

## Supplementary Information

### For

## General and Versatile Electrochemical $\alpha$ -C(sp<sup>3</sup>)-H Amidation for N-Mannich Bases Synthesis

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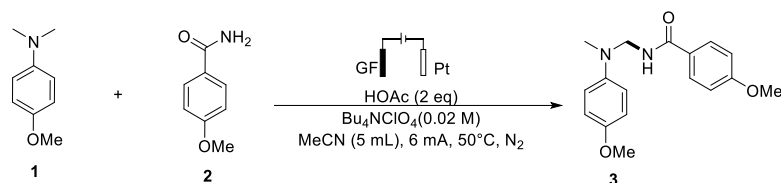
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## 1. General informations

All the reagents and solvents were purchased from commercial suppliers and used without purification unless otherwise noted. All the electrochemical reactions were performed in a Schlenk tube and monitored by TLC. Flash column chromatography was performed with silica gel (200–300 mesh). Cyclic voltammograms were recorded on a CHI 760E potentiostat in a three-electrode cell configuration with a glassy carbon working electrode (3 mm diameter) and a platinum wire counter electrode versus a SCE reference electrode. NMR spectra were recorded on a Bruker AV-400 instrument. Data were reported as chemical shifts in ppm relative to TMS (0.00 ppm) for  $^1\text{H}$  and  $\text{CDCl}_3$  (77.2 ppm). The abbreviations used for explaining the multiplicities were as follows: s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, m = multiplet. High-resolution mass spectra (HRMS) were recorded on a Bruker Esquire LC 6000 ion trap mass spectrometer using electrospray ionization.

## 2. Optimization of reaction conditions

**Table S1.** Optimization of reaction conditions.<sup>a</sup>



| Entry           | Deviation from standard conditions                                                 | Yield (%) <sup>b</sup> |
|-----------------|------------------------------------------------------------------------------------|------------------------|
| 1               | None                                                                               | 91                     |
| 2               | NO HOAc                                                                            | NR                     |
| 3               | TFA, HCO <sub>2</sub> H instead of HOAc                                            | Trace                  |
| 4               | PhCO <sub>2</sub> H, pivalic acid instead of HOAc                                  | 86, 53                 |
| 5               | TBABF <sub>4</sub> , Bu <sub>4</sub> NPF <sub>6</sub> , Bu <sub>4</sub> NOAc, TBAI | 60-85                  |
| 6 <sup>c</sup>  | DCM, DCE, Acetone as solvent                                                       | 65, 73, 42             |
| 7               | MeCN/DMSO, MeCN/DME (1/4)                                                          | Trace                  |
| 8               | rt, 60 °C, 70 °C                                                                   | 46, 88, 74             |
| 9               | GF(+) SST (-), GF(+) Ni(-), GF(+) Cu(-),                                           | 53, 72, 70             |
| 10 <sup>c</sup> | C(+) Pt (-)                                                                        | trace                  |
| 11 <sup>d</sup> | Pt(+) Pt (-)                                                                       | 26                     |
| 12              | open to air                                                                        | 81                     |
| 13              | No electricity                                                                     | NR                     |

<sup>a</sup>Reaction conditions: **1** (0.6 mmol), **2** (0.2 mmol), HOAc (0.4 mmol), Bu<sub>4</sub>NClO<sub>4</sub> (0.02 M), MeCN (5 mL), GF anode, Pt cathode, constant current (6 mA), 50 °C, N<sub>2</sub> atmosphere, 15 h (16.7 F mol<sup>-1</sup>), undivided cell.

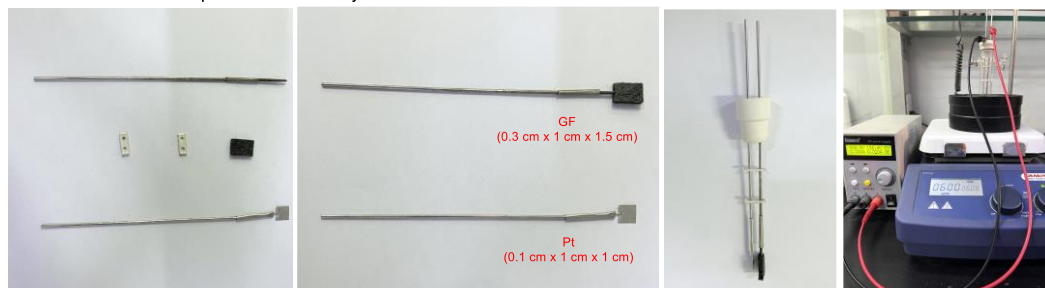
<sup>b</sup>Isolated yield. <sup>c</sup>Reaction conditions: **1** (0.5 mmol), **2** (0.25 mmol), Bu<sub>4</sub>NBF<sub>4</sub> (0.5 mmol), MeCN/HOAc (4

ml/0.5 mL), Graphite rod anode, Pt cathode, 10 mA, 60 °C, N<sub>2</sub> atmosphere. <sup>d</sup>Reaction conditions: **1** (1.0 mmol), **2** (0.5 mmol), Bu<sub>4</sub>NClO<sub>4</sub> (0.25 mmol), MeCN/MeOH (4 ml/1 mL), Pt anode, Pt cathode, 10 mA, 40 °C, N<sub>2</sub> atmosphere.

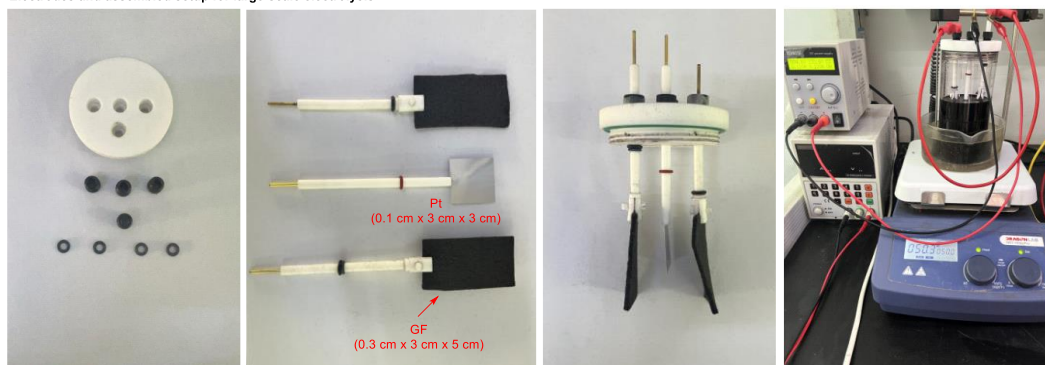
### 3. Procedures for electrolysis

**General Procedure for the electrosynthesis of N-Mannich bases.** A 10 mL dry Schlenk tube equipped with a magnetic stir bar was charged with tertiary amines (0.6 mmol, 3.0 equiv), amides (0.2 mmol), Bu<sub>4</sub>NClO<sub>4</sub> (0.02 M) in MeCN (5.0 mL). Acetic acid (0.4 mmol, 2.0 equiv) was then added via microliter syringe. The reaction vessel was fitted with a graphite felt (GF) anode (0.3 cm x 1.0 cm x 1.5 cm) and a platinum cathode (Pt) (0.1 cm x 1.0 cm x 1.0 cm). The solution was degassed by bubbling with N<sub>2</sub> for 3 minutes to remove dissolved oxygen. Constant current electrolysis was then performed at 6 mA while maintaining the reaction temperature at 50 °C for 15 hours (monitored by TLC). Upon completion, the reaction mixture was concentrated in vacuo, and the crude product was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate) to afford the desired amidation products.

Electrodes and assembled setup for small-scale electrolysis



Electrodes and assembled setup for large-scale electrolysis

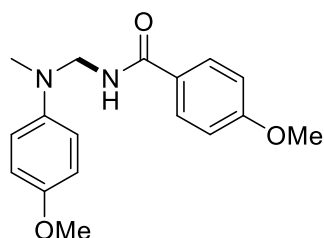


**Figure S1.** Reaction setup for electrolysis.

**Gram-scale electrosynthesis of 3.** A dry 0.5 L beaker-type electrochemical cell was charged with 4-methoxy-N, N-dimethylaniline **1** (30 mmol, 4.53 g), 4-methoxybenzylamide **2** (10

mmol, 1.51 g), Bu<sub>4</sub>NClO<sub>4</sub> (0.02 M) in MeCN (250 mL). HOAc (20 mmol) was added through syringe. Then the cell was equipped with three parallel electrodes that consist of two GF anodes (0.3 cm x 3.0 cm x 5.0 cm) and a sandwiched Pt cathode (0.1 cm x 3.0 cm x 3.0 cm). The reaction solution was degassed by N<sub>2</sub> sparging for 10 minutes to ensure complete oxygen removal. Constant current electrolysis was conducted at 120 mA while maintaining the temperature at 50 °C for 37 hours (monitored by TLC). The solution was degassed by bubbling with N<sub>2</sub> for 10 minutes to remove dissolved oxygen. Upon completion, the mixture was concentrated under reduced pressure, and the crude product was purified by flash chromatography on silica gel (PE/EA = 5/2) to afford **3** as white solid (2.43 g, 81%).

