Supporting Information

Oxy-Sulfonylation of Terminal Alkynes via C-S Coupling Enabled by Copper Photoredox Catalysis

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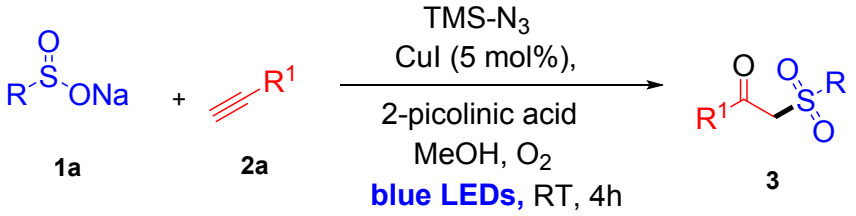
Experimental section

General: All reactions were conducted in oven-dried glasswares using a blue light-emitting diode (LED) array (24 LEDs, power density at the reaction site: 40 mW/cm^2 at 460 nm) as the visible-light source under an oxygen (O_2, 1 atm) atmosphere. All solvents were dried according to known methods, and distilled prior to use. Starting materials were commercially available (Sigma-Aldrich or Alfa-Aesar or TCI chemicals) and used as received. Aryl sodium sulfinate starting materials were prepared following literature method and used without purification.[s1a] ^{1}H NMR and ^{13}C NMR spectra were recorded at 400 and 600 MHz using deuterated $CDCl_3$ or $CDCl_3$-DMSO-d_6 mixture. Chemical shifts (δ) were reported as parts per million (ppm) and the following abbreviations were used to identify the multiplicities: s= singlet, d= doublet, t= triplet, q= quartet, m= multiplet, b= broad, and all combinations thereof can be explained by their integral parts. Unless otherwise specified, the proton/carbon signals of the above-mentioned solvent peaks (at δ 7.24 or 2.50 and δ 77.00 or 39.51 ppm, respectively) were used as the internal reference. EPR spectra were recorded using a Bruker ESP-300E instrument.

General procedure for the synthesis of β-keto sulfones

Scheme S1. General procedure for the synthesis of β-keto sulfones.



A dry test tube (20 mL) containing 5 mol% CuI, 0.5 mmol of substituted sodium sulfinate, and 20 mol% of 2-picolinic acid, dry MeOH (6 mL) was added *via* syringe and stirred for 2 minutes, and trimethylsilyl azide ($TMSN_3$) 1.1 equiv. were added and stirred again for 3 minutes. Finally, terminal acetylene (0.50 mmol) was added. The reaction mixture was then irradiated with blue LEDs (40 mW/cm^2 at 460 nm) under an oxygen atmosphere (1 atm.) at room temperature (25-28 ^{o}C) until completion of the reaction (monitored by TLC). The reaction mixture was diluted with 40 % ethyl acetate in hexane and stirred for 10 min. The mixture was filtered through celite and silica gel pads, and washed with ethyl acetate. The filtrate was concentrated and the residue was purified by column chromatography on silica gel to collect the β-keto sulfone products.

Optical pictures of the reaction mixture before and after irradiation under blue LEDs.

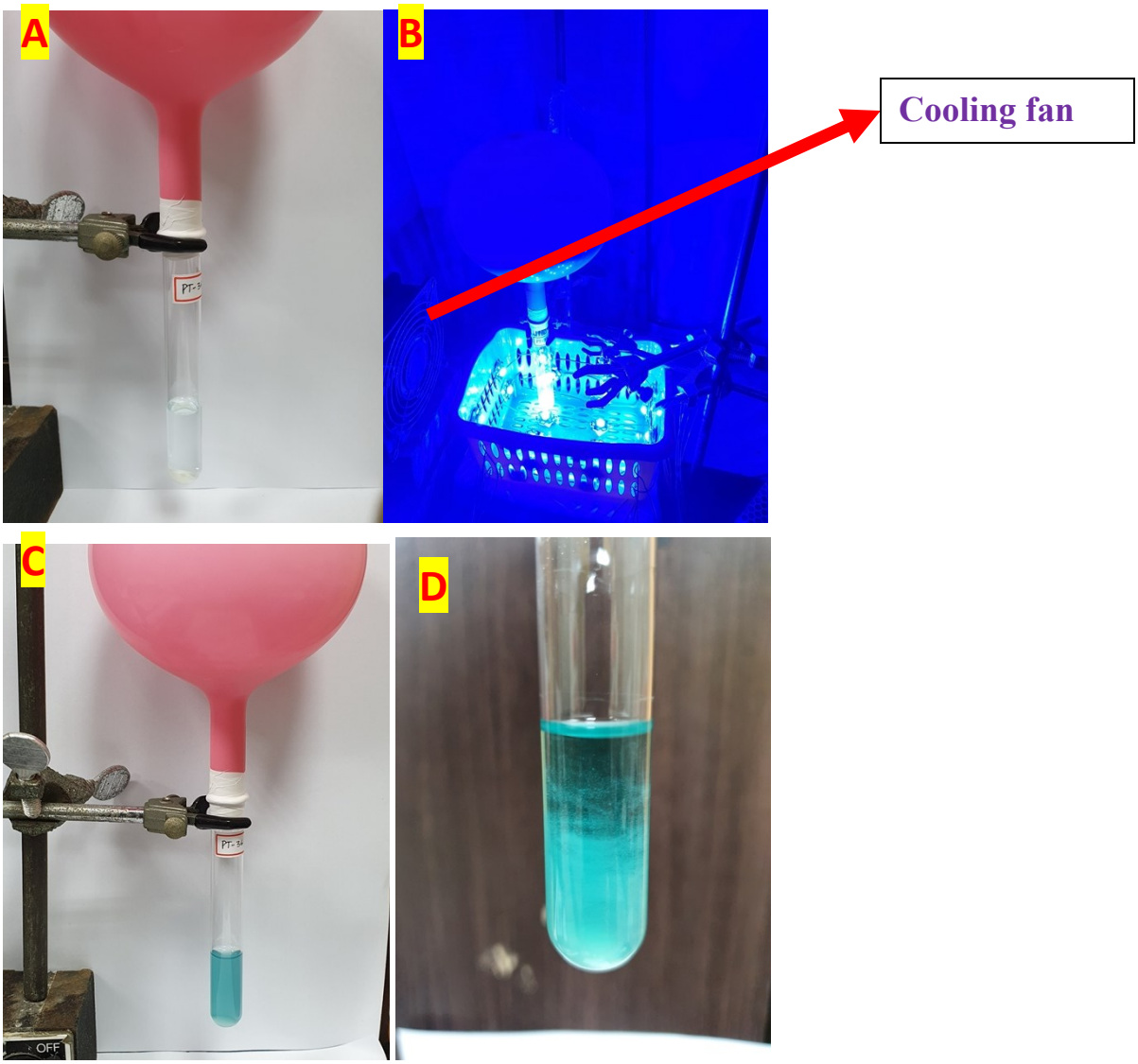


Figure S1: (**A**) Reaction mixture before irradiation. (**B**) Irradiation under blue LEDs, (**C**) & (**D**) Reaction mixture after irradiation showing formation of blue-colored $Cu^{II}(pic)_2$ complex.

Scheme S2. Comparison of various sulfonyl sources with current method to synthesize β-keto sulfones.

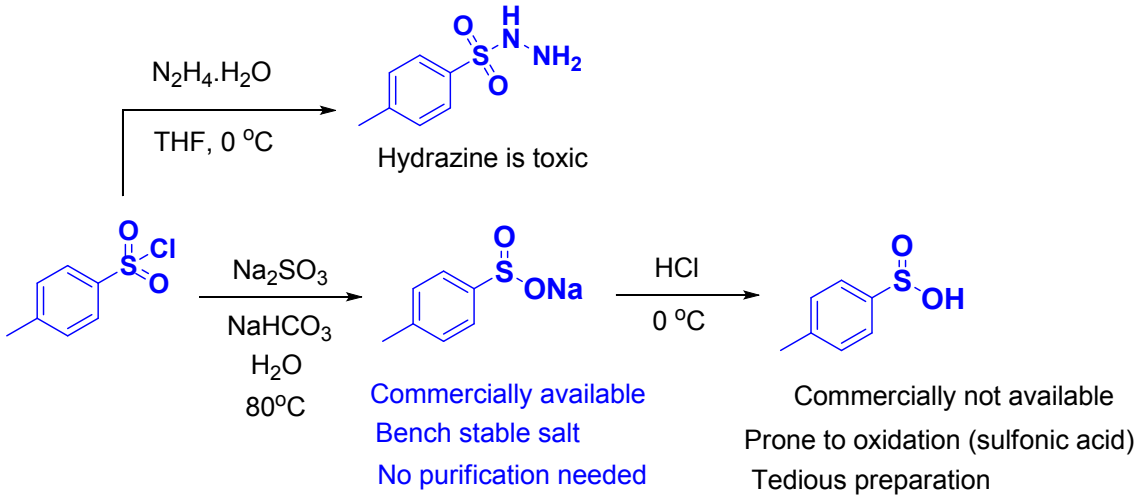


Table S1: Evaluation of Green chemistry metrics for the synthesis of **4j** (an 11 β-HSD1 inhibitor) using the current photoredox approach.

$$\textbf{Atom economy (\%) (AE)} = \frac{\text{Molecular mass of desired product}}{\text{Molecular mass of all reactants}} \times 100$$

$$\textbf{Reaction mass efficiency (\%) (RME)} = \frac{\text{Mass of desired product}}{\text{Mass of all reactants}} \times 100$$

Reactant 1	**Sodium 4-methylbenzene sulfinate**	**0.35g**	**2.0 mmol**	**FW 178.18**
Reactant 2	**1-ethynyl-4-(trifluoromethyl) benzene**	**0.34g**	**2.0 mmol**	**FW 170.13**
Reactant 3	**Trimethyl silyl azide**	**0.25g**	**2.2 mmol**	**FW 115.21**
Ligand	**2-picolinic acid**	**0.05g**	**0.41mmol**	**FW 123.11**
Solvent	**MeOH**	**15.84 g (20mL)**	**----**	**----**
Auxiliary	**----**	**----**	**----**	**----**
Recycled Solvent	**MeOH**	**8.95 g (11.3mL)**	**----**	**----**
Product	**2-tosyl-1-(4-(trifluoromethyl) phenyl)ethanone**	**0.431g**	**1.26 mmol**	**FW 342.33**
By-product	**Trimethylsilanol (TMSOH)**	**0.114g**	**1.26 mmol**	**FW 90.19**

Product yield = 63%

$$\textbf{E-factor} = \frac{0.35+0.34+0.05+0.25+15.84+0.114-0.431-8.95}{0.431\text{g}} = \textbf{17.55 Kg waste/1Kg product}$$

$$\textbf{Atom economy} = \frac{342}{479} \times 100 = \textbf{71.4\%}$$

$$\textbf{Atom efficiency} = \textbf{63\% x 71.4\% /100} = \textbf{45\%}$$

$$\textbf{Carbon efficiency} = \frac{16}{7+9+3} \times 100 = \textbf{84.2\%}$$

$$\textbf{Reaction mass efficiency} = \frac{0.431\text{g}}{0.35+0.34\text{g}} \times 100 = \textbf{62.5\%}$$

Note: The amount of TMSOH (0.114 g) was (theoretically) calculated based on the amount of product.

E-factor of current photoredox method is 17.55 kg waste per kg product, with 71.4% atom economy, 45% atom efficiency, 62.5% reaction mass efficiency and 84.2% carbon efficiency.

Table S2: Evaluation of Green chemistry metrics for the synthesis of **4j** using a literature reported thermal process[S1b].

$$\textbf{Atom economy (\%) (AE)} = \frac{\text{Molecular mass of desired product}}{\text{Molecular mass of all reactants}} \times 100$$

$$\textbf{Reaction mass efficiency (\%) (RME)} = \frac{\text{Mass of desired product}}{\text{Mass of all reactants}} \times 100$$

Reactant 1	**Sodium 4-methylbenzene sulfinate**	**0.35g**	**2.0 mmol**	**FW 178.18**
Reactant 2	**2-bromo-1-(4-(trifluoromethyl) phenyl)ethanone**	**0.534g**	**2.0 mmol**	**FW 267.04**
Solvent	**DMF**	**14.16 g (15mL)**	**----**	**----**
Auxiliary	**----**	**----**	**----**	**----**
Product	**2-tosyl-1-(4-(trifluoromethyl) phenyl)ethanone**	**0.466g**	**1.36 mmol**	**FW 342.33**

Product yield = 68%

$$\textbf{E-factor} = \frac{\textbf{0.35+ 0.534+14.16 - 0.466}}{\textbf{0.466g}} = \textbf{31.28Kg waste/ 1 Kg product}$$

$$\textbf{Atom economy} = \frac{\textbf{342}}{\textbf{445}} \times \textbf{100} = \textbf{76.8\%}$$

$$\textbf{Atom efficiency} = \textbf{68\% x 76.8\% /100} = \textbf{52.2\%}$$

$$\textbf{Carbon efficiency} = \frac{\textbf{16}}{\textbf{16}} \times \textbf{100} = \textbf{100\%}$$

$$\textbf{Reaction mass efficiency} = \frac{\textbf{0.466g}}{\textbf{0.35+0.534g}} \times \textbf{100} = \textbf{52.9\%}$$

E-factor of literature method is 31.28 kg waste per kg product, with 76.8% atom economy, 52.2% atom efficiency, 52.9% reaction mass efficiency and 100% carbon efficiency.

Table S3. Comparison of photochemical and thermal method[s1b] green chemistry metrics evaluation for the synthesis of **4j** (11 β-HSD1 inhibitor)

Parameters / **Method**	**E-factor (Kg waste/1Kg product)**	**Atom economy (%)**	**Atom efficiency (%)**	**Carbon efficiency (%)**	**Reaction mass efficiency (RME) (%)**
Photochemical	**17.55**	**71.4**	**45**	**84.2**	**62.5**
Thermal	**31.28**	**76.8**	**52.2**	**100**	**52.9**

The **E-factor** is a crucial and highly important parameter as it is the **physical parameter** in the **green chemistry analysis**. And, an overall comparison shows that the current photoredox method is **~1.8 times** better than (in terms of E-factor) previous literature method[S1b]. Besides, the current method shows higher (%) values for reaction mass efficiency (RME), and more or less same values for atom economy, and atom efficiency compared to the reported thermal method.

Table S4. EcoScale evaluation

EcoScale = 100 - Sum of individual penalties

Score on EcoScale: >75, Excellent; >50, Acceptable; <50, Inadequate

A) Calculation of penalty points:

Parameters		Penalty points
1. Yield	(100-%yield)/2 = (100-63)/2 = 14	18.5
2. Price of reaction components (To obtain 10 mmol of end product)		
a. Sodium 4-methylbenzene Sulfinate	=5.43g = $9.8	
b. 1-ethynyl-4-(trifluoromethyl)benzene	=5.2g= $139.8	
c. CuI (5.0mol%)	= 0.29g = $0.2	
d. Picolinic acid (20mol%)	= 0.75g = $0.42	
e. $TMSN_3$	= 3.87g = $44.0	
f. MeOH	= 40mL = $0.73	
Total price (USD) = $194.9		
Thus, expensive (>$50)		5
3. Safety MeOH Solvent		
Highly flammable (F)		5
4. Technical Setup		
Inconventional activation technique (Photochemical activation)		2
5. Temperature and time		
Room temperature and 24h		1
6. Workup and purification		
Removal of solvent with bp <150^{o}C		0
Classical Chromatography		10
Total Penalty Points		41.5

B) EcoScale calculation:

EcoScale = 100- 41.5 = 58.5 (an acceptable synthesis)

Preparation of copper(I) phenylacetylide:[S2] CuI (1.0 g, 5.0 mmol) was dissolved in ammonium hydroxide to form a blue solution. While stirring, phenylacetylene (0.5 g, 5.1 mmol in 50 mL ethanol) was added dropwise to the solution. The system was allowed to stand for 15 min to form a yellow precipitate suspension. The precipitate was filtered out and washed with water, ethanol, and diethyl ether, three times each. The solid was vacuum-dried, and a bright yellow solid was obtained. The spectroscopic data for the yellow solid are shown below: FT-IR (KBr, cm^{-1}) [S3] 1929 (C≡C), 1596, 1568; UV-Vis: λ_{abs}= 476 nm.

EPR measurements: EPR spectra were recorded at room temperature on a Bruker ESP-300E (X band, 9.8 GHz) with parameters setting as shown below: receiver gain= 30 n; receiver phase= 0 deg; receiver harmonic= 1; field modulation frequency=100000 Hz; microwave frequency [Hz]= 9.660469 e^{+09}; field modulation amplitude [T]= 0.00016; receiver time constant [S] = 0.32768; microwave power= 0.015 W; receiver offset [%FS]= 0; DMPO (5-,5-dimethyl-1-pyrroline N-oxide) was employed as a radical trap for the superoxide radical anion.

The reaction under standard condition (**1a**, **2a**, CuI, 2-picolinic acid and $TMSN_3$ under O_2) in CH_3OH solvent was irradiated with blue LED light for 30 min in the presence of DMPO in an EPR chamber while recording the EPR spectra. The EPR signals shown in Figure S2 is corresponding to DMPO-OO(H). Which shows that superoxide anion radical was formed in the reaction solution. No superoxide EPR signal was observed from the reaction solution under the standard condition in the absence of CuI (Figure S3). The EPR signals shown in Figure S2 is corresponding to DMPO-OO(H)

EPR spectra of reaction mixture with O_2 under blue-LEDs

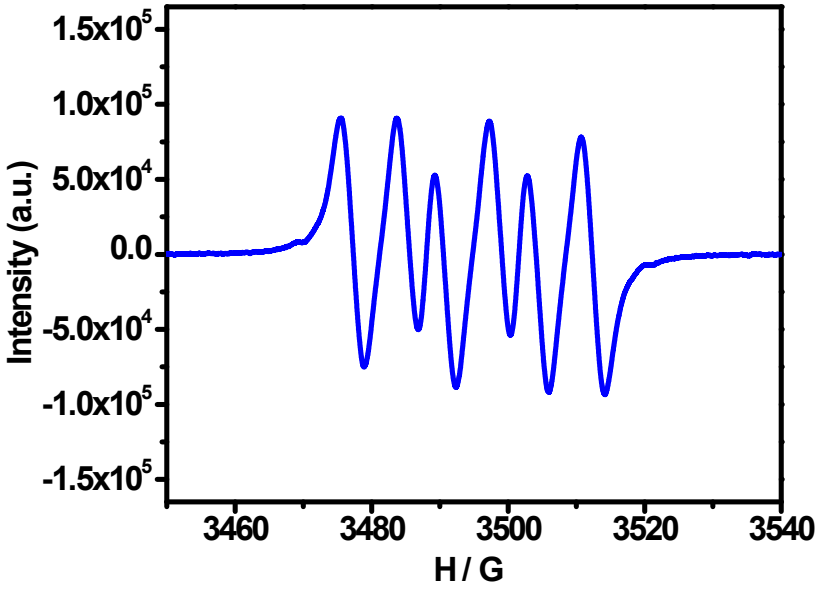


Figure S2: EPR spectra of the reaction mixture: phenylacetylene (**2a) (**0.5 mmol**)**, sodium p-toluene sulfinate (**1a**) (0.5 mmol), CuI (5mol%), 2-picolinic acid (20 mol%) and $TMSN_3$ (1.1 equiv.) in CH_3OH (6 mL), 0.5 mL of this reaction solution was taken out into a small vial, followed by the addition of 0.01 mL of DMPO (5 x 10^{-2} M). The mixture was irradiated with blue LEDs at room temperature under an oxygen atmosphere for 30 minutes. The reaction mixture was then analysed by EPR. There are 6 classical peaks, which are corresponding to the signals (DMPO-OO(H)).

EPR spectra of the reaction mixture without CuI

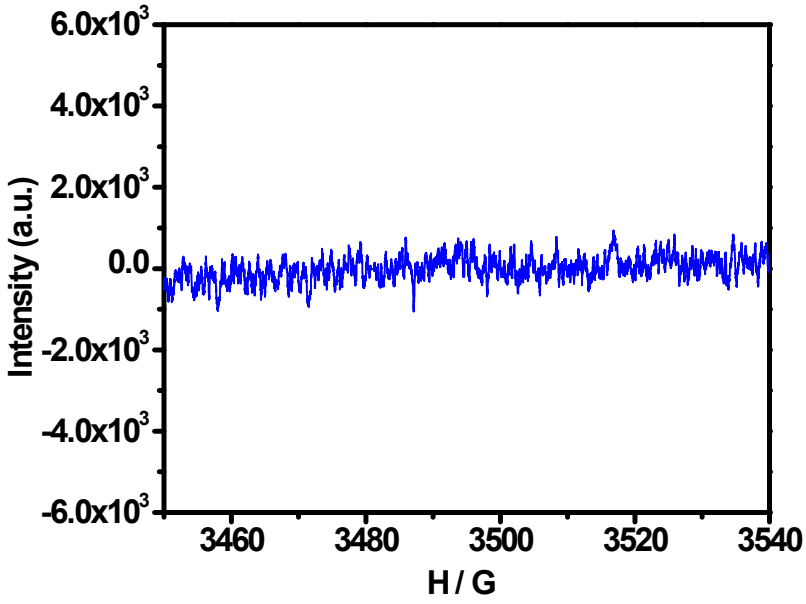


Figure S3: EPR spectra of the reaction mixture: phenylacetylene (**2a**) **(**0.5 mmol**)**, sodium p-toluene sulfinate (**1a**) (0.5 mmol), 2-picolinic acid (20 mol%) and $TMSN_3$ (1.1 equiv.) in CH_3OH (6 mL), 0.5 mL of this reaction solution was taken out into a small vial, followed by the addition of 0.01 mL of DMPO (5 x 10^{-2} M). The mixture was irradiated with blue LEDs at room temperature under an oxygen atmosphere (1 atm.) for 30 minutes (in the absence of CuI). The reaction mixture was then analysed by EPR. No signals were detected.

EPR spectra of the reaction mixture in the absence of DMPO

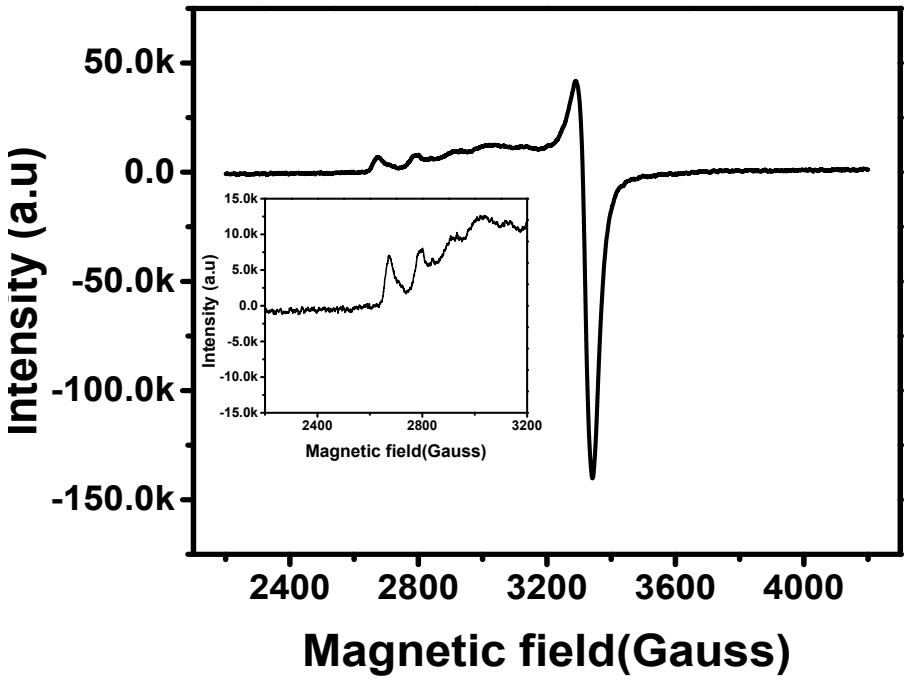


Figure S4: EPR spectra of the reaction mixture: phenylacetylene (**2a) (**0.5 mmol**)**, sodium p-toluene sulfinate (**1a**) (0.5 mmol), CuI (5mol%), 2-picolinic acid (20 mol%) and $TMSN_3$ (1.1 equiv.) in CH_3OH (6 mL). The mixture was irradiated with blue LEDs at room temperature under an oxygen atmosphere for 30 minutes. The reaction mixture was then analysed by EPR at 77 K. The observed signals are corresponding to the signals of copper (II) superoxo complex[S4].

Excitation and emission spectra of copper(I)-phenylacetylide:[S3]

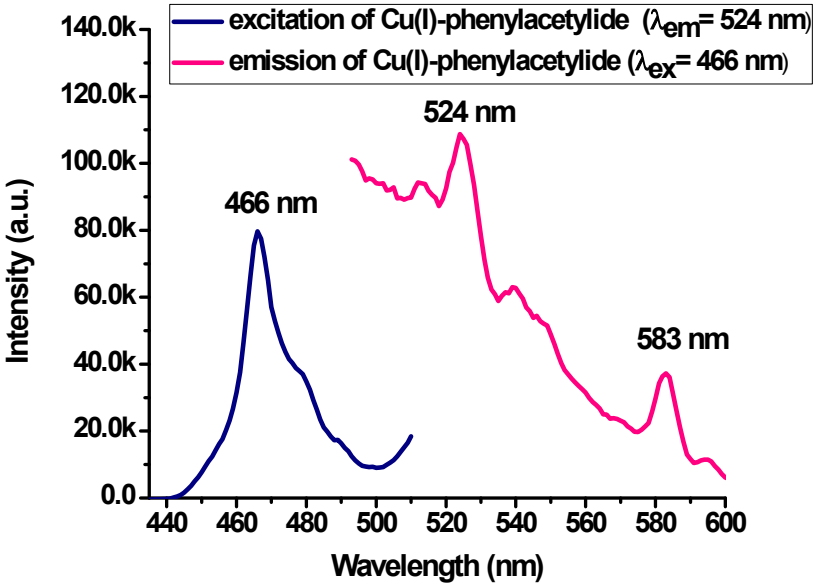
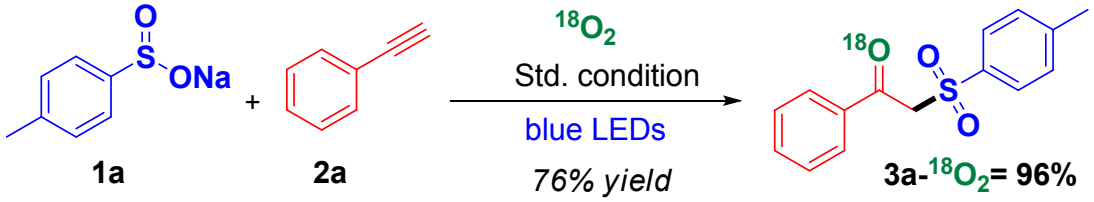


Figure S5: Excitation and emission spectra of in-situ generated copper(I)-phenylacetylide in CH_3CN solvent.

$^{18}O_2$ labeling experiment

We have performed an $^{18}O_2$-labeling experiment using anhydrous solvents under the standard condition (97% purity of $^{18}O_2$ gas, instead of $^{16}O_2$ air, was filled in the reaction system) for **1a** and **2a** in Scheme S5 under standard condition. From ESI mass, the final product **3a** was determined to contain an $^{18}O_2$ labeled β-keto sulfone in 96% exclusively, indicating that the oxygen atom in the **3a** originated from molecular O_2.

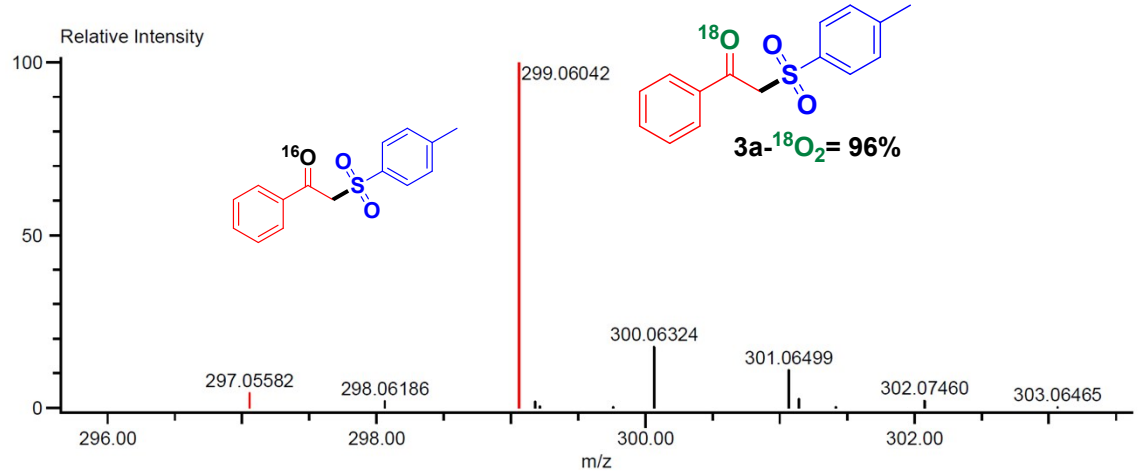
Scheme S3. $^{18}O_2$ isotope labelling experiments.



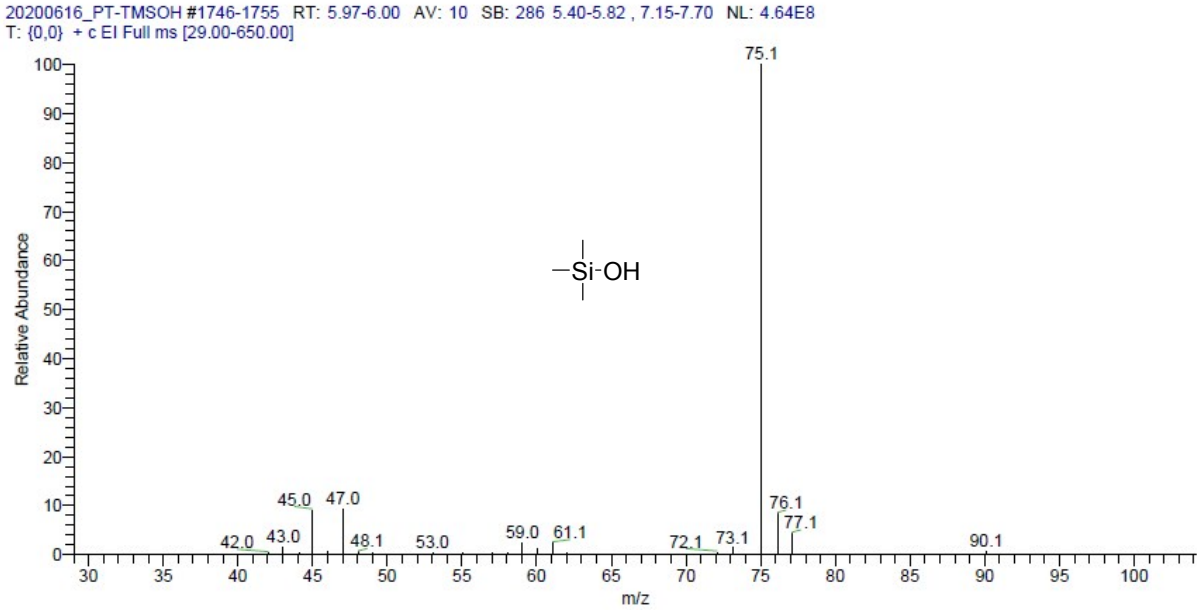
Data:PT-O18- C
Comment:
Description:
Ionization Mode:ESI+
History:Average(MS[1] 0.58..0.78)

Acquired:5/12/2020 1:51:06 PM
Operator:AccuTOF
m/z Calibration File:20200102-1TFANa...
Created:5/12/2020 2:08:56 PM
Created by:AccuTOF

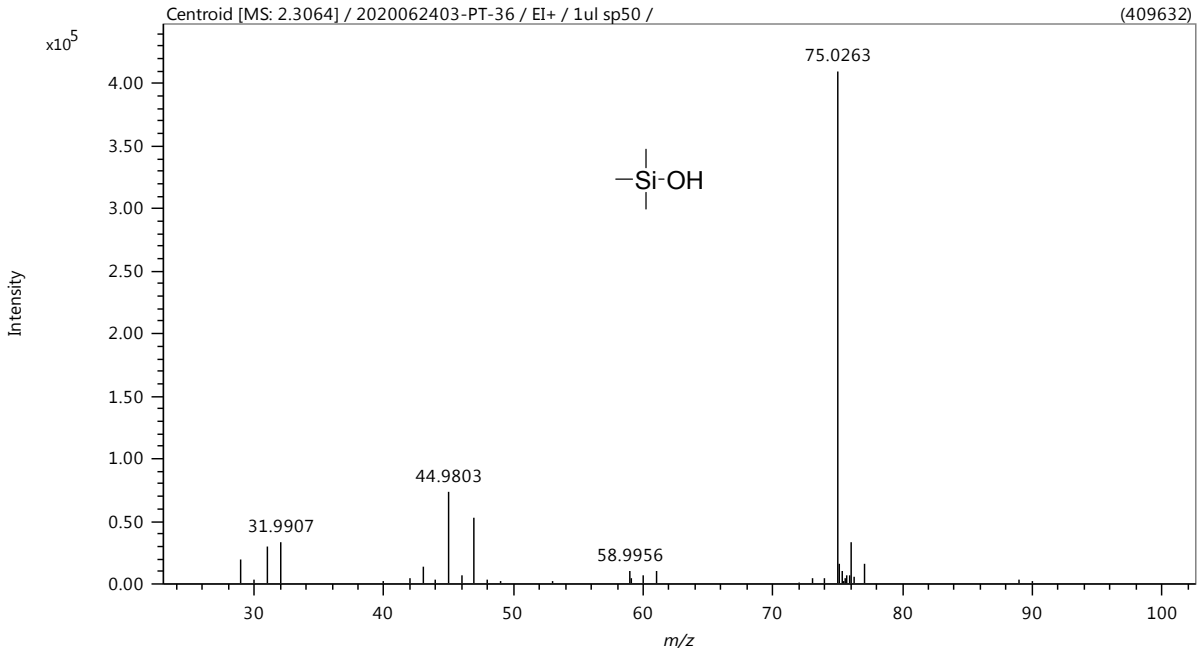
Charge number:1 Tolerance:250.00[ppm], 200.00 .. 200.... Unsaturation Number:-150.0 .. 200.0 (...
Element:^{12}C:15 .. 15, ^{1}H:14 .. 15, ^{23}Na:0 .. 1, ^{16}O:2 .. 3, ^{18}O:0 .. 3, ^{32}S:1 .. 1



Mass	Intensity	Calc. Mass	Mass Difference [mDa]	Mass Difference [ppm]	Possible Formula
297.05582	1400.83	297.05613	-0.31	-1.05	$^{12}C_{15}{}^{1}H_{14}{}^{23}Na_1{}^{16}O_3{}^{32}S_1$
299.06042	33841.54	299.06038	0.04	0.13	$^{12}C_{15}{}^{1}H_{14}{}^{23}Na_1{}^{16}O_2{}^{18}O_1{}^{32}S_1$



GC-MS of reaction mixture TMS-OH (detected): M - CH_3 peak



Supplementary references:

S1. (a) M-L. Feng, L-Y. Xi, S-Y. Chen, and X-Q. Yu, *Eur. J. Org. Chem.,* **2017**, 2746. (b)J. Xiang, M. Ipek, V. Suri, M. Tam, Y. Xing, N. Huang, Y. Zhang, J. Tobin, T. S. Mansoura and J. McKew, Bioorg. Med. Chem., **2007**, *15*, 4396.

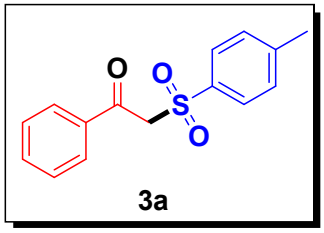
S2. (a) A. Sagadevan, A. Ragupathi, C.-C. Lin, J. R. Hwu and K. C. Hwang, *Green Chem.,* **2015**, *17*, 1113. (b) A. Sagadevan, A. Ragupathi, K. C. Hwang, *Angew. Chem., Int. Ed.* **2015**, *54*, 13896-13901.

S3. W. Shi, Y. Luo, X. Luo, L. Chao, H. Zhang, J. Wang, A. Lei, *J. Am. Chem. Soc.*, **2008**, *130*, 14713-14720.

S4. (a) E. I. Solomon, P. Chen, M. Metz, S. K. Lee, and A. E. Palmer, *Angew. Chem. Int. Ed.*, 2001, 40, 4570; (b) B. Kim, D. Jeong, T. Ohta, and J. Cho, *Commun. Chem.,* 2019, 81, 2.

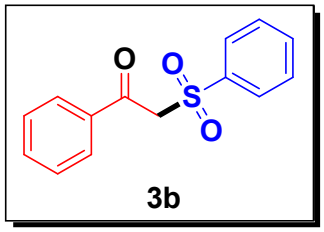
Spectroscopic Data

1-phenyl-2-tosylethanone (3a)



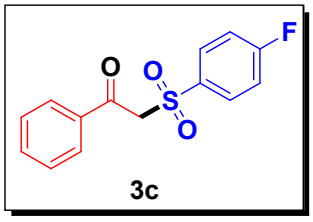
White solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.0 Hz, 2 H), 7.74 (d, *J* = 8.6 Hz, 2 H), 7.60 (t, *J* = 6.7 Hz, 1 H), 7.46 (t, *J*= 7.2 Hz, 2 H), 7.31 (d, *J* = 8.0 Hz, 2 H), 4.69 (s, 2 H), 2.41 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.1, 145.3, 135.7, 134.2, 129.8, 129.2, 128.8, 128.5, 63.5 and 21.6; HRMS: calcd for $C_{15}H_{14}NaO_3S$ (M+Na): 297.0561, found: 297.0565.

1-phenyl-2-(phenylsulfonyl)ethanone (3b)



White solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.91 (dd, *J* = 8.4 Hz, 1.2 Hz, 2 H), 7.88 (dd, *J* = 8.3 Hz, 1.0 Hz, 2 H), 7.64-7.62 (m, 1 H), 7.60-7.57 (m, 1 H), 7.53-7.50 (m, 2 H), 7.46-7.43 (m, 2 H), 4.72 (s, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.9, 138.7, 135.6, 134.3, 134.2, 129.2, 129.1, 128.8, 128.7, 128.5 and 63.3; HRMS: calcd for $C_{14}H_{12}NaO_3S$ (M+Na): 283.0405, found: 283.0404.

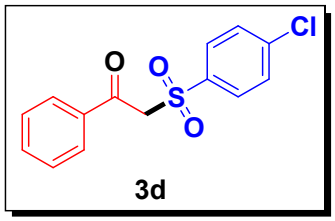
2-(4-fluorophenylsulfonyl)-1-phenylethanone (3c)



White solid; **^{1}H NMR** (600 MHz, $CDCl_3$): **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.91-7.88 (m, 4 H), 7.62-7.59 (m, 1 H), 7.47 (t, *J* = 7.8 Hz, 2 H), 7.19 (t, *J* = 8.4 Hz, 2 H), 4.72 (s, 2 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.9, 166.9 (d, $J_{C\text{-}F}$= 270 Hz), 135.5, 134.6, 134.5, 133.6, 131.6 (d, $J_{C\text{-}F}$=

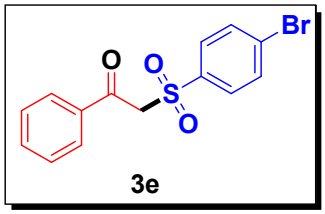
9 Hz), 130.1, 129.2, 128.9, 128.4, 116.6 (d, $J_{C\text{-}F}$= 24 Hz), and 63.3; ^{19}F NMR (563 MHz, CCl_3F): δ -101.8 (s), HRMS: calcd for $C_{14}H_{11}FNaO_3S$ (M+Na): 301.0311, found: 301.0307.

