

## **Supporting Information**

### **Electrochemical Sulfinylation of Phenols with Sulfides: A Metal- and Oxidant-free Cross-Coupling for the Synthesis of Aromatic Sulfoxides**

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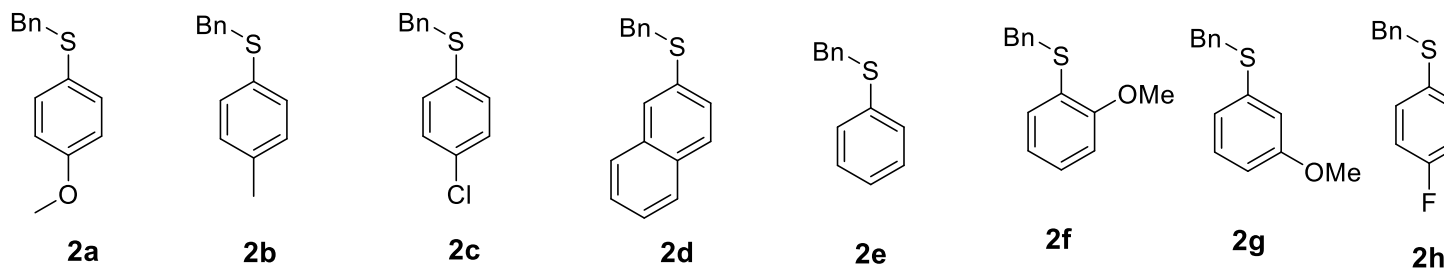
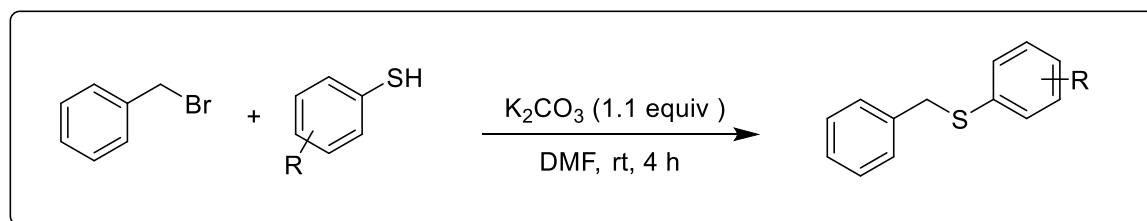
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## A. General Information

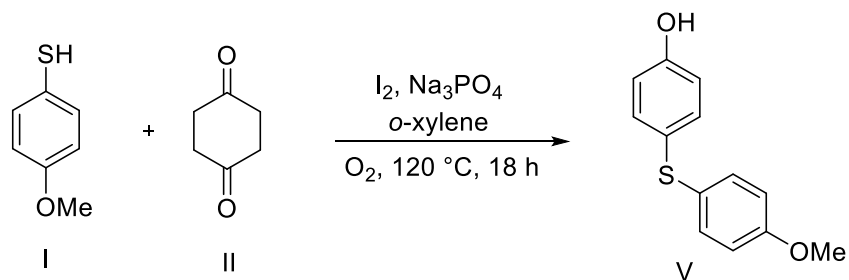
Unless noted otherwise, all reagents and solvents were purchased from commercial sources and used as received. All reactions were performed in oven dried round bottom flasks. Electrochemical reactions were performed at room temperature using DC power supply of Keysight technologies (25 V, 5A) and GW INSTEK GPP-4323 (32 V, 3 A). Electrodes were commercially available from IKA. Cyclic voltammetry analysis was carried out in CH instrument electrochemical analyzer (CHL1110C). The developed chromatogram was analyzed by UV lamp (254 nm) or p-anisaldehyde solution. Column chromatography was performed on silica gel mesh size 200-300. The proton ( $^1\text{H}$ ) and carbon  $^{13}\text{C}\{^1\text{H}\}$  NMR spectra were recorded in 400 MHz JEOL JNM ECS400 spectrometer in the  $\text{CDCl}_3$  solvent (unless otherwise mentioned) and are reported in  $\delta$  units. Chemical shifts of NMR spectra are expressed in parts per million (ppm). Coupling constants ( $J$  Values) are reported in Hz. High-resolution mass spectra (HRMS) were obtained using the electron spray ionization (ESI) technique and TOF mass analyzer. Yields refer to isolated compounds, estimated to be less than 95% pure as determined by  $^1\text{H}$ NMR. The description of the signals includes the following: s = singlet, d = doublet, dd = doublet of doublet, t = triplet, dt = doublet of triplet, q = quartet, br = broad and m = multiplet.

## B. General Procedure for the Synthesis of ArCH<sub>2</sub>SAr'<sup>1</sup>

In an oven dried 100 mL round-bottomed flask equipped with a magnetic bar, benzyl bromides (1 equiv.), thiophenols (1 equiv.) and powdered K<sub>2</sub>CO<sub>3</sub> (1.1 equiv.) in DMF were taken. The reaction was allowed to stir for 4 h and completion was monitored by TLC. After completion, water was added to the reaction mixture and extracted with ethyl acetate three times. The resulting organic layer was further washed with brine solution and dried over anhydrous sodium sulfate. The solvent was removed on a rotavap under reduced pressure, the residue was subjected to flash column chromatography to obtain the desired products.

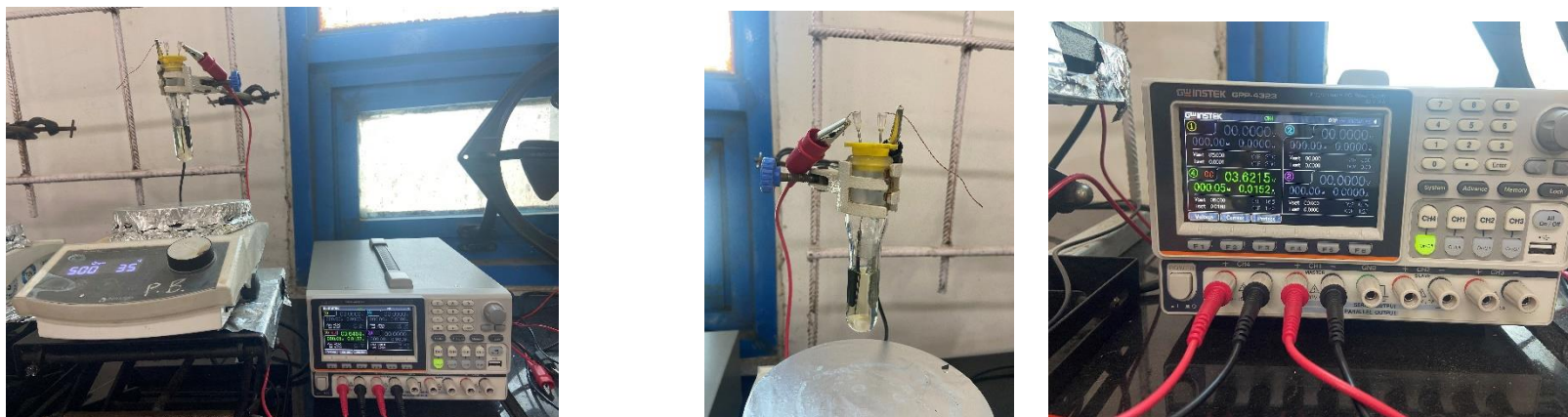


### C) General Procedure for synthesis of intermediate V<sup>3</sup>



In an oven-dried reaction vessel charged with cyclohexanones (0.5 mmol), thiophenols (0.3 mmol),  $I_2$  (0.2 mmol),  $Na_3PO_4$  (0.2 mmol), and *o*-xylene (1.5 mL). The reaction vessel was purged with oxygen three times and stirred at  $120\text{ }^\circ\text{C}$  for 18 h. After cooling to room temperature, the reaction mixture was diluted with EtOAc and filtered. The filtrate was then concentrated in vacuo, and the resulting residue was purified by column chromatography on silica gel to afford the corresponding product.

## D). General set up and Procedure for Electrochemical reaction

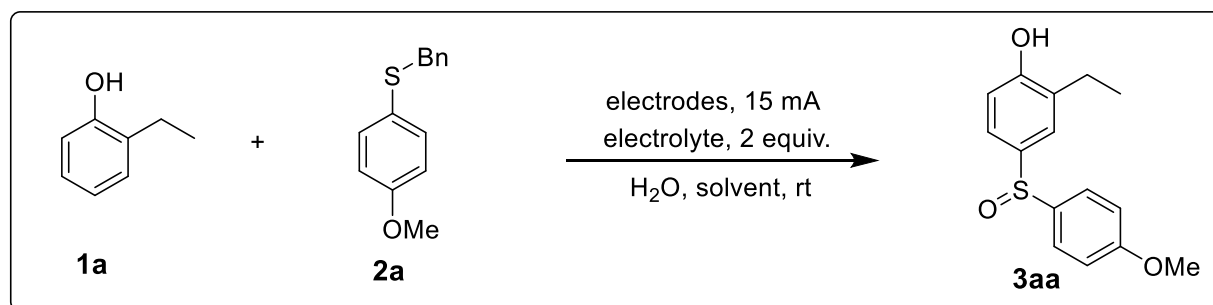


In an undivided cell equipped with magnetic bar and graphite as anode and nickel as cathode, mixture of **1a-1n** (1.0 equiv.), **2a-2e** (1.0 equiv.),  $\text{Bu}_4\text{NPF}_6$  (2.0 equiv.) and DCE: HFIP (3:1) 4 ml were added. The mixture was electrolyzed at a constant current of 15 mA at room temperature for 2-6 h in a DC power supply. Upon completion, the solvent was removed under reduced pressure and the crude was subjected to silica gel column chromatography (200-400 mesh) to afford the desired product.

### E) General procedure for the electrochemical gram-scale synthesis

In an oven-dried two-neck round-bottom flask (100 mL) equipped with a magnetic bar and graphite as both the anode and cathode, **1a** (8.18 mmol, 1.0 g), **2a** (8.18 mmol, 1.88 g), Bu<sub>4</sub>NPF<sub>6</sub> (1 equiv.), and DCE: HFIP (3:1) (24 mL) were added. The mixture was electrolyzed at a constant current of 15 mA at room temperature for 18 h in a DC power supply. After 18 h, the solvent was removed under reduced pressure, and the crude was purified by silica gel column chromatography using 6-8 % ethyl acetate in hexane to afford the desired product in 40% yield (0.9 g).

### F) Optimization studies for the synthesis of 2-ethyl-4-((4-methoxyphenyl)sulfinyl)phenol(**3aa**)





2-ethylphenol (**1a**, 0.25 mmol), benzyl(4-methoxyphenyl)sulfane (**2a**, 0.25 mmol), electrolyte (2.0 equiv.) and solvent (4 ml, 3:1) were taken in an undivided cell equipped with stir bar and graphite anode and nickel cathode. The mixture was electrolyzed at a constant current of 15 mA at room temperature for 2-6 h. After the completion of reaction, the mixture was evaporated in vacuo and the crude was purified by silica gel column chromatography using 5-10 % ethyl acetate in hexane to get the 2-ethyl-4-((4-methoxyphenyl)sulfinyl)phenol (**3aa**) in 18-60% isolated yield.

**Table ES1: Optimization of reaction conditions**

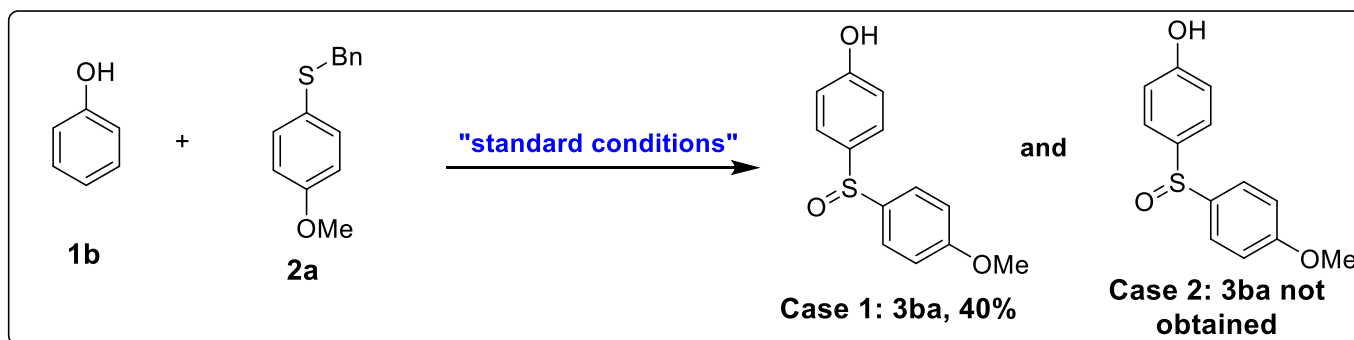
<b>Entry</b>	<b>Solvent</b>	<b>Electrolyte</b>	<b>Electrode</b>	<b>Yield(%)<sup>b</sup></b>
1 <sup>a</sup>	<b>DCE:HFIP</b>	<b>Bu<sub>4</sub>NPF<sub>6</sub></b>	<b>C(+)/Ni(-)</b>	<b>60</b>
2	DCE:HFIP	Bu <sub>4</sub> NBF <sub>4</sub>	C(+)/Ni(-)	40
3	DCE:HFIP	Et <sub>4</sub> NPF <sub>6</sub>	C(+)/Ni(-)	46

4	DCE:HFIP	LiClO <sub>4</sub>	C(+)/Ni(-)	42
5	DCE:HFIP	Et <sub>4</sub> NOTf	C(+)/Ni(-)	40
6	DCE:HFIP	Bu <sub>4</sub> NI	C(+)/Ni(-)	c.m.
7	DCE:HFIP	Bu <sub>4</sub> NBr	C(+)/Ni(-)	c.m.
8	DCE:HFIP	Bu <sub>4</sub> NCl	C(+)/Ni(-)	c.m.
9	DCE:HFIP	Bu <sub>4</sub> NHSO <sub>4</sub>	C(+)/Ni(-)	c.m.
10	DCE:HFIP	Bu <sub>4</sub> NOAc	C(+)/Ni(-)	c.m.
11	DCE	Bu <sub>4</sub> NPF <sub>6</sub>	C(+)/Ni(-)	48
12	MeOH	Bu <sub>4</sub> NPF <sub>6</sub>	C(+)/Ni(-)	35
13	THF	Bu <sub>4</sub> NPF <sub>6</sub>	C(+)/Ni(-)	n.r.
14	DCE:TFE	Bu <sub>4</sub> NPF <sub>6</sub>	C(+)/Ni(-)	18
15	DCM:HFIP	Bu <sub>4</sub> NPF <sub>6</sub>	C(+)/Ni(-)	n.r.
16	DCE:HFIP	Bu <sub>4</sub> NPF <sub>6</sub>	C(+)/C(-)	45
17	DCE:HFIP	Bu <sub>4</sub> NPF <sub>6</sub>	C(+)/Pt(-)	50
18	DCE:HFIP	Bu <sub>4</sub> NPF <sub>6</sub>	Ni(+)/Ni(-)	40

<sup>a</sup>Reaction conditions: **1a** (0.25 mmol), **2a** (0.25 mmol), Bu<sub>4</sub>NPF<sub>6</sub> as the electrolyte (0.49 mmol), DCE: HFIP (3:1) as the solvent (4 mL), 15 mA constant current, graphite anode, nickel cathode, undivided cell, 25 °C. <sup>b</sup>Isolated yield, n.r. = no reaction. c.m. = complex mixture.

## G) Mechanistic studies

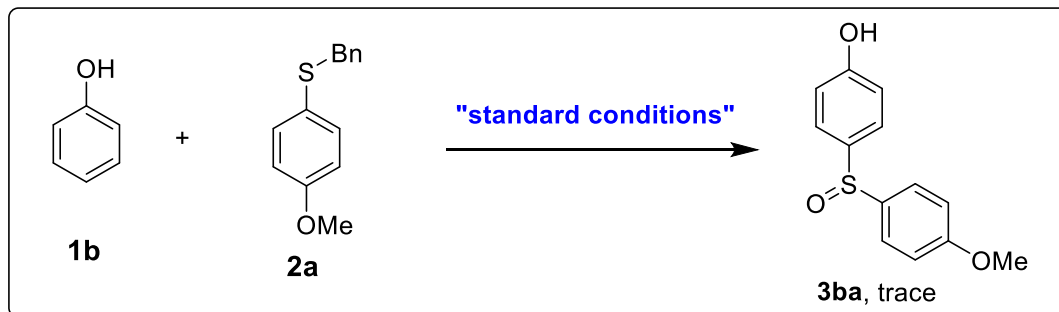
### i) Divided cell experiment



**Case 1: In anodic chamber:** A divided cell was equipped with two magnetic stir bars in anodic and cathodic chamber respectively. Further, the anodic chamber was filled with corresponding Phenol **1b** (1.0 equiv.), benzyl(4-methoxyphenyl)sulfane **2a** (1.0 equiv.), tetrabutylammonium hexafluorophosphate ( $\text{Bu}_4\text{NPF}_6$ ) (2.0 equiv.), in DCE:HFIP (3:1) solvent. The cathodic chamber was filled only with supporting electrolyte solution and the solution was electrolyzed with carbon anode (in anodic chamber) and nickel cathode (in cathodic chamber) at a constant current of 15 mA for 10 h at room temperature (25-30 °C). However, there is a slight decrease in yield as the desired product was obtained in 40 % yield.

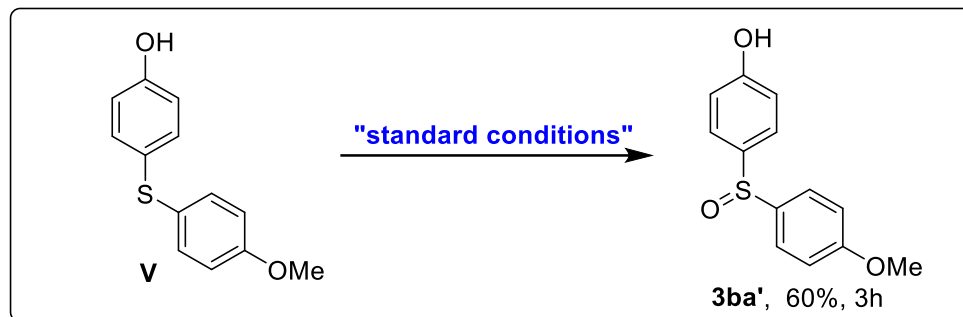
**Case 2: In cathodic chamber:** A divided cell was equipped with two magnetic stir bars in anodic and cathodic chamber respectively. Further, the cathodic chamber was filled with corresponding Phenol **1b** (1.0 equiv.), benzyl(4-methoxyphenyl)sulfane **2a** (1.0 equiv.), tetrabutylammonium hexafluorophosphate ( $\text{Bu}_4\text{NPF}_6$ ) (2.0 equiv.) in DCE:HFIP (3:1) solvent. The anodic chamber was filled with supporting electrolyte solution and the solution was electrolyzed with carbon anode (in anodic chamber) and nickel cathode (in cathodic chamber) at a constant current of 15 mA for 10 h at room temperature. The progress of the reaction was monitored by TLC, which shows that starting material remain unconsumed. This infers that the reaction takes place by the anodic oxidation.

## ii) Time based electrolysis



A test tube was equipped with a magnetic stir bar and was added corresponding Phenol **1b** (1.0 equiv.), benzyl(4-methoxyphenyl)sulfane **2a** (1.0 equiv.), tetrabutylammonium hexafluorophosphate ( $\text{Bu}_4\text{NPF}_6$ ) (2.0 equiv.) in DCE:HFIP (3:1) solvent. Further, the solution was electrolyzed with carbon anode and nickel cathode at a constant current of 15 mA for 1 h at room temperature. The progress of the reaction was monitored by TLC, which shows that starting material remain unconsumed with trace amount of product formation. After, that the reaction was stirred overnight without electricity. Again, the progress of the reaction was monitored by TLC, which indicated that a similar TLC was observed. Further, the reaction mixture was purified through column chromatography and approx. 70% of the starting material was recovered. This indicates that the chain propagation step is absent in this case.

### iii) Reaction of Intermediate **V** under standard conditions



In an undivided cell equipped with magnetic bar and graphite as anode and nickel as cathode, mixture of **V** (1.0 equiv., 0.10 mmol),  $\text{Bu}_4\text{NPF}_6$  (2.0 equiv., .21 mmol) and DCE: HFIP (3:1) 4 ml were added. The mixture was electrolyzed at a constant current of 15 mA at room temperature for 3 h in a DC power supply. Upon completion, the solvent was removed under reduced pressure and the crude was subjected to silica gel column chromatography (200-400 mesh) to afford the desired product.

#### iv) Cyclic voltammetry

Cyclic voltammetry analysis was carried out in CH instrument electrochemical analyzer (CHL1110C). Samples were prepared in 5 ml vial with 0.01 M of substrate (**1b**), 0.01 M of benzyl(4-methoxyphenyl)sulfane (**2a**) and 0.1 M of  $\text{Bu}_4\text{NPF}_6$  in DCE:HFIP (3:1) 4ml. Measurements employed glassy carbon working electrode, platinum wire counter electrode and a 3M KCl silver-silver chloride reference electrode. The sweep rate applied was 50 mV/s. The oxidation potential of **1b** was observed to be 1.10 V (vs Ag/AgCl), and 1.5 V (vs Ag/AgCl) for **2a**. However, on adding both the reagents together, a slight shift in the oxidation potential of both **1b** and **2a** to 1.37 V and 1.89 V respectively was observed (figure 1). All the CV experiments were carried out in Argon atmosphere and demonstrated as follow: (a) 0.1 M  $\text{Bu}_4\text{NBF}_4$  (black) (b) 0.01 M **1b** (red) (d) 0.01 M **2a** (blue) (e) 0.01 M **1a** and 0.01 M **2a** (pink).

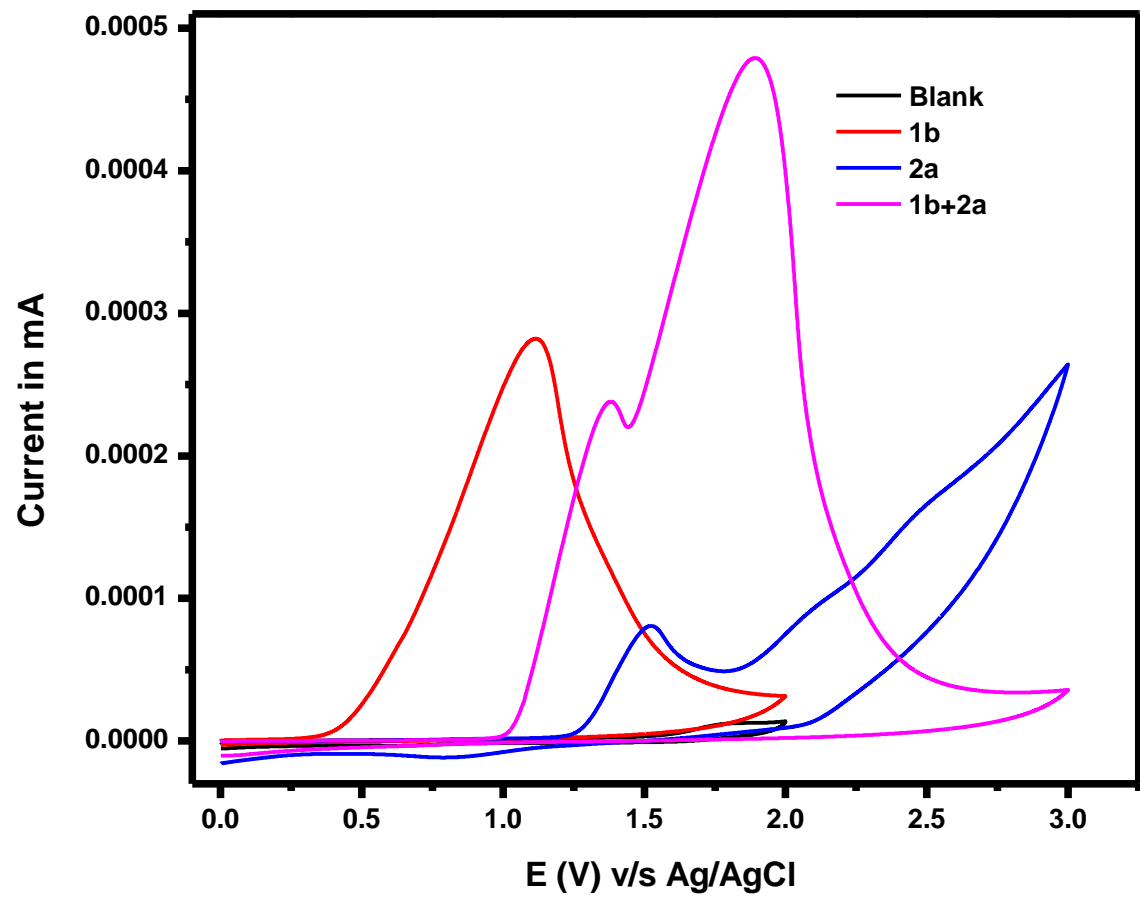
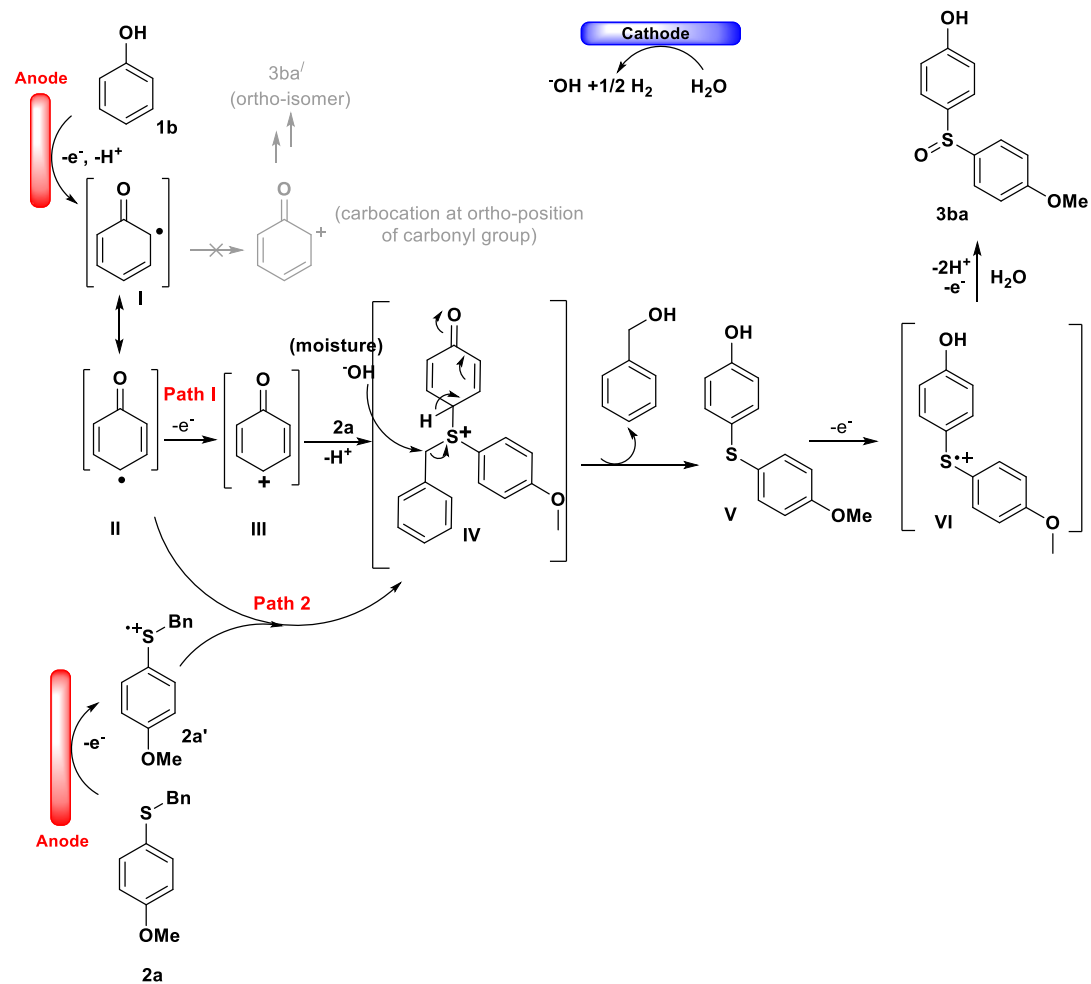


Figure 1. Cyclic Voltammetry experiment.



## v) Plausible Mechanism



Scheme 1. Plausible mechanism

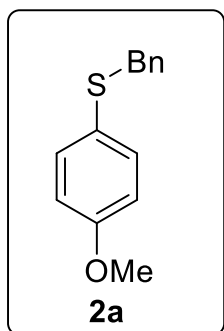
**Path 1:** The reaction gets initiated with the single-electron oxidation (SET) of phenol **1b** to generate the radical species **II**, which further undergoes anodic oxidation leading to carbocation **III**. Nucleophilic attack of sulfide **2a** on carbocation **III** generates sulfonium cation intermediate **IV**. The active benzylic position of intermediate **IV** undergoes nucleophilic attack by hydroxide ion, which is formed by the cathodic reduction of water (moisture) and afford the species **V** with benzyl alcohol as a side product (detected by GC-MS and LC-MS). Species **V** further undergoes one-electron oxidation followed by the attack of water and another anodic oxidation to deliver the sulfoxide **3ba**.

**Path 2:** Like path 1, The reaction gets initiated with the single-electron oxidation (SET) of phenol **1b** to generate the radical species **II**. At the same time the sulfide **2a** undergoes one electron oxidation leading to radical cation **2a'**. The radical-radical coupling of intermediate **II** and **2a'** generates the intermediate **IV**. Further, The active benzylic position of intermediate **IV** undergoes nucleophilic attack by hydroxide ion, which is formed by the cathodic reduction of water (moisture) and afford the species **V** with benzyl alcohol as a side product (detected by GC-MS and LC-MS). Species **V** further undergoes one-electron oxidation followed by the attack of water and another anodic oxidation to deliver the sulfoxide **3ba**.

## H) Characterization data

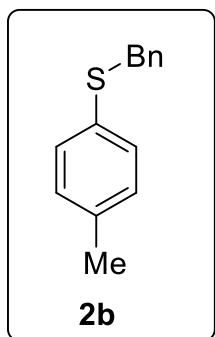
### i) Characterization data of the starting materials

#### Benzyl(4-methoxyphenyl)sulfane(2a)



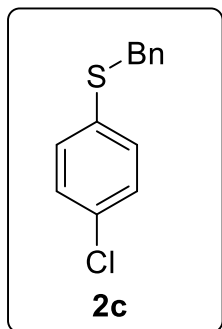
**Overall yield:** 90%, 0.438 g; **Nature:** White solid; **<sup>1</sup>H NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.27-7.20 (m, 5H), 7.19-7.17 (m, 2H), 6.80-6.76 (m, 2H), 3.97 (s, 2H), 3.77 (s, 3H).

#### Benzyl(p-tolyl)sulfane(2b)



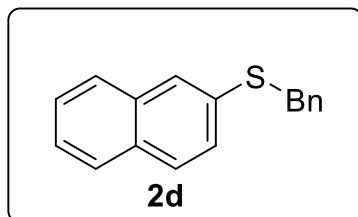
**Overall yield:** 88%, 0.467 g; **Nature:** White solid; **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.28-7.20 (m, 7H), 7.06 (d, J = 8.1 Hz, 1H), 4.07 (s, 2H), 2.31 (s, 3H).

### Benzyl(4-chlorophenyl)sulfane(2c)



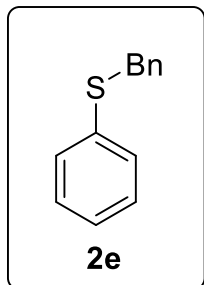
**Overall yield:** 92%, 0.450 g; **Nature:** White solid, **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.30-7.22 (m, 5H), 7.20 (s, 4H), 4.07 (s, 2H).

### Benzyl(naphthalen-2-yl)sulfane(2d)



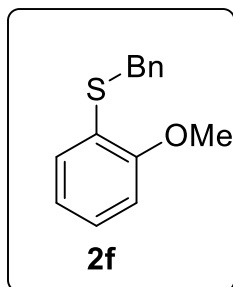
**Overall yield:** 85%, 0.402 g; **Nature:** White solid, **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.79-7.70 (m, 4H), 7.48-7.40 (m, 3H), 7.35-7.23 (m, 5H), 4.23 (s, 2H).

### Benzyl(phenyl)sulfane(2e)



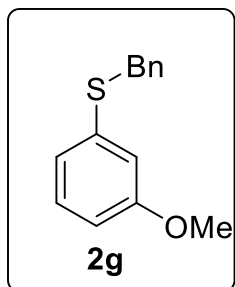
**Overall yield:** 95%, 0.515g; **Nature:** White solid;  **$^1\text{H NMR}$**  (400 MHz, CHLOROFORM-D):  $\delta$  7.33-7.20 (m, 4H), 7.20-7.15 (m, 1H), 4.11 (s, 1H).

### benzyl(2-methoxyphenyl)sulfane(2f)



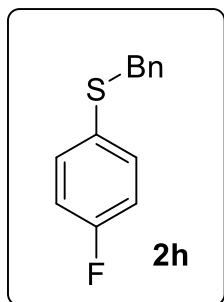
**Overall yield:** 87%, 0.430g; **Nature:** White solid;  **$^1\text{H NMR}$**  (400 MHz, CHLOROFORM-D):  $\delta$  7.31-7.16 (m, 7H), 6.86 (m, 2H), 4.09 (s, 2H), 3.88 (s, 1H).

**benzyl(3-methoxyphenyl)sulfane (2g)**



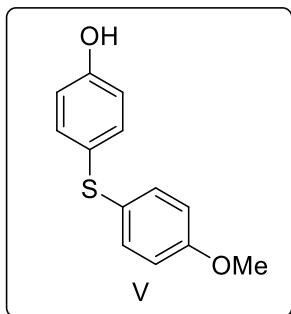
**Overall yield:** 81%, 0.4g; **Nature:** Transparent liquid;  **$^1\text{H NMR}$**  (400 MHz, CHLOROFORM-D):  $\delta$  7.33-7.21 (m, 5H), 7.19-7.14 (m, 1H), 6.91-6.88 (m, 1H), 6.82-6.81 (m, 1H), 6.73-6.70 (m, 1H), 4.12 (s, 2H), 3.73 (s, 3H).

**benzyl(4-fluorophenyl)sulfane (2h)**



**Overall yield:** 68%, 0.350g; **Nature:** Transparent liquid;  **$^1\text{H NMR}$**  (400 MHz, CHLOROFORM-D):  $\delta$  7.29-7.19 (m, 7H), 6.96-6.91 (m, 2H), 4.02 (s, 2H).

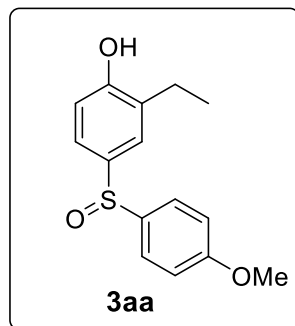
### 4-((4-methoxyphenyl)thio)phenol(V)<sup>3</sup>



**Overall yield:** 65%, 0.540g; **Nature:** yellow liquid; **<sup>1</sup>H NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.81 (d,  $J = 9.0$  Hz, 2H), 7.75 (d,  $J = 6.8$  Hz, 2H), 6.93 (d,  $J = 8.9$  Hz, 2H), 6.88 (d,  $J = 8.7$  Hz, 2H), 3.82 (s, 3H).

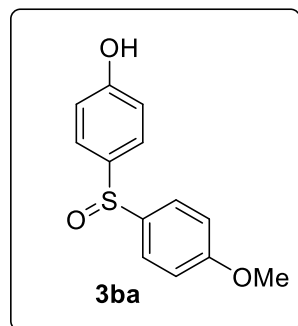
## ii) Characterization data of the products

### 2-ethyl-4-((4-methoxyphenyl)sulfinyl)phenol(3aa)



**Overall yield:** 60%, 41 mg; **Nature:** Brown liquid; **R<sub>f</sub>** = 0.4 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D): <sup>1</sup>H NMR (400 MHz, CHLOROFORM-D); δ 7.27-7.23 (m, 2H), 7.16 (d, *J* = 2.3 Hz, 1H), 7.04 (dd, *J* = 8.3, 2.3 Hz, 1H), 6.85-6.80 (m, 1H), 6.68 (d, *J* = 8.3 Hz, 1H), 4.80 (s, 1H), 3.78 (s, 3H), 2.57 (q, *J* = 7.5 Hz, 1H), 1.19 (t, *J* = 7.6 Hz, 1H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D): δ 158.9, 153.0, 132.8, 132.5, 131.1, 130.5, 127.8, 127.2, 116.1, 114.8, 55.4, 22.9, 13.9; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>17</sub>O<sub>3</sub>S 277.0898, found 277.0894.

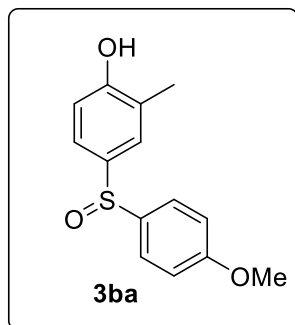
### 4-((4-methoxyphenyl)sulfinyl)phenol(3ba)<sup>2a,b</sup>



**Overall yield:** 62%, 49 mg; **Nature:** Brown semisolid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D): δ 7.28-7.26 (m, 2H), 7.22-7.19 (m, 2H), 6.84-6.81 (m, 2H), 6.76-6.71 (m, 2H), 4.87 (s, 1H), 3.78 (s, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D): δ 159.1, 154.1, 132.9, 127.8, 127.3, 116.3, 114.8, 55.4; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>13</sub>H<sub>13</sub>O<sub>3</sub>S 249.0585, found 249.580.

