **2015**,*13*, 8411−8415.

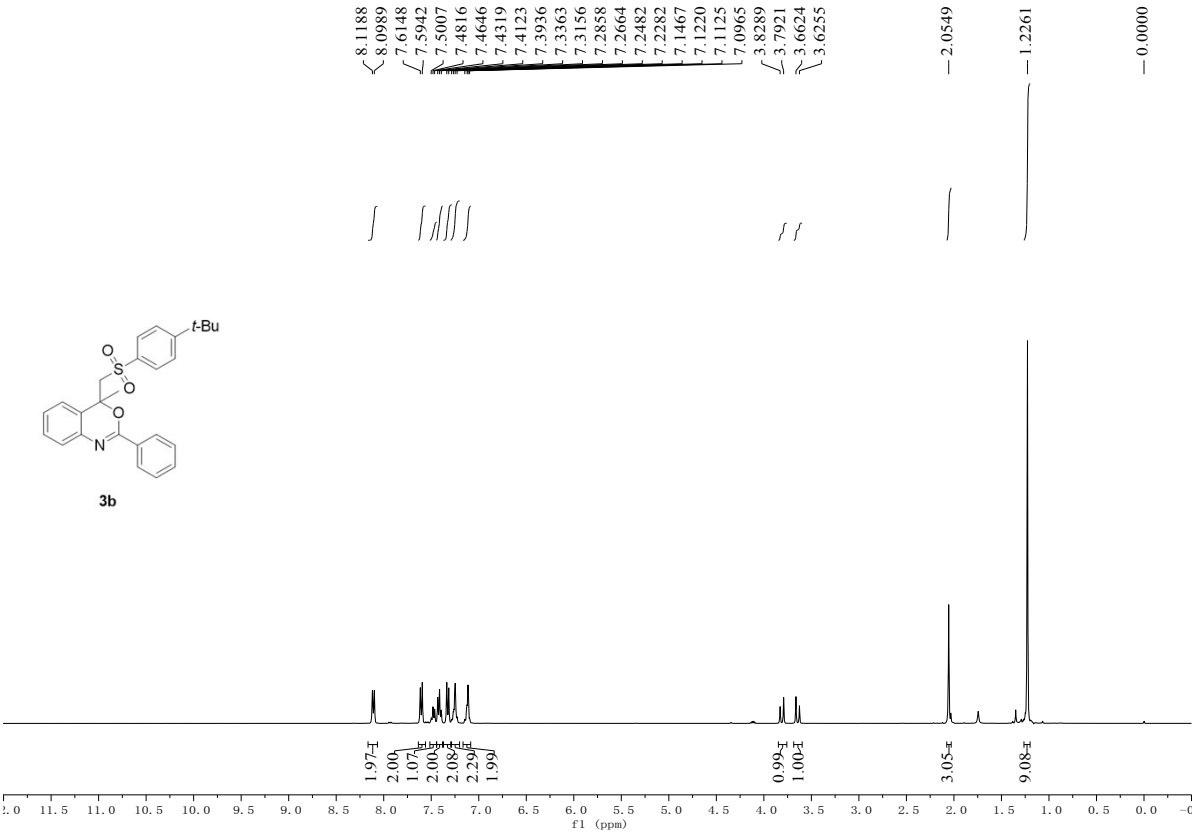
[3] Yuan, Y.; Cao, Y.; Lin, Y.; Li, Y.; Huang, Z.; Lei, A. *ACS Catal.* **2018**, *8*, 10871−10875.

[4] Liu, T.; Zheng, Q.; Li, Z.; Wu, J. *Adv. Synth. Catal.* **2018**, *360*, 865−869.

7. NMR spectra of products 3 and 4



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7.0965
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t-Bu
3b
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2.29
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1.00
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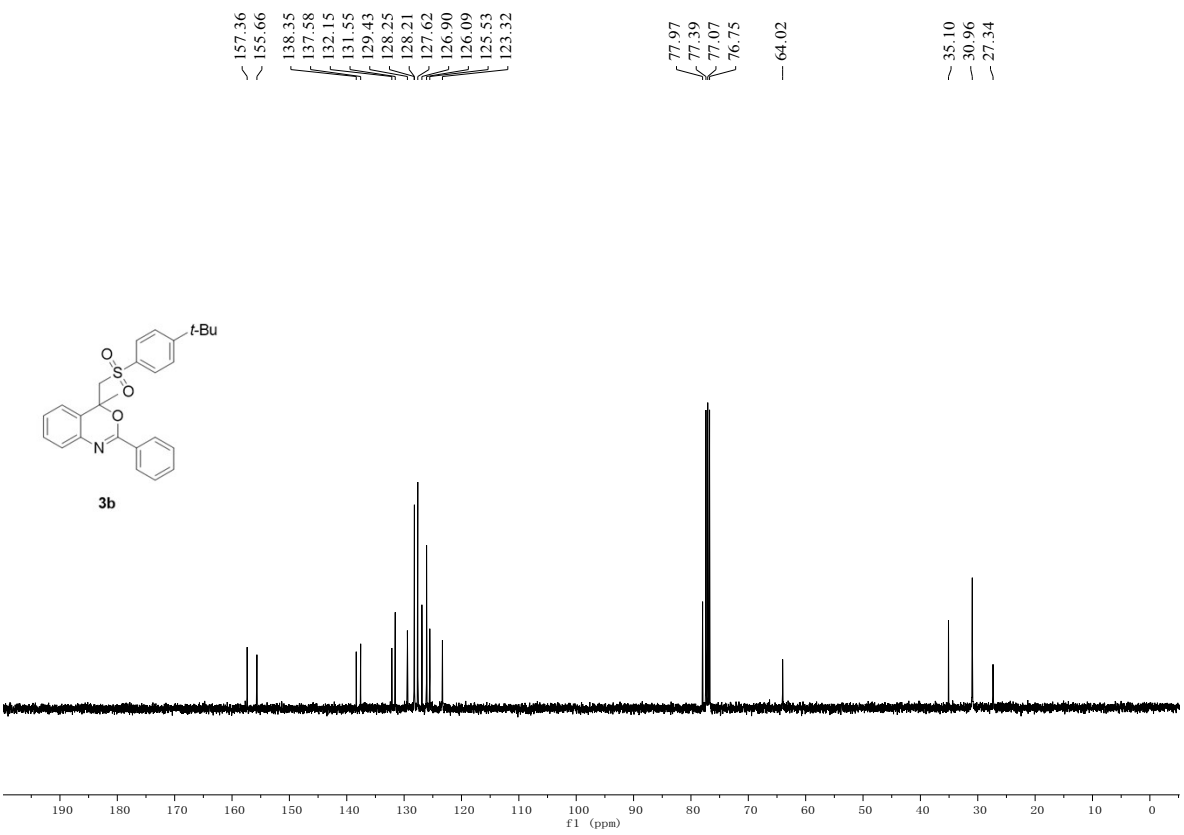
9.08
f1 (ppm)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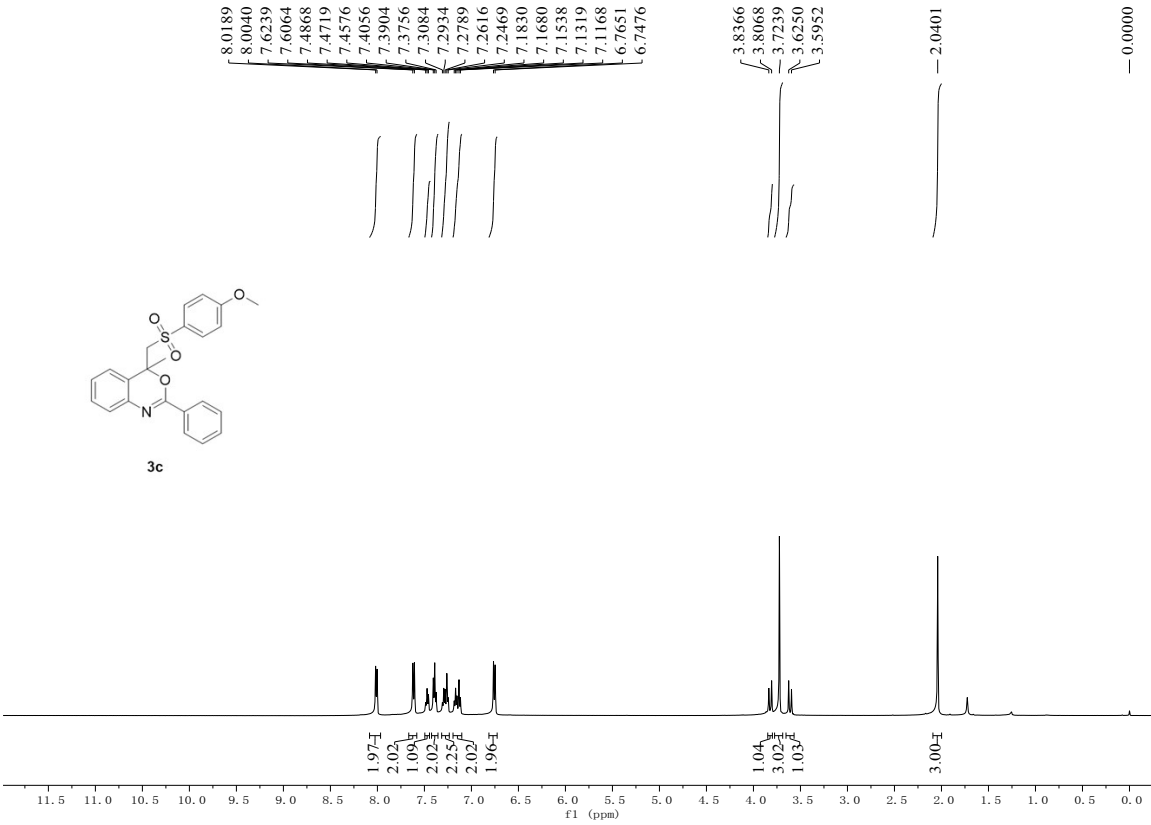
t-Bu
3b
f1 (ppm)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1.09
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2.25
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1.96
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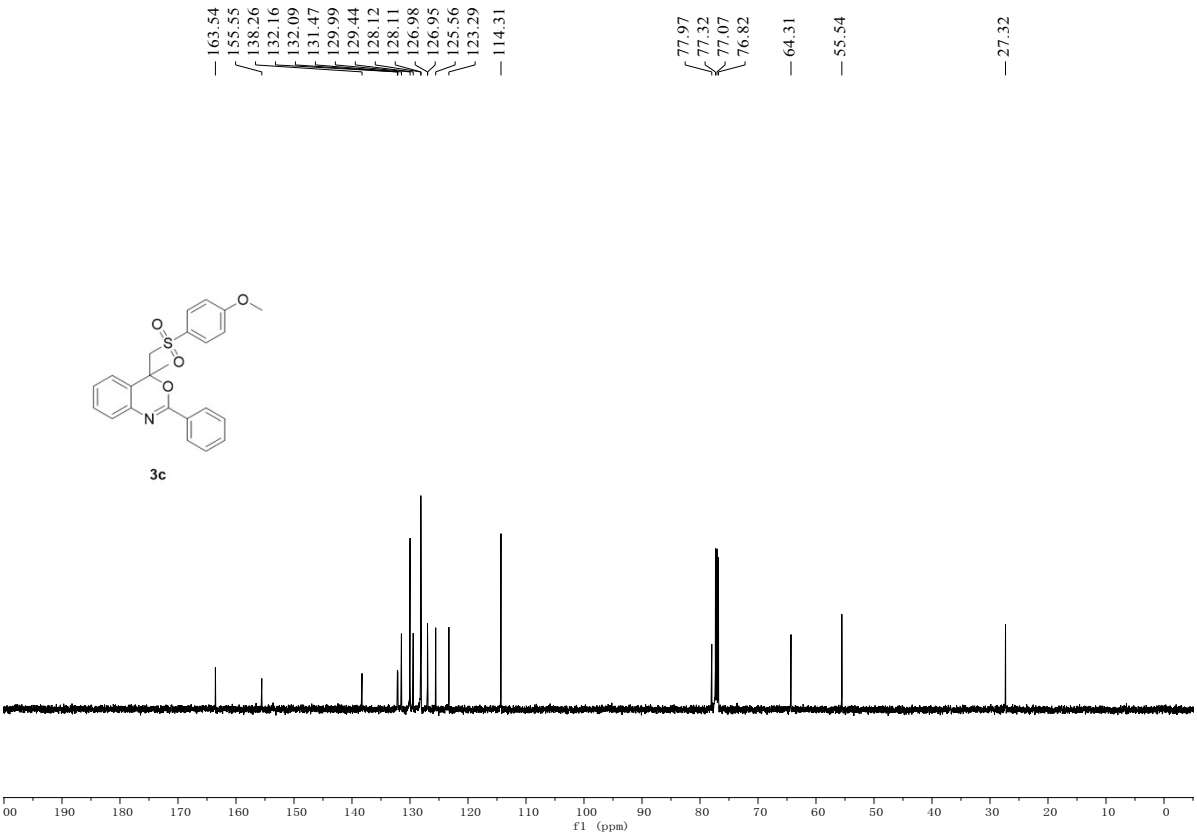
3.02
1.03
3.00
f1 (ppm)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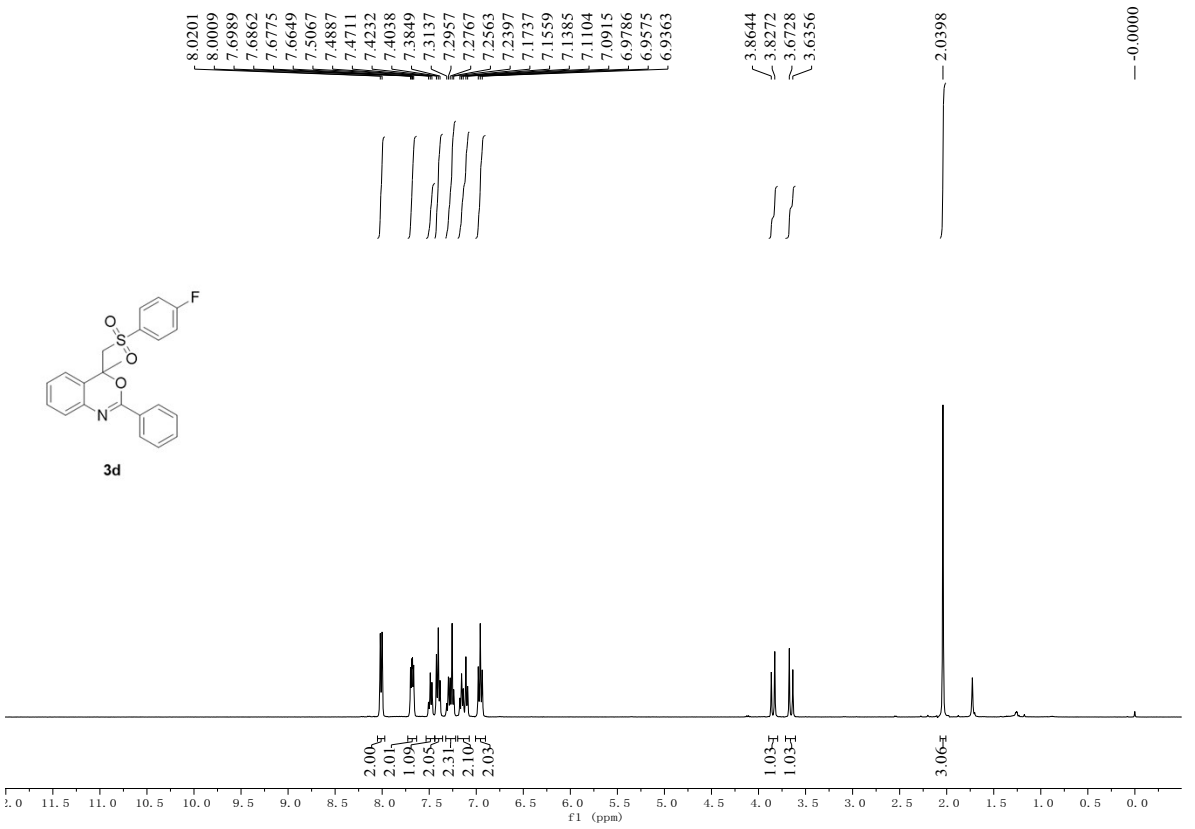
155.55
138.26
132.16
132.09
131.47
129.99
129.44
128.12
128.11
126.98
126.95
125.56
123.29
114.31
77.97
77.32
77.07
76.82
64.31
55.54
27.32
3c
f1 (ppm)


8.0201
8.0009
7.6989
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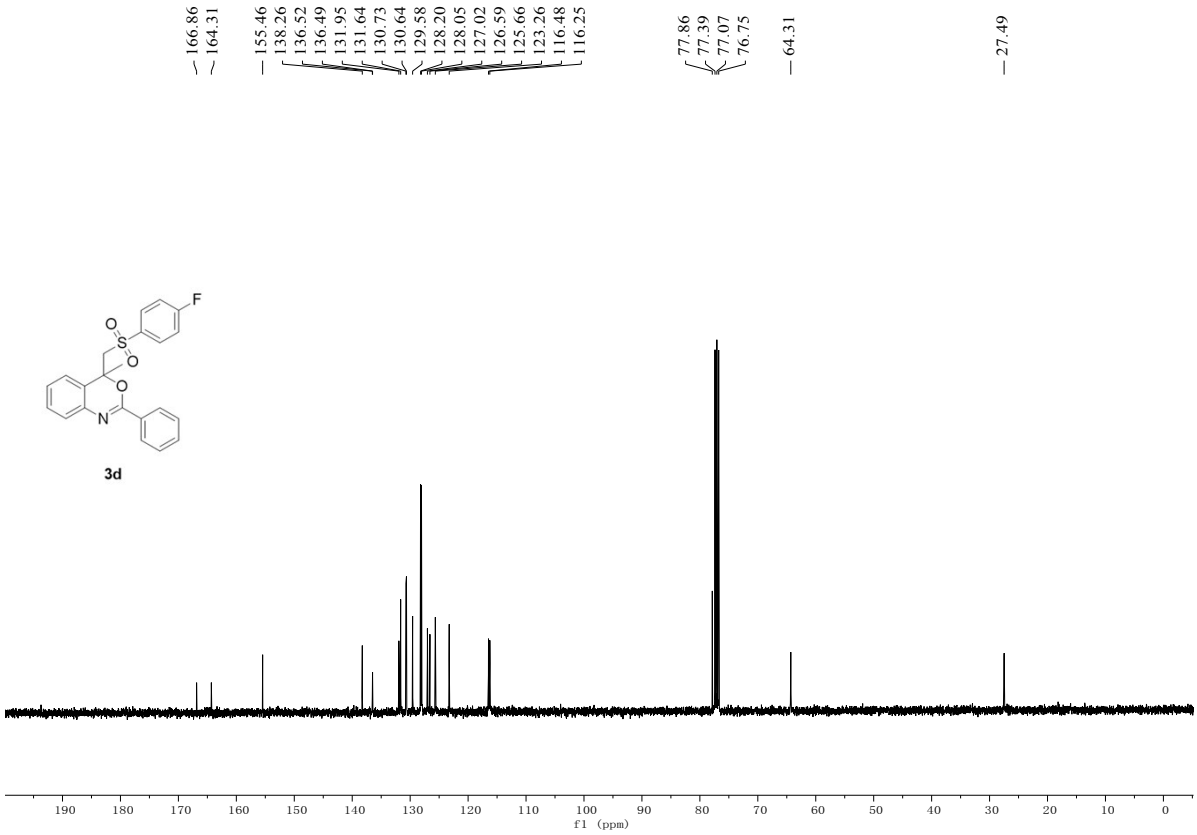
7.6775
7.6649
7.5067
7.4887
7.4711
7.4232
7.4038
7.3849
7.3137
7.2957
7.2767
7.2563
7.2397
7.1737
7.1559
7.1385
7.1104
7.0915
6.9786
6.9575
6.9363
3.8644
3.8272
3.6728
3.6356
2.0398
-0.0000
F
O
S
O
O
N
3d
2.00
2.01
1.09
2.05
2.31
2.10
2.03
1.03
1.03
3.06
f1 (ppm)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164.31
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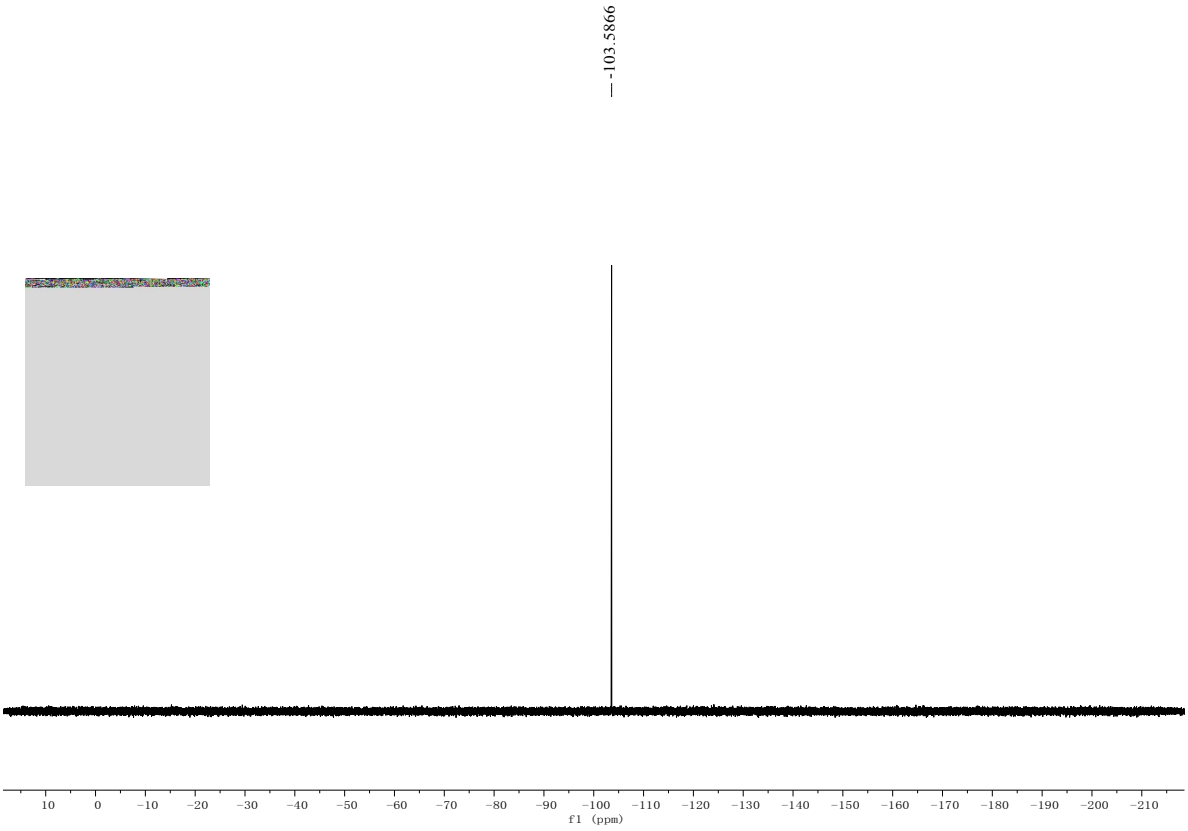
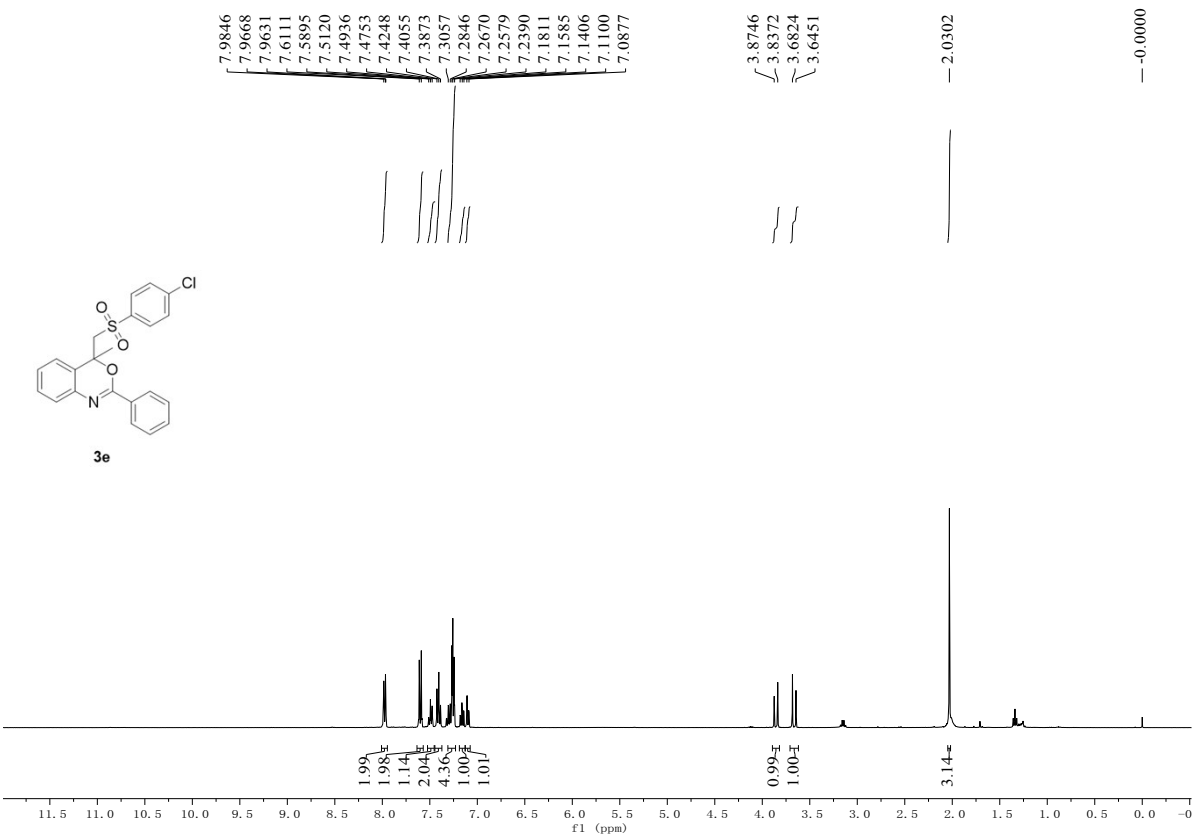
138.26
136.52
136.49
131.95
131.64
130.73
130.64
129.58
128.20
128.05
127.02
126.59
125.66
123.26
116.48
116.25
77.86
77.39
77.07
76.75
64.31
27.49
F
O
S
O
O
N
3d
f1 (ppm)