2-(4-chlorophenylsulfonyl)-1-phenylethanone (3d)



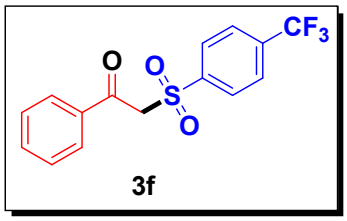
Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.91 (dd, *J* = 8.5 Hz, 1.2 Hz, 2 H), 7.81-7.79 (m, 2 H), 7.62-7.59 (m, 1 H), 7.50-745 (m, 4 H), 4.72 (s, 2 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.8, 141.0, 137.0, 135.5, 134.4, 130.1, 1297, 129.4, 129.1, 128.9, 128.8, and 63.2; HRMS: calcd for $C_{14}H_{11}ClNaO_3S$ (M+Na): 317.0015, found: 317.0013.

2-(4-bromophenylsulfonyl)-1-phenylethanone (3e)



Colorless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.90 (d, *J* = 7.7 Hz, 2 H), 7.73 (d, *J* = 8.2 Hz, 2 H), 7.64 (d, *J* = 8.2 Hz, 2 H), 7.60 (t, *J*= 7.4 Hz, 1 H), 7.46 (t, *J* = 7.4 Hz, 2 H), 4.72 (s, 2 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.6, 137.5, 135.4, 134.4, 132.4, 130.1, 129.6, 129.1, 128.8, and 63.1; HRMS: calcd for $C_{14}H_{11}BrNaO_3S$ (M+Na) : 360.9510, found: 360.9511.

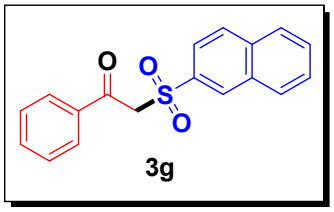
1-phenyl-2-(4-(trifluoromethyl)phenylsulfonyl)ethanone (3f)



Yellow solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.03 (d, *J* = 8.3 Hz, 2 H), 7.90 (dd, *J*= 8.0 Hz, 1.0 Hz, 2 H), 7.80 (d, *J* = 8.3 Hz, 2 H), 7.61 (t, *J*= 8.1 Hz, 1 H), 7.48 (t, *J*= 7.5 Hz, 2 H), 4.76 (s, 2 H); **^{13}CNMR** (150 MHz, $CDCl_3$): δ 187.7, 142.0, 135.6 (q, $J_{C\text{-}F}$= 33 Hz, 33 Hz), 134.6, 130.4 (t, $J_{C\text{-}F}$= 33 Hz), 129.3 (t, $J_{C\text{-}F}$= 31.5 Hz), 127.8, 127.6, 126.3, 126.2 (q, $J_{C\text{-}F}$= 97.5 Hz, 256.5 Hz),

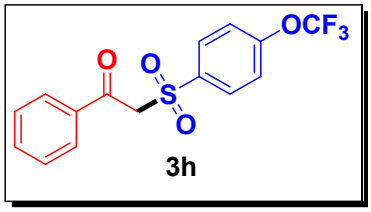
120.2, 105.3, and 63.0; ^{19}F NMR (563 MHz, CCl_3F): δ -62.7 (s), HRMS: calcd for $C_{15}H_{11}F_3O_3S$: 351.0279 (M+Na), found:351.0278.

2-(naphthalen-2-ylsulfonyl)-1-phenylethanone (3g)



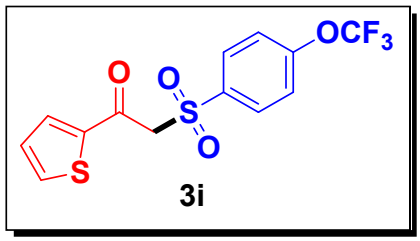
Brown solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.43 (s, 1 H), 7.97-7.89 (m, 5 H), 7.85 (dd, *J*= 8.5 Hz, 2.0 Hz, 1 H), 7.67-7.64 (m, 1 H), 7.61-7.55 (m, 2 H), 7.43 (t, *J*= 8.4 Hz, 2 H), 4.79 (s, 2 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.9, 135.7, 135.6, 135.5, 134.3, 131.9, 130.6, 129.5, 129.5, 129.4, 129.8, 129.8, 127.9, 127.7, 122.9 and 63.5; HRMS: calcd for $C_{18}H_{14}NaO_3S$ (M+Na): 333.0561, found: 333.0563.

1-phenyl-2-(4-(trifluoromethoxy)phenylsulfonyl)ethanone (3h)



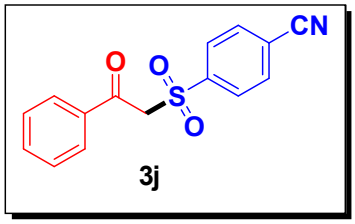
colorless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.95 (d, *J*= 9.0 Hz, 2 H), 7.90 (d, *J*= 7.4 Hz, 2 H), 7.62 (t, *J*= 7.7 Hz, 1 H), 7.46 (t, *J* = 8.4 Hz, 2 H), 7.34 (d, *J*= 8.4 Hz, 2 H), 4.74 (s, 2 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.8, 153.3, 136.7, 135.5, 134.5, 131.0, 129.1, 129.0, 128.9, 121.0 (t, $J_{C\text{-}F}$= 259.5 Hz), 120.7, and 63.2; ^{19}F NMR (563 MHz, CCl_3F): δ -57.0 (s); HRMS: calcd for $C_{15}H_{11}F_3NaO_4S$ (M+Na): 367.0227, found: 367.0228.

1-(thiophen-2-yl)-2-(4-(trifluoromethoxy)phenylsulfonyl)ethanone (3i)



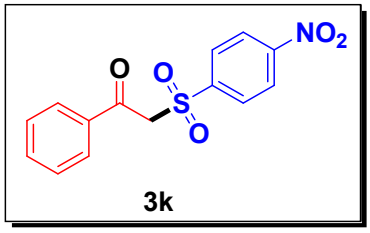
Colorless viscous liquid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.94 (d, *J*= 8.6 Hz, 2 H), 7.77-7.74 (m, 2 H), 7.34 (d, *J*= 8.9 Hz, 2 H), 7.14 (t, *J* = 4.8 Hz, 1 H), 4.63 (s, 2 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 180.0, 153.4, 142.9, 136.7, 136.5, 135.1, 131.0, 128.7, 122.7, 120.9 (t, $J_{C\text{-}F}$= 259.5 Hz), 120.7 and 64.2; ^{19}F NMR (563 MHz, CCl_3F): δ -57.0 (s) HRMS: calcd for $C_{13}H_9F_3NaO_4S_2$ (M+Na): 372.9792, found: 372.9794.

4-(2-oxo-2-phenylethylsulfonyl)benzonitrile (3j)



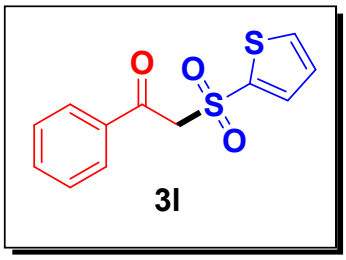
Light yellow solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.02 (d, *J* = 9.0 Hz, 2 H), 7.89 (dd, *J*= 8.2 Hz, 1.3 Hz, 2 H), 7.83 (d, *J* = 8.5 Hz, 2 H), 7.64-7.61 (m, 1 H), 7.48 (t, *J*= 8.7 Hz, 2 H), 4.77 (s, 2 H), ; **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.6, 142.5, 135.3, 134.7, 132.8, 129.4, 129.2, 129.1, 129.0, 129.8, 117.9, 117.0 and 62.8; HRMS: calcd for $C_{15}H_{11}NNaO_3S$ (M+Na): 308.0357, found: 308.0351.

2-(4-nitrophenylsulfonyl)-1-phenylethanone (3k)



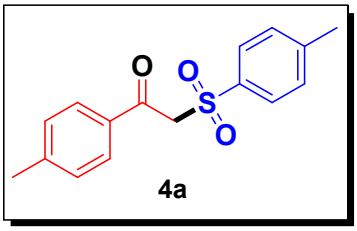
Light yellow solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.37 (d, *J*= 9.3 Hz, 2 H), 8.10 (d, *J* = 8.7 Hz, 2 H), 7.91 (d, *J*= 8.1 Hz, 2 H), 7.63 (t, *J*= 7.6 Hz, 1 H), 7.49 (t, *J*= 7.6 Hz, 2 H), 4.79 (s, 2 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.6, 151.2, 144.3, 135.6, 134.7, 130.3, 129.1, 129.0, 124.2, and 63.2; HRMS: calcd for $C_{14}H_{11}NNaO_5S$ (M+Na): 328.0256, found:328.0257.

1-phenyl-2-(thiophen-2-ylsulfonyl)ethanone (3l)



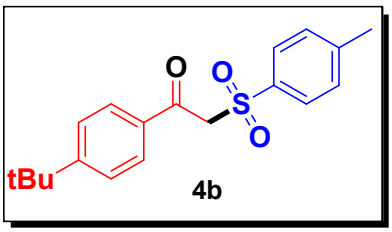
Brown solid; **^{1}H NMR** (600 MHz, CDCl3): δ 7.93 (dd, *J*= 8.3 Hz, 1.0 Hz, 2 H), 7.71 (dd, *J*= 4.9 Hz, 1.3 Hz, 1 H), 7.67 (dd, *J*= 3.7 Hz, 1.2 Hz, 1 H), 7.61-7.58 (m, 1 H), 7.46 (t, *J*= 8.5 Hz, 2 H), 7.10 (dd, *J*= 5.0 Hz, 3.7 Hz, 1 H), 4.80 (s, 2 H); **^{13}C NMR** (150 MHz, CDCl3): δ 187.7, 139.4, 135.6, 135.4, 134.9, 134.4, 129.2, 128.8, 127.9, and 64.3; HRMS: calcd for $C_{12}H_{10}NaO_3S_2$ (M+Na): 288.9969, found:288.9965.

1-p-tolyl-2-tosylethanone (4a)



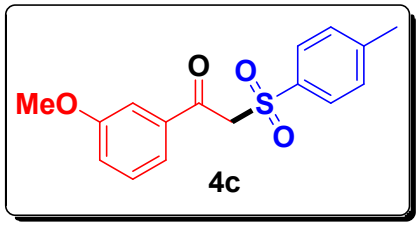
Yellowish Solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.82 (d, *J*= 8.2 Hz, 2 H), 7.73 (d, *J*= 8.2 Hz, 2 H), 7.29 (d, *J*= 7.9 Hz, 2 H) 7.25 (dd, *J*= 7.8 Hz, 0.7 Hz, 2 H), 4.66 (s, 2 H), 2.41 (s, 3H), 2.39(s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.6, 145.5, 145.2, 135.7, 133., 129.7, 129.4, 129.4, 128.5, 63.4, 21.7, and 21.6; HRMS: calcd for $C_{16}H_{16}NaO_3S$ (M+Na): 311.0718, found:311.0711.

1-(4-tert-butylphenyl)-2-tosylethanone (4b)



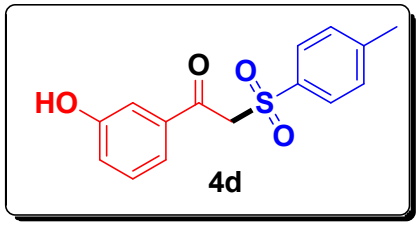
Yellowish solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.86 (d, *J*= 8.3 Hz, 2 H), 7.74 (d, *J*= 8.3 Hz, 2 H), 7.46 (d, *J*= 8.8 Hz, 2 H), 7.30-7.29 (m, 2 H), 4.67 (s, 2 H), 2.41 (s, 3 H), 1.31 (s, 9 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 1876, 158.3, 145.2, 135.7, 133.2, 129.7, 129.3, 128.5, 125.7, 63.4, 35.2, 30.9, and 21.6; HRMS: calcd for $C_{19}H_{22}NaO_3S$ (M+Na): 353.1187, found:353.1184.

1-(3-methoxyphenyl)-2-tosylethanone (4c)



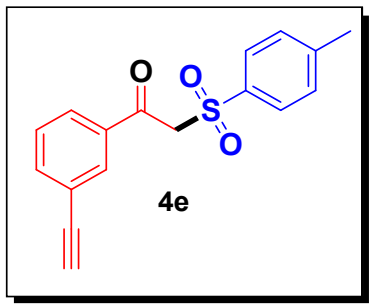
Colorless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): 7.74 (d, *J*= 8.1 Hz, 2 H), 7.48-7.47 (m, 1 H), 7.40 (dd, *J*= 2.5 Hz, 1.75 Hz, 1 H), 7.34 (t, *J*= 8.0 Hz, 1 H), 7.30 (*J*= 7.8 Hz, 2 H), 7.12-7.11 (m, 1 H), 4.67 (s, 2 H), 3.08 (s, 3 H), 2.40 (s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.9, 159.8, 145.2, 137.0, 135.7, 129.7, 128.5, 122.1, 121.0, 112.9, 63.5, 55.4, and 21.6; HRMS: calcd for $C_{16}H_{16}NaO_4S$ (M+Na): 327.0667, found: 327.0668.

1-(3-hydroxyphenyl)-2-tosylethanone (4d)



Brown solid; **^{1}H NMR** (600 MHz, d_6-DMSO): δ 9.86 (s, 1 H), 7.76 (d, *J*= 8.8 Hz, 2 H), 7.42-7.38 (m, 3 H), 7.30 (t, *J*= 8.2 Hz, 1 H), 7.24 (t, *J* = 2.4 Hz, 1 H), 7.05-7.03 (m, 1H), 5.19 (s, 2H), 2.38 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.9, 157.5, 144.5, 137.1, 136.7, 129.8, 129.6, 128.0, 121.3, 120.3, 114.8, 62.3 and 21.1; HRMS: calcd for $C_{15}H_{14}NaO_4S$ (M+Na): 313.0510, found: 313.0517.

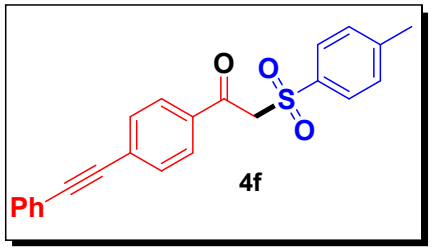
1-(3-ethynylphenyl)-2-tosylethanone (4e)



Brown solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.95 (t, *J* = 1.4 Hz, 1 H), 7.90-7.89 (m, 1 H), 7.72 (d, *J*= 8.2 Hz, 2 H), 7.68-7.66 (m, 1 H), 7.42 (t, *J* = 7.7 Hz, 1 H), 7.31 (d, *J* = 7.3 Hz, 2 H), 4.67

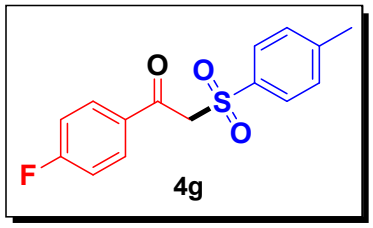
(s, 2H), 3.13 (s, 1H), 2.41 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.4, 145.5, 137.6, 135.7, 135.4, 132.7, 129.8, 129.3, 128.9, 128.5, 123.0, 82.0,78.8, 63.5, and 21.6; HRMS: calcd for $C_{17}H_{14}NaO_3S$ (M+Na): 321.0561, found: 321.0565.

1-(4-(phenylethynyl)phenyl)-2-tosylethanone (4f)



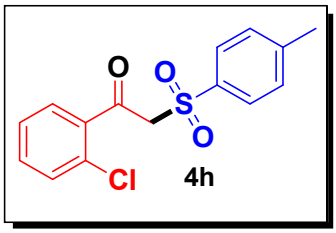
Brown solid; **^{1}H NMR** (600 MHz, CDCl3): δ 7.91 (d, *J* = 8.4 Hz, 2 H), 7.74 (d, *J* = 8.7 Hz, 2 H), 7.59 (d, *J* = 8.4 Hz, 2 H), 7.54 (dd, *J* = 5.8 Hz, 2.1 Hz, 2 H), 7.36-7.35 (m, 3 H), 7.32 (d, *J* = 7.6 Hz, 2 H), 4.68 (s, 2 H), 2.42 (s, 3 H); **^{13}C NMR** (150 MHz, CDCl3): δ 187.3, 145.4, 135.6, 134.7, 131.8, 131.7, 129.8, 129.5, 129.2, 129.0, 128.5, 128.4, 122.3, 93.8, 88.3, 63.6, and 21.6; HRMS: calcd for $C_{23}H_{18}NaO_3S$ (M+Na): 397.0874, found: 397.0873.

1-(4-fluorophenyl)-2-tosylethanone (4g)



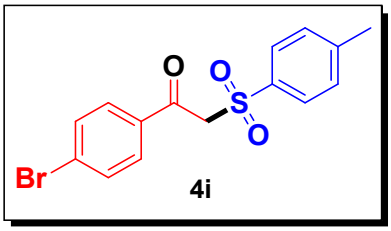
Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.98 (dd, *J* = 9.0 Hz, 5.2 Hz, 2 H), 7.72 (d, *J* = 8.4 Hz, 2 H), 7.31 (d, *J*= 7.9 Hz, 2 H), 7.10 (t, *J* = 8.7 Hz, 2 H), 4.66 (s, 2 H), 2.41 (s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 186.5, 167.2 (d, J_{C-F}= 258.0 Hz), 145.4, 138.0, 135.5, 132.8 (d, J_{C-F}= 9.0 Hz), 132.2 (d, J_{C-F}= 9.0 Hz), 130.0, 129.8, 129.6 (d, J_{C-F}= 25.5 Hz), 128.5, 116.1 (d, J_{C-F}= 21.0 Hz), 63.6 and 21.6; ^{19}F NMR (563 MHz, CCl_3F): δ -101.8 (s); HRMS: calcd for $C_{15}H_{13}FNaO_3S$ (M+Na): 315.0467, found: 315.0463.

1-(2-chlorophenyl)-2-tosylethanone (4h)



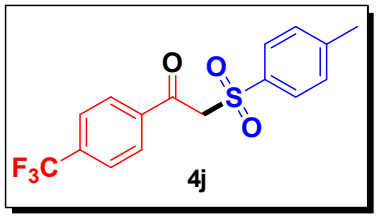
Colourless liquid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.74 (d, *J* = 7.9 Hz, 2 H), 7.53 (d, *J* = 7.7 Hz, 1 H), 7.42 (dd, *J*= 9.5 Hz, 2.0 Hz, 1 H), 7.34 (t, *J*= 8.0 Hz, 1 H), 7.31-7.30 (m, 3 H), 4.78 (s, 2 H), 2.42 (s, 3 H) ; **^{13}C NMR** (150 MHz, $CDCl_3$): δ 190.2, 145.3, 137.3, 135.9, 133.0, 131.5, 130.6, 130.5, 129.8, 128.5, 127.1, 66.4 and 21.6; HRMS: calcd for $C_{15}H_{13}ClNaO_3S$ (M+Na): 331.0172, found: 331.0175.

1-(4-bromophenyl)-2-tosylethanone (4i)



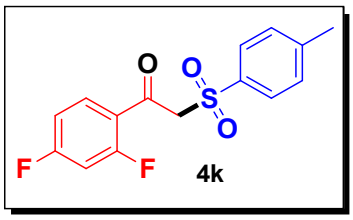
Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.80 (d, *J* = 8.8 Hz, 2 H), 7.72 (d, *J* = 8.3 Hz, 2 H), 7.61 (d, *J* = 9.2 Hz, 2 H), 7.32 (d, *J* = 8.3 Hz, 2 H), 4.65 (s, 2 H), 2.43 (s, 3 H) ; **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.2, 145.5, 135.5, 134.4, 132.2, 130.8, 129.9, 129.9, 128.5, 63.7, and 21.7; HRMS: calcd for $C_{15}H_{13}BrNaO_3S$ (M+Na): 374.9666, found: 374.9668.

2-tosyl-1-(4-(trifluoromethyl)phenyl)ethanone (4j)



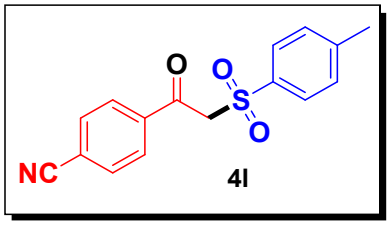
Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.04 (d, *J* = 8.0 Hz, 2 H), 7.72 (d, *J* = 7.5 Hz, 4 H), 7.32 (d, *J* = 8.5 Hz, 2 H), 4.72 (s, 2 H), 2.42 (s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.9, 145.6, 138.8, 135.5 (q, $Jc\text{-}_F$ = 19.5 Hz, 13.5 Hz), 129.9, 129.6, 128.5, 125.8, 124.2 (q, $Jc\text{-}_F$ = 237.0 Hz, 271.5 Hz), and 63.8; ^{19}F NMR (563 MHz, CCl_3F): δ -63.0 (s); HRMS: calcd for $C_{16}H_{13}F_3NaO_3S$ (M+Na): 365.0435, found: 365.0438.

1-(2,4-difluorophenyl)-2-tosylethanone (4k)



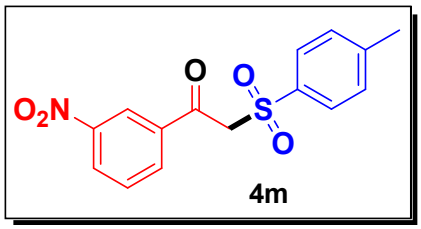
Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.86-7.82 (m, 1 H), 7.74 (d, *J* = 8.4 Hz, 2 H), 7.31 (dd, *J* = 8.3 Hz, 0.8 Hz, 2 H), 6.96-6.93 (m, 1 H), 6.85-6.81 (m, 1 H), 4.71 (s, 2 H), 2.41 (s, 3 H) ; **^{13}C NMR** (150 MHz, $CDCl_3$): δ 184.5, 167.5 (d, $Jc\text{-}_F$ = 259.5 Hz), 165.8 (d, $Jc\text{-}_F$ = 259.5 Hz), 163.6 (d, $Jc\text{-}_F$ = 12.0 Hz), 161.9 (d, $Jc\text{-}_F$ = 13.5 Hz), 145.3, 136.0, 133.2 (d, $Jc\text{-}_F$ = 7.5 Hz), 129.7, 128.4, 121.2, 112.7 (d, $Jc\text{-}_F$ = 24.0 Hz,), 105.1 (d, $Jc\text{-}_F$ = 52.5 Hz), 105.0, 66.9 (d, $Jc\text{-}_F$ = 9.0 Hz), and 21.6; ^{19}F NMR (563 MHz, CCl_3F): δ -98.0, -103.3 (s); HRMS: calcd for $C_{15}H_{12}F_2NaO_3S$ (M+Na): 333.0373, found: 333.0376.

4-(2-tosylacetyl)benzonitrile (4l)



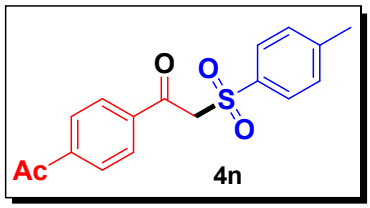
Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.05 (d, *J* = 9.1 Hz, 2 H), 7.78 (d, *J* = 8.6 Hz, 2 H), 7.72 (d, *J* = 7.6 Hz, 2 H), 7.34 (dd, *J* = 7.8 Hz, 0.7 Hz, 2 H), 4.69 (s, 2 H), 2.43 (s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.1, 145.8, 138.5, 135.3, 132.5, 130.0, 129.7, 128.4, 117.5, 117.4, 63.8, and 21.7; HRMS: calcd for $C_{16}H_{13}NNaO_3S$ (M+Na): 322.0514, found: 322.0515.

1-(3-nitrophenyl)-2-tosylethanone (4m)



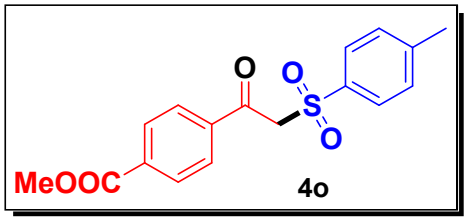
Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.70 (s, 1 H), 8.45-8.43 (m, 1 H), 8.31-8.30 (m, 1 H), 7.73-7.69 (m, 3 H), 7.34 (d, *J* = 8.1 Hz, 2 H), 4.74 (s, 2 H), 2.43 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 186.3, 148.4, 145.8, 136.9, 135.3, 134.8, 130.1, 130.0, 128.5, 128.3, 124.1, 63.8, and 21.7 ; HRMS: calcd for $C_{15}H_{13}NNaO_5S$ (M+Na): 342.0412, found: 342.0415.

1-(4-acetylphenyl)-2-tosylethanone (4n)



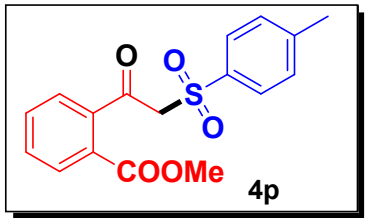
Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.01 (s, 4 H), 7.73 (d, *J* = 8.2 Hz, 2 H), 7.73 (dd, *J* = 8.74 Hz, 1.0 Hz, 2 H), 4.71 (s, 2 H), 2.62 (s, 3H), 2.42(s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 197.1, 187.7, 145.6, 140.8, 138.7, 135.5, 129.9, 129.5, 128.5, 63.9, 26.9, and 21.7; HRMS: calcd for $C_{17}H_{16}NaO_4S$ (M+Na): 339.0667, found: 339.0664.

methyl 4-(2-tosylacetyl)benzoate (4o)



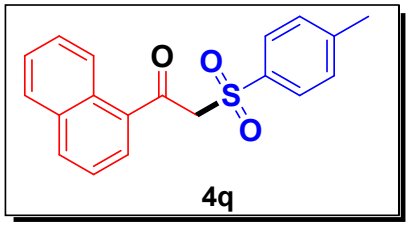
Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.10 (d, *J* = 9.2 Hz, 2 H), 7.98 (d, *J* = 8.9 Hz, 2 H), 7.73 (d, *J* = 7.7 Hz, 2 H), 7.32 (dd, *J* = 8.5 Hz, 1.0 Hz, 2 H), 4.71 (s, 2 H), 3.93 (s, 3 H), 2.42 (s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.8, 165.8, 145.6, 138.7, 135.5, 134.8, 129.9, 129.9, 129.2, 128.5, 63.8, 52.6, and 21.7; HRMS: calcd for $C_{17}H_{16}NaO_5S$ (M+Na): 355.0616, found: 355.0612.

methyl 2-(2-tosylacetyl)benzoate (4p)



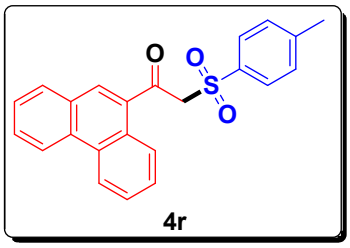
Colourless sticky liquid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.89 (dd, *J* = 7.8 Hz, 0.9 Hz, 1 H), 7.74 (d, *J* = 8.7 Hz, 2 H), 7.59-7.57 (m, 1 H), 7.51-7.48 (m, 1 H), 7.44 (dd, *J* = 7.9 Hz, 1.0 Hz, 1 H), 7.30 (d, *J* = 7.9 Hz, 2 H), 4.59 (s, 2 H), 3.82 (s, 3 H), 2.42 (s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 193.3, 166.5, 145.1, 142.0, 136.1, 132.9, 130.5, 129.7, 128.4, 127.6, 66.9, 52.6, and 21.6; HRMS: $C_{17}H_{16}NaO_5S$ (M+Na): 355.0616, found: 355.0612.

1-(naphthalen-1-yl)-2-tosylethanone (4q)



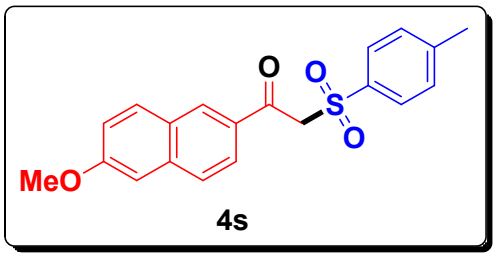
Colourless sticky liquid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.54 (d, *J* = 9.6 Hz, 1 H), 8.01 (d, *J* = 8.2 Hz, 1 H), 7.98 (d, *J* = 7.9 Hz, 1 H), 7.83 (d, *J* = 7.5 Hz, 1 H), 7.71 (d, *J* = 7.9 Hz, 2 H), 7.57-7.50 (m, 2 H), 7.46 (t, *J* = 7.5 Hz, 1 H), 7.22 (d, *J* = 7.7 Hz, 2 H), 4.82 (s, 2 H), 2.38 (s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 190.4, 145.1, 135.8, 134.5, 133.8, 133.3, 130.7, 130.3, 129.7, 128.6, 128.5, 128.5, 126.7, 125.5, 124.2, 66.2 and 21.6; HRMS calcd for $C_{19}H_{16}NaO_3S$ (M+Na): 347.0718, found: 347.0716.

1-(phenanthren-9-yl)-2-tosylethanone (4r)



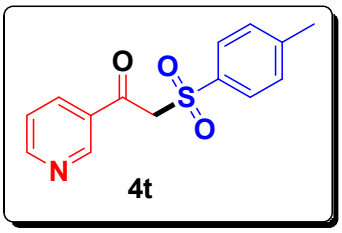
Colourless sticky liquid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.67 (dd, *J* = 8.0 Hz, 0.7 Hz, 1 H), 8.63 (d, *J* = 8.5 Hz, 1 H), 8.52 (dd, *J*= 7.7 Hz, 1.0Hz, 1 H), 8.17 (s, 1 H), 7.90 (dd, *J* = 7.9 Hz, 1.0 Hz, 1 H), 7.76-7.74 (m, 1 H), 7.71-7.65 (m, 3 H), 7.63-7.59 (m, 2 H), 7.19 (d, *J* = 8.5 Hz, 2 H), 4.90 (s, 2 H), 2.31 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 190.4, 145.2, 135.7, 133.4, 132.5, 132.4, 130.7, 130.3, 129.8, 129.7, 129.4, 128.5, 127.9, 127.8, 127.4, 127.2, 126.4, 122.7, 122.6, 66.4, and 21.5; HRMS: calcd for $C_{23}H_{18}NaO_3S$ (M+Na): 397.0874, found: 397.0873.

1-(6-methoxynaphthalen-2-yl)-2-(phenylsulfonyl)ethanone (4s)



Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.32 (s, 1 H), 7.90 (d, *J* = 8.2 Hz, 1 H), 7.80 (d, *J*= 9.0 Hz, 1 H), 7.75 (d, *J* = 8.6 Hz, 2 H), 7.72 (d, *J* = 9.0 Hz, 1 H), 7.28 (d, *J* = 8.0 Hz, 2 H), 7.19-7.17 (m, 1 H), 7.11 (s, 1 H), 4.78 (s, 2 H), 3.92 (s, 3 H), 2.37(s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.5, 160.4, 145.2, 137.8, 135.7, 131.9, 131.5, 131.1, 129.7, 128.5, 127.5, 127.3, 124.7, 119.9, 105.7, 63.6, 55.4 and 21.6; HRMS: calcd for $C_{19}H_{16}NaO_4S$ (M+Na): 363.0667, found: 363.0666.

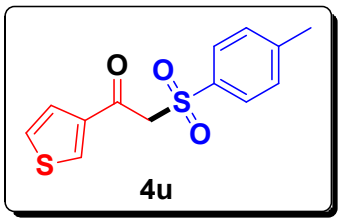
1-(pyridin-3-yl)-2-tosylethanone (4t)



4t

Colourless solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.30 (d, *J* = 8.2 Hz, 2 H), 7.73 (d, *J* = 7.7 Hz, 3 H), 7.33 (d, *J* = 8.6 Hz, 3 H), 4.70 (s, 2 H), 2.42 (s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 187.6, 153.7, 150.0, 145.7, 136.4, 135.4, 130.5, 129.9, 128.4, 127.6, 63.8, and 21.6; HRMS: calcd for $C_{14}H_{13}NNaO_3S$ (M+Na): 298.0514, found: 298.0511.

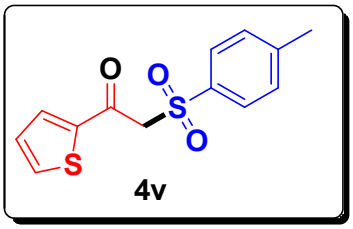
1-(thiophen-3-yl)-2-tosylethanone (4u)



4u

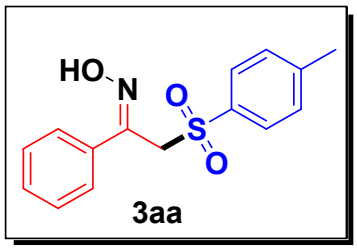
Colourless sticky liquid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.17-8.16 (m, 1 H), 7.73 (d, *J* = 8.9 Hz, 2 H), 7.50 (dd, *J*= 5.2 Hz, 1.5Hz, 1 H), 7.32-7.29 (m, 3 H), 4.56 (s, 2 H), 2.42 (s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 181.6, 145.4, 141.1, 135.5, 129.8, 128.5, 127.2, 126.8, 65.2, and 21.6; HRMS: calcd for $C_{13}H_{12}NaO_3S_2$ (M+Na): 303.0126, found: 303.0123.

1-(thiophen-2-yl)-2-tosylethanone (4v)



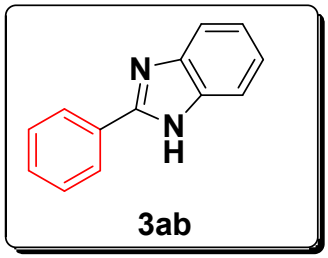
Colourless sticky liquid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.78-7.77 (m, 1 H), 7.73-7.70 (m, 3 H), 7.31 (d, *J*= 8.4 Hz, 2 H), 7.12 (t, *J* = 4.8 Hz, 1 H), 4.58 (s, 2 H), 2.40 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 180.2, 145.4, 143.1, 136.3, 135.2, 129.8, 128.6, 128.5, 64.6, and 21.6; HRMS: calcd for $C_{13}H_{12}NaO_3S_2$ (M+Na): 303.0126, found: 303.0129.

(E)-1-phenyl-2-tosylethanone oxime (3aa)



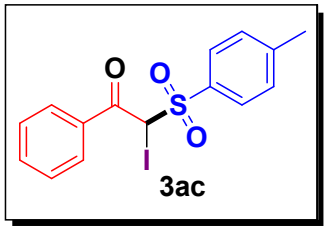
White solid; **^{1}H NMR** (600 MHz, DMSO-d_6): δ 11.80 (s, 1 H), 7.65-7.62 (m, 4 H), 7.33 (t, *J*= 8.1 Hz, 5 H), 4.91 (s, 2 H), 2.35 (s, 3 H); **^{13}C NMR** (150 MHz, DMSO-d_6): δ145.9, 144.6, 137.1, 134.7, 129.7, 129.1, 128.1, 126.6, 51.6, and 21.3; HRMS: calcd for $C_{15}H_{15}NNaO_3S$ (M+Na): 312.0670, found: 312.0668.

2-phenyl-1H-benzo[d]imidazole (3ab)

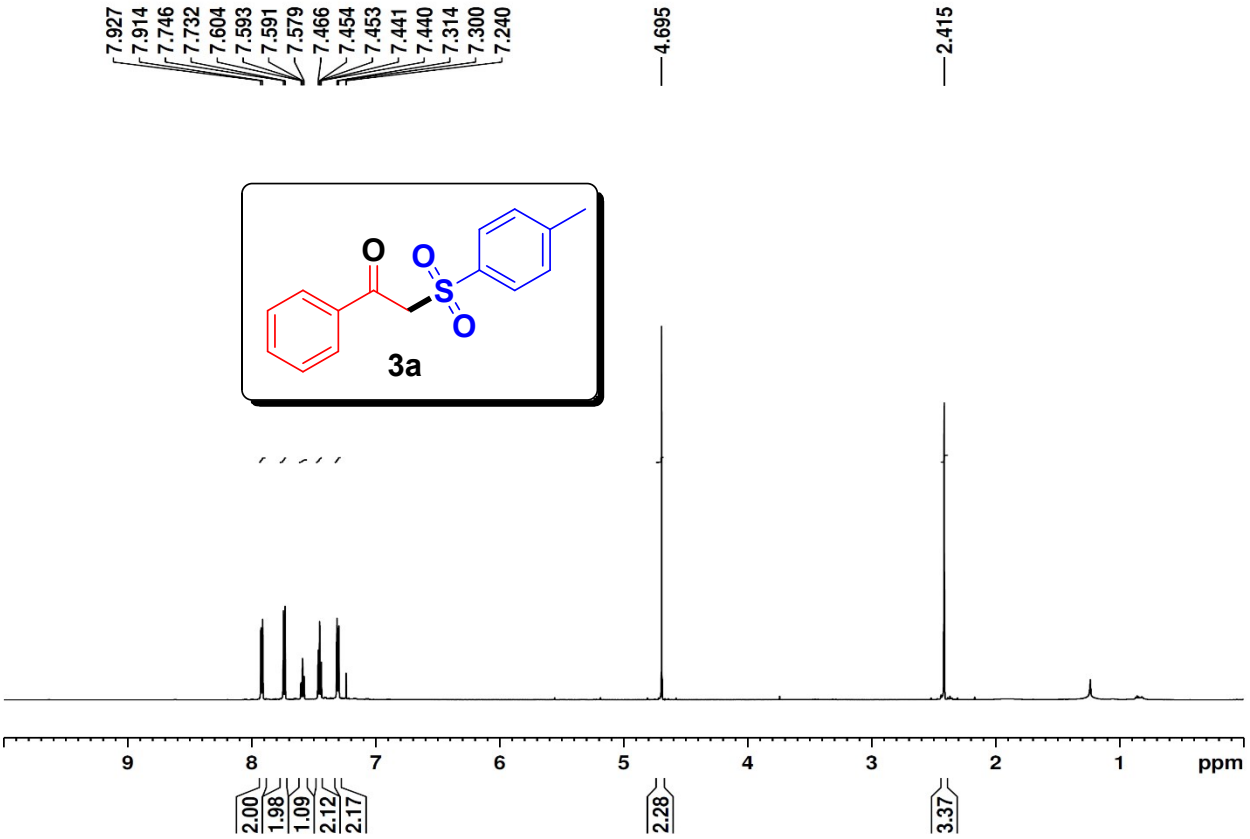


White solid; **^{1}H NMR** (600 MHz, DMSO-d_6): δ 8.17 (d, *J*= 7.8 Hz, 1 H), 7.55-7.47 (m, 5 H), 7.20 (d, *J*= 7.6 H, 2 H); **^{13}C NMR** (150 MHz, DMSO-d_6): δ 151.1, 130.3, 130.0, 129.1, 126.6, 122.1, and 119.2; HRMS: calcd for $C_{13}H_{11}N_2$ (M+H): 195.0922, found: 195.0927.