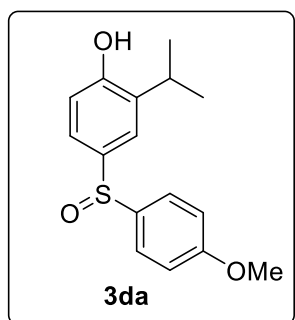


#### 4-((4-methoxyphenyl)sulfinyl)-2-methylphenol(3ca)



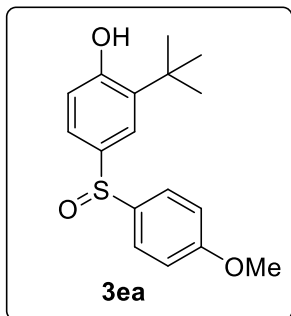
**Overall yield:** 63%, 49 mg; **Nature:** Brown liquid; **R<sub>f</sub>** = 0.4 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D): <sup>1</sup>H NMR (400 MHz, CHLOROFORM-D) δ 7.27-7.24 (m, 2H), 7.13 (d, *J* = 2.1 Hz, 1H), 7.06 (dd, *J* = 8.2, 2.3 Hz, 1H), 6.84-6.80 (m, 2H), 6.69 (d, *J* = 8.3 Hz, 1H), 4.83 (s, 1H), 3.78 (s, 3H), 2.19 (s, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D): δ 158.9, 153.4, 134.3, 132.6, 130.6, 127.7, 127.1, 124.9, 115.7, 114.8, 55.4, 15.8; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>15</sub>O<sub>3</sub>S 263.0742, found 263.0743.

#### 2-isopropyl-4-((4-methoxyphenyl)sulfinyl)phenol(3da)



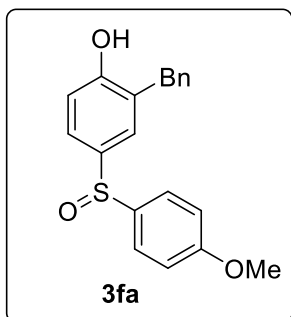
**Overall yield:** 50%, 32 mg; **Nature:** light brown liquid; **R<sub>f</sub>** = 0.4 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D): <sup>1</sup>H NMR (400 MHz, CHLOROFORM-D) δ 7.26-7.22 (m, 3H), 7.01 (dd, *J* = 8.3, 2.3 Hz, 1H), 6.84-6.80 (m, 1H), 6.66 (d, *J* = 8.3 Hz, 1H), 4.81 (s, 1H), 3.78 (s, 1H), 3.14 (dt, *J* = 13.8, 6.9 Hz, 1H), 1.21 (d, *J* = 6.9 Hz, 6H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D): δ 158.9, 152.3, 135.5, 132.4, 130.1, 127.7, 127.3, 116.2, 114.8, 55.4, 27.2, 22.6; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>19</sub>O<sub>3</sub>S 291.1055, found 291.1051.

### 2-(tert-butyl)-4-((4-methoxyphenyl)sulfinyl)phenol(3ea)



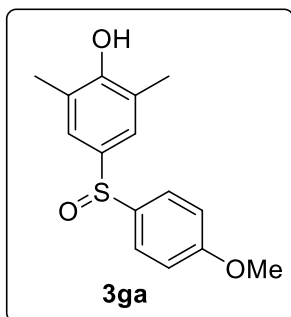
**Overall yield:** 55%, 34 mg; **Nature:** light brown liquid; **R<sub>f</sub>** = 0.4 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.29 – 7.25 (m, 3H), 7.00 (dd,  $J$  = 8.1, 2.3 Hz, 1H), 6.82 (d,  $J$  = 9.0 Hz, 2H), 6.58 (d,  $J$  = 8.1 Hz, 1H), 4.90 (s, 1H), 3.78 (s, 3H), 1.35 (s, 9H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D):  $\delta$  158.9, 153.8, 137.1, 132.6, 130.7, 130.1, 127.6, 126.9, 117.5, 114.8, 55.4, 34.8, 29.5; ; **HRMS** (ESI, Q-TOF)  $m/z$  [M + H]<sup>+</sup> Calcd for C<sub>17</sub>H<sub>21</sub>O<sub>3</sub>S 305.1211, found 305.1212.

### 2-benzyl-4-((4-methoxyphenyl)sulfinyl)phenol(3fa)



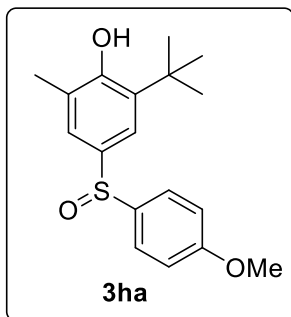
**Overall yield:** 55%, 31 mg; **Nature:** Brown liquid; **R<sub>f</sub>** = 0.4 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.29-7.25 (m, 4H), 7.22-7.17 (m, 3H), 7.13 (d,  $J$  = 2.3 Hz, 1H), 7.07 (dd,  $J$  = 8.3, 2.3 Hz, 1H), 6.83-6.81 (m, 2H), 6.70 (d,  $J$  = 8.3 Hz, 1H), 4.74 (s, 1H), 3.92 (s, 2H), 3.78 (s, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D): 159.0, 153.2, 139.4, 133.9, 132.9, 130.9, 128.7, 128.0, 127.8, 127.3, 126.6, 116.7, 114.8, 55.4, 36.4; **HRMS** (ESI, Q-TOF)  $m/z$  [M + H]<sup>+</sup> Calcd for C<sub>20</sub>H<sub>19</sub>O<sub>3</sub>S 339.1055, found 339.1050.

#### 4-((4-methoxyphenyl)sulfinyl)-2,6-dimethylphenol(3ga)



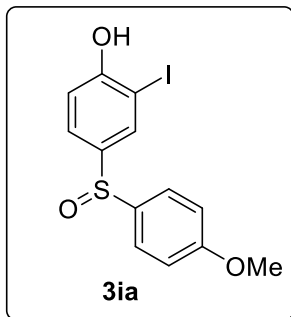
**Overall yield:** 47%, 32 mg; **Nature:** light brown liquid,  $R_f = 0.5$  (Hexane/ethyl acetate = 85:15);  **$^1\text{H-NMR}$**  (400 MHz, CHLOROFORM-D):  $\delta$  7.27-7.23 (m, 2H), 7.00 (s, 2H), 6.84-6.80 (m, 2H), 4.63 (s, 1H), 3.78 (s, 3H), 2.18 (s, 6H);  **$^{13}\text{C}\{^1\text{H}\}$  NMR** (101 MHz, CHLOROFORM-D):  $\delta$  158.8, 151.9, 132.4, 132.1, 127.9, 126.2, 124.1, 114.8, 55.4, 15.9; **HRMS** (ESI, Q-TOF)  $m/z$  [M + H]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>17</sub>O<sub>3</sub>S 277.0898, found 277.0895.

#### 2-(tert-butyl)-4-((4-methoxyphenyl)sulfinyl)-6-methylphenol(3ha)



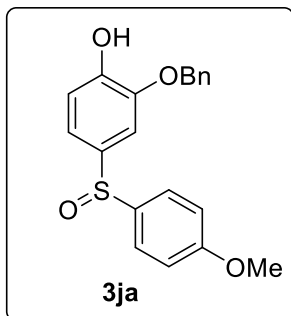
**Overall yield:** 55%, 31 mg; **Nature:** Brown liquid;  $R_f = 0.5$  (Hexane/ethyl acetate = 95:5);  **$^1\text{H-NMR}$**  (400 MHz, CHLOROFORM-D):  $\delta$  7.26-7.24 (m, 2H), 7.18 (d,  $J = 2.3$  Hz, 1H), 6.97 (d,  $J = 2.3$  Hz, 1H), 6.84-6.81 (m, 2H), 4.77 (s, 1H), 3.78 (s, 1H), 2.18 (s, 1H), 1.36 (s, 9H);  **$^{13}\text{C}\{^1\text{H}\}$  NMR** (101 MHz, CHLOROFORM-D):  $\delta$  158.8, 152.4, 136.7, 132.3, 131.8, 128.9, 127.9, 125.9, 124.2, 114.7, 55.4, 34.7, 29.7, 16.0; **HRMS** (ESI, Q-TOF)  $m/z$  [M + H]<sup>+</sup> Calcd for C<sub>18</sub>H<sub>23</sub>O<sub>3</sub>S 319.1368, found 319.1365.

### 2-iodo-4-((4-methoxyphenyl)sulfinyl)phenol(3ia)



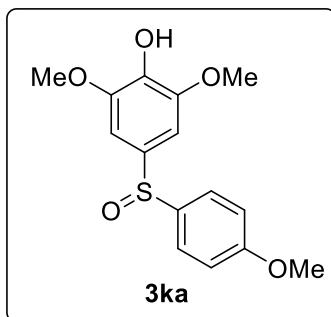
**Overall yield:** 60%, 26 mg; **Nature:** Brown liquid; **R<sub>f</sub>** = 0.4 (Hexane/ethyl acetate = 85:15); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.59 (d, *J* = 2.2 Hz, 1H), 7.32-7.28 (m, 2H), 7.17 (dd, *J* = 8.4, 2.2 Hz, 1H), 6.87 (dd, *J* = 13.9, 8.7 Hz, 3H), 5.27 (s, 1H), 3.79 (s, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D):  $\delta$  159.5, 154.0, 139.8, 133.5, 132.8, 129.0, 128.3, 115.0, 114.6, 55.4; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>13</sub>H<sub>12</sub>O<sub>3</sub>SI 374.9552, found 374.9554.

### 2-(benzyloxy)-4-((4-methoxyphenyl)sulfinyl)phenol(3ja)



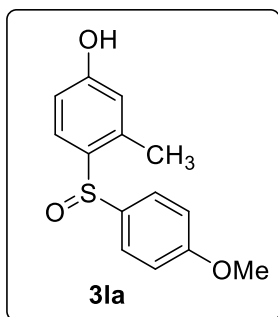
**Overall yield:** 52%, 28 mg; **Nature:** light brown liquid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 85:15); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.38-7.34 (m, 5H), 7.24 (dd, *J* = 6.7, 2.2 Hz, 3H), 6.93-6.86 (m, 2H), 6.83-6.80 (m, 2H), 5.65 (s, 1H), 5.01 (s, 2H), 3.79 (s, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D):  $\delta$  159.4, 146.0, 144.7, 136.2, 133.5, 128.7, 127.8, 124.4, 122.3, 120.1, 114.9, 112.3, 71.4, 55.4. ; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>20</sub>H<sub>19</sub>O<sub>4</sub>S 355.1004, found 355.0997.

### 2,6-dimethoxy-4-((4-methoxyphenyl)sulfinyl)phenol(3ka)



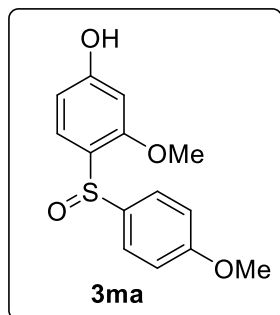
**Overall yield:** 55%, 33 mg; **Nature:** Brown liquid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 85:15); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D): δ 7.32-7.28 (m, 2H), 6.86-6.82 (m, 2H), 6.57 (q, *J* = 8.7 Hz, 2H), 5.62 (s, 1H), 3.89 (s, 3H), 3.84 (s, 3H), 3.78 (s, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D): δ 159.2, 147.3, 145.6, 139.1, 133.6, 125.7, 122.7, 121.9, 114.9, 107.2, 60.8, 56.4, 55.4; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>17</sub>O<sub>5</sub>S 309.0797, found 309.0793.

### 4-((4-methoxyphenyl)sulfinyl)-3-methylphenol(3la)



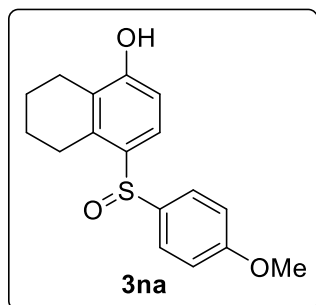
**Overall yield:** 48%, 35 mg; **Nature:** light brown liquid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 85:15); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D): δ 7.19 (d, *J* = 8.4 Hz, 1H), 7.14-7.10 (m, 2H), 6.82-6.78 (m, 2H), 6.72 (d, *J* = 2.8 Hz, 1H), 6.61 (dd, *J* = 8.4, 2.8 Hz, 1H), 4.91 (s, 1H), 3.77 (s, 3H), 2.31 (s, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D): δ 158.5, 155.5, 142.2, 135.1, 131.2, 127.7, 125.7, 117.6, 114.8, 113.8, 55.5, 20.8; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>15</sub>O<sub>3</sub>S 263.0742, found 263.0741.

### 3-methoxy-4-((4-methoxyphenyl)sulfinyl)phenol(3ma)



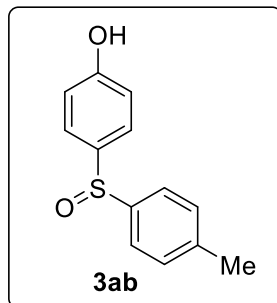
**Overall yield:** 40%, 27 mg; **Nature:** light brown liquid;  $R_f = 0.4$  (Hexane/ethyl acetate = 85:15);  $^1\text{H-NMR}$  (400 MHz, CHLOROFORM-D):  $\delta$  7.24 (d,  $J = 4.5$  Hz, 1H), 6.99 (d,  $J = 8.3$  Hz, 1H), 6.84-6.80 (m, 2H), 6.43 (d,  $J = 2.5$  Hz, 1H), 6.33 (dd,  $J = 8.3, 2.6$  Hz, 1H), 5.07 (s, 1H), 3.80 (s, 3H), 3.78 (s, 3H);  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, CHLOROFORM-D):  $\delta$  158.9, 158.9, 156.7, 133.6, 132.8, 126.2, 115.6, 114.8, 107.9, 99.6, 77.4, 77.1, 76.8, 56.0, 55.4; **HRMS** (ESI, Q-TOF)  $m/z$   $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{14}\text{H}_{15}\text{O}_4\text{S}$  279.0691, found 279.0692.

### 4-((4-methoxyphenyl)sulfinyl)-5,6,7,8-tetrahydronaphthalen-1-ol(3na)



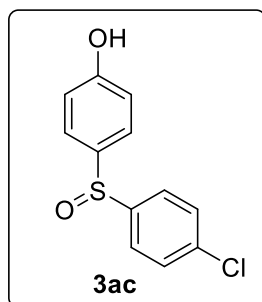
**Overall yield:** 45%, 28 mg; **Nature:** light brown liquid;  $R_f = 0.5$  (Hexane/ethyl acetate = 9:1);  $^1\text{H-NMR}$  (400 MHz, CHLOROFORM-D):  $\delta$  7.15-7.12 (m, 2H), 7.04 (d,  $J = 8.4$  Hz, 1H), 6.83-6.79 (m, 2H), 6.58 (d,  $J = 8.3$  Hz, 1H), 4.86 (s, 1H), 3.77 (s, 3H), 2.75 (t,  $J = 5.7$  Hz, 2H), 2.62 (t,  $J = 5.8$  Hz, 2H), 1.79-1.71 (m, 4H);  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, CHLOROFORM-D):  $\delta$  158.5, 153.6, 140.5, 131.4, 131.4, 127.5, 125.8, 124.8, 114.8, 112.6, 55.4, 28.07, 23.4, 22.7, 22.1; **HRMS** (ESI, Q-TOF)  $m/z$   $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{17}\text{H}_{19}\text{O}_3\text{S}$  303.1055, found 303.1045

#### 4-(p-tolylsulfinyl)phenol(3bb)



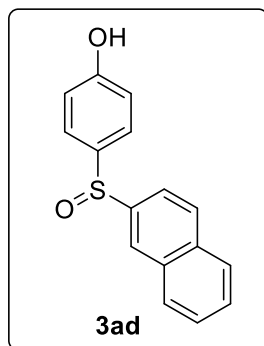
**Overall yield:** 58%, 43 mg; **Nature:** light brown liquid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.32-7.28 (m, 2H), 7.13-7.11 (m, 2H), 7.05 (d,  $J$  = 8.1 Hz, 2H), 6.83-6.77 (m, 2H), 4.96 (s, 1H), 2.29 (s, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D):  $\delta$  155.5, 136.3, 134.6, 129.9, 129.5, 125.9, 116.4, 21.1; **HRMS** (ESI, Q-TOF)  $m/z$  [M + H]<sup>+</sup> Calcd for C<sub>13</sub>H<sub>13</sub>O<sub>2</sub>S 233.0636, found 233.0628.

#### 4-((4-chlorophenyl)sulfinyl)phenol(3bc)



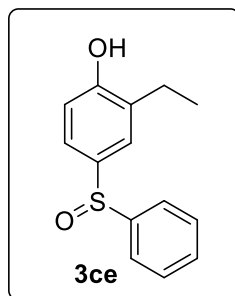
**Overall yield:** 48%, 39 mg; **Nature:** light brown liquid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D):  $\delta$  7.36 – 7.33 (m, 1H), 7.20 – 7.17 (m, 1H), 7.08 – 7.05 (m, 1H), 6.84 – 6.81 (m, 1H), 4.98 (s, 1H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D):  $\delta$  156.2, 135.8, 130.1, 129.3, 129.1, 128.5, 116.7, 115.3; **HRMS** (ESI, Q-TOF)  $m/z$  [M + H]<sup>+</sup> Calcd for C<sub>12</sub>H<sub>10</sub>O<sub>2</sub>SCl 253.0090, found 253.0082.

### 4-(naphthalen-2-ylsulfinyl)phenol(3bd)



**Overall yield:** 38%, 32 mg; **Nature:** light brown semisolid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D): δ 7.75 (d, *J* = 7.2 Hz, 1H), 7.68 (dd, *J* = 17.9, 8.0 Hz, 2H), 7.59 (s, 1H), 7.45-7.38 (m, 4H), 7.28 (dd, *J* = 8.6, 1.8 Hz, 1H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.96 (s, 1H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D): δ 155.9, 135.8, 135.5, 133.8, 131.81, 128.6, 127.8, 127.2, 126.8, 126.6, 125.8, 124.8, 116.6; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>13</sub>O<sub>2</sub>S 269.0636, found 269.0627.

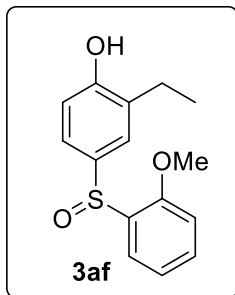
### 2-ethyl-4-(phenylsulfinyl)phenol(3ae)



**Overall yield:** 60%, 36 mg; **Nature:** Brown liquid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D): δ 7.28 (d, *J* = 2.2 Hz, 1H), 7.24-7.19 (m, 3H), 7.17-7.10 (m, 3H), 6.74 (t, *J* = 6.7 Hz, 1H), 4.93 (s, 1H), 2.60 (q, *J* = 7.6 Hz, 2H), 1.21 (t, *J* = 7.6 Hz, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D): δ 153.9, 138.8, 135.3, 133.2, 131.3, 128.9, 128.1, 125.7, 124.2, 116.3, 22.9, 13.8; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>15</sub>O<sub>2</sub>S 247.0793, found 247.0793.

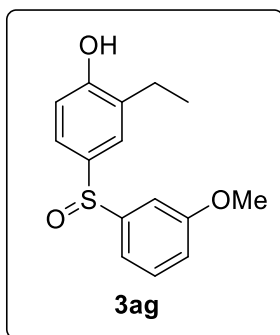


### 2-ethyl-4-((2-methoxyphenyl)sulfinyl)phenol (3af)



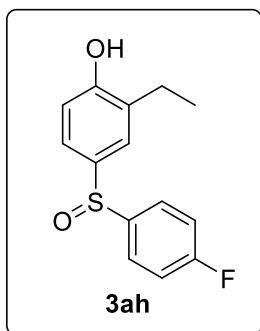
**Overall yield:** 65%, 44 mg; **Nature:** Brown liquid;  $R_f = 0.5$  (Hexane/ethyl acetate = 9:1);  **$^1\text{H-NMR}$**  (400 MHz, CHLOROFORM-D):  $\delta$  7.30 (d,  $J = 2.2$  Hz, 1H), 7.22 (dd,  $J = 8.2, 2.3$  Hz, 1H), 7.13-7.08 (m, 1H), 6.85-6.81 (m, 1H), 6.79-6.74 (m, 3H), 4.95 (s, 1H), 3.90 (s, 3H), 2.61 (q,  $J = 7.5$  Hz, 2H), 1.21 (t,  $J = 7.5$  Hz, 3H);  **$^{13}\text{C}\{^1\text{H}\}$  NMR** (101 MHz, CHLOROFORM-D):  $\delta$  155.5, 154.1, 131.4, 127.8, 126.4, 122.6, 121.3, 116.3, 110.3, 55.9, 22.9, 13.8; **HRMS** (ESI, Q-TOF)  $m/z$   $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{15}\text{H}_{17}\text{O}_3\text{S}$  277.0898, found 277.0894.

### 2-ethyl-4-((3-methoxyphenyl)sulfinyl)phenol (3ag)



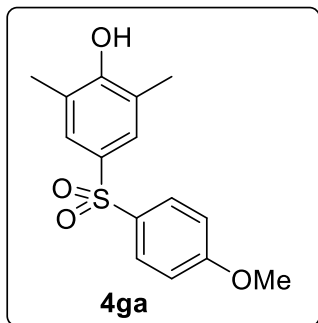
**Overall yield:** 63%, 43 mg; **Nature:** Brown liquid;  $R_f = 0.5$  (Hexane/ethyl acetate = 9:1);  **$^1\text{H-NMR}$**  (400 MHz, CHLOROFORM-D):  $\delta$  7.30-7.28 (m, 2H), 7.23-7.20 (m, 1H), 7.15-7.11 (m, 1H), 6.75 (d,  $J = 8.3$  Hz, 1H), 6.73-6.70 (m, 1H), 6.67-6.64 (m, 1H), 4.87 (s, 1H), 3.72 (s, 3H), 2.61 (q,  $J = 7.5$  Hz, 2H), 1.21 (t,  $J = 7.6$  Hz, 3H);  **$^{13}\text{C}\{^1\text{H}\}$  NMR** (101 MHz, CHLOROFORM-D):  $\delta$  160.0, 154.0, 140.4, 135.6, 133.4, 129.8, 123.7, 120.0, 116.2, 113.1, 111.2, 55.2, 22.9, 13.8; **HRMS** (ESI, Q-TOF)  $m/z$   $[\text{M} + \text{H}]^+$  Calcd for  $\text{C}_{15}\text{H}_{17}\text{O}_3\text{S}$  277.0898, found 277.0894.

**2-ethyl-4-((4-fluorophenyl)sulfinyl)phenol (3ah)**



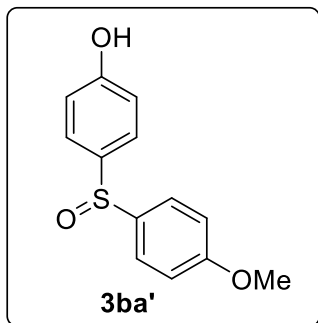
**Overall yield:** 50%, 32 mg; **Nature:** Brown liquid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D): δ 7.22 (d, *J* = 2.3 Hz, 1H), 7.20-7.12 (m, 3H), 6.97-6.92 (m, 2H), 6.73 (d, *J* = 8.3 Hz, 1H), 4.82 (s, 1H), 2.59 (q, *J* = 7.5 Hz, 2H), 1.20 (t, *J* = 7.5 Hz, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D): δ 162.8, 160.4, 153.7, 134.4, 132.2, 131.4, 130.9, 130.9, 125.2, 116.3, 116.2, 116.0, 22.9, 13.8; **HRMS** (ESI, Q-TOF) *m/z* [M + H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>14</sub>O<sub>2</sub>FS 265.0699, found 265.0674.

#### 4-((4-methoxyphenyl)sulfonyl)-2,6-dimethylphenol (4ga)



**Overall yield:** 70%, 15 mg; **Nature:** Transparent liquid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 70:); **<sup>1</sup>H-NMR** (400 MHz, CHLOROFORM-D):  $\delta$  = 7.54 -7.49 (m, 2H), 7.19 (s, 2H), 6.97-6.91 (m, 2H), 5.32 (s, 1H), 3.81 (s, 3H), 2.21 (s, 6H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D):  $\delta$  = 161.7, 155.0, 136.8, 135.8, 126.9, 125.7, 124.5, 114.7, 55.5, 16.1; **HRMS** (ESI, Q-TOF) m/z [M + H]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>17</sub>O<sub>4</sub>S 293.0848, found 293.0846.

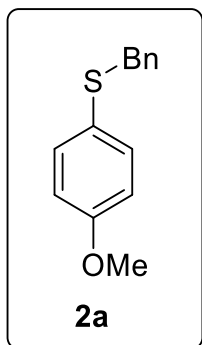
#### 4-((4-methoxyphenyl)sulfinyl)phenol (3ba')



**Overall yield:** 70%, 15 mg; **Nature:** Transparent liquid; **R<sub>f</sub>** = 0.5 (Hexane/ethyl acetate = 9:1); **<sup>1</sup>H NMR** (400 MHz, CHLOROFORM-D)  $\delta$  7.29-7.25 (m, 2H), 7.23-7.19 (m, 2H), 6.85-6.81 (m, 2H), 6.77-6.73 (m, 2H), 5.08 (s, 1H), 3.78 (s, 3H); **<sup>13</sup>C{<sup>1</sup>H} NMR** (101 MHz, CHLOROFORM-D):  $\delta$  163.2, 160.1, 129.8, 129.4, 116.1, 114.6, 55.7; **HRMS** (ESI, Q-TOF) m/z [M + H]<sup>+</sup> Calcd for C<sub>13</sub>H<sub>13</sub>O<sub>3</sub>S 249.0585, found 249.0584

### I) Copies of NMR, HRMS, GC-MS and LC-MS spectra of compounds

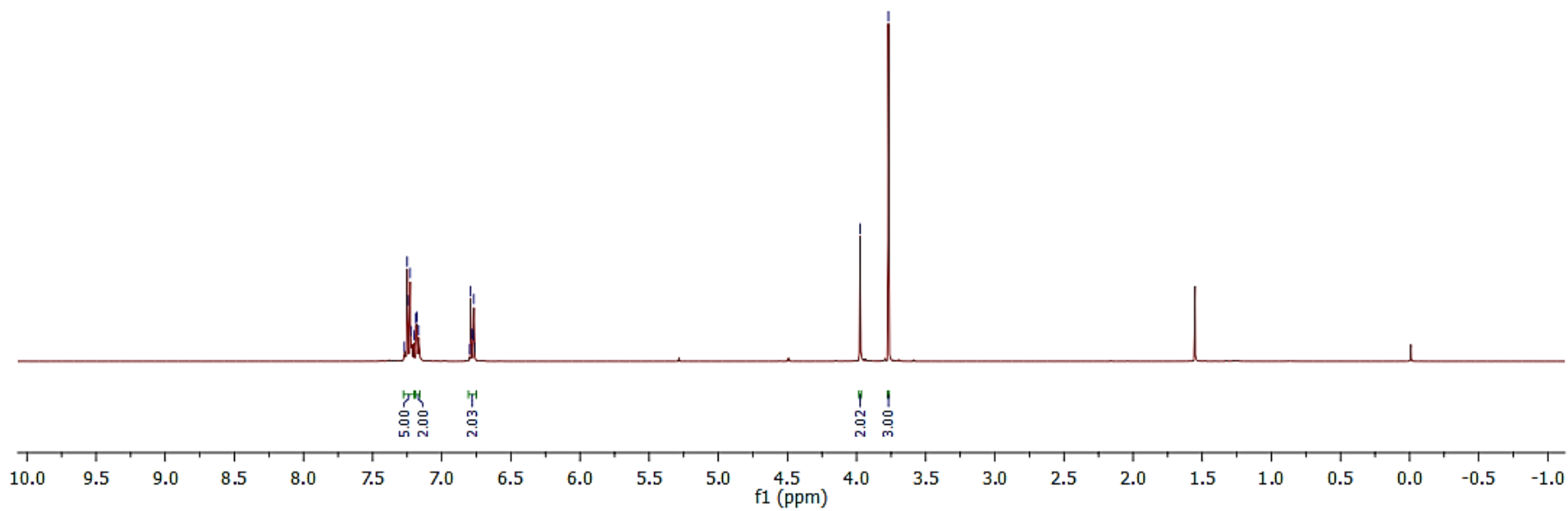
26-02-127



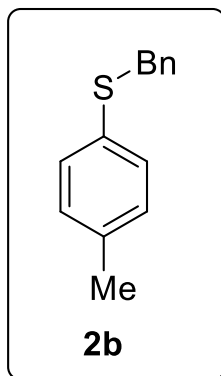
7.27  
7.25  
7.23  
7.22  
7.20  
7.19  
7.18  
7.17  
6.80  
6.79  
6.78  
6.77  
6.77  
6.76

3.97  
3.77

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 2a**



26-02-190

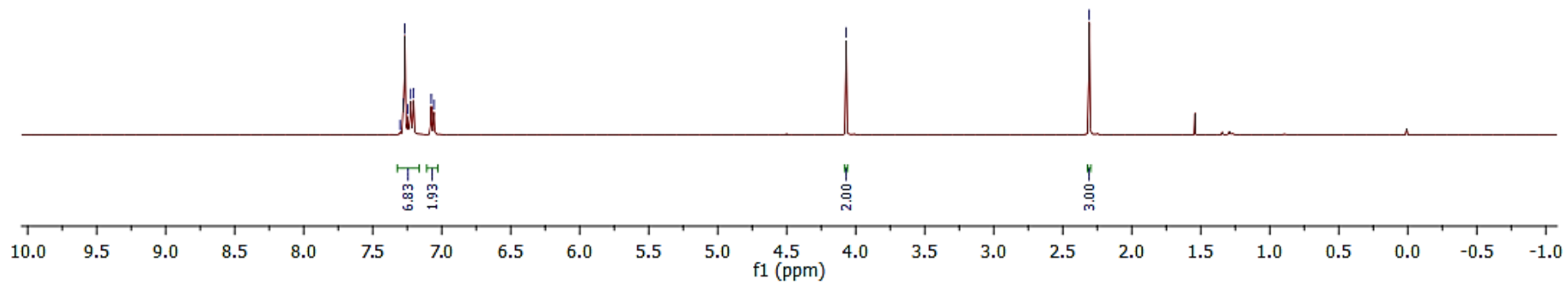


7.30  
7.28  
7.27  
7.25  
7.22  
7.20  
7.07  
7.05

4.07

2.31

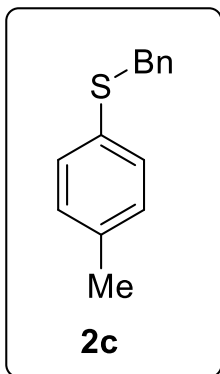
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 2b**



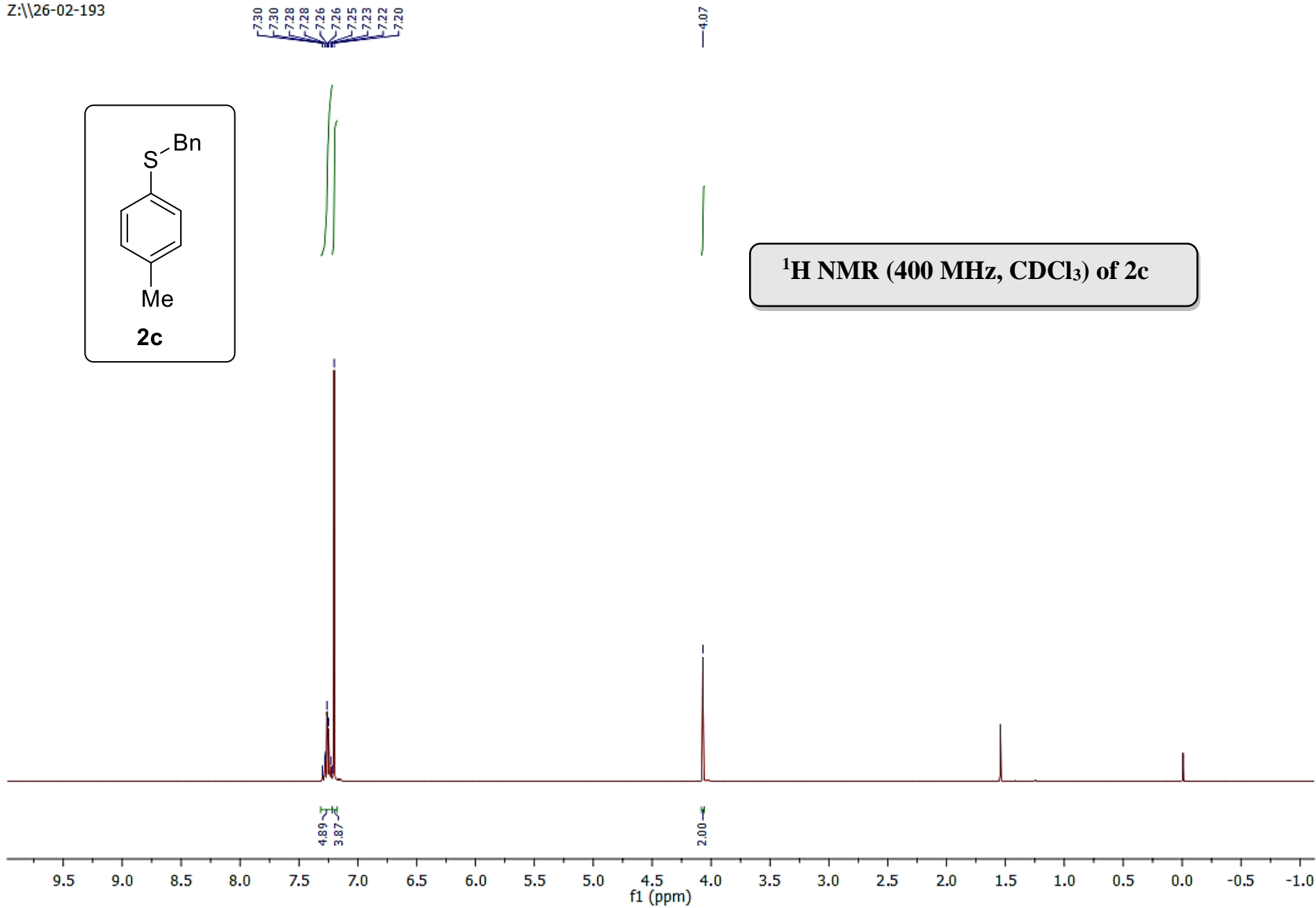
Z:\\26-02-193

7.30  
7.30  
7.28  
7.28  
7.26  
7.26  
7.25  
7.23  
7.22  
7.20

4.07



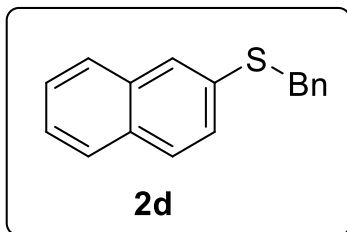
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 2c**



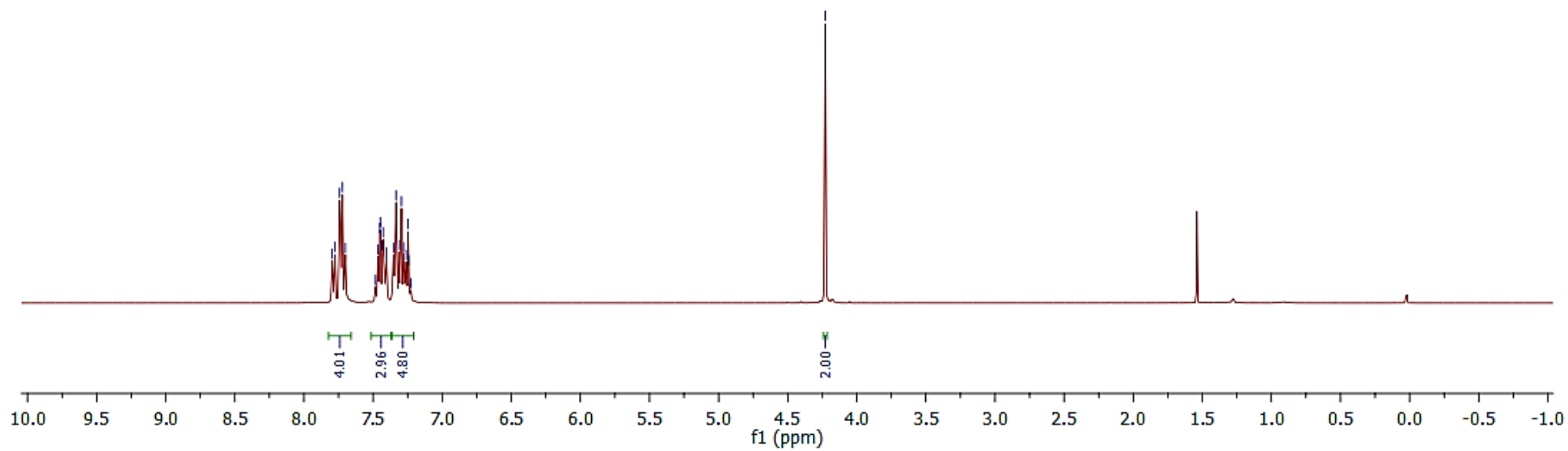
26-02-196

7.79  
7.78  
7.74  
7.72  
7.70  
7.48  
7.47  
7.45  
7.45  
7.43  
7.43  
7.40  
7.40  
7.35  
7.33  
7.31  
7.29  
7.28  
7.26  
7.25  
7.24  
7.23

