Electronic Supplementary Material (ESI) for New Journal of Chemistry.


# Visible-Light-Induced Arylation *via* Electron-Donor-Acceptor Complex: a Catalyst-Free Approach for the Synthesis of N-(Hetero) Aryl Sulfonamides

Department of Chemistry, Indian Institute of Technology (BHU), Varanasi-221005

Arsala Kamal[a], Vandana Srivastava[a], and Sundaram Singh*[a]

* Address for Correspondence

Dr. Sundaram Singh, Department of Chemistry, Indian Institute of Technology (BHU), Varanasi – 221 005, U.P., India,

E-mail: sundaram.apc@itbhu.ac.in

Tel: +91 9451658650

# Table of Content

## 1. General considerations

All commercially available reagents and solvents were used directly without further purification. All reactions were conducted under an oxygen atmosphere and oven-dried glasswares were used. All reactions were conducted using a blue light-emitting diode (LED) as the visible-light source. The progress of the reaction was measured by thin-layer chromatography (TLC) and visualized using UV light. Melting points were determined on a digital melting point apparatus and temperatures were uncorrected. UV-visible spectroscopy of the reaction solution was recorded on a SHIMADZU UV-3600 UV-visible spectrophotometer. Perkin Elmer Micro analyzer was used for (C, H, and N All the $^{1}H$, $^{13}C$, and $^{19}F$ NMR spectra were recorded through Bruker 500 MHz spectrometer ($^{1}H$ NMR at 500 MHz, $^{13}C$ NMR at 126 MHz, and $^{19}F$ NMR at 126 MHz), in DMSO-$d_6$ and $CDCl_3$ chemical shift was indicated in δ ppm, using TMS as an internal standard. HRMS (m/z) were recorded in an electron ionization or electrospray ionization (ESI) mode on Waters-Q-TOF Premier-HAB213 and Sciex X500R QTOF instruments.

**Experimental Setup**

The reactions were performed using a blue LED strip as the light source. A reaction tube was fixed at 5-6 cm from commercial 280 cm, blue LED strip (10 watts), while a fan was used to cool down the reactor (the reaction temperature within the reaction vessel was measured to be between 30-35 ºC) shown in **Figure S1.**

**1.1 The visible light irradiation setup.**

**Figure S1**. A reaction setup showing the reaction tube was fixed at 5-6 cm from the blue LED strip (280 cm). [Personal photo made by the author- Arsala Kamal]

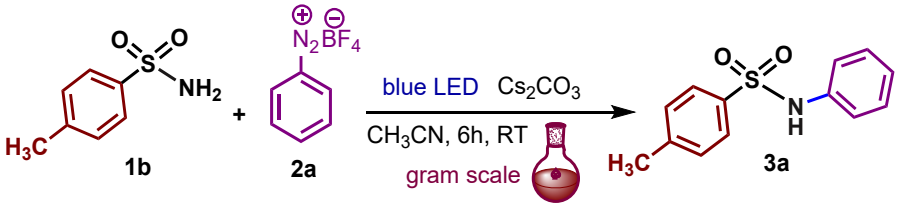
## 2. Experimental Procedures

### 2.1 General procedure for the preparation of N-Aryl Sulfonamide.

A 10 mL reaction tube equipped with a magnetic stirring bar was charged with sulfonamide derivatives **1** (0.6 mmol), diazonium salt derivatives **2** (0.3 mmol), $Cs_2CO_3$(0.6 mmol) as a base, and solvent $CH_3CN$ (4 mL) The mixture was then stirred at room temperature and irradiated with a (280 cm) blue LEDs light strips for 6 h. After completion of the reaction, the reaction mixture was extracted with ethyl acetate (3**x**50 mL). The combined organic phases were dried over anhydrous $Na_2SO_4$, and then the solvent was removed under vacuum. The residue was purified by column chromatography on neutral silica gel (ethyl acetate: hexane)

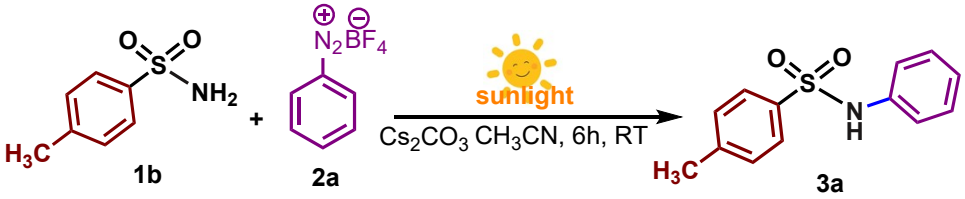
### 2.2 Gram-scale Experiments

The gram-scale synthesis of **3a** was prepared according to the general procedure with blue LED irradiation using sulfonamide derivatives **1b** (6 mmol), diazonium salt **2a** (3 mmol), $Cs_2CO_3$ (6 mmol) as base and solvent in $CH_3CN$ (20 mL) at room temperature for 6 h. The reaction vessel (round bottom flask) was 5 cm away from the blue LED. The isolated yield of **3a** (63%, 0.466 g) was given.




**Scheme S1** Gram-Scale

### 2.3 Application under the sunlight

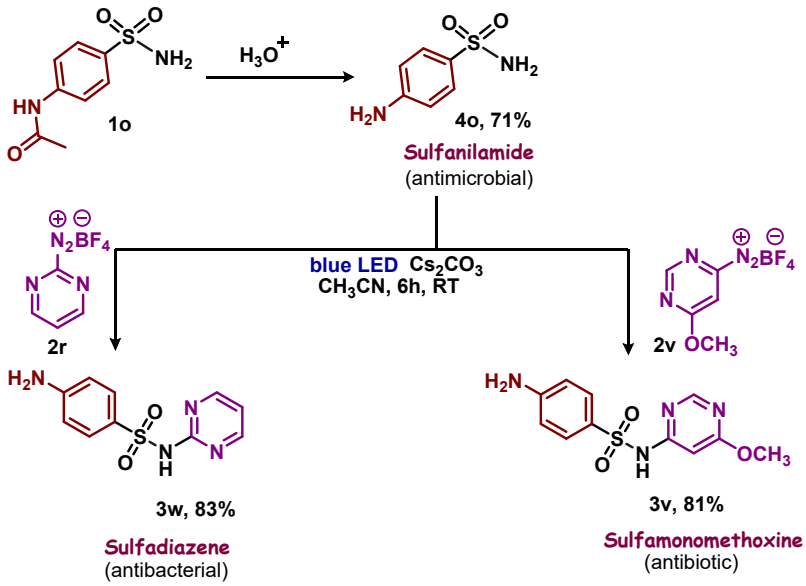



**Scheme S2** Synthesis under the sunlight

The synthesis under the sunlight: The reaction tube was charged with **1b** (0.6 mmol), **2a** (0.3 mmol), and $Cs_2CO_3$ (0.6 mmol) in $CH_3CN$ (4 mL) at room temperature, then the reaction system

was irradiated under sunlight for 6 h (from 10:00 to 16:00; 14/09/2022. IIT BHU, Varanasi, UP India. Temperature: 25 °C – 35 °C) provided the isolated yield of **3a** was 61%.

### 2.4 Synthesis of biologically relevant molecules



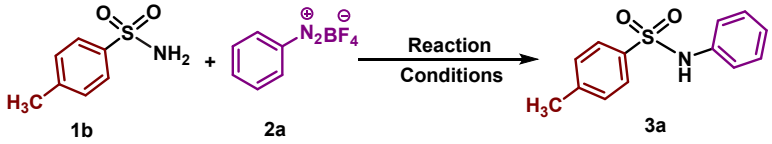

**Scheme S3** Synthesis of biologically relevant molecules

## 3. Optimizations

The reaction's optimization was carried out. Avoiding transition-metal catalysts was the original intention of this research. Based on this idea, we commenced our investigation with benzene diazonium salt **2a**, *p*-toluene sulfonamide **1b**, $NaHCO_3$ as a base in MeCN solvent under blue LED irradiation, gratifyingly desired product N-aryl sulfonamide (**3a**) was obtained in 76% isolated yield in 6 hours (**Table S1, entry 1**). Inspired by this result, benzene diazonium salt and *p*-toluene sulfonamide are selected as the model reaction to optimize reaction conditions such as base, solvents, and blue LED light (**Tables S1 & 2**). Preliminary studies revealed that the base significantly impacted the photoreaction, and $Cs_2CO_3$ was the best, giving the N-aryl sulfonamides **3a** with 90% yield (**Table S1, entry 3**). Other bases, including $Na_2CO_3$, $K_2CO_3$, $Et_3N$, and DBU aware less efficient than $Cs_2CO_3$, giving **3a** in 27−80% yields (**Table S2, entries 1−6**). With $Cs_2CO_3$ as the base, solvent screening revealed that $CH_3CN$ was the best solvent (**Table S1, entries 7−14**). No reaction occurred without a solvent and base (**Table S1, entries 15&16**). The increase in

the amount of base has no significant effect on the yield of the product (**Table S1, entries 17**).

**Table S1 Optimization of reaction conditions**[a]



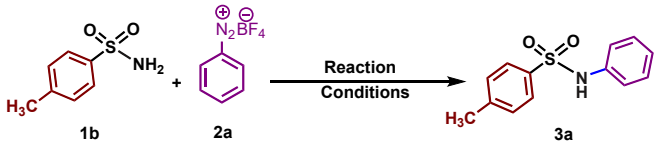

1b 2a 3a

| Entry | base | Solvent | Yield[b]% |
|---|---|---|---|
| 1 | $NaHCO_3$ | $CH_3CN$ | 76 |
| 2 | $Na_2CO_3$ | $CH_3CN$ | 74 |
| 3 | $Cs_2CO_3$ | $CH_3CN$ | 90 |
| 4 | $K_2CO_3$ | $CH_3CN$ | 80 |
| 5 | $NEt_3$ | $CH_3CN$ | 38 |
| 6 | DBU | $CH_3CN$ | 27 |
| 7 | $Cs_2CO_3$ | DCM | 40 |
| 8 | $Cs_2CO_3$ | Ethanol | 74 |
| 9 | $Cs_2CO_3$ | DMC | 78 |
| 10 | $Cs_2CO_3$ | Water | nr |
| 11 | $Cs_2CO_3$ | THF | nr |
| 12 | $Cs_2CO_3$ | 2-MeTHF | nr |
| 13 | $Cs_2CO_3$ | DMF | nr |
| 14 | $Cs_2CO_3$ | DMSO | nr |
| 15 | $Cs_2CO_3$ | - | nr |
| 16 | - | $CH_3CN$ | nr |
| 17[c] | $Cs_2CO_3$ | $CH_3CN$ | 87 |

[a]Reaction conditions: **1b** (0.6 mmol), **2a** (0.3 mmol), base (0.6 mmol), solvent (4 mL), blue LED, ambient room temperature, and 6 h. [c]1.2 mmol $Cs_2CO_3$ [b]Isolated yield.

Moreover, upon extensive reaction optimization (**Table S2**), the best reaction conditions with 90% yield of the desired product **3a** were obtained using $CH_3CN$ (4 mL), **2a**, **1b,** and cesium carbonate ($Cs_2CO_3$) as a base under blue light-emitting diodes (LEDs) at room temperature for 6 hr. (**Table S2, entry 8**). A significant drop in yield was observed while switching from blue LED to other light sources like green, purple, white LED, and 20W CFL (**Table S2, entries 2-5**). After that, the reaction without light gave no product, revealing that light is indispensable in this arylation reaction (**Table S2, entry 6**). Finally, if we prolonged the reaction time, 6 h to 8 h, the yield of the target product was still 90% (**Table S2 entry 7**).

**Table S2 Reaction conditions**[a]




1b 2a 3a

| Entry | variations in the reaction conditions | Yield (%)[b] |
|---|---|---|
| 1 | Blue LEDs | 90 |
| 2 | green LEDs instead of blue LEDs | 28 |
| 3 | Purple LEDs instead of blue LEDs | 46 |
| 4 | white LEDs instead of blue LEDs | 63 |
| 5 | 20W CFL instead of blue LEDs | 49 |
| 6 | no light | nr |
| 7 | reaction for 8 hrs | 90 |
| 8 | Standard Conditions | 90 |

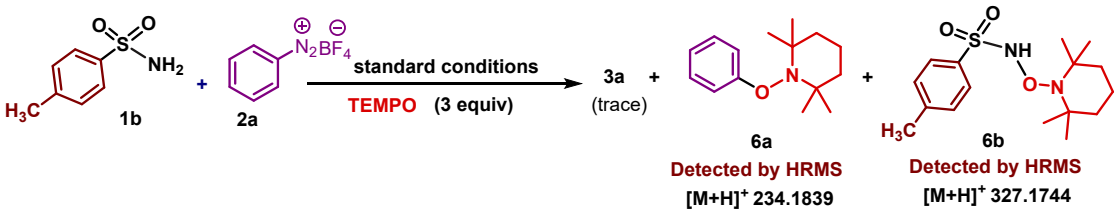
[a] Reaction conditions: **1b** (0.6 mmol), **2a** (0.3 mmol), $Cs_2CO_3$ (0.6 mmol), $CH_3CN$ as a solvent (4 mL), blue LED, ambient room temperature, and 6 h . [b]Isolated yield

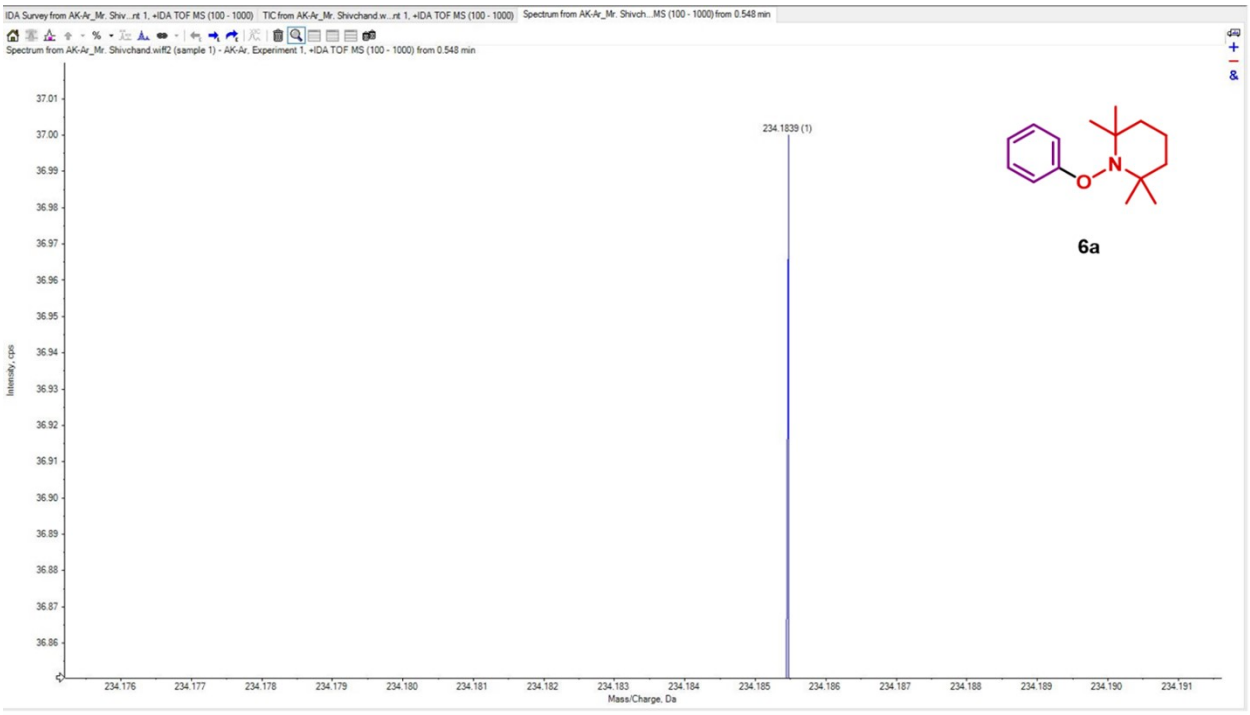
# 4 Mechanistic Studies

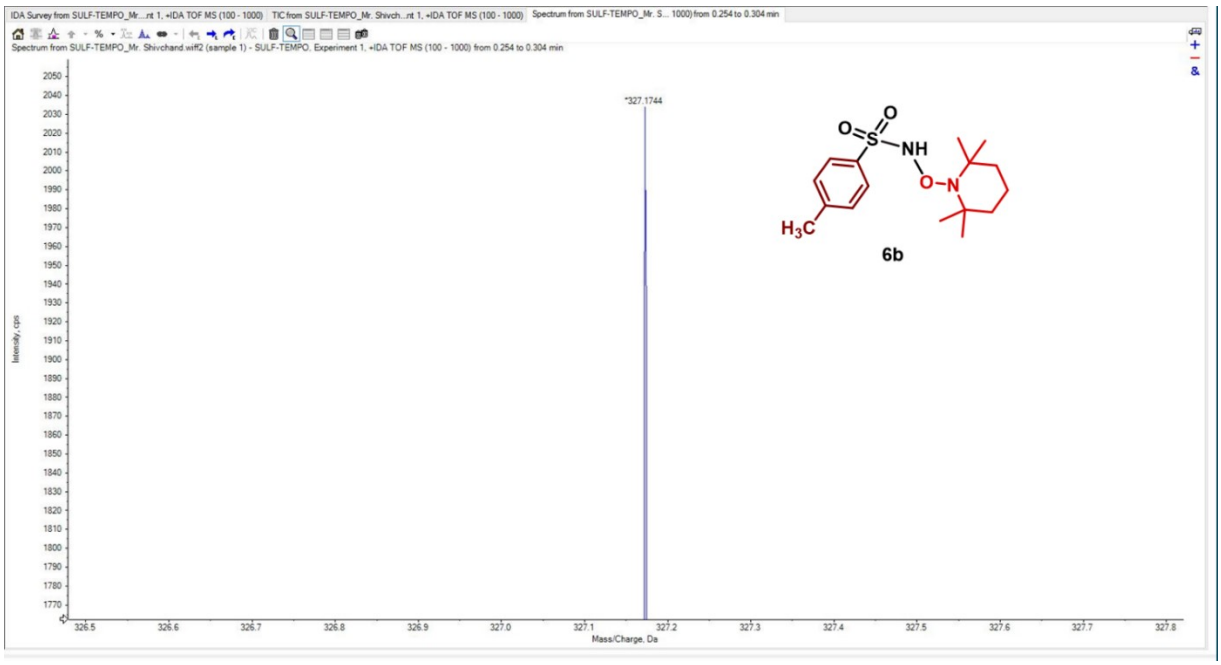
## 4.1 TEMPO radical trapping experiments

Some control experiments were performed to investigate the mechanism of the photocatalyst-free approach for the synthesis of N-aryl Sulfonamides. At first 2, 2, 6, 6-tetramethylpiperidinooxy (TEMPO) (radical scavengers) (3 equiv.) was added to the reaction system, and the trace amount of the **3a** and TEMPO adducts **6a** and **6b** were detected in HRMS data from the crude reaction mixture. These results suggested that the reaction passes through a radical pathway and aryl radical from **2a** and sulfonamide radical from **1b** were involved as a reaction intermediate.

.




IDA Survey from AK-Ar_Mr. Shiv...nt 1, +IDA TOF MS (100 - 1000)
TIC from AK-Ar_Mr. Shivchand.w...nt 1, +IDA TOF MS (100 - 1000)
Spectrum from AK-Ar_Mr. Shivch...MS (100 - 1000) from 0.548 min
Spectrum from AK-Ar_Mr. Shivchand.wiff2 (sample 1) - AK-Ar, Experiment 1, +IDA TOF MS (100 - 1000) from 0.548 min
234.1839 (1)
Intensity, cps
Mass/Charge, Da
6a


IDA Survey from SULF-TEMPO_Mr...nt 1, +IDA TOF MS (100 - 1000)
TIC from SULF-TEMPO_Mr. Shivch...nt 1, +IDA TOF MS (100 - 1000)
Spectrum from SULF-TEMPO_Mr. S... 1000) from 0.254 to 0.304 min
Spectrum from SULF-TEMPO_Mr. Shivchand.wiff2 (sample 1) - SULF-TEMPO, Experiment 1, +IDA TOF MS (100 - 1000) from 0.254 to 0.304 min
*327.1744
Intensity, cps
Mass/Charge, Da
6b

## 4.2 UV/Vis absorption spectrometry.

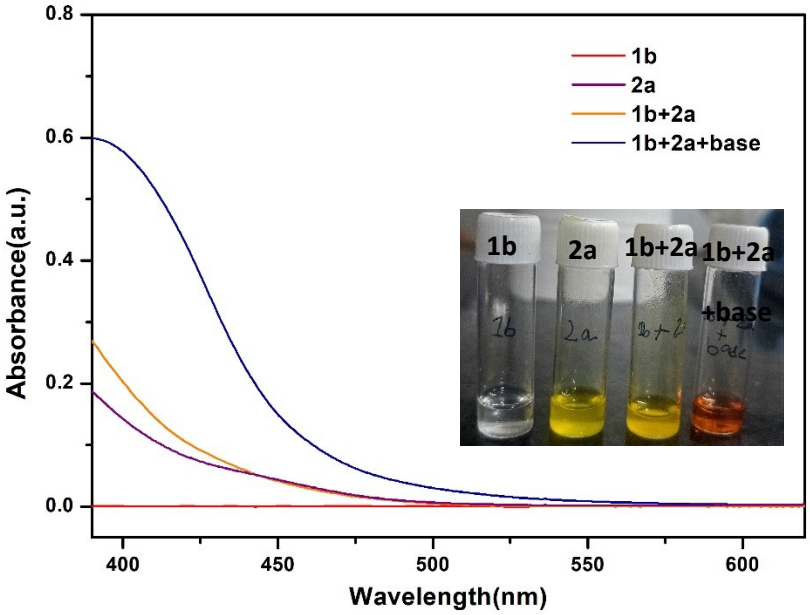
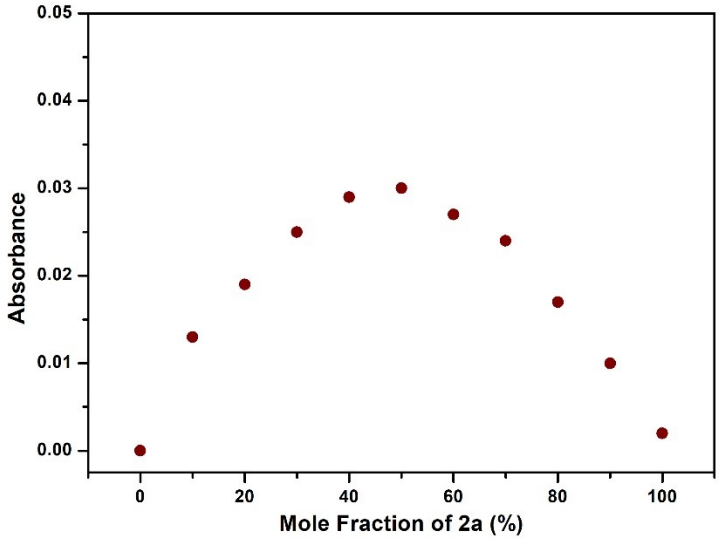



**Figure S2.** Absorption spectra of **1b**, **2a**, and mixture of **1b+2a & 1b+2a + base**. (Dissolved in MeCN).

## 4.3 Stoichiometry of the EDA complex 1b+2a (Jobs plot)




The stoichiometry of the EDA complexes **1b &2a** was calculated using the Job's plot method. The Job's plot of the EDA complex between **1b** and **2a** was calculated by measuring the absorption of Acetonitrile solutions at 490 nm with different donor/acceptor ratios with constant concentration

(0.02 M) of the two components. The absorbance values were plotted against the molar fraction (%) of **2a**. The Job's plot analysis of the EDA complex between **1b** and **2a** showed a maximal absorbance at 50% molar fraction of **2a** indicating the 1:1 stoichiometry of the EDA complex in solution.

## 4.4 ON-OFF Experiments

The reaction between **1b** and **2a** was conducted under standard conditions. The reaction mixture was subjected to sequential periods of stirring under visible-light irradiation (blue LED) followed by stirring in the absence of light. At each time point, one reaction system was suspended, which was then purified with column chromatography to give the corresponding products **3a**. The yield of **3a** was measured by the weight of the product.

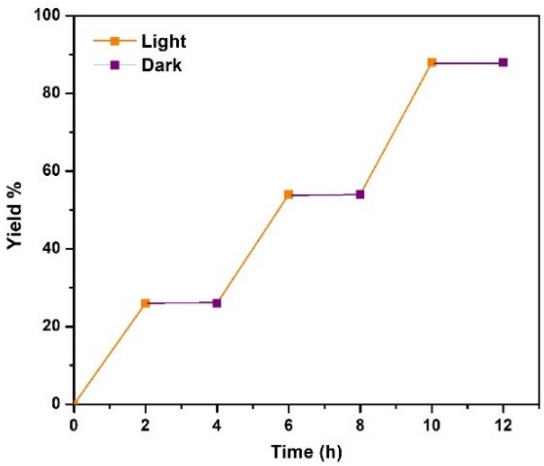
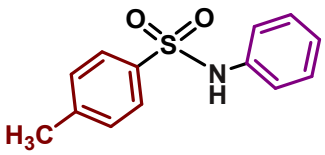


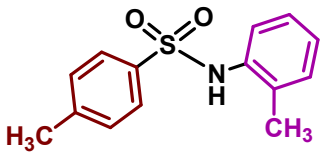

**Figure S3.** On-Off Experiments

## 5. Characterization of the Target Products

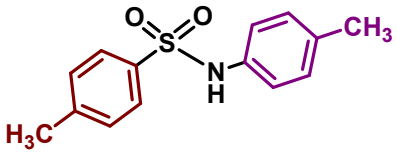



4-methyl-N-phenylbenzenesulfonamide **(3a)** 88% yield. Pale green solid. m.p.: 104-105 °C $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 10.13 (s, 1H), 7.73 (d, $J$ = 9.6 Hz, 2H), 7.59 (t, $J$ = 8.0 Hz, 1H), 7.52-7.55 (m, 2H), 7.02 (d, $J$ = 8.3 Hz, 2H), 6.97 (d, $J$ = 8.5 Hz, 2H), 2.17 (s, 3H). $^{13}$C NMR (126

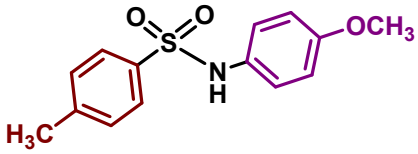
MHz, DMSO $d_6$) δ 139.98, 135.45, 133.88, 133.23, 130.03, 129.64, 127.11, 121.11, 20.75. HRMS (ESI) m/z: [M+H] + calculated for $C_{13}H_{14}NO_2S$ 248.0745; found: 248.0733



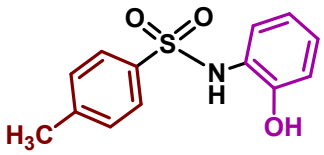
4-methyl-N-(o-tolyl)benzenesulfonamide **(3b)** 84% yield. Pink solid. m.p.: 106-107 °C $^1$H NMR (500 MHz, DMSO $d_6$) δ 9.59 (s, 1H), 7.48 (d, $J$ = 8.0 Hz, 2H), 7.30-7.34 (m, 2H), 7.13-7.29 (m, 2H), 7.12 (d, $J$ = 7.8 Hz, 2H), 2.30 (d, $J$ = 6.9 Hz, 6H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 146.04, 138.18, 131.80, 131.58, 128.56, 128.15, 127.64, 125.95, 123.18, 21.25, 17.29 HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{16}NO_2S$ 262.0901; found: 263.0917



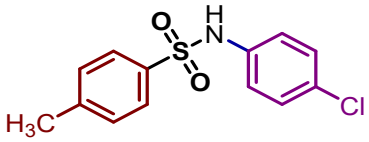
4-methyl-N-(p-tolyl)benzenesulfonamide **(3c)** 86% yield. White solid. m.p.: 114-116 °C $^1$H NMR (500 MHz, DMSO $d_6$) δ 9.95 (s, 1H), 7.51 (d, $J$ = 7.9 Hz, 2H), 7.31 (d, $J$ = 8.3 Hz, 2H), 7.26 (d, $J$ = 8.2 Hz, 2H), 7.14 (d, $J$ = 7.9 Hz, 2H), 2.32 (s, 3H), 2.30 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 145.53, 138.53, 138.40, 130.68, 129.21, 128.68, 125.95, 123.54, 21.26, 20.99. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{16}NO_2S$ 262.0901; found: 262.0912



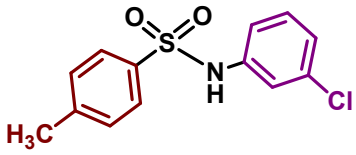
N-(4-methoxyphenyl)-4-methylbenzenesulfonamide **(3d)** 85% yield. Pale pink solid. m.p.: 115-117 °C $^1$H NMR (500 MHz, DMSO $d_6$) δ 7.97 (s, 1H), 7.68 (d, $J$ = 8.2 Hz, 2H), 7.38 (d, $J$ = 8.0 Hz, 2H), 7.14 (d, $J$ = 8.7 Hz, 2H), 6.84 (d, $J$ = 8.7 Hz, 2H), 3.72 (s, 3H), 2.39 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 158.92, 142.96, 138.35, 130.04, 129.97, 129.39, 127.01, 114.09, 55.53, 21.43 HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{16}NO_3S$ 278.0850; found: 278.0852



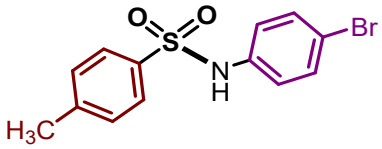
N-(2-hydroxyphenyl)-4-methylbenzenesulfonamide **(3e)** 84% yield. Gray solid. m.p.: 137-138 °C $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 10.70 (s, 1H), 9.73 (s, 1H), 7.50 (d, *J* = 8.1 Hz, 2H), 7.25-7.29 (m, 2H), 7.13 (d, *J* = 7.8 Hz, 2H), 7.02 (d, *J* = 8.1 Hz, 1H), 6.89 (t, *J* = 8.3 Hz, 1H), 2.29 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 151.17, 145.96, 138.24, 129.90, 128.58, 125.96, 124.52, 119.96, 119.32, 116.62, 21.26. HRMS (ESI) m/z: [M+H] + calculated for C13H14NO3S 264.0694; found: 264.0698.



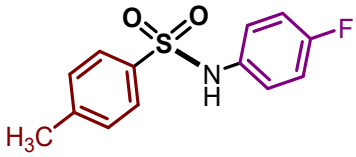
N-(4-chlorophenyl)-4-methylbenzenesulfonamide **(3f):** 87% yield. White solid. m.p.: 120-122 °C $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.21 (d, *J* = 7.9 Hz, 2H), 7.06 (d, *J* = 8.8 Hz, 2H), 2.40 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 144.23, 135.61, 135.22, 130.77, 129.82, 129.40, 127.29, 122.79, 21.57. HRMS (ESI) m/z: [M+H] + calculated for $C_{13}H_{13}NO_2SCl$ 282.0355; found: 282.0358



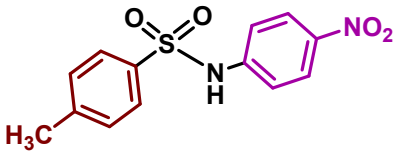
N-(3-chlorophenyl)-4-methylbenzenesulfonamide **(3g)** 87% yield. White solid. m.p.: 114-115 °C $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.50 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.19 (s, 1H), 7.14 (d, *J* = 7.8 Hz, 2H), 7.08 (d, *J* = 8.0 Hz, 1H), 6.89 (t, *J* = 7.6 Hz, 1H), 2.30 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 145.71, 139.43, 138.41, 130.00, 128.63, 128.51, 125.96, 122.22, 121.34, 119.53, 21.26. HRMS (ESI) m/z: [M+H] + calculated for $C_{13}H_{13}NO_2SCl$ 282.0355; found: 282.0362



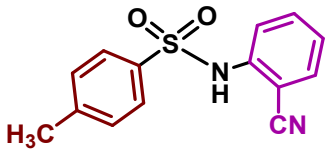

N-(4-bromophenyl)-4-methylbenzenesulfonamide **(3h):** 92% yield. White solid. m.p.: 140-145°C $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.21 (d, *J* = 8.8 Hz, 2H), 7.05 (d, *J* = 8.8 Hz, 2H), 2.40 (s, 26H).$^{13}$C NMR (126 MHz, $CDCl_3$) δ 144.23, 135.62, 135.16, 130.84, 129.81, 129.42, 127.28, 122.86, 21.59.HRMS (ESI) m/z: [M+H] + calculated for $C_{13}H_{13}NO_2SBr$ 325.9850; found: 325.9851



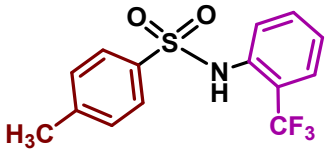

N-(4-fluorophenyl)-4-methylbenzenesulfonamide **(3i)**: 86% yield. White solid. m.p.: 98-99 °C $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 10.17 (s, 1H), 7.60 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.08 (d, *J* = 6.8 Hz, 4H), 2.33 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 144.24, 142.13, 136.78, 130.38, 127.17, 127.03, 127.00, 119.09, 21.42 $^{19}$F NMR (471 MHz, CDCl3) δ -116.24 – -116.48. HRMS (ESI) m/z: [M+H] + calculated for $C_{13}H_{13}NO_2SF$ 266.0651; found: 266.0654



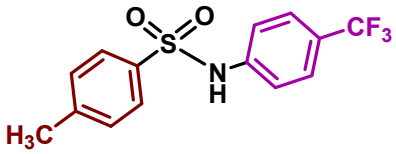

4-methyl-N-(4-nitrophenyl)benzenesulfonamide **(3*J*):** 91% yield. Pale gray solid. m.p.: 180-182 °C. $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.95 (d, *J* = 9.2 Hz, 2H), 7.52 (d, *J* = 8.1 Hz, 2H), 7.16 (d, *J* = 8.9 Hz, 2H), 6.63 (d, *J* = 9.2 Hz, 2H), 2.29 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 155.84, 145.25, 138.71, 136.28, 128.73, 126.83, 125.97, 113.12, 21.25. HRMS (ESI) m/z: [M+H] + calculated for $C_{13}H_{13}N_2O_4S$ 293.0596; found: 293.0599



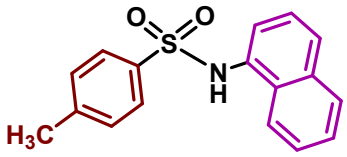
N-(2-cyanophenyl)-4-methylbenzenesulfonamide **(3k)** 86% yield. White solid. m.p.: 172-173 °C $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.51 (d, $J$ = 8.1 Hz, 2H), 7.32-7.39 (m, 1H), 7.29-7.31 (m,1H), 7.14-7.16 (m, 2H), 6.80-6.82 (m, 1H), 6.60-6.63 (m,1H), 2.30 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 151.61, 145.41, 138.60, 134.45, 132.93, 128.69, 125.98, 118.53, 116.71, 115.92, 94.23, 21.26. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S$ 273.0697; found: 273.0699



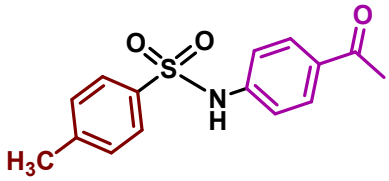
4-methyl-N-(2-(trifluoromethyl)phenyl)benzenesulfonamide **(3l):** 84% yield. White solid. m.p.: 124-125 °C $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 10.83 (s, 1H), 7.72 (d, $J$ = 8.3 Hz, 2H), 7.60 (d, $J$ = 8.6 Hz, 2H), 7.37 (d, $J$ = 8.1 Hz, 2H), 7.29 (d, $J$ = 8.5 Hz, 2H), 2.34 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 144.24, 142.13, 136.78, 130.38, 127.17, 127.03, 127.00, 124.00, 123.00, 119.09, 21.42 $^{19}$F NMR (471 MHz, DMSO $d_6$) δ -61.39. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_2SF_3$ 316.0619; found: 316.0619



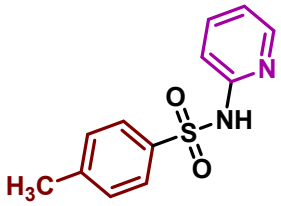
4-methyl-N-(4-(trifluoromethyl)phenyl)benzenesulfonamide **(3m):** 88% yield. Cream solid. m.p.: 144-145 °C, $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, $J$ = 8.1 Hz, 2H), 7.50 (d, $J$ = 8.1 Hz, 2H), 7.28-7.30 (m,2H), 7.21 (t, $J$ = 8.6 Hz, 2H), 2.44 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 144.57, 140.03, 135.69, 129.96, 127.27, 126.62, 126.59, 125.01, 122.85, 119.60, 21.54. $^{19}$F NMR (471 MHz, $CDCl_3$) δ -62.20.HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_2SF_3$ 316.0619; found: 315.0620



4-methyl-N-(naphthalen-1-yl)benzenesulfonamide **(3n):** 90% yield. brownish gray solid. m.p.: 109-111°C $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 10.49 (s, 1H), 7.80 (d, *J* = 7.9 Hz, 3H), 7.71 (d, *J* = 8.3 Hz, 2H), 7.58 (s, 1H), 7.42-7.45 (m,1H), 7.38 (t, *J* = 6.9 Hz, 1H), 7.30-7.32 (m, 3H), 2.27 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 143.76, 137.06, 135.98, 133.70, 130.40, 130.15, 129.48, 127.95, 127.62, 127.23, 127.11, 125.44, 120.70, 116.20, 21.37. HRMS (ESI) m/z: [M+H] + calculated for $C_{17}H_{16}NO_2S$ 298.0901; found: 298.0912

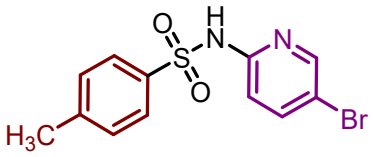


N-(4-acetylphenyl)-4-methylbenzenesulfonamide **(3o):** 79% yield. Brown solid. m.p.: 198-200 $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.84 (d, *J* = 8.7 Hz, 2H), 7.50 (d, *J* = 8.1 Hz, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 6.96 (d, *J* = 8.6 Hz, 2H), 2.47 (s, 3H), 2.30 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 196.31, 145.50, 138.54, 130.73, 128.68, 125.96, 117.61, 26.72, 21.26. HRMS (ESI) m/z: [M+H] + calculated for $C_{15}H_{16}NO_3S$ 290.0850; found: 290.0844

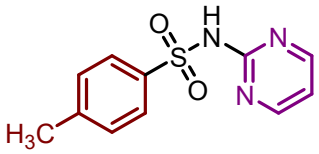


4-methyl-N-(pyridin-2-yl)benzenesulfonamide **(3p):** 79% yield. light yellow solid m.p.: 213-214 °C $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 11.87 (s, 1H), 8.02 (d, *J* = 6.1 Hz, 1H), 7.76 (d, *J* = 8.3 Hz, 2H), 7.70 (t, *J* = 8.9 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.6 Hz, 1H), 6.86-7.88 (m, 1H),

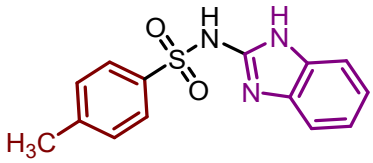
2.34 (s, 3H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 153.44, 142.94, 140.60, 139.39, 129.85, 127.08, 116.25, 114.01, 21.40. HRMS (ESI) m/z: [M+H] + calculated for $C_{12}H_{13}N_2O_2S$ 249.0697; found: 249.0699



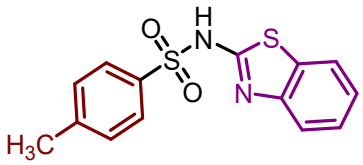
N-(5-bromopyridin-2-yl)-4-methylbenzenesulfonamide **(3q):** 81% yield. Yellow liquid b.p.: 222-224 °C (reptd.) $^1$H NMR (500 MHz, $CDCl_3$) δ 7.76 (s, 2H), 7.53-7.56 (m, 1H), 7.37 (d, *J* = 8.9 Hz, 1H), 7.27 (d, *J* = 8.2 Hz, 2H), 6.49 (d, *J* = 8.8 Hz, 1H), 4.84 (s, 1H), 2.41 (s, 3H).$^{13}$C NMR (126 MHz, $CDCl_3$) δ 156.76, 149.18, 144.39, 141.43, 140.75, 129.91, 127.18, 113.47, 110.57, 21.60. HRMS (ESI) m/z: [M+H] + calculated for $C_{12}H_{12}N_2O_2SBr$ 326.9802; found: 326. 97.98



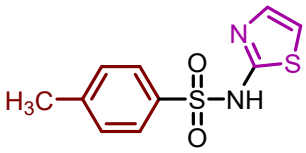
4-methyl-N-(pyrimidin-2-yl)benzenesulfonamide **(3r):** 85% yield. White solid. m.p.: 213-214 °C $^1$H NMR (500 MHz, DMSO $d_6$) δ 8.65 (d, *J* = 7.6 Hz, 2H), 7.54 (d, *J* = 7.8 Hz, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 7.00 (d, *J* = 8.8 Hz, 1H), 2.29 (s, 3H). HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{14}N_3O_2S$ 250.0650; found: 250.0654



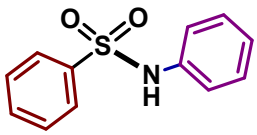
N-(1H-benzo[d]imidazol-2-yl)-4-methylbenzenesulfonamide **(3s)**: 86% yield. White solid. m.p.: 213-214 °C $^1$H NMR (500 MHz, DMSO $d_6$) δ 8.44 (s, 2H), 7.52 (d, *J* = 7.6 Hz, 2H), 7.39 – 7.33 (m, 1H), 7.24 – 7.19 (m, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 2.28 (s, 3H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 151.06, 145.30, 138.70, 130.06, 128.73, 125.9, 123.54, 111.81, 21.24. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{14}N_3O_2S$ 288.0806; found: 288.0802



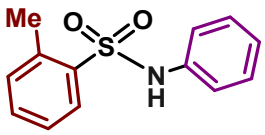
N-(benzo[d]thiazol-2-yl)-4-methylbenzenesulfonamide **(3t):** 79% yield. Cream white solid. m.p.: 243-245 °C $^1$H NMR (500 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 6.3 Hz, 4H), 7.13 (d, *J* = 4.6 Hz, 2H), 6.52 (d, *J* = 4.6 Hz, 1H), 5.60 (s, 1H), 2.42 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 169.79, 143.21, 138.10, 129.46, 126.75, 124.38, 107.84, 29.72, 21.56. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 304.0340; found: 304.0339



4-methyl-N-(thiazol-2-yl)benzenesulfonamide **(3u):** 78% yield. Yellow solid. m.p.: 137-138 °C $^1$H NMR (500 MHz, DMSO $d_6$) δ 12.69 (s, 1H), 7.69 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.24 (d, *J* = 7.6 Hz, 1H), 6.82 (d, *J* = 7.6 Hz, 1H), 2.35 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 169.27, 142.62, 140.00, 129.80, 126.29, 124.89, 108.60, 21.40. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 254.0184; found: 254.0183

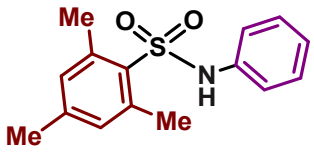


N-phenylbenzenesulfonamide **(4a):** 92% yield, White solid. m.p.: 105-106 °C, $^1$H NMR (500 MHz, DMSO $d_6$) δ 7.60 (t, *J* = 7.9 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 4H), 7.46 (s, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.14 (d, *J* = 8.3 Hz, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 145.53, 138.53, 138.40, 130.68, 129.21, 128.68, 125.95, 123.54. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 233.0510; found: 233.0509

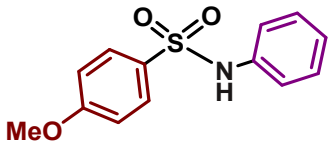


2-methyl-N-phenylbenzenesulfonamide **(4b):** 82% yield, White solid. m.p.: 68-70 °C $^1$H NMR (500 MHz, DMSO $d_6$) δ 9.59 (s, 1H), 7.65 (d, *J* = 7.3 Hz, 3H), 7.55 (t, *J* = 7.8 Hz, 2H), 7.15 – 7.12

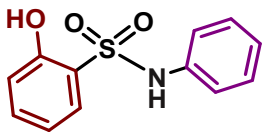
(m, 1H), 7.08-7.10 (m,2H), 6.98 – 6.94 (m, 1H), 1.96 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 141.01, 135.21, 134.61, 133.18, 131.19, 129.64, 126.97, 126.95, 126.79, 17.99. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 247.0667; found: 247.0668



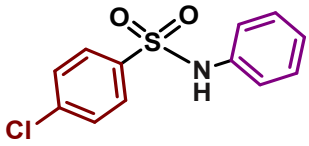

2,4,6-trimethyl-N-phenylbenzenesulfonamide **(4c):** 48% yield, White solid. m.p.: 168-169 °C, $^1$H NMR (500 MHz, DMSO $d_6$) δ 10.18 (s, 1H), 7.21 (t, $J$ = 7.9 Hz, 2H), 7.01 – 6.95 (m, 5H), 2.55 (s, 6H), 2.22 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 142.48, 139.09, 138.11, 134.30, 132.25, 129.60, 123.99, 119.55, 22.91, 20.83. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 275.0980; found: 275.0982



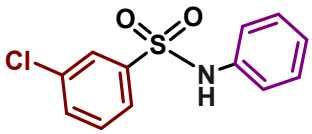

4-methoxy-N-phenylbenzenesulfonamide **(4d):** 93% yield, White solid. m.p.: 105-106 °C $^1$H NMR (500 MHz, DMSO $d_6$) δ 9.92 (s, 1H), 7.67-7.69 (m, 2H), 7.58-7.61 (m, 1H), 7.53 (t, $J$ = 7.5 Hz, 2H), 6.97 (d, $J$ = 9.0 Hz, 2H), 6.80 (d, $J$ = 9.0 Hz, 2H), 3.66 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 157.00, 139.91, 133.17, 130.52, 129.59, 127.13, 123.93, 114.74, 55.59. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 263.0616; found: 263.0318



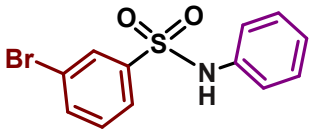

2-hydroxy-N-phenylbenzenesulfonamide **(4e):** 88% yield, brown solid. m.p.: 121-122 °C, $^1$H NMR (500 MHz, DMSO $d_6$) δ 9.23 (s, 2H), 7.73 (d, $J$ = 7.3 Hz, 2H), 7.53 (t, $J$ = 7.3 Hz, 1H), 7.47 (t, $J$ = 7.4 Hz, 2H), 7.07 (d, $J$ = 7.8 Hz, 1H), 6.80 (t, $J$ = 7.6 Hz, 1H), 6.67 (d, $J$ = 7.7 Hz, 1H), 6.62 (t, $J$ = 7.6 Hz, 1H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 150.53, 142.56, 132.23, 129.12, 127.00, 124.60, 123.47, 119.23, 114.89. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 249.0460; found: 249.0461



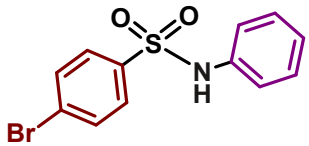
4-chloro-N-phenylbenzenesulfonamide **(4f)** 83% yield, White solid. m.p.: 103-105 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.73-7.75 (m, 2H), 7.55 (t, $J$ = 6.7 Hz, 1H), 7.50-7.54 (m, 2H), 7.20-7.22 (m, 2H), 7.02-7.04 (m, 2H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 141.33, 139.84, 132.71, 129.51, 129.29, 126.99, 126.79, 122.09. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 267.0120; found: 267.0117



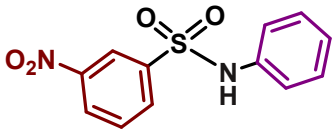
3-chloro-N-phenylbenzenesulfonamide **(4g):** 81% yield, White solid. m.p.: 90-92 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.74 (d, $J$ = 9.6 Hz, 2H), 7.46 (d, $J$ = 7.3 Hz, 3H), 7.04 (t, $J$ = 8.1 Hz, 1H), 6.94 (s, 1H), 6.83 (d, $J$ = 8.2 Hz, 1H), 6.71 (d, $J$ = 9.7 Hz, 1H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 144.14, 133.31, 131.39, 130.82, 130.39, 129.06, 126.80, 119.63, 119.16, 115.36. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 267.0120; found: 267.0115



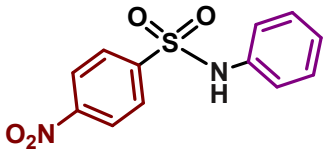
3-bromo-N-phenylbenzenesulfonamide **(4h):** 87% yield, White solid. m.p.: 105-106 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.94-7.95 (m, 2H), 7.80 (d, $J$ = 8.2 Hz, 2H), 7.49 (d, $J$ = 7.3 Hz, 2H), 7.01 (d, $J$ = 9.3 Hz, 1H), 6.74 (s, 1H), 6.60 (d, $J$ = 9.2 Hz, 1H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 138.83, 136.09, 131.91, 129.28, 126.88, 126.76, 125.68, 119.03, 112.84 HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 310.9616; found: 310.9614



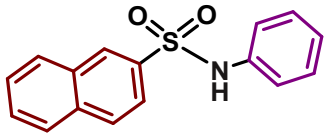
4-bromo-N-phenylbenzenesulfonamide **(4i):** 87% yield, yellowish White solid. m.p.: 120-121 °C, $^1$H NMR (500 MHz, DMSO $d_6$) δ 7.76 (d, *J* = 8.2 Hz, 2H), 7.46-7.52 (m, 3H), 7.15 (d, *J* = 8.9 Hz, 2H), 6.97 (d, *J* = 8.9 Hz, 2H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 142.49, 132.13, 129.31, 129.07, 126.91, 124.53, 122.09. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 310.9616; found: 310.9617



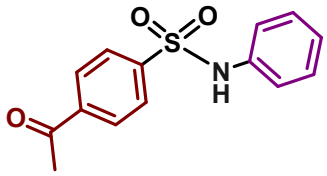
3-nitro-N-phenylbenzenesulfonamide **(4*J*):** 83% yield, orange solid. m.p.: 110-111 °C, $^1$H NMR (500 MHz, DMSO $d_6$) δ 7.72-7.73 (m, *J* = 7.4 Hz, 2H), 7.65 (t, *J* = 7.2 Hz, 1H), 7.37 (d, *J* = 7.7 Hz, 3H), 7.28 (d, *J* = 6.7 Hz, 1H), 7.15 (t, *J* = 7.9 Hz, 1H), 7.11 (d, *J* = 7.4 Hz, 1H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 152.01, 148.77, 146.64, 130.14, 129.26, 128.62, 127.57, 126.57, 113.57, 110.92, HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 278.0361; found: 278.0360



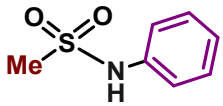
4-nitro-N-phenylbenzenesulfonamide **(4k):** 87% yield, Yellow solid. m.p.: 137-138 °C $^1$H NMR (500 MHz, DMSO $d_6$) δ 7.72 (d, *J* = 7.8 Hz, 2H), 7.45-7.46 (m, 3H), 7.08 (d, *J* = 8.0 Hz, 2H), 6.92-6.94 (m, 2H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 150.15, 143.79, 131.65, 129.10, 128.85, 126.84, 122.10, 115.62. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}N_2O_2S_2$ 278.0361; found: 278.0363



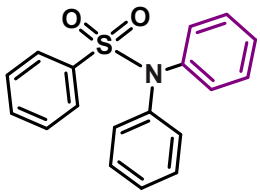
N-phenylnaphthalene-2-sulfonamide **(4l):** 87% yield, white solid. m.p.: 133-134 °C, $^1$H NMR (500 MHz, DMSO $d_6$) δ 7.74-7.75 (m, 2H), 7.59 (m, *J* = 8.4 Hz, 1H), 7.52-7.56 (m, 2H), 7.37 (s, 3H), 7.22 (d, *J* = 8.9 Hz, 2H), 7.04 (d, *J* = 8.9 Hz, 2H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 141.08, 139.45, 132.83, 129.56, 129.34, 128.80, 127.01, 124.73, 123.61, 122.08. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 283.0667; found: 283.0666