(S)-2-iodo-1-phenyl-2-tosylethanone (3ac)



Coloress solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.98 (dd, *J* = 8.5 Hz, *J* = 1.0 Hz, 1 H), 7.83 (d, *J* = 8.2 Hz, 2 H), 7.65-7.62 (m, 1H), 7.51 (t, *J* = 7.6 Hz, 2 H), 7.36 (d, *J* = 8.4 Hz, 2 H), 6.19 (s, 1 H), 2.45 (s, 3 H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 186.7, 146.4, 134.7, 134.3, 131.7, 130.9, 129.5, 129.5, 129.1, 128.9 and 60.0; EI-HRMS: calcd for $C_{15}H_{13}IO_3S$: 399.9630, found: 399.9620.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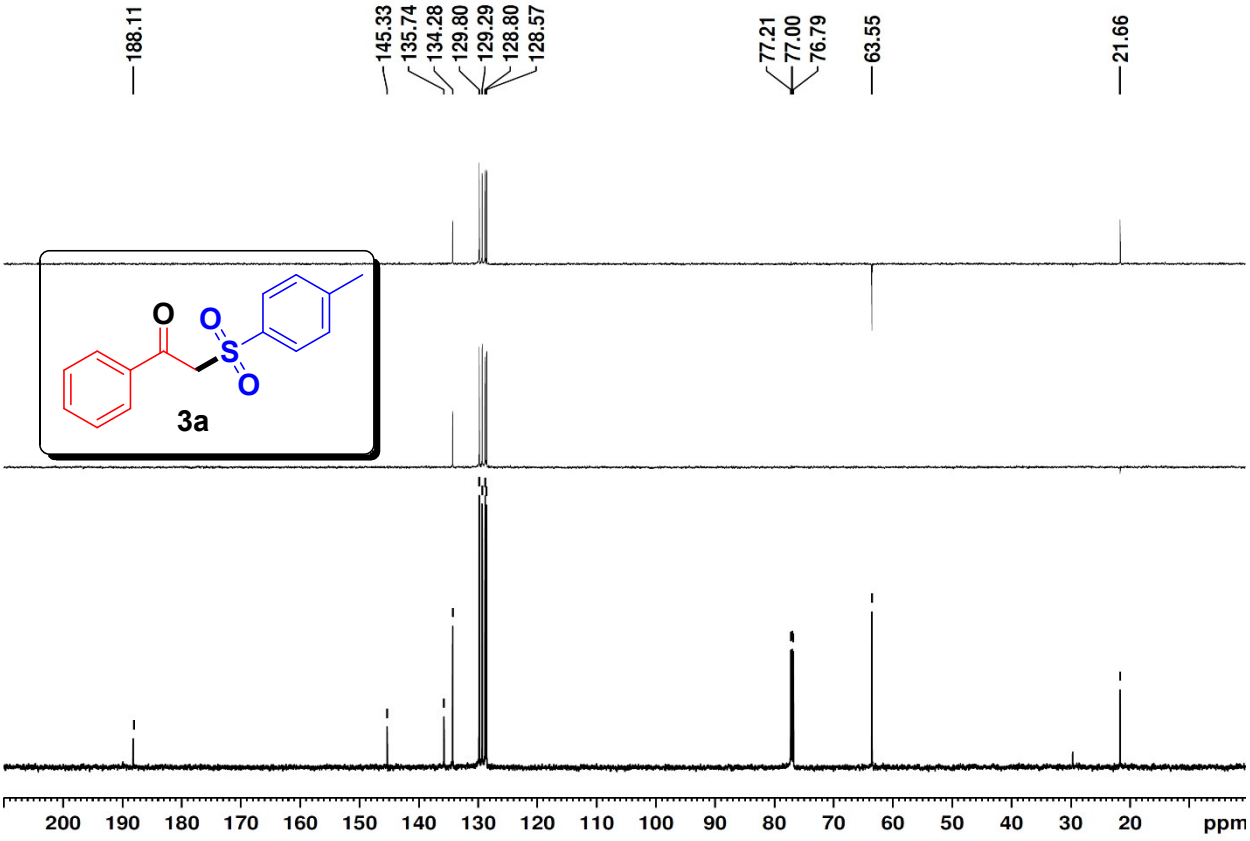
7.453
7.441
7.440
7.314
7.300
7.240
4.695
2.415
3a
9
8
7
6
5
4
3
2
1
ppm
2.00
1.98
1.09
2.12
2.17
2.28
3.37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128.80
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77.00
76.79
63.55
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3a
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120
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20
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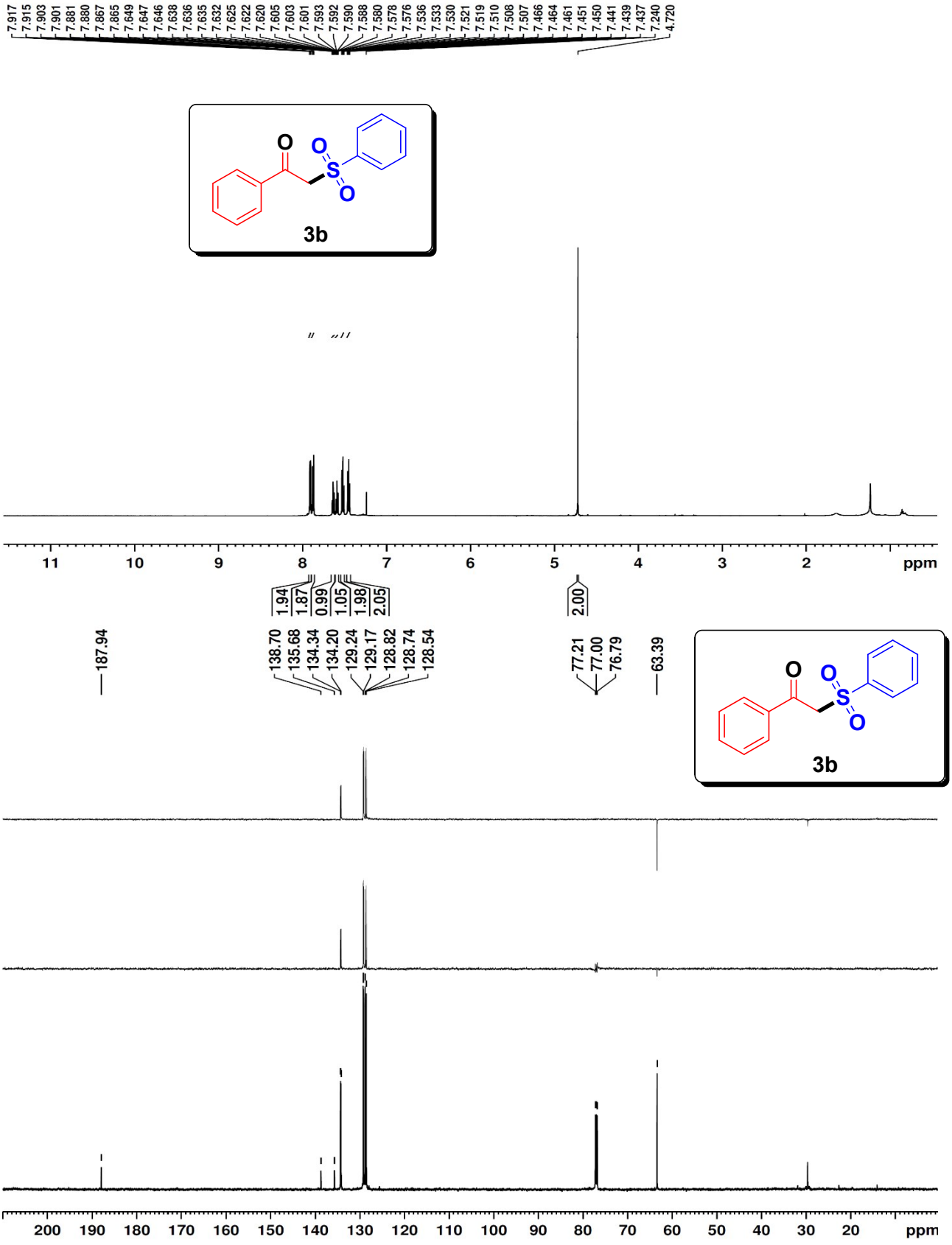
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128.74
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77.21
77.00
76.79
63.39
3b
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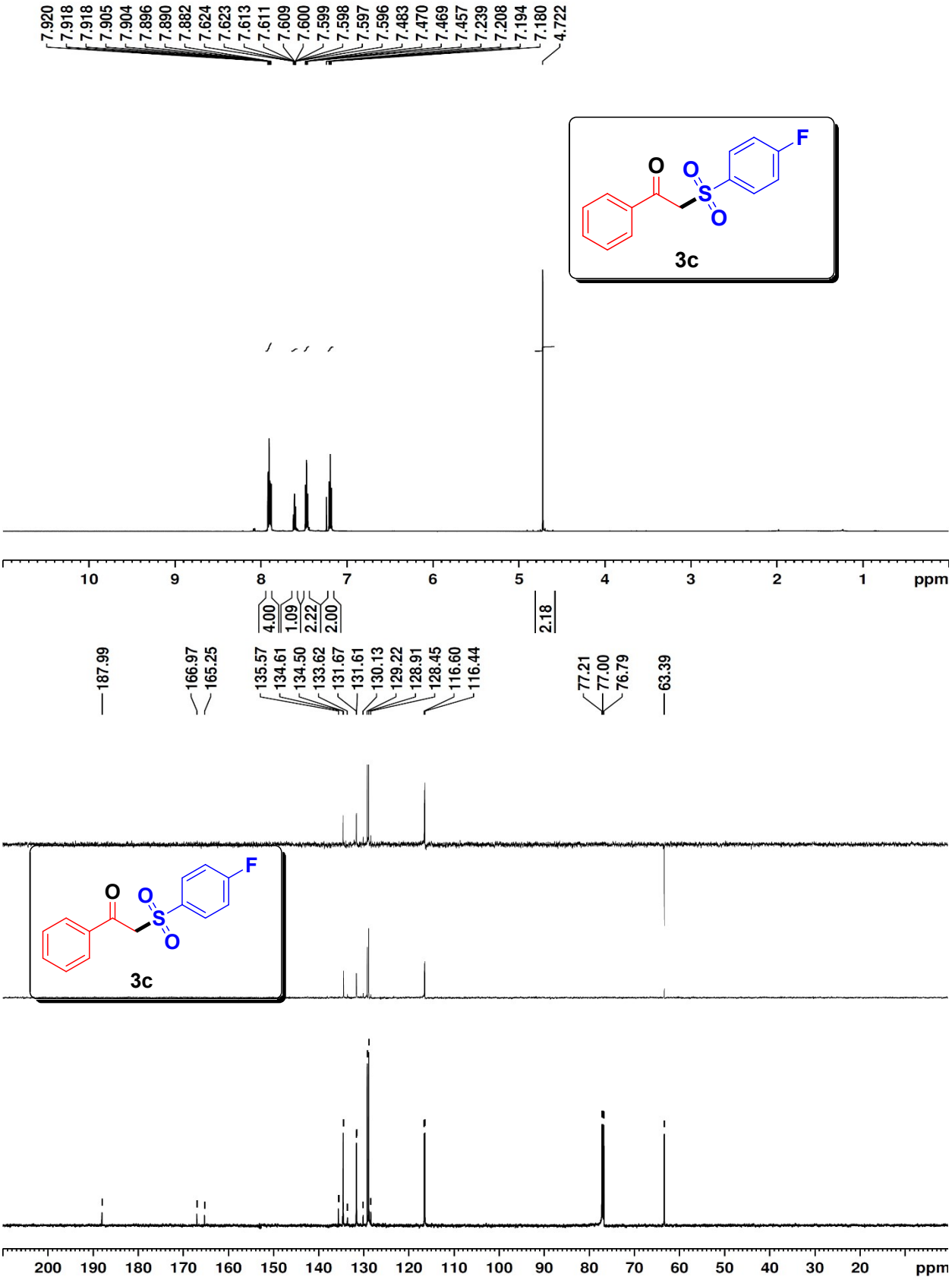
20
ppm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O
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S
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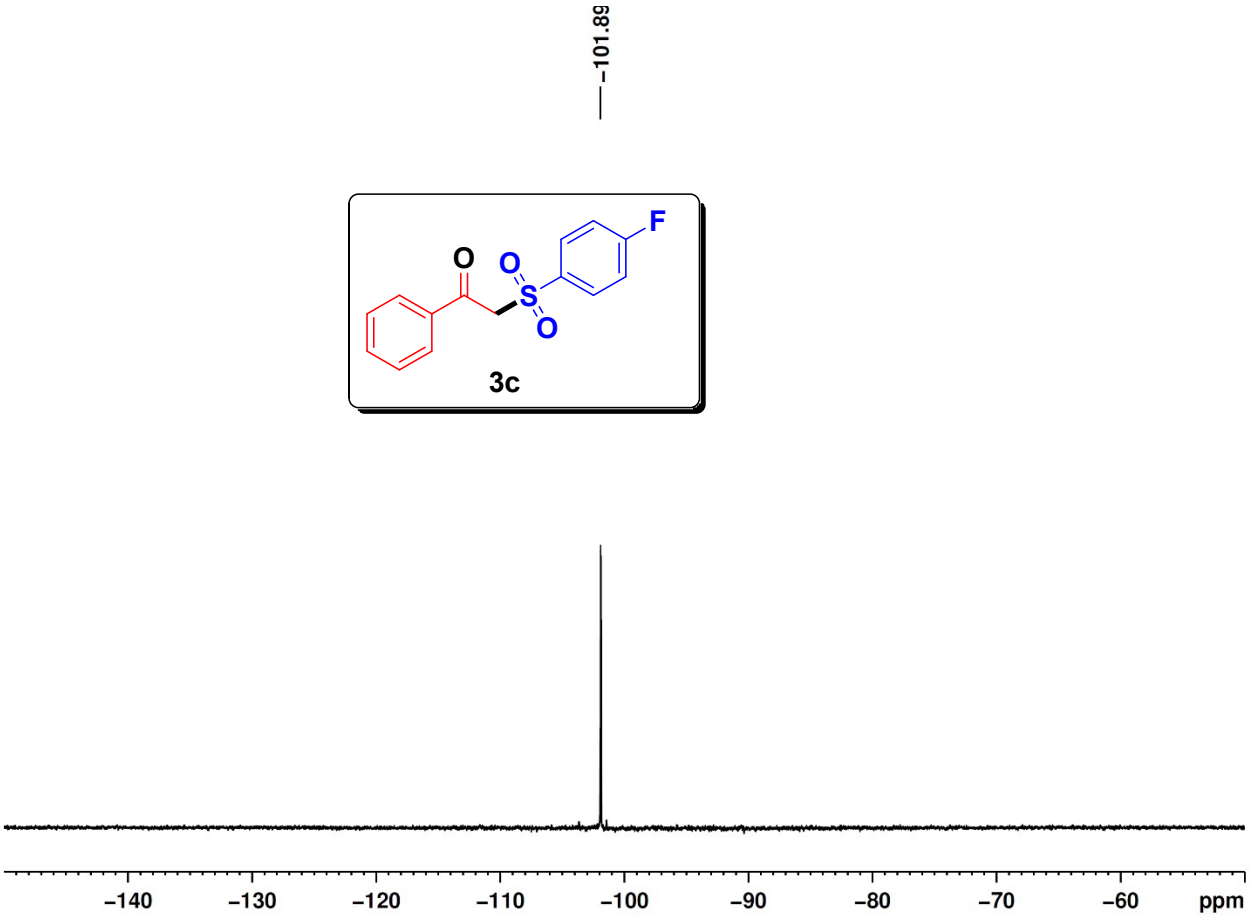
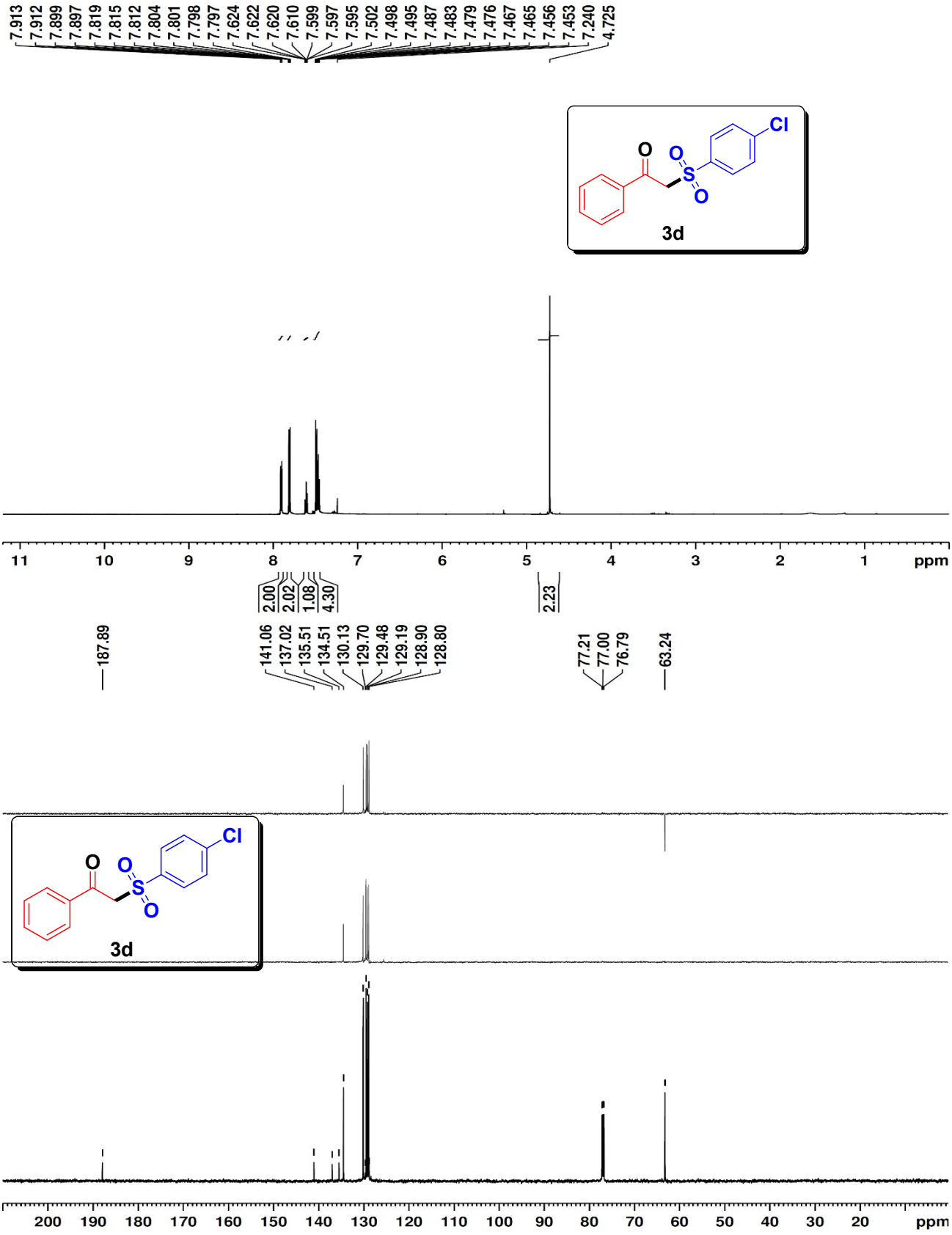
^{19}F NMR

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120
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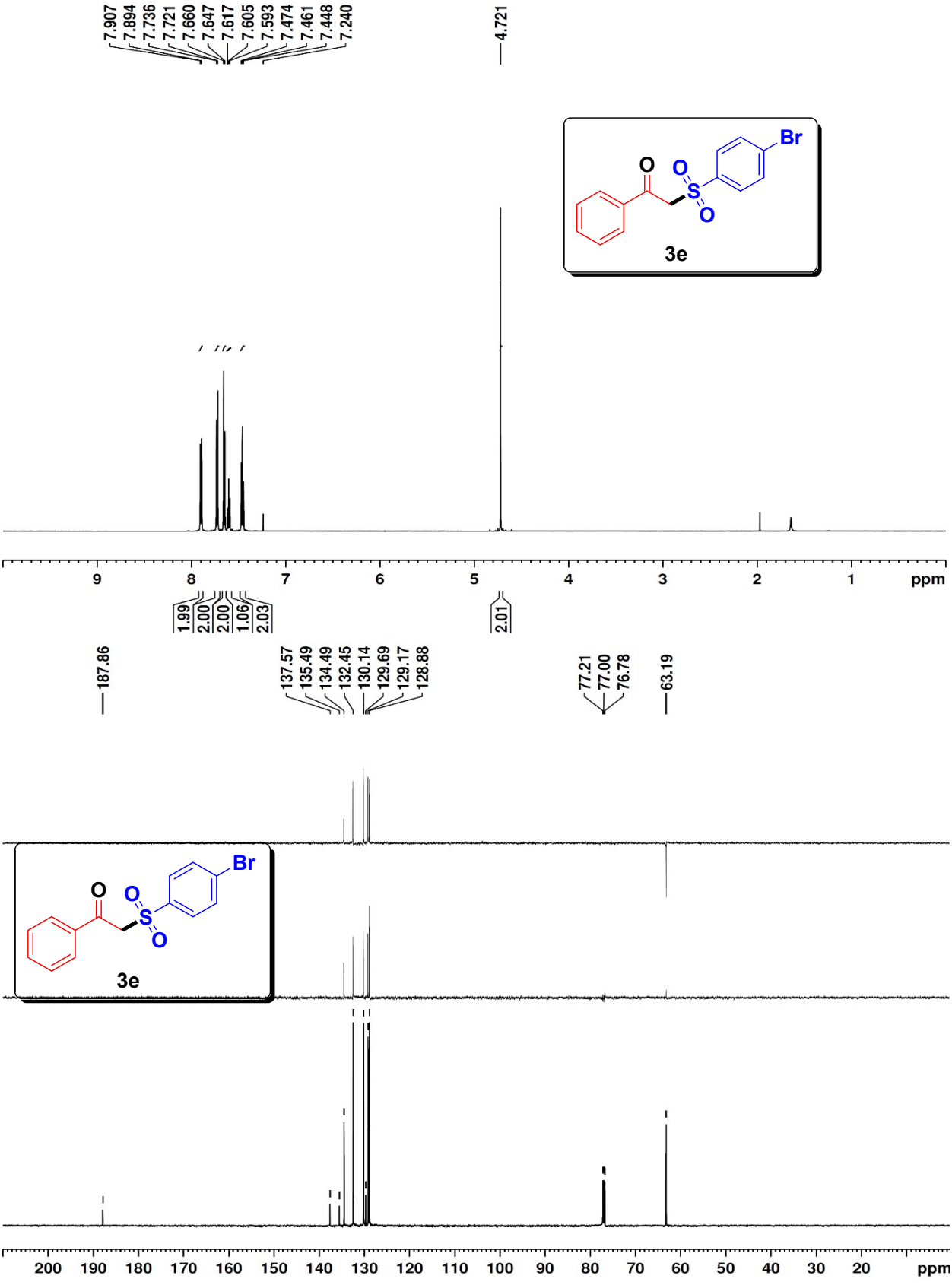
ppm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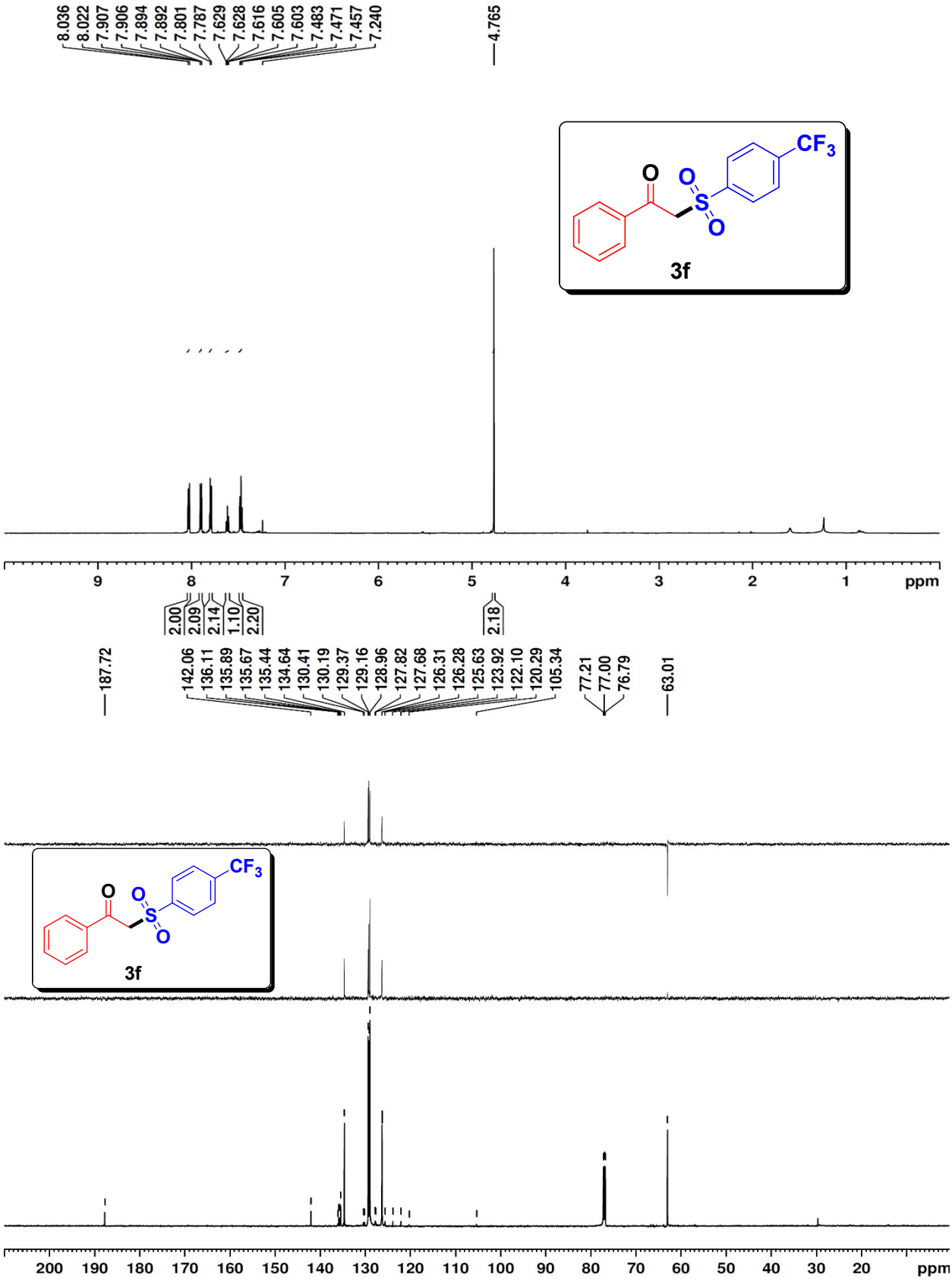
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7.603
7.483
7.471
7.457
7.240
4.765
CF3
O
O
S
O
3f
9
8
7
6
5
4
3
2
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ppm
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2.09
2.14
1.10
2.20
2.18
187.72
142.06
136.11
135.89
135.67
135.44
134.64
130.41
130.19
129.37
129.16
128.96
127.82
127.68
126.31
126.28
125.63
123.92
122.10
120.29
105.34
77.21
77.00
76.79
63.01
CF3
O
O
S
O
3f
200
190
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100
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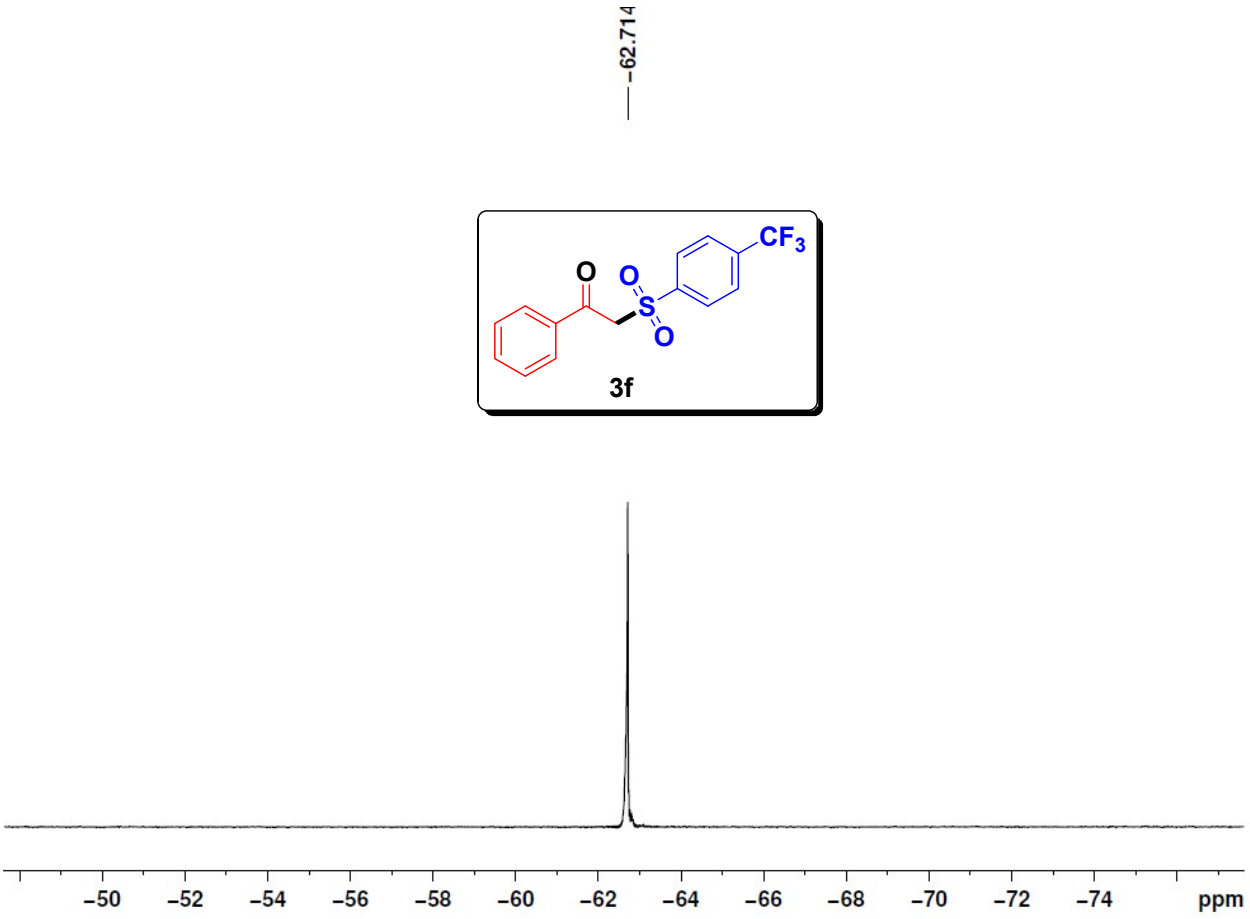
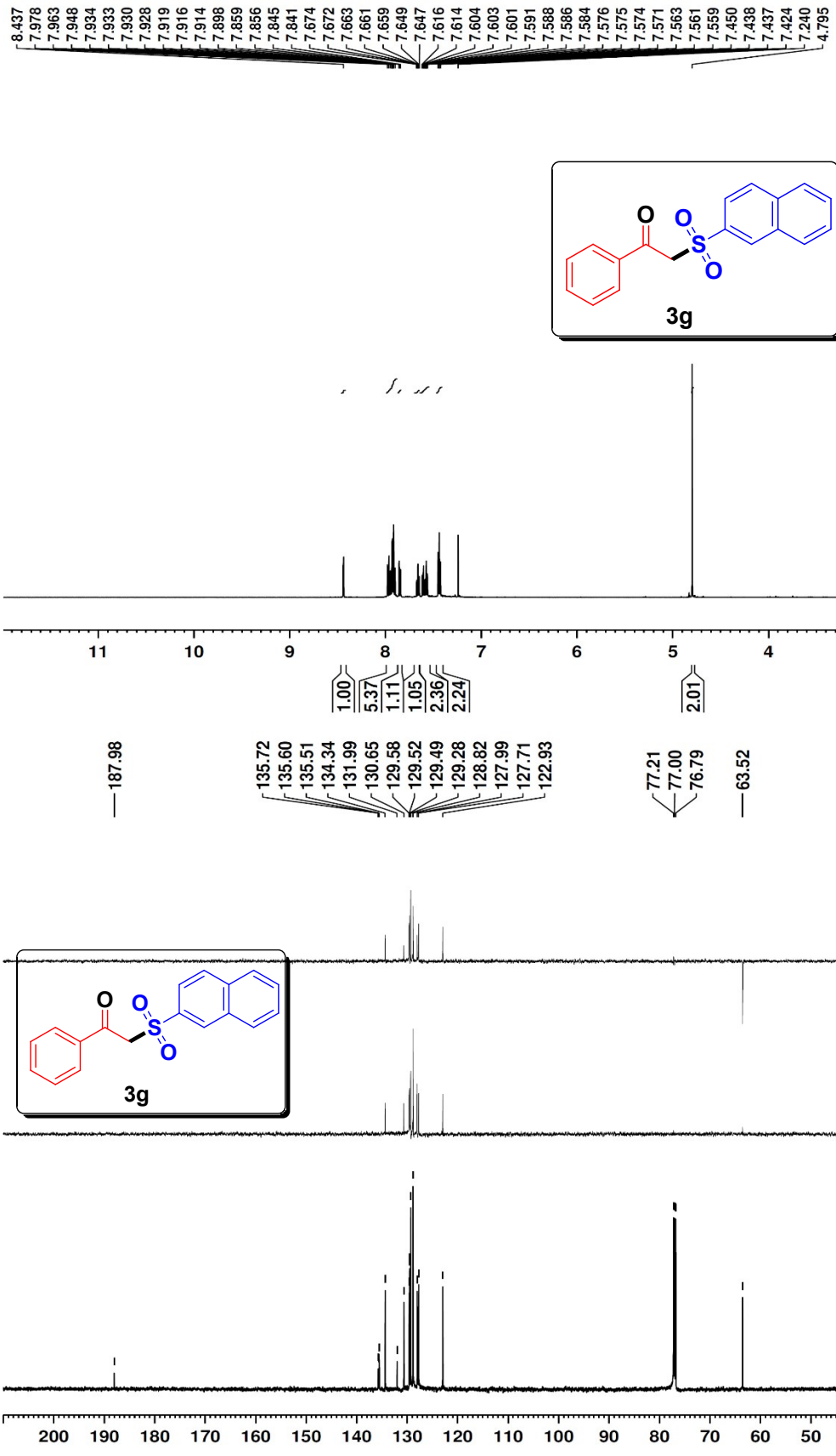
ppm