#### 4. Characterization data for products

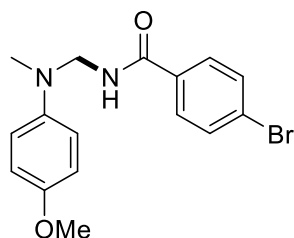


**4-methoxy-N-(((4-methoxyphenyl)(methyl)amino)methyl)benzamide (3).** White solid, mp 118.2-119.1 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 5/2, Y = 91%, (54.6 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.97 – 7.58 (m, 2H), 7.02 – 6.72 (m, 7H), 4.97 (d, *J* = 5.6 Hz, 2H), 3.78 (s, 3H), 3.72 (s, 3H), 2.94 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.56, 162.27, 152.75, 142.47, 128.92, 126.41, 115.64, 114.84, 113.69, 59.16, 55.65, 55.37, 38.23.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>17</sub>H<sub>20</sub>N<sub>2</sub>NaO<sub>3</sub> 323.1366, obsd 323.1373.

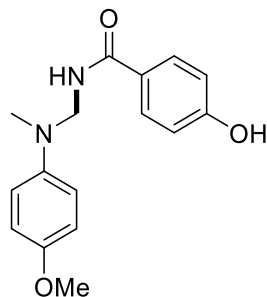


**4-bromo-N-(((4-methoxyphenyl)(methyl)amino)methyl)benzamide (4).** Brown solid, mp 73.5-74.0 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 43%, (30.0 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.66 – 7.51 (m, 4H), 6.95 – 6.82 (m, 4H), 6.48 (s, 1H), 5.06 (d, *J* = 5.6 Hz, 2H), 3.79 (s, 3H), 2.99 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.10, 153.20, 142.34, 133.15, 132.00, 128.75, 126.56, 115.95, 115.12, 59.55, 55.85, 38.48.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>16</sub>H<sub>17</sub>BrN<sub>2</sub>NaO<sub>2</sub> 371.0366, obsd 371.0366.

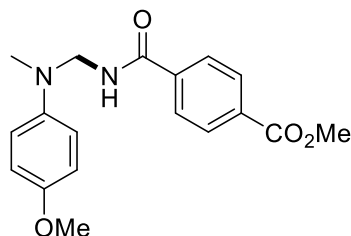


**4-hydroxy-N-(((4-methoxyphenyl)(methyl)amino)methyl)benzamide (5).** Brown oil. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/2, Y = 69%, (39.5 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.55 (d, *J* = 8.5 Hz, 2H), 6.83 (s, 4H), 6.80 (d, *J* = 8.4 Hz, 2H), 6.61 (t, *J* = 5.6 Hz, 1H), 4.99 (d, *J* = 5.6 Hz, 2H), 3.74 (s, 3H), 2.93 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 168.71, 160.38, 153.14, 142.40, 129.18, 125.19, 116.13, 115.77, 115.12, 59.63, 55.87, 38.42.

**ESI HRMS** *m/z* (M+H)<sup>+</sup> calcd for C<sub>16</sub>H<sub>19</sub>N<sub>2</sub>O<sub>3</sub> 287.1390, obsd 287.1395.

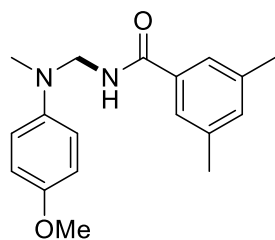


**methyl 4-(((4-methoxyphenyl)(methyl)amino)methyl)carbamoylbenzoate (6).** Brown solid, mp 78.3-79.1 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 5/2, Y = 95%, (62.3 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.02 (d, *J* = 7.5 Hz, 2H), 7.76 (d, *J* = 8.4 Hz, 2H), 6.85 (s, 4H), 6.81 – 6.74 (m, 1H), 5.03 (d, *J* = 5.6 Hz, 2H), 3.91 (s, 3H), 3.75 (s, 3H), 2.97 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.29, 166.37, 153.07, 142.28, 138.22, 132.89, 129.90, 127.19, 115.82, 115.01, 59.50, 55.77, 52.53, 38.43.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>18</sub>H<sub>20</sub>N<sub>2</sub>NaO<sub>4</sub> 351.1315, obsd 351.1324.

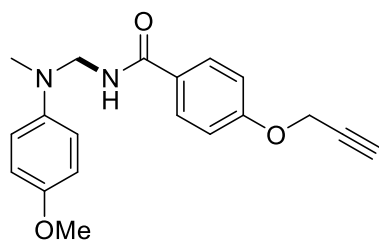


**N-(((4-methoxyphenyl)(methyl)amino)methyl)-3,5-dimethylbenzamide (7).** Brown solid, mp 89.7-90.6 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 95%, (56.6 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.32 (s, 2H), 7.10 (s, 1H), 6.85 (s, 4H), 6.58 (t, *J* = 5.4 Hz, 1H), 5.02 (d, *J* = 5.7 Hz, 2H), 3.75 (s, 3H), 2.97 (s, 3H), 2.31 (s, 6H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 168.43, 152.94, 142.50, 138.38, 134.27, 133.37, 124.85, 115.76, 114.99, 59.23, 55.78, 38.33, 21.31.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>18</sub>H<sub>22</sub>N<sub>2</sub>NaO<sub>2</sub> 321.1573, obsd 321.1578.



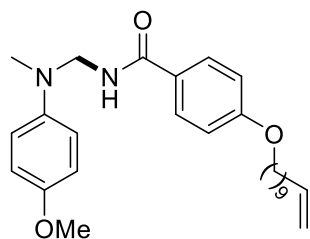
**N-(((4-methoxyphenyl)(methyl)amino)methyl)-4-(prop-2-yn-1-yloxy)benzamide (8).**

Brown oil. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 5/2, Y = 84%, (54.4 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.70 (d, *J* = 8.8 Hz, 2H), 6.97 (d, *J* = 8.9 Hz, 2H), 6.86 (s, 4H), 6.44 (s, 1H), 5.03 (d, *J* = 5.7 Hz, 2H), 4.72 (d, *J* = 2.5 Hz, 2H), 3.77 (s, 3H), 2.97 (s, 3H), 2.53 (d, *J* = 2.4 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.40, 160.29, 153.09, 142.49, 128.94, 127.46, 115.95, 115.08, 114.85, 78.02, 76.22, 59.37, 55.97, 55.85, 38.43.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>19</sub>H<sub>20</sub>N<sub>2</sub>NaO<sub>3</sub> 347.1366, obsd 347.1368.



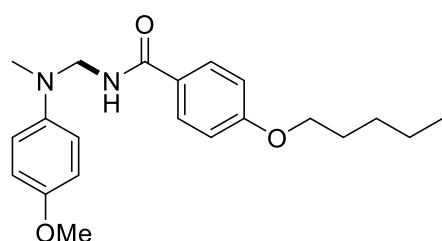
**N-(((4-methoxyphenyl)(methyl)amino)methyl)-4-(undec-10-en-1-yloxy)benzamide (9).**

White solid, mp 80.5-81.4 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 85%, (74.5 mg).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.67 (d, *J* = 8.8 Hz, 2H), 6.89 – 6.83 (m, 6H), 6.50 (q, *J* = 4.9, 4.4 Hz, 1H), 5.81 (ddt, *J* = 16.9, 10.1, 6.6 Hz, 1H), 5.16 – 4.80 (m, 4H), 3.96 (t, *J* = 6.6 Hz, 2H), 3.76 (s, 3H), 2.96 (s, 3H), 2.04 (q, *J* = 7.0 Hz, 2H), 1.77 (p, *J* = 6.8 Hz, 2H), 1.53 – 1.21 (m, 12H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.56, 162.07, 153.00, 142.56, 139.35, 128.92 (2C), 126.23, 115.87 (2C), 115.03 (2C), 114.35 (2C), 114.29, 68.31, 59.29, 55.80, 38.37, 33.93, 29.62, 29.54, 29.47, 29.24, 29.04 (2C), 26.10.

ESI HRMS *m/z* (M+H)<sup>+</sup> calcd for C<sub>27</sub>H<sub>39</sub>N<sub>2</sub>O<sub>3</sub> 439.2955, obsd 439.2952.

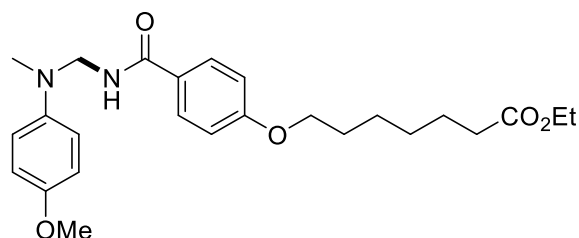


**N-(((4-methoxyphenyl)(methyl)amino)methyl)-4-(pentyloxy)benzamide (10).** Brown solid, mp 79.7-80.7 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 96%, (68.4 mg).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.67 (d, *J* = 8.8 Hz, 2H), 6.96 – 6.76 (m, 6H), 6.63 – 6.49 (m, 1H), 5.01 (d, *J* = 5.6 Hz, 2H), 3.96 (t, *J* = 6.6 Hz, 2H), 3.75 (s, 3H), 2.95 (s, 3H), 1.78 (dt, *J* = 14.5, 6.8 Hz, 2H), 1.40 (dddd, *J* = 21.4, 15.4, 8.3, 5.4 Hz, 4H), 0.92 (t, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.58, 162.04, 152.95, 142.55, 128.92, 126.21, 115.83, 115.00, 114.32, 68.27, 59.26, 55.78, 38.35, 28.92, 28.23, 22.55, 14.13.