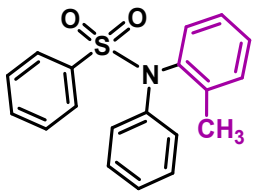
4-acetyl-N-phenylbenzenesulfonamide **(4m):** 87% yield, Cream white solid. m.p.: 134-135 °C $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.76 (d, *J* = 7.9 Hz, 1H), 7.64-7.67 (m, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 1H), 6.56 (d, *J* = 8.8 Hz, 2H), 6.04 (s, 1H), 2.38 (s, 3H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 195.40, 154.09, 131.04, 130.00, 129.06, 126.77, 125.28, 119.14, 112.91, 26.32. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 275.0616; found: 275.0615



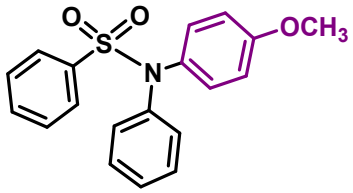
N-phenylmethanesulfonamide **(4n):** 88% yield, White solid. m.p.: 100-101 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.34 (t, *J* = 7.9 Hz, 2H), 7.21-7.22 (m, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 2.98 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 138.93, 129.76, 124.26, 120.23, 39.64 HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 173.0354; found: 173.0355



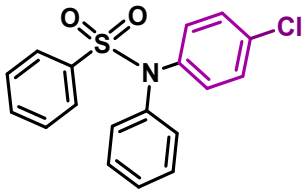
N,N-diphenylbenzenesulfonamide **(5a):** 71% yield, White solid. m.p.: 135-136 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.73 (d, *J* = 9.5 Hz, 2H), 7.54 – 7.48 (m, 3H), 7.15-7.18 (m, 4H), 7.00 (d, *J* = 8.7 Hz, 2H), 6.76 (d, *J* = 8.6 Hz, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 157.79, 132.37, 129.83, 129.39, 129.16, 126.94, 122.08, 119.25, 115.69, HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 309.0823; found: 309.0821



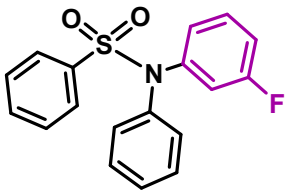
N-phenyl-N-(o-tolyl)benzenesulfonamide **(5b):** 68% yield, White solid. m.p.: 128-129 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.63-7.67 (m, 3H), 7.55 (t, *J* = 7.6 Hz, 2H), 7.12-7.18 (m, 2H), 7.08-7.10 (m, 3H), 6.98 – 6.95 (m, 1H), 6.81 – 6.72 (m, 3H), 1.97 (s, 3H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 157.77, 141.03, 135.22, 134.63, 133.17, 131.19, 129.84, 129.63, 126.98, 126.94, 126.78, 119.28, 115.69, 17.99. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 323.0980; found: 323.0981



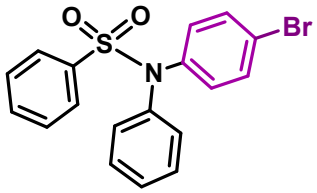

N-(4-methoxyphenyl)-N-phenylbenzenesulfonamide **(5c)** 78% yield, White solid. m.p.: 112-113 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.67-7.69 (m, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.53 (t, *J* = 7.5 Hz, 2H), 7.18 – 7.15 (m, 2H), 6.97 (d, *J* = 7.0 Hz, 2H), 6.80 (d, *J* = 9.0 Hz, 2H), 6.77 – 6.75 (m, 3H), 3.66 (s, 3H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 157.77, 157.00, 139.88, 133.18, 130.51, 129.84, 129.59, 127.13, 123.93, 119.27, 115.68, 114.73, 55.58. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 339.0929; found: 339.0931



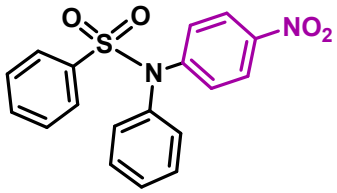

N-(4-chlorophenyl)-N-phenylbenzenesulfonamide (**5d**) 76% yield, White solid. m.p.: 138-139 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.72-7.74 (m, 2H), 7.45-7.46(m, 3H), 7.15-7.18 (m, 2H), 7.04 (t, *J* = 8.1 Hz, 1H), 6.93 (t, *J* = 7.1 Hz, 1H), 6.83 (d, *J* = 9.0 Hz, 1H), 6.75-6.78 (m, 3H), 6.70 (d, *J* = 9.1 Hz, 1H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 157.79, 133.30, 131.36, 130.38, 129.84, 129.05, 126.78, 119.59, 119.25, 119.17, 115.69. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 343.0434; found: 343.0433



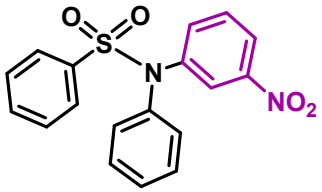
N-(3-fluorophenyl)-N-phenylbenzenesulfonamide (**5e**) 76 % yield, White solid. m.p.: 137-138 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.74 (d, *J* = 8.0 Hz, 1H), 7.48-7.53 (m, 2H), 7.15-7-18 (m, 5H), 7.00 (d, *J* = 8.9 Hz, 1H), 6.75-6.78 (m, 5H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 157.79, 132.31, 129.84, 129.37, 129.14, 126.94, 122.08, 119.25, 115.69. $^{19}$F NMR (471 MHz, DMSO $d_6$) δ -105.59. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 327.0729; found: 327.0731



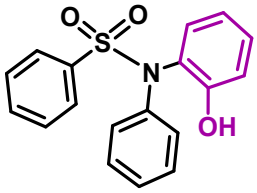

N-(4-bromophenyl)-N-phenylbenzenesulfonamide (**5f**) 77% yield, White solid. m.p.: 137-138 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.75 (d, *J* = 7.0 Hz, 2H), 7.58 (t, *J* = 7.9 Hz, 1H), 7.52 (t, *J* = 7.3 Hz, 2H), 7.24 (d, *J* = 8.9 Hz, 2H), 7.16 (t, *J* = 7.2 Hz, 2H), 7.06 (d, *J* = 8.9 Hz, 2H), 6.77 (t, *J* = 6.6 Hz, 3H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 157.80, 140.76, 138.95, 132.98, 129.83, 129.61, 129.40, 127.41, 127.04, 122.08, 119.25, 115.69. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 386.9929; found: 386.9926



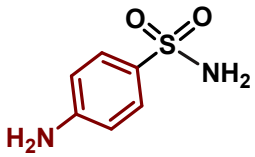

N-(4-nitrophenyl)-N-phenylbenzenesulfonamide (**5g**) 83% yield, Yellow solid. m.p.: 130-131 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 8.24 (s, 2H), 7.93 (d, *J* = 8.8 Hz, 2H), 7.14-7.18 (m, 2H), 7.01 (t, *J* = 8.9 Hz, 2H), 6.89 (d, *J* = 7.3 Hz, 2H), 6.75-6.78 (m, 3H), 6.63-6.68 (m, 1H).$^{13}$C NMR (126 MHz, DMSO $d_6$) δ 157.80, 129.81, 128.89, 128.84, 128.08, 128.05, 124.23, 124.19, 121.29, 119.22, 115.69. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 354.0674; found: 354.0679



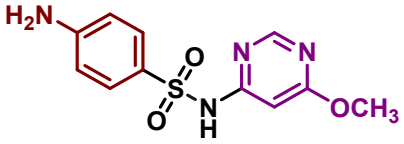
N-(3-nitrophenyl)-N-phenylbenzenesulfonamide (**5h**) 81% yield, orange solid. m.p.: 136-137 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.72 (s, 1H), 7.47-7.53 (m, 2H), 7.15-7.18 (m, 5H), 7.00 (d, *J* = 8.9 Hz, 1H), 6.76 (s, 5H), 13C NMR (126 MHz, DMSO $d_6$) δ 157.89, 129.84, 129.49, 129.14, 126.94, 122.17, 119.25, 115.69. HRMS (ESI) m/z: [M+H] + calculated for $C_{14}H_{13}NO_3S$ 354.0674; found: 354.0671



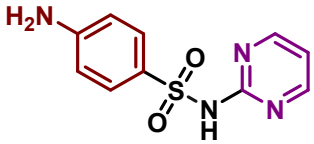
N-(2-hydroxyphenyl)-N-phenylbenzenesulfonamide (**5i**) 84% yield, White solid. m.p.: 112-113 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 9.16 (s, 1H), 7.73 (d, *J* = 8.6 Hz, 3H), 7.58 – 7.53 (m, 2H), 7.49 (t, *J* = 7.5 Hz, 3H), 7.20 – 7.13 (m, 1H), 7.10 – 7.07 (m, 1H), 6.84 (d, *J* = 7.8 Hz, 1H), 6.76 (d, *J* = 8.6 Hz, 1H), 6.69 (d, *J* = 8.0 Hz, 1H), 6.64 (d, *J* = 6.3 Hz, 1H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 157.78, 150.62, 142.08, 132.47, 129.83, 129.18, 127.05, 126.44, 125.30, 124.01, 119.28, 119.26, 115.69, 115.29.



4-aminobenzenesulfonamide *(Sulfanilamide*) **(4o)** 71% yield, White solid. m.p.: 164-166 °C
$^{1}$H NMR (500 MHz, DMSO $d_6$) δ 7.47 – 7.44 (m, 2H), 6.89 (s, 2H), 6.61 – 6.57 (m, 2H), 5.81 (s, 2H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 152.38, 130.50, 127.89, 112.89.

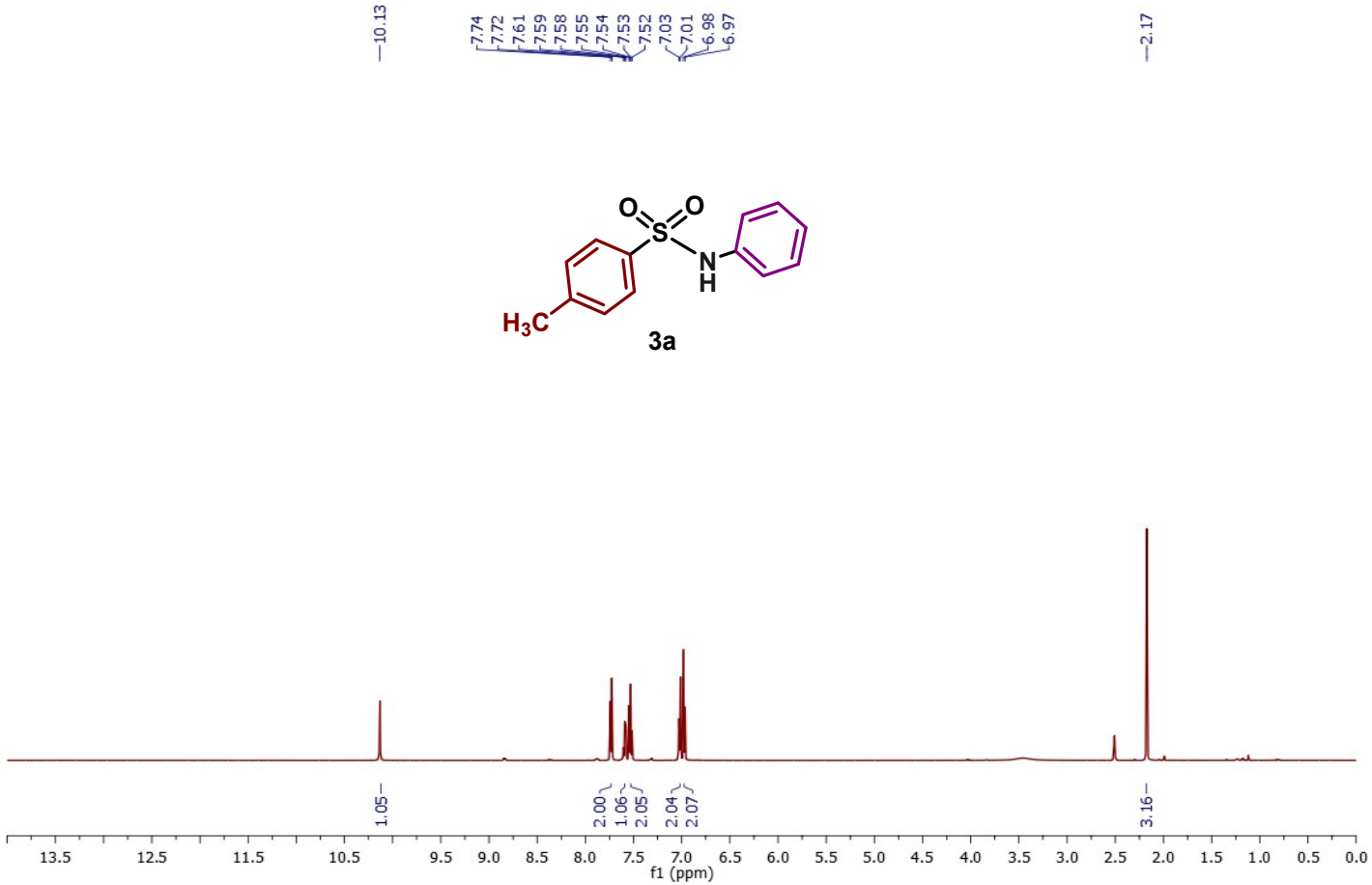


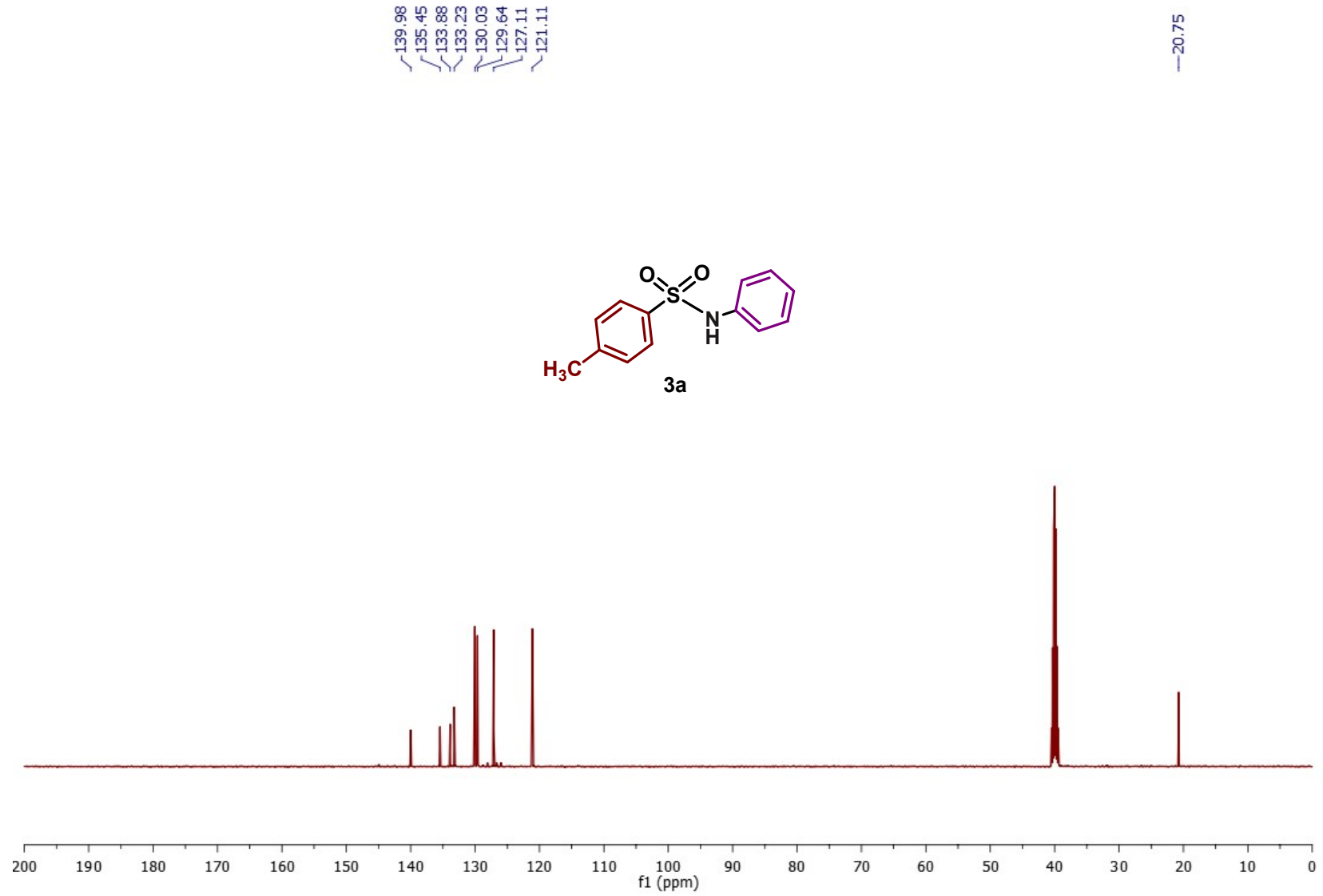
4-amino-N-(6-methoxypyrimidin-4-yl)benzenesulfonamide (*Sulfamonomethoxine*) **(3v)** 81% yield, White solid. m.p.: 206-208 °C, $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 11.32 (s, 1H), 8.39 (d, *J* = 0.9 Hz, 1H), 7.57 – 7.53 (m, 2H), 6.61 – 6.56 (m, 2H), 6.31 (d, *J* = 0.9 Hz, 1H), 6.09 (s, 2H), 3.84 (s, 3H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 170.25, 159.26, 153.81, 129.66, 113.00, 91.07, 54.35.

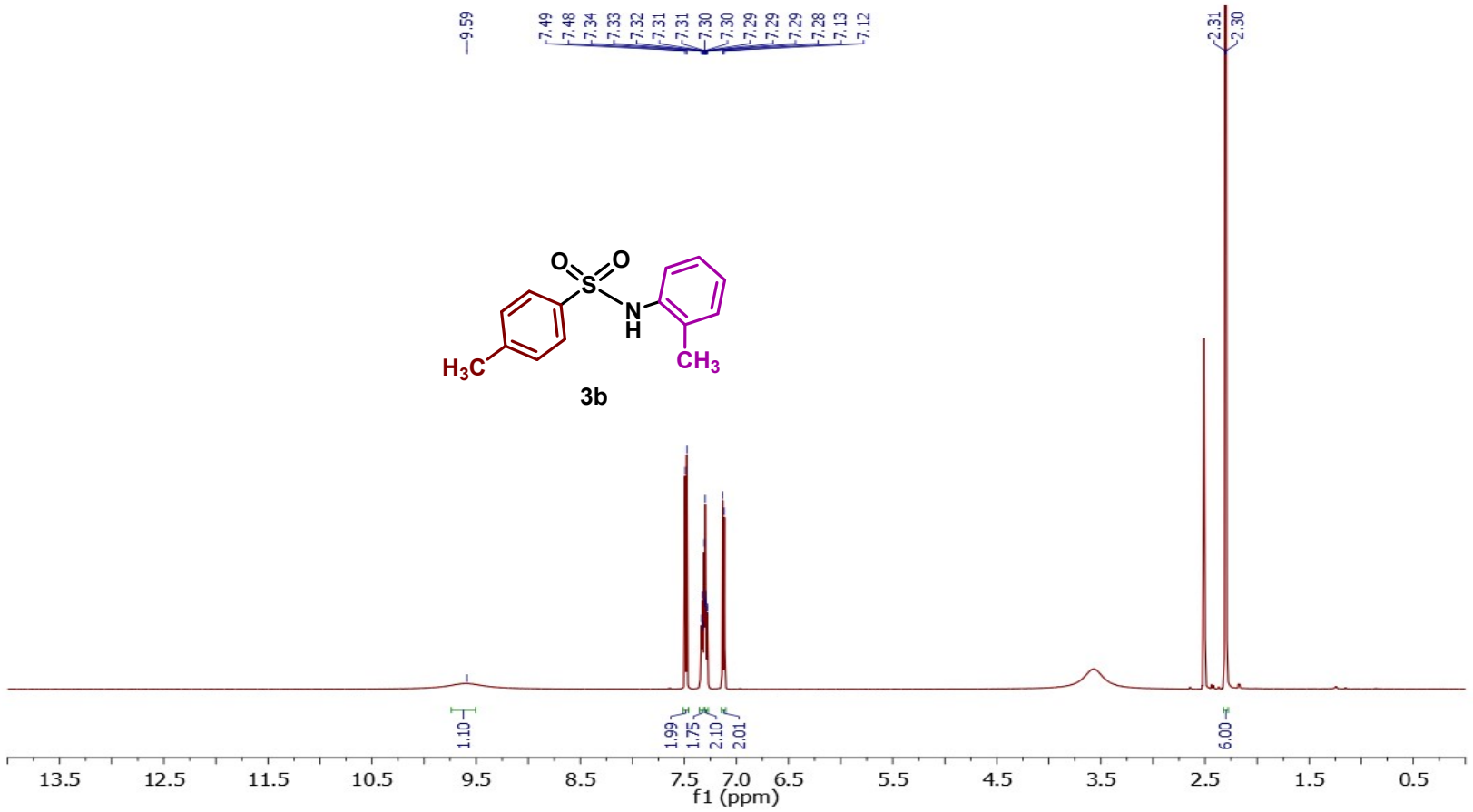


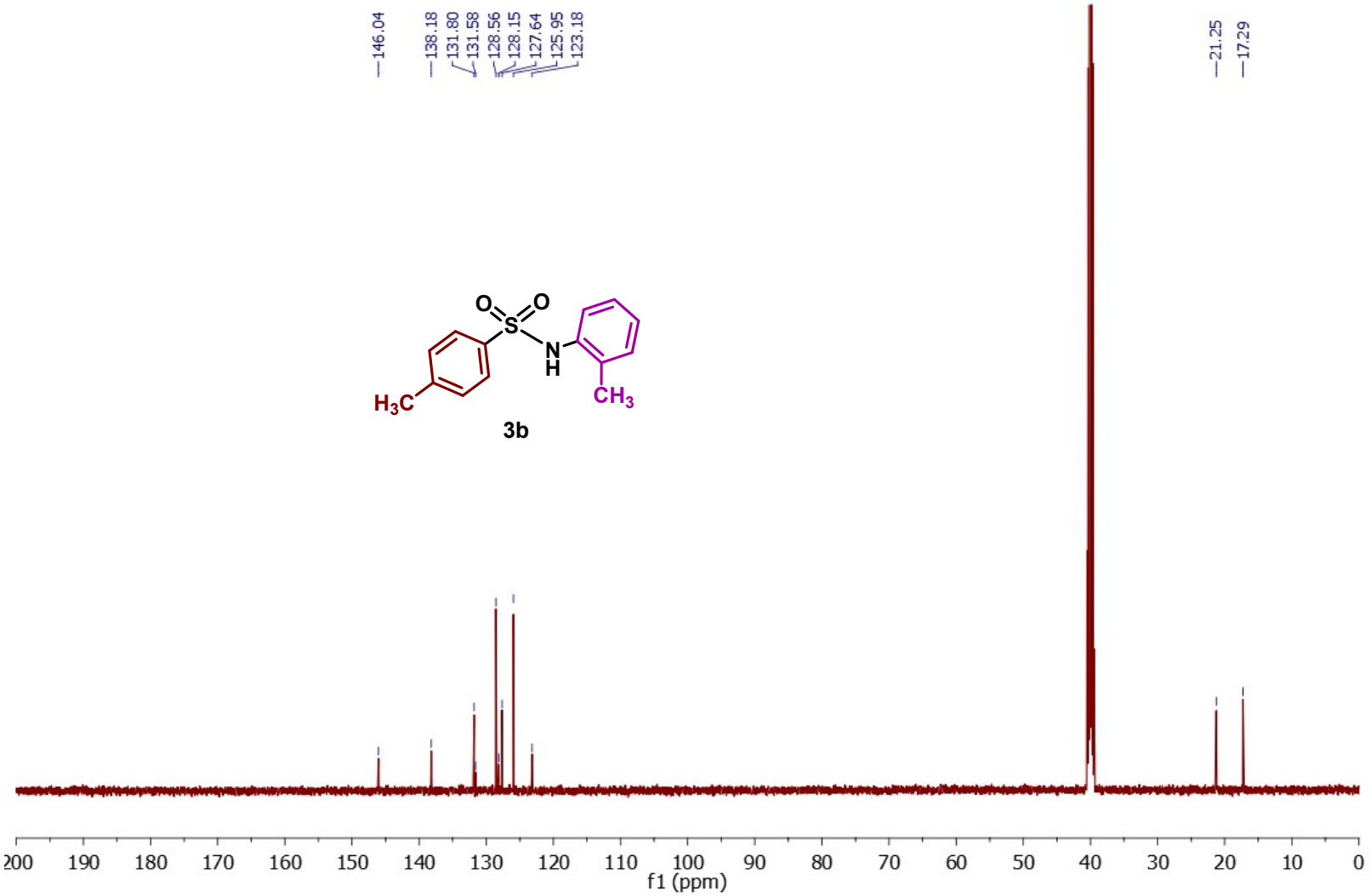

4-amino-N-(pyrimidin-2-yl)benzenesulfonamide (*Sulfadiazine*) **(3w)** 83% yield, White solid. m.p.: 252-256 °C $^{1}$H NMR (500 MHz, DMSO $d_6$) δ 11.25 (s, 1H), 8.48 (d, *J* = 4.8 Hz, 2H), 7.63 – 7.59 (m, 2H), 7.01 (t, *J* = 4.8 Hz, 1H), 6.59 – 6.54 (m, 2H), 6.00 (s, 2H). $^{13}$C NMR (126 MHz, DMSO $d_6$) δ 158.72, 157.72, 153.49, 130.28, 125.37, 119.81, 115.98, 112.59.

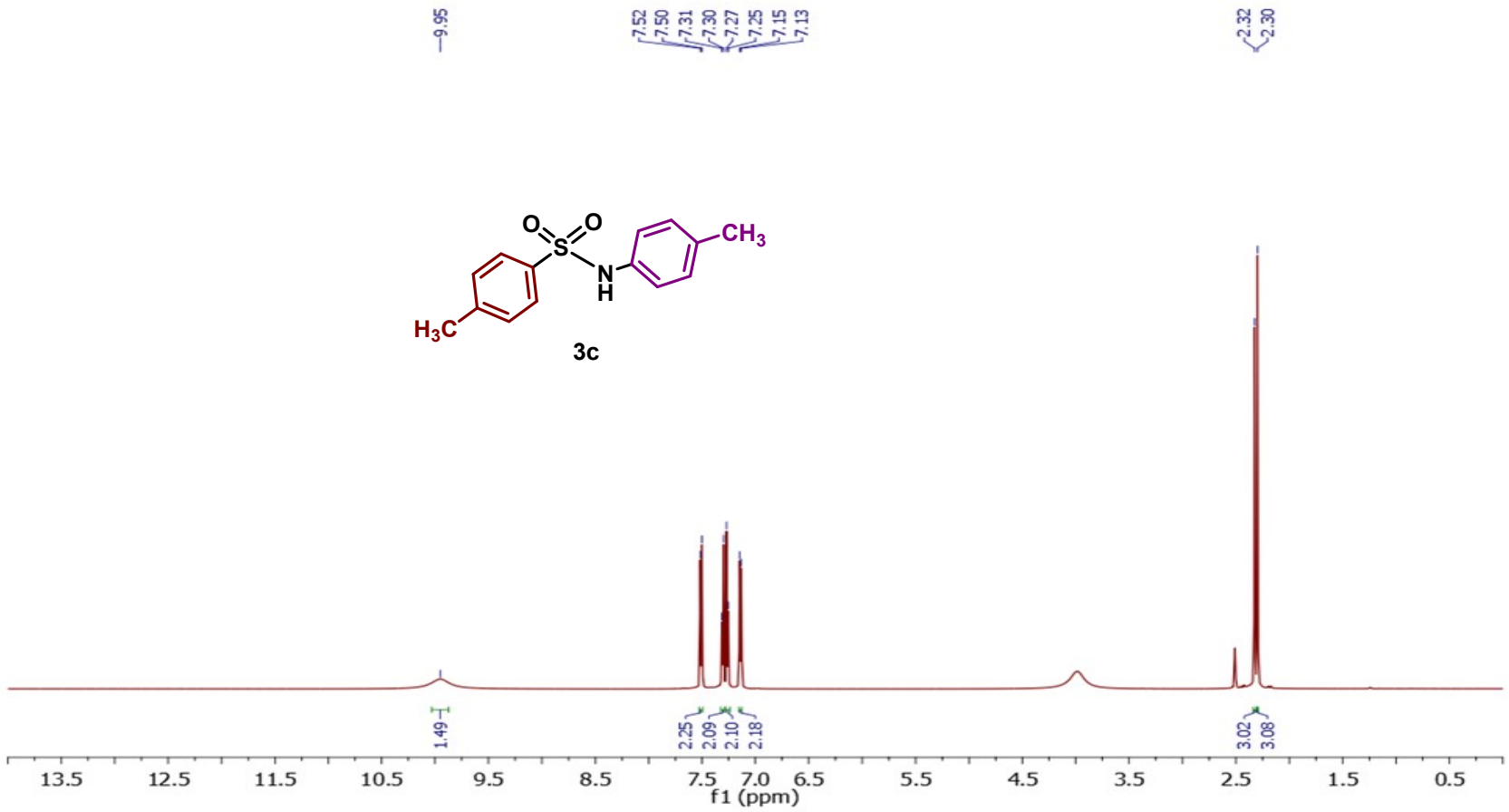
# $^{1}$H NMR and $^{13}$C NMR Spectra

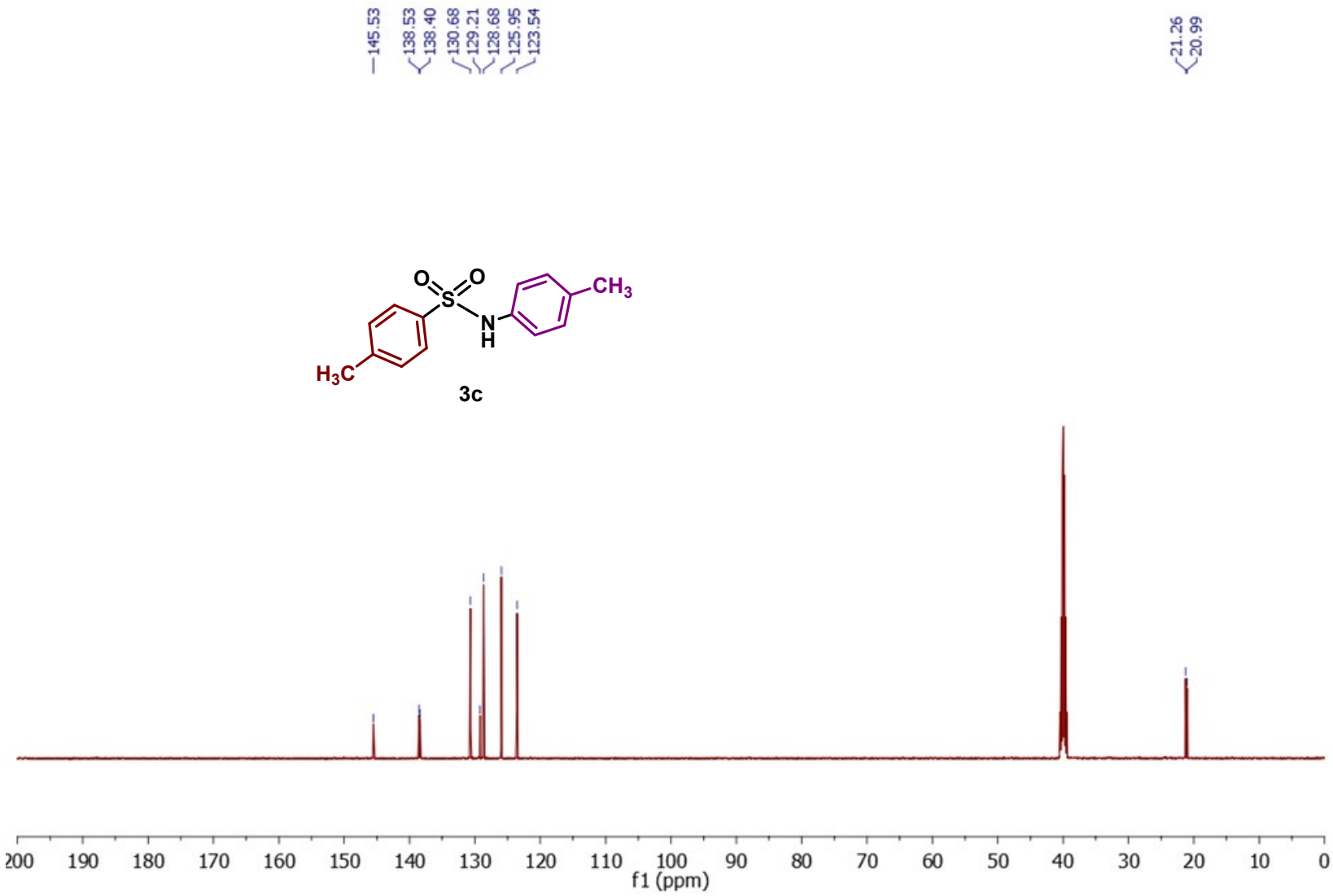


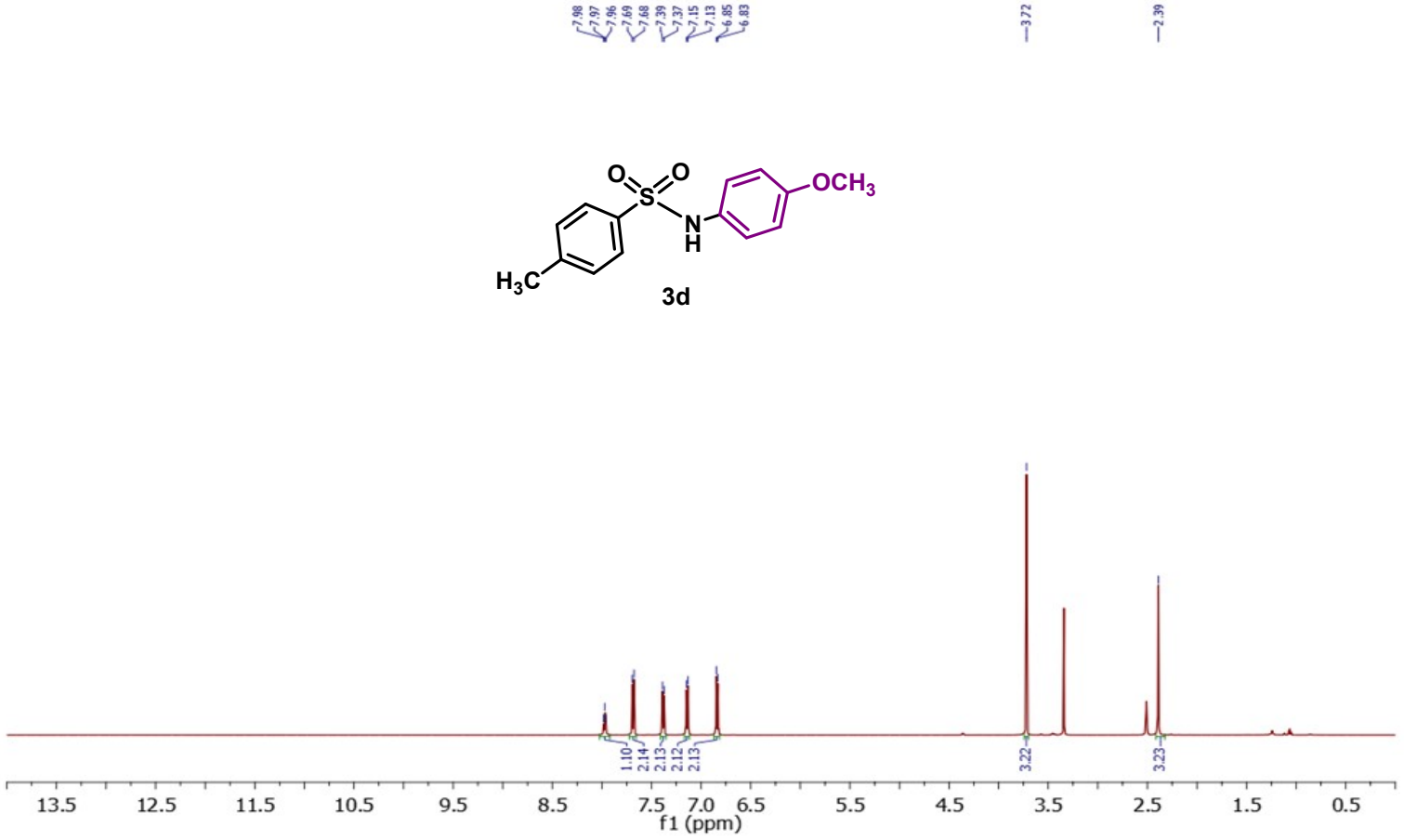

139.98
135.45
133.88
133.23
130.03
129.64
127.11
121.11
20.75
O
O
S
N
H
H3C
3a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


9.59
7.49
7.48
7.34
7.33
7.32
7.31
7.31
7.30
7.30
7.29
7.29
7.29
7.28
7.13
7.12
2.31
2.30
O
O
S
N
H
H3C
CH3
3b
1.10
1.99
1.75
2.10
2.01
6.00
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

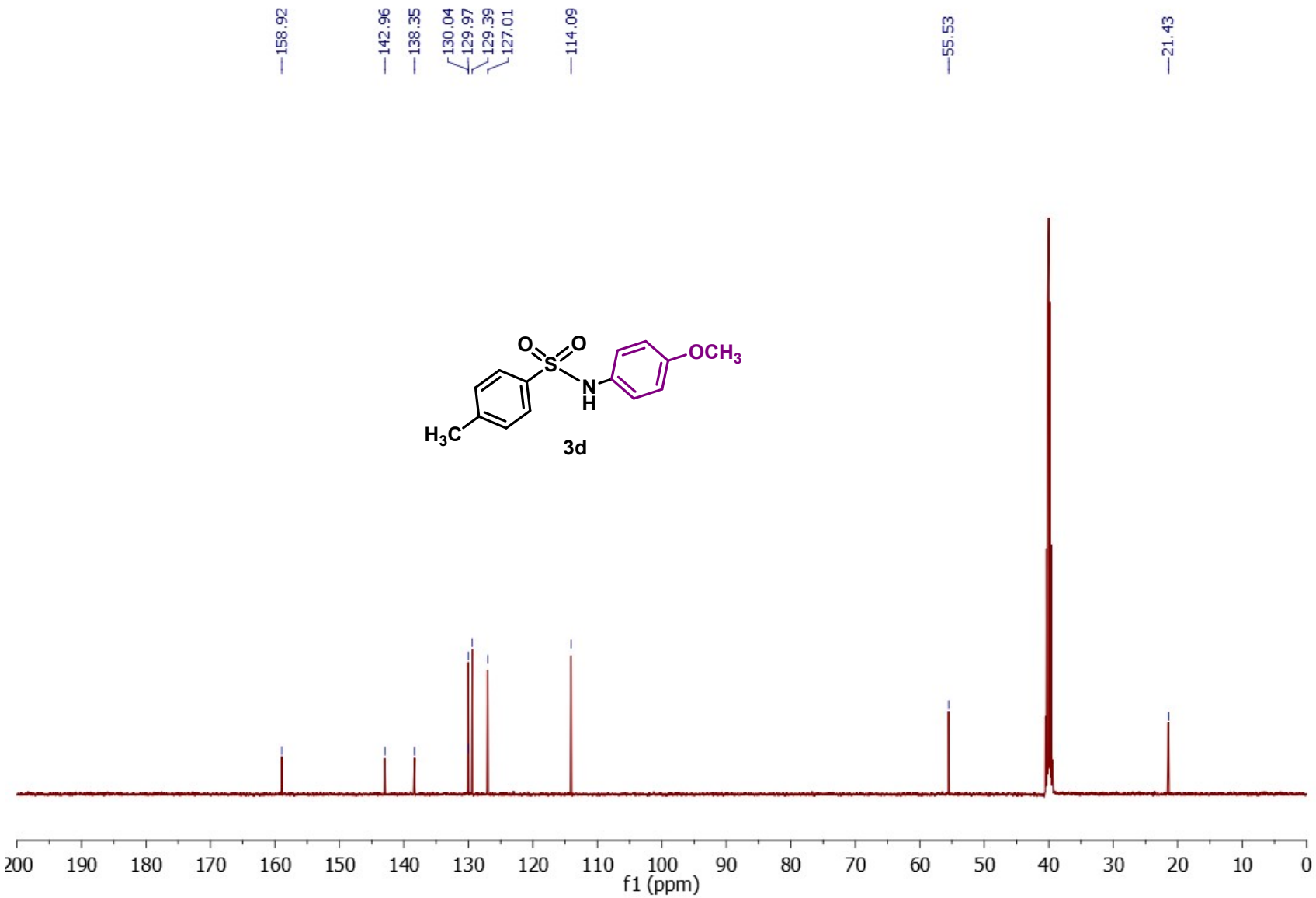

146.04
138.18
131.80
131.58
128.56
128.15
127.64
125.95
123.18
21.25
17.29
O
O
S
N
H
H3C
CH3
3b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

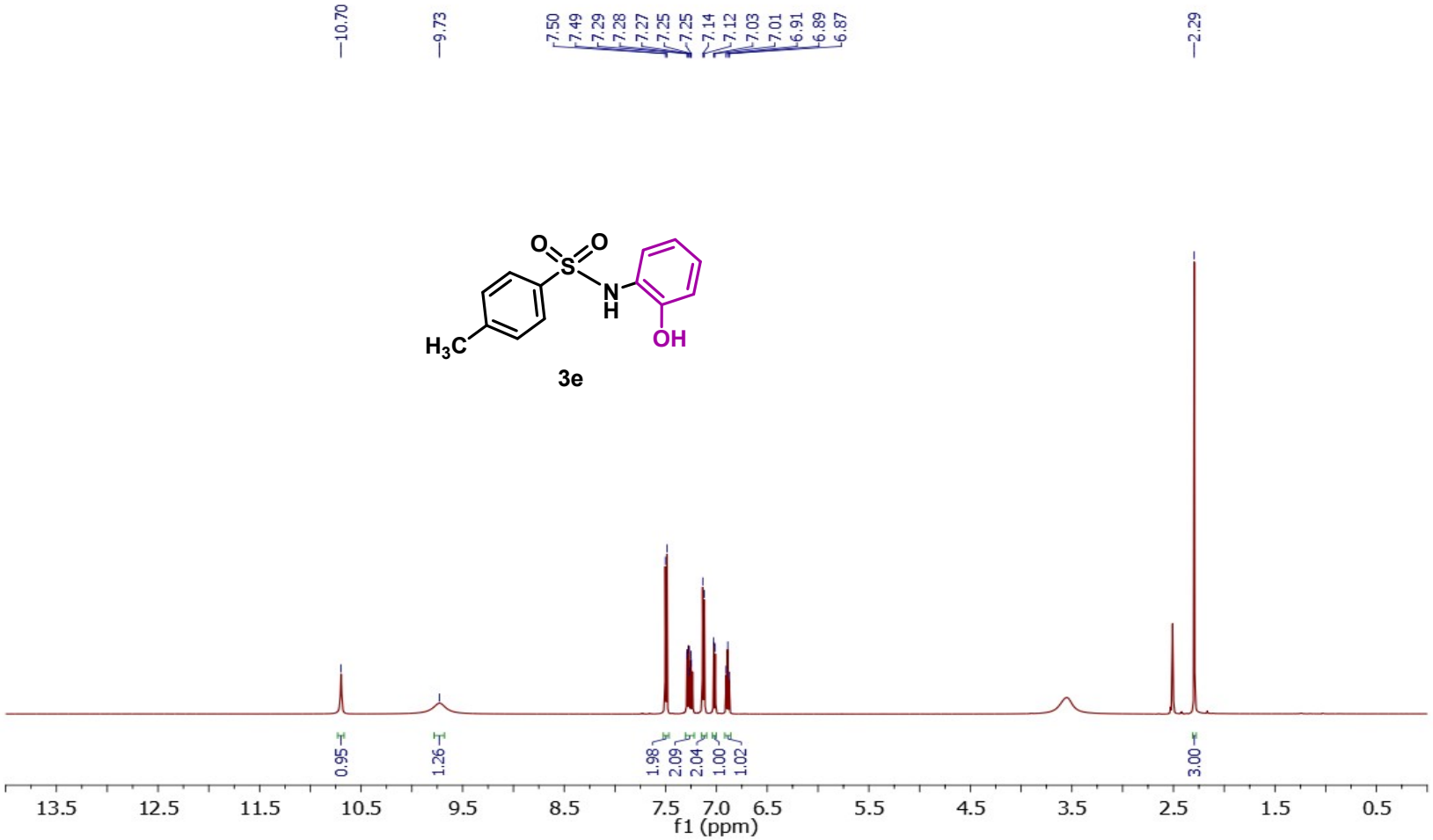

9.95
7.52
7.50
7.31
7.30
7.27
7.25
7.15
7.13
2.32
2.30
O
O
S
N
H
CH3
H3C
3c
1.49
2.25
2.09
2.10
2.18
3.02
3.08
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

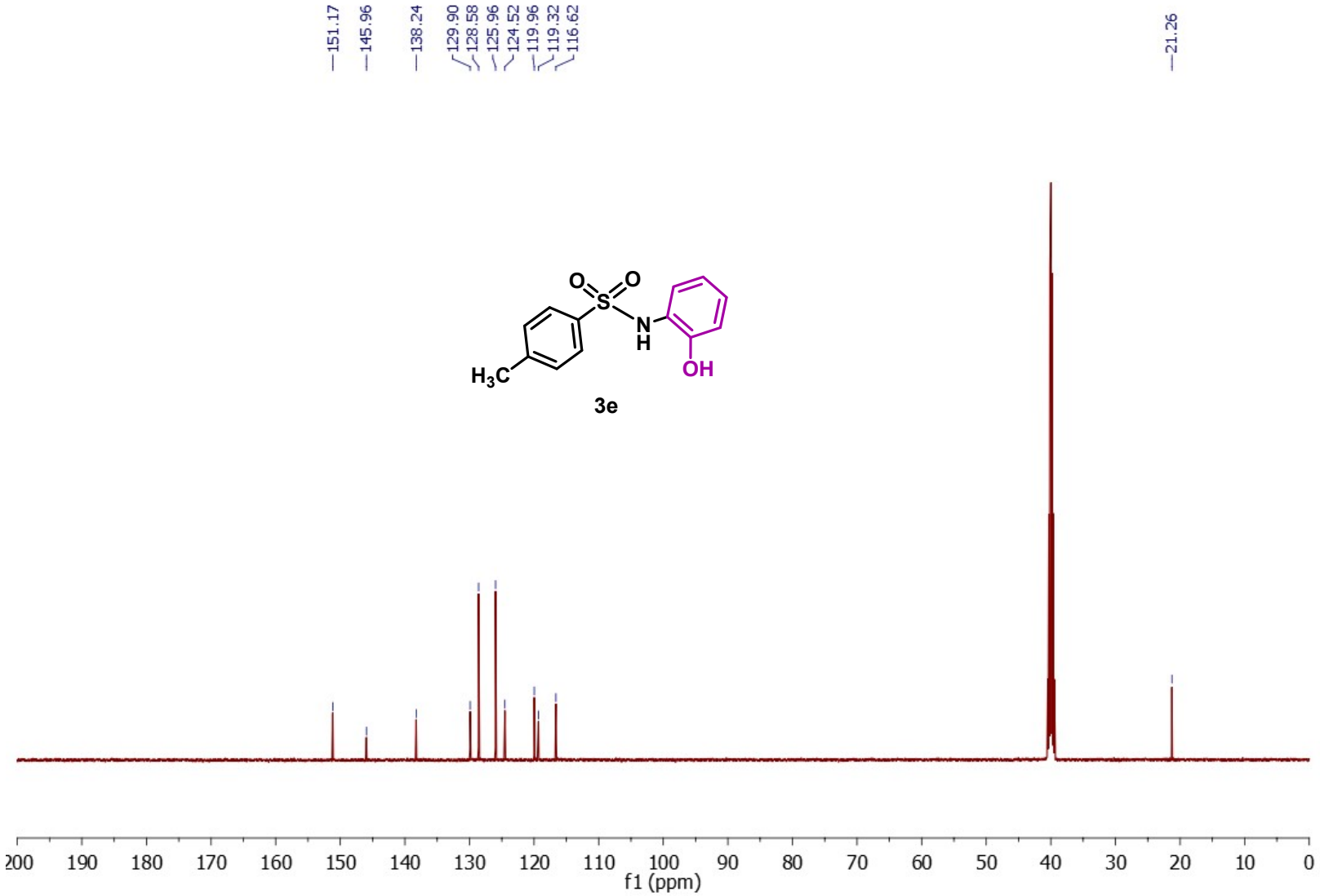

145.53
138.53
138.40
130.68
129.21
128.68
125.95
123.54
21.26
20.99
O
O
S
N
H
CH3
H3C
3c
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

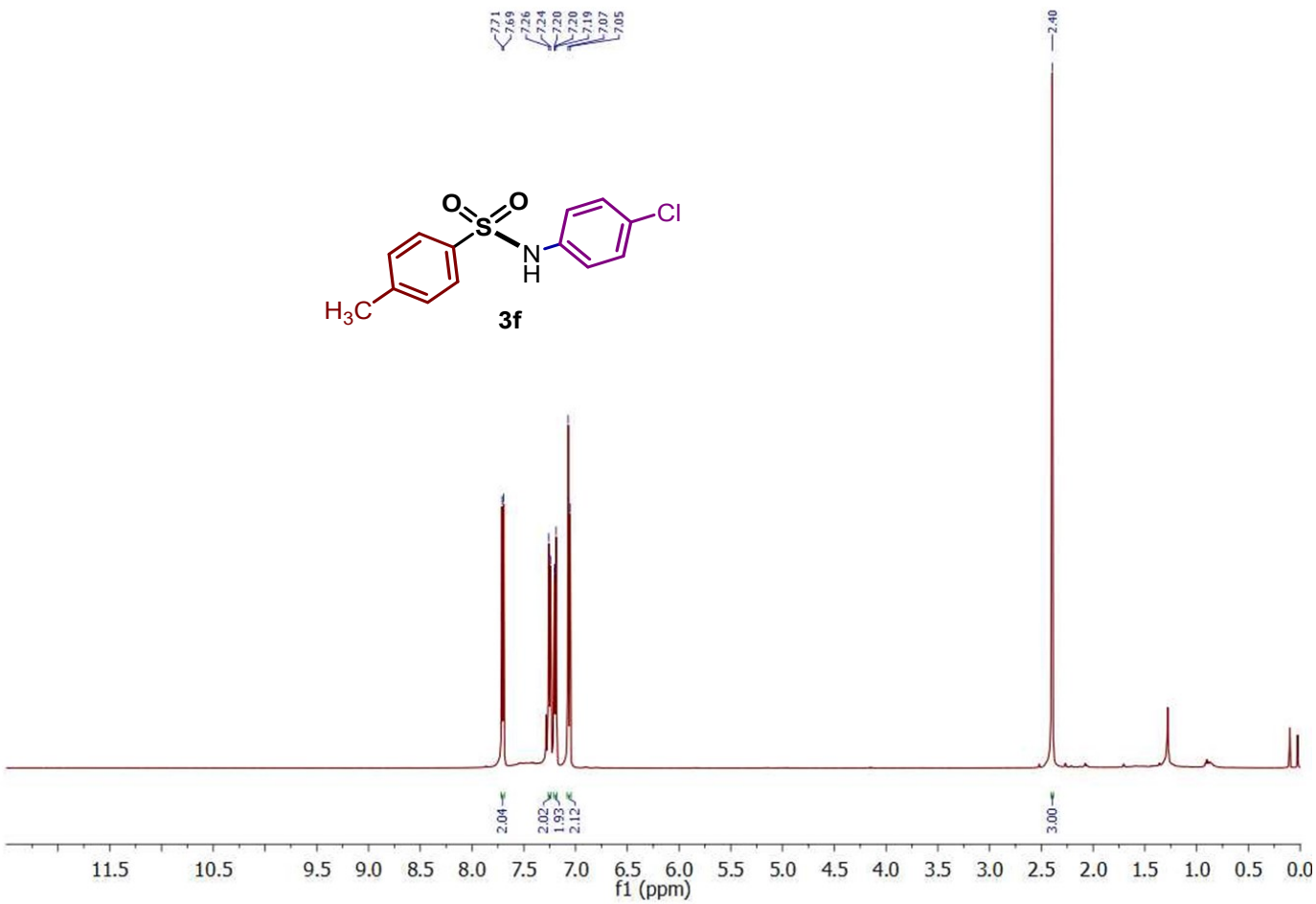

7.98
7.97
7.96
7.69
7.68
7.39
7.37
7.15
7.13
6.85
6.83
3.72
2.39
O
O
S
N
H
OCH3
H3C
1.10
2.14
2.13
2.12
2.13
3.22
3.23
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