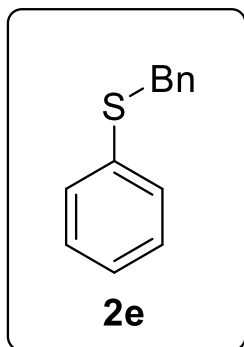
4.23



**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 2d**



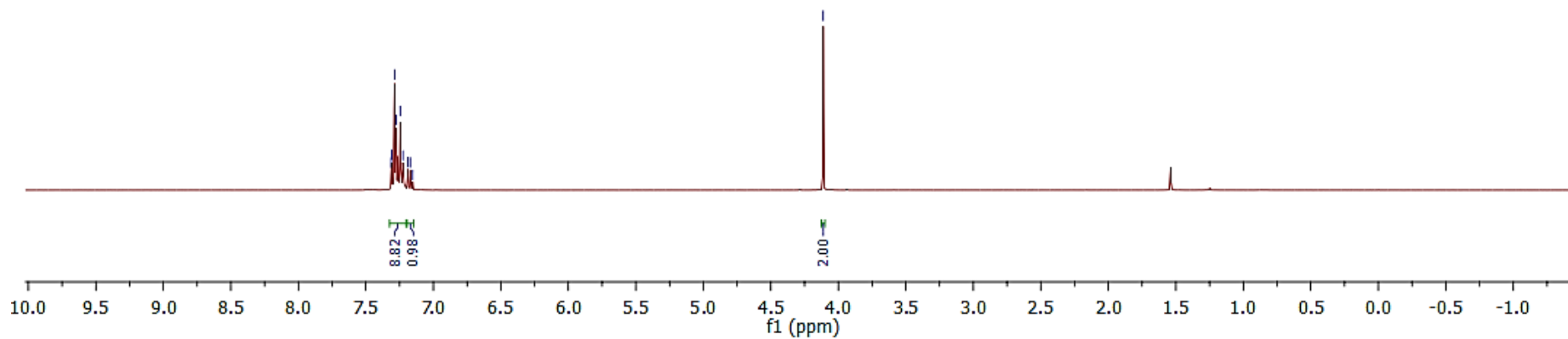
26-02-139



7.31  
7.31  
7.29  
7.28  
7.25  
7.23  
7.19  
7.17  
7.15

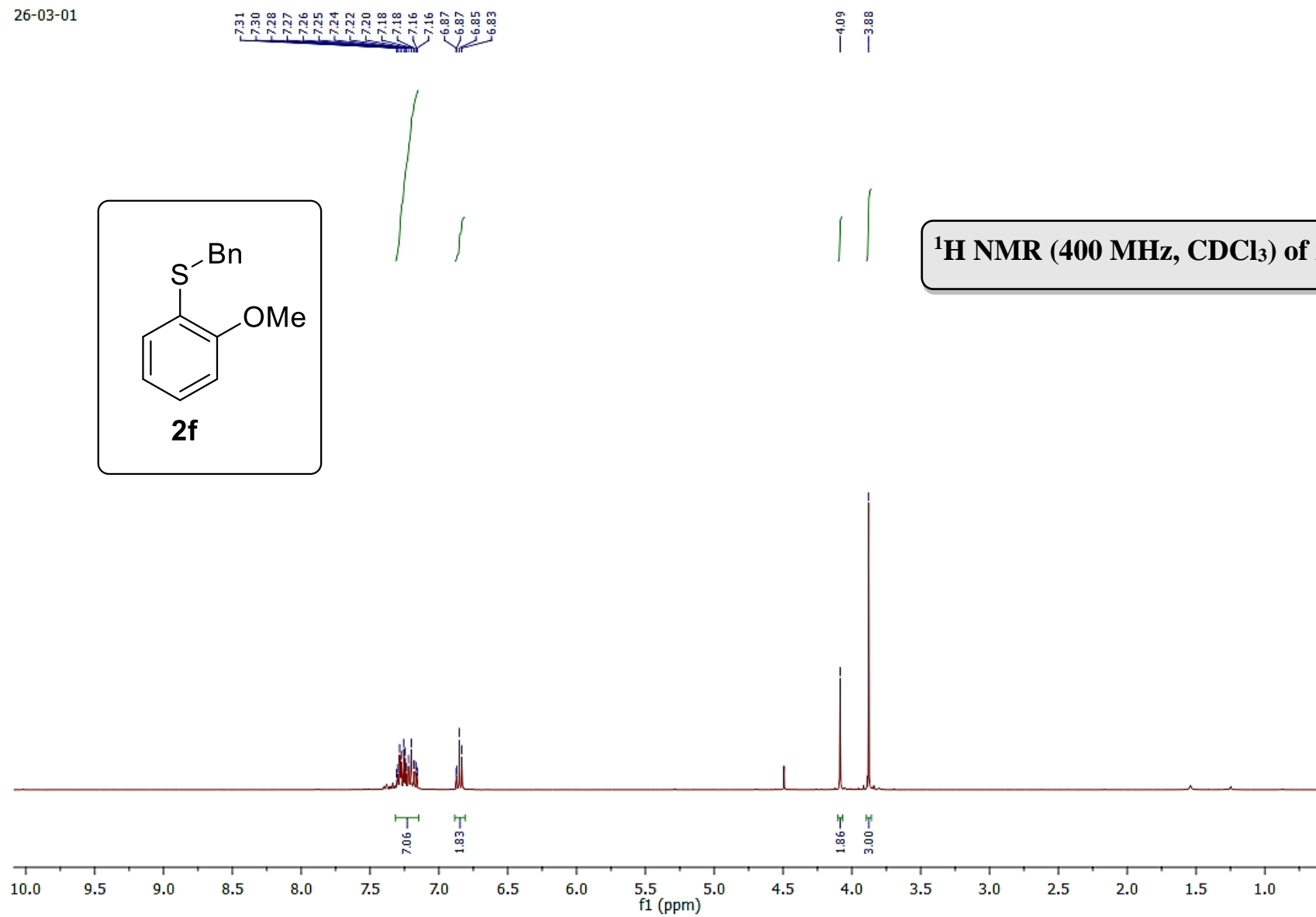
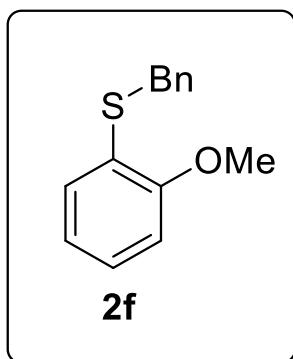
4.11

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 2e**

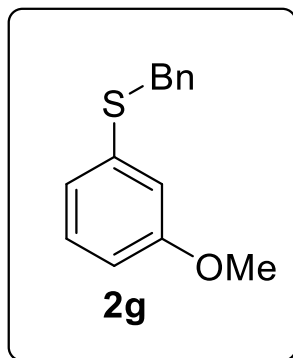




26-03-01



26-03-02

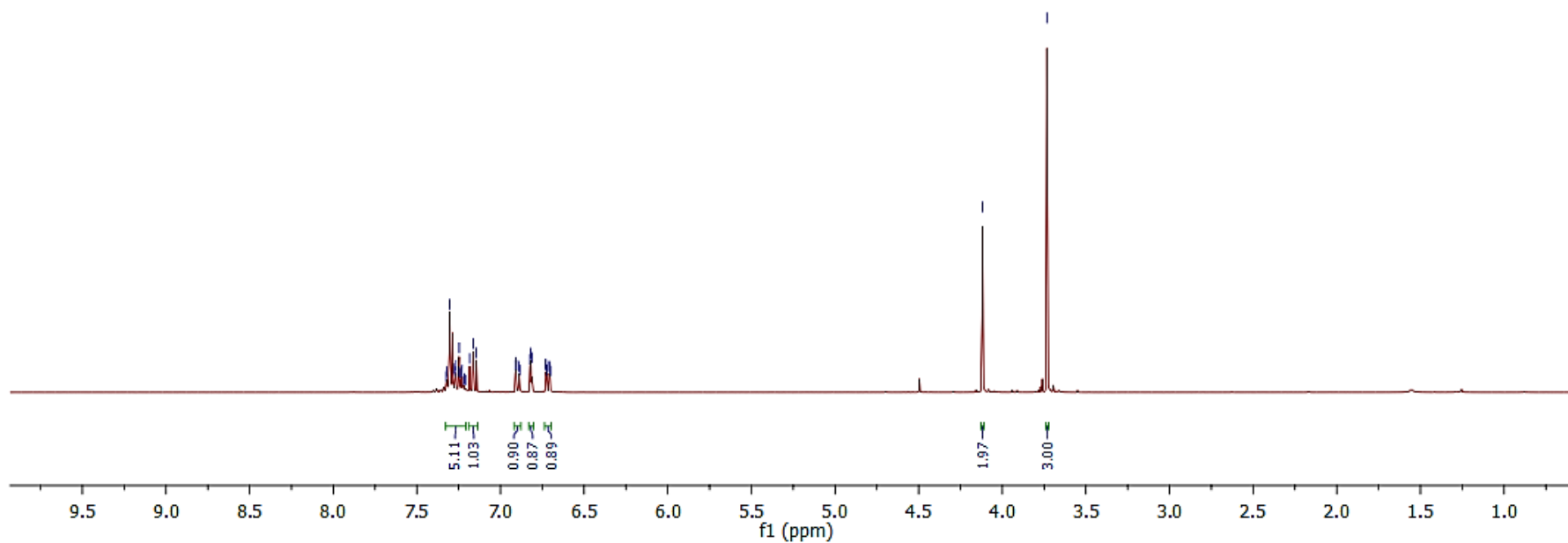


7.33  
7.32  
7.30  
7.28  
7.27  
7.27  
7.25  
7.25  
7.24  
7.23  
7.22  
7.22  
7.21  
7.18  
7.16  
7.14  
6.91  
6.91  
6.90  
6.89  
6.88  
6.82  
6.82  
6.81  
6.73  
6.73  
6.72  
6.72  
6.71  
6.71  
6.70  
6.70

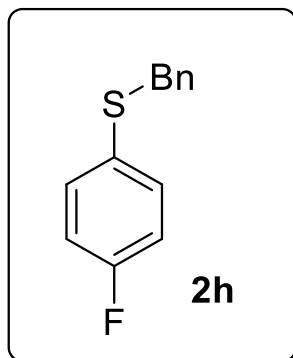
4.12

3.73

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 2g**



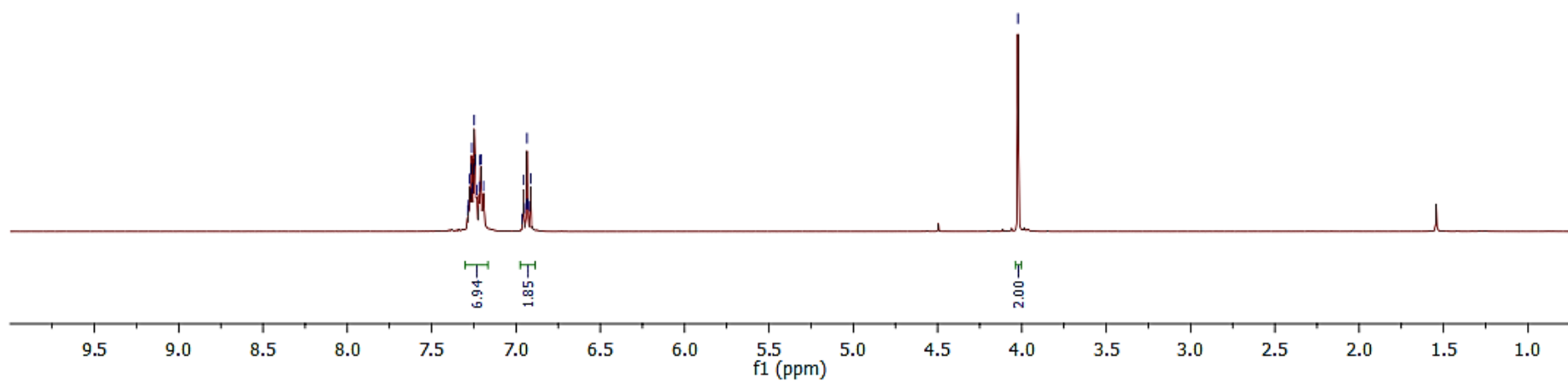
26-03-08



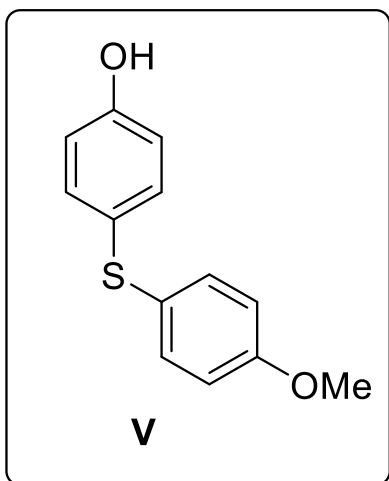
7.29  
7.28  
7.28  
7.27  
7.27  
7.26  
7.26  
7.25  
7.25  
7.24  
7.23  
7.22  
7.22  
7.21  
7.21  
7.19  
6.96  
6.96  
6.95  
6.94  
6.93  
6.93  
6.92  
6.91

4.02

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 2h**



26-03



7.82  
7.80  
7.76  
7.74

6.94  
6.92  
6.89  
6.87



**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of V**

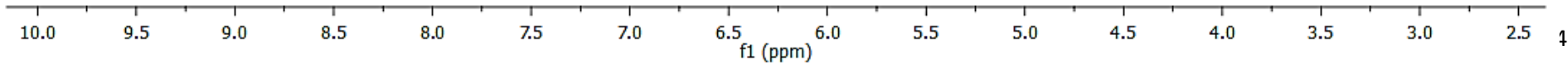
3.82



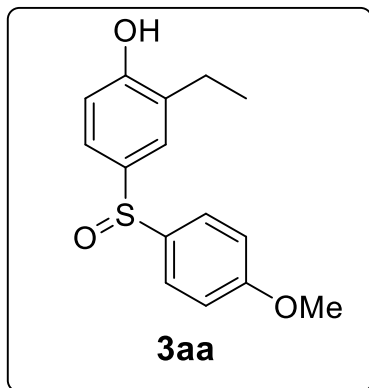
2.18  
2.19

2.17  
2.24

3.00



26-02-133RV



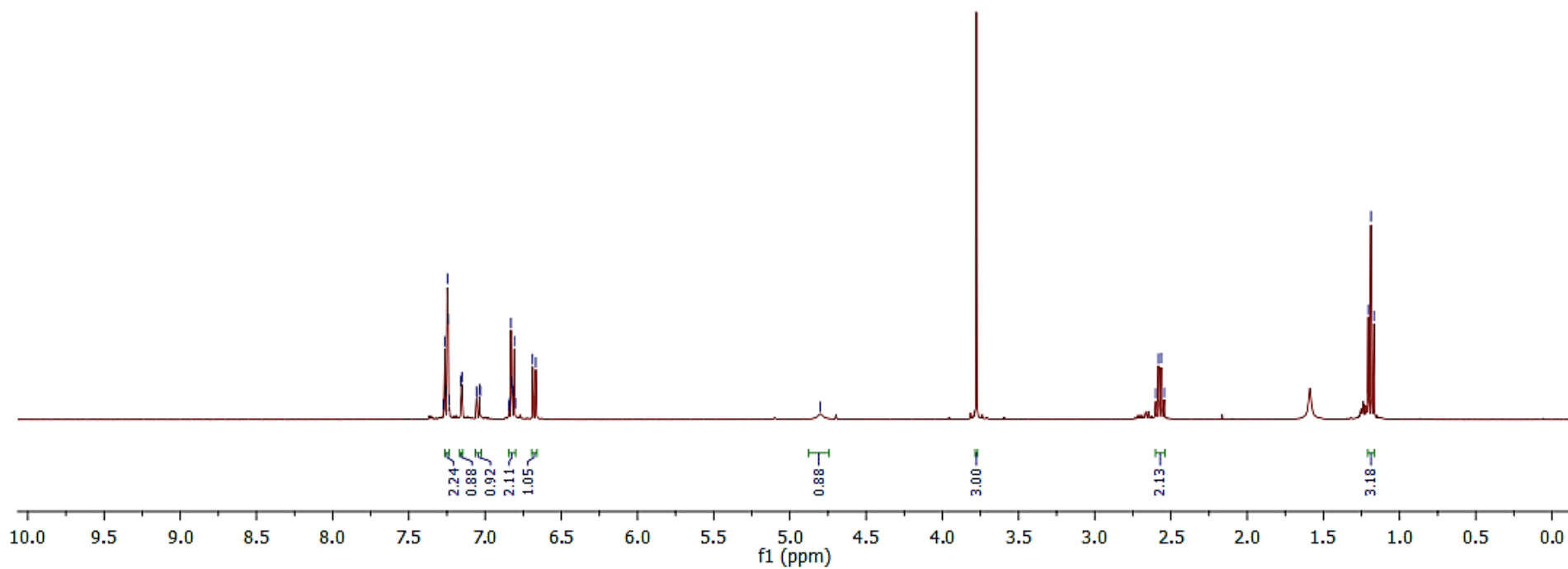
7.27  
7.26  
7.26  
7.25  
7.24  
7.23  
7.16  
7.15  
7.06  
7.05  
7.04  
7.03  
6.84  
6.84  
6.83  
6.82  
6.81  
6.80  
6.69  
6.67

4.80

2.60  
2.58  
2.56  
2.54

1.21  
1.19  
1.17

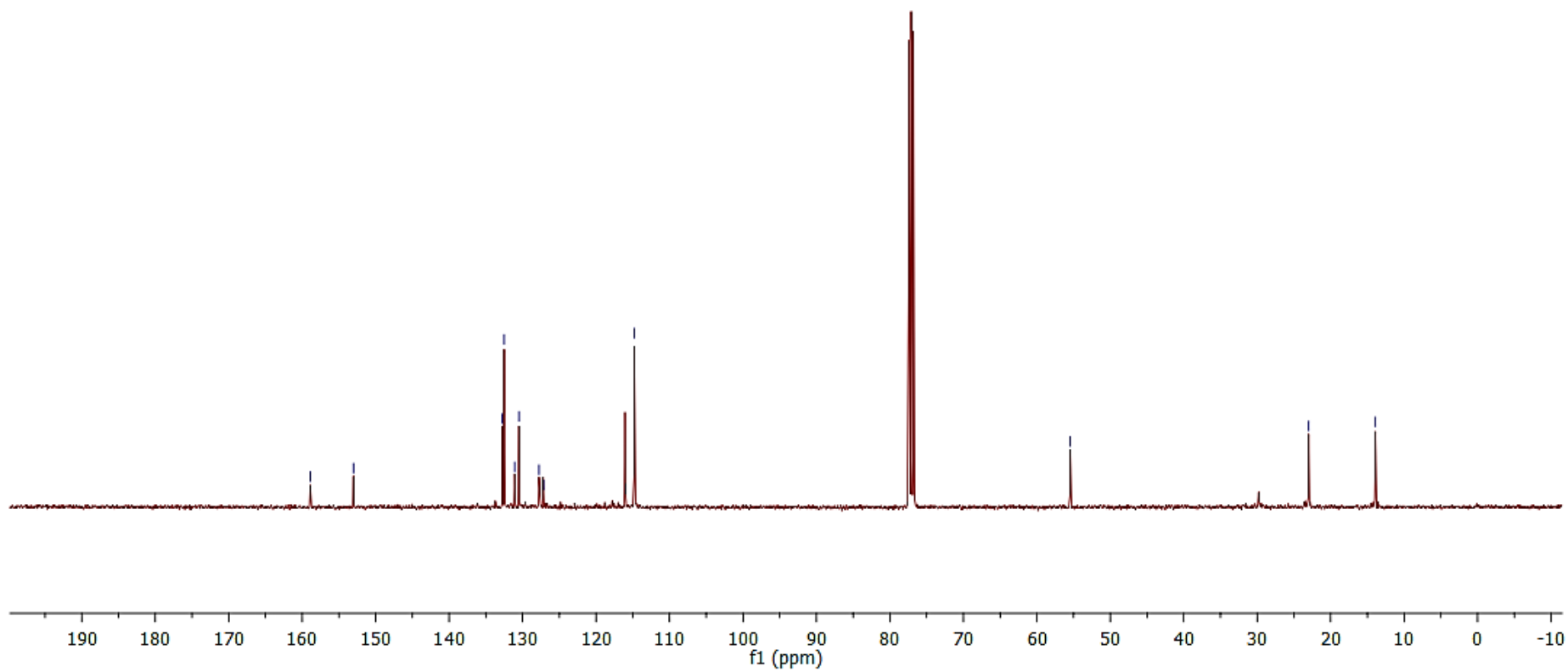
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3aa**



26-02-133

158.90  
153.02  
132.76  
132.51  
131.08  
130.49  
127.76  
127.14  
115.95  
114.79  
55.44  
22.99  
13.89

**$^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz,  $\text{CDCl}_3$ ) of 3aa**



Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

282 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-5 O: 0-6 S: 0-1

Sample Name : 26\_02\_125

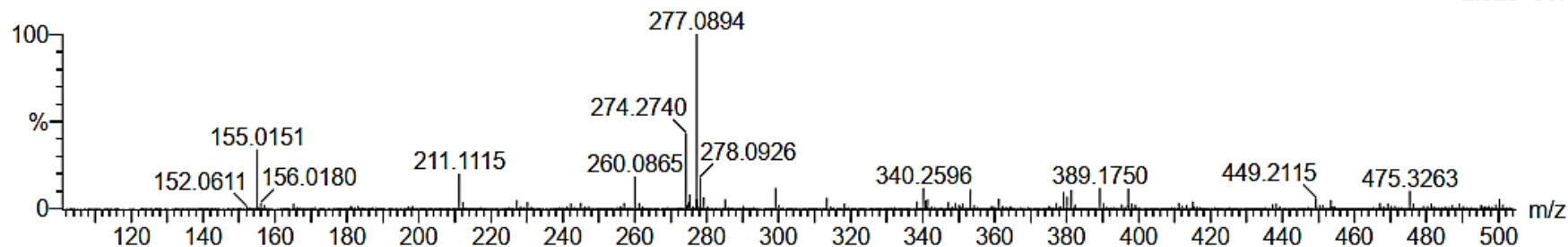
IITRPR

XEVO G2-XS QTOF

Test Name :

01042022\_26\_02\_125 5 (0.124)

1: TOF MS ES+  
2.32e+007



Minimum: -1.5  
Maximum: 2.0 10.0 50.0

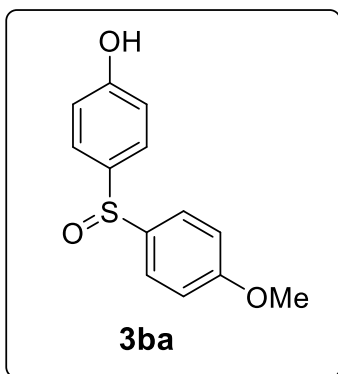
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
277.0894	277.0898	-0.4	-1.4	7.5	1051.2	n/a	n/a	C15 H17 O3 S

26-02-170

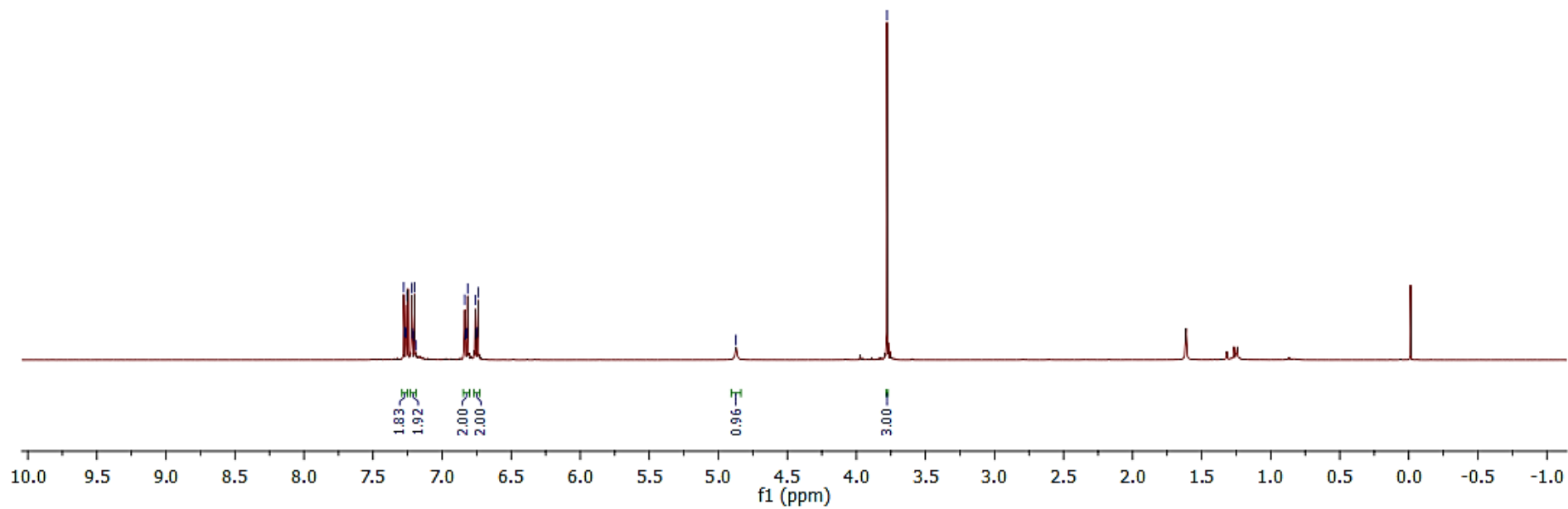
7.28  
7.27  
7.26  
7.26  
7.22  
7.22  
7.20  
7.20  
7.19  
6.84  
6.83  
6.82  
6.81  
6.76  
6.76  
6.74  
6.74

4.87

3.78

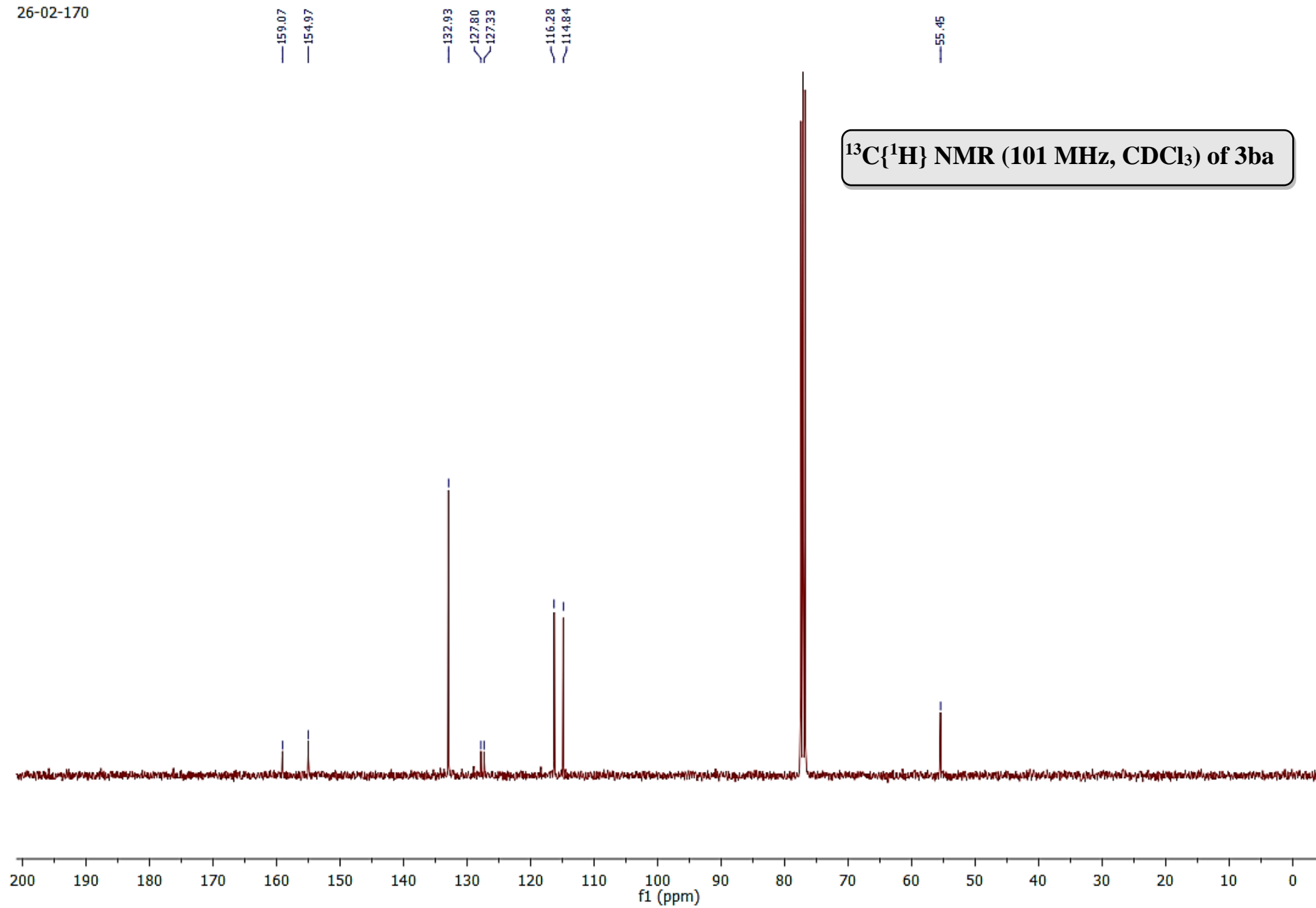


**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3ba**





26-02-170



Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

85 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 O: 0-10 S: 0-2

Sample Name : 26\_02\_170

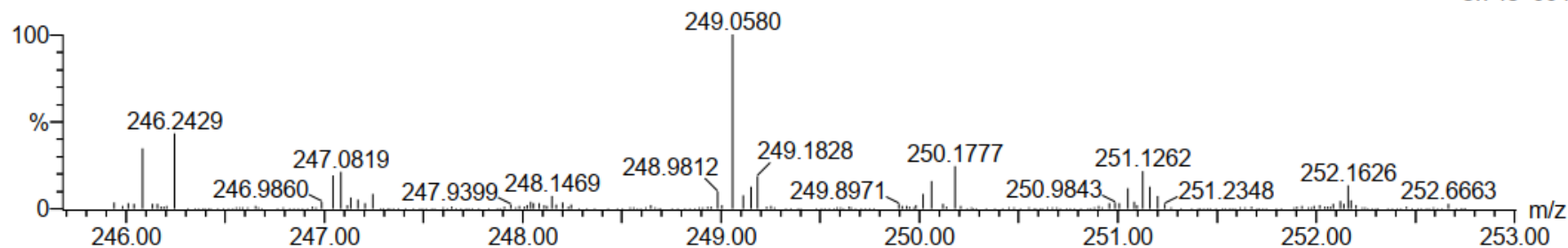
Test Name :

100522\_26\_02\_170 19 (0.418)

IITRPR

XEVO G2-XS QTOF

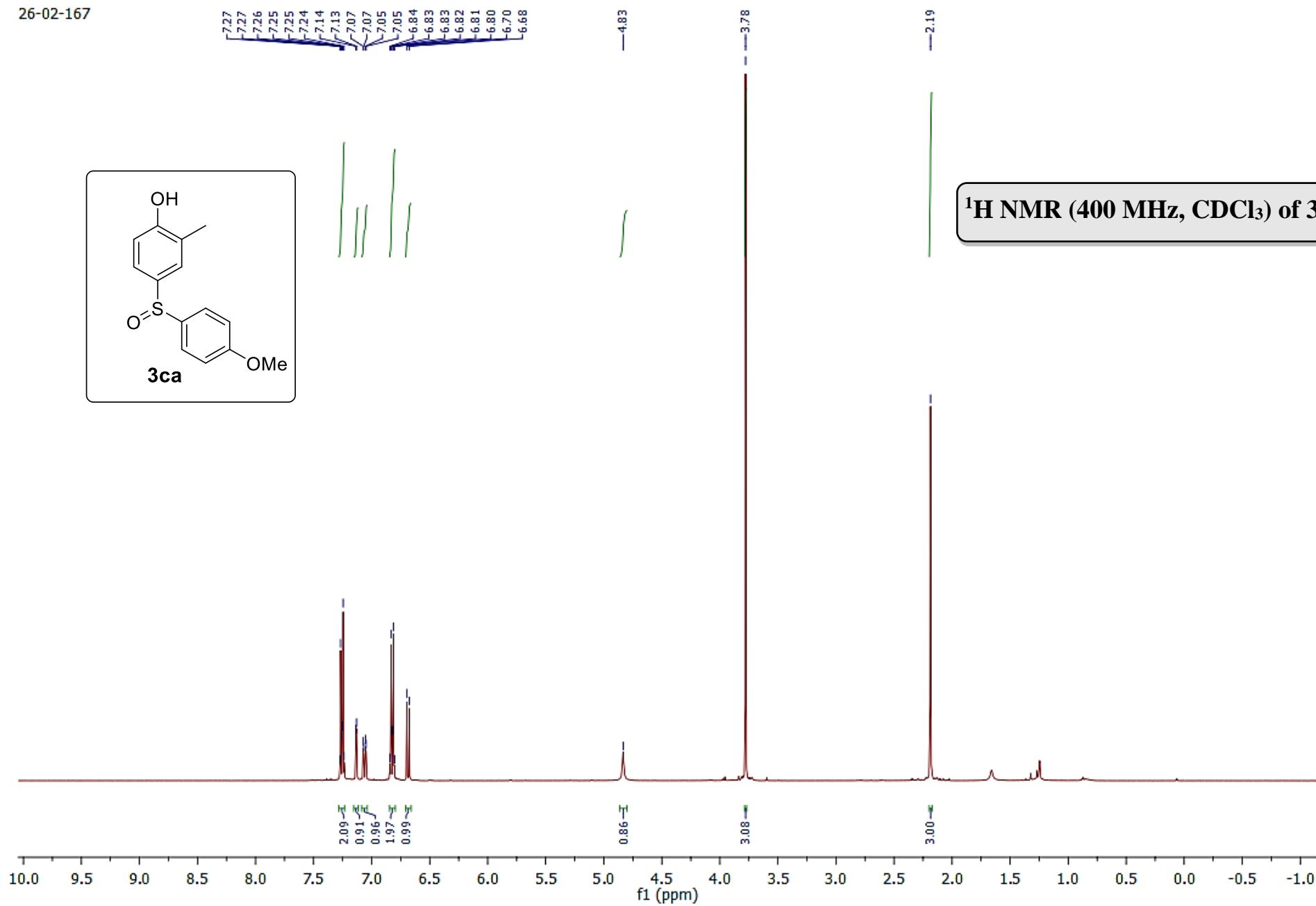
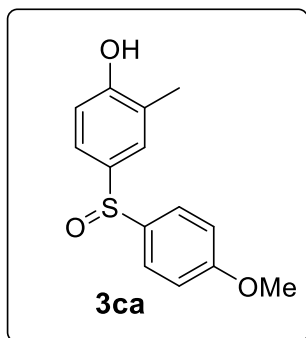
1: TOF MS ES+  
5.74e+004



Minimum: -1.5  
Maximum: 2.0 5.0 50.0

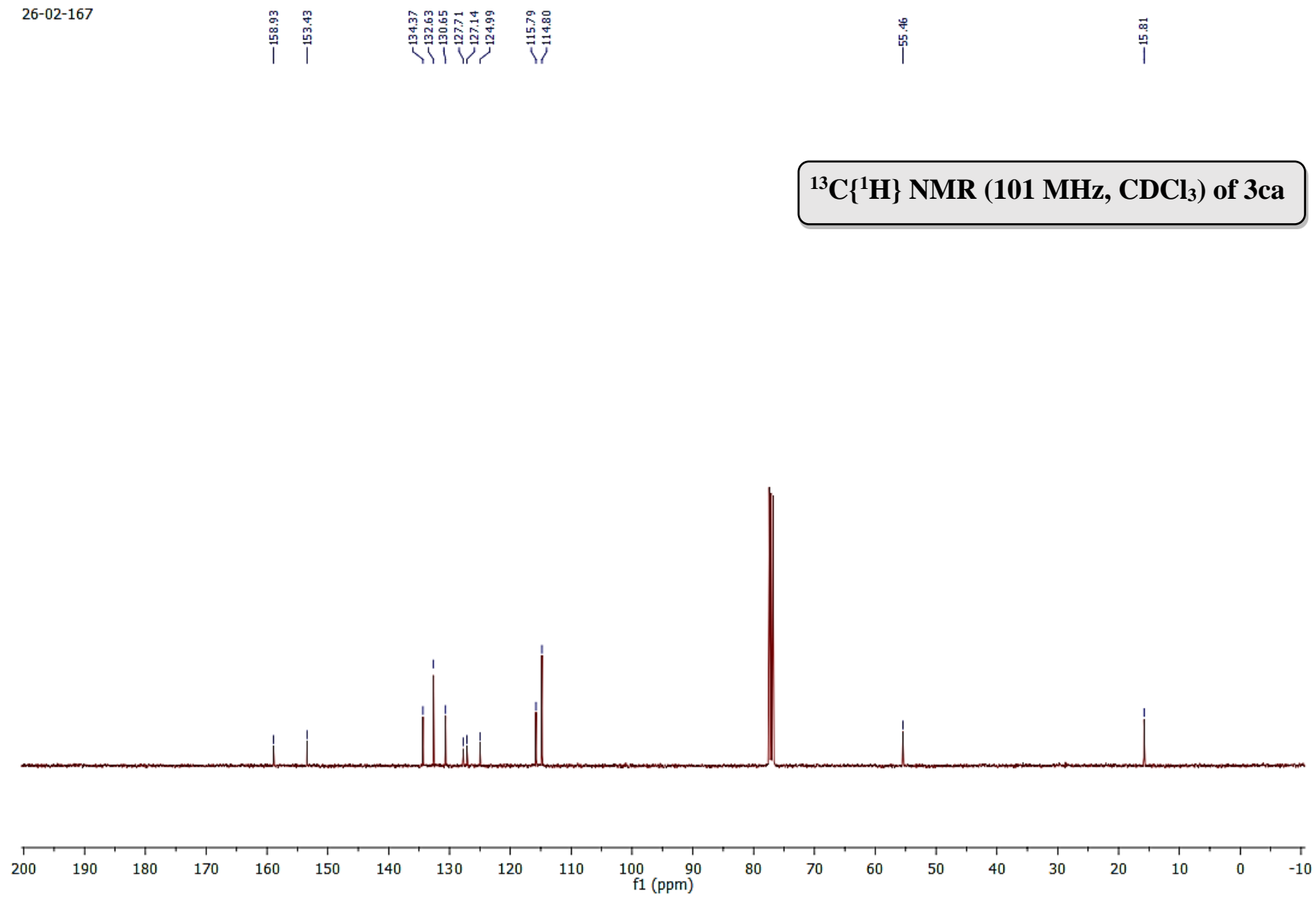
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
249.0580	249.0585	-0.5	-2.0	7.5	1419.6	n/a	n/a	C13 H13 O3 S

26-02-167



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3ca

26-02-167



Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

132 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

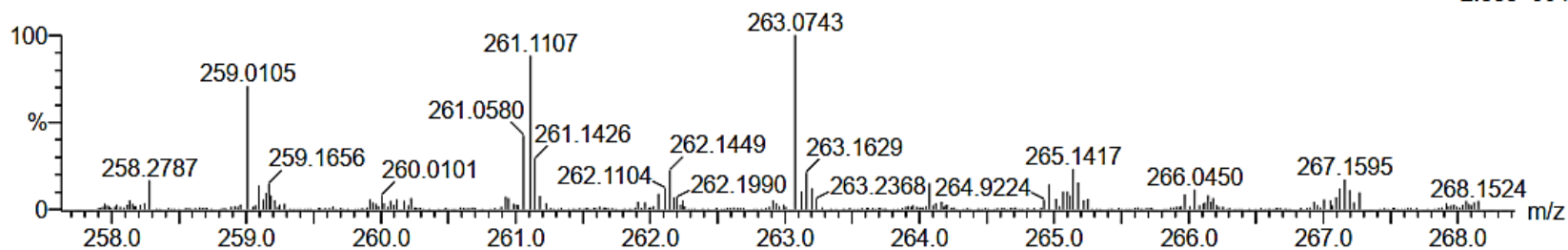
C: 0-50 H: 0-50 O: 0-10 S: 0-2 I: 0-3

Sample Name : 26\_02\_167  
 Test Name :  
 100522\_26\_02\_167 28 (0.605)