-103.5866
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)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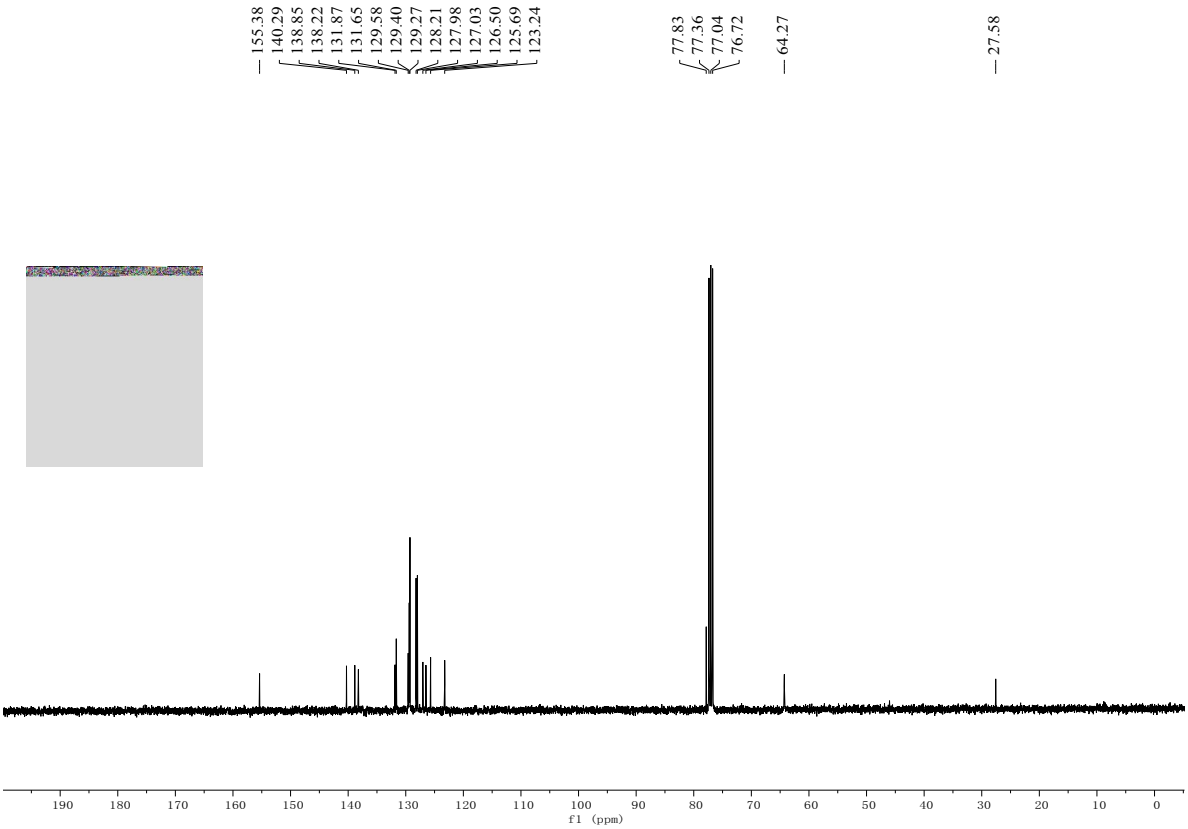
7.5895
7.5120
7.4936
7.4753
7.4248
7.4055
7.3873
7.3057
7.2846
7.2670
7.2579
7.2390
7.1811
7.1585
7.1406
7.1100
7.0877
3.8746
3.8372
3.6824
3.6451
2.0302
-0.0000
Cl
O
S
O
O
N
3e
1.99
1.98
1.14
2.04
4.36
1.00
1.01
0.99
1.00
3.14
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0
f1 (ppm)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140.29
138.85
138.22
131.87
131.65
129.58
129.40
129.27
128.21
127.98
127.03
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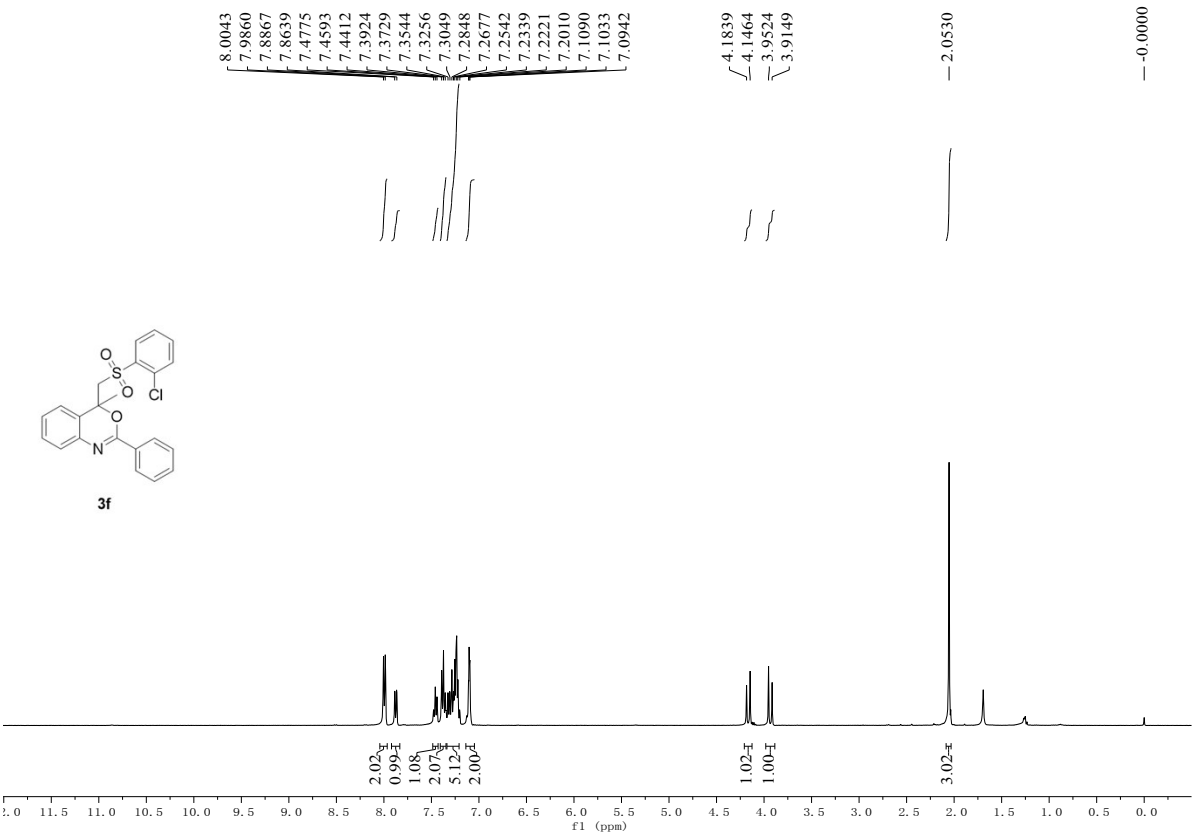
77.04
76.72
64.27
27.58
f1 (ppm)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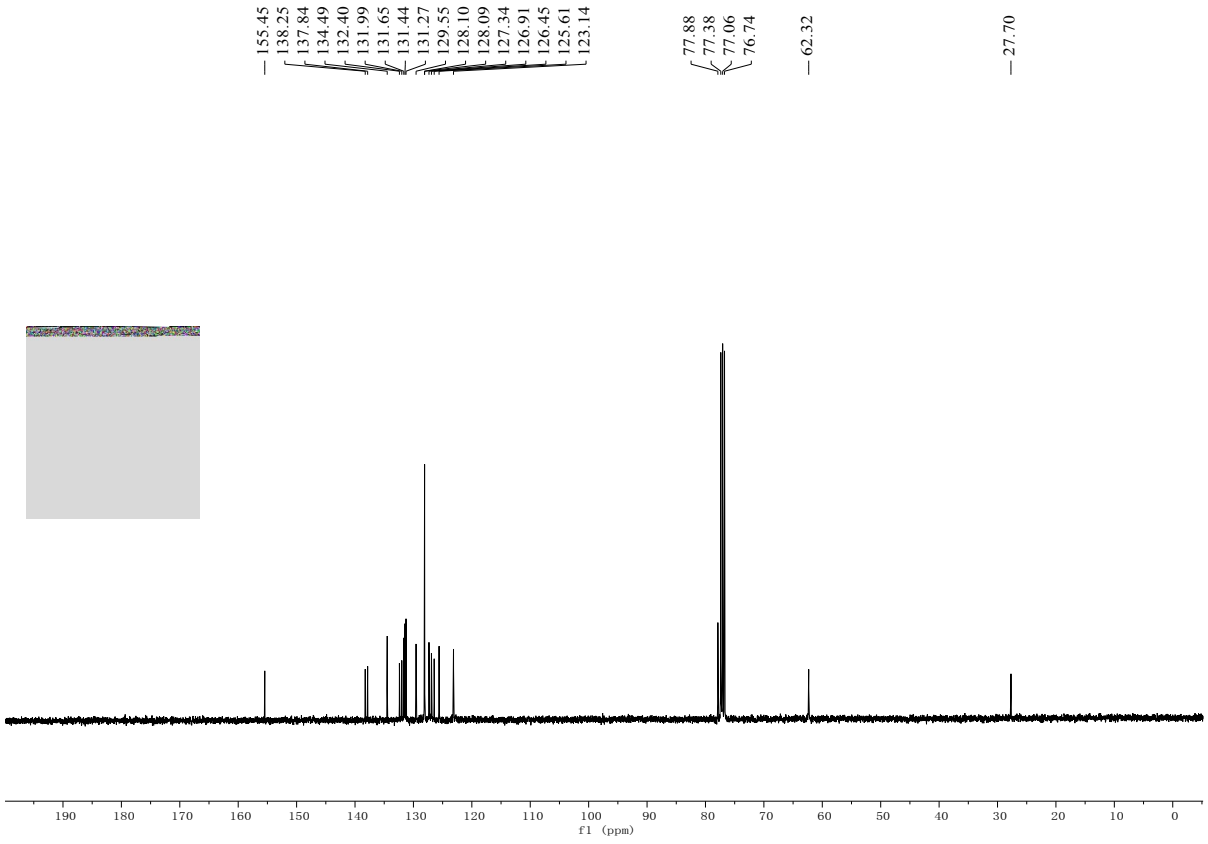
7.4775
7.4593
7.4412
7.3924
7.3729
7.3544
7.3256
7.3049
7.2848
7.2677
7.2542
7.2339
7.2221
7.2010
7.1090
7.1033
7.0942
4.1839
4.1464
3.9524
3.9149
2.0530
-0.0000
2.02
0.99
1.08
2.07
5.12
2.00
1.02
1.00
3.02
f1 (ppm)

O
S
O
Cl
O
N

3f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134.49
132.40
131.99
131.65
131.44
131.27
129.55
128.10
128.09
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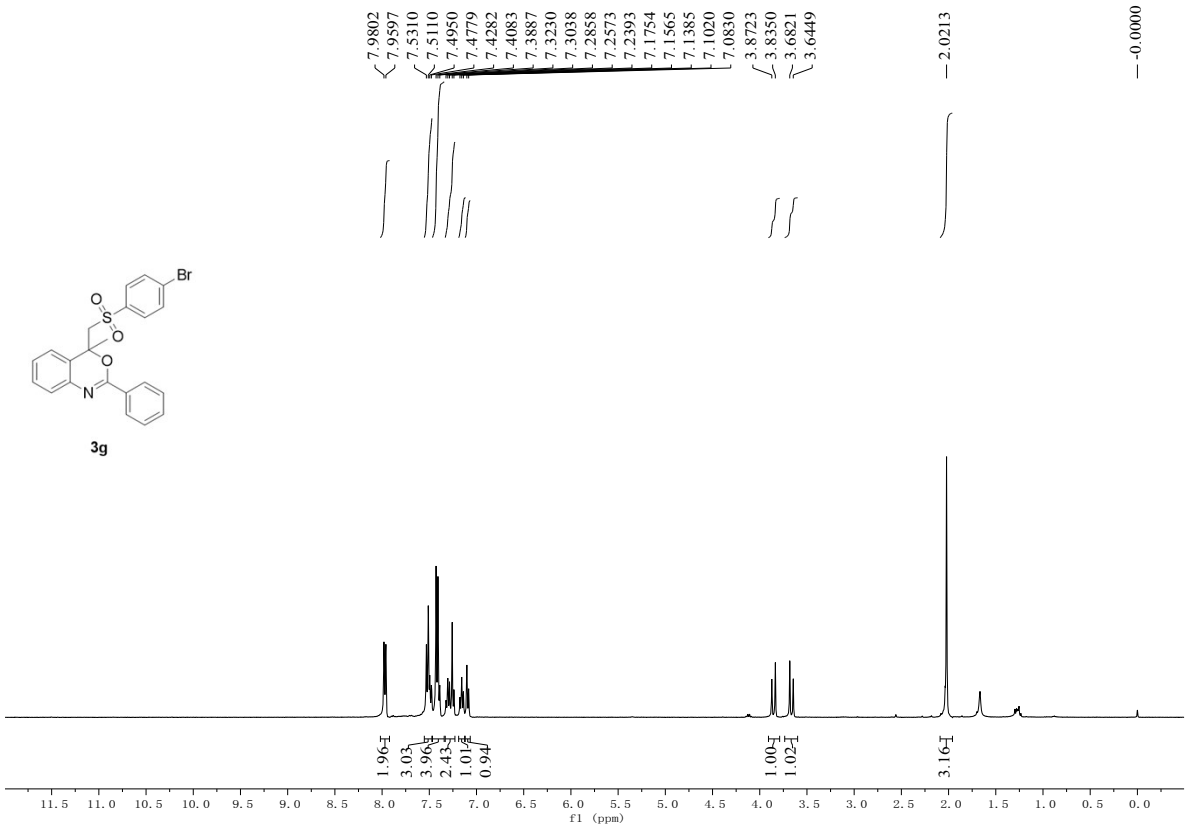
77.06
76.74
62.32
27.70
f1 (ppm)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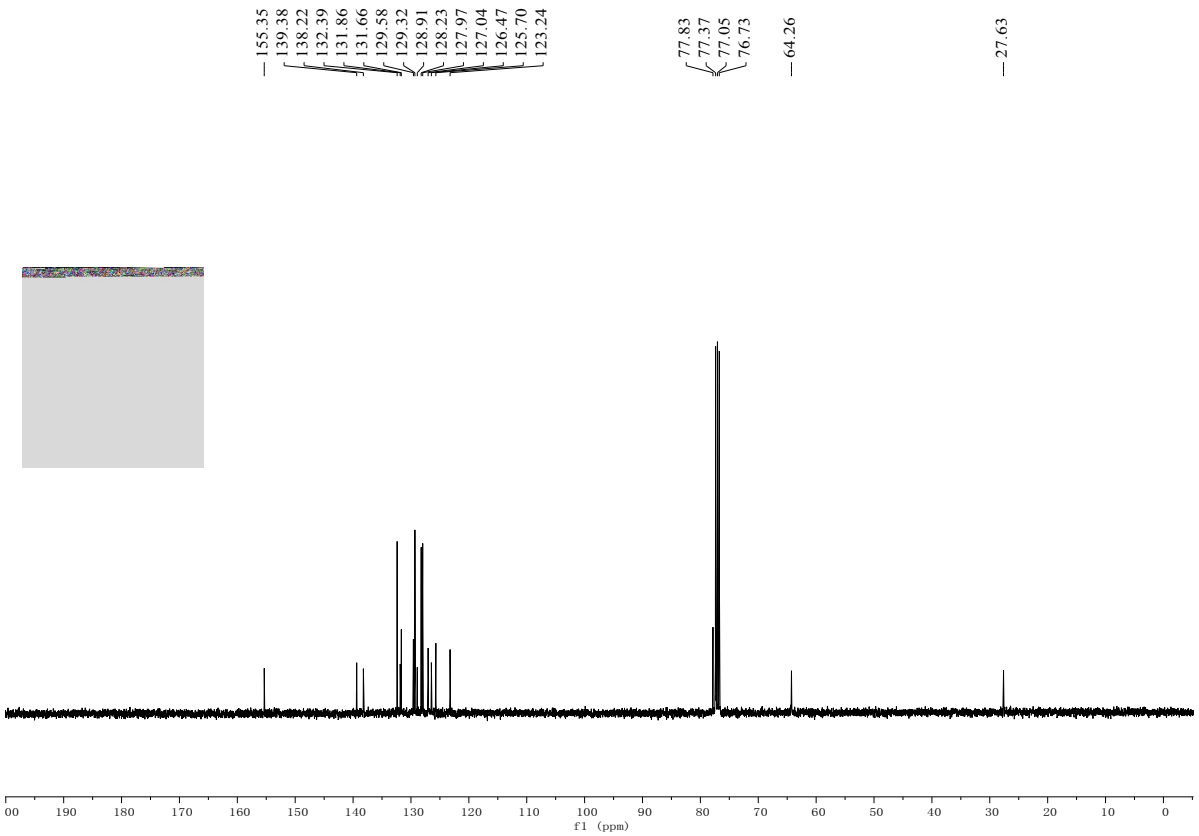
7.4950
7.4779
7.4282
7.4083
7.3887
7.3230
7.3038
7.2858
7.2573
7.2393
7.1754
7.1565
7.1385
7.1020
7.0830
3.8723
3.8350
3.6821
3.6449
2.0213
-0.0000
1.96
3.03
3.96
2.43
1.01
0.94
1.00
1.02
3.16
f1 (ppm)