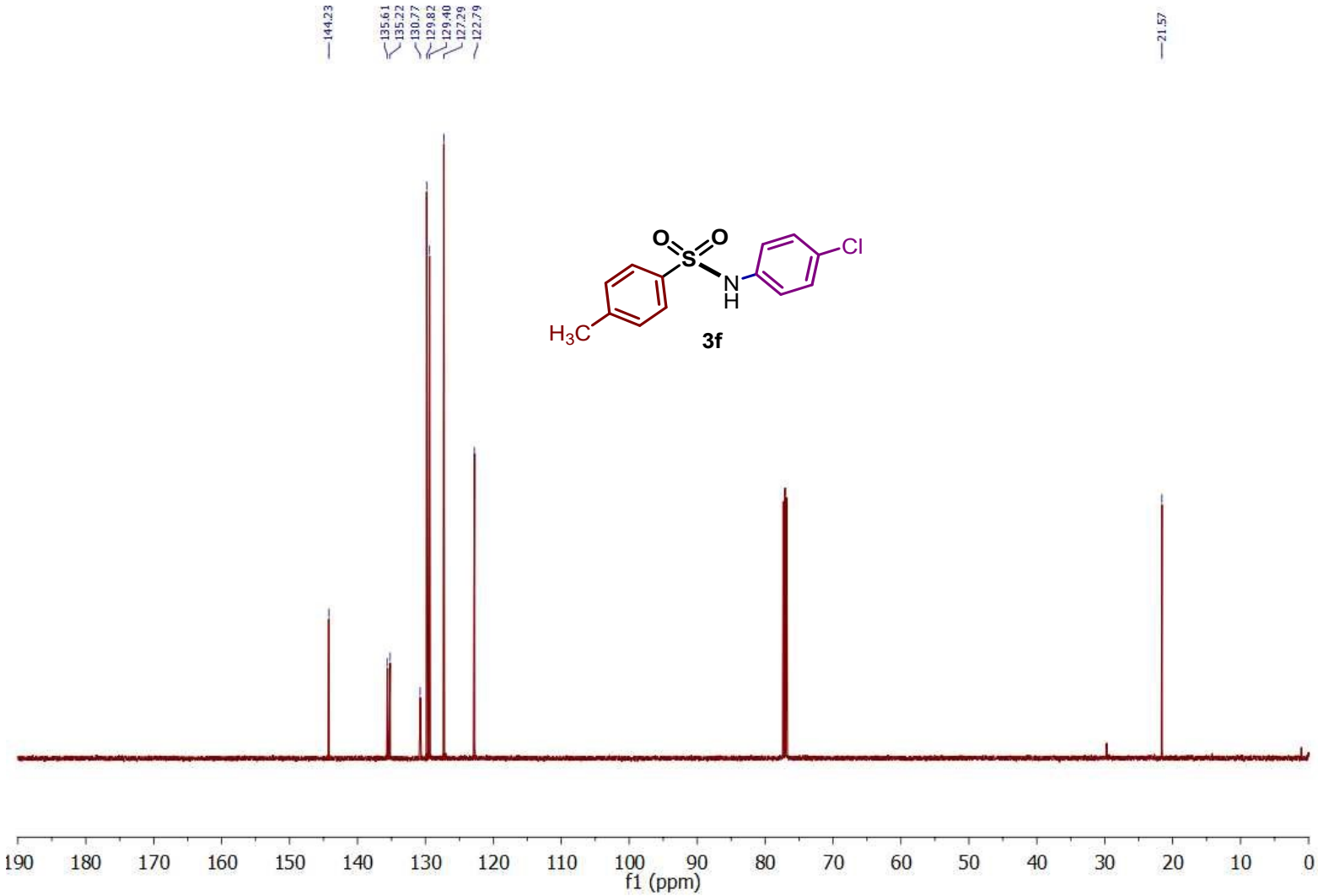
**3d**

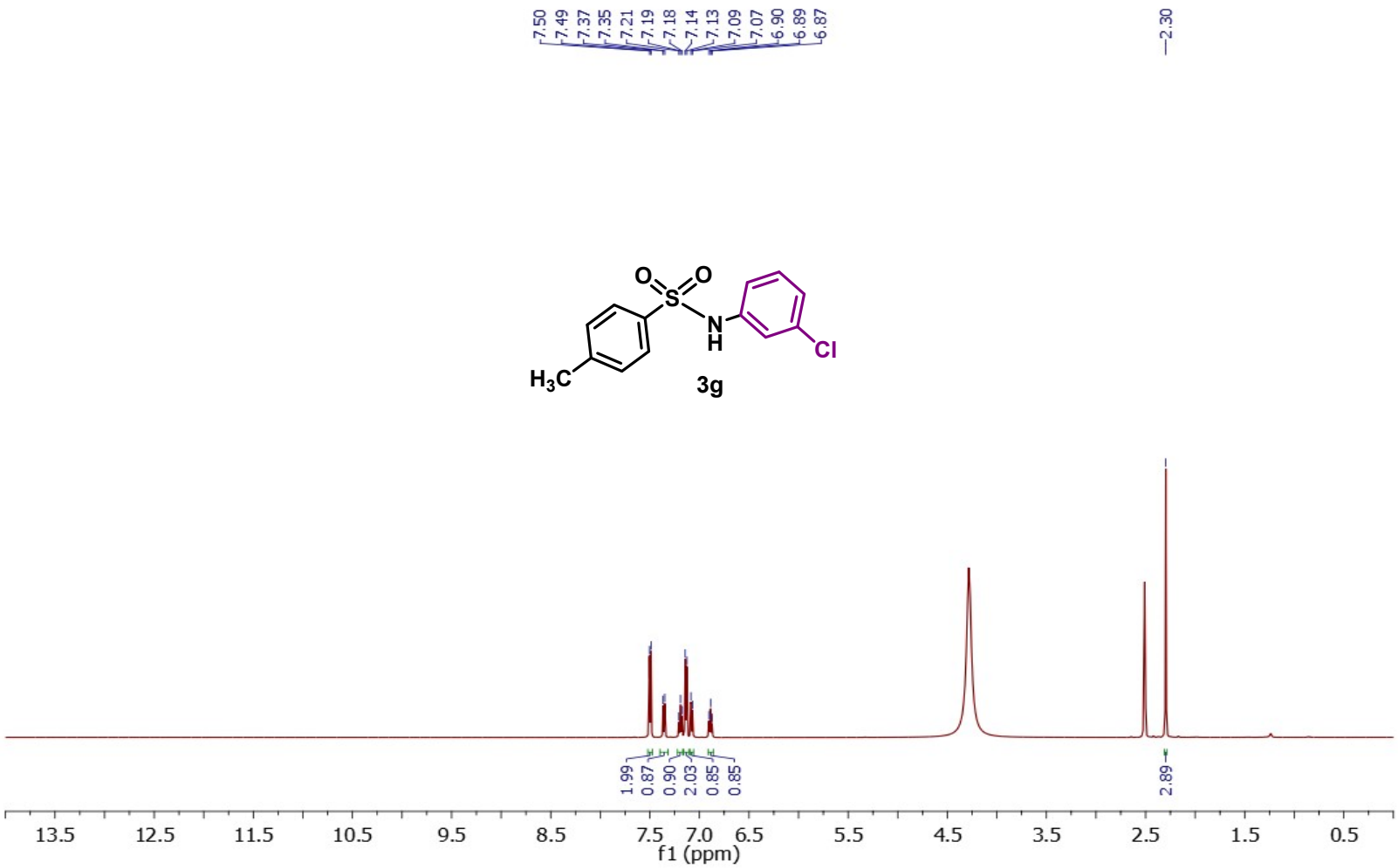

158.92
142.96
138.35
130.04
129.97
129.39
127.01
114.09
55.53
21.43
O
O
S
N
H
OCH3
H3C
3d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

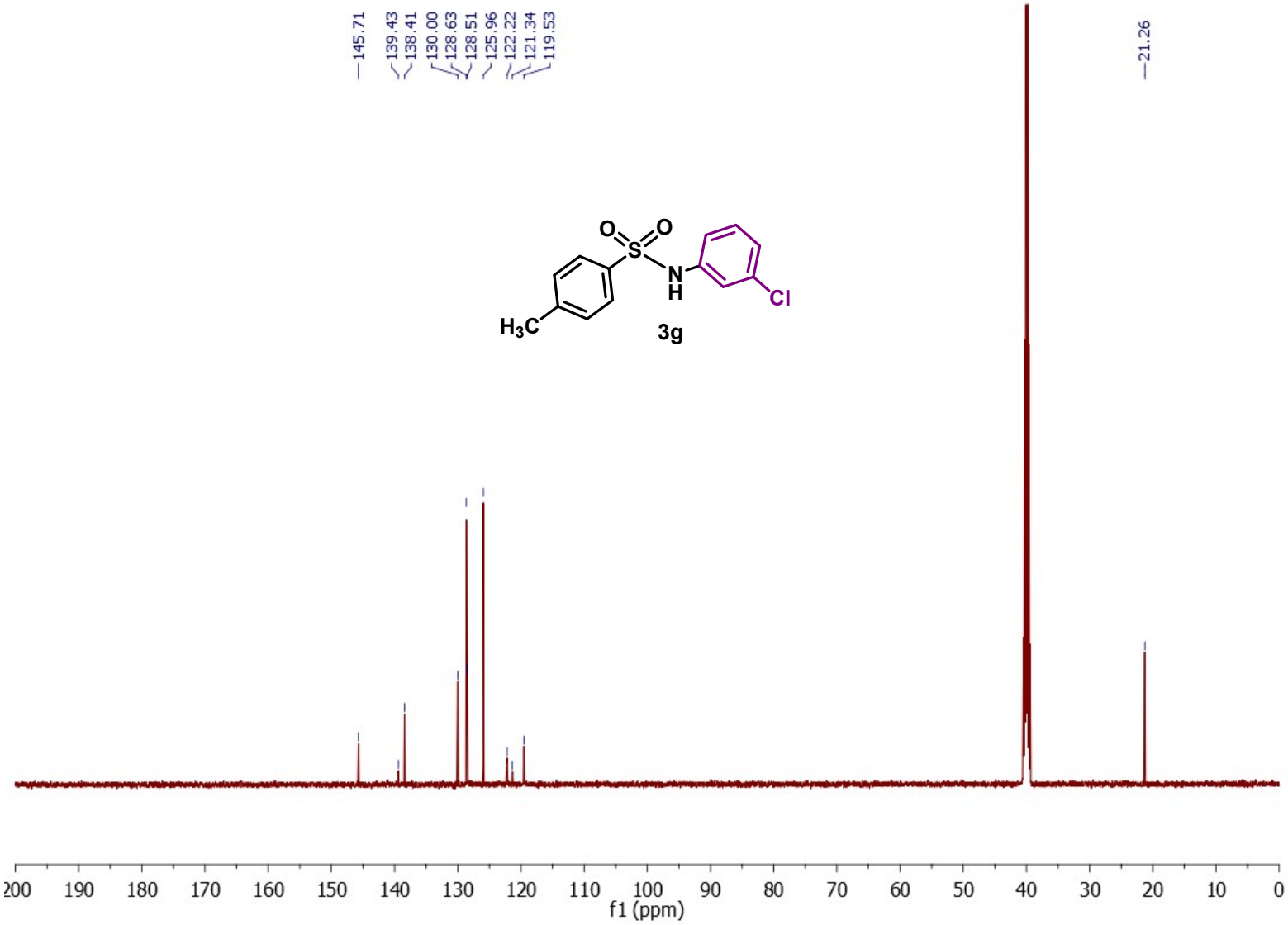

10.70
9.73
7.50
7.49
7.29
7.28
7.27
7.25
7.25
7.14
7.12
7.03
7.01
6.91
6.89
6.87
2.29
O
O
S
N
H
OH
H3C
3e
0.95
1.26
1.98
2.09
2.04
1.00
1.02
3.00
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

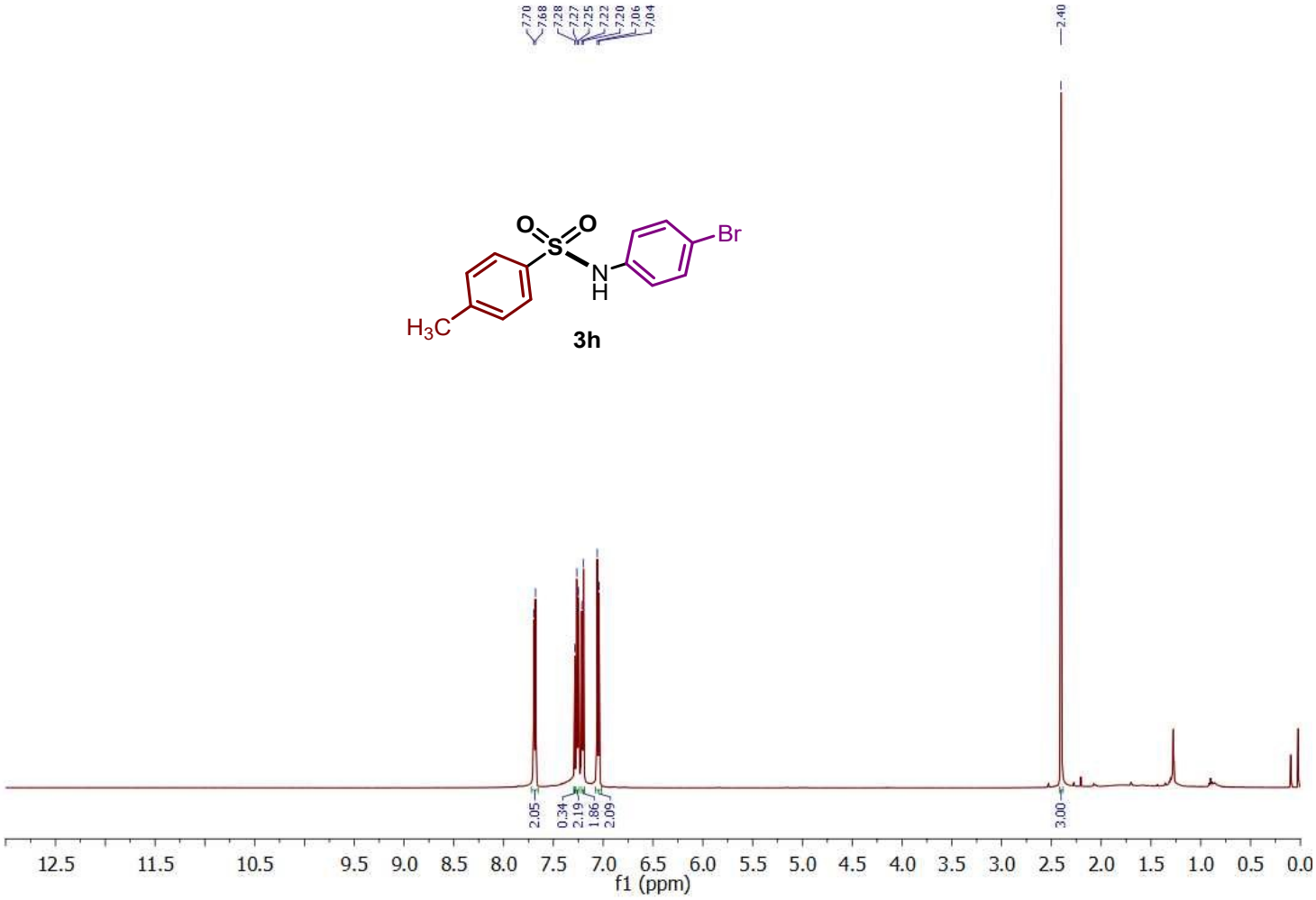

151.17
145.96
138.24
129.90
128.58
125.96
124.52
119.96
119.32
116.62
21.26
O
O
S
N
H
OH
H3C
3e
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

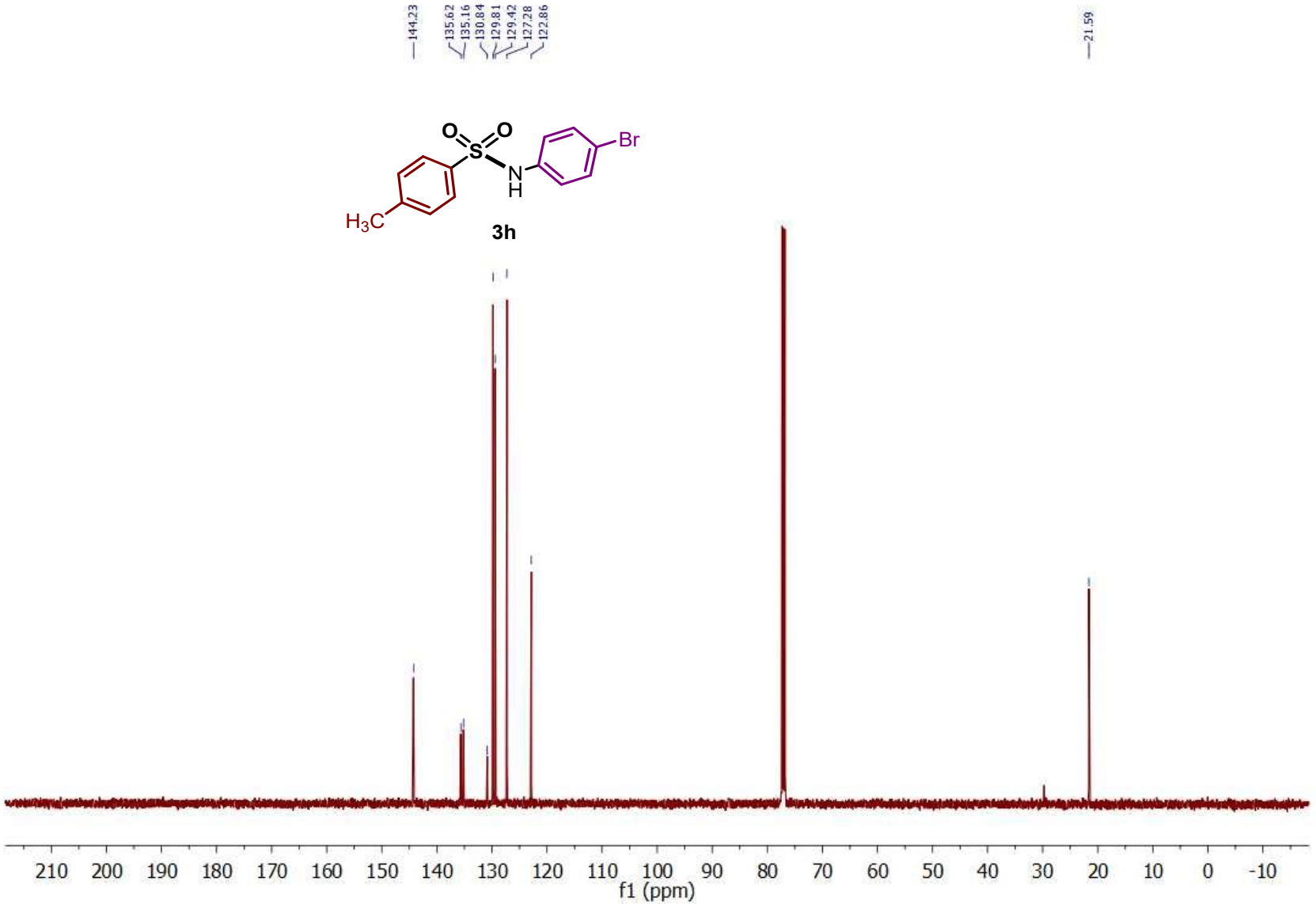

7.71
7.69
7.26
7.24
7.20
7.20
7.19
7.07
7.05
2.40
O
O
S
N
H
Cl
H3C
3f
2.04
2.02
1.93
2.12
3.00
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

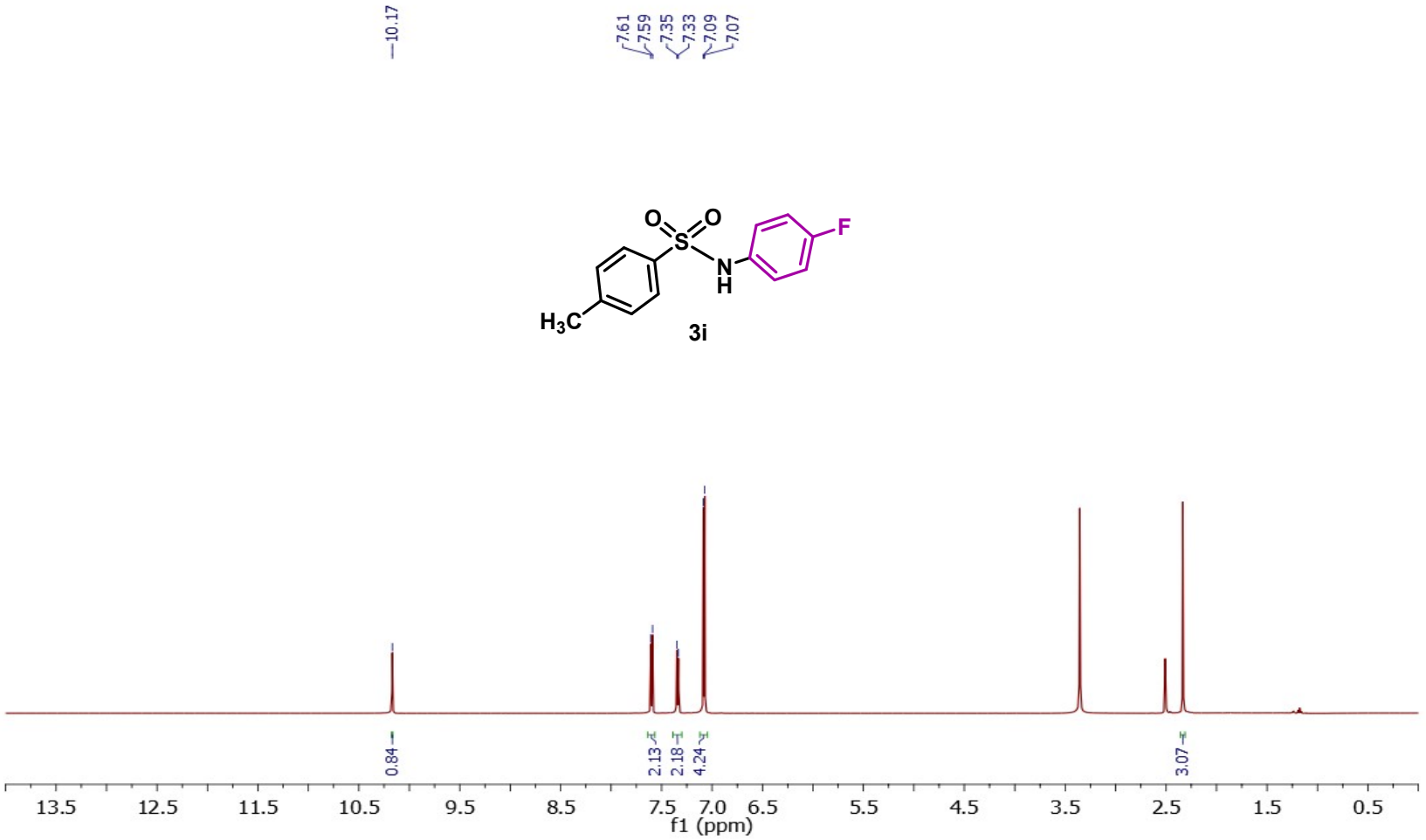

144.23
135.61
135.22
130.77
129.82
129.40
127.29
122.79
21.57
O
O
S
N
H
Cl
H3C
3f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

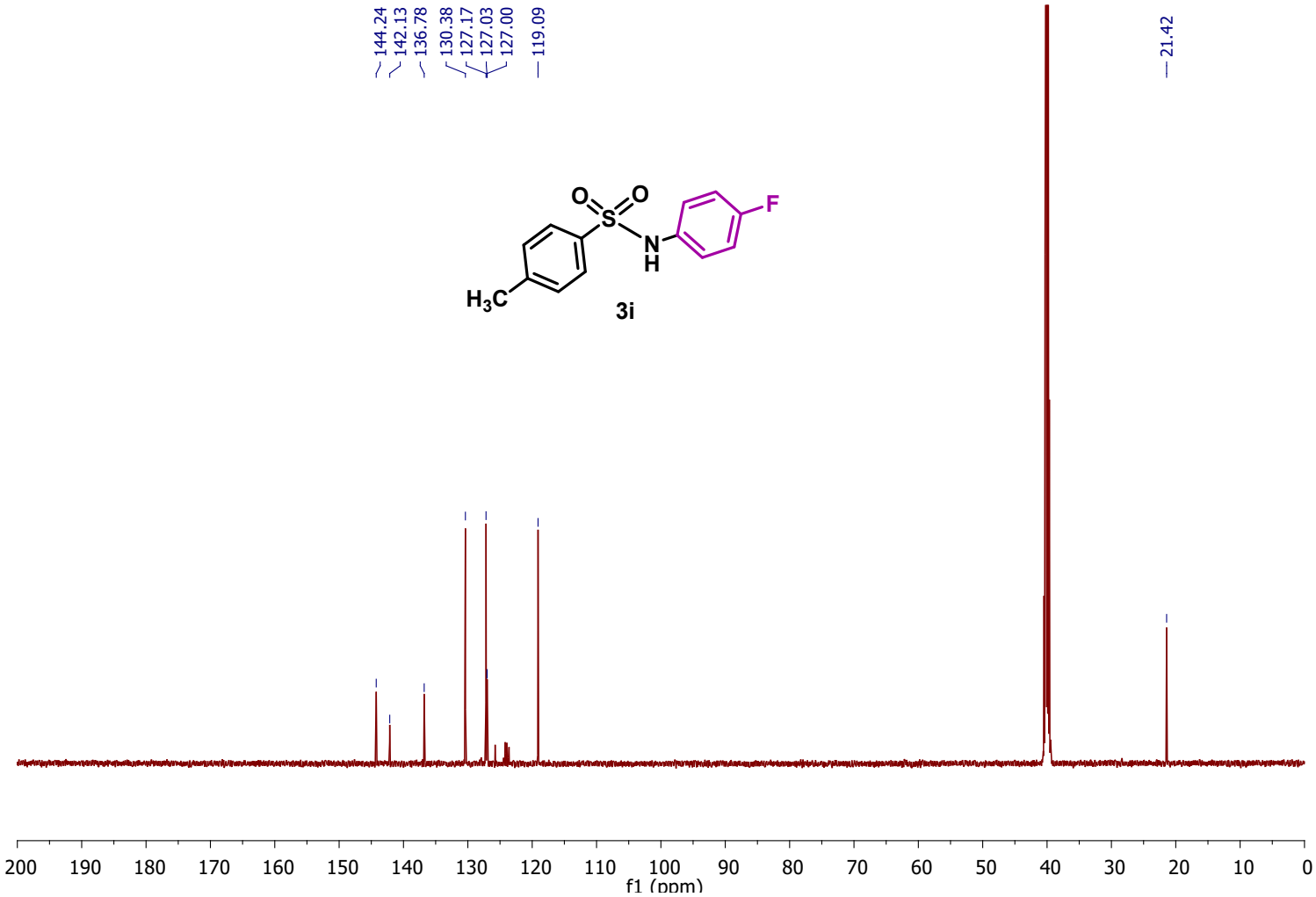

7.50
7.49
7.37
7.35
7.21
7.19
7.18
7.14
7.13
7.09
7.07
6.90
6.89
6.87
2.30
O
O
S
N
H
Cl
H3C
3g
1.99
0.87
0.90
2.03
0.85
0.85
2.89
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

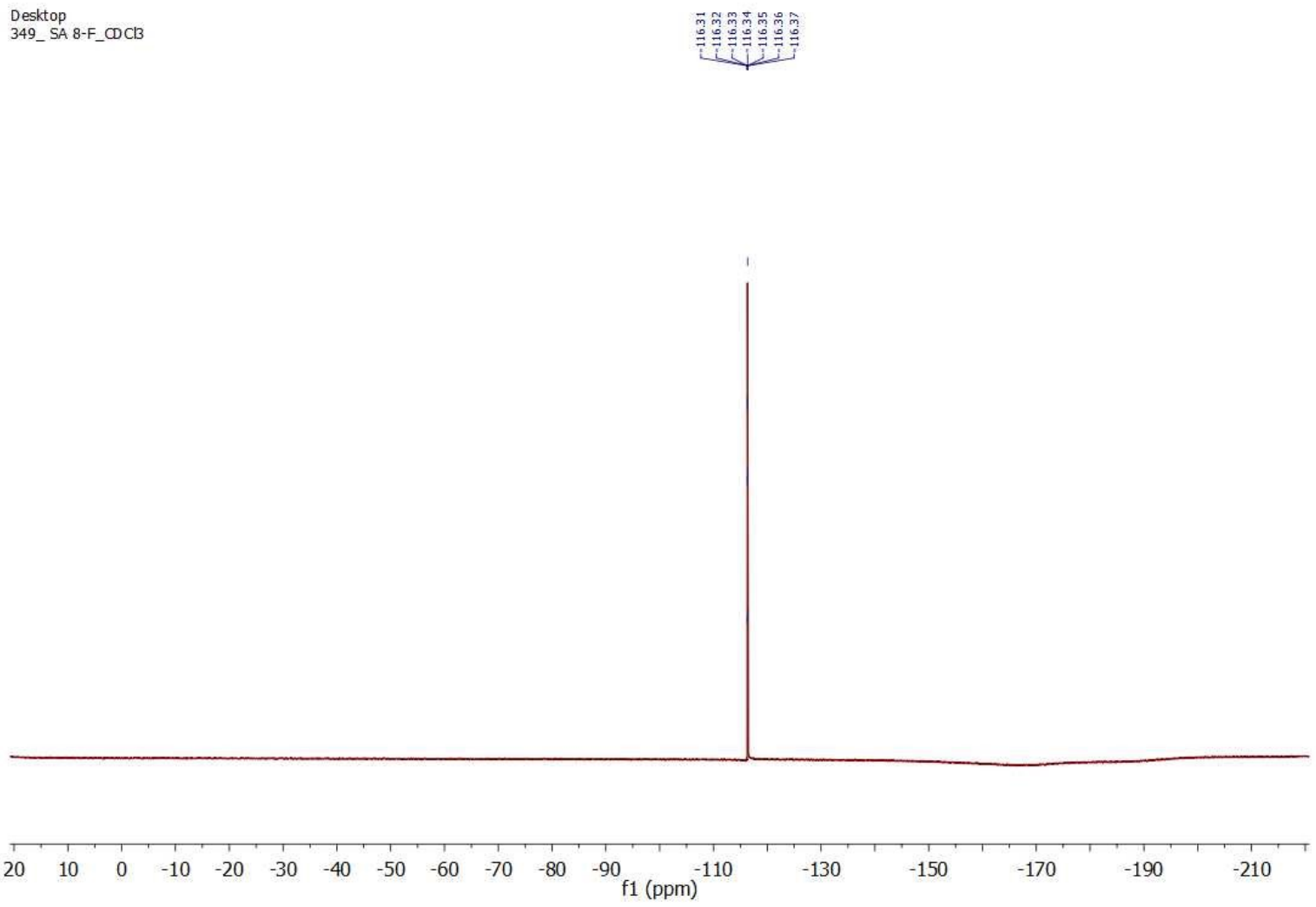

145.71
139.43
138.41
130.00
128.63
128.51
125.96
122.22
121.34
119.53
21.26
O
O
S
N
H
Cl
H3C
3g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

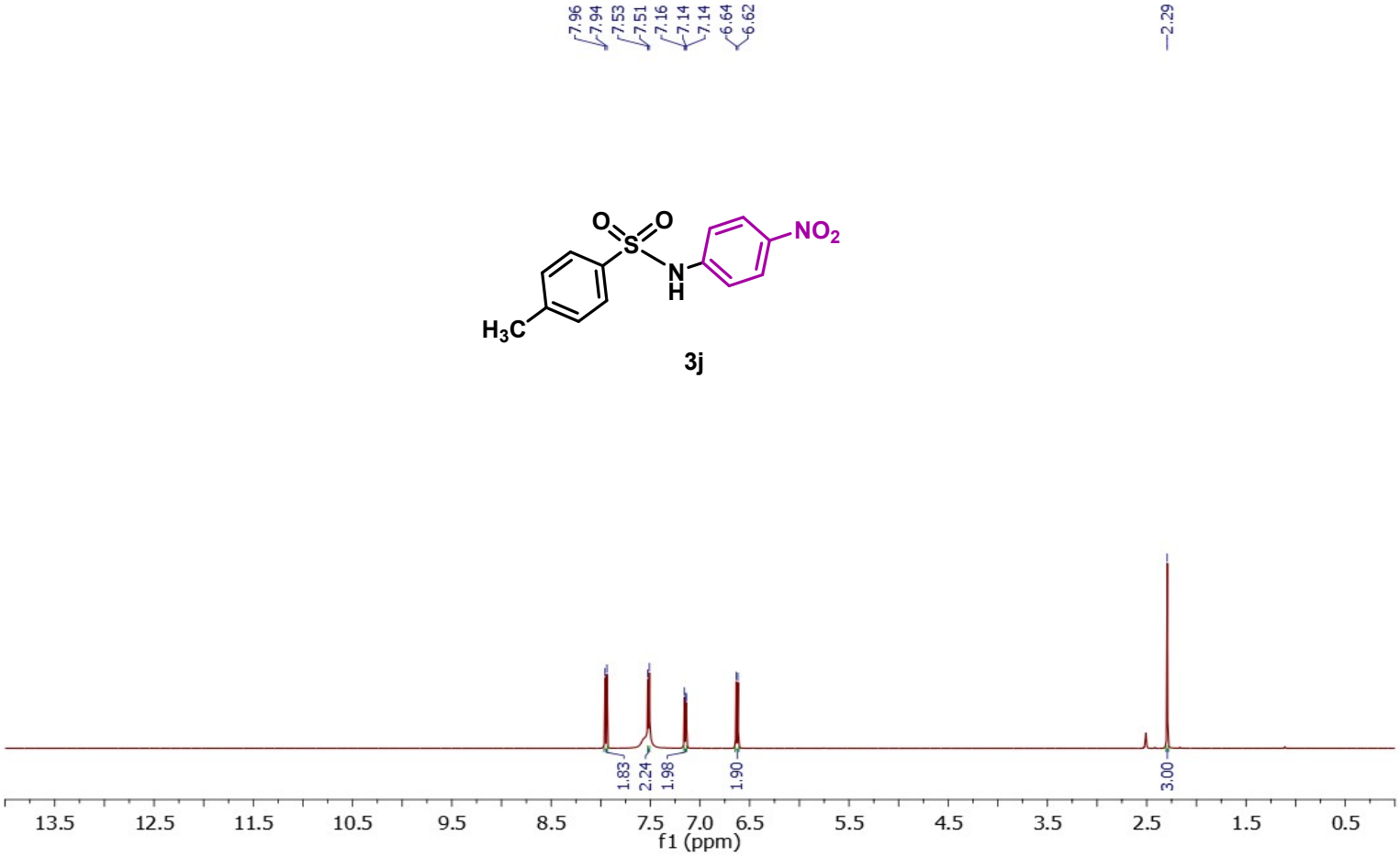

7.70
7.68
7.28
7.27
7.25
7.22
7.20
7.06
7.04
2.40
O
O
S
N
H
Br
H3C
3h
2.05
0.34
2.19
1.86
2.09
3.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


144.23
135.62
135.16
130.84
129.81
129.42
127.28
122.86
21.59
O
O
S
N
H
Br
H3C
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

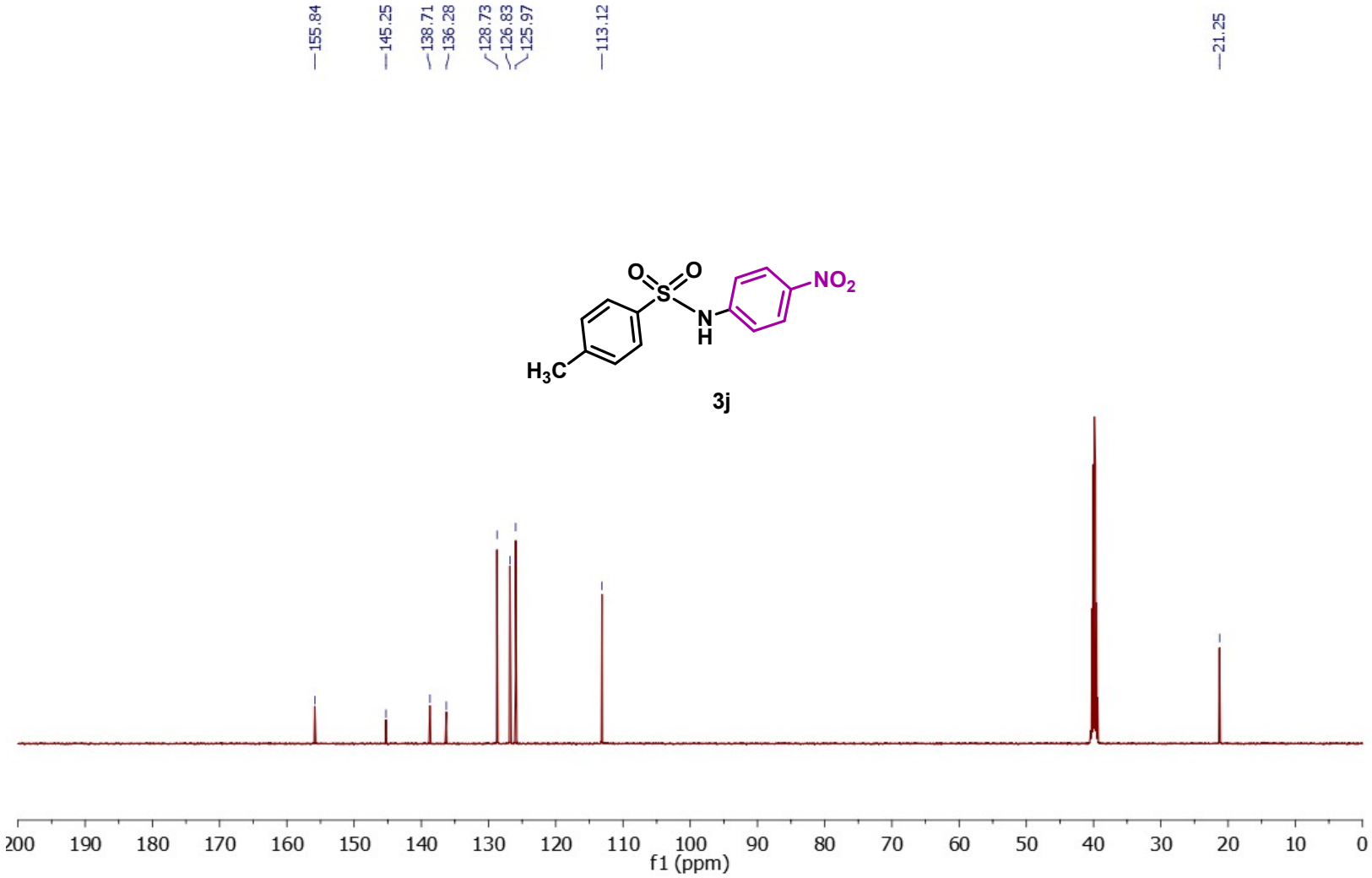

10.17
7.61
7.59
7.35
7.33
7.09
7.07
O
O
S
N
H
F
H3C
3i
0.84
2.13
2.18
4.24
3.07
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

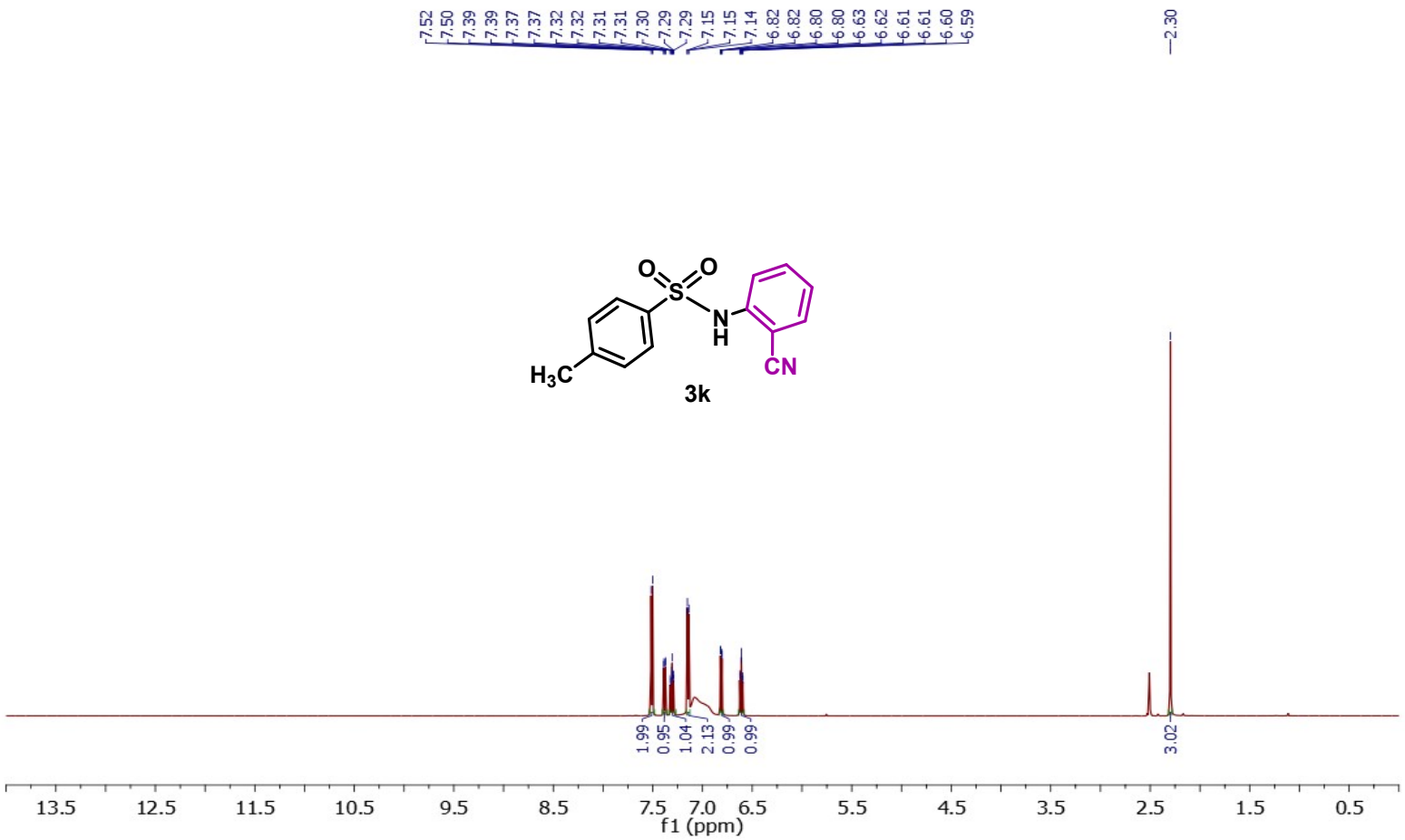

144.24
142.13
136.78
130.38
127.17
127.03
127.00
119.09
21.42
O
O
S
N
H
F
H3C
3i
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

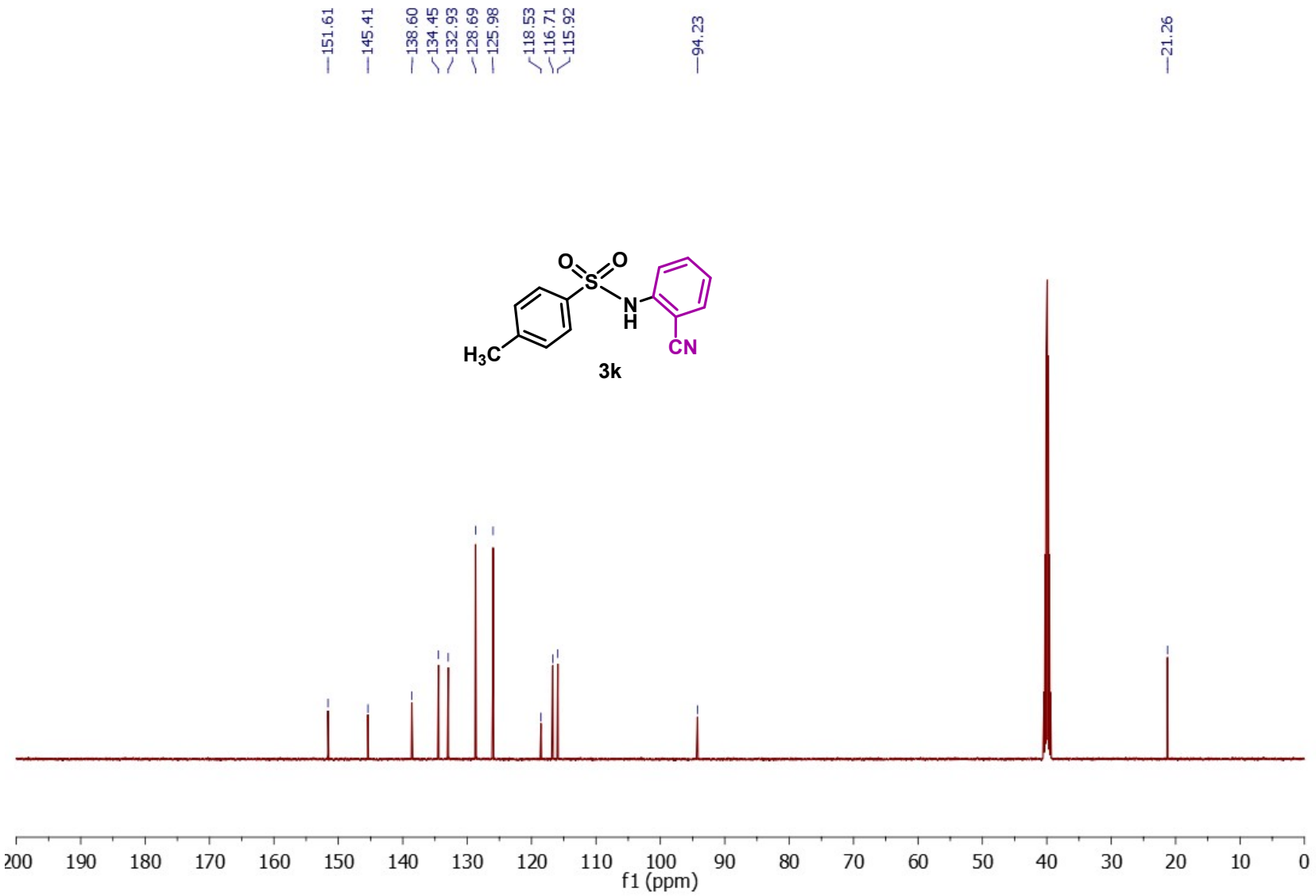

Desktop
349_ SA 8-F_CDCl3
116.31
116.32
116.33
116.34
116.35
116.36
116.37
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

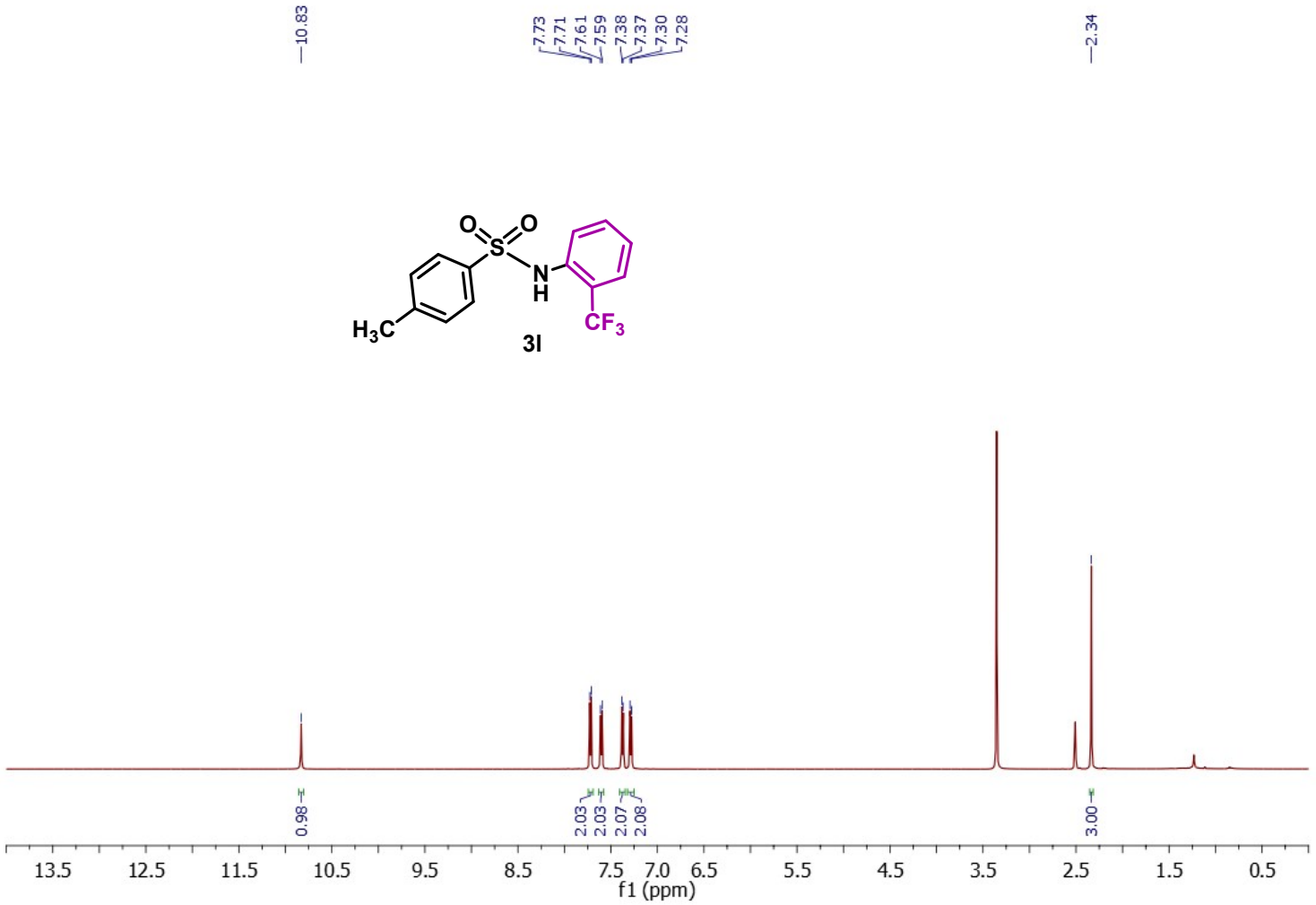

7.96
7.94
7.53
7.51
7.16
7.14
7.14
6.64
6.62
2.29
O
O
S
N
H
NO2
H3C
1.83
2.24
1.98
1.90
3.00
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