ESI HRMS *m/z* (M+Na)<sup>+</sup> calcd for C<sub>21</sub>H<sub>28</sub>N<sub>2</sub>NaO<sub>3</sub> 379.1992, obsd 379.1997.



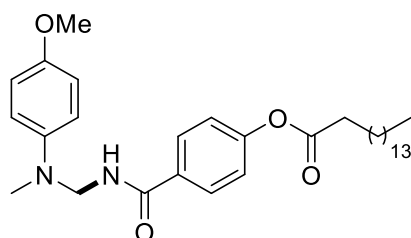
**ethyl 7-(4-(((4-methoxyphenyl)(methyl)amino)methyl)carbamoyl)phenoxy)heptanoate (11).** Brown solid, mp 82.3-83.1 °C. The title compound was prepared according to general

procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 5/2, Y = 97%, (79.6 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.68 (d, *J* = 8.8 Hz, 1H), 6.96 – 6.79 (m, 6H), 6.62 (t, *J* = 5.7 Hz, 1H), 5.01 (d, *J* = 5.7 Hz, 2H), 4.11 (q, *J* = 7.1 Hz, 2H), 3.95 (t, *J* = 6.4 Hz, 2H), 3.75 (s, 3H), 2.96 (s, 3H), 2.30 (t, *J* = 7.5 Hz, 2H), 1.78 (p, *J* = 6.7 Hz, 2H), 1.65 (p, *J* = 7.5 Hz, 2H), 1.52 – 1.43 (m, 2H), 1.42 – 1.34 (m, 2H), 1.24 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 173.86 (C=O), 167.55 (C=O), 161.93 (Cq), 152.90 (Cq), 142.52 (Cq), 128.92 (CH, 2C), 126.25 (Cq), 115.78 (CH, 2C), 114.94 (CH, 2C), 114.26 (CH, 2C), 68.05 (CH<sub>2</sub>), 60.33 (CH<sub>2</sub>), 59.23 (CH<sub>2</sub>), 55.74 (OCH<sub>3</sub>), 38.32(NCH<sub>3</sub>), 34.31 (CH<sub>2</sub>), 28.99 (CH<sub>2</sub>), 28.90 (CH<sub>2</sub>), 25.76 (CH<sub>2</sub>), 24.91 (CH<sub>2</sub>), 14.34 (CH<sub>3</sub>).

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>25</sub>H<sub>34</sub>N<sub>2</sub>NaO<sub>5</sub> 465.2360, obsd 465.2366.

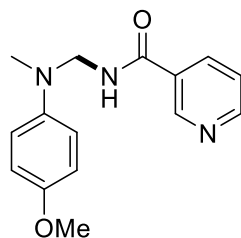


**4-((((4-methoxyphenyl)(methylamino)methyl)carbamoyl)phenyl) palmitate (12).** White solid, mp 92.5-93.4 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 64%, (45.6 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.72 (d, *J* = 8.5 Hz, 2H), 7.10 (d, *J* = 8.6 Hz, 2H), 6.85 (s, 4H), 6.54 (t, *J* = 5.7 Hz, 1H), 5.02 (d, *J* = 5.6 Hz, 2H), 3.76 (s, 3H), 2.96 (s, 3H), 2.55 (t, *J* = 7.5 Hz, 2H), 1.74 (p, *J* = 7.4 Hz, 2H), 1.47 – 1.17 (m, 24H), 0.88 (t, *J* = 6.8 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.08, 167.21, 153.47, 153.15, 142.38, 131.79, 128.58, 121.94, 115.99, 115.07, 59.47, 55.82, 38.42, 34.51, 32.08, 29.84, 29.83, 29.81, 29.75, 29.60, 29.51, 29.39, 29.23, 24.99, 22.85, 14.28.

**ESI HRMS** *m/z* (M+H)<sup>+</sup> calcd for C<sub>32</sub>H<sub>49</sub>N<sub>2</sub>O<sub>4</sub> 525.3687, obsd 525.3682.



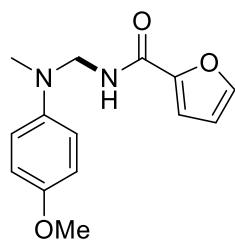
**N-((((4-methoxyphenyl)(methylamino)methyl)nicotinamide (13).** Brown solid, mp 71.4-72.4 °C. The title compound was prepared according to general procedure, electricity =

16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 2/3, Y = 40%, (21.7 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.87 (d, *J* = 2.2 Hz, 1H), 8.63 (d, *J* = 4.8 Hz, 1H), 8.05 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.32 (dd, *J* = 8.0, 4.9 Hz, 1H), 6.96 (t, *J* = 5.6 Hz, 1H), 6.84 (s, 4H), 5.03 (d, *J* = 5.6 Hz, 2H), 3.75 (s, 3H), 2.97 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 166.29, 153.15, 152.38, 148.05, 142.27, 135.33, 130.12, 123.61, 115.96, 115.03, 59.52, 55.78, 38.43.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>15</sub>H<sub>17</sub>N<sub>3</sub>NaO<sub>2</sub> 294.1213, obsd 294.1217.

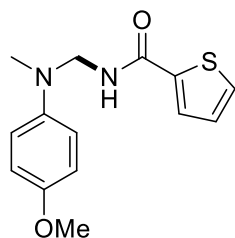


**N-(((4-methoxyphenyl)(methyl)amino)methyl)furan-2-carboxamide** Brown oil. **(14).** The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 2/1, Y = 90%, (46.8 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.45 – 7.36 (m, 1H), 7.12 (d, *J* = 3.5 Hz, 1H), 6.86 (s, 4H), 6.77 (s, 1H), 6.47 (dd, *J* = 3.5, 1.8 Hz, 1H), 5.01 (d, *J* = 5.9 Hz, 2H), 3.76 (s, 3H), 2.96 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 158.84, 153.02, 147.66, 144.15, 142.33, 115.83, 115.00, 114.84, 112.34, 58.27, 55.78, 38.28.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>14</sub>H<sub>16</sub>N<sub>2</sub>NaO<sub>3</sub> 283.1053, obsd 283.1055.

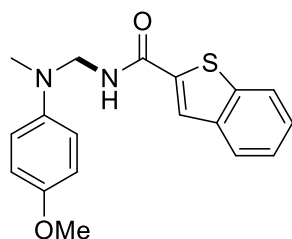


**N-(((4-methoxyphenyl)(methyl)amino)methyl)thiophene-2-carboxamide** **(15).** Brown solid, mp 72.1-73.0 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 87%, (48.0 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.46 (dt, *J* = 4.6, 1.7 Hz, 2H), 7.03 (dd, *J* = 4.9, 3.8 Hz, 1H), 6.85 (s, 4H), 6.56 (t, *J* = 5.8 Hz, 1H), 5.00 (d, *J* = 5.8 Hz, 2H), 3.76 (s, 3H), 2.96 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 162.45, 153.05, 142.39, 138.77, 130.52, 128.34, 127.77, 115.91, 115.01, 59.26, 55.80, 38.33.

**ESI HRMS**  $m/z$  ( $M+Na$ )<sup>+</sup> calcd for C<sub>14</sub>H<sub>16</sub>N<sub>2</sub>NaO<sub>2</sub>S 299.0825, obsd 299.0828.



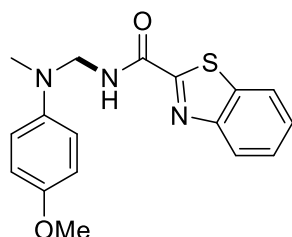
**N-(((4-methoxyphenyl)(methyl)amino)methyl)benzo[b]thiophene-2-carboxamide (16).**

Brown solid, mp 68.6-69.5 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 90%, (58.7 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.81 (d,  $J$  = 7.7 Hz, 1H), 7.75 (d,  $J$  = 7.7 Hz, 1H), 7.70 (s, 1H), 7.47 – 7.32 (m, 2H), 6.86 (s, 4H), 6.73 (t,  $J$  = 5.8 Hz, 1H), 5.03 (d,  $J$  = 5.7 Hz, 2H), 3.75 (s, 3H), 2.97 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 162.94, 153.15, 142.33, 141.09, 139.14, 138.29, 126.55, 125.47, 125.20, 125.05, 122.82, 116.01, 115.05, 59.49, 55.79, 38.39.

**ESI HRMS**  $m/z$  ( $M+Na$ )<sup>+</sup> calcd for C<sub>18</sub>H<sub>18</sub>N<sub>2</sub>NaO<sub>2</sub>S 349.0981, obsd 349.0988.



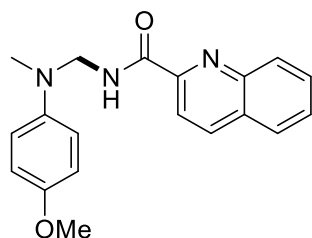
**N-(((4-methoxyphenyl)(methyl)amino)methyl)benzo[d]thiazole-2-carboxamide (17).**

Brown solid, mp 130.5-131.2 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/2, Y = 86%, (56.3 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.07 (s, 1H), 8.40 (d,  $J$  = 1.7 Hz, 1H), 8.09 (d,  $J$  = 8.5 Hz, 1H), 7.80 (d,  $J$  = 8.4 Hz, 1H), 6.87 (s, 4H), 6.75 (t,  $J$  = 5.8 Hz, 1H), 5.08 (d,  $J$  = 5.6 Hz, 2H), 3.76 (s, 3H), 3.00 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.43, 156.85, 155.27, 153.14, 142.36, 134.25, 131.72, 124.83, 123.67, 121.88, 115.92, 115.09, 59.64, 55.82, 38.49.