IITRPR

XEVO G2-XS QTOF

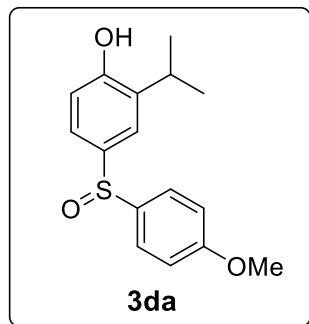
1: TOF MS ES+  
 2.88e+004



Minimum: -1.5  
 Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
263.0743	263.0742	0.1	0.4	7.5	1562.5	n/a	n/a	C14 H15 O3 S

Z:\\26-02-157RV



7.26  
7.25  
7.25  
7.24  
7.24  
7.22  
7.22  
7.02  
7.02  
7.00  
7.00  
6.84  
6.83  
6.83  
6.81  
6.80  
6.67  
6.65

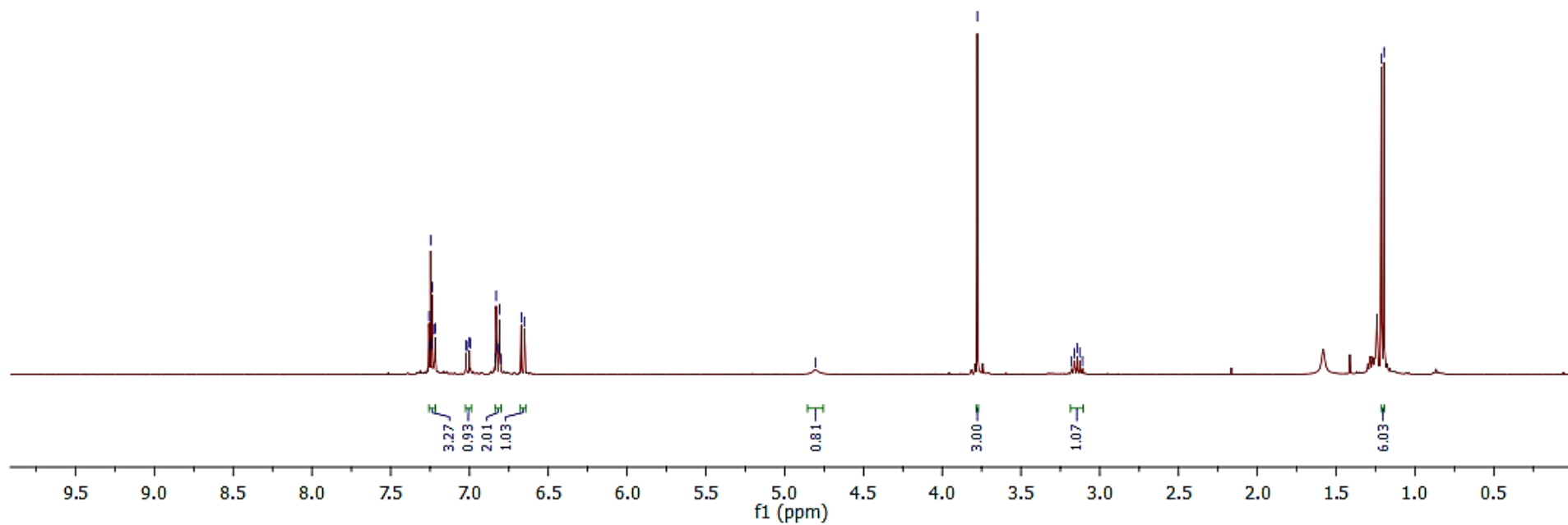
4.81

3.78

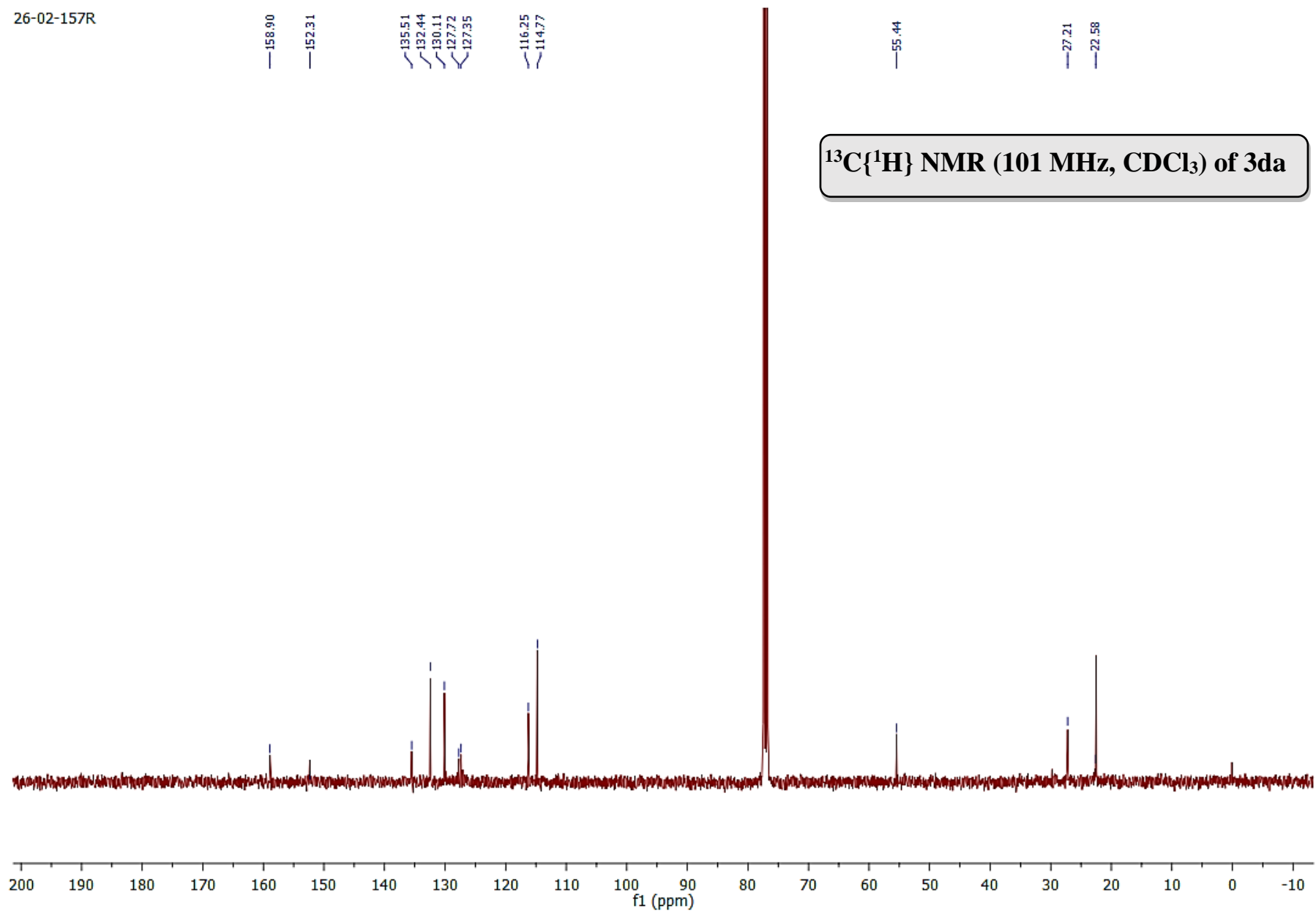
3.18  
3.16  
3.14  
3.13  
3.11

1.21  
1.20

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3da**



26-02-157R



# HRMS of 3da

## Elemental Composition Report

Page 1

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

160 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 O: 0-10 S: 0-2 I: 0-3

Sample Name : 26\_02\_157

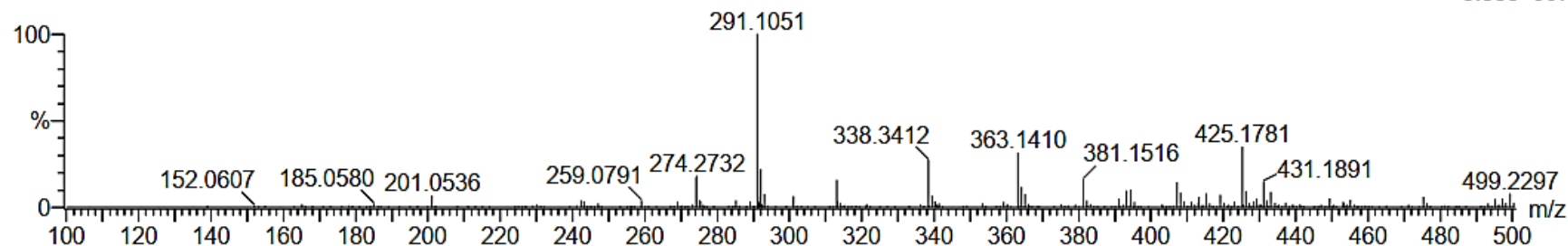
IITRPR

XEVO G2-XS QTOF

Test Name :

100522\_26\_02\_157 7 (0.169)

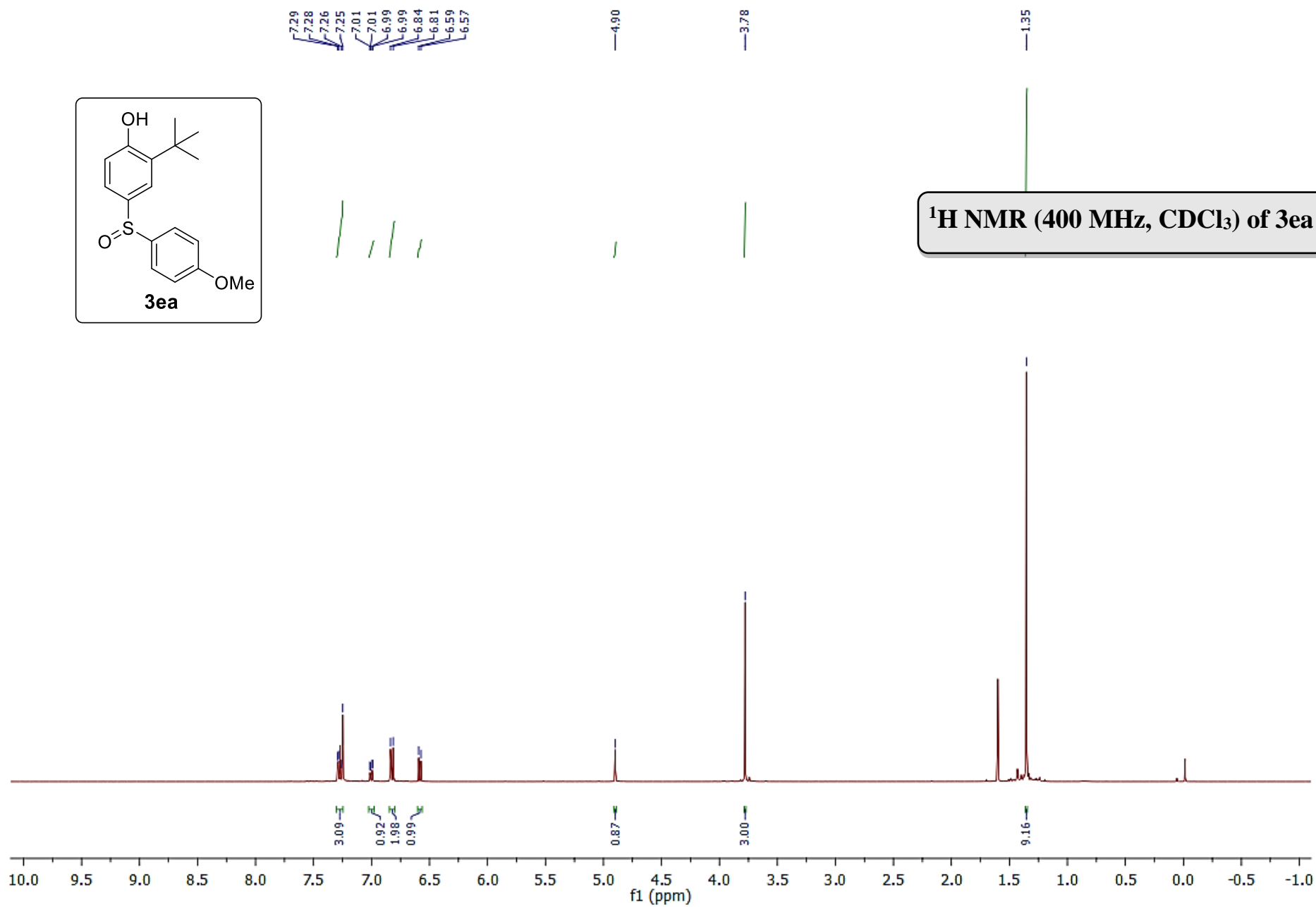
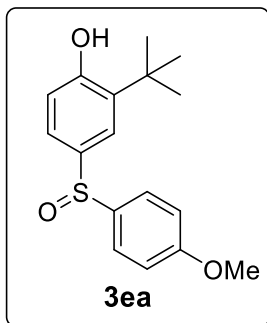
1: TOF MS ES+  
3.55e+007

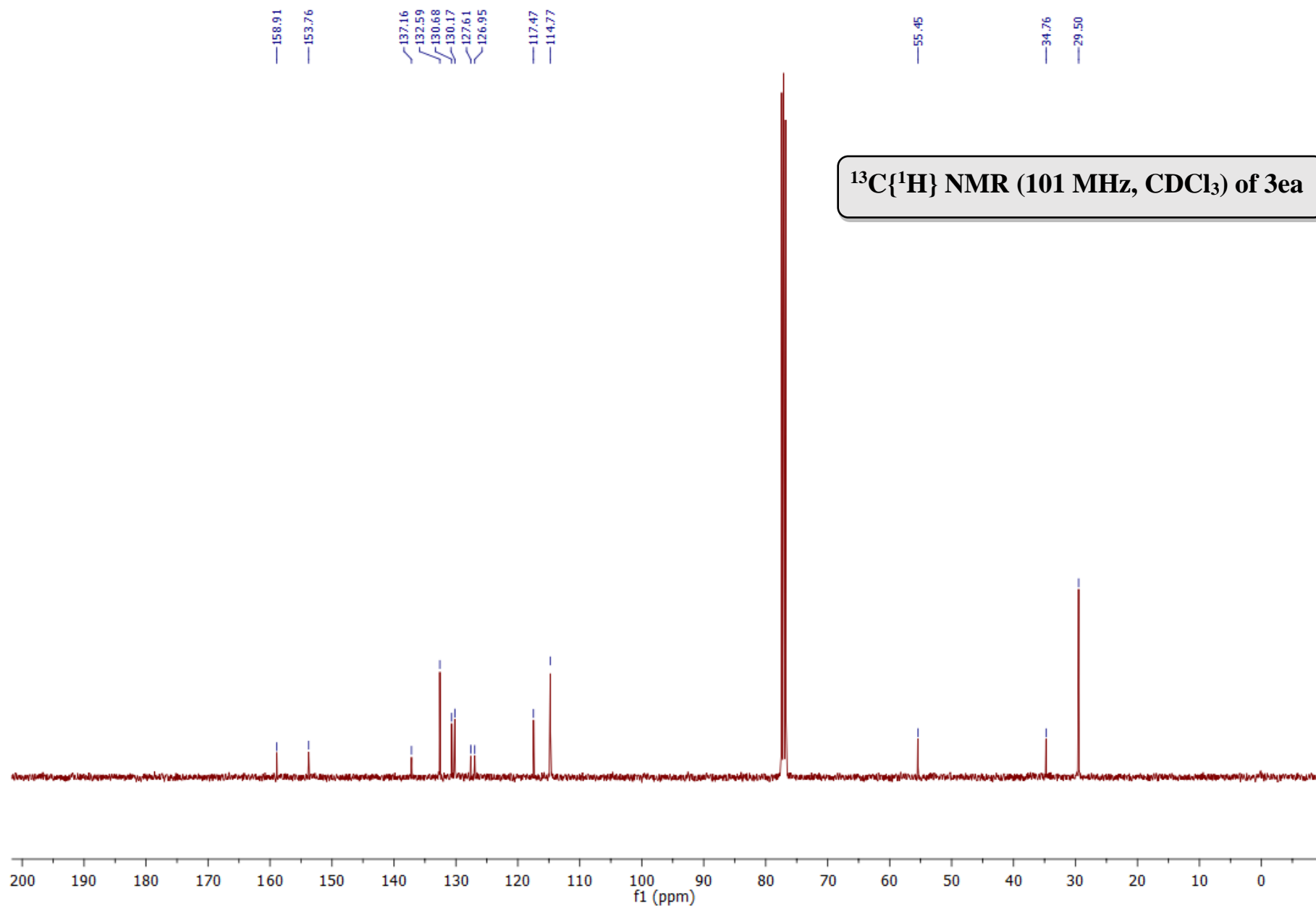


Minimum: -1.5  
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
291.1051	291.1055	-0.4	-1.4	7.5	2332.7	n/a	n/a	C16 H19 O3 S







# HRMS of 3ea

## Elemental Composition Report

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

572 formula(e) evaluated with 3 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 1-100 H: 1-100 N: 0-10 O: 0-10 S: 1-2

Sample Name : 26\_02\_211

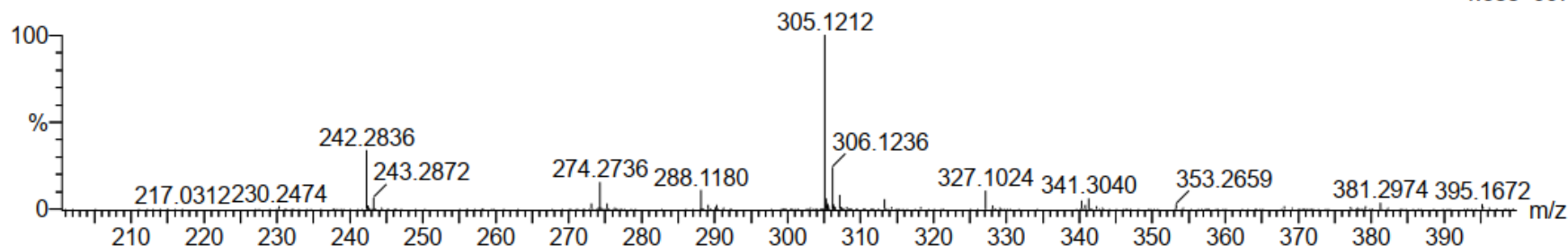
Test Name :

170622\_26\_02\_211 12 (0.143)

IITRPR

XEVO G2-XS QTOF

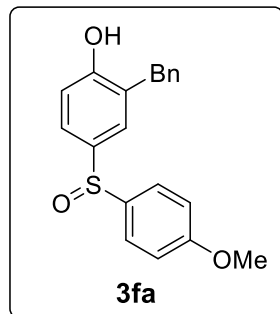
1: TOF MS ES+  
4.65e+007



Minimum: -1.5  
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
305.1212	305.1211	0.1	0.3	7.5	921.6	n/a	n/a	C17 H21 O3 S

26-02-158

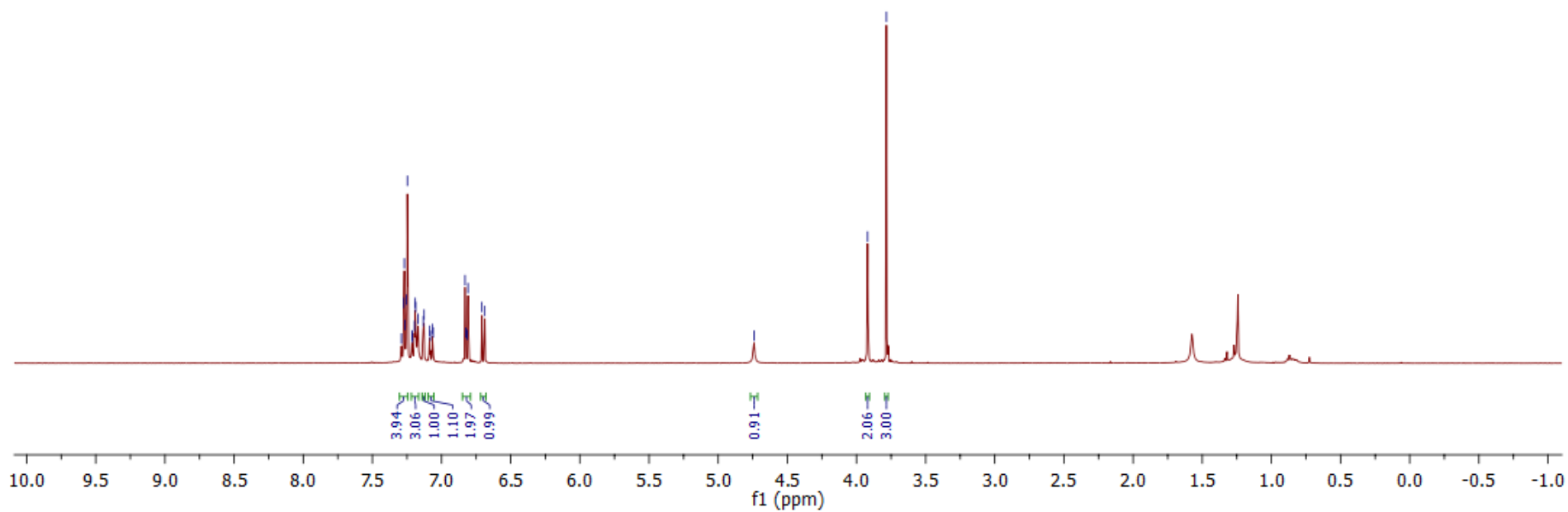


7.29  
7.27  
7.27  
7.26  
7.25  
7.25  
7.21  
7.21  
7.20  
7.19  
7.19  
7.17  
7.13  
7.13  
7.09  
7.08  
7.07  
7.06  
6.83  
6.83  
6.81  
6.81  
6.71  
6.69

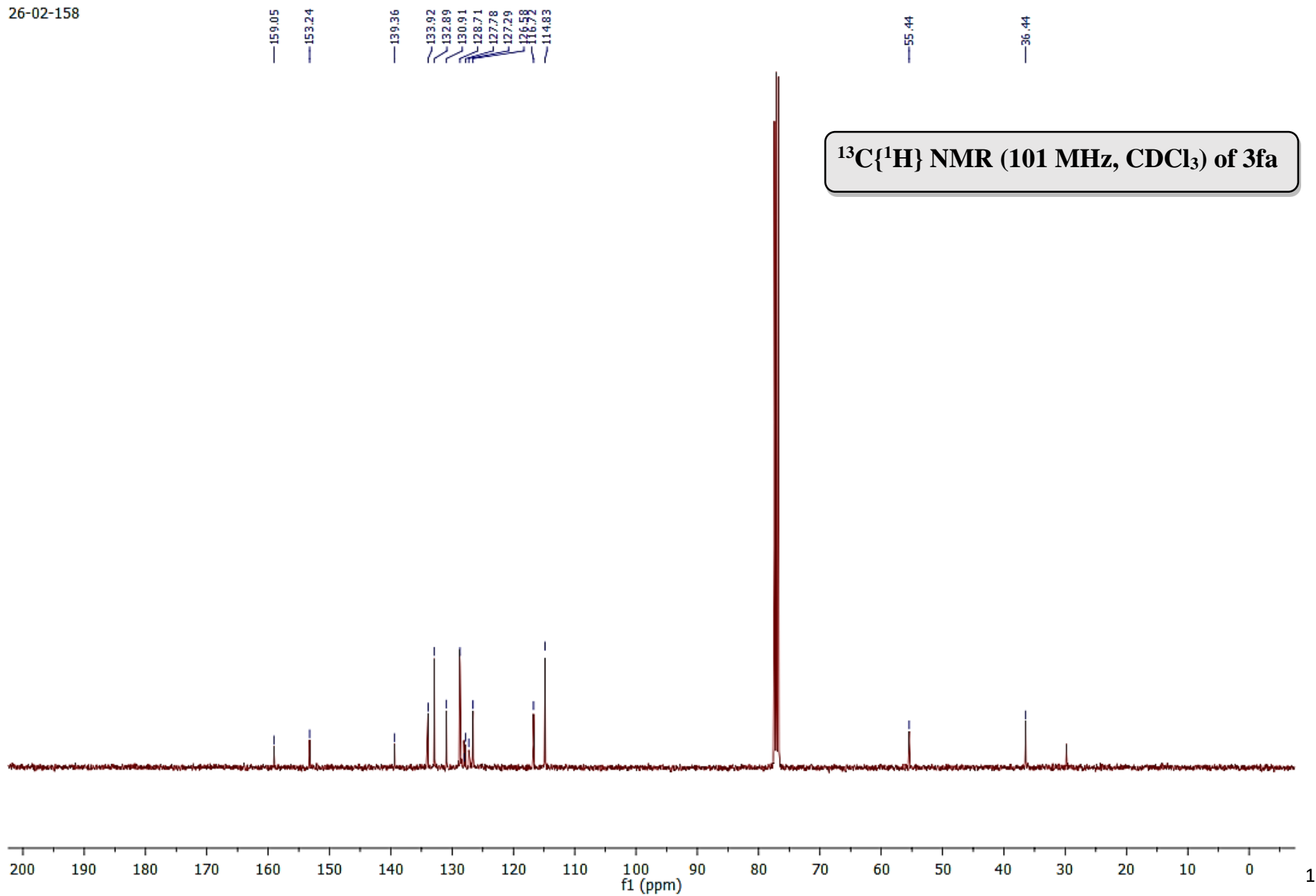
4.74

3.92  
3.78

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3fa**



26-02-158



# HRMS of 3fa

## Elemental Composition Report

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

138 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 O: 0-12 S: 0-2

Sample Name : 26\_02\_158

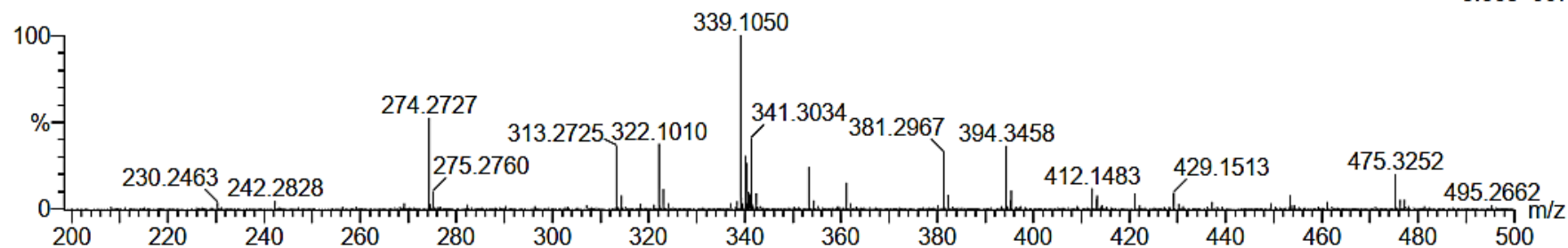
Test Name :

180522\_26\_02\_158 10 (0.125)

IITRPR

XEVO G2-XS QTOF

1: TOF MS ES+  
3.66e+007



Minimum: -1.5  
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
339.1050	339.1055	-0.5	-1.5	11.5	1337.0	n/a	n/a	C20 H19 O3 S

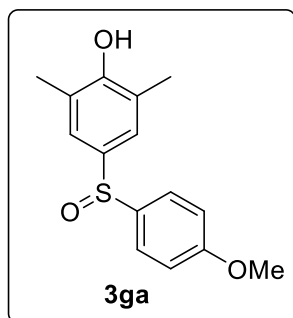
Z:\\26-02-154R

7.27  
7.26  
7.25  
7.25  
7.24  
7.24  
7.23  
7.00  
6.84  
6.83  
6.82  
6.81  
6.81  
6.80

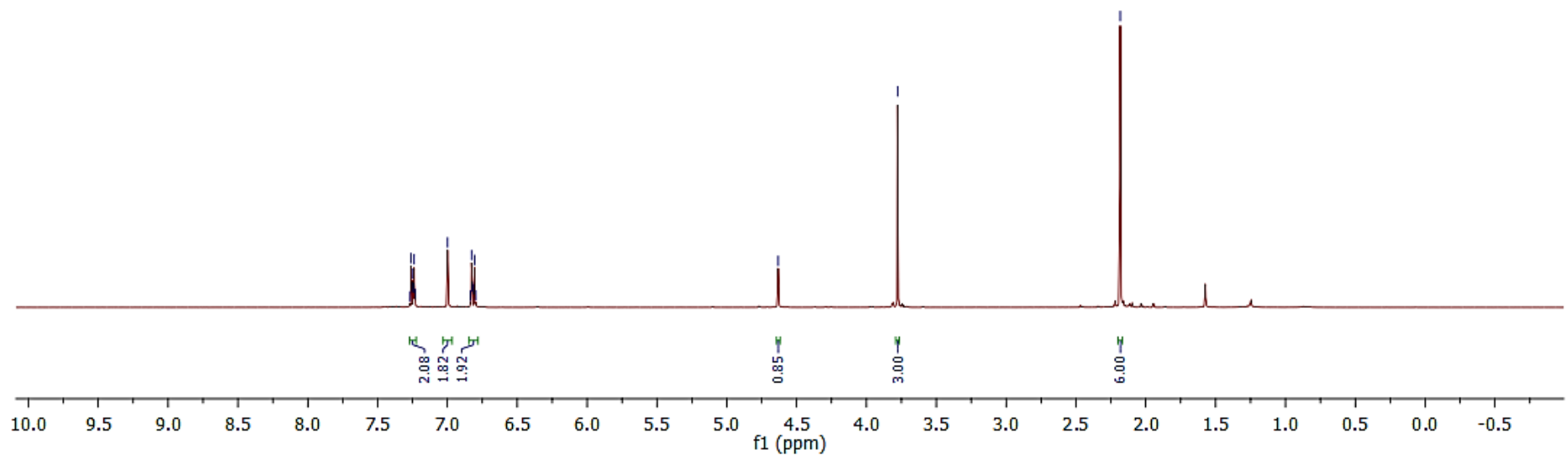
4.63

3.78

2.18



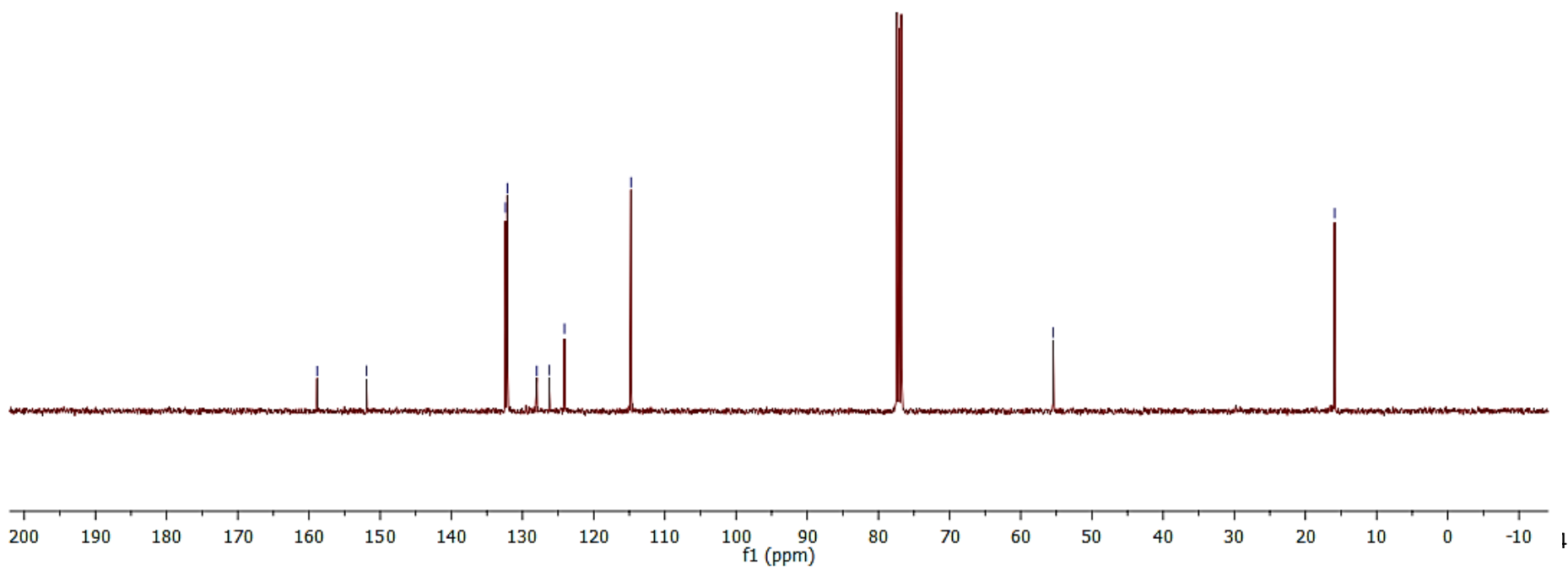
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3ga**



26-02-154R

158.86  
151.91  
132.41  
132.15  
127.99  
126.25  
124.10  
114.76  
55.43  
15.90

**$^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz,  $\text{CDCl}_3$ ) of 3ga**





# HRMS of 3ga

## Elemental Composition Report

Page 1

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

96 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 O: 0-10 S: 0-2

Sample Name : 26\_02\_154

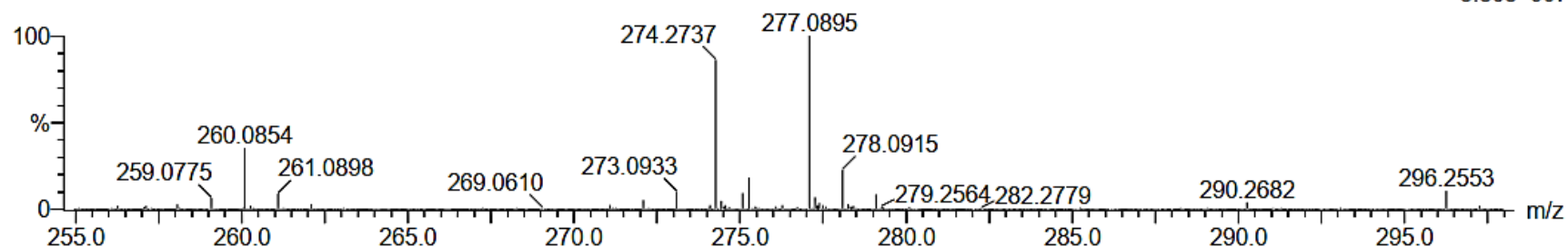
Test Name :

100522\_26\_02\_154 5 (0.124)

IITRPR

XEVO G2-XS QTOF

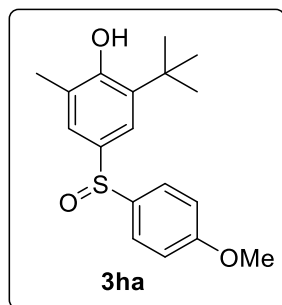
1: TOF MS ES+  
3.80e+007



Minimum: -1.5  
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
277.0895	277.0898	-0.3	-1.1	7.5	2326.5	n/a	n/a	C15 H17 O3 S

26-02-173R



7.26  
7.25  
7.24  
7.24  
7.18  
7.17  
6.98  
6.97  
6.84  
6.83  
6.82  
6.81  
6.81

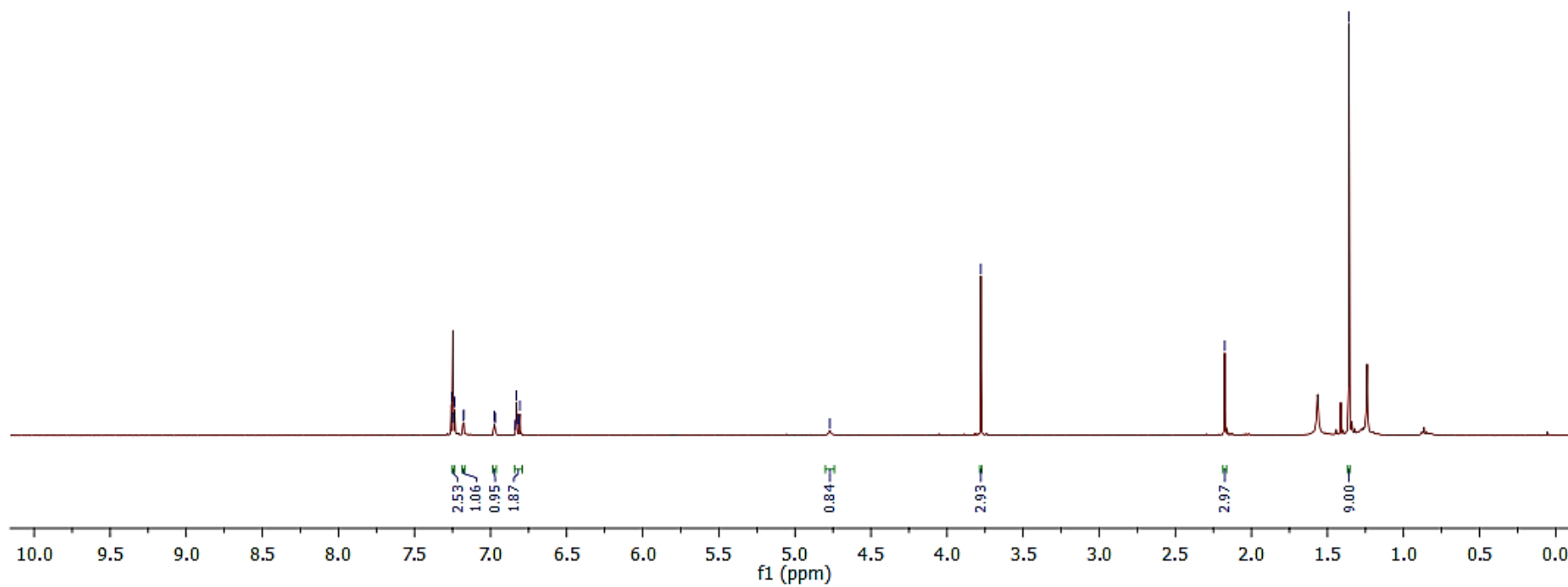
4.77

3.78

2.18

1.36

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3ha**



26-02-173

158.81  
152.38  
136.66  
132.34  
131.82  
128.91  
127.92  
125.92  
124.20  
114.74