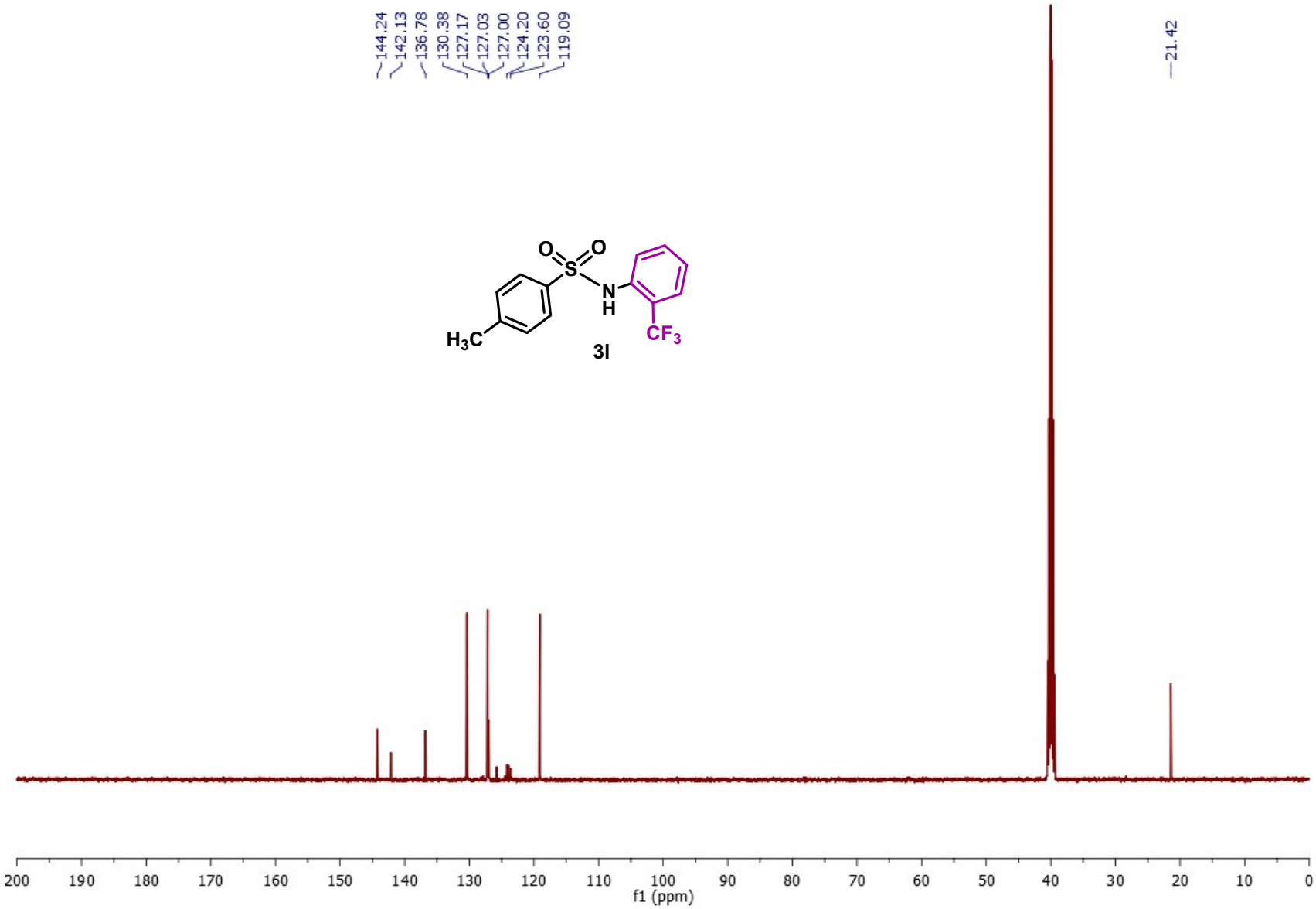
**3j**

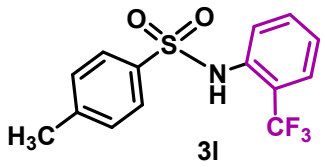

155.84
145.25
138.71
136.28
128.73
126.83
125.97
113.12
21.25
O
O
S
N
H
NO2
H3C
3j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.52
7.50
7.39
7.39
7.37
7.37
7.32
7.32
7.31
7.31
7.30
7.29
7.29
7.15
7.15
7.14
6.82
6.82
6.80
6.80
6.63
6.62
6.61
6.61
6.60
6.59
2.30
O
O
S
N
H
CN
H3C
3k
1.99
0.95
1.04
2.13
0.99
0.99
3.02
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

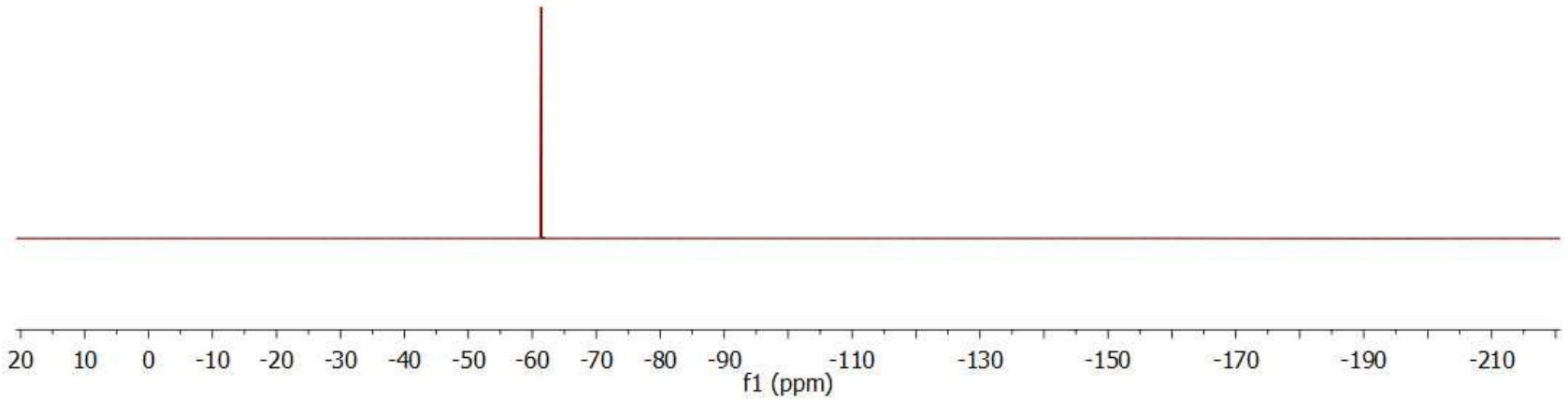

151.61
145.41
138.60
134.45
132.93
128.69
125.98
118.53
116.71
115.92
94.23
21.26
O
S
O
N
H
CN
H3C
3k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

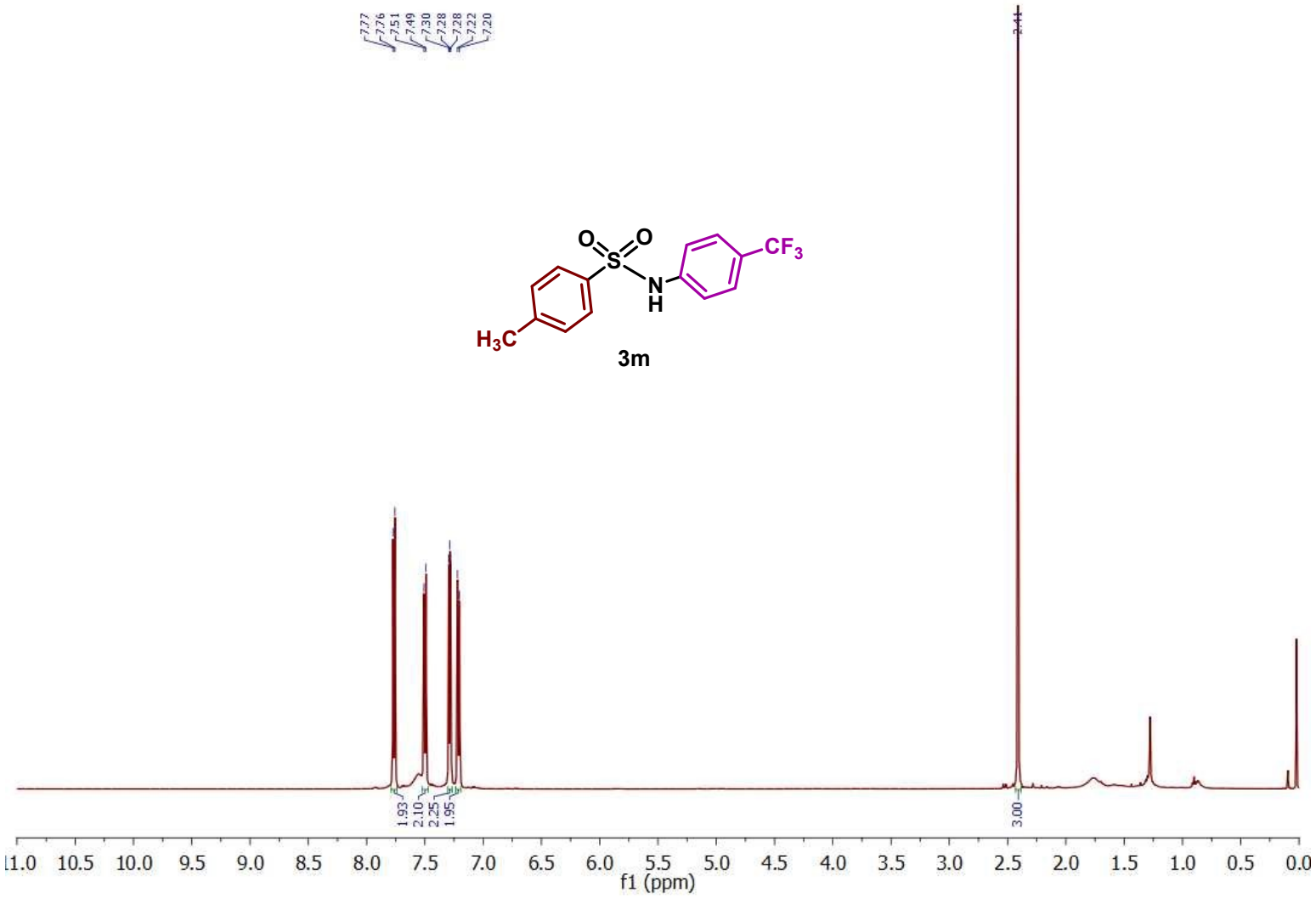

10.83
7.73
7.71
7.61
7.59
7.38
7.37
7.30
7.28
2.34
O
O
S
N
H
H3C
CF3
3l
0.98
2.03
2.03
2.07
2.08
3.00
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

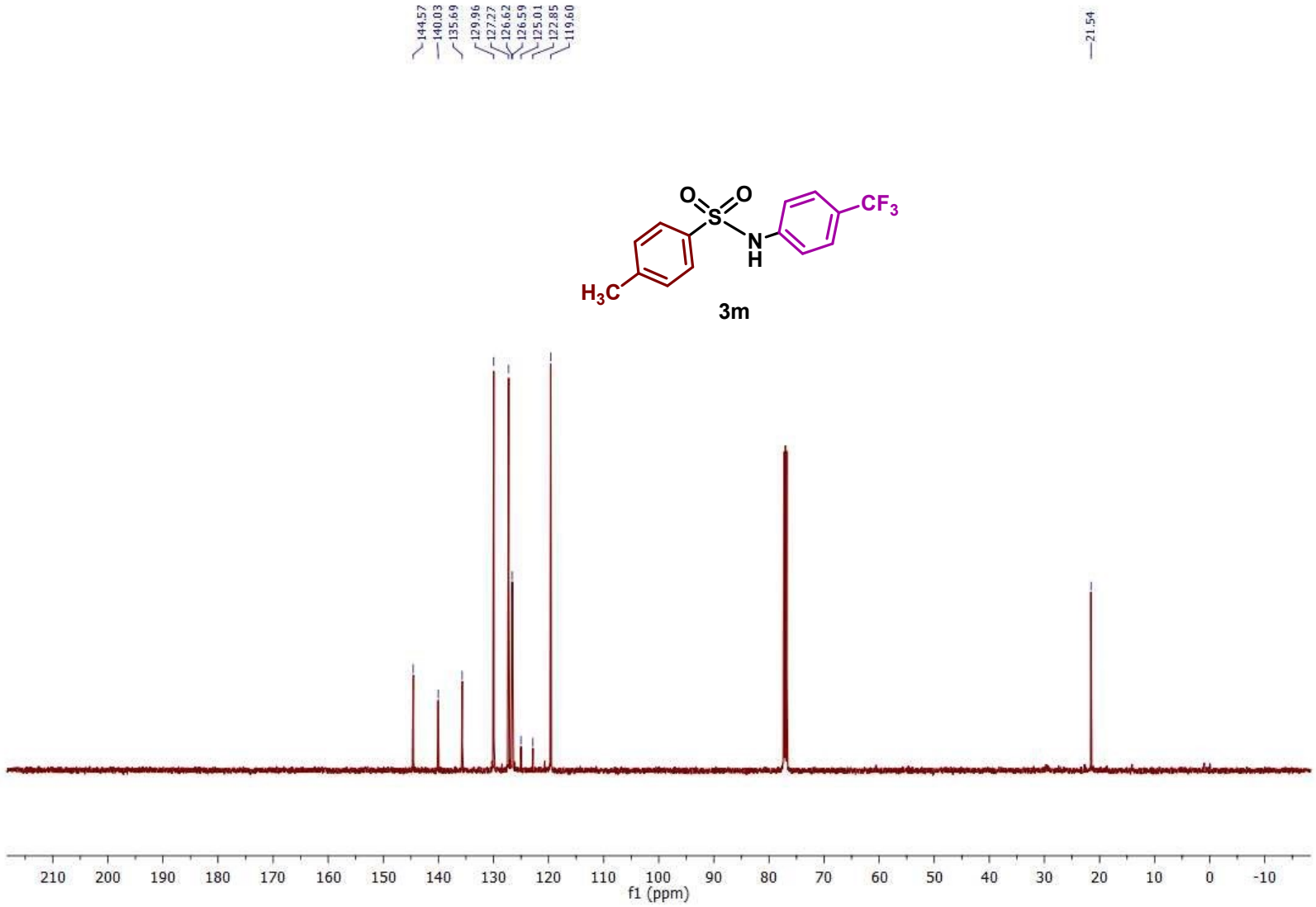

144.24
142.13
136.78
130.38
127.17
127.03
127.00
124.20
123.60
119.09
21.42
O
O
S
N
H
H3C
CF3
3l
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

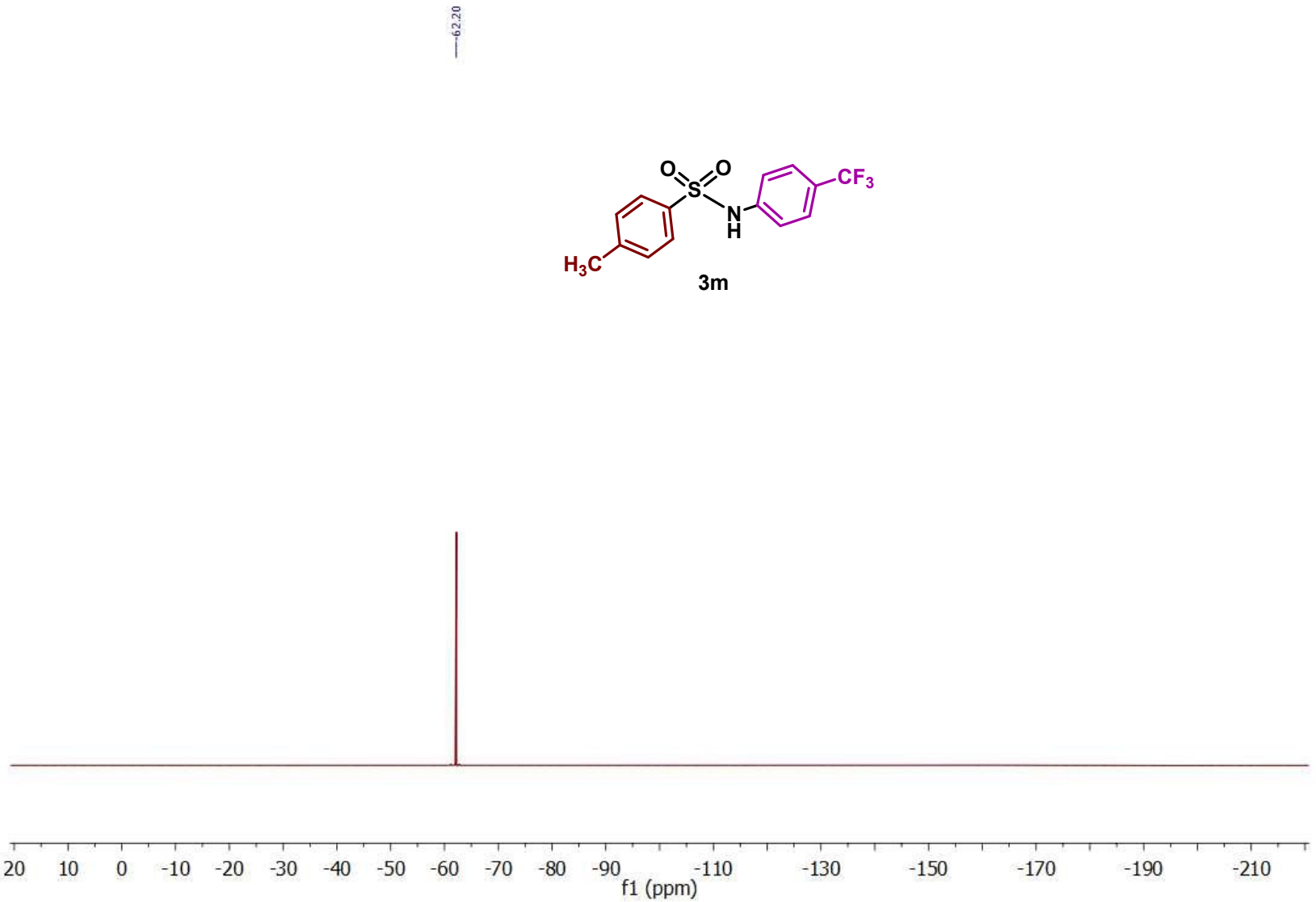

O
O
S
N
H
$H_3C$
$CF_3$

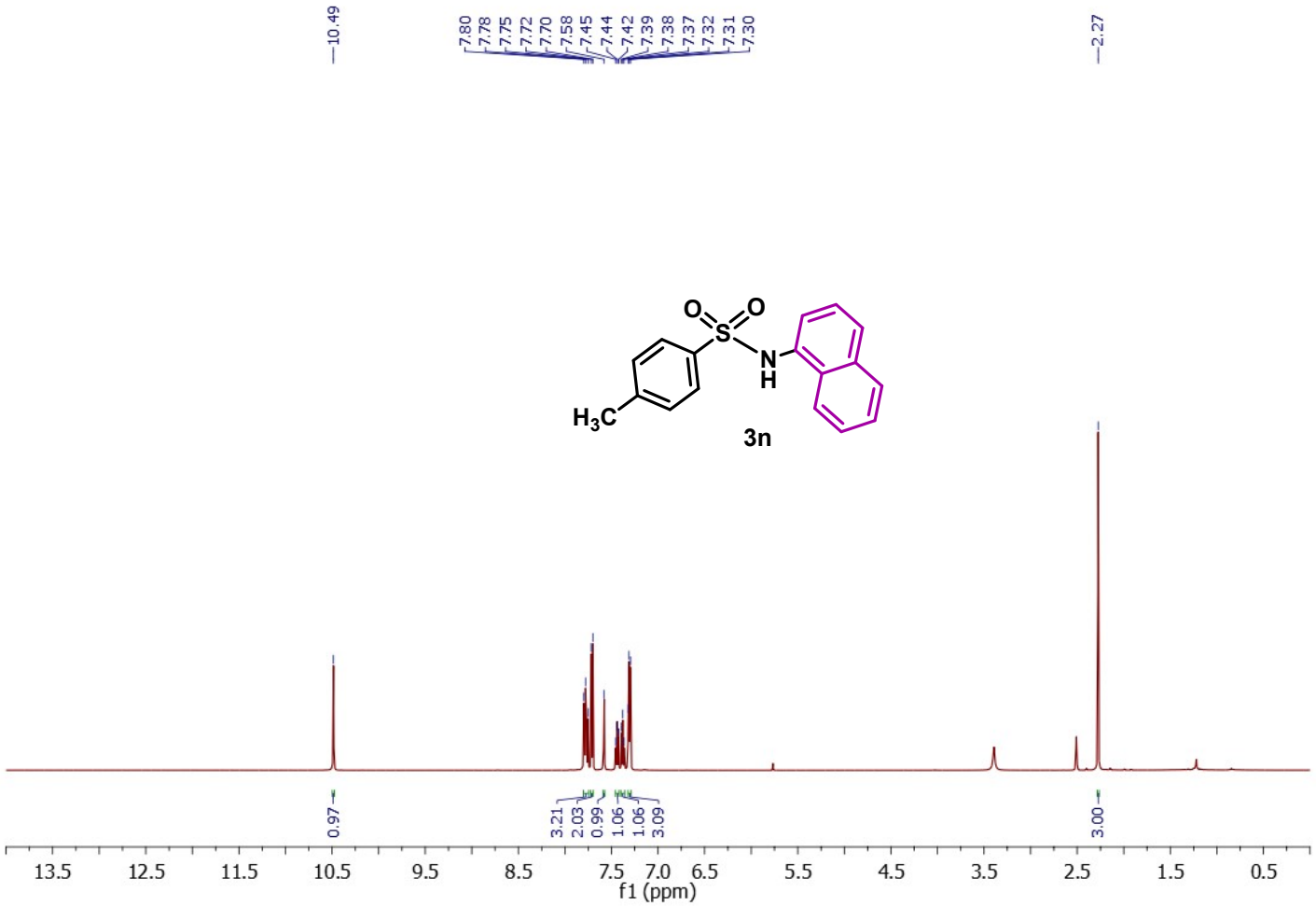
3l

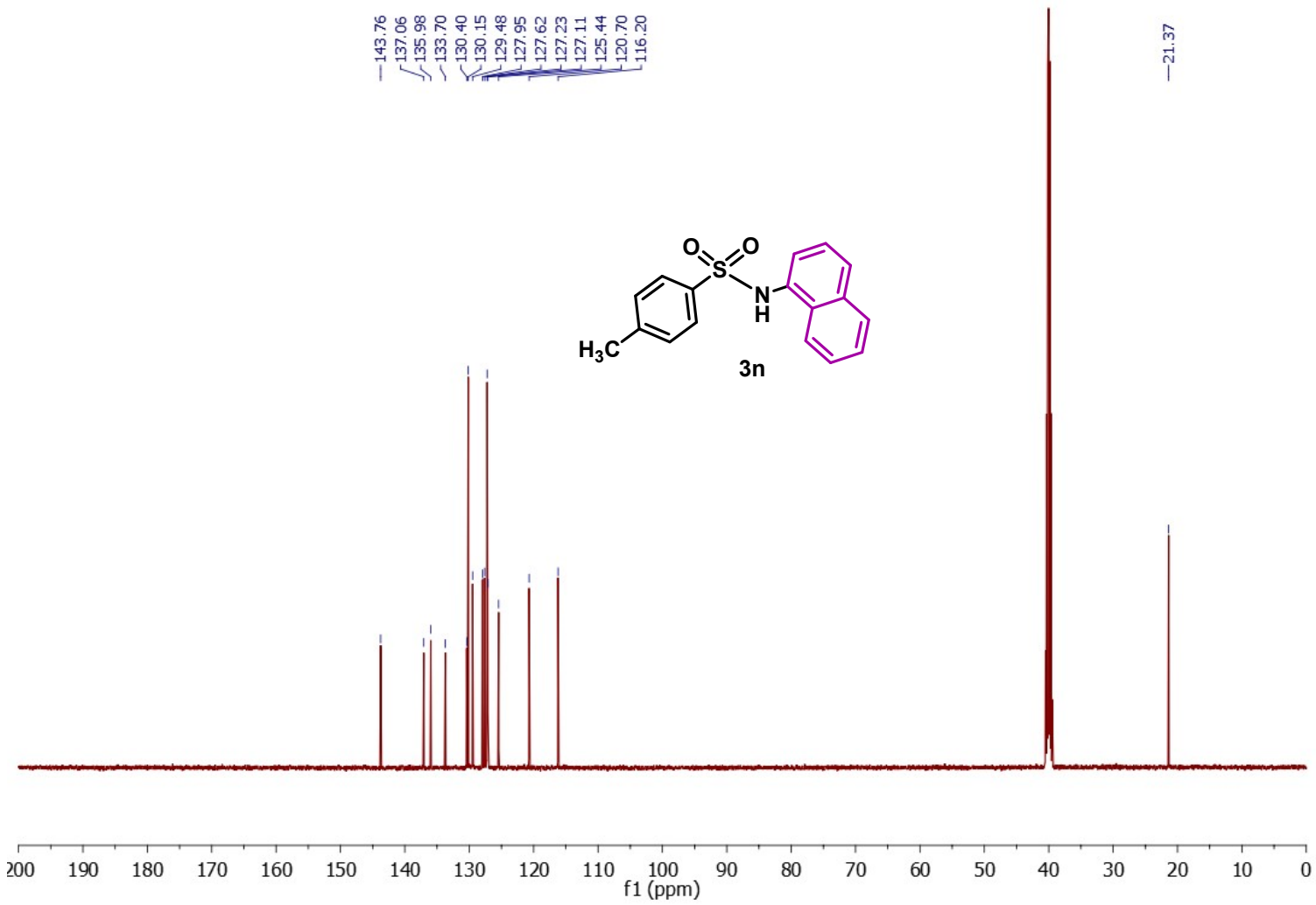

61.39
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

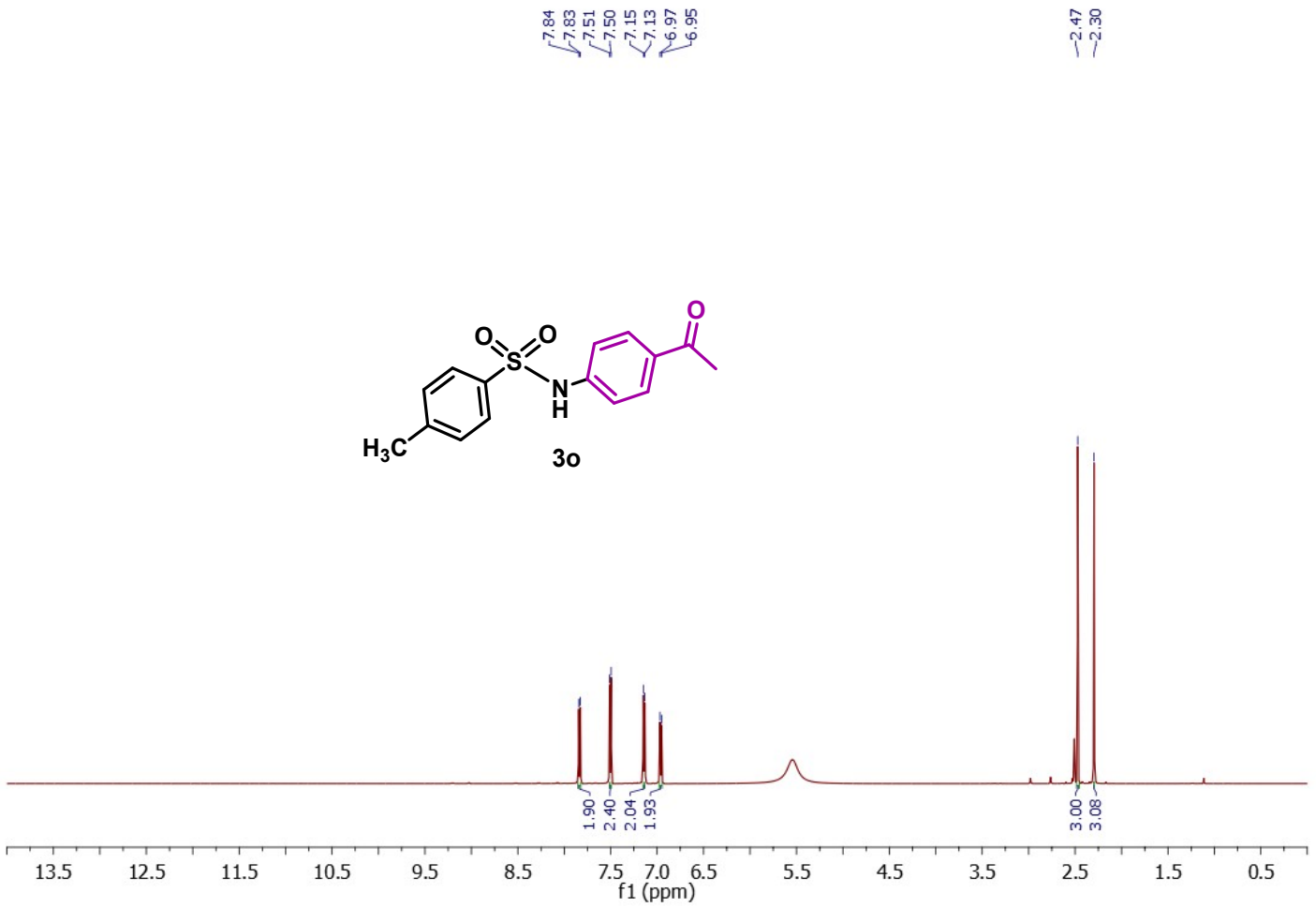

7.77
7.76
7.51
7.49
7.30
7.28
7.28
7.22
7.20
2.41
O
O
S
N
H
CF3
H3C
3m
1.93
2.10
2.25
1.95
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

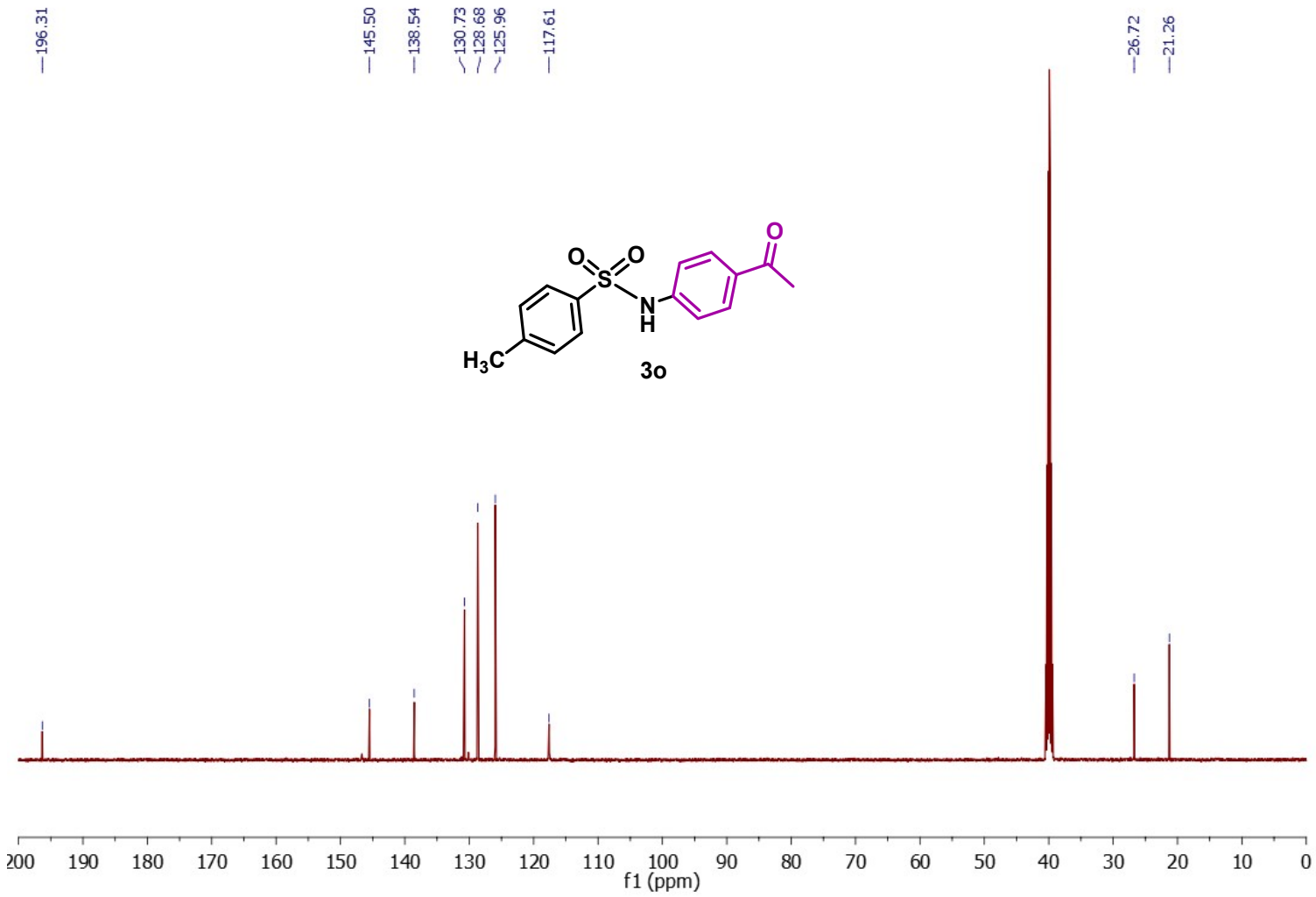

144.57
140.03
135.69
129.96
127.27
126.62
126.59
125.01
122.85
119.60
21.54
O
O
S
N
H
CF3
H3C
3m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

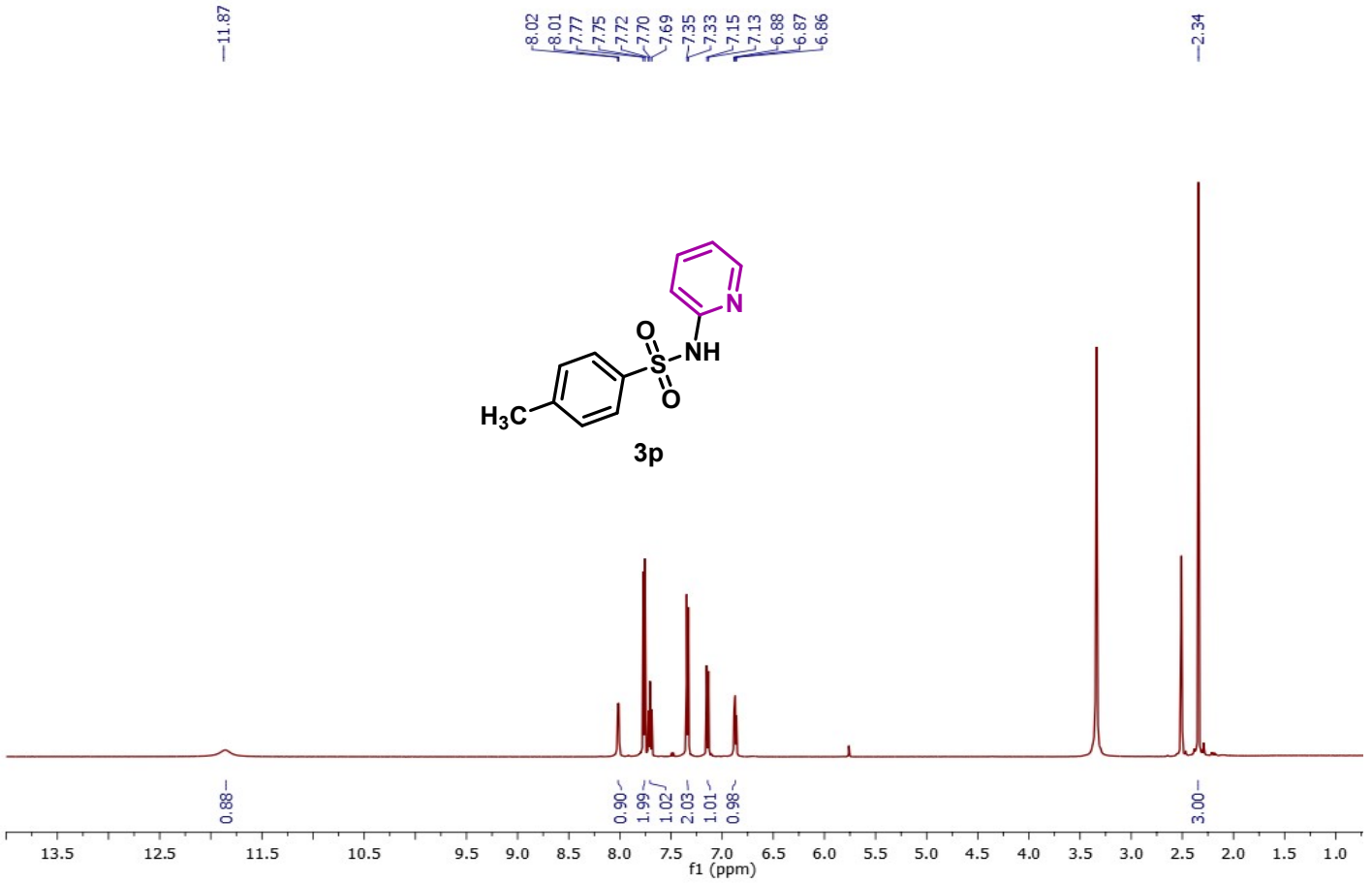

-62.20
O
O
S
N
H
CF3
H3C
3m
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

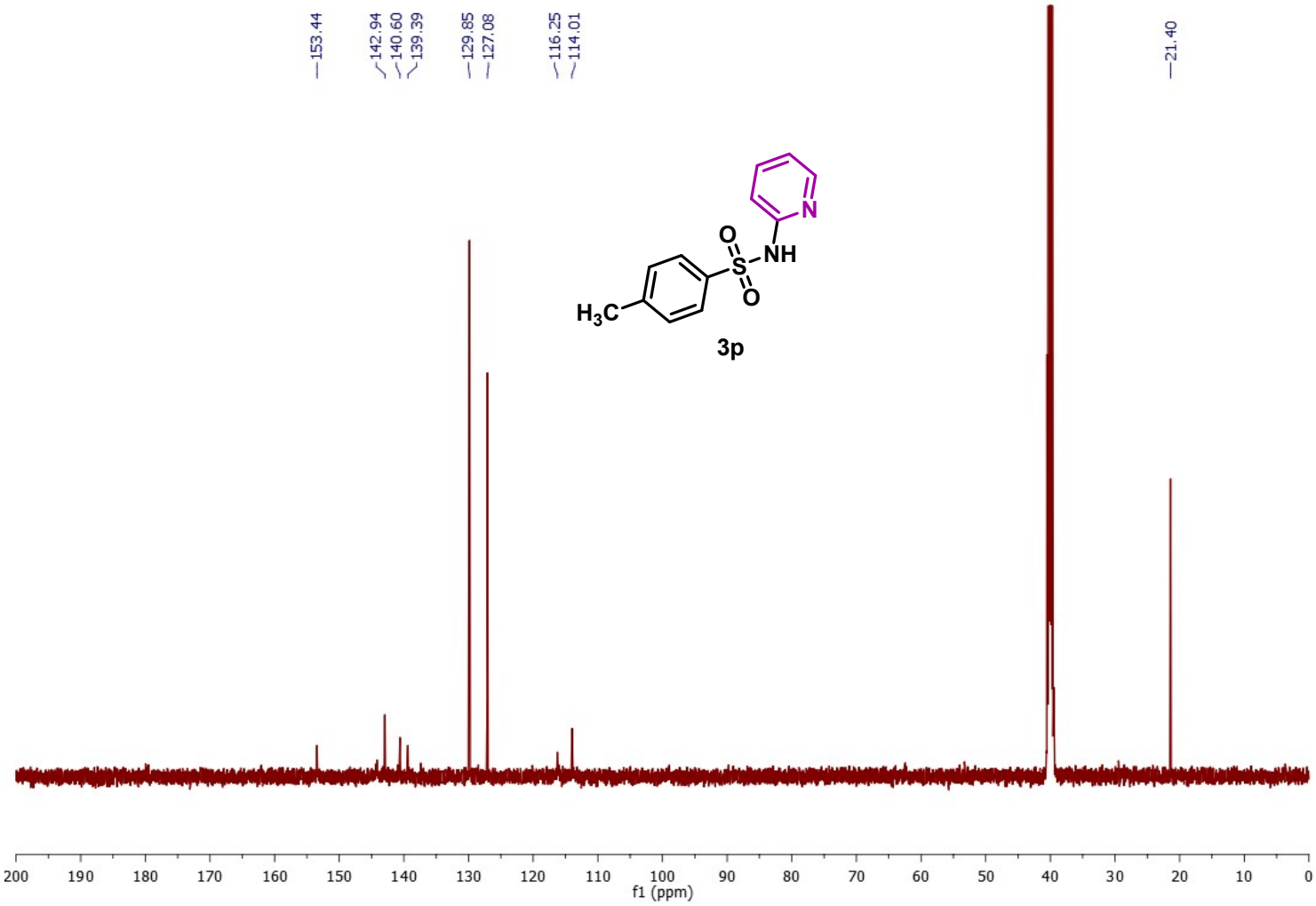

10.49
7.80
7.78
7.75
7.72
7.70
7.58
7.45
7.44
7.42
7.39
7.38
7.37
7.32
7.31
7.30
2.27
O
O
S
N
H
H3C
3n
0.97
3.21
2.03
0.99
1.06
1.06
3.09
3.00
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

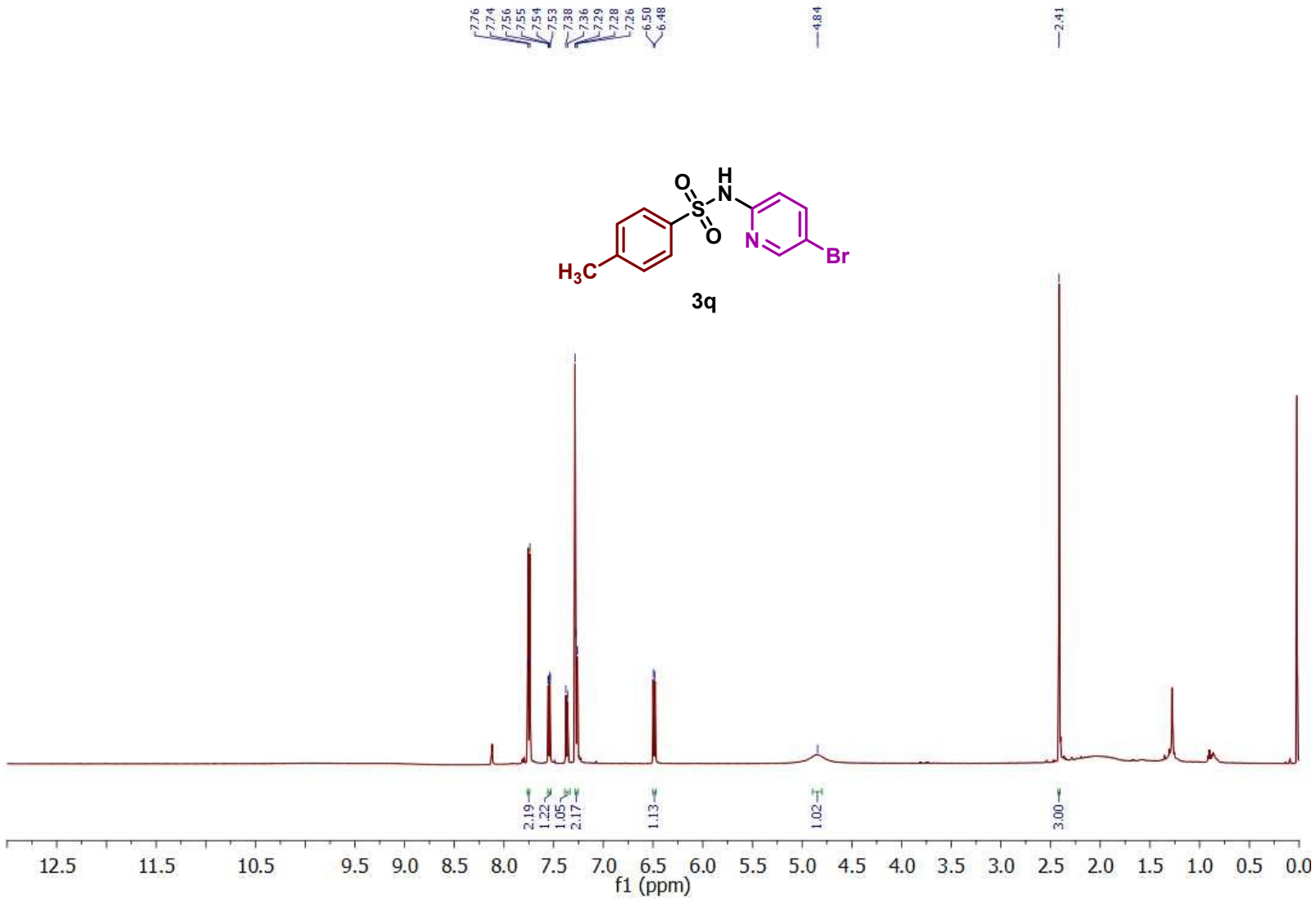

143.76
137.06
135.98
133.70
130.40
130.15
129.48
127.95
127.62
127.23
127.11
125.44
120.70
116.20
21.37
O
O
S
N
H
H3C
3n
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

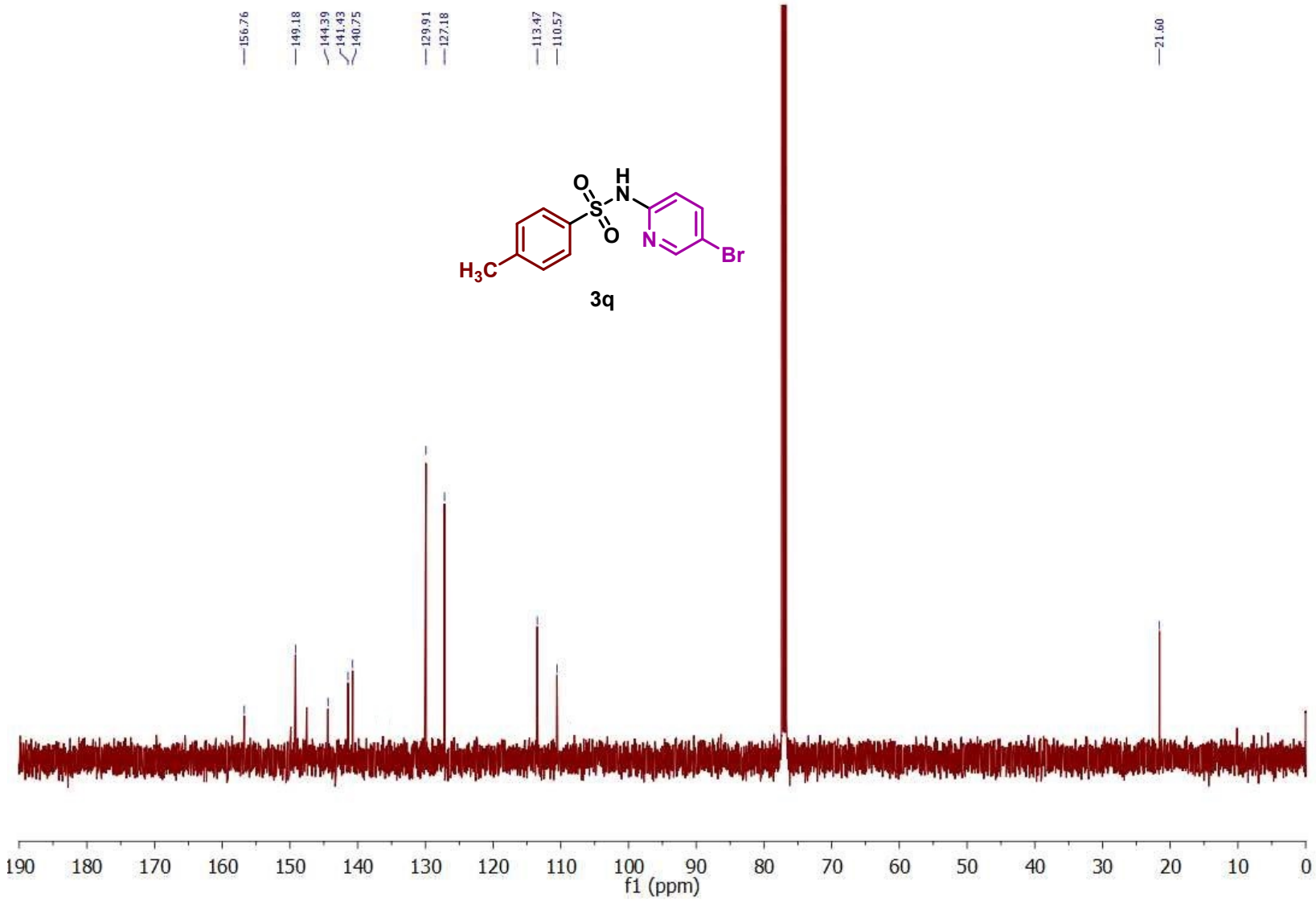

7.84
7.83
7.51
7.50
7.15
7.13
6.97
6.95
2.47
2.30
O
O
O
S
N
H
H3C
3o
1.90
2.40
2.04
1.93
3.00
3.08
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

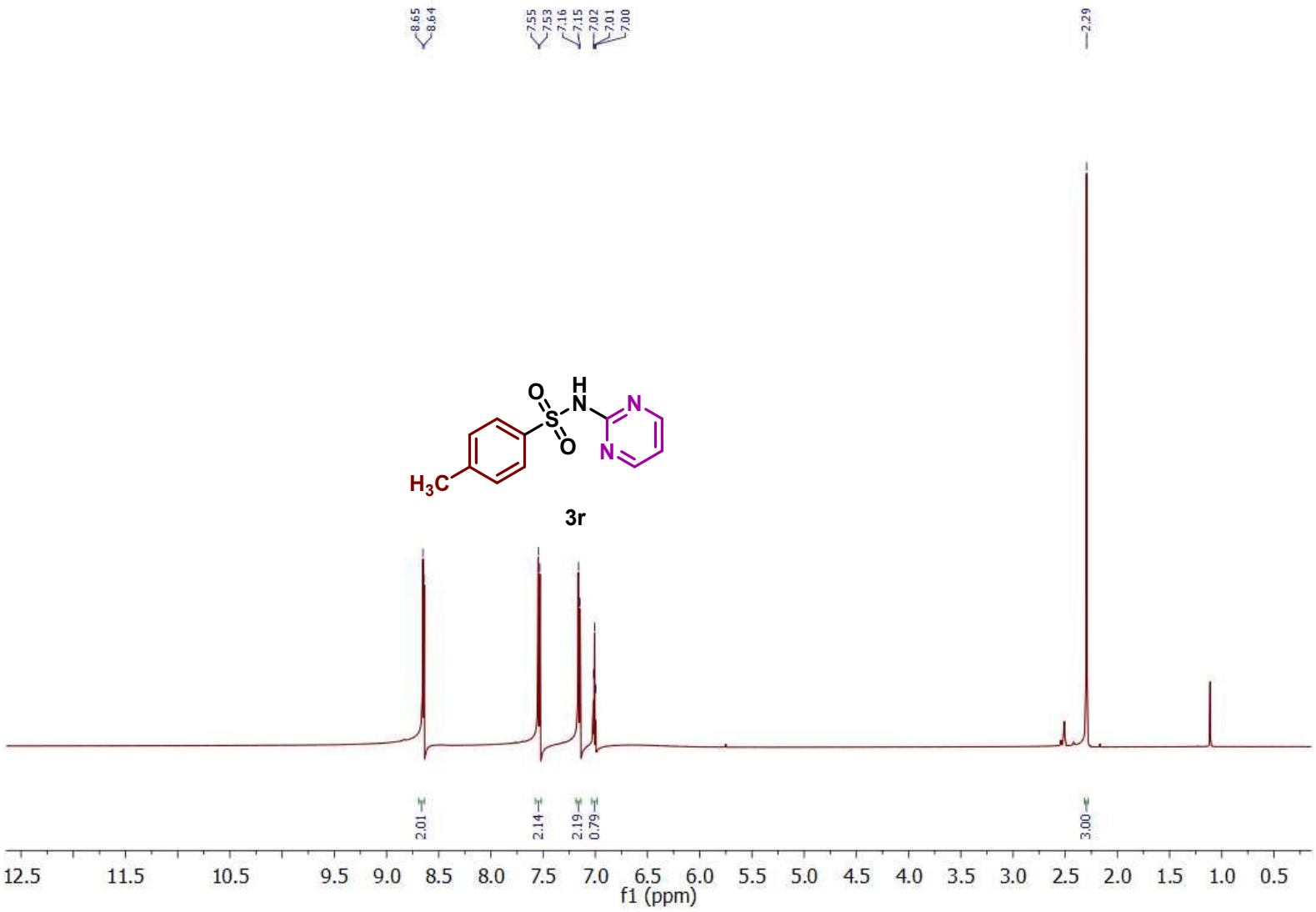

196.31
145.50
138.54
130.73
128.68
125.96
117.61
26.72
21.26
O
O
S
O
N
H
H3C
3o
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