**ESI HRMS**  $m/z$  ( $M+Na$ )<sup>+</sup> calcd for C<sub>17</sub>H<sub>17</sub>N<sub>3</sub>NaO<sub>2</sub>S 350.0934, obsd 350.0935.

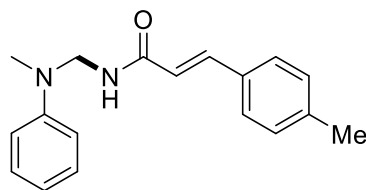


**N-(((4-methoxyphenyl)(methyl)amino)methyl)quinoline-2-carboxamide (18).** Brown solid, mp 73.0-73.7 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 60%, (38.5 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.64 (t, *J* = 6.2 Hz, 1H), 8.30 (s, 2H), 8.06 (d, *J* = 8.5 Hz, 1H), 7.86 (dd, *J* = 8.3, 1.4 Hz, 1H), 7.74 (ddd, *J* = 8.5, 6.9, 1.5 Hz, 1H), 7.60 (ddd, *J* = 8.2, 6.9, 1.2 Hz, 1H), 7.02 – 6.79 (m, 4H), 5.13 (d, *J* = 6.1 Hz, 2H), 3.78 (s, 3H), 3.03 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 165.12, 152.97, 149.54, 146.58, 142.68, 137.69, 130.26, 129.90, 129.52, 128.15, 127.88, 119.05, 115.87, 115.01, 58.88, 55.85, 38.40.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>NaO<sub>2</sub> 344.1369, obsd 344.1376.

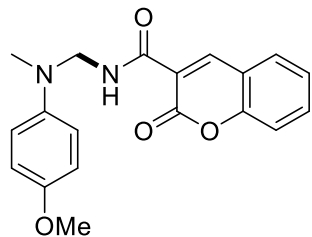


**(E)-N-((methyl(phenyl)amino)methyl)-3-(p-tolyl)acrylamide (19).** White solid, mp 98.4-99.2 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 5/2, Y = 47%, (26.3 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.61 (d, *J* = 15.6 Hz, 1H), 7.35 (d, *J* = 8.1 Hz, 2H), 7.32 – 7.22 (m, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 6.95 – 6.74 (m, 3H), 6.30 (d, *J* = 15.6 Hz, 1H), 6.18 (s, 1H), 5.03 (d, *J* = 5.7 Hz, 2H), 3.02 (s, 3H), 2.34 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 166.66, 148.03, 141.91, 140.30, 132.00, 129.70, 129.65, 127.98, 119.27, 118.30, 113.38, 57.67, 38.11, 21.56.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>18</sub>H<sub>20</sub>N<sub>2</sub>NaO 303.1468, obsd 303.1472.



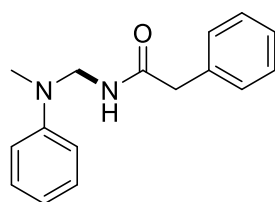
**N-(((4-methoxyphenyl)(methyl)amino)methyl)-2-oxo-2H-chromene-3-carboxamide. (20).**

Brown solid, mp 124.5-125.3 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 2/1, Y = 95%, (64.2 mg).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.17 (t, *J* = 5.8 Hz, 1H), 8.89 (s, 1H), 7.65 (ddd, *J* = 13.9, 7.7, 1.5 Hz, 2H), 7.36 (t, *J* = 7.8 Hz, 2H), 6.94 – 6.81 (m, 4H), 5.03 (d, *J* = 5.7 Hz, 2H), 3.75 (s, 3H), 3.00 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 162.05, 161.37, 154.54, 152.96, 148.75, 142.38, 134.29, 129.94, 125.41, 118.65, 118.32, 116.73, 115.83, 114.90, 59.08, 55.73, 38.49.

ESI HRMS *m/z* (M+Na)<sup>+</sup> calcd for C<sub>19</sub>H<sub>18</sub>N<sub>2</sub>NaO<sub>4</sub> 361.1159, obsd 361.1161.



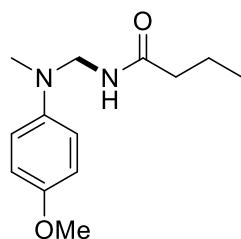
**N-((methyl(phenyl)amino)methyl)-2-phenylacetamide (21).** White solid, mp 74.8-75.5 °C.

The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 88%, (44.7 mg).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.36 – 7.14 (m, 7H), 6.79 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.76 – 6.71 (m, 2H), 5.87 (s, 1H), 4.86 (d, *J* = 5.7 Hz, 2H), 3.54 (s, 2H), 2.93 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 171.65, 147.97, 134.78, 129.55, 129.44, 129.13, 127.51, 118.43, 113.52, 57.95, 43.96, 38.05.

ESI HRMS *m/z* (M+Na)<sup>+</sup> calcd for C<sub>16</sub>H<sub>18</sub>N<sub>2</sub>NaO 277.1311, obsd 277.1318.



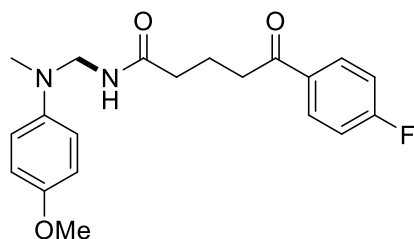
**N-(((4-methoxyphenyl)(methyl)amino)methyl)butyramide (22).** White solid, mp 66.6-67.5

°C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/2, Y = 93%, (43.9 mg).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.85 – 6.65 (m, 4H), 5.88 (s, 1H), 4.76 (d, *J* = 5.8 Hz, 2H), 3.69 (s, 3H), 2.82 (s, 3H), 2.05 (t, *J* = 7.5 Hz, 2H), 1.55 (h, *J* = 7.0 Hz, 2H), 0.84 (t, *J* = 7.4 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 173.70, 152.92, 142.44, 115.77, 114.97, 58.47, 55.80, 38.75, 38.25, 19.17, 13.84.

**ESI HRMS**  $m/z$  ( $M+Na$ )<sup>+</sup> calcd for C<sub>13</sub>H<sub>20</sub>N<sub>2</sub>NaO<sub>2</sub> 259.1417, obsd 259.1414.



**5-(4-fluorophenyl)-N-(((4-methoxyphenyl)(methyl)amino)methyl)-5-oxopentanimide**

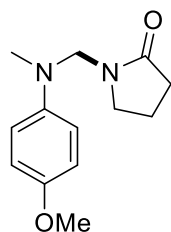
**(23).** Brown solid, mp 96.5-97.8 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 1/1, Y = 69%, (49.6 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.05 – 7.83 (m, 2H), 7.11 (t,  $J$  = 8.6 Hz, 2H), 6.80 (q,  $J$  = 9.2 Hz, 4H), 6.09 (t,  $J$  = 5.9 Hz, 1H), 4.84 (d,  $J$  = 5.8 Hz, 2H), 3.74 (s, 3H), 2.97 (t,  $J$  = 7.0 Hz, 2H), 2.90 (s, 3H), 2.26 (t,  $J$  = 7.1 Hz, 2H), 2.07 – 2.00 (m, 2H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 198.39, 173.09, 165.89 (d,  $J$  = 254.6 Hz), 152.95, 142.26, 133.27, 133.24, 130.83 (d,  $J$  = 9.5 Hz), 115.95, 115.73, 114.97, 58.52, 55.77, 38.38, 37.34, 35.50, 20.14.

**<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>) δ -105.12.

**ESI HRMS**  $m/z$  ( $M+Na$ )<sup>+</sup> calcd for C<sub>20</sub>H<sub>23</sub>FN<sub>2</sub>NaO<sub>3</sub> 381.1585, obsd 381.1592.

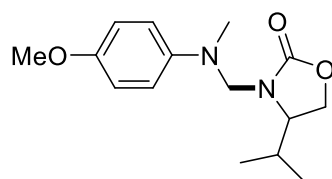


**1-(((4-methoxyphenyl)(methyl)amino)methyl)pyrrolidin-2-one (24).** Brown oil. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 1/1, Y = 79%, (37.0 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 6.83 (t,  $J$  = 7.2 Hz, 4H), 4.81 (s, 2H), 3.75 (s, 3H), 3.25 (t,  $J$  = 7.0 Hz, 2H), 2.92 (s, 3H), 2.37 (t,  $J$  = 8.1 Hz, 2H), 1.93 (p,  $J$  = 7.6 Hz, 2H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 175.60, 152.56, 142.58, 114.94, 114.80, 61.15, 55.69, 46.58, 38.43, 30.94, 17.86.

**ESI HRMS**  $m/z$  ( $M+Na$ )<sup>+</sup> calcd for C<sub>13</sub>H<sub>18</sub>N<sub>2</sub>NaO<sub>2</sub> 257.1260, obsd 257.1260.