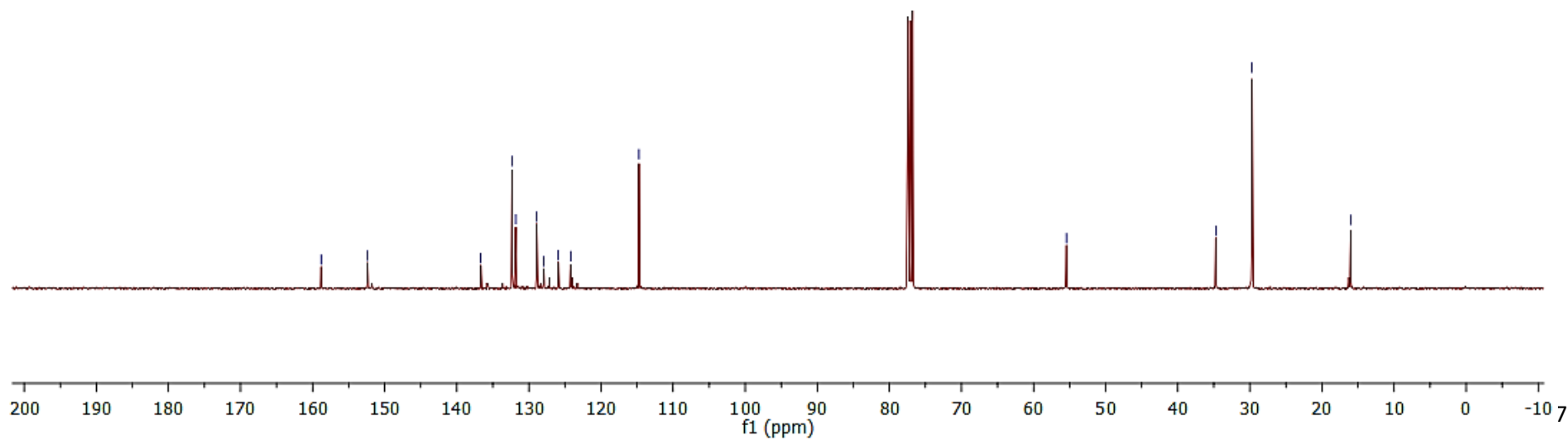
55.44

34.73

29.70

16.03

**$^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz,  $\text{CDCl}_3$ ) of 3ha**



# HRMS of 3ha

## Elemental Composition Report

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

543 formula(e) evaluated with 3 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-4 O: 0-10 S: 0-2

Sample Name : 26\_02\_173

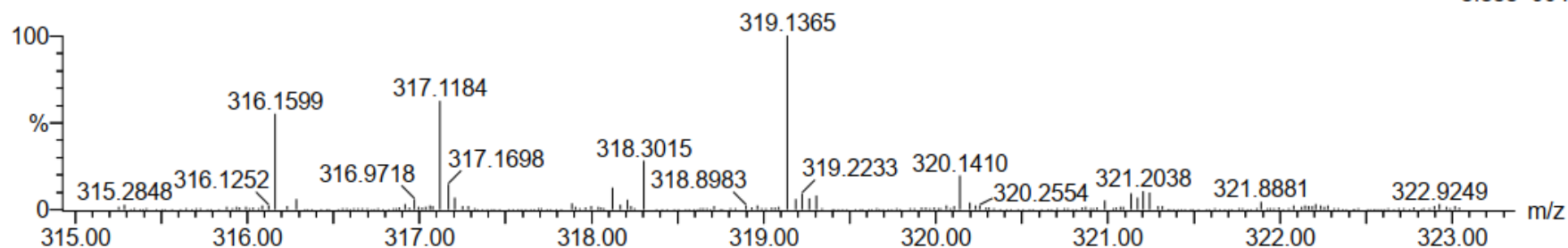
Test Name :

100522\_26\_02\_173 20 (0.435)

IITRPR

XEVO G2-XS QTOF

1: TOF MS ES+  
5.85e+04



Minimum: -1.5  
Maximum: 2.0 5.0 50.0

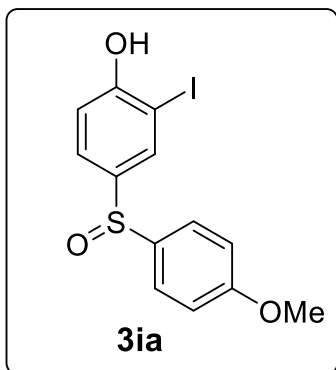
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
319.1365	319.1368	-0.3	-0.9	7.5	1432.4	n/a	n/a	C18 H23 O3 S

Z:\\26-02-161R

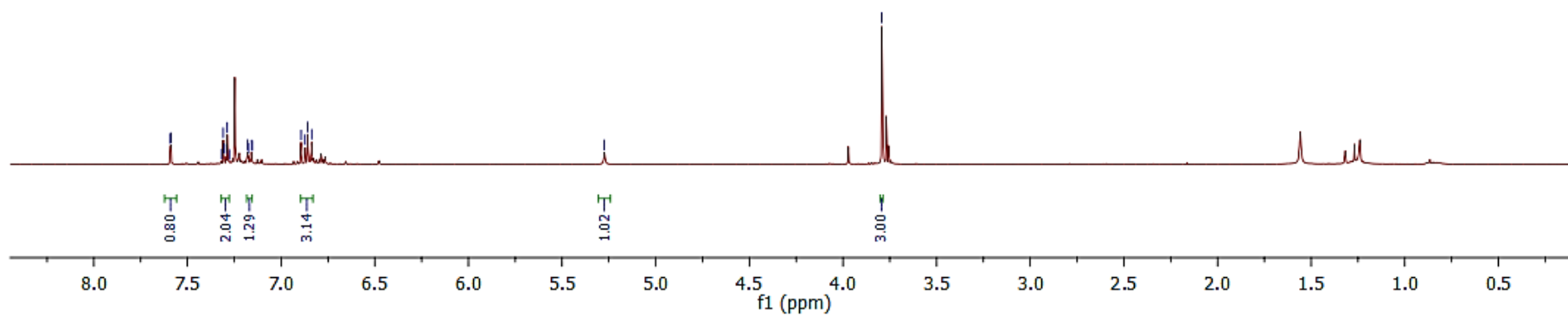
7.59  
7.59  
7.31  
7.29  
7.18  
6.88  
6.87  
6.86  
6.84

5.27

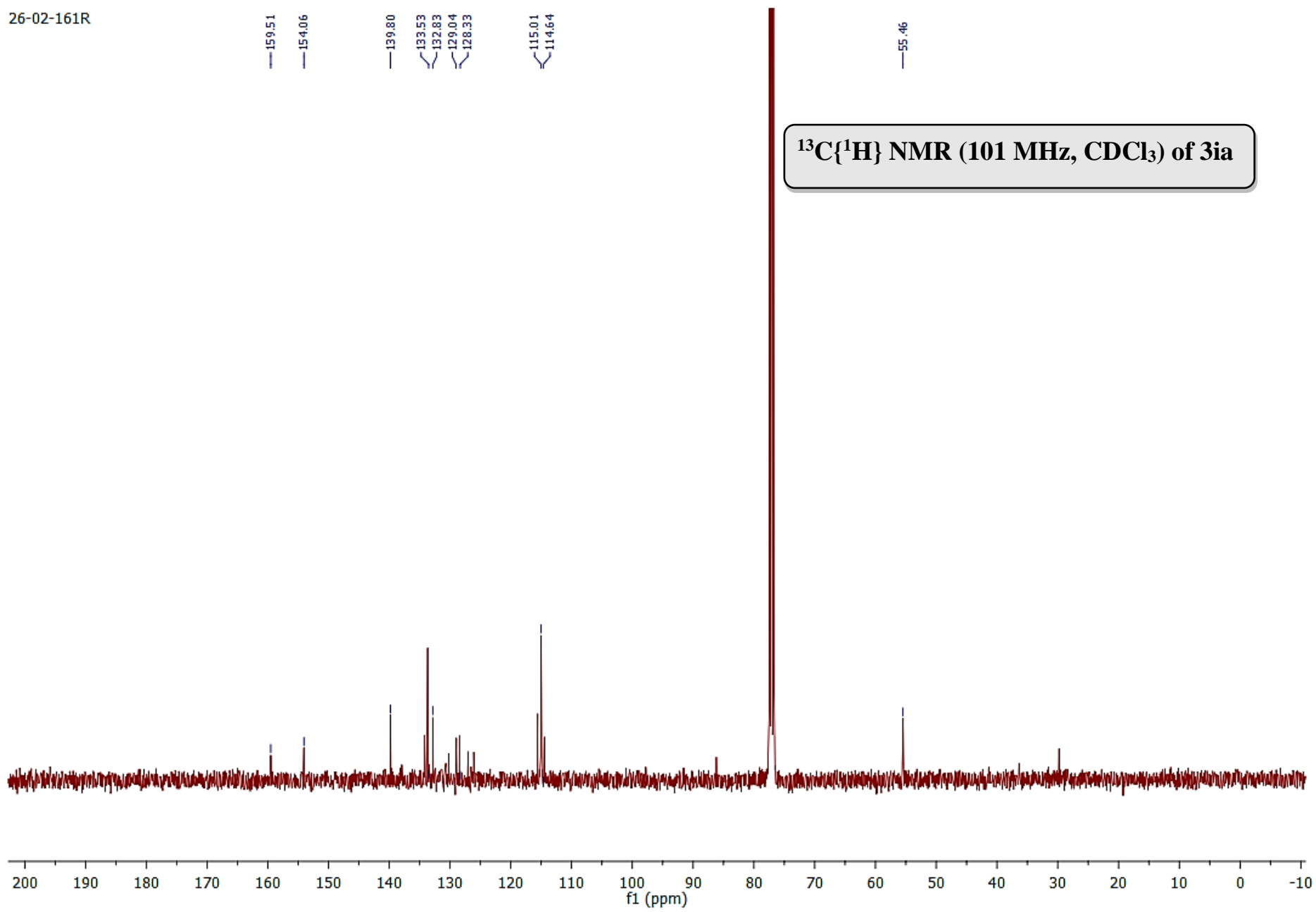
3.79



**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3ia**



26-02-161R



# HRMS of 3ia

## Elemental Composition Report

Page 1

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

243 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

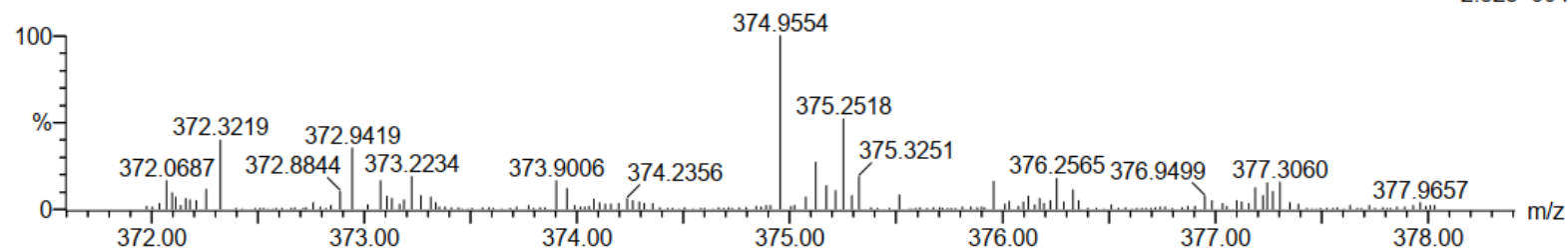
C: 0-50 H: 0-50 O: 0-10 S: 0-2 I: 0-3

Sample Name : 26\_02\_161R  
Test Name :  
100522\_26\_02\_161R 12 (0.265)

IITRPR

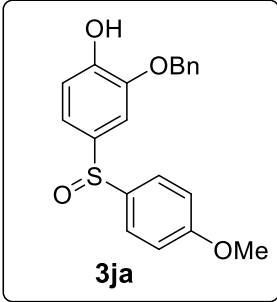
XEVO G2-XS QTOF

1: TOF MS ES+  
2.32e+004



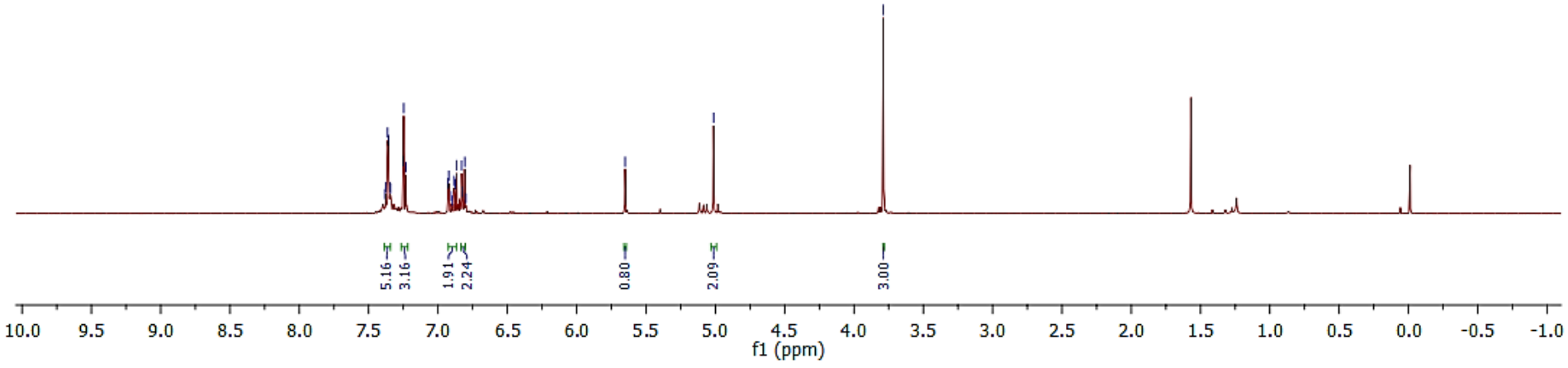
Minimum: -1.5  
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
374.9554	374.9552	0.2	0.5	7.5	1138.2	n/a	n/a	C13 H12 O3 S I



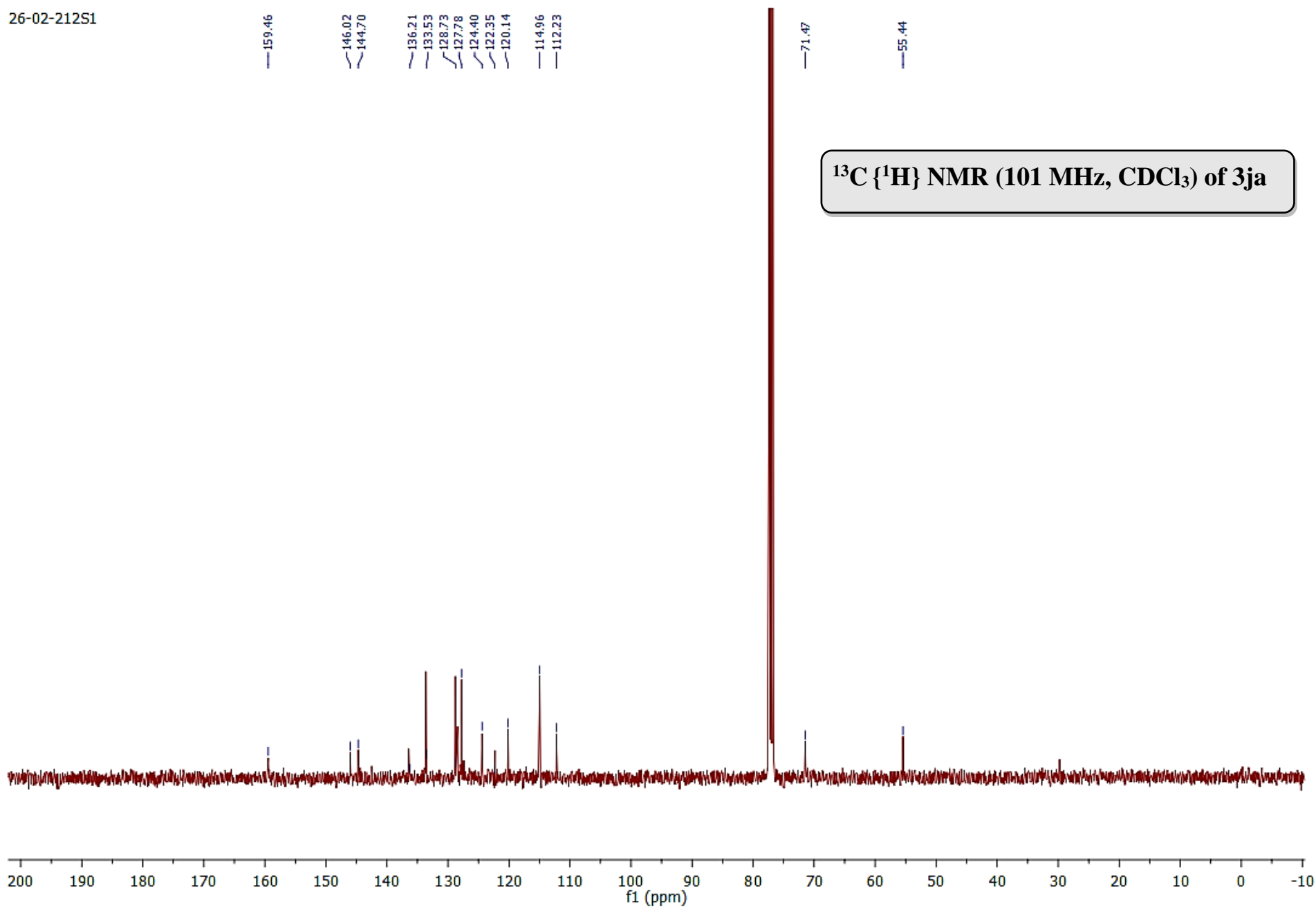
7.38  
7.38  
7.36  
7.36  
7.35  
7.34  
7.25  
7.23  
6.93  
6.92  
6.90  
6.88  
6.88  
6.86  
6.83  
6.82  
6.81  
5.80  
5.01  
3.79

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3ja**





26-02-212S1



# HRMS of 3ja

## Elemental Composition Report

Page 1

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

77 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 1-100 H: 1-100 O: 0-10 S: 1-2

Sample Name : 26\_02\_212

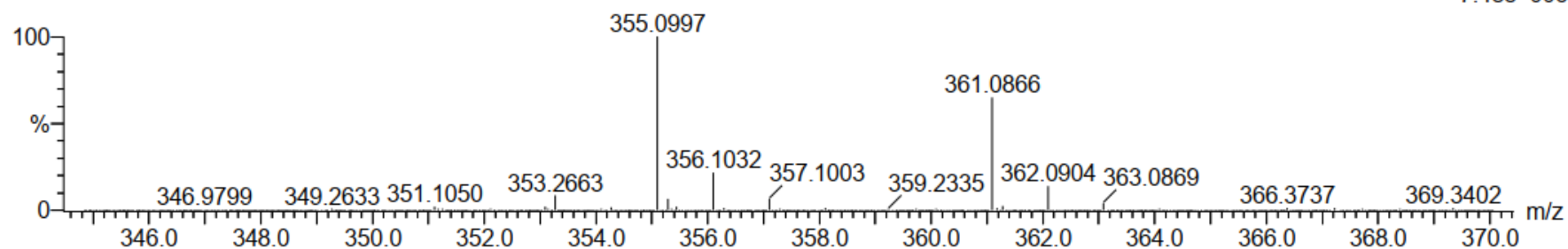
IITRPR

XEVO G2-XS QTOF

Test Name :

170622\_26\_02\_212 9 (0.117)

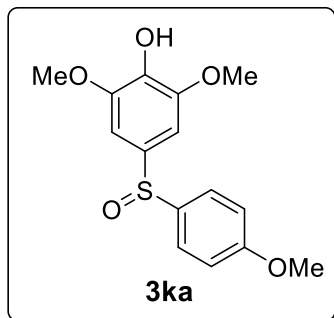
1: TOF MS ES+  
7.48e+006



Minimum: -1.5  
Maximum: 2.0 5.0 50.0

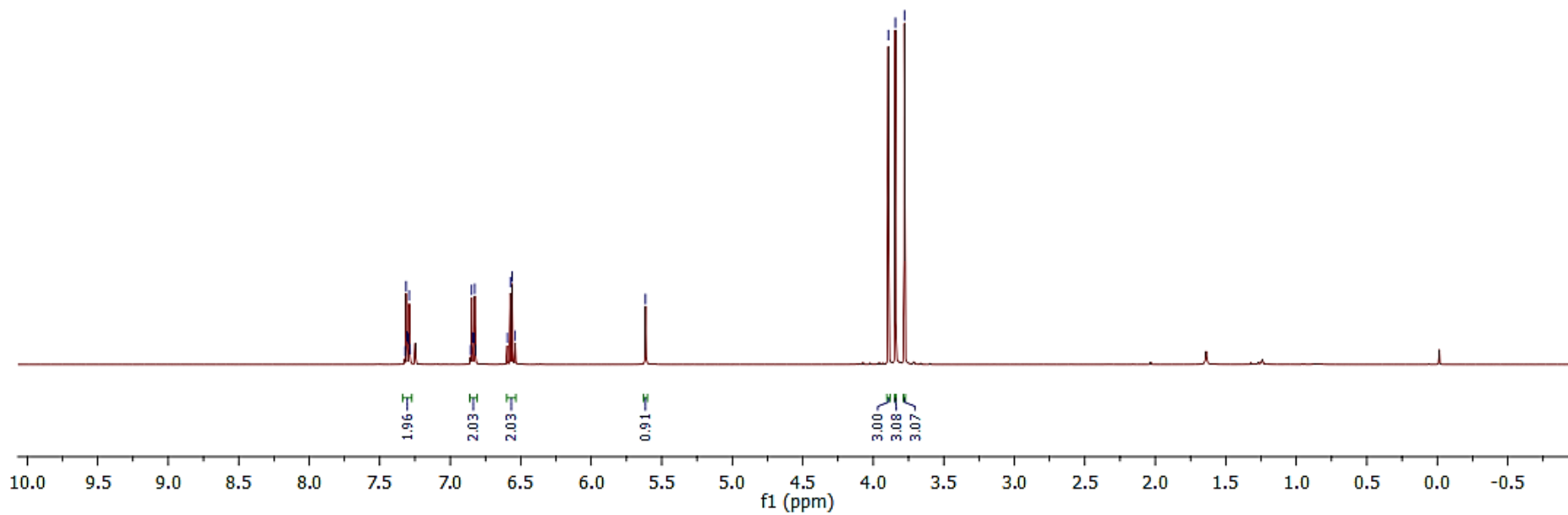
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
355.0997	355.1004	-0.7	-2.0	11.5	1659.6	n/a	n/a	C20 H19 O4 S

Z:\\26-02-181



7.32  
7.31  
7.31  
7.30  
7.29  
7.28  
6.86  
6.85  
6.84  
6.83  
6.83  
6.82  
6.60  
6.57  
6.56  
6.54  
5.62  
3.89  
3.84  
3.78

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3ka

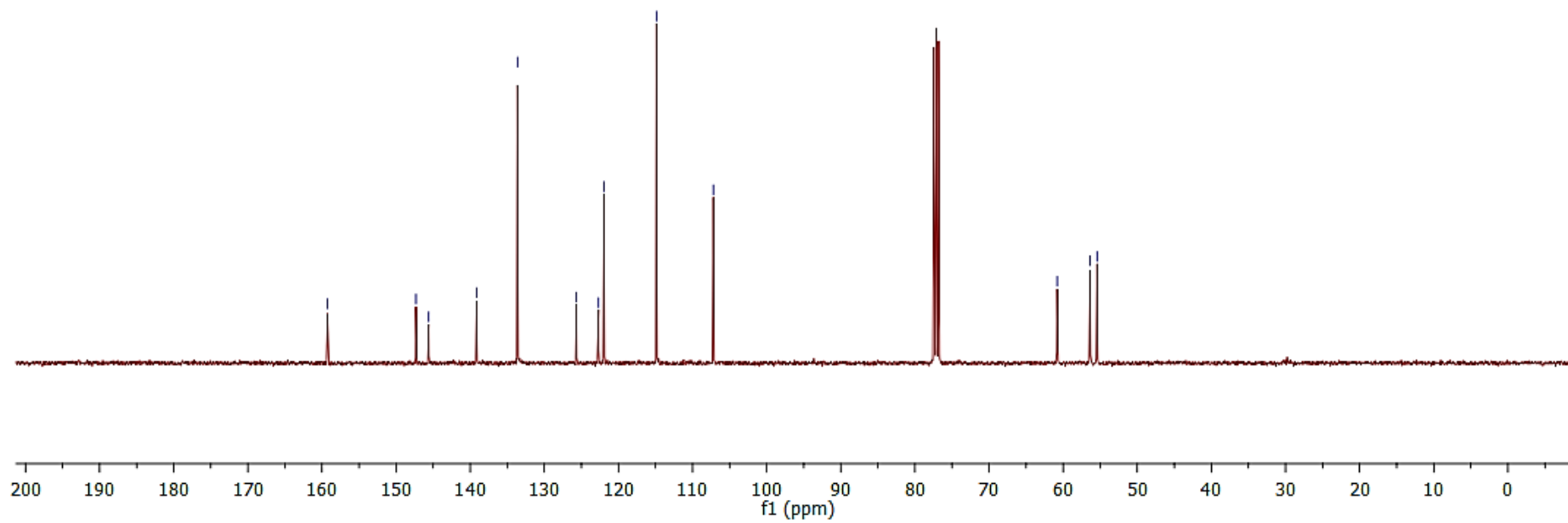


26-02-181

159.27  
147.32  
145.60  
139.13  
133.63  
125.67  
122.68  
121.93  
114.87  
107.19

60.80  
56.36  
55.43

$^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz,  $\text{CDCl}_3$ ) of 3ka



Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0  
 Element prediction: Off  
 Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

121 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 O: 0-12 S: 0-2

Sample Name : 26\_02\_181

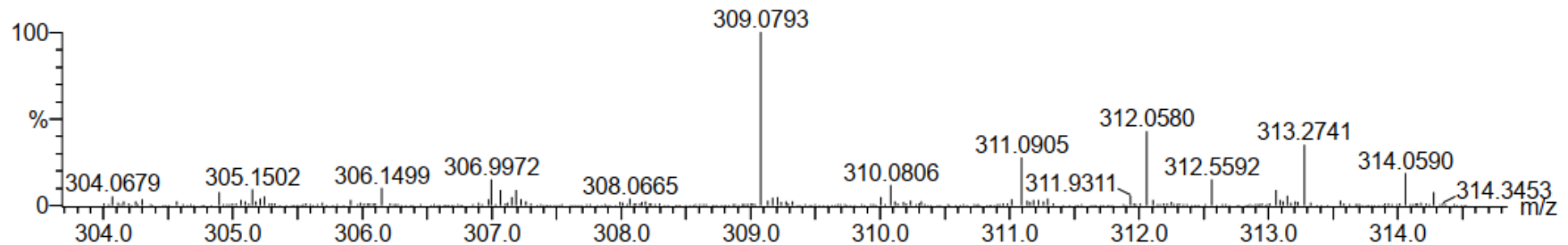
Test Name :

180522\_26\_02\_181 40 (0.417)

IITRPR

XEVO G2-XS QTOF

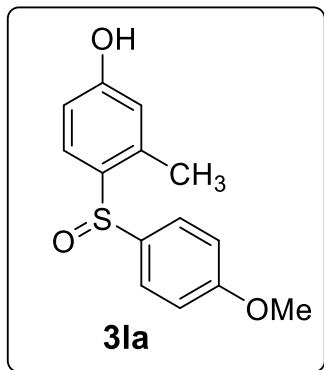
1: TOF MS ES+  
3.61e+004



Minimum: -1.5  
 Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
309.0793	309.0797	-0.4	-1.3	7.5	1375.8	n/a	n/a	C15 H17 O5 S

Z:\\26-02-164R1



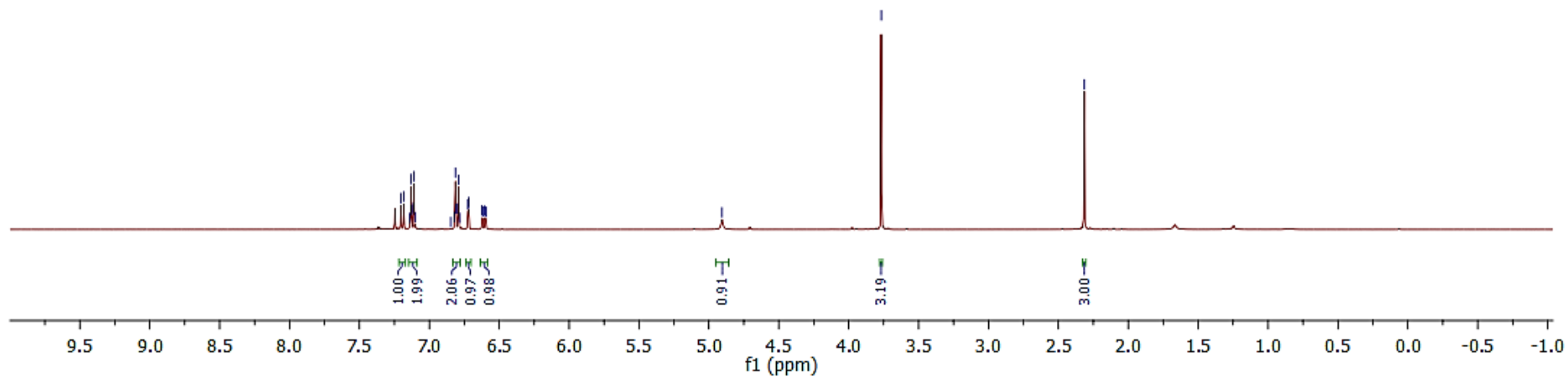
7.20  
7.18  
7.14  
7.13  
7.12  
7.11  
6.85  
6.82  
6.81  
6.81  
6.80  
6.79  
6.78  
6.72  
6.62  
6.62  
6.60  
6.60

4.91

3.77

2.31

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3la**



H

26-02-164R1

158.49  
155.55

142.19

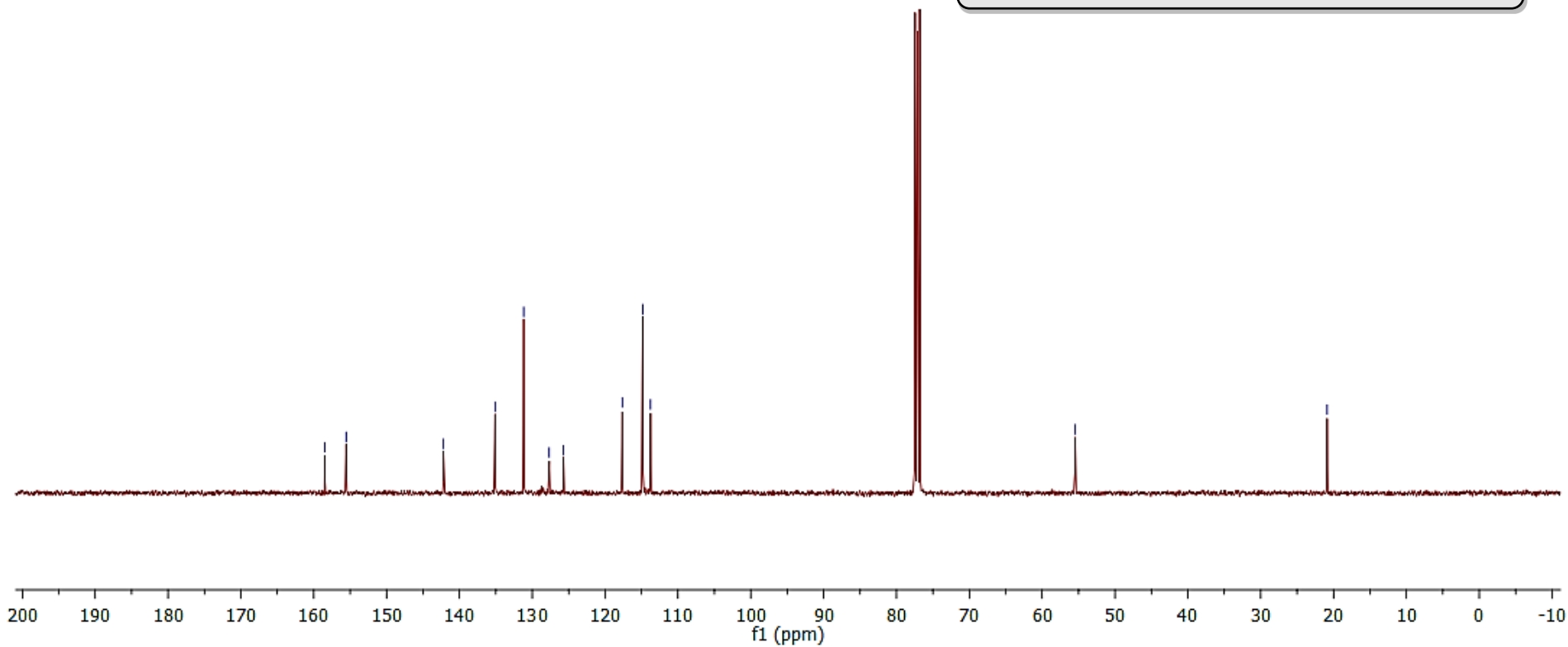
135.10  
131.17  
127.67  
125.71

117.64  
114.84  
113.77

55.46

20.87

**$^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz,  $\text{CDCl}_3$ ) of 3la**



# HRMS of 3la

## Elemental Composition Report

Page 1

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

433 formula(e) evaluated with 2 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-4 O: 0-10 S: 0-2

Sample Name : 26\_02\_164

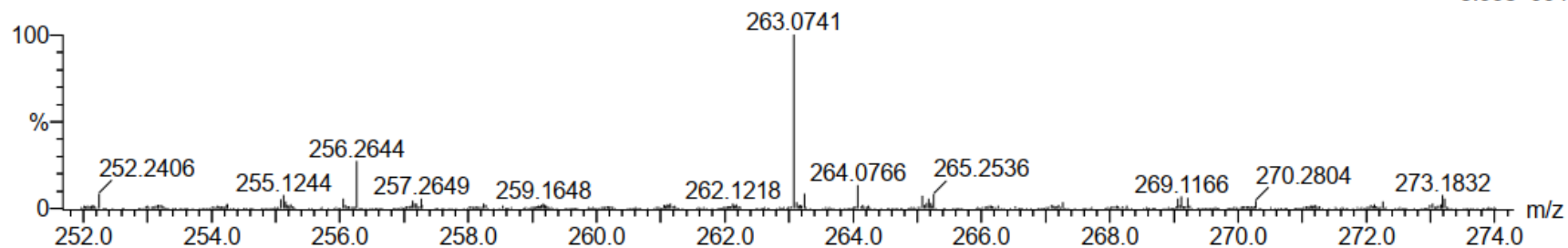
Test Name :

100522\_26\_02\_164 3 (0.151)

IITRPR

XEVO G2-XS QTOF

2: TOF MS ES+  
3.99e+004

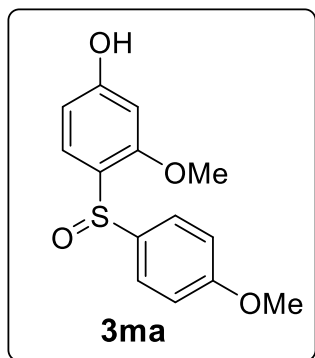


Minimum: -1.5  
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
263.0741	263.0742	-0.1	-0.4	7.5	983.6	n/a	n/a	C14 H15 O3 S



26-02-165R

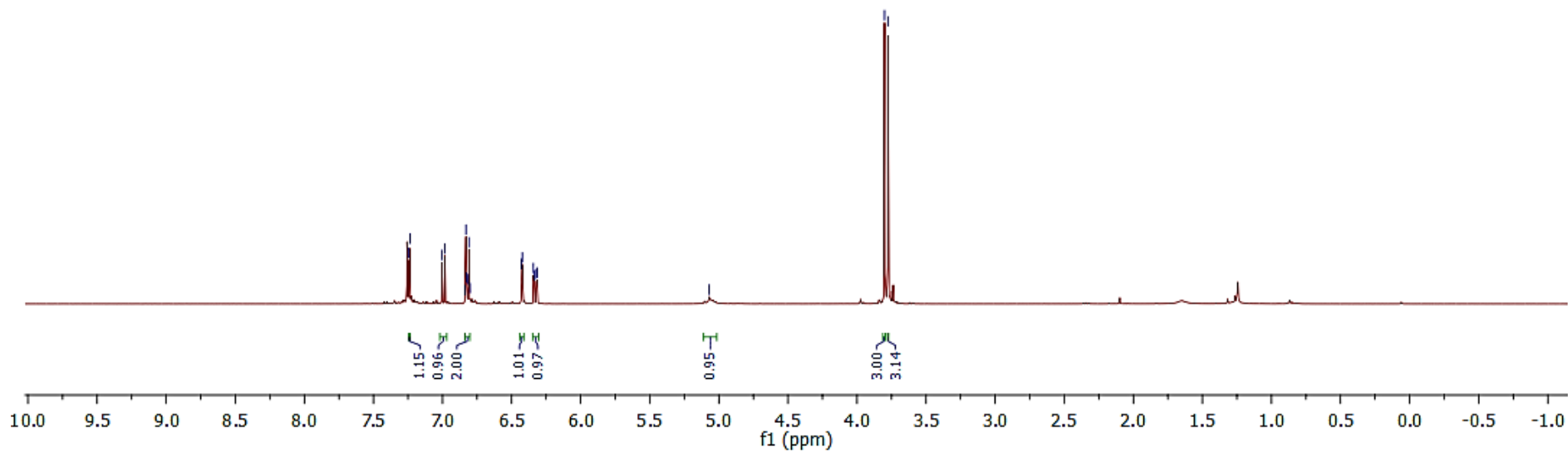


7.25  
7.24  
7.00  
6.98  
6.84  
6.83  
6.83  
6.81  
6.81  
6.80  
6.43  
6.42  
6.34  
6.34  
6.32  
6.32