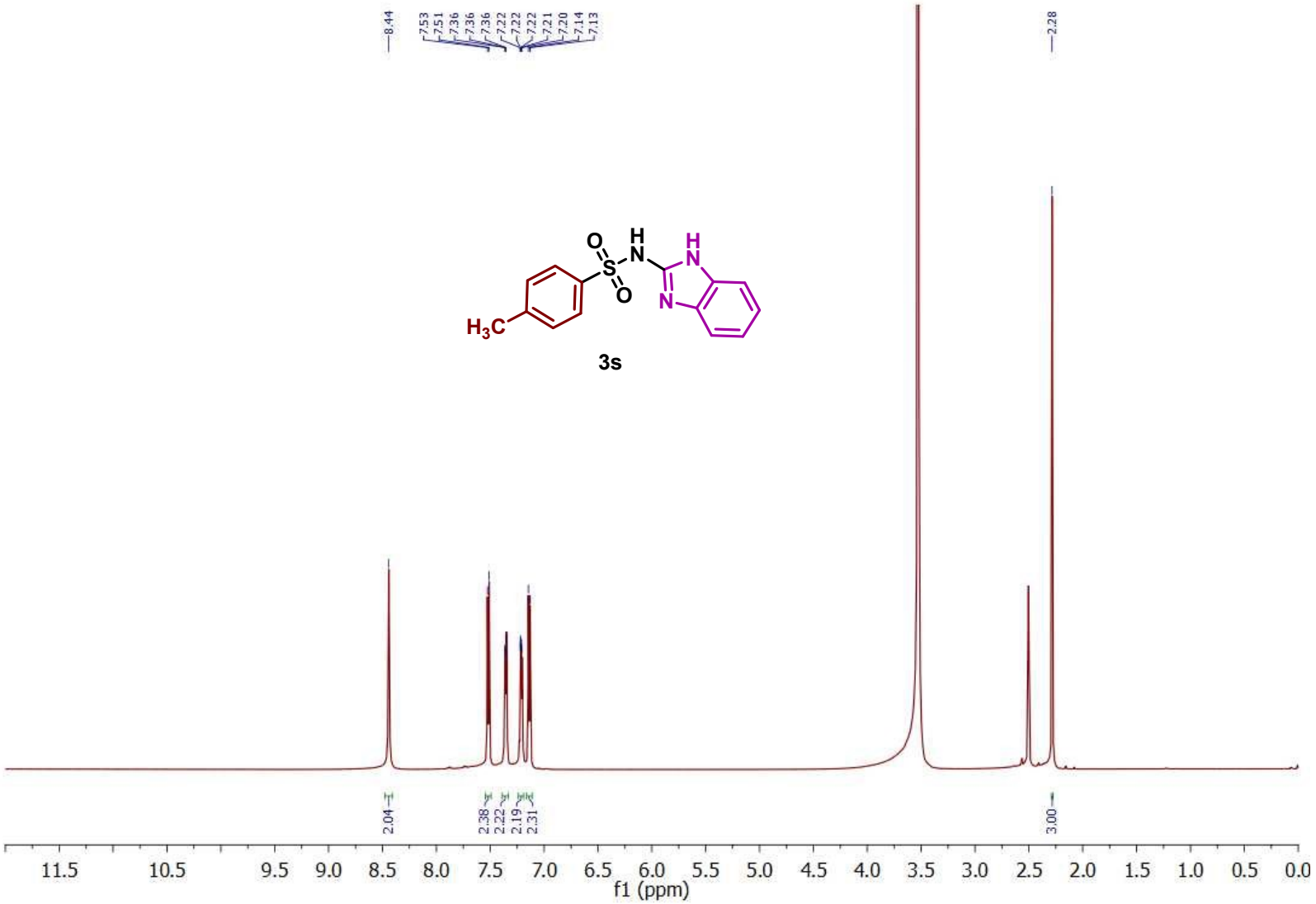

11.87
8.02
8.01
7.77
7.75
7.72
7.70
7.69
7.35
7.33
7.15
7.13
6.88
6.87
6.86
2.34
N
O
NH
S
O
H3C
3p
0.88
0.90
1.99
1.02
2.03
1.01
0.98
3.00
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)

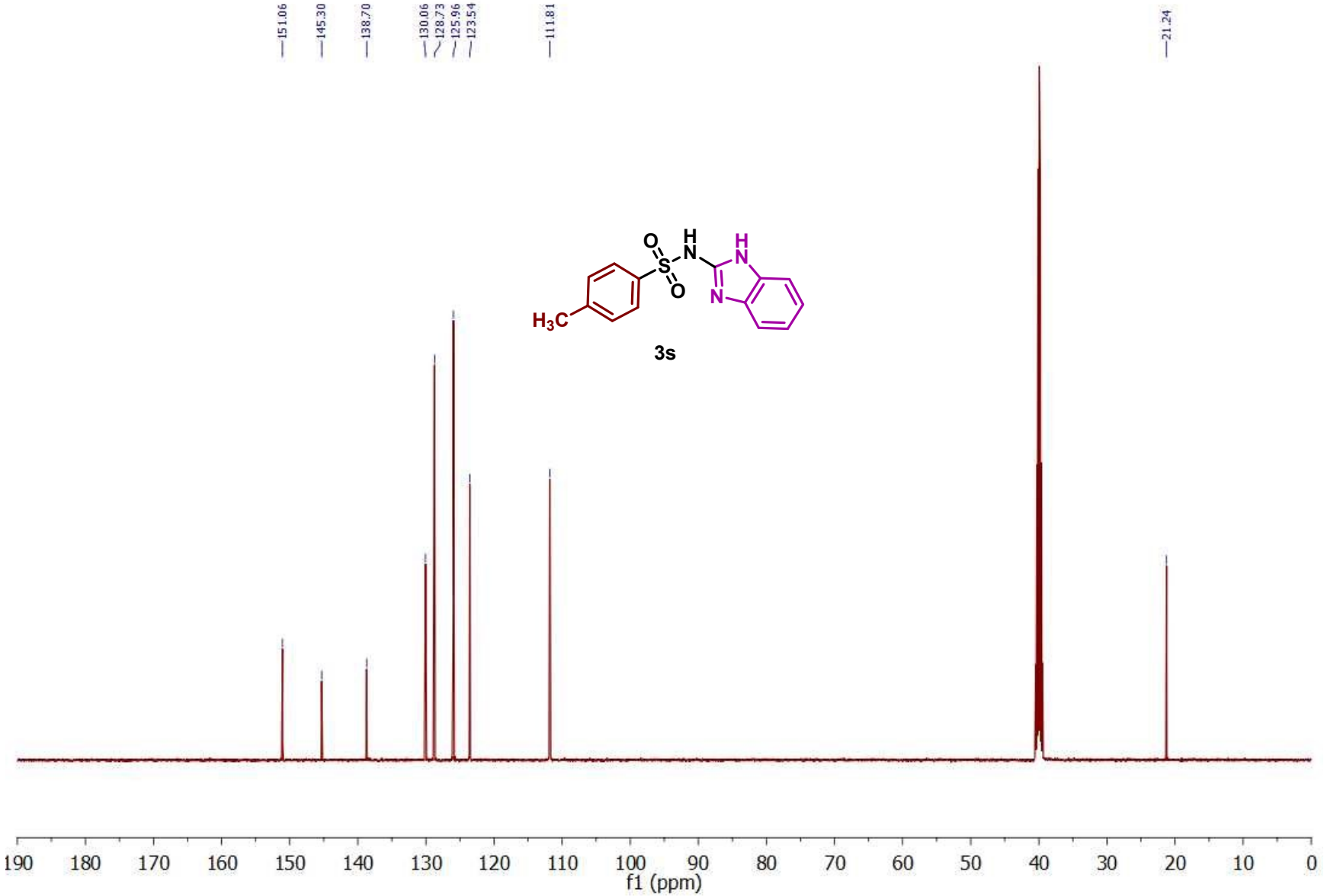

153.44
142.94
140.60
139.39
129.85
127.08
116.25
114.01
21.40
N
O
NH
S
O
H3C
3p
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

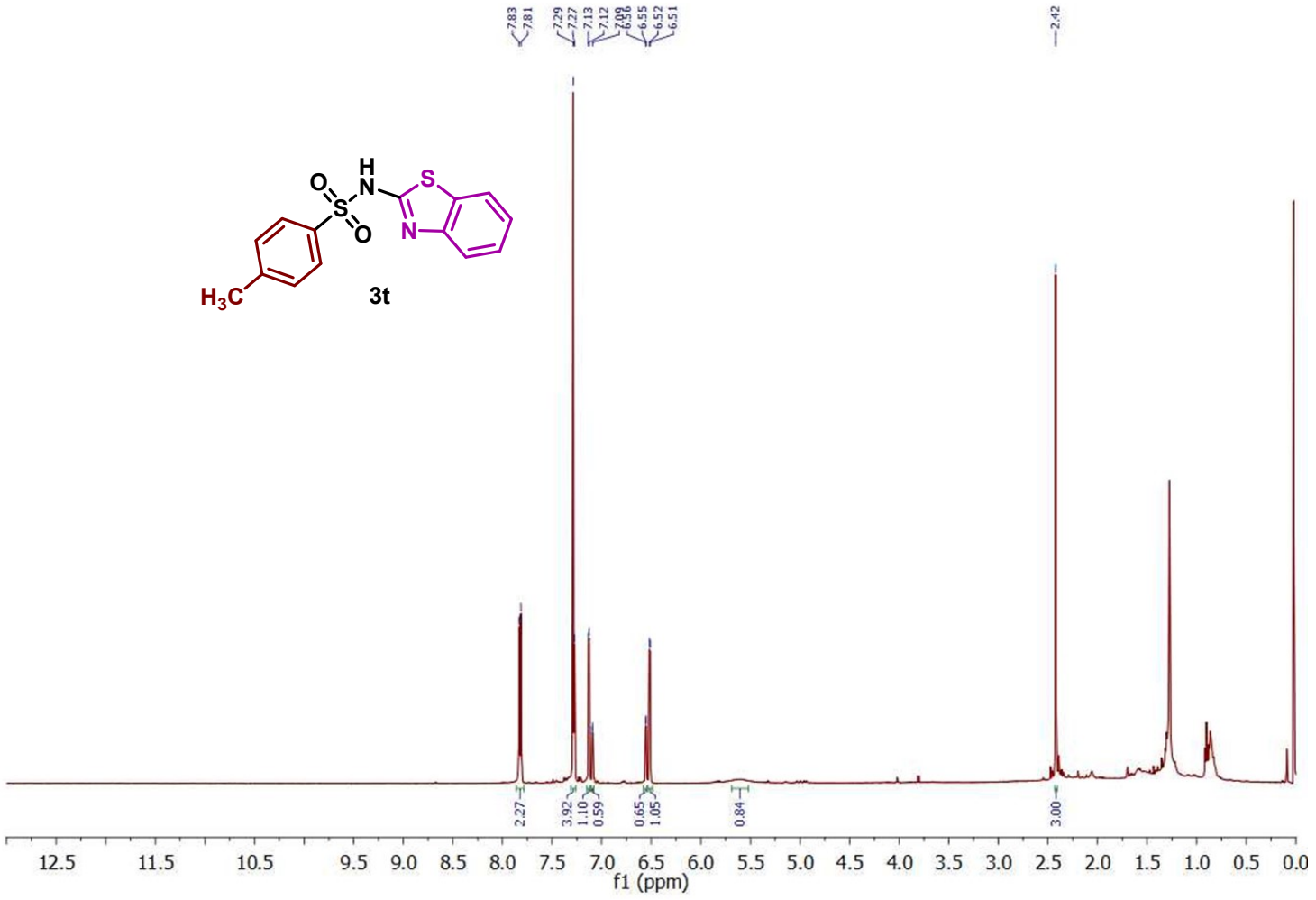

7.76
7.74
7.56
7.55
7.54
7.53
7.38
7.36
7.29
7.28
7.26
6.50
6.48
4.84
2.41
O
H
N
S
O
N
Br
H3C
3q
2.19
1.22
1.05
2.17
1.13
1.02
3.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

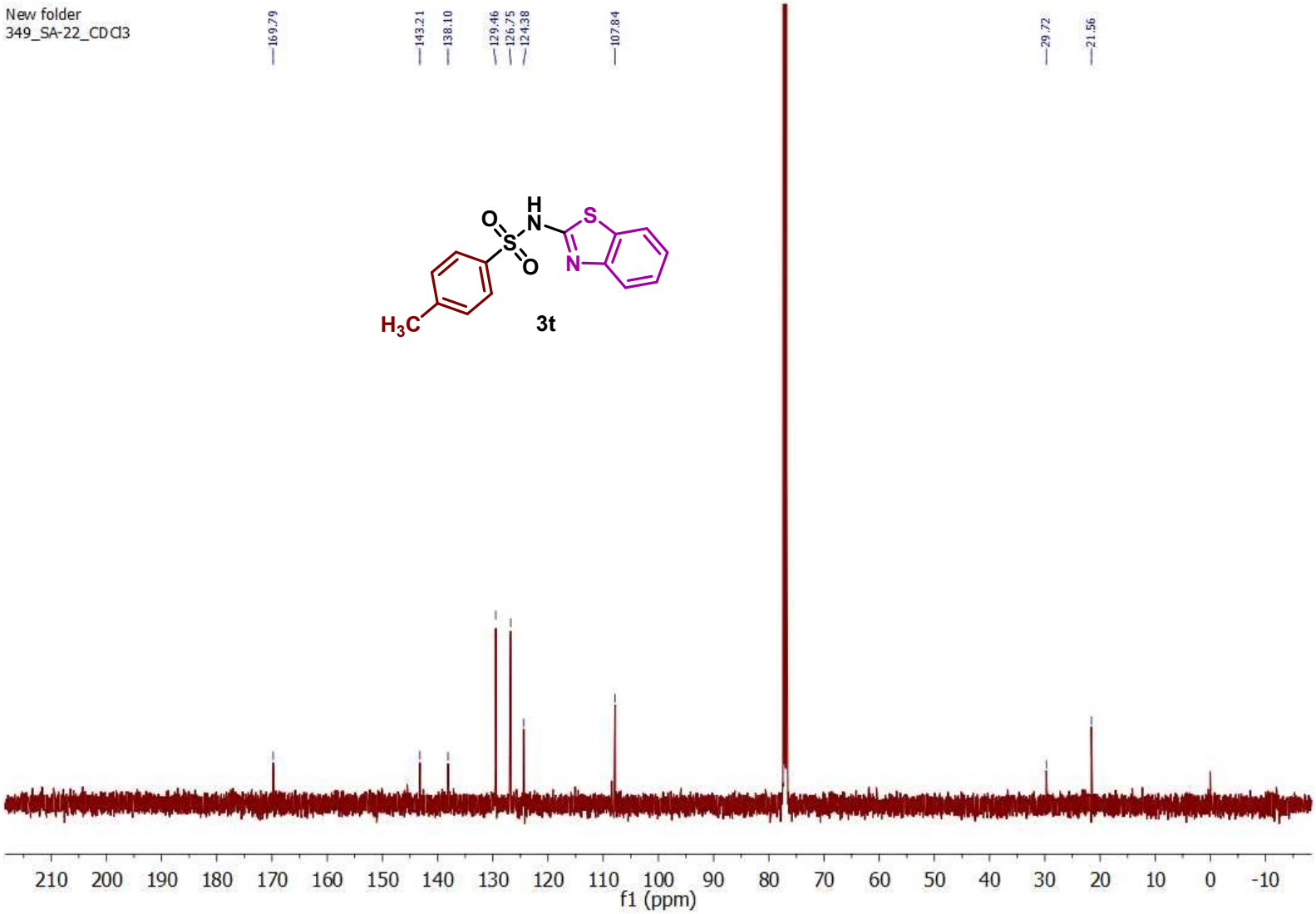

156.76
149.18
144.39
141.43
140.75
129.91
127.18
113.47
110.57
21.60
O
S
O
H
N
N
Br
H3C
3q
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

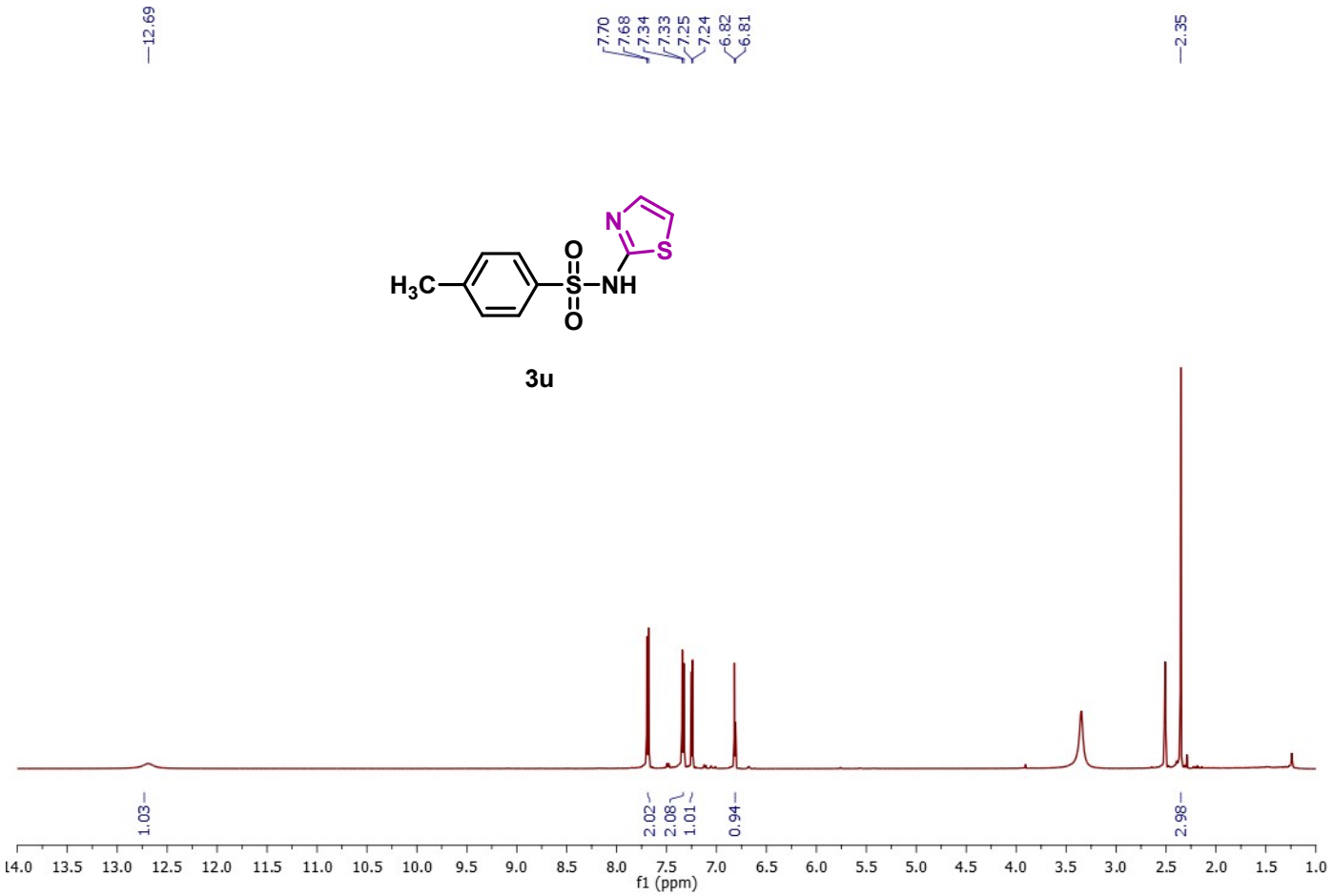

8.65
8.64
7.55
7.53
7.16
7.15
7.02
7.01
7.00
2.29
O
H
N
S
O
N
N
H3C
3r
2.01
2.14
2.19
0.79
3.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


8.44
7.53
7.51
7.36
7.36
7.36
7.22
7.22
7.22
7.21
7.20
7.14
7.13
2.28
O
H
N
S
O
H
N
N
H3C
3s
2.04
2.38
2.22
2.19
2.31
3.00
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

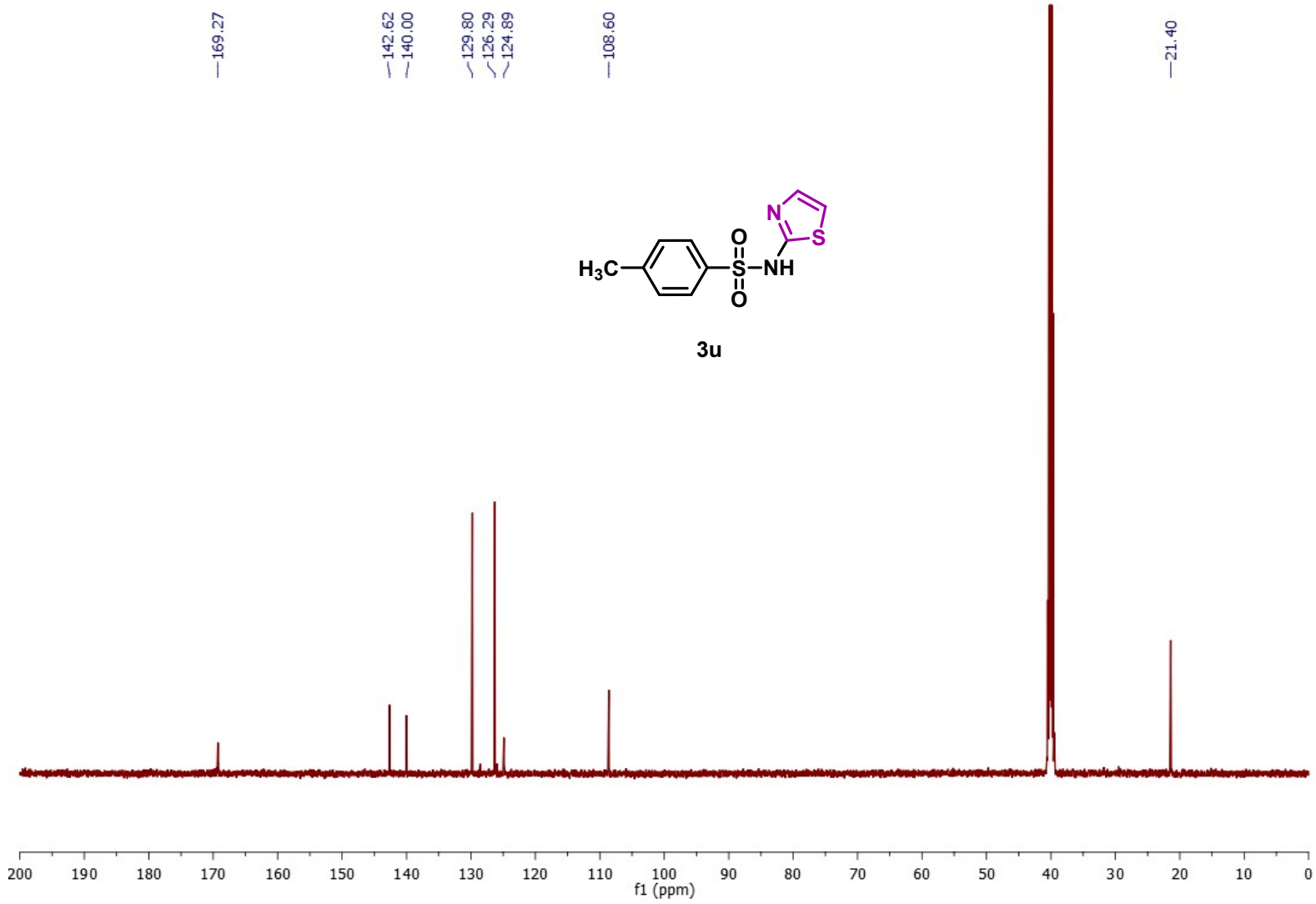

151.06
145.30
138.70
130.06
128.73
125.96
123.54
111.81
21.24
O
H
N
S
O
H
N
N
H3C
3s
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.83
7.81
7.29
7.27
7.13
7.12
7.09
7.08
6.55
6.52
6.51
2.42
O
H
N
S
O
S
O
N
H3C
3t
2.27
3.92
1.10
0.59
0.65
1.05
0.84
3.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


New folder
349_SA-22_CDCl3
169.79
143.21
138.10
129.46
126.75
124.38
107.84
29.72
21.56
O
S
O
H
N
S
N
H3C
3t
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

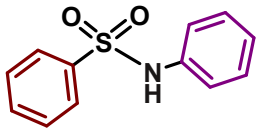

12.69
7.70
7.68
7.34
7.33
7.25
7.24
6.82
6.81
2.35
N
S
O
S
NH
O
H3C
1.03
2.02
2.08
1.01
0.94
2.98
14.0 13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)

**3u**

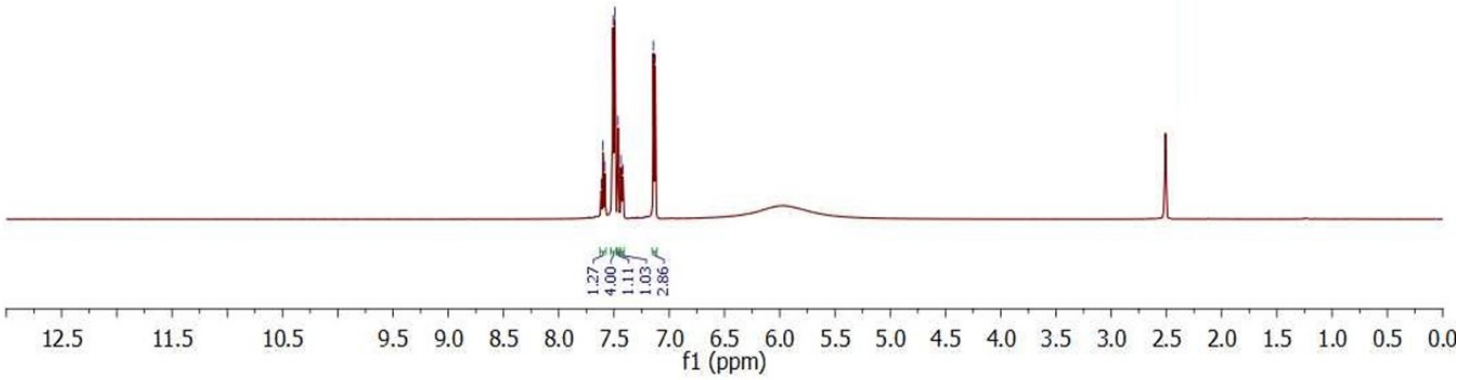

169.27
142.62
140.00
129.80
126.29
124.89
108.60
21.40
N
S
O
S
NH
O
H3C
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

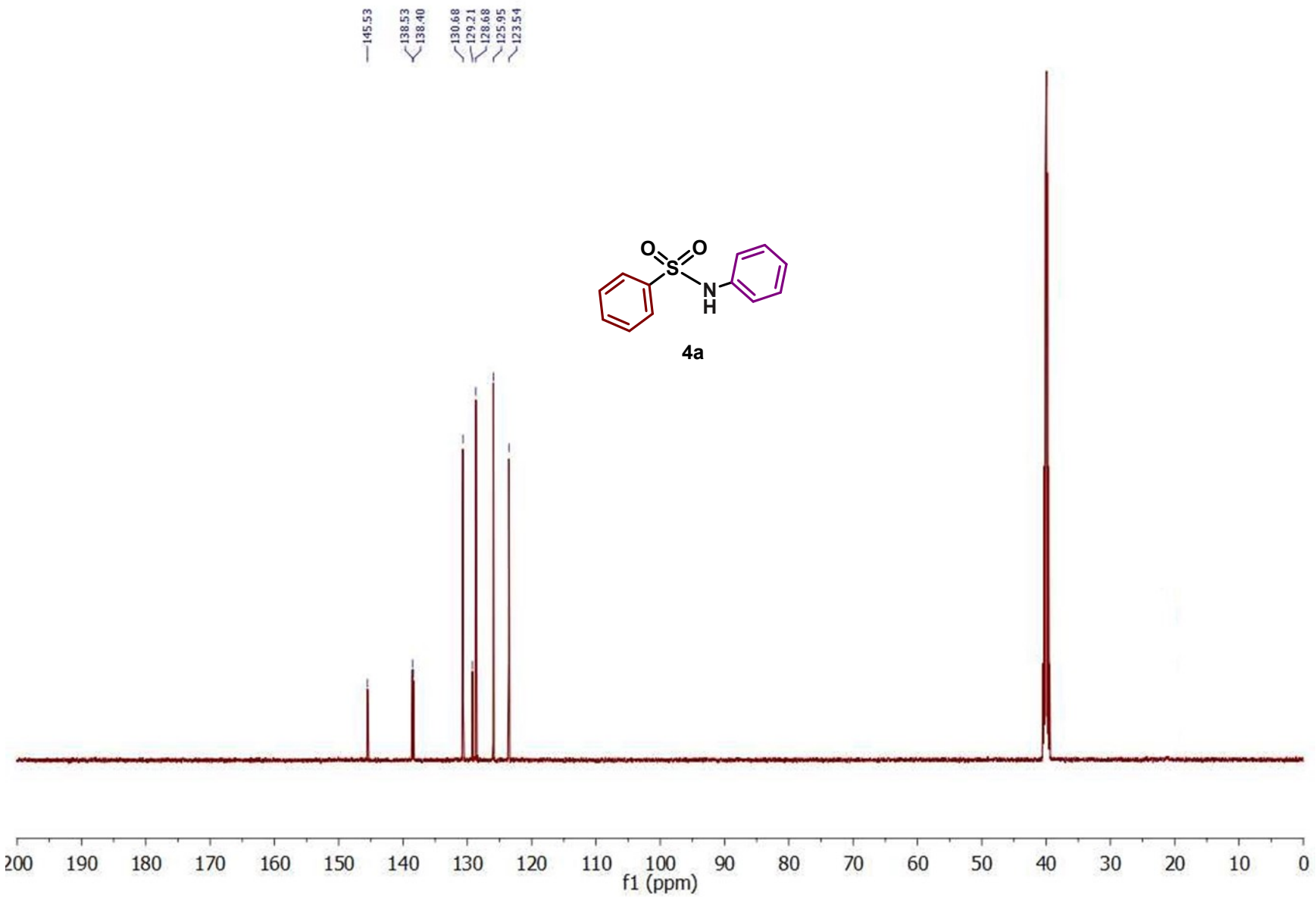
**3u**

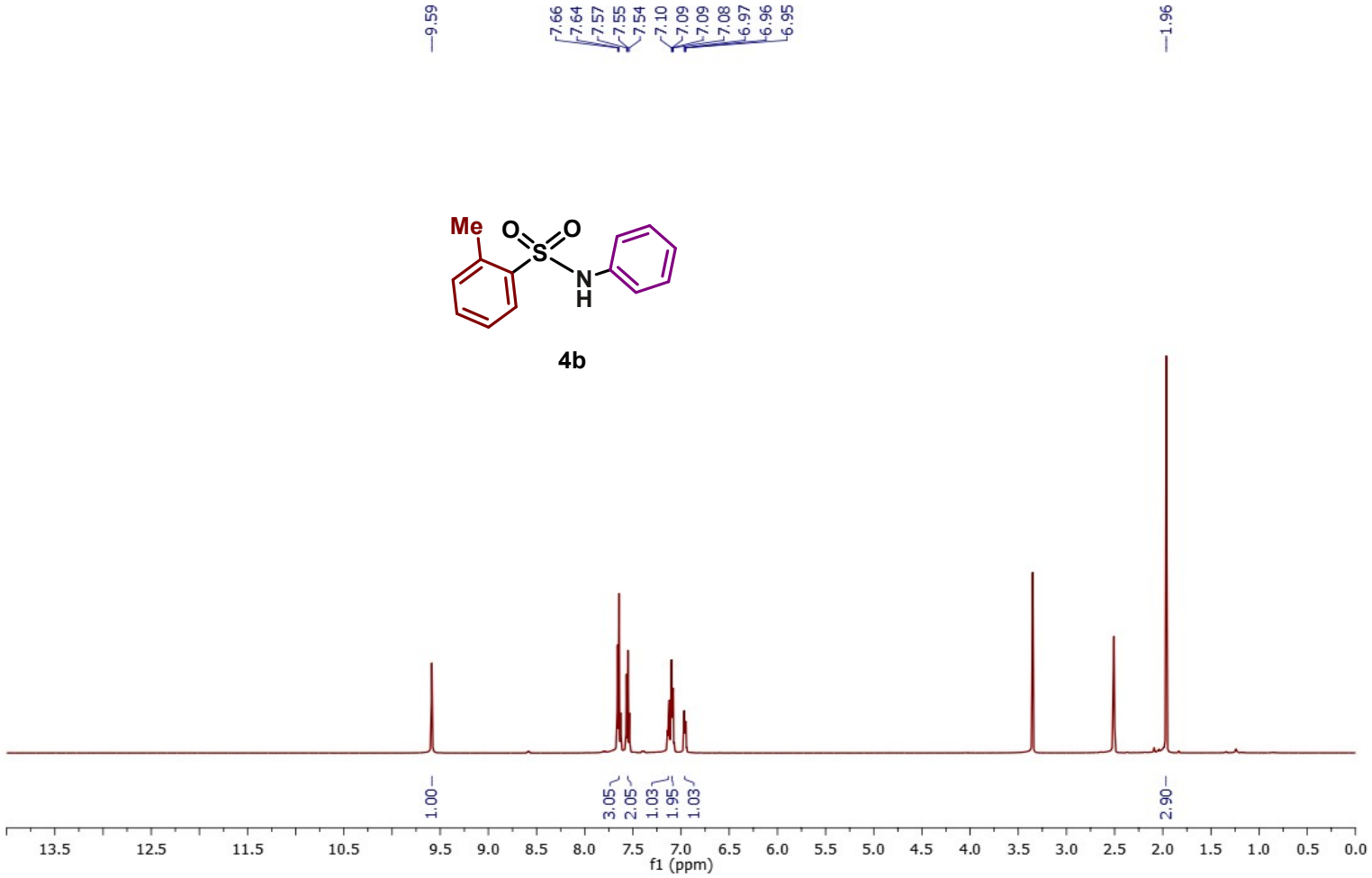

7.62
7.60
7.58
7.51
7.49
7.46
7.44
7.42
7.14
7.13

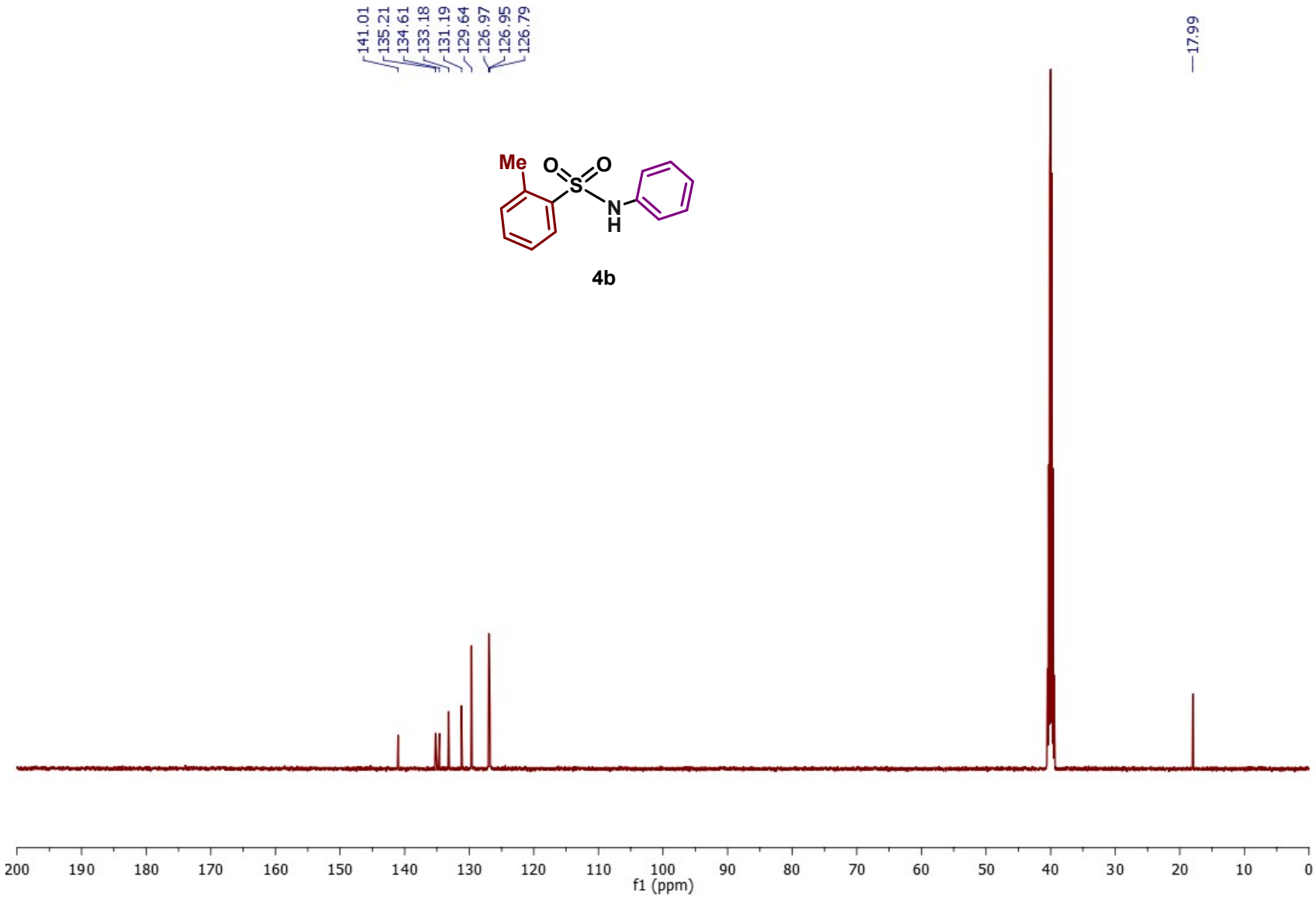

O
O
S
N
H

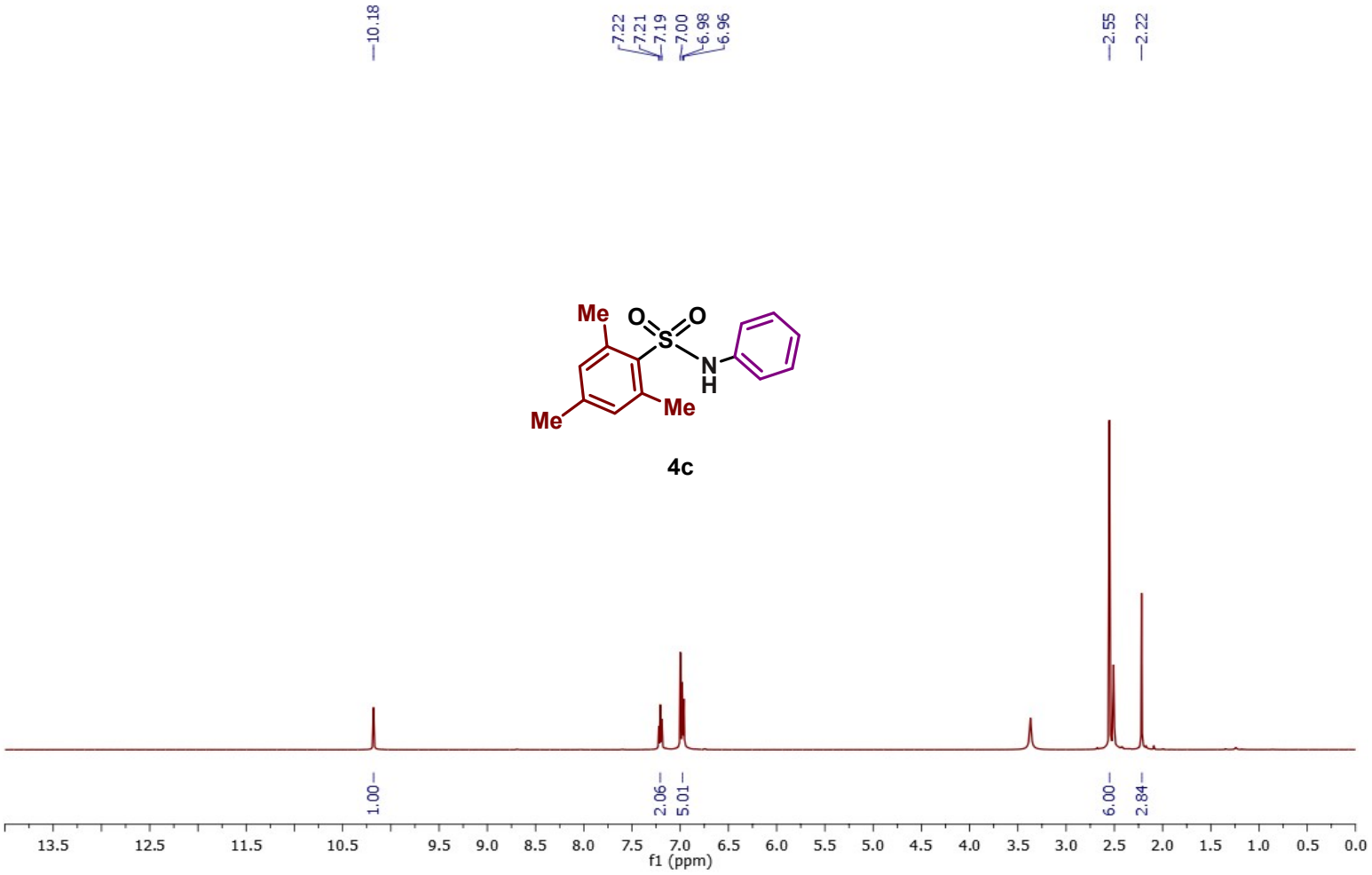
**4a**


1.27
4.00
1.11
1.03
2.86
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

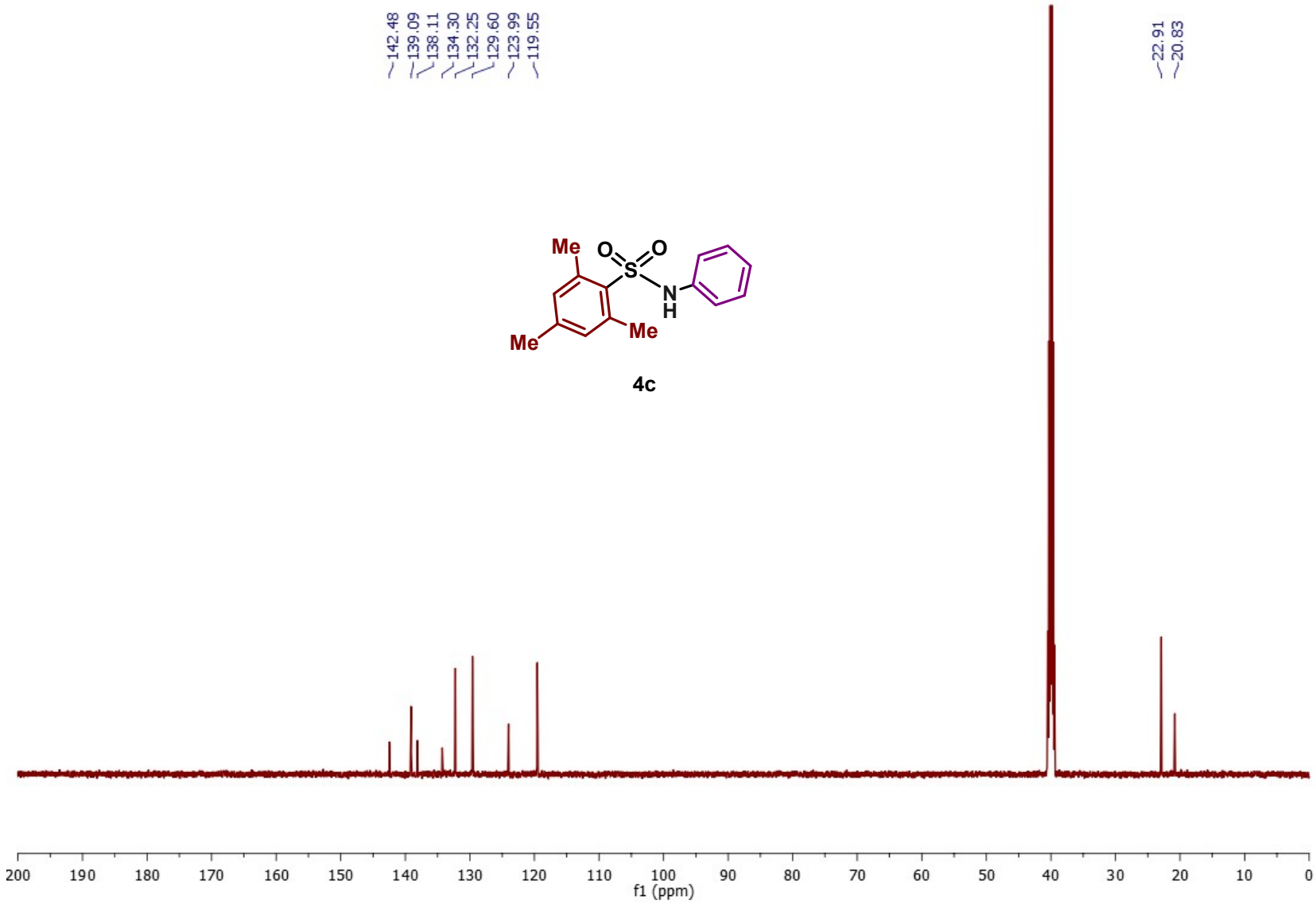

145.53
138.53
138.40
130.68
129.21
128.68
125.95
123.54
O
O
S
N
H
4a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


9.59
7.66
7.64
7.57
7.55
7.54
7.10
7.09
7.09
7.08
6.97
6.96
6.95
1.96
Me
O
O
S
N
H
4b
1.00
3.05
2.05
1.03
1.95
1.03
2.90
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

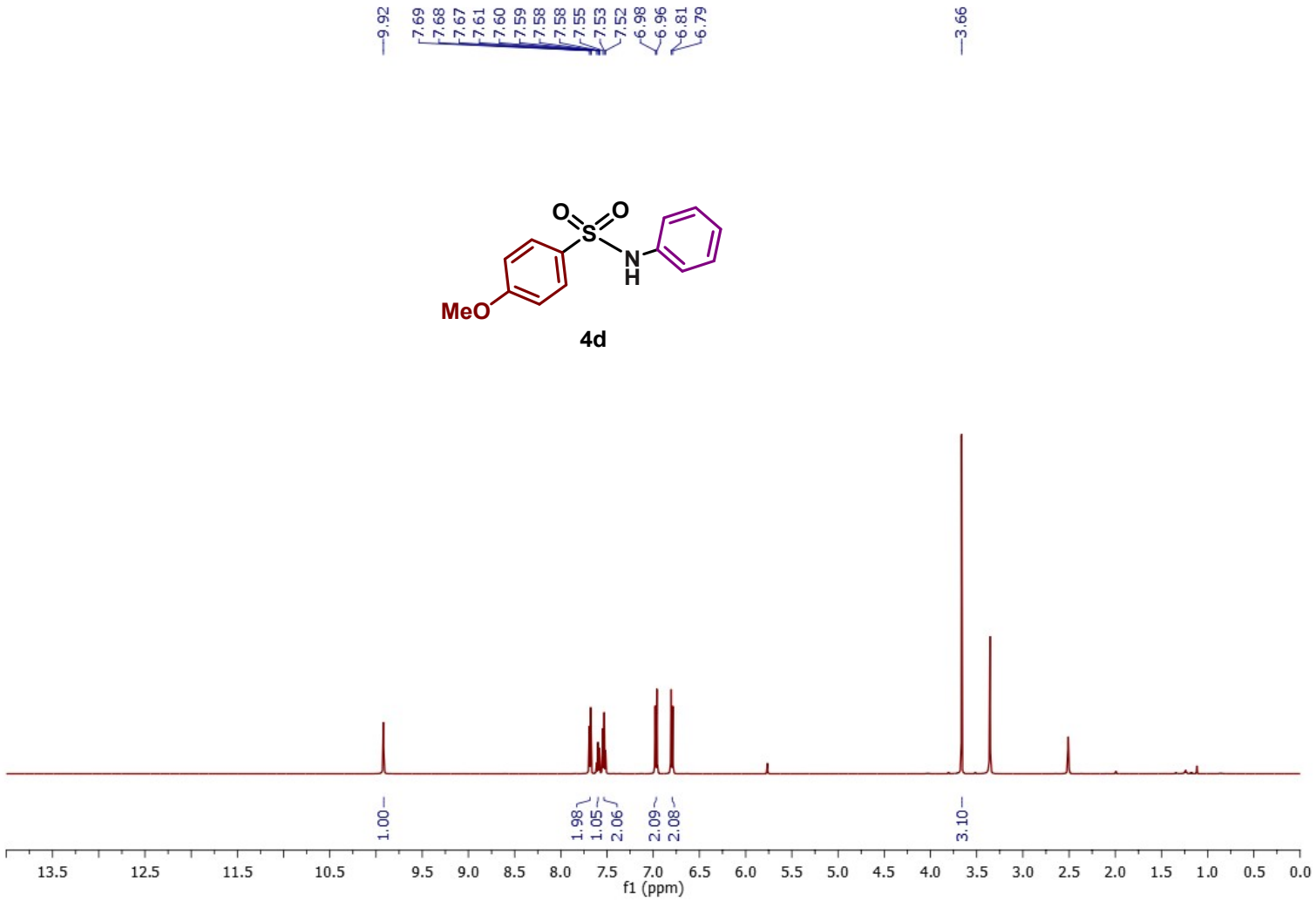

141.01
135.21
134.61
133.18
131.19
129.64
126.97
126.95
126.79
17.99
Me
O
O
S
N
H
4b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


10.18
7.22
7.21
7.19
7.00
6.98
6.96
2.55
2.22
Me
O
O
S
N
H
Me
Me
1.00
2.06
5.01
6.00
2.84
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