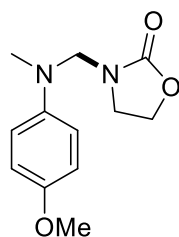


**4-isopropyl-3-(((4-methoxyphenyl)(methyl)amino)methyl)oxazolidin-2-one (25).** Brown oil. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 90%, (50.2 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 6.85 (s, 4H), 5.02 (d, *J* = 14.2 Hz, 1H), 4.58 (d, *J* = 14.2 Hz, 1H), 4.13 (t, *J* = 9.0 Hz, 1H), 4.04 (dd, *J* = 9.1, 4.9 Hz, 1H), 3.77 (s, 3H), 3.59 (dt, *J* = 8.7, 4.3 Hz, 1H), 2.91 (s, 3H), 2.04 (pd, *J* = 6.9, 6.4, 2.9 Hz, 1H), 0.83 (d, *J* = 6.9 Hz, 3H), 0.78 (d, *J* = 7.0 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 158.61(C=O), 153.32 (Cq), 142.76 (Cq), 116.37 (CH, 2C), 114.91 (CH, 2C), 62.74 (CH<sub>2</sub>), 61.61 (CH<sub>2</sub>), 58.18 (CH), 55.78 (OCH<sub>3</sub>), 38.37 (NCH<sub>3</sub>), 27.33 (CH), 17.92 (CH<sub>3</sub>), 14.13 (CH<sub>3</sub>).

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>15</sub>H<sub>22</sub>N<sub>2</sub>NaO<sub>3</sub> 301.1523, obsd 301.1527.

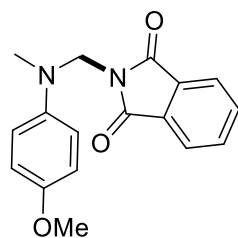


**3-(((4-methoxyphenyl)(methyl)amino)methyl)oxazolidin-2-one (26).** Brown solid, mp 163.5-164.3 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/2, Y = 90%, (42.5 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 6.85 (s, 4H), 4.83 (s, 2H), 4.25 (dd, *J* = 8.8, 7.2 Hz, 2H), 3.76 (s, 3H), 3.55 – 3.36 (m, 2H), 2.95 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 158.51, 152.97, 142.44, 115.44, 114.90, 63.63, 61.93, 55.73, 44.19, 38.28.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>12</sub>H<sub>16</sub>N<sub>2</sub>NaO<sub>3</sub> 259.1053, obsd 256.1054.

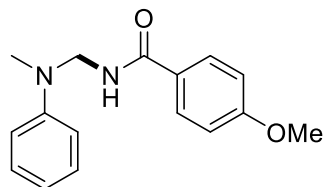


**2-(((4-methoxyphenyl)(methyl)amino)methyl)isoindoline-1,3-dione (27).** White solid, mp 146.0-146.8 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 2/1, Y = 66%, (39.1 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.83 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.70 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.09 – 6.99 (m, 2H), 6.90 – 6.82 (m, 2H), 5.20 (s, 2H), 3.75 (s, 3H), 3.07 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 168.97, 153.02, 141.96, 134.28, 132.15, 123.58, 115.98, 114.65, 57.62, 55.74, 39.28.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>17</sub>H<sub>17</sub>N<sub>2</sub>O<sub>3</sub> 297.1234, obsd 297.1237.

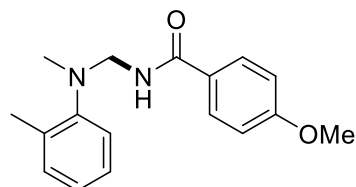


**4-methoxy-N-((methyl(phenyl)amino)methyl)benzamide (28).** White solid, mp 115.1-116.0 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 87%, (47.0 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.70 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 7.8 Hz, 2H), 6.88 (t, *J* = 9.0 Hz, 4H), 6.82 (t, *J* = 7.3 Hz, 1H), 6.50 (s, 1H), 5.11 (d, *J* = 5.6 Hz, 2H), 3.83 (s, 3H), 3.06 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.53, 162.48, 148.08, 129.68, 128.99, 126.42, 118.30, 113.90, 113.33, 58.21, 55.57, 38.16.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>16</sub>H<sub>18</sub>N<sub>2</sub>NaO<sub>2</sub> 293.1260, obsd 293.1262.

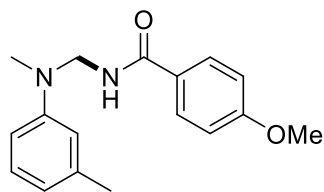


**4-methoxy-N-((methyl(o-tolyl)amino)methyl)benzamide (29).** White solid, mp 87.5-88.4 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 64%, (36.3 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.73 (d, *J* = 8.8 Hz, 2H), 7.26 – 7.17 (m, 2H), 7.11 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.05 (td, *J* = 7.4, 1.3 Hz, 1H), 6.99 – 6.89 (m, 2H), 6.30 (s, 1H), 4.73 (d, *J* = 6.1 Hz, 2H), 3.86 (s, 3H), 2.84 (s, 3H), 2.39 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.55, 162.44, 148.98, 133.08, 131.59, 128.91, 126.69, 126.62, 123.96, 121.32, 113.93, 59.91, 55.56, 39.29, 18.46.

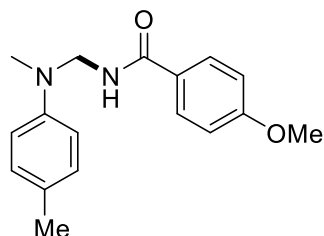
**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>17</sub>H<sub>20</sub>N<sub>2</sub>NaO<sub>2</sub> 307.1417, obsd 307.1425.



**4-methoxy-N-((methyl(m-tolyl)amino)methyl)benzamide (30).** White solid, mp 92.3-93.5 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 61%, (34.6 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.69 (d, *J* = 8.8 Hz, 2H), 7.17 (dd, *J* = 9.2, 7.3 Hz, 1H), 6.89 (d, *J* = 8.8 Hz, 2H), 6.76 – 6.59 (m, 3H), 6.44 (s, 1H), 5.10 (d, *J* = 5.5 Hz, 2H), 3.83 (s, 3H), 3.04 (s, 3H), 2.33 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.56, 162.53, 148.25, 139.49, 129.55, 129.00, 126.58, 119.32, 114.24, 113.94, 110.60, 58.32, 55.58, 38.14, 22.07.

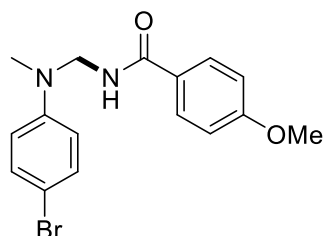
ESI HRMS *m/z* (M+Na)<sup>+</sup> calcd for C<sub>17</sub>H<sub>20</sub>N<sub>2</sub>NaO<sub>2</sub> 307.1417, obsd 307.1421.



**4-methoxy-N-((methyl(p-tolyl)amino)methyl)benzamide (31).** White solid, mp 121.9-122.7 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 82%, (46.6 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.68 (d, *J* = 8.8 Hz, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 6.79 (d, *J* = 8.6 Hz, 2H), 6.47 (s, 1H), 5.08 (d, *J* = 5.5 Hz, 2H), 3.82 (s, 3H), 3.01 (s, 3H), 2.27 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.54, 162.47, 145.97, 130.18, 128.97, 127.79, 126.52, 113.89, 113.86, 58.55, 55.56, 38.18, 20.44.

ESI HRMS *m/z* (M+Na)<sup>+</sup> calcd for C<sub>17</sub>H<sub>20</sub>N<sub>2</sub>NaO<sub>2</sub> 307.1417, obsd 307.1424.



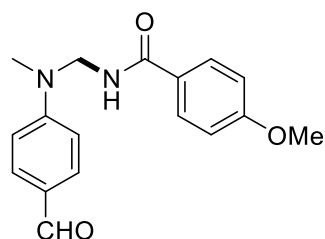
**N-(((4-bromophenyl)(methyl)amino)methyl)-4-methoxybenzamide (32).** Brown solid, mp 103.6-104.5 °C. The title compound was prepared according to general procedure, electricity

= 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 89%, (61.9 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.69 (d, *J* = 8.8 Hz, 2H), 7.29 (d, *J* = 9.0 Hz, 2H), 7.01 – 6.79 (m, 3H), 6.70 (d, *J* = 9.0 Hz, 2H), 5.00 (d, *J* = 5.6 Hz, 2H), 3.80 (s, 3H), 3.00 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.62, 162.48, 147.08, 132.14, 129.01, 126.21, 114.92, 113.84, 110.16, 58.09, 55.51, 38.23.

**ESI HRMS** *m/z* (M+H)<sup>+</sup> calcd for C<sub>16</sub>H<sub>18</sub>BrN<sub>2</sub>O<sub>2</sub> 349.0546, obsd 349.0551.

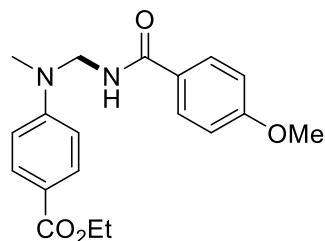


**4-formyl-N-(((4-methoxyphenyl)(methyl)amino)methyl)benzamide (33).** Brown oil. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/2, Y = 61%, (36.7 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.75 (s, 1H), 7.74 (dd, *J* = 8.9, 3.1 Hz, 4H), 6.89 (t, *J* = 8.7 Hz, 5H), 5.16 (d, *J* = 5.7 Hz, 2H), 3.83 (s, 3H), 3.21 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 190.70, 167.63, 162.74, 152.87, 132.33, 129.13, 126.77, 126.01, 114.01, 112.03, 57.39, 55.60, 38.60.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>17</sub>H<sub>18</sub>N<sub>2</sub>NaO<sub>3</sub> 321.1210, obsd 321.1217.

