

## Electrochemically Driven Michael Reaction: Synthesis of Hydroquinone Thioethers

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### Supplementary Information

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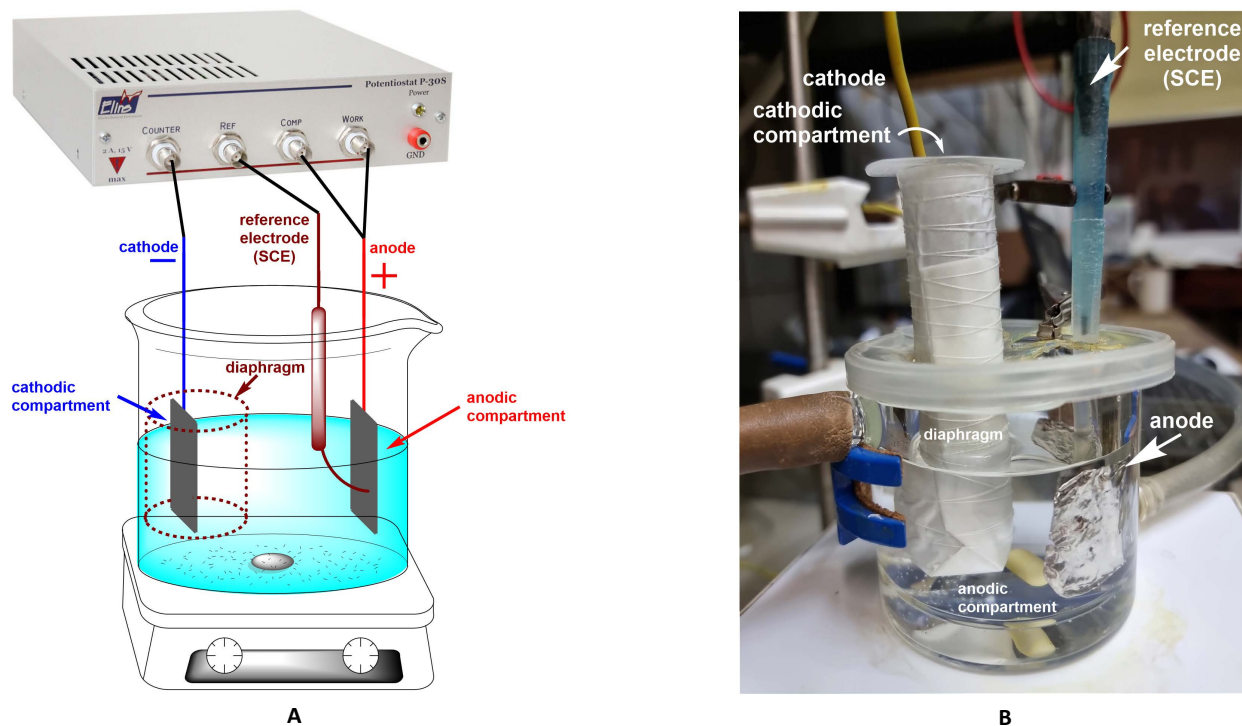
## General information

The  $^1\text{H}$  and  $^{13}\text{C}$  spectra were recorded in  $\text{DMSO-}d_6$  on a Bruker Avance 300 (300 MHz for  $^1\text{H}$  and 75 MHz for  $^{13}\text{C}$ ) or Bruker DRX 500 (126 MHz for  $^{13}\text{C}$ ). Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference:  $^1\text{H}$  ( $\text{DMSO-}d_6$   $\delta = 2.50$  ppm),  $^{13}\text{C}$  ( $\text{DMSO-}d_6$   $\delta = 39.5$  ppm).<sup>1</sup> Splitting patterns are designated as s, singlet; d, doublet; t, triplet; m, multiplet; dd, double doublet; br., broad.

High resolution mass-spectra (HRMS) were measured on the Bruker micrOTOF II instrument using electrospray ionization. The measurements were performed in a positive ion mode (interface capillary voltage – 4500 V), mass range from  $m/z$  50 to  $m/z$  3000.

Starting compounds **1a-e** and **2a-g**, MeCN (for HPLC), MeOH, EtOH, acetone,  $\text{NaClO}_4$ ,  $\text{Bu}_4\text{NClO}_4$ ,  $\text{Bu}_4\text{NBF}_4$ , anhydrous  $\text{Na}_2\text{SO}_4$  aq.  $\text{HClO}_4$  (70%) were purchased from commercial sources and were used as is. Samples of *para*-quinone and 2-(benzothiazol-2-ylthio)benzene-1,4-diol **3aa** for CV studies (see below) were prepared as reported previously.<sup>2,3</sup> Petroleum ether (PE, 40/70), ethyl acetate (EtOAc) and water were used after distillation. Column chromatography was performed using silica gel (0.040 – 0.063 mm, 60 Å).

Electrochemical experiments (cyclic voltammetry, CV and controlled potential electrolysis, CPE) were carried out using a computer-assisted potentiostat P-30JM (Elins, Russia) and a temperature-controlled ( $20 \pm 1$  °C) divided glass cell ( $V_{\text{anodic compartment}} = 60$  mL,  $V_{\text{cathodic compartment}} = 10$  mL) equipped with a tracing-paper diaphragm (see also **Figure S1**).

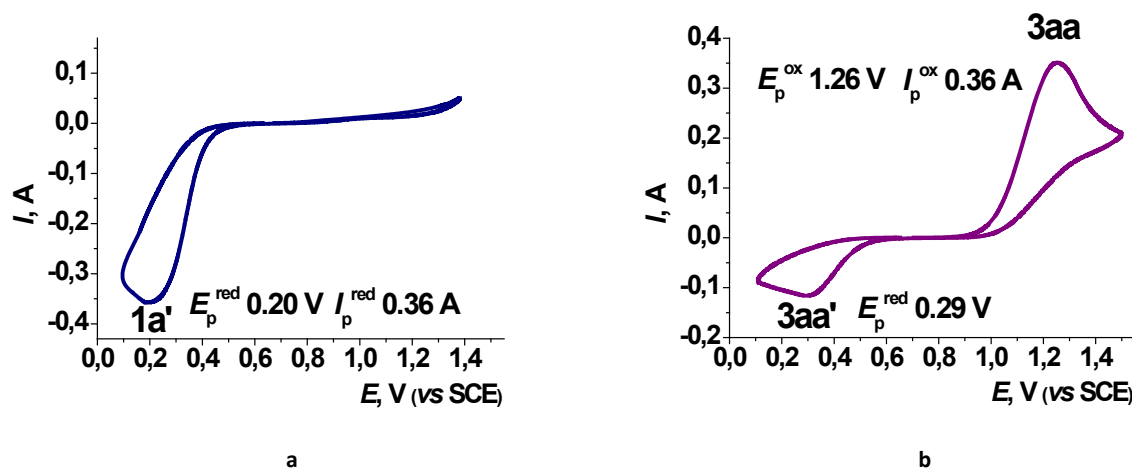


**Figure S1** Electrochemical system (A) and photo of electrochemical cell (B)

## Experimental procedures and additional data.

### Experimental procedures for Scheme 1 and Table 1. CV studies and two-stage synthesis of thioether **3aa**.

CV studies were carried out at a scan rate of  $0.10 \text{ V} \cdot \text{s}^{-1}$  using 3 electrodes (a Pt disk,  $d = 2 \text{ mm}$ , as working electrode; a Pt plate as counter electrode,  $S = 3.5 \text{ cm}^2$ ; saturated calomel electrode, SCE, as reference electrode). The working electrode was polished with chromium oxide paste ( $\sim 8 - 17 \mu\text{m}$  particle size) and carefully washed with acetone before each measurement. The concentration of the studied compounds was  $\sim 5.50 - 33.34 \text{ mM}$  in the  $0.1 \text{ M NaClO}_4$  in MeCN as supporting electrolyte.



**Figure S2** CV measurements of authentic samples of protonated *para*-quinone **1a'** and 2-(benzothiazol-2-ylthio)benzene-1,4-diol **3aa** (Pt working electrode, SCE ref. electrode,  $0.1 \text{ M NaClO}_4$  in MeCN (60 mL), initial anodic scan, scan rate  $0.10 \text{ V} \cdot \text{s}^{-1}$ ). Curve *a*: *para*-quinone (16.67 mM, 1 mmol) after adding of 2-fold excess of  $\text{HClO}_4$  (33.34 mM, 2 mmol); Curve *b*: prepared<sup>3</sup> 2-(benzothiazol-2-ylthio)benzene-1,4-diol **3aa**.

Synthesis of thioether **3aa** involved Stage 1 and Stage 2.

*Stage 1.* The anodic compartment contained  $0.1 \text{ M}$  solution of  $\text{NaClO}_4$  ( $\text{Bu}_4\text{NClO}_4$ , or  $\text{Bu}_4\text{NBF}_4$ ) in MeCN (MeOH, EtOH, or  $\text{H}_2\text{O}$ ) (60 mL) with dissolved arene **1a** (1 mmol, 0.110 g) and the cathodic compartment contained  $0.5 \text{ M}$  solution of  $\text{NaClO}_4$  ( $\text{Bu}_4\text{NClO}_4$  in entry 2, or  $\text{Bu}_4\text{NBF}_4$  in entry 3) in MeCN (MeOH in entry 5, EtOH in entry 6, or  $\text{H}_2\text{O}$  in entry 7) (10 mL). CPE was performed with stirring at  $E_{\text{anode}} = 1.15 \text{ V}$  (vs. SCE) using Pt electrodes ( $S_{\text{anode}} = 16.5 \text{ cm}^2$ ,  $S_{\text{cathode}} = 3.5 \text{ cm}^2$ ) and 5-layer tracing paper diaphragm (1-layer in entry 4). The amount of passed electricity (Q) was 193 C, which is theoretically necessary for the two-electron oxidation of arene into the corresponding protonated *p*-quinone **1a'** ( $Q_t$ , calculated according to Faraday's law<sup>4</sup>).

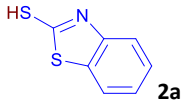
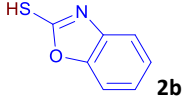
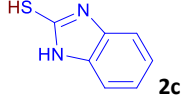
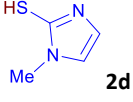
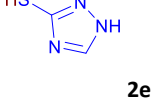
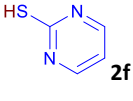
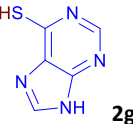
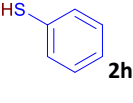
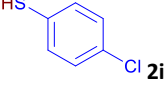
*Stage 2.* After CPE, thiol **2a** (1 mmol, 0.167 g) was added into the anodic compartment. The reaction was stirring for 12h. After the anolyte was analyzed by CV (in entries 1 – 7) or (in entry 1) was concentrated *in vacuo* (to  $\sim 3 - 5 \text{ mL}$ ) and poured into water (75 mL). The resulting precipitate was filtered off and dried in air. Further purification of the residue by column chromatography on silica gel (PE / EtOAc as eluent) gave pure target product **3aa** with yield 95% (0.262 g, 0.95 mmol).

## Experimental procedures for Scheme 2. Two-stage synthesis of thioethers **3aa-eg**, **4ea,ef,eg**.

*Stage 1.* The anodic compartment contained 0.1 M solution of NaClO<sub>4</sub> in MeCN (60 mL) with dissolved arenes **1a-e** (1 mmol, 0.110 – 0.166 g) and the cathodic compartment contained 0.5 M solution of NaClO<sub>4</sub> in MeCN (10 mL). CPE was performed with stirring at  $E_{\text{anode}} = E_{\text{p}}^{\text{ox}}_{\text{1a-e}} = 0.97 - 1.25$  V (vs. SCE) using Pt electrodes ( $S_{\text{anode}} = 16.5$  cm<sup>2</sup>,  $S_{\text{cathode}} = 3.5$  cm<sup>2</sup>) and 5-layer tracing paper diaphragm. The amount of passed electricity (Q) was 193 C.

*Stage 2.* After CPE, thiol **2a-g** (1 mmol, 0.101 – 0.167 g) was added into the anodic compartment. The reaction was stirring for 12 h and the mixture was concentrated *in vacuo* (to ~3 – 5 mL). In entries involved thiol **2a** the concentrate was poured into water (75 mL) and resulting precipitates were filtered off, dried in air and purified by column chromatography on silica gel (PE / EtOAc from 10 / 1 to 1 / 1 as eluent) gave pure target products **3aa-da** (or mixture of regioisomers **3ea + 4ea**) with yields 85 – 95% (0.258 – 0.292 g, 0.85 – 0.95 mmol). In entries involved thiols **2b-g** the concentrate was dissolved in water (15 mL) followed by extraction with EtOAc (3 × 25 mL). Combined extracts were dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and evaporated *in vacuo*. The residue was purified by column chromatography on silica gel (PE / EtOAc / MeOH from 5 / 1 / 0 to 1 / 1 / 0 and 1 / 2 / 0.3 as eluent), which gave pure target products **3ab-ai** (or mixture of regioisomers **3ef + 4ef** and **3eg + 4eg**) with yields 36 – 99% (0.083 – 0.284 g, 0.36 – 0.99 mmol).

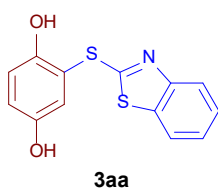
**Table S1** Oxidation potentials ( $E_p^{ox}$ ) and acidity ( $pK_a$ ) of thiols **2a-i**

Thiol	$E_p^{ox}$	$pK_a$
 <b>2a</b>	1.17	6.94 <sup>5</sup>
 <b>2b</b>	1.24	6.3 <sup>5</sup>
 <b>2c</b>	0.93	10.07 <sup>5</sup>
 <b>2d</b>	0.65	11.64 <sup>6</sup>
 <b>2e</b>	0.95	~13 <sup>7</sup>
 <b>2f</b>	2.37	7.14 <sup>8</sup>
 <b>2g</b>	2.37	7.77 <sup>9</sup>
 <b>2h</b>	1.57	7.2 <sup>10</sup>
 <b>2i</b>	1.67	7.0 <sup>10</sup>

Pt working electrode, SCE ref. electrode, 0.1 M NaClO<sub>4</sub> in MeCN, initial anodic scan, scan rate 0.10 V · s<sup>-1</sup>.

## Characterization of compounds

### 2-(Benzo[d]thiazol-2-ylthio)benzene-1,4-diol (**3aa**).<sup>3, 11-13</sup>



White solid. M. p. 230 – 232 °C (lit.<sup>3</sup> 224 – 226 °C).

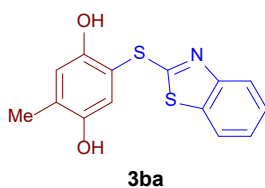
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.68 (s, 1H), 9.19 (s, 1H), 7.89 (d, <sup>3</sup>*J* = 7.8 Hz, 1H), 7.80 (d, <sup>3</sup>*J* = 7.8 Hz, 1H), 7.43 (t, <sup>3</sup>*J* = 7.8 Hz, 1H), 7.31 (t, <sup>3</sup>*J* = 7.8 Hz, 1H), 6.99 (d, <sup>4</sup>*J* = 2.7 Hz, 1H), 6.90 (d, <sup>3</sup>*J* = 8.4 Hz, 1H), 6.85 (dd, <sup>3</sup>*J* = 8.4 Hz, <sup>4</sup>*J* = 2.7 Hz, 1H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 170.0, 153.6, 151.2, 150.4, 134.9, 126.2, 124.1, 122.0, 121.6, 121.1, 120.2, 117.5, 114.0.

Yield 95% (262 mg, 0.95 mmol).

HRMS (ESI-TOF) calc. for [C<sub>13</sub>H<sub>10</sub>NO<sub>2</sub>S<sub>2</sub>]<sup>+</sup> [M + H]<sup>+</sup> 276.0147, found 276.0151.

### 2-(Benzo[d]thiazol-2-ylthio)-5-methylbenzene-1,4-diol (**3ba**).



White solid. M. p. 224 – 226 °C.

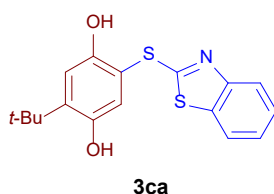
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.56 (s, 1H), 9.11 (s, 1H), 7.87 (d, <sup>3</sup>*J* = 7.7 Hz, 1H), 7.78 (d, <sup>3</sup>*J* = 7.7 Hz, 1H), 7.42 (t, <sup>3</sup>*J* = 7.7 Hz, 1H), 7.29 (t, <sup>3</sup>*J* = 7.7 Hz, 1H), 6.98 (s, 1H), 6.81 (s, 1H), 2.14 (s, 3H).

<sup>13</sup>C NMR (126 MHz, DMSO-*d*<sub>6</sub>) δ 171.2, 153.8, 151.3, 148.7, 134.9, 130.3, 126.2, 124.0, 121.6, 121.4, 121.0, 118.7, 110.3, 16.3.

Yield 89% (258 mg, 0.89 mmol).

HRMS (ESI-TOF) calc. for [C<sub>14</sub>H<sub>12</sub>NO<sub>2</sub>S<sub>2</sub>]<sup>+</sup> [M + H]<sup>+</sup> 290.0304, found 290.0297.

### 2-(Benzo[d]oxazol-2-ylthio)-5-(*tert*-butyl)benzene-1,4-diol (**3ca**).



White solid. M. p. 230 – 232 °C.

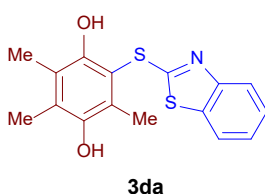
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.51 (s, 1H), 9.16 (s, 1H), 7.90 (d, <sup>3</sup>*J* = 7.5 Hz, 1H), 7.79 (d, <sup>3</sup>*J* = 7.5 Hz, 1H), 7.42 (t, <sup>3</sup>*J* = 7.5 Hz, 1H), 7.29 (t, <sup>3</sup>*J* = 7.5 Hz, 1H), 6.99 (s, 1H), 6.95 (s, 1H), 1.37 (s, 9H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 170.5, 153.7, 151.0, 148.9, 141.1, 135.0, 126.1, 124.0, 122.5, 121.6, 121.0, 115.1, 110.3, 34.6, 29.0 (3C).

Yield 88% (292 mg, 0.88 mmol).

HRMS (ESI-TOF) calc. for [C<sub>17</sub>H<sub>18</sub>NO<sub>2</sub>S<sub>2</sub>]<sup>+</sup> [M + H]<sup>+</sup> 332.0773, found 332.0770.

### 2-(Benzo[d]thiazol-2-ylthio)-3,5,6-trimethylbenzene-1,4-diol (**3da**).



Cream-colored solid. M. p. 164 – 166 °C.

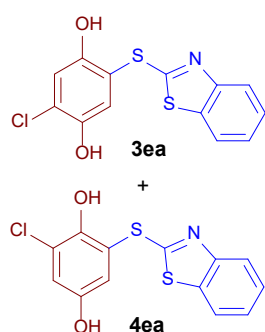
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 8.50 (s, 1H), 7.94 (s, 1H), 7.85 (d, <sup>3</sup>*J* = 8.4 Hz, 1H), 7.78 (d, <sup>3</sup>*J* = 8.4 Hz, 1H), 7.41 (t, <sup>3</sup>*J* = 8.4 Hz, 1H), 7.28 (t, <sup>3</sup>*J* = 8.4 Hz, 1H), 2.33 (s, 3H), 2.20 (s, 3H), 2.16 (s, 3H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 171.0, 154.0, 150.1, 146.5, 134.8, 130.3, 127.1, 126.1, 123.8, 122.8, 121.5, 120.9, 112.5, 14.8, 13.5, 13.2.

Yield 90% (286 mg, 0.90 mmol).

HRMS (ESI-TOF) calc. for [C<sub>16</sub>H<sub>16</sub>NO<sub>2</sub>S<sub>2</sub>]<sup>+</sup> [M + H]<sup>+</sup> 316.0460, found 316.0456.

### 2-(Benzo[d]thiazol-2-ylthio)-5-chlorobenzene-1,4-diol (**3ea**) and 2-(benzo[d]thiazol-2-ylthio)-6-chlorobenzene-1,4-diol (**4ea**), mixture of regioisomers (~1 : 0.4).



White solid. M. p. 183 – 185 °C.

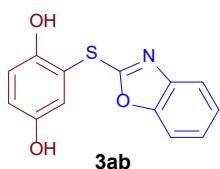
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 10.13 (s, 1H, **3ea**), 9.92 (s, 1H, **3ea**), 9.68 (s, 1H, **4ea**), 9.59 (s, 1H, **4ea**), 7.97 – 7.89 (m, 1H, **3ea** + **4ea**), 7.88 – 7.78 (m, 1H, **3ea** + **4ea**), 7.49 – 7.39 (m, 1H, **3ea** + **4ea**), 7.38 – 7.28 (m, 1H, **3ea** + **4ea**), 7.20 (s, 1H, **3ea**), 7.05 (s, 1H, **3ea**), 7.04 (d, <sup>4</sup>*J* = 2.7 Hz, 1H, **4ea**), 7.03 (d, <sup>4</sup>*J* = 2.7 Hz, 1H, **4ea**)

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 169.3 (**3ea**), 168.4 (**4ea**), 153.6 (**3ea**), 153.5 (**4ea**), 151.4 (**3ea**), 150.9 (**4ea**), 146.6 (**4ea**), 146.3 (**3ea**), 135.0 (**4ea**), 134.9 (**3ea**), 126.4 (**4ea**), 126.3 (**3ea**), 124.5 (**4ea**), 124.3 (**3ea**), 124.0 (**4ea**), 123.2 (**3ea**), 122.9 (**4ea**), 121.8 (**4ea**), 121.7 (**3ea**), 121.4 (**3ea**), 121.3 (**4ea**), 121.2 (**3ea**), 119.9 (**3ea**), 118.3 (**4ea**), 117.5 (**3ea**), 113.4 (**4ea**)

Yield 85% (263 mg, 0.85 mmol).

HRMS (ESI-TOF) calc. for [C<sub>13</sub>H<sub>9</sub>ClNO<sub>2</sub>S<sub>2</sub>]<sup>+</sup> [M + H]<sup>+</sup> 309.9758, found 309.9767.

### 2-(Benzo[d]oxazol-2-ylthio)benzene-1,4-diol (**3ab**).



**3ab**

White solid. M. p. 221 – 223 °C.

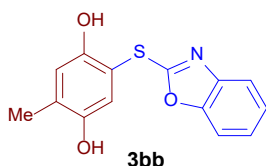
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.56 (s, 1H), 9.10 (s, 1H), 7.69 – 7.56 (m, 2H), 7.37 – 7.26 (m, 2H), 6.96 (d, <sup>4</sup>*J* = 3.0 Hz, 1H), 6.82 (d, <sup>3</sup>*J* = 8.5 Hz, 1H), 6.81 (dd, <sup>3</sup>*J* = 8.5 Hz, <sup>4</sup>*J* = 3.0 Hz, 1H).

<sup>13</sup>C NMR (126 MHz, DMSO-*d*<sub>6</sub>) δ 162.6, 151.2, 150.4, 150.1, 141.5, 124.6, 124.4, 120.9, 118.9, 118.5, 116.9, 112.0, 110.2.

Yield 86% (224 mg, 0.86 mmol).

HRMS (ESI-TOF) calc. for [C<sub>13</sub>H<sub>10</sub>NO<sub>3</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 260.0376, found 260.0375.

### 2-(Benzo[d]oxazol-2-ylthio)-5-methylbenzene-1,4-diol (**3bb**).



**3bb**

Cream-colored solid. M. p. 203 – 205 °C.

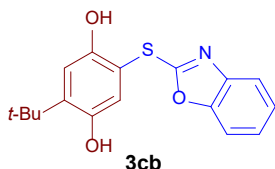
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.45 (s, 1H), 9.02 (s, 1H), 7.66 – 7.56 (m, 2H), 7.35 – 7.24 (m, 2H), 6.95 (s, 1H), 6.74 (s, 1H), 2.12 (s, 3H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 163.2, 151.3, 150.6, 148.3, 141.6, 128.9, 124.6, 124.3, 120.7, 118.5, 118.3, 110.3, 108.0, 16.2.

Yield 77% (210 mg, 0.77 mmol).

HRMS (ESI-TOF) calc. for [C<sub>14</sub>H<sub>12</sub>NO<sub>3</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 274.0532, found 274.0537.

### 2-(Benzo[d]oxazol-2-ylthio)-5-(*tert*-butyl)benzene-1,4-diol (**3cb**).



**3cb**

White solid. M. p. 174 – 176 °C.

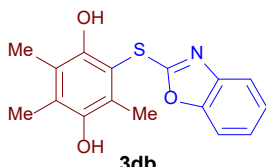
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.39 (s, 1H), 9.08 (s, 1H), 7.66 – 7.57 (m, 2H), 7.36 – 7.26 (m, 2H), 6.94 (s, 1H), 6.87 (s, 1H), 1.35 (s, 9H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 162.9, 151.2, 150.3, 148.7, 141.5, 139.8, 124.6, 124.3, 121.7, 118.5, 114.7, 110.2, 108.0, 34.5, 29.1 (3C).

Yield 90% (284 mg, 0.90 mmol).

HRMS (ESI-TOF) calc. for [C<sub>17</sub>H<sub>18</sub>NO<sub>3</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 316.1002, found 316.0999.

### 2-(Benzo[d]oxazol-2-ylthio)-3,5,6-trimethylbenzene-1,4-diol (**3db**).



**3db**

White solid. M. p. 181 – 183 °C.

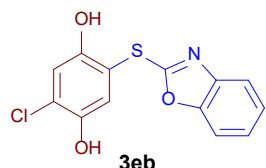
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.12 (s, 1H), 8.36 (s, 1H), 7.24 – 6.55 (m, 4H), 2.24 (s, 3H), 2.13 (s, 3H), 2.06 (s, 3H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 160.4, 149.4, 148.5, 141.7, 135.9, 125.5, 123.4, 121.2, 119.7, 118.7, 117.6, 116.6, 116.0, 15.1, 12.8, 12.4.

Yield 65% (196 mg, 0.65 mmol).

HRMS (ESI-TOF) calc. for [C<sub>16</sub>H<sub>16</sub>NO<sub>3</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 302.0845, found 302.0853.

### 2-(Benzo[d]oxazol-2-ylthio)-5-chlorobenzene-1,4-diol (**3eb**).



**3eb**

Cream-colored solid. M. p. 232 – 234 °C.

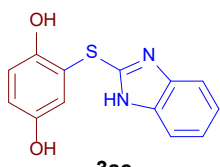
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 10.02 (s, 1H), 9.82 (s, 1H), 7.69 – 7.56 (m, 2H), 7.38 – 7.26 (m, 2H), 7.17 (s, 1H), 6.97 (s, 1H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 162.1, 151.2, 150.6, 146.0, 141.4, 124.7, 124.5, 122.7, 122.2, 118.6, 116.9, 111.3, 110.3.

Yield 91% (267 mg, 0.91 mmol).

HRMS (ESI-TOF) calc. for [C<sub>13</sub>H<sub>9</sub>ClNO<sub>3</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 293.9986, found 293.9995.

### 2-((1*H*-Benzo[d]imidazol-2-yl)thio)benzene-1,4-diol (**3ac**).<sup>3, 14</sup>



**3ac**

White solid. M.p. 218 – 220 °C (lit.<sup>3</sup> 218 – 220 °C).

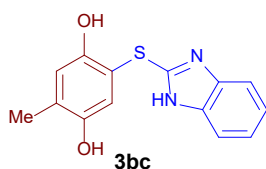
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 12.59 (br. s, 1H), 9.51 (br. s, 1H), 8.94 (s, 1H), 7.76 – 7.30 (m, 2H), 7.26 – 6.96 (m, 2H), 6.75 (d, <sup>3</sup>*J* = 8.4 Hz, 1H), 6.67 – 6.35 (m, 2H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 150.3 (2C), 148.5 (2C), 147.1, 122.0, 117.9 (2C), 117.4, 116.7 (2C), 116.4 (2C)

Yield 65% (168 mg, 0.65 mmol).

HRMS (ESI-TOF) calc. for [C<sub>13</sub>H<sub>11</sub>N<sub>2</sub>O<sub>2</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 259.0536, found 259.0536.

### 2-((1H-Benzo[d]imidazol-2-yl)thio)-5-methylbenzene-1,4-diol (**3bc**).



Cream-colored solid. M. p. 169 – 171 °C.

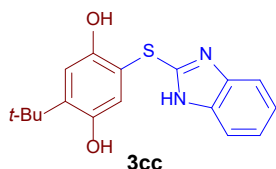
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.19 (br. s, 3H), 7.72 – 7.57 (m, 2H), 7.49 – 7.35 (m, 2H), 6.95 (s, 1H), 6.80 (s, 1H), 2.14 (s, 3H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 150.7, 150.5, 148.8, 133.1, 130.1, 125.0 (2C), 120.1, 118.8, 113.4 (2C), 106.1, 39.5, 16.3.

Yield 73% (199 mg, 0.73 mmol).

HRMS (ESI-TOF) calc. for [C<sub>14</sub>H<sub>13</sub>N<sub>2</sub>O<sub>2</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 273.0692, found 273.0699.

### 2-((1H-Benzo[d]imidazol-2-yl)thio)-5-(*tert*-butyl)benzene-1,4-diol (**3cc**).



Cream-colored solid. M. p. 134 – 136 °C.

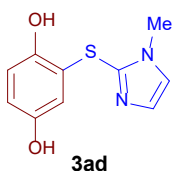
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.28 (s, 1H), 7.77 – 7.61 (m, 2H), 7.52 – 7.39 (m, 2H), 6.94 (s, 1H), 6.91 (s, 1H), 1.35 (s, 9H). The signals of the OH-groups were not observed, probably merging with residual water from DMSO-*d*<sub>6</sub>.

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 150.2 (2C), 149.2, 140.9, 132.5 (2C), 125.4 (2C), 121.0, 115.1, 113.4 (2C), 106.2, 34.7, 29.0 (3C).

Yield 77% (242 mg, 0.77 mmol).

HRMS (ESI-TOF) calc. for [C<sub>17</sub>H<sub>19</sub>N<sub>2</sub>O<sub>2</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 315.1162, found 315.1162.

### 2-((1-Methyl-1H-imidazol-2-yl)thio)benzene-1,4-diol (**3ad**).<sup>3</sup>



Cream-colored solid. M. p. 193 – 195 °C (lit.<sup>3</sup> 189 – 191 °C).

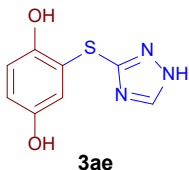
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.45 (br. s, 1H), 8.80 (s, 1H), 7.47 (s, 1H), 7.10 (s, 1H), 6.63 (d, <sup>3</sup>J = 8.1 Hz, 1H), 6.43 (dd, <sup>3</sup>J = 8.1 Hz, <sup>4</sup>J = 2.1 Hz, 1H), 5.86 (d, <sup>4</sup>J = 2.1 Hz, 1H), 3.60 (s, 3H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 150.5, 146.3, 136.2, 129.5, 124.9, 121.9, 116.1, 113.9, 113.5, 33.4.

Yield 63% (140 mg, 0.63 mmol).

HRMS (ESI-TOF) calc. for [C<sub>10</sub>H<sub>11</sub>N<sub>2</sub>O<sub>2</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 223.0536, found 223.0541.

### 2-((1H-1,2,4-Triazol-3-yl)thio)benzene-1,4-diol (**3ae**).



White solid. M. p. 190 – 192 °C.

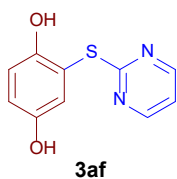
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 14.31 (br. s, 1H), 9.29 (br. s, 1H), 8.81 (s, 1H), 8.55 (br. s, 1H), 6.66 (d, <sup>3</sup>J = 8.6 Hz, 1H), 6.50 (dd, <sup>3</sup>J = 8.6 Hz, <sup>4</sup>J = 3.0 Hz, 1H), 6.39 (d, <sup>4</sup>J = 3.0 Hz, 1H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 150.2, 147.3 (2C), 146.0, 119.7, 116.1, 116.0, 114.9.

Yield 55% (115 mg, 0.55 mmol).

HRMS (ESI-TOF) calc. for [C<sub>8</sub>H<sub>8</sub>N<sub>3</sub>O<sub>2</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 210.0332, found 210.0328.

### 2-(Pyrimidin-2-ylthio)benzene-1,4-diol (**3af**).<sup>3, 13, 15</sup>



Yellowish solid. M. p. 200 – 202 °C (lit.<sup>3</sup> 202 – 204 °C).

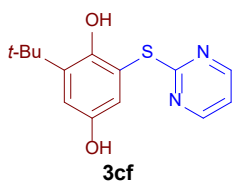
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.10 (s, 1H), 8.91 (s, 1H), 8.54 (d, <sup>3</sup>J = 4.8 Hz, 2H), 7.17 (t, <sup>3</sup>J = 4.8 Hz, 1H), 6.84 (d, <sup>4</sup>J = 2.8 Hz, 1H), 6.76 (d, <sup>3</sup>J = 8.4 Hz, 1H), 6.70 (dd, <sup>3</sup>J = 8.4 Hz, <sup>4</sup>J = 2.8 Hz, 1H)

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 171.4, 157.8 (2C), 151.2, 149.8, 122.5, 118.3, 117.5, 116.7, 114.6.

yield 98% (216 mg, 0.98 mmol)

HRMS (ESI-TOF) calc. for [C<sub>10</sub>H<sub>9</sub>N<sub>2</sub>O<sub>2</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 221.0379, found 221.0388.

### 2-(*Tert*-butyl)-6-(pyrimidin-2-ylthio)benzene-1,4-diol (**3cf**).



White solid. M. p. 211 – 213 °C.

<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 8.88 (s, 1H), 8.58 (d, <sup>3</sup>J = 4.8 Hz, 2H), 8.18 (s, 1H), 7.21 (t, <sup>3</sup>J = 4.8 Hz, 1H), 6.78 (d, <sup>4</sup>J = 2.9 Hz, 1H), 6.73 (d, <sup>4</sup>J = 2.9 Hz, 1H), 1.34 (s, 9H).

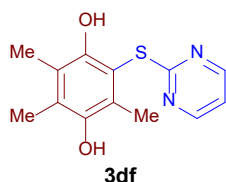
<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 171.2, 158.0 (2C), 149.6, 149.4, 138.5, 119.7, 117.7, 116.8, 116.1, 34.8, 29.4 (3C).

yield 81% (224 mg, 0.81 mmol)

HRMS (ESI-TOF) calc. for [C<sub>14</sub>H<sub>17</sub>N<sub>2</sub>O<sub>2</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 277.1005, found 277.1001.



### 2,3,5-Trimethyl-6-(pyrimidin-2-ylthio)benzene-1,4-diol (**3df**).



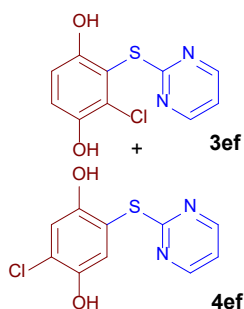
Yellowish solid. M. p. 212 – 214 °C.

$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  8.53 (d,  $^3J = 4.8$  Hz, 2H), 7.98 (s, 1H), 7.70 (s, 1H), 7.17 (t,  $^3J = 4.8$  Hz, 1H), 2.18 (s, 3H), 2.14 (s, 3H), 2.09 (s, 3H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  171.1, 157.8 (2C), 149.9, 145.9, 128.4, 127.3, 121.5, 117.5, 112.3, 15.0, 13.3, 13.1.

yield 36% (94 mg, 0.36 mmol) HRMS (ESI-TOF) calc. for  $[\text{C}_{13}\text{H}_{15}\text{N}_2\text{O}_2\text{S}]^+ [\text{M} + \text{H}]^+$  263.0849, found 263.0847.

### 2-Chloro-3-(pyrimidin-2-ylthio)benzene-1,4-diol (**3ef**) and 2-chloro-5-(pyrimidin-2-ylthio)benzene-1,4-diol (**4ef**), mixture of regioisomers (~1 : 0.2)



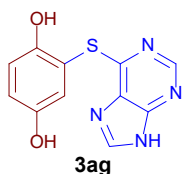
Yellowish solid. M. p. 199 – 201 °C.

$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  9.61 (s, 1H, **4ef**), 9.57 (s, 1H, **4ef**), 9.42 (s, 1H, **3ef**), 9.03 (s, 1H, **3ef**), 8.58 (d,  $^3J = 4.8$  Hz, 2H, **3ef**), 8.55 (d,  $^3J = 4.8$  Hz, 2H, **4ef**), 7.21 (t,  $^3J = 4.8$  Hz, 1H, **3ef**), 7.19 (t,  $^3J = 4.8$  Hz, 1H, **4ef**), 7.06 (s, 1H, **4ef**), 6.92, 6.91 (merged d,  $^3J = 8.8$  Hz, 1H, **3ef**, s, 1H, **4ef**), and d,  $^3J = 8.8$  Hz, 1H, **3ef**).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  170.9 (**4ef**), 170.6 (**3ef**), 158.0 (2C, **3ef**), 157.9 (2C, **4ef**), 151.5 (**4ef**), 150.2 (**3ef**), 146.9 (**3ef**), 145.7 (**4ef**), 123.8 (**4ef**), 122.0 (**4ef**), 121.9 (**3ef**), 121.8 (**3ef**), 118.5 (**3ef**), 118.2 (**3ef**), 117.8 (**3ef**), 117.6 (**4ef**), 116.7 (**4ef**), 113.9 (**4ef**).

yield 62% (158 mg, 0.62 mmol) HRMS (ESI-TOF) calc. for  $[\text{C}_{10}\text{H}_8\text{ClN}_2\text{O}_2\text{S}]^+ [\text{M} + \text{H}]^+$  254.9990, found 255.0000.

### 2-((9H-Purin-6-yl)thio)benzene-1,4-diol (**3ag**).



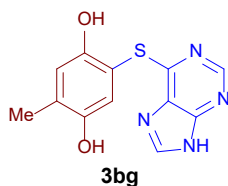
Yellow solid. M. p. 240 – 242 °C.

$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  13.40 (br. s), 9.19 (br. s, 1H), 8.97 (br. s, 1H), 8.53 (s, 1H), 8.47 (s, 1H), 6.88 (d,  $^3J = 2.7$  Hz, 1H), 6.79 (d,  $^3J = 8.6$  Hz, 1H), 6.78 (dd,  $^3J = 8.6$  Hz,  $^4J = 2.7$  Hz, 1H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  157.9, 151.5, 151.4 (2C), 149.9, 143.5 (2C), 122.8, 118.5, 116.7, 112.6.

yield 99% (258 mg, 0.99 mmol) HRMS (ESI-TOF) calc. for  $[\text{C}_{11}\text{H}_9\text{N}_4\text{O}_2\text{S}]^+ [\text{M} + \text{H}]^+$  261.0441, found 261.0444.

### 2-((9H-Purin-6-yl)thio)-5-methylbenzene-1,4-diol (**3bg**).



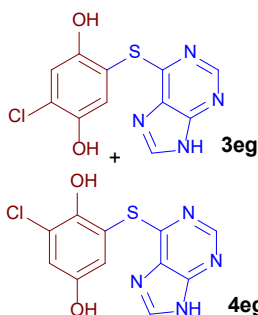
Cream-colored solid. M. p. 229 – 231 °C.

$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  13.49 (br. s, 1H), 9.06 (br. s, 1H), 8.85 (s, 1H), 8.51, 8.47 (merged s, 2H), 6.85 (s, 1H), 6.71 (s, 1H), 2.12 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz, DMSO- $d_6$ )  $\delta$  159.2, 151.6 (2C), 149.5, 148.1, 143.0, 129.9, 128.1, 122.3, 118.1, 108.7, 16.3.

yield 48% (132 mg, 0.48 mmol) HRMS (ESI-TOF) calc. for  $[\text{C}_{12}\text{H}_{11}\text{N}_4\text{O}_2\text{S}]^+ [\text{M} + \text{H}]^+$  275.0597, found 275.0605.

### 2-((9H-Purin-6-yl)thio)-5-chlorobenzene-1,4-diol (**3eg**) and 2-((9H-purin-6-yl)thio)-6-chlorobenzene-1,4-diol (**4eg**), mixture of regioisomers (~0.3 : 1).



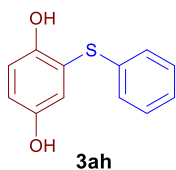
White solid. M. p. 194 – 196 °C.

$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  13.56 (br. s, 1H, **3eg** + **4eg**), 9.65, 9.47, 9.30, 9.20 (merged br. s, 2H, **3eg** + **4eg**), 8.57 (s, 1H, **4eg**), 8.54 (s, 1H, **3eg**), 8.50 (s, 1H, **4eg**), 8.47 (s, 1H, **3eg**), 7.08 (s, 1H, **3eg**), 6.94 (merged d,  $^4J = 3.0$  Hz, 1H, **4eg** and s, 1H, **3eg**), 6.91 (d,  $^4J = 3.0$  Hz, 1H, **4eg**)

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  151.6 (2C, **4eg**), 150.31 (2C, **3eg**), 150.32 (2C, **4eg**), 147.1 (**4eg**), 145.8 (**4eg**), 122.2 (2C, **3eg**), 122.1 (2C, **4eg**), 118.6 (**4eg**), 116.7 (2C, **3eg**), 116.6 9 (2C, **4eg**). The 4 signals corresponding to the 5 carbon atoms of the minor isomer (**3eg**) were not detected due to their low intensity.

yield 70% (206 mg, 0.70 mmol) HRMS (ESI-TOF) calc. for  $[\text{C}_{11}\text{H}_8\text{ClN}_4\text{O}_2\text{S}]^+ [\text{M} + \text{H}]^+$  295.0051, found 295.0060.

**2-(phenylthio)benzene-1,4-diol (3ah).<sup>16</sup>**



Yellow oil.

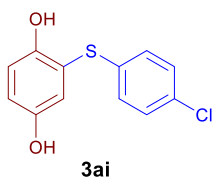
<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.15 (s, 1H), 8.84 (s, 1H), 7.43 – 7.16 (m, 5H), 6.72 (d, <sup>3</sup>*J* = 8.6 Hz, 1H), 6.59 (d, <sup>3</sup>*J* = 8.6 Hz, 1H), 6.48 (s, 1H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 150.3, 148.8, 135.2, 129.9 (2C), 129.3 (2C), 126.6, 120.0, 118.3, 116.3, 115.9.

yield 38 % (83 mg, 0.38 mmol)

HRMS (ESI-TOF) calc. for [C<sub>12</sub>H<sub>11</sub>O<sub>2</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 218.0396, found 218.0402.

**2-((4-chlorophenyl)thio)benzene-1,4-diol (3ai).<sup>16</sup>**



White solid. M. p. 120 – 121 °C. (lit.<sup>16</sup> 119.5 – 120 °C.)

<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ 9.23 (s, 1H), 8.90 (s, 1H), 7.37 (d, <sup>3</sup>*J* = 8.3 Hz, 2H), 7.21 (d, <sup>3</sup>*J* = 8.3 Hz, 2H), 6.75 (d, <sup>3</sup>*J* = 8.9 Hz, 1H), 6.64 (d, <sup>3</sup>*J* = 8.9 Hz, 1H), 6.54 (s, 1H).

<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 150.3, 149.2, 134.8, 131.0, 130.8 (2C), 129.1 (2C), 118.9, 118.8, 116.6, 116.5.

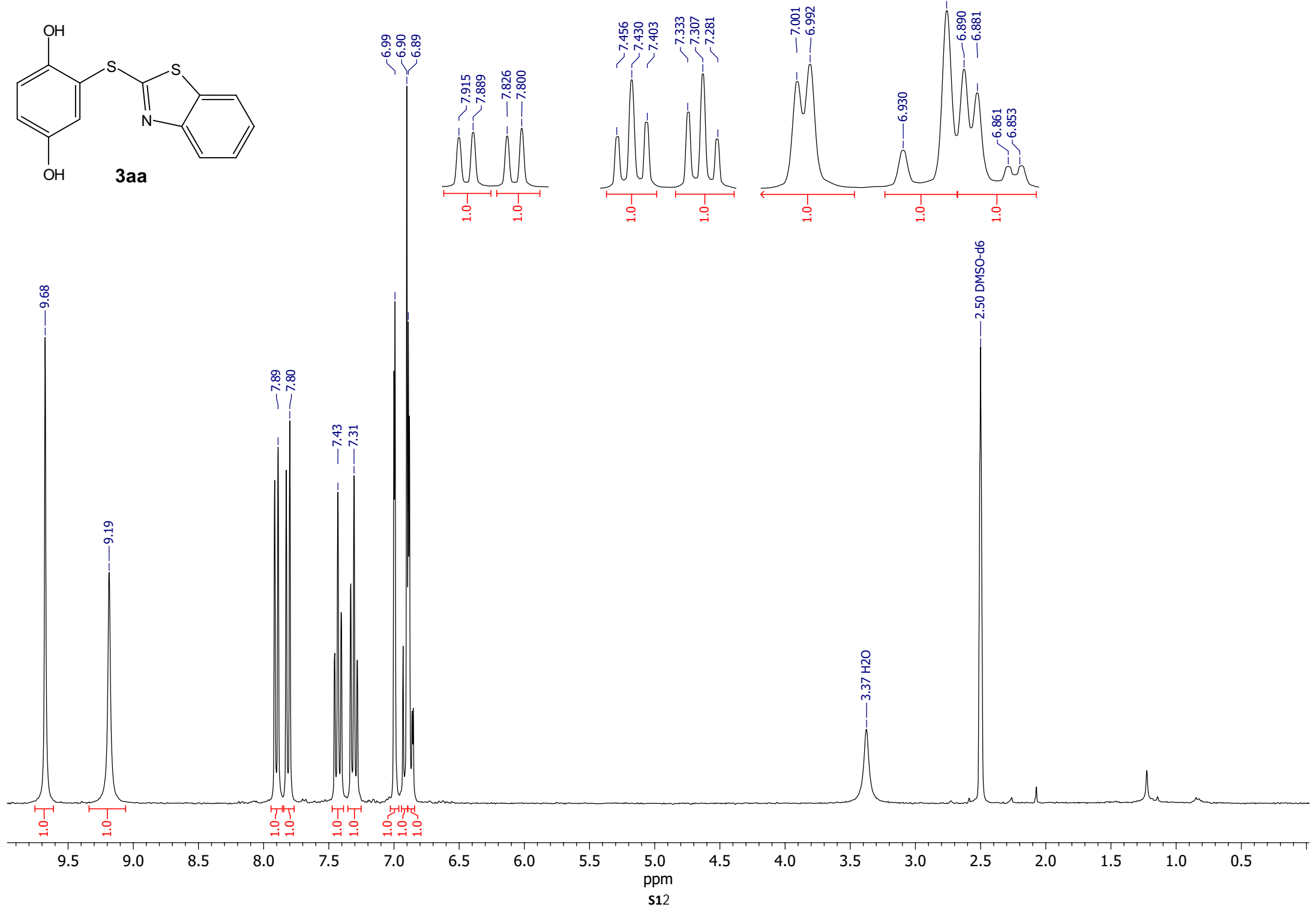
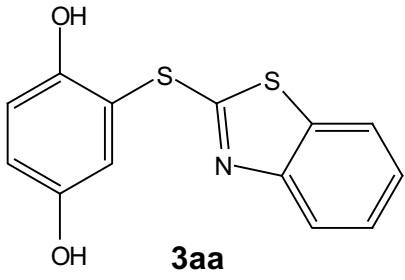
yield 53% (134 mg, 0.53 mmol)

HRMS (ESI-TOF) calc. for [C<sub>12</sub>H<sub>10</sub>ClO<sub>2</sub>S]<sup>+</sup> [M + H]<sup>+</sup> 253.0085, found 253.0088.