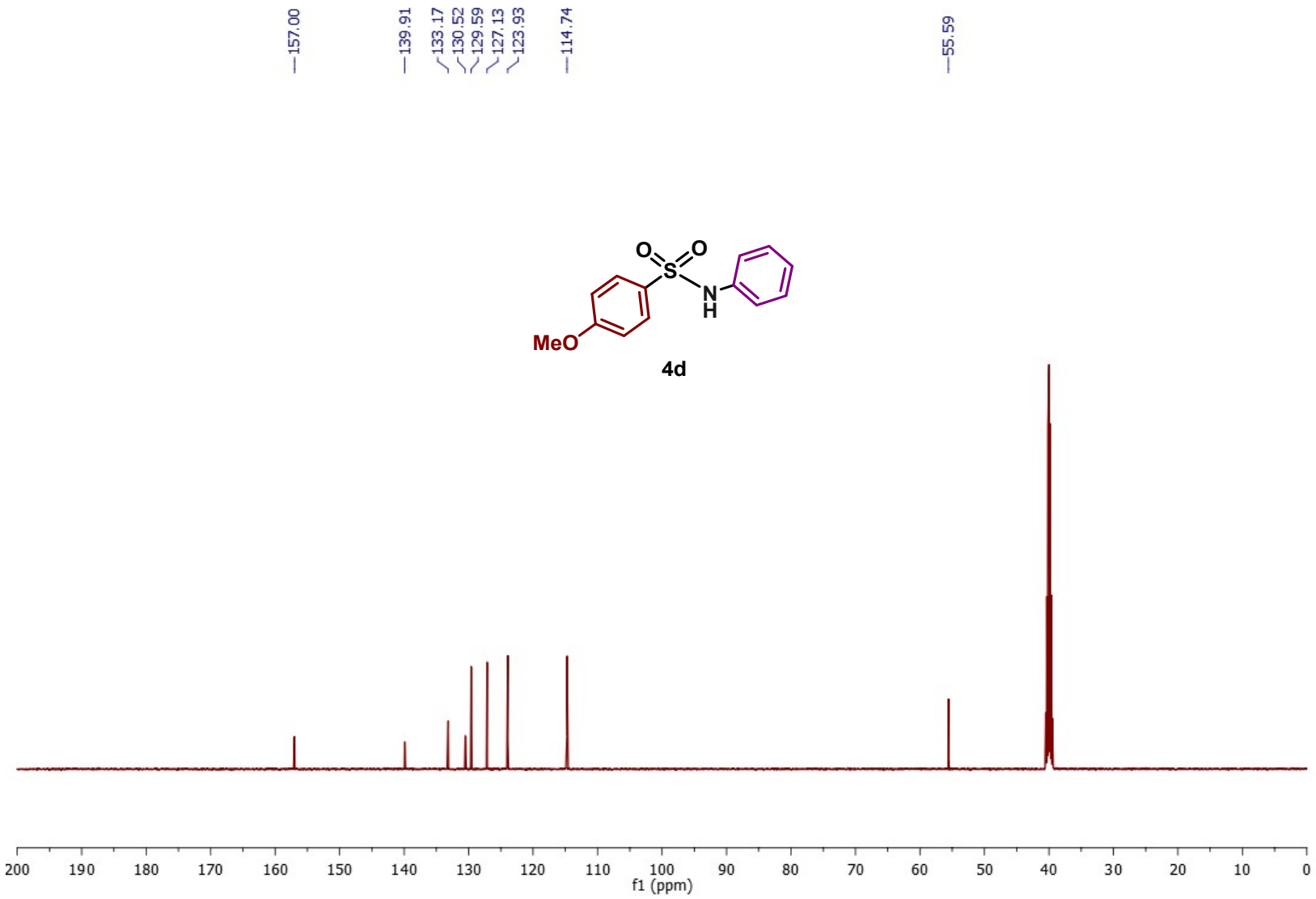
**4c**

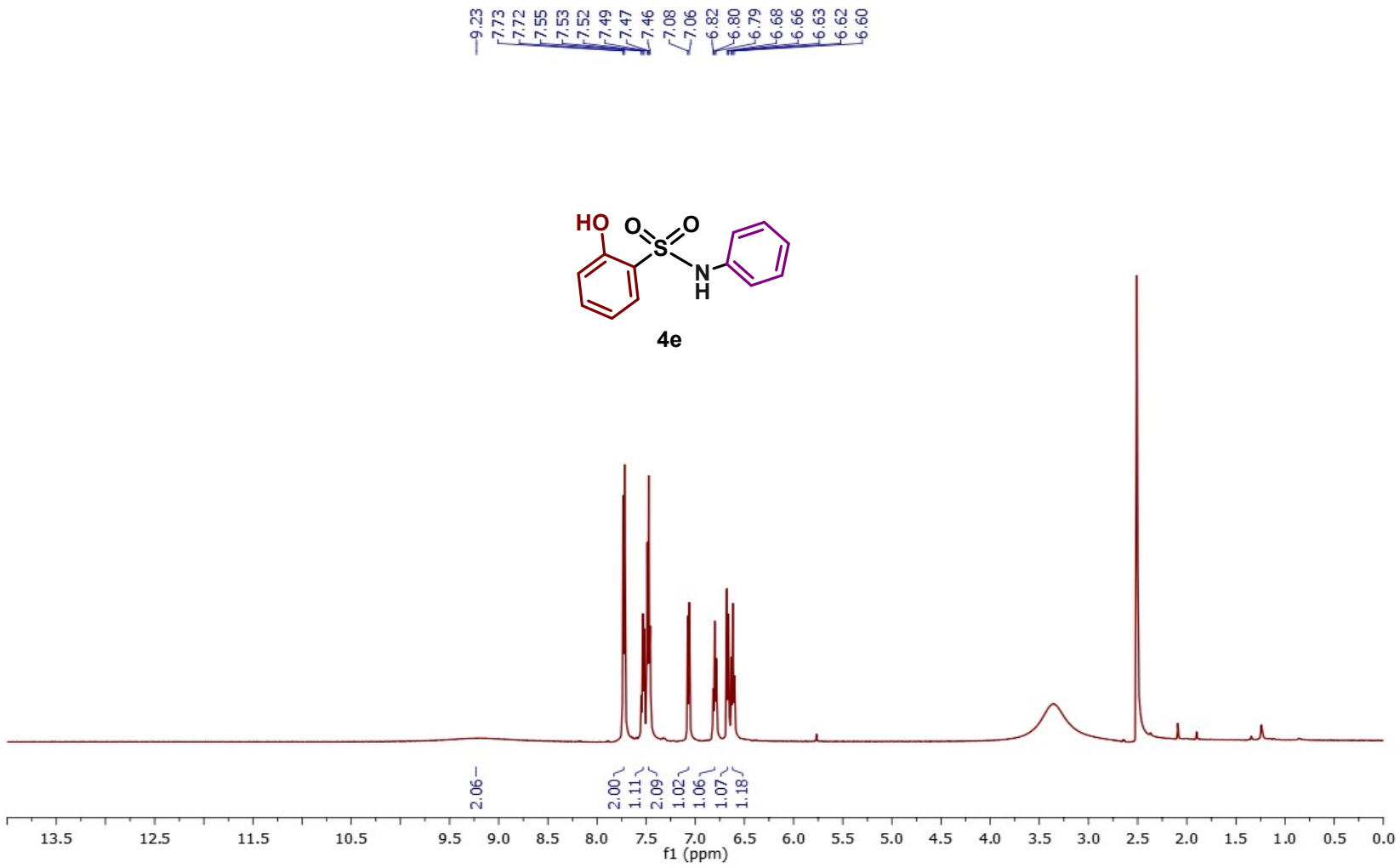

142.48
139.09
138.11
134.30
132.25
129.60
123.99
119.55
22.91
20.83
Me
O
O
S
N
H
Me
Me
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

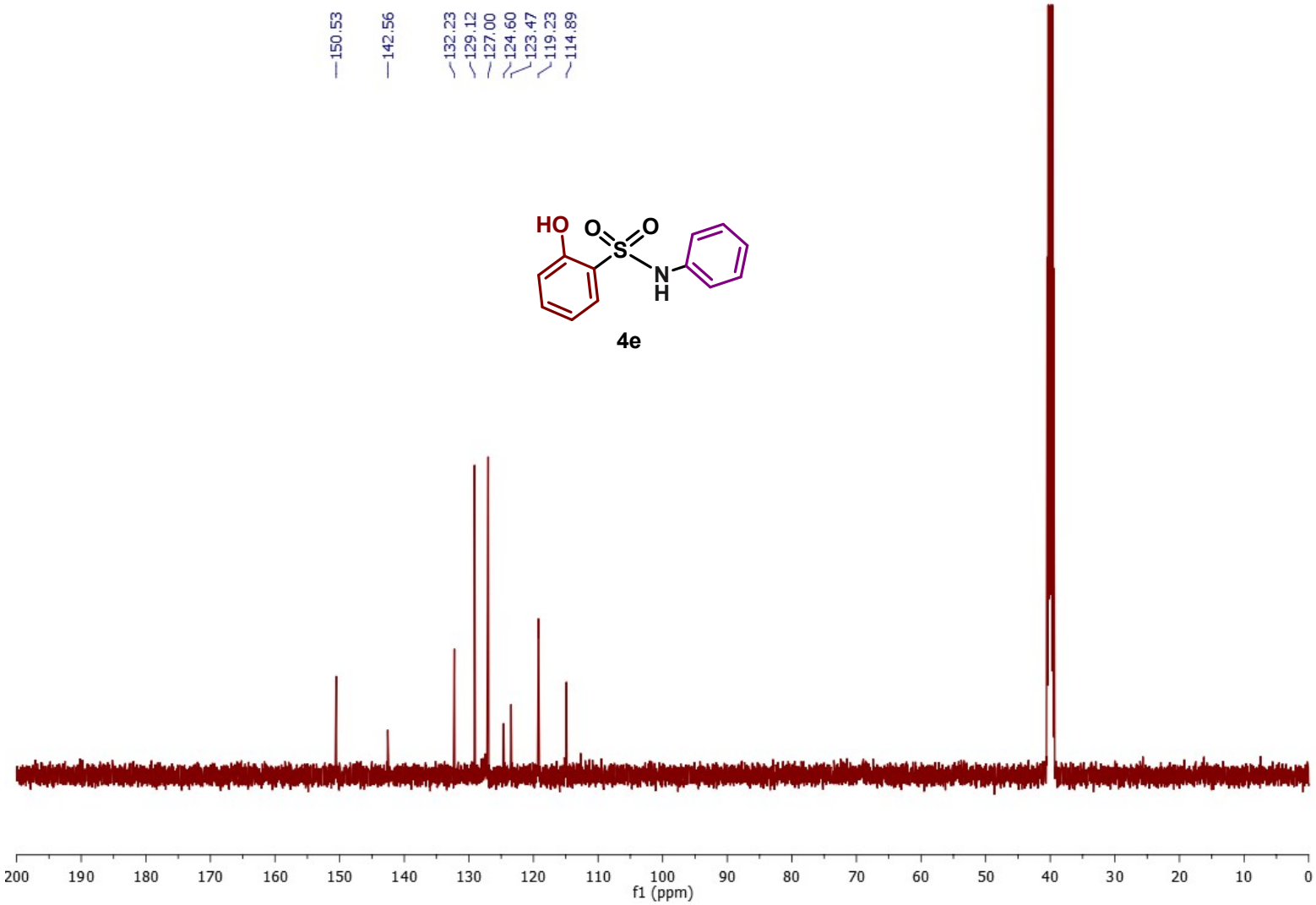
**4c**


9.92
7.69
7.68
7.67
7.61
7.60
7.59
7.58
7.58
7.55
7.53
7.52
6.98
6.96
6.81
6.79
3.66
O
O
S
N
H
MeO
1.00
1.98
1.05
2.06
2.09
2.08
3.10
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

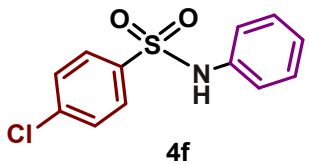
**4d**


157.00
139.91
133.17
130.52
129.59
127.13
123.93
114.74
55.59
O
O
S
N
H
MeO
4d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

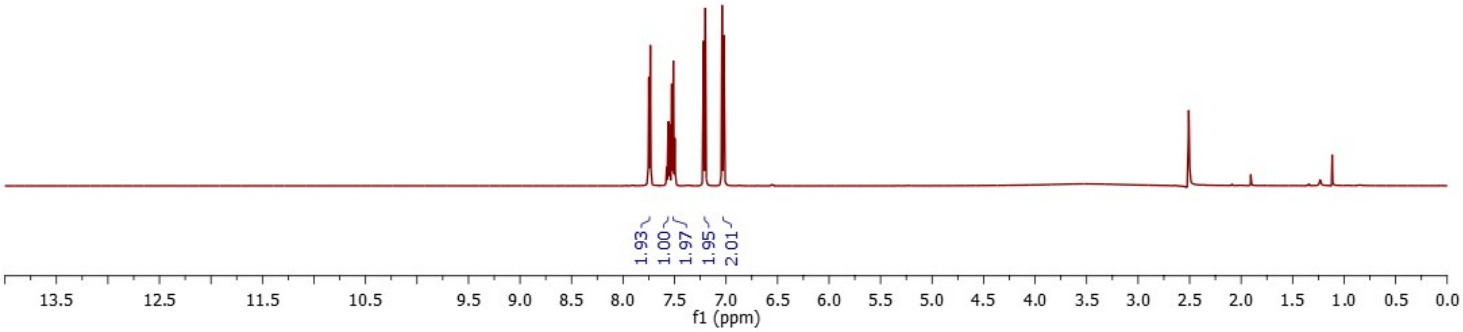

9.23
7.73
7.72
7.55
7.53
7.52
7.49
7.47
7.46
7.08
7.06
6.82
6.80
6.79
6.68
6.66
6.63
6.62
6.60
HO
O
S
O
N
H
4e
2.06
2.00
1.11
2.09
1.02
1.06
1.07
1.18
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

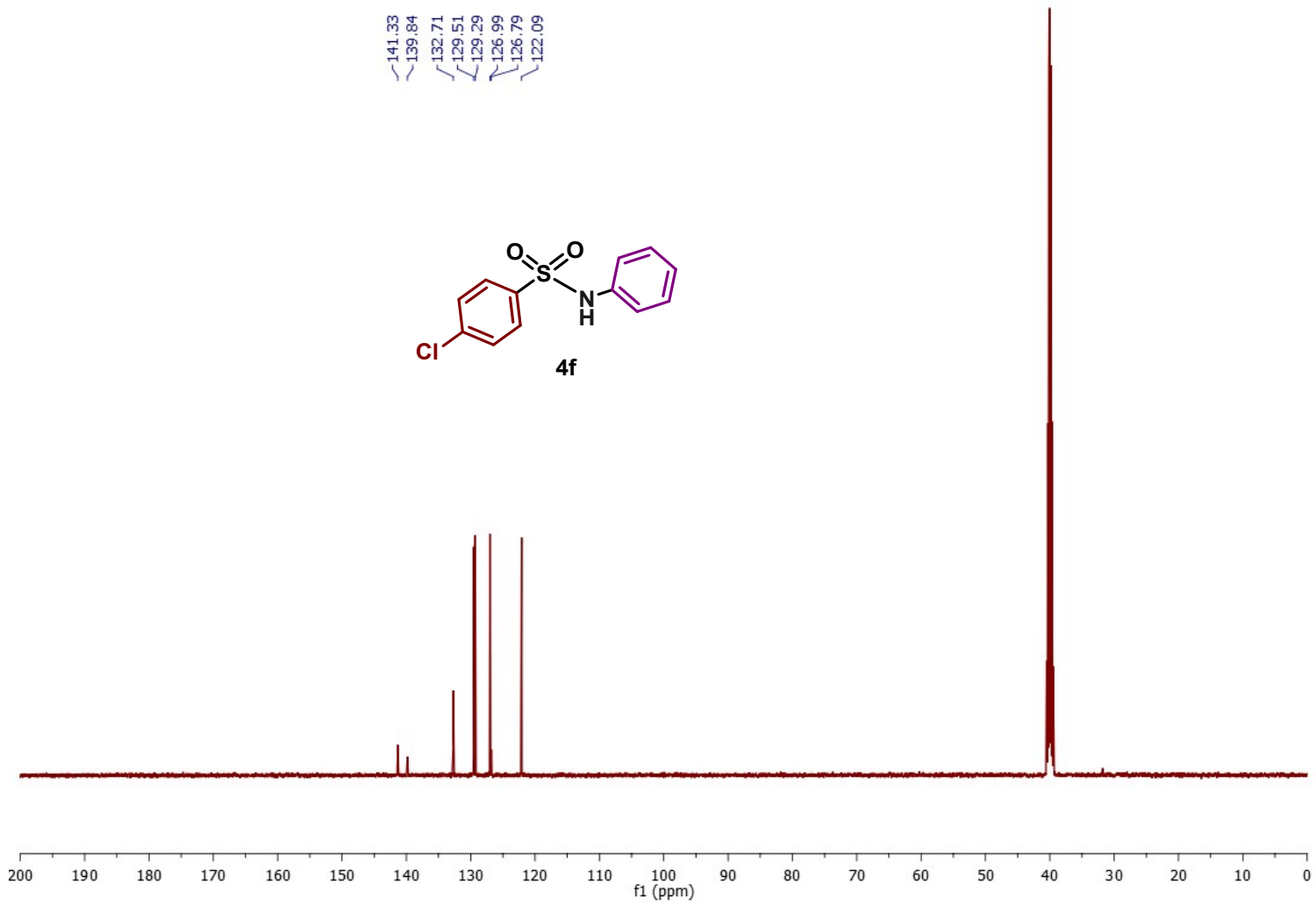

150.53
142.56
132.23
129.12
127.00
124.60
123.47
119.23
114.89
HO
O
O
S
N
H
4e
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.75
7.75
7.73
7.73
7.56
7.55
7.55
7.54
7.53
7.51
7.51
7.50
7.22
7.21
7.20
7.20
7.04
7.03
7.03
7.02

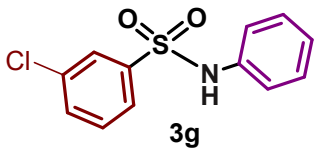

O
O
S
N
H
Cl

**4f**

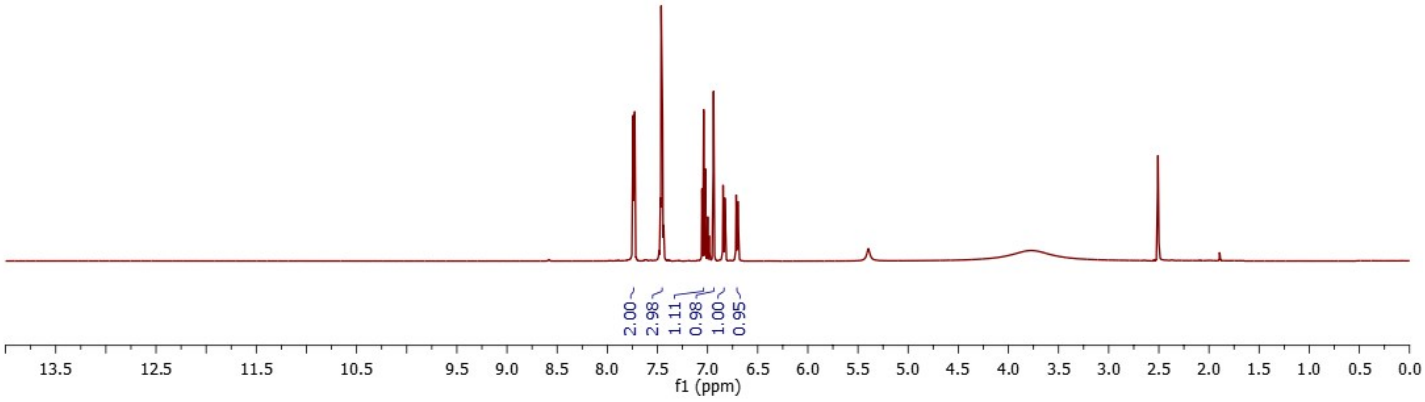

1.93
1.00
1.97
1.95
2.01
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

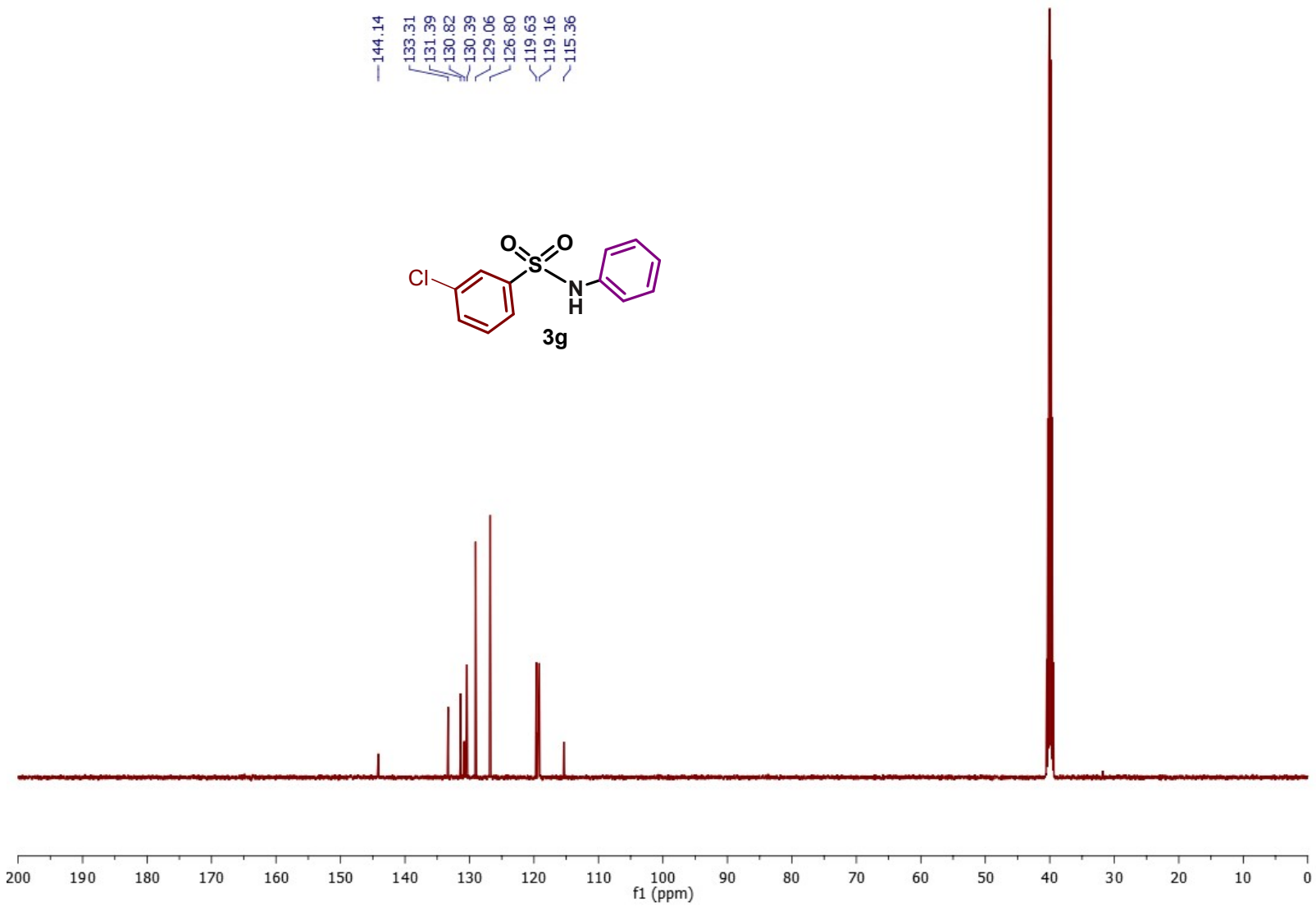

141.33
139.84
132.71
129.51
129.29
126.99
126.79
122.09
O
O
S
N
H
Cl
4f
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.75
7.73
7.46
7.45
7.06
7.04
7.02
6.94
6.84
6.83
6.72
6.70

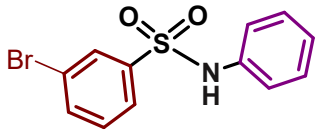

O
O
S
N
H
Cl

**3g**

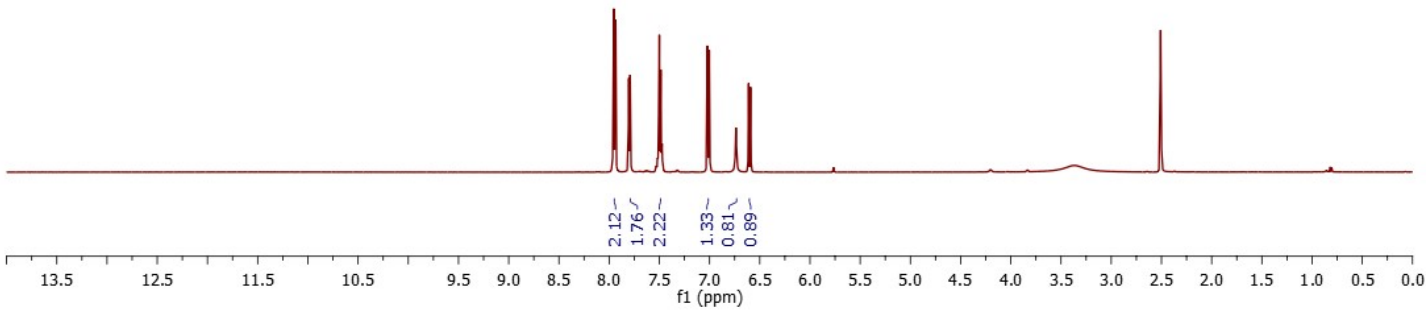

2.00
2.98
1.11
0.98
1.00
0.95
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

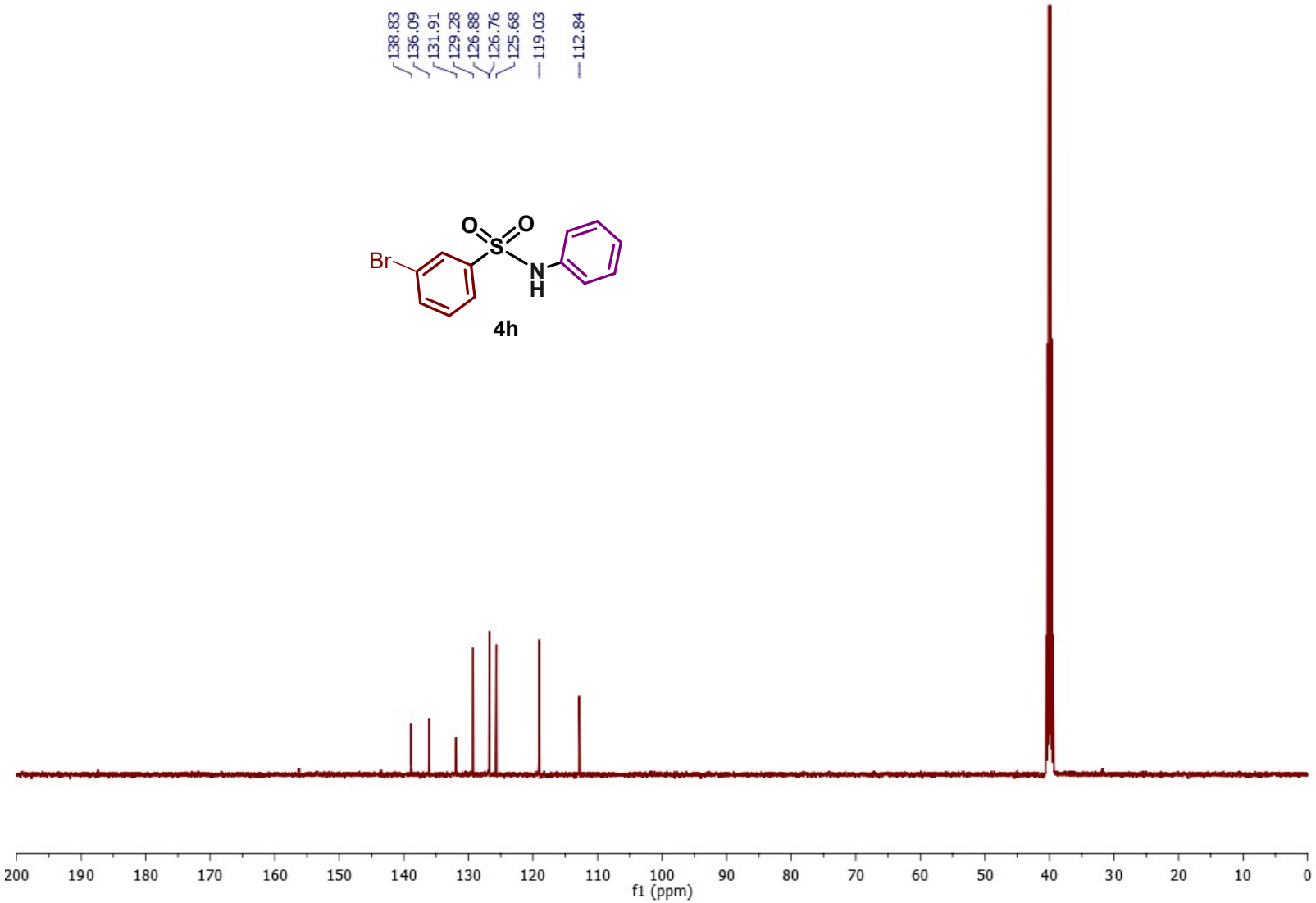

144.14
133.31
131.39
130.82
130.39
129.06
126.80
119.63
119.16
115.36
O
O
S
N
H
Cl
3g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.95
7.94
7.94
7.80
7.79
7.52
7.50
7.49
7.02
7.00
6.74
6.61
6.59

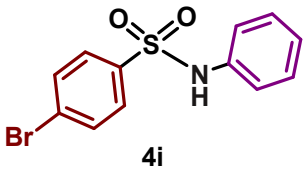

O
O
S
N
H
Br

**4h**

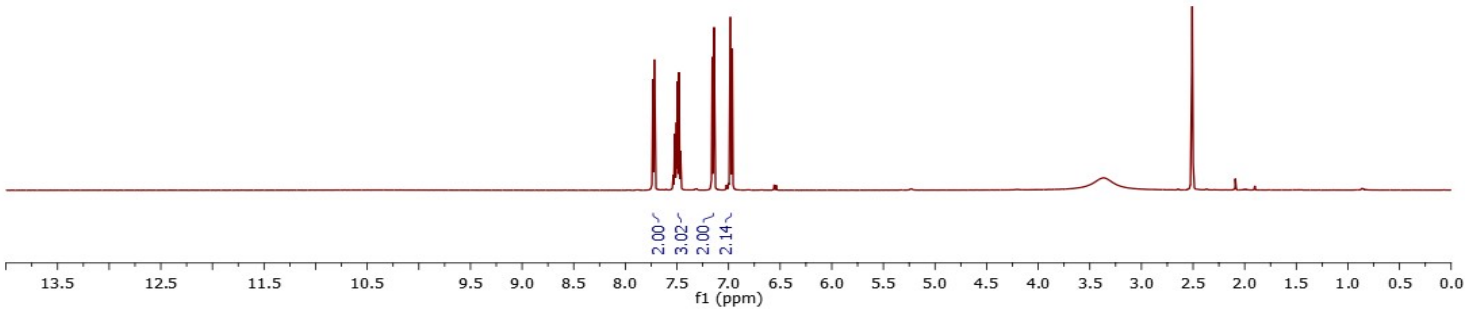

2.12
1.76
2.22
1.33
0.81
0.89
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

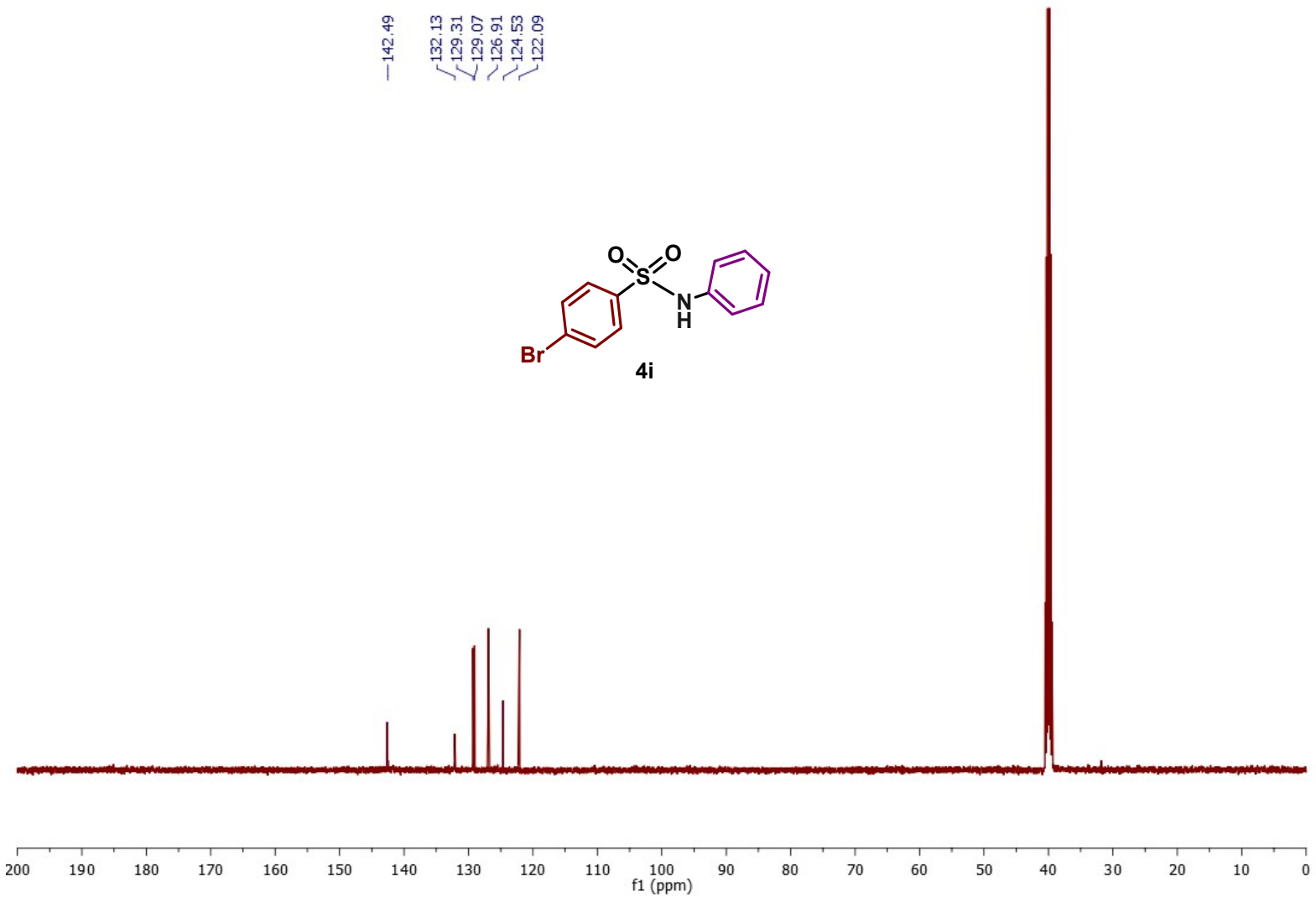

138.83
136.09
131.91
129.28
126.88
126.76
125.68
119.03
112.84
Br
O
O
S
N
H
4h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.73
7.72
7.72
7.52
7.49
7.48
7.46
7.16
7.14
6.98
6.96

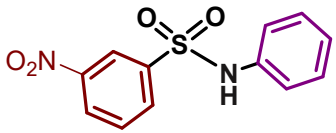

O
O
S
N
H
Br

4i

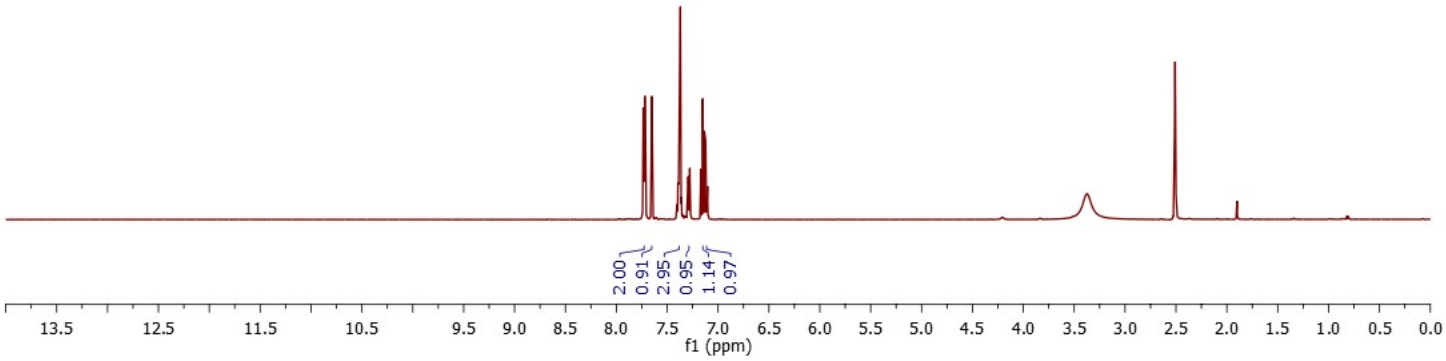

2.00
3.02
2.00
2.14
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

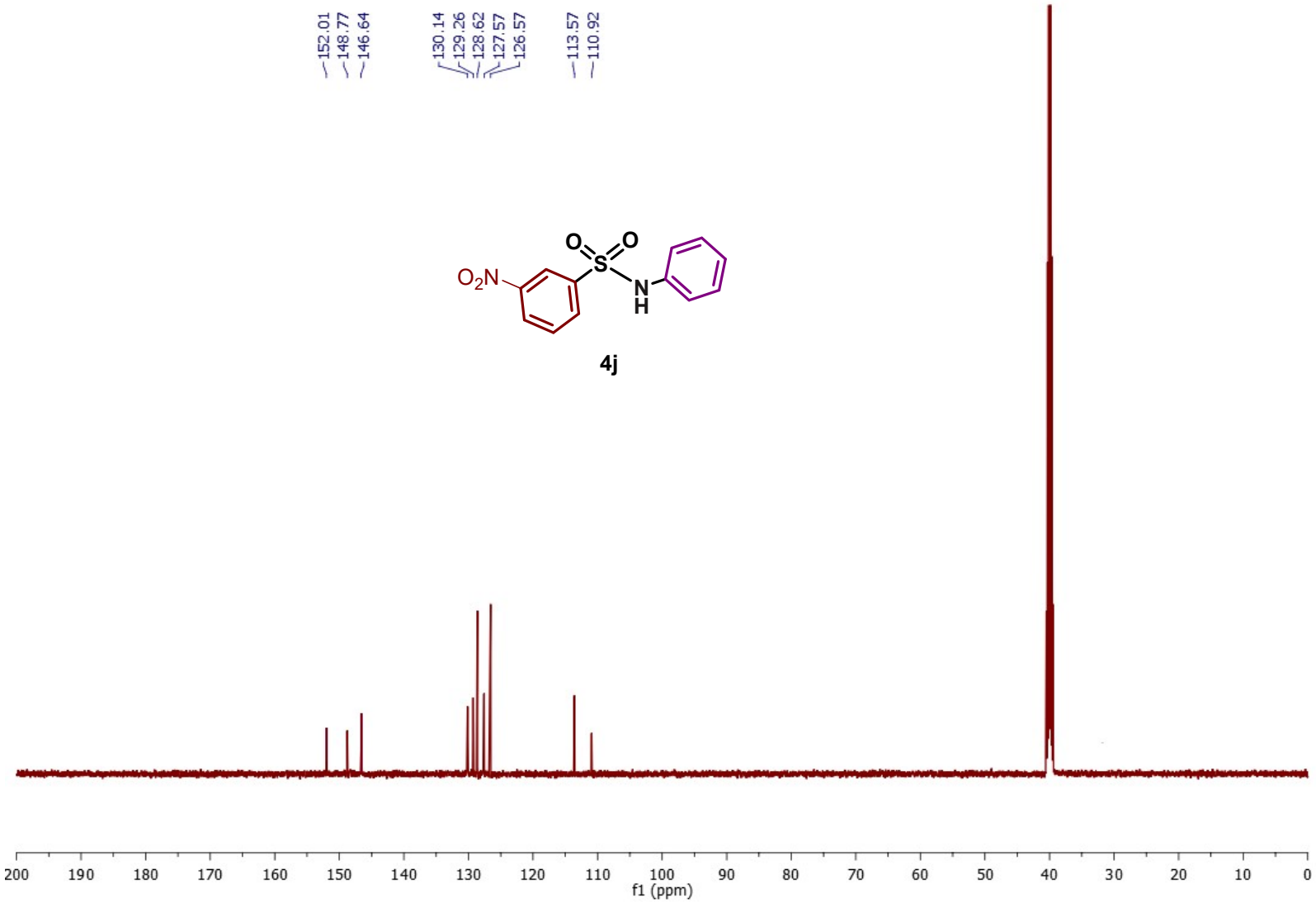

142.49
132.13
129.31
129.07
126.91
124.53
122.09
O
O
S
N
H
Br
4i
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.73
7.72
7.72
7.65
7.65
7.65
7.37
7.37
7.29
7.28
7.17
7.15
7.14
7.12
7.11

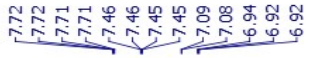

O
O
S
O2N
N
H

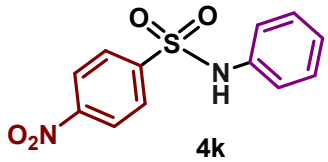
**4j**


2.00
0.91
2.95
0.95
1.14
0.97
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

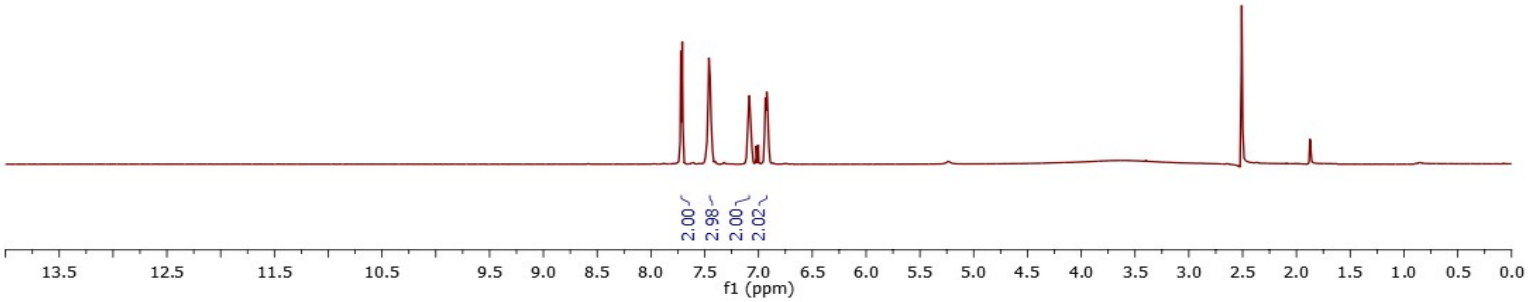

152.01
148.77
146.64
130.14
129.26
128.62
127.57
126.57
113.57
110.92
O
O
S
N
H
O2N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

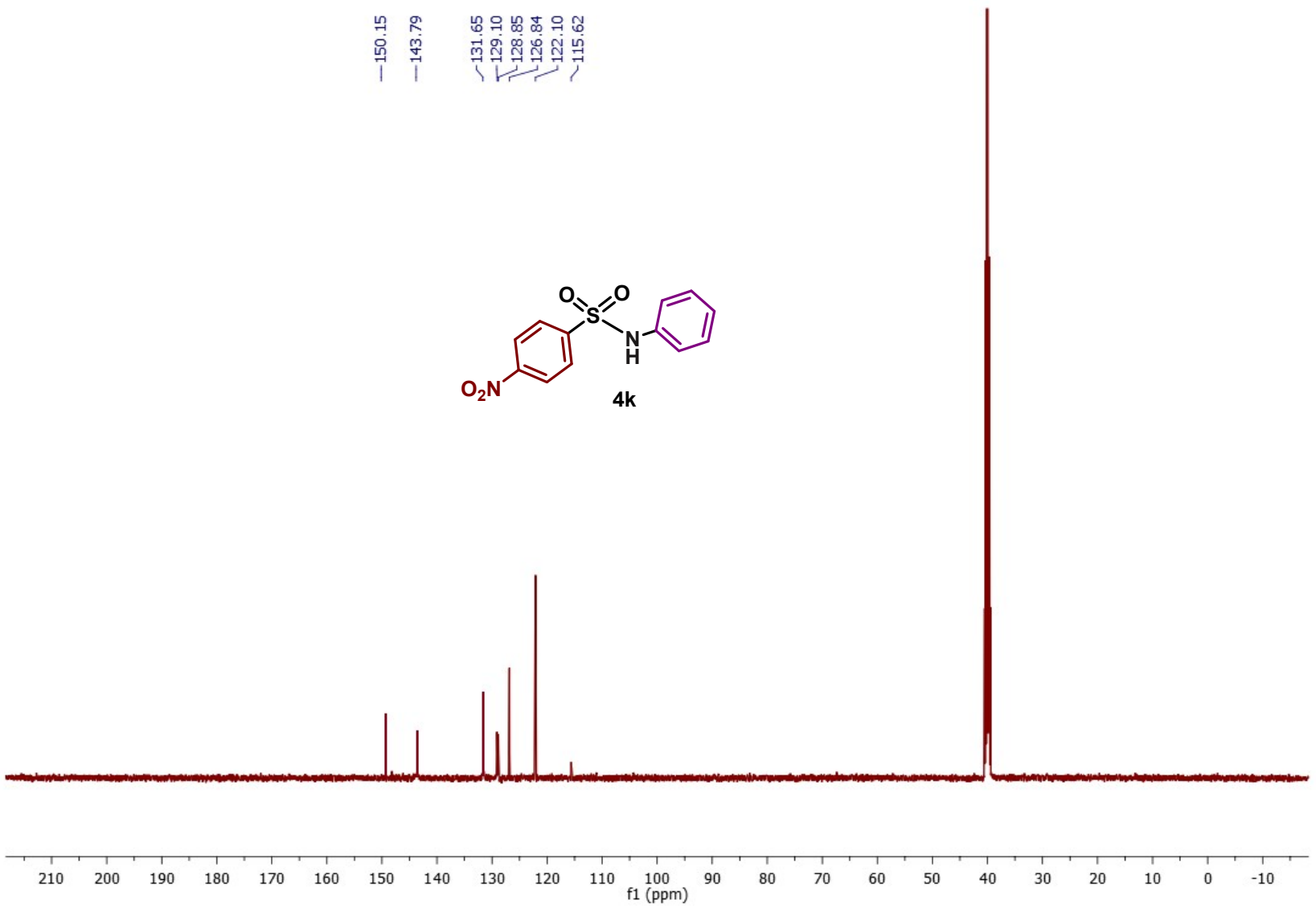
**4j**


7.72
7.72
7.71
7.71
7.46
7.46
7.45
7.45
7.09
7.08
6.94
6.92
6.92

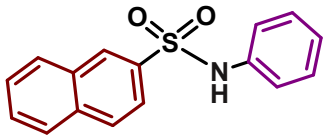

O
O
S
N
H
O2N

**4k**

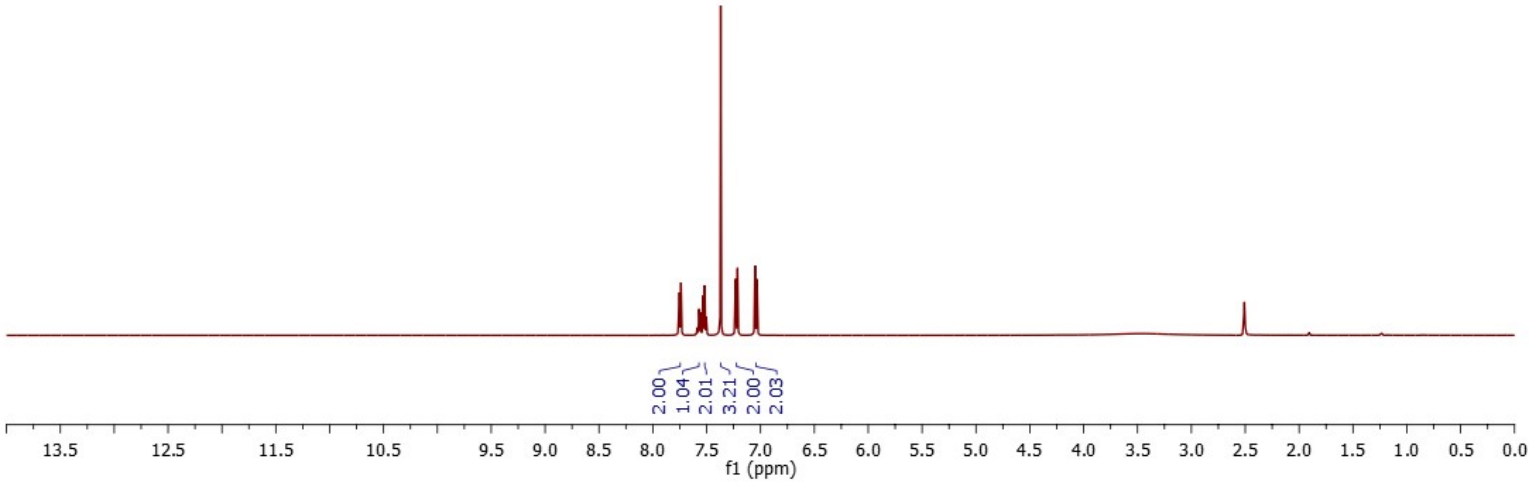

2.00
2.98
2.00
2.02
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

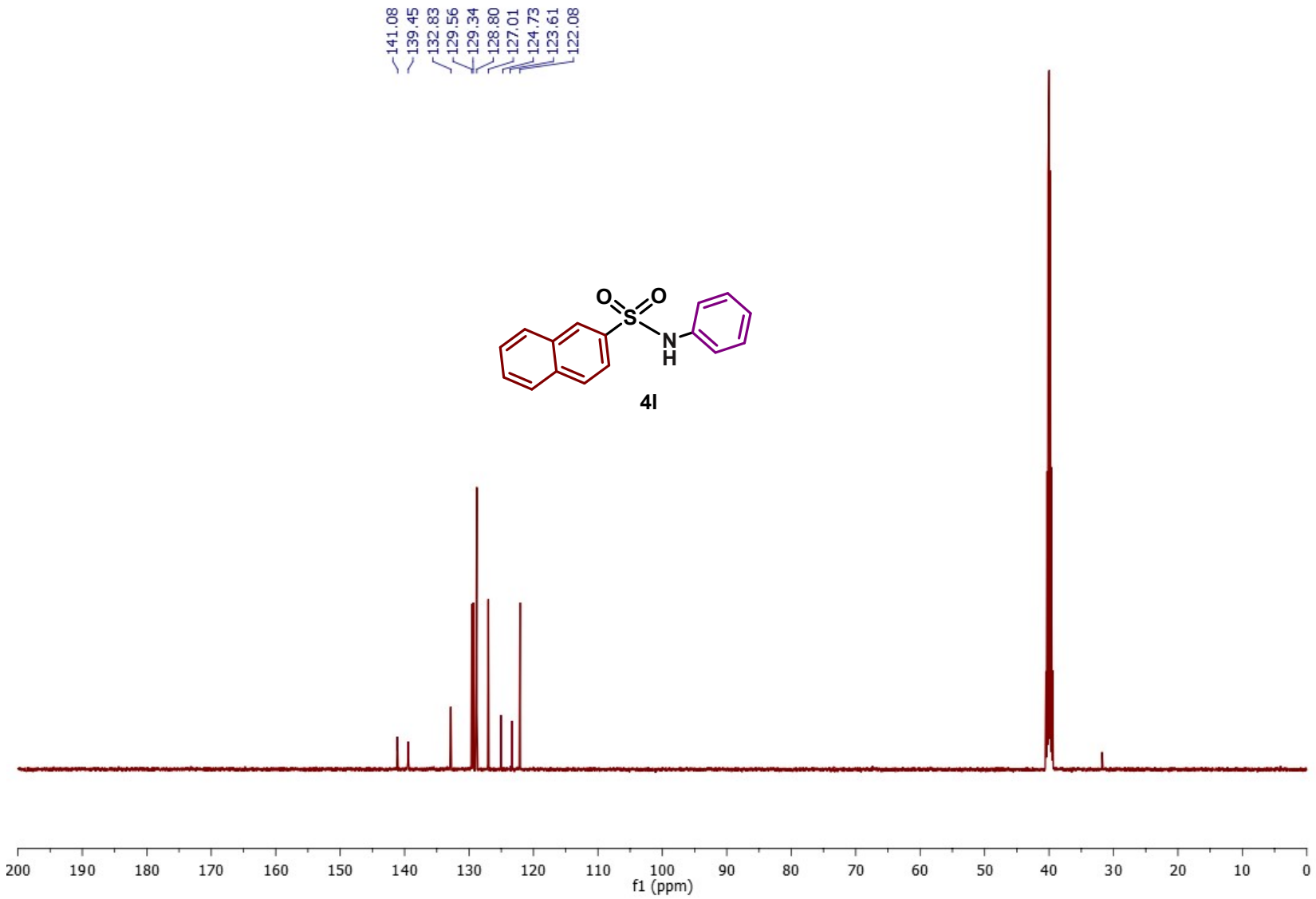

150.15
143.79
131.65
129.10
128.85
126.84
122.10
115.62
O
O
S
N
H
O2N
4k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