Br
O
S
O
O
N

3g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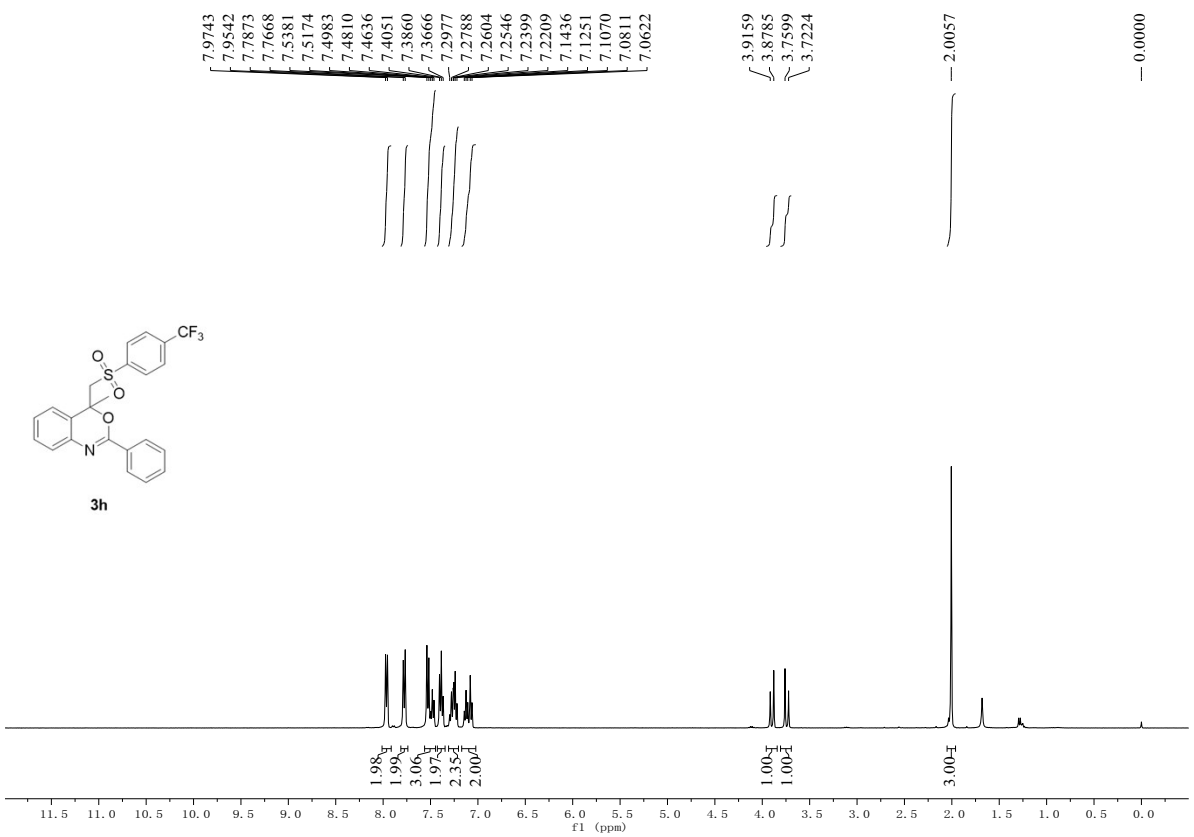
139.38
138.22
132.39
131.86
131.66
129.58
129.32
128.91
128.23
127.97
127.04
126.47
125.70
123.24
77.83
77.37
77.05
76.73
64.26
27.63
f1 (ppm)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7.5174
7.4983
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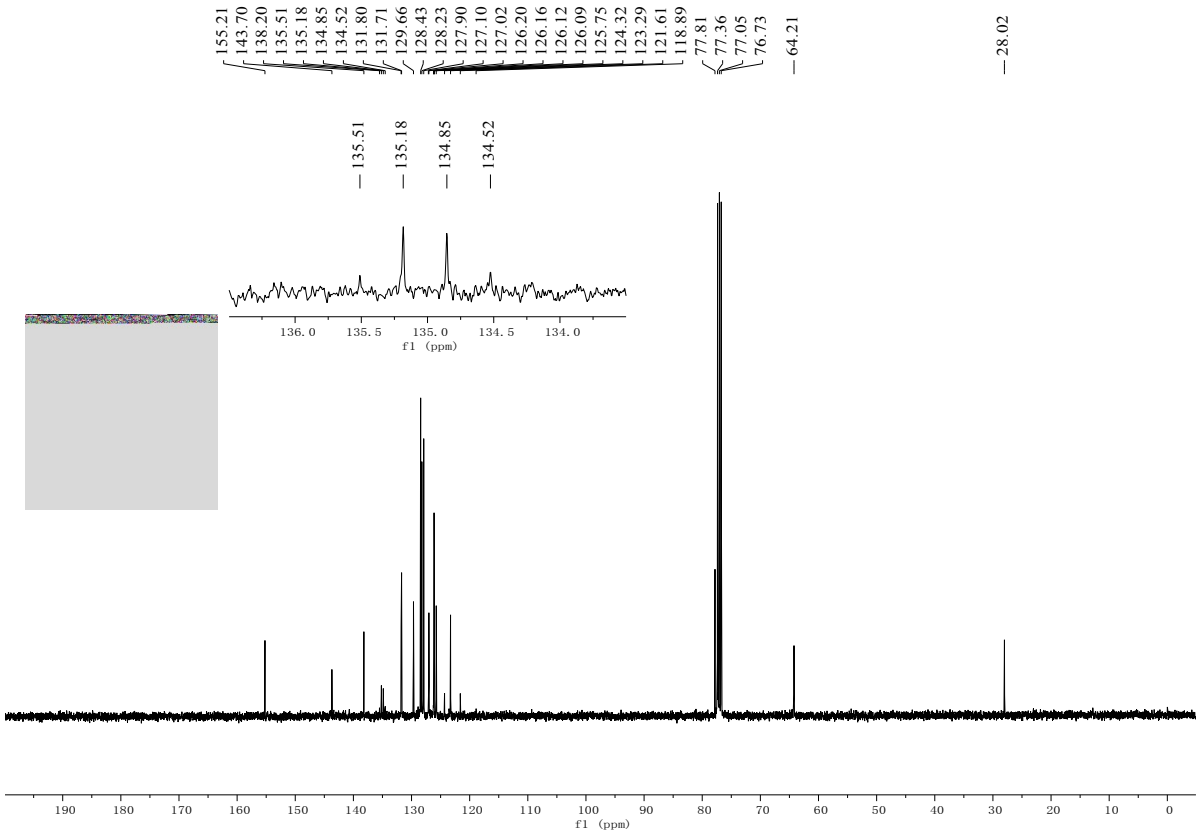
7.4636
7.4051
7.3860
7.3666
7.2977
7.2788
7.2604
7.2546
7.2399
7.2209
7.1436
7.1251
7.1070
7.0811
7.0622
3.9159
3.8785
3.7599
3.7224
2.0057
0.0000
1.98
1.99
3.06
1.97
2.35
2.00
1.00
1.00
3.00
f1 (ppm)

CF3
O
S
O
O
N

3h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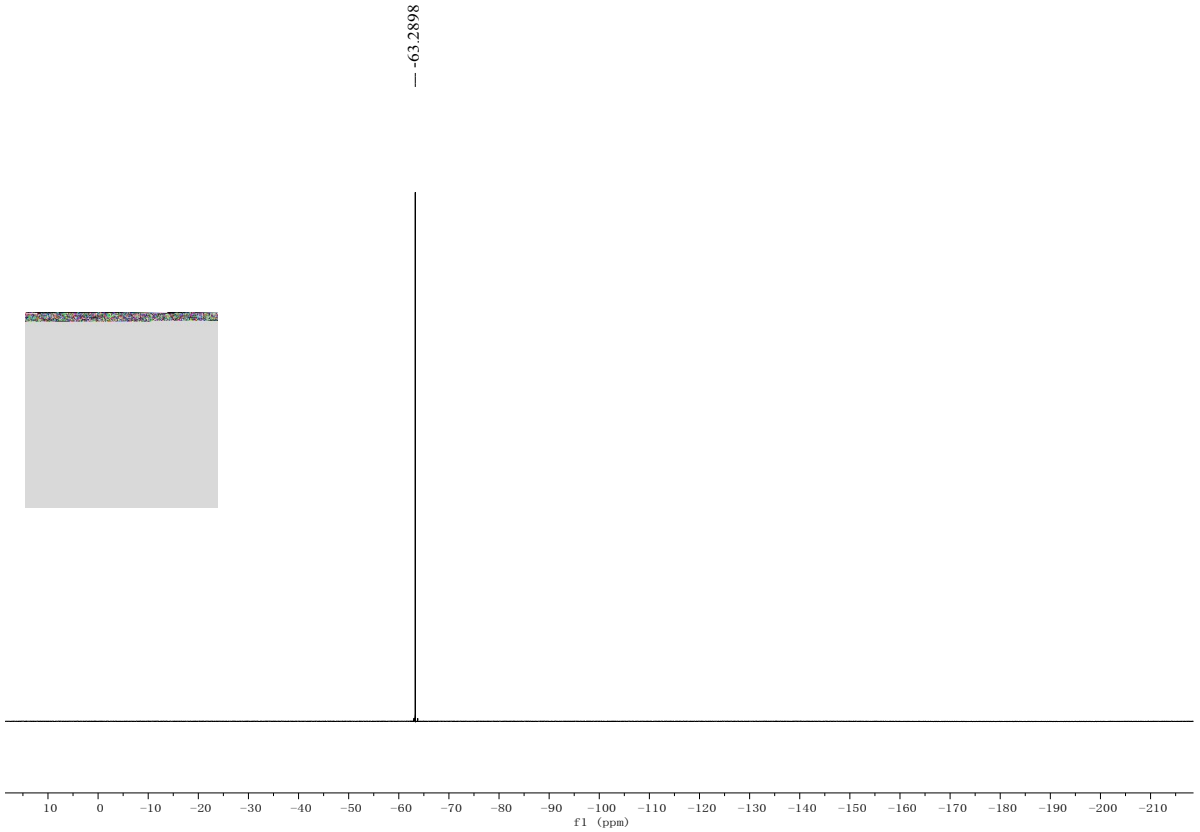
138.20
135.51
135.18
134.85
134.52
131.80
131.71
129.66
128.43
128.23
127.90
127.10
127.02
126.20
126.16
126.12
126.09
125.75
124.32
123.29
121.61
118.89
77.81
77.36
77.05
76.73
64.21
28.02
135.51
135.18
134.85
134.52
136.0
135.5
135.0
134.5
134.0
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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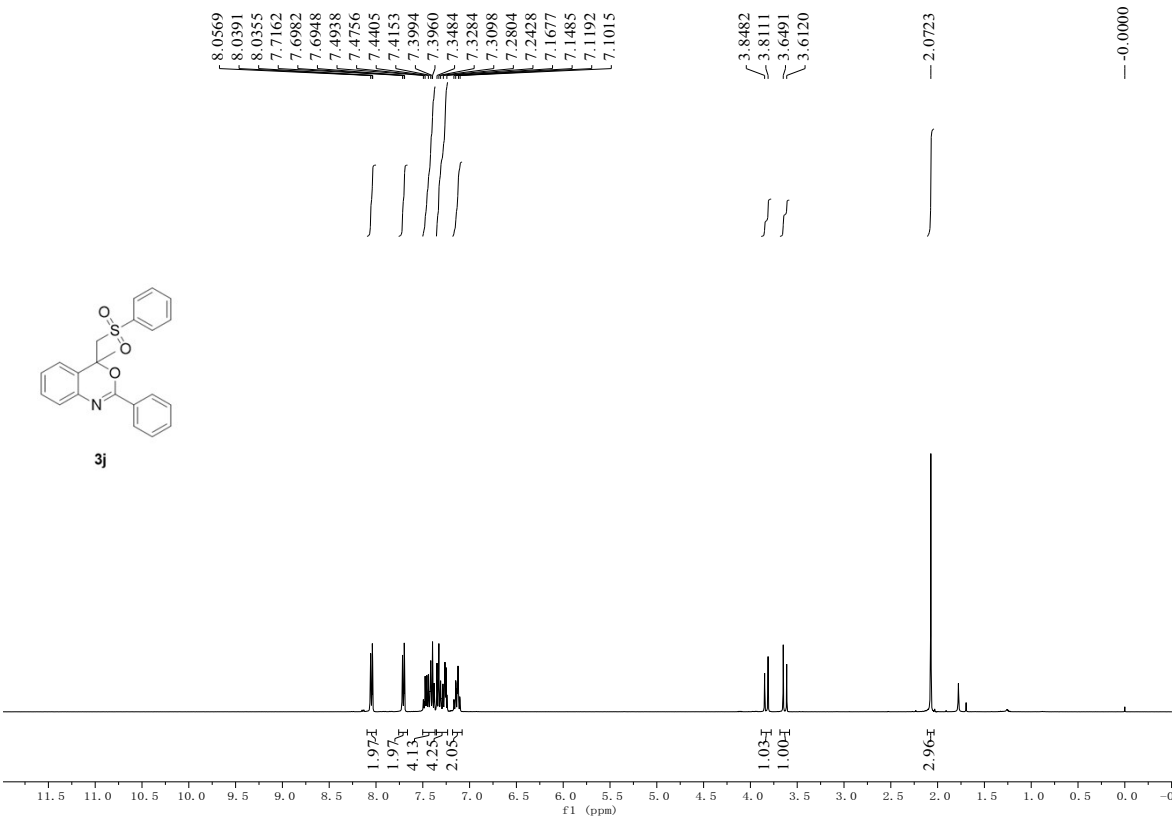
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
f1 (ppm)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8.0355
7.7162
7.6982
7.6948
7.4938
7.4756
7.4405
7.4153
7.3994
7.3960
7.3484
7.3284
7.3098
7.2804
7.2428
7.1677
7.1485
7.1192
7.1015
3.8482
3.8111
3.6491
3.6120
2.0723
-0.0000
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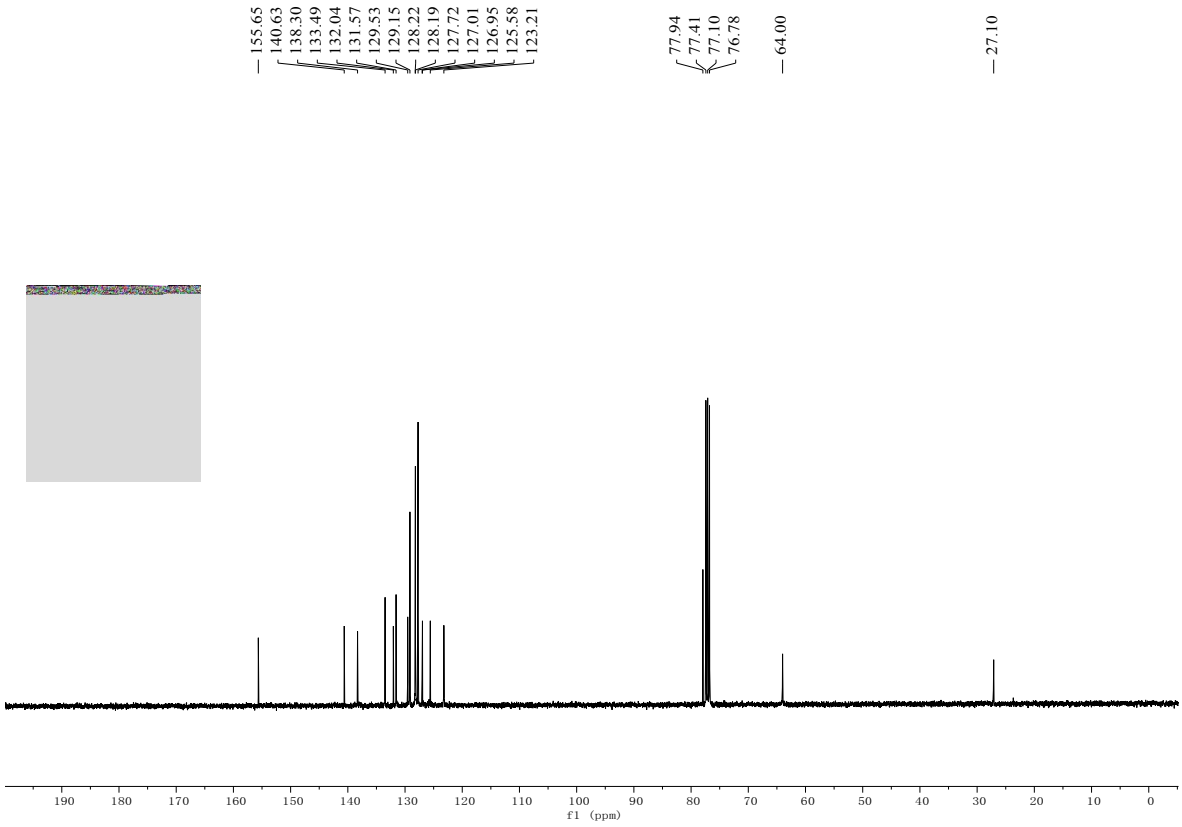
1.97
4.13
4.25
2.05
1.03
1.00
2.96
f1 (ppm)

3j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140.63
138.30
133.49
132.04
131.57
129.53
129.15
128.22
128.19
127.72
127.01
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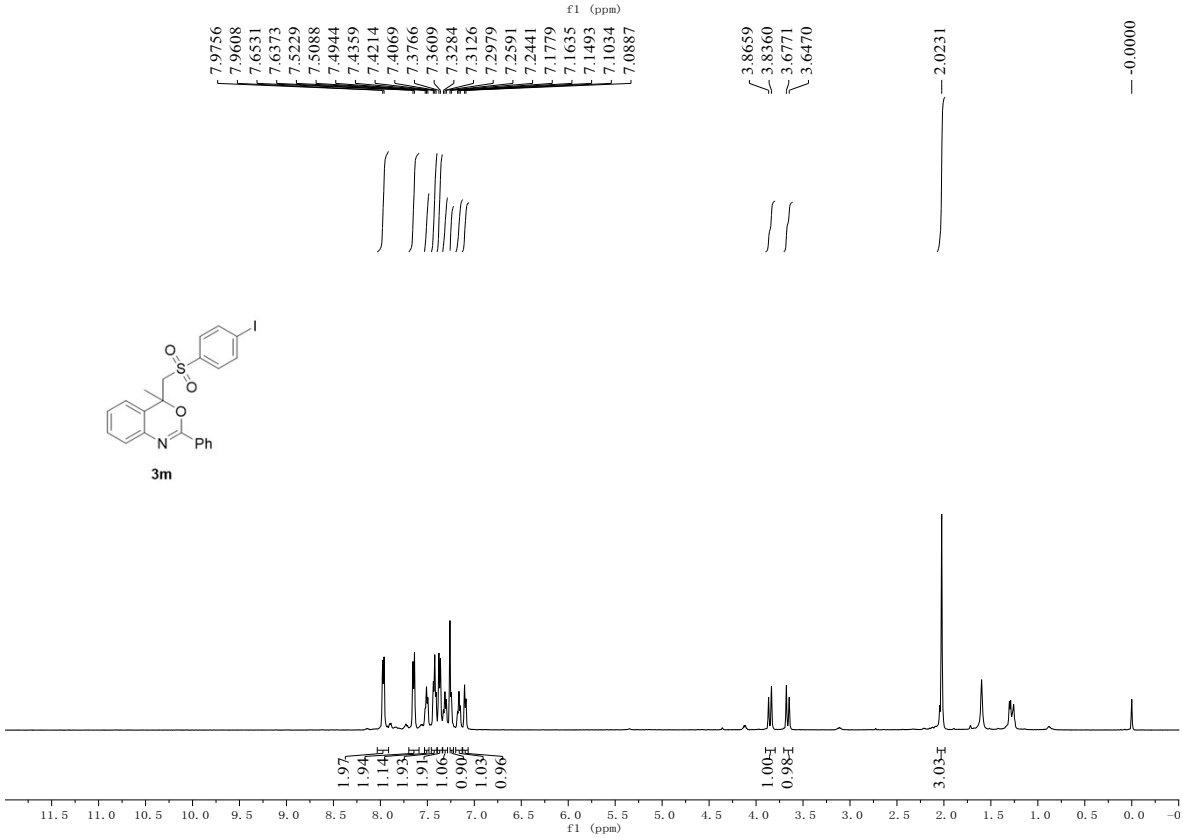
77.10
76.78
64.00
27.10
f1 (ppm)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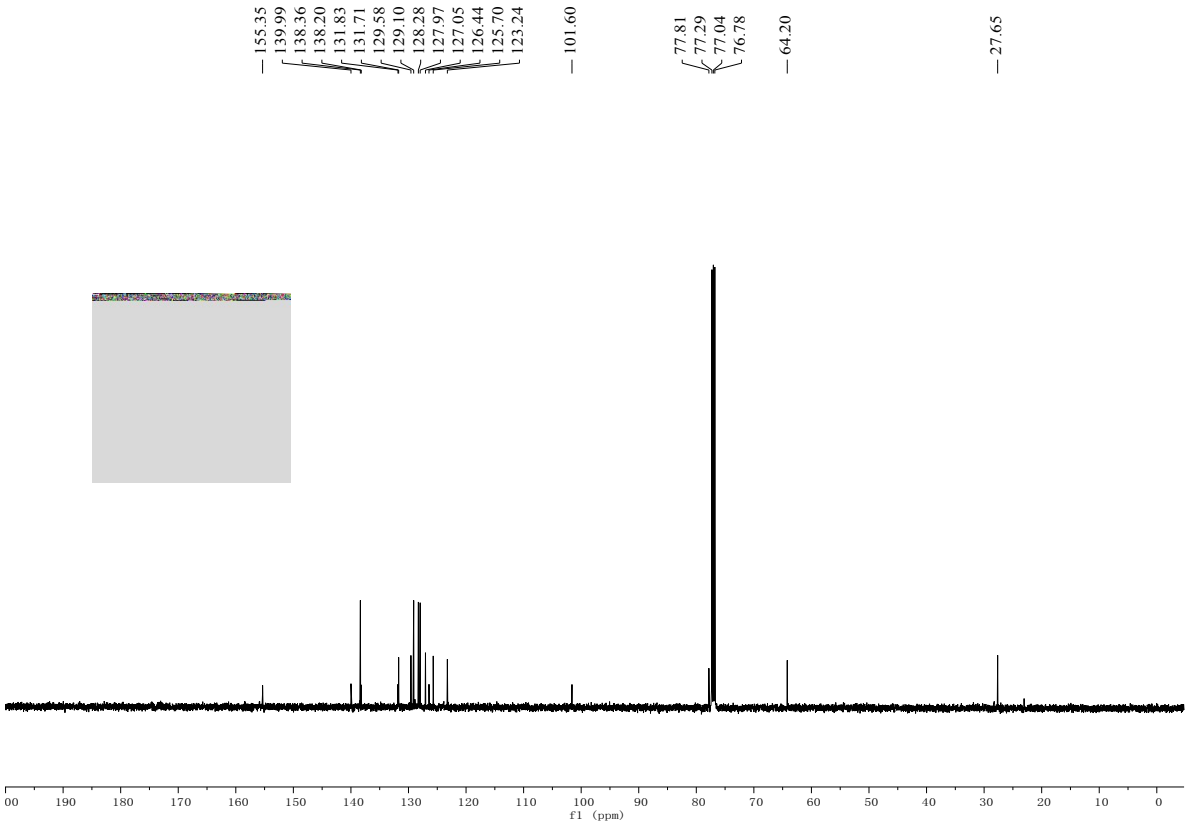
7.5229
7.5088
7.4944
7.4359
7.4214
7.4069
7.3766
7.3609
7.3284
7.3126
7.2979
7.2591
7.2441
7.1779
7.1635
7.1493
7.1034
7.0887
3.8659
3.8360
3.6771
3.6470
2.0231
-0.0000
1.97
1.94
1.14
1.93
1.91
1.06
0.90
1.03
0.96
1.00
0.98
3.03
f1 (ppm)