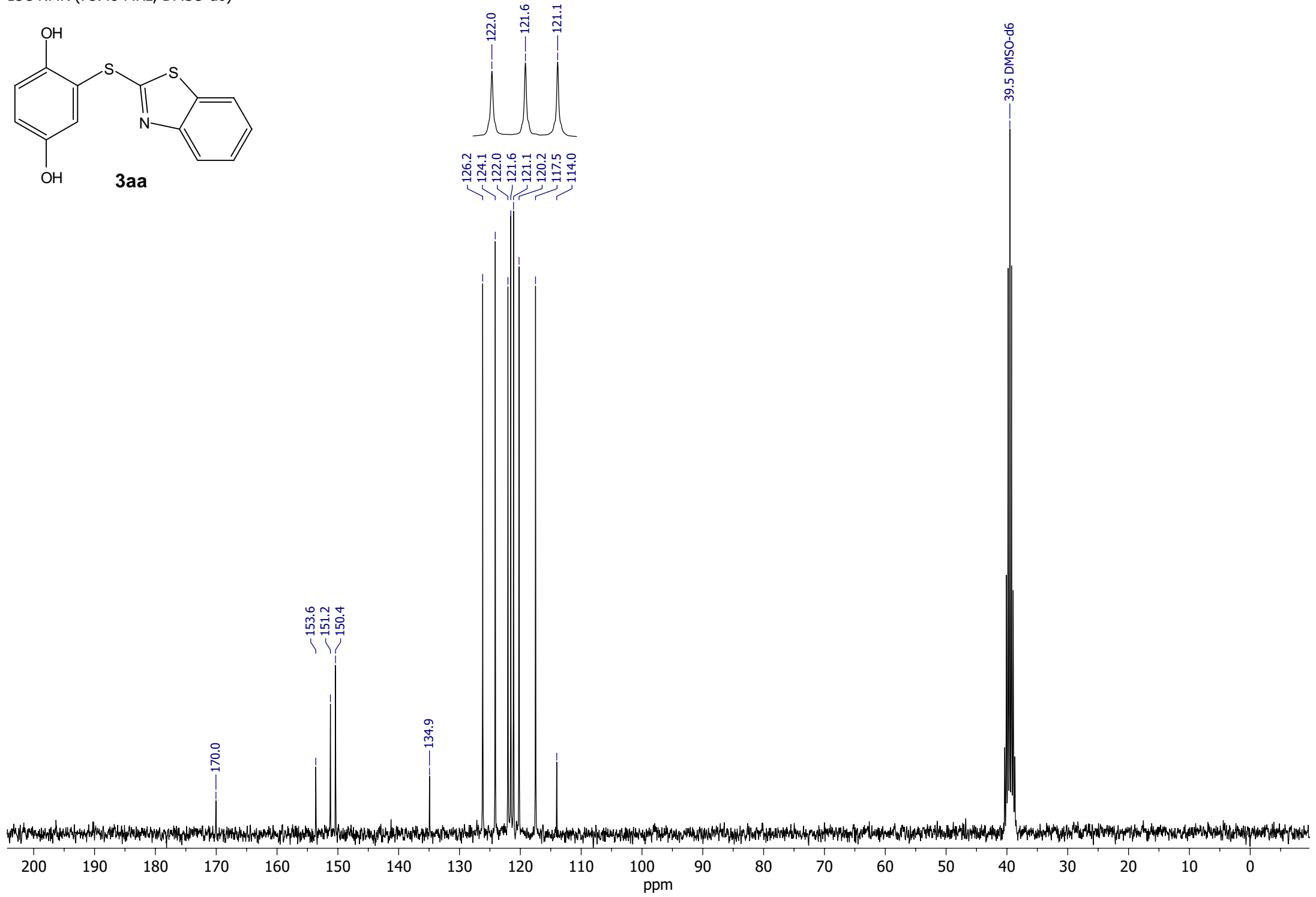
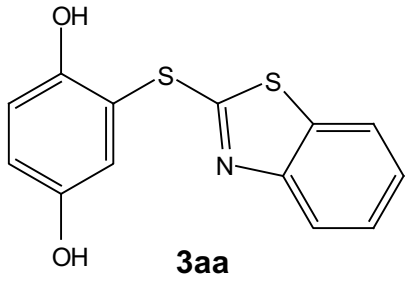
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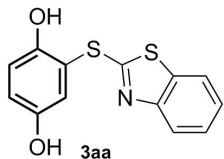
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<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)





Chemical Formula: C<sub>13</sub>H<sub>9</sub>NO<sub>2</sub>S<sub>2</sub>  
 Exact Mass: 275,01

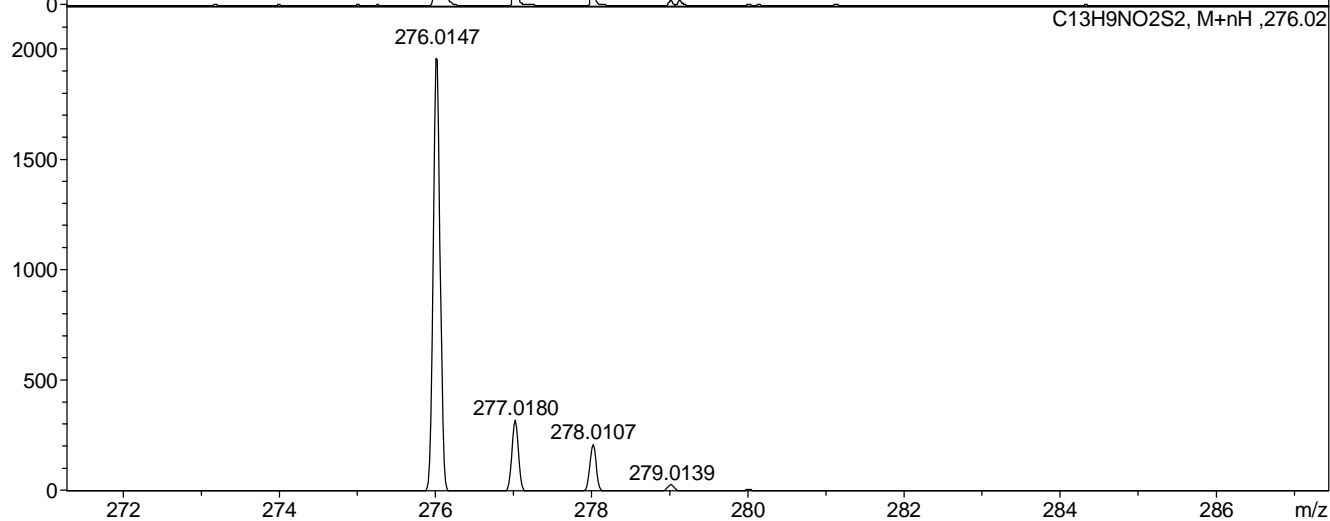
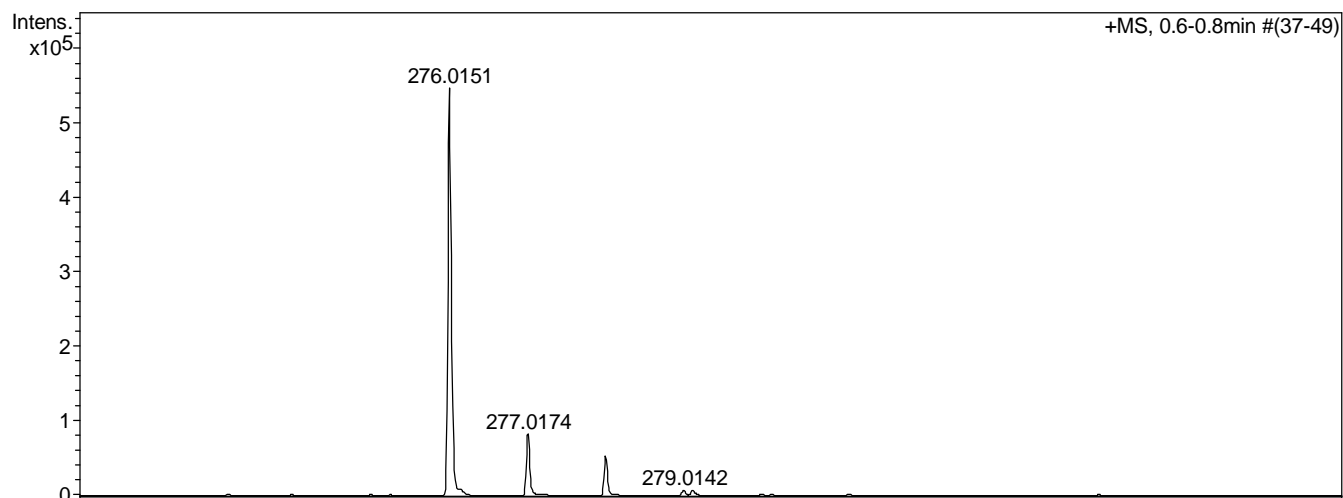
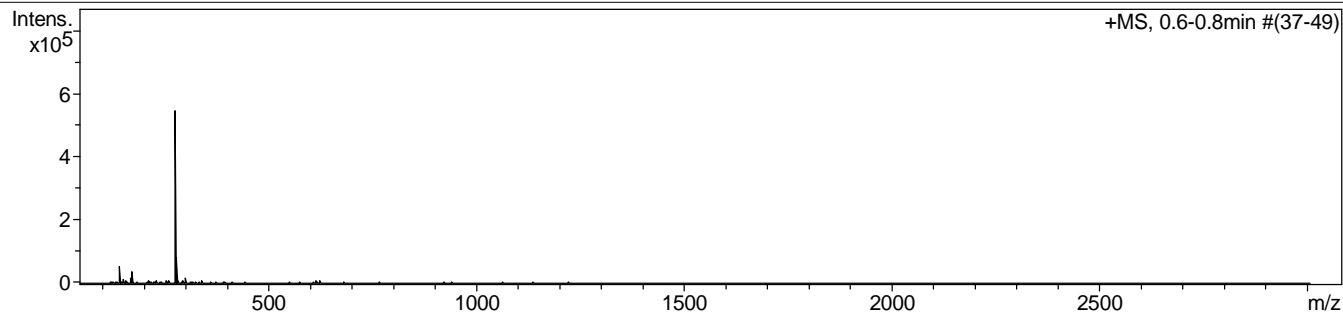
**Analysis Info**

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 Sample Name /VAPP MNV307  
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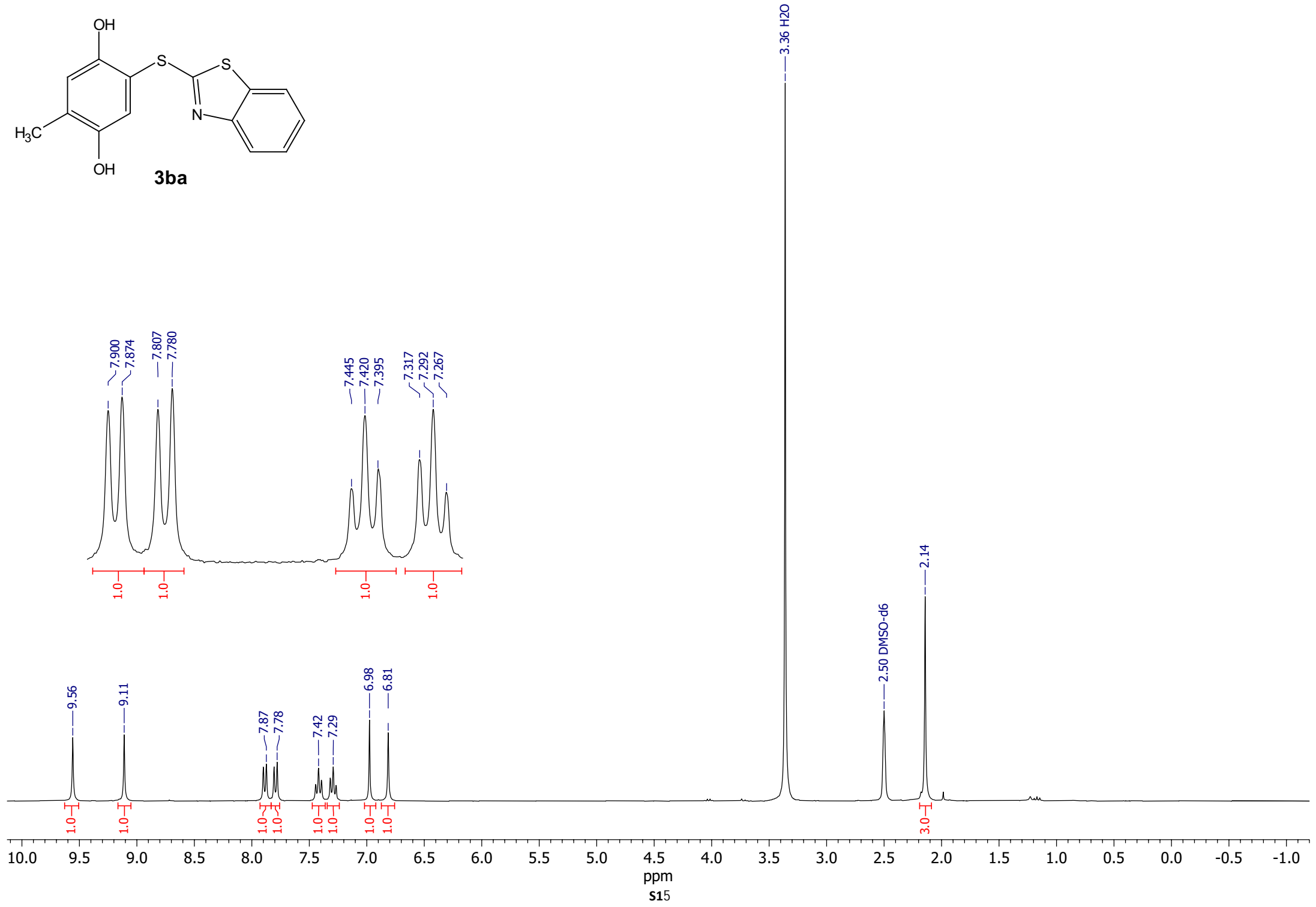
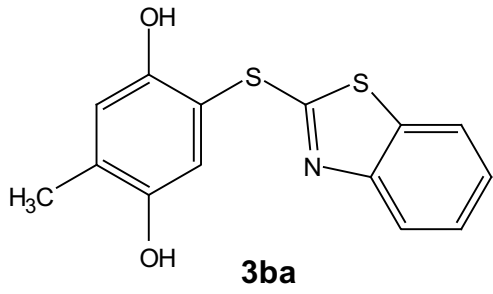
Acquisition Date 15.05.2024 10:24:07  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

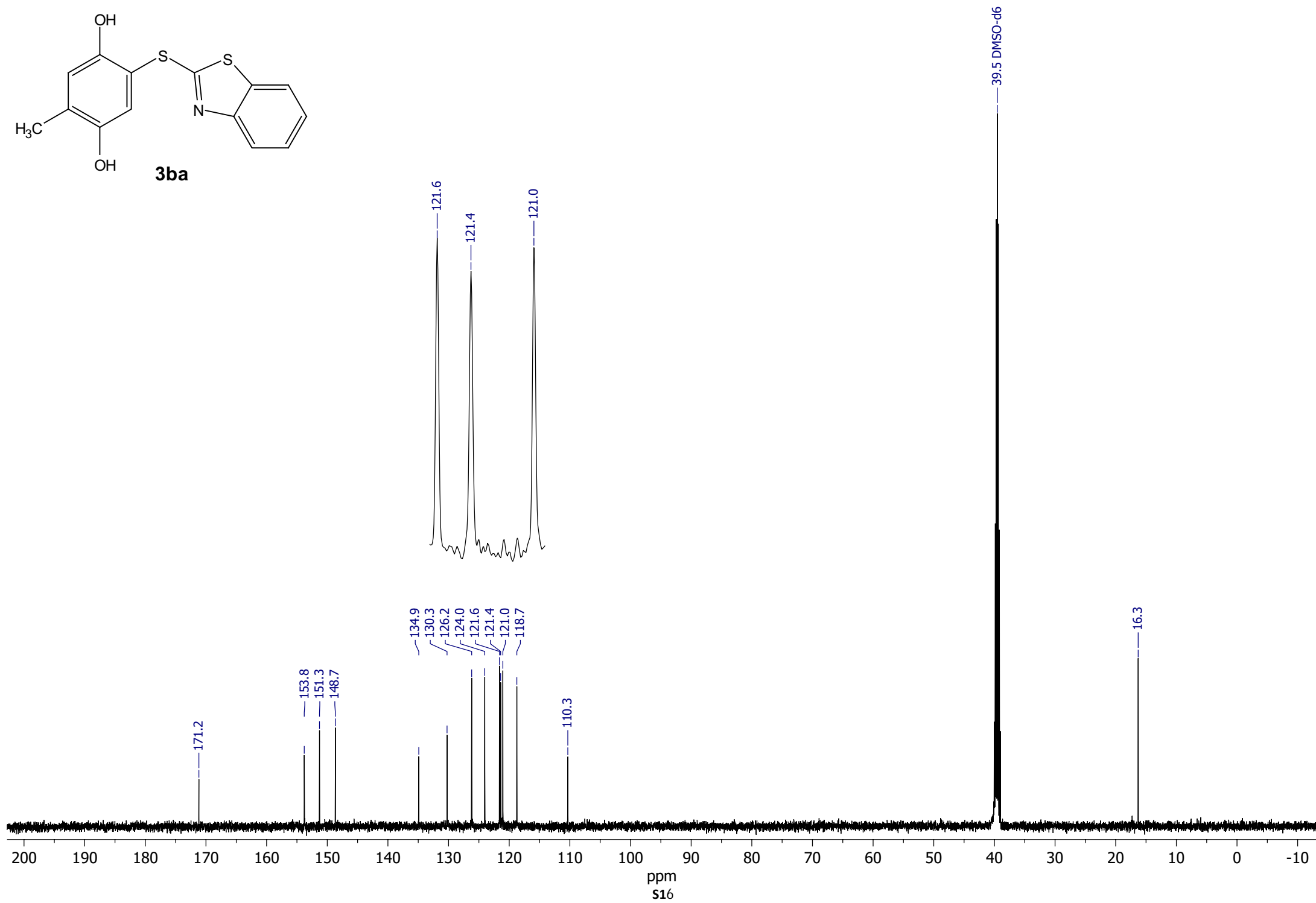
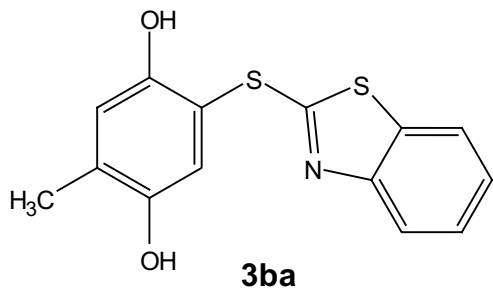
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Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



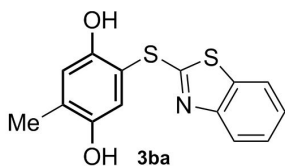
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



<sup>13</sup>C NMR (125.77 MHz, DMSO-d<sub>6</sub>)







Chemical Formula: C<sub>14</sub>H<sub>11</sub>NO<sub>2</sub>S<sub>2</sub>  
 Exact Mass: 289,02

**Analysis Info**

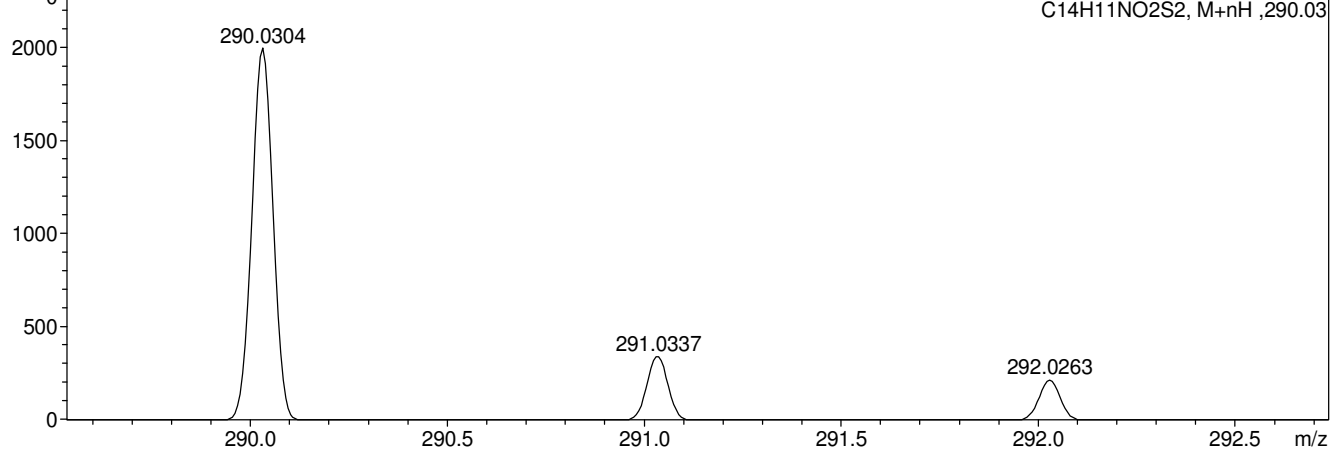
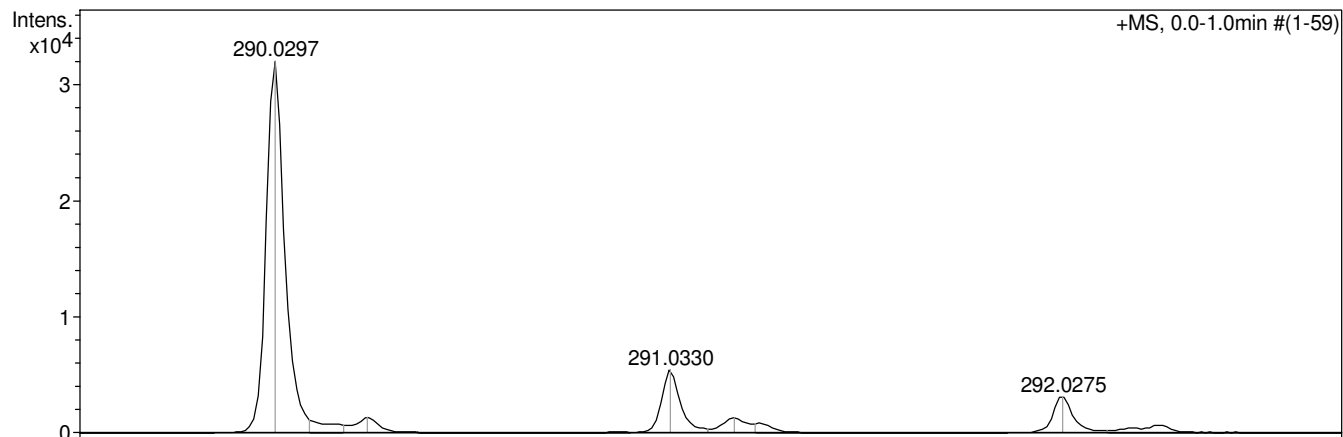
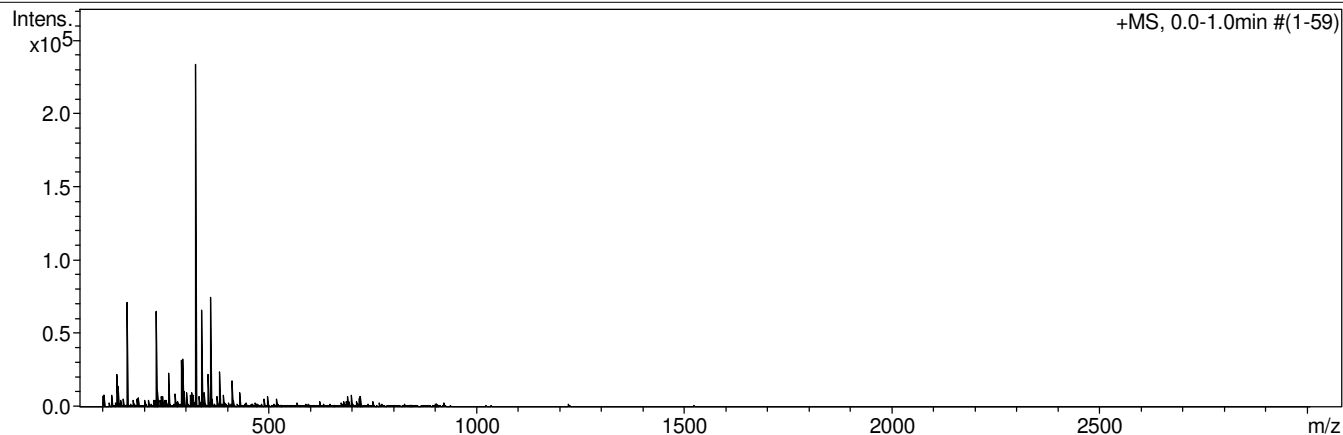
Analysis Name D:\Data\Chizhov\Egorov\Moiseeva\mnv297\_&clblow.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV297  
 Comment CH3CN 100 %, dil. 200, low conc. calibrant added

Acquisition Date 15.01.2024 14:23:29

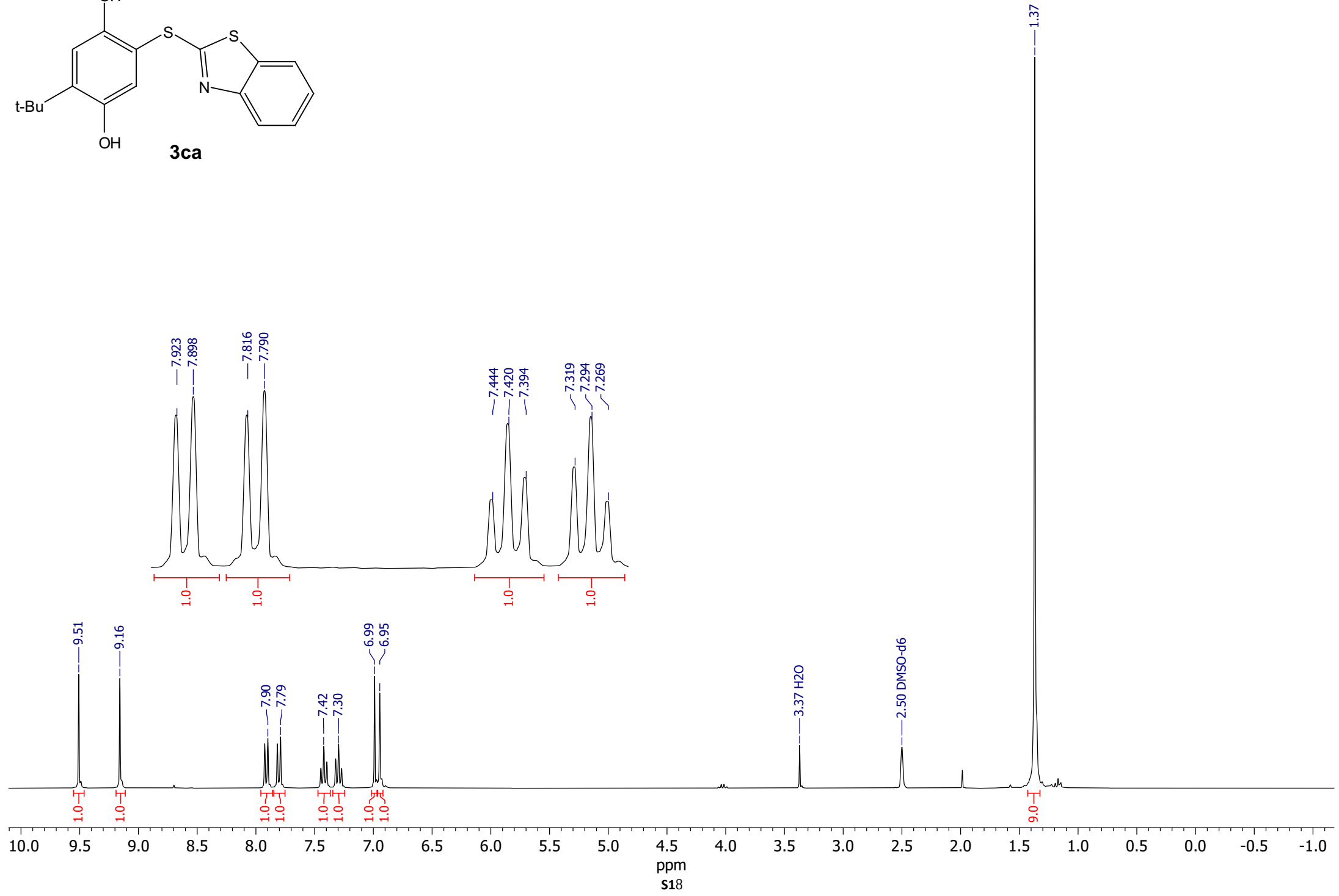
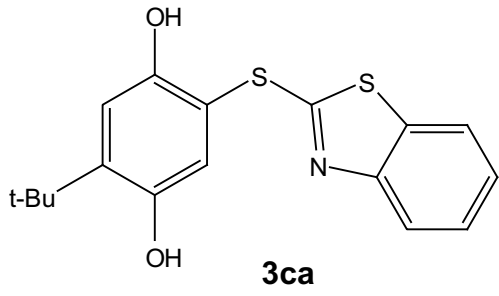
Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

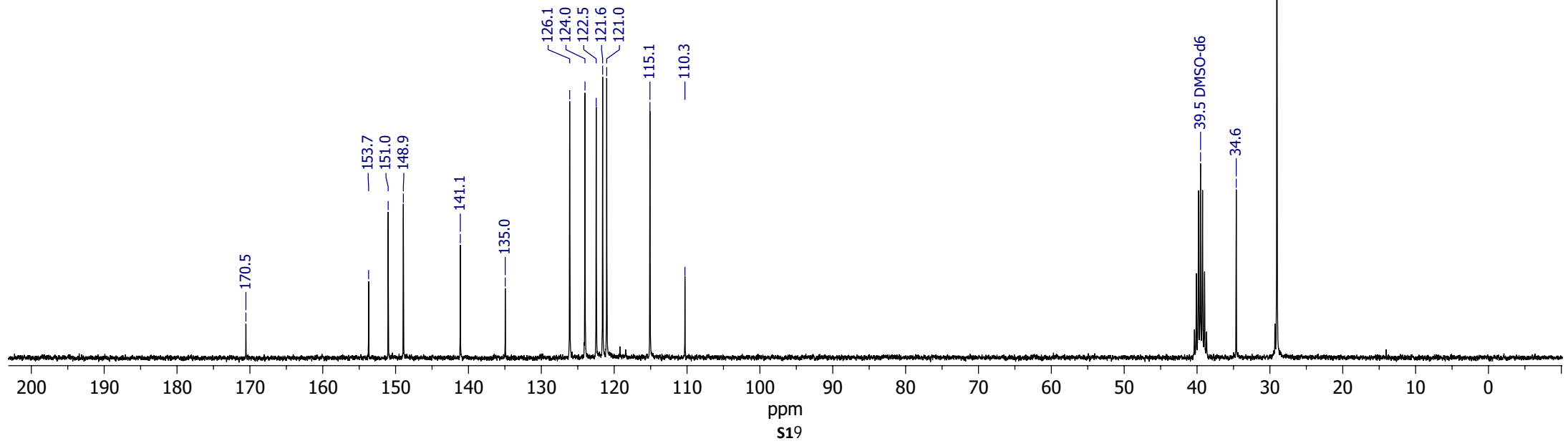
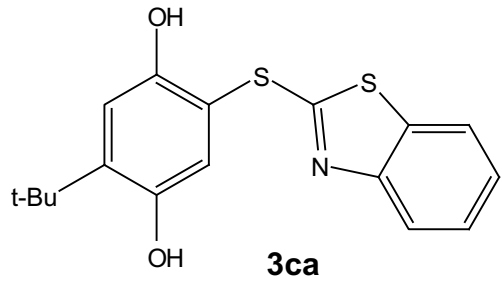
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Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

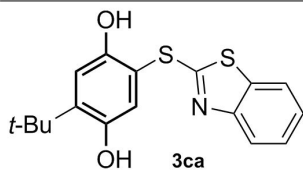


<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)





Chemical Formula: C<sub>17</sub>H<sub>17</sub>NO<sub>2</sub>S<sub>2</sub>  
 Exact Mass: 331,07

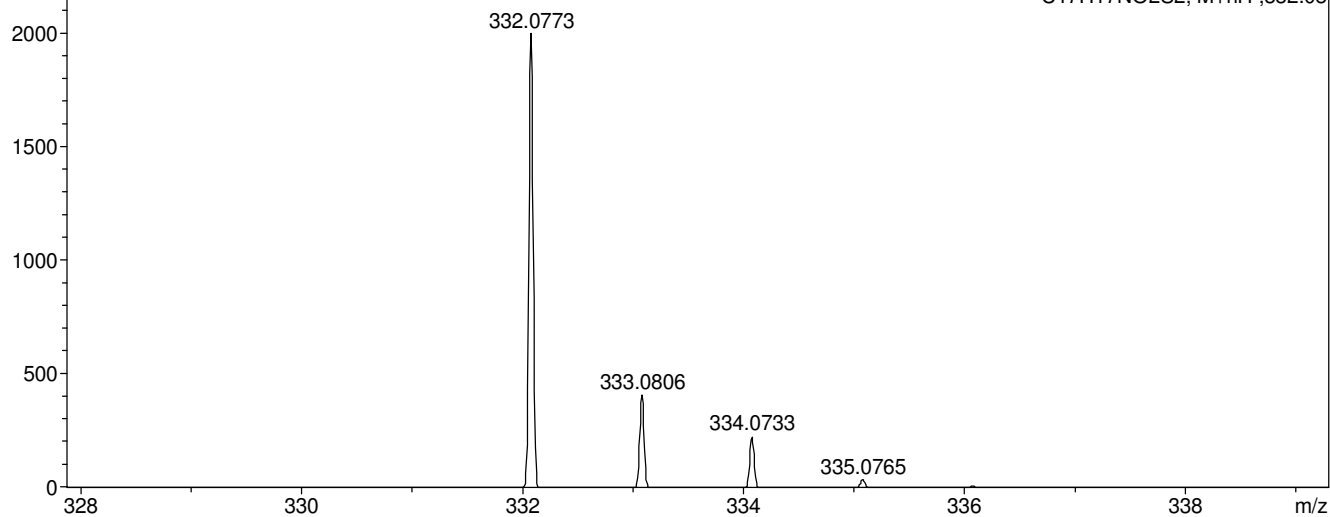
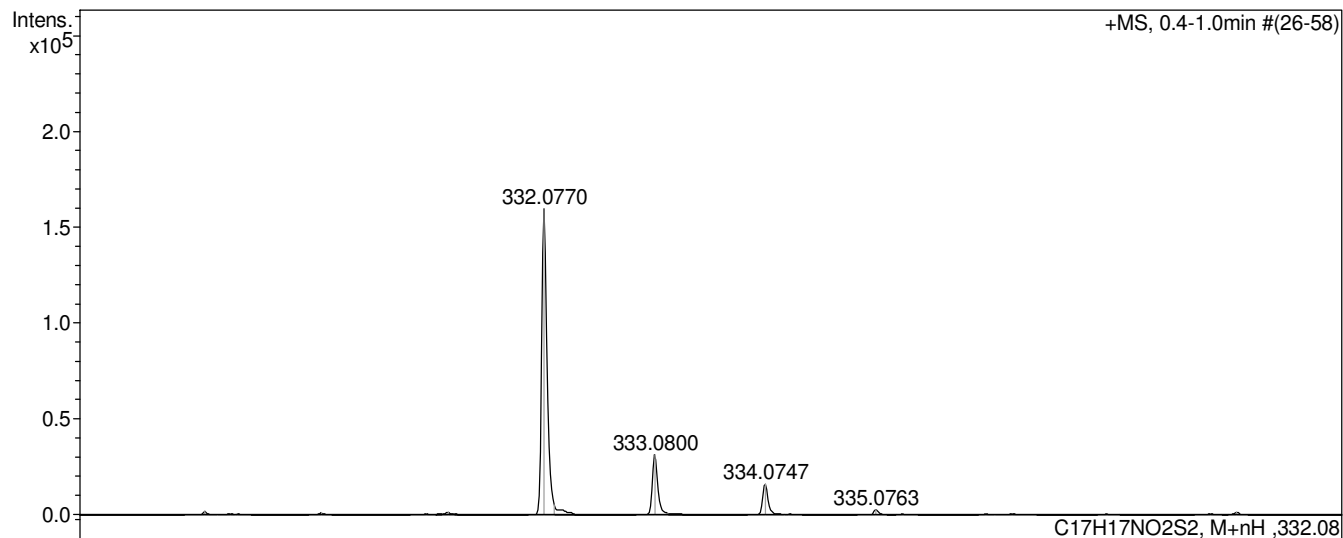
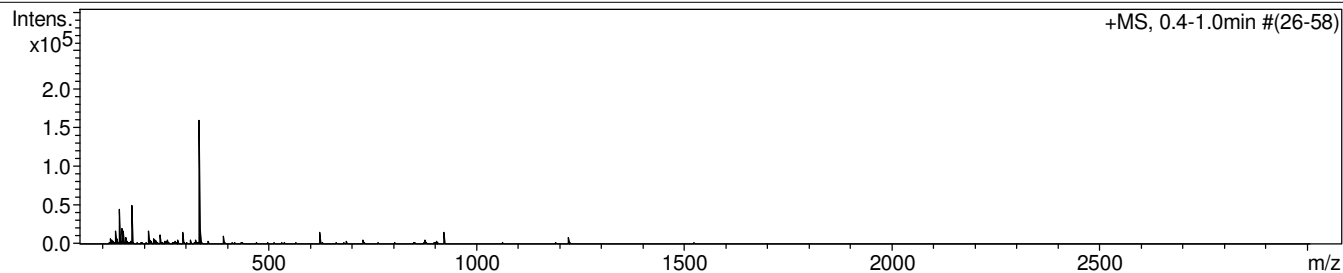
**Analysis Info**

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 Sample Name /VAPP MNV 319  
 Comment C17H17NO2S2 mH332.0773 calibrant added CH3CN

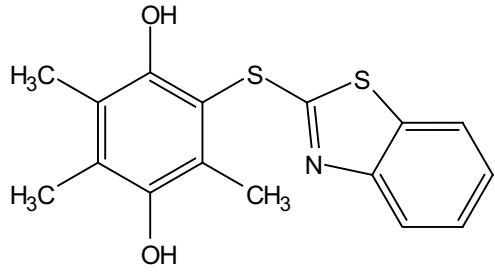
Acquisition Date 17.05.2024 9:33:48  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

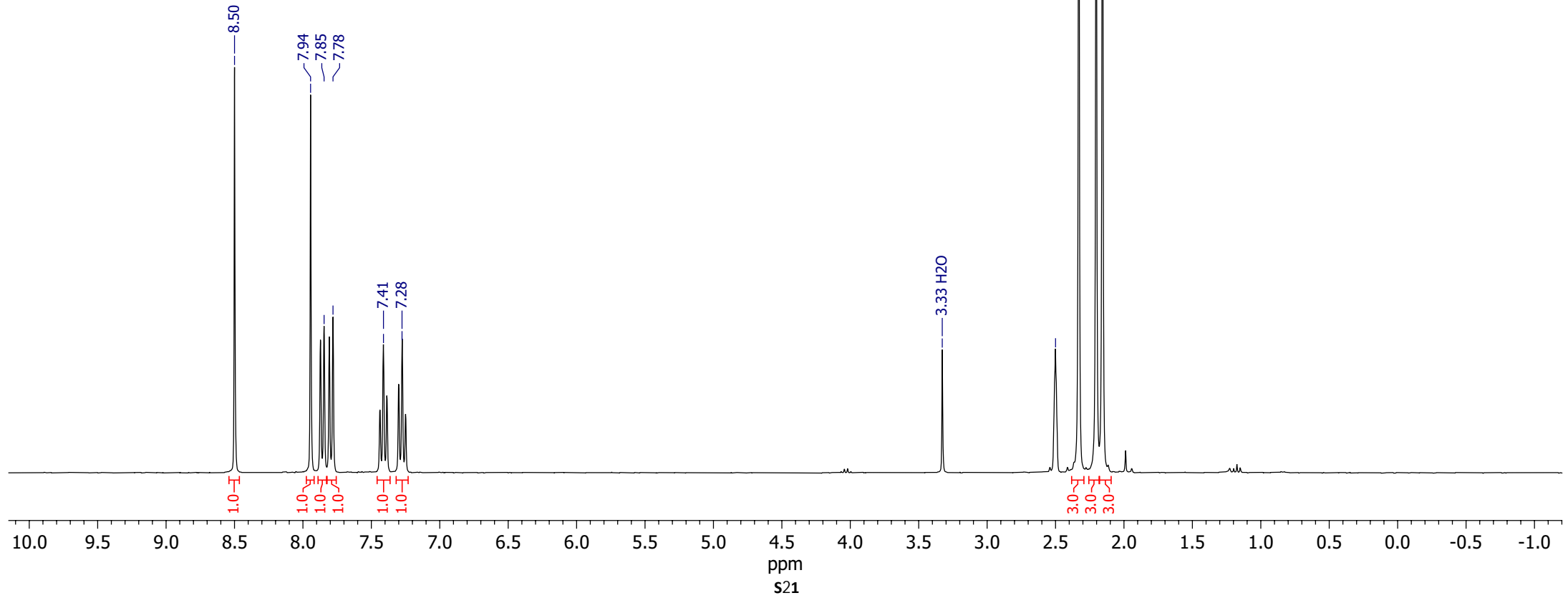
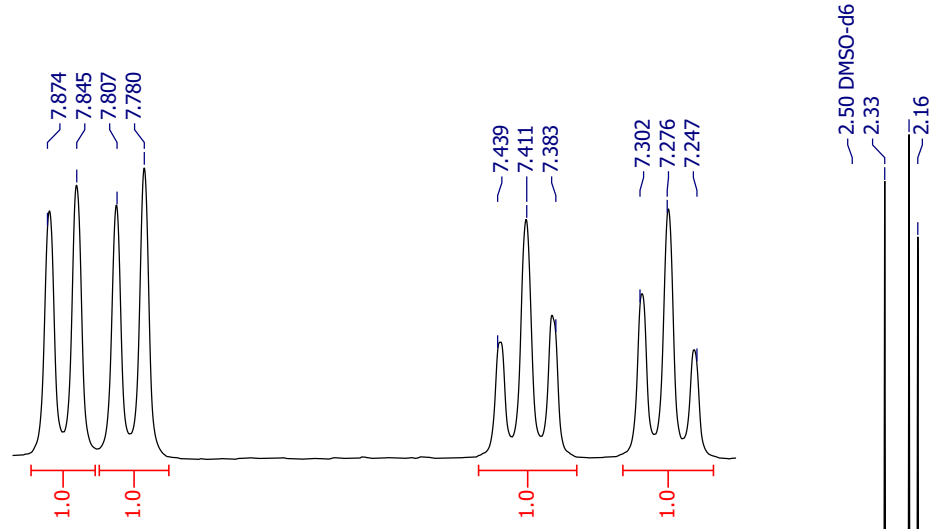
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Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



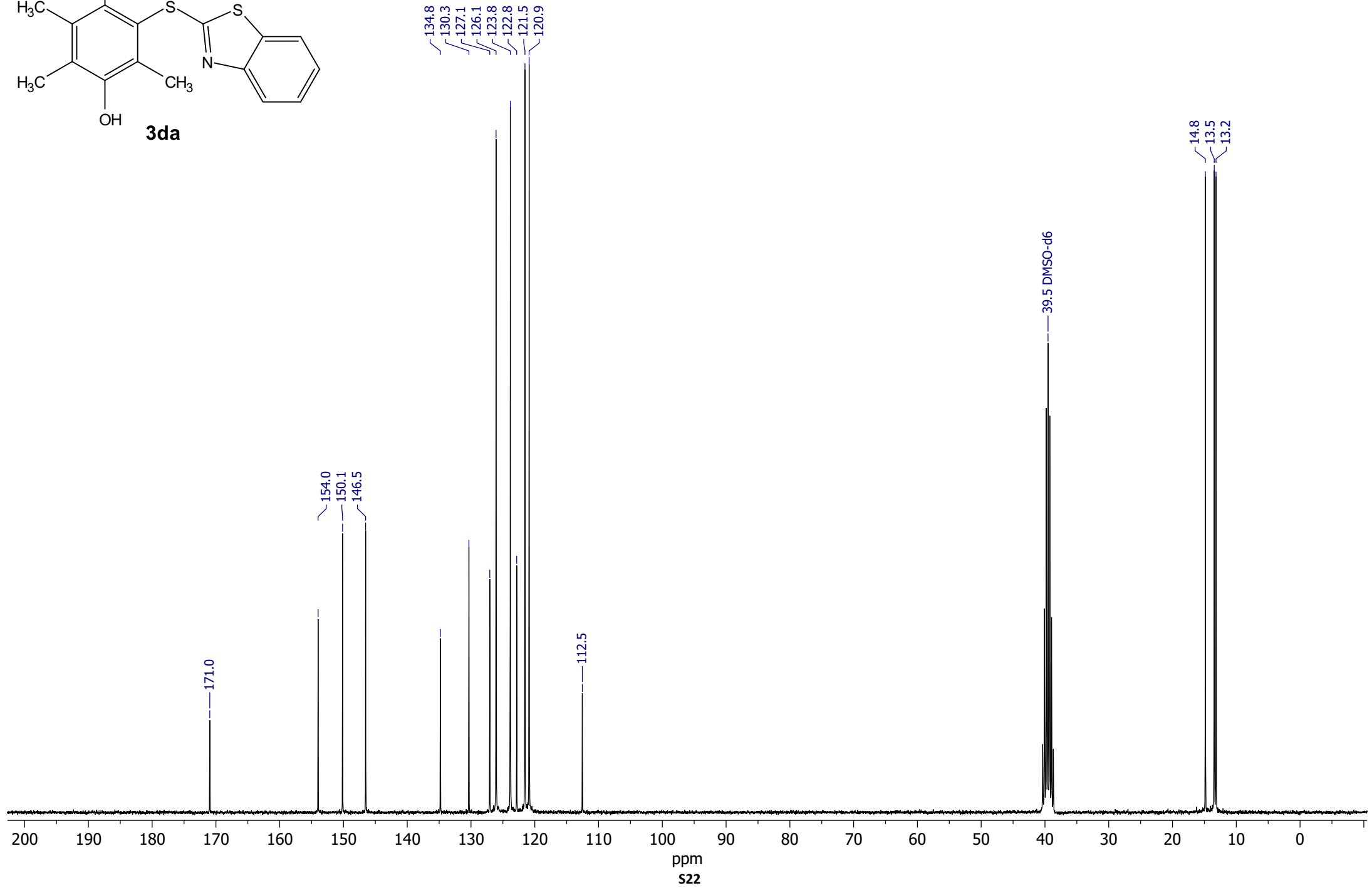
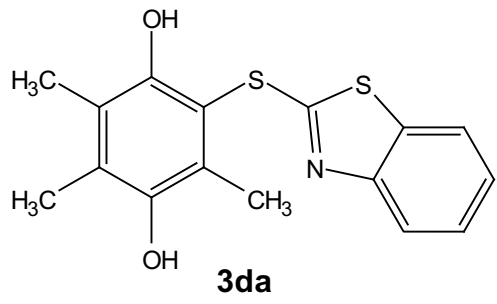
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)

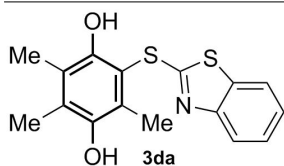


**3da**



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)





Chemical Formula: C<sub>16</sub>H<sub>15</sub>NO<sub>2</sub>S<sub>2</sub>  
 Exact Mass: 317,05

**Analysis Info**

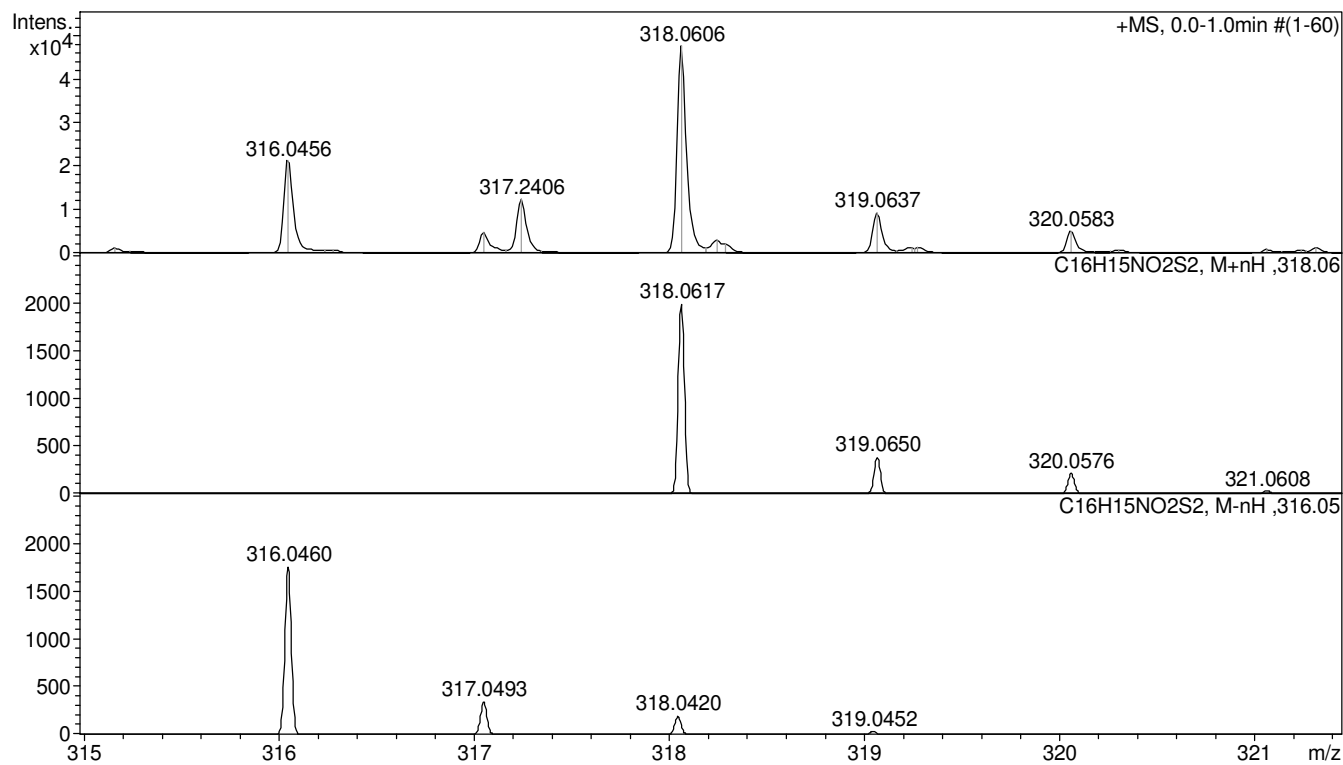
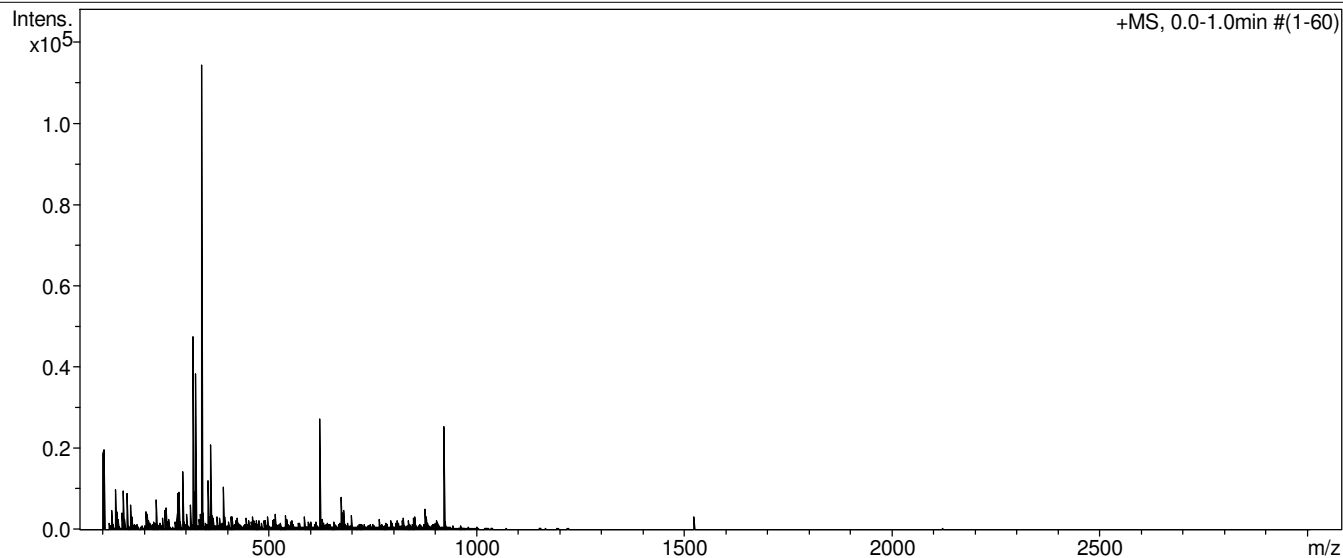
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 Sample Name /VAPP MNV316  
 Comment CH3CN 100 %, dil. 20, calibrant added

Acquisition Date 16.02.2024 16:15:27

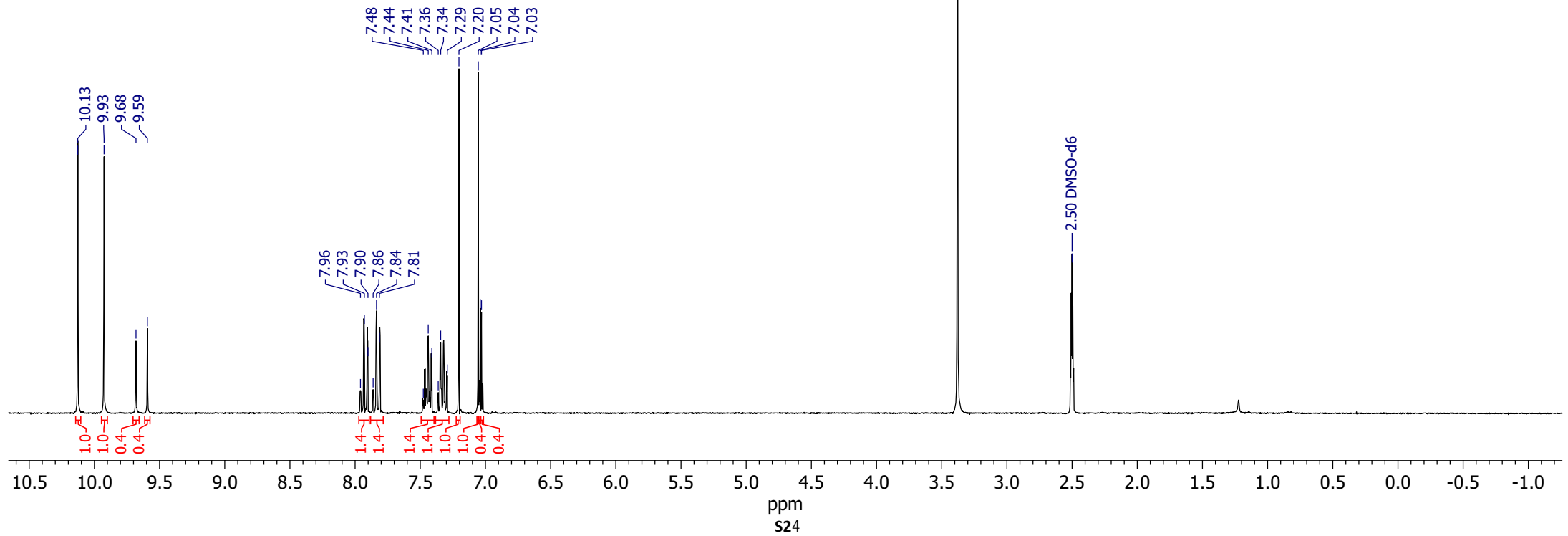
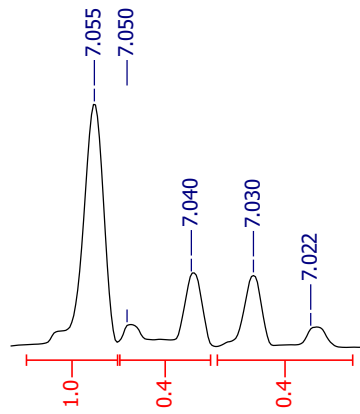
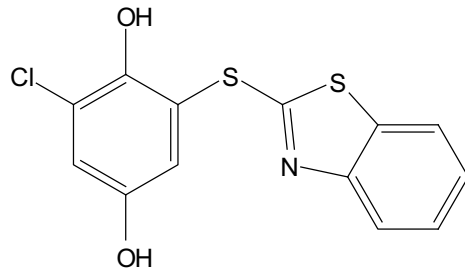
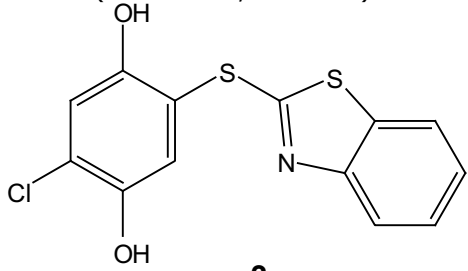
Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