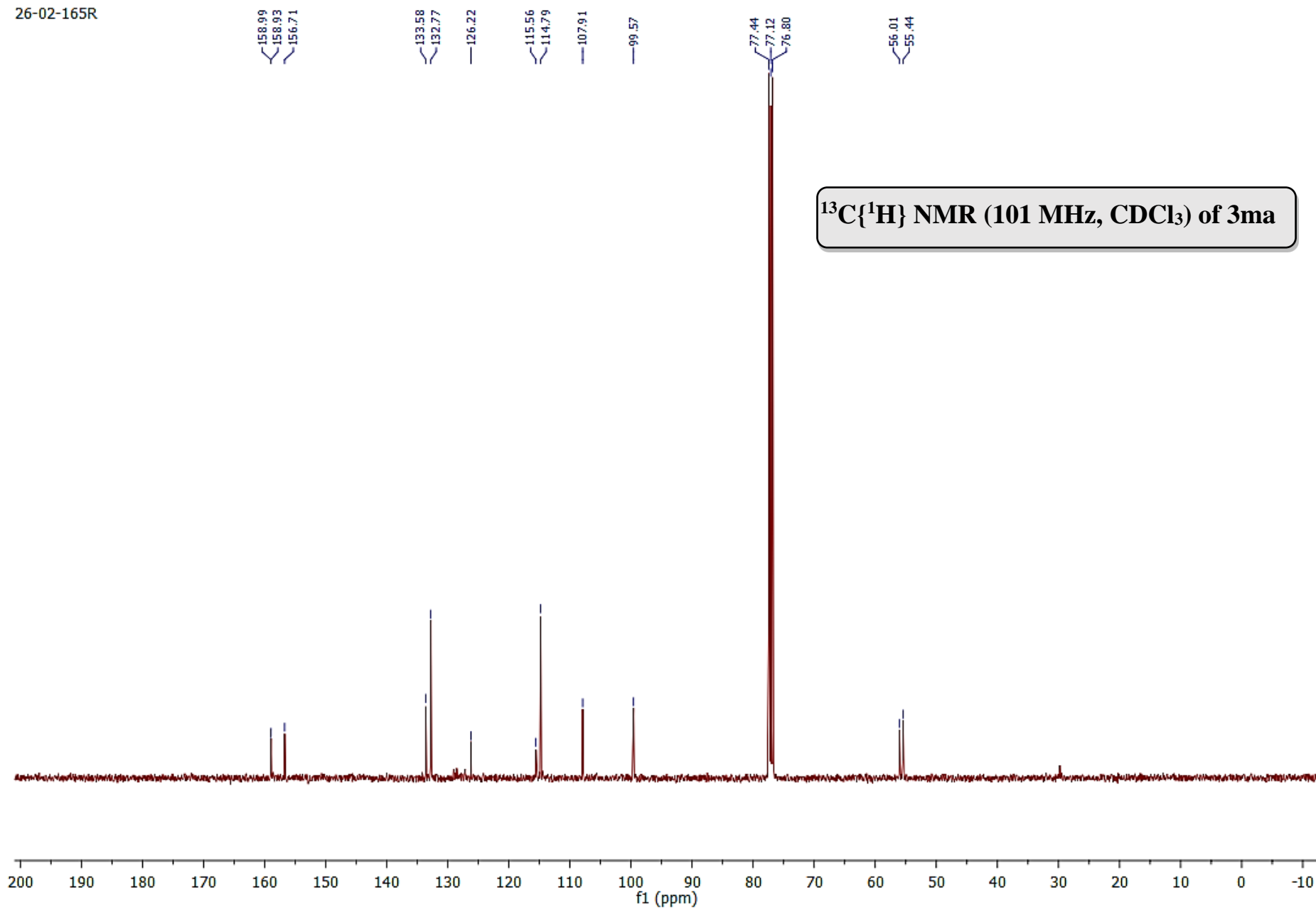
5.07

3.80  
3.78

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3ma**



26-02-165R



Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

146 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

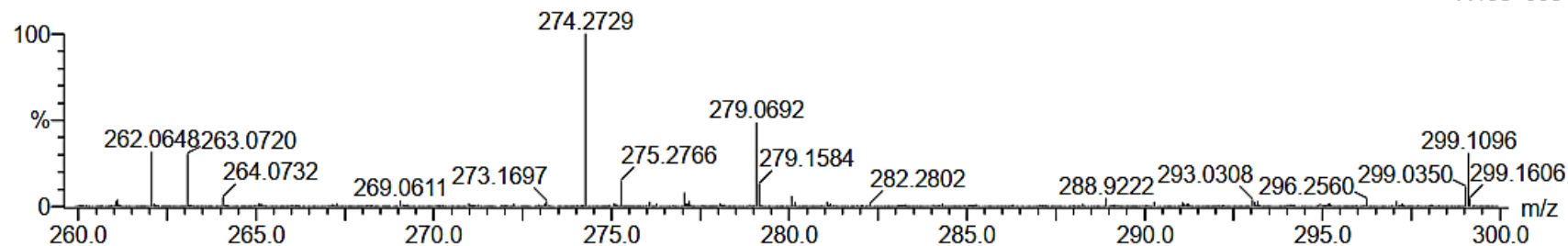
C: 0-50 H: 0-50 O: 0-10 S: 0-2 I: 0-3

Sample Name : 26\_02\_165  
 Test Name :  
 100522\_26\_02\_165 10 (0.232)

IITRPR

XEVO G2-XS QTOF

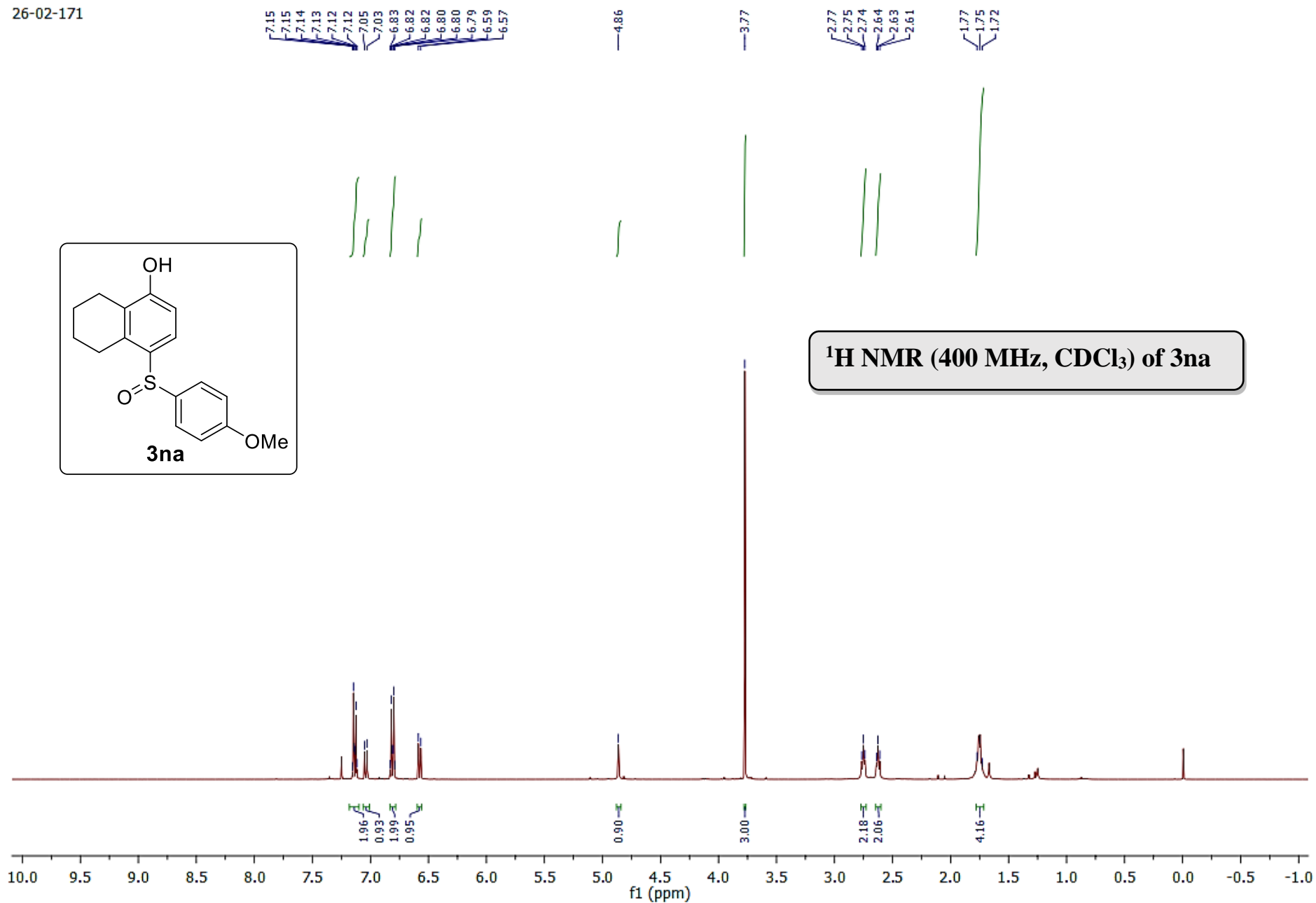
1: TOF MS ES+  
 7.19e+005



Minimum: -1.5  
 Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
279.0692	279.0691	0.1	0.4	7.5	1819.7	n/a	n/a	C14 H15 O4 S

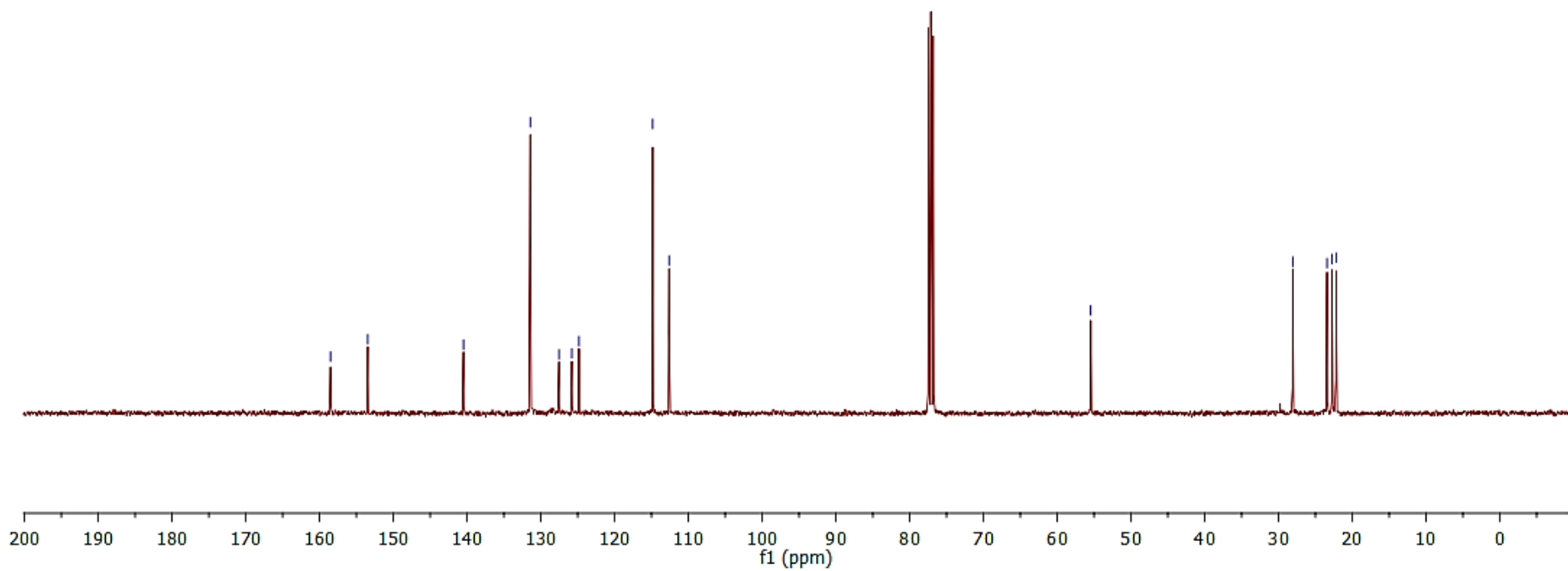
26-02-171



26-02-171

158.51  
153.46  
140.48  
131.43  
131.40  
127.53  
125.79  
124.82  
114.84  
112.61  
55.44  
28.07  
23.42  
22.74  
22.19

$^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz,  $\text{CDCl}_3$ ) of 3na



# HRMS of 3na

## Elemental Composition Report

Page 1

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

171 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 O: 0-10 S: 0-2 I: 0-3

Sample Name : 26\_02\_171

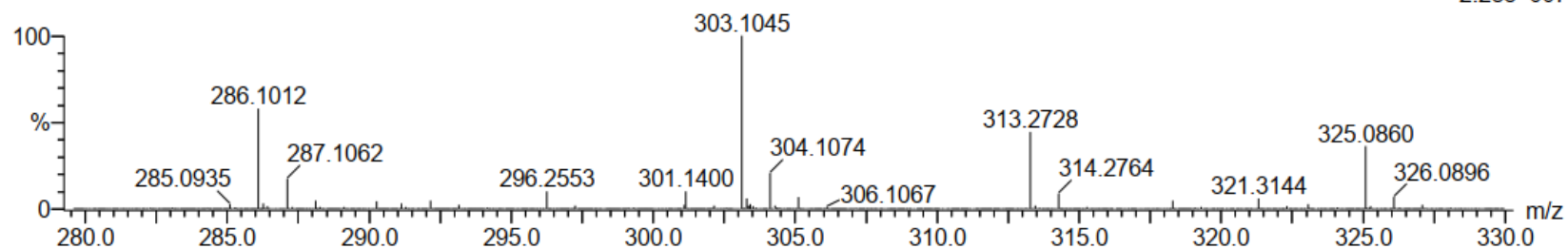
IITRPR

XEVO G2-XS QTOF

Test Name :

100522\_26\_02\_171 5 (0.124)

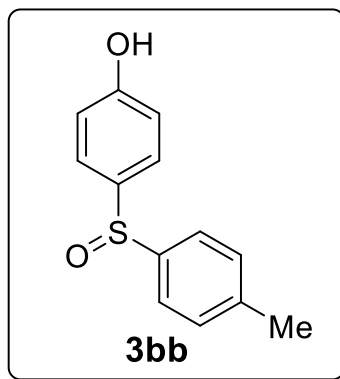
1: TOF MS ES+  
2.28e+007



Minimum: -1.5  
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
303.1045	303.1055	-1.0	-3.3	8.5	2127.6	n/a	n/a	C17 H19 O3 S

26-02-202R

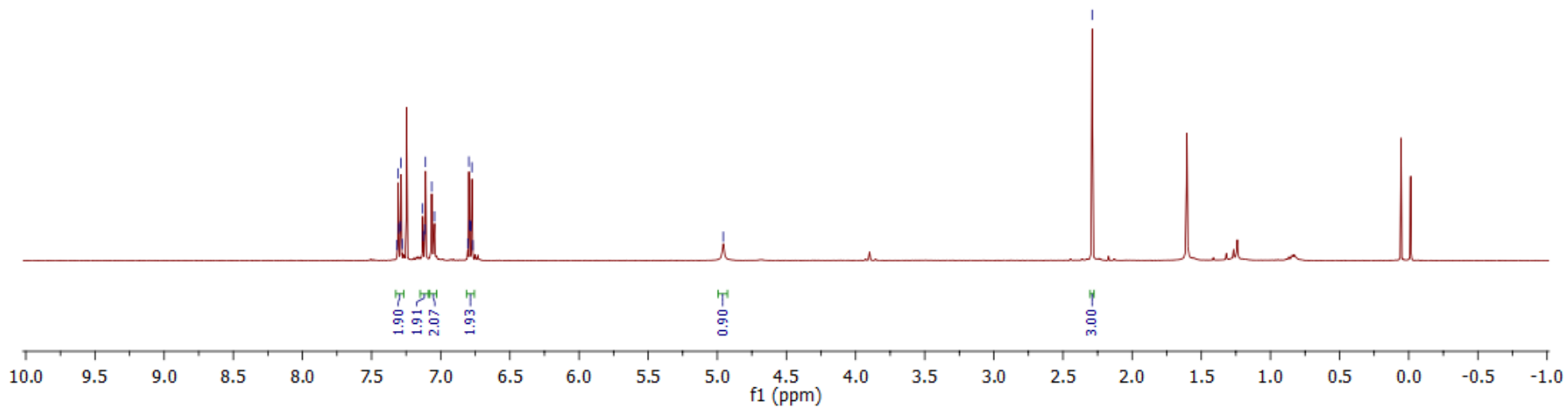


7.32  
7.31  
7.30  
7.29  
7.28  
7.13  
7.12  
7.11  
7.07  
7.04  
6.80  
6.80  
6.79  
6.78  
6.77

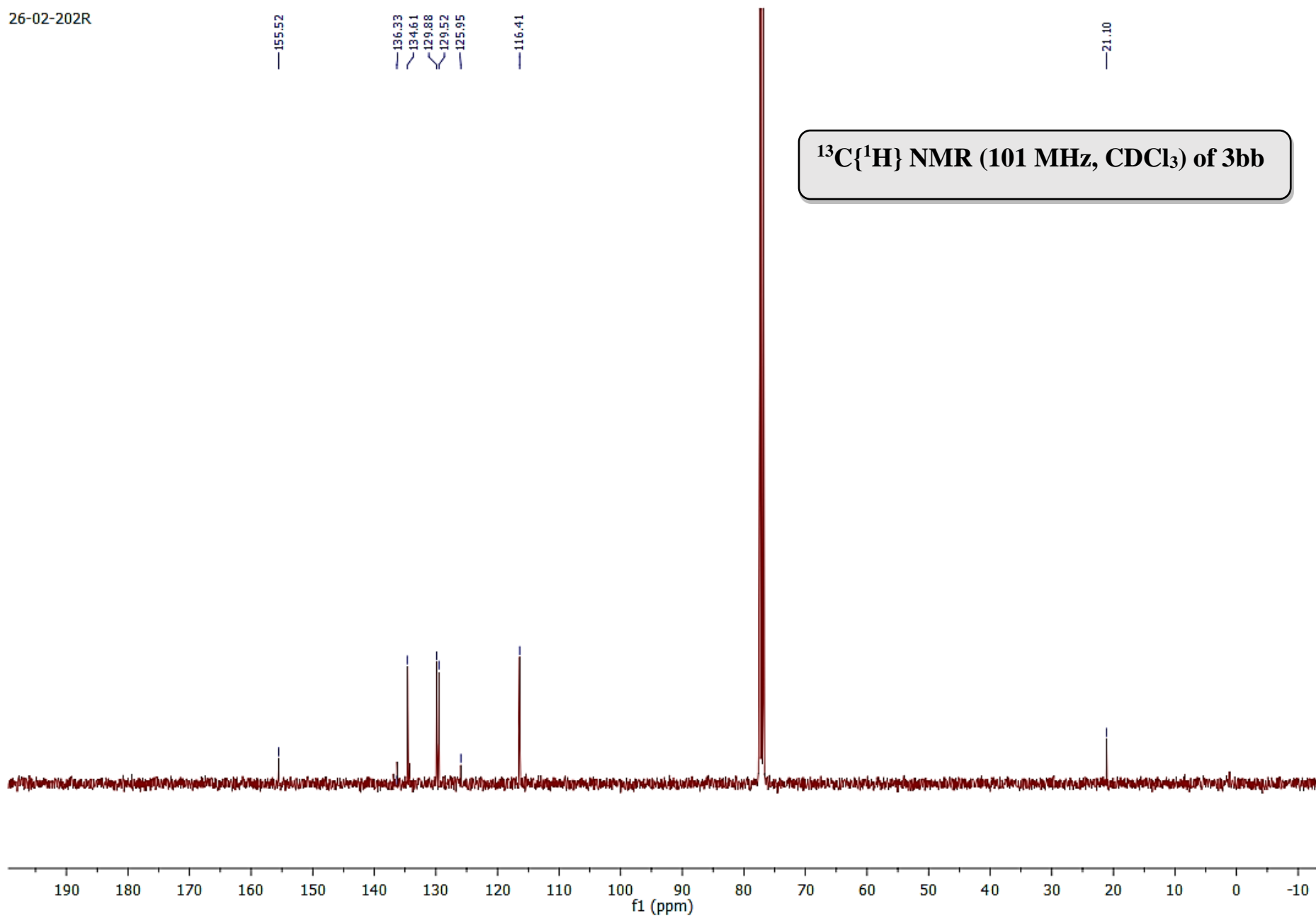
4.96

2.29

**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of 3bb**



26-02-202R





Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

46 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 1-100 H: 1-100 O: 0-10 S: 1-2

Sample Name : 26\_02\_202

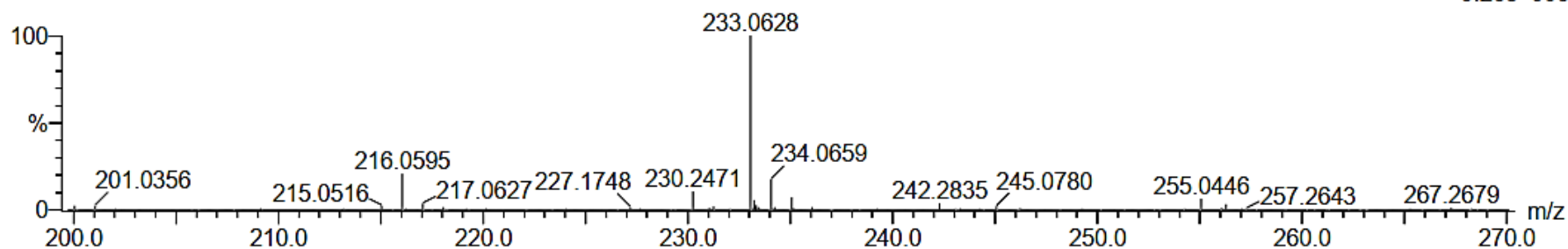
Test Name :

140622\_26\_02\_202 9 (0.117)

IITRPR

XEVO G2-XS QTOF

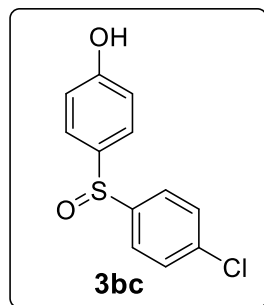
1: TOF MS ES+  
9.26e+006



Minimum: -1.5  
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
233.0628	233.0636	-0.8	-3.4	7.5	2246.2	n/a	n/a	C13 H13 O2 S

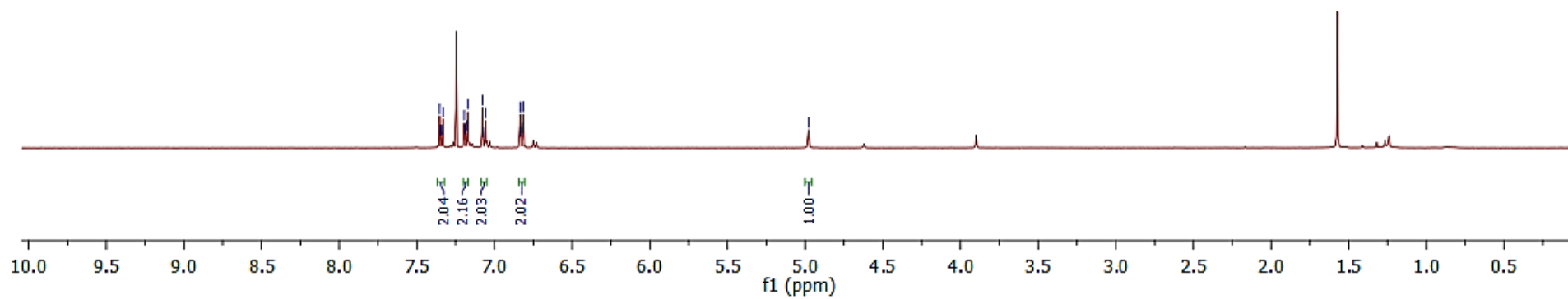
26-02-203-1



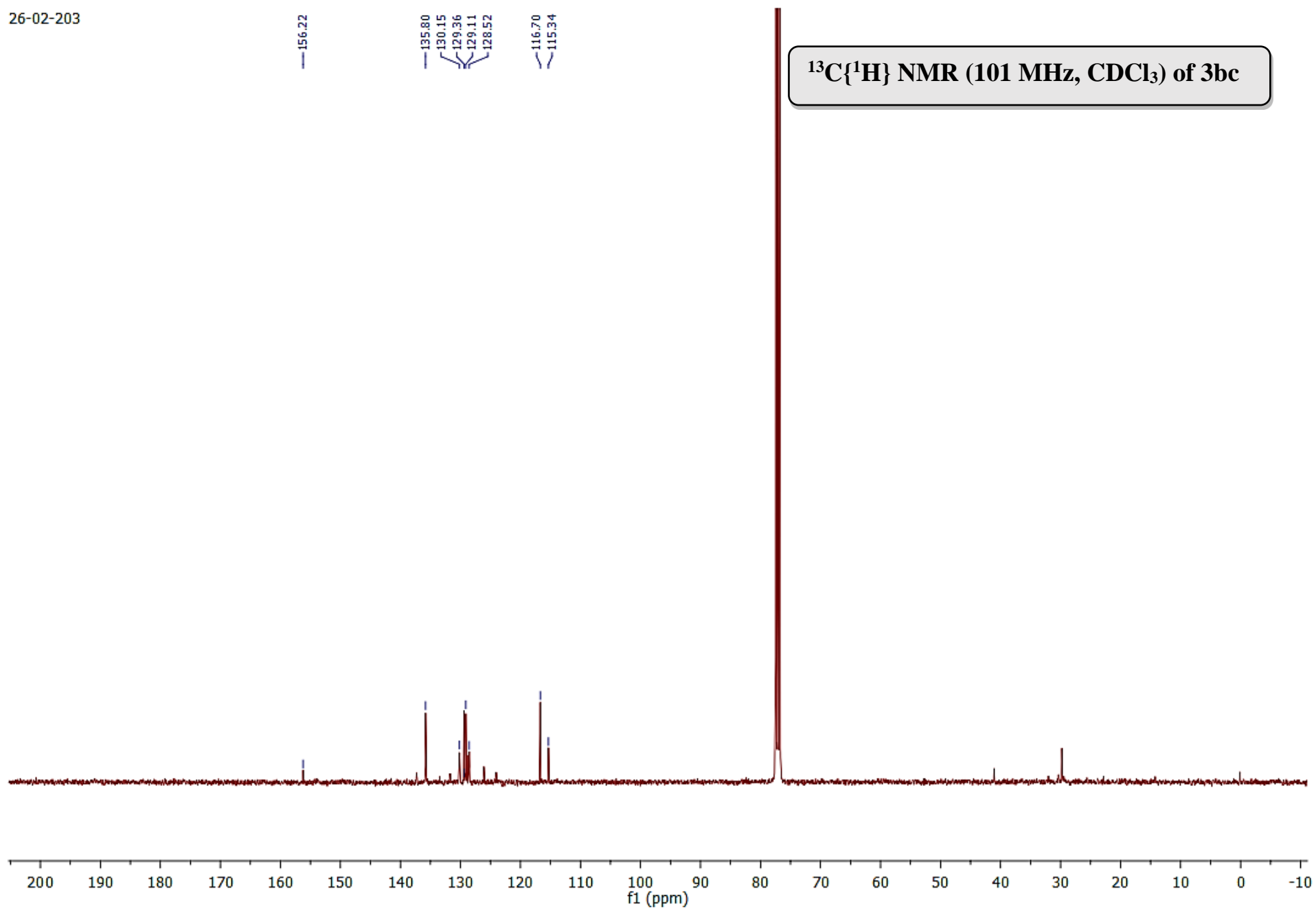
7.35  
7.35  
7.34  
7.33  
7.19  
7.19  
7.18  
7.17  
7.08  
7.07  
7.06  
6.84  
6.83  
6.82  
6.81

4.98

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3bc**



26-02-203



## Elemental Composition Report

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

38 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 1-100 H: 1-100 O: 0-10 S: 1-2 Cl: 1-1

Sample Name : 26\_02\_203

IITRPR

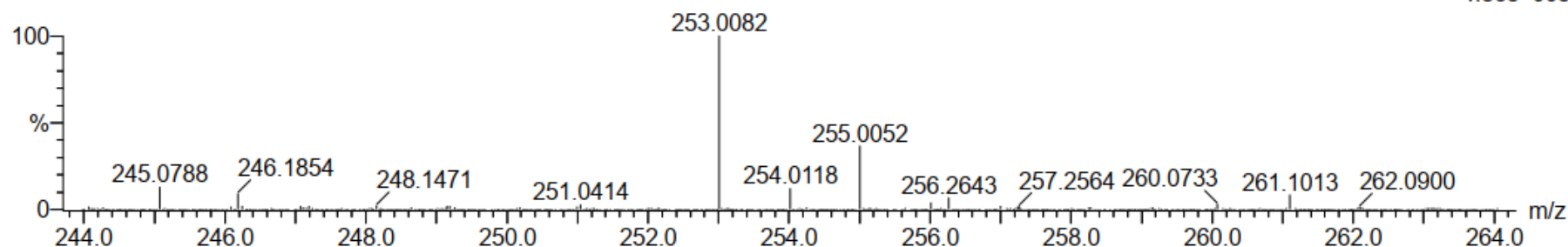
XEVO G2-XS QTOF

Test Name :

140622\_26\_02\_203 16 (0.177)

1: TOF MS ES+

4.86e+005



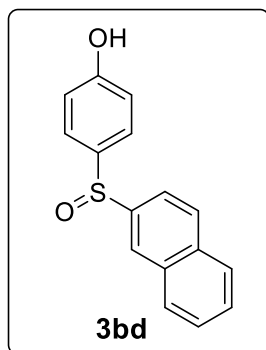
Minimum: -1.5  
 Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
253.0082	253.0090	-0.8	-3.2	7.5	1645.3	n/a	n/a	C12 H10 O2 S Cl

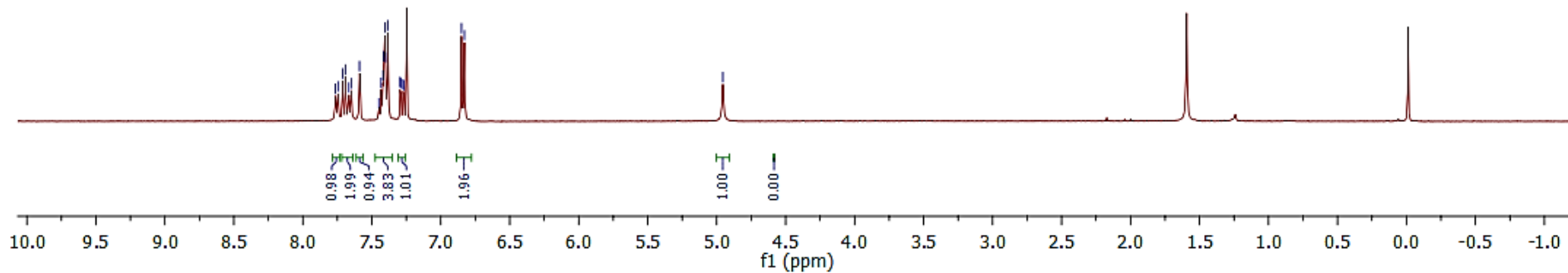
26-02-205R

7.76  
7.75  
7.71  
7.69  
7.67  
7.65  
7.59  
7.45  
7.43  
7.42  
7.41  
7.41  
7.40  
7.38  
7.30  
7.29  
7.27  
7.27  
6.85  
6.83

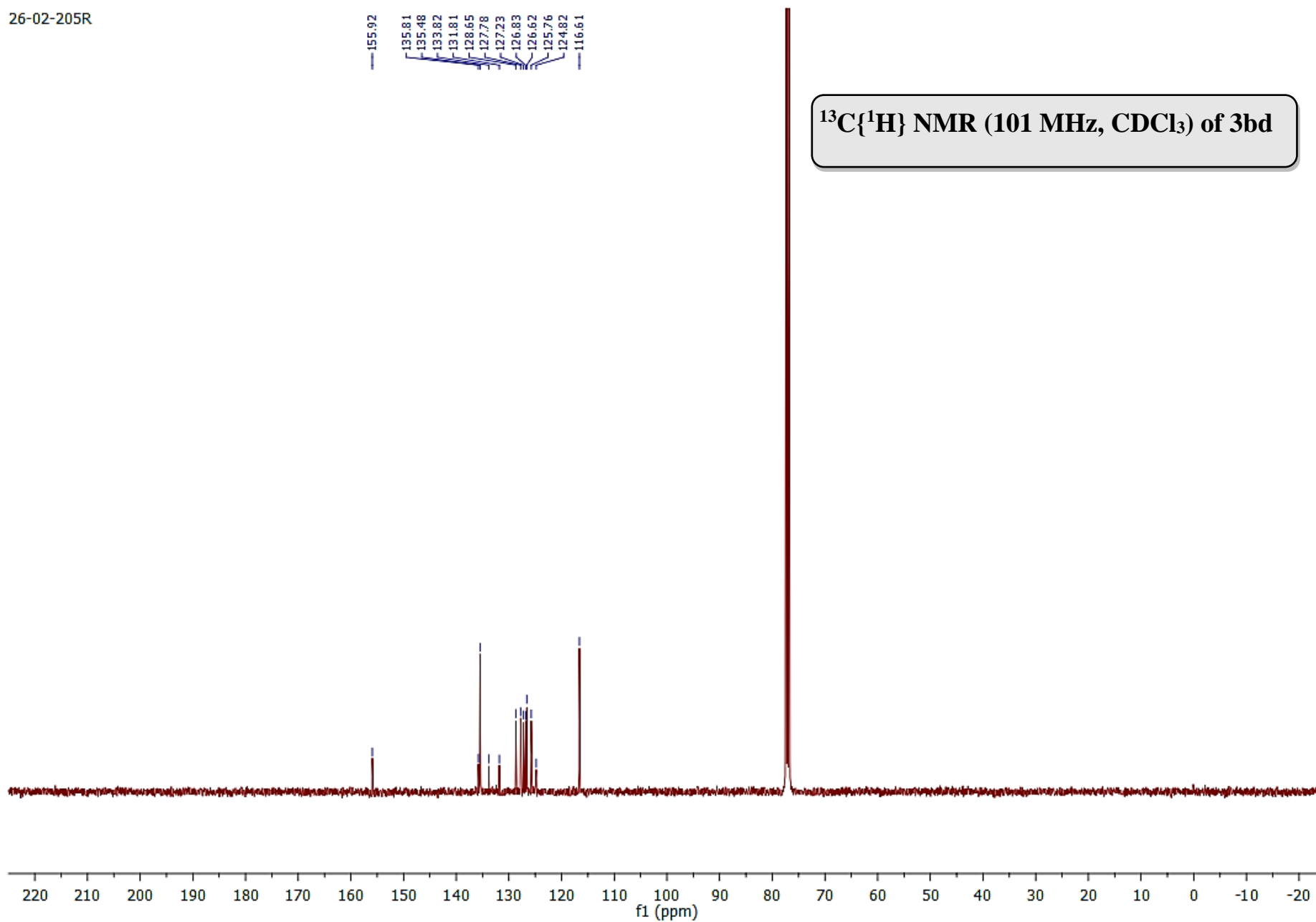
4.96



**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3bd**



26-02-205R



Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

58 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 1-100 H: 1-100 O: 0-10 S: 1-2

Sample Name : 26\_02\_205

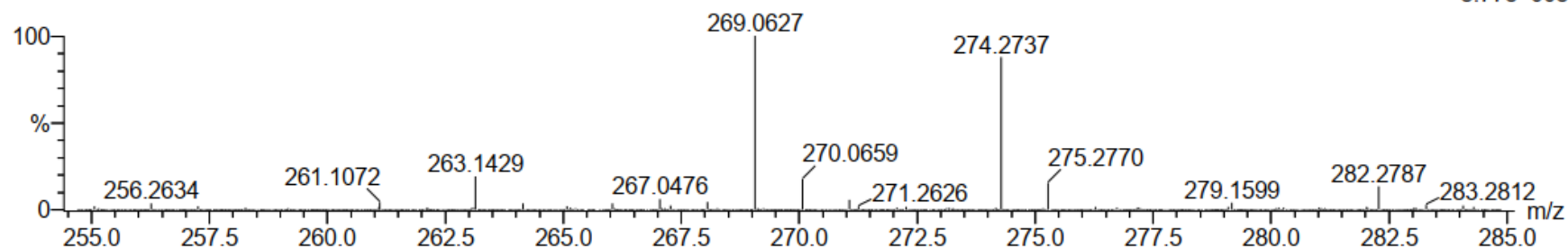
Test Name :

140622\_26\_02\_205 17 (0.197)

IITRPR

XEVO G2-XS QTOF

1: TOF MS ES+  
8.77e+005



Minimum: -1.5  
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
269.0627	269.0636	-0.9	-3.3	10.5	1670.1	n/a	n/a	C16 H13 O2 S

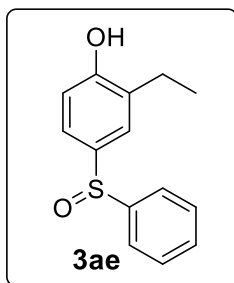
Z:\\26-02-169R

7.29  
7.28  
7.24  
7.24  
7.23  
7.22  
7.22  
7.21  
7.21  
7.20  
7.20  
7.19  
7.19  
7.15  
7.15  
7.15  
7.14  
7.13  
7.13  
7.13  
7.12  
7.12  
7.11  
7.10  
7.10  
7.10