O
S
O
O
N
Ph
I

3m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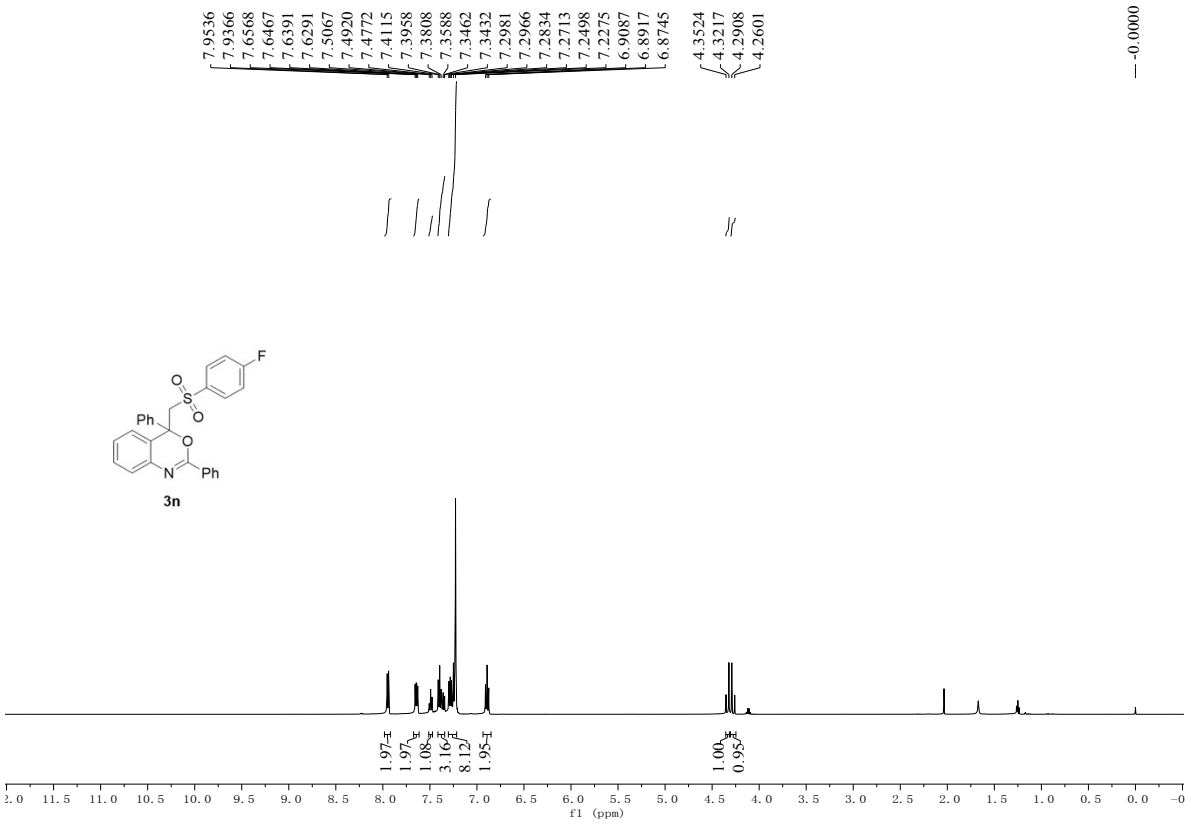
139.99
138.36
138.20
131.83
131.71
129.58
129.10
128.28
127.97
127.05
126.44
125.70
123.24
101.60
77.81
77.29
77.04
76.78
64.20
27.65
f1 (ppm)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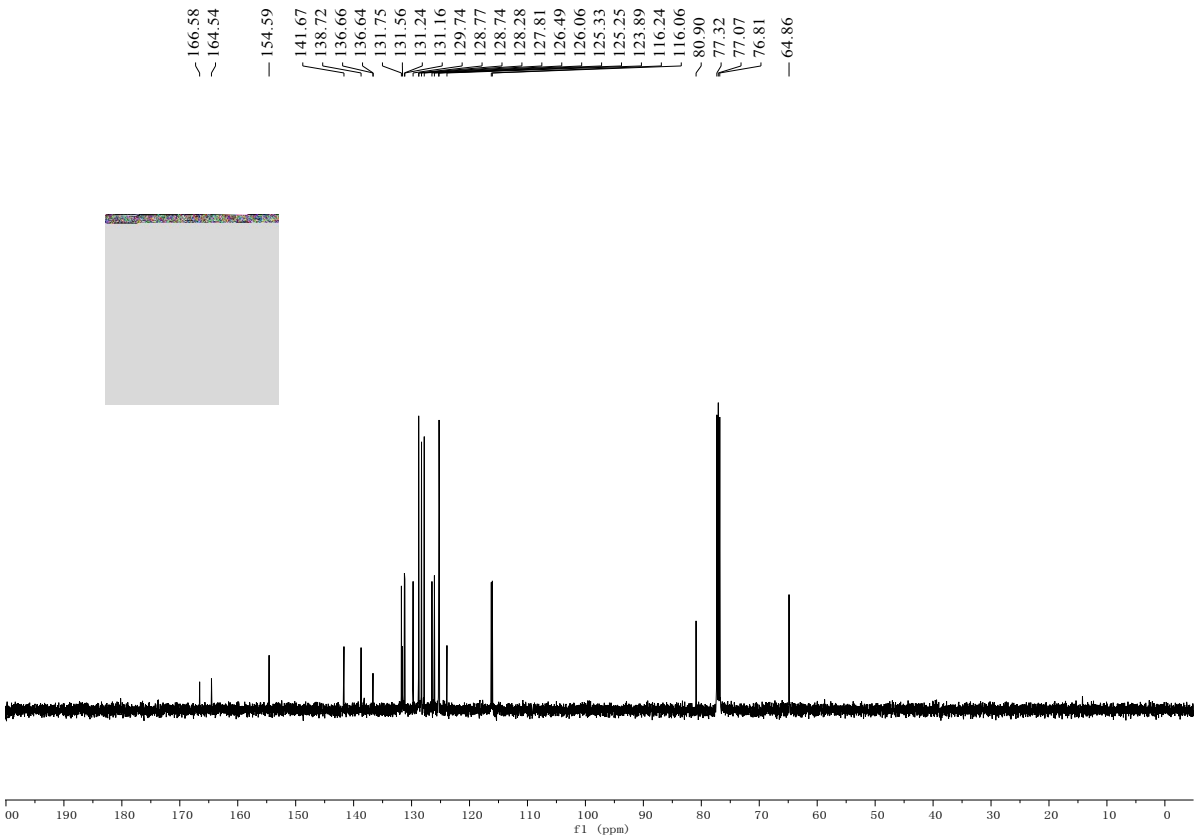
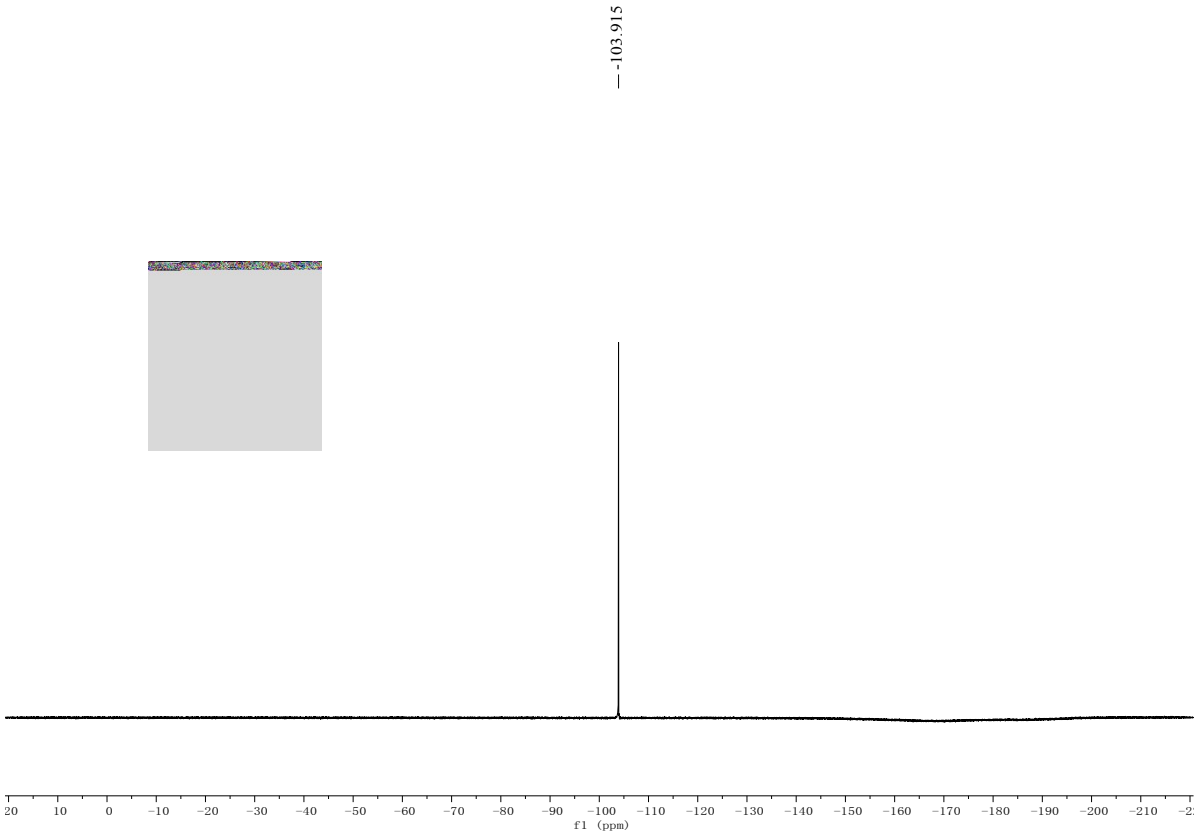
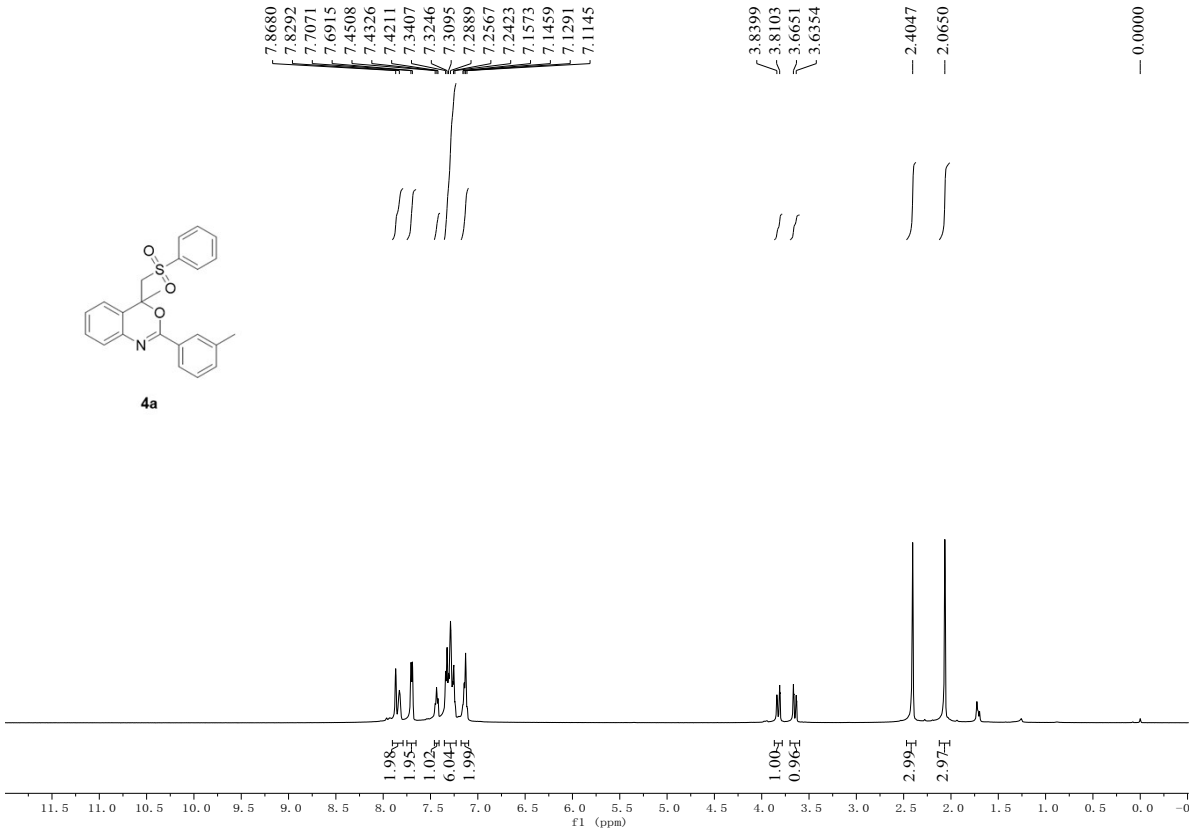
7.4920
7.4772
7.4115
7.3958
7.3808
7.3588
7.3462
7.3432
7.2981
7.2966
7.2834
7.2713
7.2498
7.2275
6.9087
6.8917
6.8745
4.3524
4.3217
4.2908
4.2601
-0.0000
F
O
S
O
Ph
O
N
Ph
3n
1.97
1.97
1.08
3.16
8.12
1.95
1.00
0.95
f1 (ppm)


166.58
164.54
154.59
141.67
138.72
136.66
136.64
131.75
131.56
131.24
131.16
129.74
128.77
128.74
128.28
127.81
126.49
126.06
125.33
125.25
123.89
116.24
116.06
80.90
77.32
77.07
76.81
64.86
f1 (ppm)


-103.915
f1 (ppm)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7.7071
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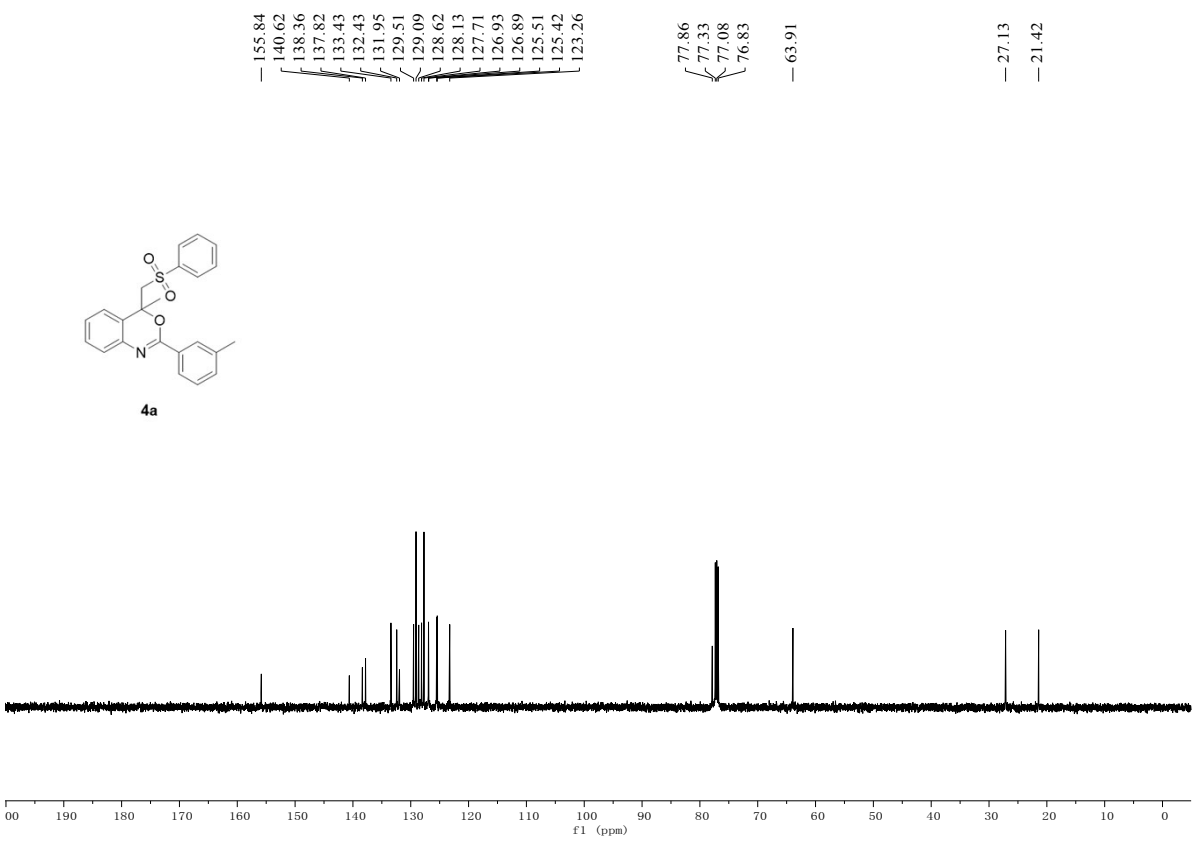
7.4508
7.4326
7.4211
7.3407
7.3246
7.3095
7.2889
7.2567
7.2423
7.1573
7.1459
7.1291
7.1145
3.8399
3.8103
3.6651
3.6354
2.4047
2.0650
0.0000
1.98
1.95
1.02
6.04
1.99
1.00
0.96
2.99
2.97
f1 (ppm)

4a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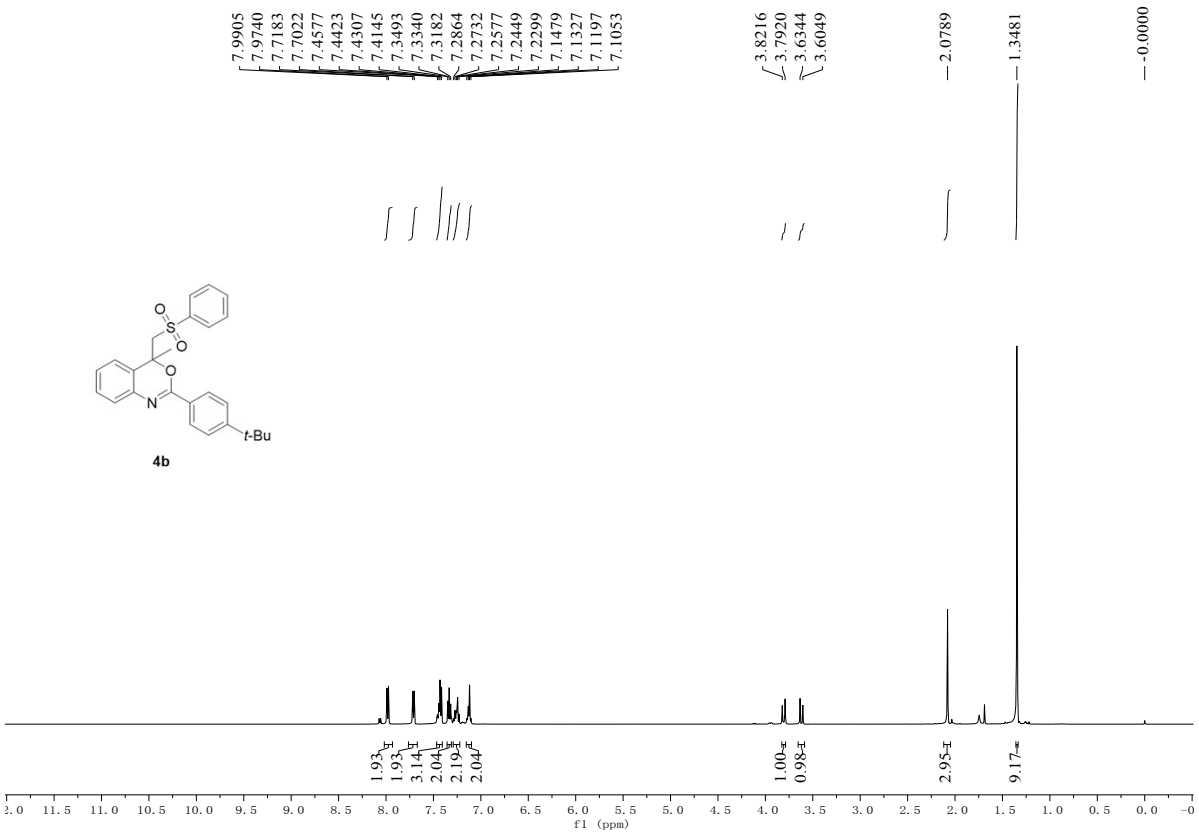
137.82
133.43
132.43
131.95
129.51
129.09
128.62
128.13
127.71
126.93
126.89
125.51
125.42
123.26
77.86
77.33
77.08
76.83
63.91
27.13
21.42
f1 (ppm)