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Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

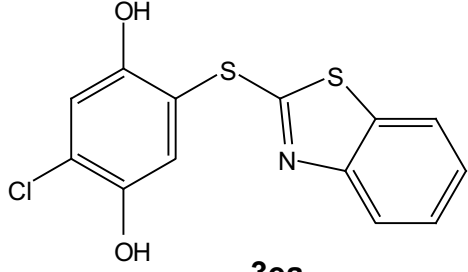


<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)

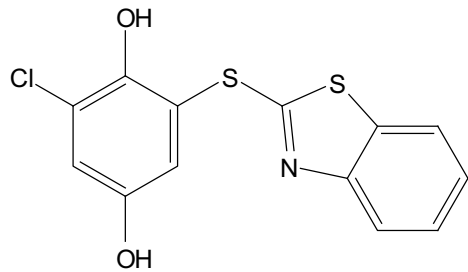




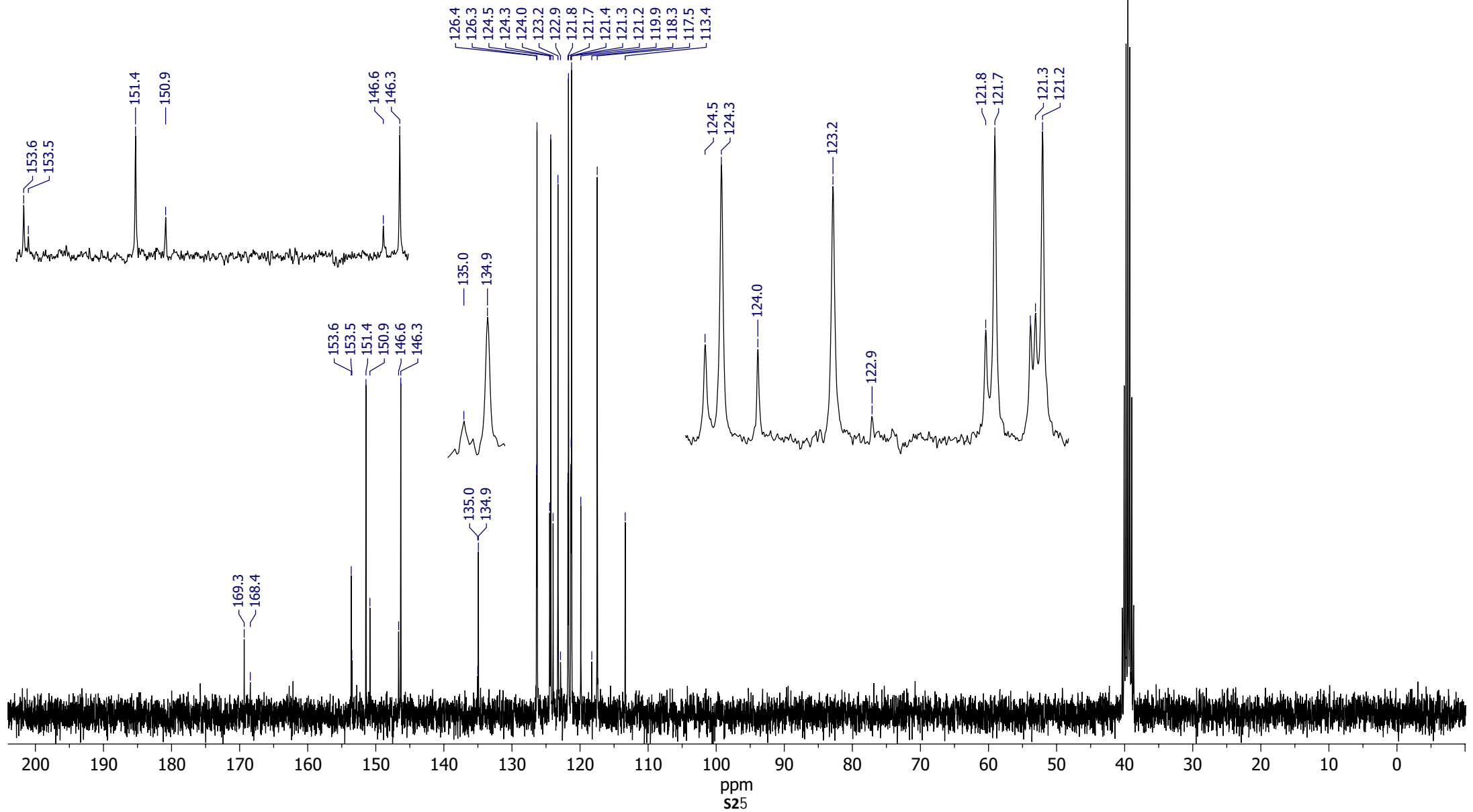
<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)

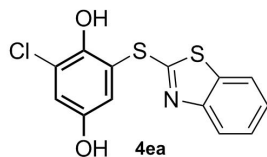
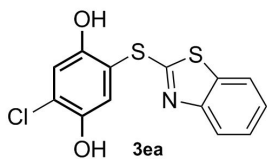


**3ea**



**4ea**





Chemical Formula: C<sub>13</sub>H<sub>8</sub>ClNO<sub>2</sub>S<sub>2</sub>  
Exact Mass: 308,97

**Analysis Info**

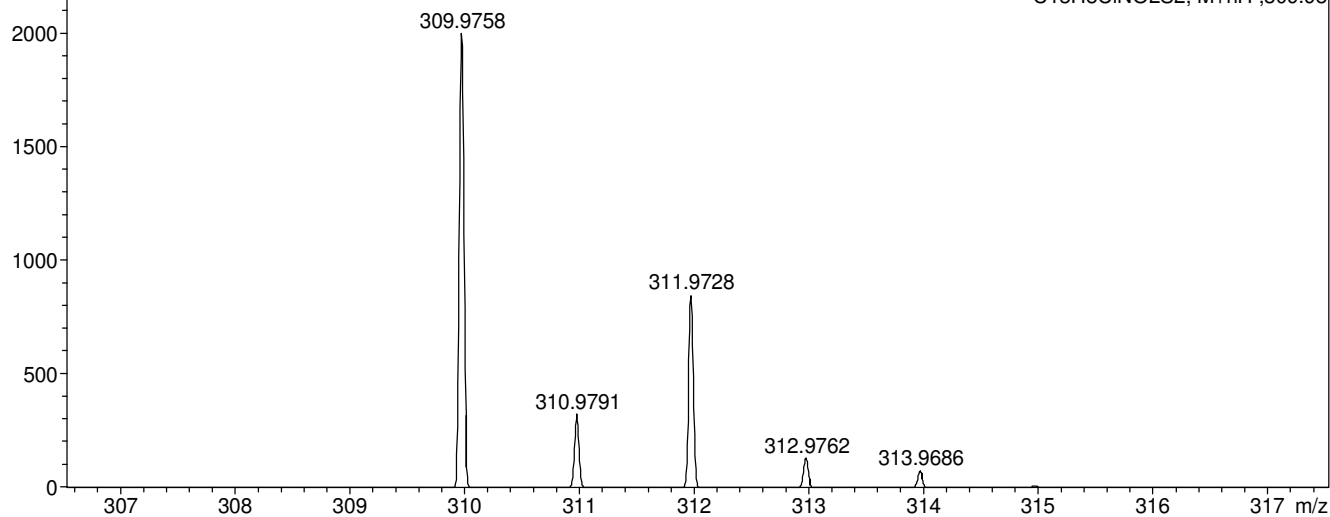
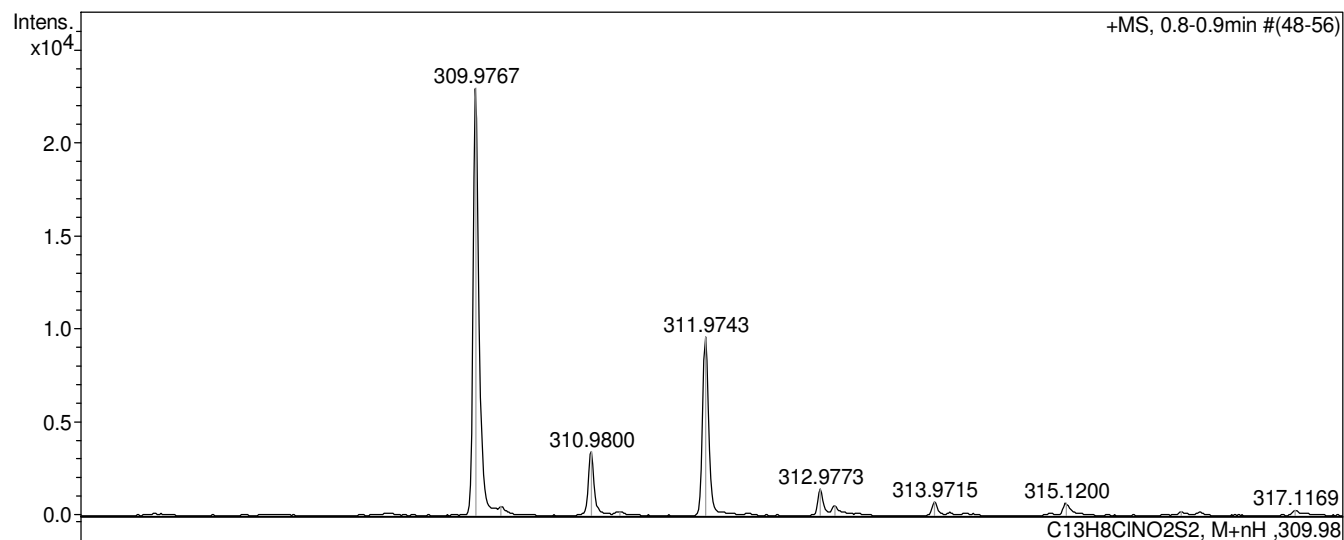
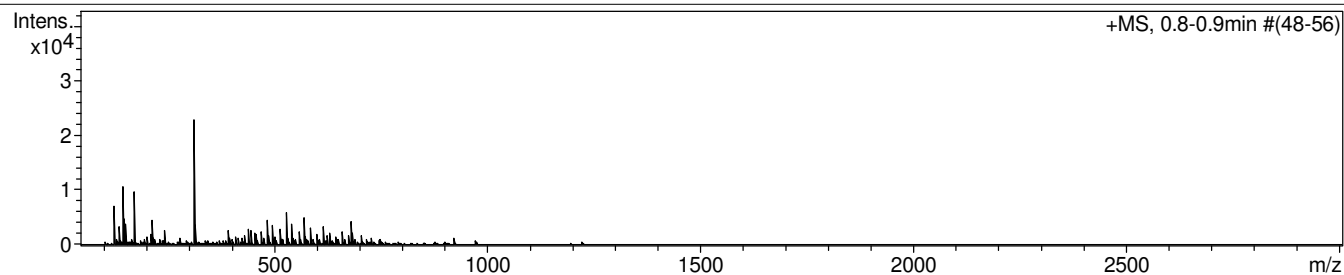
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Sample Name /VAPP MNV382  
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Acquisition Date 16.07.2024 10:41:11

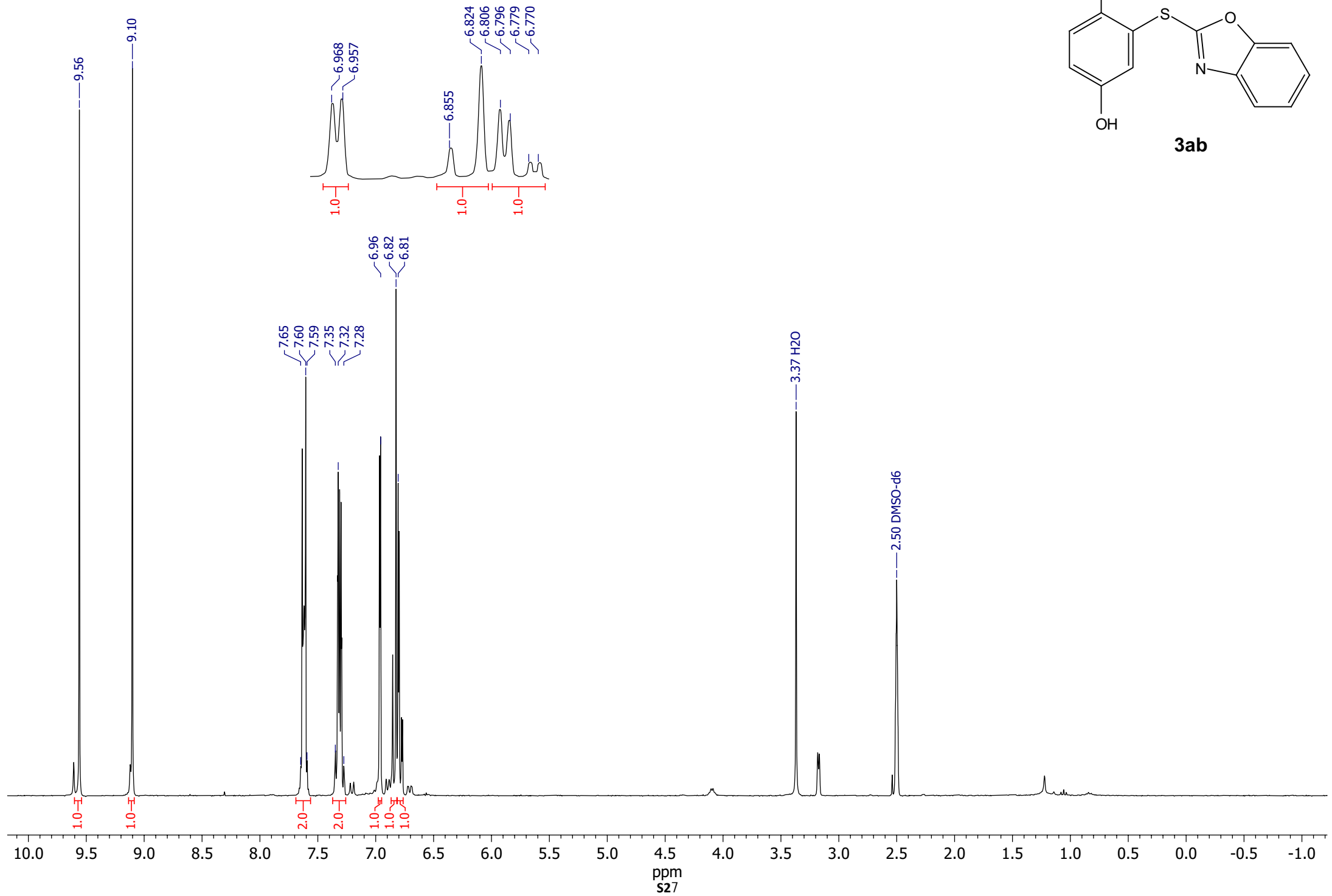
Operator BDAL@DE  
Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

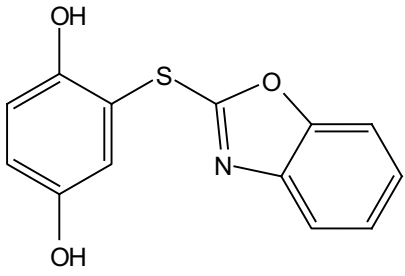
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Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



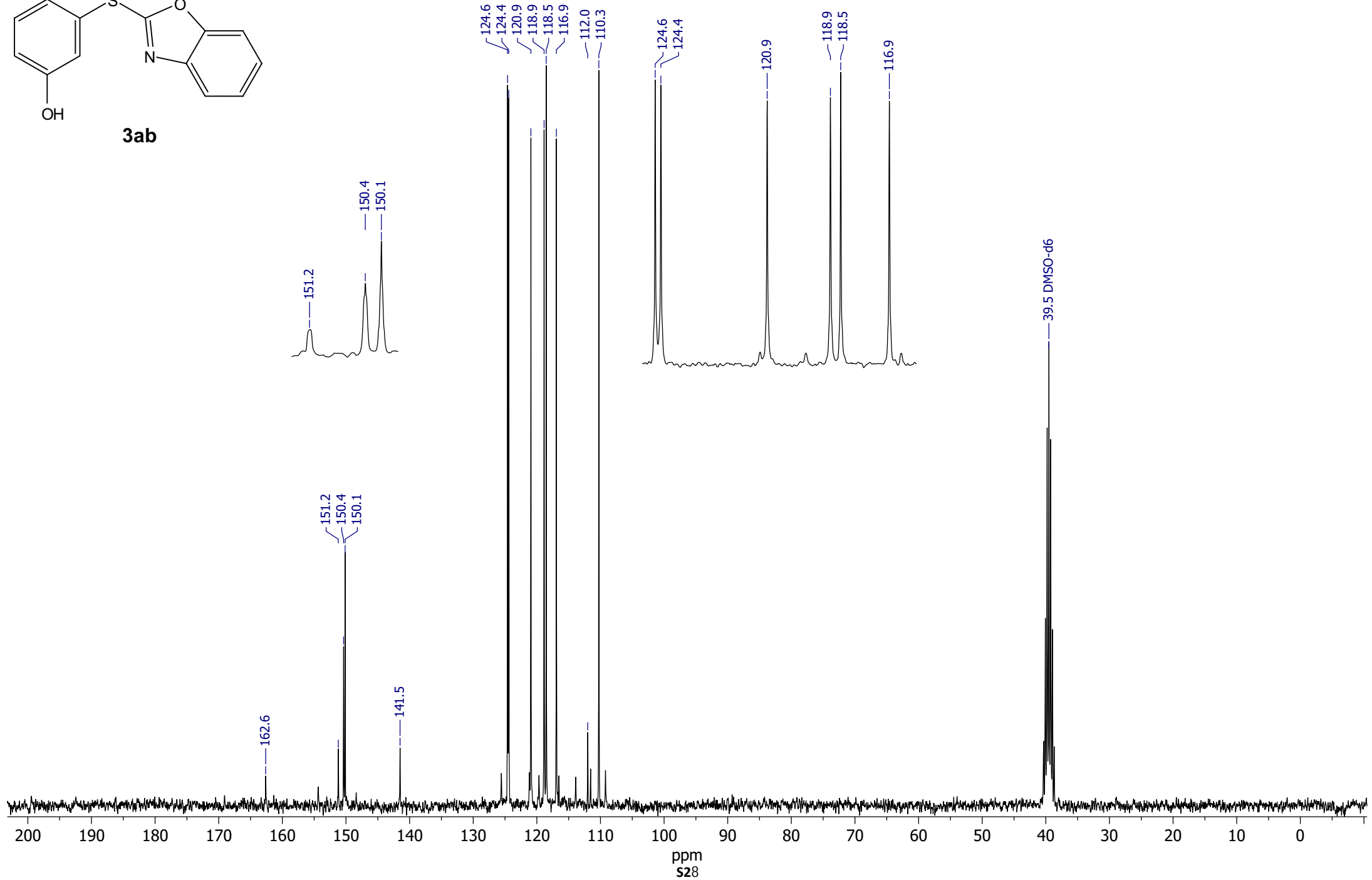
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)

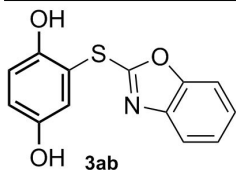


<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)



**3ab**





Chemical Formula: C<sub>13</sub>H<sub>9</sub>NO<sub>3</sub>S  
 Exact Mass: 259,03

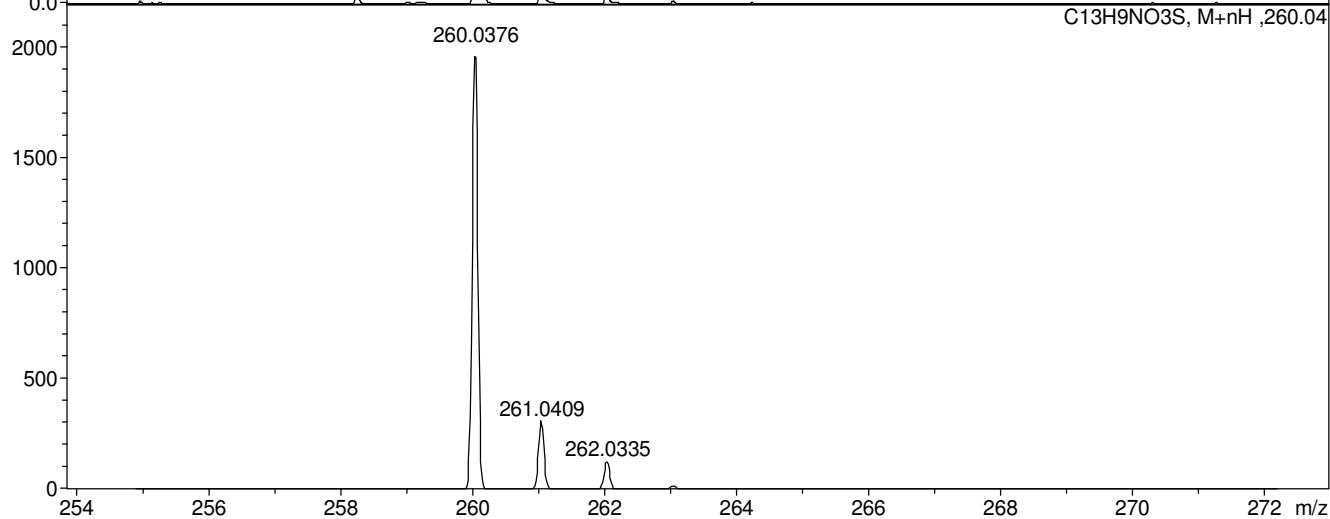
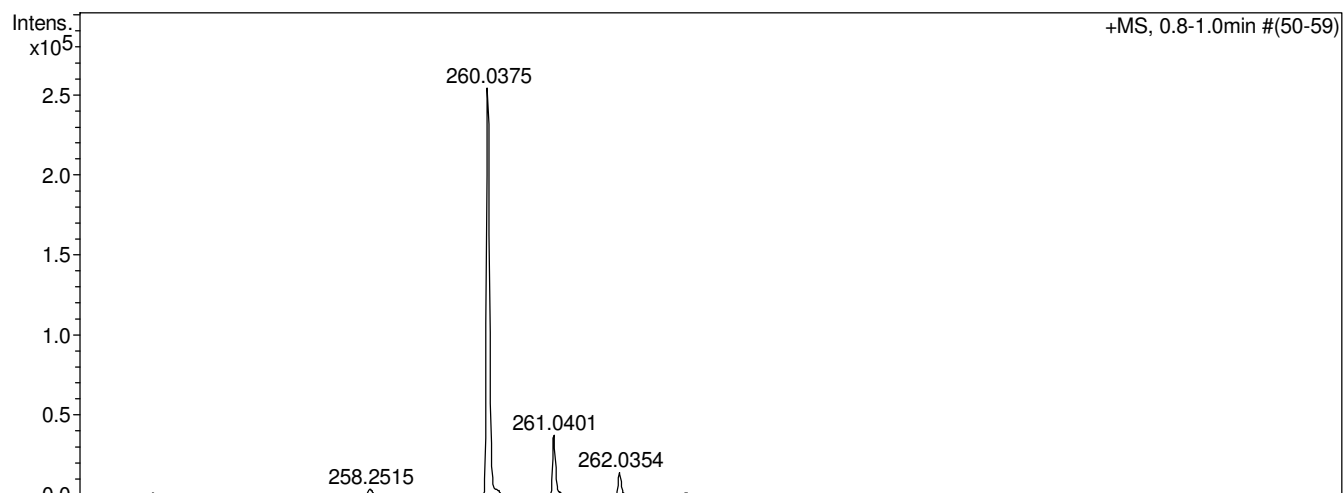
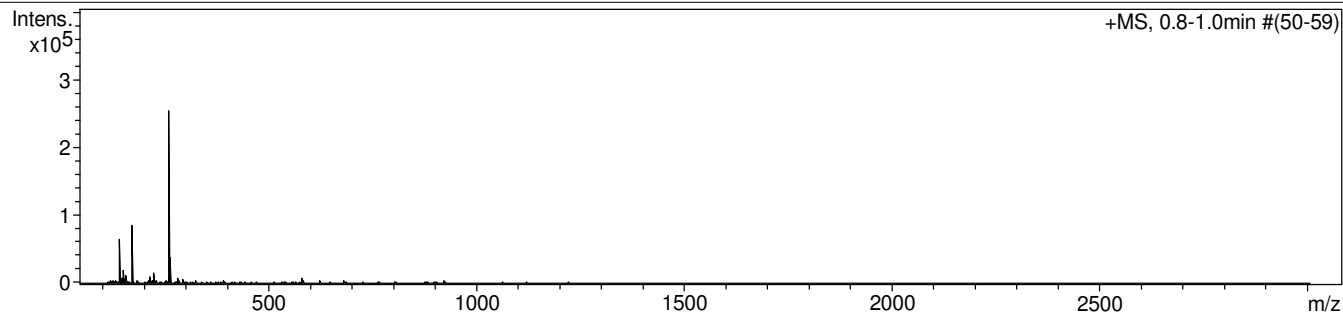
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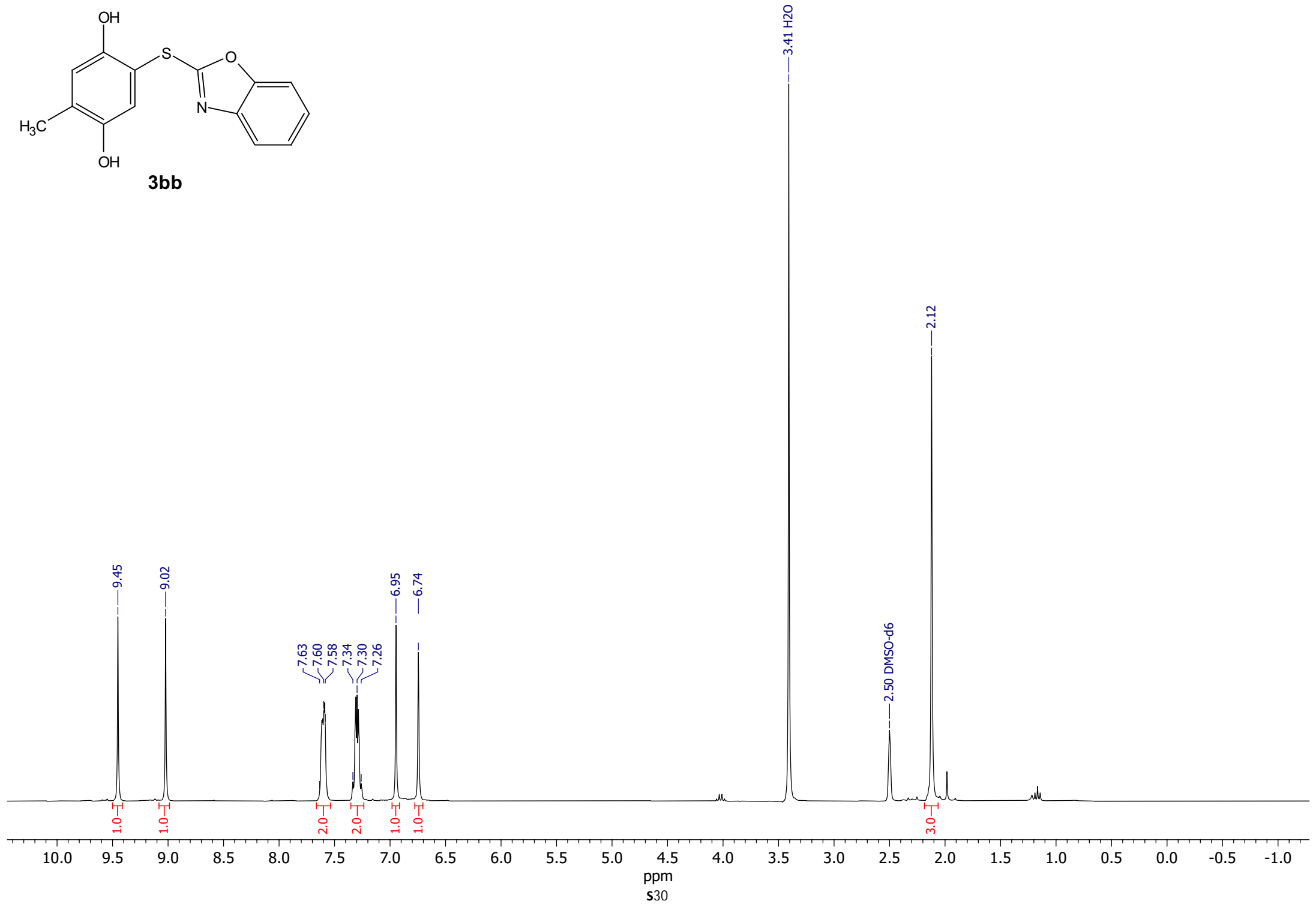
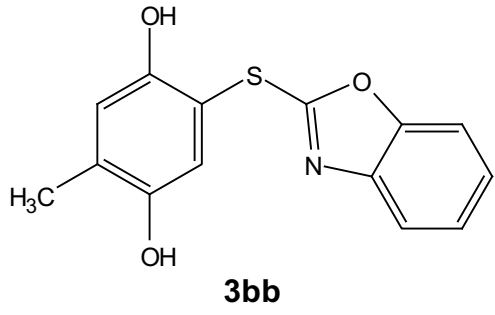
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 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

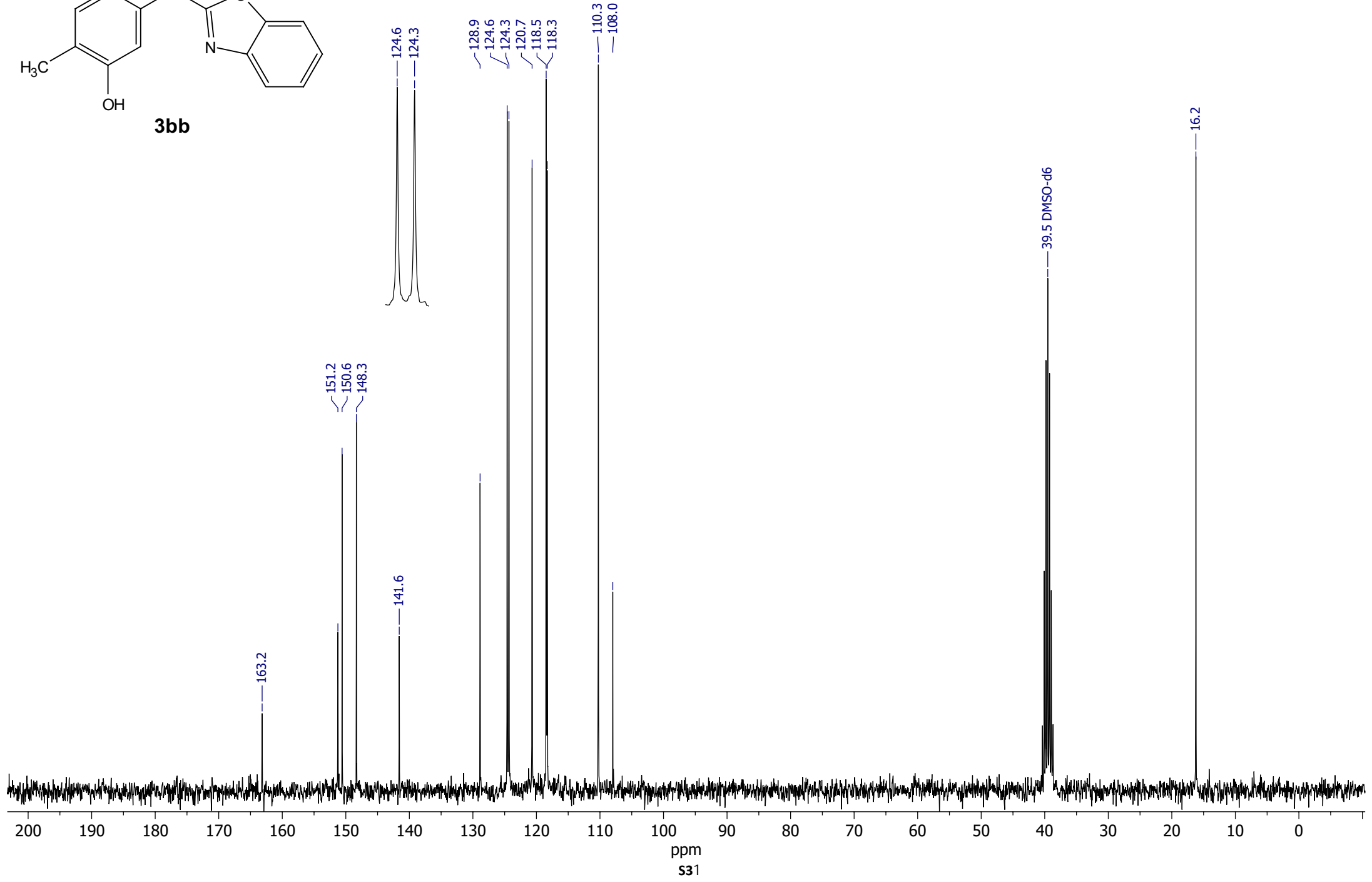
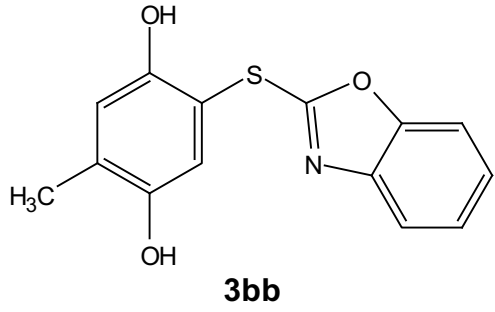
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Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

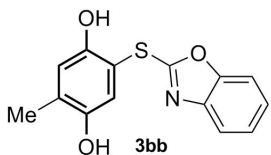


<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)





Chemical Formula: C<sub>14</sub>H<sub>11</sub>NO<sub>3</sub>S  
 Exact Mass: 273,05

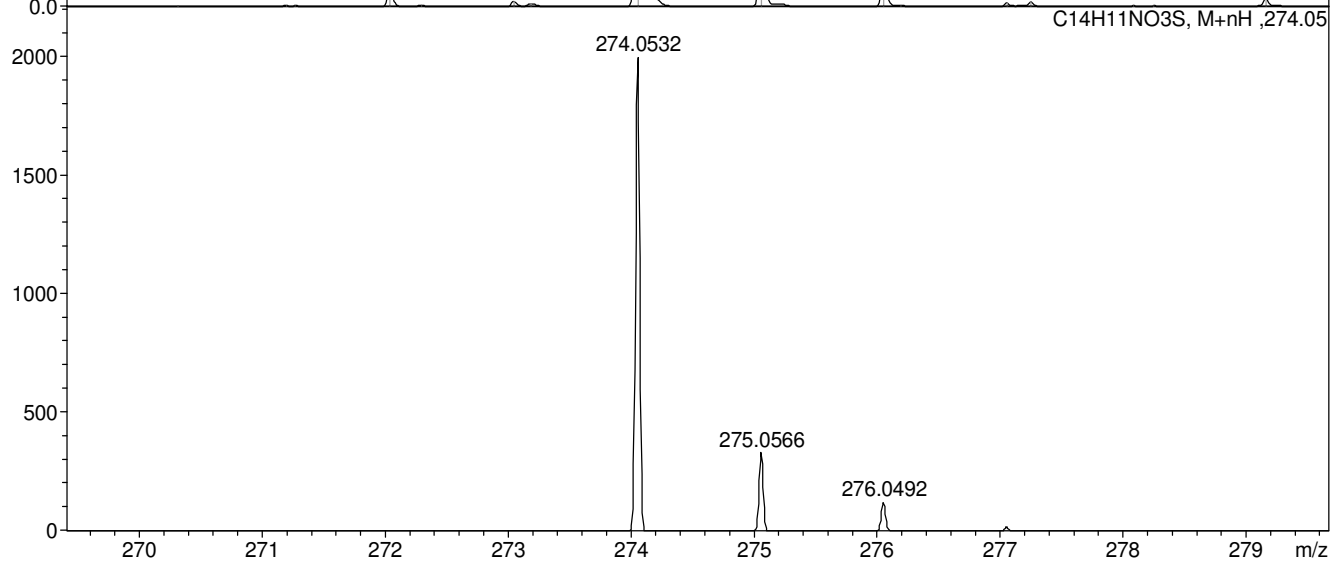
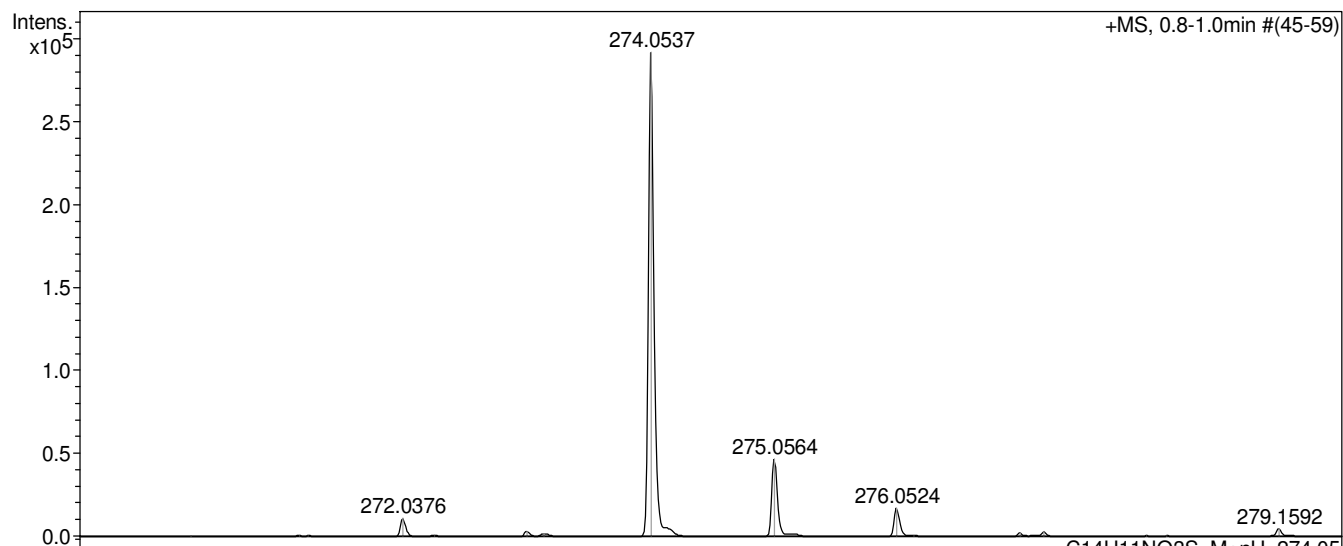
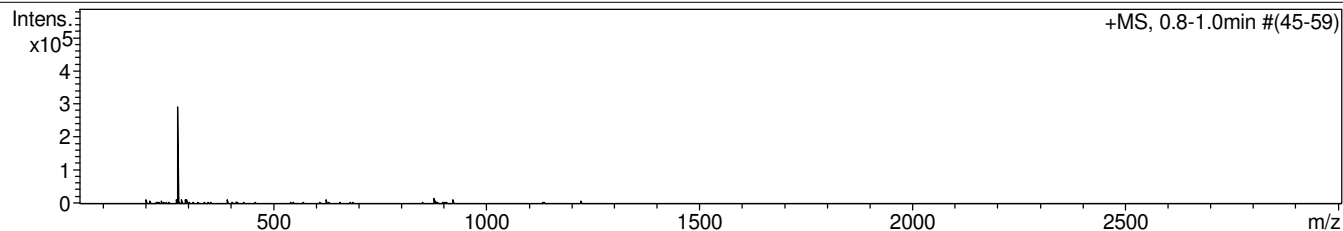
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 Sample Name /VAPP MMV348  
 Comment C14H11NO3S mH274.0532 calibrant added CH3CN

Acquisition Date 04.04.2024 16:54:12  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

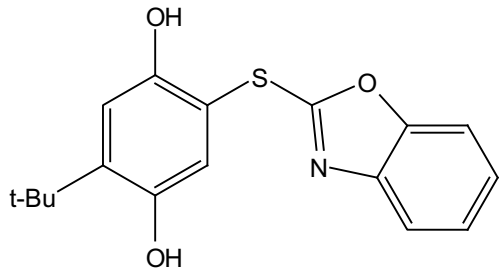
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Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

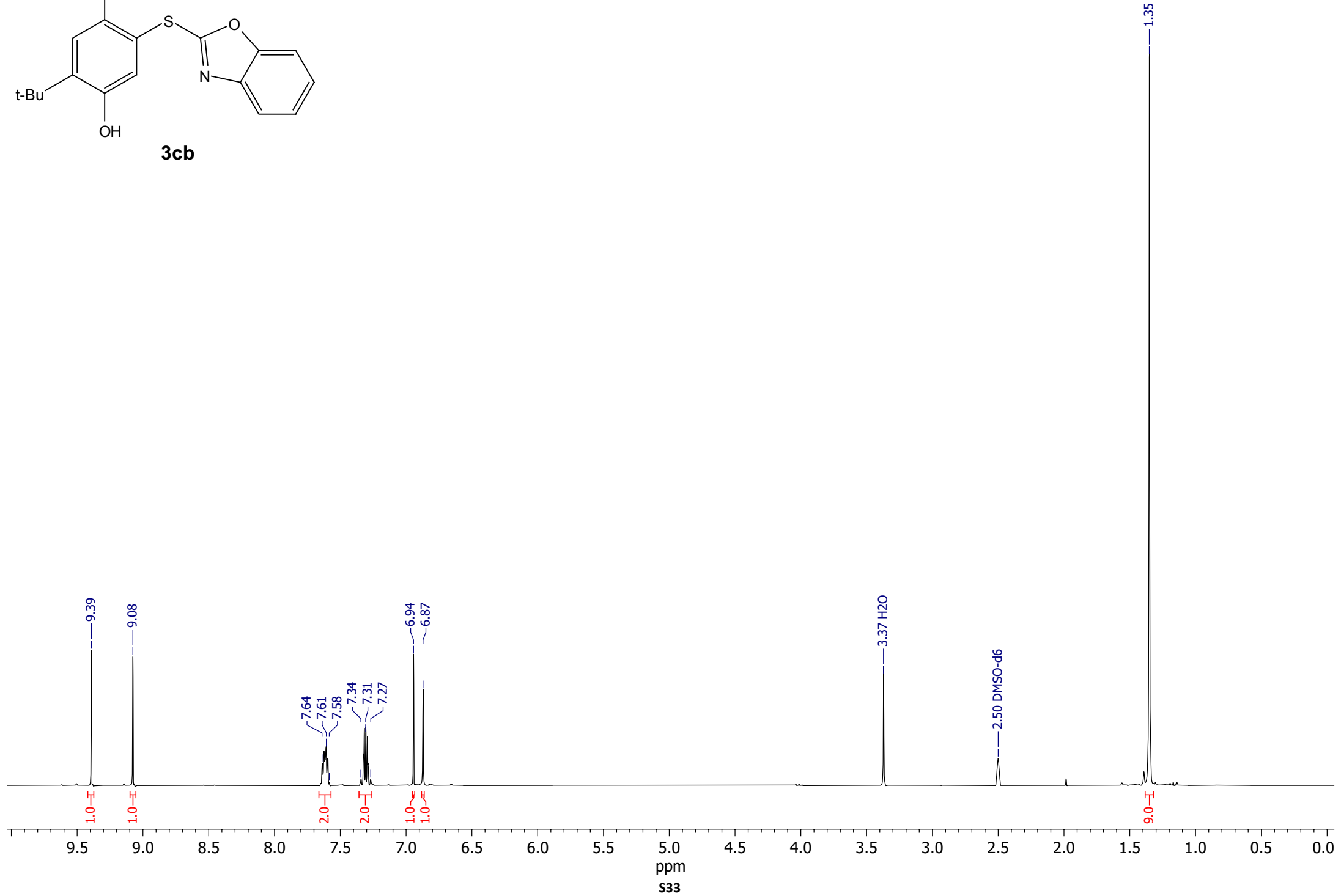




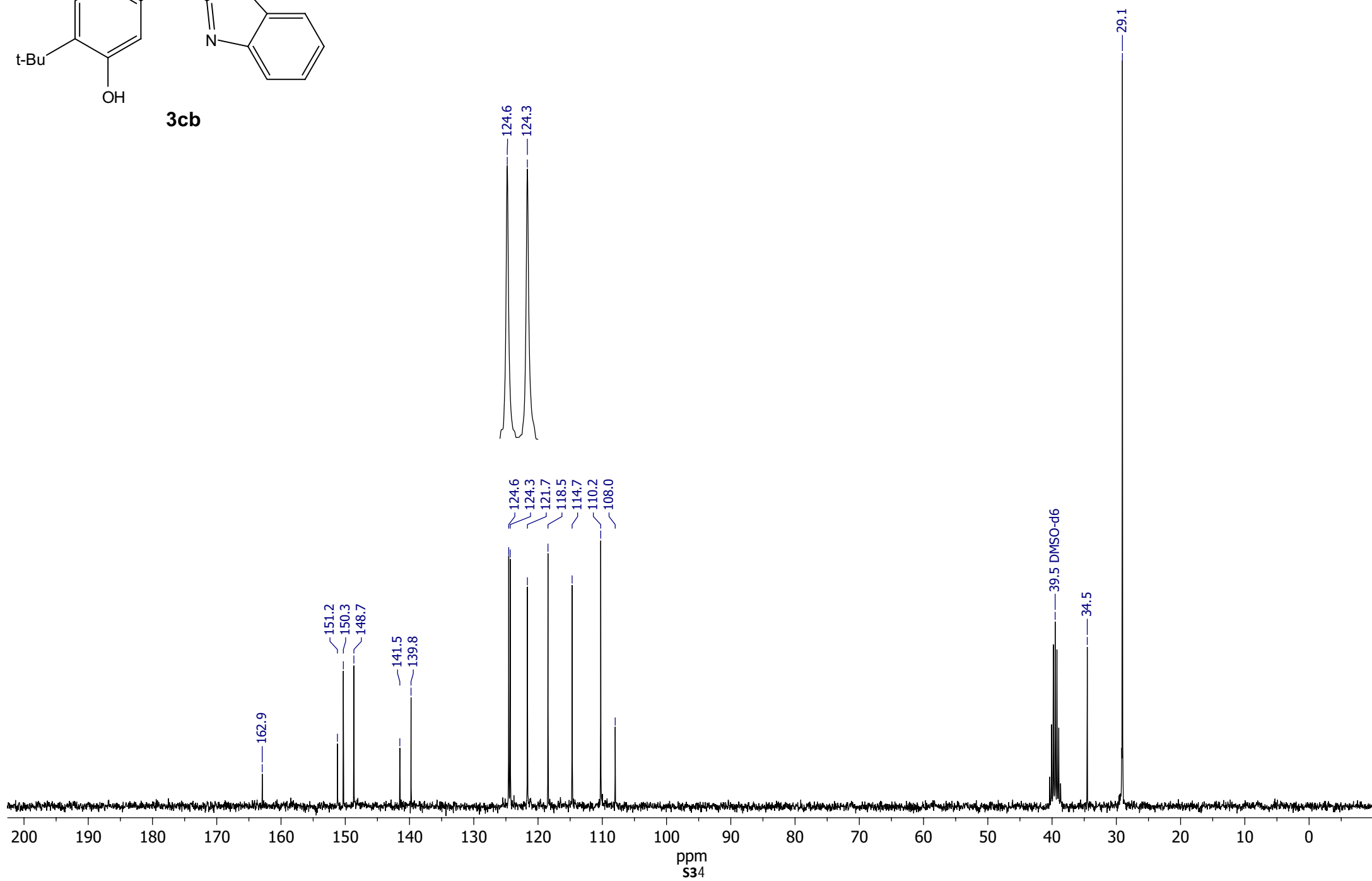
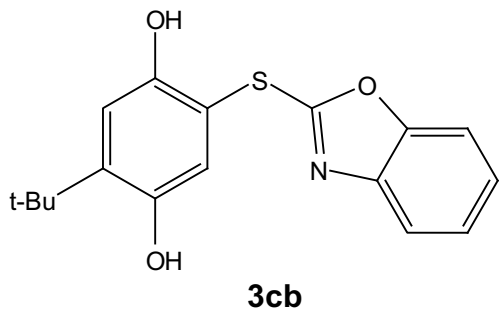
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)

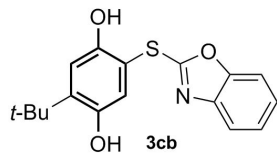


**3cb**



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)





Chemical Formula: C<sub>17</sub>H<sub>17</sub>NO<sub>3</sub>S  
 Exact Mass: 315,09