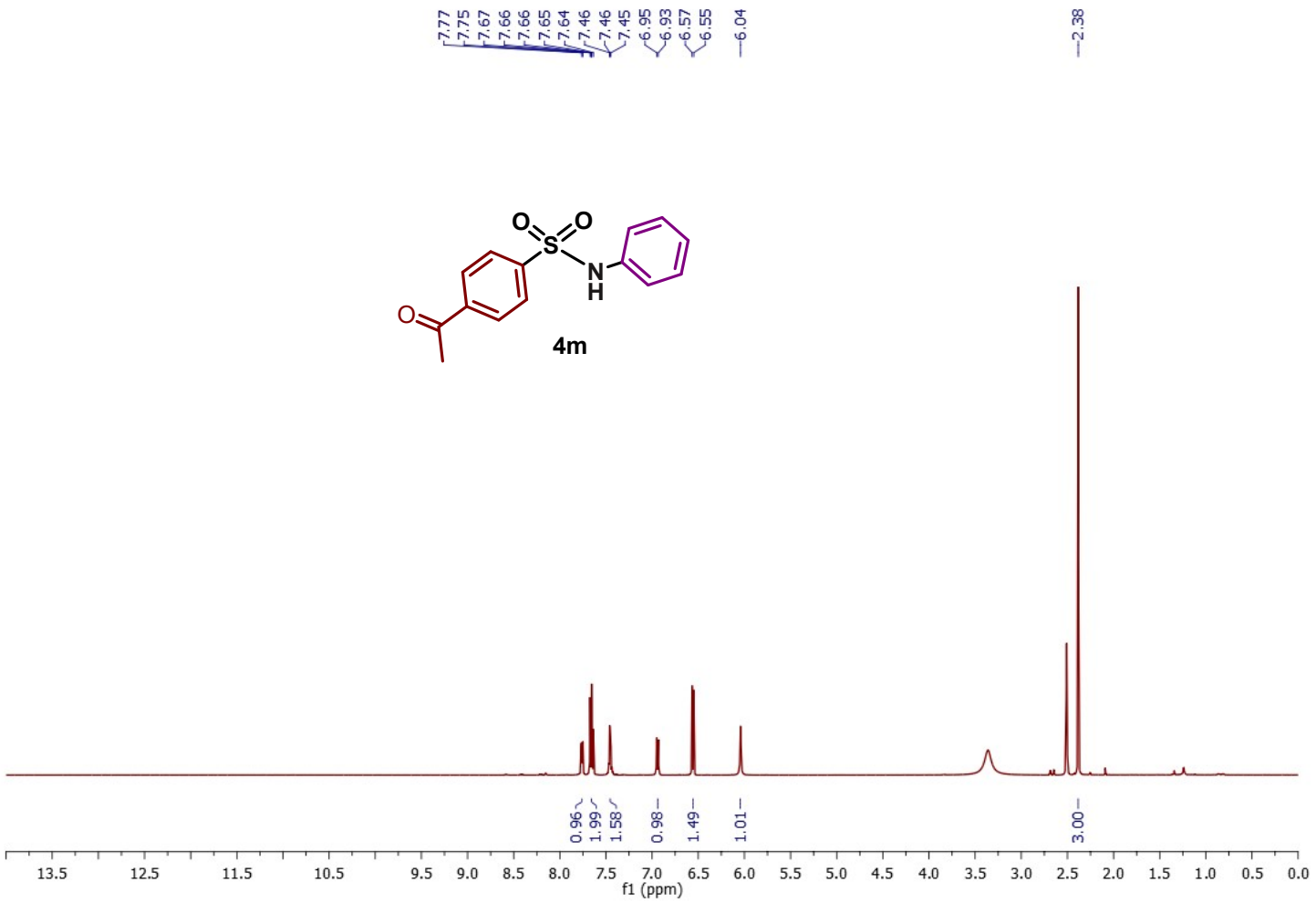

7.75
7.74
7.74
7.57
7.56
7.56
7.53
7.52
7.52
7.37
7.23
7.21
7.05
7.03

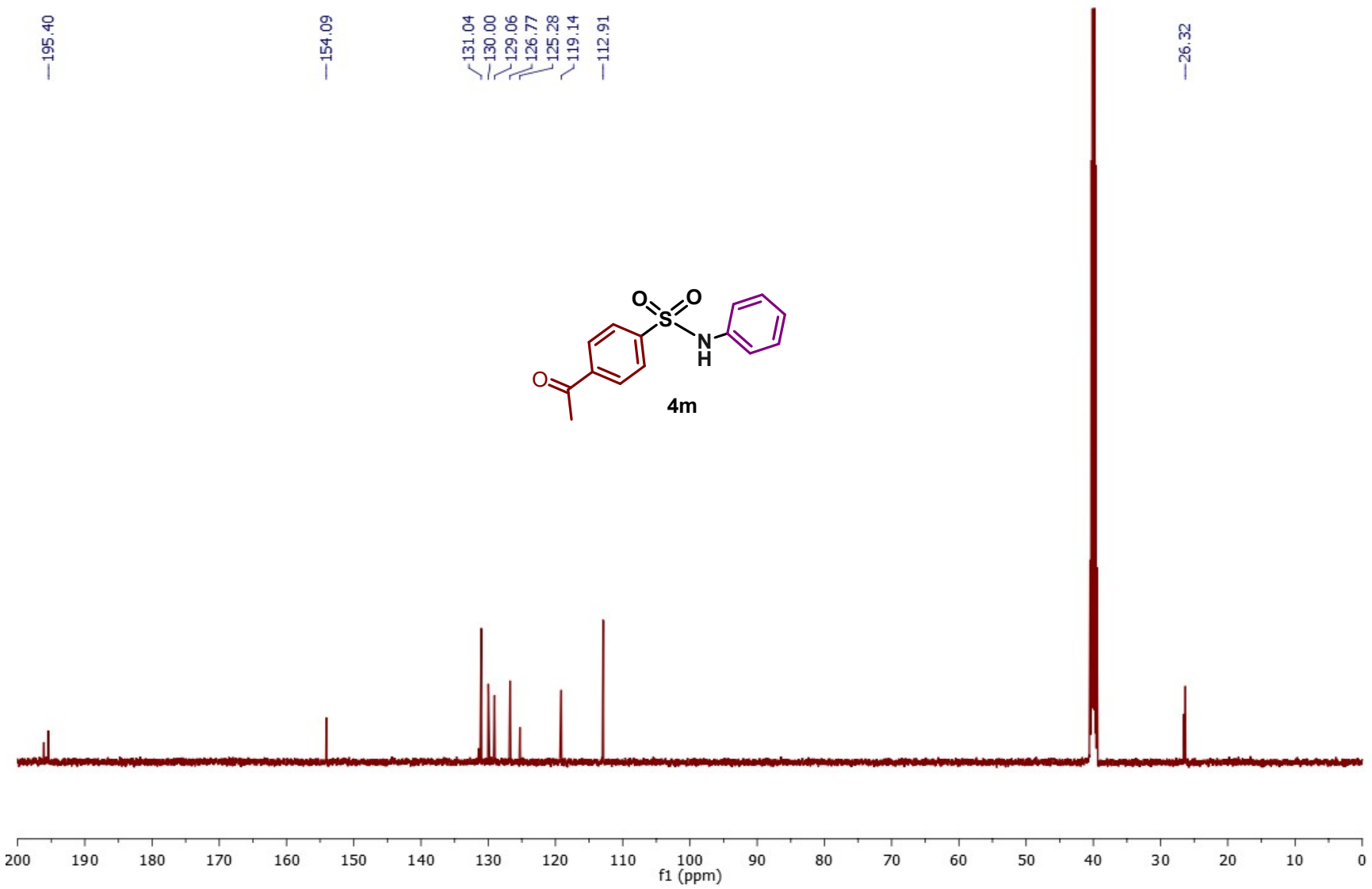

O
O
S
N
H

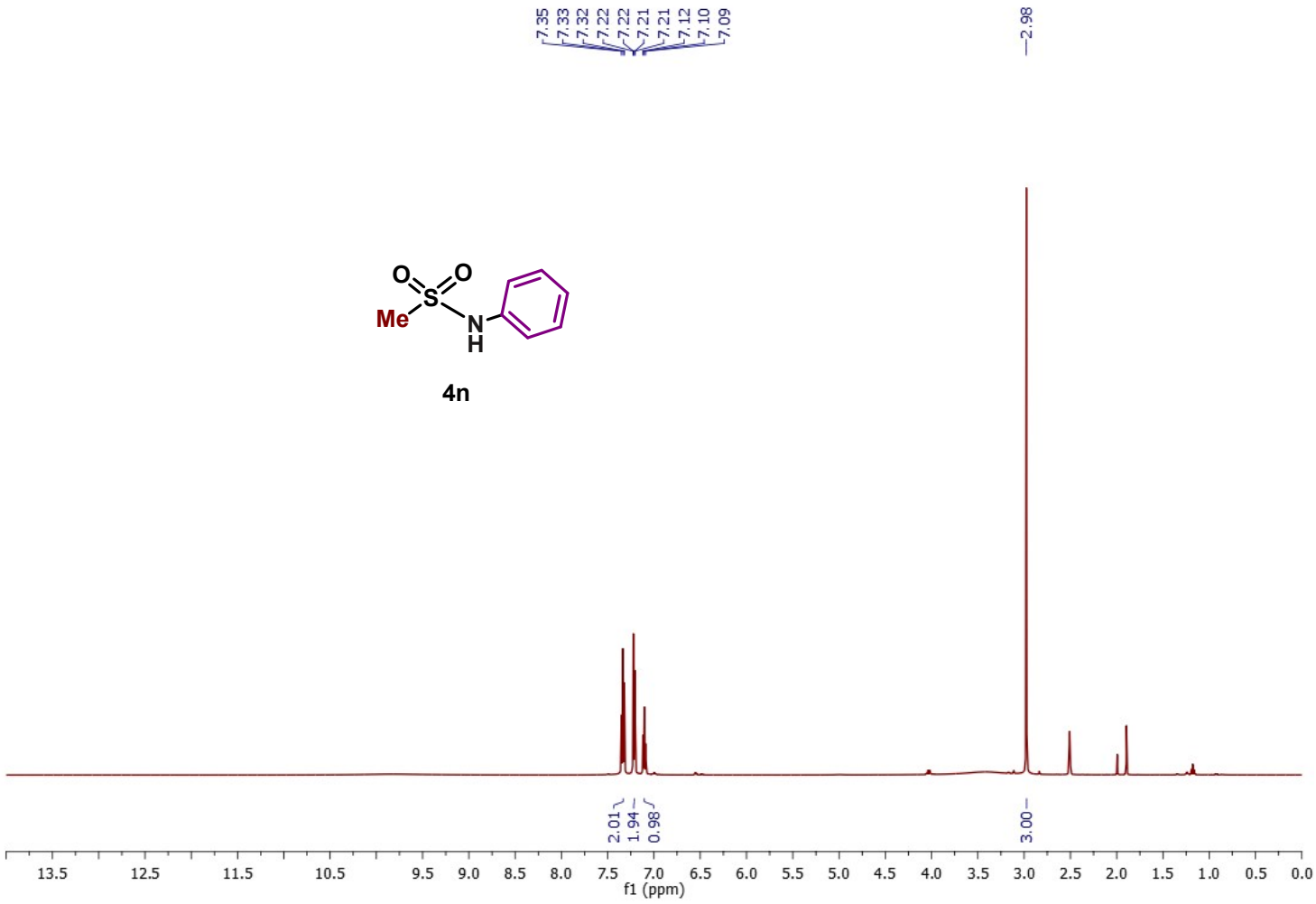
**4l**


2.00
1.04
2.01
3.21
2.00
2.03
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

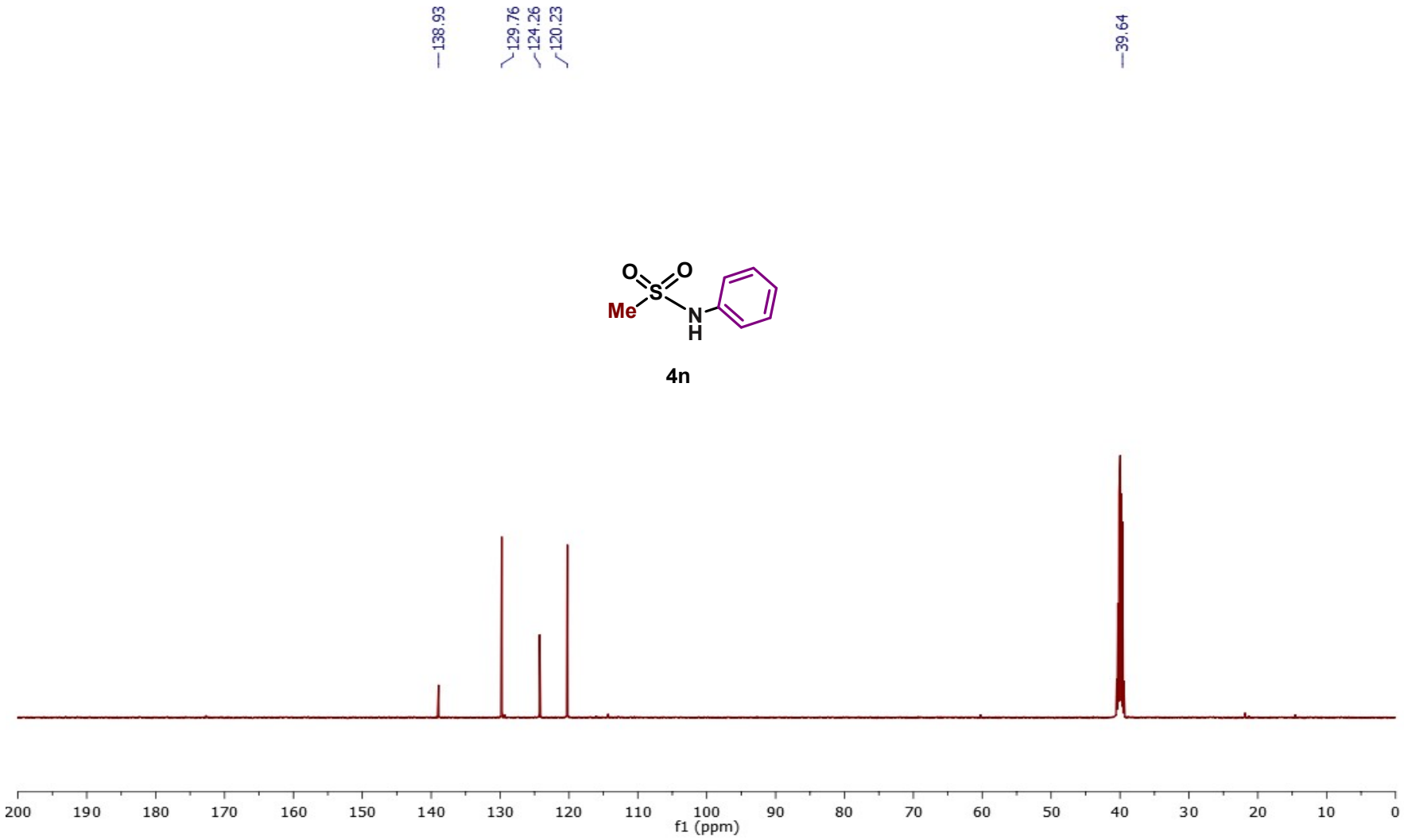

141.08
139.45
132.83
129.56
129.34
128.80
127.01
124.73
123.61
122.08
O
O
S
N
H
4l
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.77
7.75
7.67
7.66
7.66
7.65
7.64
7.46
7.46
7.45
6.95
6.93
6.57
6.55
6.04
2.38
O
O
S
N
H
O
4m
0.96
1.99
1.58
0.98
1.49
1.01
3.00
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


195.40
154.09
131.04
130.00
129.06
126.77
125.28
119.14
112.91
26.32
O
O
S
N
H
O
4m
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

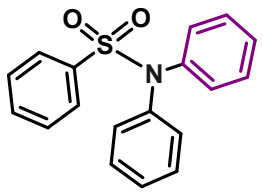

7.35
7.33
7.32
7.22
7.22
7.21
7.21
7.12
7.10
7.09
2.98
O
O
S
Me
N
H
2.01
1.94
0.98
3.00
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

**4n**

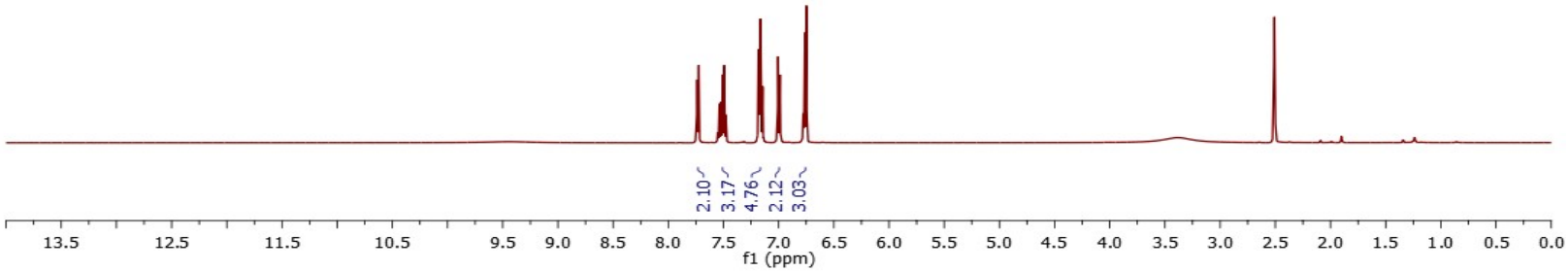

138.93
129.76
124.26
120.23
39.64
O
O
S
Me
N
H
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

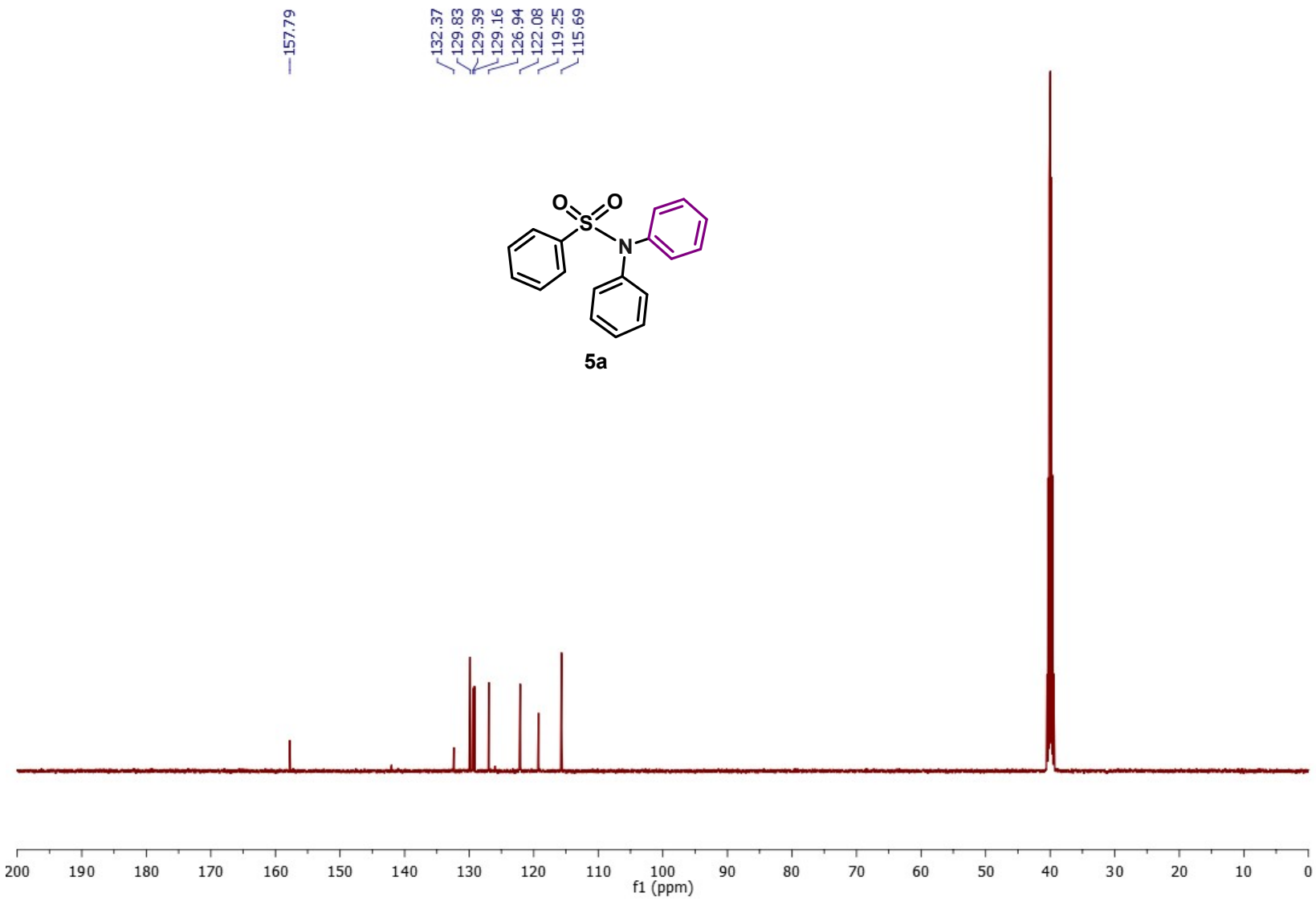
**4n**


7.74
7.72
7.54
7.53
7.51
7.49
7.48
7.18
7.18
7.17
7.15
7.01
6.99
6.78
6.76
6.75

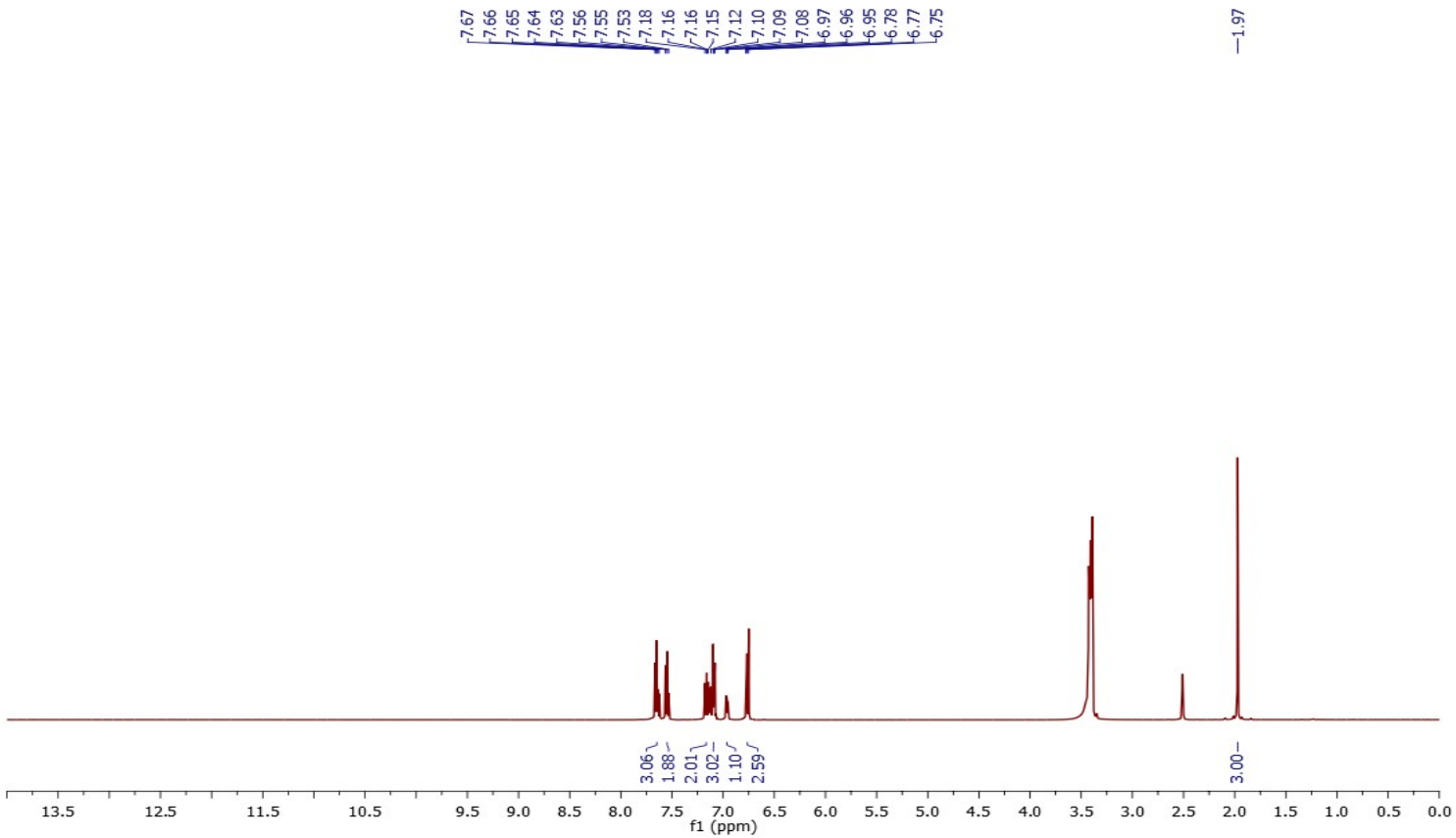

O
O
S
N

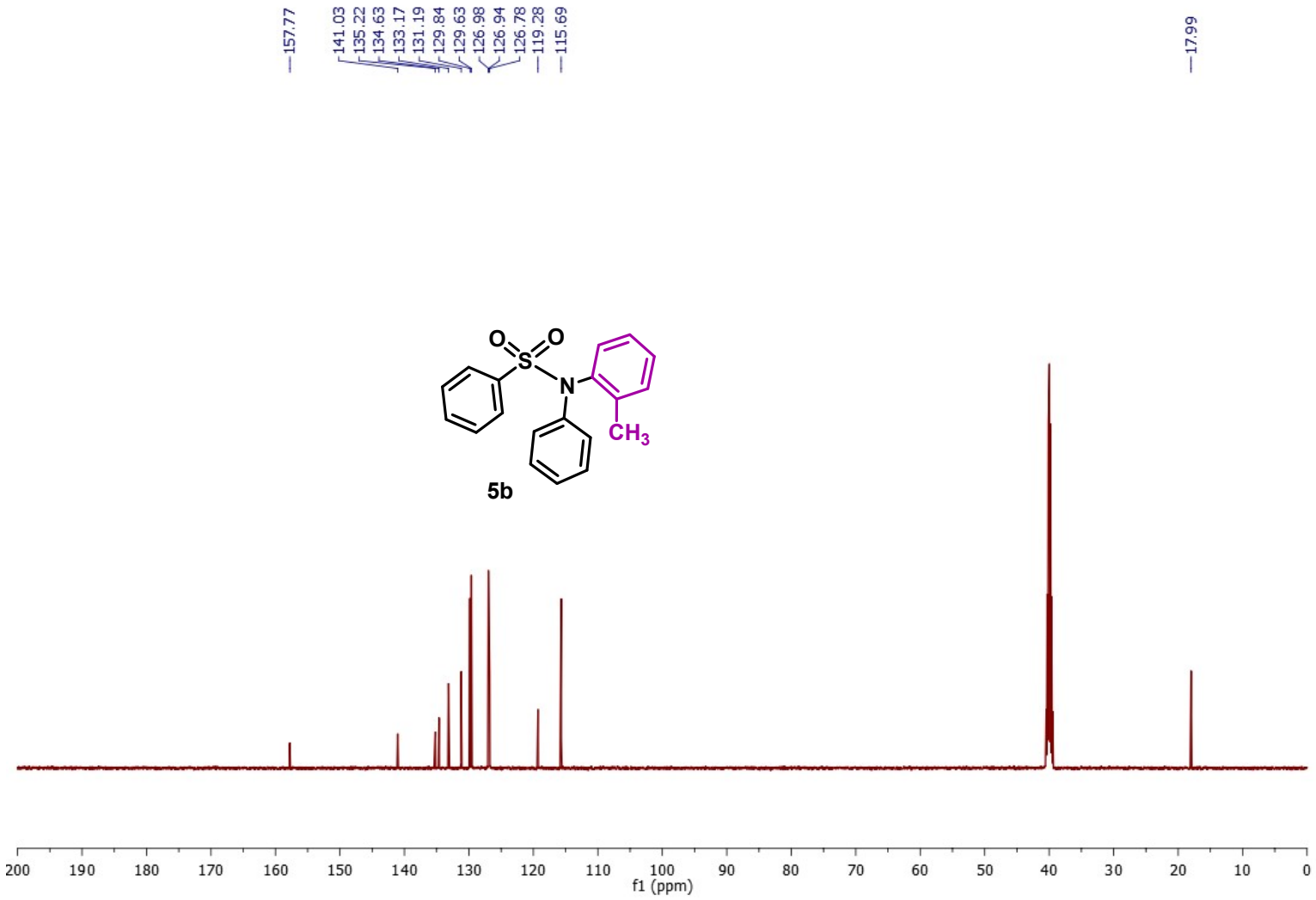
**5a**

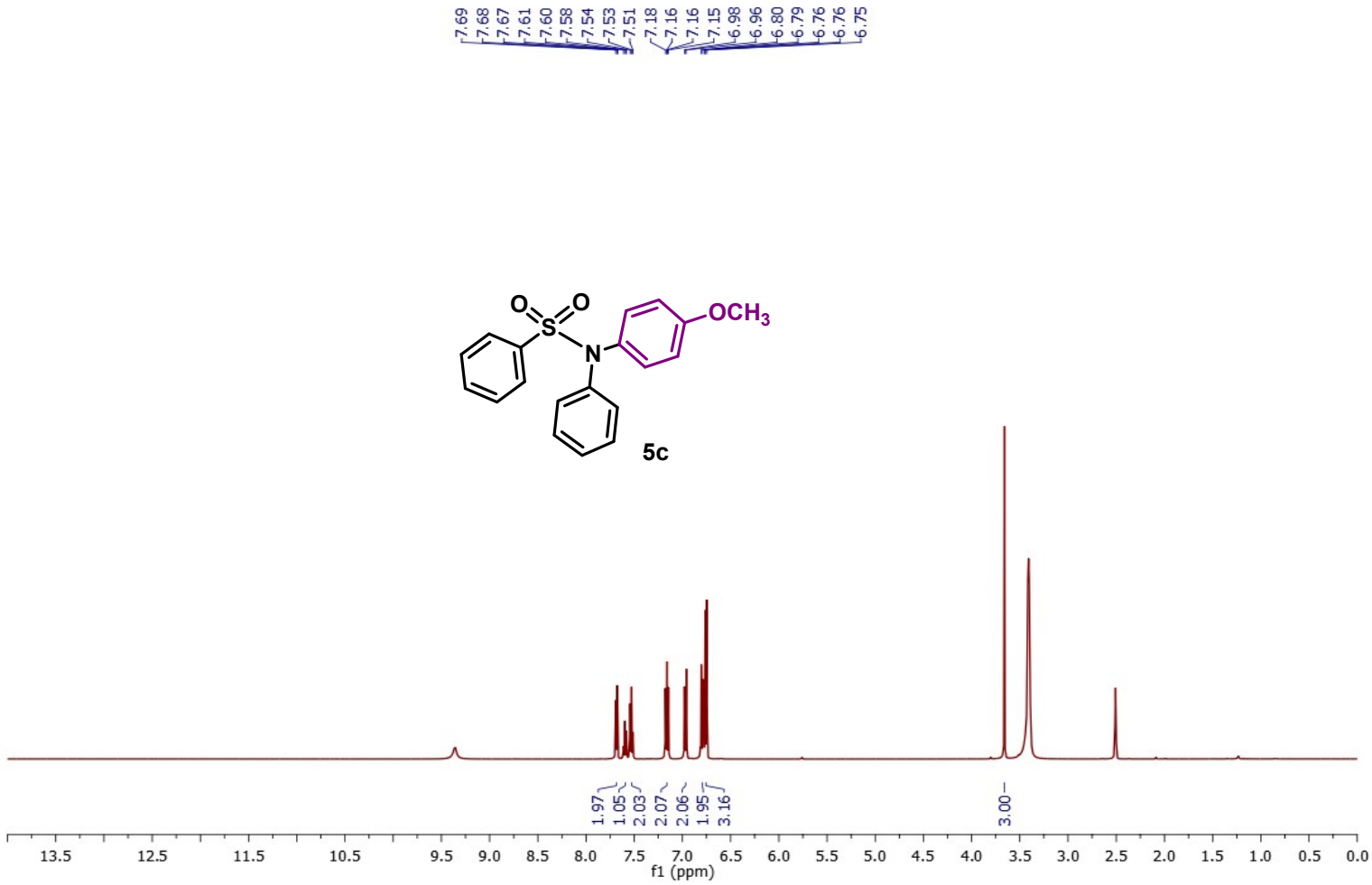

2.10
3.17
4.76
2.12
3.03
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

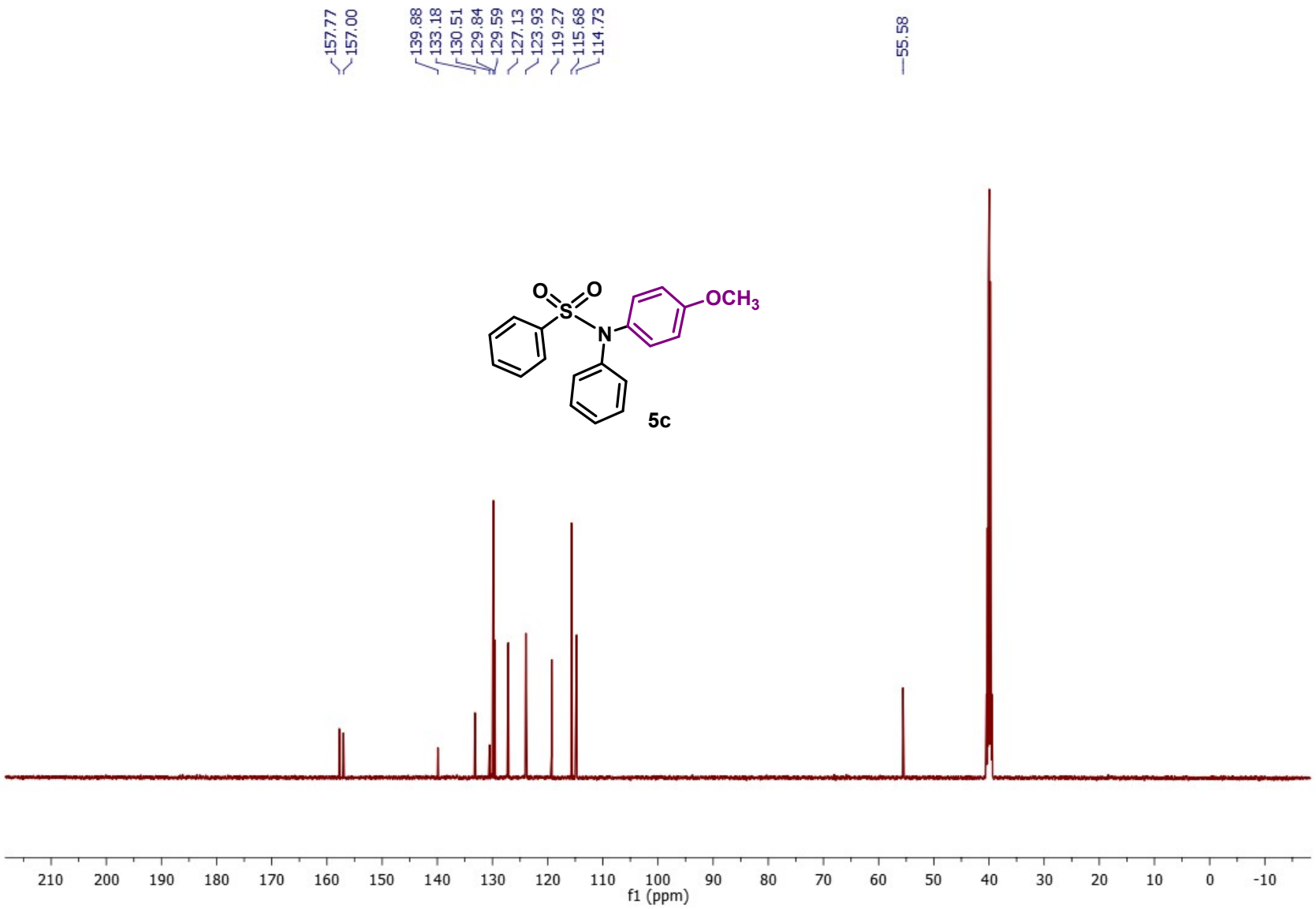

157.79
132.37
129.83
129.39
129.16
126.94
122.08
119.25
115.69
O
O
S
N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

**5a**

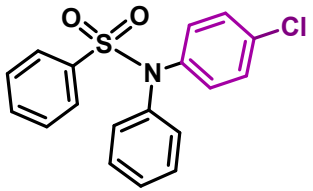

7.67
7.66
7.65
7.64
7.63
7.56
7.55
7.53
7.18
7.16
7.16
7.15
7.12
7.10
7.09
7.08
6.97
6.96
6.95
6.78
6.77
6.75
1.97
3.06
1.88
2.01
3.02
1.10
2.59
3.00
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


157.77
141.03
135.22
134.63
133.17
131.19
129.84
129.63
126.98
126.94
126.78
119.28
115.69
17.99
O
O
S
N
CH3
5b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

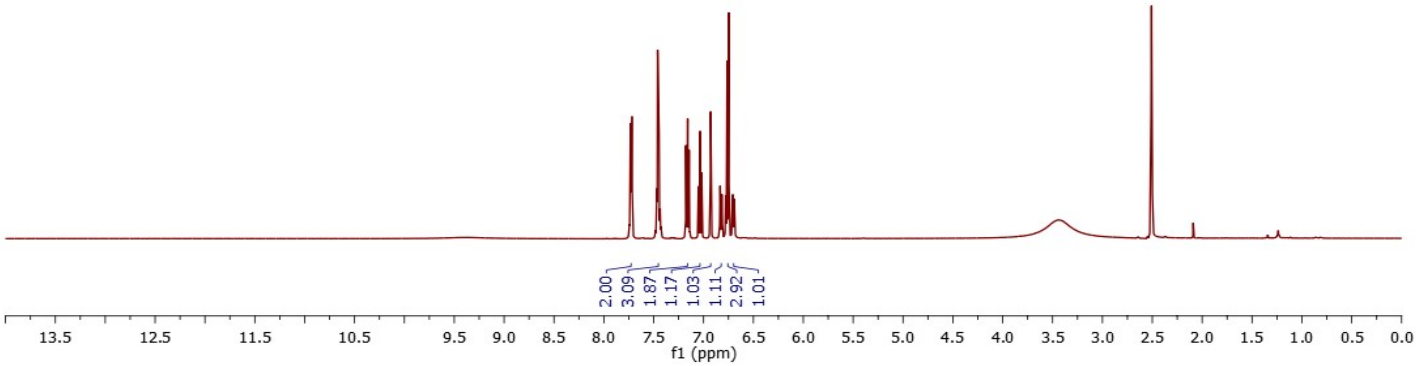

7.69
7.68
7.67
7.61
7.60
7.58
7.54
7.53
7.51
7.18
7.16
7.16
7.15
6.98
6.96
6.80
6.79
6.76
6.76
6.75
O
O
S
N
OCH3
5c
1.97
1.05
2.03
2.07
2.06
1.95
3.16
3.00
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

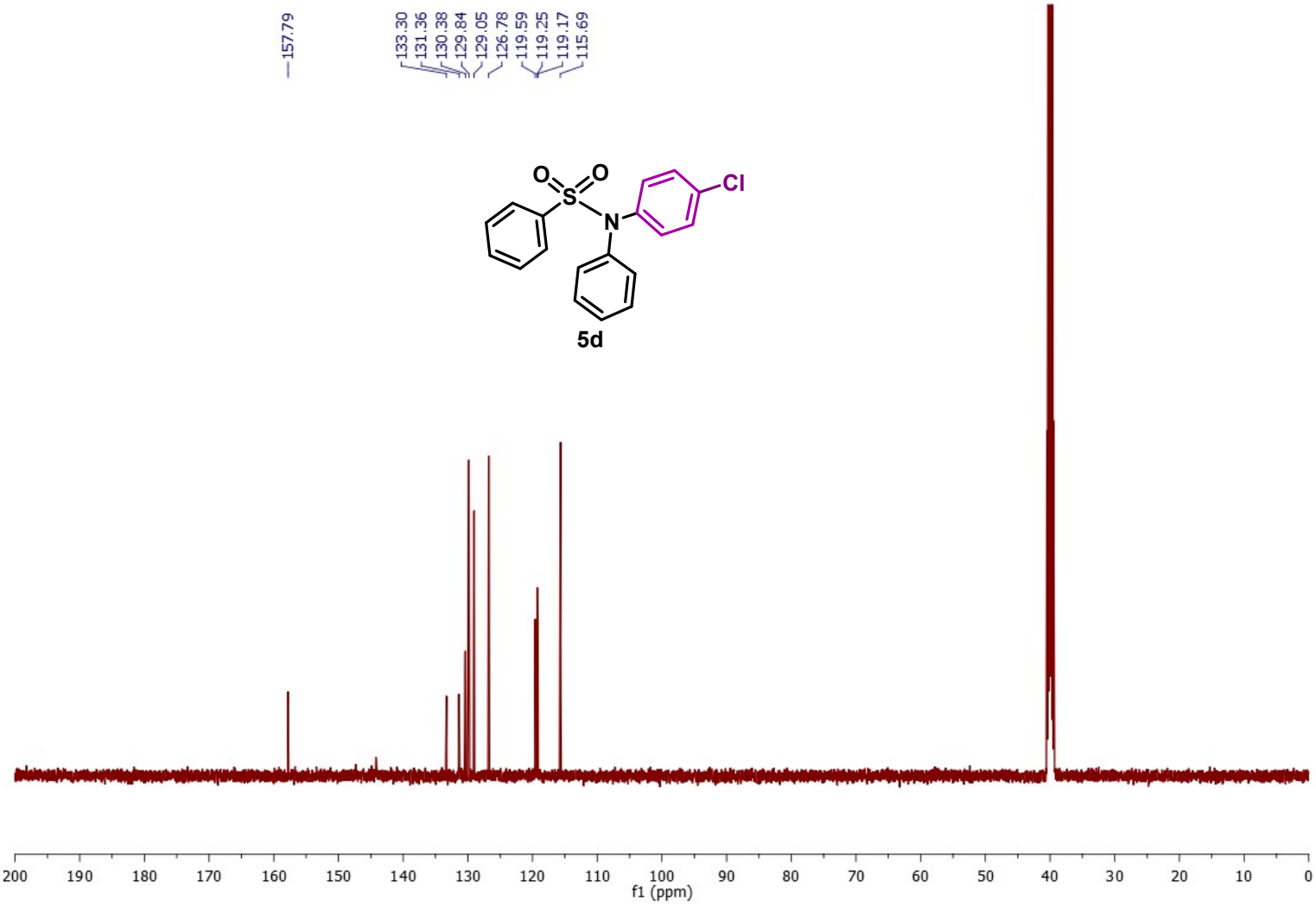

157.77
157.00
139.88
133.18
130.51
129.84
129.59
127.13
123.93
119.27
115.68
114.73
55.58
O
O
S
N
OCH3
5c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

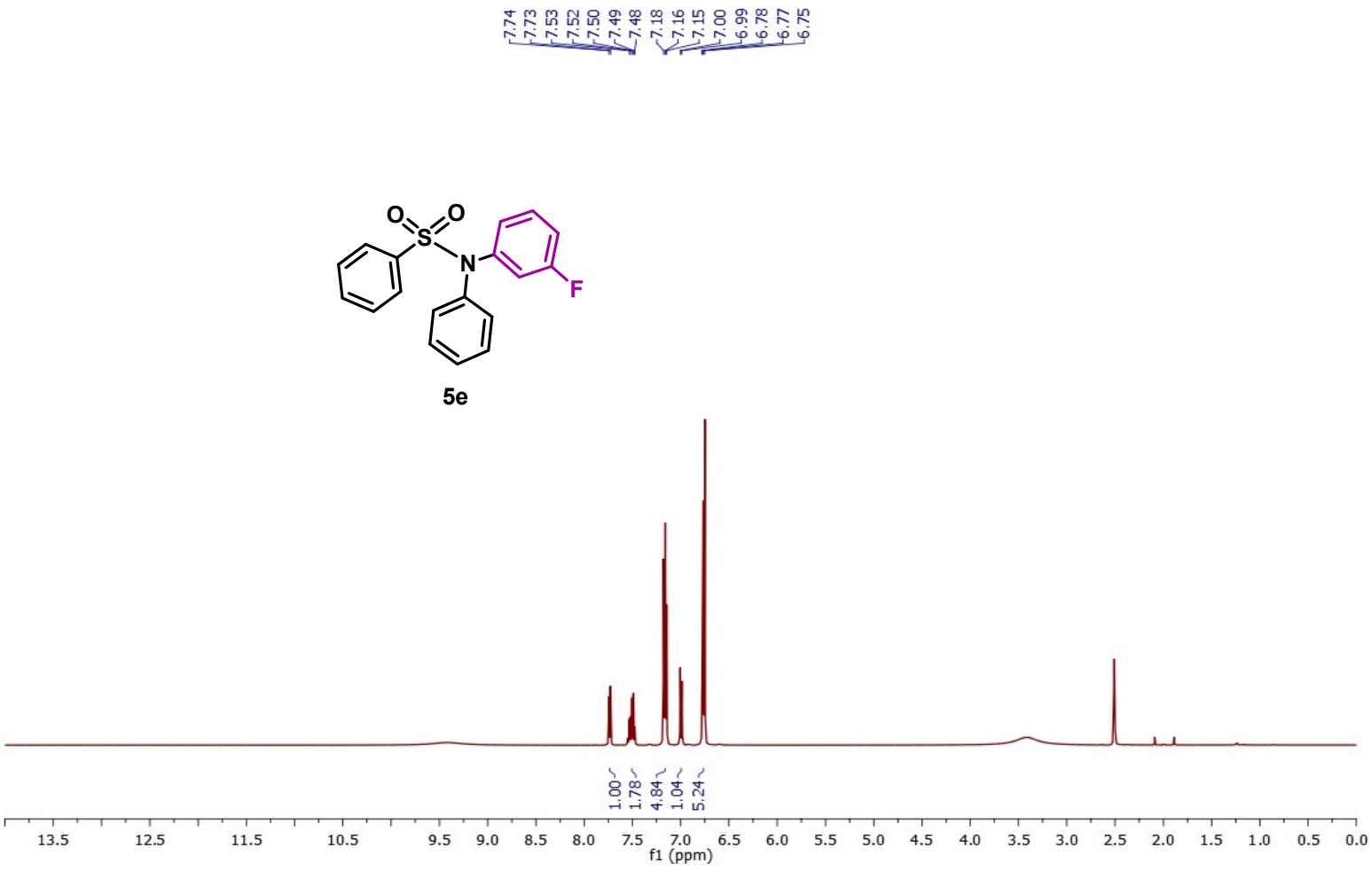

7.74
7.73
7.72
7.72
7.46
7.45
7.18
7.16
7.15
7.05
7.04
7.02
6.93
6.93
6.92
6.84
6.82
6.78
6.76
6.75
6.71
6.69


O
O
S
N
Cl

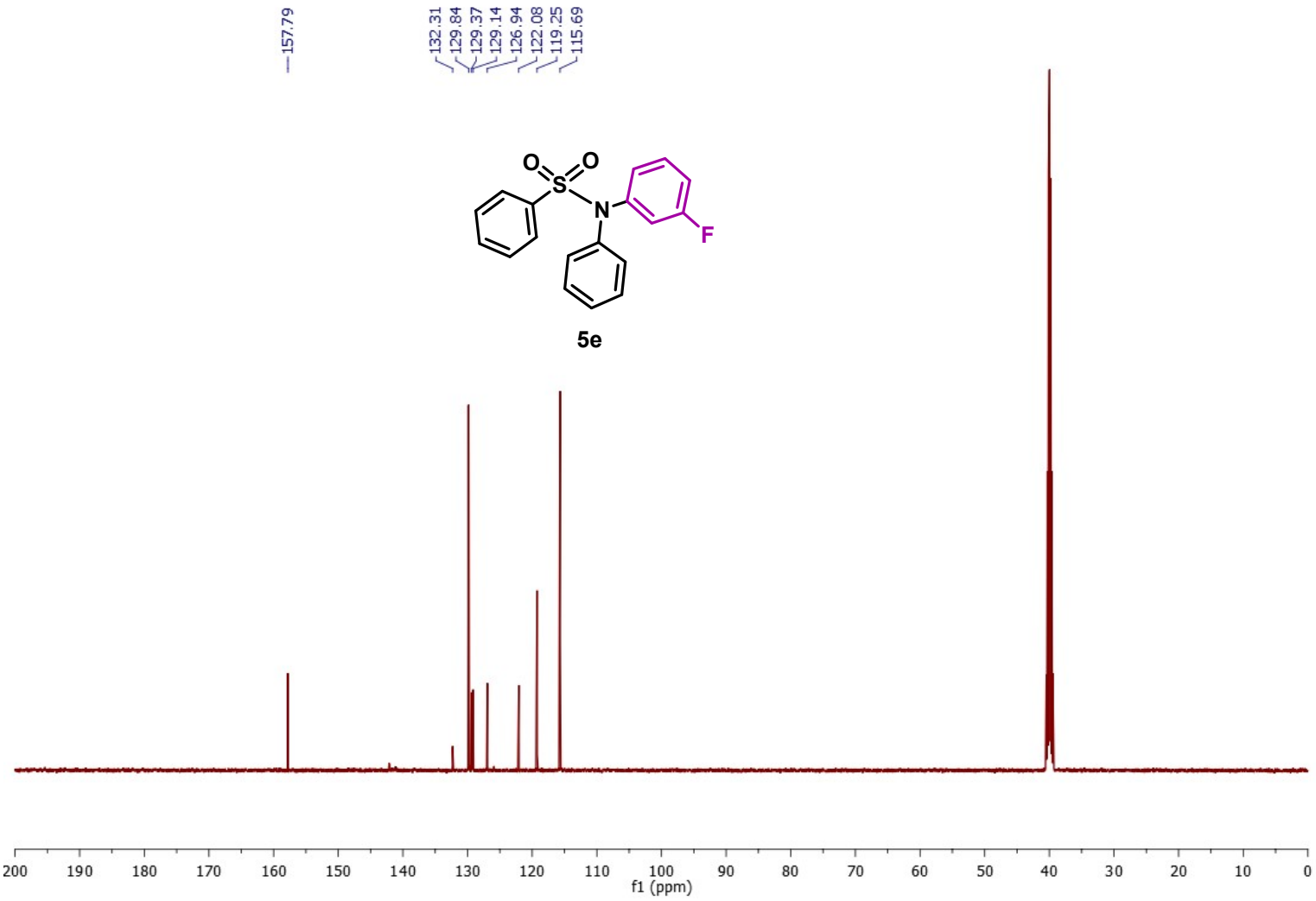
**5d**

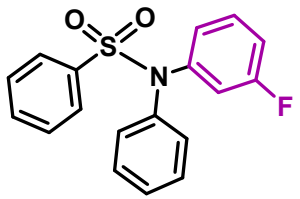

2.00
3.09
1.87
1.17
1.03
1.11
2.92
1.01
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


157.79
133.30
131.36
130.38
129.84
129.05
126.78
119.59
119.25
119.17
115.69
O
O
S
N
Cl
5d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.74
7.73
7.53
7.52
7.50
7.49
7.48
7.18
7.16
7.15
7.00
6.99
6.78
6.77
6.75
O
O
S
N
F
1.00
1.78
4.84
1.04
5.24
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

**5e**

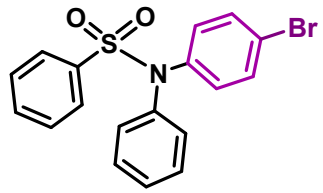

157.79
132.31
129.84
129.37
129.14
126.94
122.08
119.25
115.69
O
O
S
N
F
5e
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
O
S
N
F