4a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7.4577
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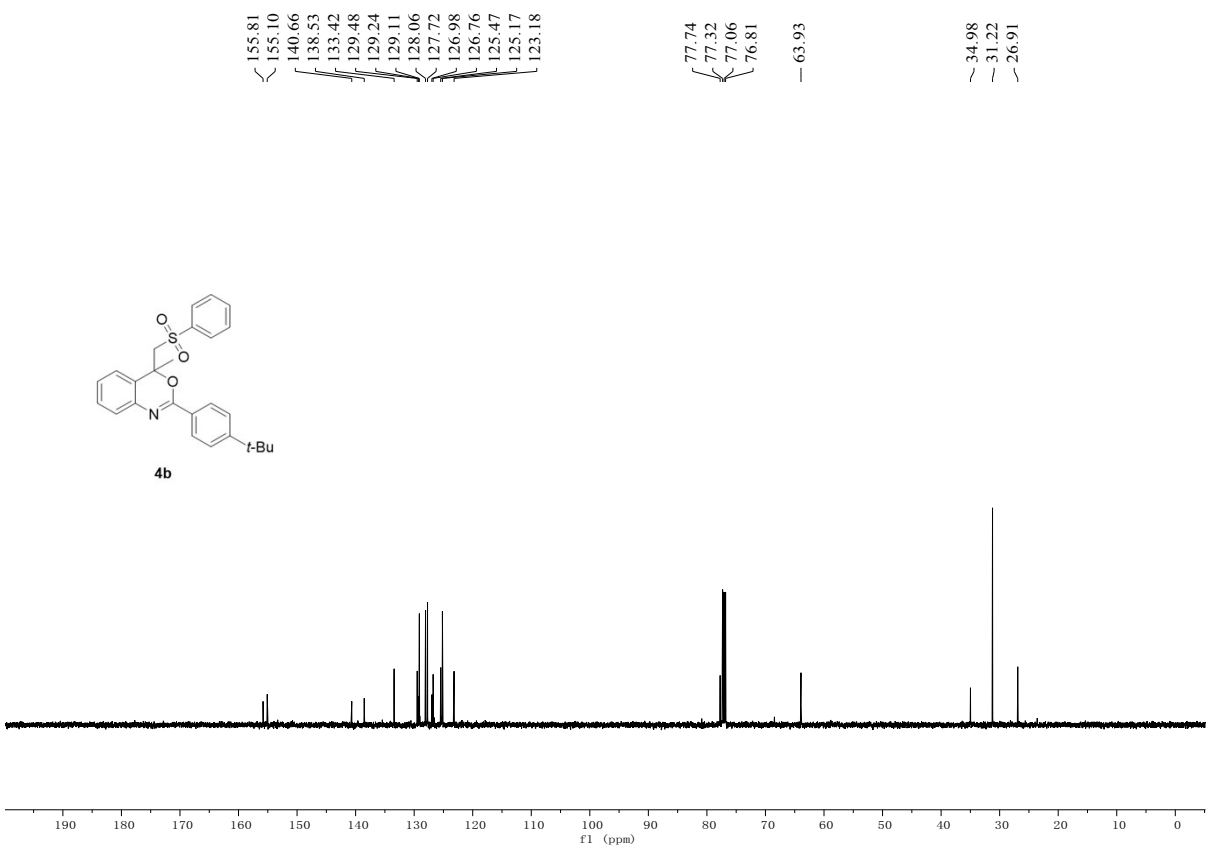
7.4307
7.4145
7.3493
7.3340
7.3182
7.2864
7.2732
7.2577
7.2449
7.2299
7.1479
7.1327
7.1197
7.1053
3.8216
3.7920
3.6344
3.6049
2.0789
1.3481
-0.0000
1.93
1.93
3.14
2.04
2.19
2.04
1.00
0.98
2.95
9.17
f1 (ppm)
t-Bu
4b


155.81
155.10
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133.42
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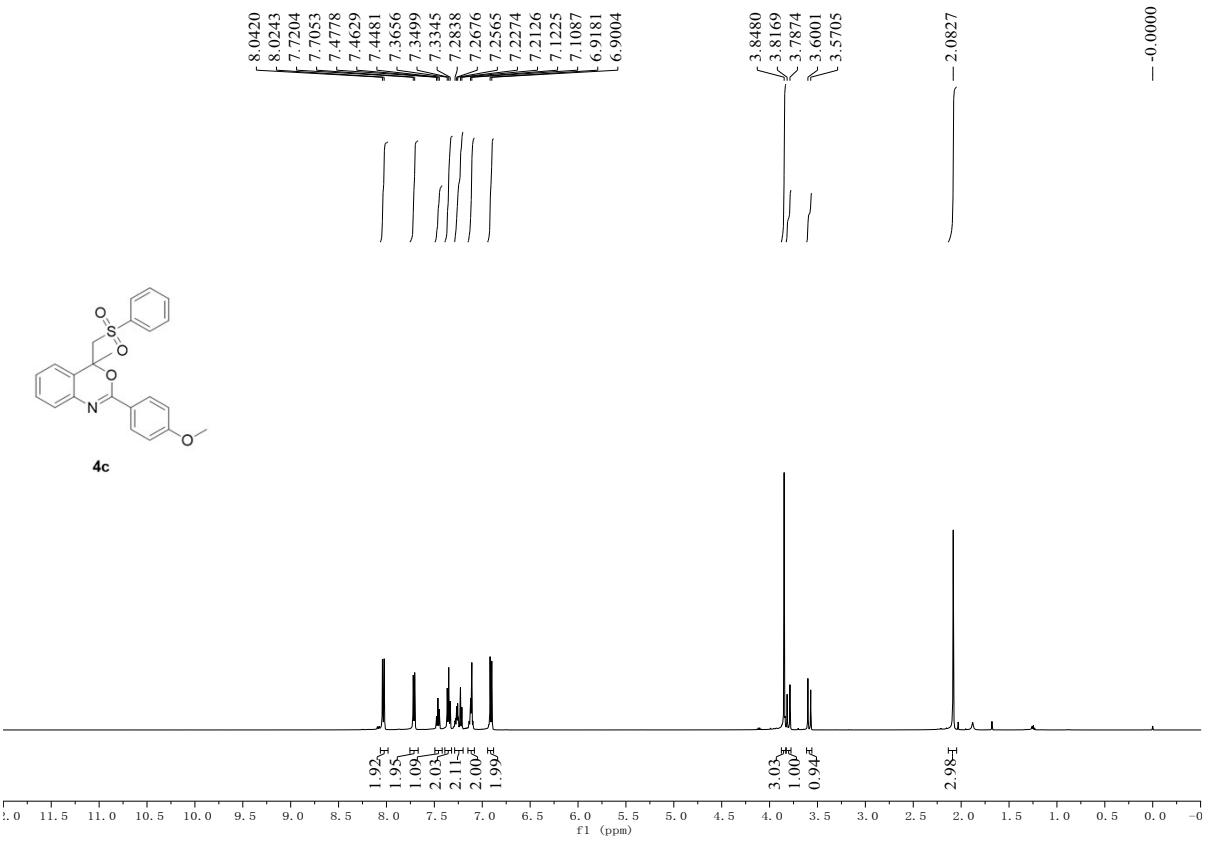
129.24
129.11
128.06
127.72
126.98
126.76
125.47
125.17
123.18
77.74
77.32
77.06
76.81
63.93
34.98
31.22
26.91
f1 (ppm)
t-Bu
4b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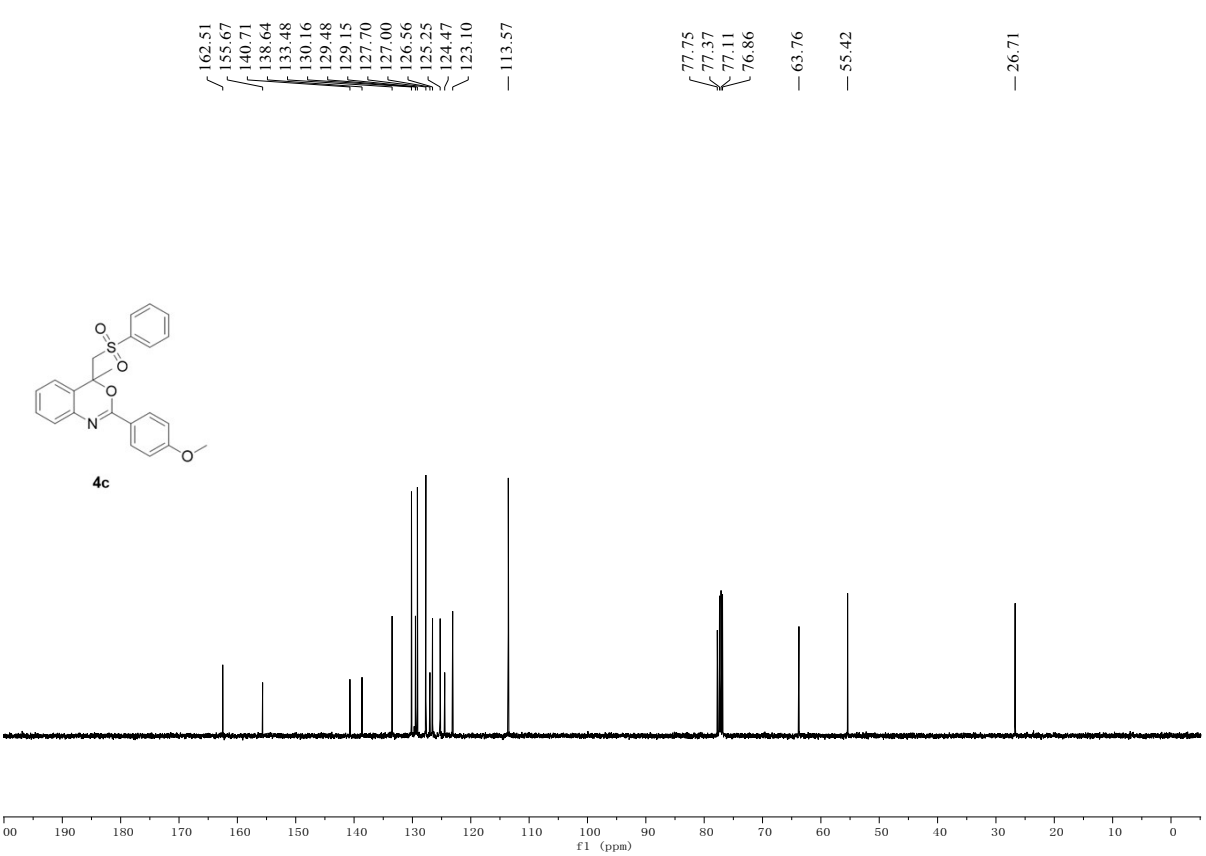
8.0243
7.7204
7.7053
7.4778
7.4629
7.4481
7.3656
7.3499
7.3345
7.2838
7.2676
7.2565
7.2274
7.2126
7.1225
7.1087
6.9181
6.9004
3.8480
3.8169
3.7874
3.6001
3.5705
2.0827
-0.0000
1.92
1.95
1.09
2.03
2.11
2.00
1.99
3.03
1.00
0.94
2.98
f1 (ppm)

4c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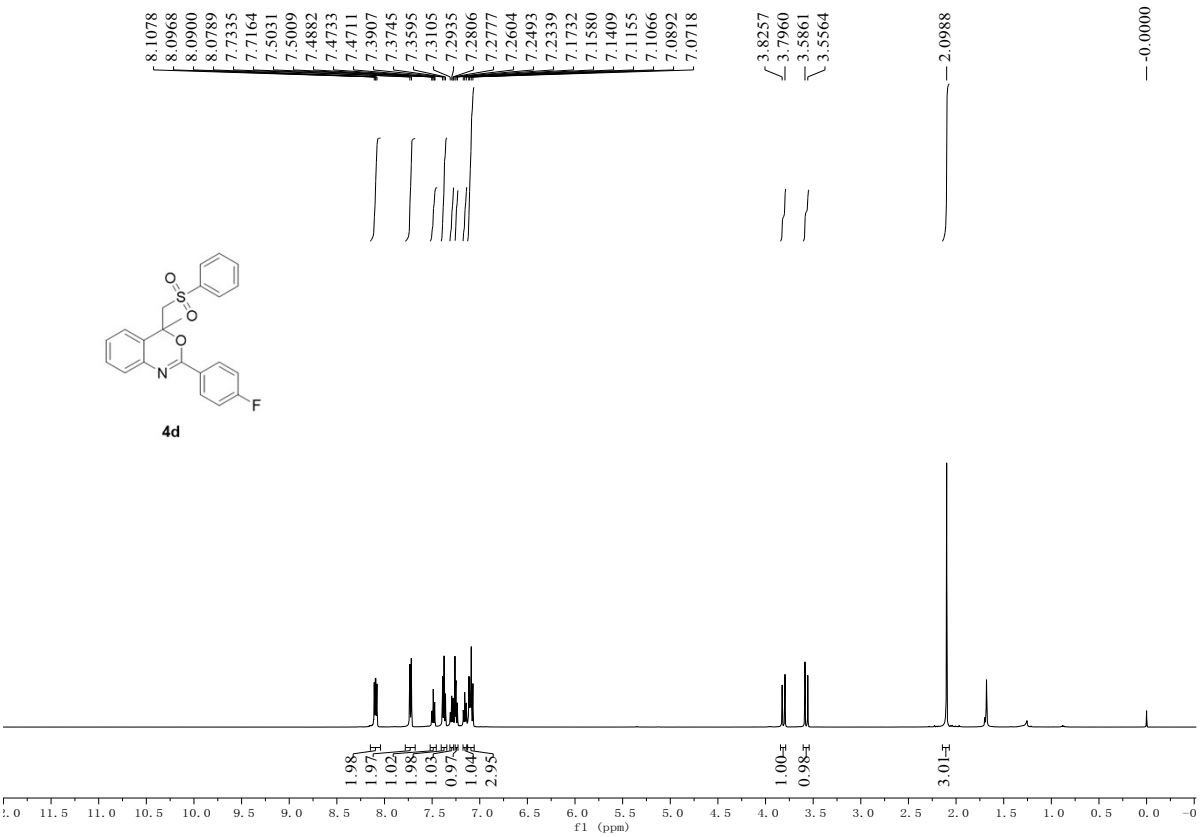
130.16
129.48
129.15
127.70
127.00
126.56
125.25
124.47
123.10
113.57
77.75
77.37
77.11
76.86
63.76
55.42
26.71
f1 (ppm)

4c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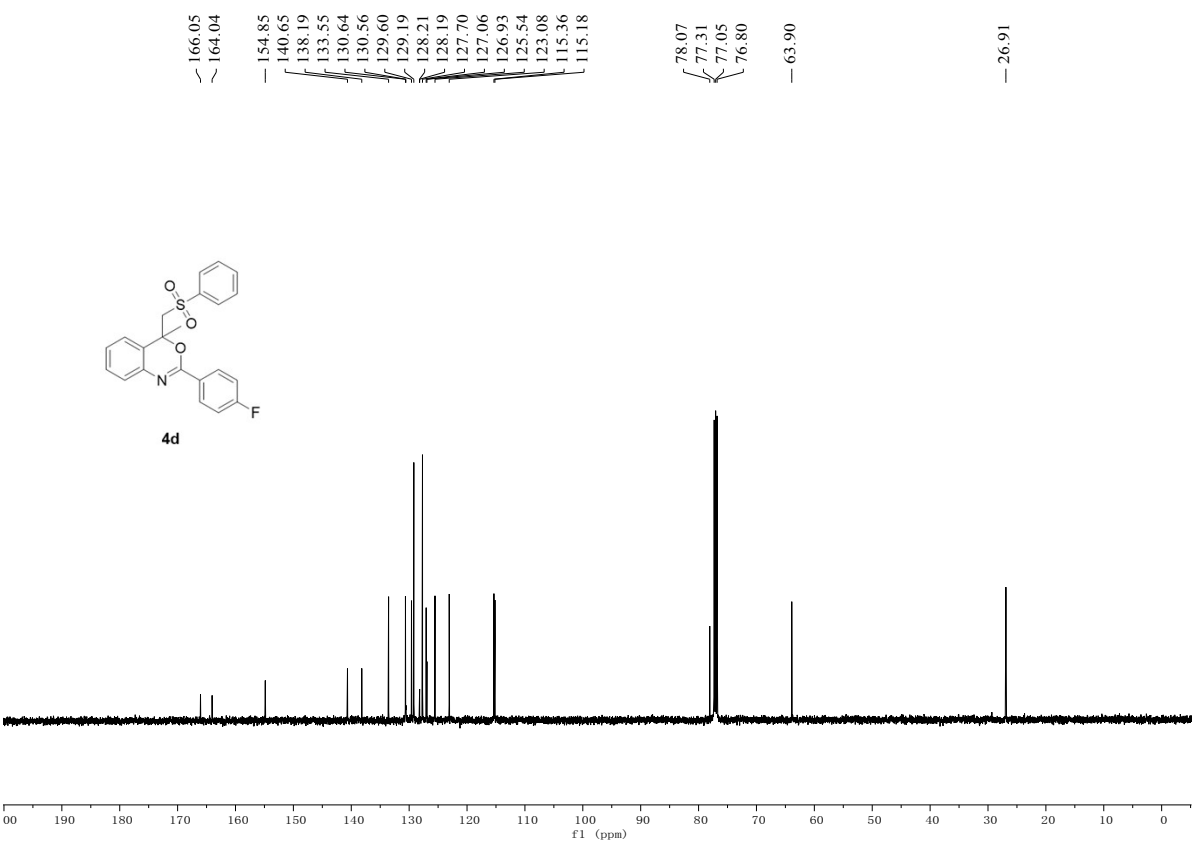
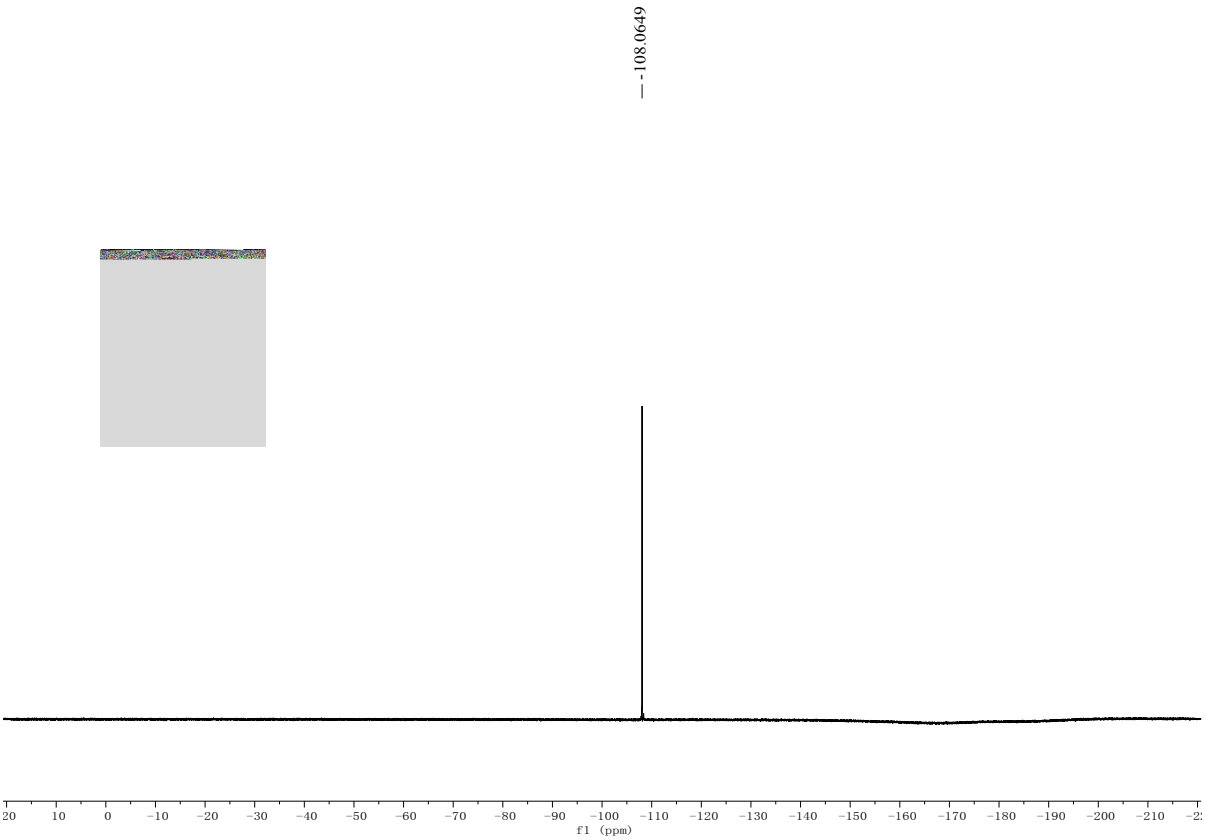
8.0900
8.0789
7.7335
7.7164
7.5031
7.5009
7.4882
7.4733
7.4711
7.3907
7.3745
7.3595
7.3105
7.2935
7.2806
7.2777
7.2604
7.2493
7.2339
7.1732
7.1580
7.1409
7.1155
7.1066
7.0892
7.0718
3.8257
3.7960
3.5861
3.5564
2.0988
-0.0000
1.98
1.97
1.02
1.98
1.03
0.97
1.04
2.95
1.00
0.98
3.01
f1 (ppm)

4d


166.05
164.04
154.85
140.65
138.19
133.55
130.64
130.56
129.60
129.19
128.21
128.19
127.70
127.06
126.93
125.54
123.08
115.36
115.18
78.07
77.31
77.05
76.80
63.90
26.91
f1 (ppm)