**Analysis Info**

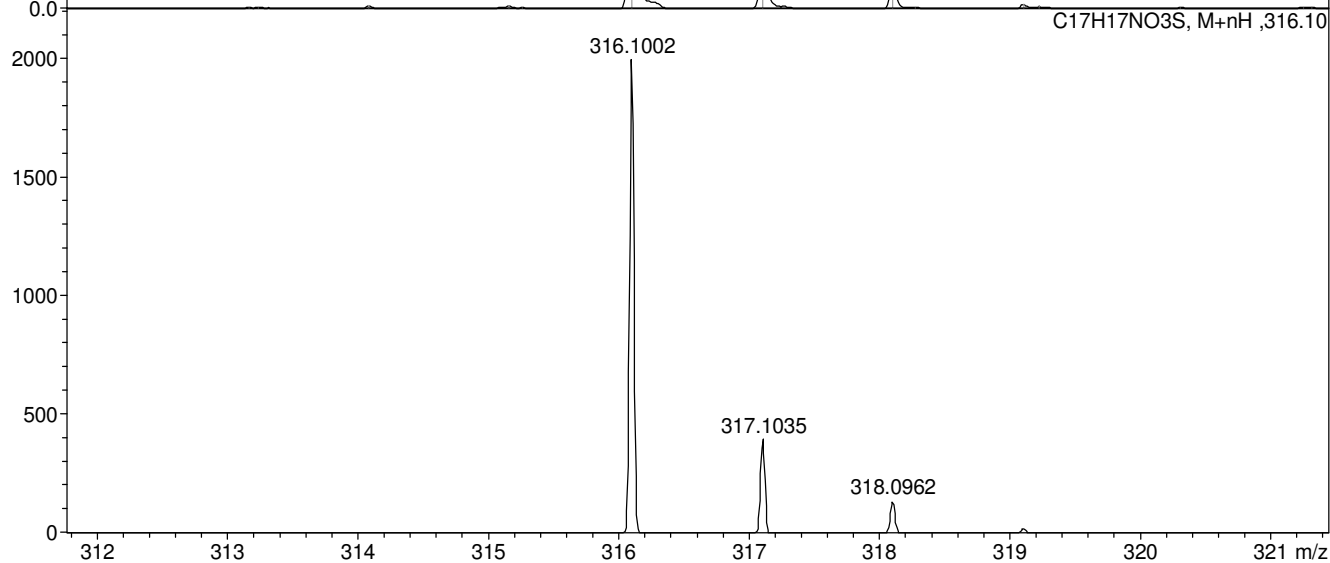
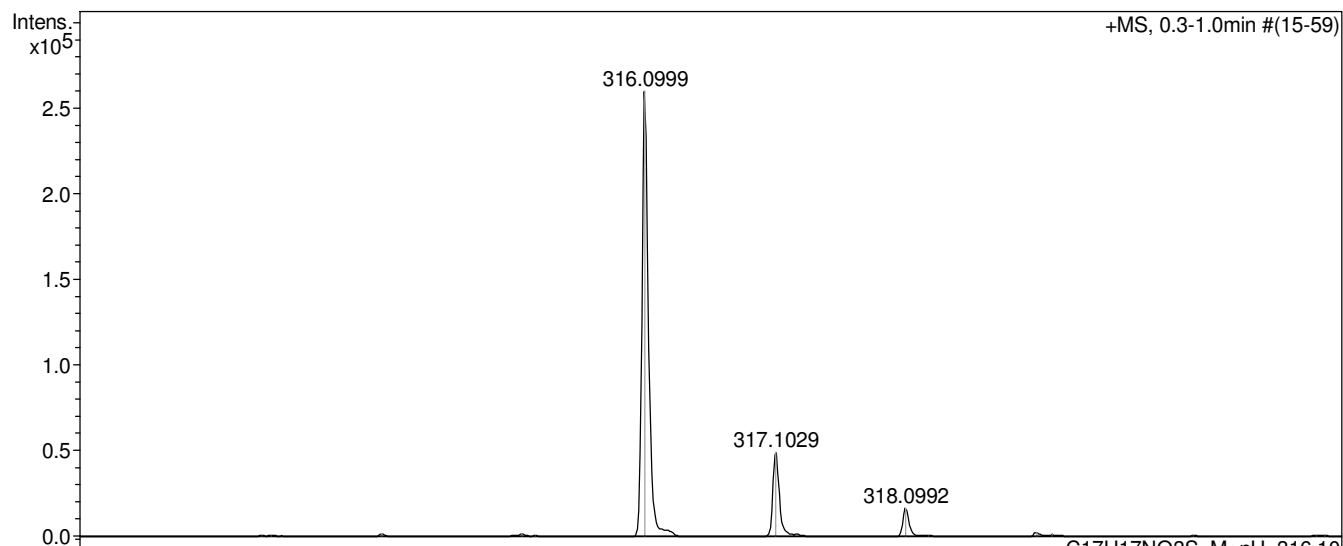
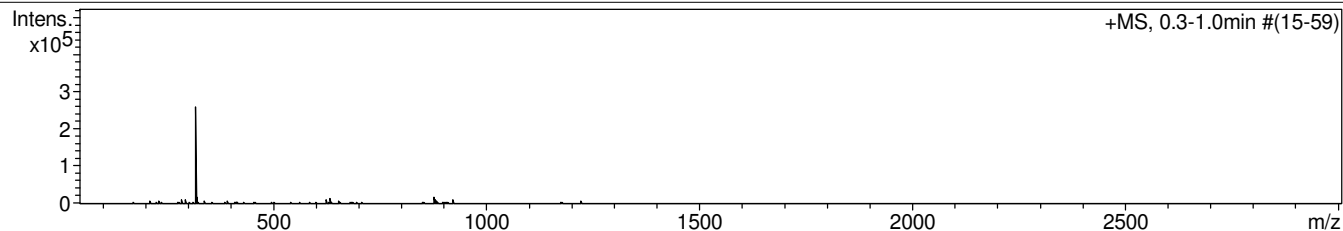
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 Sample Name /VAPP MMV344  
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Acquisition Date 04.04.2024 16:59:36

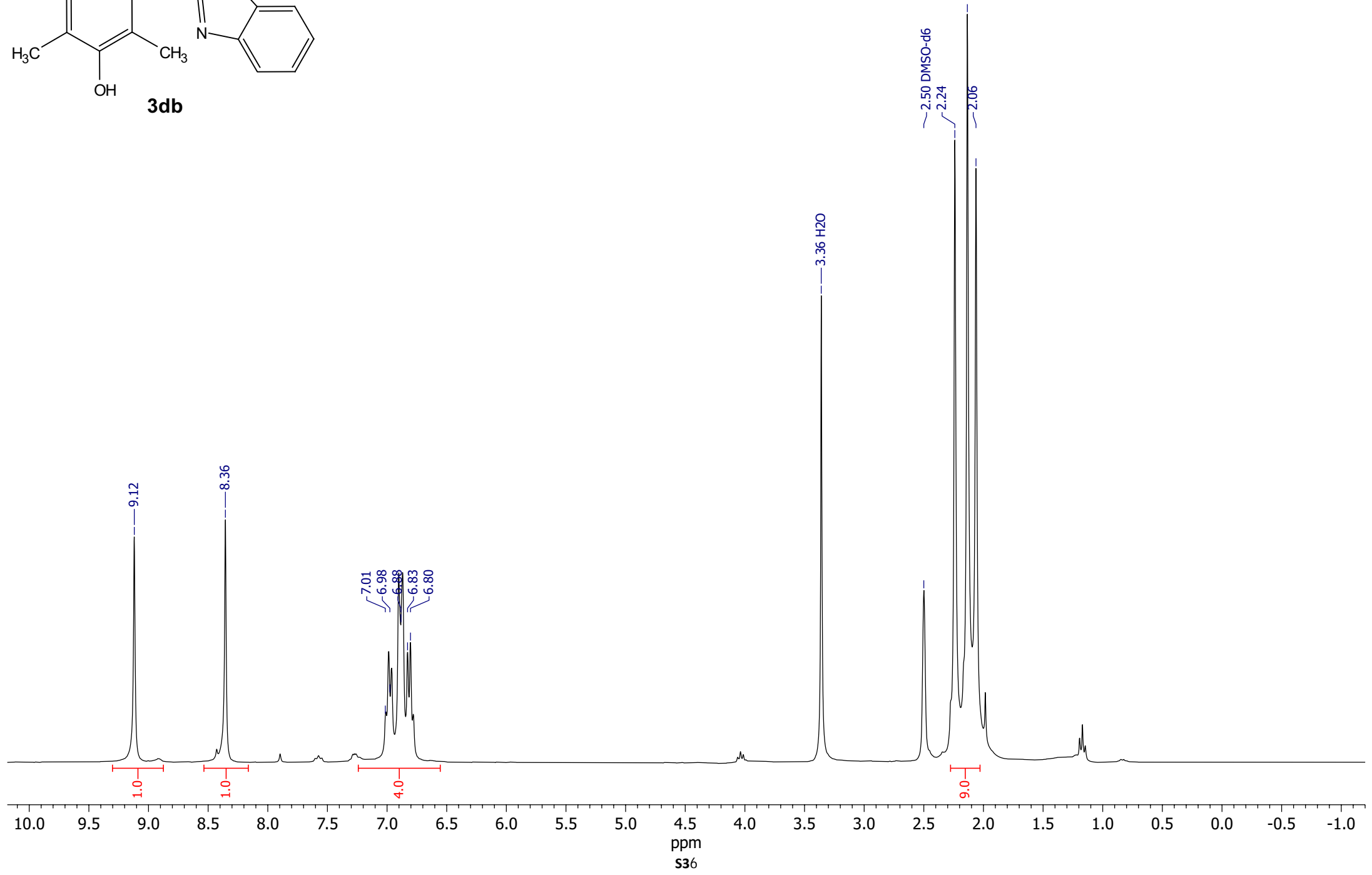
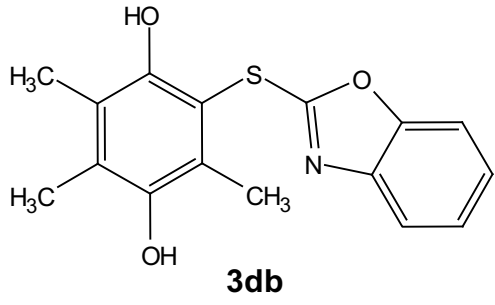
Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

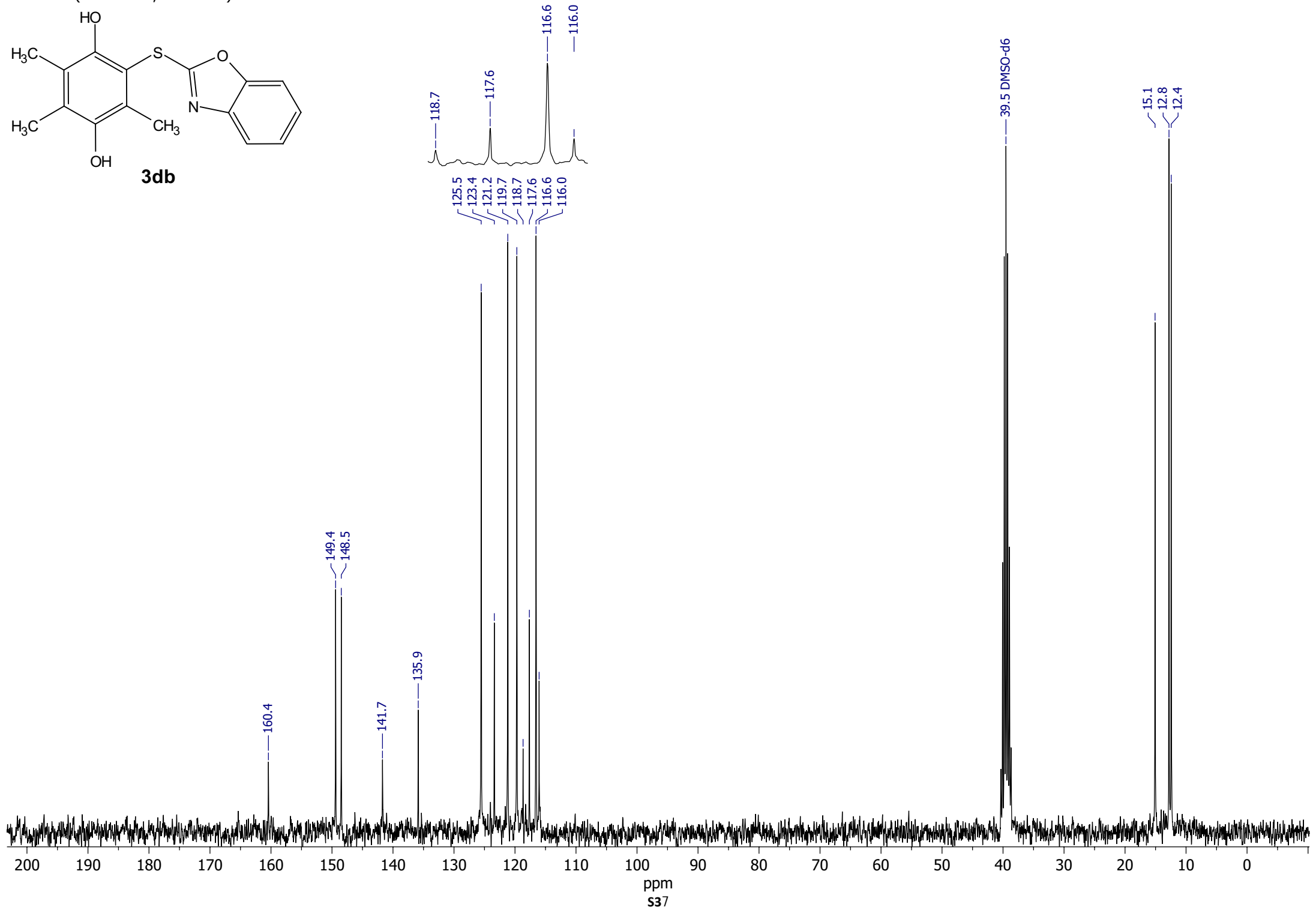
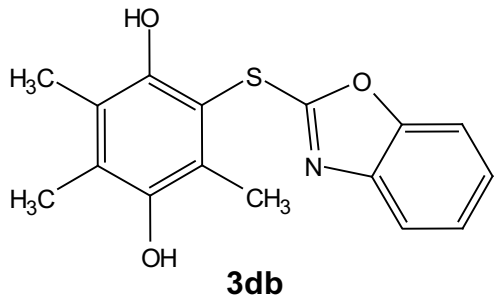
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

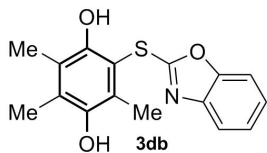


<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)





Chemical Formula: C<sub>16</sub>H<sub>15</sub>NO<sub>3</sub>S  
 Exact Mass: 301.08

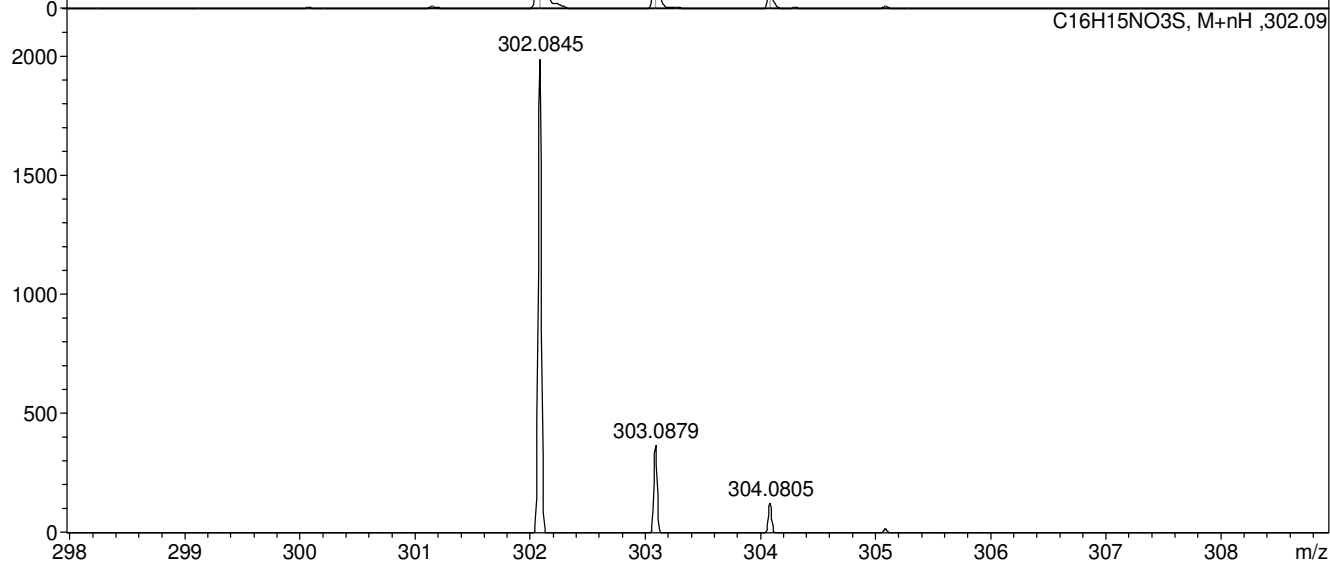
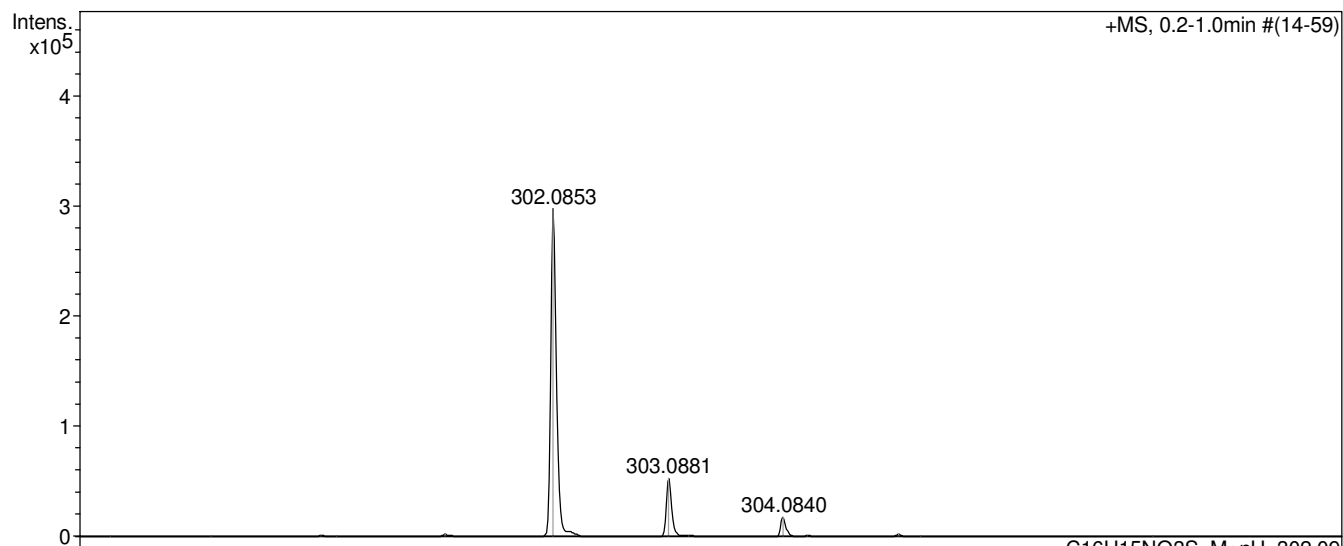
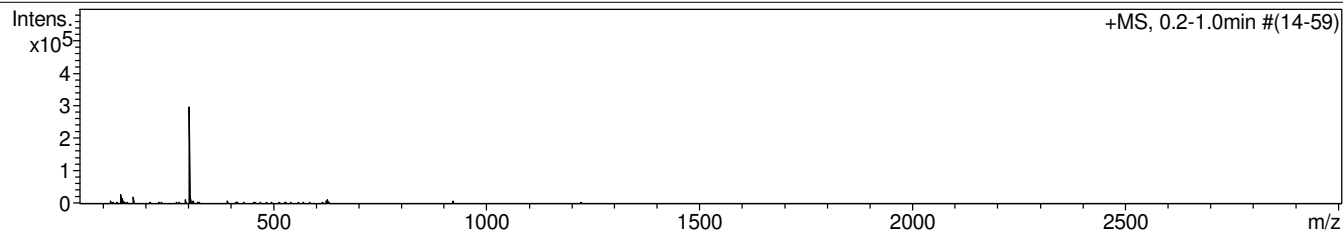
**Analysis Info**

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\0611034.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV374  
 Comment C16H15NO3S mH 302.0845 calibrant added CH3CN

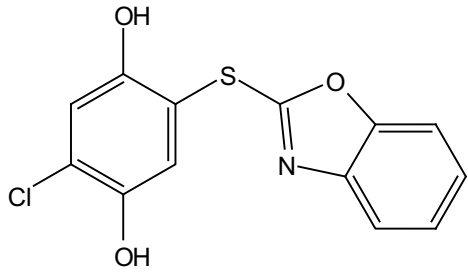
Acquisition Date 11.06.2024 15:28:55  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

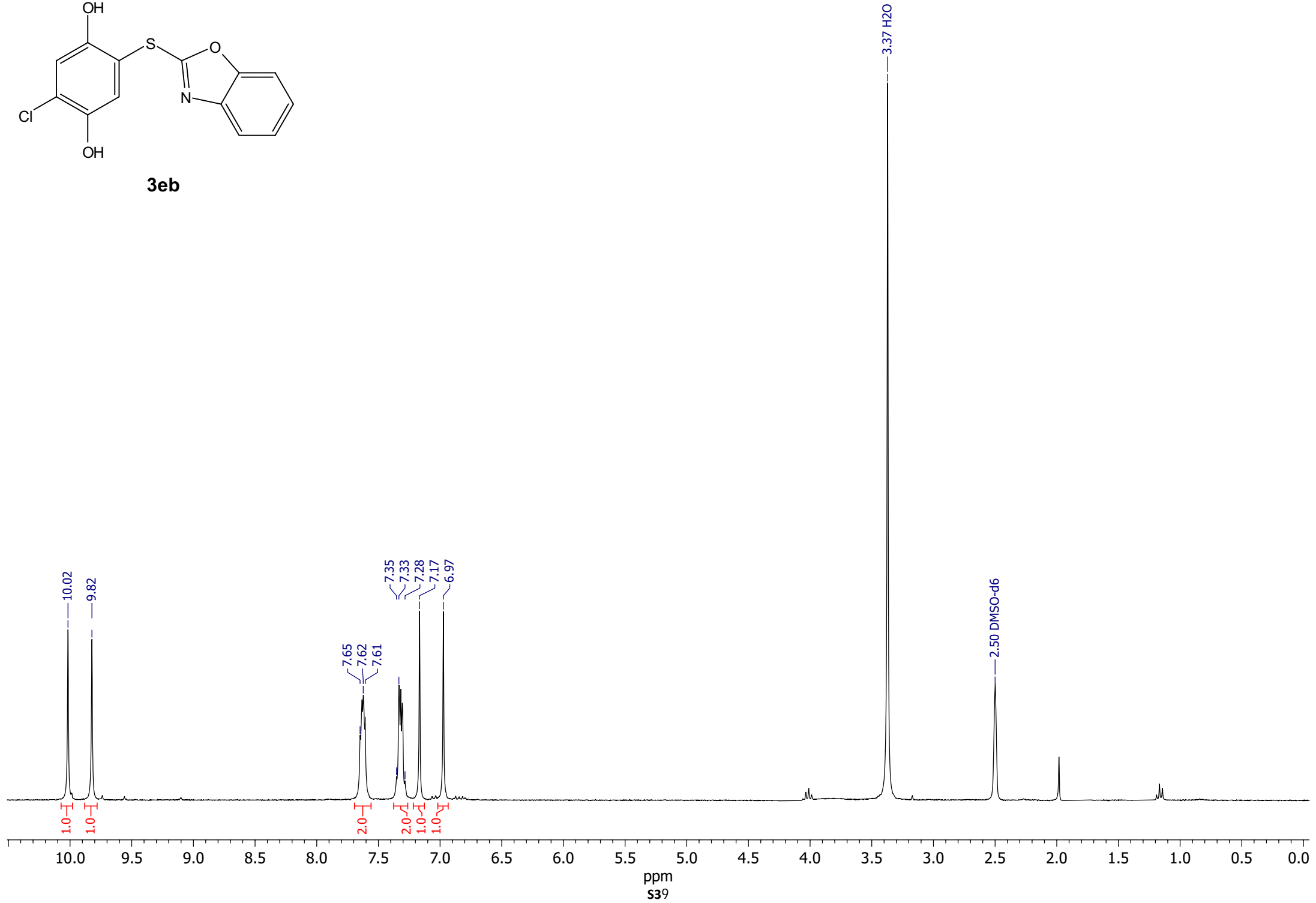
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



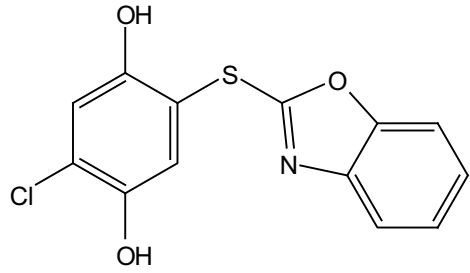
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



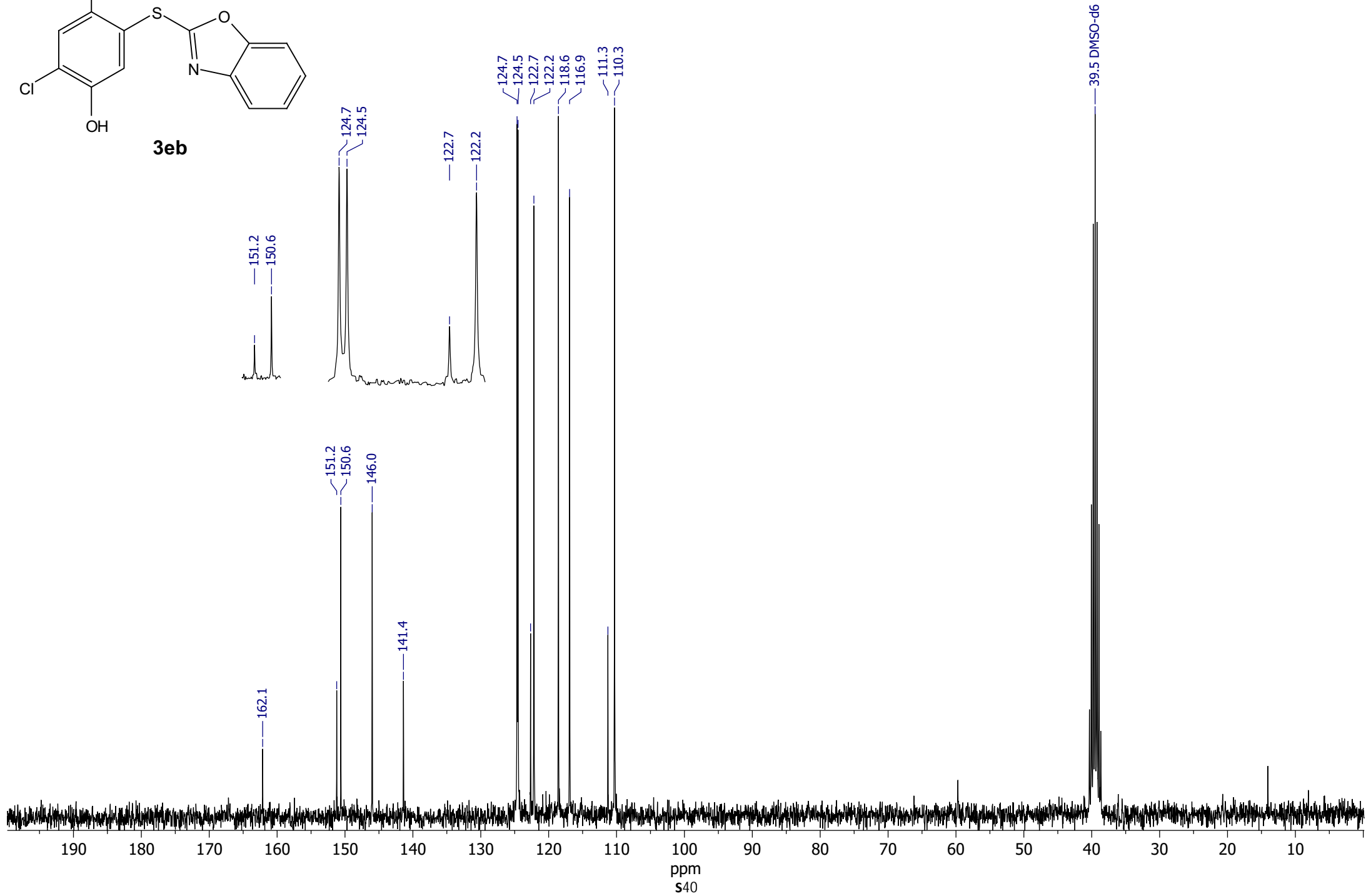
**3eb**



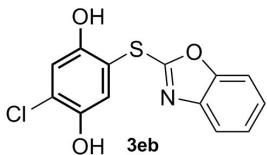
<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)



**3eb**







Chemical Formula: C<sub>13</sub>H<sub>8</sub>ClNO<sub>3</sub>S  
 Exact Mass: 292,99

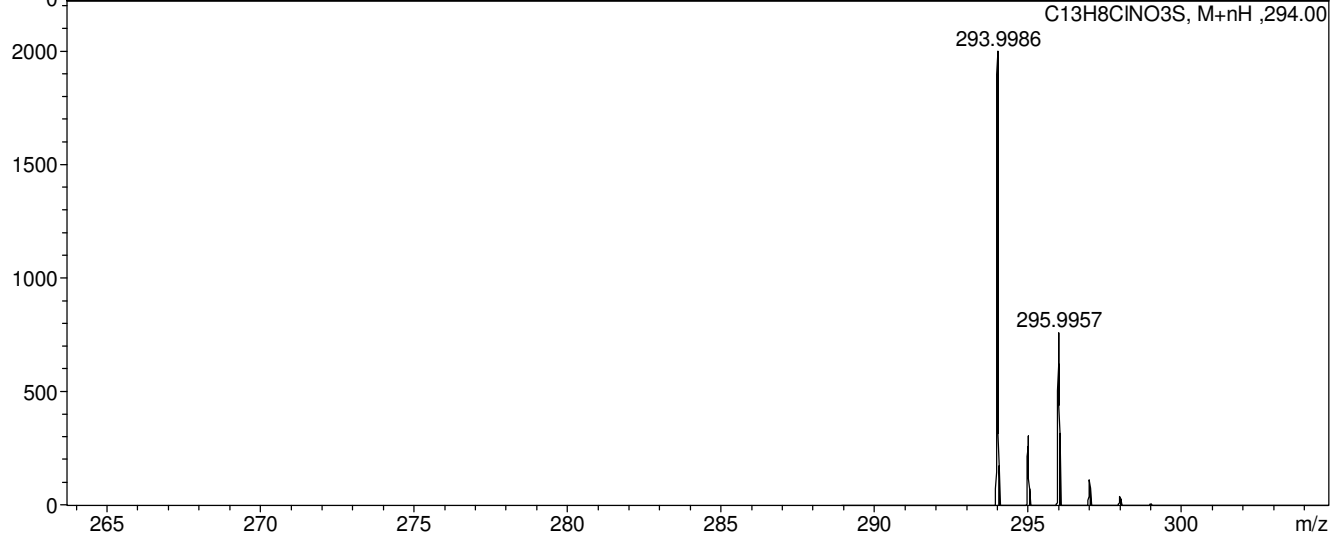
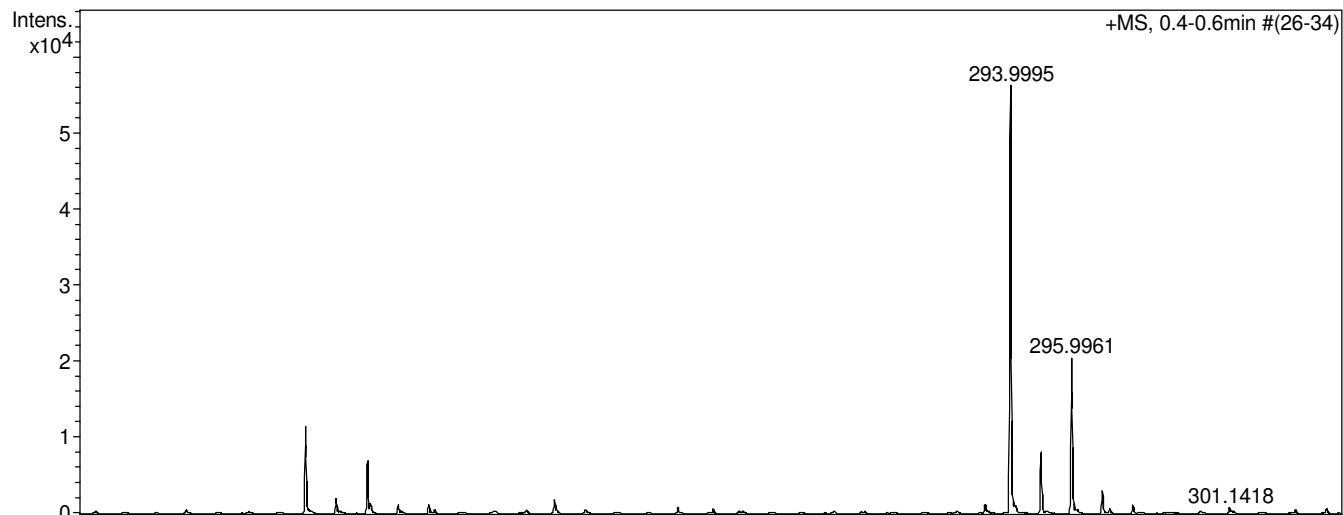
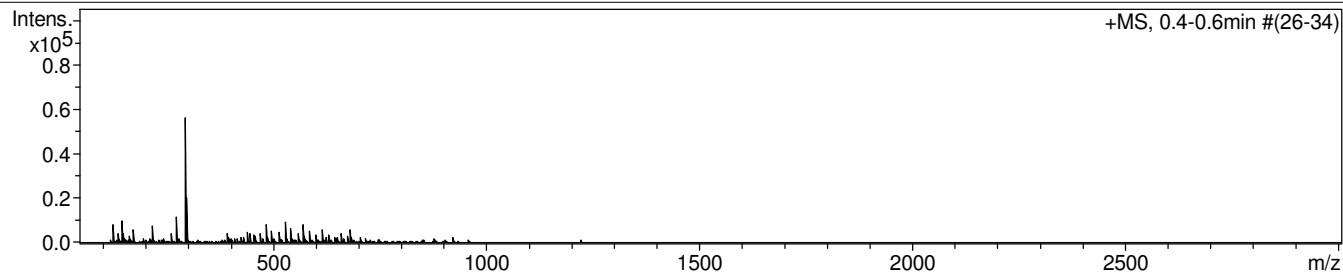
**Analysis Info**

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\0716008.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV392  
 Comment C13H8ClNO3S mH293.9986 calibrant added CH3CN

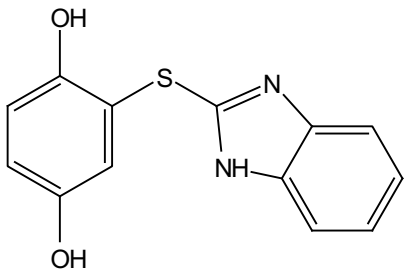
Acquisition Date 16.07.2024 10:55:54  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

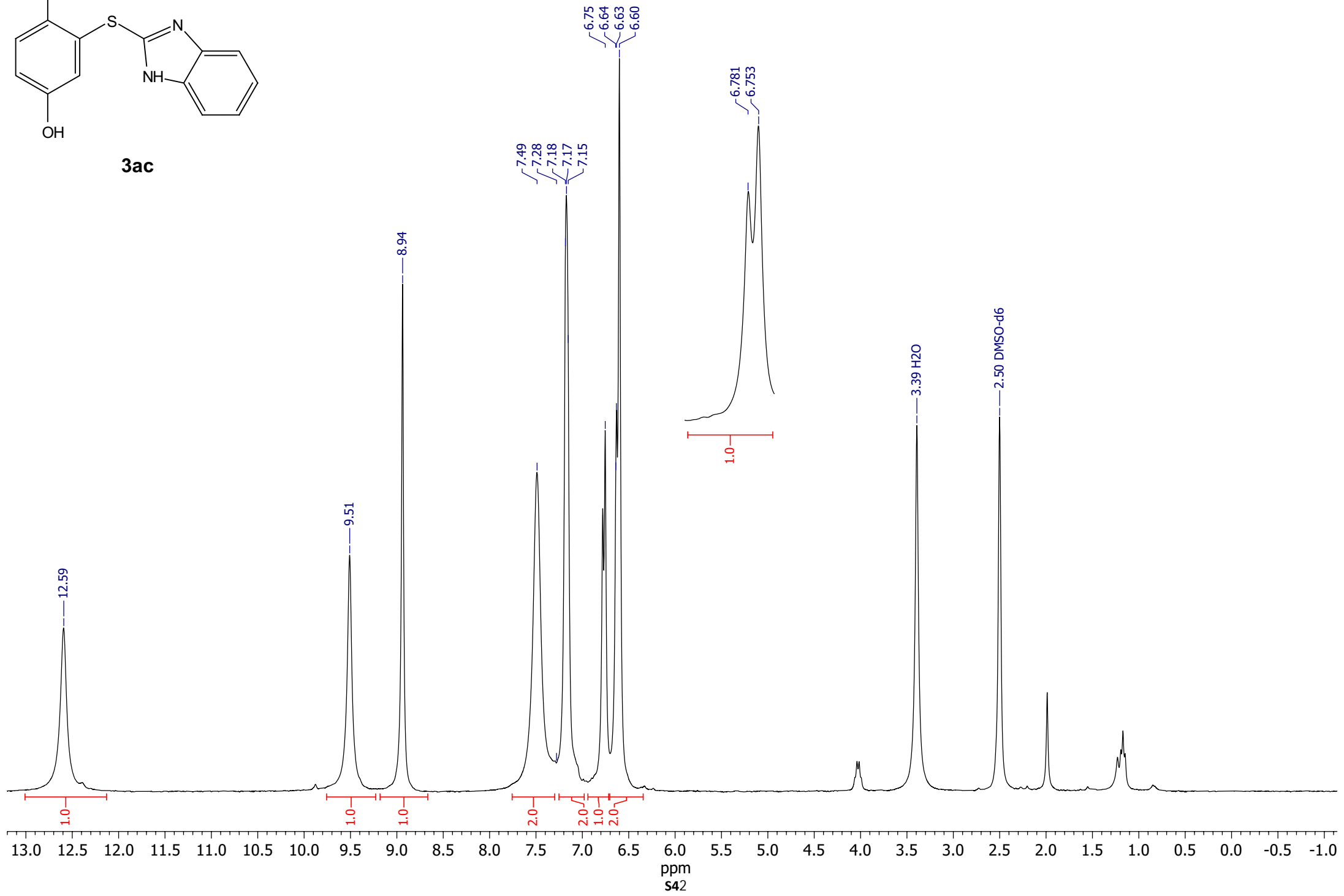
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



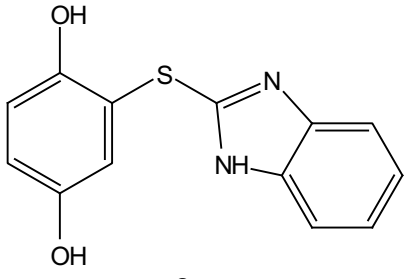
1H NMR (300.13 MHz, DMSO-d6)



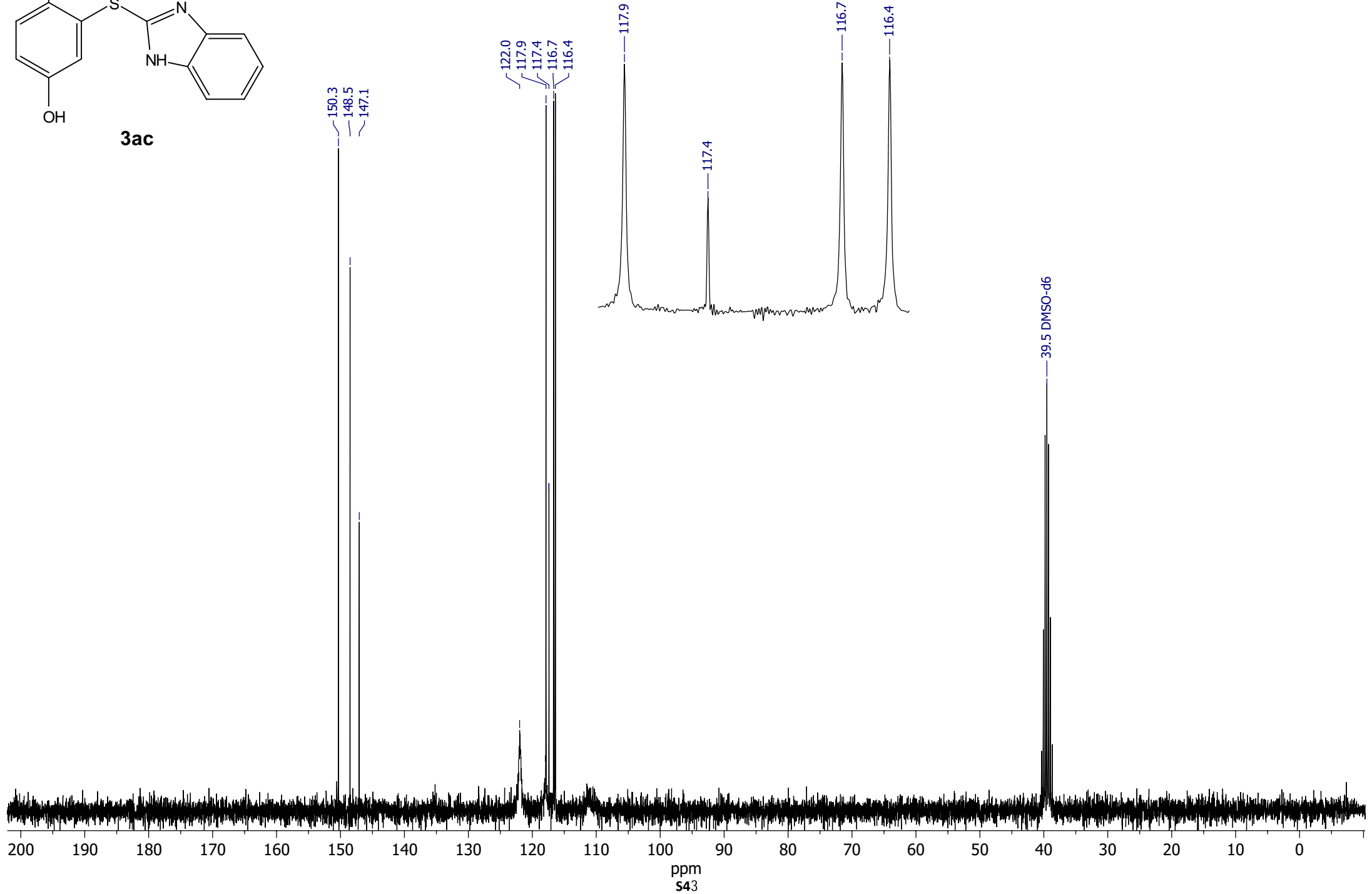
**3ac**

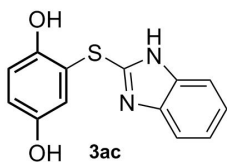


<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)



**3ac**





Chemical Formula: C<sub>13</sub>H<sub>10</sub>N<sub>2</sub>O<sub>2</sub>S  
 Exact Mass: 258,05

**Analysis Info**

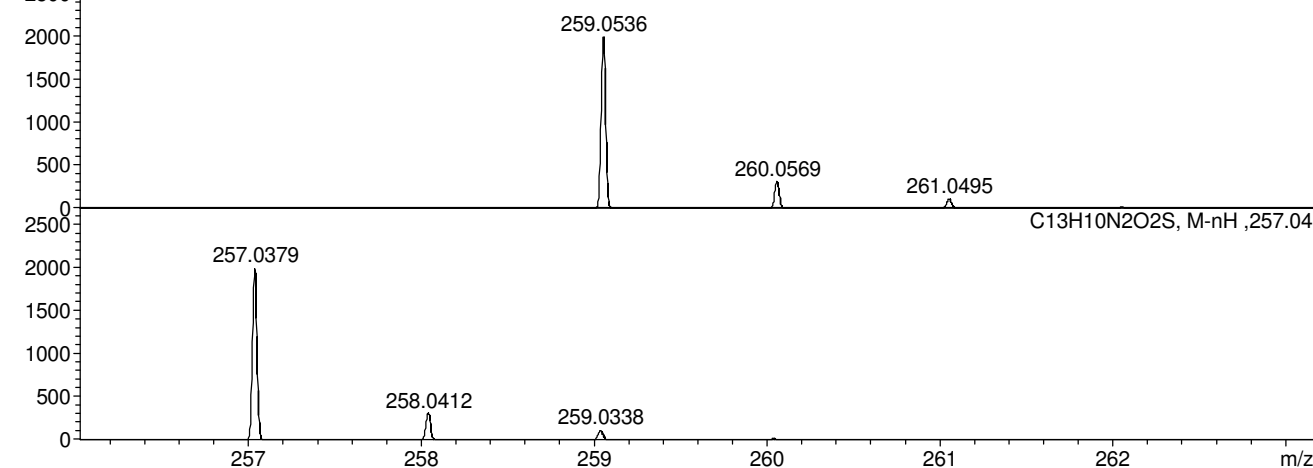
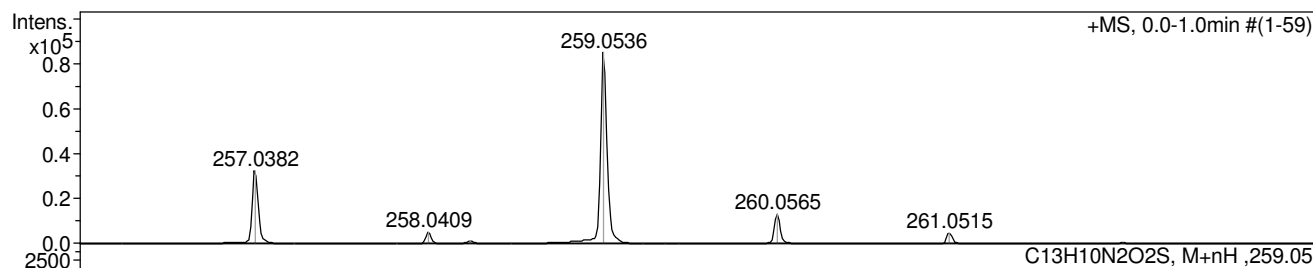
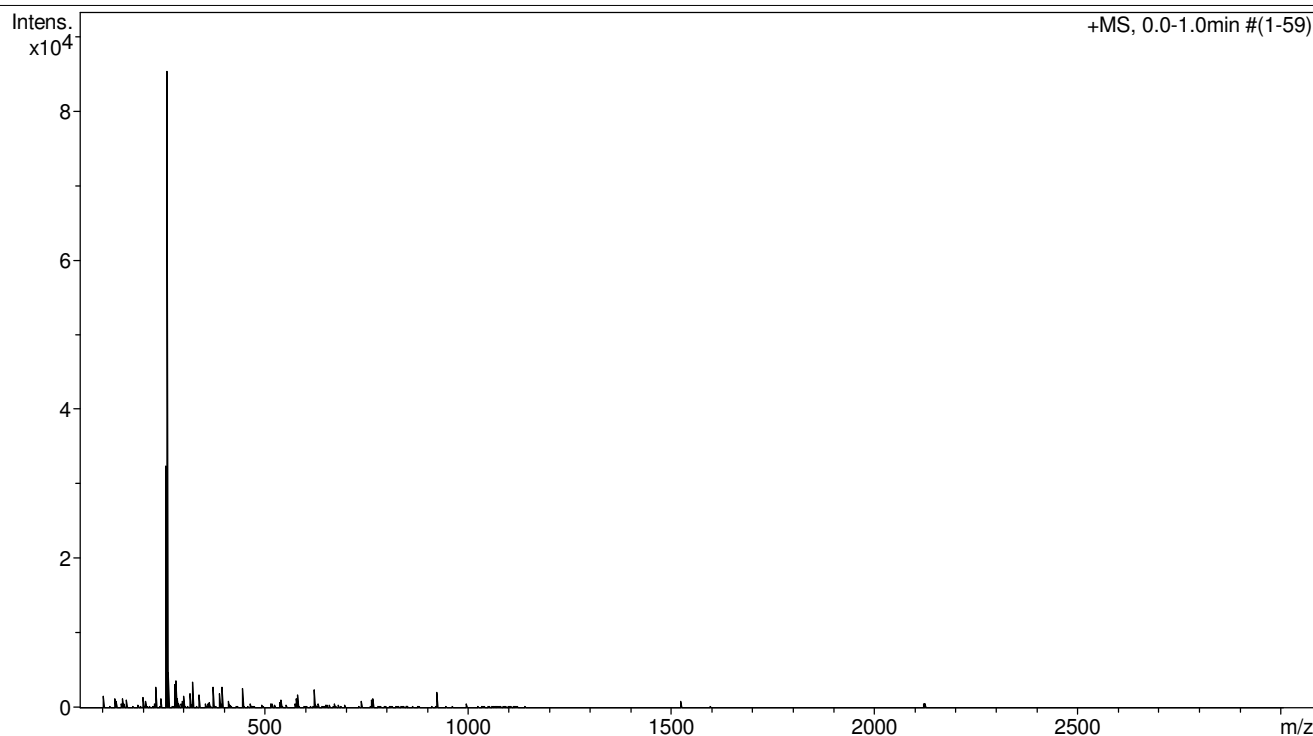
Analysis Name D:\Data\Chizhov\Egorov\Moiseeva\mnv367\_&clblow.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV367  
 Comment CH3CN 100 %, dil. 200, calibrant added

Acquisition Date 17.04.2024 17:06:51

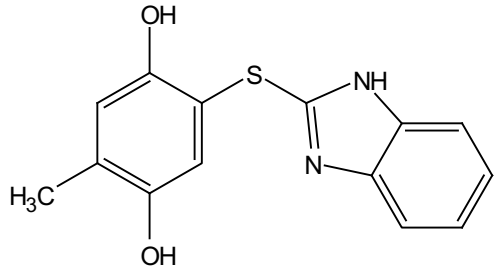
Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

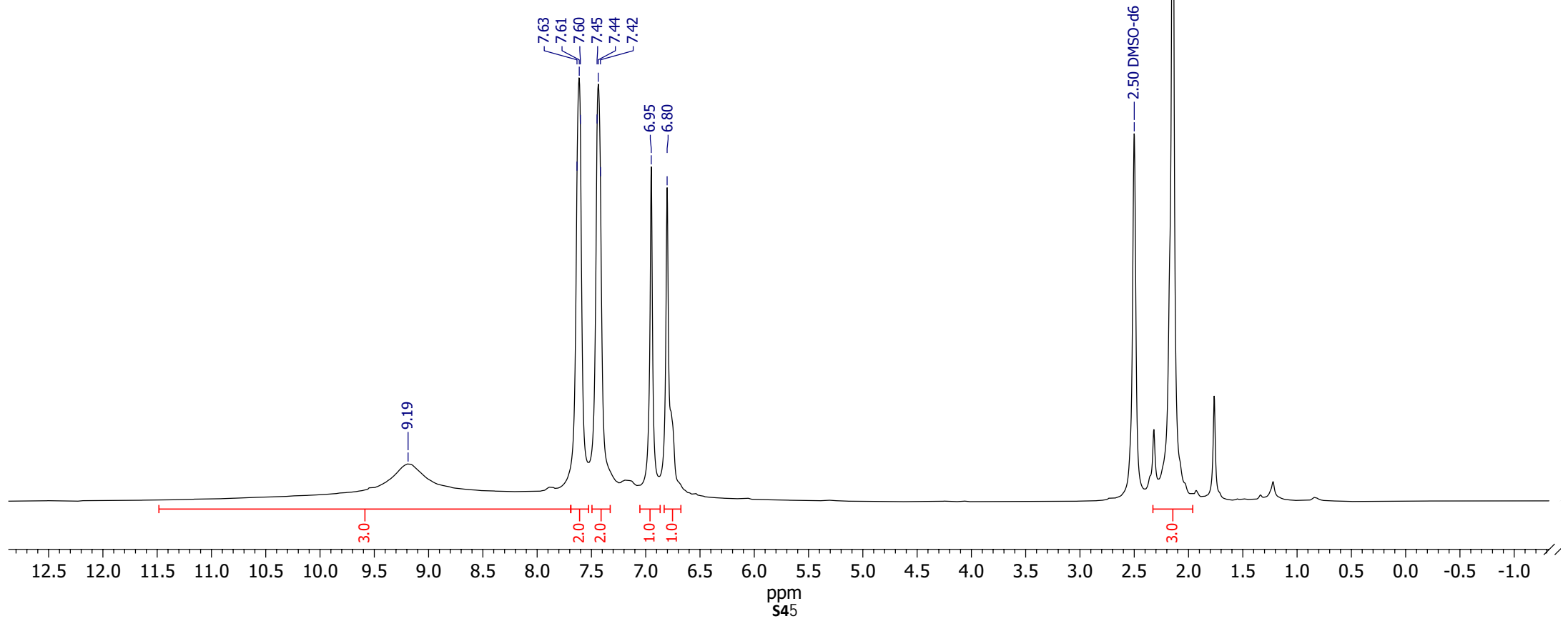
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



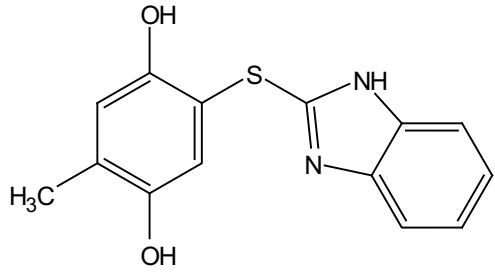
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