^{19}F NMR

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7.856
7.845
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7.674
7.672
7.663
7.661
7.659
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7.616
7.614
7.604
7.603
7.601
7.591
7.588
7.586
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7.571
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7.559
7.450
7.438
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7.240
4.795
3g
11
10
9
8
7
6
5
4
3
2
ppm
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1.11
1.05
2.36
2.24
2.01
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3g
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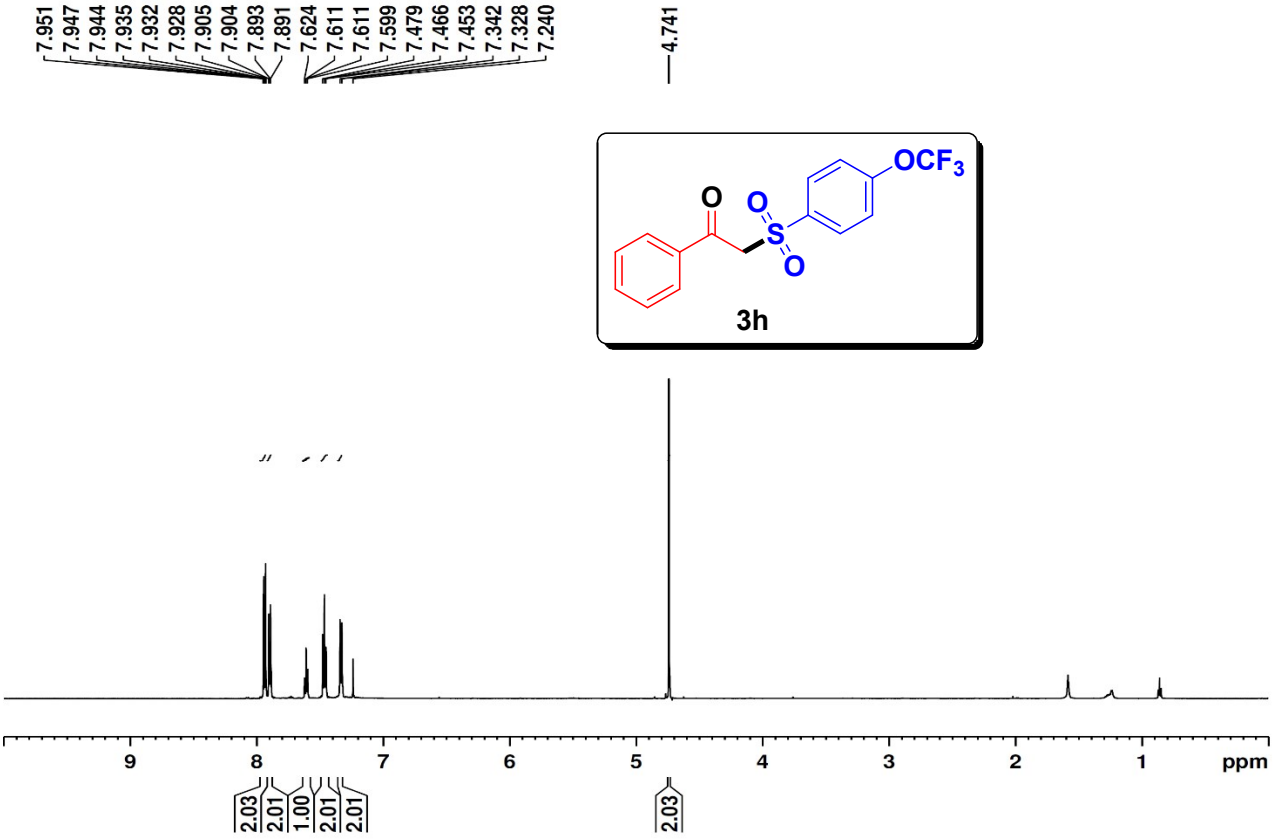
ppm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7.891
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7.611
7.611
7.599
7.479
7.466
7.453
7.342
7.328
7.240
4.741
OCF3
O
O
S
O
3h
9
8
7
6
5
4
3
2
1
ppm
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2.01
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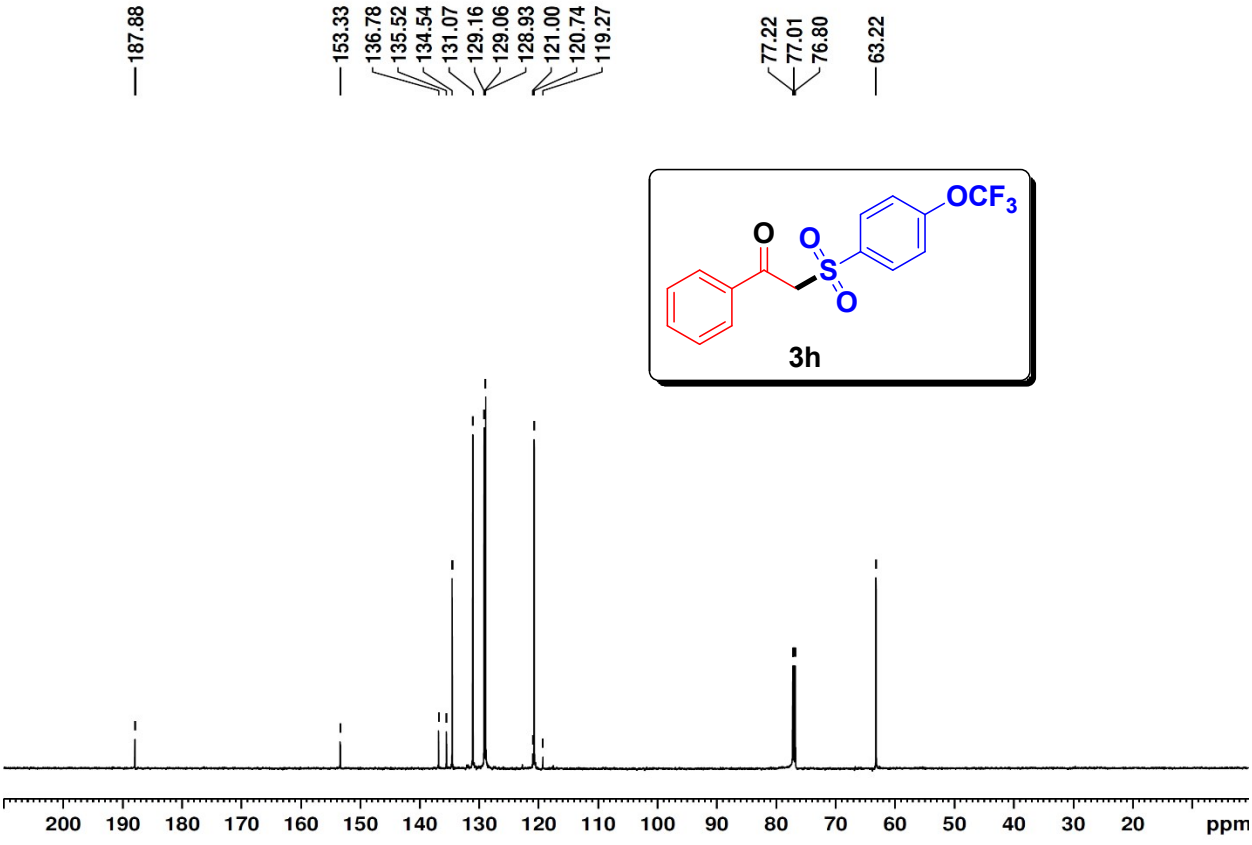
2.01
2.01
2.03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129.06
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121.00
120.74
119.27
77.22
77.01
76.80
63.22
OCF3
O
O
S
O
3h
200
190
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100
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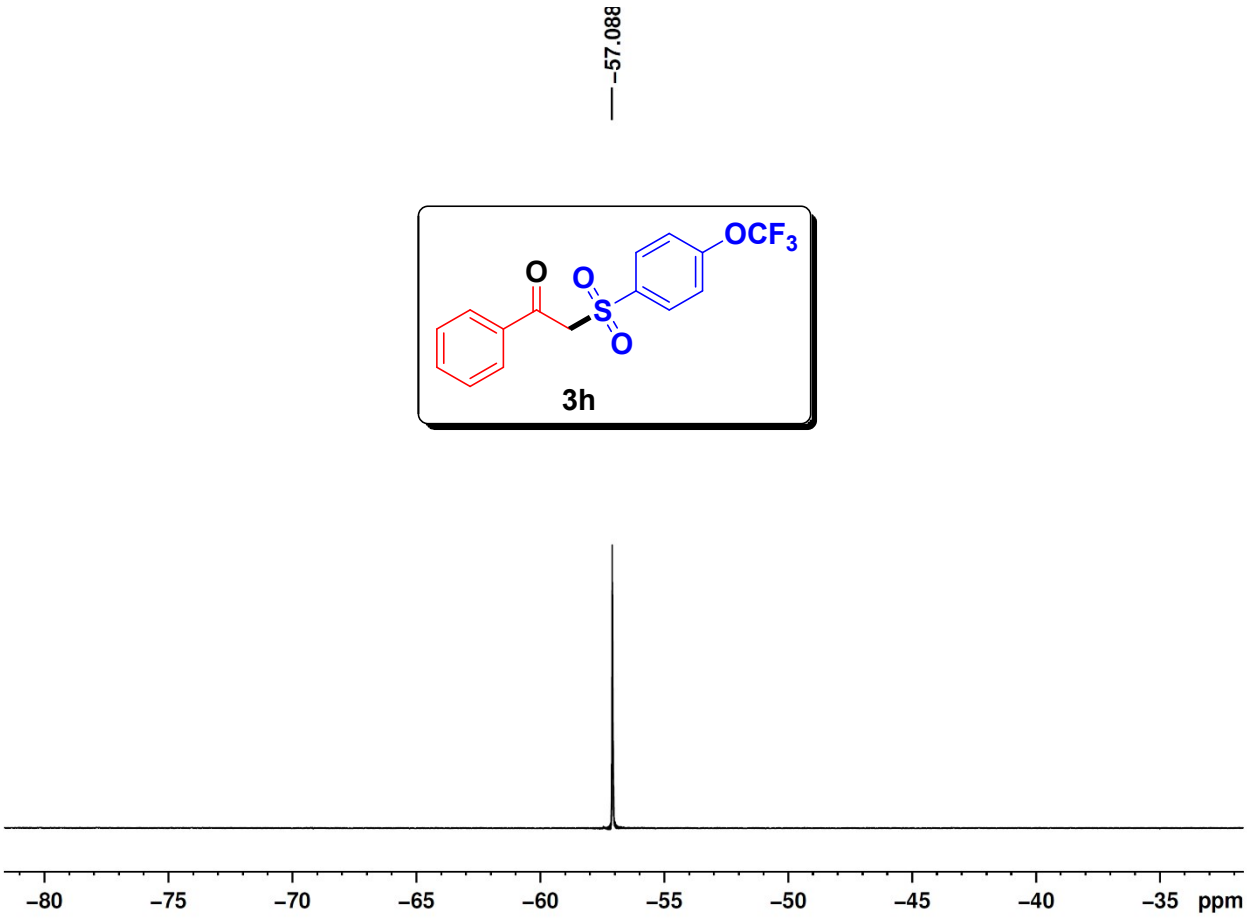
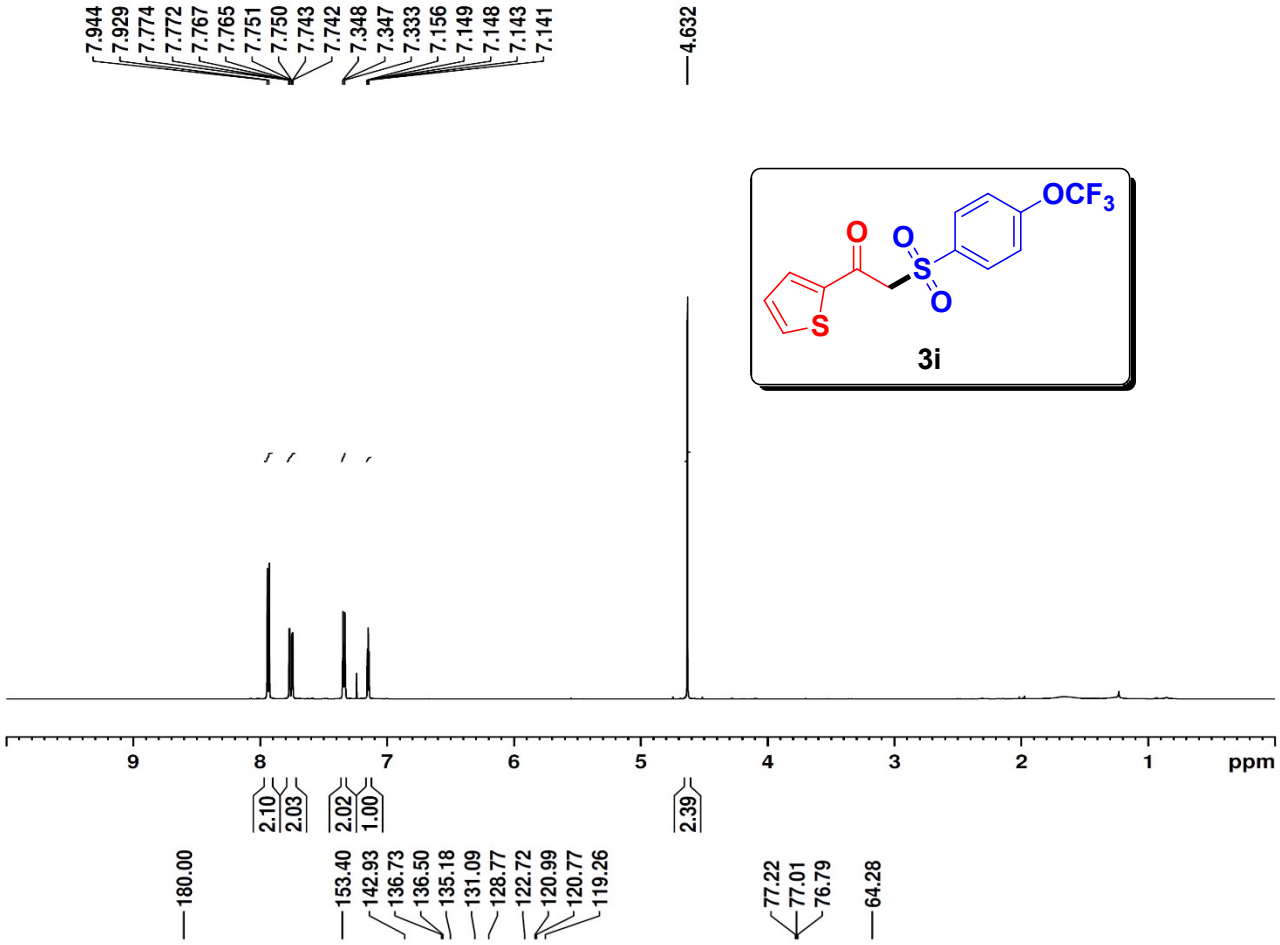
20
ppm

^{19}F NMR

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7.333
7.156
7.149
7.148
7.143
7.141
4.632
OCF3
O
O
S
O
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6
5
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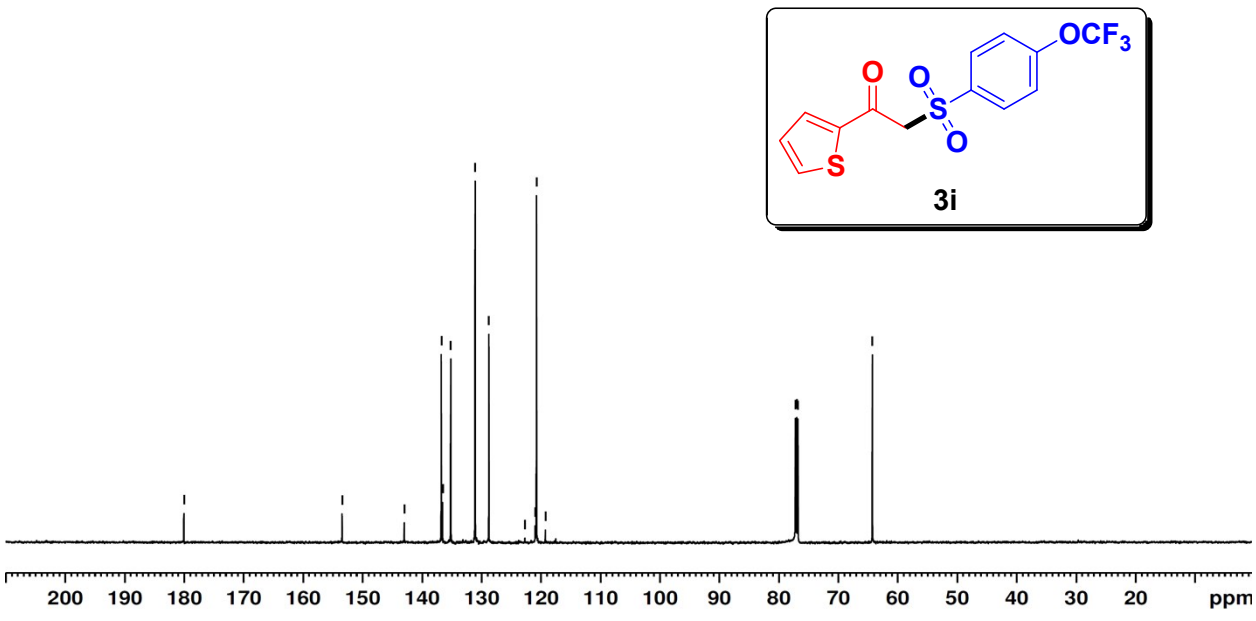
2.02
1.00
2.39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128.77
122.72
120.99
120.77
119.26
77.22
77.01
76.79
64.28
OCF3
O
O
S
O
S
3i
200
190
180
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140
130
120
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100
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80
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60
50
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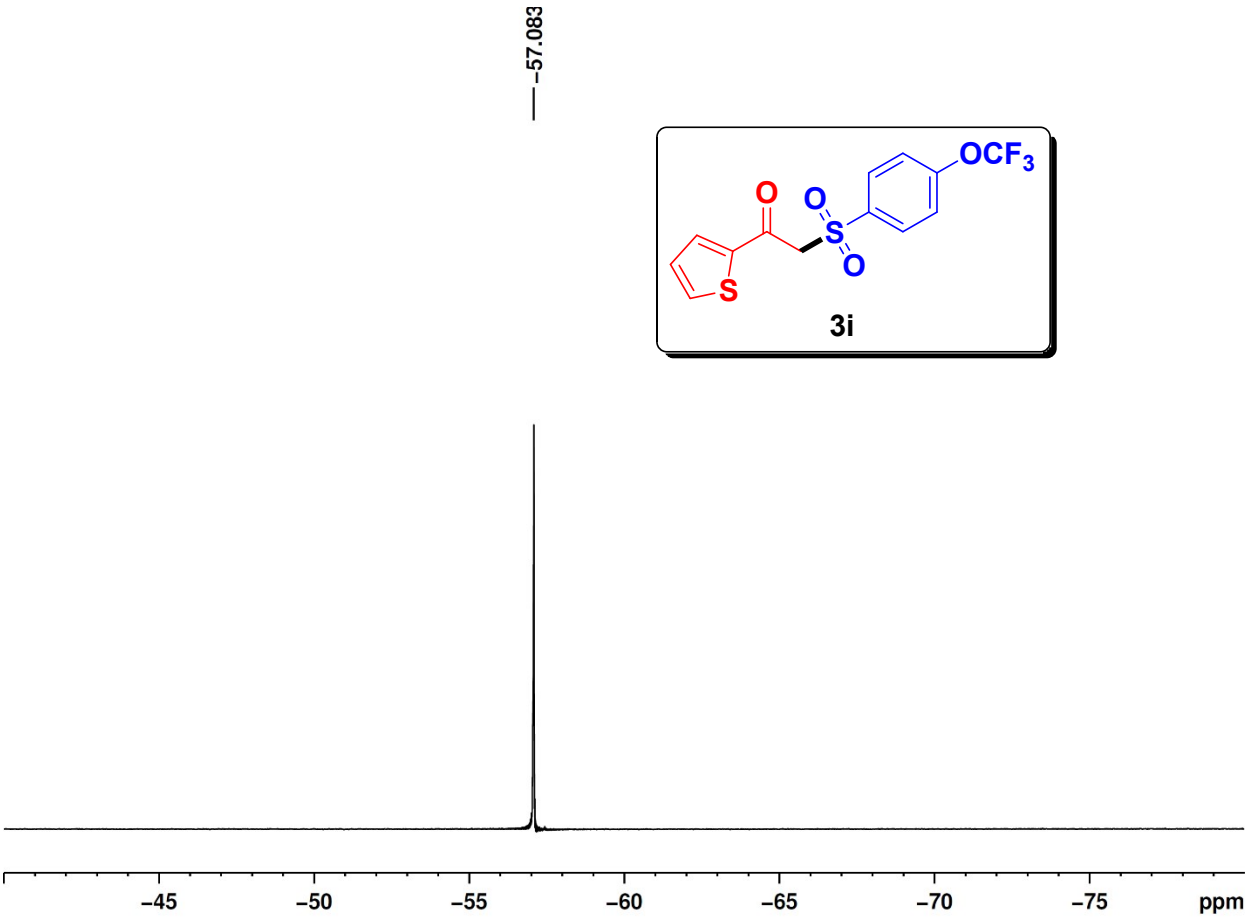
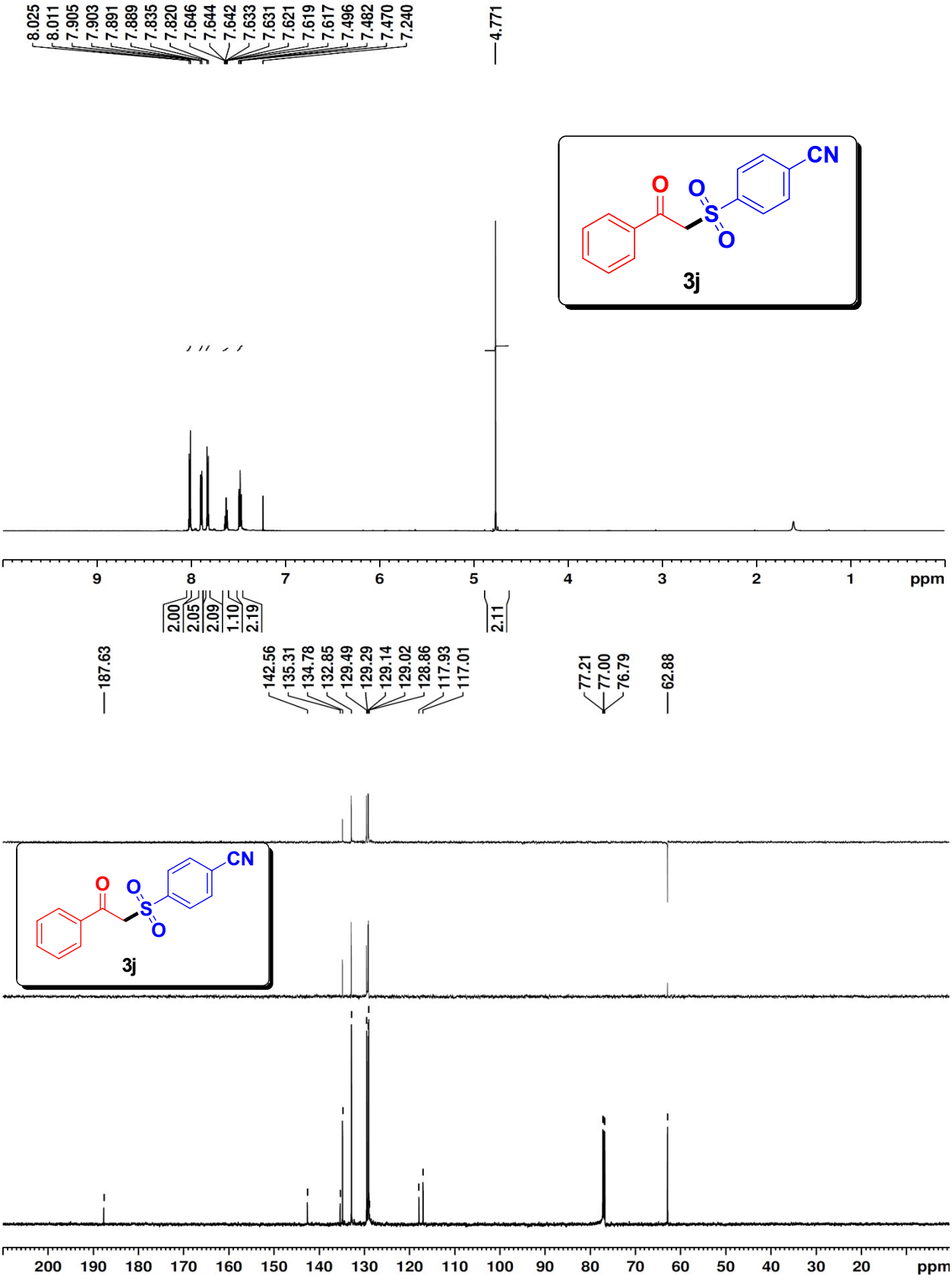
30
20
ppm