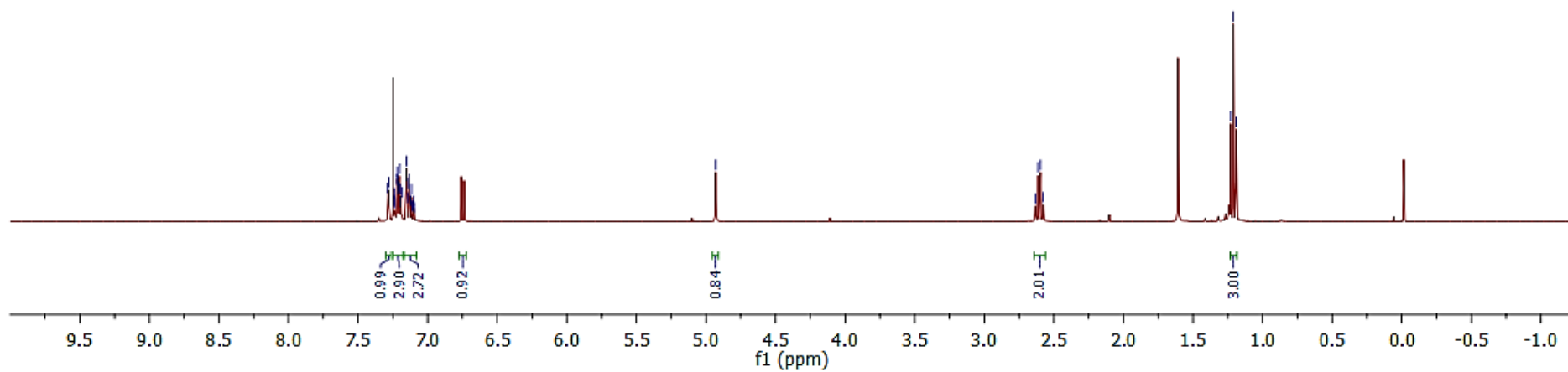
4.93

2.63  
2.61  
2.60  
2.58

1.23  
1.21  
1.19

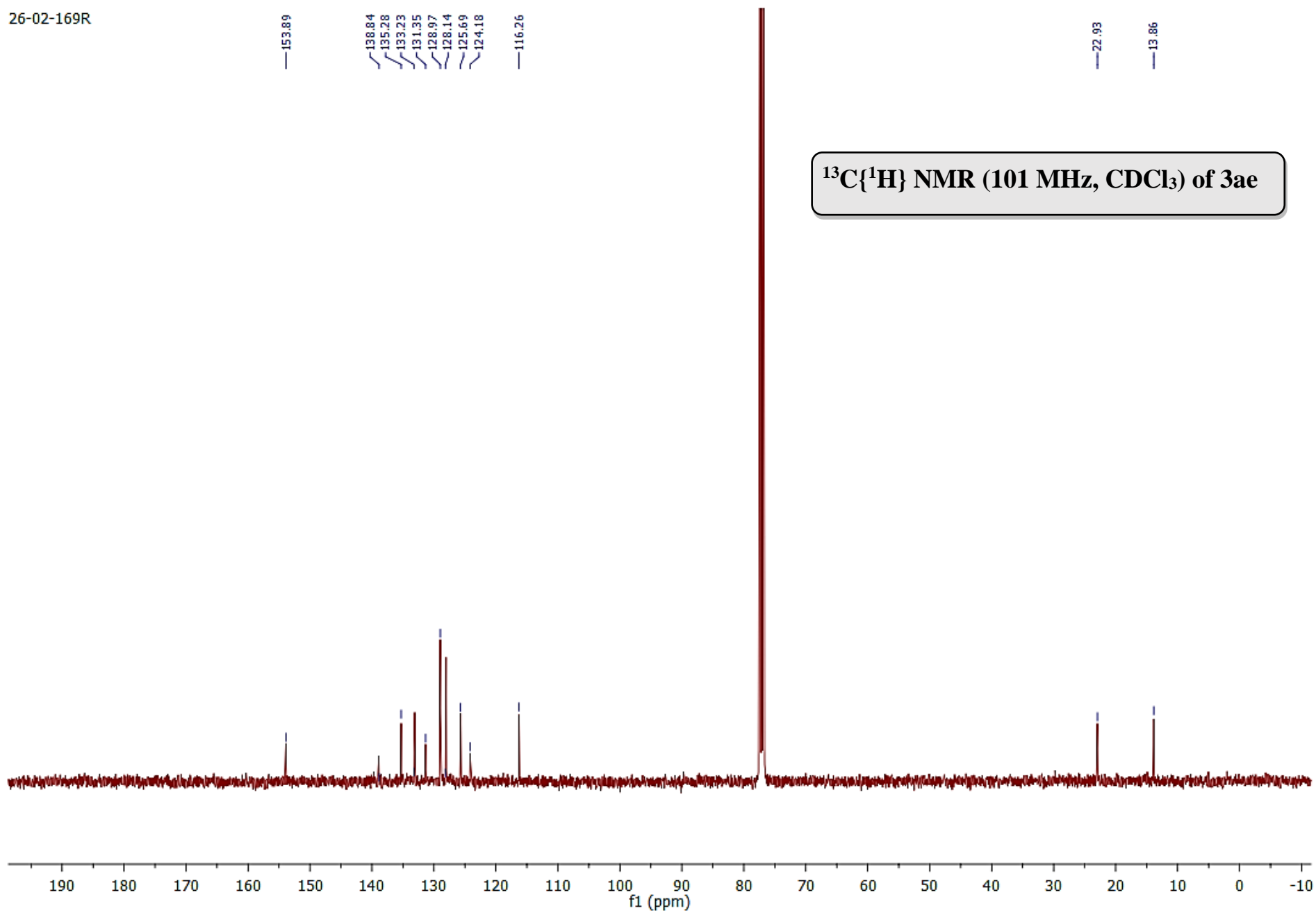


**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 3ae**





26-02-169R



# HRMS of 3ae

## Elemental Composition Report

Page 1

### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

117 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 O: 0-10 S: 0-2 I: 0-3

Sample Name : 26\_02\_169

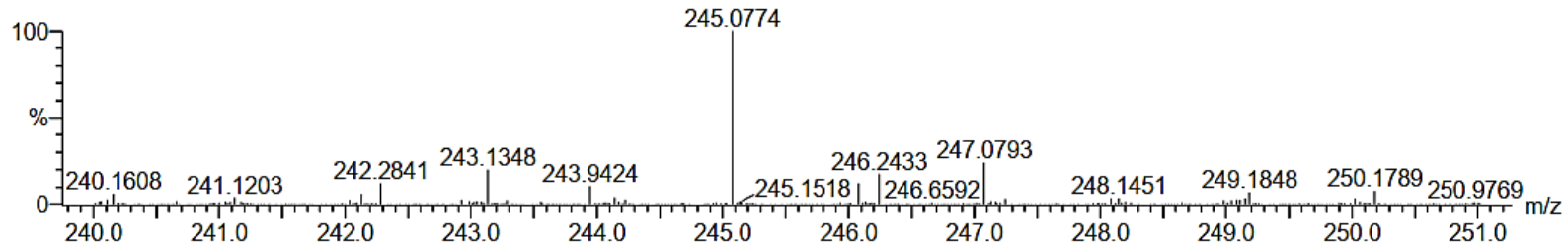
IITRPR

XEVO G2-XS QTOF

Test Name :

100522\_26\_02\_169 16 (0.356)

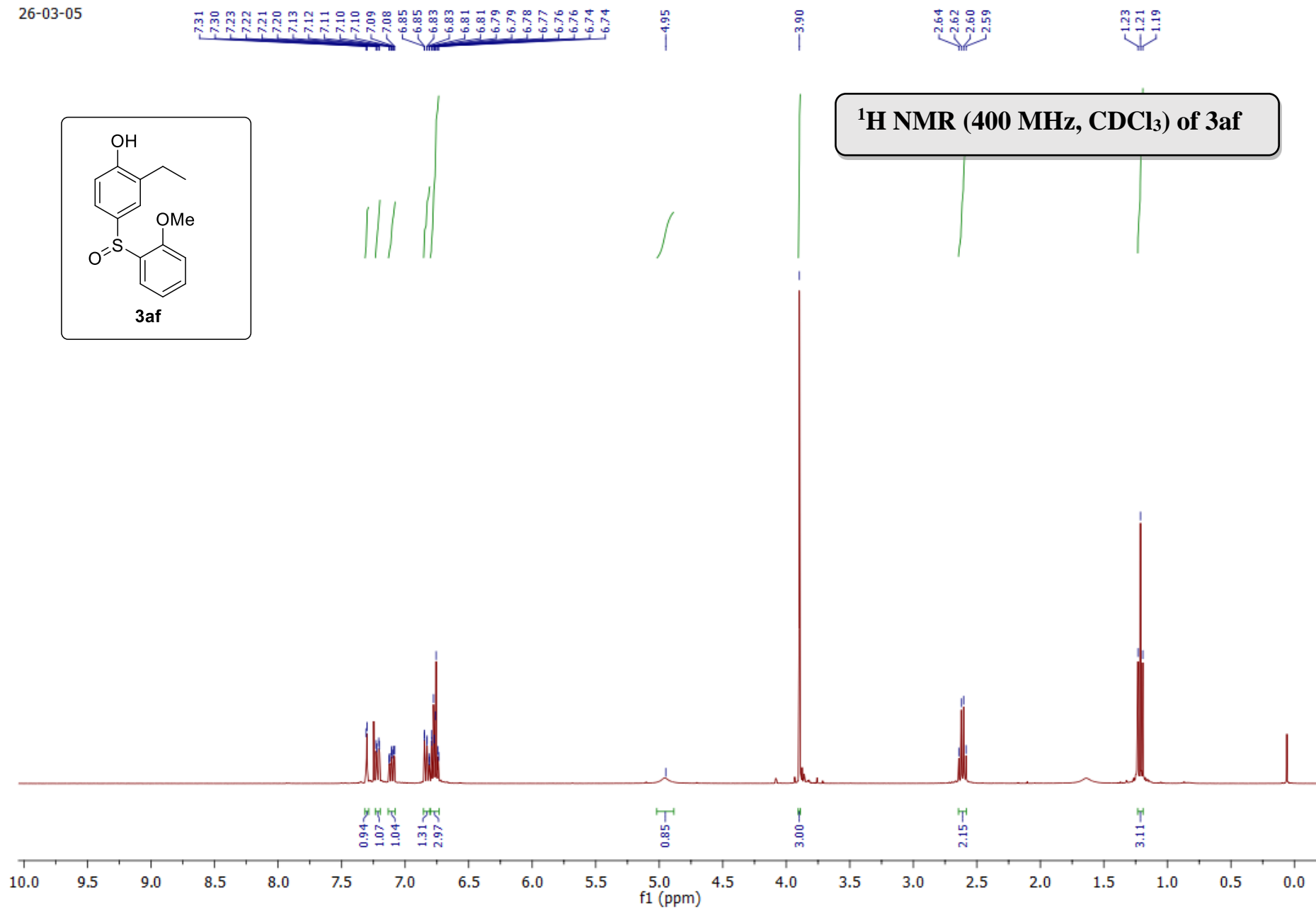
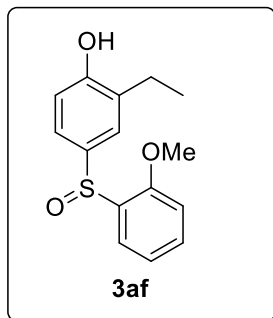
1: TOF MS ES+  
1.76e+005



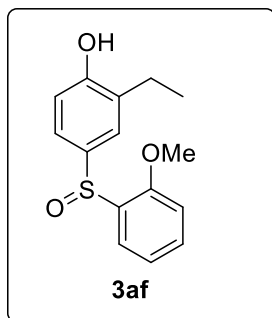
Minimum: -1.5  
Maximum: 2.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
247.0793	247.0793	0.0	0.0	7.5	1429.5	n/a	n/a	C14 H15 O2 S

26-03-05



26-03-05



155.56  
154.06

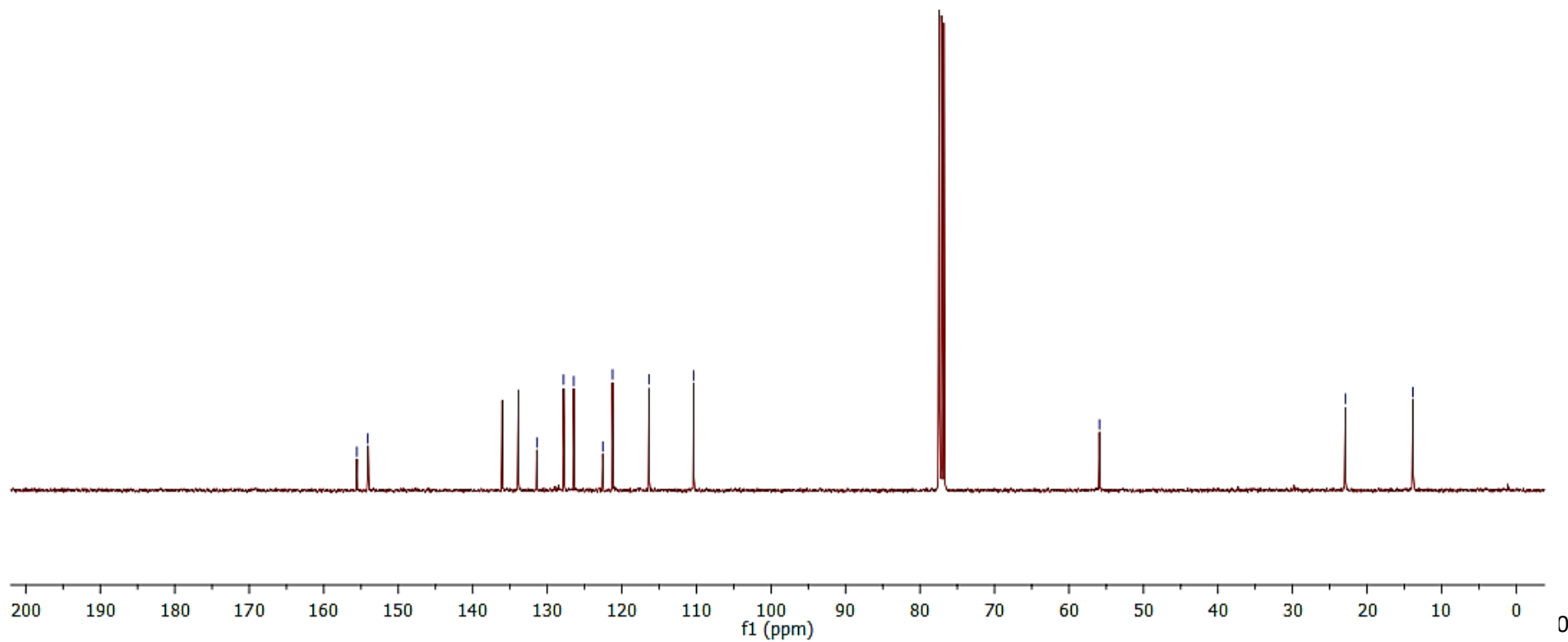
131.41  
127.82  
126.46  
122.56  
121.26  
116.34  
110.37

55.92

22.91

13.83

<sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, CDCl<sub>3</sub>) of 3af



Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

282 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-5 O: 0-6 S: 0-1

Sample Name : 26\_02\_125

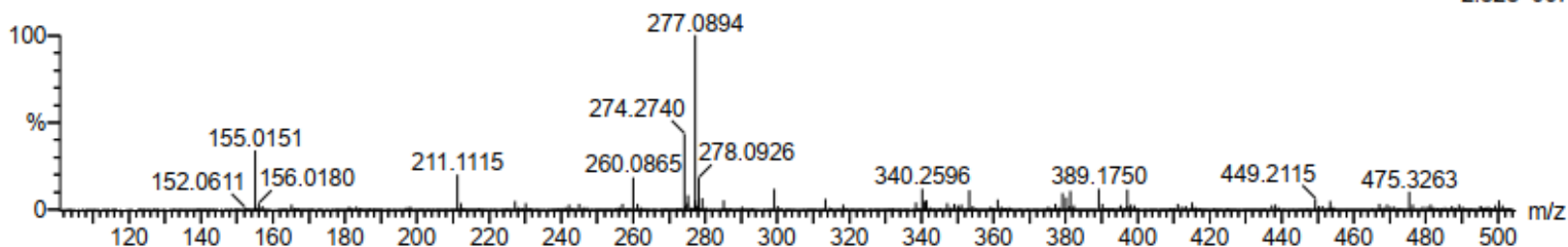
IITRPR

XEVO G2-XS QTOF

Test Name :

01042022\_26\_02\_125 5 (0.124)

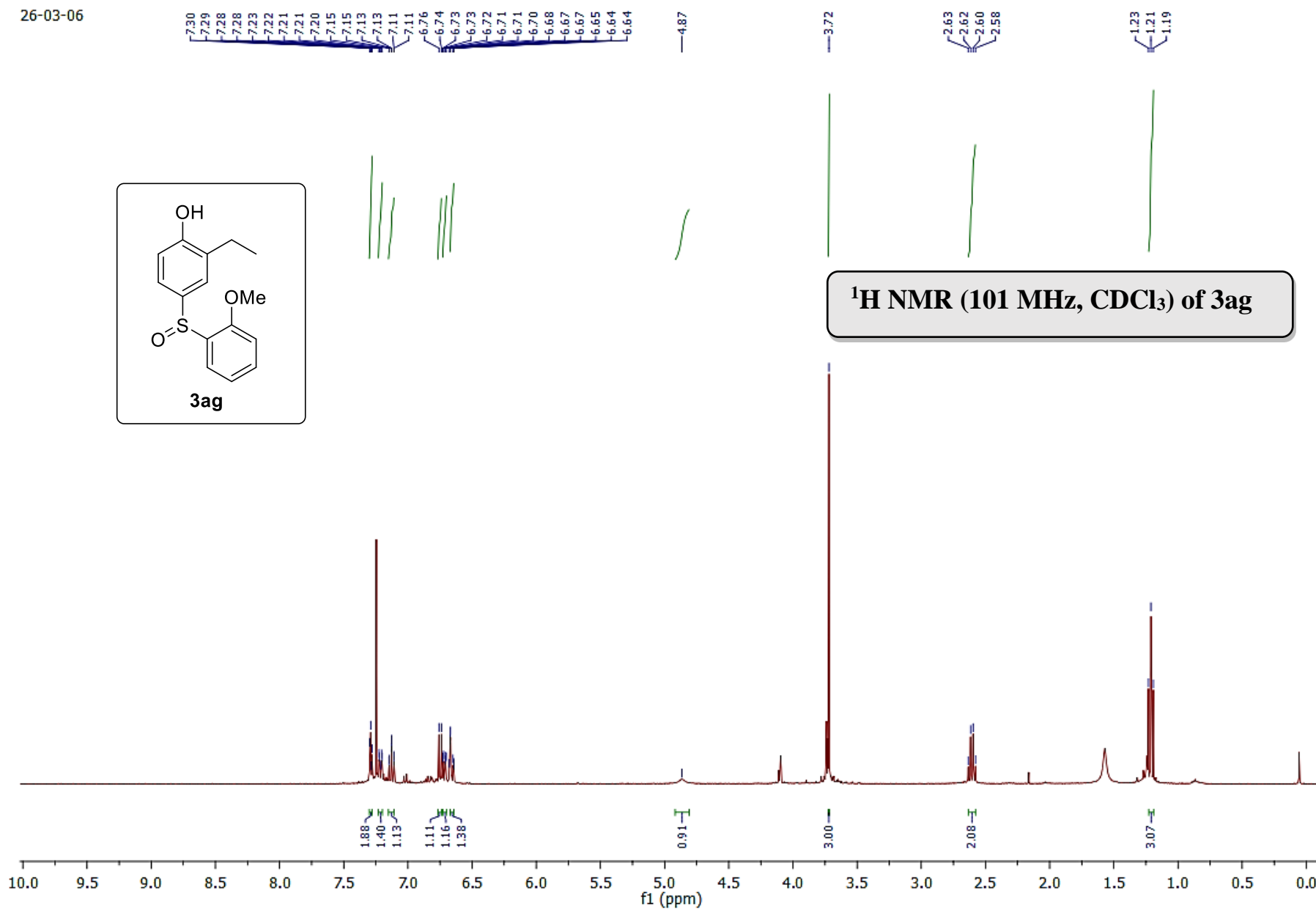
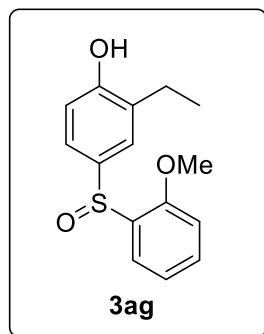
1: TOF MS ES+  
2.32e+007



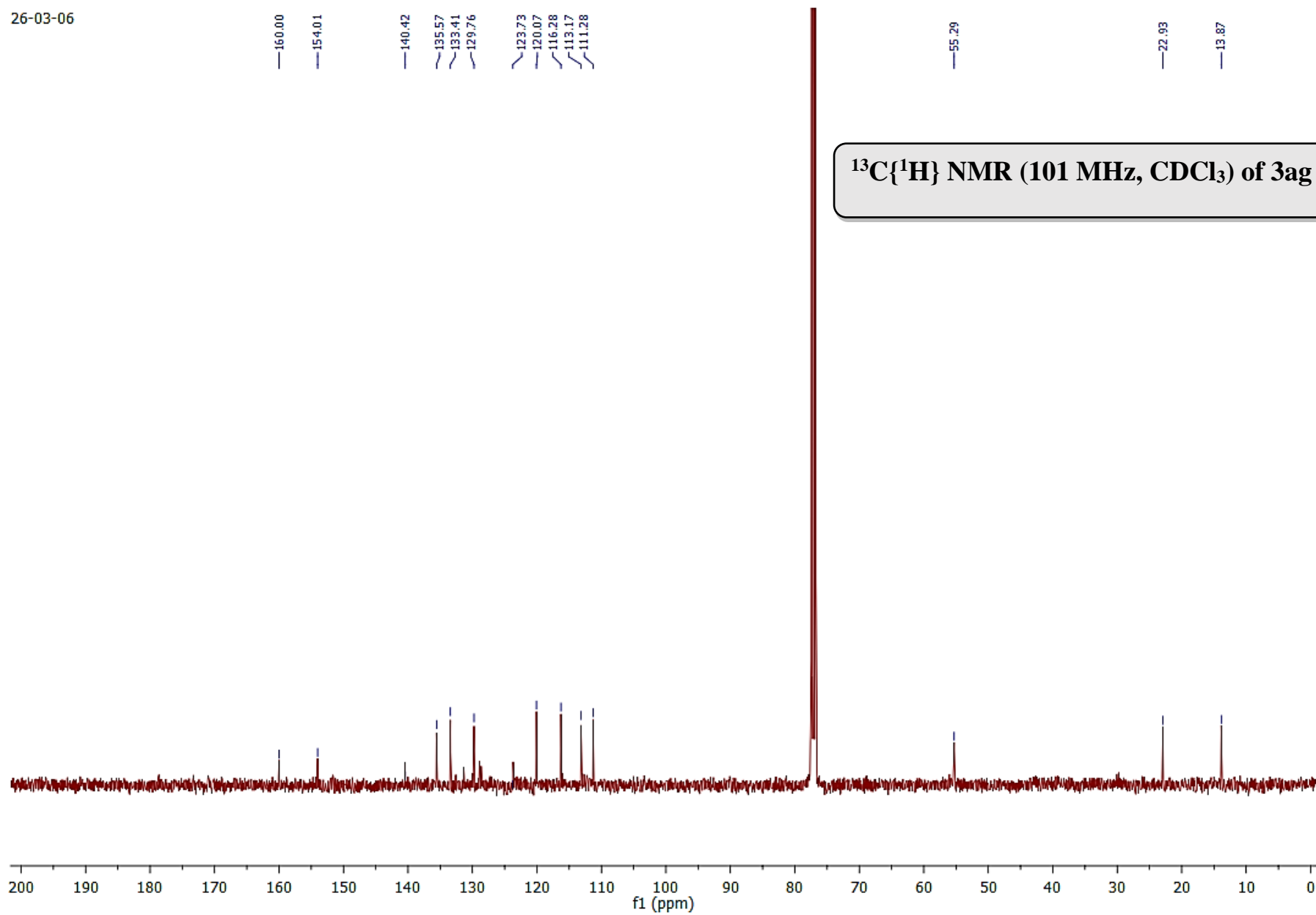
Minimum: -1.5  
Maximum: 2.0 10.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
277.0894	277.0898	-0.4	-1.4	7.5	1051.2	n/a	n/a	C15 H17 O3 S

26-03-06



26-03-06



Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions

282 formula(e) evaluated with 1 results within limits (up to 1 closest results for each mass)

Elements Used:

C: 0-50 H: 0-50 N: 0-5 O: 0-6 S: 0-1

Sample Name : 26\_02\_125

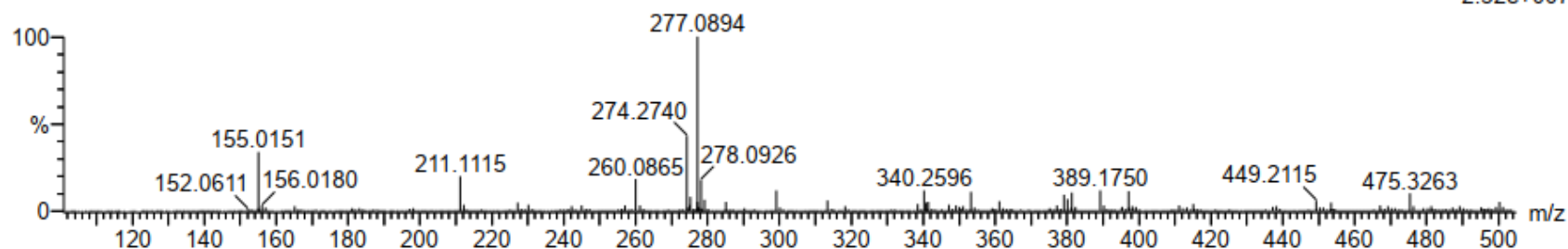
IITRPR

XEVO G2-XS QTOF

Test Name :

01042022\_26\_02\_125 5 (0.124)

1: TOF MS ES+  
2.32e+007

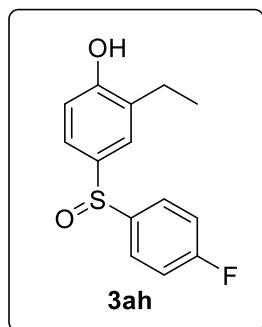


Minimum: -1.5  
Maximum: 2.0 10.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
277.0894	277.0898	-0.4	-1.4	7.5	1051.2	n/a	n/a	C15 H17 O3 S



26-03-10



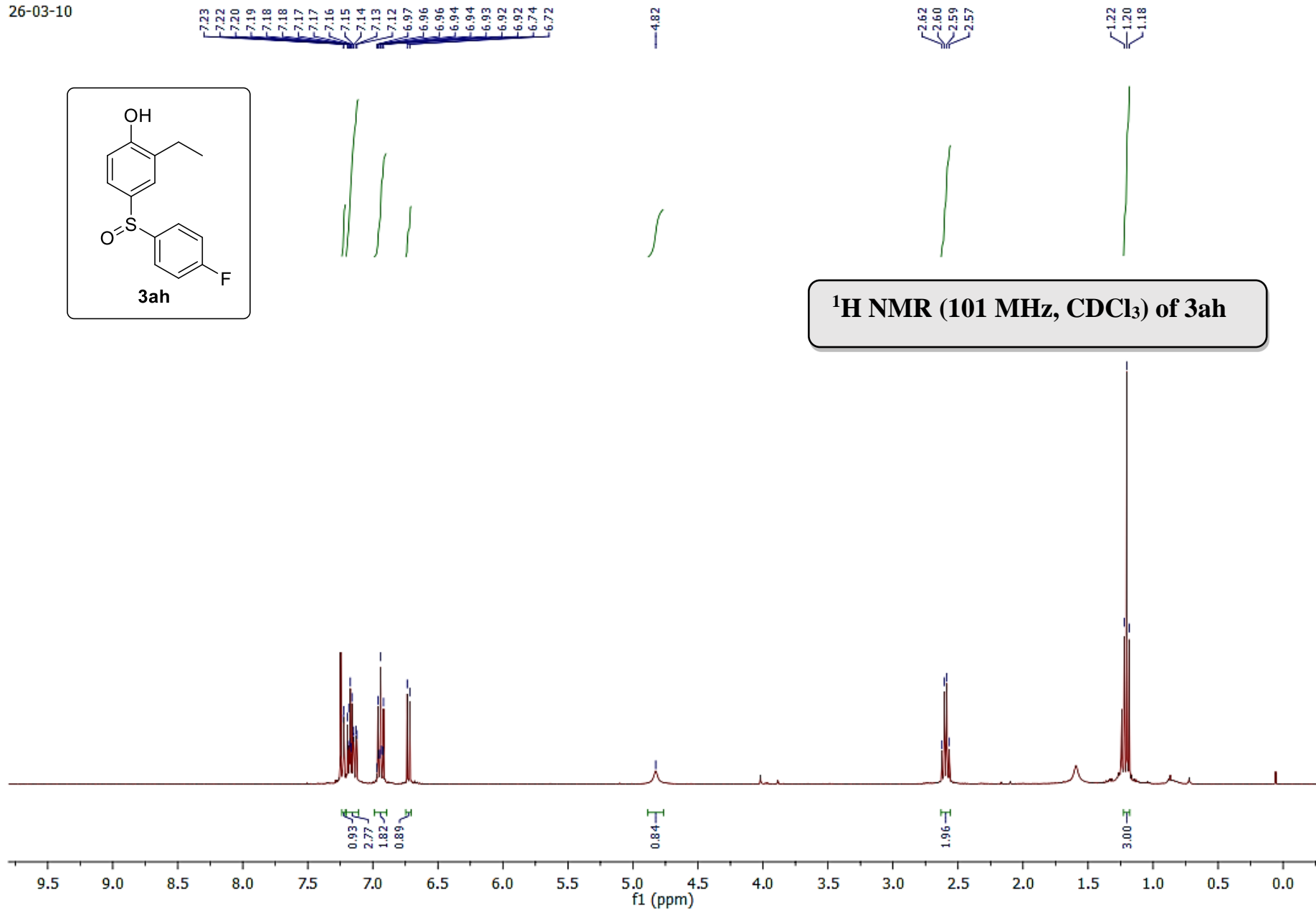
7.23  
7.22  
7.20  
7.19  
7.18  
7.18  
7.17  
7.17  
7.16  
7.15  
7.14  
7.13  
7.12  
6.97  
6.96  
6.96  
6.94  
6.94  
6.93  
6.92  
6.92  
6.74  
6.72

4.82

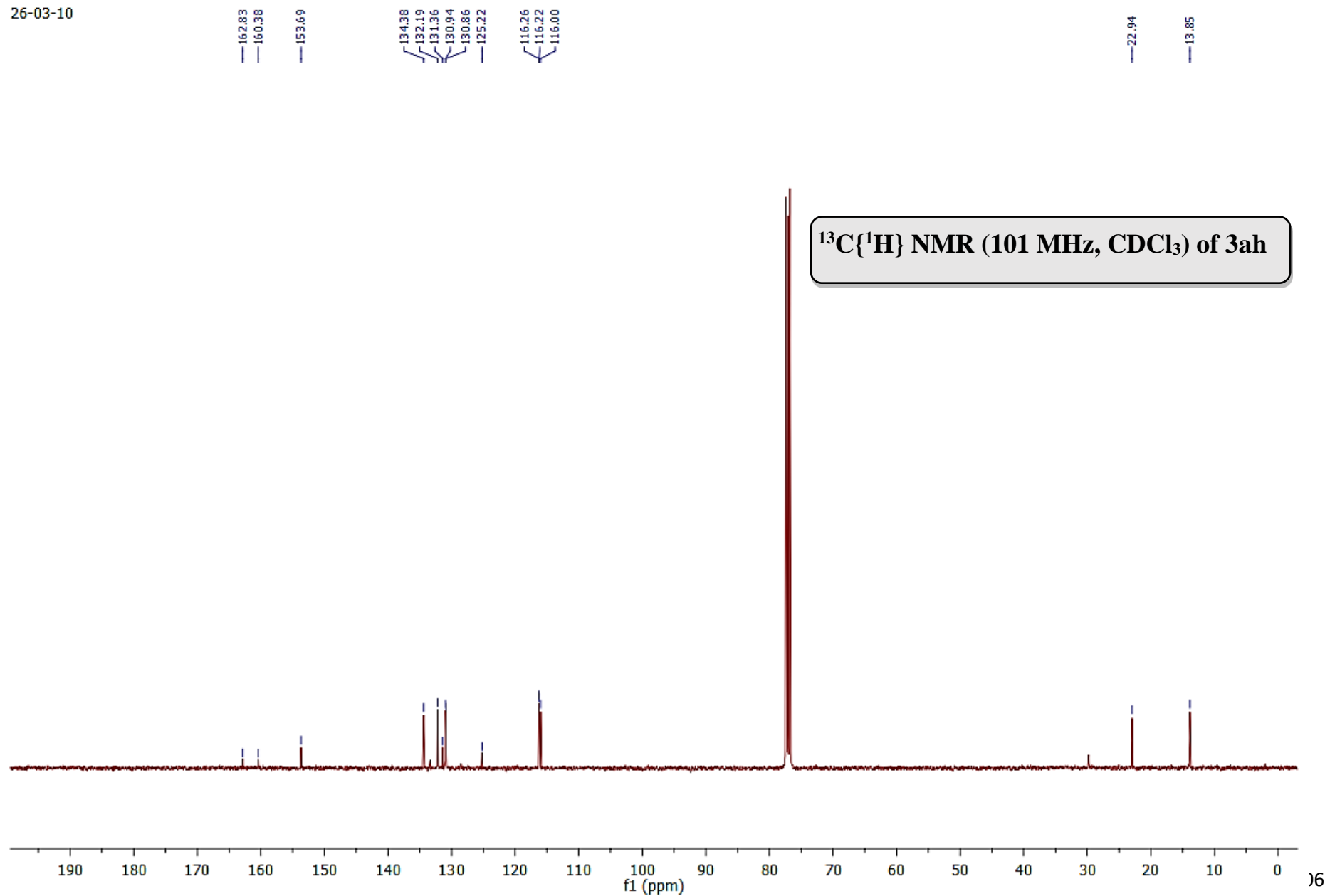
2.62  
2.60  
2.59  
2.57

1.22  
1.20  
1.18

**<sup>1</sup>H NMR (101 MHz, CDCl<sub>3</sub>) of 3ah**



26-03-10



Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

10 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

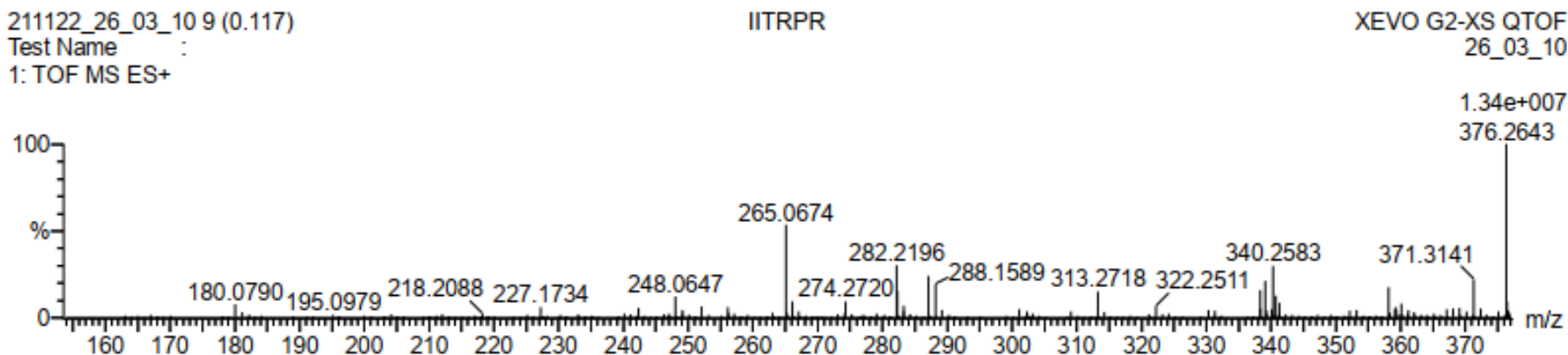
Elements Used:

C: 0-14 H: 0-14 O: 0-2 F: 0-1 S: 0-1

211122\_26\_03\_10 9 (0.117)

Test Name :

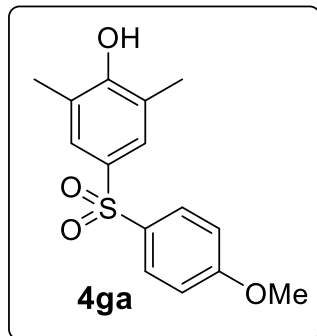
1: TOF MS ES+



Minimum: -1.5  
Maximum: 5.0 10.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
265.0674	265.0699	-2.5	-9.4	7.5	1212.7	n/a	n/a	C14 H14 O2 F S