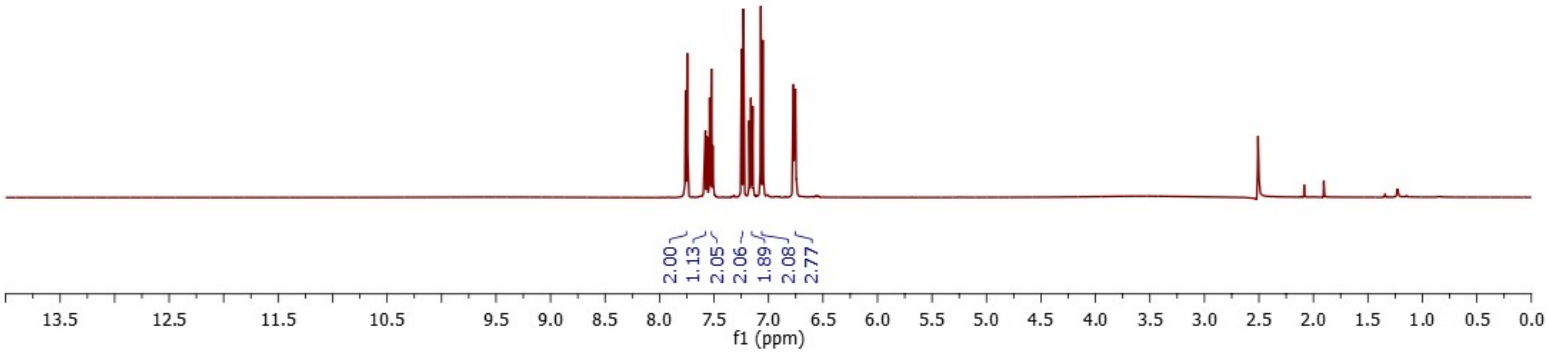
**5e**

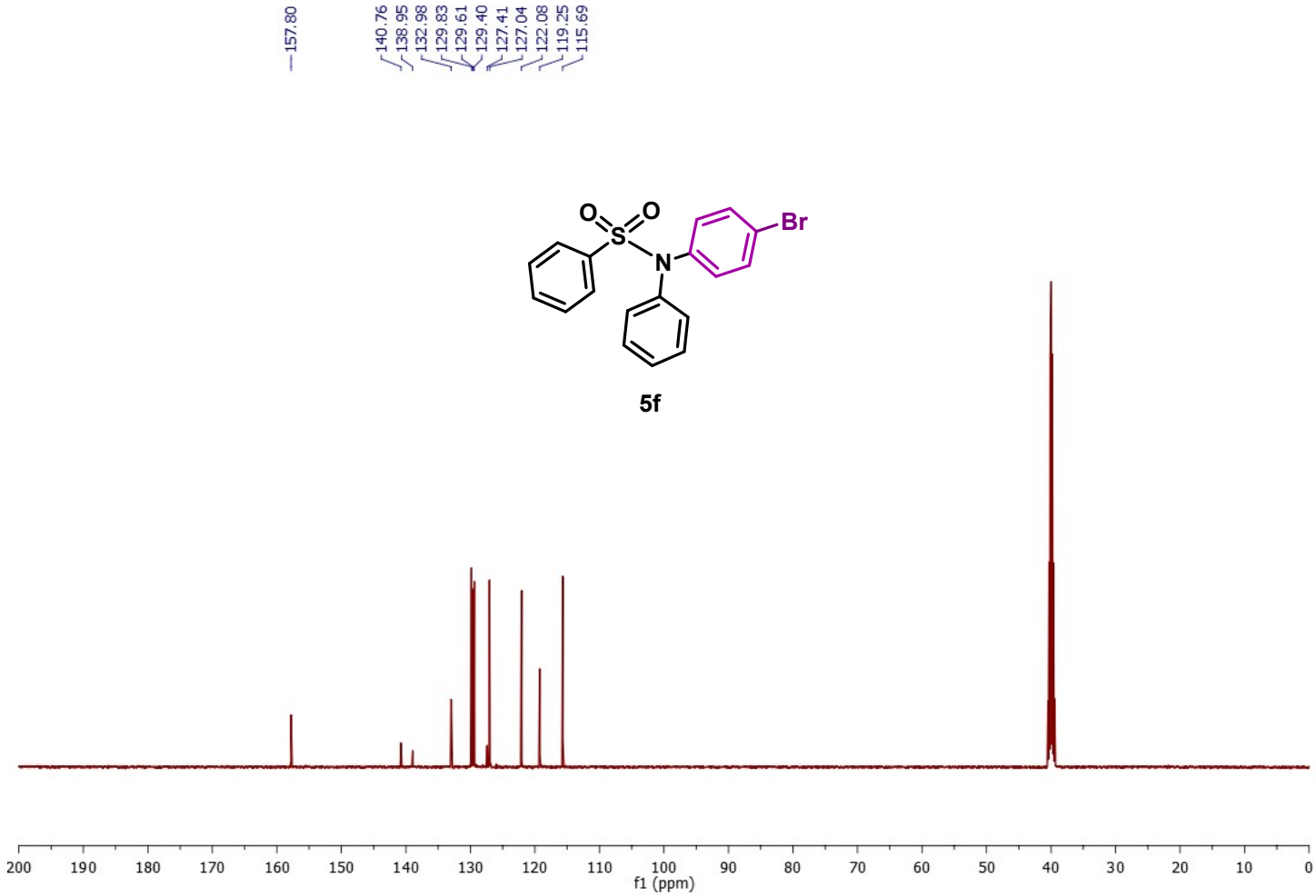

-105.59
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-105
-115
-125
-135
f1 (ppm)

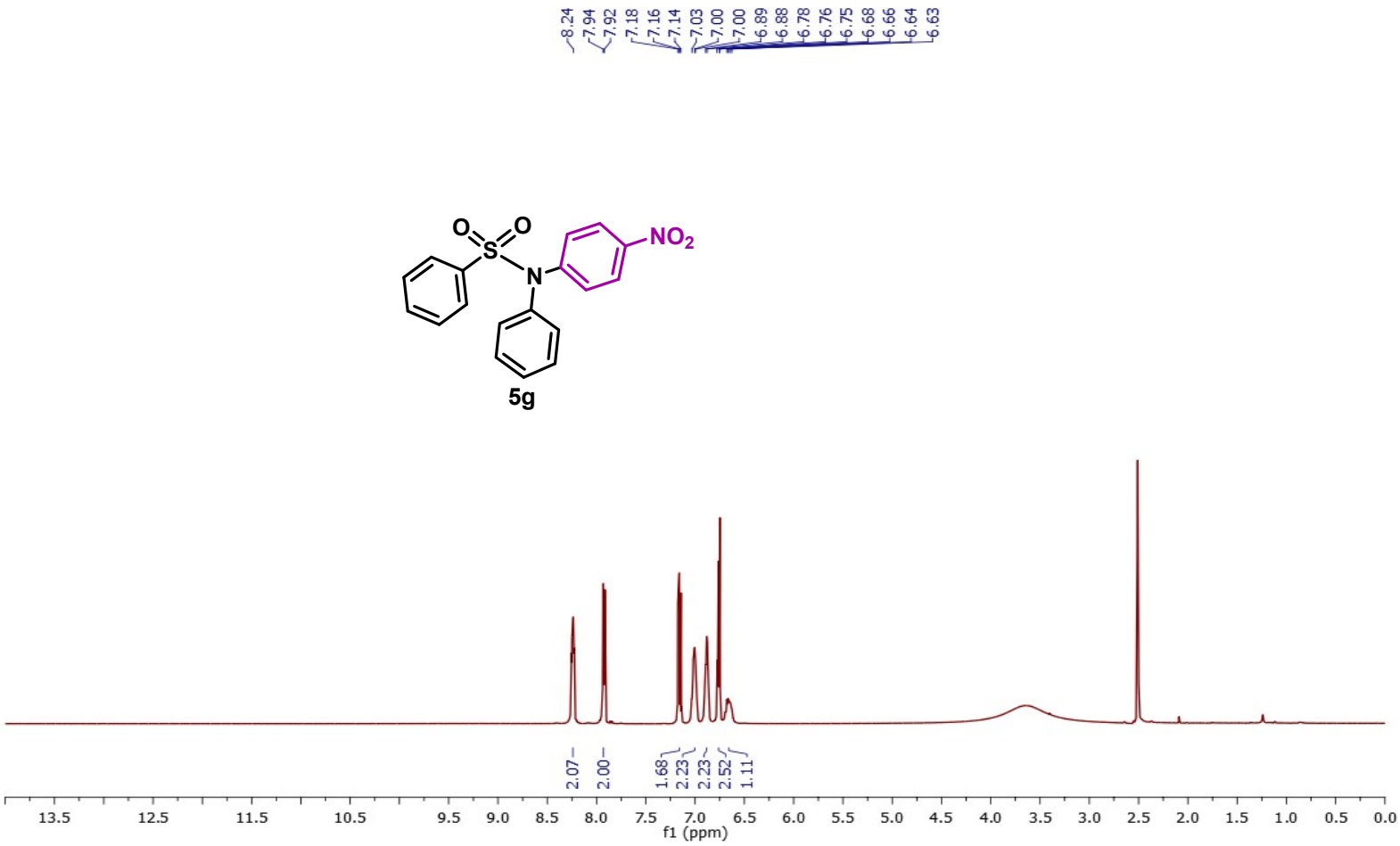

7.76
7.75
7.60
7.58
7.56
7.54
7.52
7.51
7.25
7.23
7.18
7.16
7.15
7.07
7.05
6.78
6.77
6.75

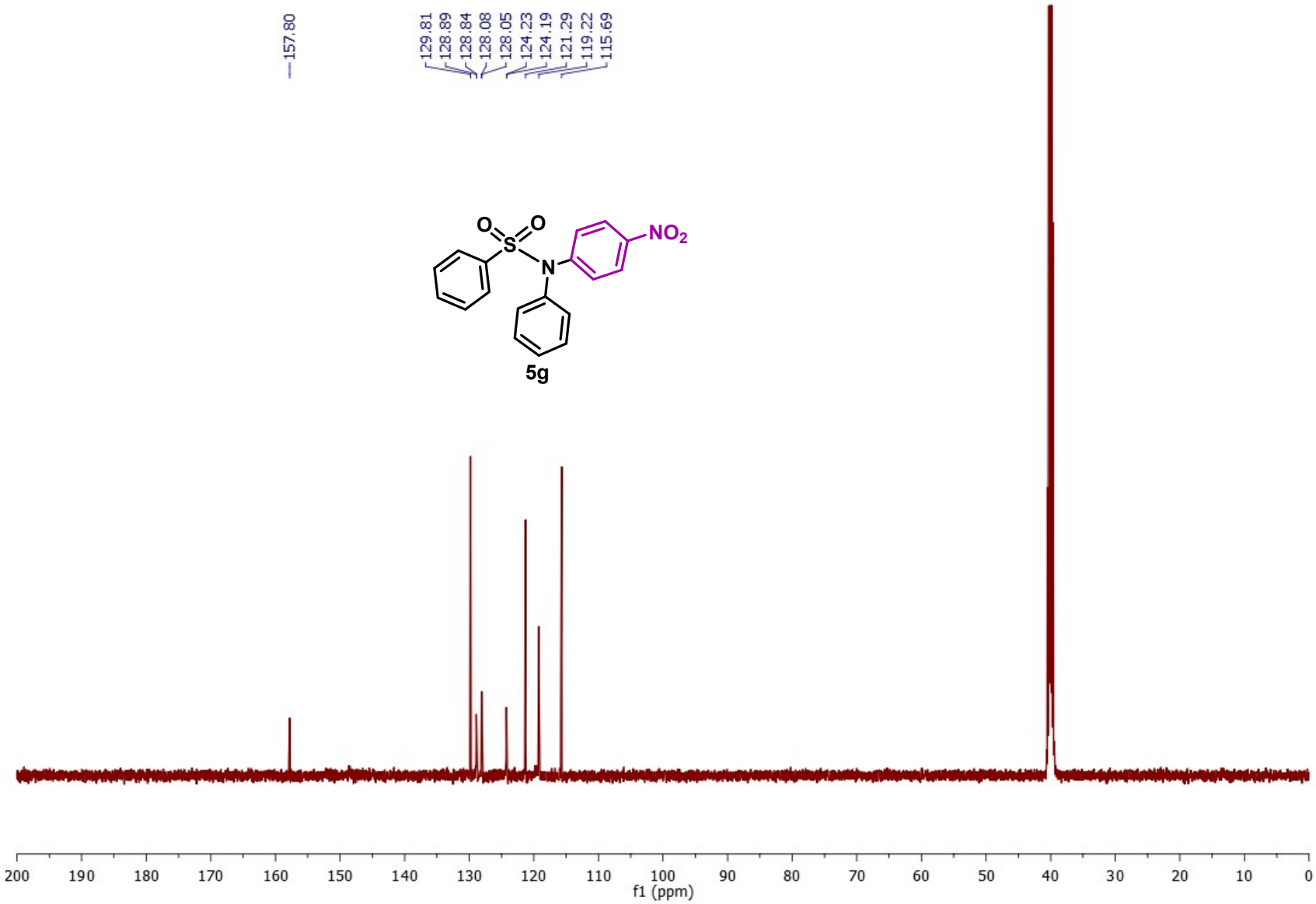

O
O
S
N
Br

**5f**

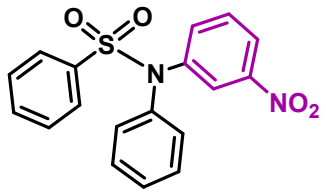

2.00
1.13
2.05
2.06
1.89
2.08
2.77
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


157.80
140.76
138.95
132.98
129.83
129.61
129.40
127.41
127.04
122.08
119.25
115.69
O
O
S
N
Br
5f
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

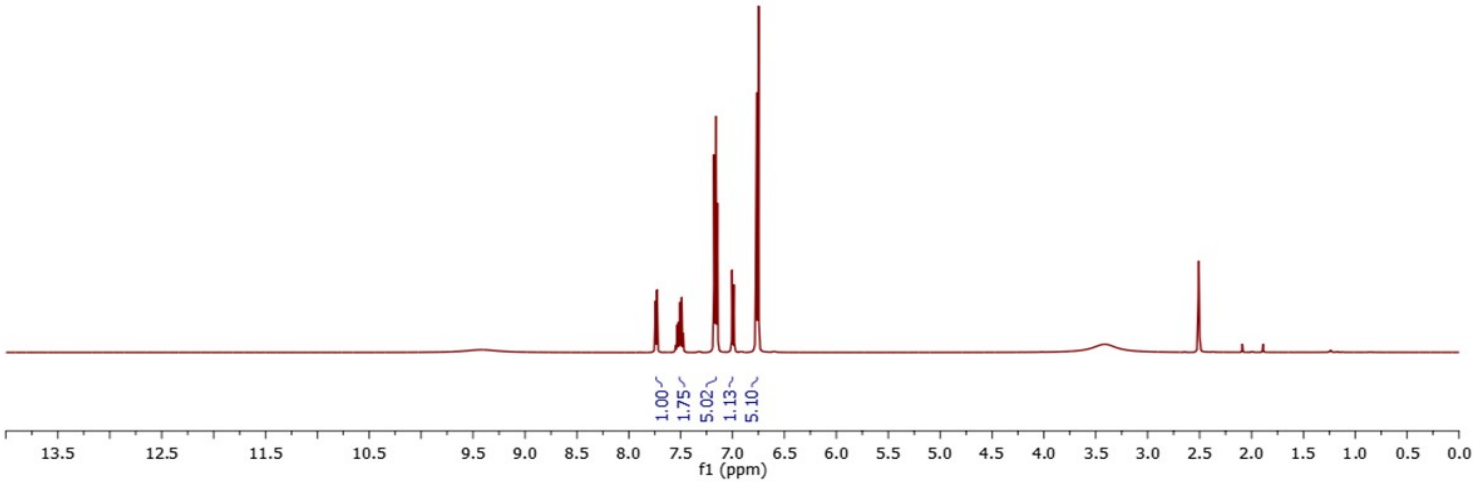

8.24
7.94
7.92
7.18
7.16
7.14
7.03
7.00
7.00
6.89
6.88
6.78
6.76
6.75
6.68
6.66
6.64
6.63
O
O
S
N
NO2
5g
2.07
2.00
1.68
2.23
2.23
2.52
1.11
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

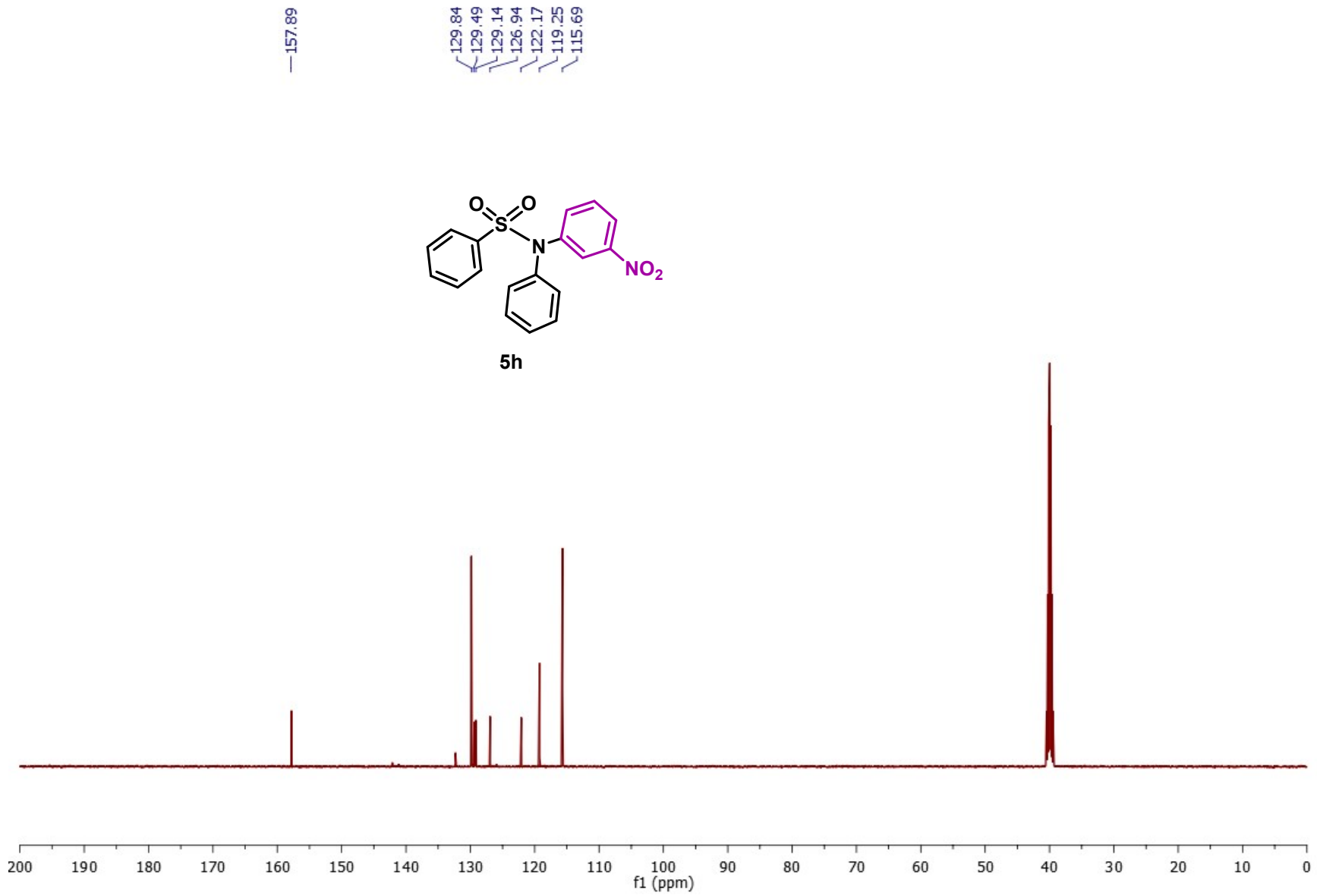

157.80
129.81
128.89
128.84
128.08
128.05
124.23
124.19
121.29
119.22
115.69
O
O
S
N
NO2
5g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.75
7.72
7.53
7.52
7.50
7.49
7.47
7.18
7.17
7.15
7.00
6.99
6.78
6.76
6.74


O
O
S
N
NO2

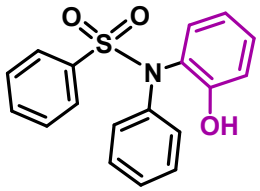
**5h**


1.00
1.75
5.02
1.13
5.10
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

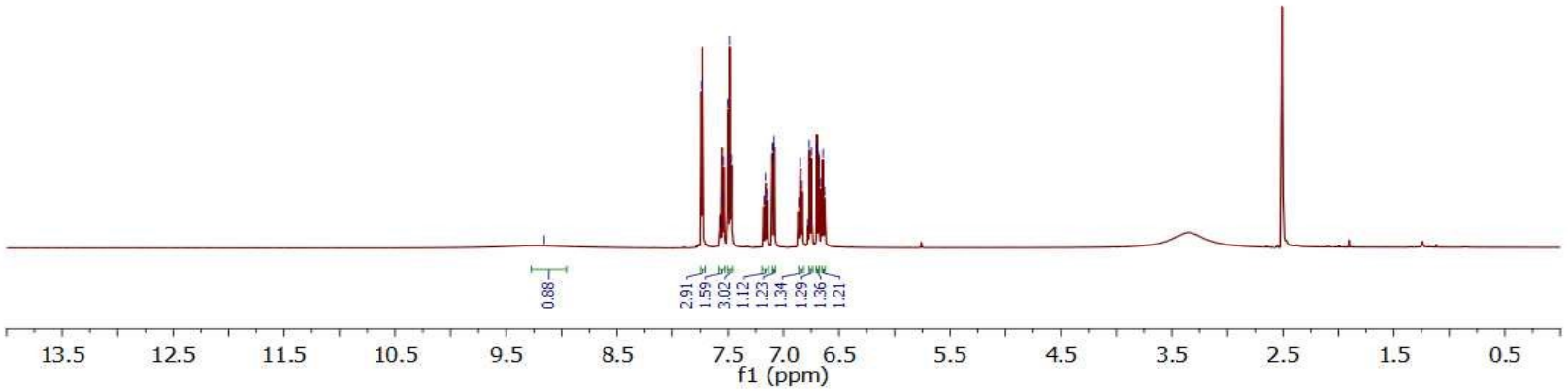

157.89
129.84
129.49
129.14
126.94
122.17
119.25
115.69
O
O
S
N
NO2
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

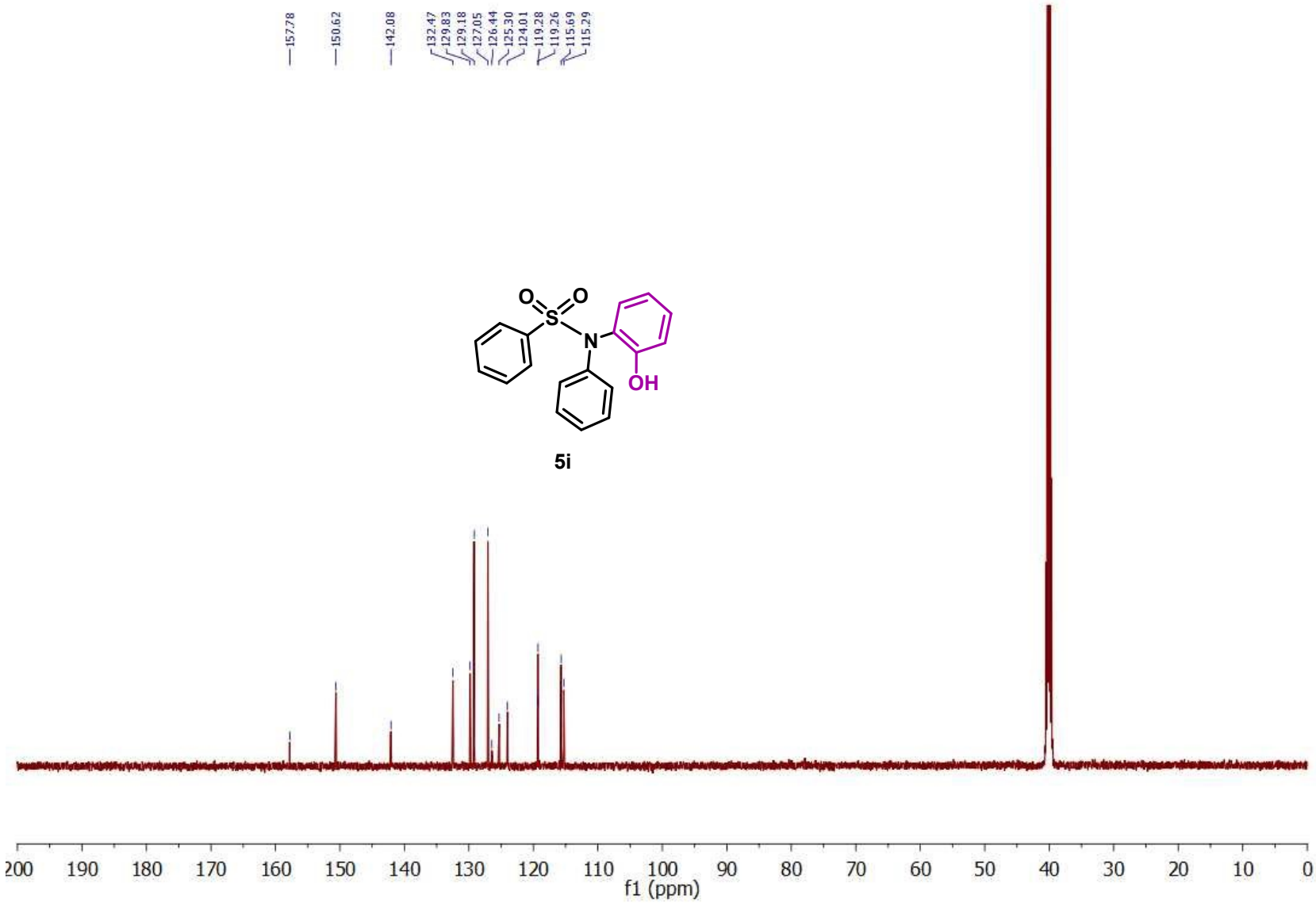
**5h**


9.16
7.74
7.73
7.57
7.55
7.54
7.50
7.49
7.47
7.18
7.16
7.15
7.10
7.10
7.09
7.08
6.86
6.85
6.83
6.78
6.77
6.75
6.70
6.69
6.66
6.64
6.63

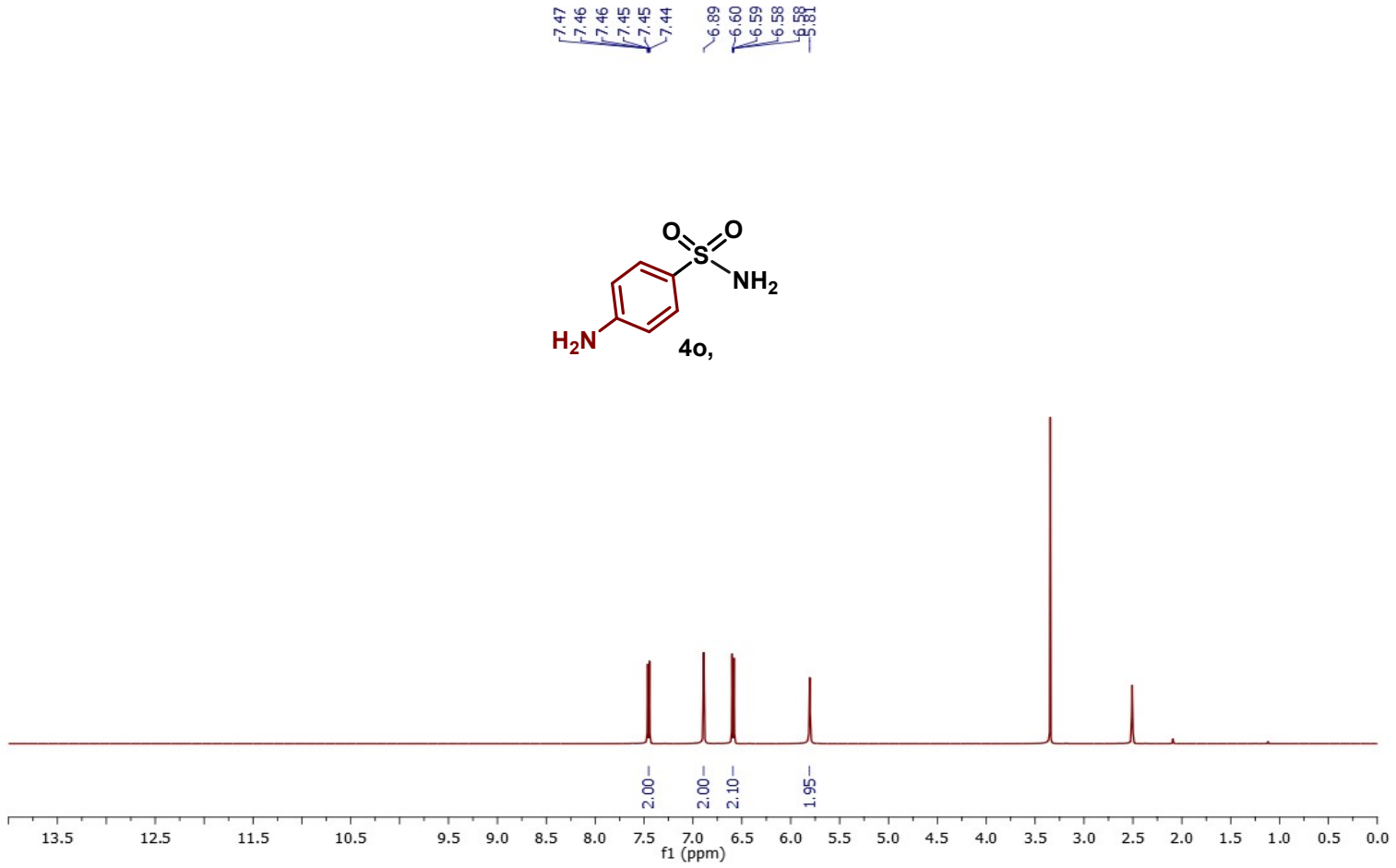

O
O
S
N
OH

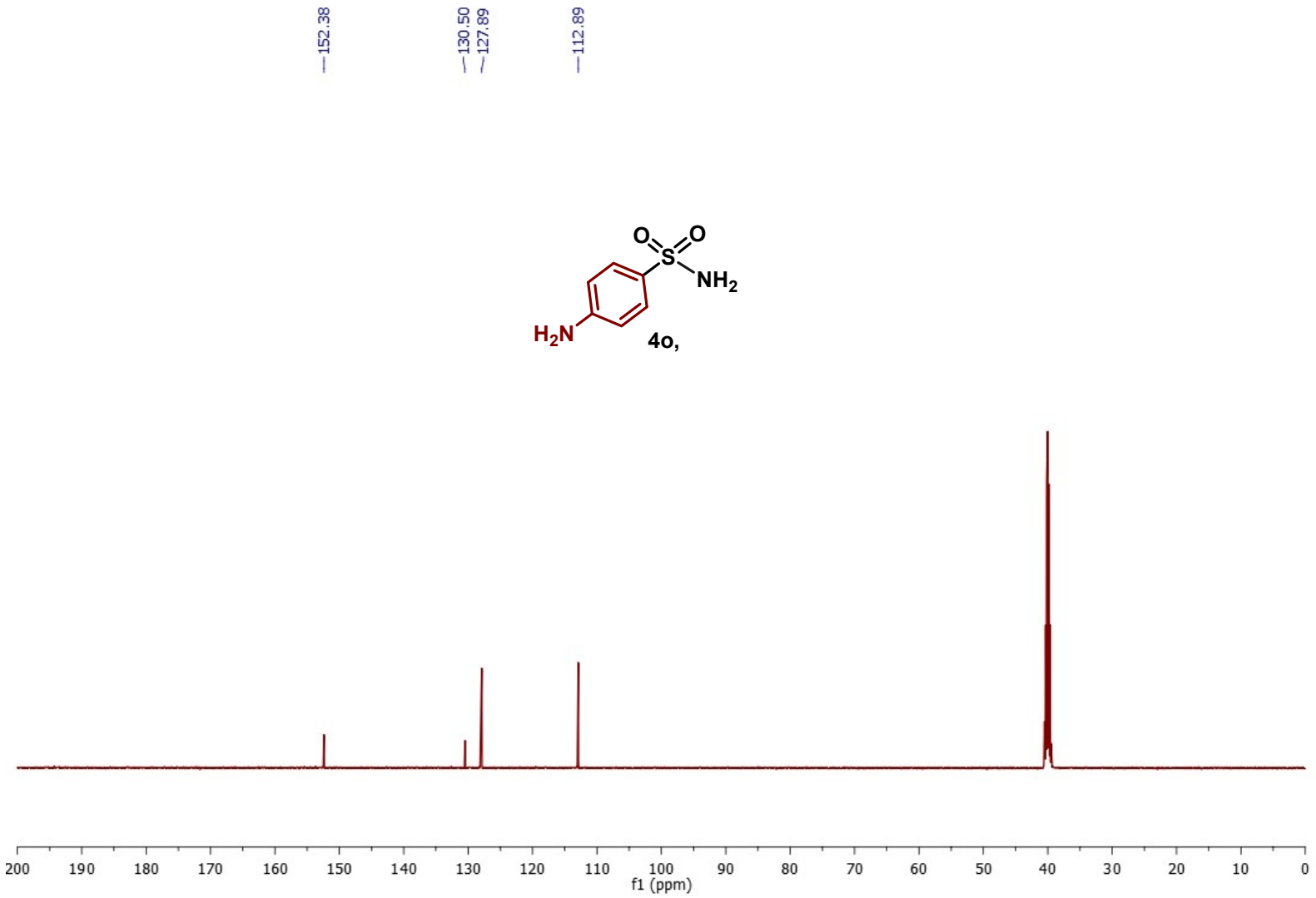
**5i**

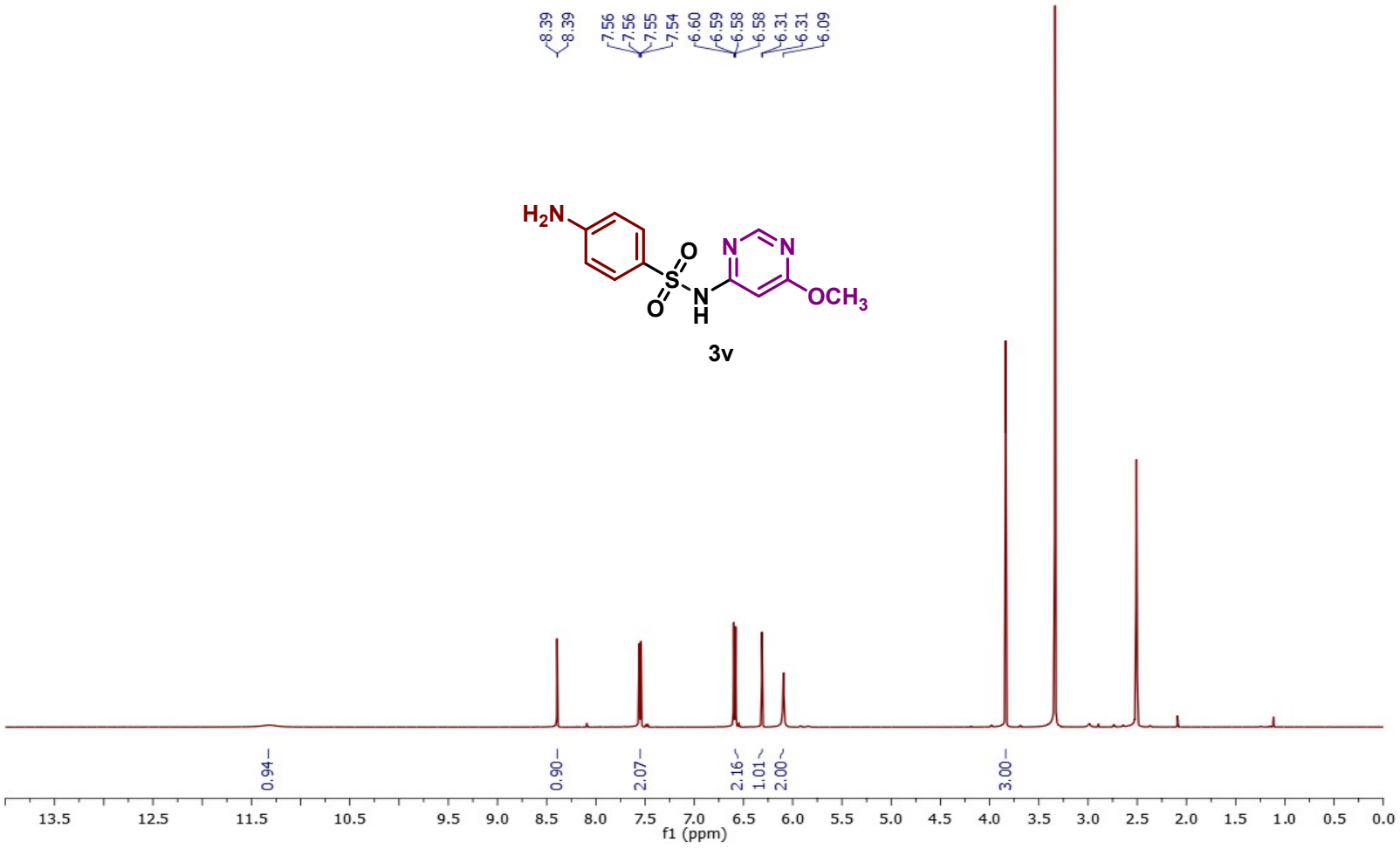

0.88
2.91
1.59
3.02
1.12
1.23
1.34
1.29
1.36
1.21
13.5
12.5
11.5
10.5
9.5
8.5
7.5
7.0
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

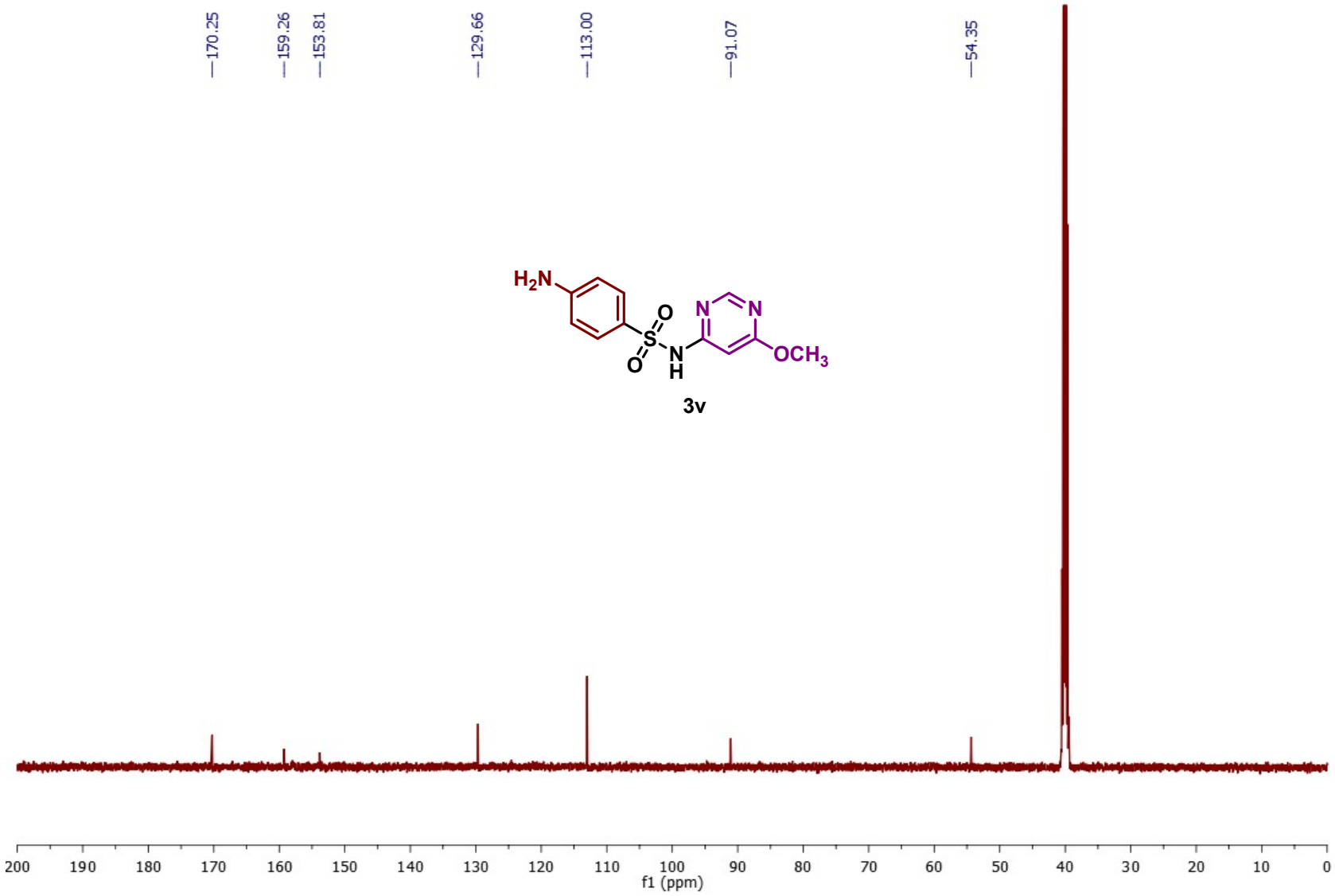

157.78
150.62
142.08
132.47
129.83
129.18
127.05
126.44
125.30
124.01
119.28
119.26
115.69
115.29
O
O
S
N
OH
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

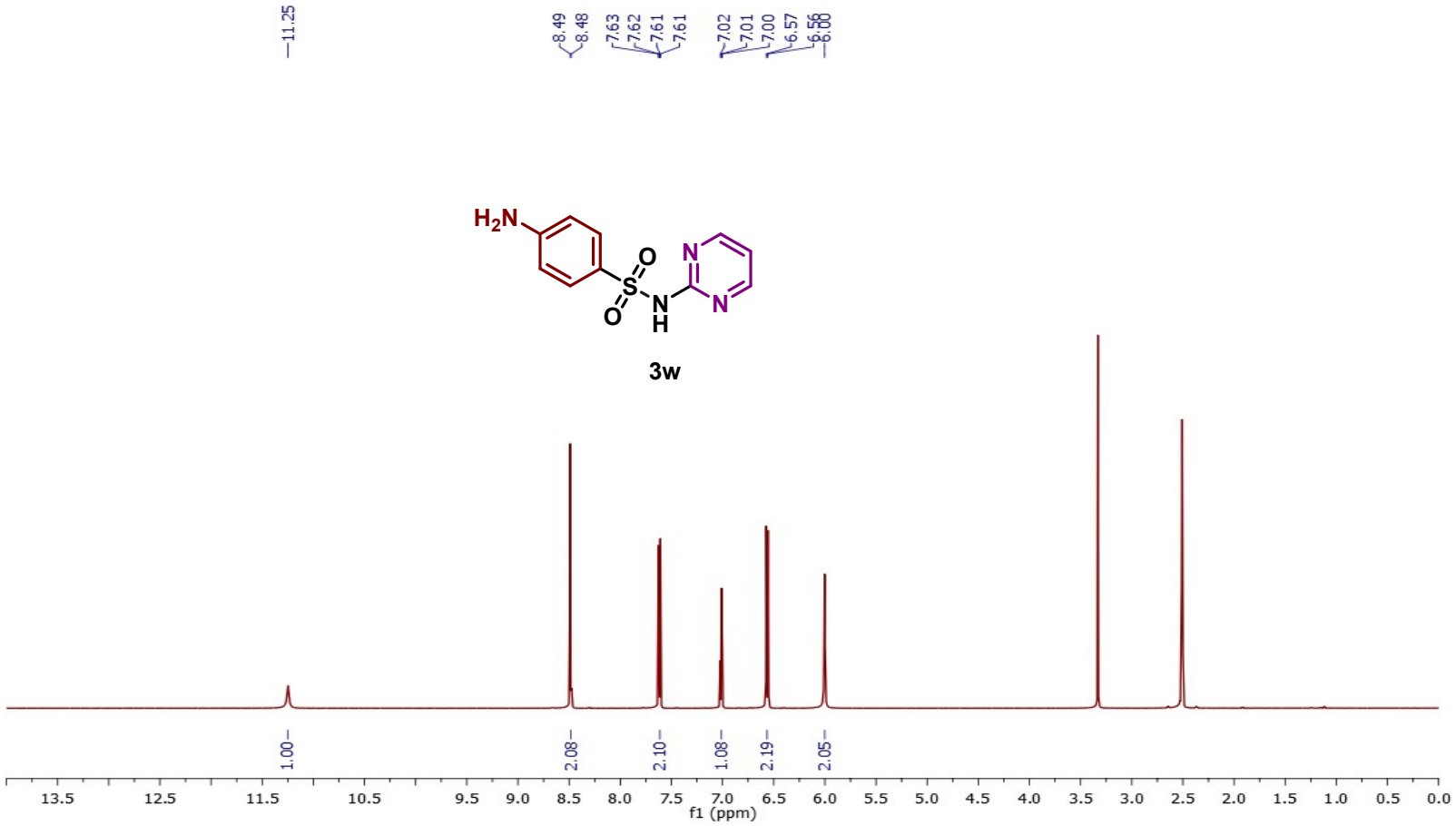
**5i**


7.47
7.46
7.46
7.45
7.45
7.44
6.89
6.60
6.59
6.58
6.58
5.81
O
O
S
NH2
H2N
4o,
2.00
2.00
2.10
1.95
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


152.38
130.50
127.89
112.89
O
O
S
NH2
H2N
4o,
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.39
8.39
7.56
7.56
7.55
7.54
6.60
6.59
6.58
6.58
6.31
6.31
6.09
H2N
O
S
O
N
H
N
N
OCH3
3v
0.94
0.90
2.07
2.16
1.01
2.00
3.00
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


170.25
159.26
153.81
129.66
113.00
91.07
54.35
H2N
O
S
O
N
H
N
N
OCH3
3v
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


11.25
8.49
8.48
7.63
7.62
7.61
7.61
7.02
7.01
7.00
6.57
6.56
6.00
H2N
O
S
O
N
H
N
N
3w
1.00
2.08
2.10
1.08
2.19
2.05
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

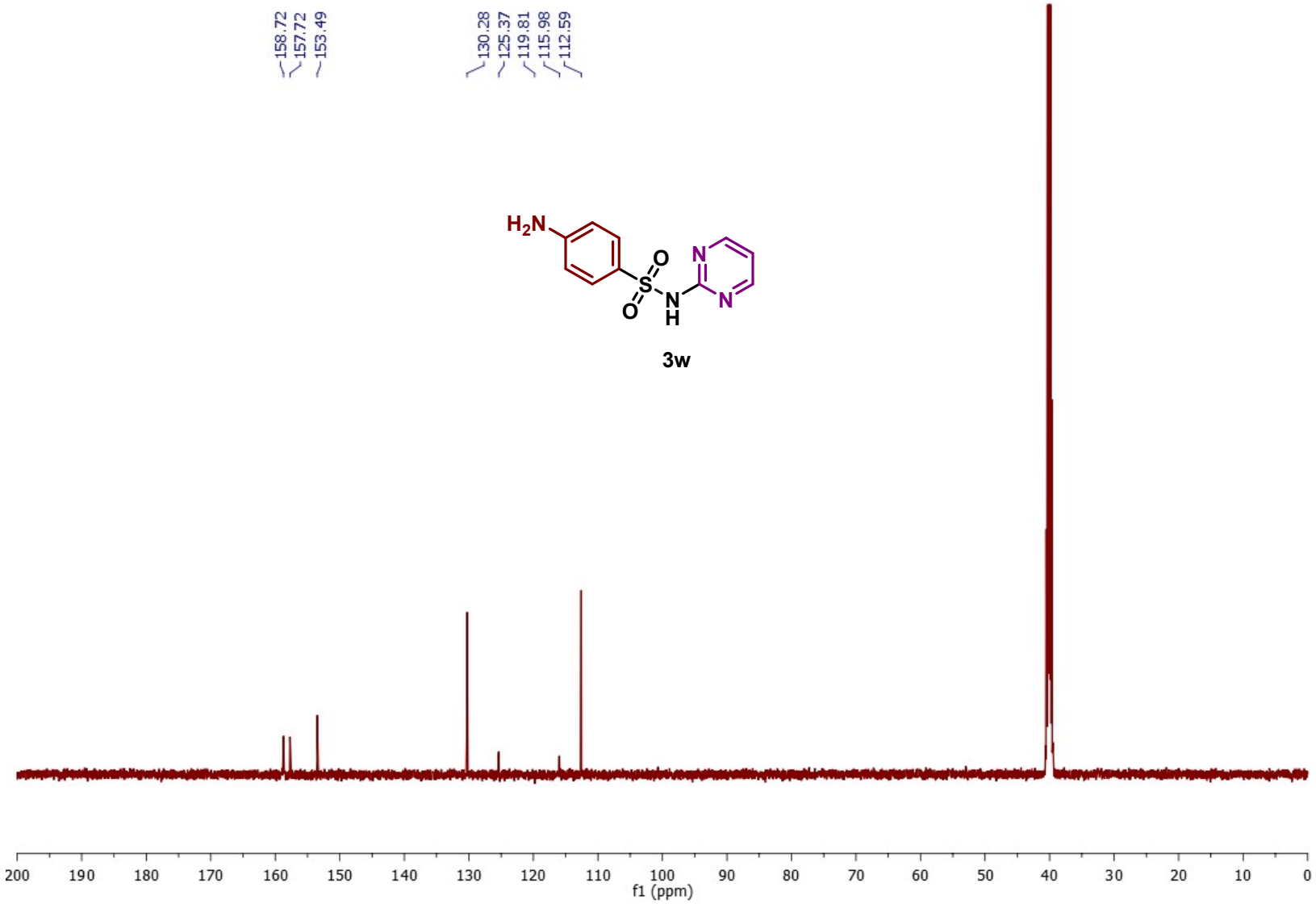
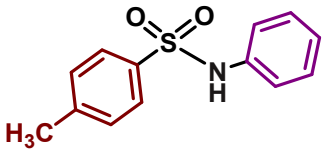
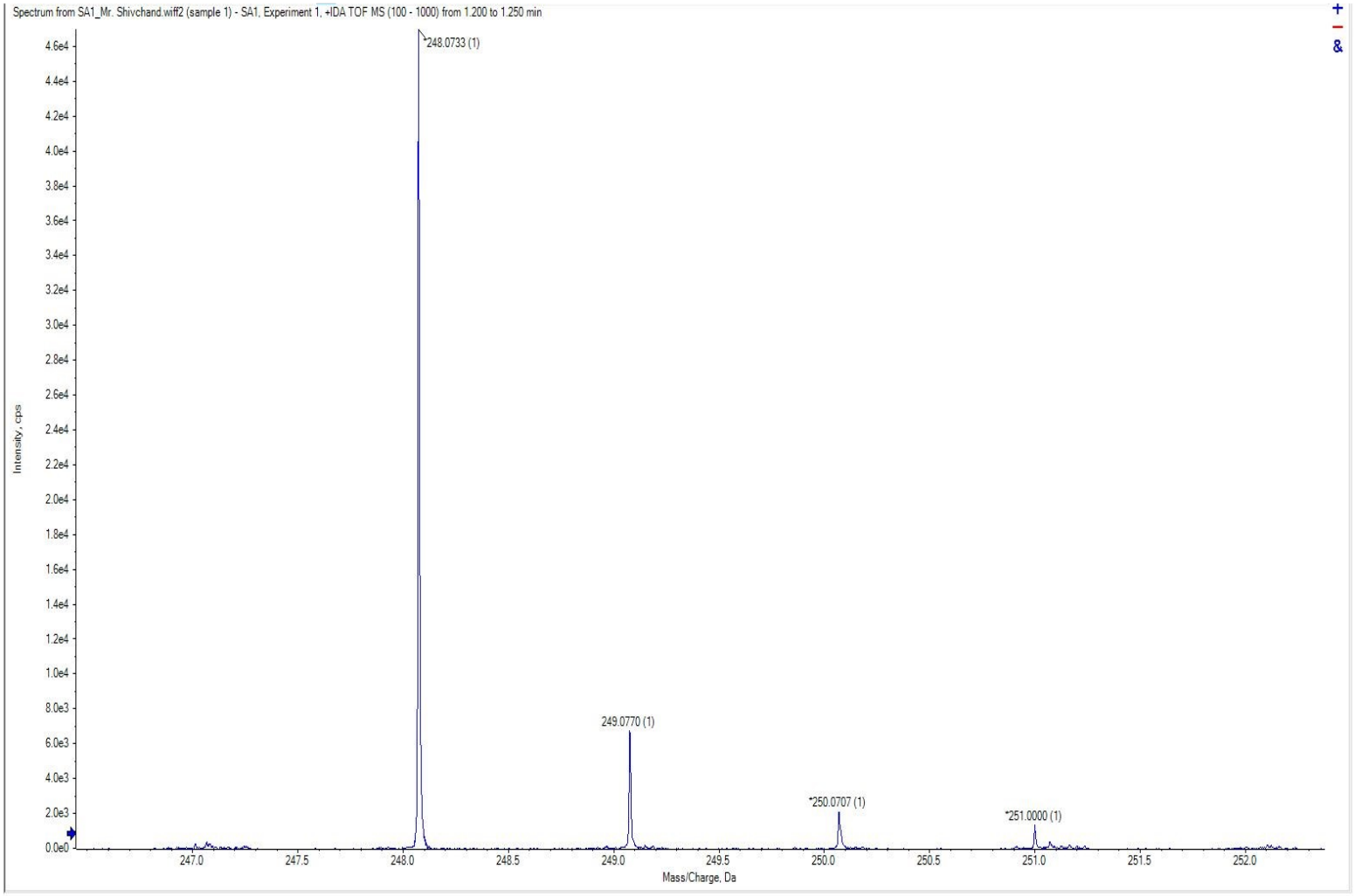

158.72
157.72
153.49
130.28
125.37
119.81
115.98
112.59
H2N
O
S
O
N
H
N
N
3w
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

**Figure S4. HRMS spectra of 3a**



# Figure S5. HRMS spectra of 4c