4d


-108.0649
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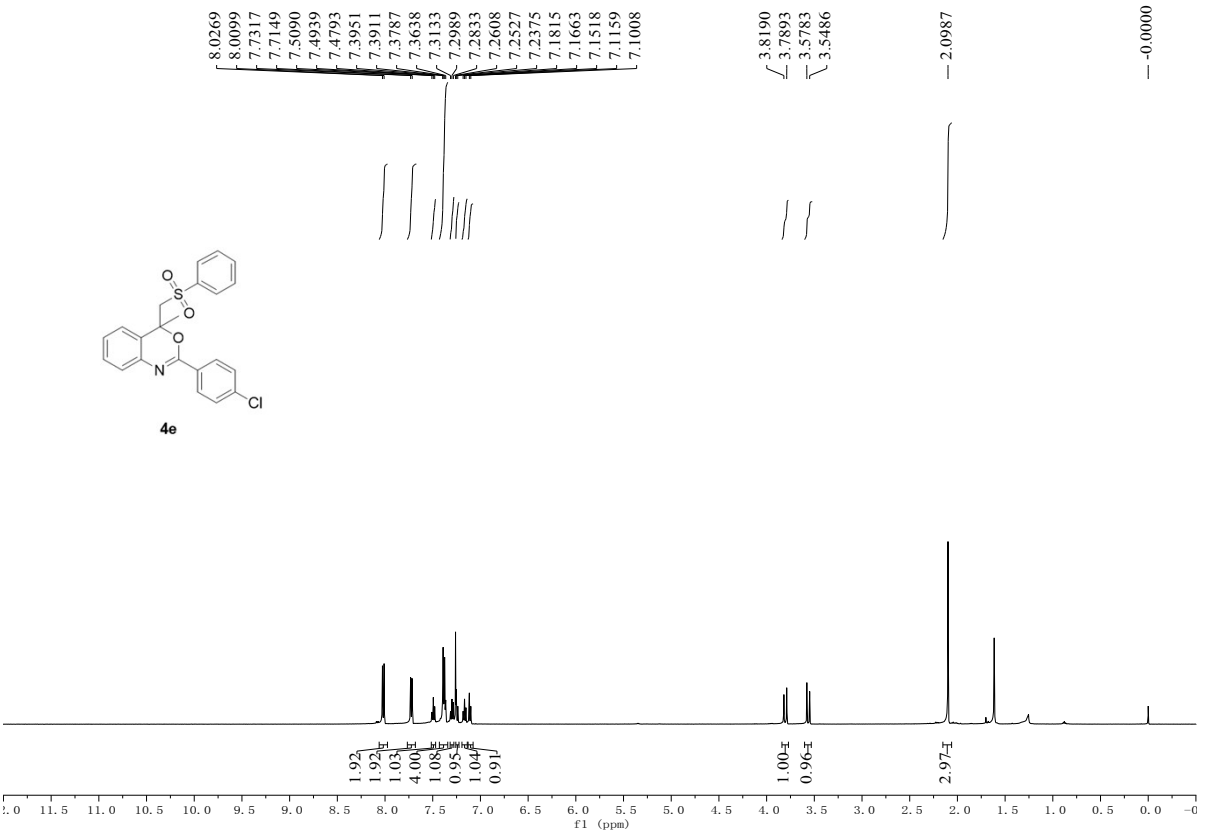
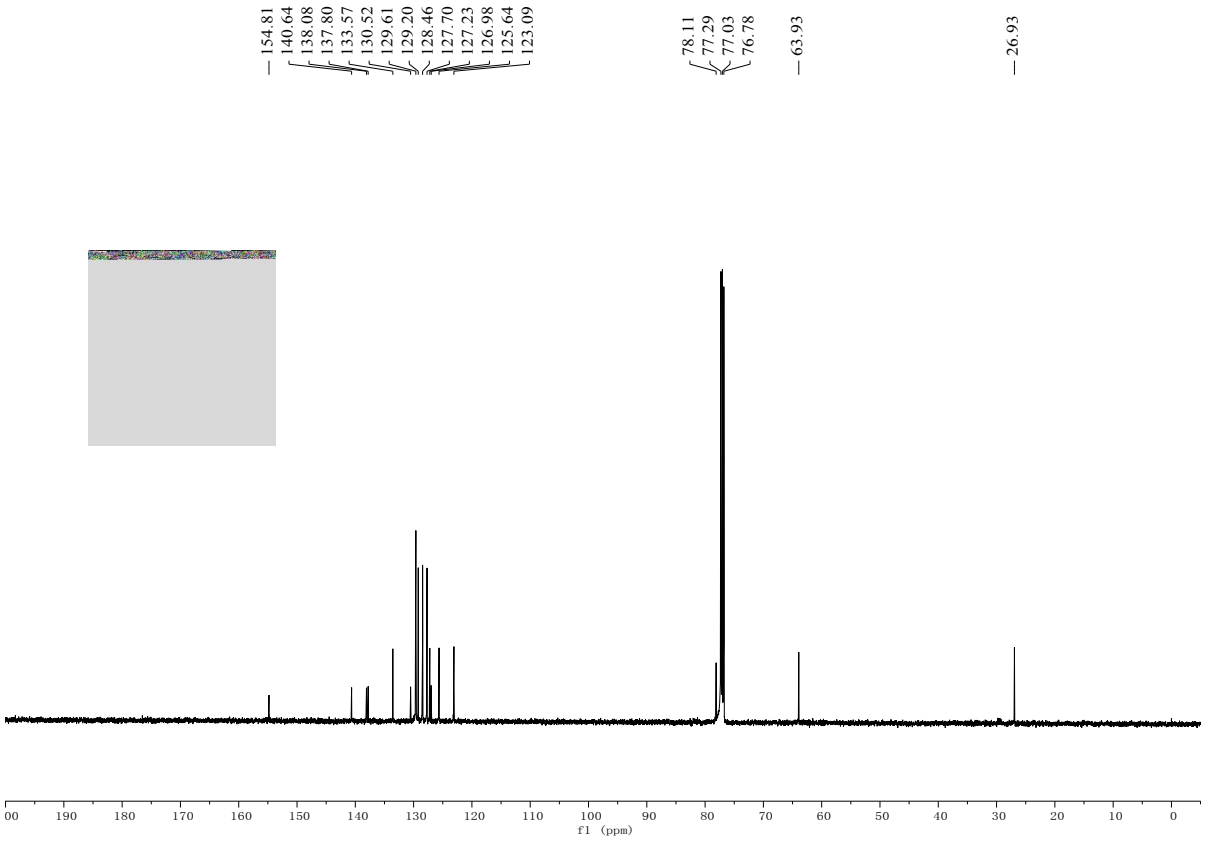
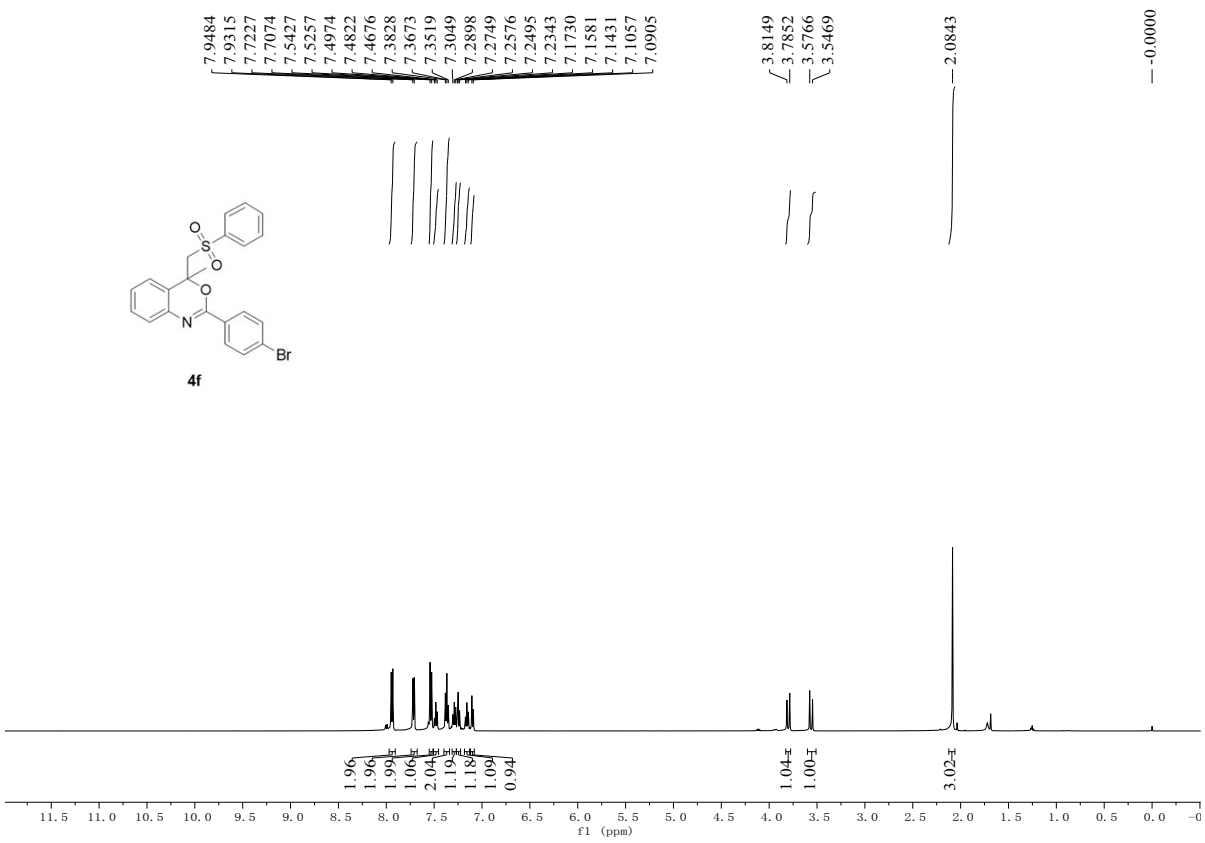

8.0269
8.0099
7.7317
7.7149
7.5090
7.4939
7.4793
7.3951
7.3911
7.3787
7.3638
7.3133
7.2989
7.2833
7.2608
7.2527
7.2375
7.1815
7.1663
7.1518
7.1159
7.1008
3.8190
3.7893
3.5783
3.5486
2.0987
-0.0000
1.92
1.92
1.03
4.00
1.08
0.95
1.04
0.91
1.00
0.96
2.97
f1 (ppm)

O
S
O
O
N
Cl

4e


154.81
140.64
138.08
137.80
133.57
130.52
129.61
129.20
128.46
127.70
127.23
126.98
125.64
123.09
78.11
77.29
77.03
76.78
63.93
26.93
f1 (ppm)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7.7227
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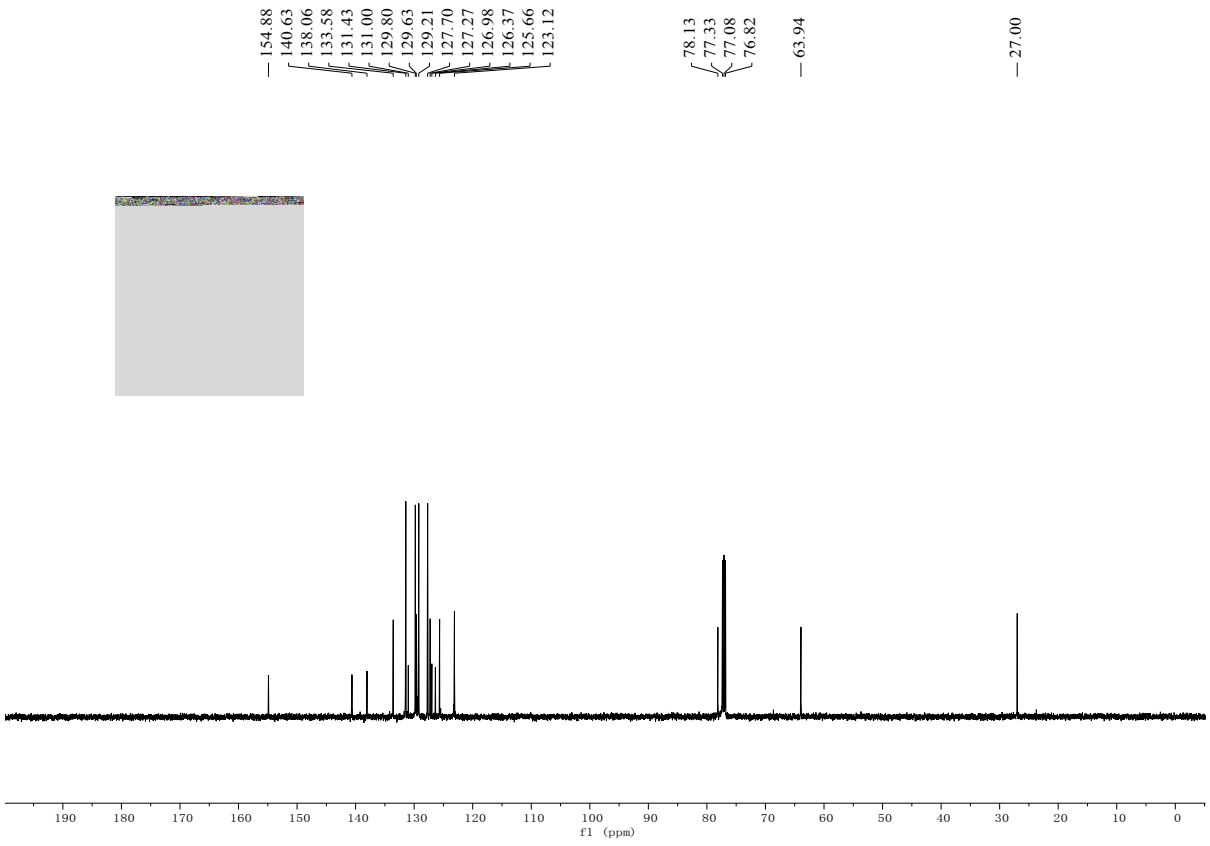
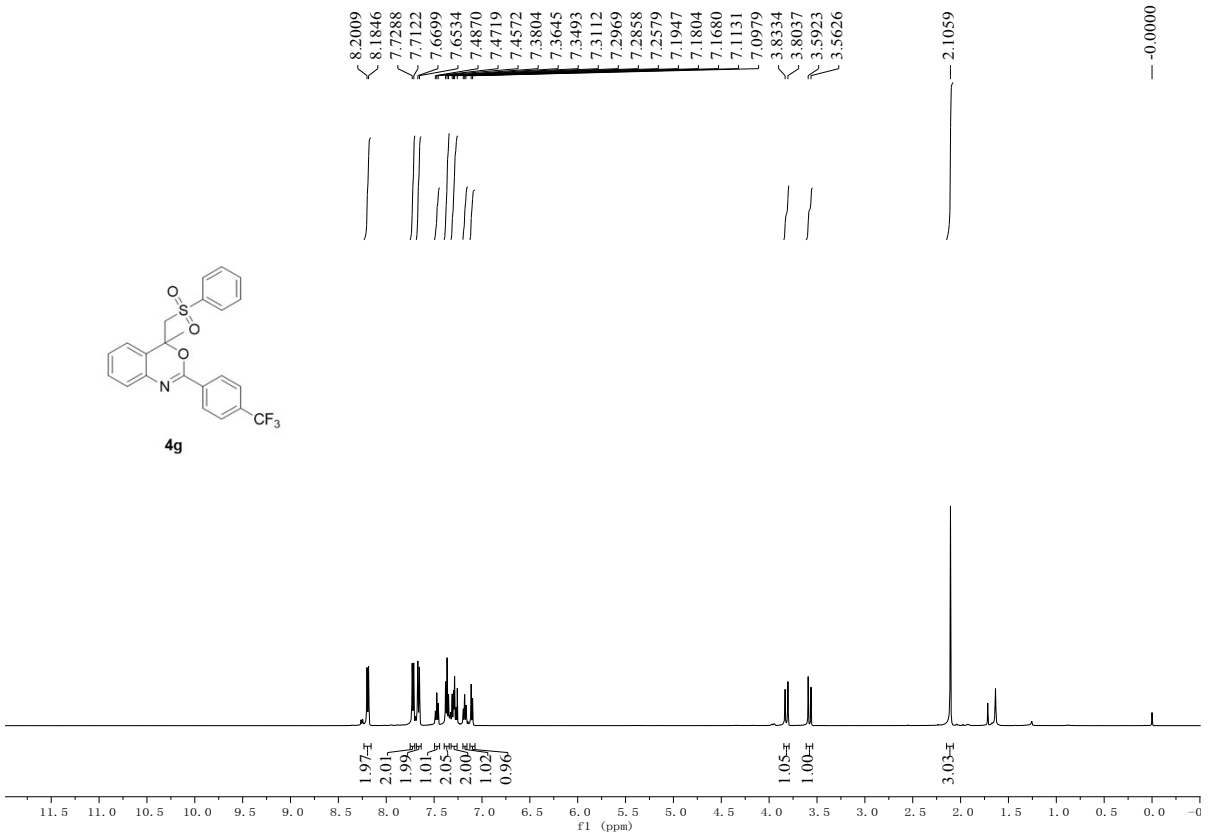
7.5427
7.5257
7.4974
7.4822
7.4676
7.3828
7.3673
7.3519
7.3049
7.2898
7.2749
7.2576
7.2495
7.2343
7.1730
7.1581
7.1431
7.1057
7.0905
3.8149
3.7852
3.5766
3.5469
2.0843
-0.0000
1.96
1.96
1.99
1.06
2.04
1.19
1.18
1.09
0.94
1.04
1.00
3.02
f1 (ppm)

O
S
O
O
N
Br

4f


154.88
140.63
138.06
133.58
131.43
131.00
129.80
129.63
129.21
127.70
127.27
126.98
126.37
125.66
123.12
78.13
77.33
77.08
76.82
63.94
27.00
f1 (ppm)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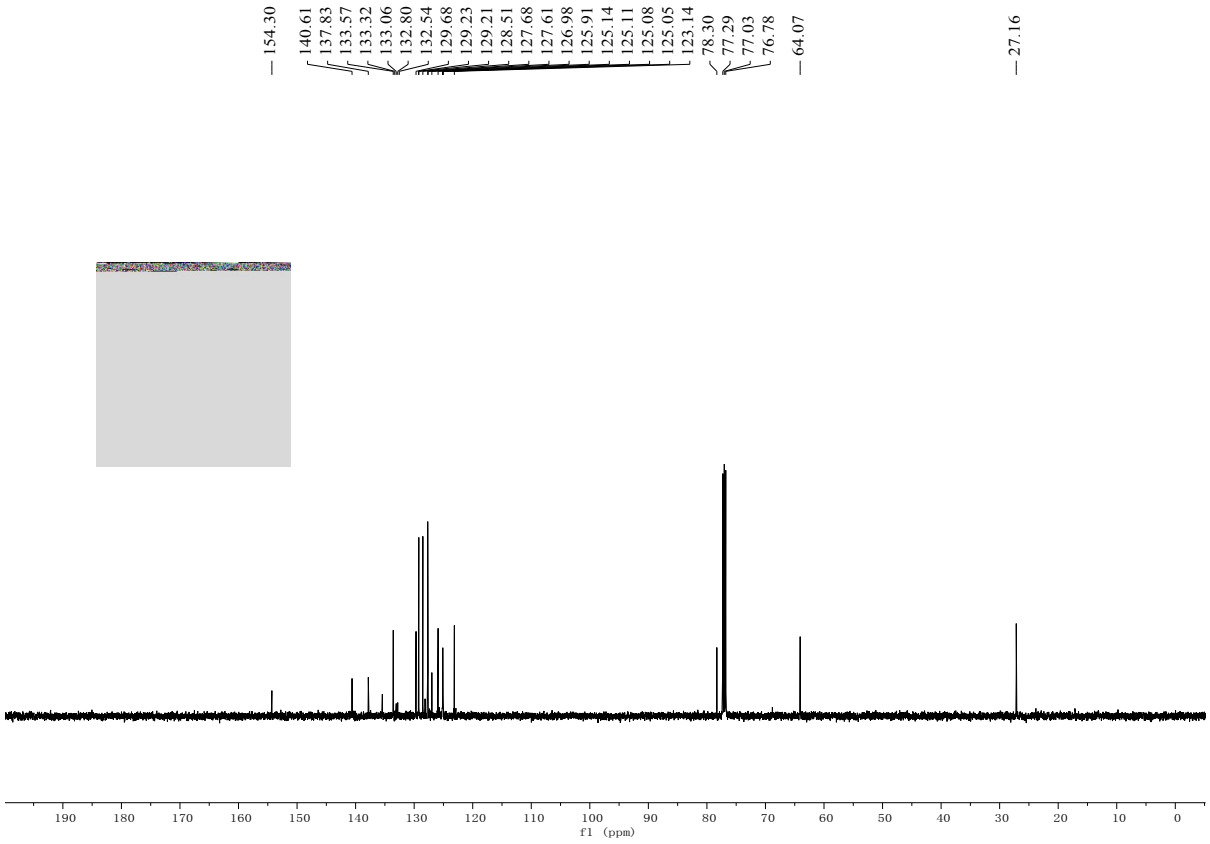
8.1846
7.7288
7.7122
7.6699
7.6534
7.4870
7.4719
7.4572
7.3804
7.3645
7.3493
7.3112
7.2969
7.2858
7.2579
7.1947
7.1804
7.1680
7.1131
7.0979
3.8334
3.8037
3.5923
3.5626
2.1059
-0.0000
1.97
2.01
1.99
1.01
2.05
2.00
1.02
0.96
1.05
1.00
3.03
f1 (ppm)