^{19}F NMR

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7.644
7.642
7.633
7.631
7.621
7.619
7.617
7.496
7.482
7.470
7.240
4.771
CN
O
O
S
O
3j
9
8
7
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5
4
3
2
1
ppm
2.00
2.05
2.09
1.10
2.19
2.11
187.63
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135.31
134.78
132.85
129.49
129.29
129.14
129.02
128.86
117.93
117.01
77.21
77.00
76.79
62.88
CN
O
O
S
O
3j
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150
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130
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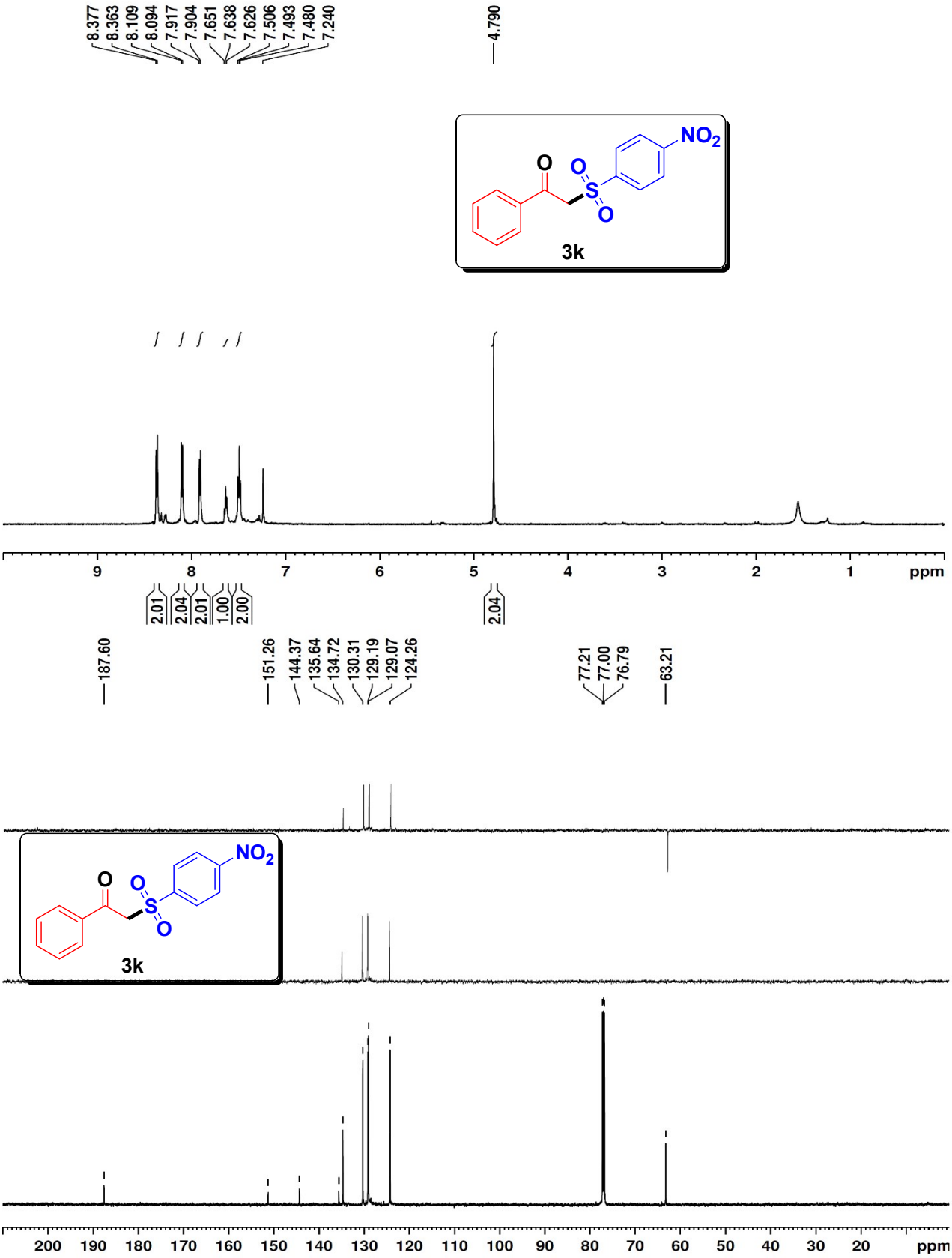
20
ppm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7.638
7.626
7.506
7.493
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7.240
4.790
NO2
O
O
S
O
3k
9
8
7
6
5
4
3
2
1
ppm
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2.04
2.01
1.00
2.00
2.04
187.60
151.26
144.37
135.64
134.72
130.31
129.19
129.07
124.26
77.21
77.00
76.79
63.21
NO2
O
O
S
O
3k
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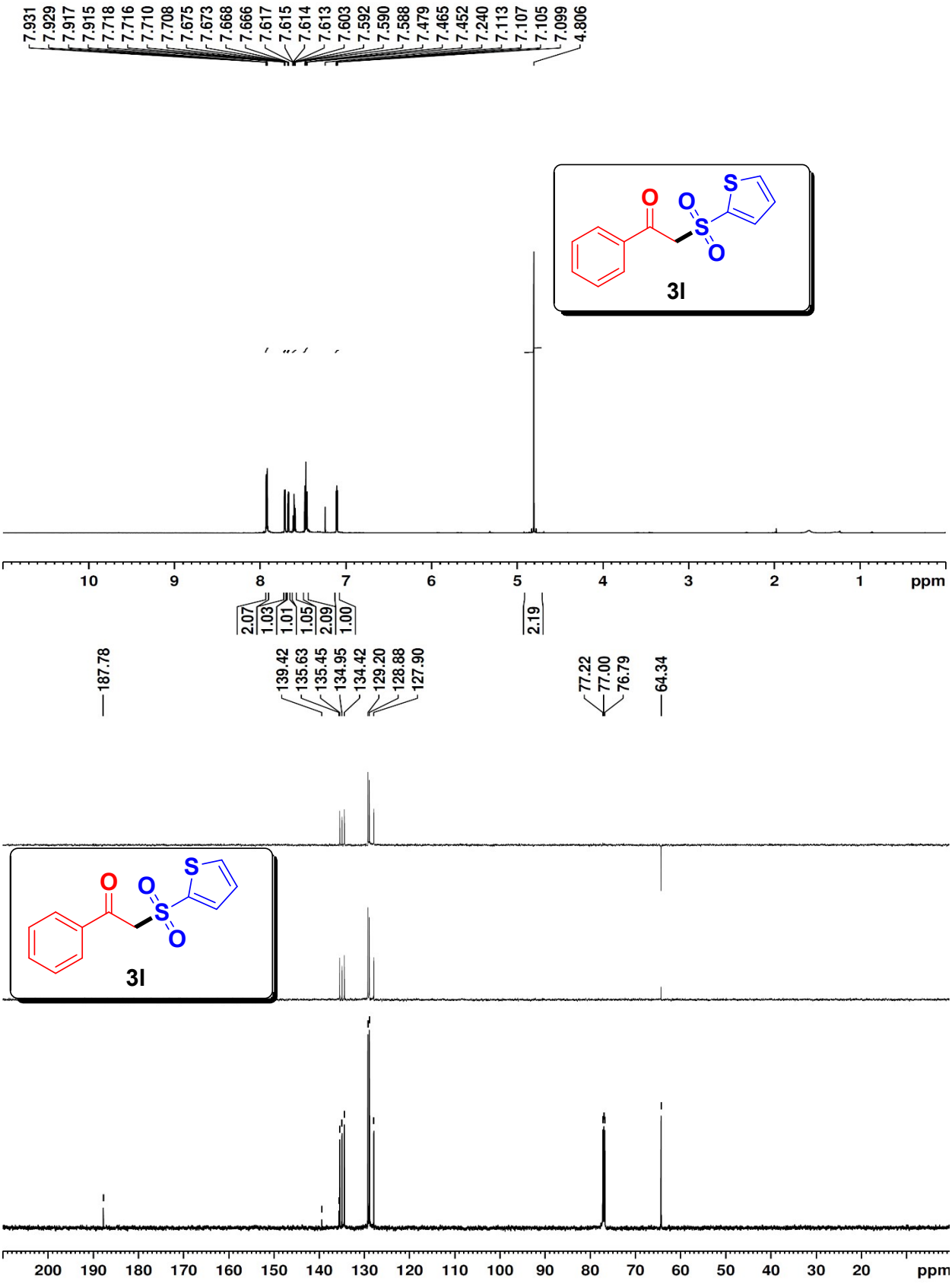
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.675
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7.666
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7.615
7.614
7.613
7.603
7.592
7.590
7.588
7.479
7.465
7.452
7.240
7.113
7.107
7.105
7.099
4.806
3l
10
9
8
7
6
5
4
3
2
1
ppm
2.07
1.03
1.01
1.05
2.09
1.00
2.19
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135.63
135.45
134.95
134.42
129.20
128.88
127.90
77.22
77.00
76.79
64.34
3l
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160
150
140
130
120
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80
70
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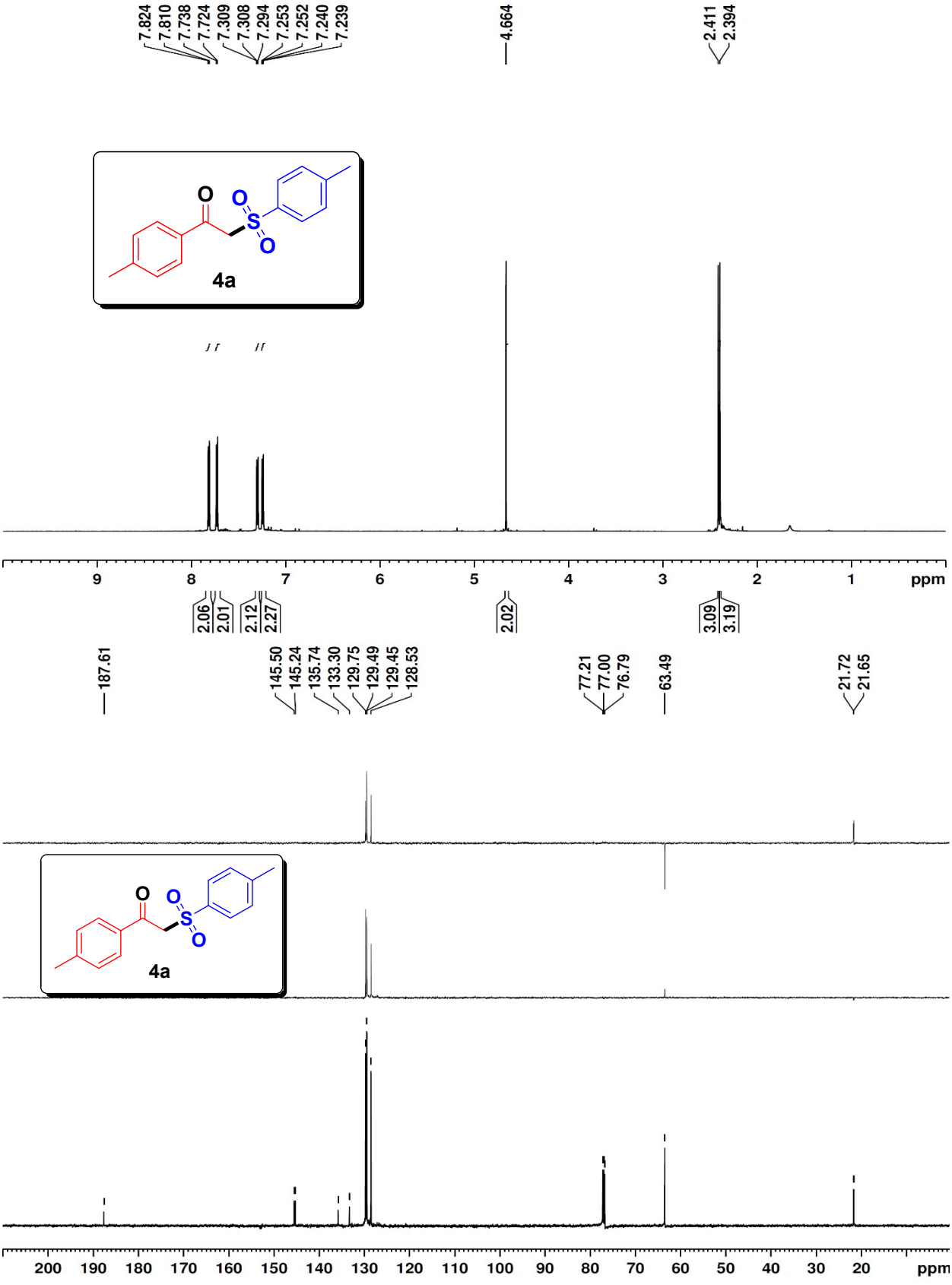
50
40
30
20
ppm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7.252
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2.394
4a
9
8
7
6
5
4
3
2
1
ppm
2.06
2.01
2.12
2.27
2.02
3.09
3.19
187.61
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145.24
135.74
133.30
129.75
129.49
129.45
128.53
77.21
77.00
76.79
63.49
21.72
21.65
4a
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80
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20
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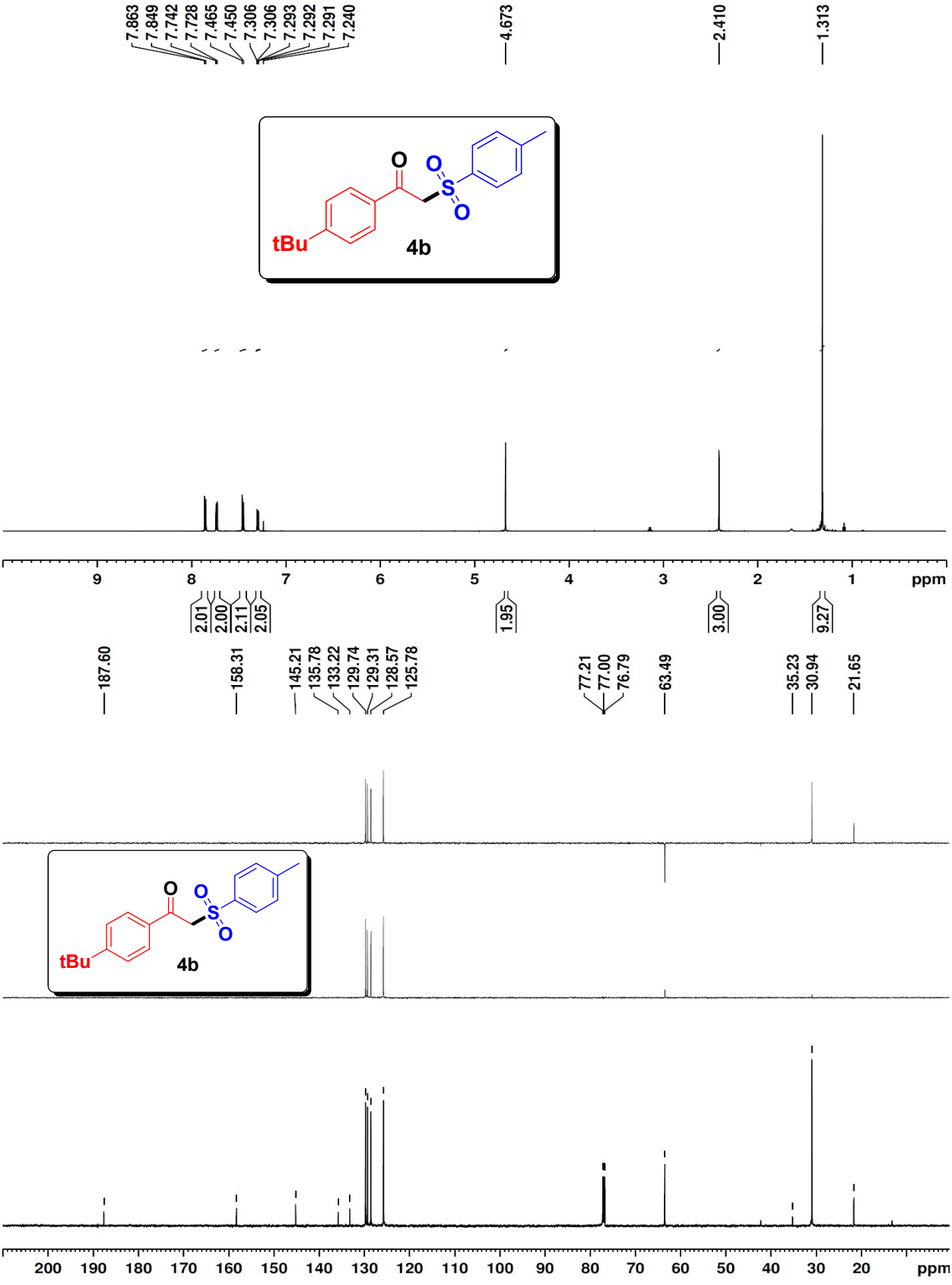
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7.306
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7.292
7.291
7.240
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2.410
1.313
tBu
4b
9
8
7
6
5
4
3
2
1
ppm
2.01
2.00
2.11
2.05
1.95
3.00
9.27
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158.31
145.21
135.78
133.22
129.74
129.31
128.57
125.78
77.21
77.00
76.79
63.49
35.23
30.94
21.65
tBu
4b
200
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160
150
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130
120
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80
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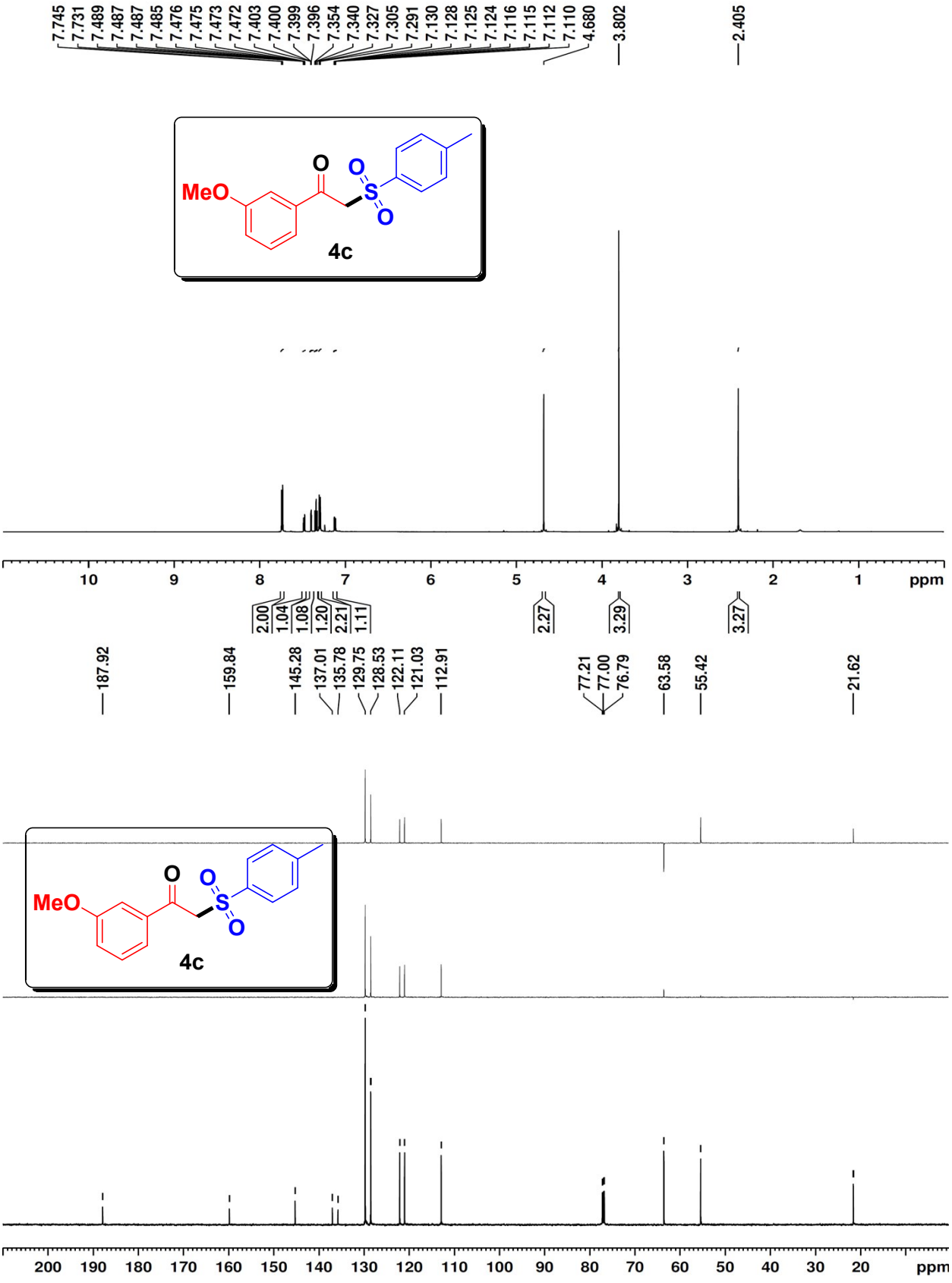
40
30
20
ppm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7.399
7.396
7.354
7.340
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7.291
7.130
7.128
7.125
7.124
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7.115
7.112
7.110
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3.802
2.405
MeO
O
O
S
O
4c
10
9
8
7
6
5
4
3
2
1
ppm
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1.04
1.08
1.20
2.21
1.11
2.27
3.29
3.27
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159.84
145.28
137.01
135.78
129.75
128.53
122.11
121.03
112.91
77.21
77.00
76.79
63.58
55.42
21.62
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O
O
S
O
4c
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150
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100
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80
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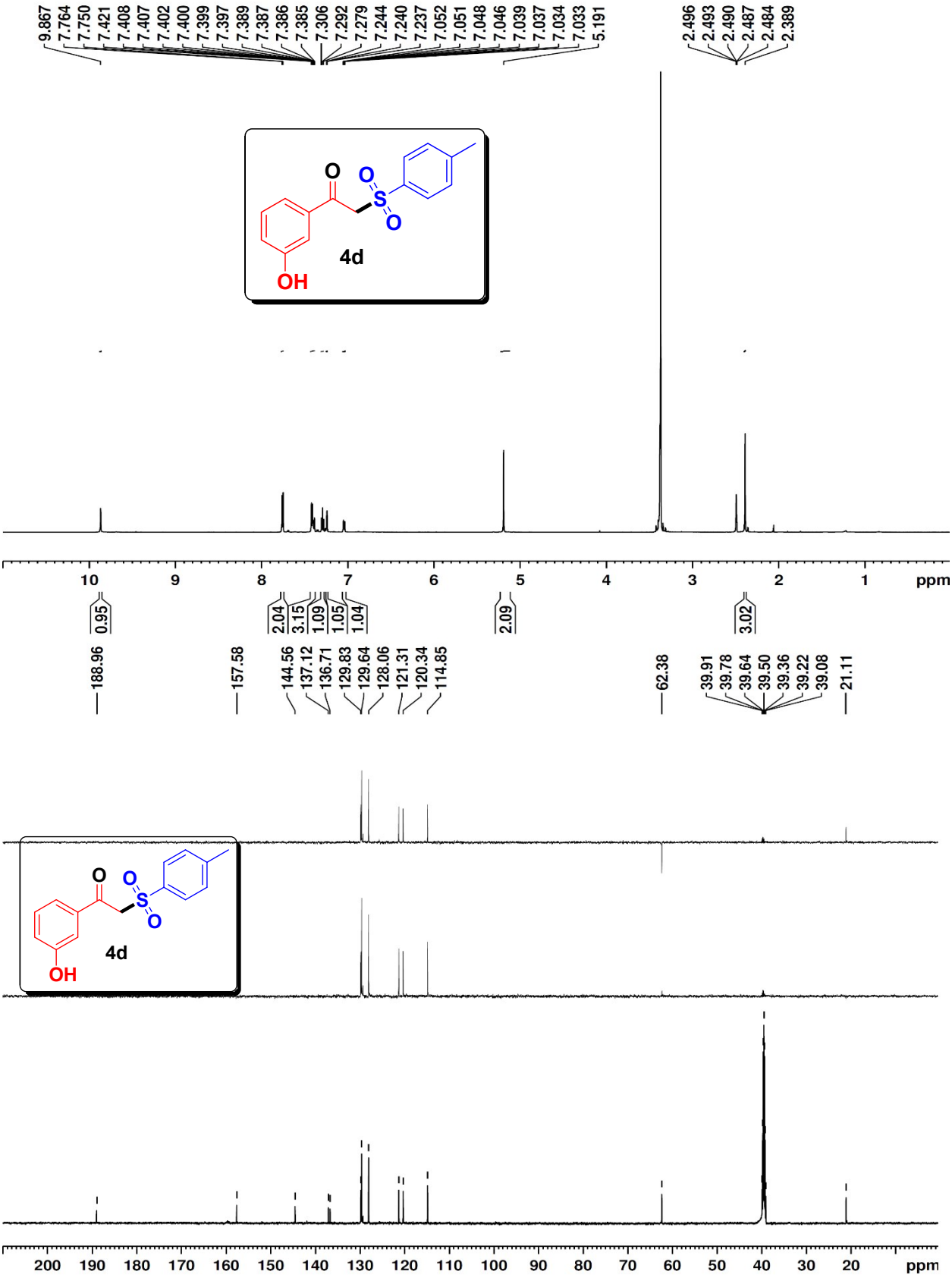
20
ppm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7.385
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7.244
7.240
7.237
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7.051
7.048
7.046
7.039
7.037
7.034
7.033
5.191
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2.493
2.490
2.487
2.484
2.389
O
O
S
O
OH
4d
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1.05
1.04
2.09
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121.31
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62.38
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39.78
39.64
39.50
39.36
39.22
39.08
21.11
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150
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40
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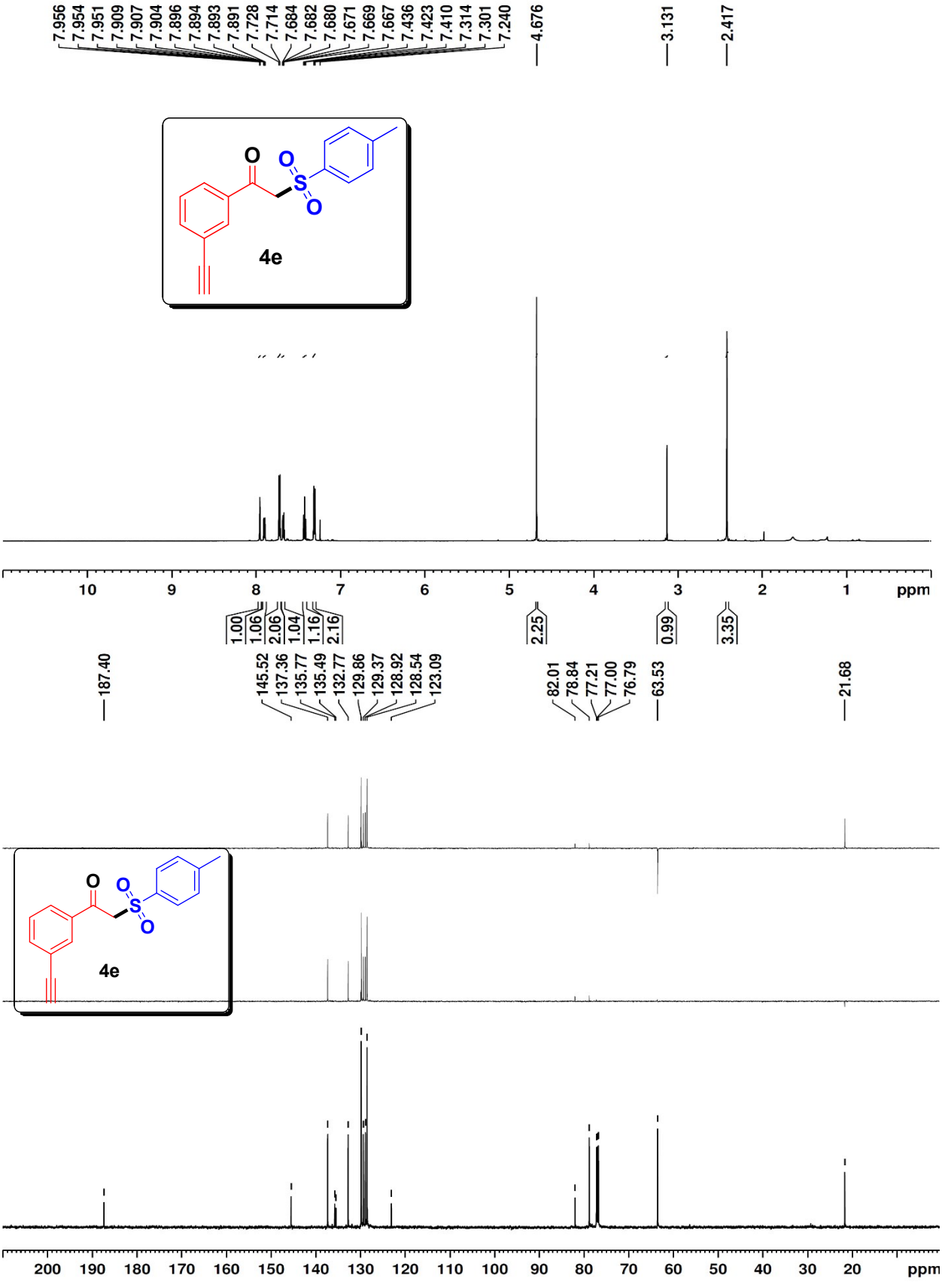
20
ppm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7.682
7.680
7.671
7.669
7.667
7.436
7.423
7.410
7.314
7.301
7.240
4.676
3.131
2.417
4e
10
9
8
7
6
5
4
3
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1.06
2.06
1.04
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2.16
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137.36
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4e
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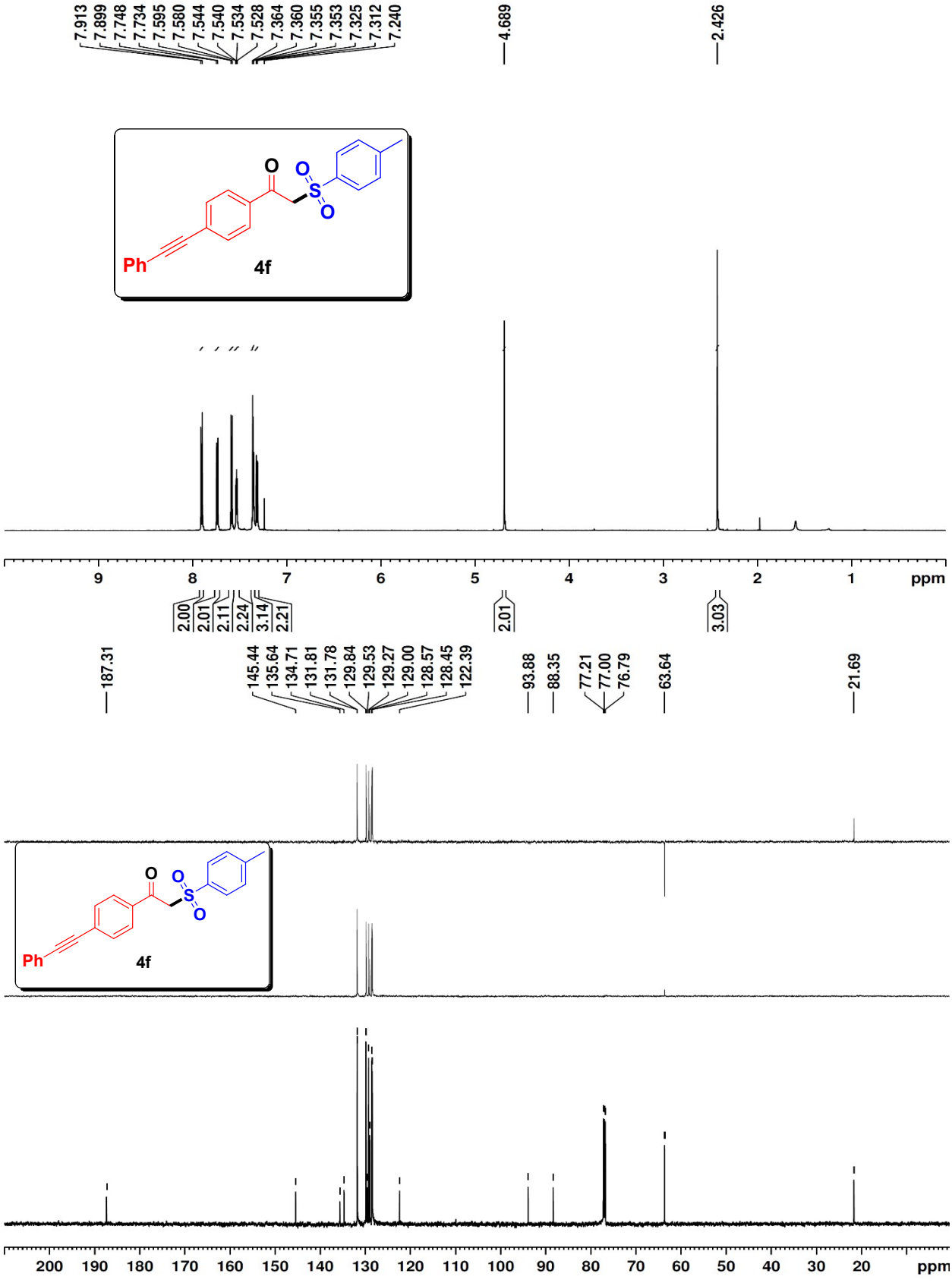
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7.534
7.528
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7.360
7.355
7.353
7.325
7.312
7.240
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2.426
Ph
4f
9
8
7
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5
4
3
2
1
ppm
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2.01
2.11
2.24
3.14
2.21
2.01
3.03
187.31
145.44
135.64
134.71
131.81
131.78
129.84
129.53
129.27
129.00
128.57
128.45
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93.88
88.35
77.21
77.00
76.79
63.64
21.69
Ph
4f
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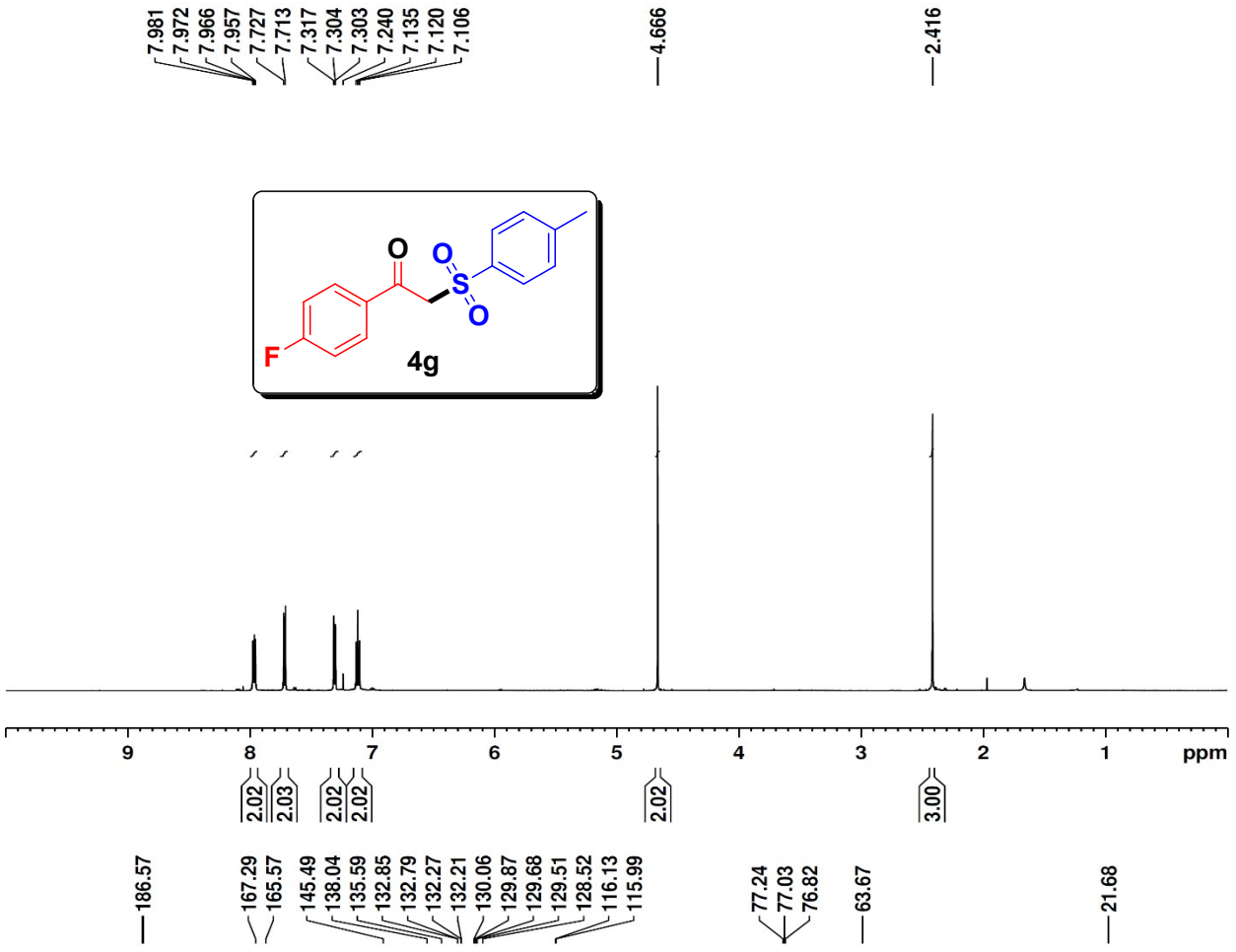
20
ppm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7.303
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7.120
7.106
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4g
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5
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3
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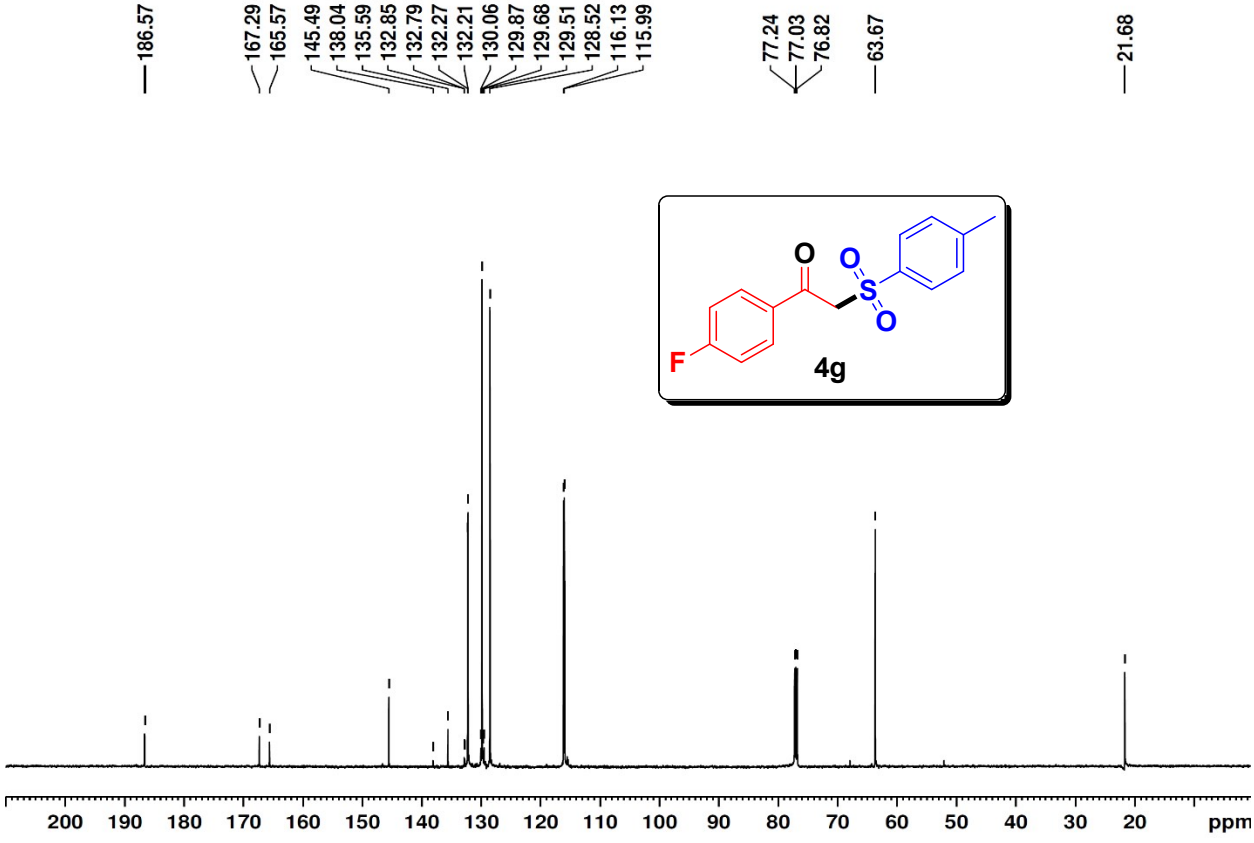
1
ppm
2.02
2.03
2.02
2.02
2.02
3.00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129.51
128.52
116.13
115.99
77.24
77.03
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63.67
21.68
4g
200
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100
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80
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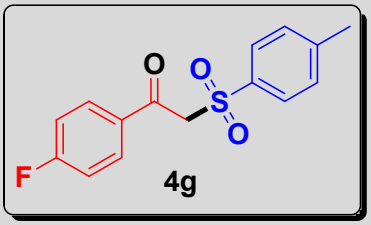
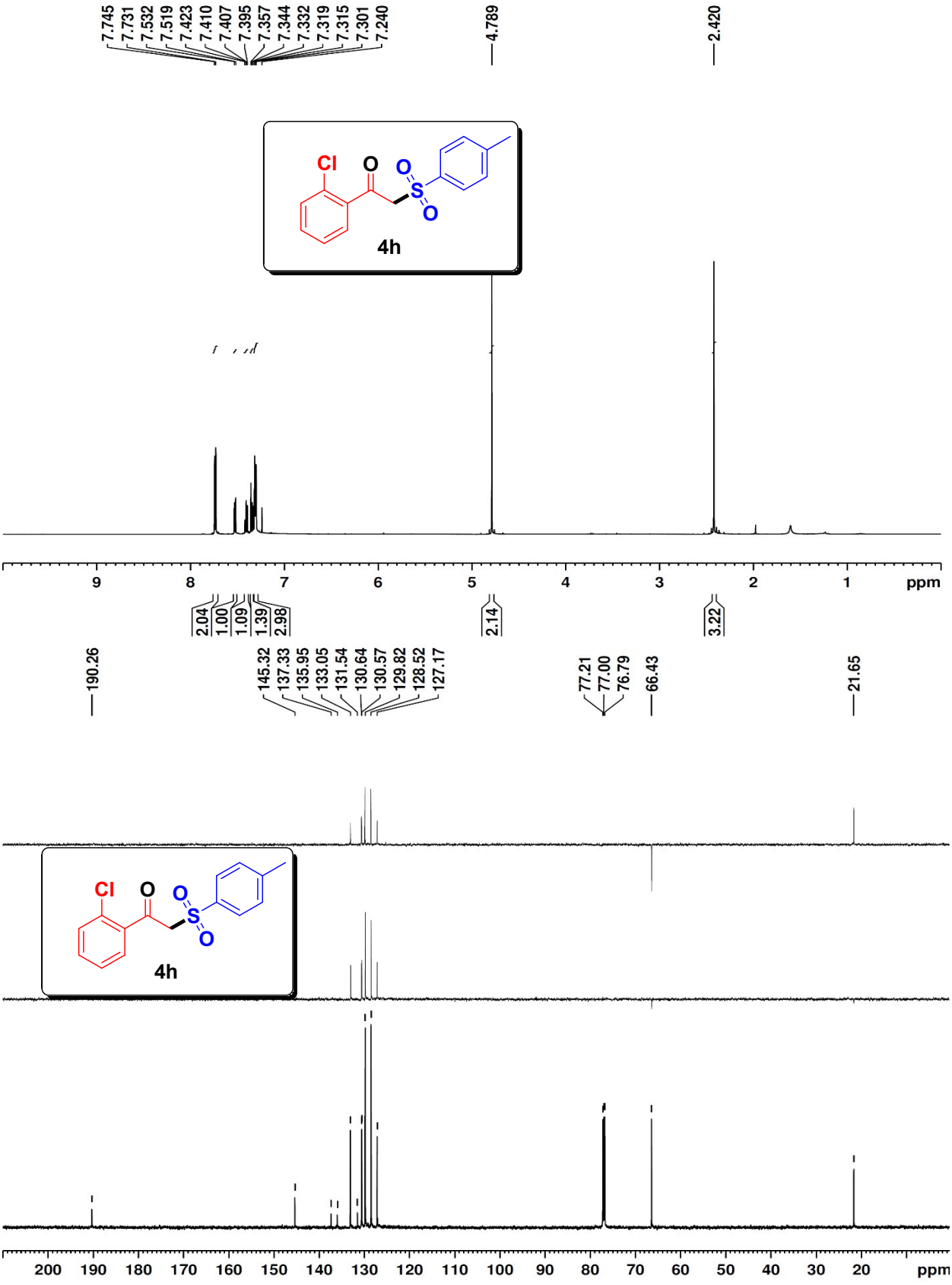
60
50
40
30
20
ppm

^{19}F NMR

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7.301
7.240
4.789
2.420
Cl
O
O
S
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4h
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1.00
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1.39
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4h
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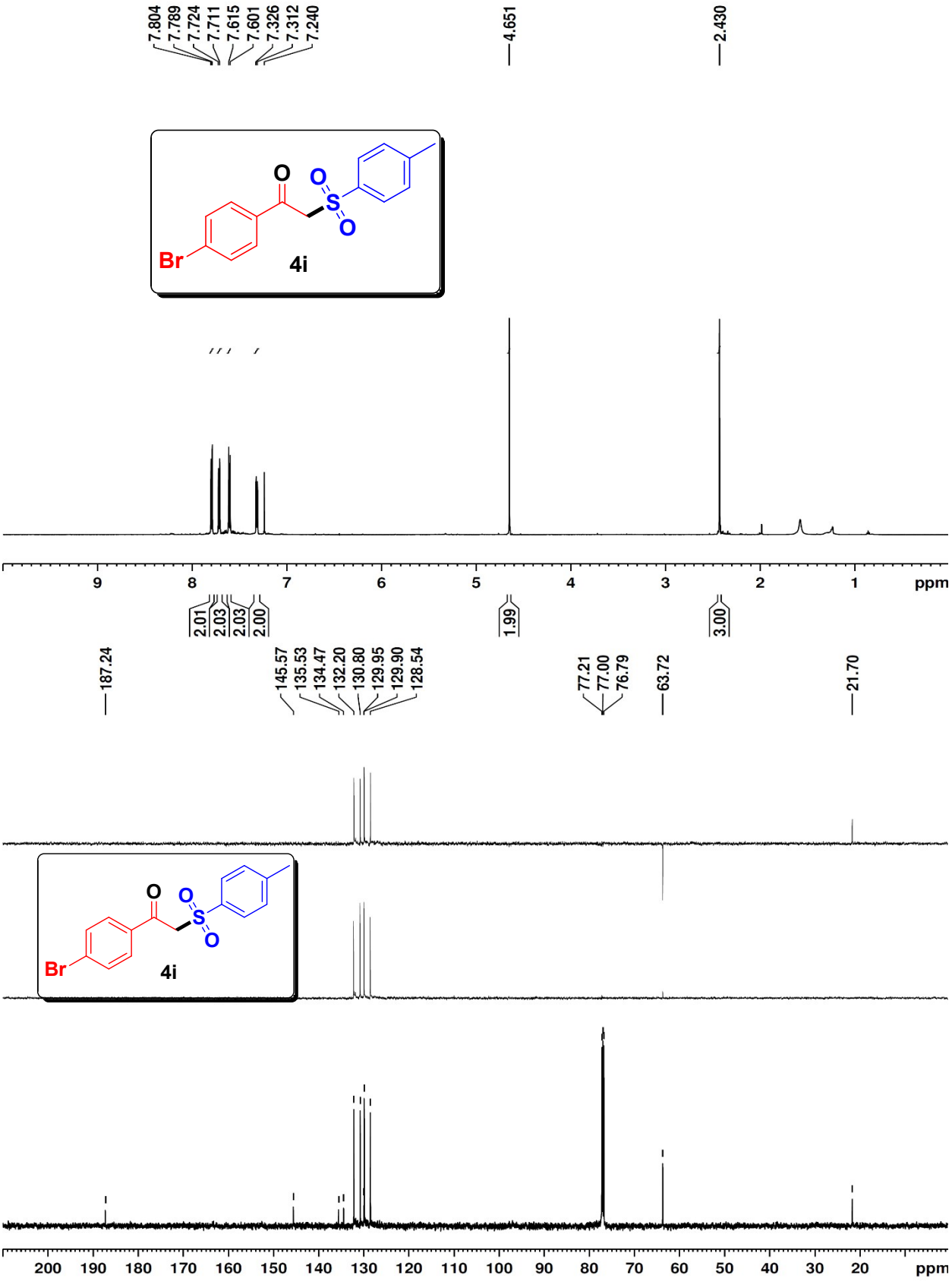
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4.651
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Br
4i
9
8
7
6
5
4
3
2
1
ppm
2.01
2.03
2.03
2.00
1.99
3.00
187.24
145.57
135.53
134.47
132.20
130.80
129.95
129.90
128.54
77.21
77.00
76.79
63.72
21.70
Br
4i
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150
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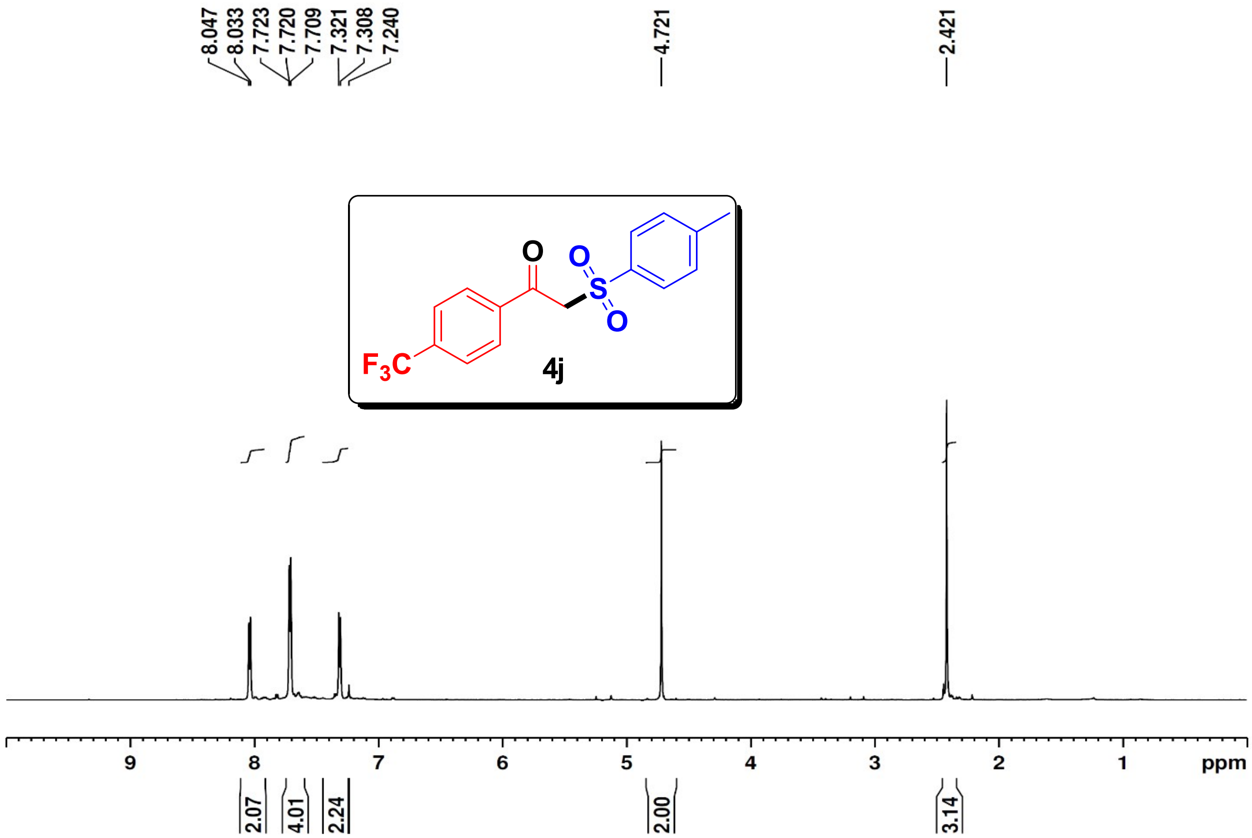
30
20
ppm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8.033
7.723
7.720
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7.321
7.308
7.240
4.721
2.421
O
O
S
O
F3C
4j
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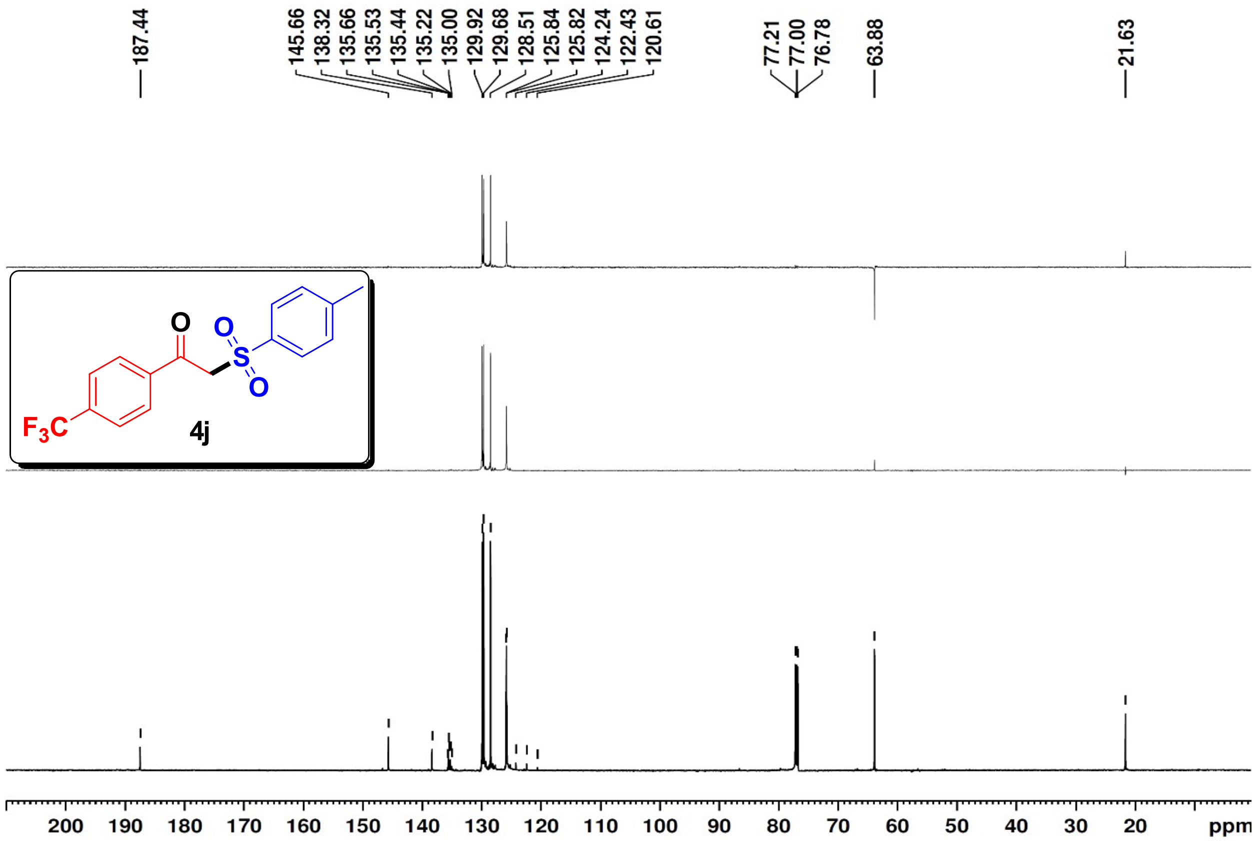
8
7
6
5
4
3
2
1
ppm
2.07
4.01
2.24
2.00
3.14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125.84
125.82
124.24
122.43
120.61
77.21
77.00
76.78
63.88
21.63
O
O
S
O
F3C
4j
200
190
180
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140
130
120
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100
90
80
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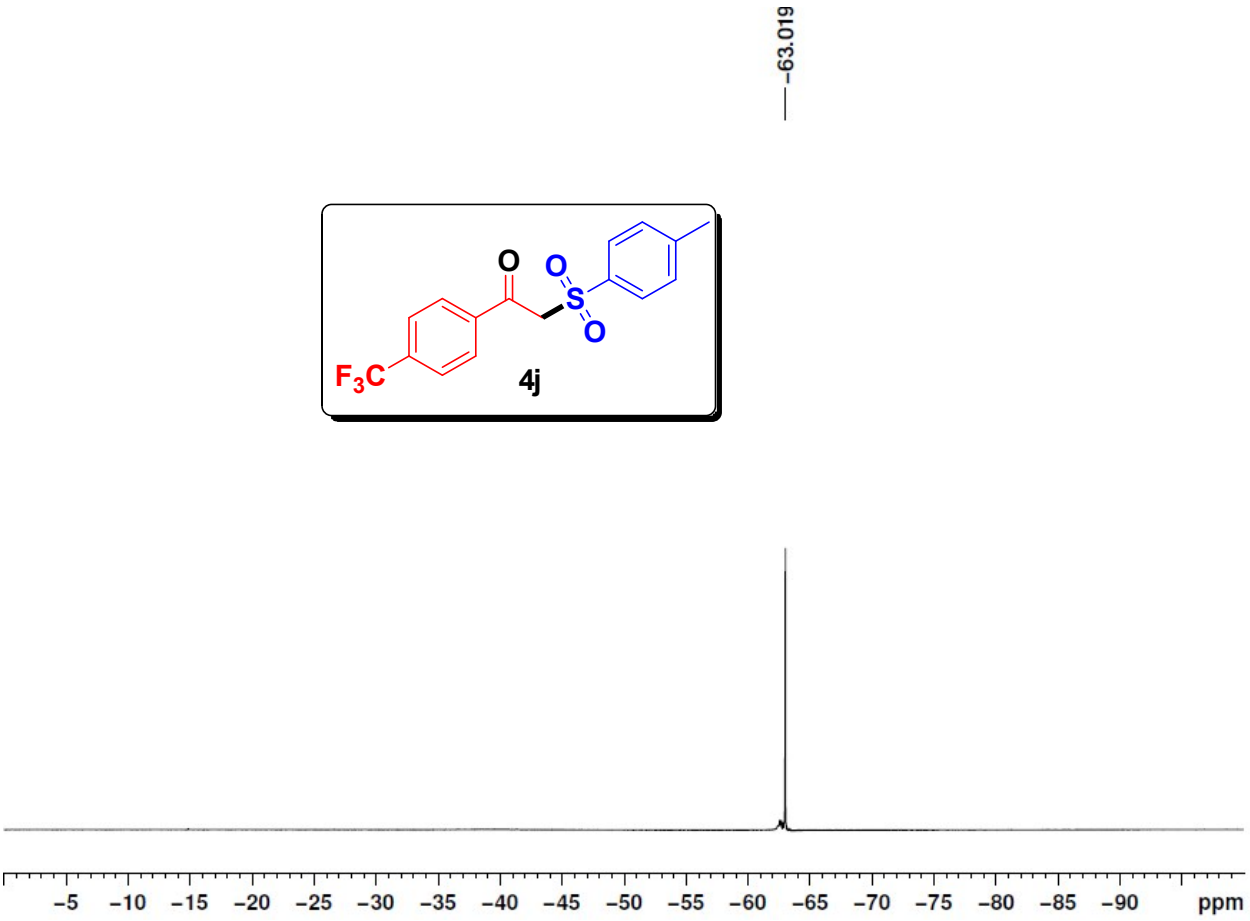
60
50
40
30
20
ppm