26-02-248

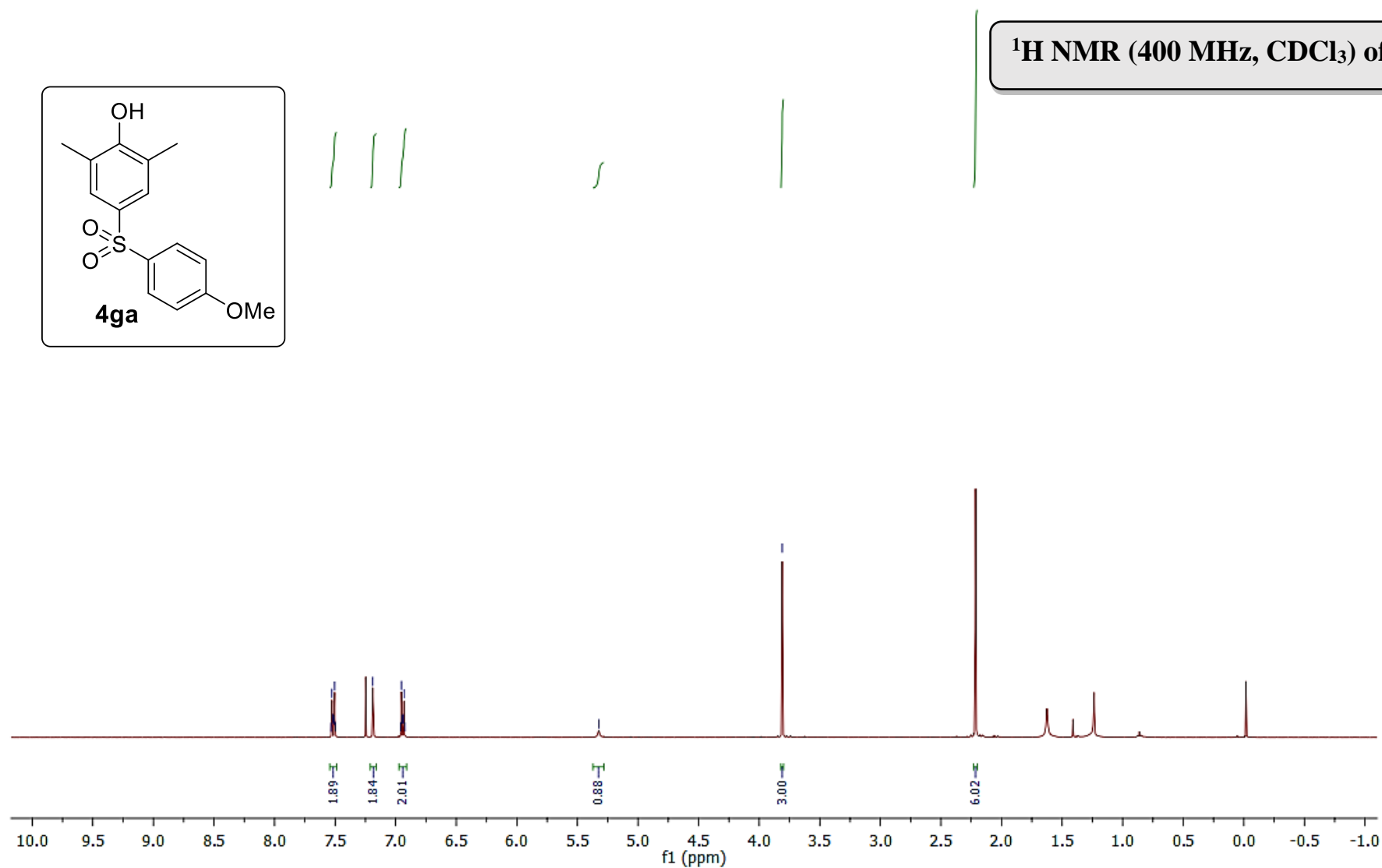


7.53  
7.53  
7.52  
7.51  
7.50  
7.19  
6.96  
6.95  
6.94  
6.93

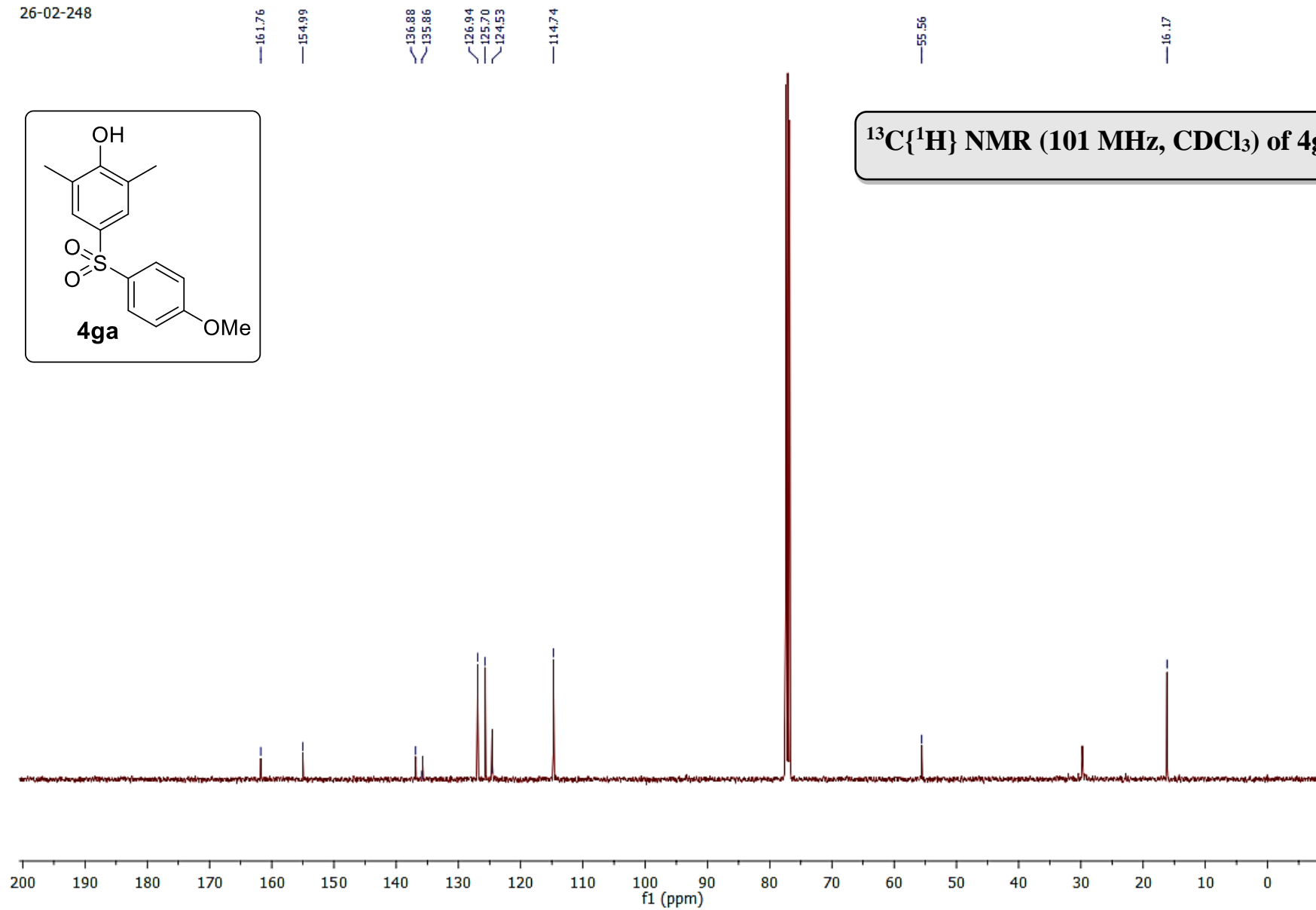
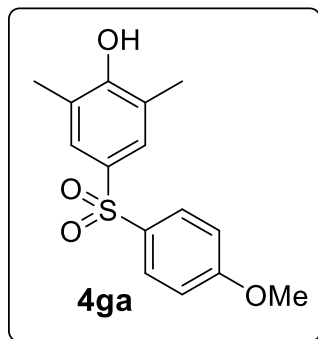
5.32

3.81

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of 4ga**



26-02-248



Elemental Composition Report

Single Mass Analysis

Tolerance = 100.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

2 formula(e) evaluated with 1 results within limits (up to 100 best isotopic matches for each mass)

Elements Used:

C: 15-15 H: 1-100 O: 4-4 S: 0-1

Sample Name : 26-02-247

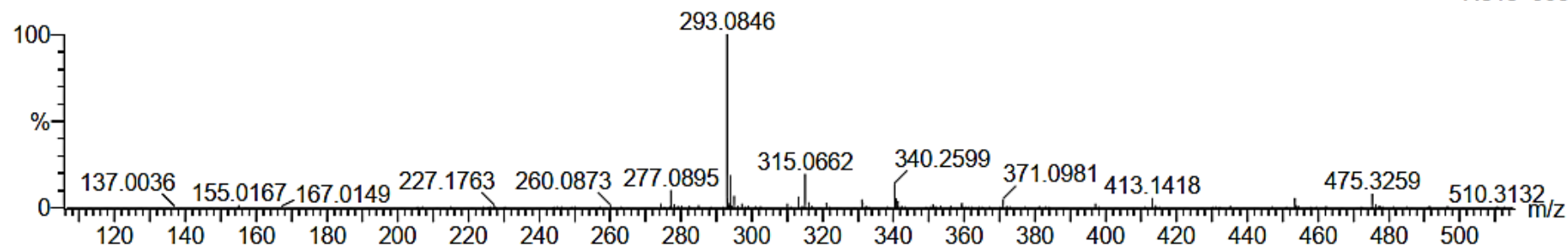
Test Name :

080922\_26-02-247-HRMS 16 (0.177)

IITRPR

XEVO G2-XS QTOF

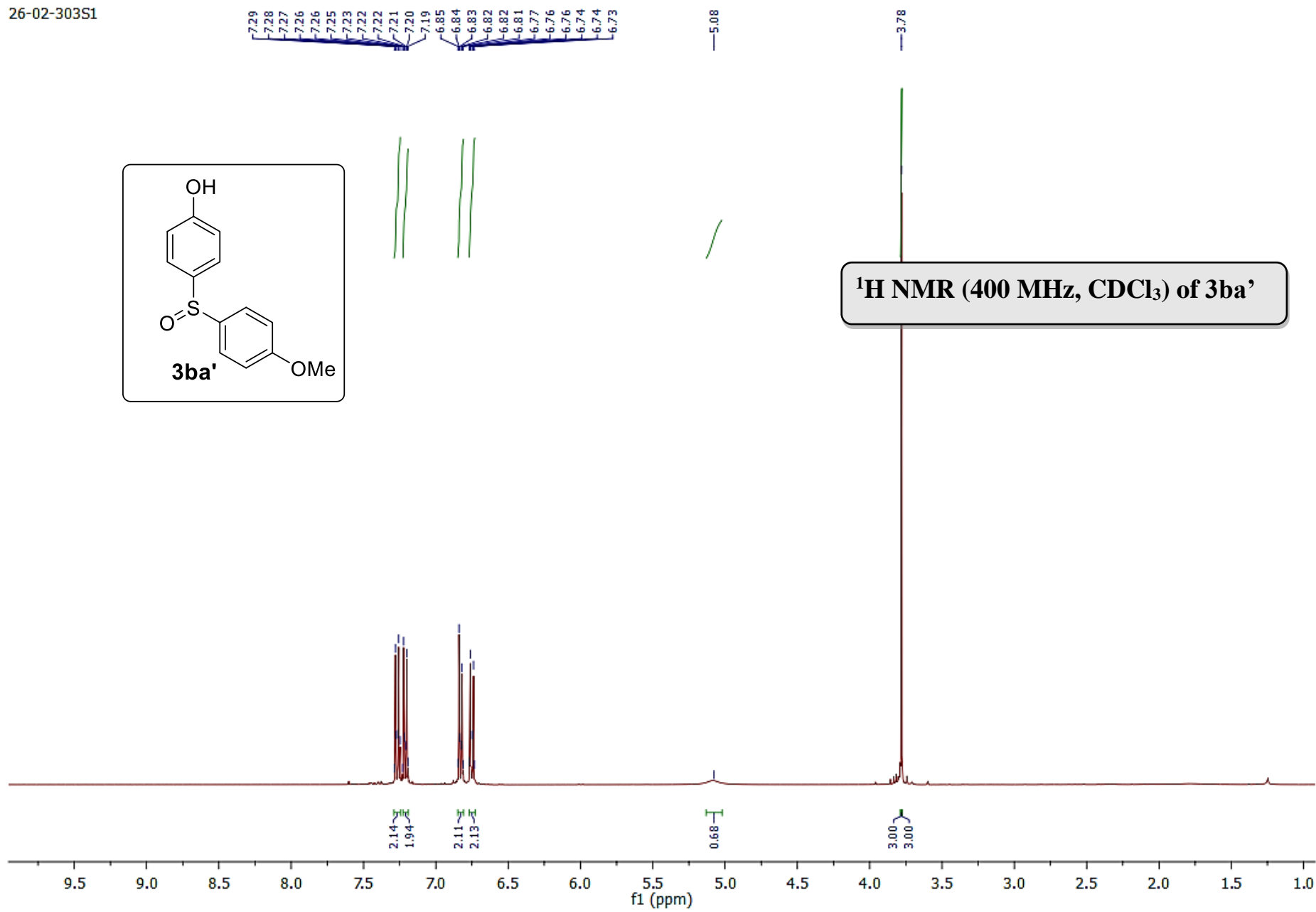
1: TOF MS ES+  
7.51e+006

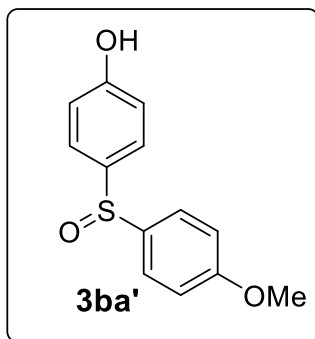


Minimum: -1.5  
Maximum: 10.0 100.0 50.0

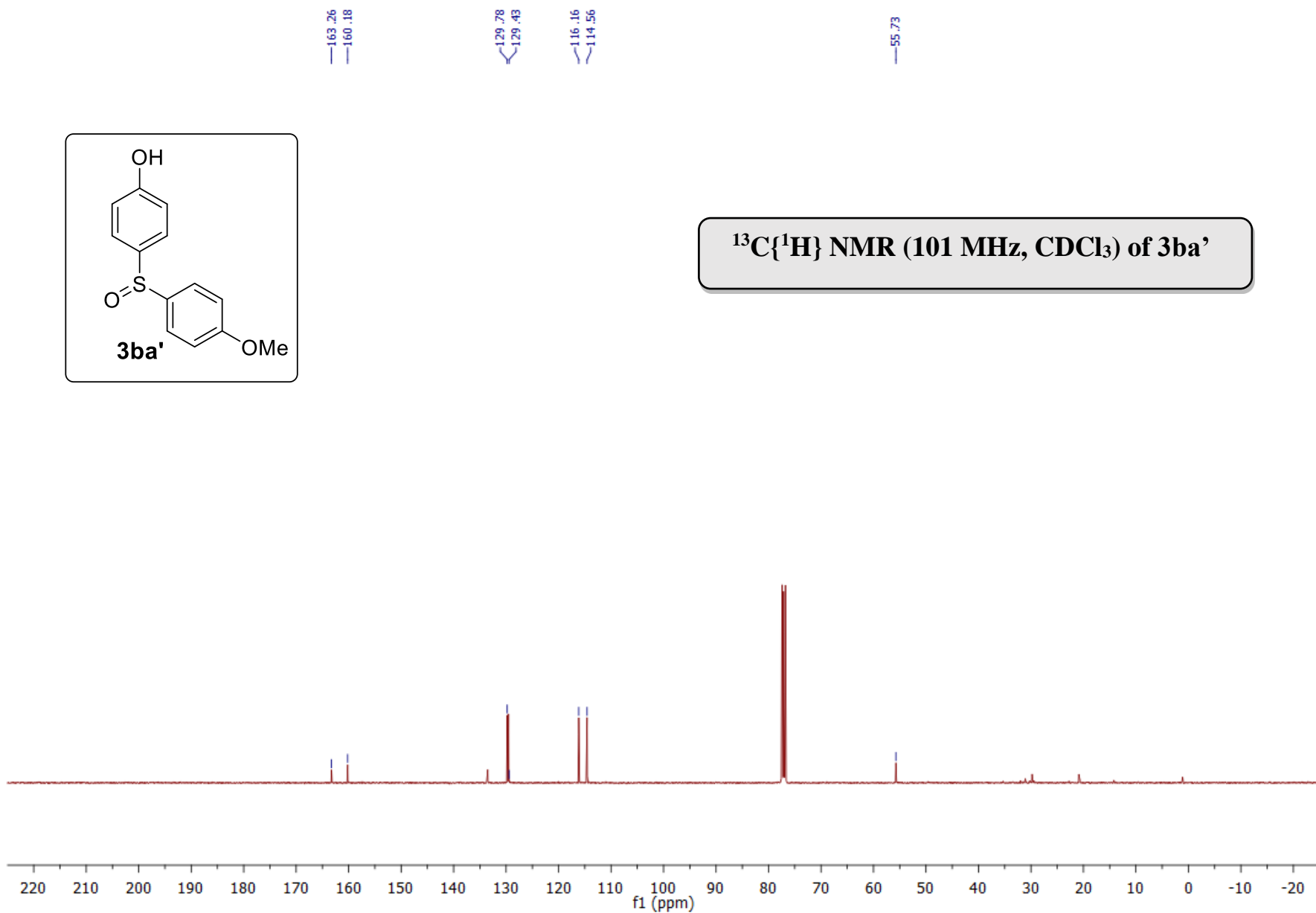
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
293.0846	293.0848	-0.2	-0.7	7.5	1073.4	n/a	n/a	C15 H17 O4 S

26-02-303S1





**$^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz,  $\text{CDCl}_3$ ) of 3ba'**





**HRMS of 3ba'**

**Elemental Composition Report**

**Single Mass Analysis**

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

8 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-13 H: 0-15 O: 0-4 S: 0-1

181122\_26\_02\_304R 14 (0.160)

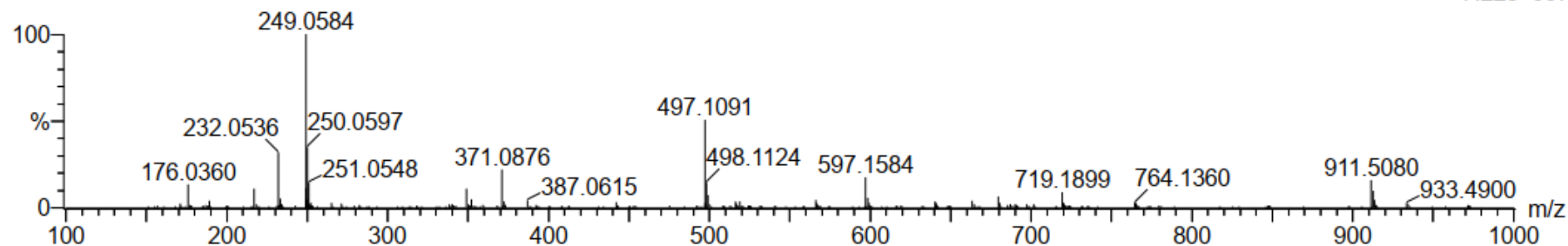
IITRPR

XEVO G2-XS QTOF  
26\_02\_304R

Test Name :

1: TOF MS ES+

7.22e+007



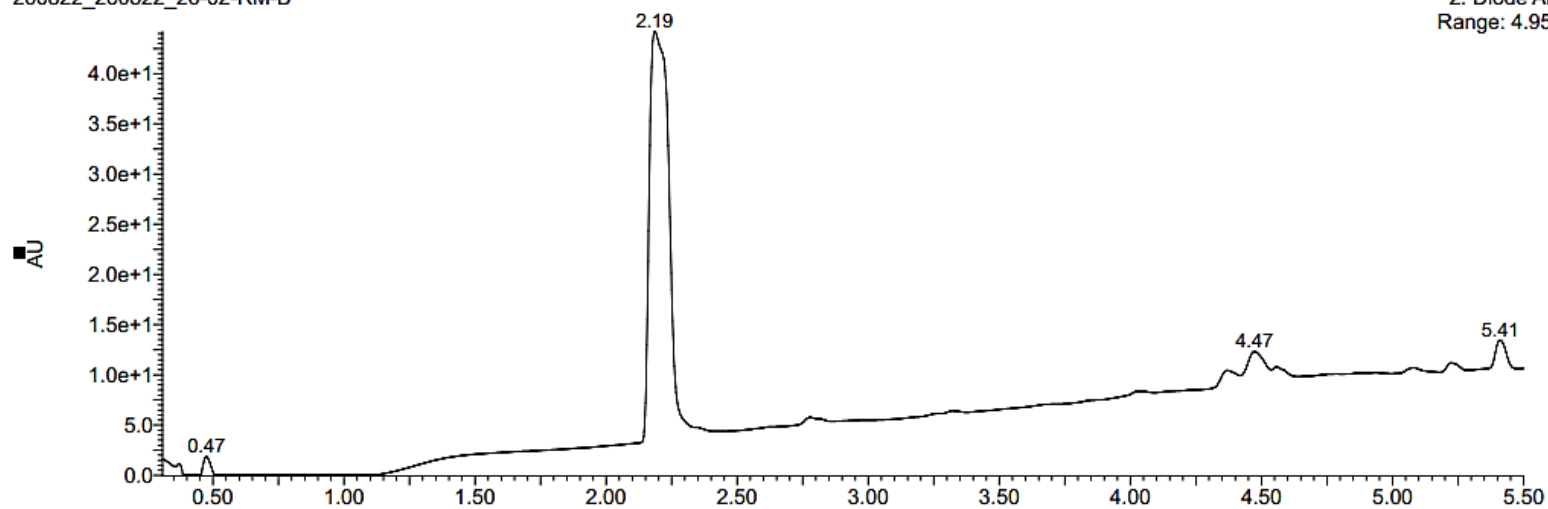
Minimum: -1.5  
Maximum: 5.0 10.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
249.0584	249.0585	-0.1	-0.4	7.5	1626.7	n/a	n/a	C13 H13 O3 S

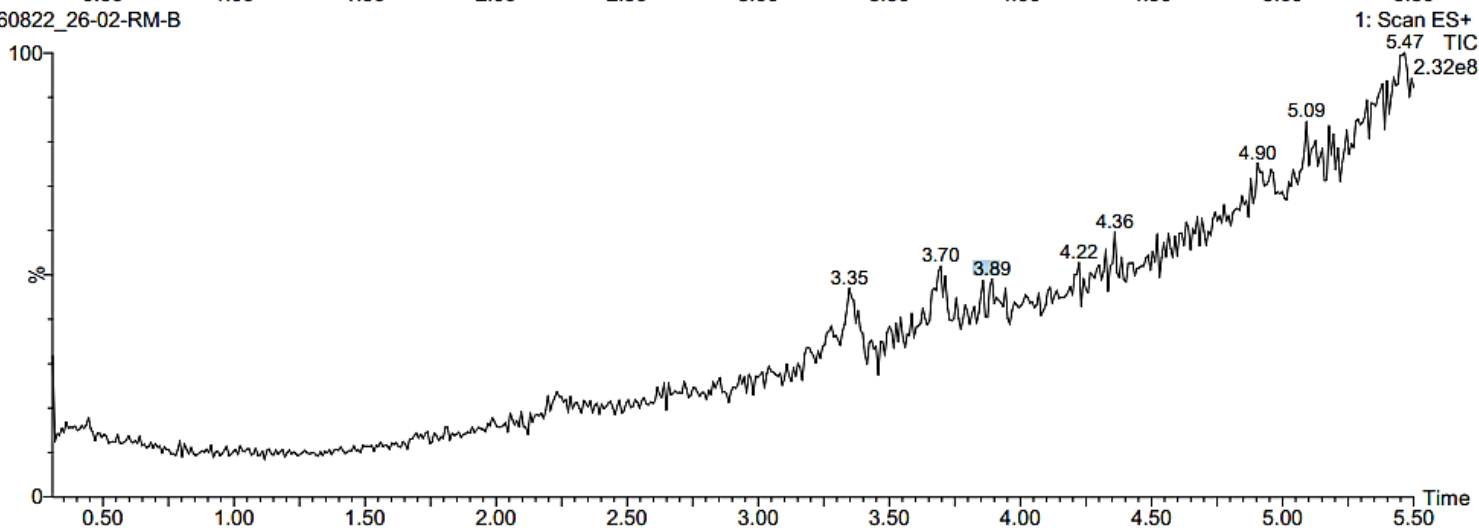
# LC-MS of Reaction Mixture

26-02-RM-B  
260822\_260822\_26-02-RM-B

2: Diode Array  
Range: 4.95e+1



260822\_260822\_26-02-RM-B

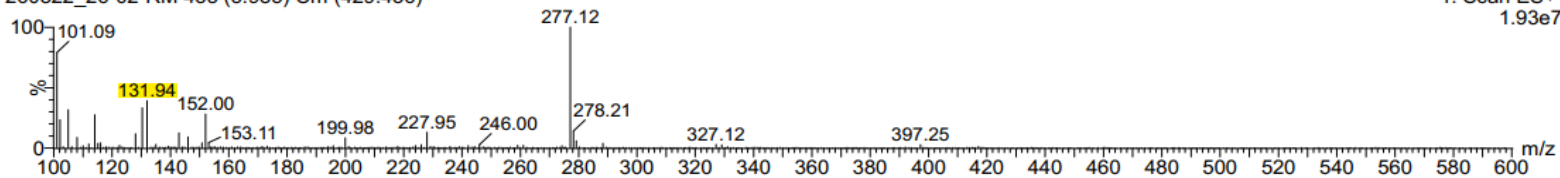


## LC-MS Spectra of Reaction Mixture

$[M+Na] = 131.90$  for Benzyl alcohol

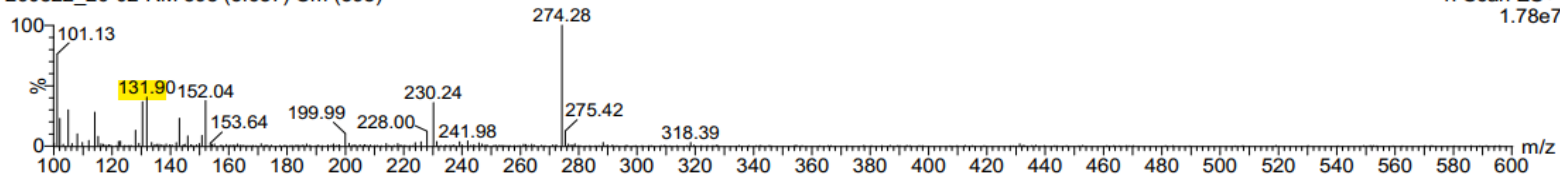
260822\_26-02-RM 433 (3.985) Cm (429:436)

1: Scan ES+  
1.93e7



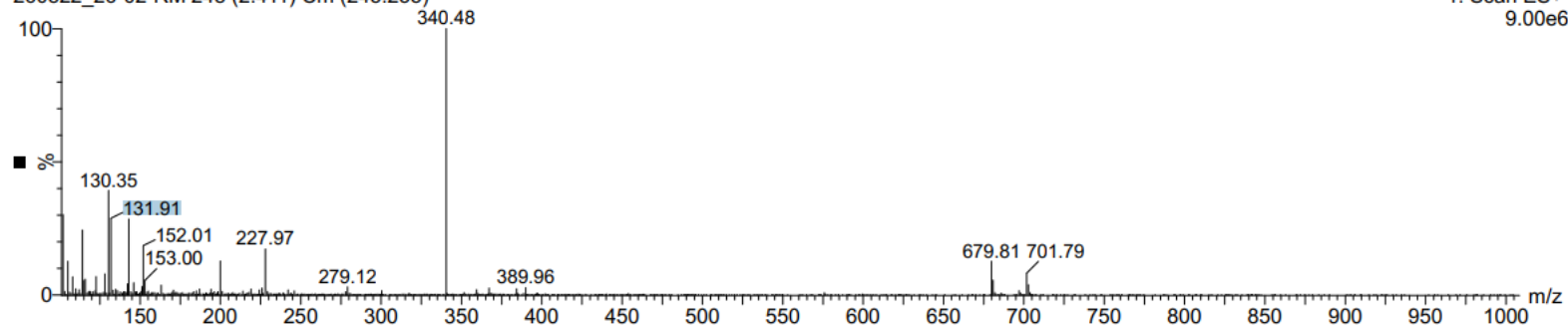
260822\_26-02-RM 398 (3.687) Cm (398)

1: Scan ES+  
1.78e7



260822\_26-02-RM 248 (2.411) Cm (245:255)

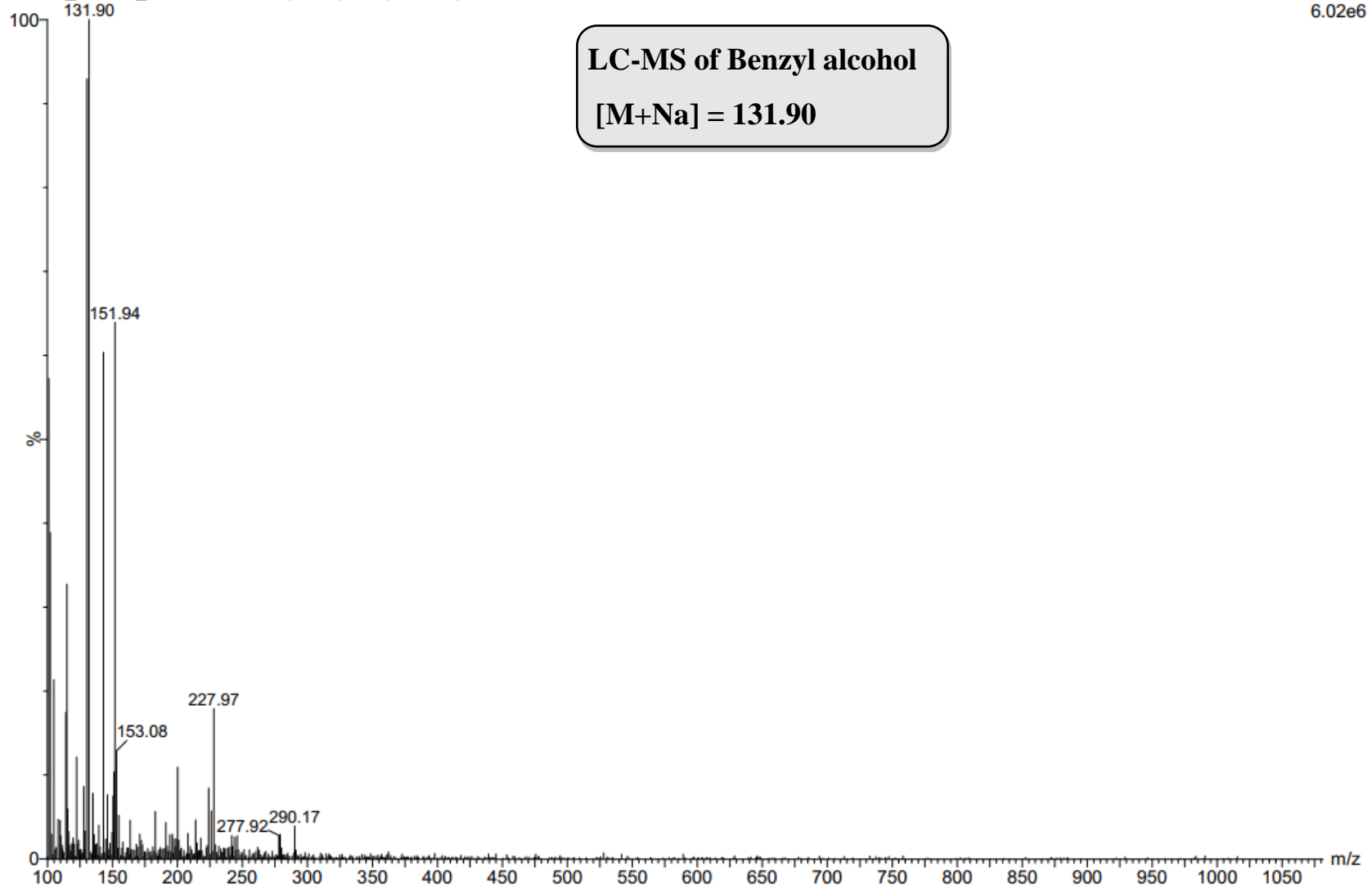
1: Scan ES+  
9.00e6



26-02-RM-B

260822\_260822\_26-02-RM-B 227 (2.232) Cm (226:229)

1: Scan ES+  
6.02e6



LC-MS of Benzyl alcohol

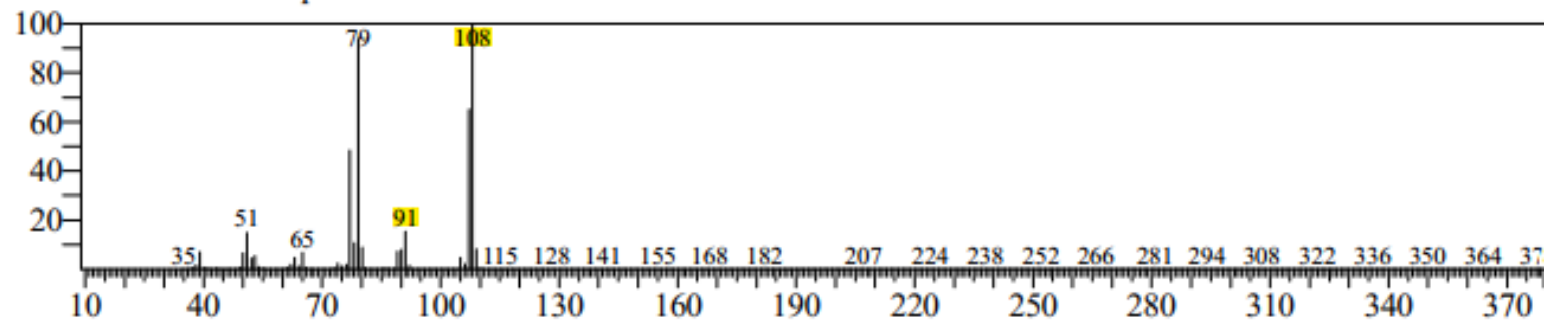
[M+Na] = 131.90

**GC-MS of Benzyl alcohol**

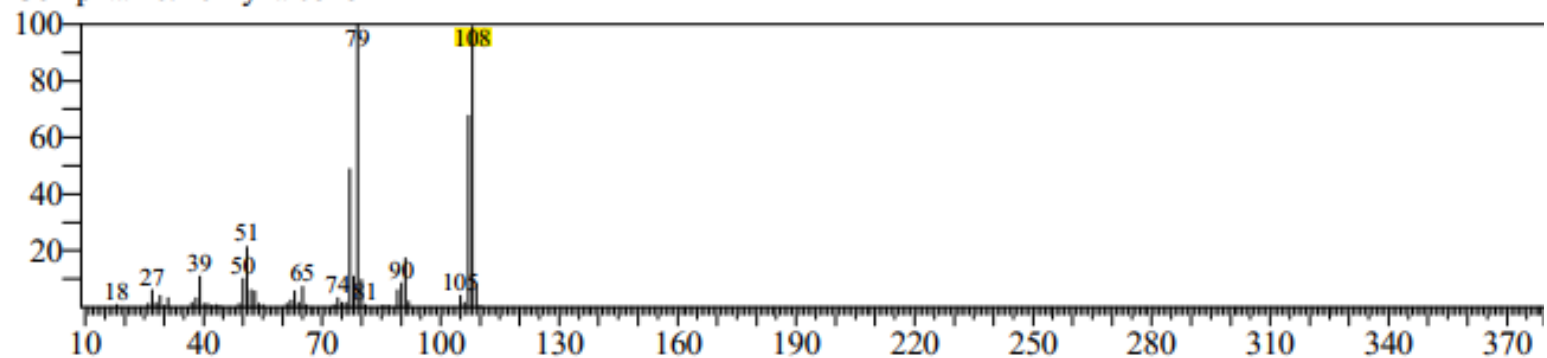
**Base peak- 108**

<< Target >>

Line#:1 R.Time:9.350(Scan#:1671) MassPeaks:463  
RawMode:Single 9.350(1671) BasePeak:108.10(10000)  
BG Mode:None Group 1 - Event 1 Scan



Hit#:1 Entry:5979 Library:NIST17.lib  
SI:98 Formula:C7H8O CAS:100-51-6 MolWeight:108 RetIndex:1036  
CompName:Benzy alcohol



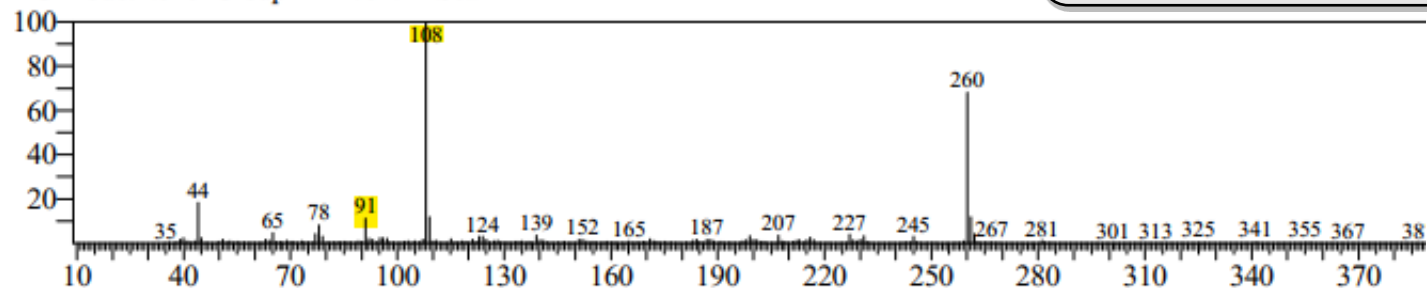
Hit#:2 Entry:5969 Library:NIST17.lib

<< Target >>

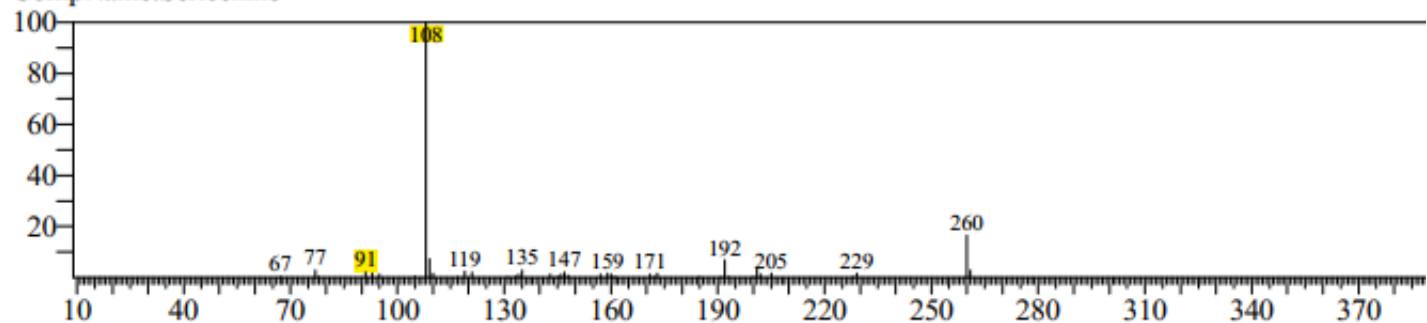
Line#:1 R.Time:24.655(Scan#:4732) MassPeaks:465  
RawMode:Single 24.655(4732) BasePeak:108.10(10000)  
BG Mode:None Group 1 - Event 1 Scan

GC-MS of Reaction mixture

Base peak- 108



Hit#:1 Entry:133422 Library:NIST17.lib  
SI:65 Formula:C<sub>16</sub>H<sub>20</sub>O<sub>3</sub> CAS:0-00-0 MolWeight:260 RetIndex:1971  
CompName:Sericenine



## H) References

- 1) Yu, M.; Xie, Y.; Xie, C.; Zhang, Y. *Org. Lett.* 2012, 14, 2164.
- 2) (a) Liu, Q.; Wang, L.; Yue, H.; Li, J. S.; Luod, Z.; Wei, W. *Green Chem.* **2019**, 21, 1609-1613; (b) Suzuki, M.; Kanemoto, K.; Nakamura, Y.; Hosoya, T.; Yoshida, S. *Org. Lett.* **2021**, 23, 3793-3797.
- 3) Xiao, F.; Tang, M.; Huang, H.; Guo-Jun Deng, G. J. *J. Org. Chem.*, **2022**, 87, 512-523.