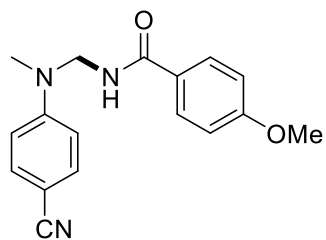


**ethyl 4-(((4-methoxybenzamido)methyl)(methyl)amino)benzoate (34).** White solid, mp 123.1-124.1 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 2/1, Y = 80%, (54.7 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.88 (d, *J* = 8.7 Hz, 2H), 7.74 (d, *J* = 8.7 Hz, 2H), 7.26 (t, *J* = 6.8 Hz, 1H), 6.81 (dd, *J* = 20.2, 8.8 Hz, 4H), 5.08 (d, *J* = 5.6 Hz, 2H), 4.27 (q, *J* = 7.1 Hz, 2H), 3.79 (s, 3H), 3.12 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.77, 167.03, 162.47, 151.49, 131.47, 129.10, 126.11, 118.95, 113.78, 111.59, 60.43, 57.47, 55.44, 38.32, 14.48.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>19</sub>H<sub>22</sub>N<sub>2</sub>NaO<sub>4</sub> 365.1472, obsd 365.1480.

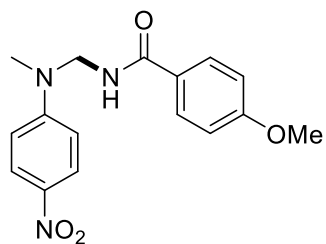


**N-(((4-cyanophenyl)(methyl)amino)methyl)-4-methoxybenzamide (35).** Brown solid, mp 151.2-152.0 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 2/1, Y = 81%, (47.8 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.76 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.7 Hz, 3H), 6.85 (t, *J* = 7.6 Hz, 4H), 5.06 (d, *J* = 5.7 Hz, 2H), 3.80 (s, 3H), 3.13 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.77, 162.56, 150.98, 133.61, 129.14, 125.85, 120.54, 113.81, 112.42, 98.85, 57.21, 55.47, 38.26.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>17</sub>H<sub>17</sub>N<sub>3</sub>NaO<sub>2</sub> 318.1213, obsd 318.1219.

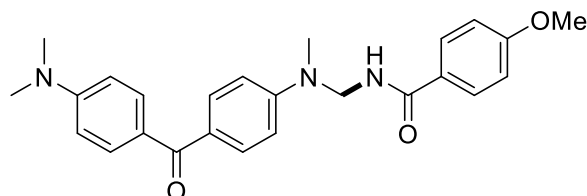


**4-methoxy-N-((methyl(4-nitrophenyl)amino)methyl)benzamide (36).** Brown solid, mp 111.3-112.2 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 2/1, Y = 47%, (29.6 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.07 (d, *J* = 9.4 Hz, 2H), 7.91 – 7.74 (m, 2H), 6.91 (d, *J* = 8.9 Hz, 3H), 6.79 (d, *J* = 9.4 Hz, 2H), 5.15 (d, *J* = 5.9 Hz, 2H), 3.84 (s, 3H), 3.22 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.67, 162.82, 152.95, 138.31, 129.18, 126.34, 125.80, 114.04, 111.36, 57.35, 55.62, 38.70.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>16</sub>H<sub>17</sub>N<sub>3</sub>NaO<sub>4</sub> 338.1111, obsd 338.1110.



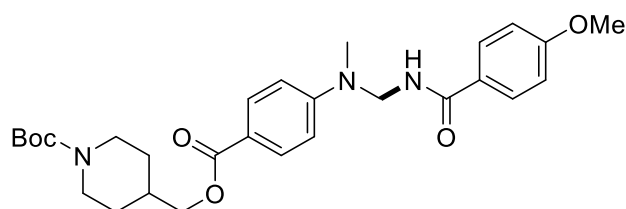
**N-(((4-(4-(dimethylamino)benzoyl)phenyl)(methyl)amino)methyl)-4-methoxybenzamide (37).** Brown solid, mp 173.0-173.8 °C. The title compound was prepared according to general

procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/2, Y = 43%, (35.8 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.87 – 7.66 (m, 6H), 7.23 – 7.14 (m, 1H), 6.84 (dd, *J* = 8.8, 6.3 Hz, 4H), 6.65 (d, *J* = 9.0 Hz, 2H), 5.14 (d, *J* = 5.7 Hz, 2H), 3.80 (s, 3H), 3.16 (s, 3H), 3.05 (s, 6H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 194.28, 167.75, 162.52, 153.01, 150.89, 132.49, 132.45, 129.20, 128.01, 126.28, 125.89, 113.87, 111.54, 110.68, 57.59, 55.53, 40.23, 38.46.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>25</sub>H<sub>27</sub>N<sub>3</sub>NaO<sub>3</sub> 440.1945, obsd 440.1953.



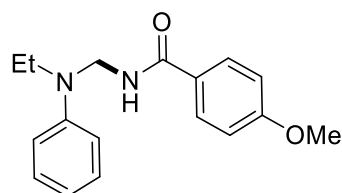
**tert-butyl**

**4-(((4-((4-methoxybenzamido)methyl)(methylamino)benzoyl)oxy)methyl)piperidine-1-carboxylate (38).** Brown oil. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 2/1, Y = 96%, (98.1 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.90 (d, *J* = 8.8 Hz, 2H), 7.73 (d, *J* = 8.9 Hz, 2H), 7.05 – 6.97 (m, 1H), 6.91 – 6.78 (m, 4H), 5.12 (d, *J* = 5.8 Hz, 2H), 4.10 (d, *J* = 6.5 Hz, 4H), 3.82 (s, 3H), 3.16 (s, 3H), 2.72 (s, 2H), 1.97 – 1.86 (m, 1H), 1.74 (d, *J* = 13.0 Hz, 2H), 1.46 (s, 9H), 1.35 – 1.14 (m, 2H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.63, 166.80, 162.57, 154.98, 151.62, 131.59, 129.09, 126.14, 118.79, 113.87, 111.68, 79.56, 68.36, 57.47, 55.51, 43.68, 38.43, 35.94, 28.90, 28.56.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>28</sub>H<sub>37</sub>N<sub>3</sub>NaO<sub>6</sub> 534.2575, obsd 534.2582.

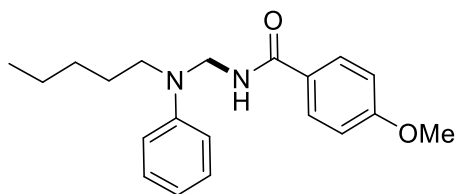


**N-((ethyl(phenyl)amino)methyl)-4-methoxybenzamide (39).** White solid, mp 92.8-93.8 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 51%, (30.0 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.70 (d, *J* = 8.8 Hz, 2H), 7.25 (t, *J* = 8.0 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.82 (d, *J* = 8.2 Hz, 2H), 6.77 (t, *J* = 7.2 Hz, 1H), 6.57 (s, 1H), 5.06 (d, *J* = 5.2 Hz, 2H), 3.81 (s, 3H), 3.51 (q, *J* = 7.0 Hz, 2H), 1.21 (t, *J* = 7.1 Hz, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  167.42, 162.40, 146.85, 129.72, 128.94, 126.46, 117.71, 113.84, 112.89, 56.58, 55.50, 44.86, 13.14.

ESI HRMS  $m/z$  ( $\text{M}+\text{Na}$ ) $^+$  calcd for  $\text{C}_{17}\text{H}_{20}\text{N}_2\text{NaO}_2$  307.1417, obsd 307.1423.

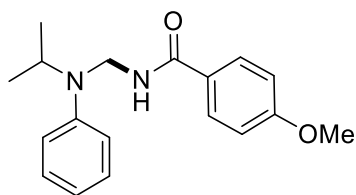


**4-methoxy-N-((pentyl(phenyl)amino)methyl)benzamide (40).** White solid, mp 62.5-63.3 °C. The title compound was prepared according to general procedure, electricity = 16.7 F  $\text{mol}^{-1}$ . Purified by flash silica column chromatography with PE/EA = 3/1, Y = 47%, (30.7 mg).

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70 (d,  $J$  = 8.8 Hz, 2H), 7.32 – 7.19 (m, 2H), 6.87 (d,  $J$  = 8.8 Hz, 2H), 6.83 – 6.72 (m, 3H), 6.62 – 6.49 (m, 1H), 5.06 (d,  $J$  = 5.2 Hz, 2H), 3.81 (s, 3H), 3.49 – 3.35 (m, 2H), 1.63 (p,  $J$  = 7.4 Hz, 2H), 1.39 – 1.26 (m, 4H), 0.89 (t,  $J$  = 6.7 Hz, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  167.40, 162.38, 147.04, 129.68, 128.93, 126.47, 117.56, 113.82, 112.73, 57.07, 55.49, 50.79, 29.40, 27.62, 22.68, 14.23.

ESI HRMS  $m/z$  ( $\text{M}+\text{Na}$ ) $^+$  calcd for  $\text{C}_{20}\text{H}_{26}\text{N}_2\text{NaO}_2$  349.1886, obsd 349.1886.