^{19}F NMR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O
O
S
O
F3C
4j
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−10
−15
−20
−25
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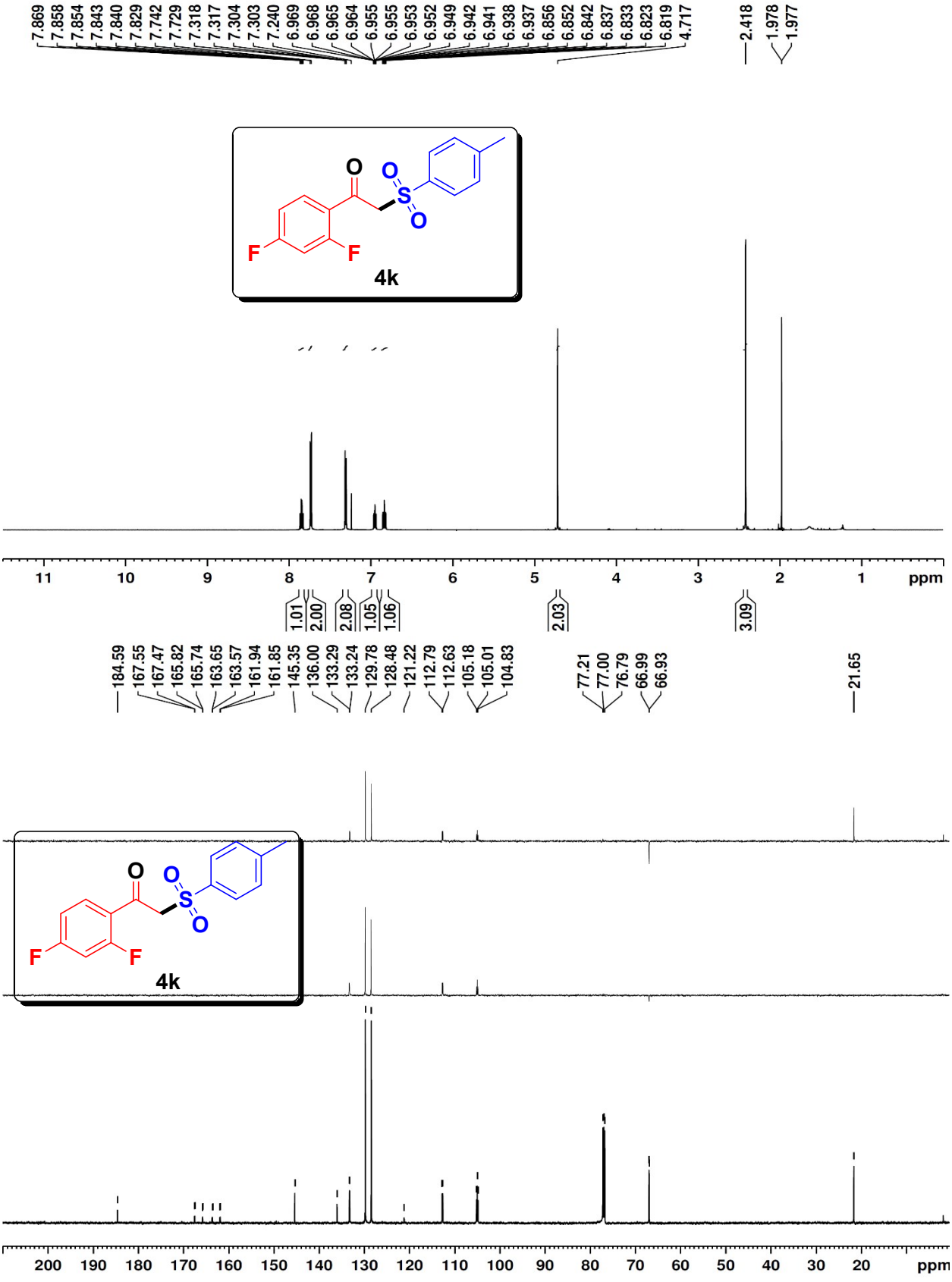
−40
−45
−50
−55
−60
−65
−70
−75
−80
−85
−90
ppm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7.303
7.240
6.969
6.968
6.965
6.964
6.955
6.955
6.953
6.952
6.949
6.942
6.941
6.938
6.937
6.856
6.852
6.842
6.837
6.833
6.823
6.819
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2.418
1.978
1.977
4k
11
10
9
8
7
6
5
4
3
2
1
ppm
1.01
2.00
2.08
1.05
1.06
2.03
3.09
184.59
167.55
167.47
165.82
165.74
163.65
163.57
161.94
161.85
145.35
136.00
133.29
133.24
129.78
128.48
121.22
112.79
112.63
105.18
105.01
104.83
77.21
77.00
76.79
66.99
66.93
21.65
4k
200
190
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100
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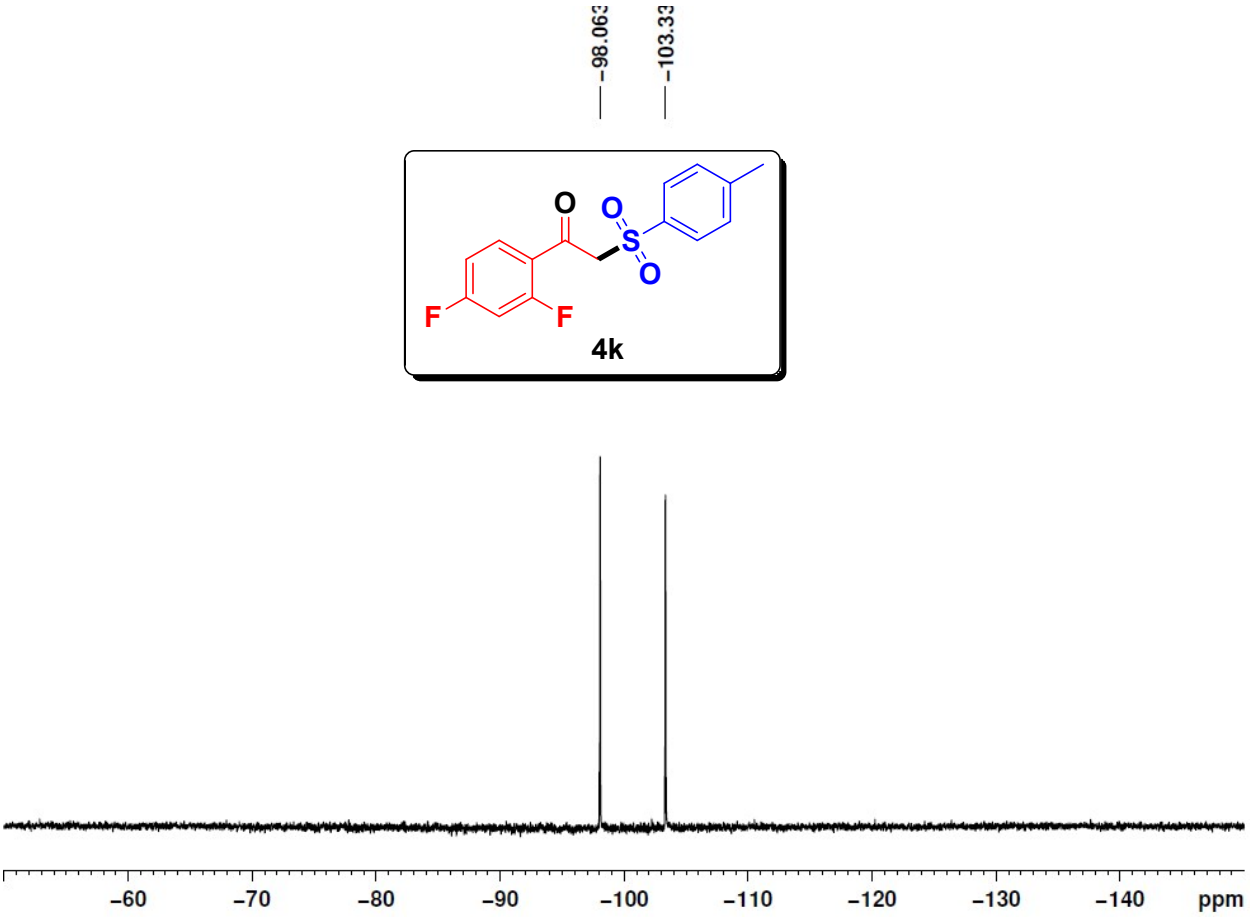
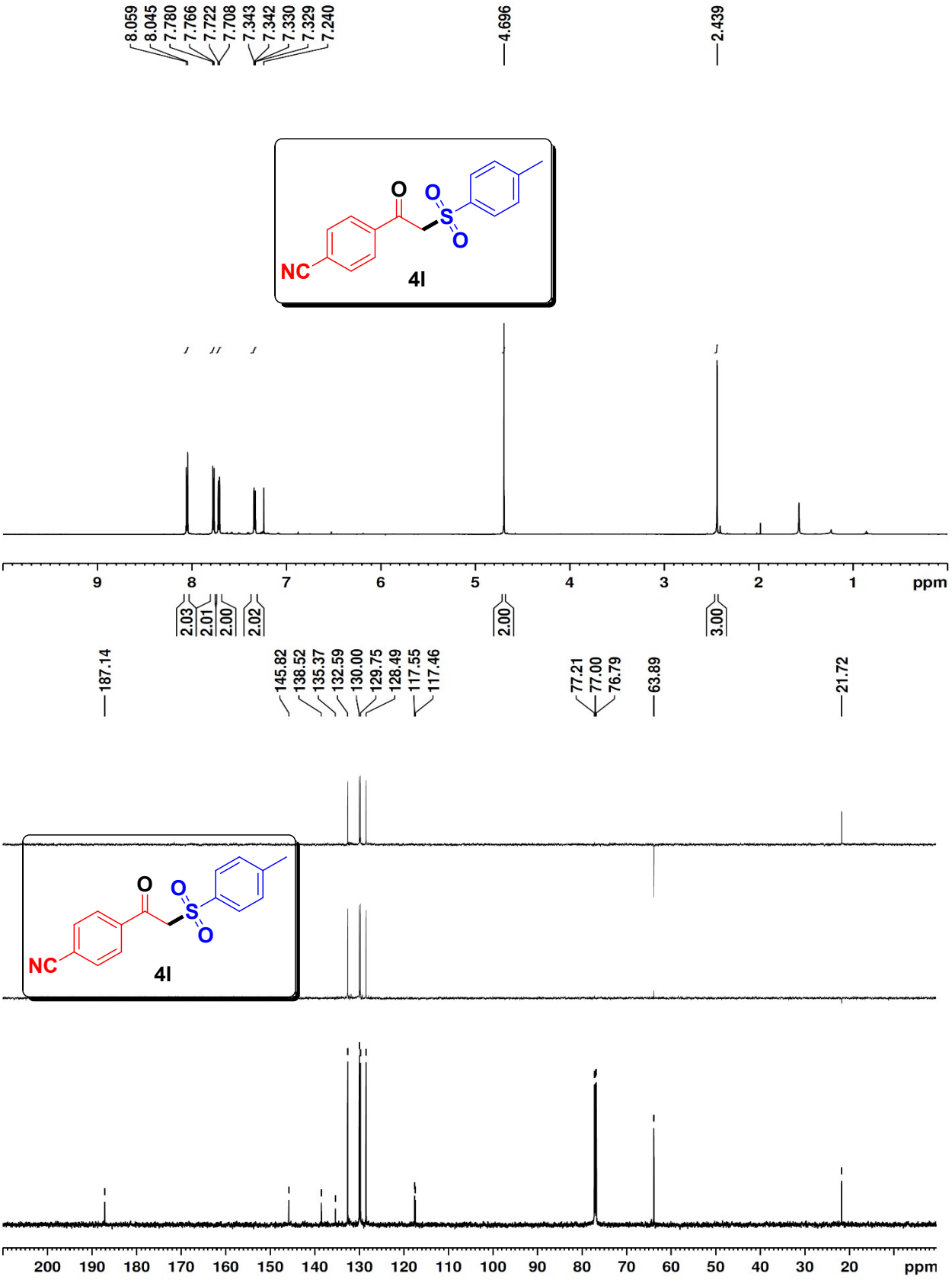
40
30
20
ppm

^{19}F NMR

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7.708
7.343
7.342
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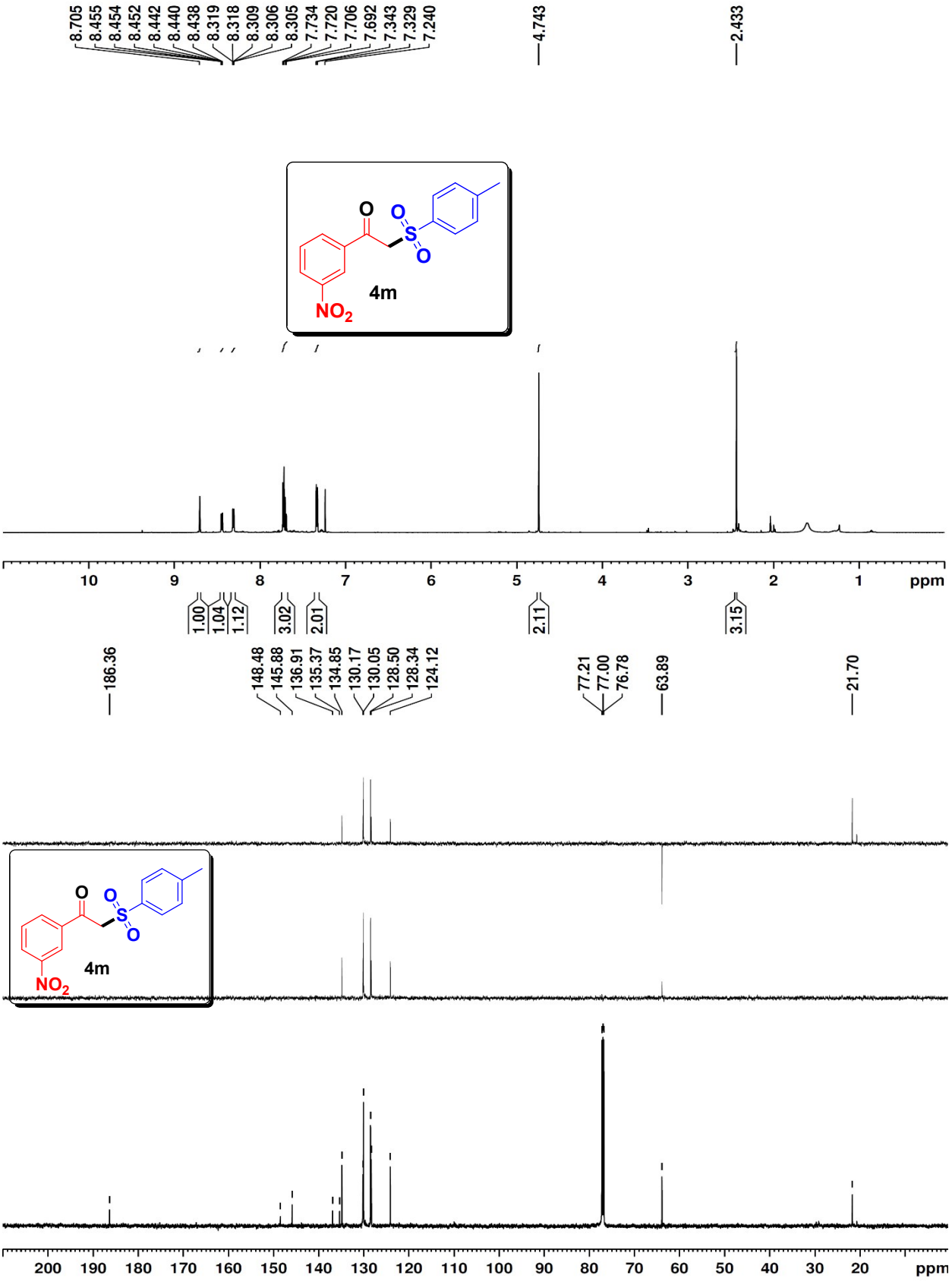
7.329
7.240
4.696
2.439
O
O
S
O
NC
4l
9
8
7
6
5
4
3
2
1
ppm
2.03
2.01
2.00
2.02
2.00
3.00
187.14
145.82
138.52
135.37
132.59
130.00
129.75
128.49
117.55
117.46
77.21
77.00
76.79
63.89
21.72
O
O
S
O
NC
4l
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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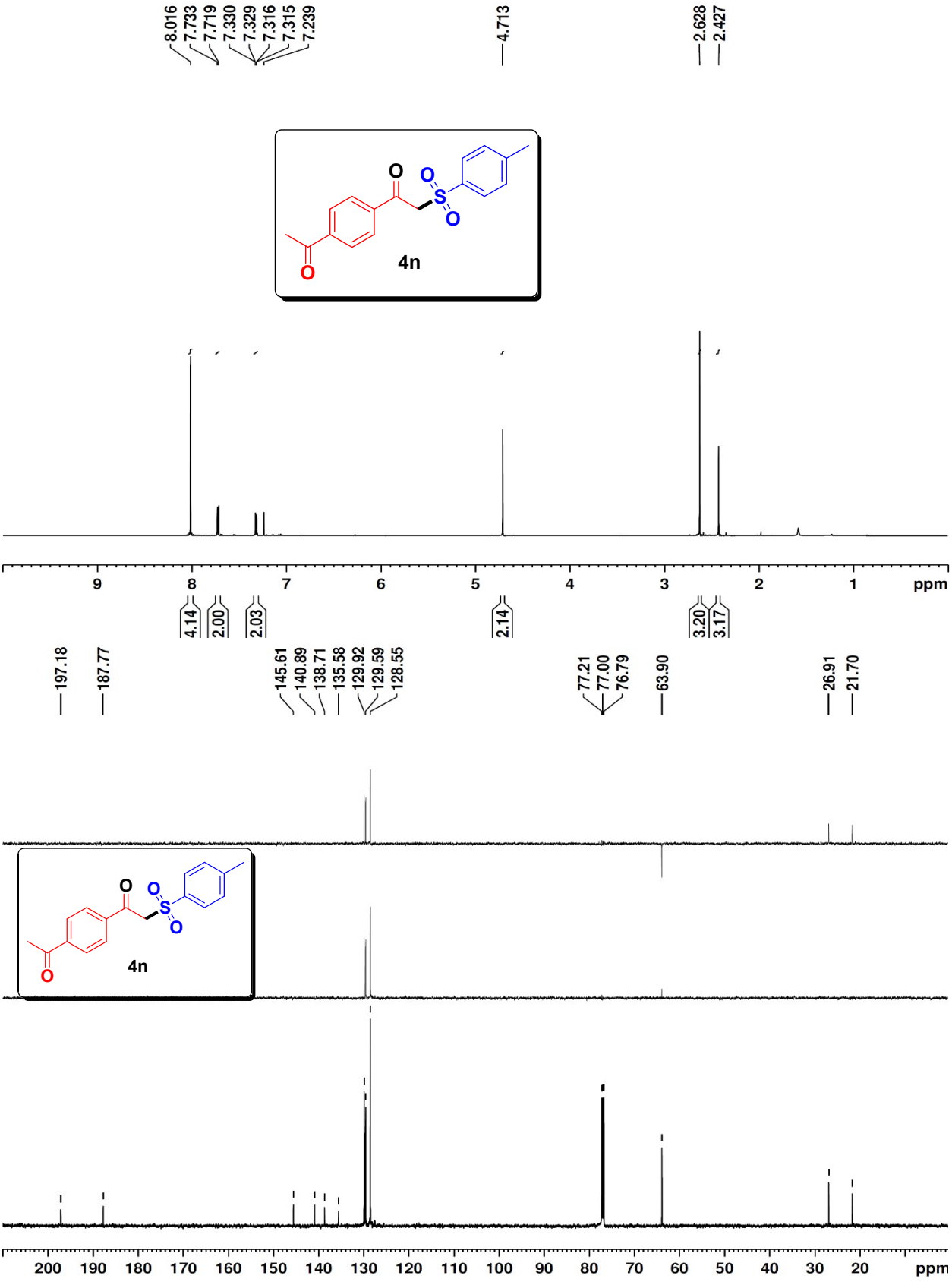
8.454
8.452
8.442
8.440
8.438
8.319
8.318
8.309
8.306
8.305
7.734
7.720
7.706
7.692
7.343
7.329
7.240
4.743
2.433
4m
NO2
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.04
1.12
3.02
2.01
2.11
3.15
186.36
148.48
145.88
136.91
135.37
134.85
130.17
130.05
128.50
128.34
124.12
77.21
77.00
76.78
63.89
21.70
4m
NO2
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.329
7.316
7.315
7.239
4.713
2.628
2.427
4n
9
8
7
6
5
4
3
2
1
ppm
4.14
2.00
2.03
2.14
3.20
3.17
197.18
187.77
145.61
140.89
138.71
135.58
129.92
129.59
128.55
77.21
77.00
76.79
63.90
26.91
21.70
4n
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
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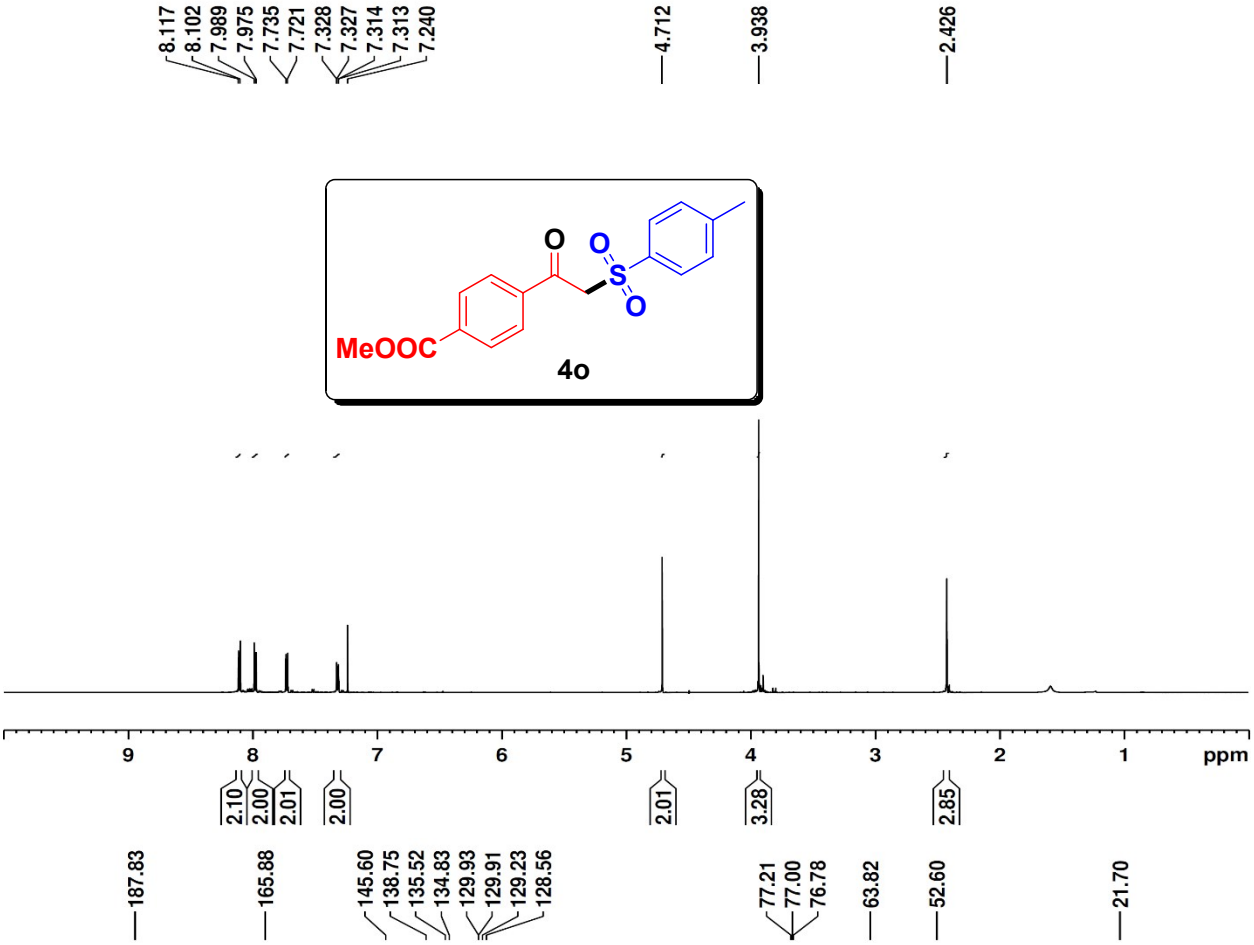
30
20
ppm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7.721
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7.327
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7.313
7.240
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3.938
2.426
MeOOC
4o
9
8
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6
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4
3
2
1
ppm
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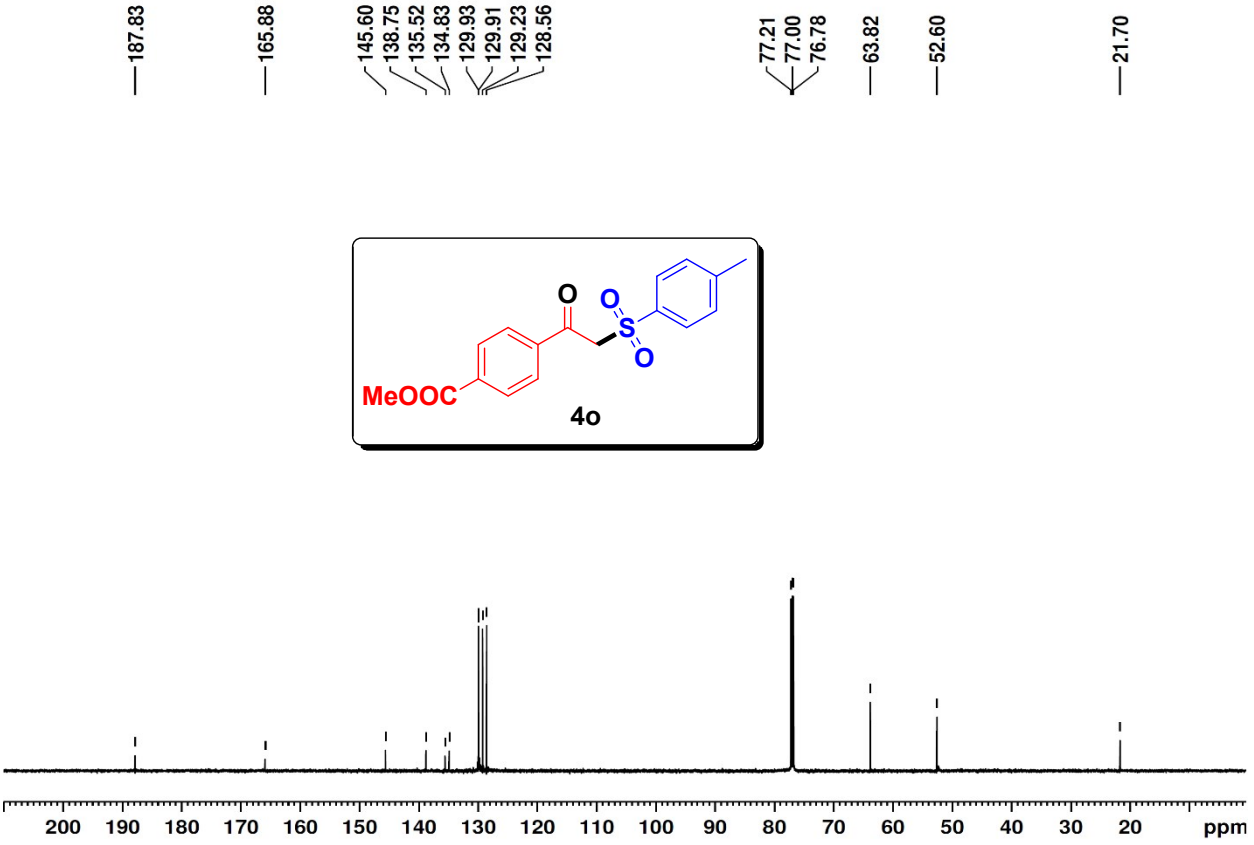
2.00
2.01
2.00
2.01
3.28
2.85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134.83
129.93
129.91
129.23
128.56
77.21
77.00
76.78
63.82
52.60
21.70
MeOOC
4o
200
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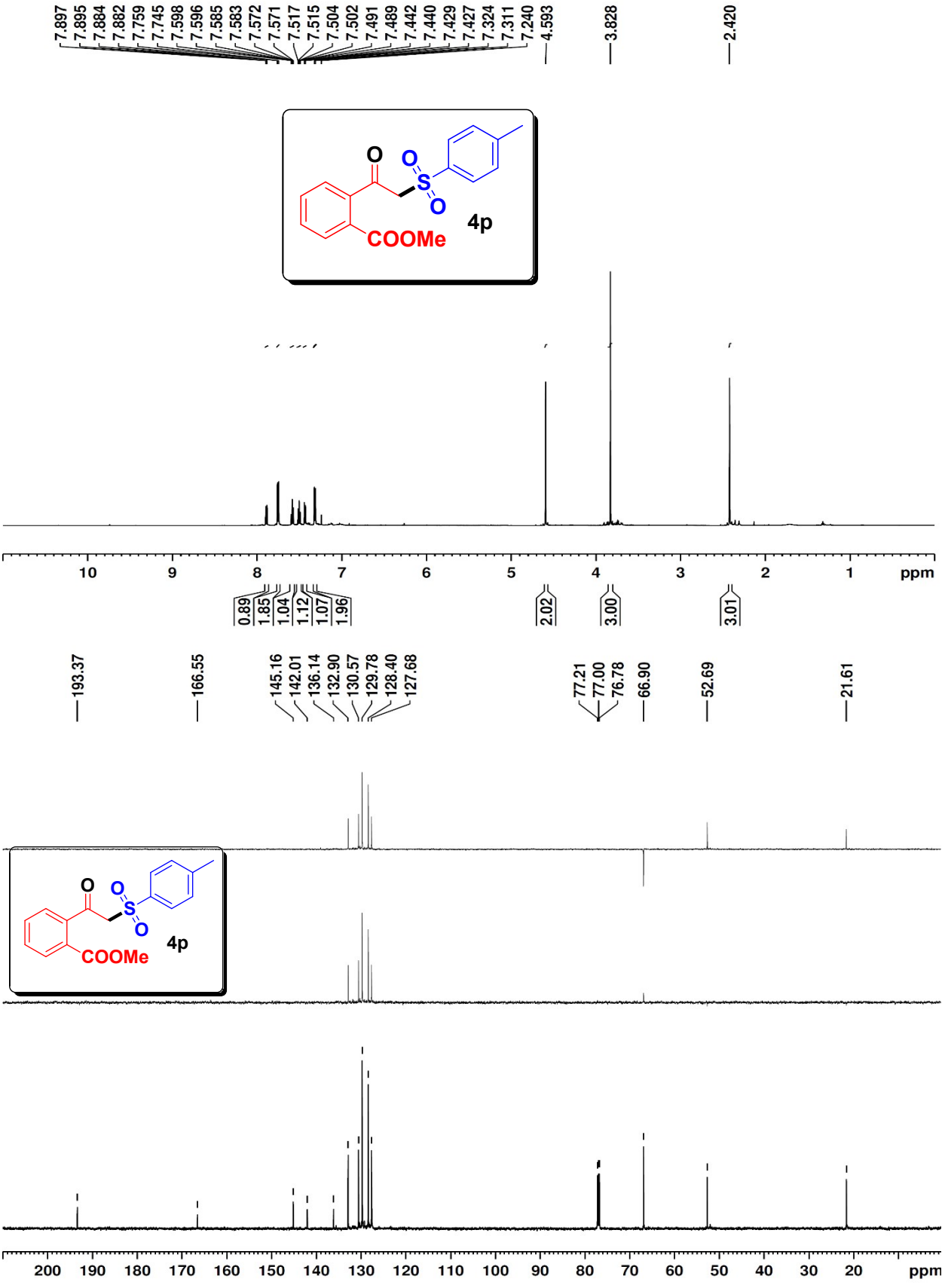
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.745
7.598
7.596
7.585
7.583
7.572
7.571
7.517
7.515
7.504
7.502
7.491
7.489
7.442
7.440
7.429
7.427
7.324
7.311
7.240
4.593
3.828
2.420
O
O
S
O
COOMe
4p
10
9
8
7
6
5
4
3
2
1
ppm
0.89
1.85
1.04
1.12
1.07
1.96
2.02
3.00
3.01
193.37
166.55
145.16
142.01
136.14
132.90
130.57
129.78
128.40
127.68
77.21
77.00
76.78
66.90
52.69
21.61
O
O
S
O
COOMe
4p
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160
150
140
130
120
110
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80
70
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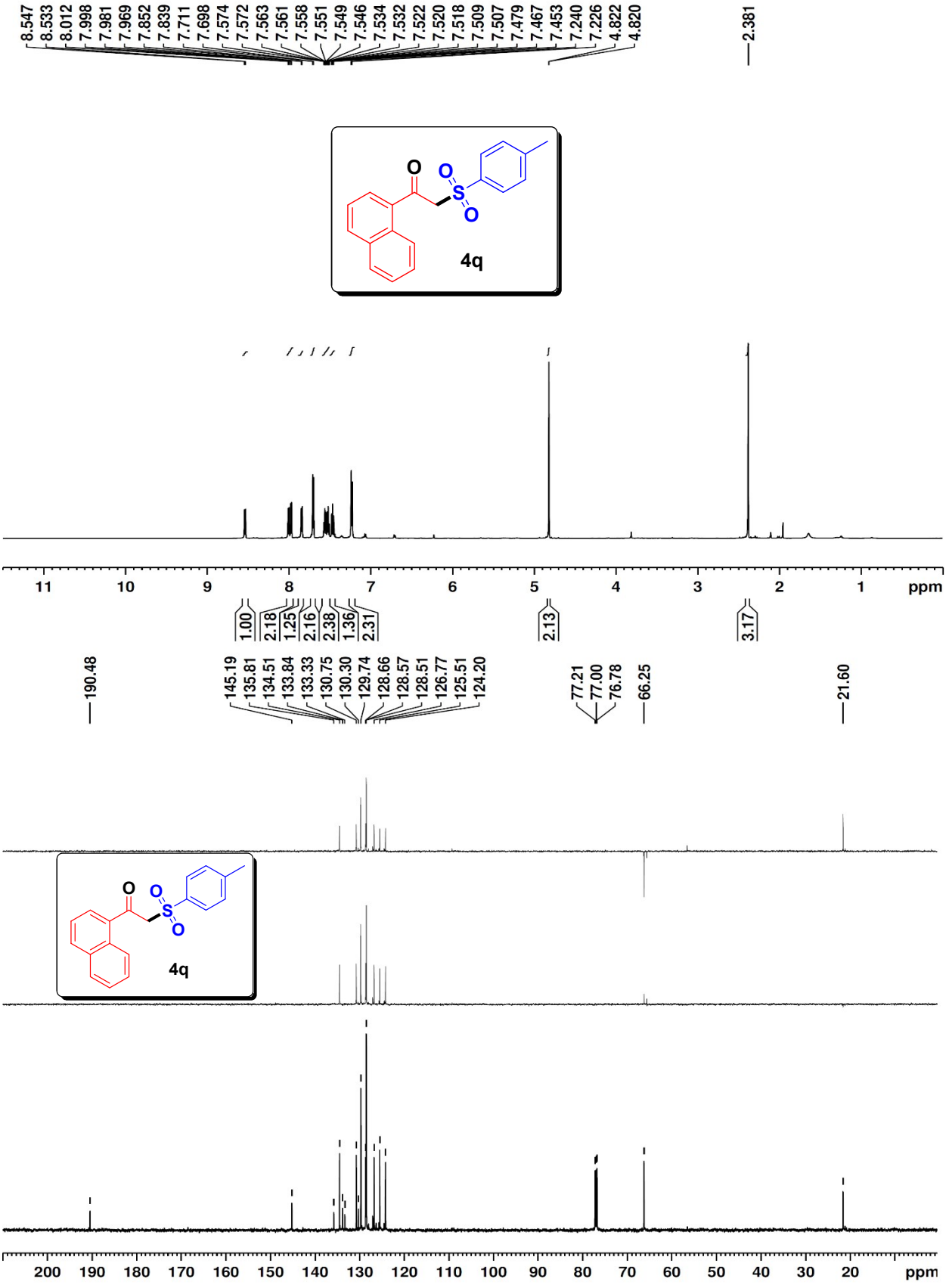
50
40
30
20
ppm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7.572
7.563
7.561
7.558
7.551
7.549
7.546
7.534
7.532
7.522
7.520
7.518
7.509
7.507
7.479
7.467
7.453
7.240
7.226
4.822
4.820
2.381
4q
11
10
9
8
7
6
5
4
3
2
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ppm
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1.25
2.16
2.38
1.36
2.31
2.13
3.17
190.48
145.19
135.81
134.51
133.84
133.33
130.75
130.30
129.74
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128.57
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126.77
125.51
124.20
77.21
77.00
76.78
66.25
21.60
4q
200
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180
170
160
150
140
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80
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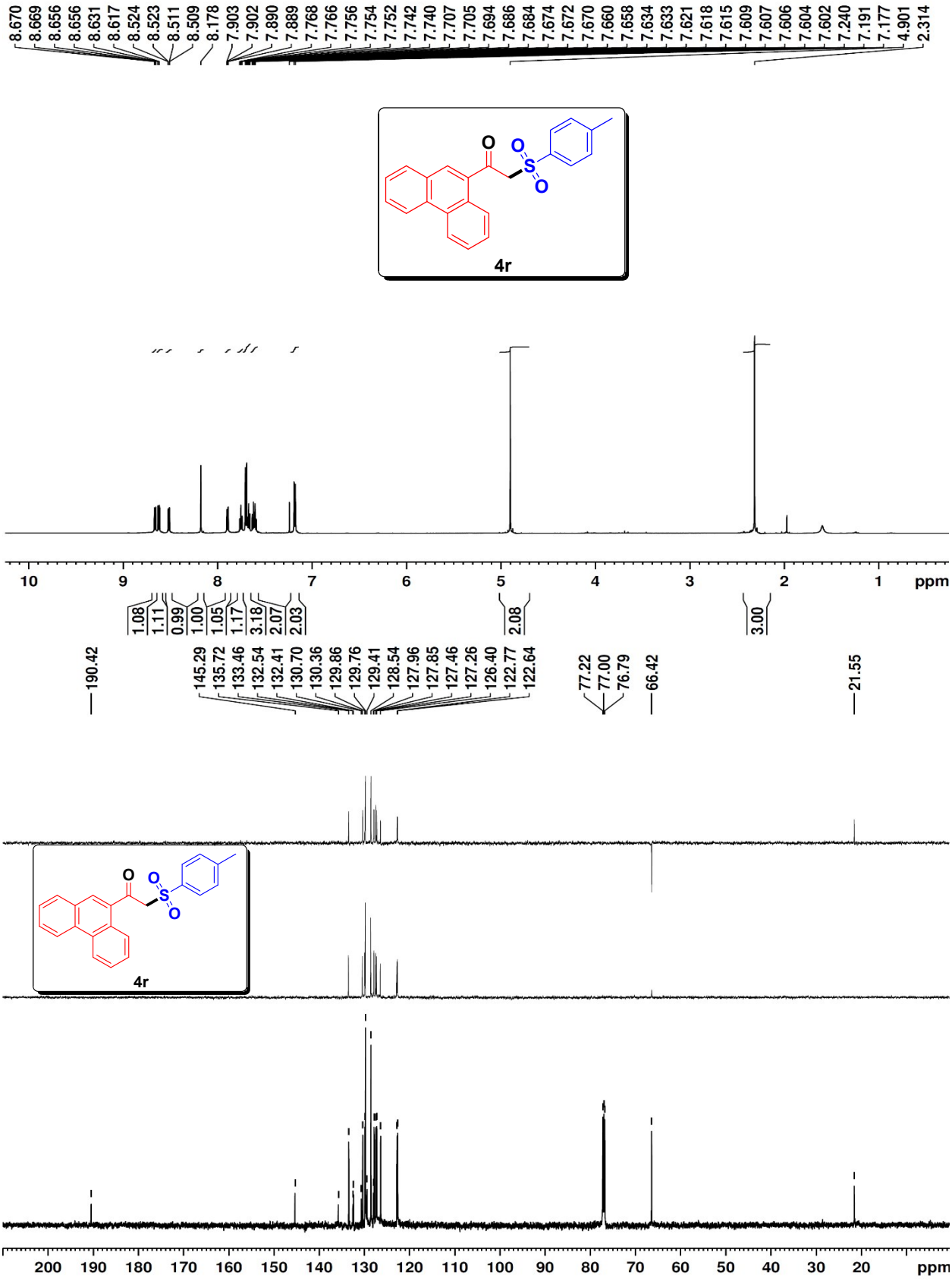
30
20
ppm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7.768
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7.754
7.752
7.742
7.740
7.707
7.705
7.694
7.686
7.684
7.674
7.672
7.670
7.660
7.658
7.634
7.633
7.621
7.618
7.615
7.609
7.607
7.606
7.604
7.602
7.240
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7.177
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2.314
4r
10
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8
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6
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4
3
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1.11
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1.17
3.18
2.07
2.03
2.08
3.00
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135.72
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132.54
132.41
130.70
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129.41
128.54
127.96
127.85
127.46
127.26
126.40
122.77
122.64
77.22
77.00
76.79
66.42
21.55
4r
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150
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130
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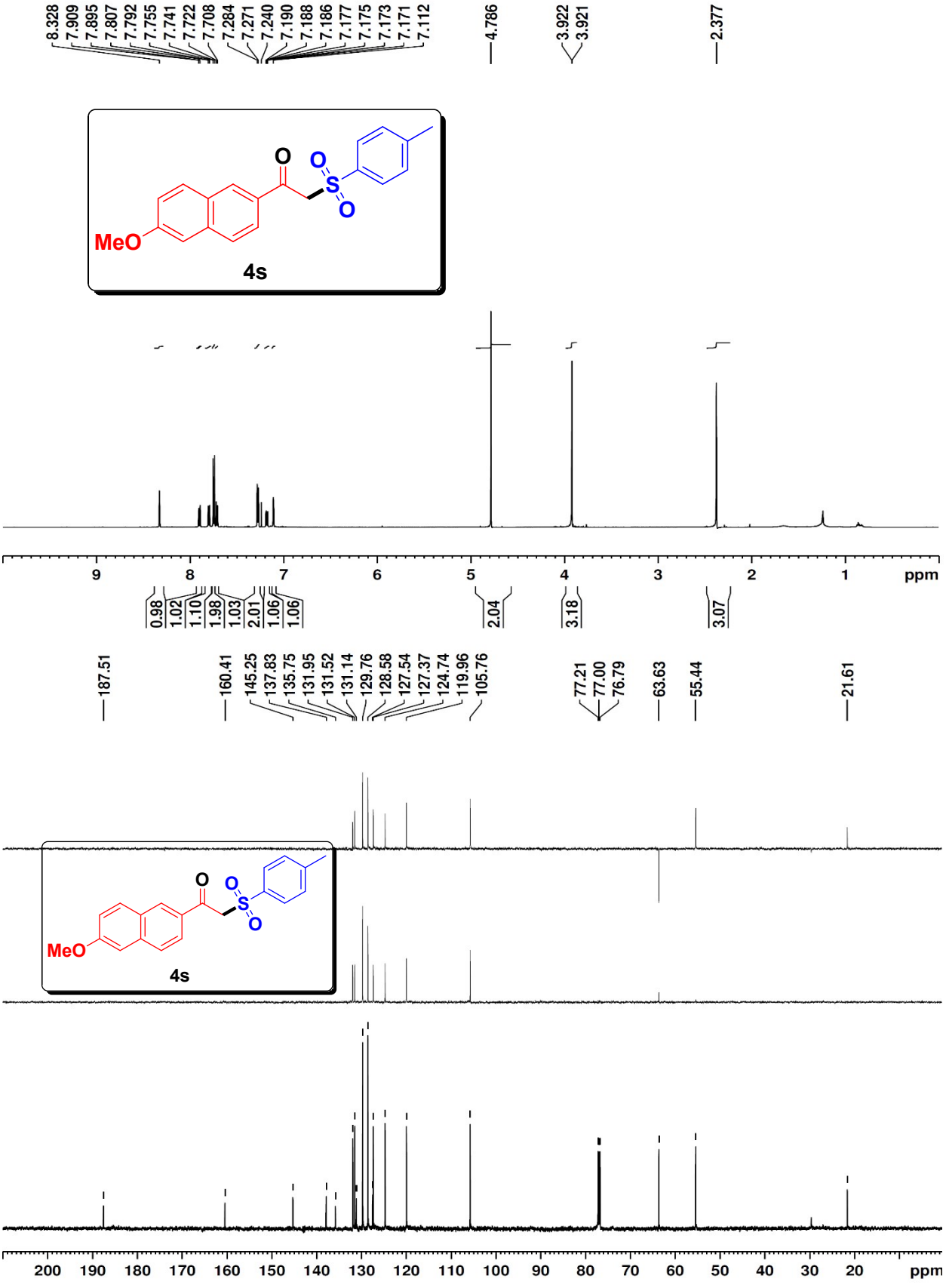
ppm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7.271
7.240
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7.188
7.186
7.177
7.175
7.173
7.171
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3.921
2.377
MeO
4s
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ppm
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1.10
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1.03
2.01
1.06
1.06
2.04
3.18
3.07
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137.83
135.75
131.95
131.52
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129.76
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127.54
127.37
124.74
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77.00
76.79
63.63
55.44
21.61
MeO
4s
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120
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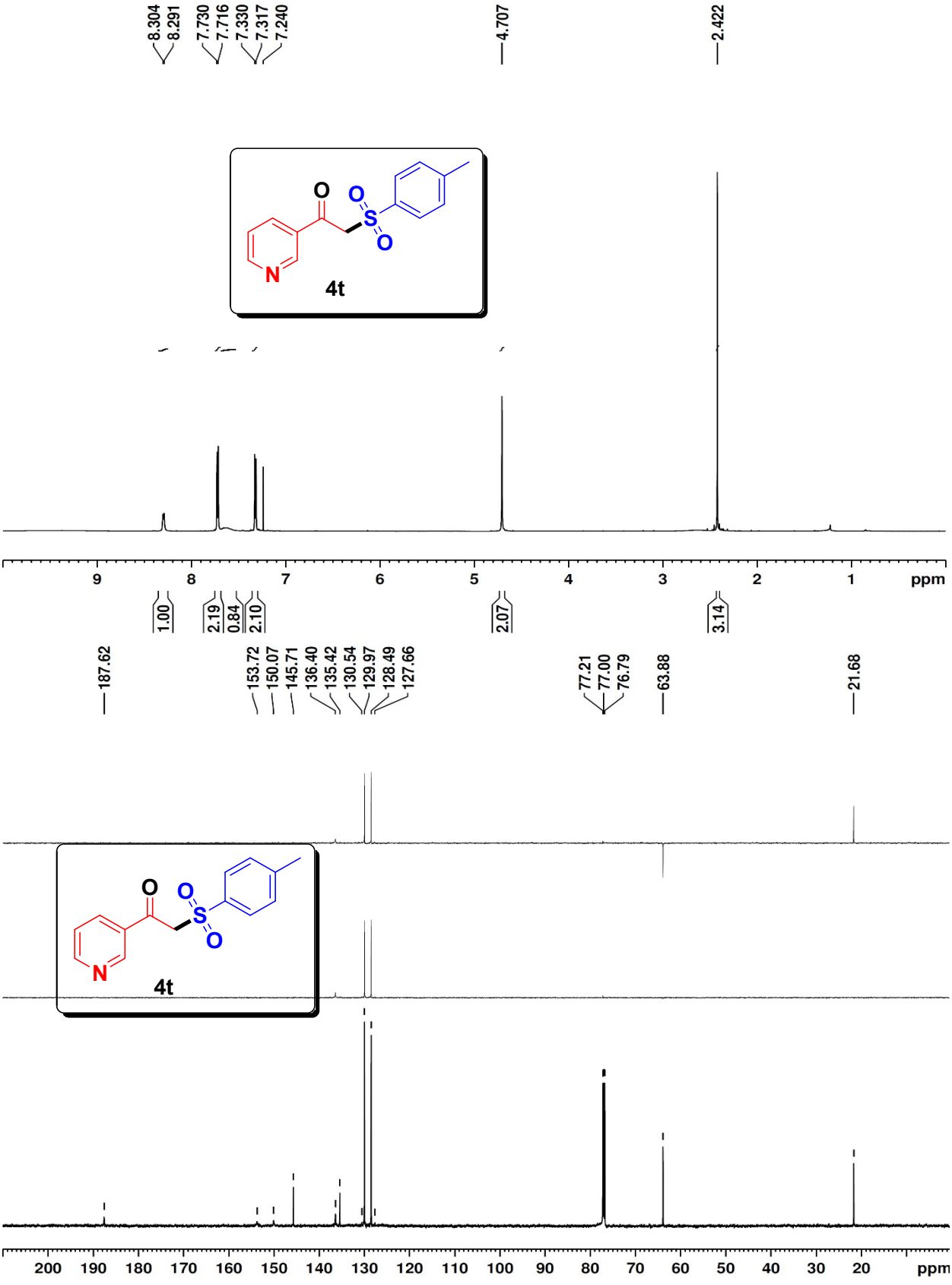
40
30
20
ppm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6
5
4
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2
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ppm
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3.14
187.62
153.72
150.07
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135.42
130.54
129.97
128.49
127.66
77.21
77.00
76.79
63.88
21.68
4t
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80
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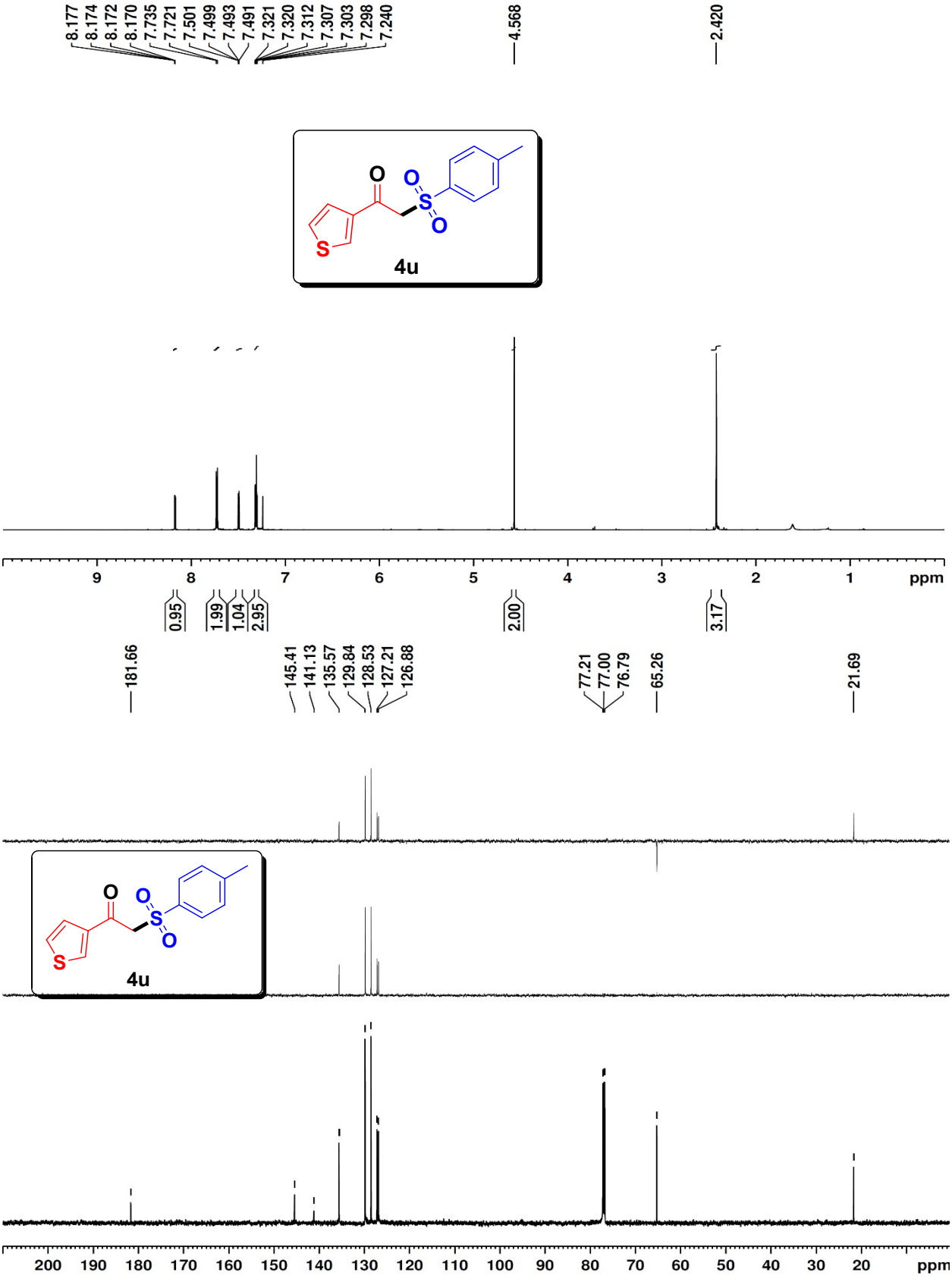
30
20
ppm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7.321
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7.312
7.307
7.303
7.298
7.240
4.568
2.420
4u
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8
7
6
5
4
3
2
1
ppm
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1.99
1.04
2.95
2.00
3.17
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135.57
129.84
128.53
127.21
126.88
77.21
77.00
76.79
65.26
21.69
4u
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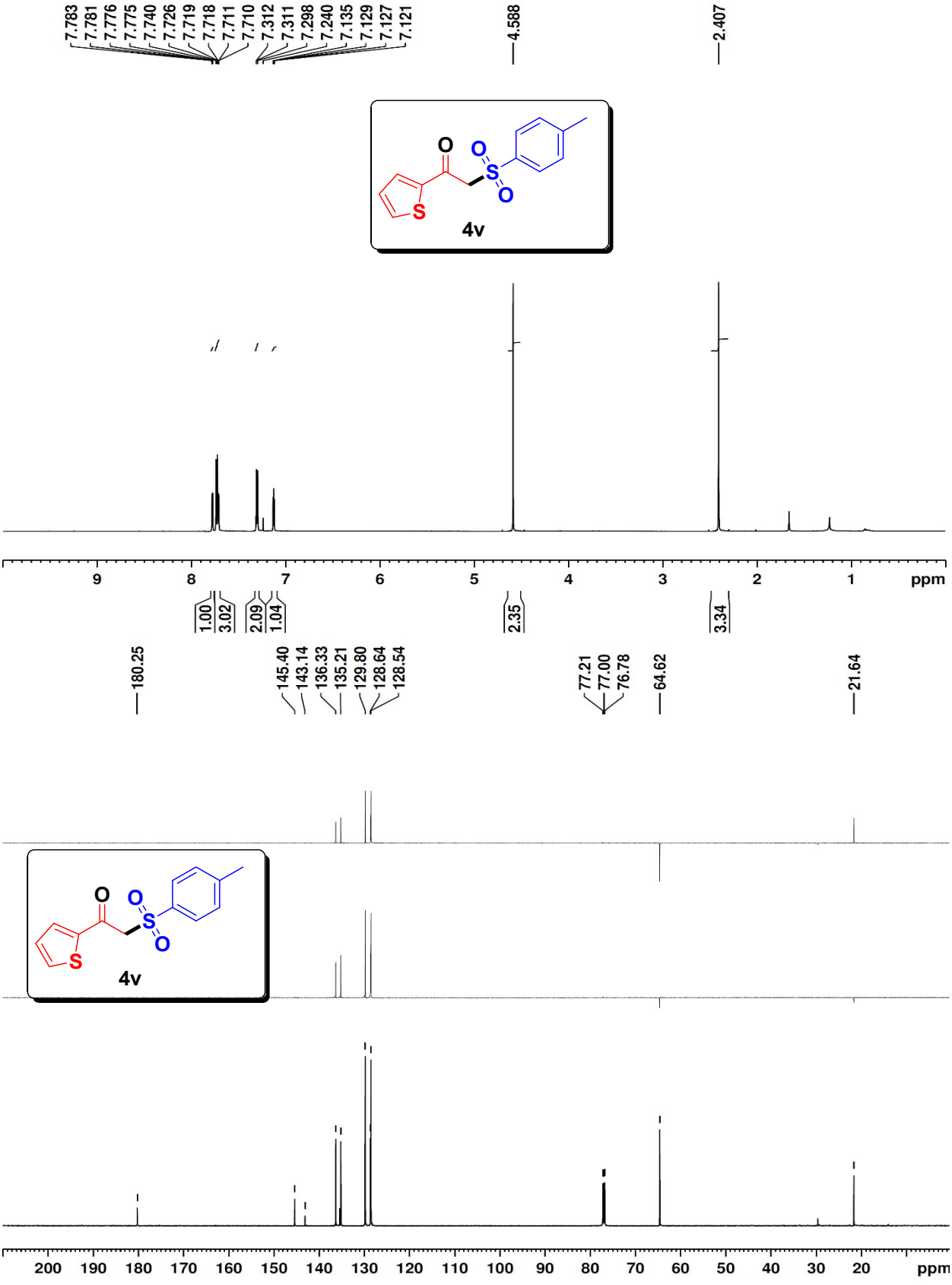
ppm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7.711
7.710
7.312
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7.298
7.240
7.135
7.129
7.127
7.121
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7
6
5
4
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2
1
ppm
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3.02
2.09
1.04
2.35
3.34
180.25
145.40
143.14
136.33
135.21
129.80
128.64
128.54
77.21
77.00
76.78
64.62
21.64
4v
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180
170
160
150
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130
120
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80
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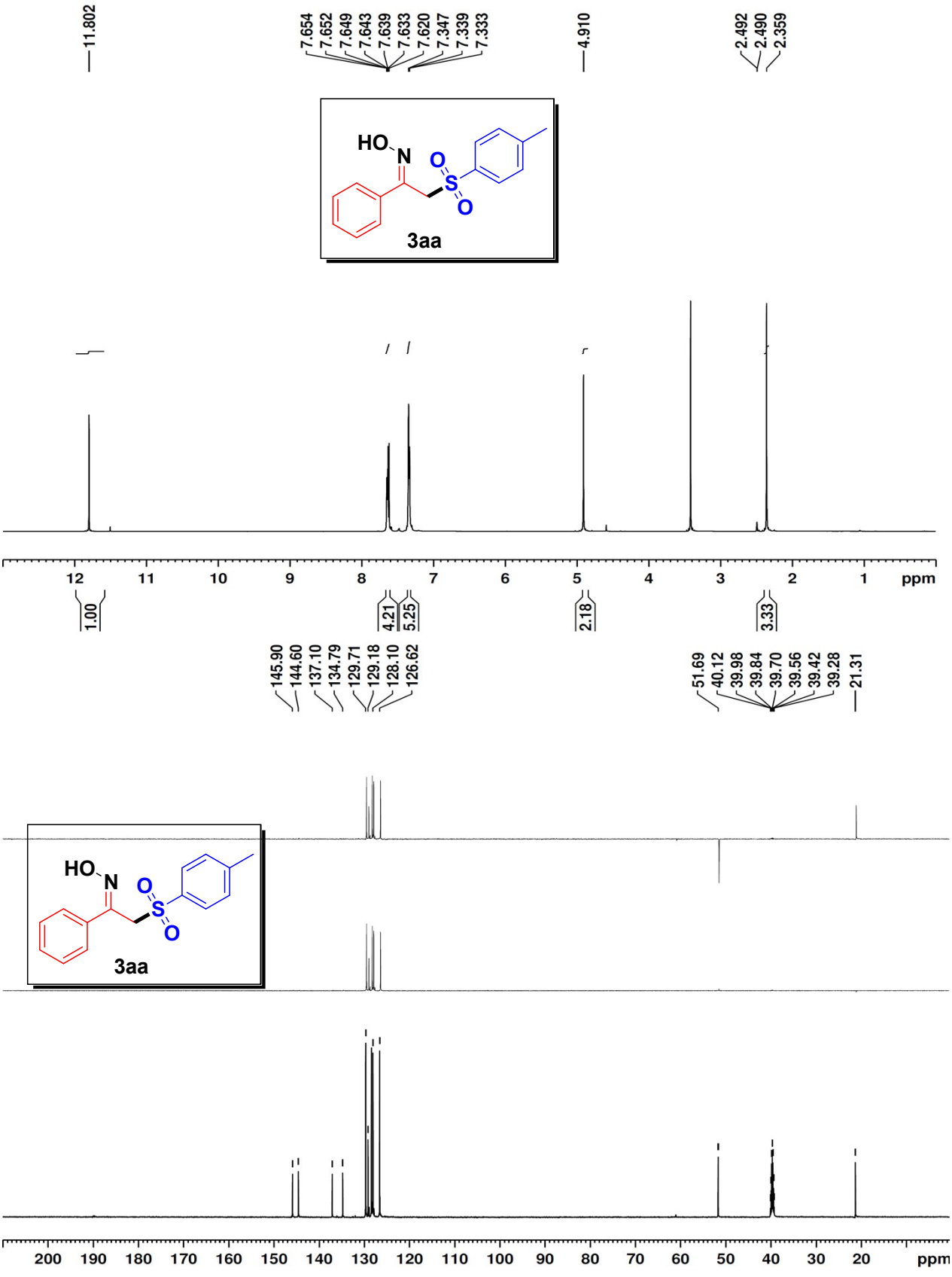
30
20
ppm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7.333
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2.490
2.359
HO
N
O
S
O
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11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
4.21
5.25
2.18
3.33
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144.60
137.10
134.79
129.71
129.18
128.10
126.62
51.69
40.12
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21.31
HO
N
O
S
O
3aa
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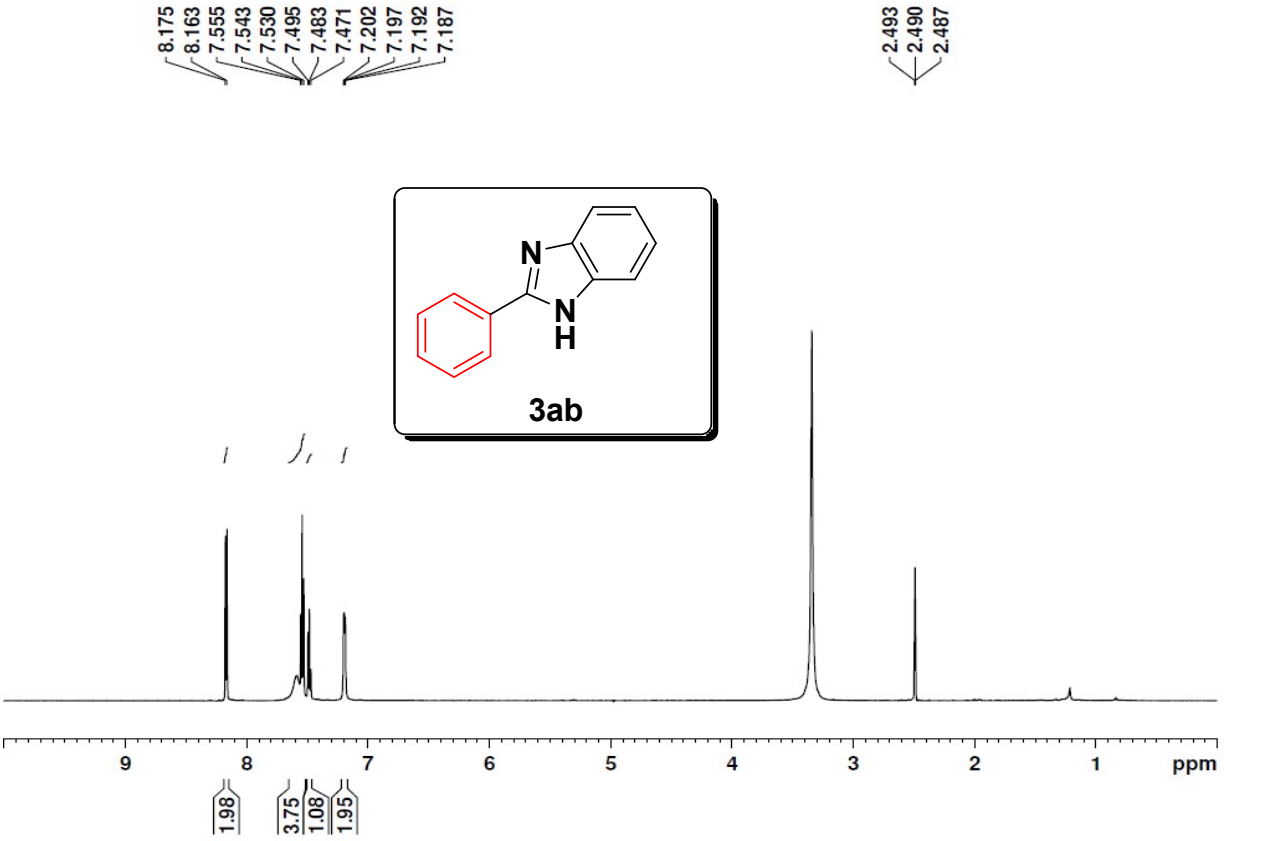
20
ppm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7.187
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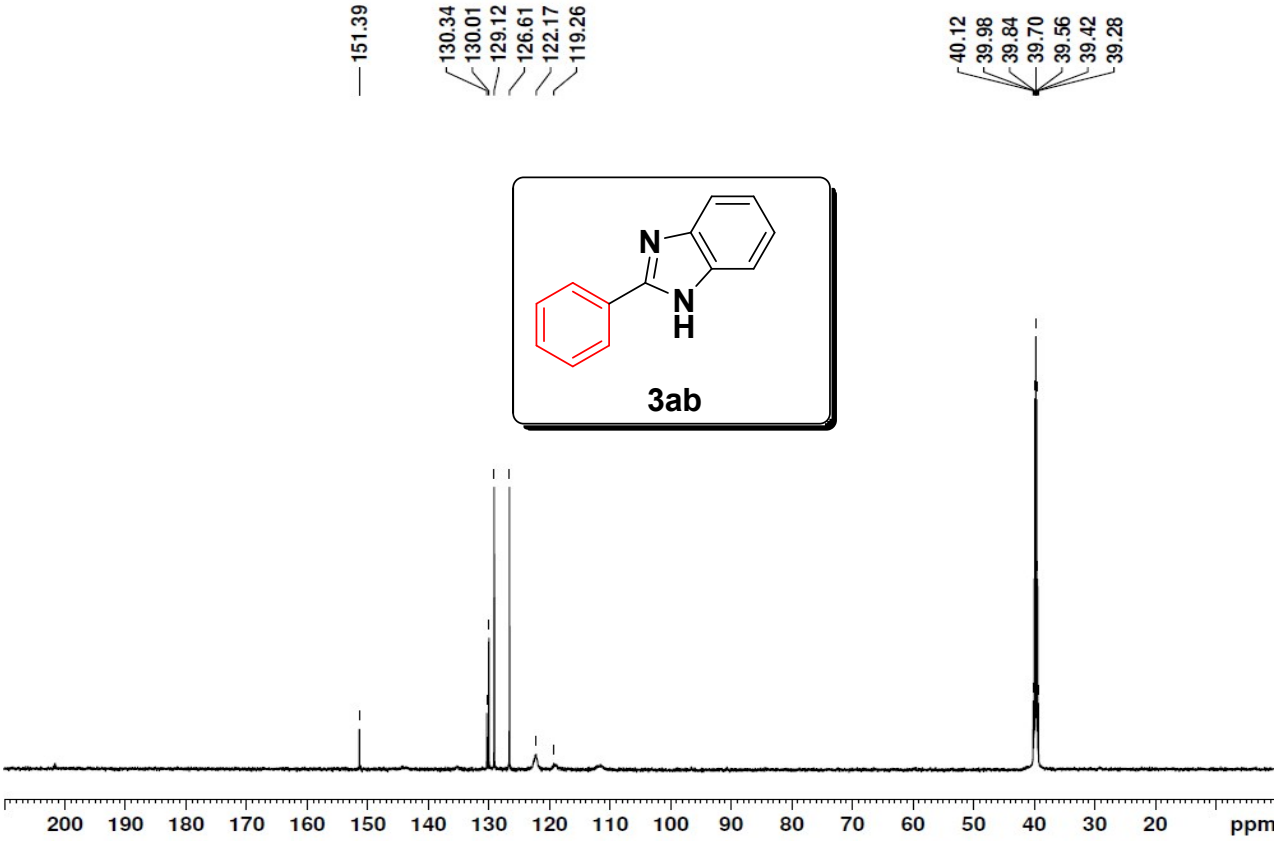
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39.84
39.70
39.56
39.42
39.28
N
N
H
3ab
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