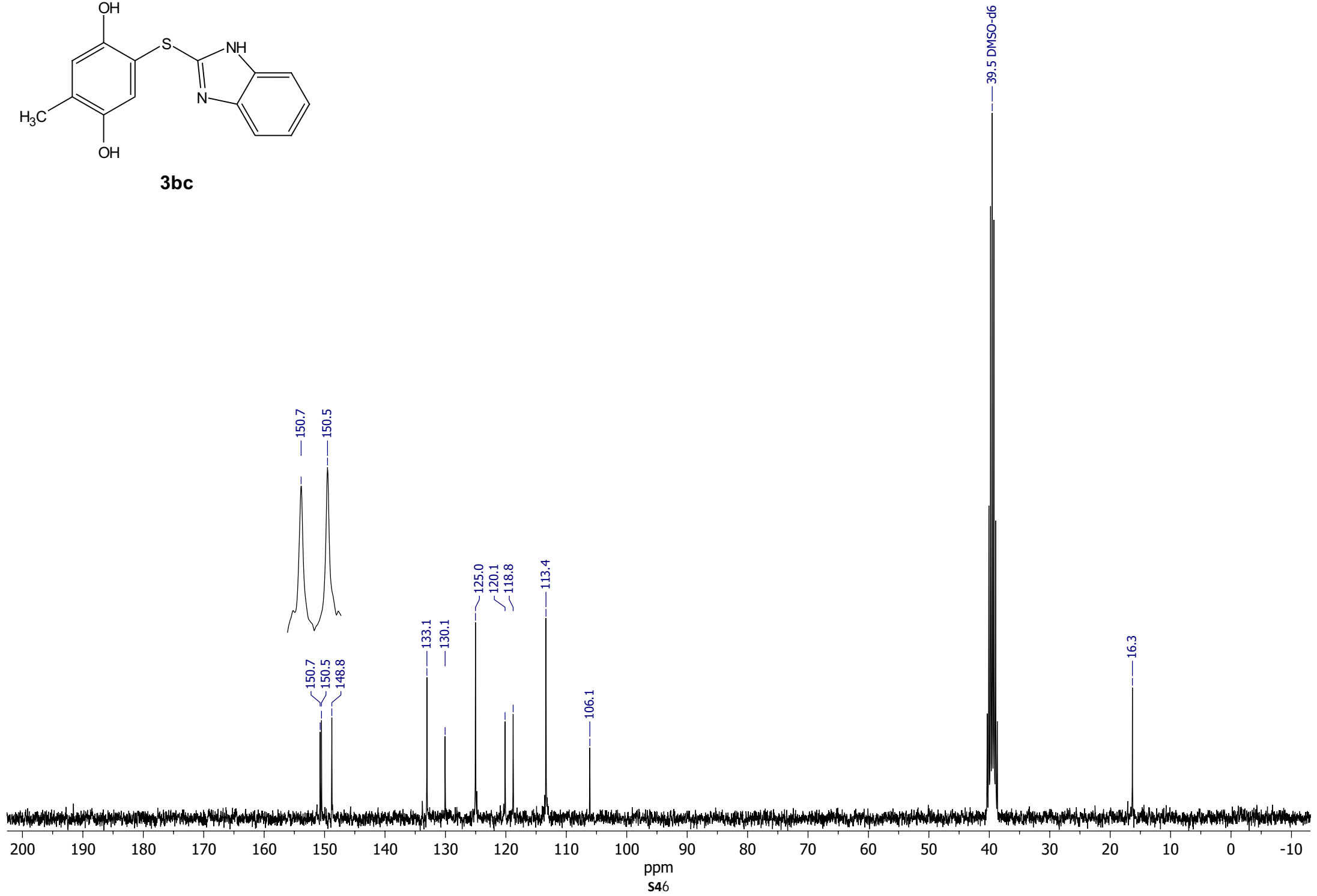
**3bc**

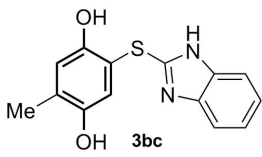


<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)



**3bc**





Chemical Formula: C<sub>14</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>S  
 Exact Mass: 272,06

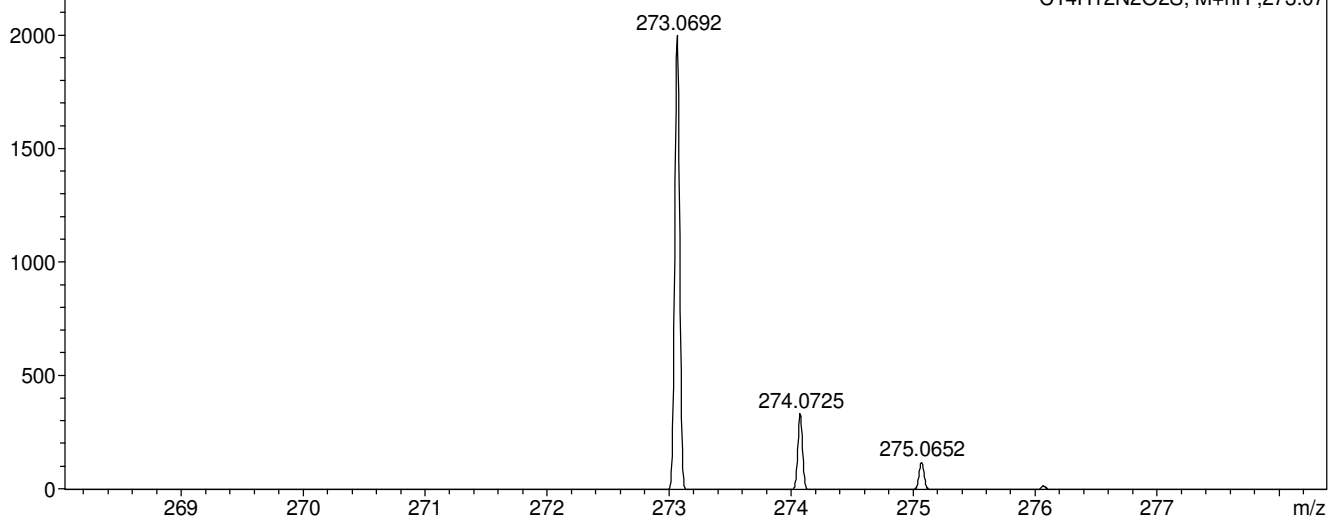
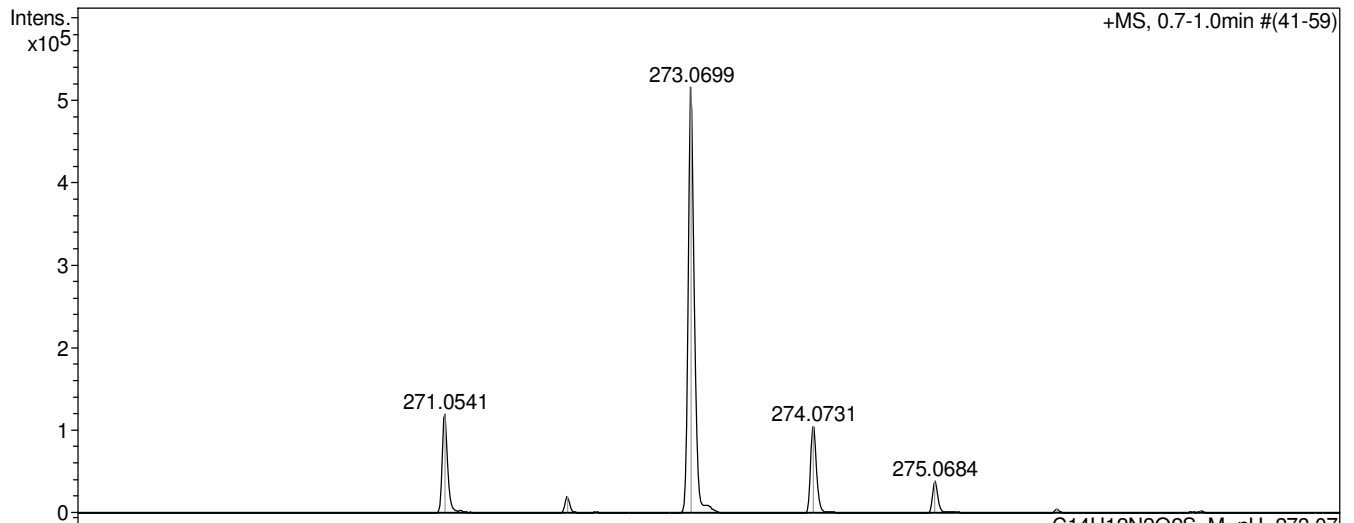
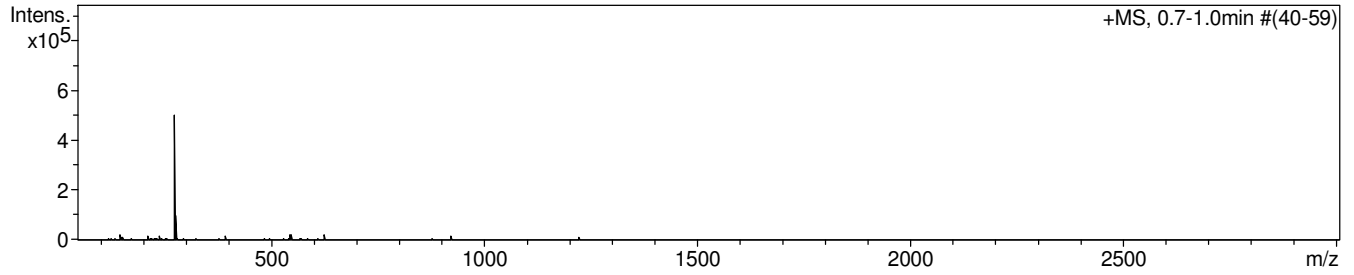
**Analysis Info**

Analysis Name D:\Data\Kolotyrykina\2024\Moiseeva\0716007.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV364  
 Comment C14H12N2O2S mH262.9909 calibrant added CH3CN

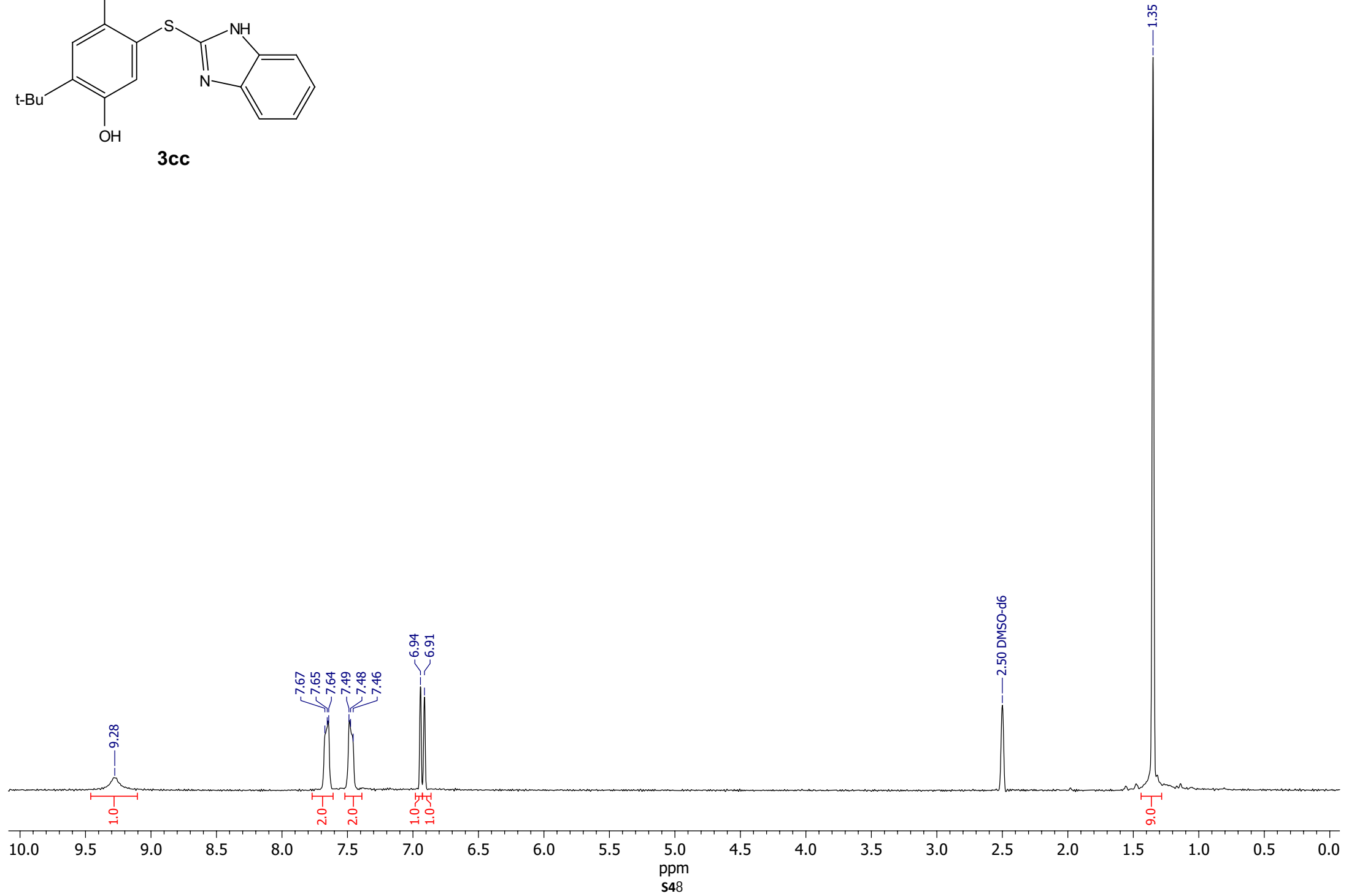
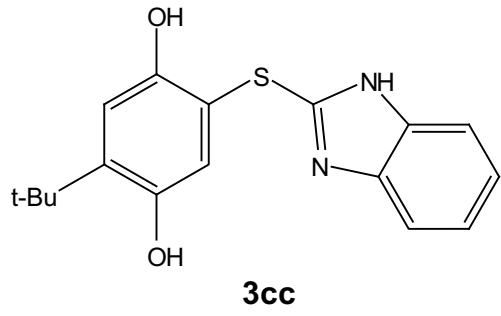
Acquisition Date 16.07.2024 10:46:36  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

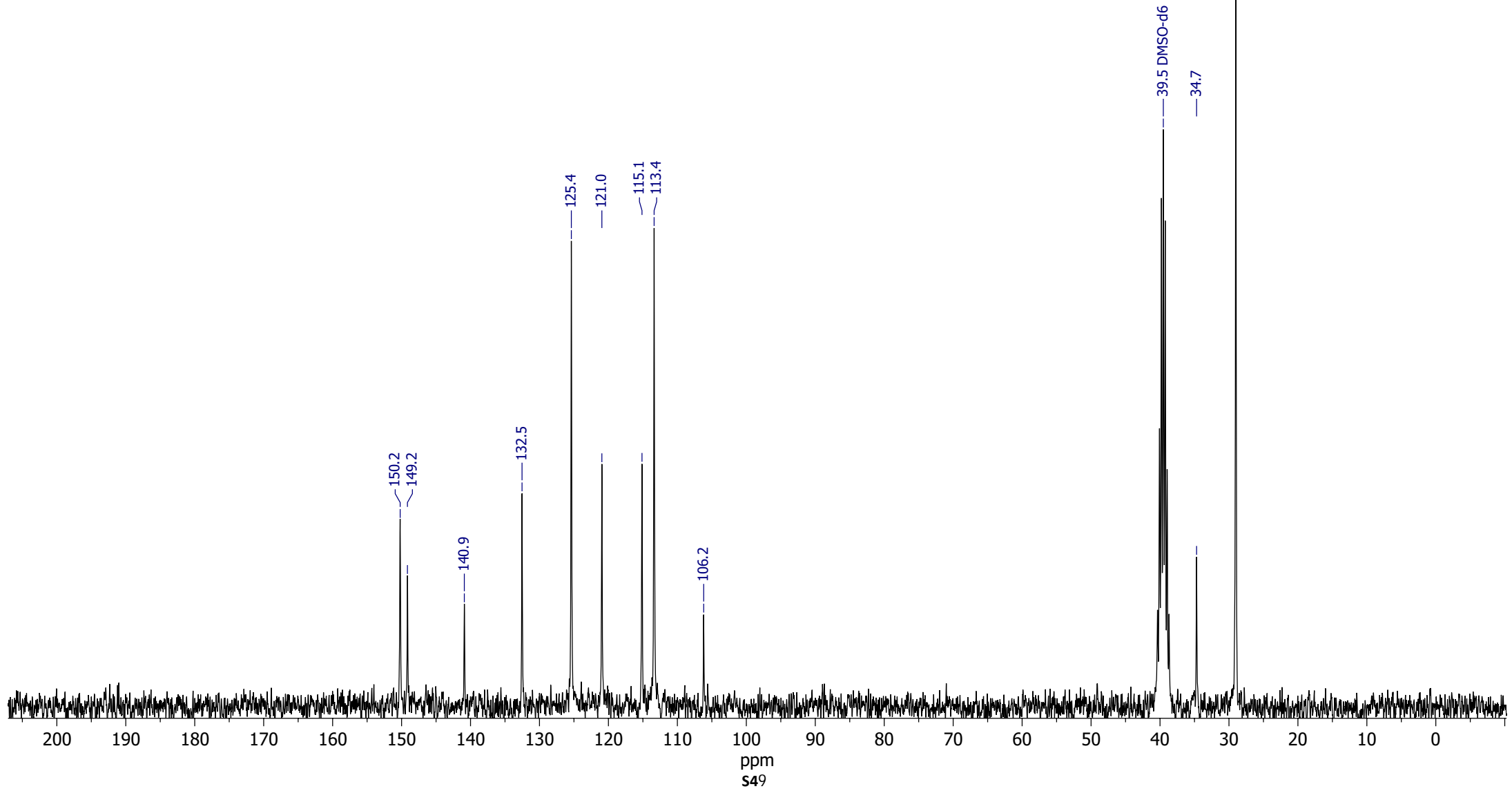
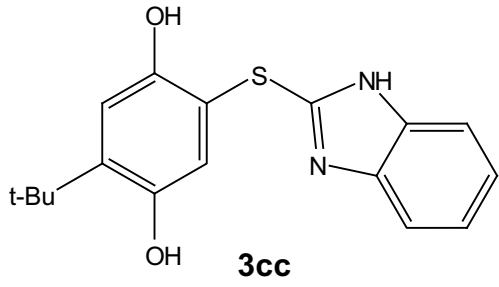


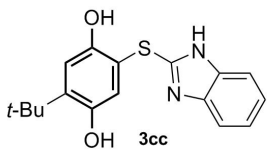
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)





<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)





Chemical Formula: C<sub>17</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>S  
Exact Mass: 314,11

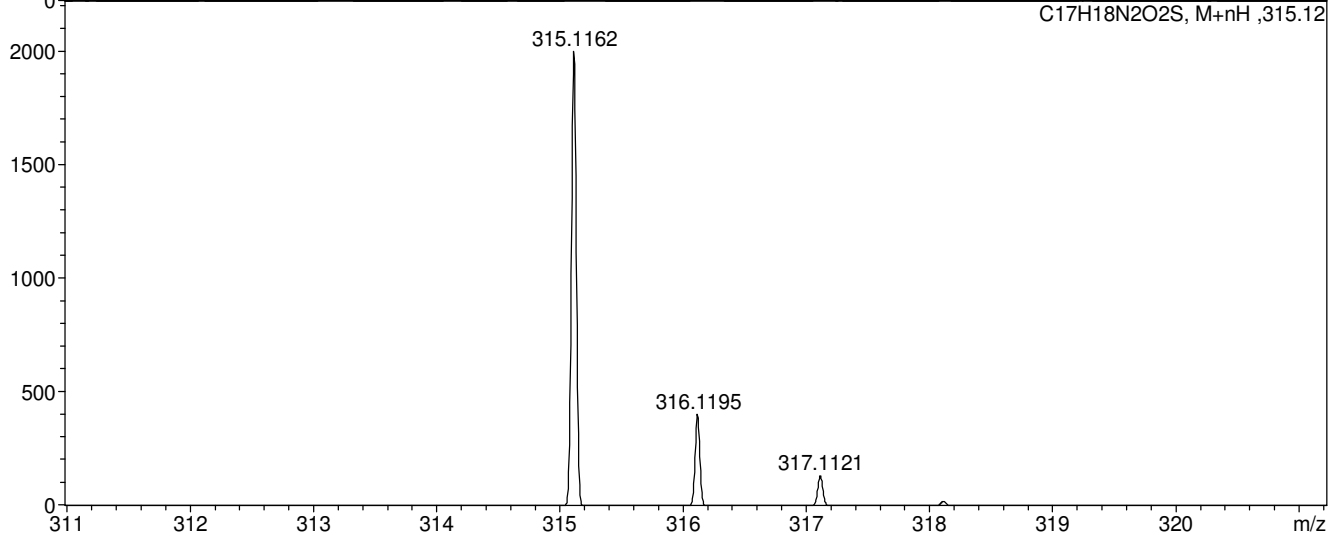
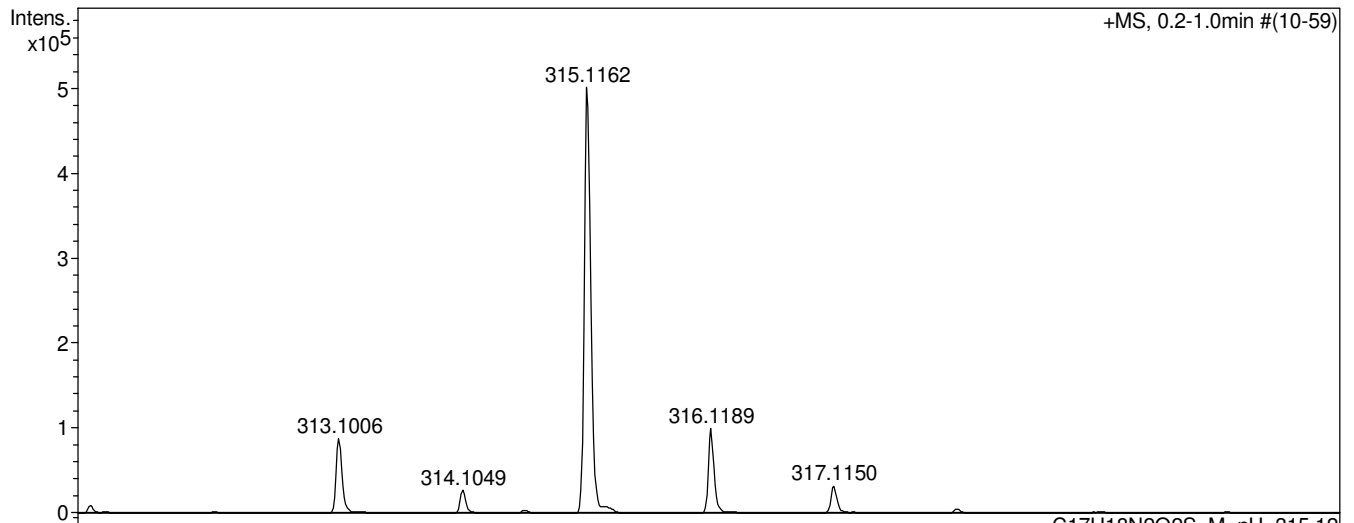
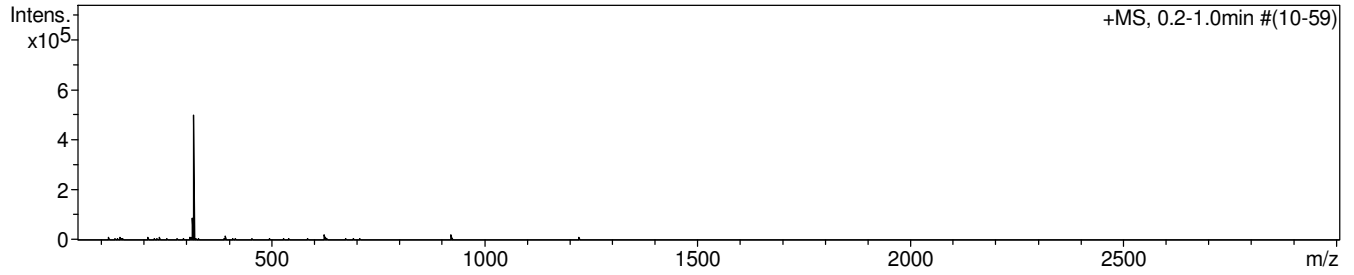
**Analysis Info**

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\0716004.d  
Method tune\_low.m  
Sample Name /VAPP MNV370  
Comment C17H18N2O2S mH315.1161 calibrant added CH3CN

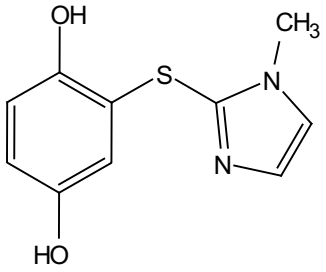
Acquisition Date 16.07.2024 10:30:11  
Operator BDAL@DE  
Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

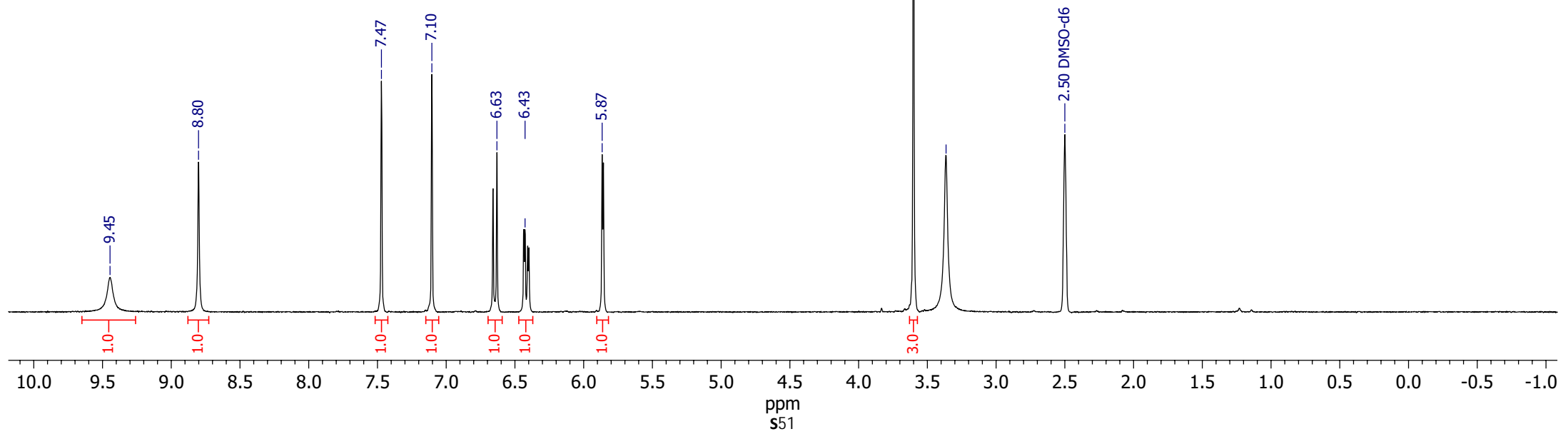
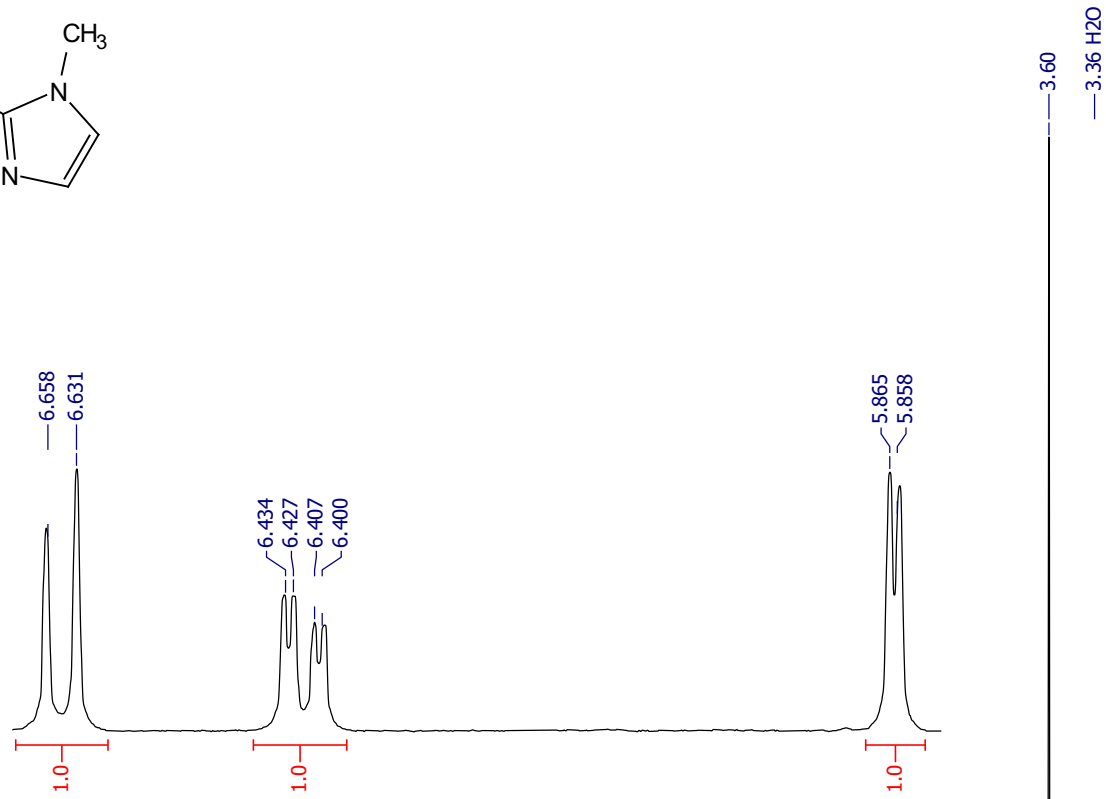
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



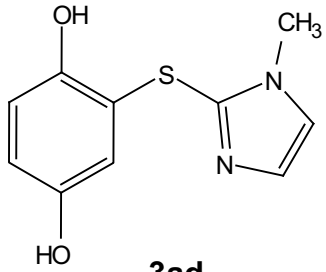
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



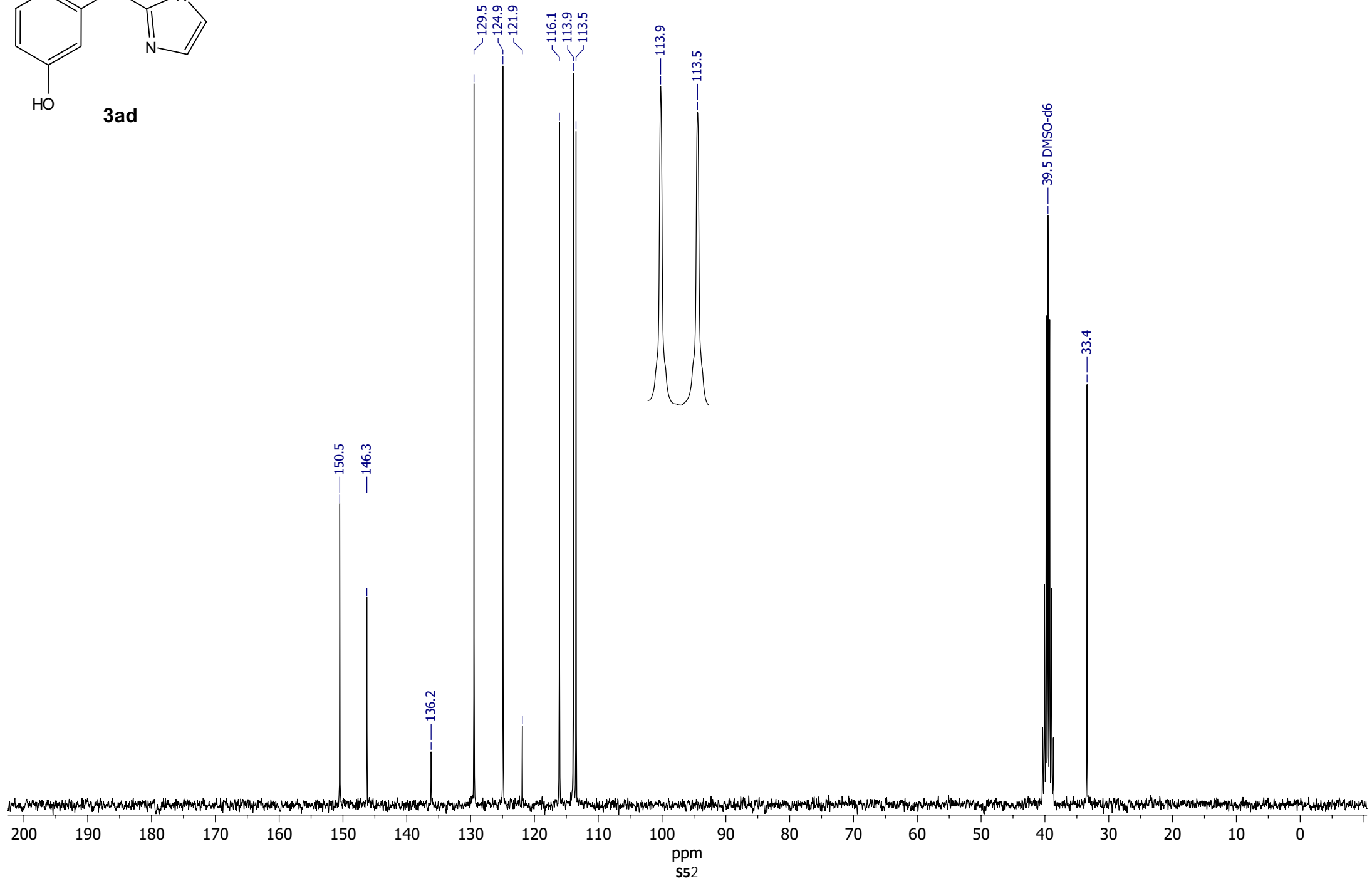
**3ad**

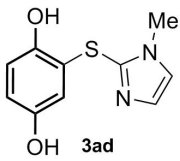


<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)



**3ad**





Chemical Formula: C<sub>10</sub>H<sub>10</sub>N<sub>2</sub>O<sub>2</sub>S  
 Exact Mass: 222,05

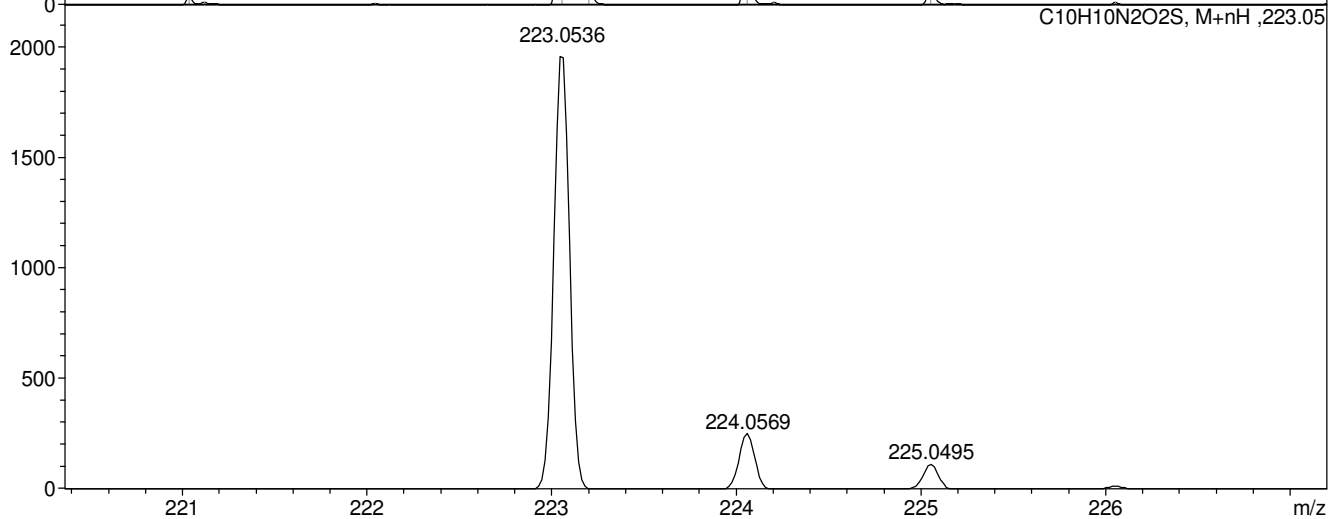
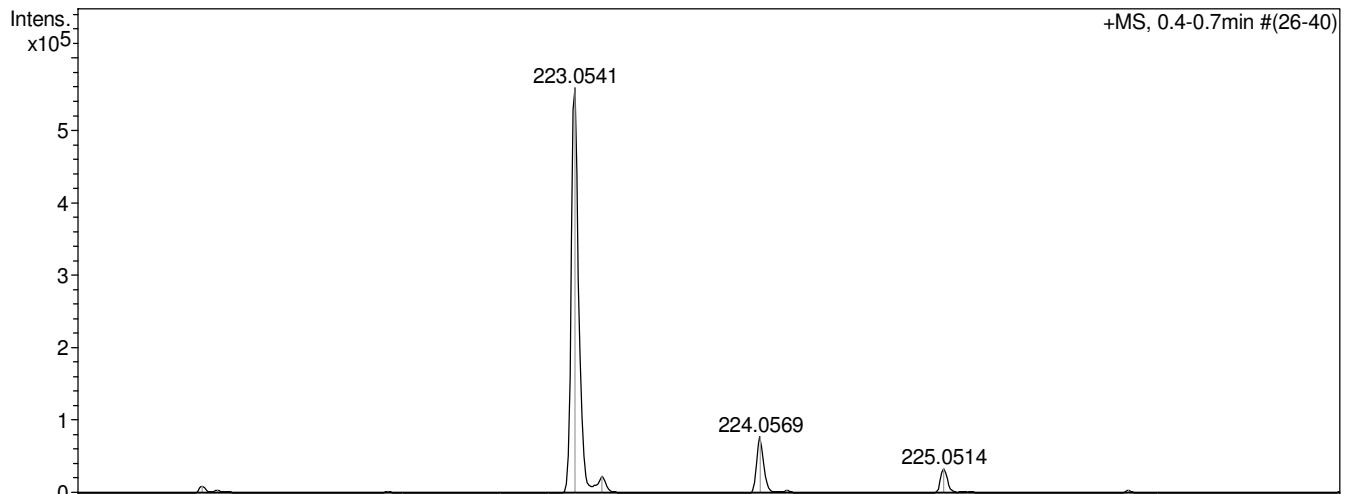
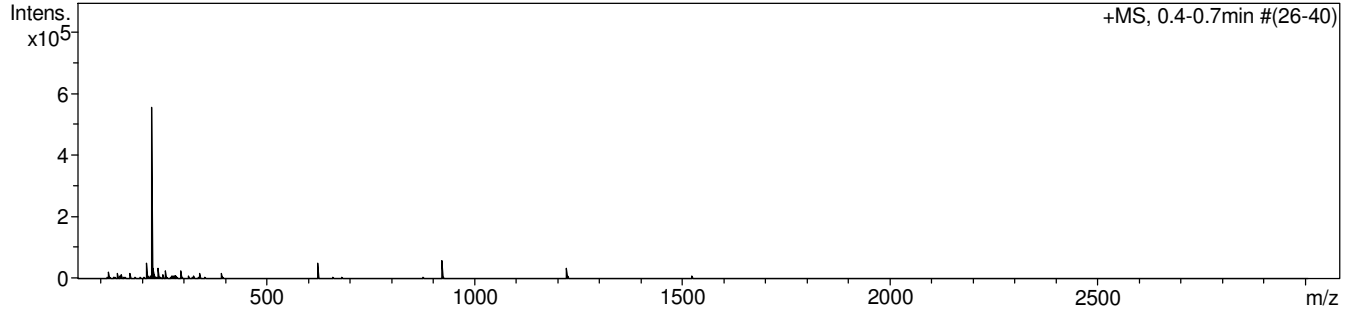
**Analysis Info**

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\0515008.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV354  
 Comment C10H10N2O2S mH223.0535 calibrant added CH3CN

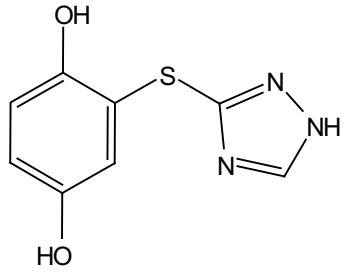
Acquisition Date 15.05.2024 10:29:04  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

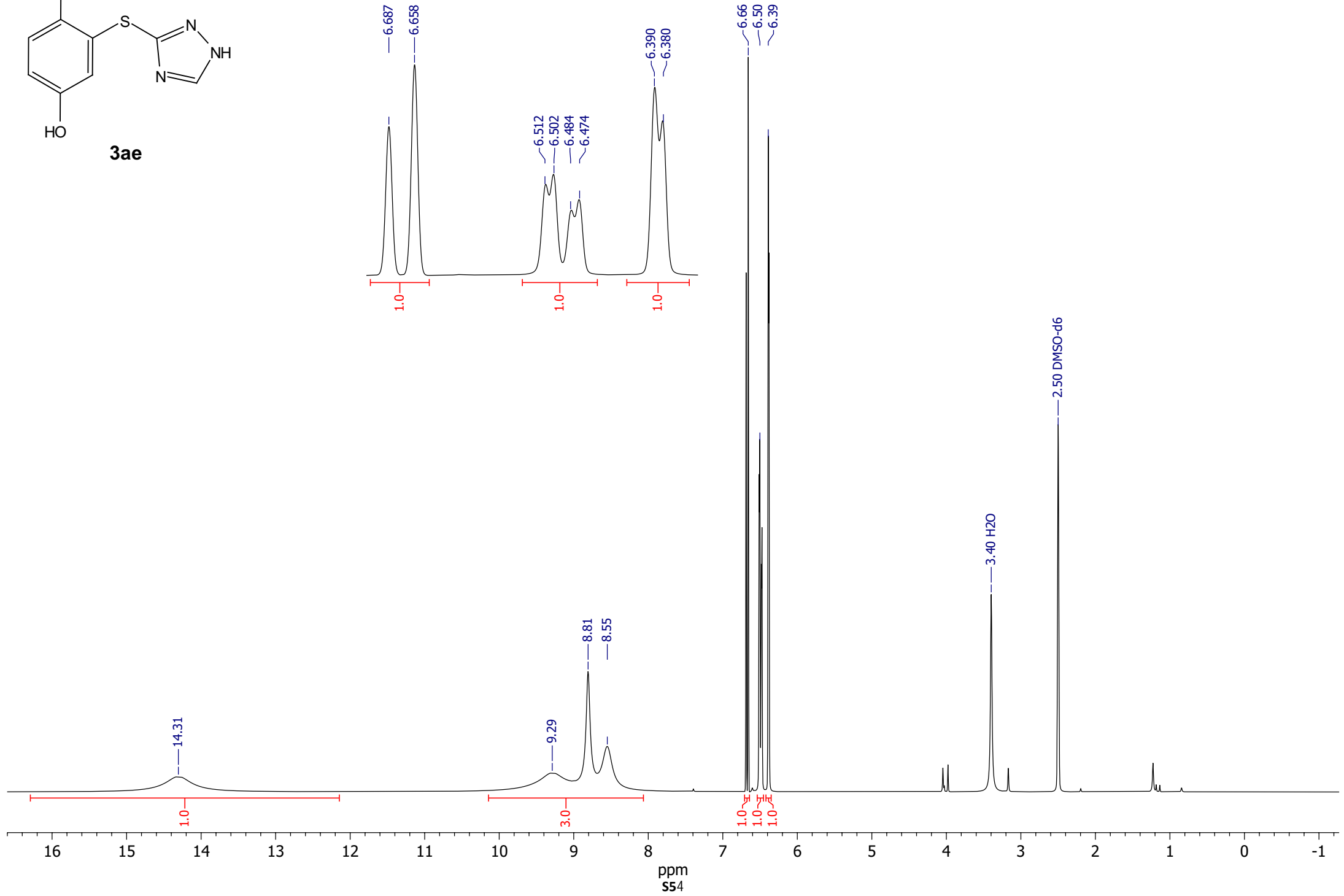
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



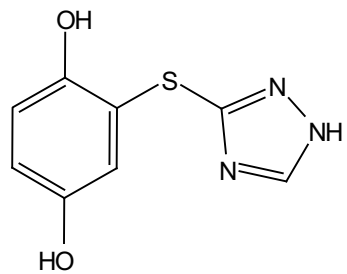
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



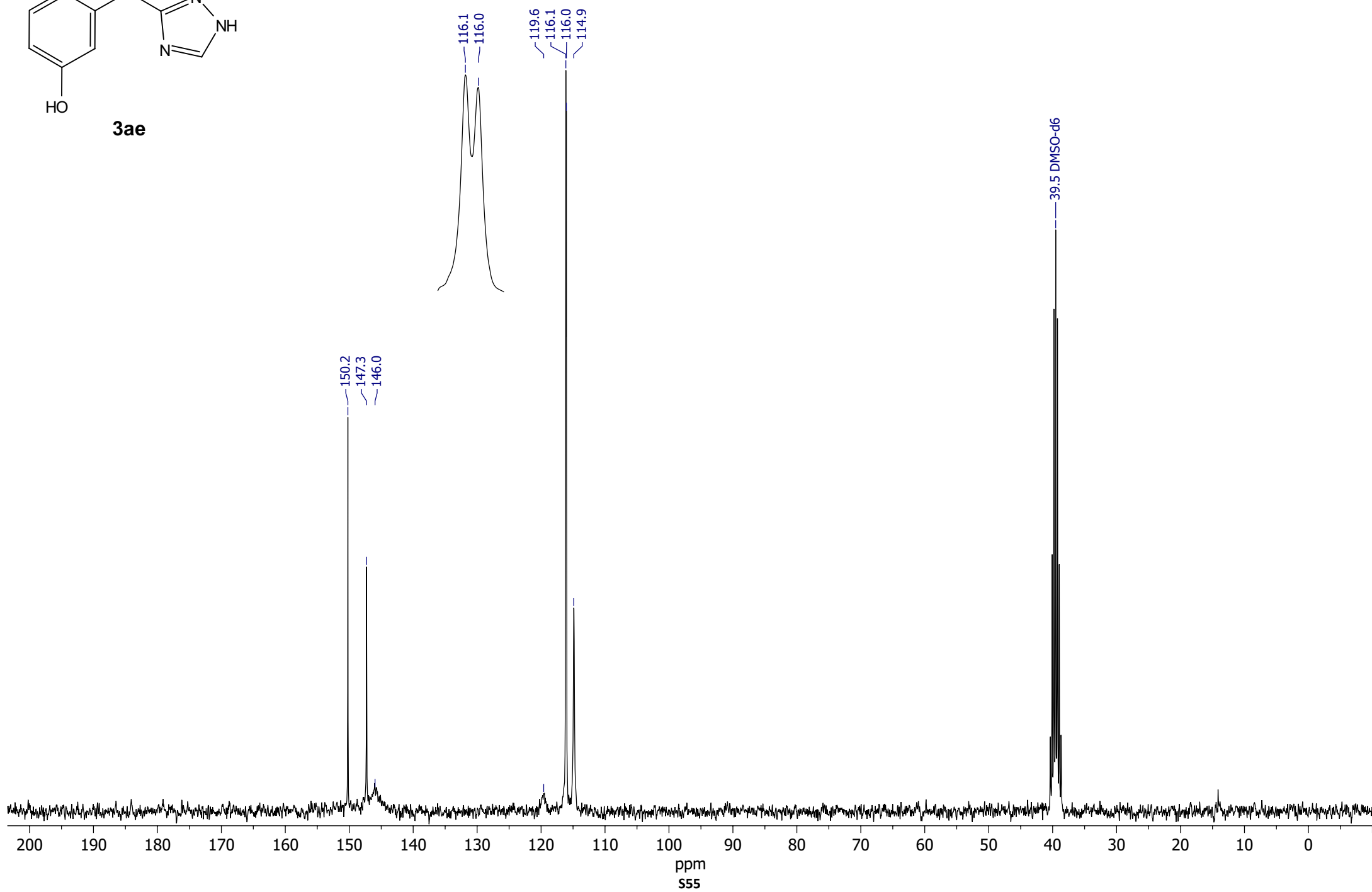
**3ae**

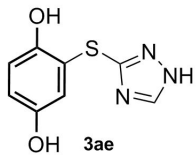


<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)



**3ae**





Chemical Formula: C<sub>8</sub>H<sub>7</sub>N<sub>3</sub>O<sub>2</sub>S  
 Exact Mass: 209,03

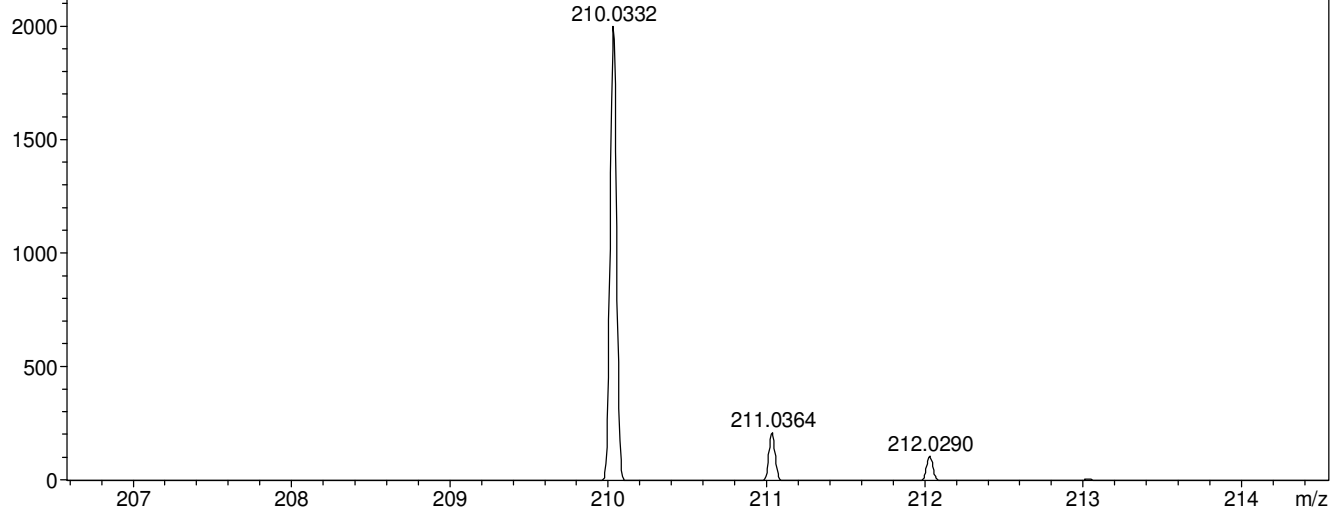
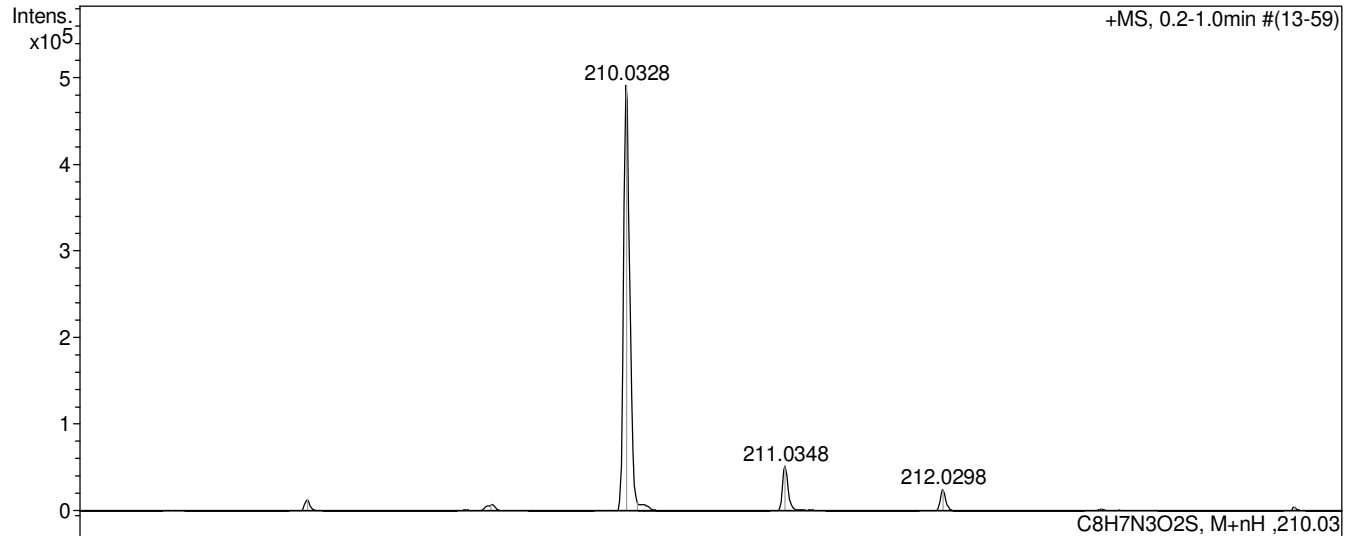
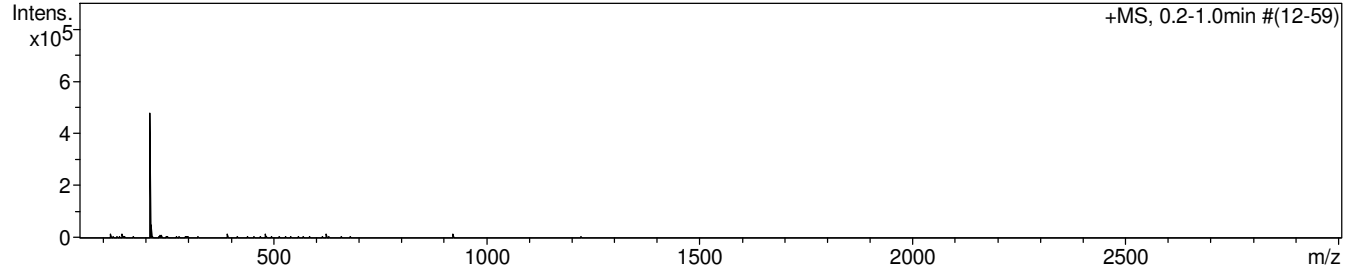
**Analysis Info**