O
S
O
O
N
CF3

4g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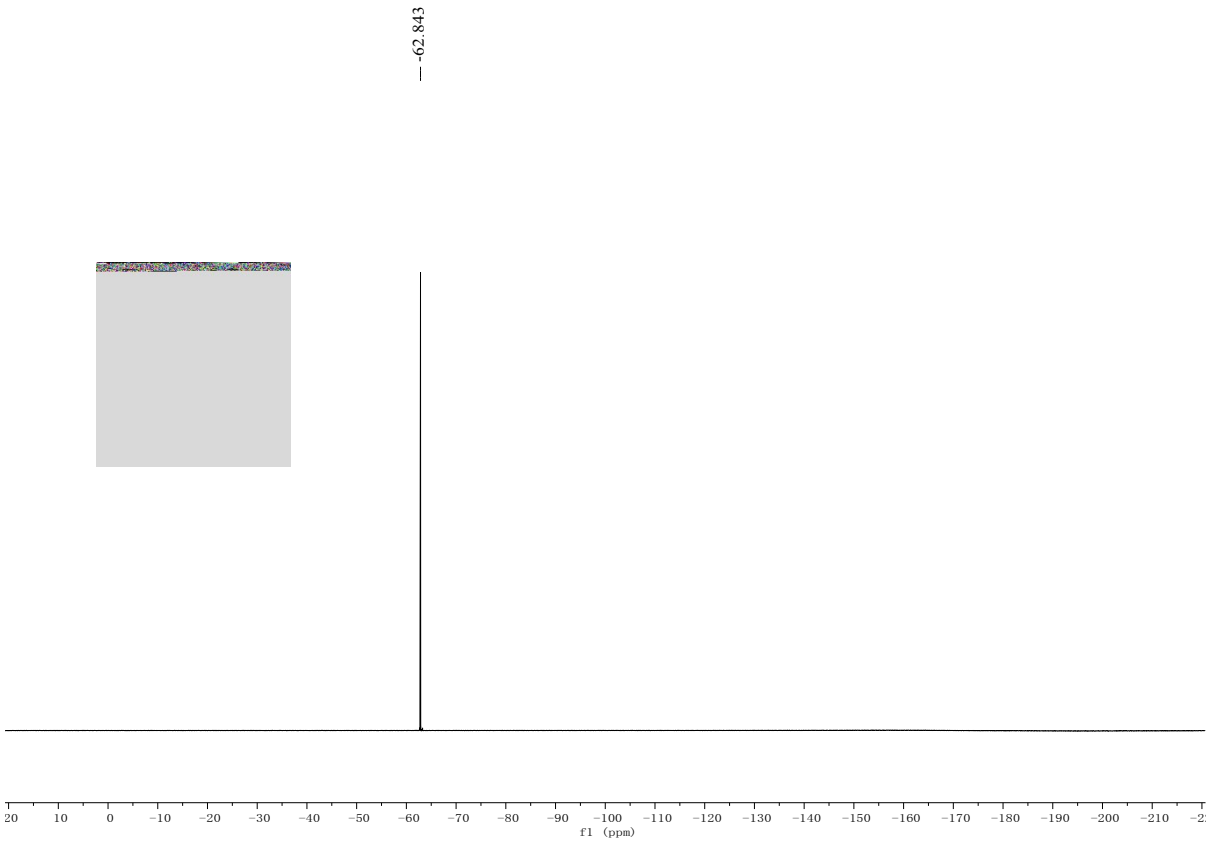
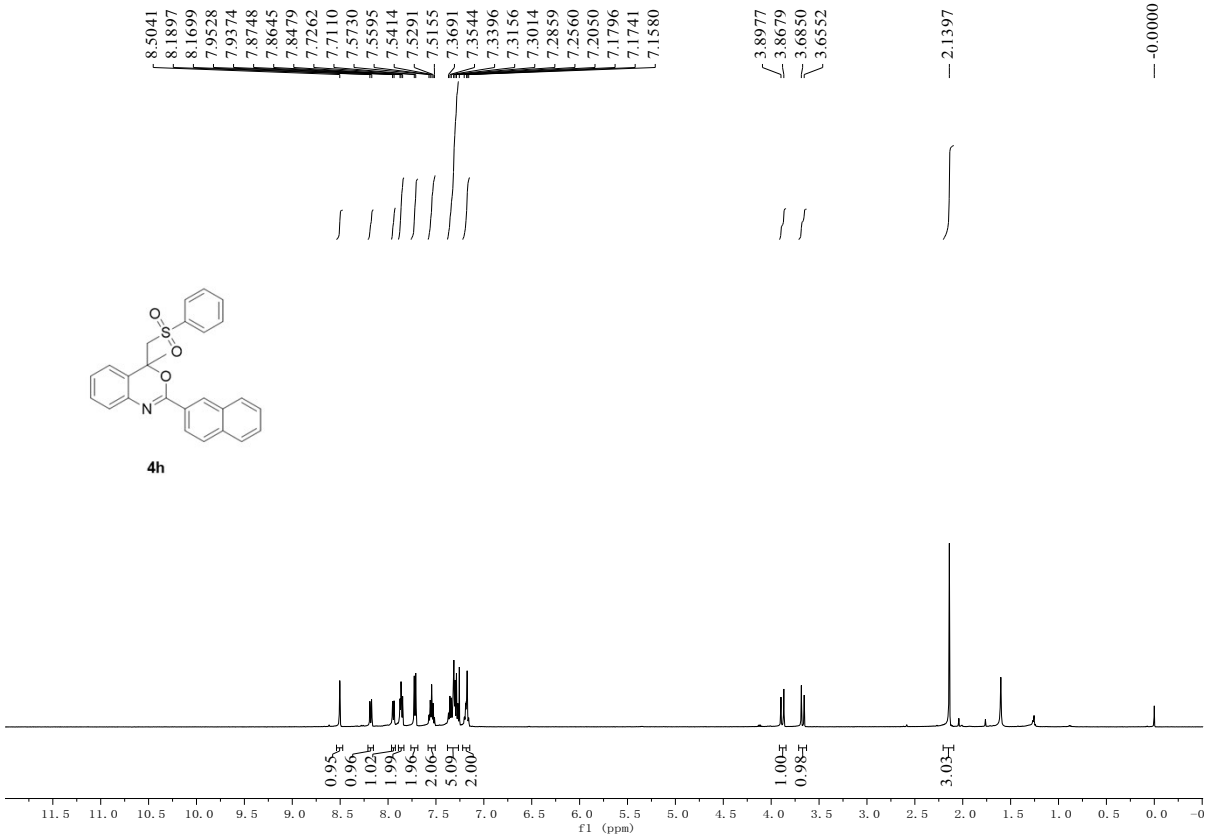
140.61
137.83
133.57
133.32
133.06
132.80
132.54
129.68
129.23
129.21
128.51
127.68
127.61
126.98
125.91
125.14
125.11
125.08
125.05
123.14
78.30
77.29
77.03
76.78
64.07
27.16
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-62.843
f1 (ppm)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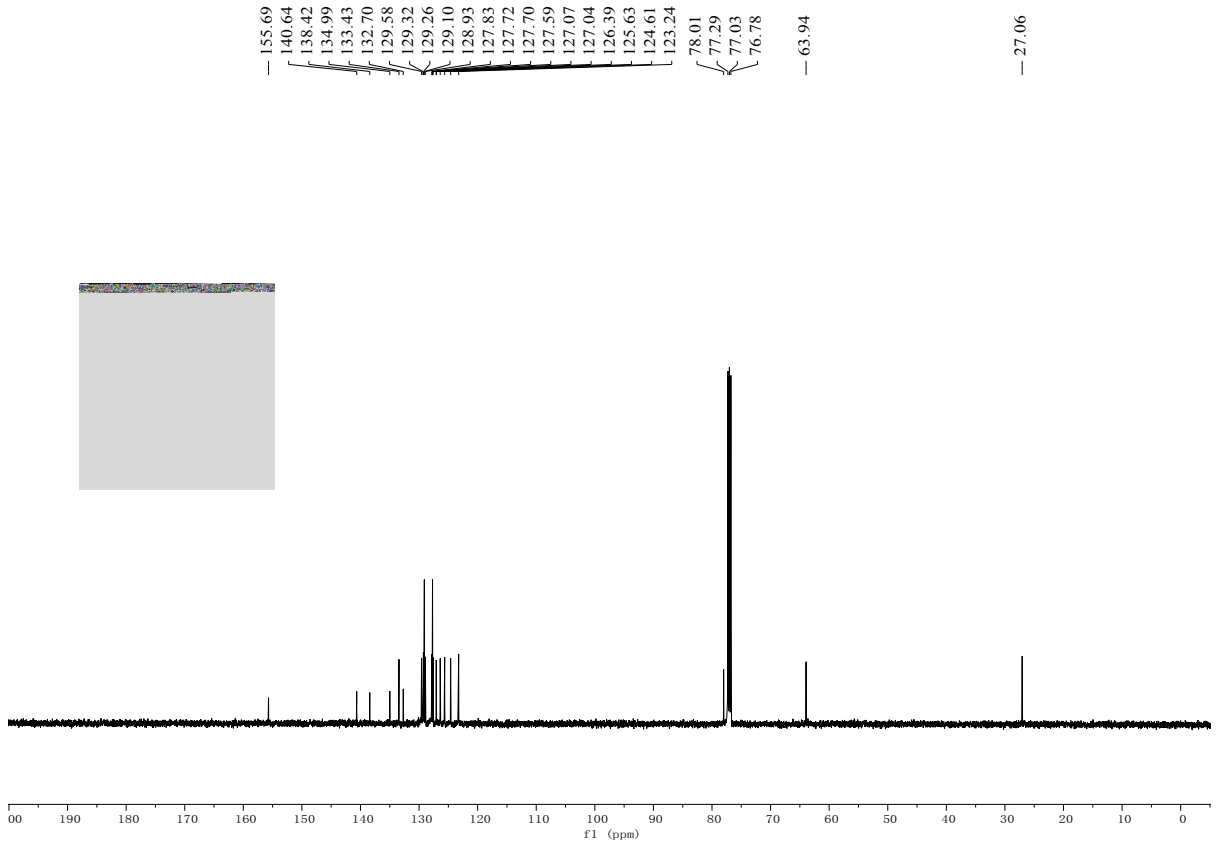
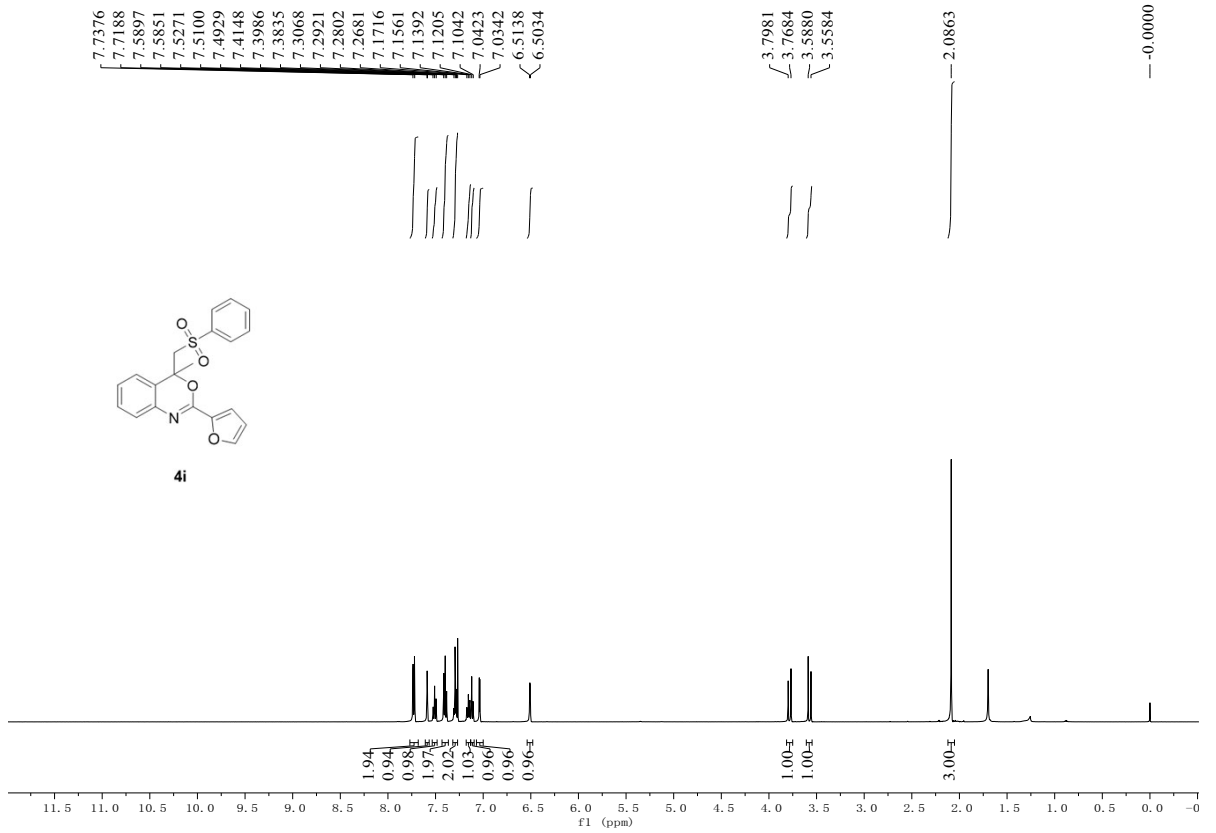
8.1897
8.1699
7.9528
7.9374
7.8748
7.8645
7.8479
7.7262
7.7110
7.5730
7.5595
7.5414
7.5291
7.5155
7.3691
7.3544
7.3396
7.3156
7.3014
7.2859
7.2560
7.2050
7.1796
7.1741
7.1580
3.8977
3.8679
3.6850
3.6552
2.1397
-0.0000
0.95
0.96
1.02
1.99
1.96
2.06
5.09
2.00
1.00
0.98
3.03
f1 (ppm)

4h


155.69
140.64
138.42
134.99
133.43
132.70
129.58
129.32
129.26
129.10
128.93
127.83
127.72
127.70
127.59
127.07
127.04
126.39
125.63
124.61
123.24
78.01
77.29
77.03
76.78
63.94
27.06
f1 (ppm)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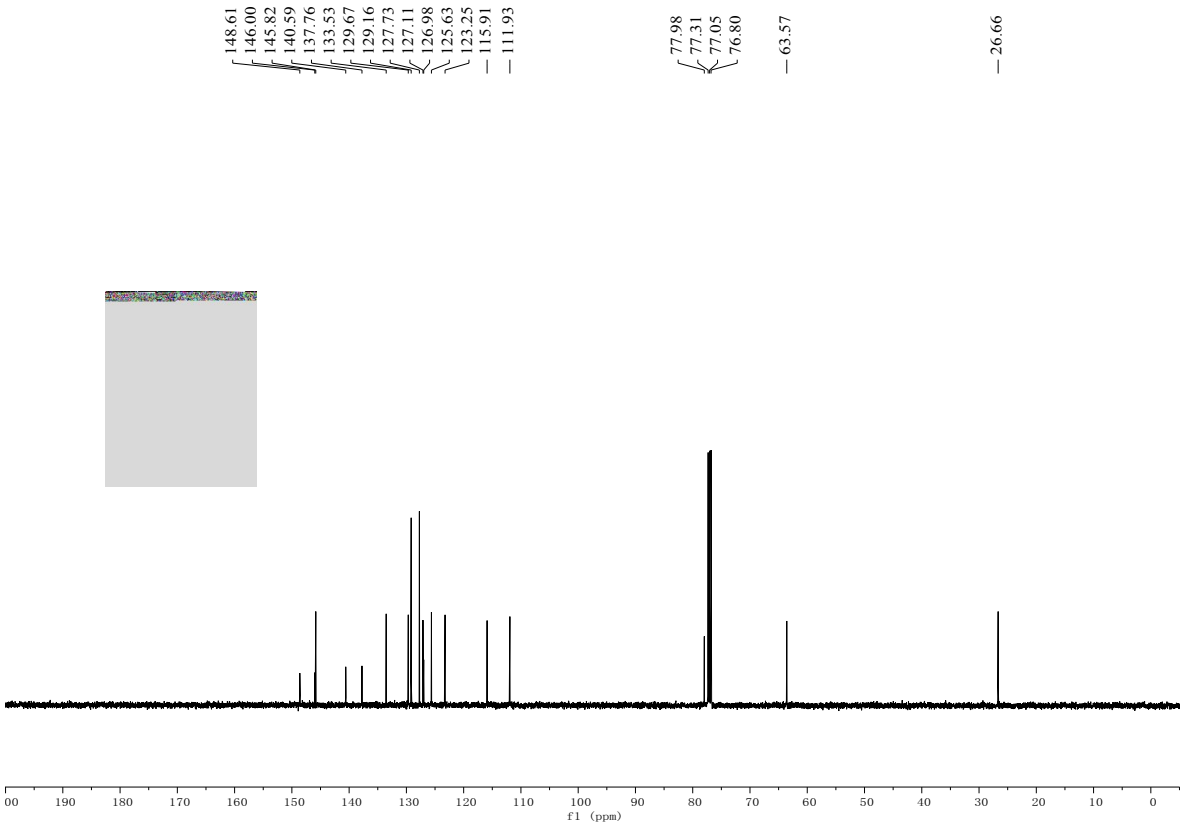
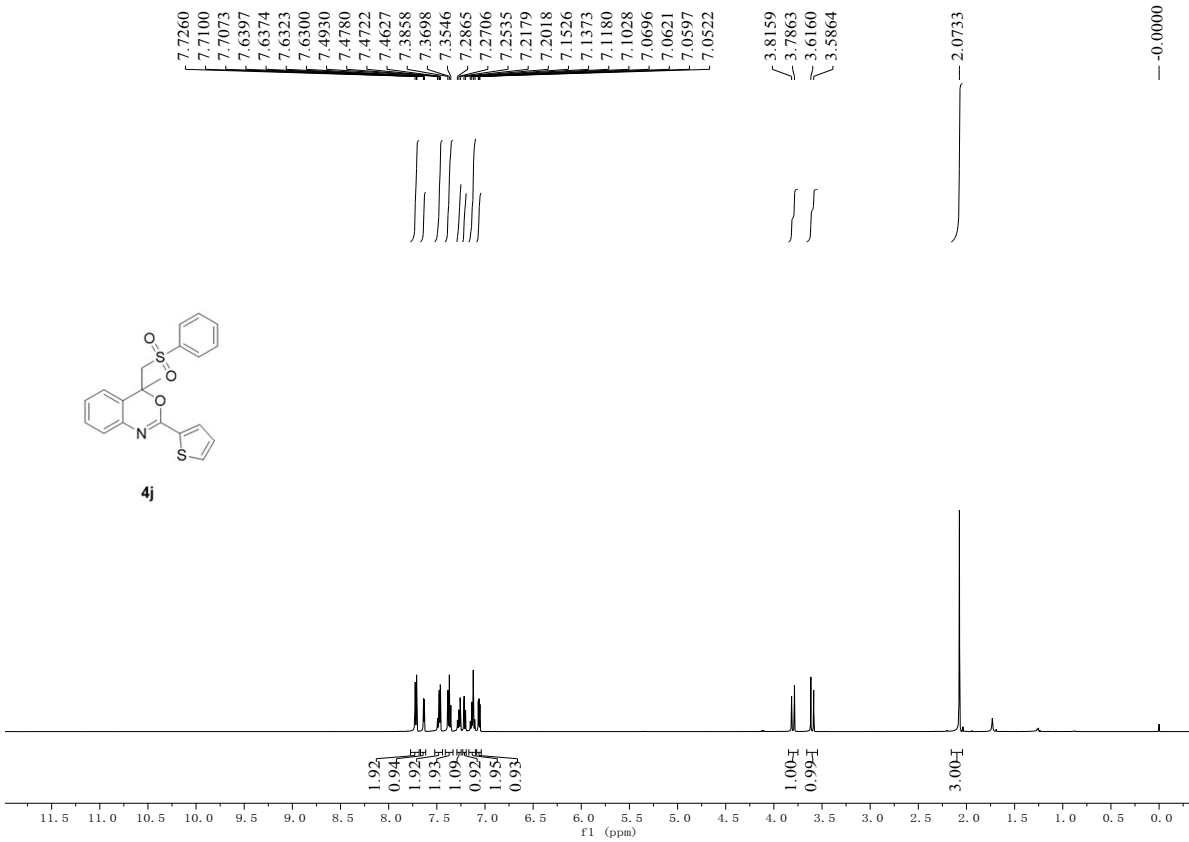
7.5897
7.5851
7.5271
7.5100
7.4929
7.4148
7.3986
7.3835
7.3068
7.2921
7.2802
7.2681
7.1716
7.1561
7.1392
7.1205
7.1042
7.0423
7.0342
6.5138
6.5034
3.7981
3.7684
3.5880
3.5584
2.0863
-0.0000
4i
1.94
0.94
0.98
1.97
2.02
1.03
0.96
0.96
1.00
1.00
3.00
f1 (ppm)