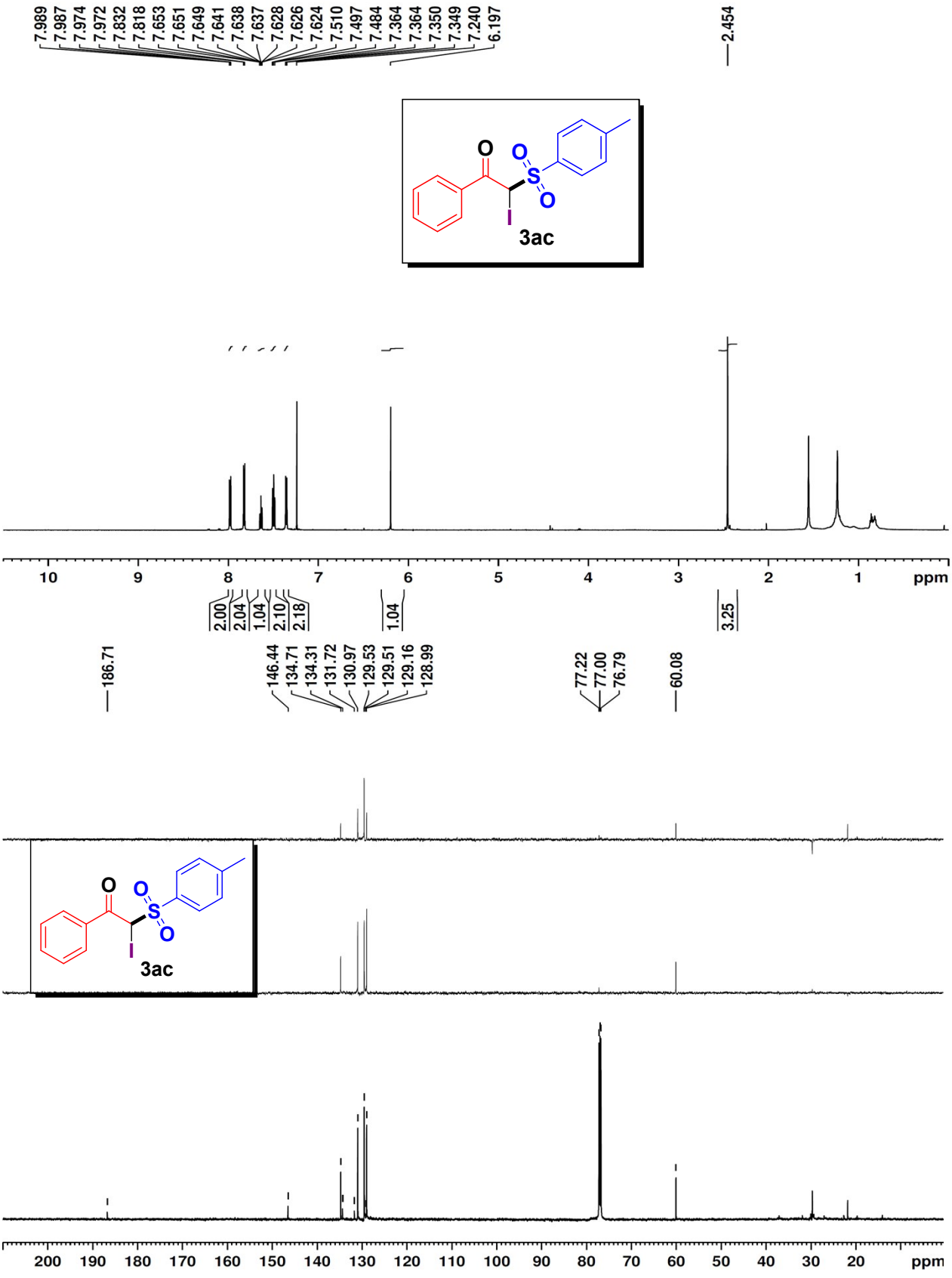

7.989
7.987
7.974
7.972
7.832
7.818
7.653
7.651
7.649
7.641
7.638
7.637
7.628
7.626
7.624
7.510
7.497
7.484
7.364
7.364
7.350
7.349
7.240
6.197
2.454
3ac
10
9
8
7
6
5
4
3
2
1
ppm
2.00
2.04
1.04
2.10
2.18
1.04
3.25
186.71
146.44
134.71
134.31
131.72
130.97
129.53
129.51
129.16
128.99
77.22
77.00
76.79
60.08
3ac
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Figure S6. ORTEP diagram of compound **3d (CCDC: 2057129)**

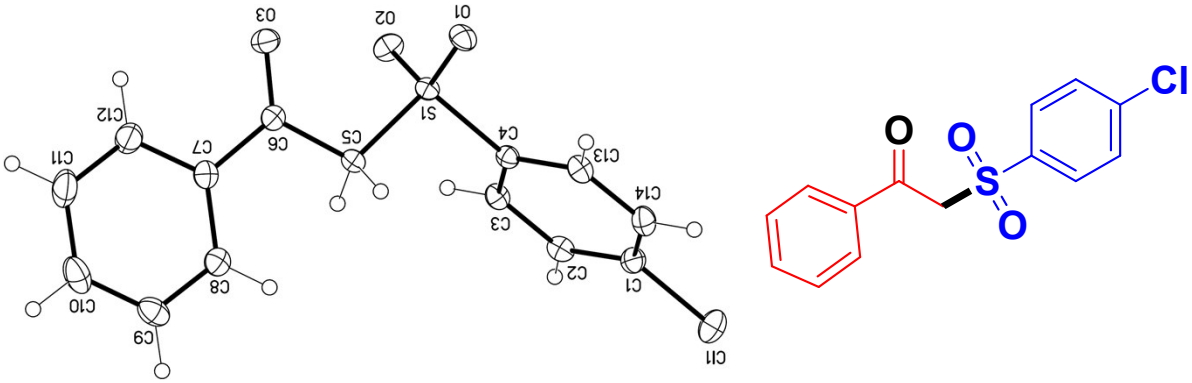


Table S5. Crystal data and structure refinement for 210103lt.

Identification code	210103LT	
Empirical formula	C14 H11 Cl O3 S	
Formula weight	294.74	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 13.3682(5) Å	a= 90°.
	b = 7.2588(3) Å	b= 90°.
	c = 26.6061(10) Å	g = 90°.
Volume	2581.78(17) $Å^3$	
Z	8	
Density (calculated)	1.517 Mg/m^3	
Absorption coefficient	0.457 mm^{-1}	
F(000)	1216	
Crystal size	0.20 x 0.20 x 0.05 mm^3	
Theta range for data collection	2.160 to 26.415°.	
Index ranges	-16<=h<=16, -8<=k<=9, -33<=l<=32	
Reflections collected	21704	
Independent reflections	2647 [R(int) = 0.0364]	
Completeness to theta = 25.242°	100.0 %	

Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7454 and 0.6680
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2647 / 0 / 172
Goodness-of-fit on F^2	1.043
Final R indices [I>2sigma(I)]	R1 = 0.0295, wR2 = 0.0744
R indices (all data)	R1 = 0.0335, wR2 = 0.0771
Extinction coefficient	n/a
Largest diff. peak and hole	0.431 and -0.360 e.Å^{-3}

Table S6. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for 210103lt. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	2051(1)	9614(1)	700(1)	25(1)
S(1)	3998(1)	11712(1)	2749(1)	13(1)
O(1)	5050(1)	11992(2)	2665(1)	18(1)
O(2)	3415(1)	13182(2)	2966(1)	19(1)
O(3)	4589(1)	11323(2)	3799(1)	20(1)
C(1)	2598(1)	10191(2)	1270(1)	17(1)
C(2)	1987(1)	10758(2)	1660(1)	16(1)
C(3)	2417(1)	11218(2)	2114(1)	16(1)
C(4)	3448(1)	11093(2)	2170(1)	13(1)
C(5)	3833(1)	9695(2)	3117(1)	14(1)
C(6)	4184(1)	9916(2)	3657(1)	14(1)
C(7)	4014(1)	8321(2)	3997(1)	16(1)
C(8)	3645(1)	6636(2)	3826(1)	18(1)
C(9)	3525(1)	5177(2)	4156(1)	23(1)
C(10)	3752(1)	5406(3)	4658(1)	28(1)
C(11)	4110(2)	7081(3)	4833(1)	30(1)

C(12)	4244(1)	8533(2)	4504(1)	23(1)
C(13)	4056(1)	10509(2)	1778(1)	16(1)
C(14)	3629(1)	10062(2)	1322(1)	19(1)

Table S7. Bond lengths [Å] and angles [°] for 210103lt.

Cl(1)-C(1)	1.7344(16)
S(1)-O(1)	1.4394(11)
S(1)-O(2)	1.4424(11)
S(1)-C(4)	1.7646(15)
S(1)-C(5)	1.7750(15)
O(3)-C(6)	1.2163(19)
C(1)-C(2)	1.384(2)
C(1)-C(14)	1.388(2)
C(2)-C(3)	1.380(2)
C(2)-H(11)	0.9500
C(3)-C(4)	1.389(2)
C(3)-H(2)	0.9500
C(4)-C(13)	1.389(2)
C(5)-C(6)	1.520(2)
C(5)-H(7)	0.9900
C(5)-H(8)	0.9900
C(6)-C(7)	1.487(2)
C(7)-C(12)	1.392(2)
C(7)-C(8)	1.395(2)
C(8)-C(9)	1.384(2)
C(8)-H(3)	0.9500
C(9)-C(10)	1.382(3)
C(9)-H(6)	0.9500
C(10)-C(11)	1.387(3)
C(10)-H(1)	0.9500

C(11)-C(12)	1.382(3)
C(11)-H(5)	0.9500
C(12)-H(4)	0.9500
C(13)-C(14)	1.379(2)
C(13)-H(10)	0.9500
C(14)-H(9)	0.9500
O(1)-S(1)-O(2)	119.08(7)
O(1)-S(1)-C(4)	107.95(7)
O(2)-S(1)-C(4)	108.30(7)
O(1)-S(1)-C(5)	108.82(7)
O(2)-S(1)-C(5)	108.78(7)
C(4)-S(1)-C(5)	102.68(7)
C(2)-C(1)-C(14)	122.07(14)
C(2)-C(1)-Cl(1)	118.59(12)
C(14)-C(1)-Cl(1)	119.34(12)
C(3)-C(2)-C(1)	118.96(14)
C(3)-C(2)-H(11)	120.5
C(1)-C(2)-H(11)	120.5
C(2)-C(3)-C(4)	119.34(14)
C(2)-C(3)-H(2)	120.3
C(4)-C(3)-H(2)	120.3
C(13)-C(4)-C(3)	121.37(14)
C(13)-C(4)-S(1)	119.37(12)
C(3)-C(4)-S(1)	119.26(11)
C(6)-C(5)-S(1)	113.31(11)
C(6)-C(5)-H(7)	108.9
S(1)-C(5)-H(7)	108.9
C(6)-C(5)-H(8)	108.9
S(1)-C(5)-H(8)	108.9
H(7)-C(5)-H(8)	107.7
O(3)-C(6)-C(7)	122.18(14)
O(3)-C(6)-C(5)	121.34(14)
C(7)-C(6)-C(5)	116.47(13)

C(12)-C(7)-C(8)	119.39(15)
C(12)-C(7)-C(6)	118.03(15)
C(8)-C(7)-C(6)	122.58(14)
C(9)-C(8)-C(7)	120.36(15)
C(9)-C(8)-H(3)	119.8
C(7)-C(8)-H(3)	119.8
C(10)-C(9)-C(8)	119.74(16)
C(10)-C(9)-H(6)	120.1
C(8)-C(9)-H(6)	120.1
C(9)-C(10)-C(11)	120.36(16)
C(9)-C(10)-H(1)	119.8
C(11)-C(10)-H(1)	119.8
C(12)-C(11)-C(10)	120.06(16)
C(12)-C(11)-H(5)	120.0
C(10)-C(11)-H(5)	120.0
C(11)-C(12)-C(7)	120.09(17)
C(11)-C(12)-H(4)	120.0
C(7)-C(12)-H(4)	120.0
C(14)-C(13)-C(4)	119.39(14)
C(14)-C(13)-H(10)	120.3
C(4)-C(13)-H(10)	120.3
C(13)-C(14)-C(1)	118.87(15)
C(13)-C(14)-H(9)	120.6
C(1)-C(14)-H(9)	120.6

Symmetry transformations used to generate equivalent atoms:

Table S8. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 210103lt. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}

Cl(1)	28(1)	31(1)	17(1)	-2(1)	-5(1)	1(1)
S(1)	10(1)	14(1)	15(1)	1(1)	0(1)	0(1)
O(1)	12(1)	22(1)	21(1)	3(1)	0(1)	-3(1)
O(2)	18(1)	17(1)	22(1)	-3(1)	-2(1)	4(1)
O(3)	20(1)	18(1)	21(1)	-2(1)	-2(1)	-3(1)
C(1)	21(1)	16(1)	15(1)	2(1)	-3(1)	0(1)
C(2)	13(1)	16(1)	20(1)	3(1)	-1(1)	1(1)
C(3)	14(1)	15(1)	17(1)	2(1)	2(1)	2(1)
C(4)	13(1)	13(1)	14(1)	2(1)	-1(1)	0(1)
C(5)	13(1)	14(1)	16(1)	1(1)	1(1)	-1(1)
C(6)	10(1)	17(1)	16(1)	-2(1)	1(1)	1(1)
C(7)	12(1)	19(1)	17(1)	0(1)	2(1)	3(1)
C(8)	14(1)	21(1)	19(1)	-1(1)	2(1)	2(1)
C(9)	19(1)	19(1)	30(1)	2(1)	6(1)	1(1)
C(10)	34(1)	23(1)	26(1)	10(1)	8(1)	5(1)
C(11)	42(1)	32(1)	16(1)	2(1)	2(1)	4(1)
C(12)	28(1)	23(1)	18(1)	-1(1)	0(1)	1(1)
C(13)	13(1)	18(1)	19(1)	3(1)	2(1)	2(1)
C(14)	20(1)	20(1)	17(1)	2(1)	4(1)	2(1)

Table S9. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 210103lt.

	x	y	z	U(eq)
H(11)	1283	10831	1615	19
H(2)	2012	11616	2387	19
H(7)	3115	9358	3117	17
H(8)	4207	8669	2959	17
H(3)	3476	6489	3482	21

H(6)	3287	4024	4036	27
H(1)	3664	4409	4886	33
H(5)	4262	7231	5179	36
H(4)	4493	9677	4624	28
H(10)	4759	10418	1823	20
H(9)	4033	9672	1048	23

Figure S7. ORTEP diagram of compound **4v (CCDC: 2008686)**

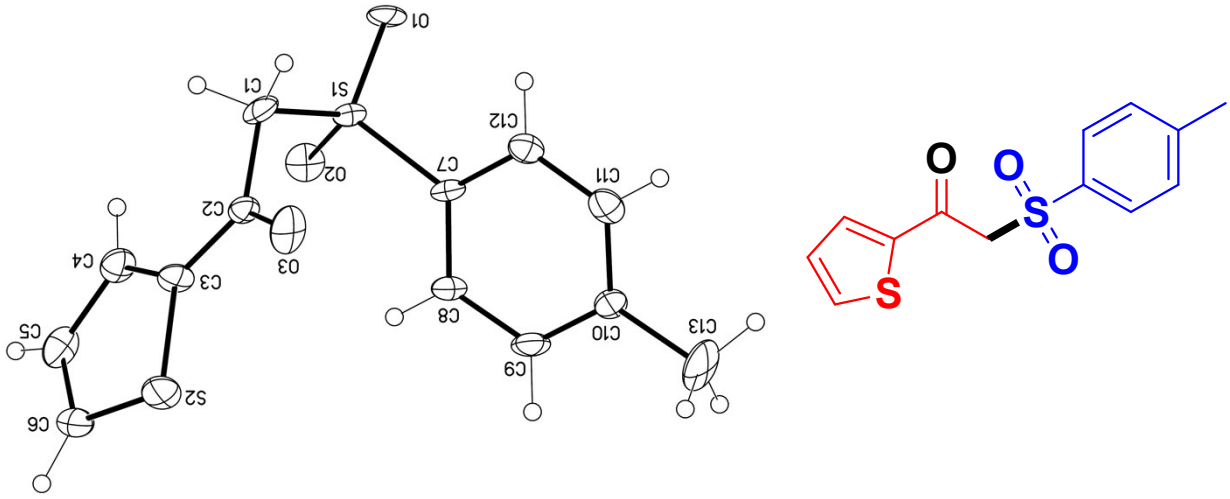


Table S10. Crystal data and structure refinement for 180534lt_0m_a.