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\0716010.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV368  
 Comment C8H7N3O2S mH210.0331 calibrant added CH3CN

Acquisition Date 16.07.2024 11:10:48  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

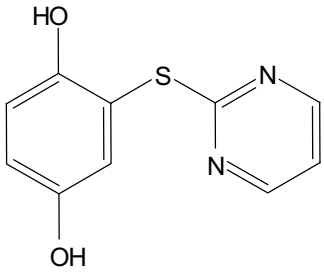
**Acquisition Parameter**

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

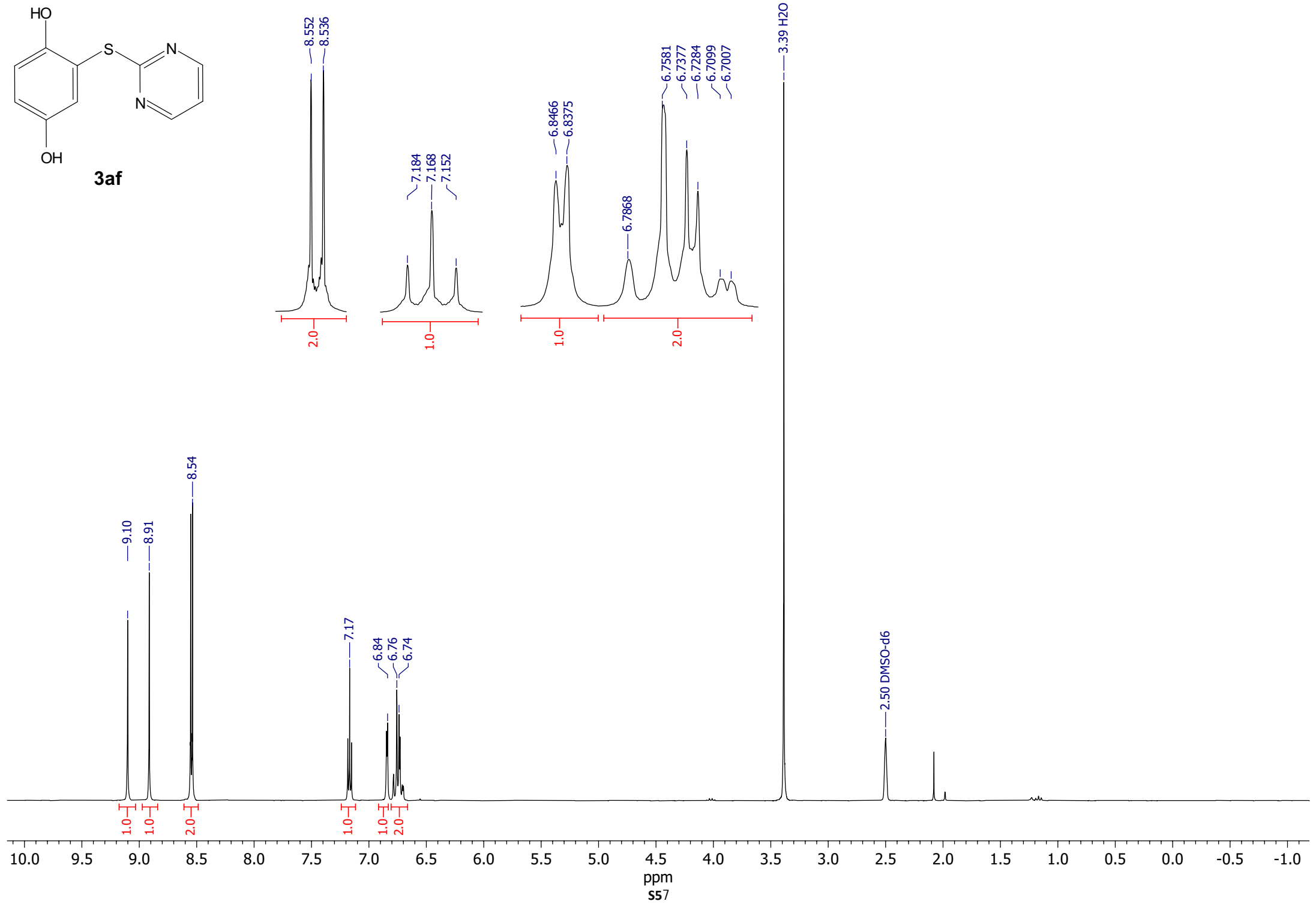




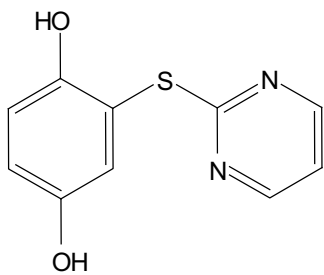
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



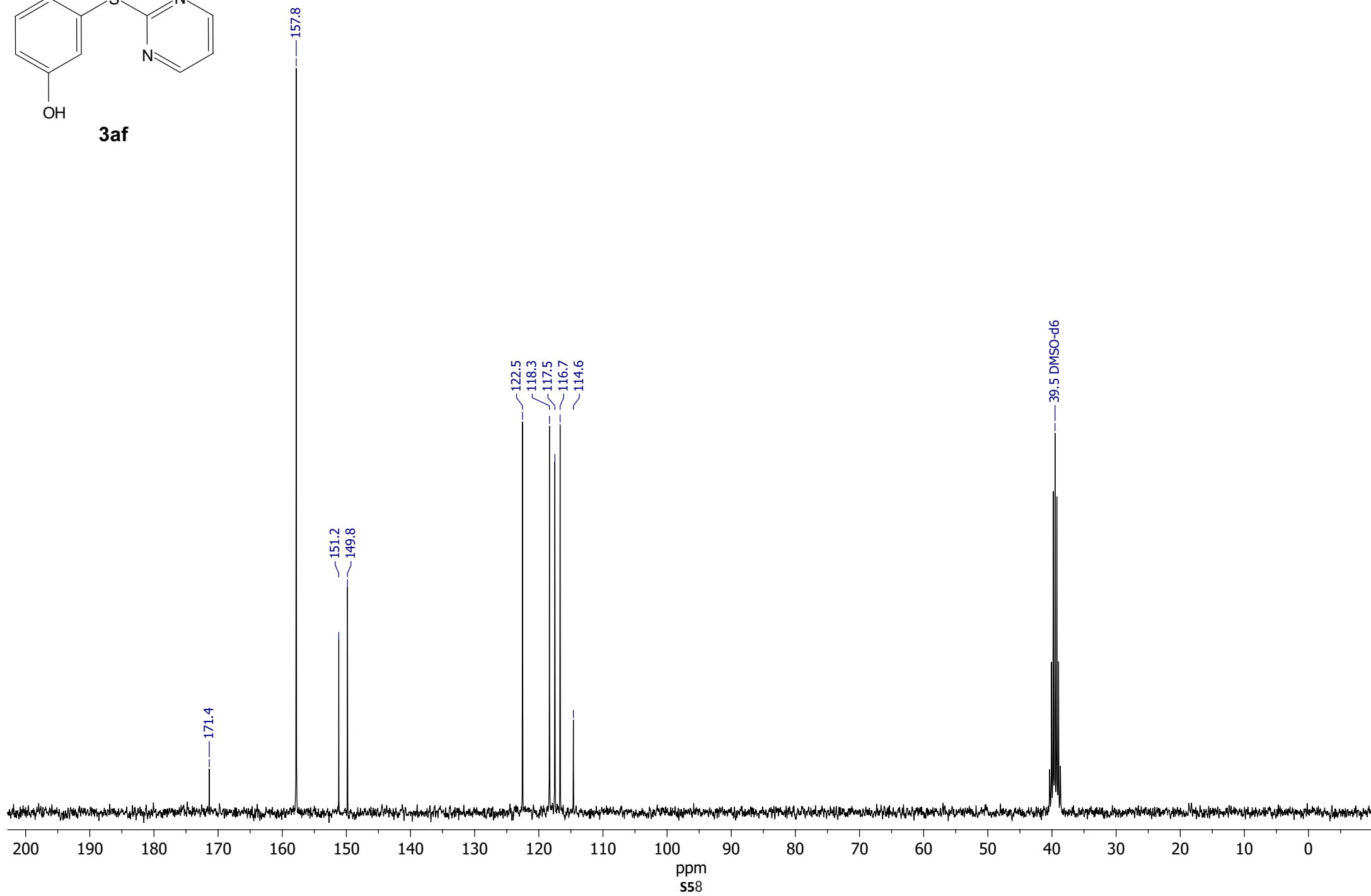
**3af**



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)



**3af**





Chemical Formula: C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>O<sub>2</sub>S  
 Exact Mass: 220,03

**Analysis Info**

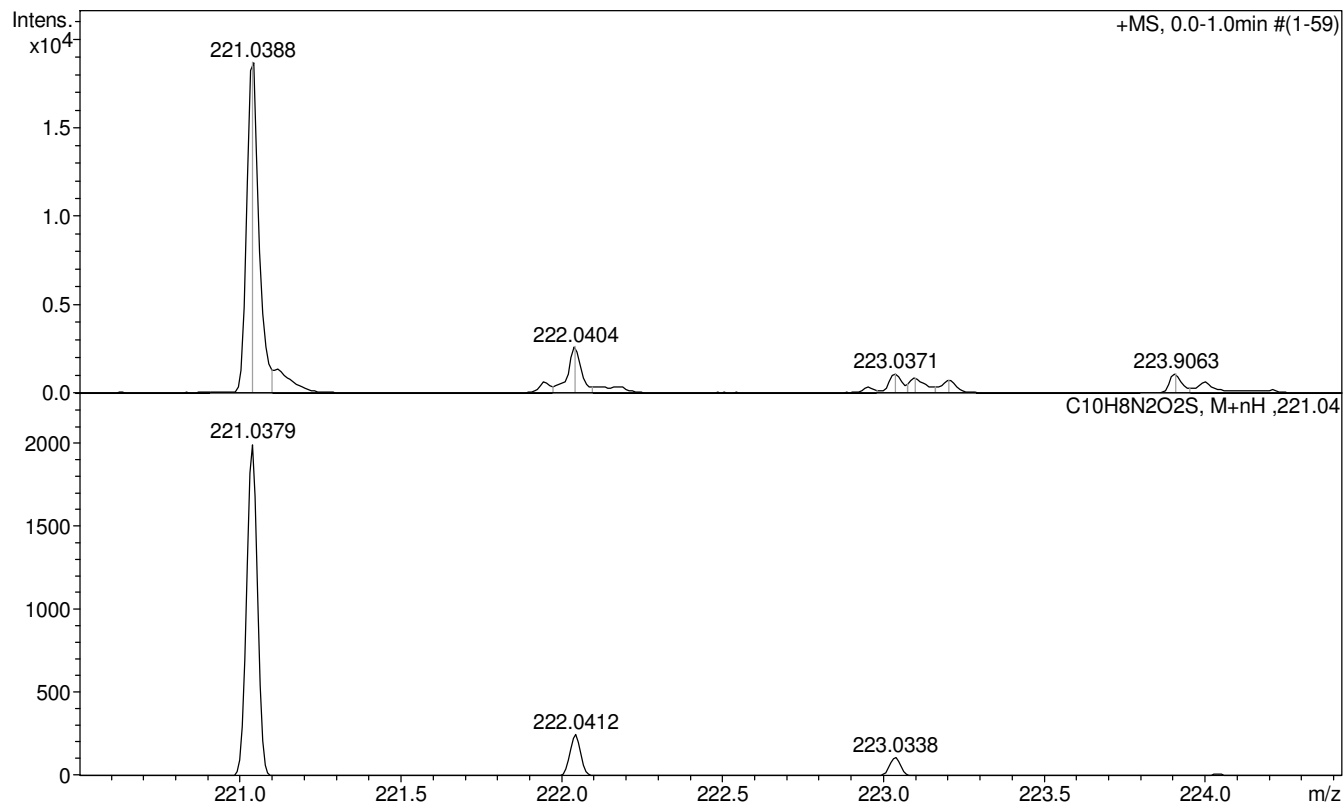
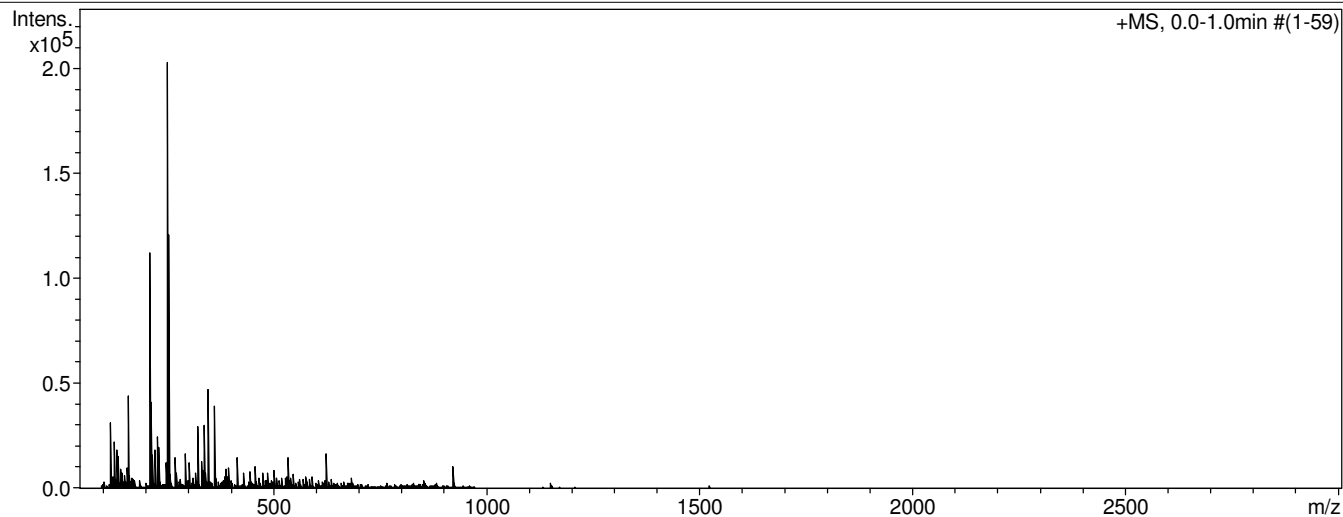
Analysis Name D:\Data\Chizhov\Egorov\Moiseeva\mnv385\_&clblow.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV-385  
 Comment CH3CN 100 %, dil. 200, calibrant added

Acquisition Date 28.06.2024 11:48:35

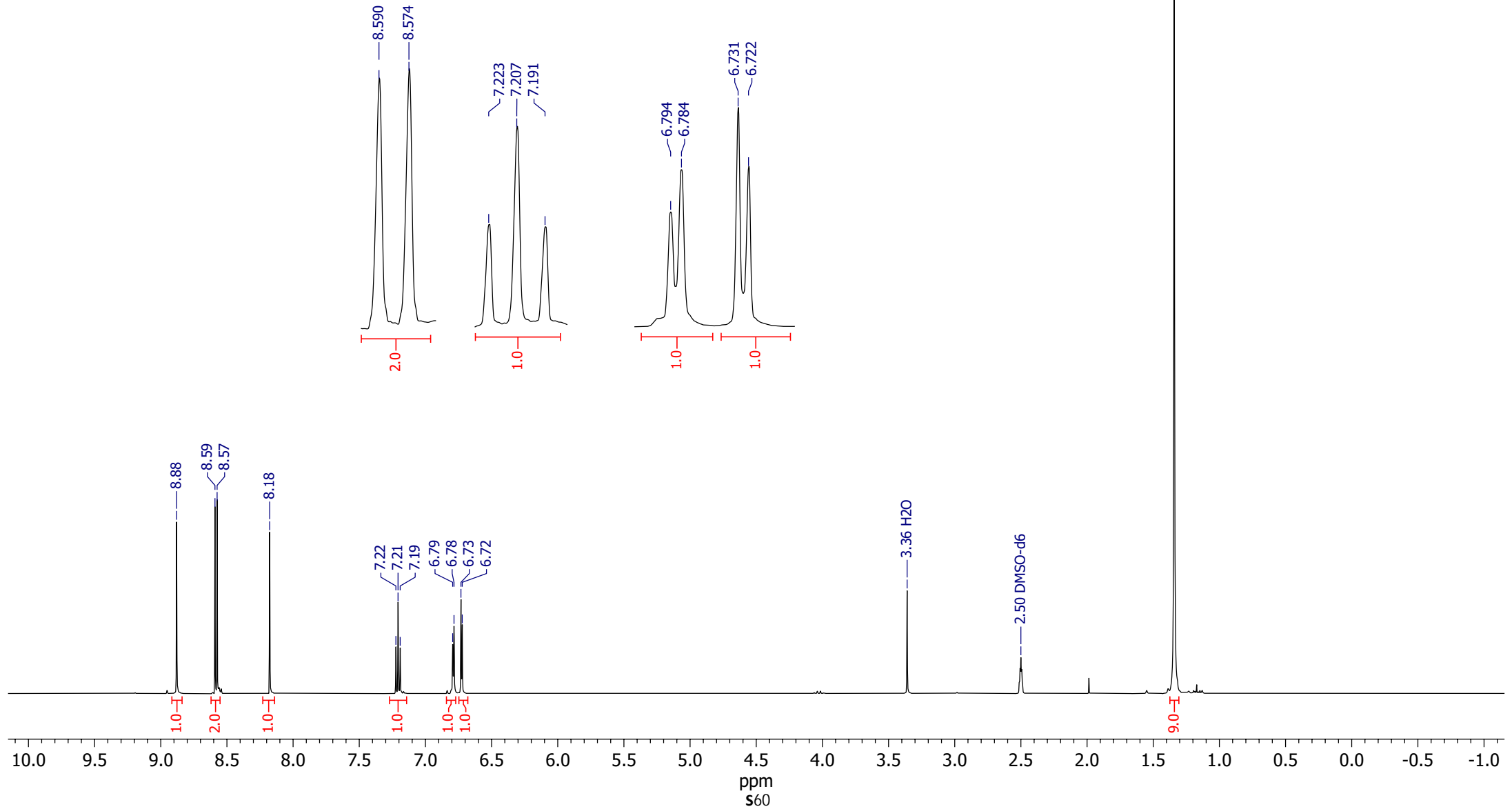
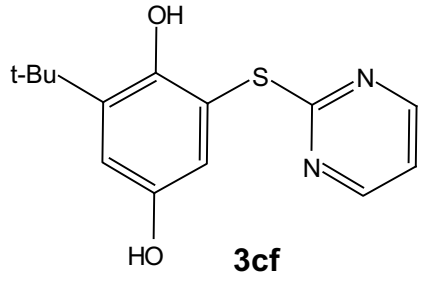
Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

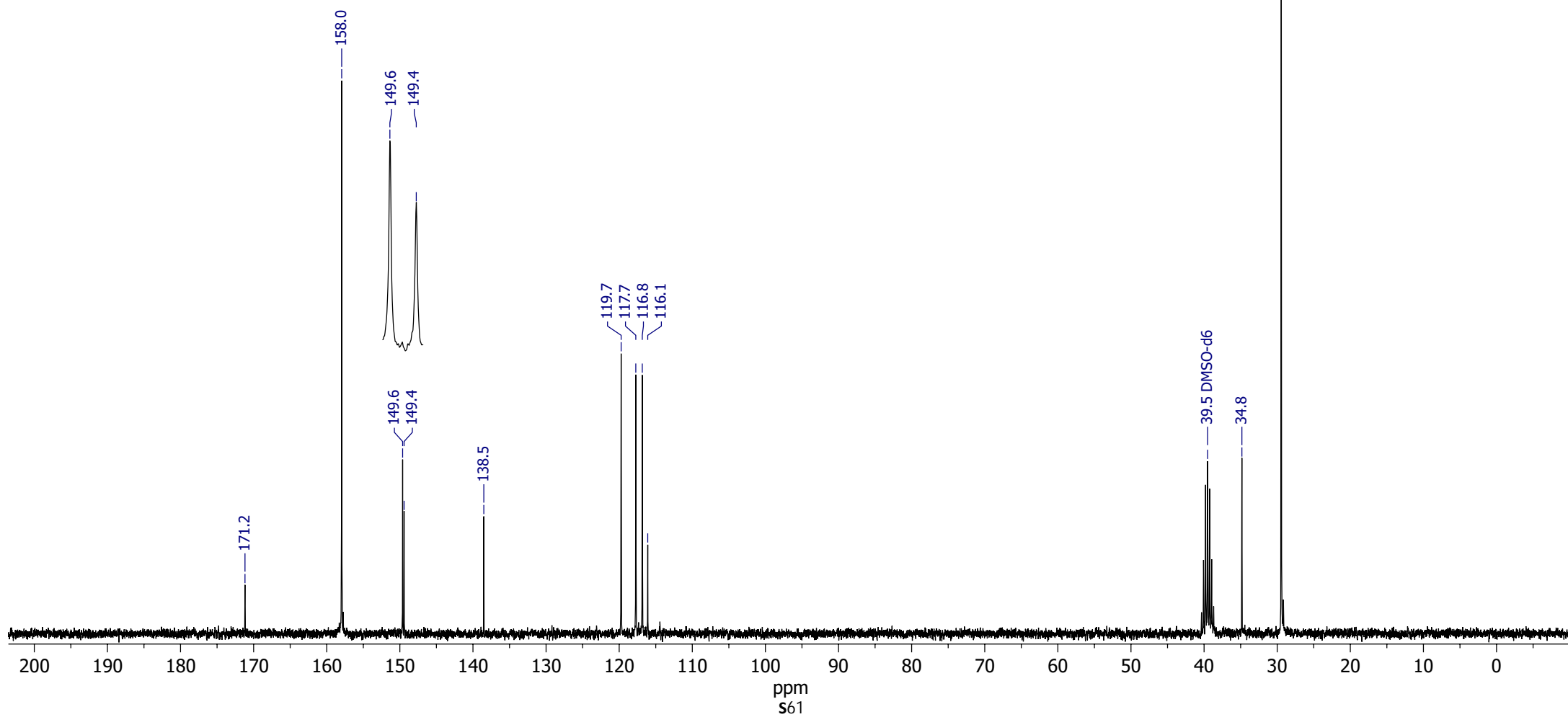
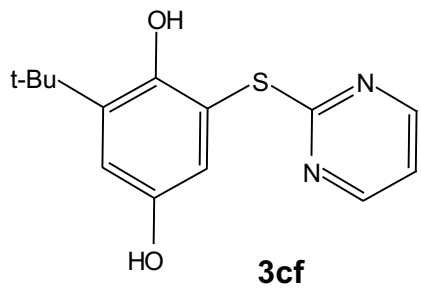
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



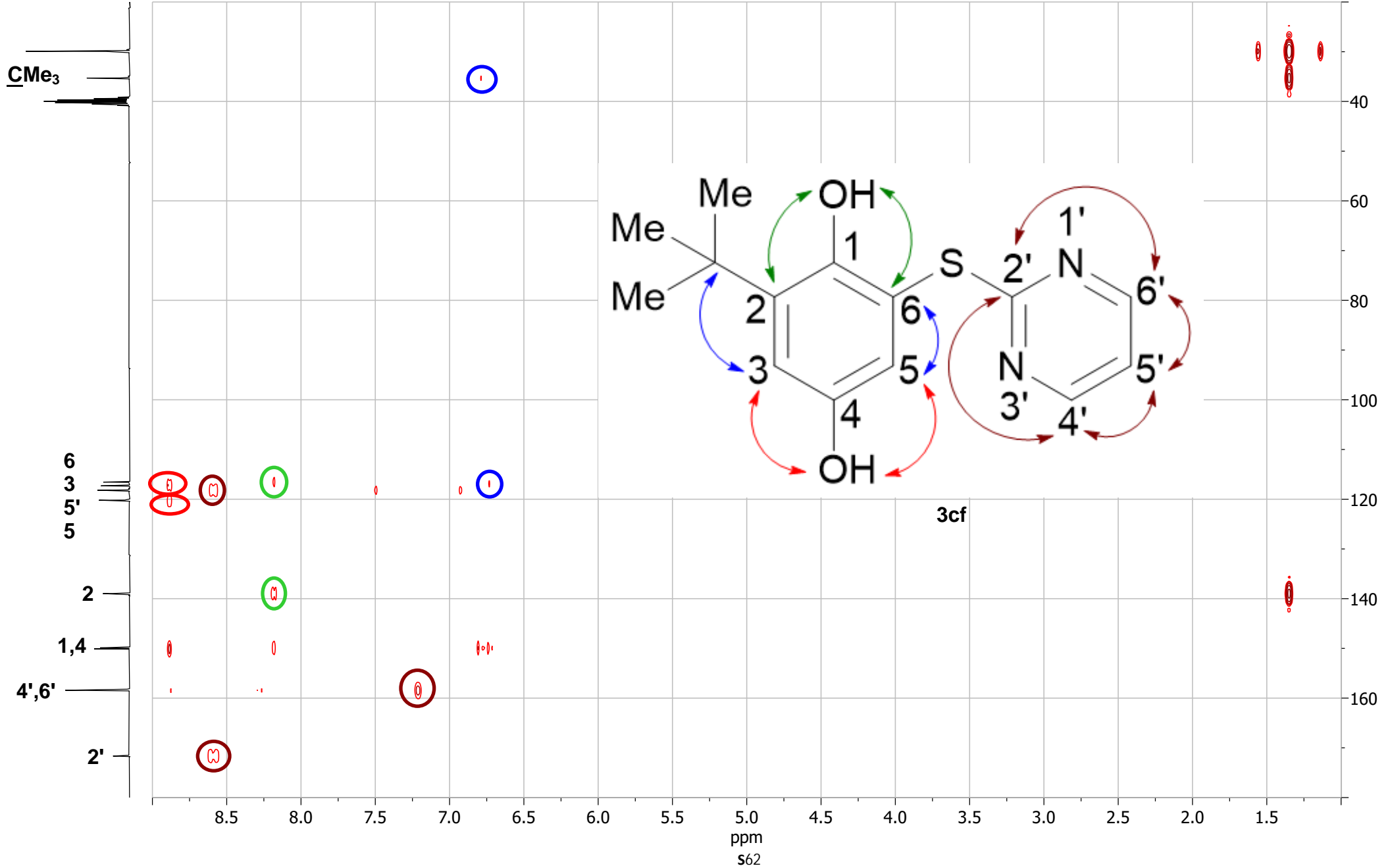
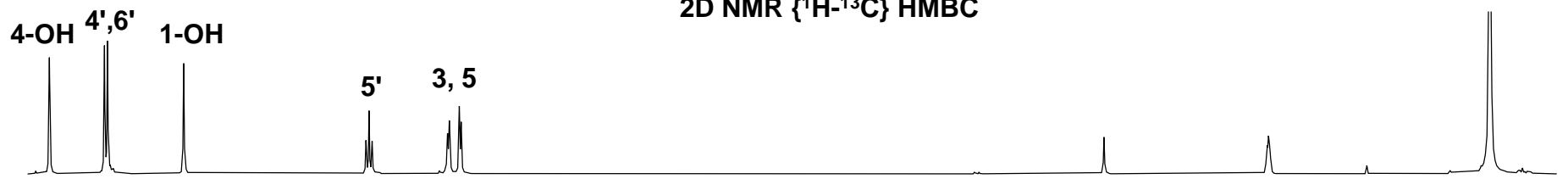
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



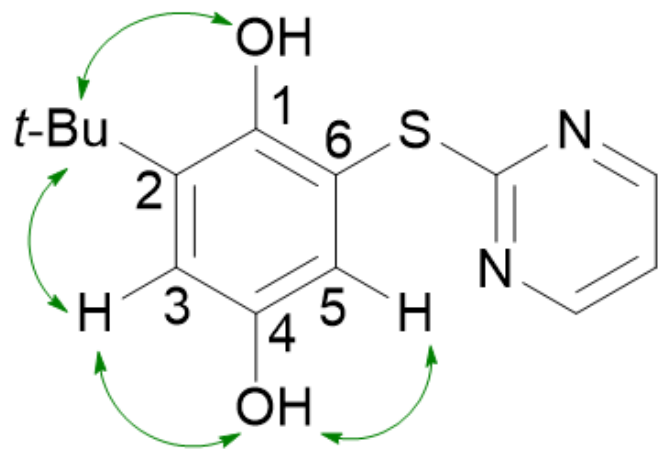
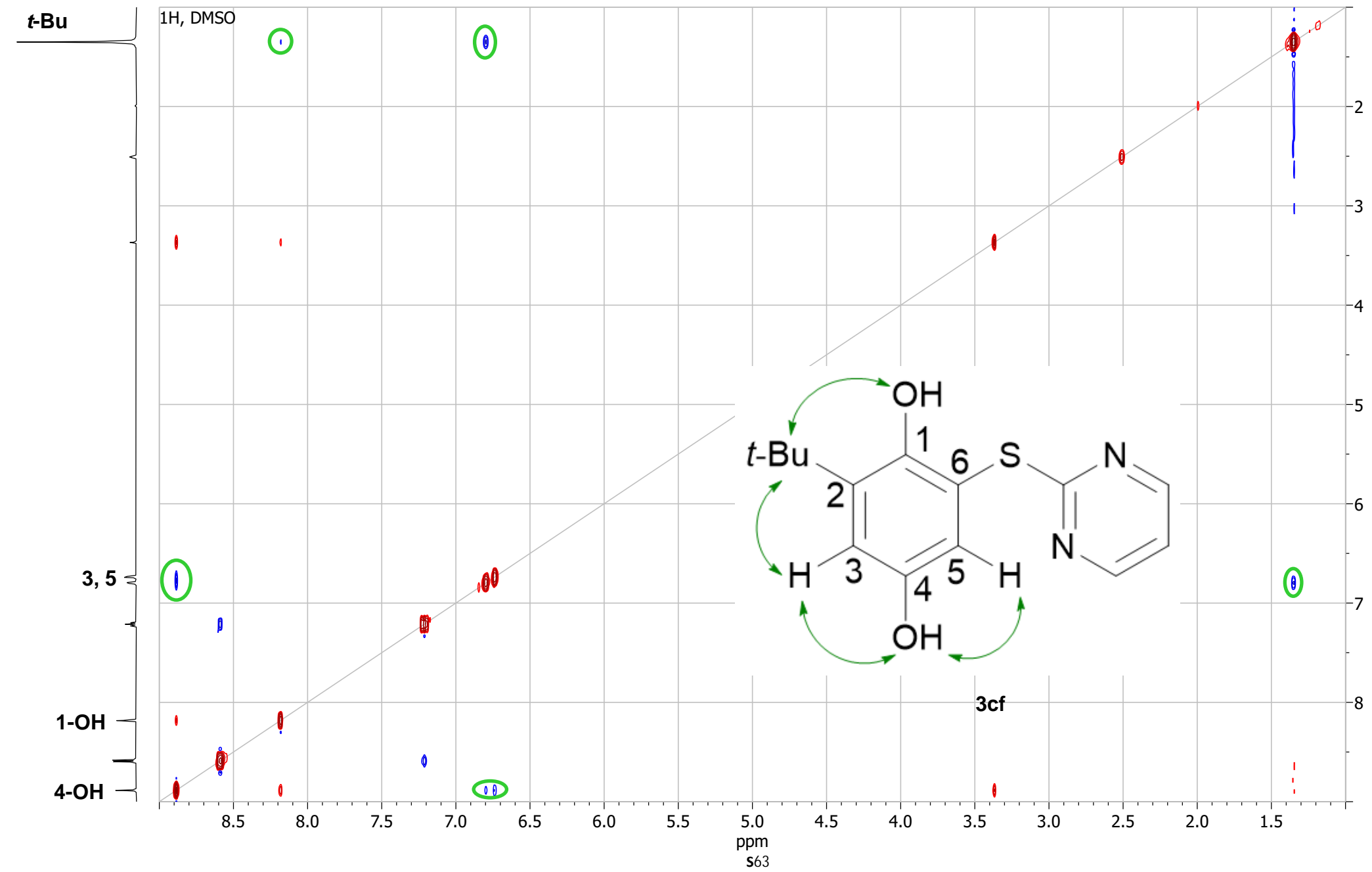
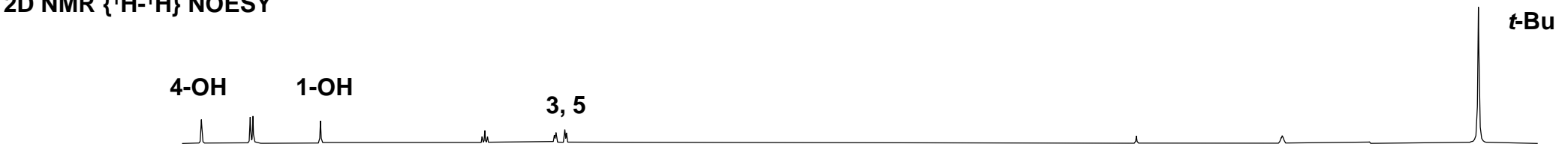
<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)



2D NMR  $\{^1\text{H}-^{13}\text{C}\}$  HMBC



2D NMR  $\{^1\text{H}-^1\text{H}\}$  NOESY



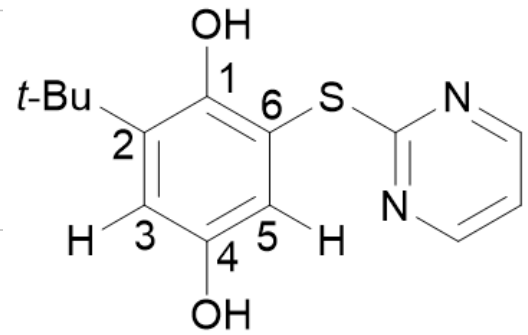
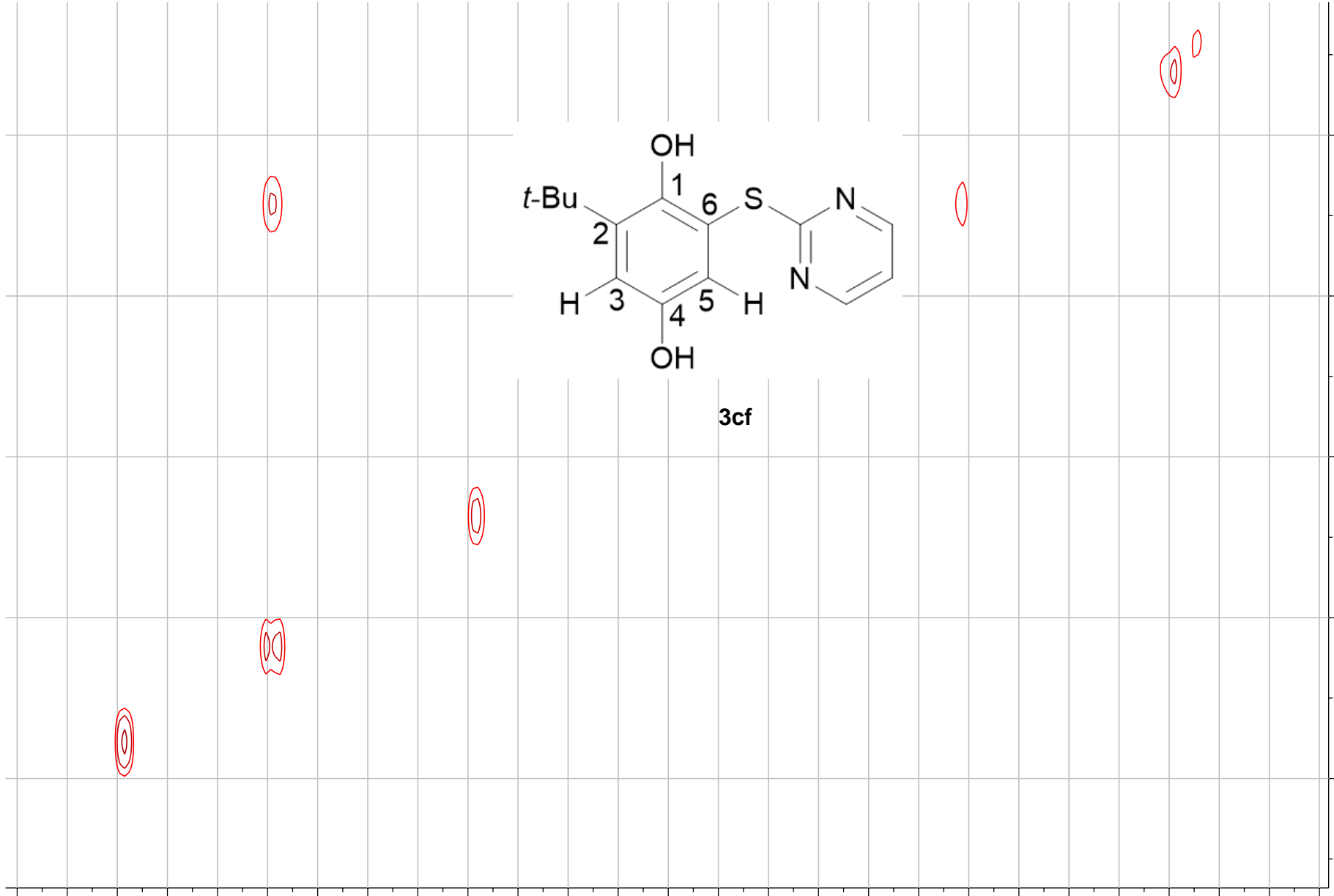
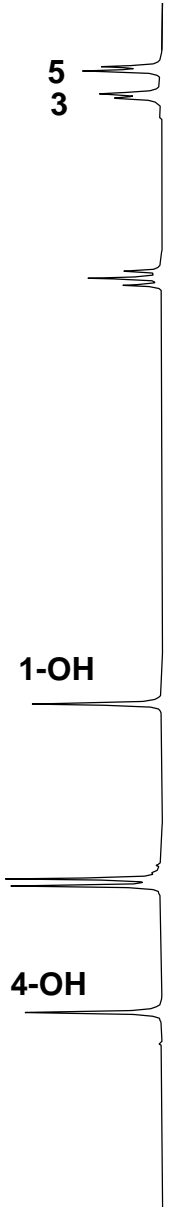
2D NMR  $\{^1\text{H}-^1\text{H}\}$  COSY

4-OH

1-OH

3

5



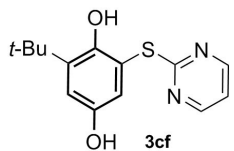
3cf

9.1 9.0 8.9 8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5

ppm  
s64

f1 (ppm)





Chemical Formula: C<sub>14</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub>S  
 Exact Mass: 276,09

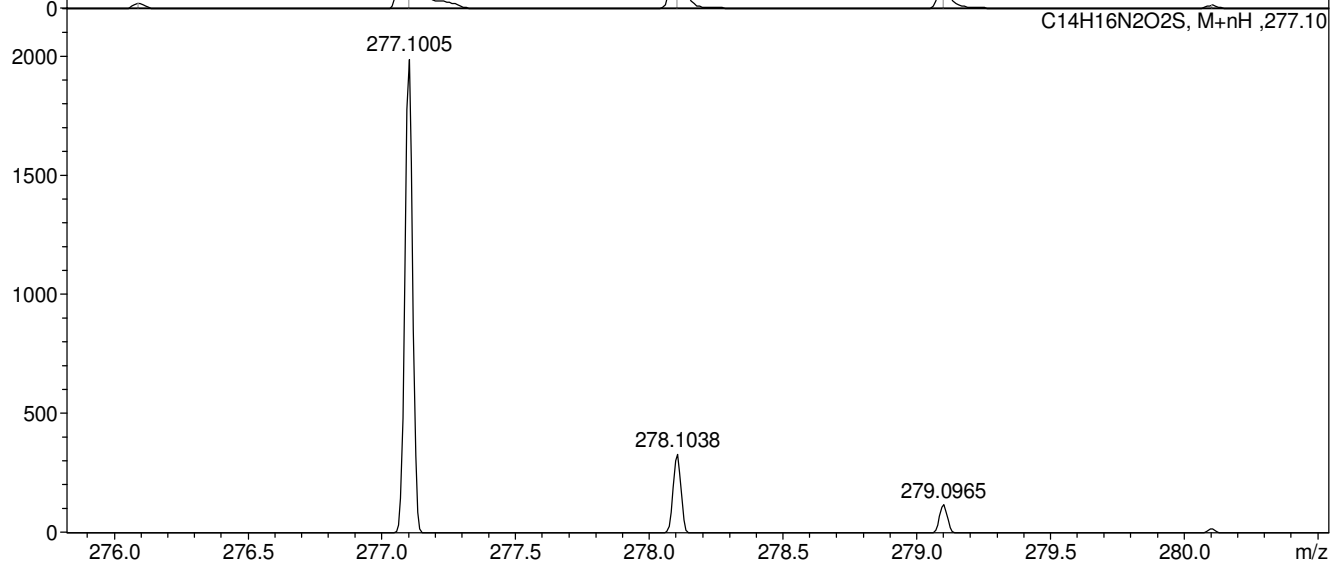
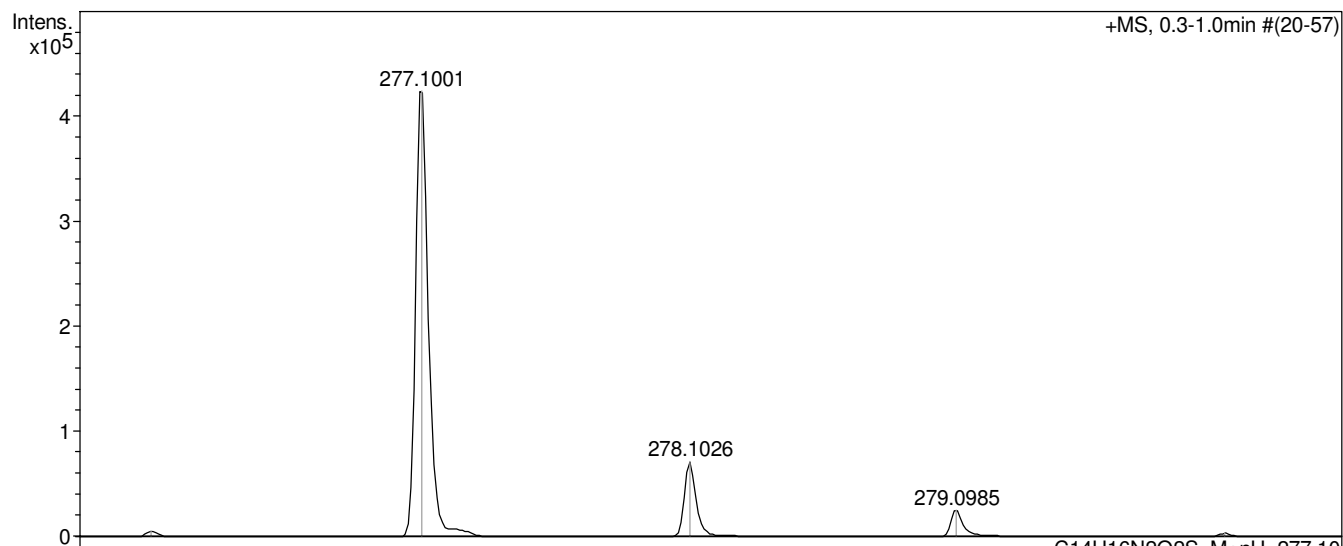
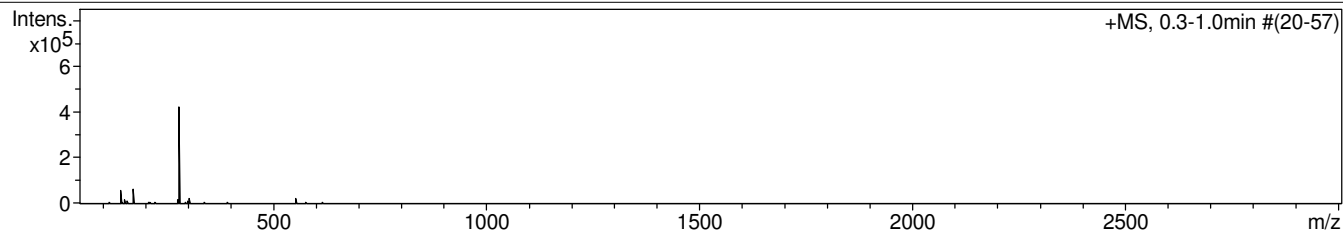
**Analysis Info**

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\0611035.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV379  
 Comment C14H16N2O2S mH277.1005 calibrant added CH3CN

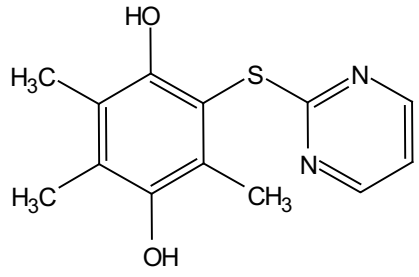
Acquisition Date 11.06.2024 15:33:16  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

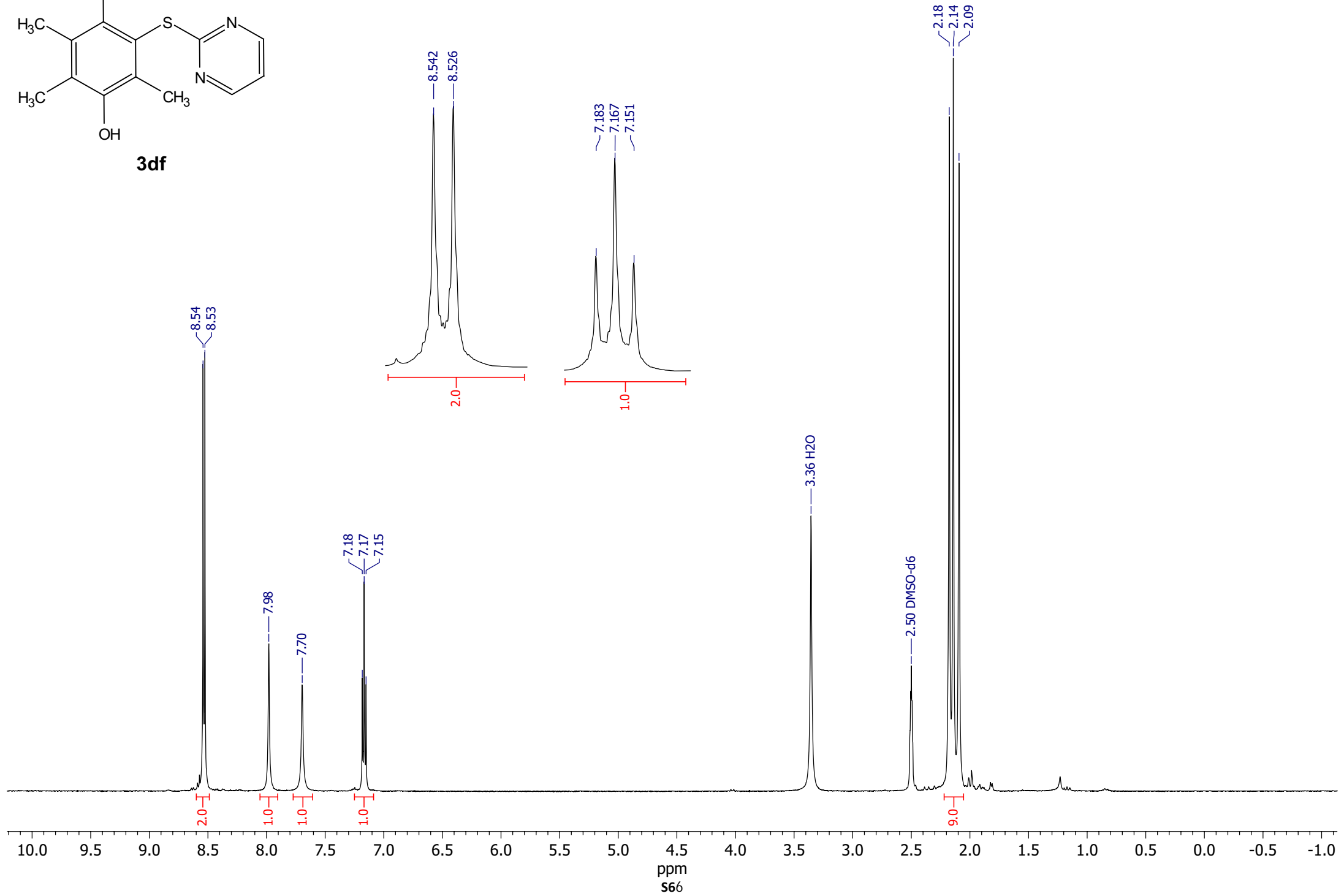
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