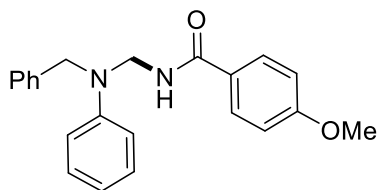


**N-((isopropyl(phenyl)amino)methyl)-4-methoxybenzamide (41).** Brown oil. The title compound was prepared according to general procedure, electricity = 16.7 F  $\text{mol}^{-1}$ . Purified by flash silica column chromatography with PE/EA = 3/1, Y = 55%, (32.8 mg).

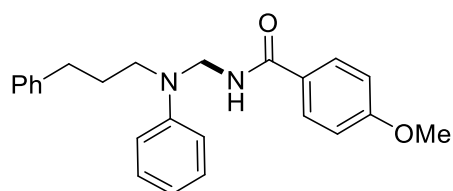
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.82 – 7.62 (m, 2H), 7.27 (t,  $J$  = 8.0 Hz, 2H), 6.89 (t,  $J$  = 9.0 Hz, 4H), 6.79 (t,  $J$  = 7.3 Hz, 1H), 6.16 (s, 1H), 5.04 (d,  $J$  = 4.5 Hz, 2H), 4.12 (p,  $J$  = 6.6 Hz, 1H), 3.83 (s, 3H), 1.28 (d,  $J$  = 6.6 Hz, 6H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  166.78, 162.44, 147.66, 129.82, 128.86, 126.61, 118.07, 113.93, 113.30, 55.58, 52.09, 47.97, 20.70.

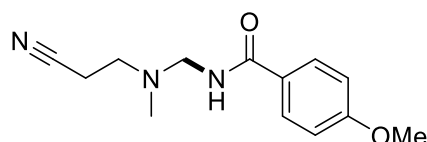
ESI HRMS  $m/z$  ( $\text{M}+\text{Na}$ ) $^+$  calcd for  $\text{C}_{18}\text{H}_{22}\text{N}_2\text{NaO}_2$  321.1573, obsd 321.1580.



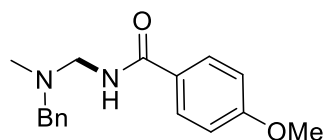
**N-((benzyl(phenyl)amino)methyl)-4-methoxybenzamide (42).** Brown solid, mp 93.2-94.3 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 5/2, Y = 52%, (36.0 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.71 – 7.57 (m, 2H), 7.36 – 7.26 (m, 5H), 7.26 – 7.20 (m, 2H), 6.91 – 6.84 (m, 4H), 6.79 (t, *J* = 7.3 Hz, 1H), 6.46 (t, *J* = 5.5 Hz, 1H), 5.19 (d, *J* = 5.4 Hz, 2H), 4.71 (s, 2H), 3.83 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.35, 162.52, 147.42, 138.92, 129.74, 128.96, 128.91, 127.27, 126.87, 126.42, 118.33, 113.91, 113.26, 57.29, 55.58, 54.58. ESI HRMS *m/z* (M+Na)<sup>+</sup> calcd for C<sub>22</sub>H<sub>22</sub>N<sub>2</sub>NaO<sub>2</sub> 369.1573, obsd 369.1582.



**4-methoxy-N-((phenyl(3-phenylpropyl)amino)methyl)benzamide (43).** Brown oil. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 50%, (37.4 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.69 (d, *J* = 8.4 Hz, 2H), 7.33 – 7.21 (m, 3H), 7.22 – 7.15 (m, 4H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.76 (t, *J* = 8.4 Hz, 3H), 6.44 (s, 1H), 5.07 (d, *J* = 5.3 Hz, 2H), 3.82 (s, 3H), 3.46 (t, *J* = 7.6 Hz, 2H), 2.67 (t, *J* = 7.7 Hz, 2H), 1.98 (p, *J* = 7.7 Hz, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.40, 162.45, 146.94, 141.66, 129.74, 128.95, 128.55, 128.51, 126.44, 126.09, 117.90, 113.89, 113.02, 57.11, 55.55, 50.23, 33.39, 29.37. ESI HRMS *m/z* (M+Na)<sup>+</sup> calcd for C<sub>24</sub>H<sub>26</sub>N<sub>2</sub>NaO<sub>2</sub> 397.1886, obsd 397.1895.



**N-(((2-cyanoethyl)(methyl)amino)methyl)-4-methoxybenzamide (44).** White solid, mp 123.1-123.9 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/2, Y = 57%, (28.2 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.87 – 7.71 (m, 2H), 7.00 – 6.89 (m, 2H), 6.67 (t, *J* = 6.5 Hz, 1H), 4.36 (d, *J* = 6.2 Hz, 2H), 3.85 (s, 3H), 2.82 (t, *J* = 6.7 Hz, 2H), 2.63 (t, *J* = 6.6 Hz, 2H), 2.43 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 168.02, 162.62, 129.01, 126.23, 119.28, 114.00, 60.09, 55.59, 49.50, 40.15, 16.92. ESI HRMS *m/z* (M+Na)<sup>+</sup> calcd for C<sub>13</sub>H<sub>17</sub>N<sub>3</sub>NaO<sub>2</sub> 270.1213, obsd 270.1221.

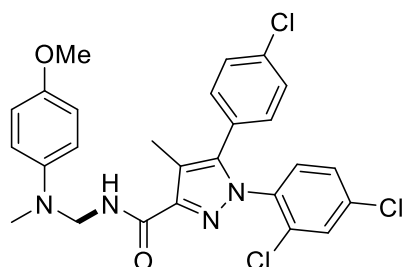


**N-((benzyl(methyl)amino)methyl)-4-methoxybenzamide (45).** White solid, 105.8-106.7 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 1/1, Y = 33%, (18.7 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.78 – 7.69 (m, 2H), 7.32 (d, *J* = 4.4 Hz, 4H), 7.29 – 7.24 (m, 1H), 6.90 (d, *J* = 8.8 Hz, 2H), 6.66 – 6.56 (m, 1H), 4.37 (d, *J* = 6.1 Hz, 2H), 3.83 (s, 3H), 3.66 (s, 2H), 2.30 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.58, 162.33, 138.74, 128.99, 128.92, 128.52, 127.29, 126.66, 113.82, 61.32, 59.30, 55.50, 39.75.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>17</sub>H<sub>20</sub>N<sub>2</sub>NaO<sub>2</sub> 307.1417, obsd 307.1419.

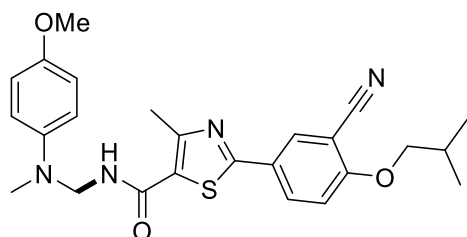


**5-(4-chlorophenyl)-1-(2,4-dichlorophenyl)-N-(((4-methoxyphenyl)(methyl)amino)methyl)-4-methyl-1H-pyrazole-3-carboxamide (46).** Brown oil. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 63%, (72.1 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.39 (d, *J* = 2.1 Hz, 1H), 7.35 (t, *J* = 6.1 Hz, 1H), 7.31 – 7.22 (m, 4H), 7.08 – 7.02 (m, 2H), 6.85 (s, 4H), 5.00 (d, *J* = 6.1 Hz, 2H), 3.74 (s, 3H), 2.99 (s, 3H), 2.38 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 163.21, 152.79, 144.69, 143.24, 142.64, 136.12, 135.96, 135.06, 133.03, 130.95, 130.69, 130.41, 129.03, 128.01, 127.27, 118.04, 115.68, 114.93, 58.11, 55.80, 38.32, 9.61.

**ESI HRMS** *m/z* (M+H)<sup>+</sup> calcd for C<sub>26</sub>H<sub>24</sub>Cl<sub>3</sub>N<sub>4</sub>O<sub>2</sub> 529.0959, obsd 529.0959.

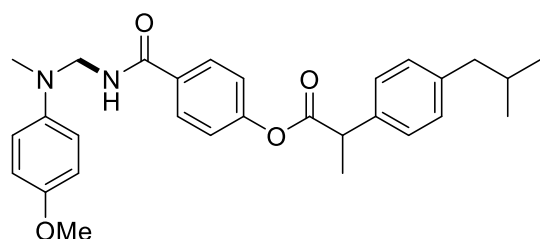


**2-(3-cyano-4-isobutoxyphenyl)-N-(((4-methoxyphenyl)(methyl)amino)methyl)-4-methylthiazole-5-carboxamide (47).** Brown oil. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 2/1, Y = 90%, (83.5 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.08 (t, *J* = 2.4 Hz, 1H), 8.00 (dt, *J* = 8.8, 2.1 Hz, 1H), 6.98 (d, *J* = 8.7 Hz, 1H), 6.87 (s, 4H), 6.38 – 6.20 (m, 1H), 5.01 (d, *J* = 5.7 Hz, 2H), 3.88 (d, *J* = 6.4 Hz, 2H), 3.77 (s, 3H), 2.98 (s, 3H), 2.63 (d, *J* = 1.1 Hz, 3H), 2.19 (dt, *J* = 13.3, 6.6 Hz, 1H), 1.09 (d, *J* = 6.7 Hz, 3H), 1.07 (d, *J* = 6.7 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 164.89, 162.48, 162.13, 156.33, 153.24, 142.17, 132.60, 132.01, 125.97, 125.90, 116.04, 115.58, 115.07, 112.74, 102.97, 75.79, 59.50, 55.79, 38.52, 28.27, 19.18, 17.52.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>25</sub>H<sub>28</sub>N<sub>4</sub>NaO<sub>3</sub>S 487.1774, obsd 487.1780.