Identification code	180534lt_0m_a	
Empirical formula	C13 H12 O3 S2	
Formula weight	280.35	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 5.5069(6) Å	□= 84.488(5)°.
	b = 13.3044(16) Å	□= 88.558(5)°.
	c = 35.296(4) Å	□ = 89.975(5)°.
Volume	2573.2(5) $Å^3$	
Z	8	
Density (calculated)	1.447 Mg/m^3	
Absorption coefficient	0.410 mm^{-1}	
F(000)	1168	
Crystal size	0.20 x 0.03 x 0.03 mm^3	
Theta range for data collection	1.160 to 26.479°.	

Index ranges	-6<=h<=5, -16<=k<=16, -44<=l<=42
Reflections collected	34484
Independent reflections	10470 [R(int) = 0.0626]
Completeness to theta = 25.242°	98.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9485 and 0.8205
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	10470 / 0 / 653
Goodness-of-fit on F^2	1.214
Final R indices [I>2sigma(I)]	R1 = 0.1323, wR2 = 0.3010
R indices (all data)	R1 = 0.1488, wR2 = 0.3075
Extinction coefficient	n/a
Largest diff. peak and hole	1.451 and -0.852 e.$Å^{-3}$

Table S11. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 180534lt_0m_a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	11696(17)	1101(7)	2303(3)	22(2)
C(2)	11073(17)	-19(6)	2310(2)	18(2)
C(3)	9030(17)	-413(6)	2553(3)	19(2)
C(4)	7608(18)	56(7)	2801(3)	23(2)
C(5)	5911(18)	-592(8)	2990(3)	26(2)
C(6)	6040(20)	-1557(8)	2883(3)	32(2)
C(7)	9519(16)	1268(6)	1568(2)	16(2)
C(8)	7539(16)	659(6)	1528(3)	18(2)
C(9)	7221(18)	259(7)	1184(3)	23(2)
C(10)	8900(20)	447(7)	883(3)	25(2)
C(11)	10904(18)	1036(7)	935(3)	23(2)

C(12)	11231(17)	1455(7)	1274(3)	21(2)
C(13)	8520(30)	23(9)	506(3)	46(3)
C(14)	7071(19)	3926(7)	2739(3)	26(2)
C(15)	6133(17)	5008(7)	2707(2)	18(2)
C(16)	4154(18)	5291(7)	2462(2)	21(2)
C(17)	2926(18)	4740(7)	2221(2)	22(2)
C(18)	1076(19)	5277(8)	2028(3)	26(2)
C(19)	890(20)	6242(8)	2123(3)	30(2)
C(20)	4758(15)	3681(6)	3469(2)	14(2)
C(21)	2722(17)	4283(7)	3507(3)	22(2)
C(22)	2254(19)	4680(7)	3854(3)	25(2)
C(23)	3838(18)	4490(7)	4156(3)	23(2)
C(24)	5886(19)	3905(7)	4110(3)	24(2)
C(25)	6366(17)	3508(6)	3765(3)	20(2)
C(26)	3250(20)	4882(9)	4538(3)	41(3)
C(27)	418(16)	7971(6)	4047(2)	15(2)
C(28)	-1605(18)	7453(7)	4189(3)	24(2)
C(29)	-2660(20)	6867(7)	3924(3)	32(2)
C(30)	-1319(17)	6917(7)	3589(3)	24(2)
C(31)	1967(16)	8663(7)	4226(2)	16(2)
C(32)	1275(16)	8923(6)	4626(2)	15(2)
C(33)	1815(17)	8468(7)	5406(2)	17(2)
C(34)	2930(16)	9161(6)	5617(2)	17(2)
C(35)	1865(17)	9364(7)	5957(2)	18(2)
C(36)	-259(16)	8891(6)	6094(2)	17(2)
C(37)	-1349(17)	8189(6)	5875(2)	18(2)
C(38)	-340(17)	7978(7)	5532(2)	19(2)
C(39)	-1392(17)	9098(7)	6468(2)	22(2)
C(40)	6279(16)	6058(6)	10382(2)	15(2)
C(41)	6906(16)	6280(6)	10788(2)	16(2)
C(42)	5324(16)	6966(7)	10974(2)	18(2)
C(43)	3258(18)	7472(7)	10846(3)	25(2)
C(44)	2252(18)	8058(7)	11119(3)	26(2)

C(45)	3506(19)	7989(7)	11445(3)	27(2)
C(46)	6952(16)	6568(6)	9609(2)	16(2)
C(47)	8002(16)	5874(6)	9390(2)	17(2)
C(48)	6957(16)	5704(7)	9046(2)	18(2)
C(49)	4860(16)	6211(7)	8926(2)	16(2)
C(50)	3835(16)	6914(7)	9156(2)	18(2)
C(51)	4850(16)	7094(6)	9495(2)	16(2)
C(52)	3751(18)	6037(7)	8556(2)	23(2)
O(1)	11137(13)	2809(4)	1892(2)	24(2)
O(2)	7395(12)	1934(5)	2159(2)	22(1)
O(3)	12274(14)	-546(5)	2113(2)	30(2)
O(4)	2901(12)	2998(5)	2881(2)	22(1)
O(5)	6651(13)	2201(5)	3138(2)	28(2)
O(6)	7140(13)	5605(5)	2901(2)	27(2)
O(7)	5543(12)	8589(6)	4938(2)	31(2)
O(8)	2754(16)	7160(5)	4918(2)	36(2)
O(9)	3759(12)	9048(5)	4065(2)	23(1)
O(10)	10624(12)	6382(6)	10064(2)	32(2)
O(11)	7906(17)	7827(5)	10109(2)	39(2)
O(12)	8662(12)	5891(5)	10942(2)	22(1)
S(1)	9795(4)	1883(2)	1988(1)	18(1)
S(2)	8256(5)	-1662(2)	2545(1)	28(1)
S(3)	5230(4)	3086(2)	3048(1)	19(1)
S(4)	2990(5)	6502(2)	2448(1)	26(1)
S(5)	3113(4)	8211(2)	4966(1)	20(1)
S(6)	1087(5)	7697(2)	3590(1)	26(1)
S(7)	8206(4)	6781(2)	10048(1)	21(1)
S(8)	5951(5)	7206(2)	11434(1)	26(1)

Table S12. Bond lengths [Å] and angles [°] for 180534lt_0m_a.

C(1)-C(2)	1.528(12)
C(1)-S(1)	1.802(9)
C(1)-H(1A)	0.9900
C(1)-H(1B)	0.9900
C(2)-O(3)	1.218(11)
C(2)-C(3)	1.463(13)
C(3)-C(4)	1.357(13)
C(3)-S(2)	1.719(9)
C(4)-C(5)	1.385(14)
C(4)-H(4)	0.9500
C(5)-C(6)	1.375(14)
C(5)-H(5)	0.9500
C(6)-S(2)	1.699(11)
C(6)-H(6)	0.9500
C(7)-C(8)	1.377(12)
C(7)-C(12)	1.387(13)
C(7)-S(1)	1.772(9)
C(8)-C(9)	1.384(12)
C(8)-H(8)	0.9500
C(9)-C(10)	1.395(13)
C(9)-H(9)	0.9500
C(10)-C(11)	1.381(14)
C(10)-C(13)	1.513(13)
C(11)-C(12)	1.384(13)
C(11)-H(11)	0.9500
C(12)-H(12)	0.9500
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-C(15)	1.524(13)
C(14)-S(3)	1.783(10)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900

C(15)-O(6)	1.237(11)
C(15)-C(16)	1.437(13)
C(16)-C(17)	1.365(12)
C(16)-S(4)	1.731(9)
C(17)-C(18)	1.398(15)
C(17)-H(17)	0.9500
C(18)-C(19)	1.360(14)
C(18)-H(18)	0.9500
C(19)-S(4)	1.708(11)
C(19)-H(19)	0.9500
C(20)-C(25)	1.388(12)
C(20)-C(21)	1.389(12)
C(20)-S(3)	1.761(8)
C(21)-C(22)	1.397(13)
C(21)-H(21)	0.9500
C(22)-C(23)	1.399(14)
C(22)-H(22)	0.9500
C(23)-C(24)	1.386(14)
C(23)-C(26)	1.518(13)
C(24)-C(25)	1.393(13)
C(24)-H(24)	0.9500
C(25)-H(25)	0.9500
C(26)-H(26A)	0.9800
C(26)-H(26B)	0.9800
C(26)-H(26C)	0.9800
C(27)-C(28)	1.368(13)
C(27)-C(31)	1.453(12)
C(27)-S(6)	1.719(8)
C(28)-C(29)	1.411(14)
C(28)-H(28)	0.9500
C(29)-C(30)	1.374(15)
C(29)-H(29)	0.9500
C(30)-S(6)	1.684(10)

C(30)-H(30)	0.9500
C(31)-O(9)	1.214(11)
C(31)-C(32)	1.527(11)
C(32)-S(5)	1.789(9)
C(32)-H(32A)	0.9900
C(32)-H(32B)	0.9900
C(33)-C(34)	1.391(12)
C(33)-C(38)	1.397(13)
C(33)-S(5)	1.757(9)
C(34)-C(35)	1.374(12)
C(34)-H(34)	0.9500
C(35)-C(36)	1.382(13)
C(35)-H(35)	0.9500
C(36)-C(37)	1.411(12)
C(36)-C(39)	1.496(12)
C(37)-C(38)	1.373(12)
C(37)-H(37)	0.9500
C(38)-H(38)	0.9500
C(39)-H(39A)	0.9800
C(39)-H(39B)	0.9800
C(39)-H(39C)	0.9800
C(40)-C(41)	1.538(11)
C(40)-S(7)	1.778(8)
C(40)-H(40A)	0.9900
C(40)-H(40B)	0.9900
C(41)-O(12)	1.213(11)
C(41)-C(42)	1.452(12)
C(42)-C(43)	1.385(14)
C(42)-S(8)	1.727(9)
C(43)-C(44)	1.400(13)
C(43)-H(43)	0.9500
C(44)-C(45)	1.354(15)
C(44)-H(44)	0.9500

C(45)-S(8)	1.705(10)
C(45)-H(45)	0.9500
C(46)-C(47)	1.379(12)
C(46)-C(51)	1.400(12)
C(46)-S(7)	1.761(9)
C(47)-C(48)	1.392(12)
C(47)-H(47)	0.9500
C(48)-C(49)	1.392(12)
C(48)-H(48)	0.9500
C(49)-C(50)	1.405(12)
C(49)-C(52)	1.494(12)
C(50)-C(51)	1.375(12)
C(50)-H(50)	0.9500
C(51)-H(51)	0.9500
C(52)-H(52A)	0.9800
C(52)-H(52B)	0.9800
C(52)-H(52C)	0.9800
O(1)-S(1)	1.445(6)
O(2)-S(1)	1.444(7)
O(4)-S(3)	1.435(7)
O(5)-S(3)	1.429(7)
O(7)-S(5)	1.428(7)
O(8)-S(5)	1.438(7)
O(10)-S(7)	1.434(7)
O(11)-S(7)	1.437(7)

C(2)-C(1)-S(1)	112.4(6)
C(2)-C(1)-H(1A)	109.1
S(1)-C(1)-H(1A)	109.1
C(2)-C(1)-H(1B)	109.1
S(1)-C(1)-H(1B)	109.1
H(1A)-C(1)-H(1B)	107.9
O(3)-C(2)-C(3)	122.7(8)

O(3)-C(2)-C(1)	119.1(9)
C(3)-C(2)-C(1)	118.2(8)
C(4)-C(3)-C(2)	129.9(8)
C(4)-C(3)-S(2)	112.1(7)
C(2)-C(3)-S(2)	118.0(6)
C(3)-C(4)-C(5)	112.3(8)
C(3)-C(4)-H(4)	123.9
C(5)-C(4)-H(4)	123.9
C(6)-C(5)-C(4)	113.0(9)
C(6)-C(5)-H(5)	123.5
C(4)-C(5)-H(5)	123.5
C(5)-C(6)-S(2)	111.6(8)
C(5)-C(6)-H(6)	124.2
S(2)-C(6)-H(6)	124.2
C(8)-C(7)-C(12)	120.9(8)
C(8)-C(7)-S(1)	119.6(7)
C(12)-C(7)-S(1)	119.3(7)
C(7)-C(8)-C(9)	119.2(8)
C(7)-C(8)-H(8)	120.4
C(9)-C(8)-H(8)	120.4
C(8)-C(9)-C(10)	121.0(9)
C(8)-C(9)-H(9)	119.5
C(10)-C(9)-H(9)	119.5
C(11)-C(10)-C(9)	118.6(8)
C(11)-C(10)-C(13)	120.6(10)
C(9)-C(10)-C(13)	120.8(10)
C(10)-C(11)-C(12)	121.1(9)
C(10)-C(11)-H(11)	119.4
C(12)-C(11)-H(11)	119.4
C(11)-C(12)-C(7)	119.1(9)
C(11)-C(12)-H(12)	120.4
C(7)-C(12)-H(12)	120.4
C(10)-C(13)-H(13A)	109.5

C(10)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(10)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(15)-C(14)-S(3)	112.9(7)
C(15)-C(14)-H(14A)	109.0
S(3)-C(14)-H(14A)	109.0
C(15)-C(14)-H(14B)	109.0
S(3)-C(14)-H(14B)	109.0
H(14A)-C(14)-H(14B)	107.8
O(6)-C(15)-C(16)	123.2(8)
O(6)-C(15)-C(14)	117.0(9)
C(16)-C(15)-C(14)	119.8(8)
C(17)-C(16)-C(15)	130.2(9)
C(17)-C(16)-S(4)	110.3(8)
C(15)-C(16)-S(4)	119.5(7)
C(16)-C(17)-C(18)	113.7(9)
C(16)-C(17)-H(17)	123.2
C(18)-C(17)-H(17)	123.2
C(19)-C(18)-C(17)	112.5(9)
C(19)-C(18)-H(18)	123.8
C(17)-C(18)-H(18)	123.8
C(18)-C(19)-S(4)	111.9(8)
C(18)-C(19)-H(19)	124.0
S(4)-C(19)-H(19)	124.0
C(25)-C(20)-C(21)	120.7(8)
C(25)-C(20)-S(3)	119.6(7)
C(21)-C(20)-S(3)	119.6(7)
C(20)-C(21)-C(22)	119.1(9)
C(20)-C(21)-H(21)	120.4
C(22)-C(21)-H(21)	120.4
C(21)-C(22)-C(23)	120.5(9)

C(21)-C(22)-H(22)	119.7
C(23)-C(22)-H(22)	119.7
C(24)-C(23)-C(22)	119.5(9)
C(24)-C(23)-C(26)	120.2(9)
C(22)-C(23)-C(26)	120.2(9)
C(23)-C(24)-C(25)	120.3(9)
C(23)-C(24)-H(24)	119.9
C(25)-C(24)-H(24)	119.9
C(20)-C(25)-C(24)	119.8(9)
C(20)-C(25)-H(25)	120.1
C(24)-C(25)-H(25)	120.1
C(23)-C(26)-H(26A)	109.5
C(23)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
C(23)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
C(28)-C(27)-C(31)	129.9(8)
C(28)-C(27)-S(6)	110.6(7)
C(31)-C(27)-S(6)	119.5(7)
C(27)-C(28)-C(29)	113.2(9)
C(27)-C(28)-H(28)	123.4
C(29)-C(28)-H(28)	123.4
C(30)-C(29)-C(28)	111.4(9)
C(30)-C(29)-H(29)	124.3
C(28)-C(29)-H(29)	124.3
C(29)-C(30)-S(6)	112.5(7)
C(29)-C(30)-H(30)	123.7
S(6)-C(30)-H(30)	123.7
O(9)-C(31)-C(27)	122.2(8)
O(9)-C(31)-C(32)	119.3(8)
C(27)-C(31)-C(32)	118.4(7)
C(31)-C(32)-S(5)	109.6(6)

C(31)-C(32)-H(32A)	109.8
S(5)-C(32)-H(32A)	109.8
C(31)-C(32)-H(32B)	109.8
S(5)-C(32)-H(32B)	109.8
H(32A)-C(32)-H(32B)	108.2
C(34)-C(33)-C(38)	121.8(8)
C(34)-C(33)-S(5)	119.5(7)
C(38)-C(33)-S(5)	118.7(7)
C(35)-C(34)-C(33)	118.6(8)
C(35)-C(34)-H(34)	120.7
C(33)-C(34)-H(34)	120.7
C(34)-C(35)-C(36)	121.5(8)
C(34)-C(35)-H(35)	119.3
C(36)-C(35)-H(35)	119.3
C(35)-C(36)-C(37)	118.7(8)
C(35)-C(36)-C(39)	121.5(8)
C(37)-C(36)-C(39)	119.8(8)
C(38)-C(37)-C(36)	121.1(8)
C(38)-C(37)-H(37)	119.4
C(36)-C(37)-H(37)	119.4
C(37)-C(38)-C(33)	118.2(8)
C(37)-C(38)-H(38)	120.9
C(33)-C(38)-H(38)	120.9
C(36)-C(39)-H(39A)	109.5
C(36)-C(39)-H(39B)	109.5
H(39A)-C(39)-H(39B)	109.5
C(36)-C(39)-H(39C)	109.5
H(39A)-C(39)-H(39C)	109.5
H(39B)-C(39)-H(39C)	109.5
C(41)-C(40)-S(7)	109.5(6)
C(41)-C(40)-H(40A)	109.8
S(7)-C(40)-H(40A)	109.8
C(41)-C(40)-H(40B)	109.8

S(7)-C(40)-H(40B)	109.8
H(40A)-C(40)-H(40B)	108.2
O(12)-C(41)-C(42)	122.2(8)
O(12)-C(41)-C(40)	120.4(8)
C(42)-C(41)-C(40)	117.4(7)
C(43)-C(42)-C(41)	130.4(8)
C(43)-C(42)-S(8)	111.3(7)
C(41)-C(42)-S(8)	118.3(7)
C(42)-C(43)-C(44)	112.1(9)
C(42)-C(43)-H(43)	124.0
C(44)-C(43)-H(43)	124.0
C(45)-C(44)-C(43)	112.8(9)
C(45)-C(44)-H(44)	123.6
C(43)-C(44)-H(44)	123.6
C(44)-C(45)-S(8)	112.9(7)
C(44)-C(45)-H(45)	123.5
S(8)-C(45)-H(45)	123.5
C(47)-C(46)-C(51)	121.3(8)
C(47)-C(46)-S(7)	119.9(7)
C(51)-C(46)-S(7)	118.7(7)
C(46)-C(47)-C(48)	119.0(8)
C(46)-C(47)-H(47)	120.5
C(48)-C(47)-H(47)	120.5
C(47)-C(48)-C(49)	121.0(8)
C(47)-C(48)-H(48)	119.5
C(49)-C(48)-H(48)	119.5
C(48)-C(49)-C(50)	118.6(8)
C(48)-C(49)-C(52)	121.1(8)
C(50)-C(49)-C(52)	120.3(8)
C(51)-C(50)-C(49)	121.1(8)
C(51)-C(50)-H(50)	119.5
C(49)-C(50)-H(50)	119.5
C(50)-C(51)-C(46)	118.9(8)

C(50)-C(51)-H(51)	120.5
C(46)-C(51)-H(51)	120.5
C(49)-C(52)-H(52A)	109.5
C(49)-C(52)-H(52B)	109.5
H(52A)-C(52)-H(52B)	109.5
C(49)-C(52)-H(52C)	109.5
H(52A)-C(52)-H(52C)	109.5
H(52B)-C(52)-H(52C)	109.5
O(2)-S(1)-O(1)	118.9(4)
O(2)-S(1)-C(7)	107.3(4)
O(1)-S(1)-C(7)	108.1(4)
O(2)-S(1)-C(1)	109.2(4)
O(1)-S(1)-C(1)	105.8(4)
C(7)-S(1)-C(1)	107.1(4)
C(6)-S(2)-C(3)	91.0(5)
O(5)-S(3)-O(4)	119.5(4)
O(5)-S(3)-C(20)	108.2(4)
O(4)-S(3)-C(20)	107.0(4)
O(5)-S(3)-C(14)	106.5(5)
O(4)-S(3)-C(14)	108.7(4)
C(20)-S(3)-C(14)	106.2(4)
C(19)-S(4)-C(16)	91.6(5)
O(7)-S(5)-O(8)	117.6(5)
O(7)-S(5)-C(33)	108.9(4)
O(8)-S(5)-C(33)	108.9(4)
O(7)-S(5)-C(32)	109.7(4)
O(8)-S(5)-C(32)	107.3(4)
C(33)-S(5)-C(32)	103.4(4)
C(30)-S(6)-C(27)	92.2(5)
O(10)-S(7)-O(11)	117.2(5)
O(10)-S(7)-C(46)	109.3(4)
O(11)-S(7)-C(46)	109.0(4)
O(10)-S(7)-C(40)	109.8(4)

O(11)-S(7)-C(40)	107.8(4)
C(46)-S(7)-C(40)	102.7(4)
C(45)-S(8)-C(42)	90.9(5)

Symmetry transformations used to generate equivalent atoms:

Table S13. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 180534lt_0m_a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	20(5)	23(5)	22(5)	-6(4)	-11(4)	-3(4)
C(2)	24(5)	11(4)	19(4)	-2(3)	-5(4)	-1(3)
C(3)	23(5)	9(4)	25(5)	-6(3)	1(4)	1(3)
C(4)	31(6)	16(4)	23(5)	-5(4)	-3(4)	2(4)
C(5)	18(5)	38(6)	23(5)	-11(4)	0(4)	5(4)
C(6)	36(6)	35(6)	26(5)	-7(4)	4(4)	-20(5)
C(7)	13(4)	12(4)	23(4)	-5(3)	-5(3)	-2(3)
C(8)	15(4)	16(4)	24(5)	-5(3)	0(4)	-4(3)
C(9)	25(5)	17(4)	29(5)	-8(4)	0(4)	-8(4)
C(10)	41(6)	17(4)	19(5)	-9(4)	4(4)	-9(4)
C(11)	30(5)	14(4)	25(5)	-2(4)	5(4)	2(4)
C(12)	23(5)	15(4)	25(5)	-2(3)	1(4)	-2(4)
C(13)	90(11)	29(6)	21(5)	-17(4)	-3(6)	3(6)
C(14)	33(6)	26(5)	19(5)	-10(4)	2(4)	5(4)
C(15)	22(5)	19(4)	12(4)	-5(3)	7(3)	-6(4)
C(16)	32(6)	15(4)	16(4)	-4(3)	3(4)	-3(4)
C(17)	30(5)	23(5)	15(4)	-8(4)	5(4)	-13(4)
C(18)	30(6)	34(5)	16(4)	-9(4)	-2(4)	-11(4)
C(19)	36(6)	30(5)	24(5)	-5(4)	-5(4)	5(5)
C(20)	12(4)	11(4)	20(4)	-4(3)	0(3)	0(3)

C(21)	18(5)	20(5)	28(5)	0(4)	-3(4)	5(4)
C(22)	25(5)	19(5)	32(5)	-6(4)	-4(4)	0(4)
C(23)	31(6)	12(4)	26(5)	-2(3)	2(4)	-4(4)
C(24)	36(6)	16(4)	22(5)	-3(4)	-9(4)	0(4)
C(25)	22(5)	11(4)	26(5)	0(3)	-7(4)	3(3)
C(26)	56(8)	40(7)	28(6)	-15(5)	-1(5)	8(6)
C(27)	17(4)	15(4)	15(4)	-7(3)	-5(3)	5(3)
C(28)	23(5)	23(5)	26(5)	-7(4)	3(4)	-5(4)
C(29)	31(6)	19(5)	47(6)	-9(4)	-2(5)	-9(4)
C(30)	19(5)	23(5)	34(5)	-10(4)	-6(4)	0(4)
C(31)	12(4)	20(4)	15(4)	-3(3)	-2(3)	3(3)
C(32)	20(5)	12(4)	13(4)	-4(3)	0(3)	-6(3)
C(33)	20(5)	17(4)	14(4)	-1(3)	0(3)	4(3)
C(34)	13(4)	17(4)	20(4)	-3(3)	3(3)	1(3)
C(35)	21(5)	17(4)	17(4)	-3(3)	-3(3)	3(4)
C(36)	19(5)	15(4)	17(4)	-2(3)	-2(3)	9(3)
C(37)	17(5)	15(4)	19(4)	1(3)	-1(3)	0(3)
C(38)	20(5)	17(4)	18(4)	0(3)	-5(4)	-2(4)
C(39)	21(5)	25(5)	19(4)	2(4)	-1(4)	-3(4)
C(40)	13(4)	14(4)	18(4)	-8(3)	5(3)	-1(3)
C(41)	16(4)	14(4)	17(4)	1(3)	-4(3)	-5(3)
C(42)	17(5)	24(5)	14(4)	-6(3)	2(3)	-5(4)
C(43)	24(5)	23(5)	29(5)	-5(4)	1(4)	-4(4)
C(44)	17(5)	21(5)	40(6)	-7(4)	6(4)	-2(4)
C(45)	33(6)	13(4)	34(5)	-9(4)	12(4)	1(4)
C(46)	14(4)	18(4)	16(4)	-1(3)	-3(3)	-8(3)
C(47)	15(4)	18(4)	17(4)	1(3)	0(3)	0(3)
C(48)	15(5)	17(4)	22(4)	-3(3)	6(3)	-7(3)
C(49)	14(4)	20(4)	15(4)	2(3)	-2(3)	1(3)
C(50)	13(4)	18(4)	21(4)	5(3)	0(3)	-3(3)
C(51)	17(5)	16(4)	17(4)	-4(3)	4(3)	-2(3)
C(52)	26(5)	27(5)	16(4)	-2(4)	-6(4)	-3(4)
O(1)	34(4)	8(3)	31(4)	-3(3)	-5(3)	-9(3)

O(2)	21(4)	18(3)	29(4)	-6(3)	2(3)	10(3)
O(3)	39(4)	29(4)	22(3)	-11(3)	6(3)	14(3)
O(4)	21(4)	15(3)	30(4)	-2(3)	2(3)	-2(3)
O(5)	36(4)	15(3)	33(4)	-7(3)	2(3)	9(3)
O(6)	35(4)	26(4)	22(3)	-11(3)	-3(3)	-8(3)
O(7)	11(3)	72(6)	13(3)	-11(3)	2(2)	8(3)
O(8)	69(6)	17(3)	24(4)	-7(3)	-3(4)	23(4)
O(9)	24(4)	24(3)	21(3)	-6(3)	1(3)	-7(3)
O(10)	6(3)	67(5)	22(3)	-7(3)	-1(3)	-8(3)
O(11)	75(6)	17(4)	27(4)	-7(3)	3(4)	-21(4)
O(12)	23(4)	22(3)	23(3)	-3(3)	-1(3)	5(3)
S(1)	19(1)	14(1)	21(1)	-6(1)	-2(1)	-4(1)
S(2)	42(2)	15(1)	28(1)	-10(1)	7(1)	-4(1)
S(3)	24(1)	14(1)	20(1)	-6(1)	-1(1)	0(1)
S(4)	36(2)	16(1)	28(1)	-10(1)	-6(1)	-2(1)
S(5)	22(1)	23(1)	17(1)	-6(1)	-1(1)	11(1)
S(6)	35(2)	28(1)	18(1)	-11(1)	2(1)	-7(1)
S(7)	21(1)	24(1)	17(1)	-6(1)	1(1)	-13(1)
S(8)	30(1)	30(1)	20(1)	-11(1)	1(1)	2(1)

Table S14. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for 180534lt_0m_a.

	x	y	z	U(eq)
H(1A)	13415	1206	2220	26
H(1B)	11508	1310	2564	26
H(4)	7759	749	2841	28
H(5)	4773	-390	3175	31
H(6)	5026	-2098	2985	39
H(8)	6405	515	1733	22

H(9)	5839	-150	1154	28
H(11)	12080	1157	734	28
H(12)	12610	1865	1306	25
H(13A)	9599	372	311	69
H(13B)	6830	122	433	69
H(13C)	8898	-700	531	69
H(14A)	8747	3917	2834	31
H(14B)	7133	3683	2482	31
H(17)	3294	4055	2189	27
H(18)	62	4999	1851	31
H(19)	-267	6715	2020	36
H(21)	1662	4423	3301	26
H(22)	850	5082	3885	30
H(24)	6968	3774	4314	29
H(25)	7790	3119	3732	24
H(26A)	3532	4346	4743	61
H(26B)	1550	5093	4547	61
H(26C)	4303	5460	4571	61
H(28)	-2234	7485	4441	29
H(29)	-4114	6486	3971	38
H(30)	-1706	6551	3380	29
H(32A)	-462	8765	4682	18
H(32B)	1521	9654	4644	18
H(34)	4398	9486	5527	20
H(35)	2605	9841	6102	22
H(37)	-2806	7858	5966	21
H(38)	-1088	7510	5384	22
H(39A)	-901	8575	6666	33
H(39B)	-3165	9096	6450	33
H(39C)	-852	9760	6534	33
H(40A)	4561	6233	10333	18
H(40B)	6499	5330	10355	18
H(43)	2601	7426	10602	30

H(44)	840	8461	11079	31
H(45)	3069	8341	11659	32
H(47)	9417	5516	9472	20
H(48)	7687	5235	8892	22
H(50)	2416	7272	9075	22
H(51)	4136	7567	9649	20
H(52A)	4317	6559	8358	34
H(52B)	1977	6068	8582	34
H(52C)	4230	5371	8484	34

CheckCIF file for compound 3d (CCDC: 2057129)

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 210103LT

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 210103LT

```
Bond precision:  C-C = 0.0022 A          Wavelength=0.71073

Cell:            a=13.3682(5)     b=7.2588(3)     c=26.6061(10)
                 alpha=90         beta=90         gamma=90
Temperature:     100 K

                Calculated                Reported
Volume          2581.78(17)               2581.78(17)
Space group     P b c a                   P b c a
Hall group      -P 2ac 2ab                -P 2ac 2ab
Moiety formula  C14 H11 Cl O3 S           ?
Sum formula     C14 H11 Cl O3 S           C14 H11 Cl O3 S
Mr              294.74                    294.74
Dx,g cm-3       1.517                     1.517
Z               8                         8
Mu (mm-1)       0.457                     0.457
F000            1216.0                    1216.0
F000'           1218.71
h,k,lmax        16,9,33                   16,9,33
Nref            2647                      2647
Tmin,Tmax       0.913,0.977               0.668,0.745
Tmin'           0.913

Correction method= # Reported T Limits: Tmin=0.668 Tmax=0.745
AbsCorr = MULTI-SCAN

Data completeness= 1.000          Theta(max)= 26.415

R(reflections)= 0.0295( 2391)     wR2(reflections)= 0.0771( 2647)

S = 1.043                 Npar= 172
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level G

```
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .     Please Do !
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          1 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.        15 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   0 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   3 ALERT level G = General information/check it is not something unexpected

   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   1 ALERT type 2 Indicator that the structure model may be wrong or deficient
   1 ALERT type 3 Indicator that the structure quality may be low
   0 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

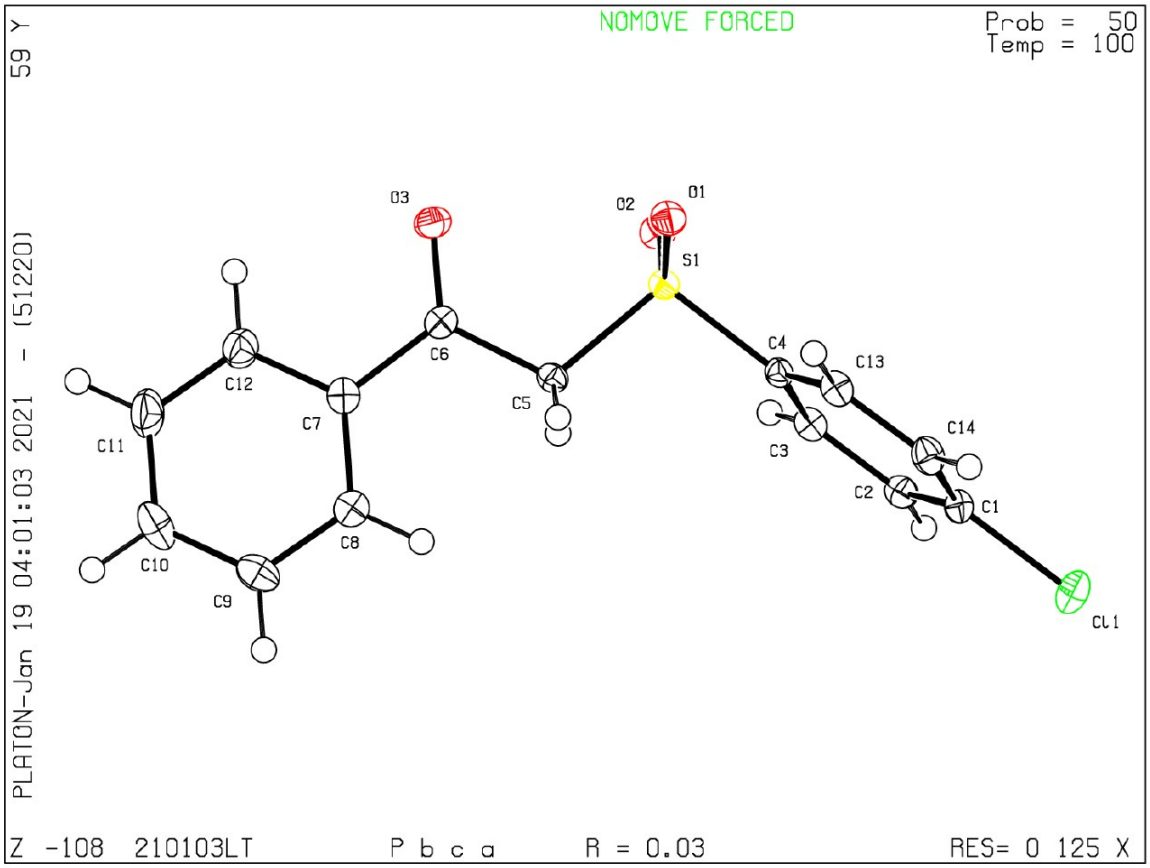
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 05/12/2020; check.def file version of 05/12/2020

Datablock 210103LT - ellipsoid plot



CheckCIF file for compound 4v (CCDC: 2008686)

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 180534lt_0m_a

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: 180534lt_0m_a

```
Bond precision:  C-C = 0.0129 A          Wavelength=0.71073

Cell:          a=5.5069(6)       b=13.3044(16)    c=35.296(4)
               alpha=84.488(5)   beta=88.558(5)   gamma=89.975(5)
Temperature:   100 K

                Calculated                Reported
Volume          2573.2(5)                 2573.2(5)
Space group     P -1                      P -1
Hall group      -P 1                      -P 1
Moiety formula  C13 H12 O3 S2             ?
Sum formula     C13 H12 O3 S2             C13 H12 O3 S2
Mr              280.35                    280.35
Dx,g cm-3       1.447                     1.447
Z               8                         8
Mu (mm-1)       0.410                     0.410
F000            1168.0                    1168.0
F000'           1170.49
h,k,lmax        6,16,44                   6,16,44
Nref            10613                     10470
Tmin,Tmax       0.985,0.988               0.821,0.948
Tmin'           0.921

Correction method= # Reported T Limits: Tmin=0.821 Tmax=0.948
AbsCorr = MULTI-SCAN

Data completeness= 0.987          Theta(max)= 26.479

R(reflections)= 0.1323( 8563)     wR2(reflections)= 0.3075( 10470)

S = 1.214                 Npar= 653
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

```
PLAT340_ALERT_3_B Low Bond Precision on  C-C Bonds ..............    0.01287 Ang.
PLAT930_ALERT_2_B FCF-based   Twin Law ( 6 0 1)[ 1 0 0] Est.d BASF      0.21 Check
PLAT930_ALERT_2_B FCF-based   Twin Law ( 1 0 0)[ 1 0 0] Est.d BASF      0.21 Check
```

Alert level C

```
DIFMX02_ALERT_1_C  The maximum difference density is > 0.1*ZMAX*0.75
           The relevant atom site should be identified.
PLAT082_ALERT_2_C High R1 Value .................................      0.13 Report
PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) ..................      0.31 Report
PLAT097_ALERT_2_C Large Reported Max.  (Positive) Residual Density     1.45 eA-3
PLAT213_ALERT_2_C Atom C13            has ADP max/min Ratio .....      3.3 prolat
PLAT213_ALERT_2_C Atom O7             has ADP max/min Ratio .....      3.2 prolat
PLAT213_ALERT_2_C Atom O8             has ADP max/min Ratio .....      3.4 prolat
PLAT213_ALERT_2_C Atom O10            has ADP max/min Ratio .....      3.8 prolat
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......   27.481 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......    4.492 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......    2.264 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600       99 Report
```

Alert level G

```
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT  Unusually Large     57.32 Why ?
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)     0.005 Degree
PLAT870_ALERT_4_G ALERTS Related to Twinning Effects Suppressed ..         ! Info
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .   Please Do !
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).         1 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        43 Note
PLAT931_ALERT_5_G CIFcalcFCF  Twin Law ( 6 0 1)         Est.d BASF      0.21 Check
PLAT931_ALERT_5_G CIFcalcFCF  Twin Law         [ 1 0 0] Est.d BASF      0.21 Check
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........      3.3 Low
```

```
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   2 ALERT type 5 Informative message, check
```

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PLATON version of 04/06/2020; check.def file version of 02/06/2020

Datablock 180534lt_0m_a - ellipsoid plot