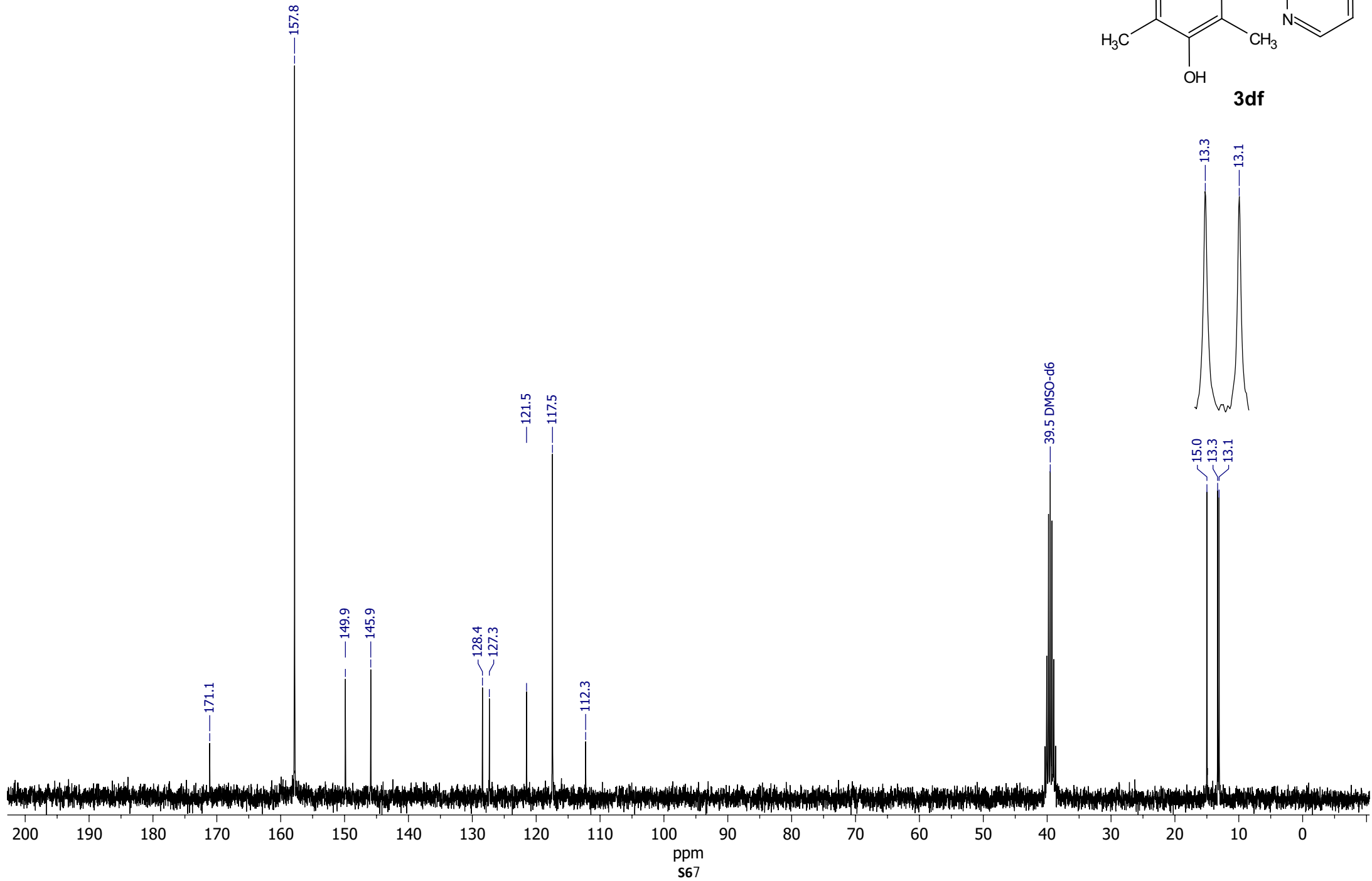
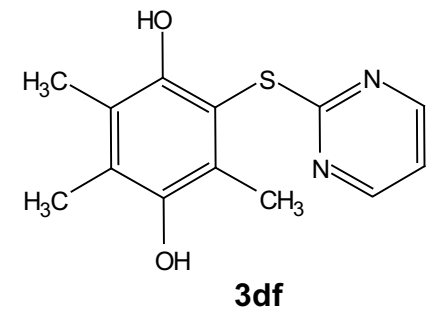
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)

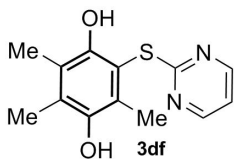


**3df**



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)





Chemical Formula: C<sub>13</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub>S  
 Exact Mass: 262,08

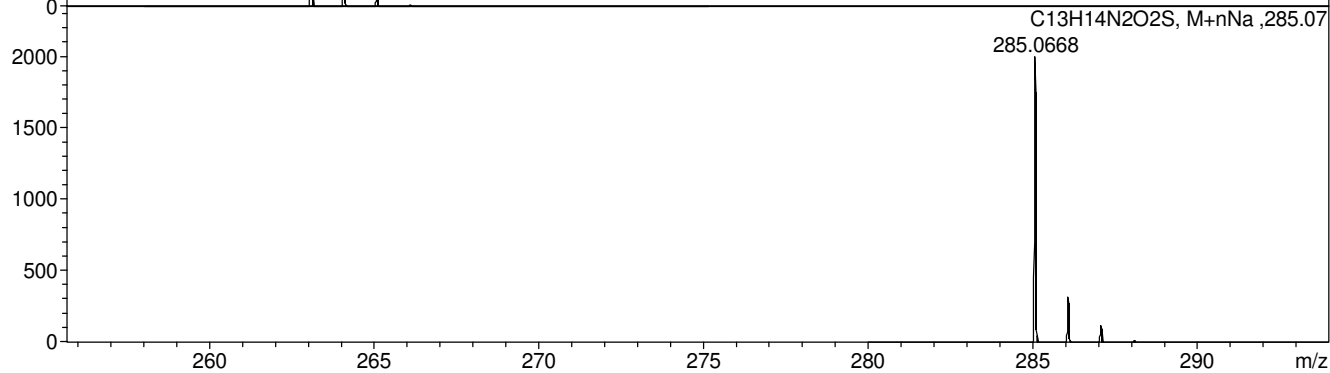
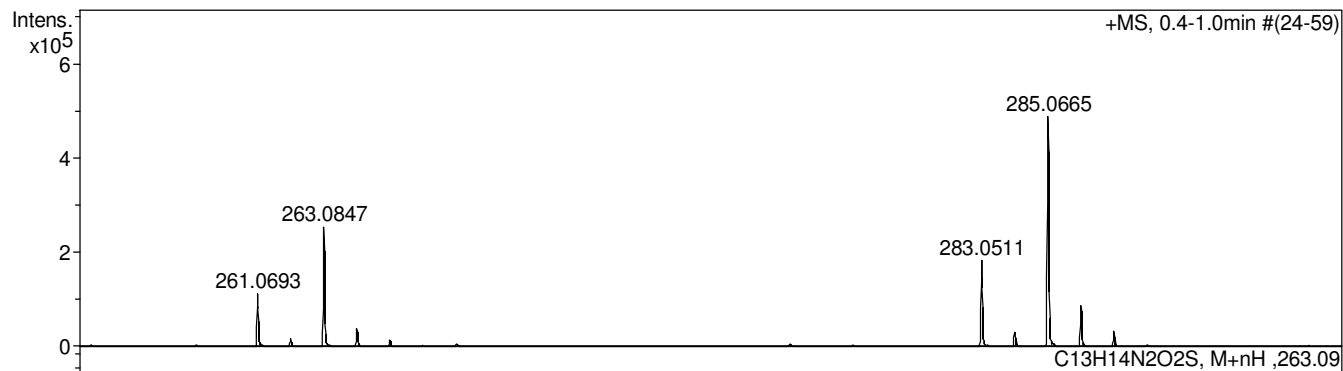
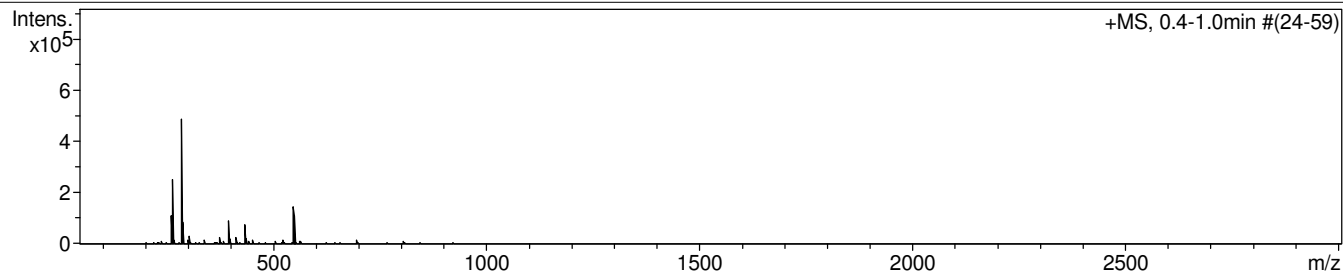
**Analysis Info**

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\0711021.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV386  
 Comment C13H14N2O2S mH263.0848 calibrant added CH3OH

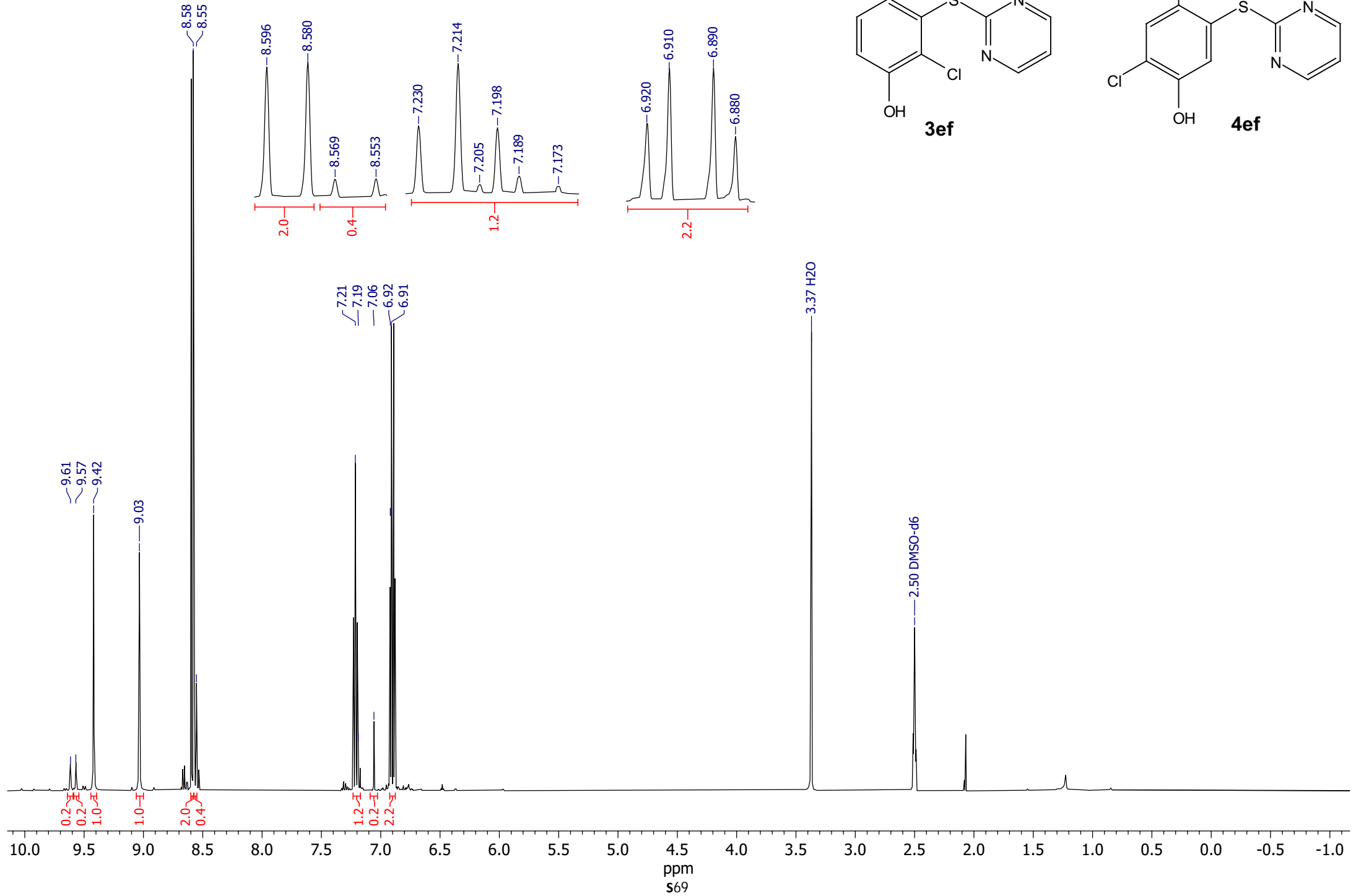
Acquisition Date 11.07.2024 16:46:54  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

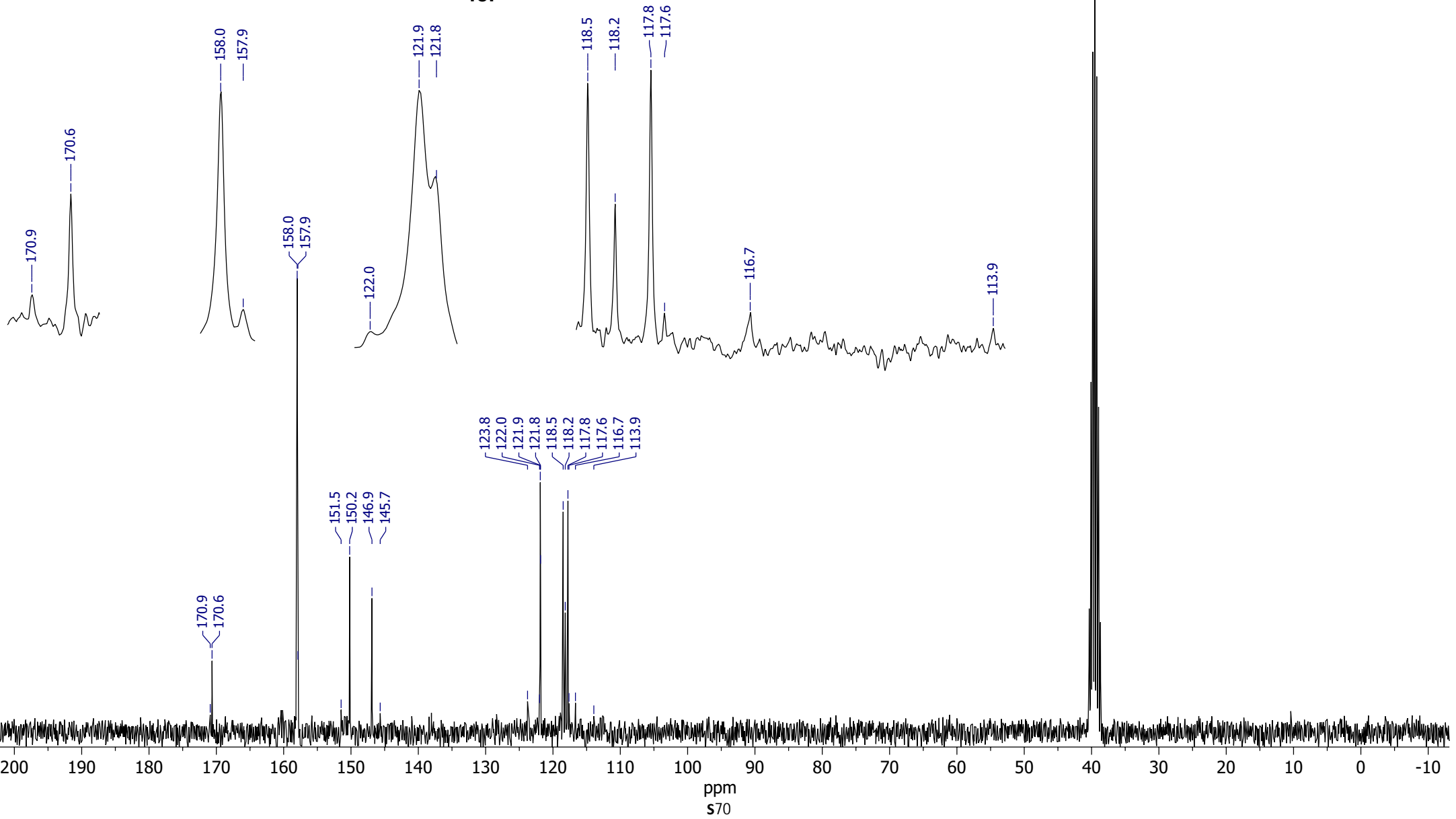
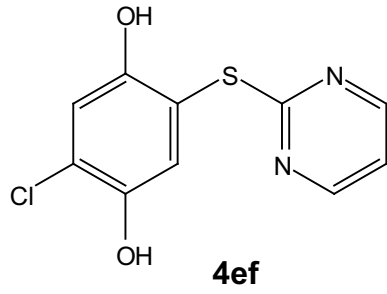
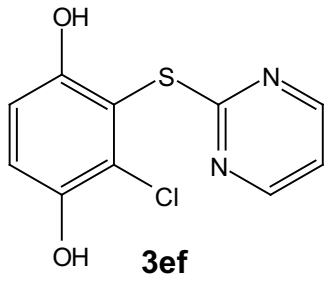
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



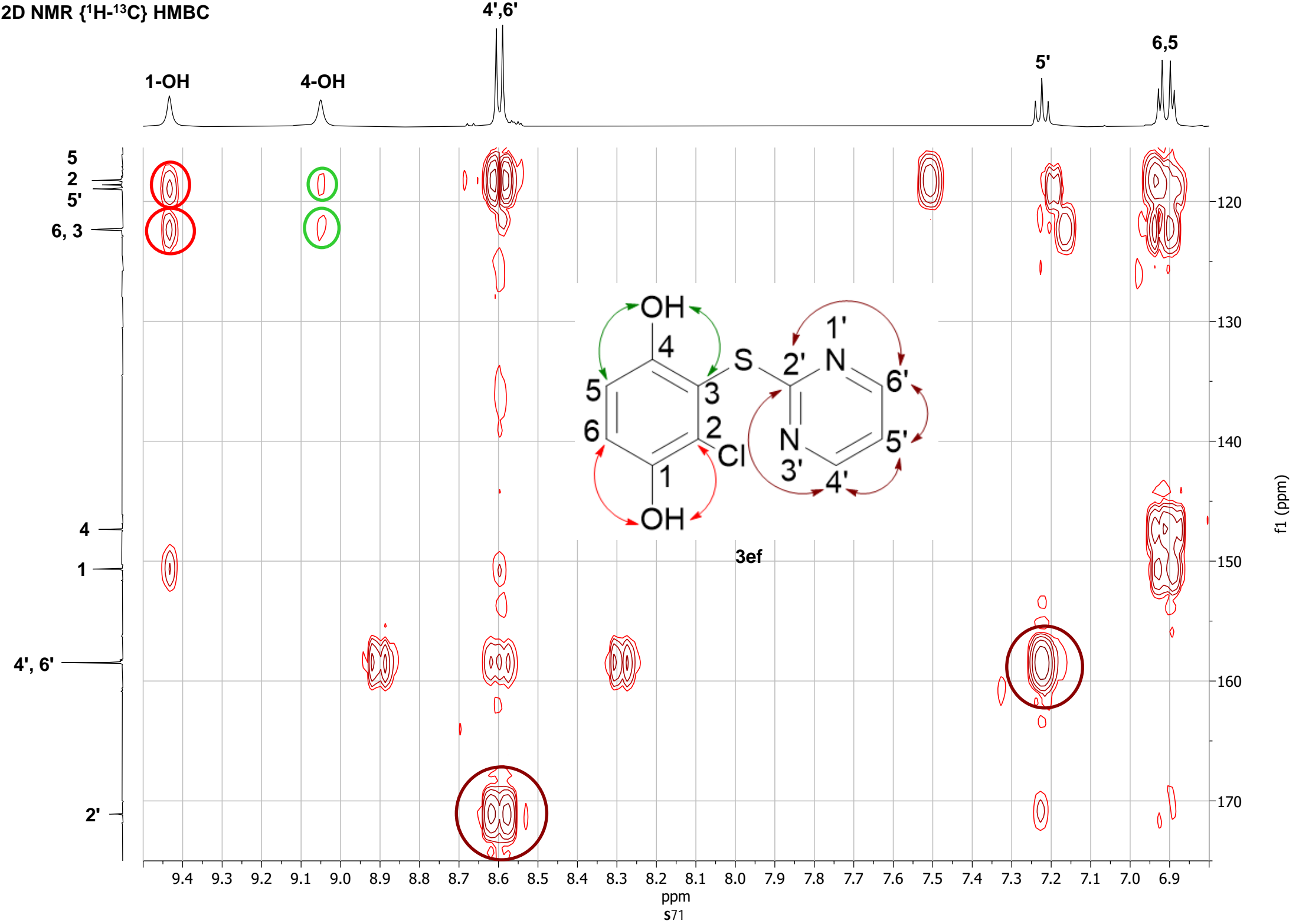
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



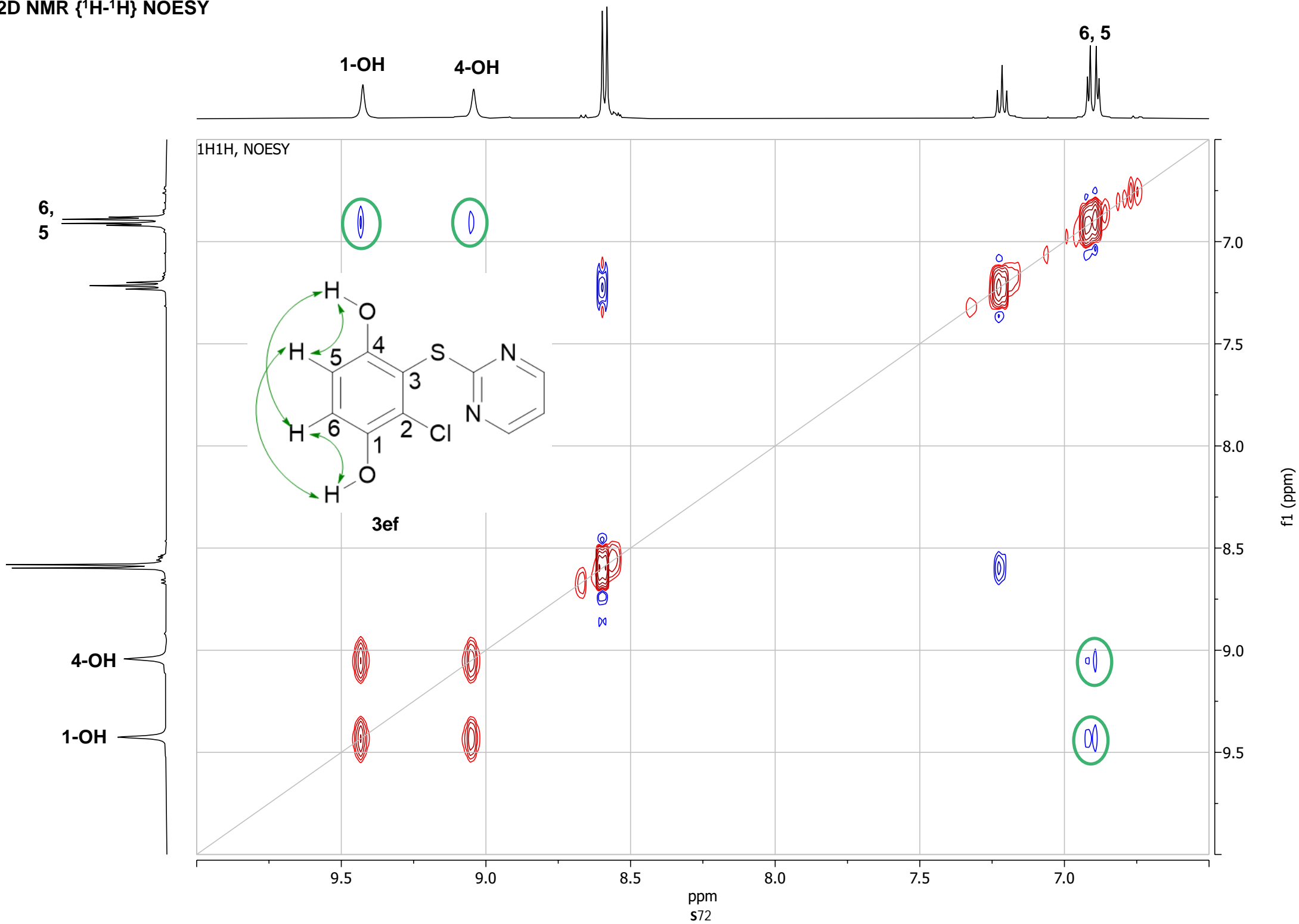
<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)



2D NMR  $\{^1\text{H}-^{13}\text{C}\}$  HMBC

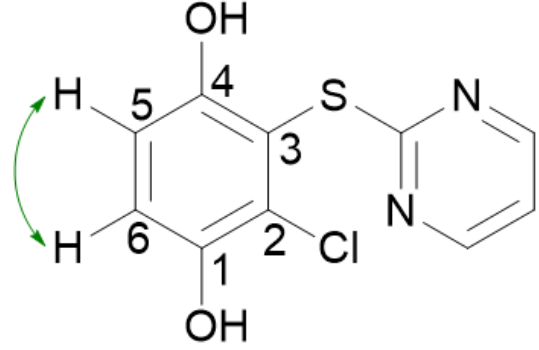
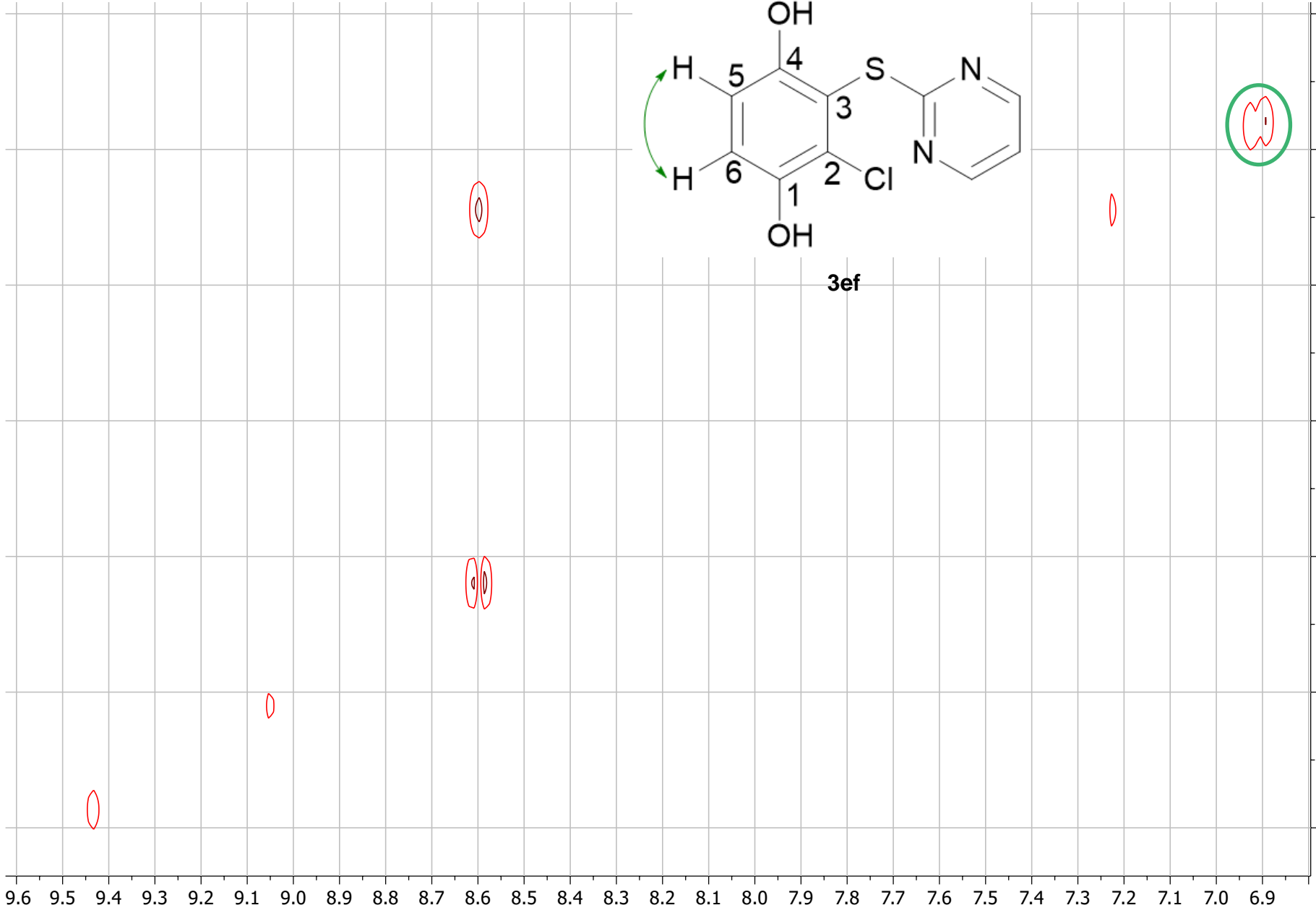
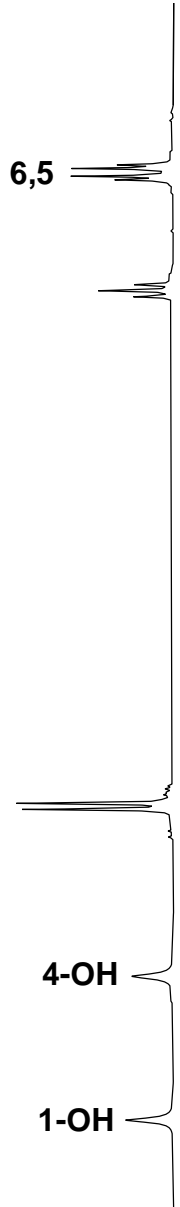


2D NMR {<sup>1</sup>H-<sup>1</sup>H} NOESY





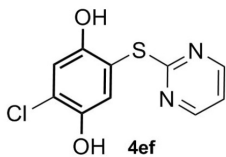
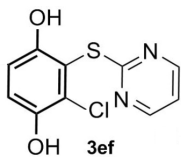
2D NMR  $\{^1\text{H}-^1\text{H}\}$  COSY



3ef

f1 (ppm)

ppm  
s73



Chemical Formula: C<sub>10</sub>H<sub>7</sub>ClN<sub>2</sub>O<sub>2</sub>S  
Exact Mass: 253,99

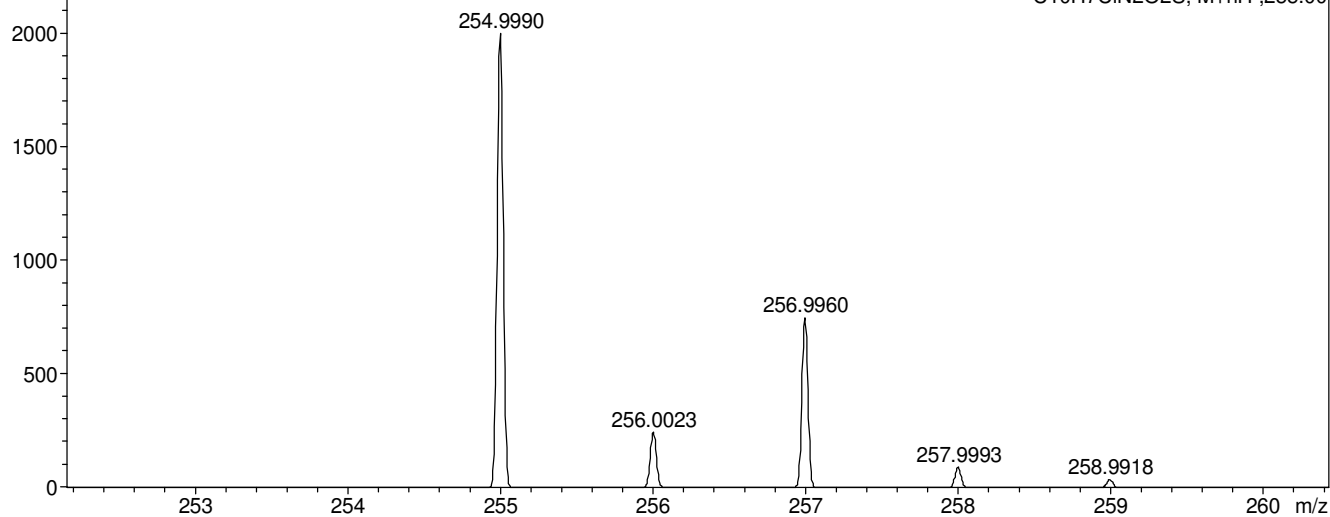
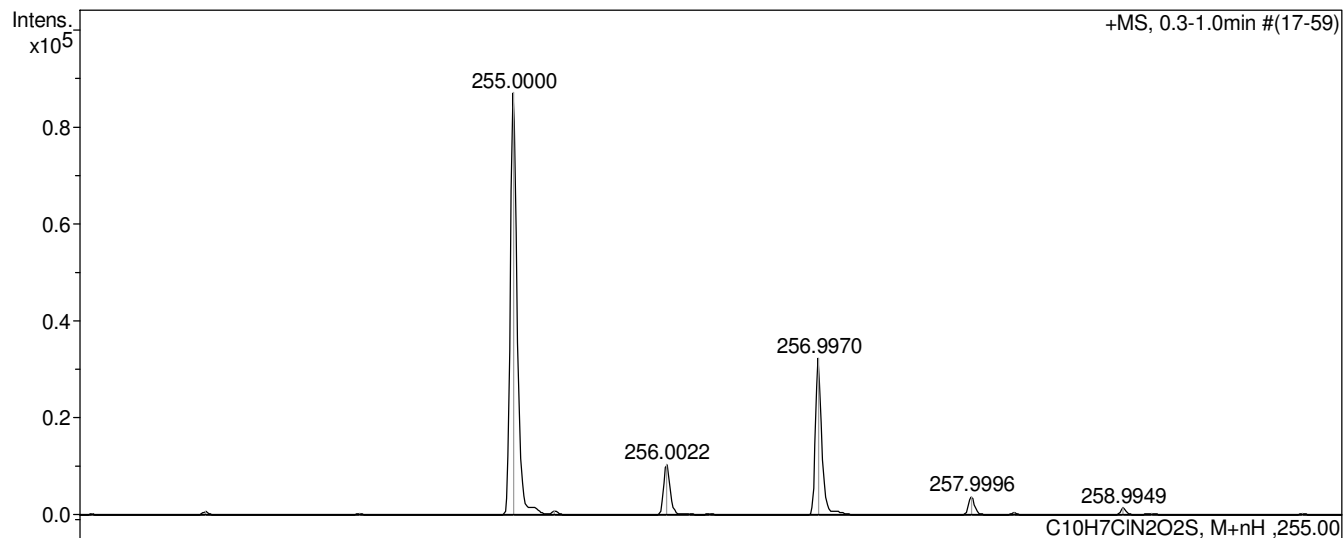
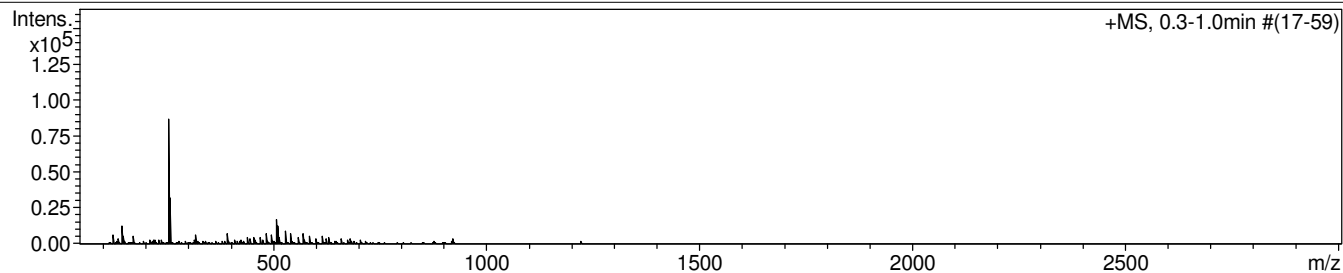
**Analysis Info**

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\0716005.d  
Method tune\_low.m  
Sample Name /VAPP MNV384  
Comment C10H7ClN2O2S mH254.9989 calibrant added CH3CN

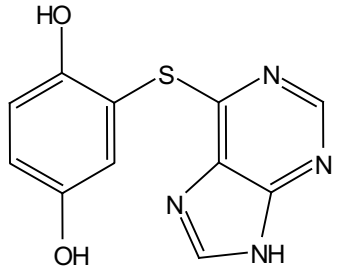
Acquisition Date 16.07.2024 10:34:41  
Operator BDAL@DE  
Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

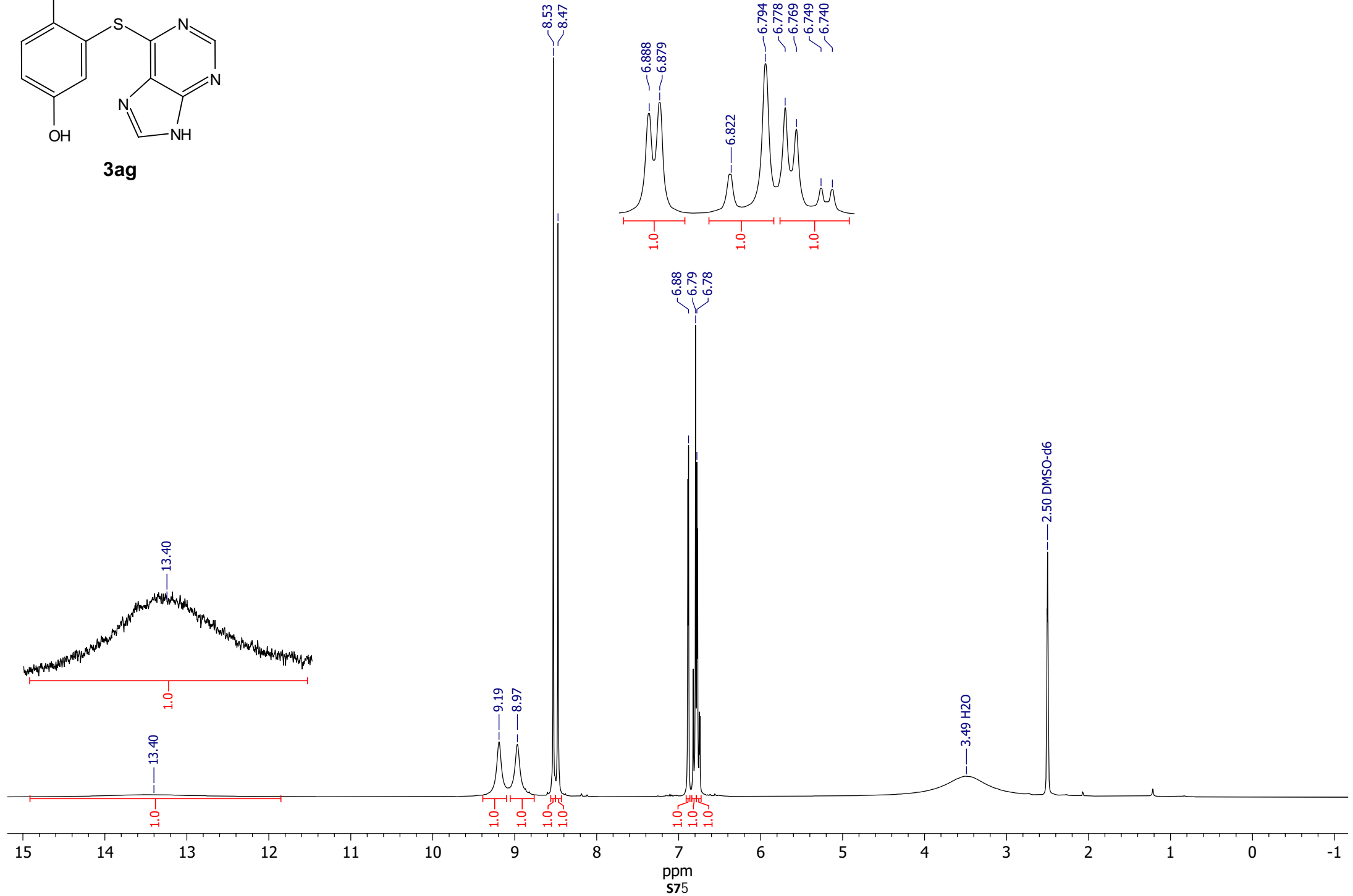
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



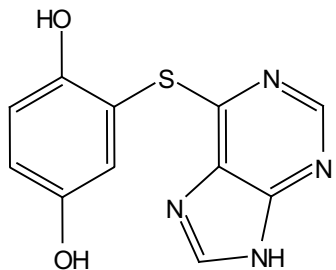
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



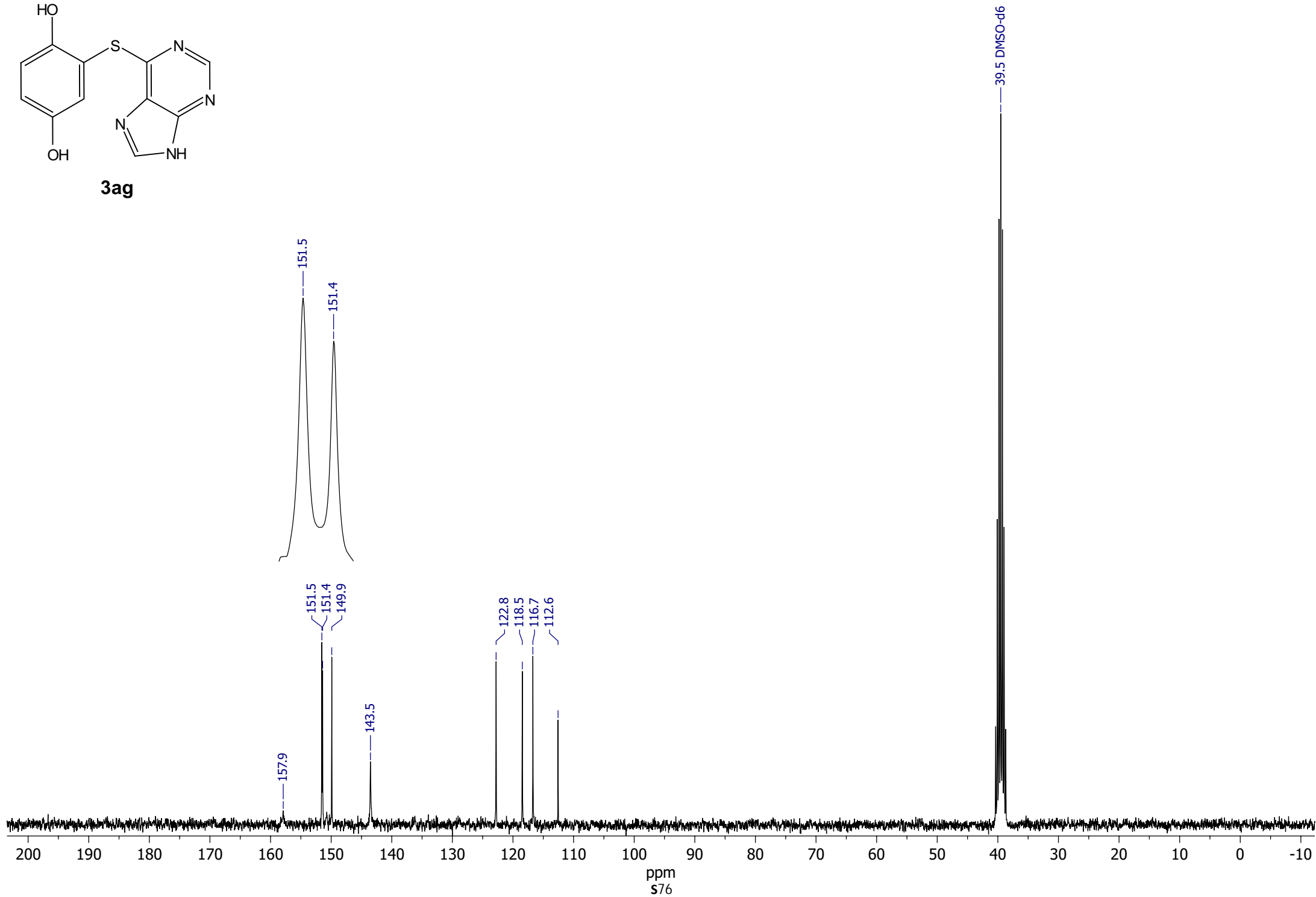
**3ag**

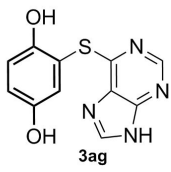


<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)



**3ag**





Chemical Formula: C<sub>11</sub>H<sub>8</sub>N<sub>4</sub>O<sub>2</sub>S  
 Exact Mass: 260,04

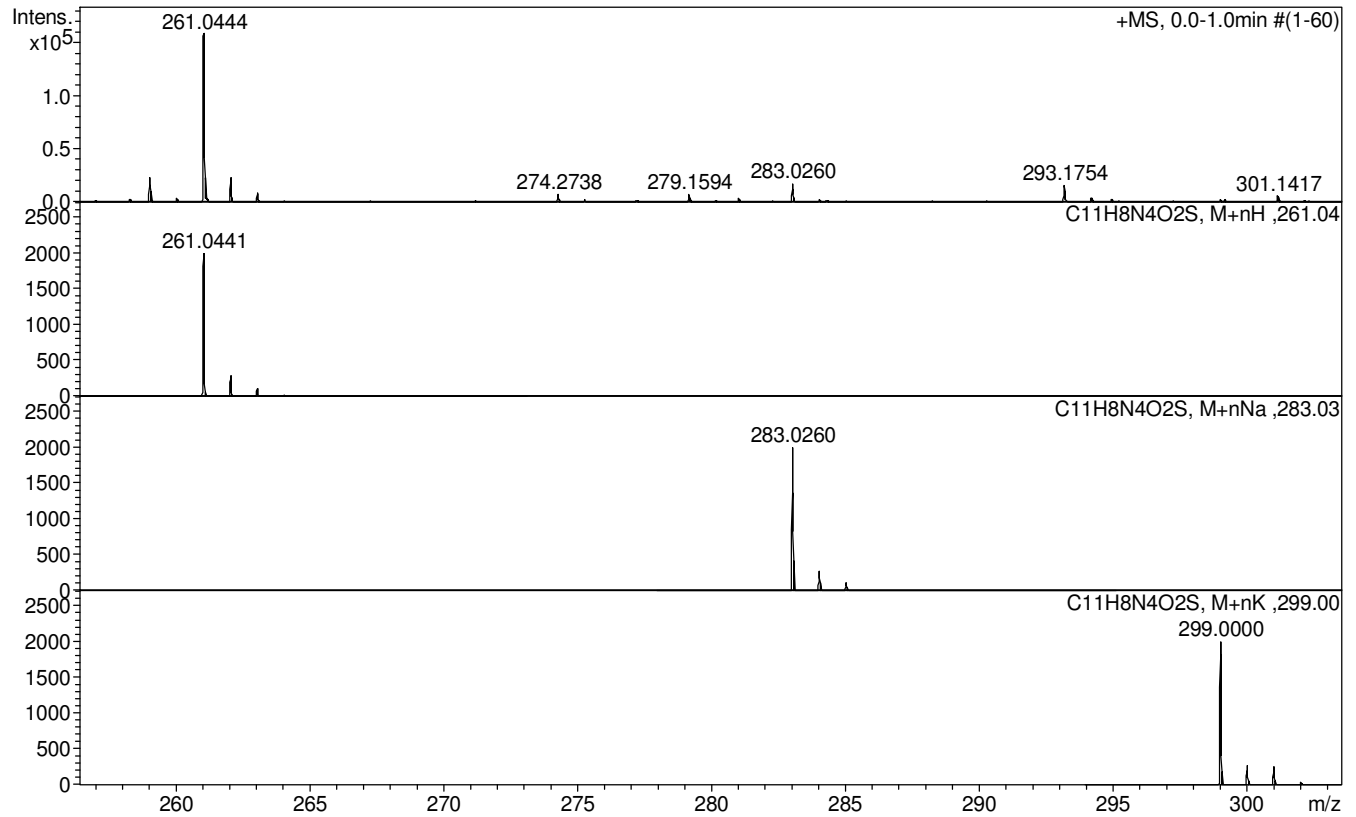
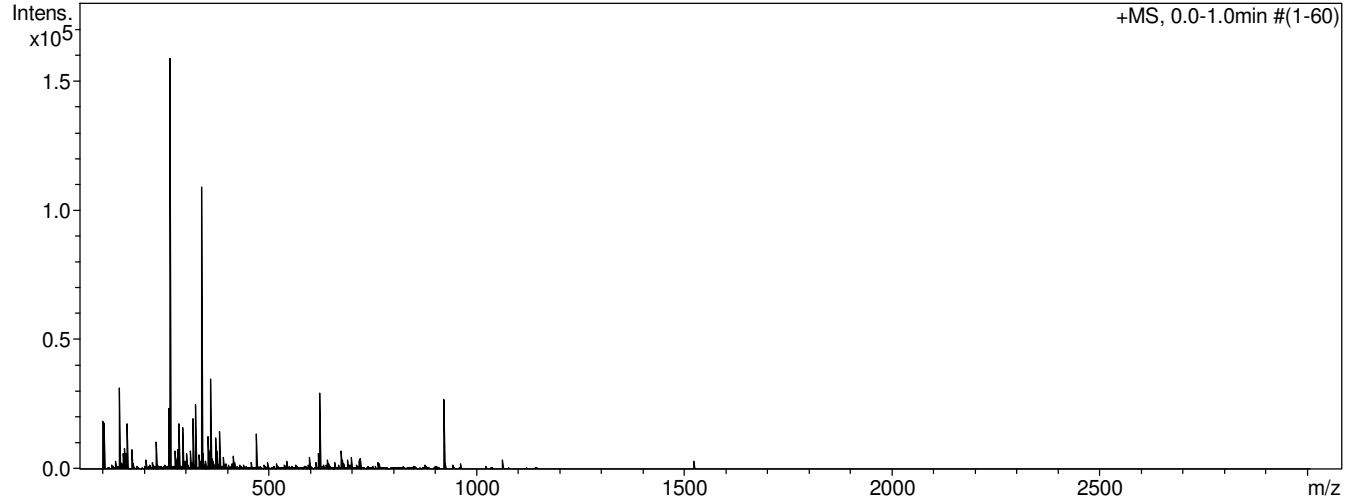
**Analysis Info**

Analysis Name D:\Data\Chizhov\Egorov\Moiseeva\mnv363\_&clblow.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV363  
 Comment CH3CN 100 %, dil. 200, calibrant added

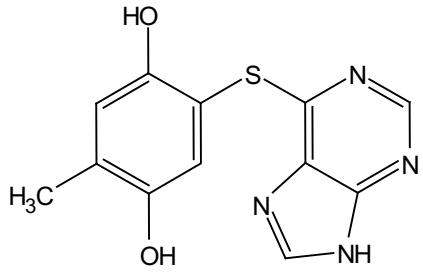
Acquisition Date 17.05.2024 15:27:21  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