148.61
146.00
145.82
140.59
137.76
133.53
129.67
129.16
127.73
127.11
126.98
125.63
123.25
115.91
111.93
77.98
77.31
77.05
76.80
63.57
26.66
f1 (ppm)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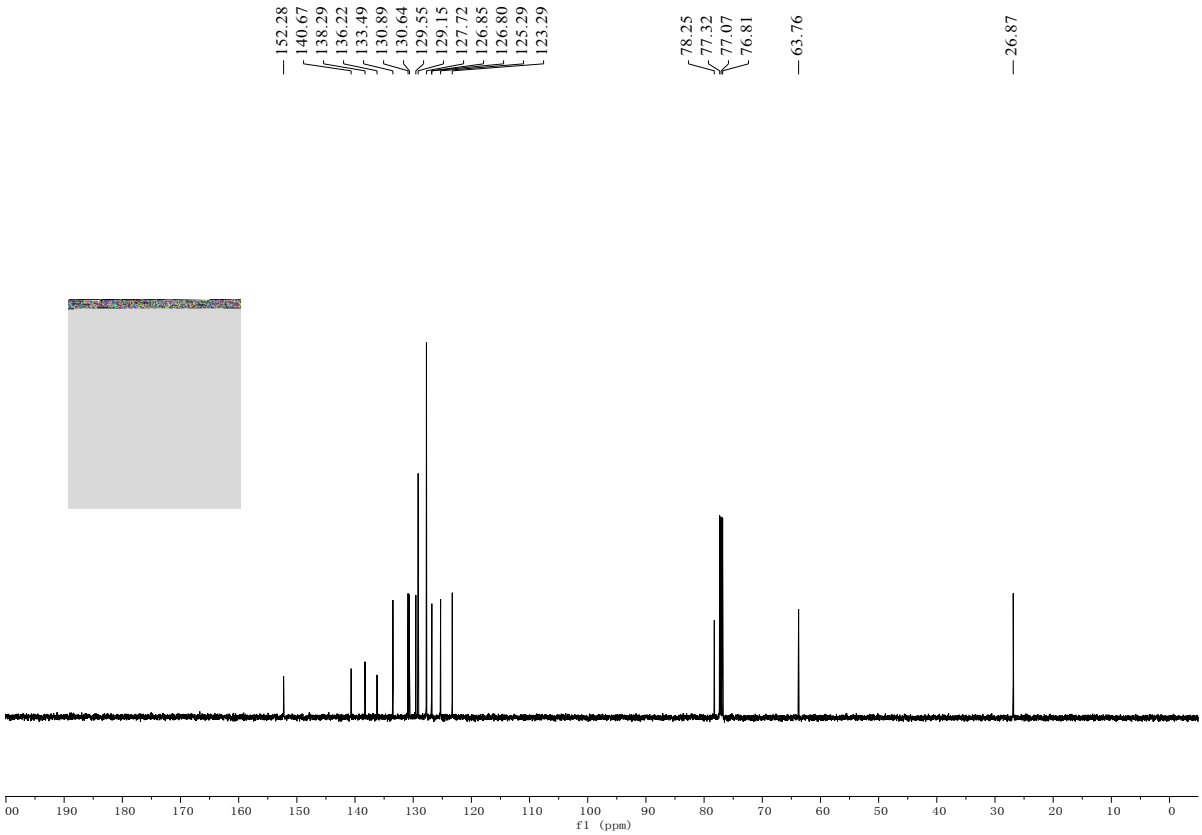
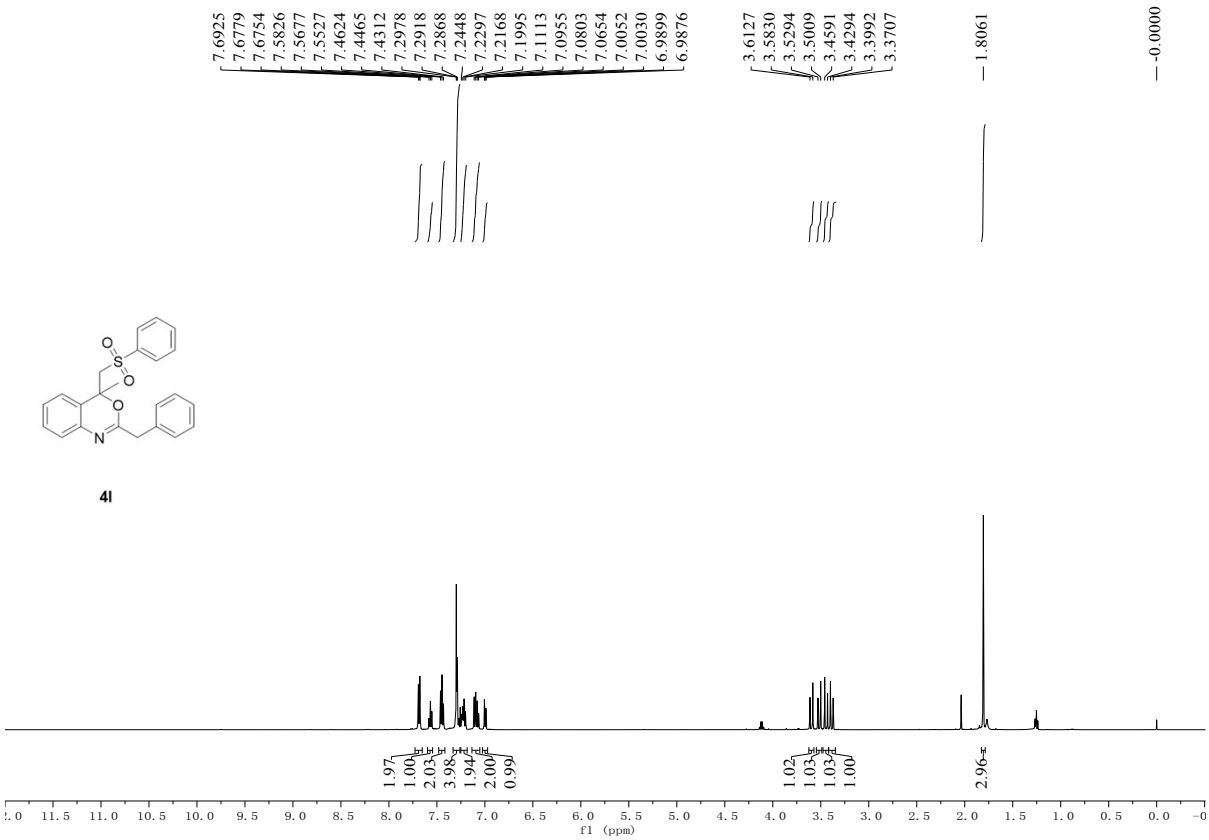
7.7073
7.6397
7.6374
7.6323
7.6300
7.4930
7.4780
7.4722
7.4627
7.3858
7.3698
7.3546
7.2865
7.2706
7.2535
7.2179
7.2018
7.1526
7.1373
7.1180
7.1028
7.0696
7.0621
7.0597
7.0522
3.8159
3.7863
3.6160
3.5864
2.0733
-0.0000
1.92
0.94
1.92
1.93
1.09
0.92
1.95
0.93
1.00
0.99
3.00
f1 (ppm)

4j


152.28
140.67
138.29
136.22
133.49
130.89
130.64
129.55
129.15
127.72
126.85
126.80
125.29
123.29
78.25
77.32
77.07
76.81
63.76
26.87
f1 (ppm)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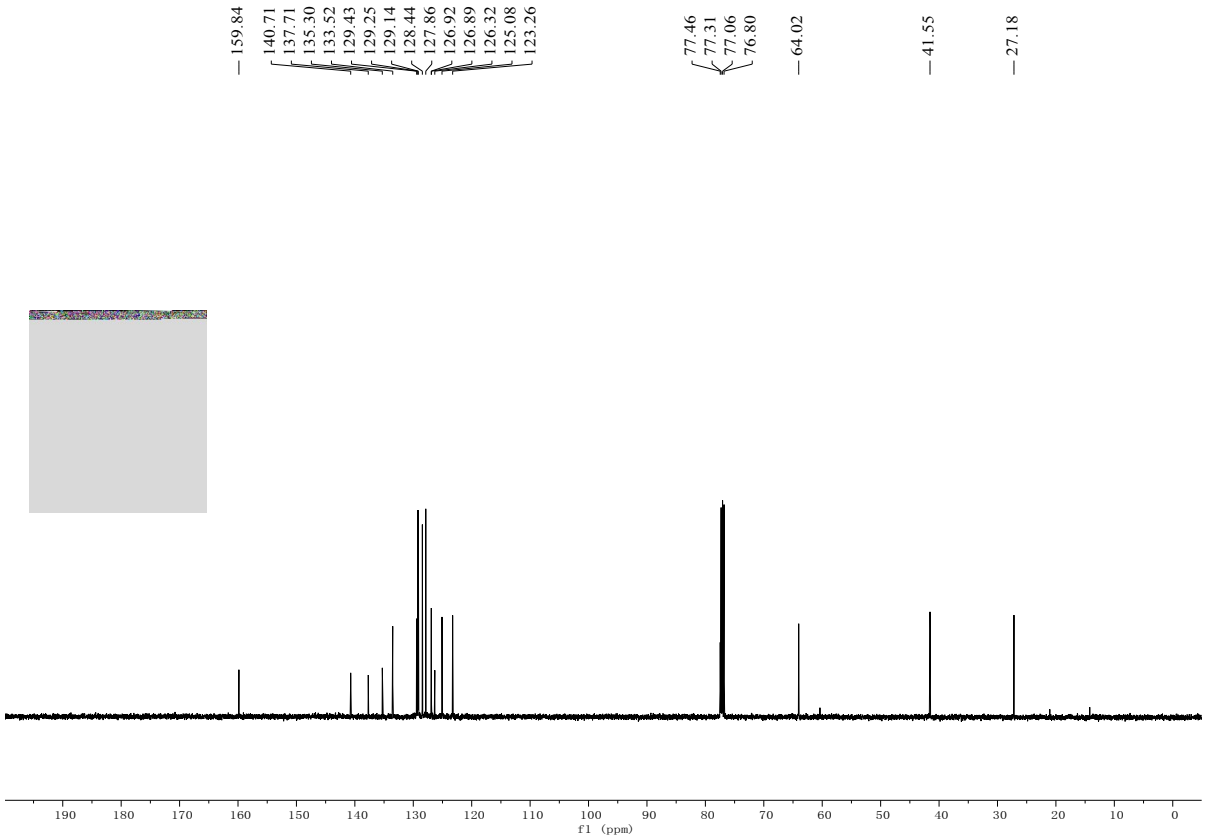
7.6754
7.5826
7.5677
7.5527
7.4624
7.4465
7.4312
7.2978
7.2918
7.2868
7.2448
7.2297
7.2168
7.1995
7.1113
7.0955
7.0803
7.0654
7.0052
7.0030
6.9899
6.9876
3.6127
3.5830
3.5294
3.5009
3.4591
3.4294
3.3992
3.3707
1.8061
-0.0000
O
S
O
O
N
4l
1.97
1.00
2.03
3.98
1.94
2.00
0.99
1.02
1.03
1.03
1.00
2.96
f1 (ppm)


159.84
140.71
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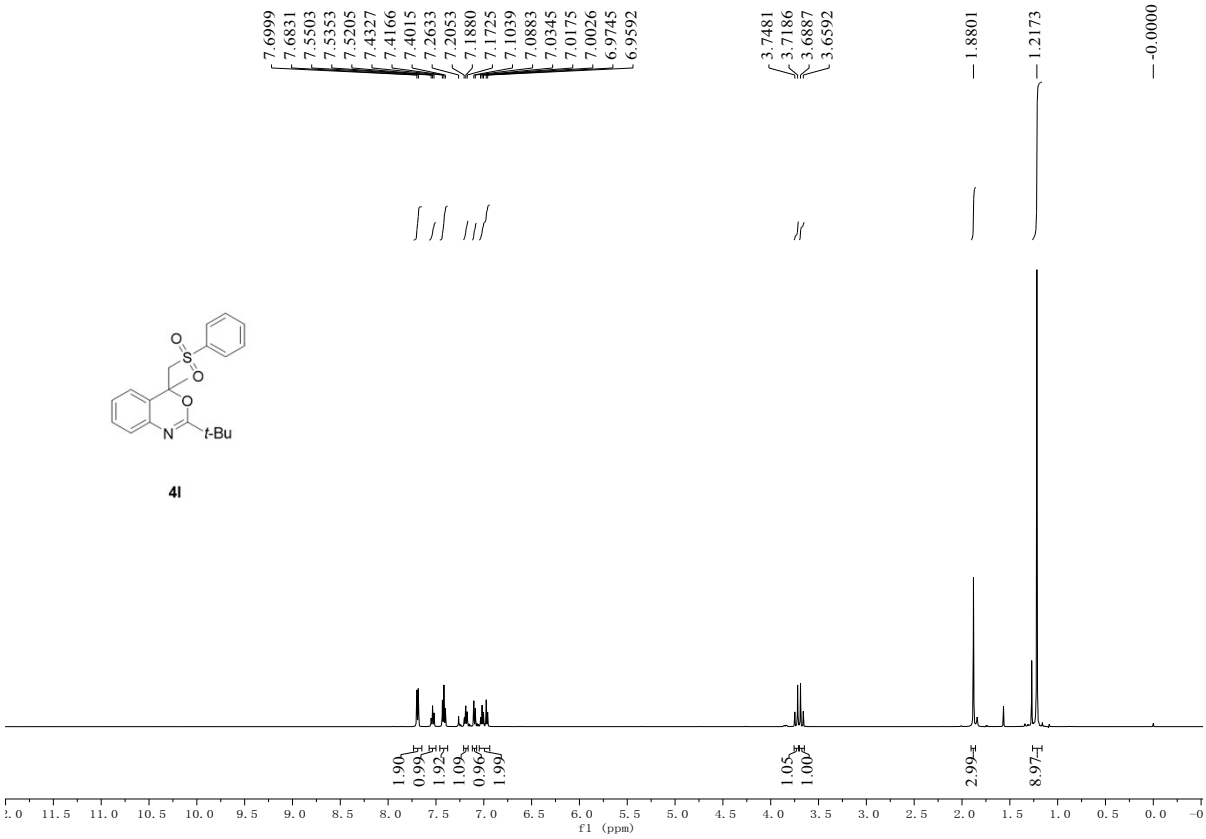
135.30
133.52
129.43
129.25
129.14
128.44
127.86
126.92
126.89
126.32
125.08
123.26
77.46
77.31
77.06
76.80
64.02
41.55
27.18
f1 (ppm)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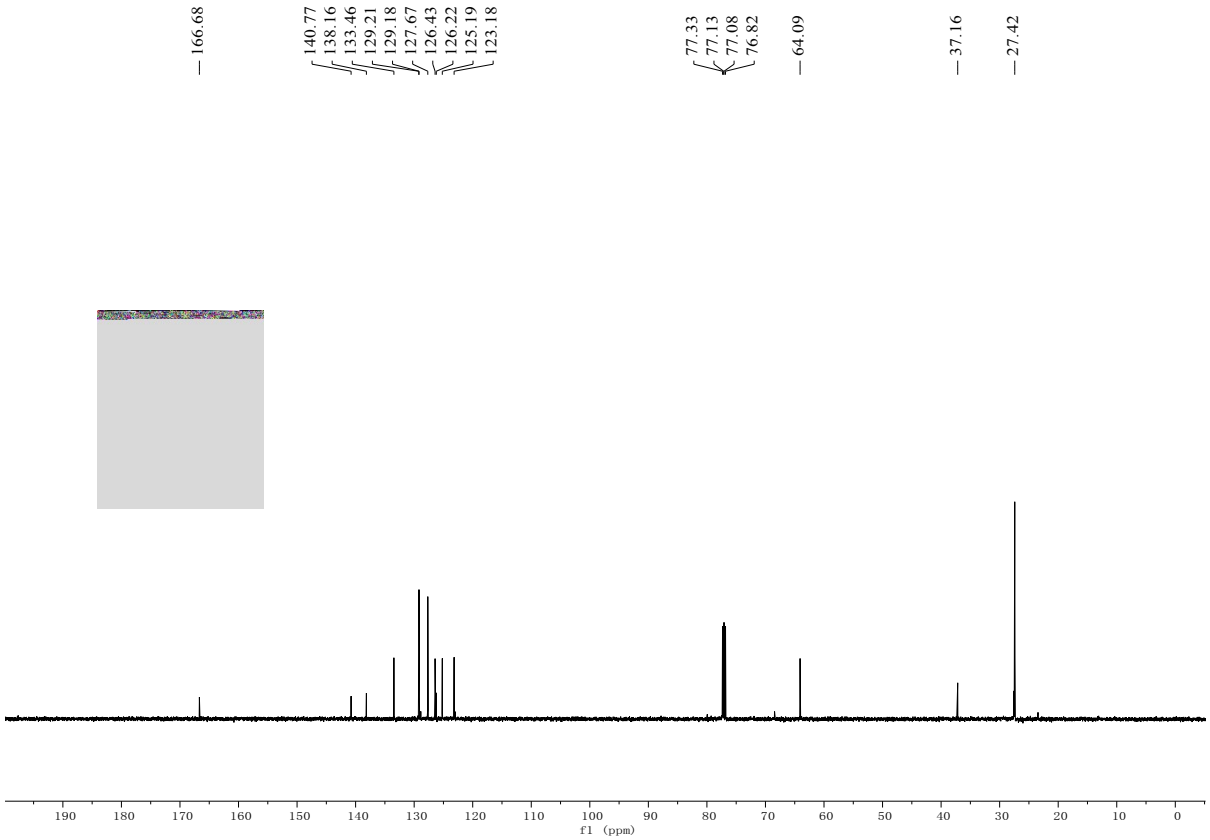
7.6831
7.5503
7.5353
7.5205
7.4327
7.4166
7.4015
7.2633
7.2053
7.1880
7.1725
7.1039
7.0883
7.0345
7.0175
7.0026
6.9745
6.9592
3.7481
3.7186
3.6887
3.6592
1.8801
1.2173
-0.0000
1.90
0.99
1.92
1.09
0.96
1.99
1.05
1.00
2.99
8.97
f1 (ppm)

O
S
O
O
N
t-Bu

4l


166.68
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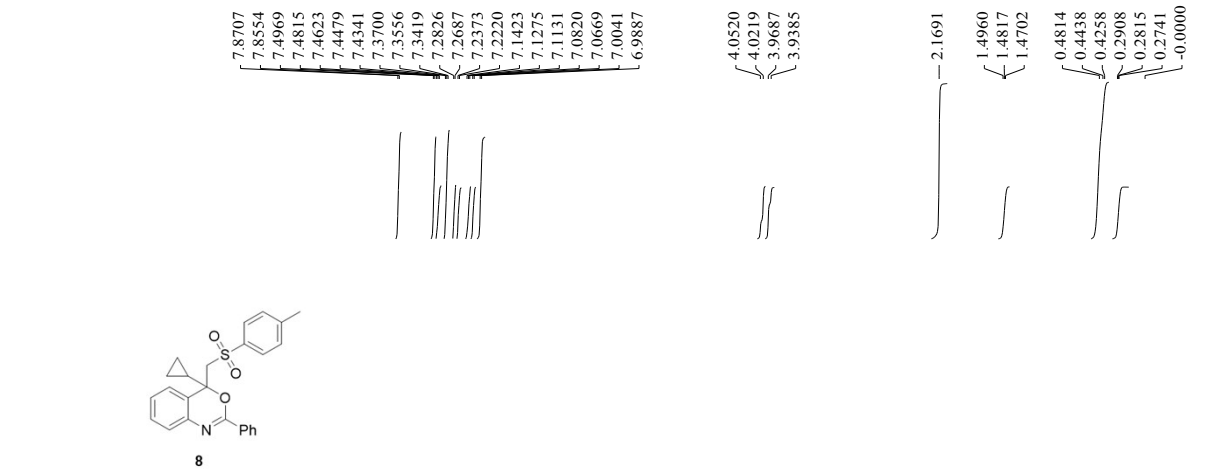
133.46
129.21
129.18
127.67
126.43
126.22
125.19
123.18
77.33
77.13
77.08
76.82
64.09
37.16
27.42
f1 (ppm)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7.3556
7.3419
7.2826
7.2687
7.2373
7.2220
7.1423
7.1275
7.1131
7.0820
7.0669
7.0041
6.9887
4.0520
4.0219
3.9687
3.9385
2.1691
1.4960
1.4817
1.4702
0.4814
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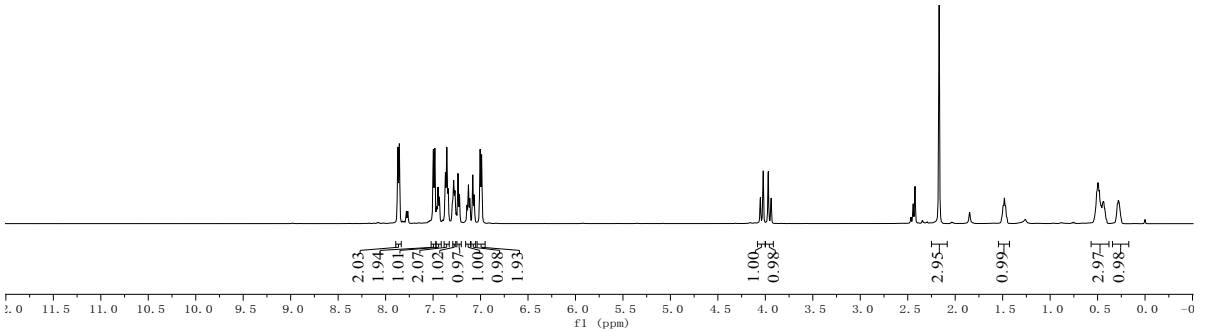
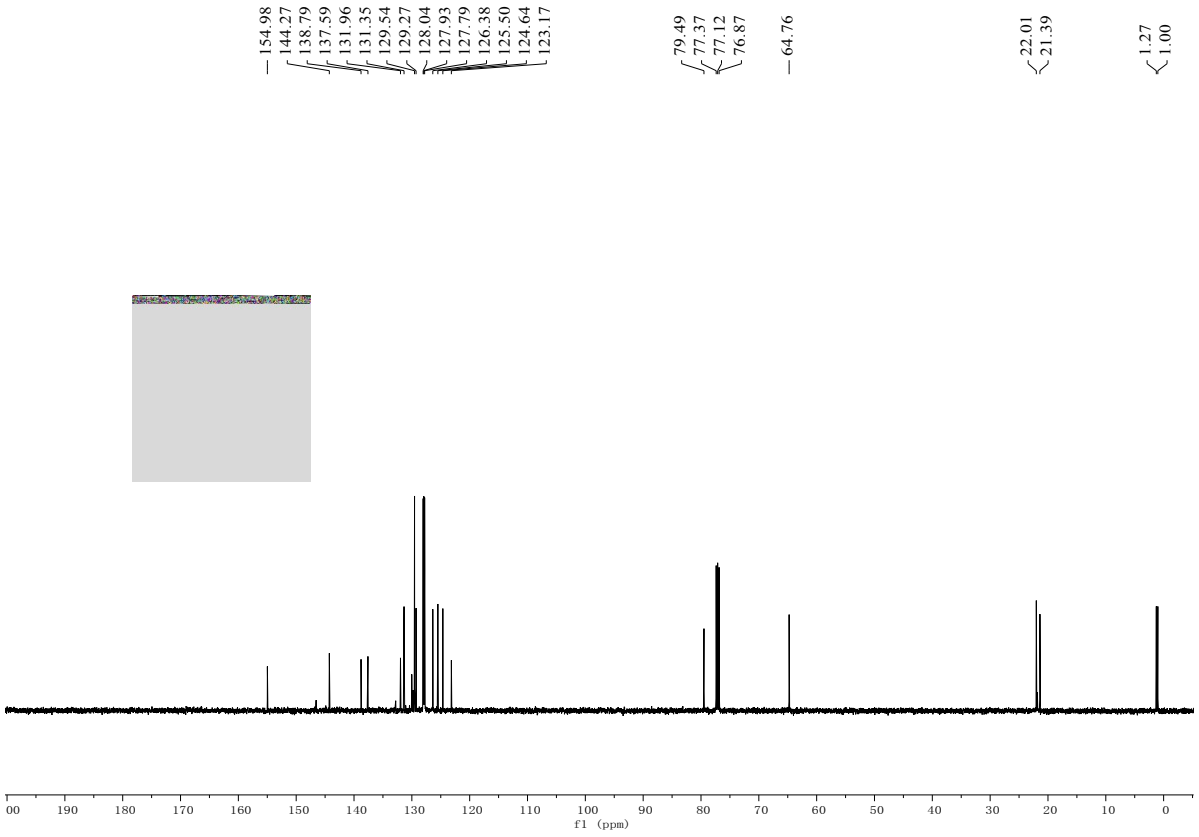
0.2908
0.2815
0.2741
-0.0000

O
S
O
O
N
Ph

8


2.03
1.94
1.01
2.07
1.02
0.97
1.00
0.98
1.93
1.00
0.98
2.95
0.99
2.97
0.98
f1 (ppm)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144.27
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137.59
131.96
131.35
129.54
129.27
128.04
127.93
127.79
126.38
125.50
124.64
123.17
79.49
77.37
77.12
76.87
64.76
22.01
21.39
1.27
1.00
f1 (ppm)