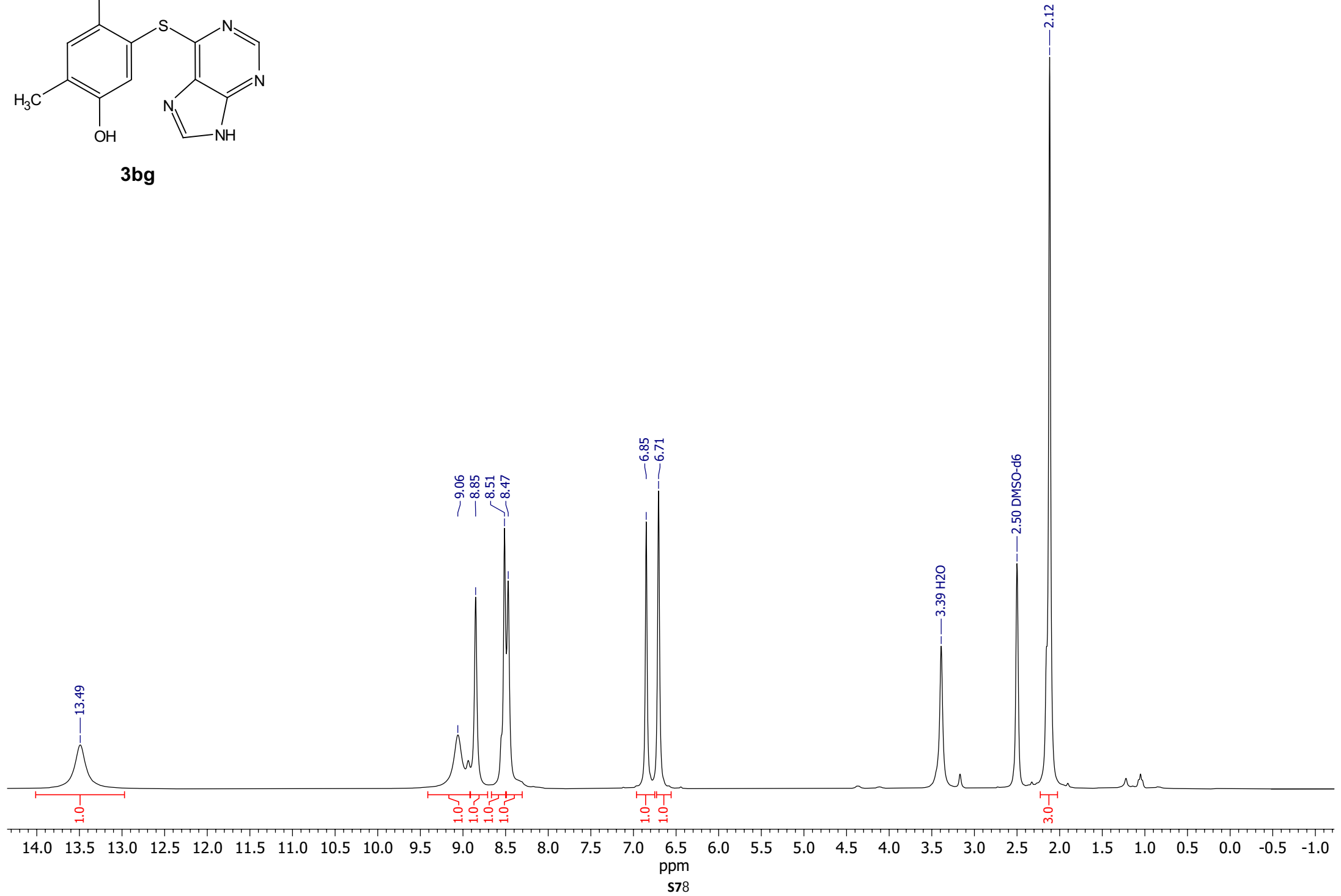
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



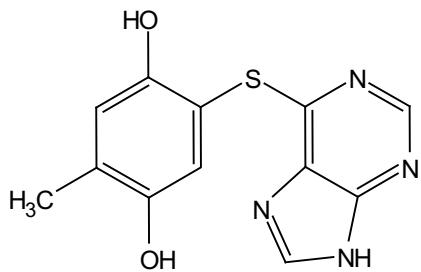
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



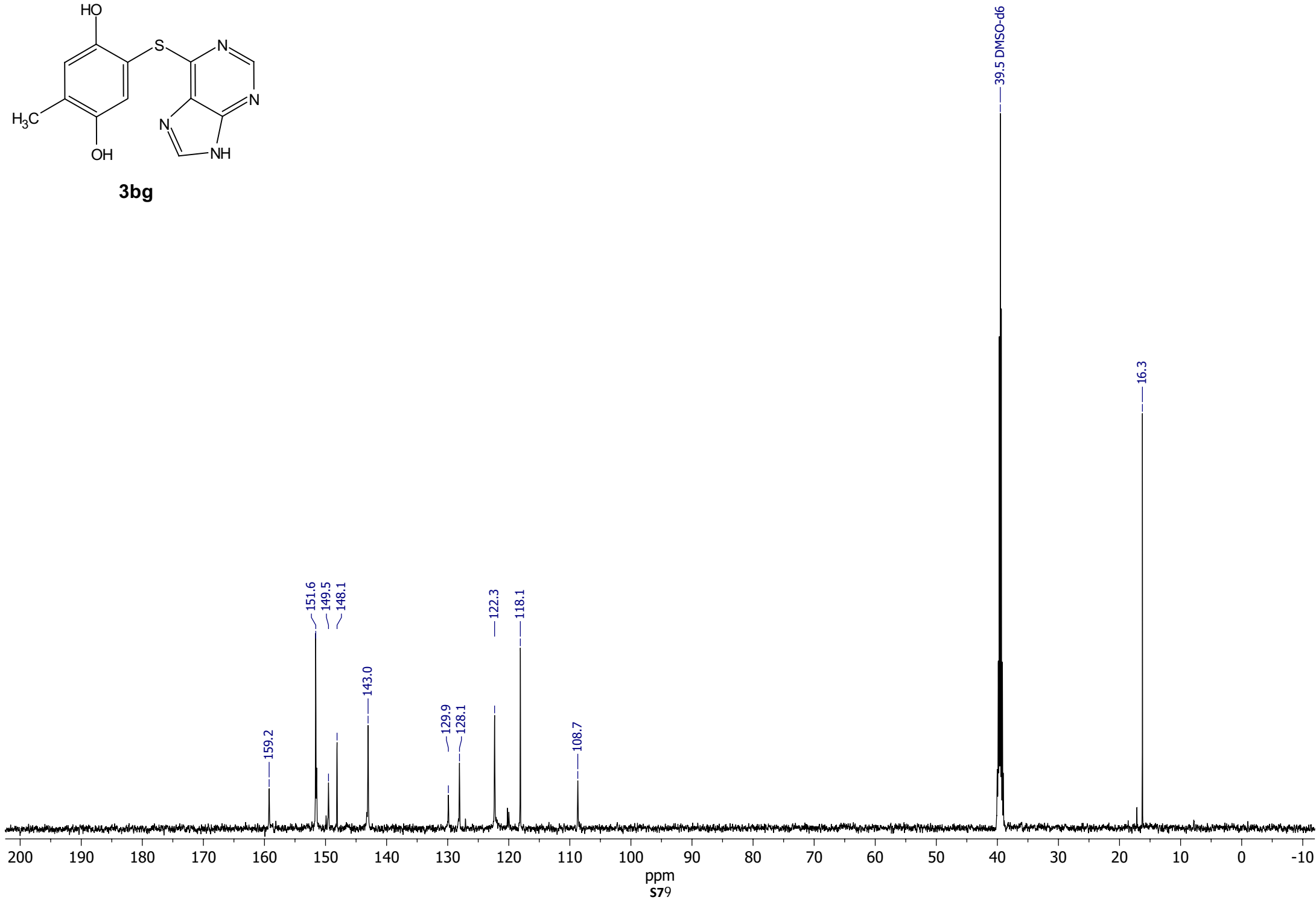
**3bg**

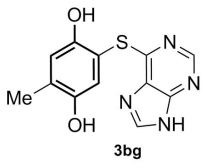


<sup>13</sup>C NMR (125.77 MHz, DMSO-d<sub>6</sub>)



**3bg**





Chemical Formula: C<sub>12</sub>H<sub>10</sub>N<sub>4</sub>O<sub>2</sub>S  
 Exact Mass: 274,05

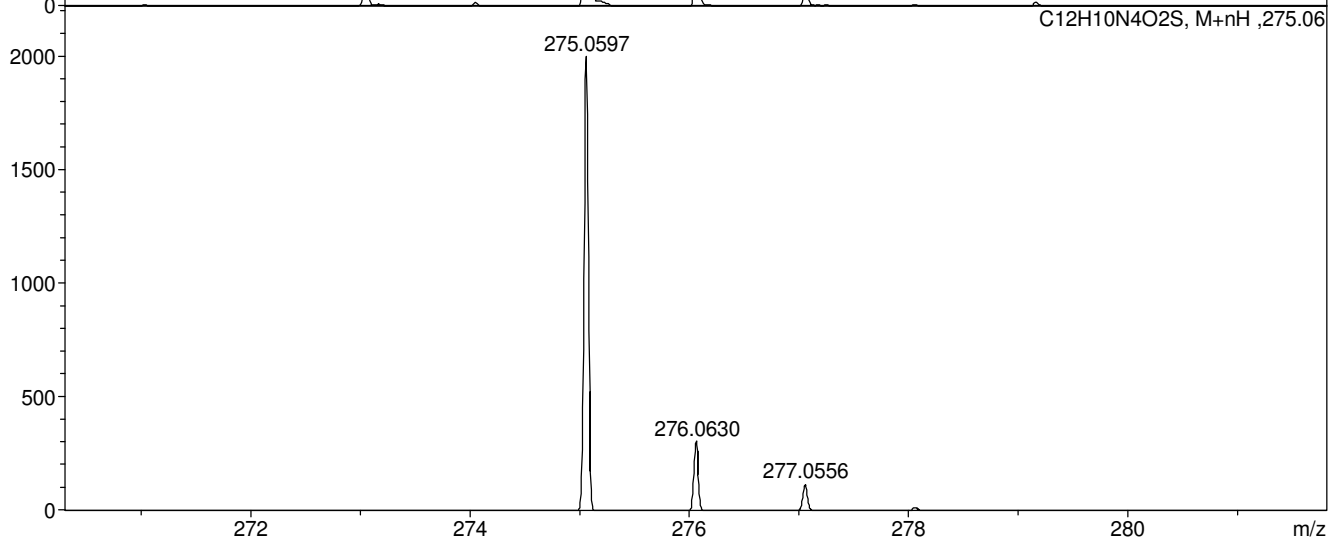
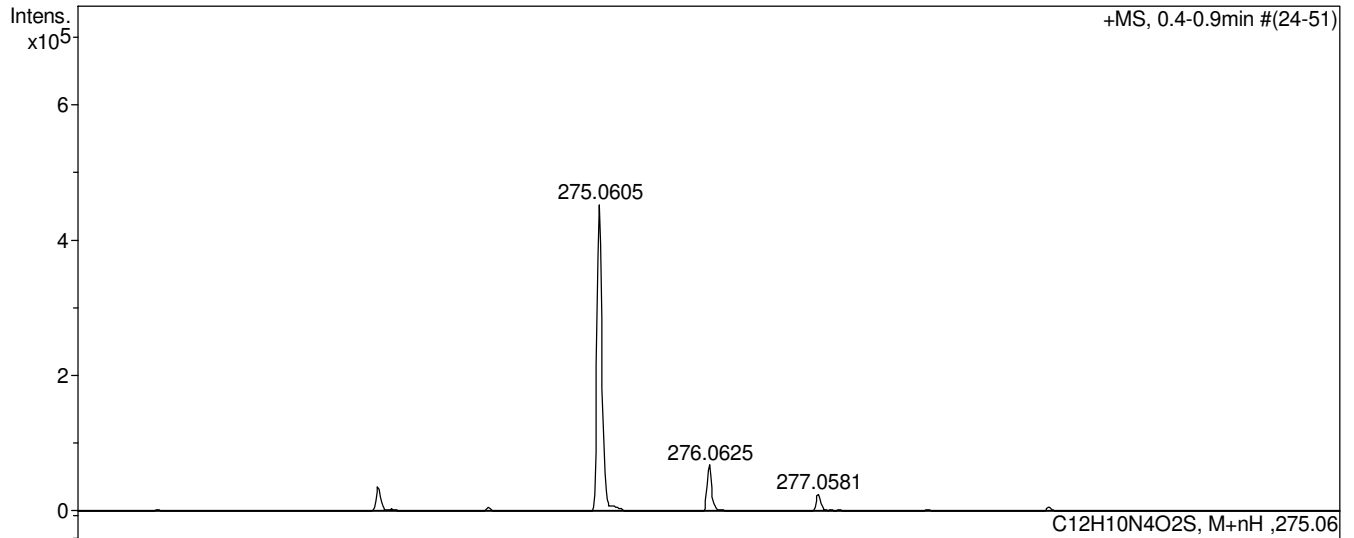
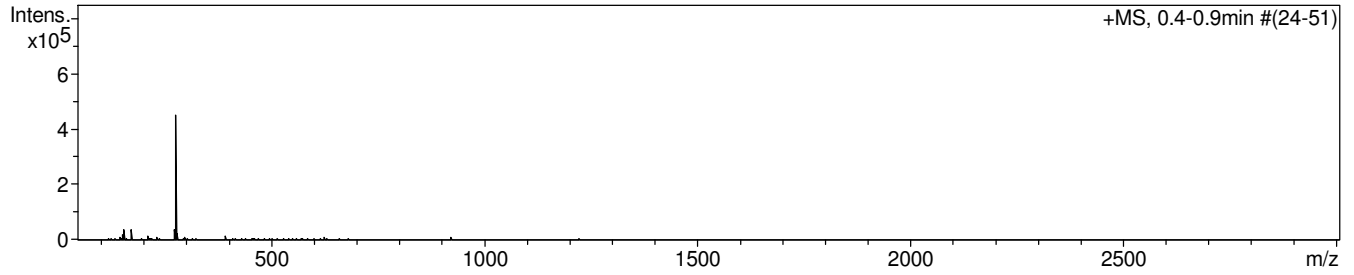
**Analysis Info**

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\0716003.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV361  
 Comment C12H10N4O2S mH275.0597 calibrant added CH3CN

Acquisition Date 16.07.2024 10:25:09  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

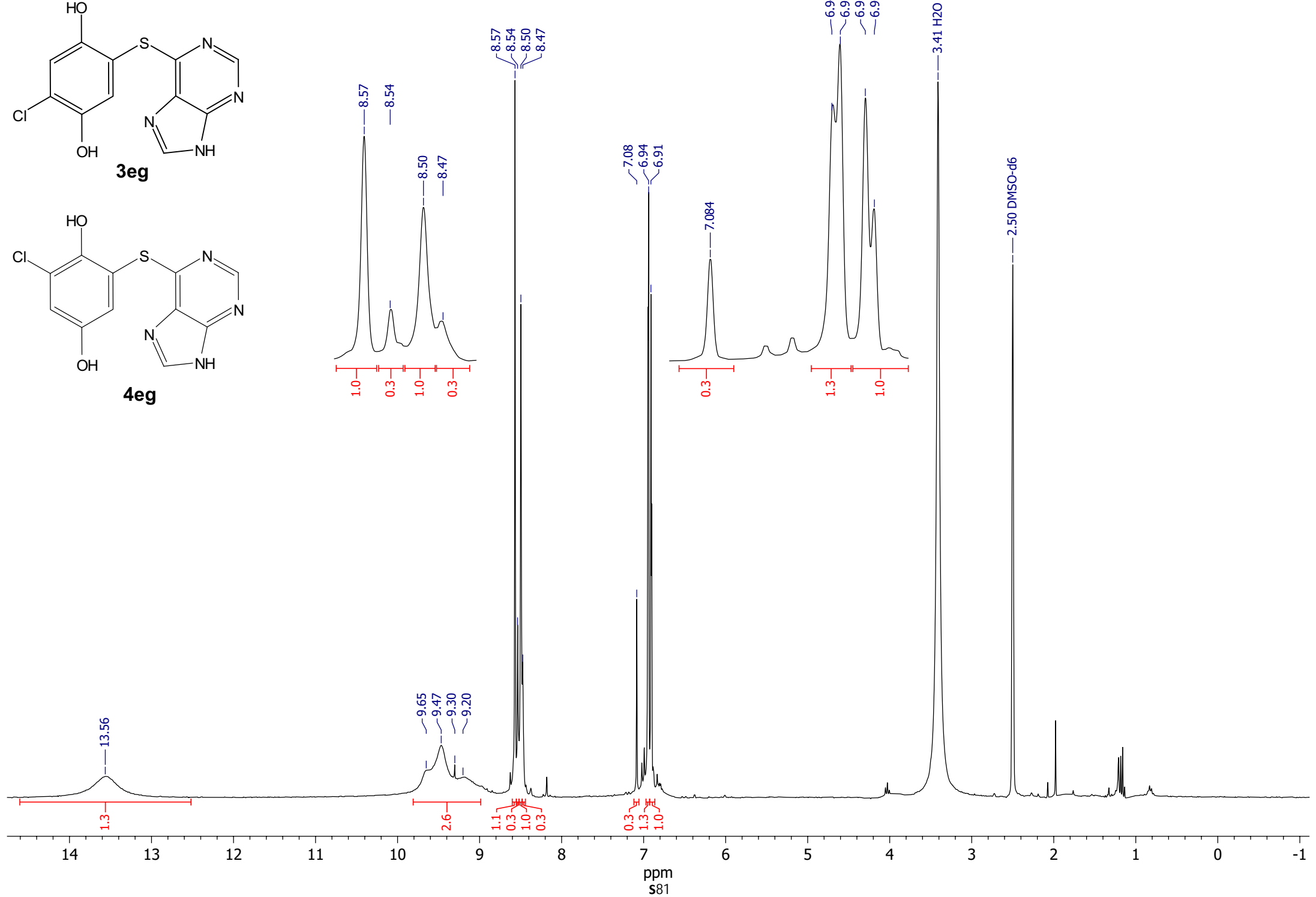
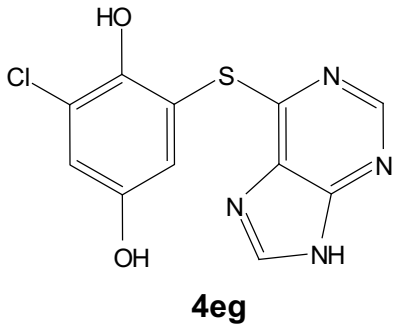
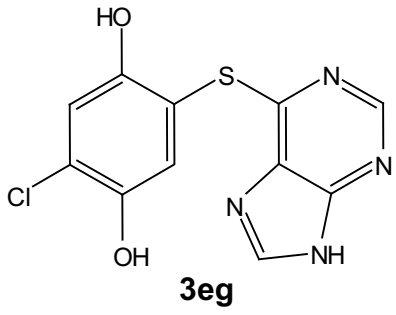
**Acquisition Parameter**

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

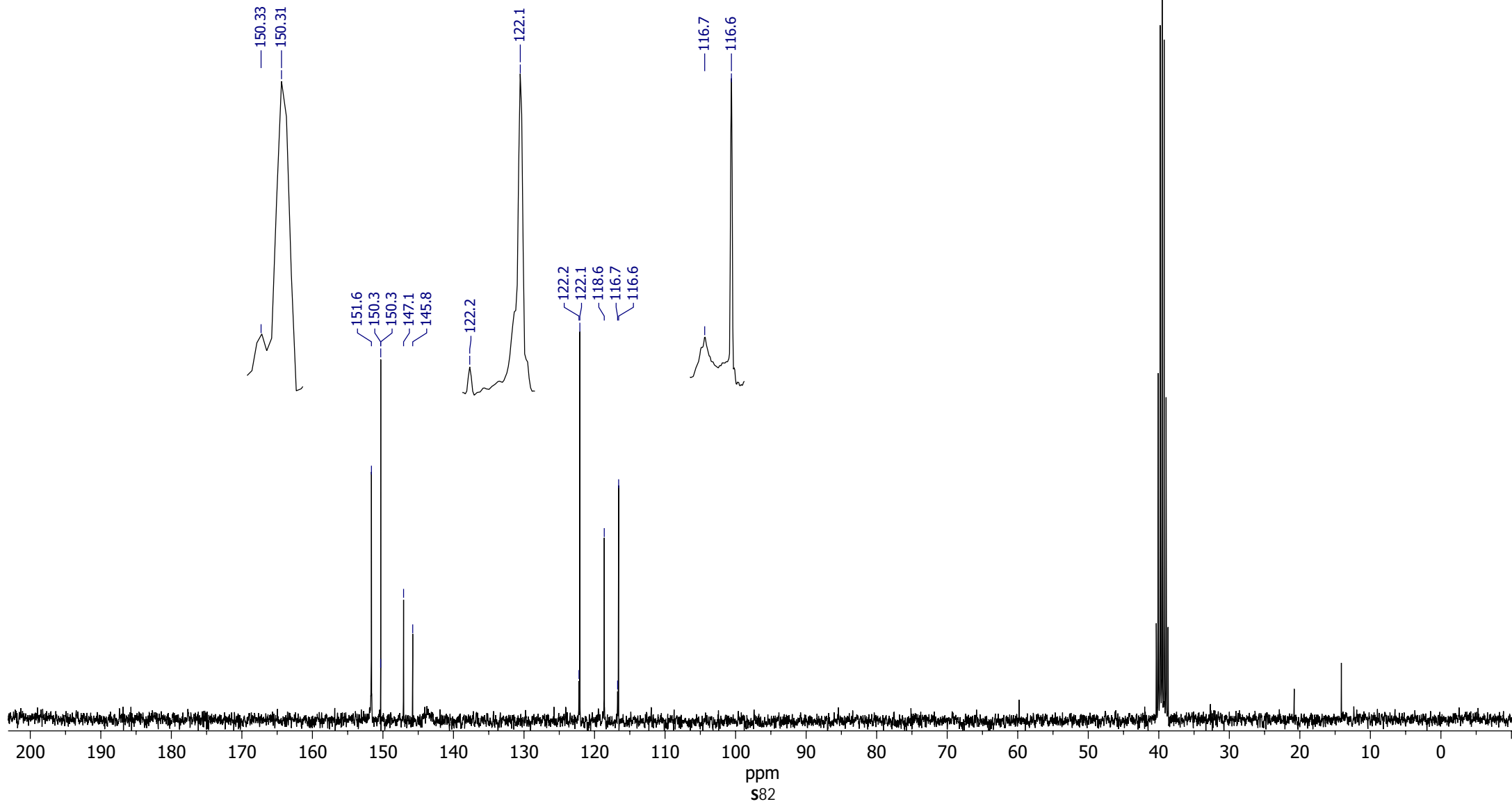
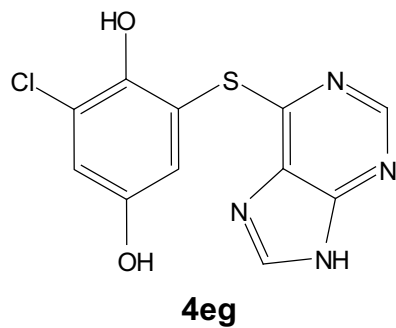
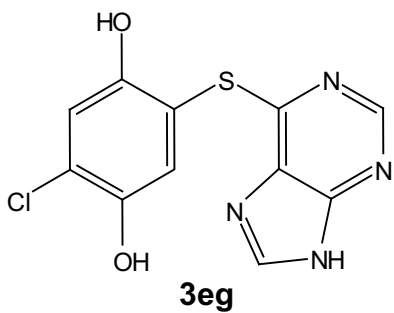


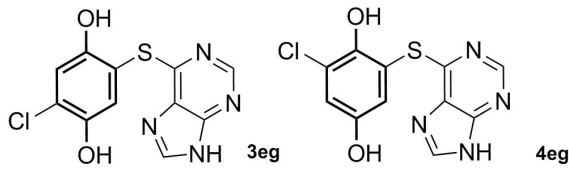


<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)





Chemical Formula: C<sub>11</sub>H<sub>7</sub>ClN<sub>4</sub>O<sub>2</sub>S  
 Exact Mass: 294,00

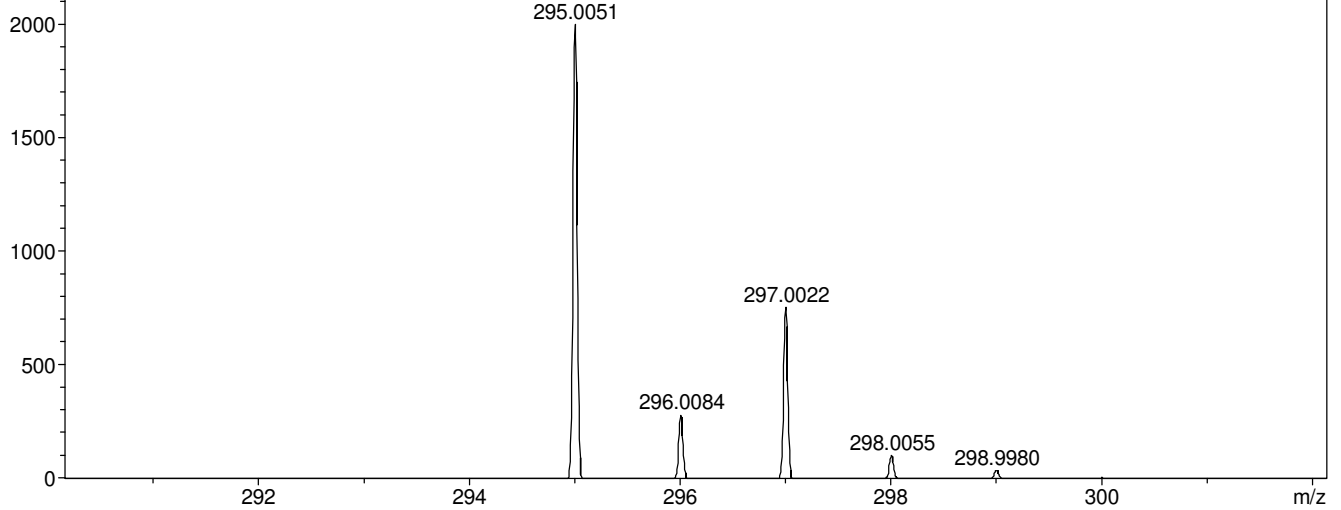
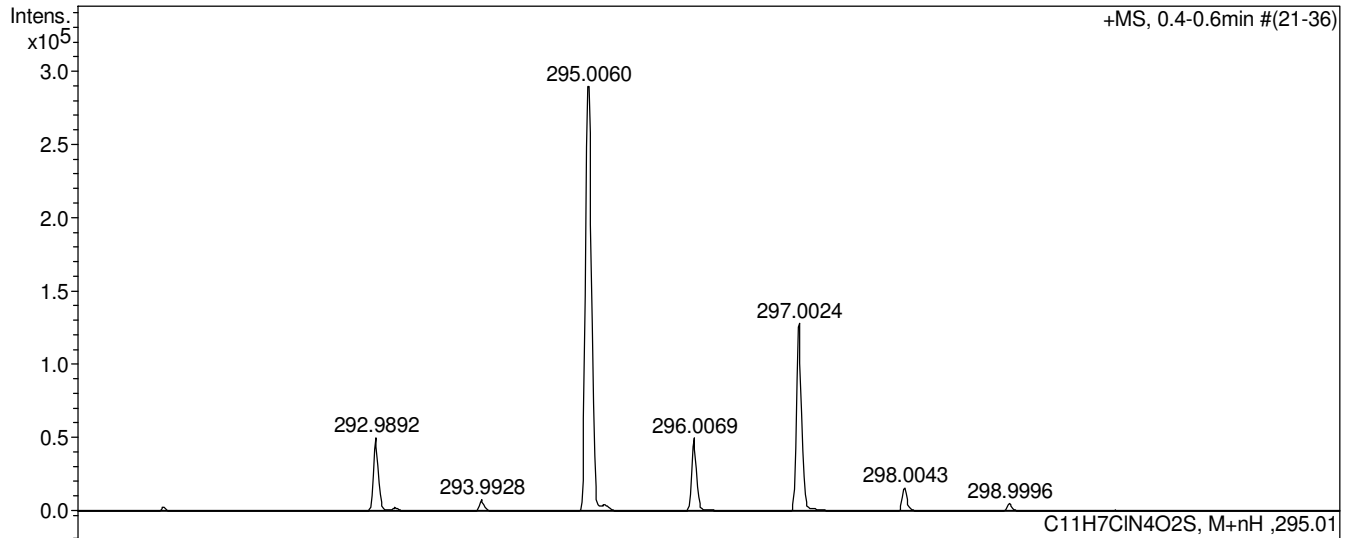
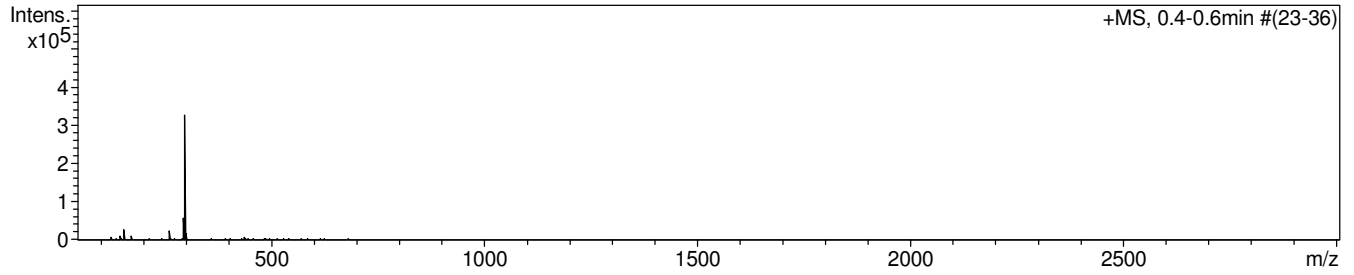
**Analysis Info**

Analysis Name D:\Data\Kolotyrykina\2024\Moiseeva\0716009.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV391  
 Comment C11H7ClN4O2S mH295.0051 calibrant added CH3CN

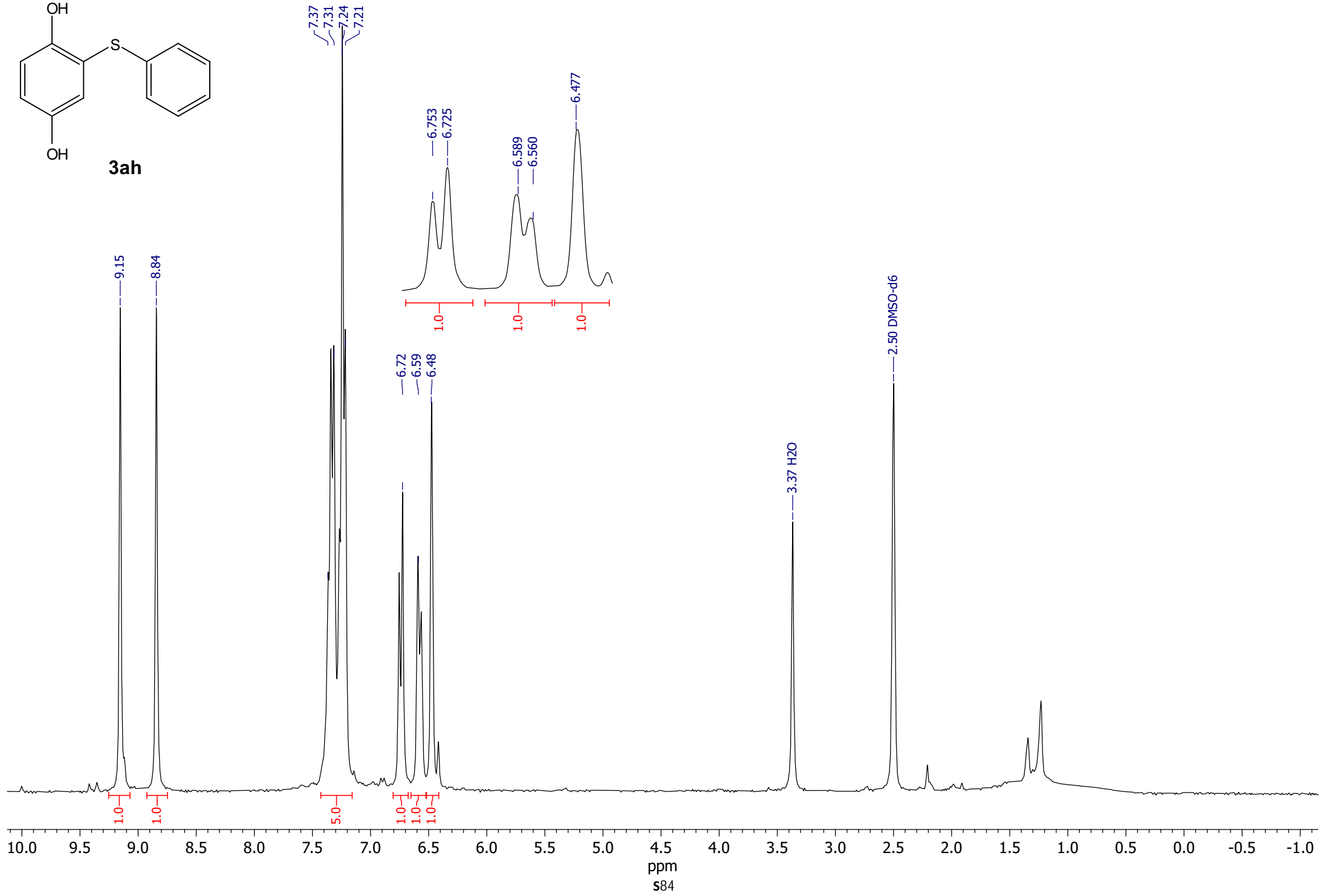
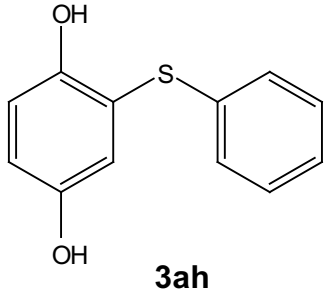
Acquisition Date 16.07.2024 11:03:48  
 Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

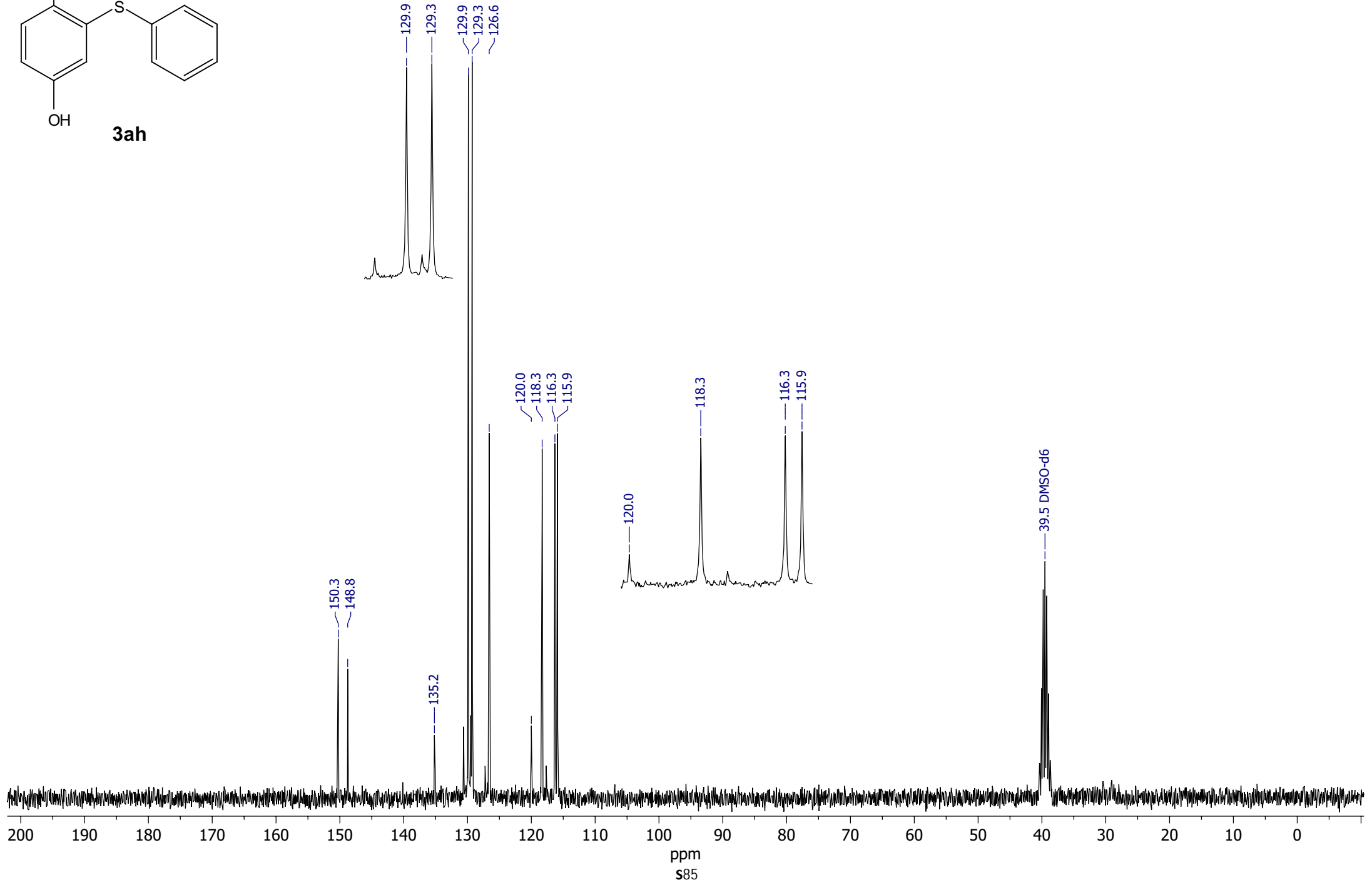
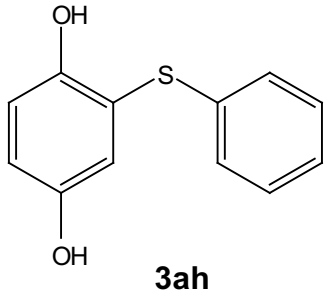
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

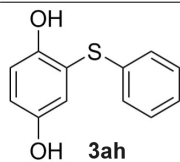


<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)





Chemical Formula: C<sub>12</sub>H<sub>10</sub>O<sub>2</sub>S  
Exact Mass: 218,04

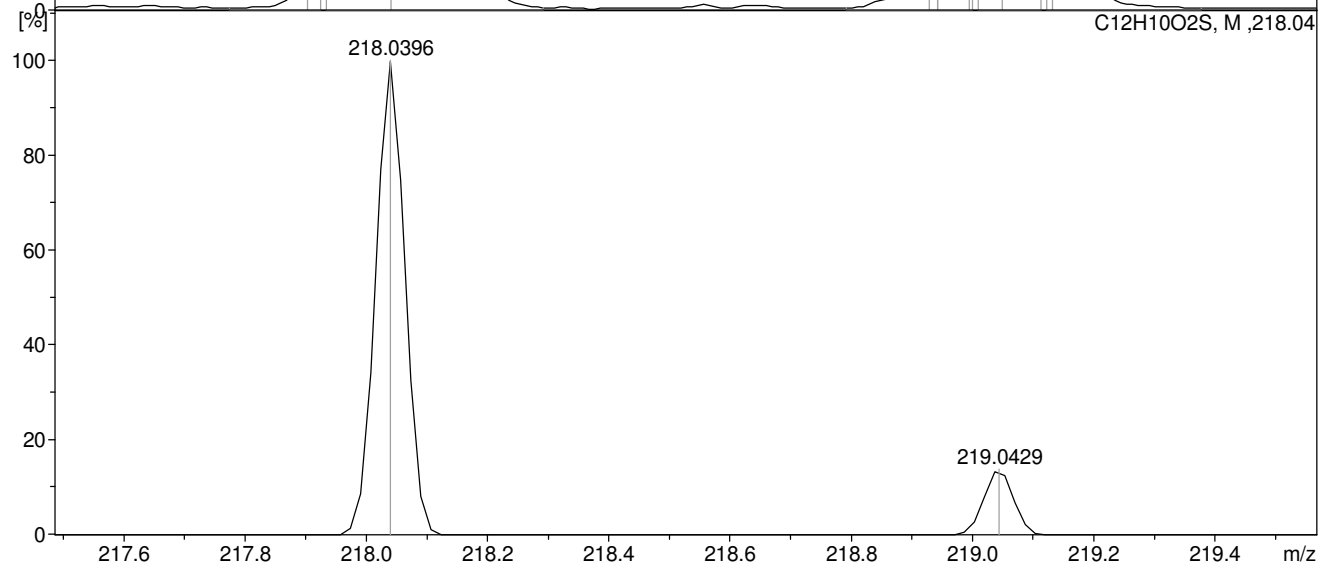
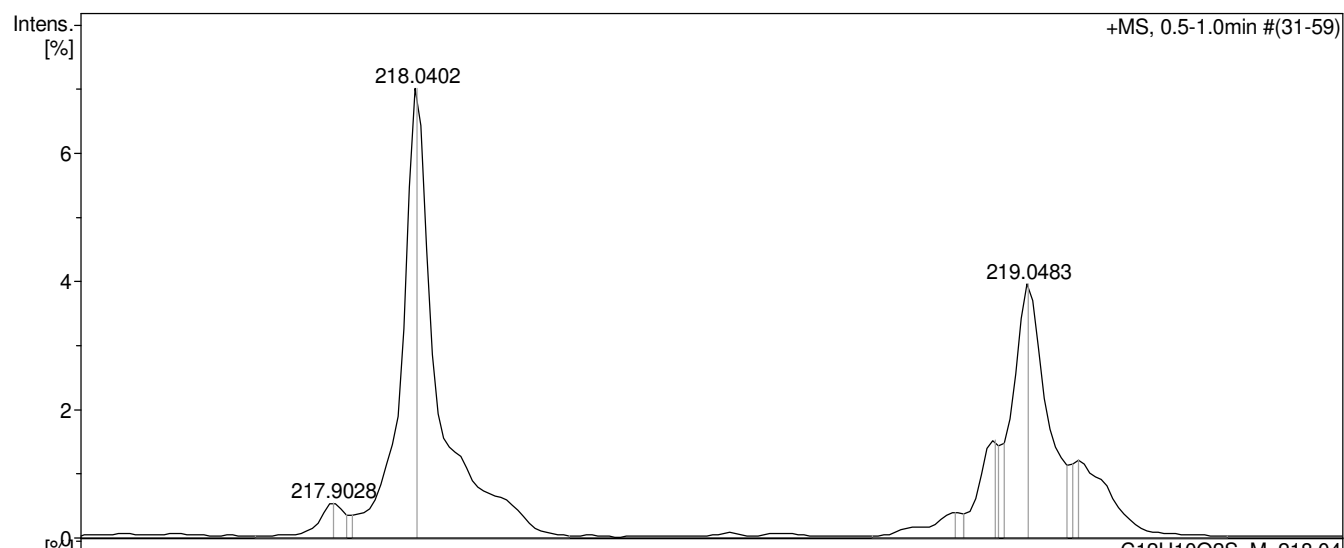
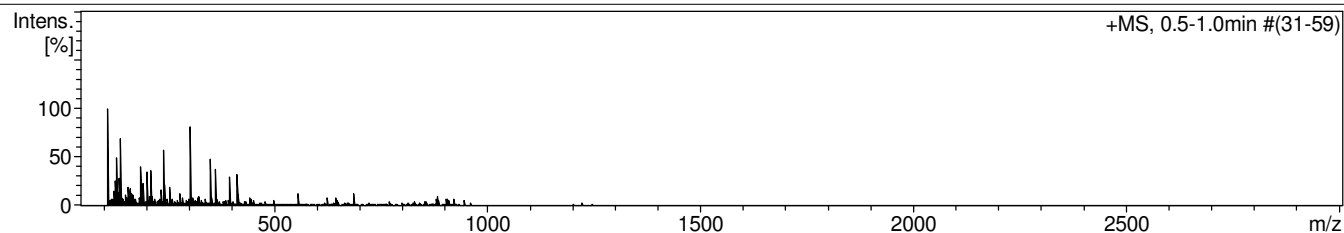
### Analysis Info

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\1128050.d  
Method tune\_low.m  
Sample Name /VAPP MNV406  
Comment C12H10O2S clb added CH3OH

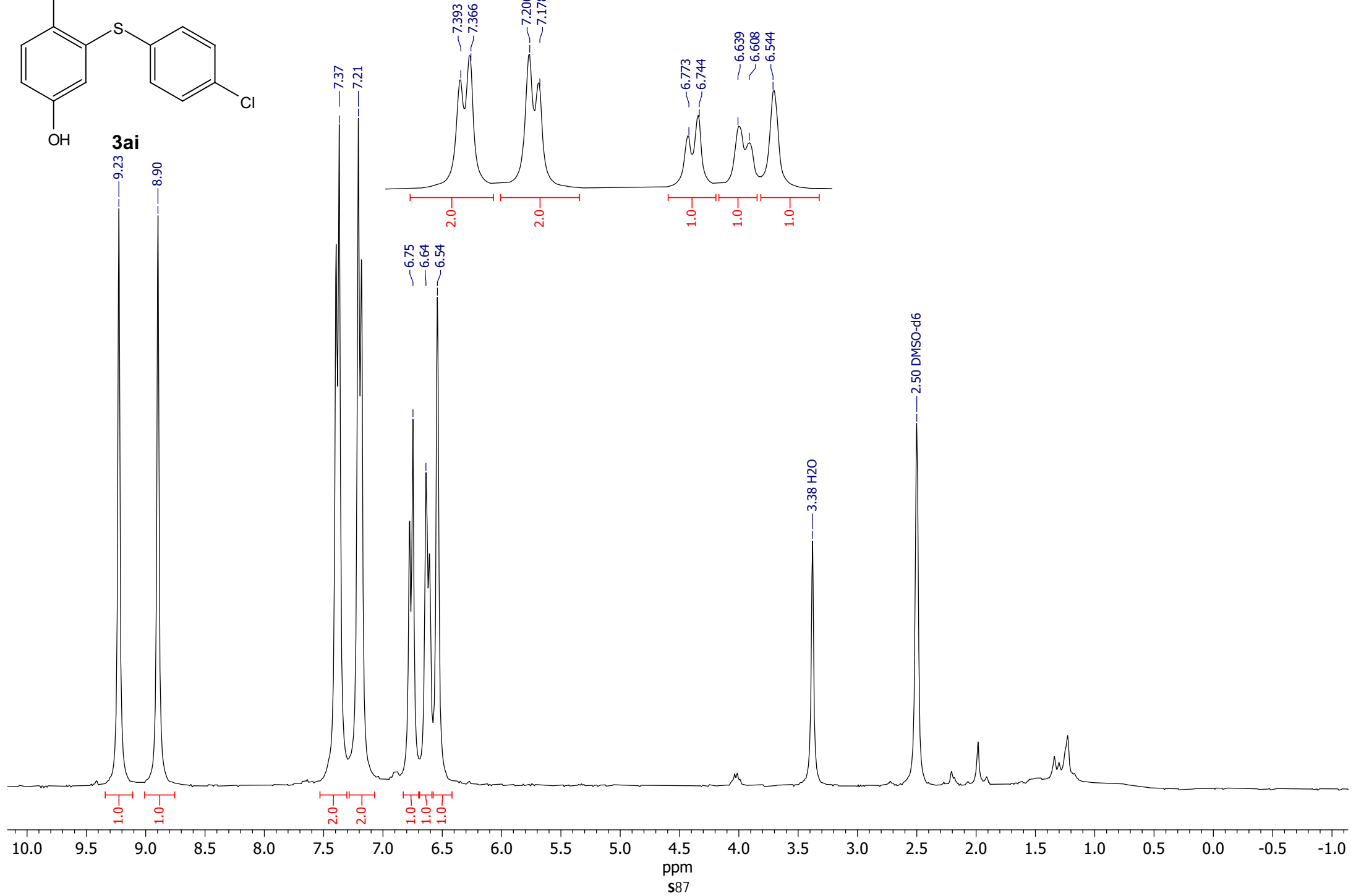
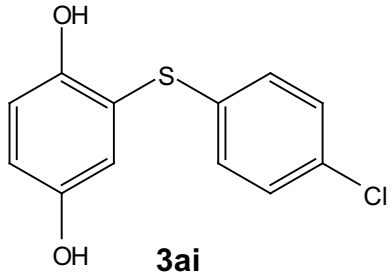
Acquisition Date 28.11.2024 18:31:46  
Operator BDAL@DE  
Instrument / Ser# micrOTOF 10248

### Acquisition Parameter

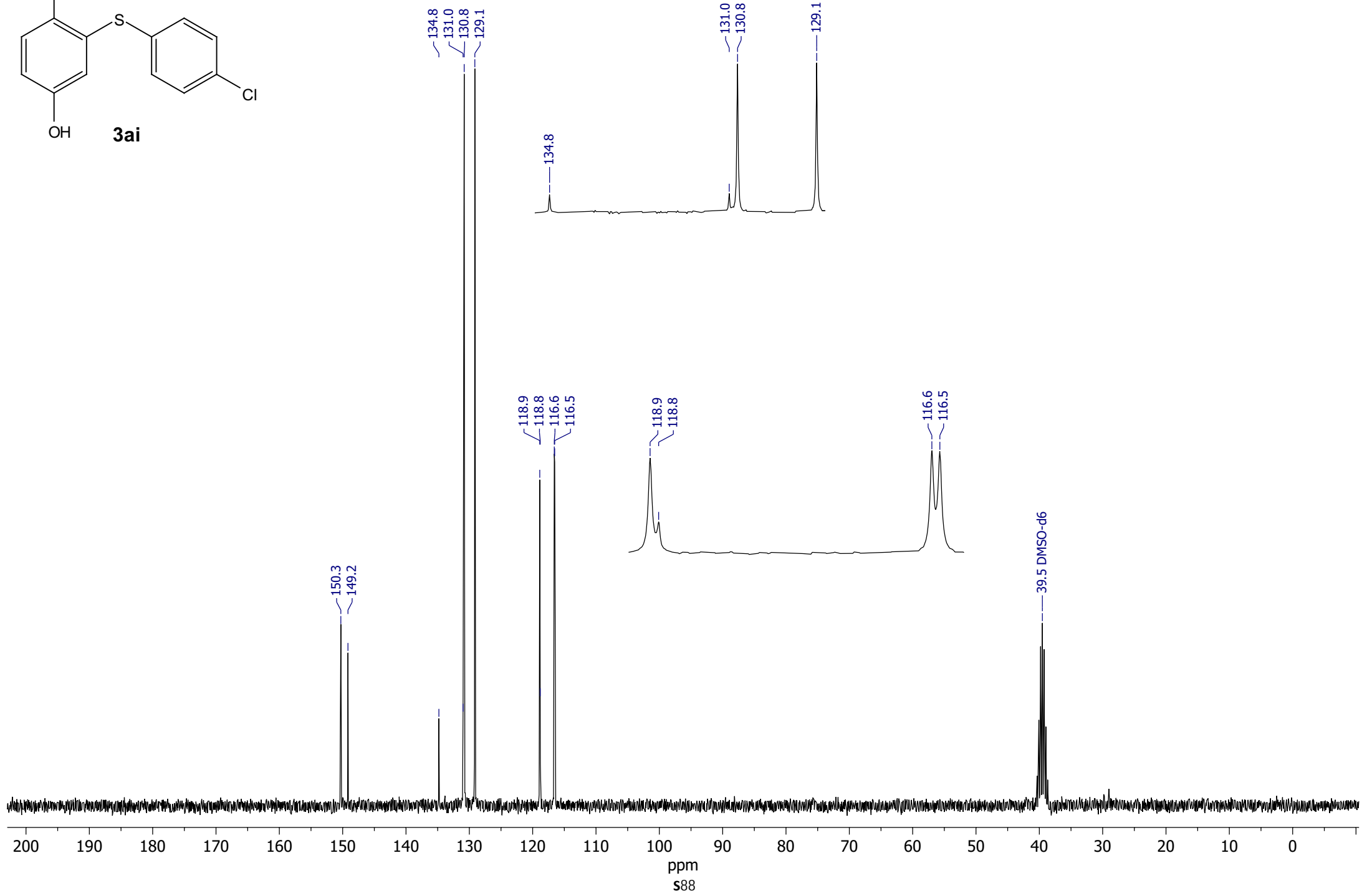
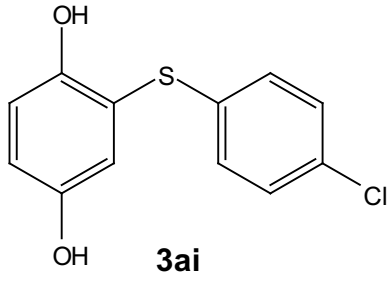
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



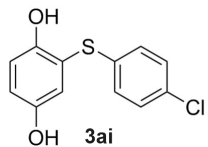
<sup>1</sup>H NMR (300.13 MHz, DMSO-d<sub>6</sub>)



<sup>13</sup>C NMR (75.48 MHz, DMSO-d<sub>6</sub>)







Chemical Formula: C<sub>12</sub>H<sub>9</sub>ClO<sub>2</sub>S  
 Exact Mass: 252,00

**Analysis Info**

Analysis Name D:\Data\Kolotyrkina\2024\Moiseeva\1128055.d  
 Method tune\_low.m  
 Sample Name /VAPP MNV401  
 Comment C12H9ClO2S clb added CH3CN

Acquisition Date 28.11.2024 19:12:26

Operator BDAL@DE  
 Instrument / Ser# micrOTOF 10248

**Acquisition Parameter**

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