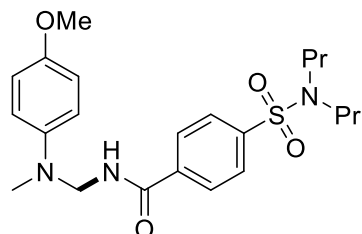
#### 4-(((4-methoxyphenyl)(methyl)amino)methyl)carbamoyl)phenyl

**2-(4-isobutylphenyl)propanoate (48).** Brown solid, 66.3-67.2 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 3/1, Y = 92%, (87.2 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.66 (d, *J* = 8.6 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.98 (d, *J* = 8.7 Hz, 2H), 6.83 (s, 4H), 6.65 (t, *J* = 5.7 Hz, 1H), 4.97 (d, *J* = 5.7 Hz, 2H), 3.92 (q, *J* = 7.1 Hz, 1H), 3.74 (s, 3H), 2.93 (s, 3H), 2.46 (d, *J* = 7.2 Hz, 2H), 1.86 (dp, *J* = 13.5, 6.8 Hz, 1H), 1.59 (d, *J* = 7.2 Hz, 3H), 0.90 (d, *J* = 6.6 Hz, 6H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 173.01, 167.21, 153.48, 153.01, 142.38, 141.11, 136.99, 131.76, 129.70, 128.50, 127.29, 121.71, 115.87, 114.98, 59.37, 55.75, 45.34, 45.13, 38.35, 30.29, 22.50, 18.57.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>29</sub>H<sub>34</sub>N<sub>2</sub>NaO<sub>4</sub> 497.2411, obsd 497.2418.



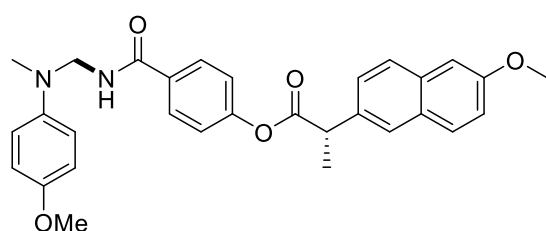
**4-(N,N-dipropylsulfamoyl)-N-(((4-methoxyphenyl)(methyl)amino)methyl)benzamide**

**(49).** Brown solid, 69.5-70.4 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 5/2, Y = 66%, (57.1 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.79 (d, *J* = 8.3 Hz, 2H), 7.73 (d, *J* = 8.3 Hz, 2H), 6.97 – 6.81 (m, 5H), 5.04 (d, *J* = 5.6 Hz, 2H), 3.75 (s, 3H), 3.09 – 3.00 (m, 4H), 2.99 (s, 3H), 1.61 – 1.43 (m, 4H), 0.85 (t, *J* = 7.4 Hz, 6H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 166.86, 152.99, 142.84, 142.24, 137.94, 127.93, 127.26, 115.68, 114.99, 59.47, 55.78, 50.03, 38.50, 22.01, 11.26.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>22</sub>H<sub>31</sub>N<sub>3</sub>NaO<sub>4</sub>S 456.1927, obsd 456.1935.



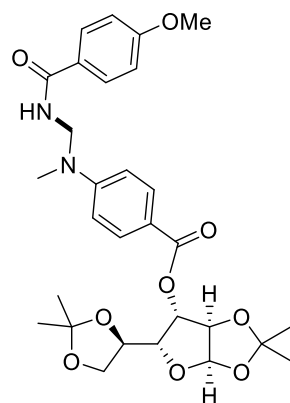
**4-(((4-methoxyphenyl)(methyl)amino)methyl)carbamoyl)phenyl**

**(S)-2-(6-methoxynaphthalen-2-yl)propanoate (50).** Brown solid, 127.0-128.0 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 5/2, Y = 84%, (83.7 mg).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.80 – 7.70 (m, 3H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.46 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.19 – 7.10 (m, 2H), 6.97 (d, *J* = 8.4 Hz, 2H), 6.82 (s, 4H), 6.57 (t, *J* = 5.6 Hz, 1H), 4.96 (d, *J* = 5.7 Hz, 2H), 4.08 (q, *J* = 7.1 Hz, 1H), 3.90 (s, 3H), 3.73 (s, 3H), 2.92 (s, 3H), 1.67 (d, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.95, 167.17, 157.94, 153.47, 153.04, 142.38, 134.89, 133.99, 131.82, 129.43, 129.08, 128.50, 127.61, 126.30, 126.11, 121.72, 119.34, 115.88, 115.00, 105.73, 59.38, 55.76, 55.45, 45.67, 38.35, 18.55.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>30</sub>H<sub>30</sub>N<sub>2</sub>NaO<sub>5</sub> 521.2047, obsd 521.2050.



**(3aR,5R,6S,6aR)-5-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-yl 4-(((4-methoxybenzamido)methyl)(methyl)amino)benzoate (51).** Brown solid, 80.2-81.1 °C. The title compound was prepared according to general procedure, electricity = 16.7 F mol<sup>-1</sup>. Purified by flash silica column chromatography with PE/EA = 2/1, Y = 57%, (63.4 mg).

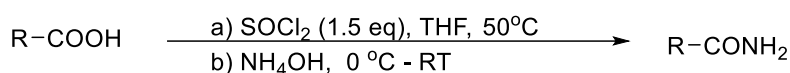
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.97 – 7.86 (m, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 6.97 – 6.86 (m, 2H), 6.82 (d, *J* = 8.6 Hz, 2H), 6.74 (s, 1H), 5.93 (d, *J* = 3.7 Hz, 1H), 5.45 (d, *J* = 2.2 Hz, 1H), 5.20 – 5.07 (m, 2H), 4.60 (d, *J* = 3.7 Hz, 1H), 4.35 (d, *J* = 3.1 Hz, 2H), 4.09 (d, *J* = 4.4 Hz, 2H), 3.83 (s, 3H), 3.17 (s, 3H), 1.55 (s, 3H), 1.41 (s, 3H), 1.31 (s, 3H), 1.27 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.57, 165.40, 162.69, 151.95, 131.90, 129.07, 126.05, 118.06, 113.98, 112.42, 111.75, 109.40, 105.27, 83.60, 80.09, 76.15, 72.85, 67.17, 57.42, 55.58, 38.48, 26.94, 26.90, 26.36, 25.40.

**ESI HRMS** *m/z* (M+Na)<sup>+</sup> calcd for C<sub>29</sub>H<sub>36</sub>N<sub>2</sub>NaO<sub>9</sub> 579.2313, obsd 579.2314.

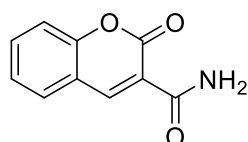
## 5. Synthesis of Substrates

Most of the tertiary amines and amides used in this work were commercially available. For non-commercial substrates, synthesis was performed according to published procedures.



**Procedure for the synthesis of amides from carboxylic acids:** To a dried round-bottom flask equipped with a stir bar, carboxylic acids (3.0 mmol) was dissolved in THF (10.0 mL). Thionyl chloride (4.5 mmol, 1.5 equiv) was added dropwise at room temperature, and the mixture was heated to 50 °C for 2 h. After cooling to room temperature, the solution was transferred into a flask containing ice-cooled ammonium hydroxide (25%, 10.0 mL) at 0 °C. The mixture was stirred vigorously for 30 min at temperature, then extracted with DCM (3 × 20 mL). The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under reduced pressure. The crude solid was triturated with PE/EA, filtered, and dried under vacuum to afford the solid amides.

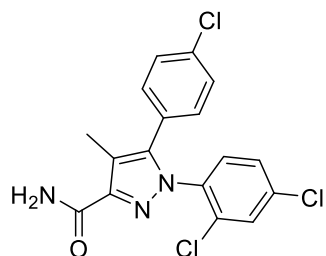
### Characterization data for representative examples:



**2-oxo-2H-chromene-3-carboxamide.**<sup>1</sup> The title compound was prepared according the above procedure from 2-oxo-2-(2-oxo-2H-chromen-3-yl)acetic acid.

**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ 8.88 (s, 1H), 8.09 (s, 1H), 7.98 (d, *J* = 7.7 Hz, 1H), 7.93 (s, 1H), 7.76 (t, *J* = 7.8 Hz, 1H), 7.50 (d, *J* = 8.4 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 1H).

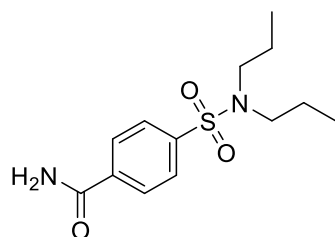
**<sup>13</sup>C NMR** (101 MHz, DMSO-*d*<sub>6</sub>) δ 162.52, 160.33, 154.04, 147.82, 134.10, 130.26, 125.08, 119.28, 118.45, 116.12.



**5-(4-chlorophenyl)-1-(2,4-dichlorophenyl)-4-methyl-1H-pyrazole-3-carboxamide.**<sup>2</sup> The title compound was prepared according to the above procedure from 5-(4-chlorophenyl)-1-(2,4-dichlorophenyl)-4-methyl-1H-pyrazole-3-carboxylic acid.

**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.81 – 7.69 (m, 2H), 7.58 (dd, *J* = 8.4, 2.4 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 2H), 7.31 (s, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 2.26 (s, 3H).

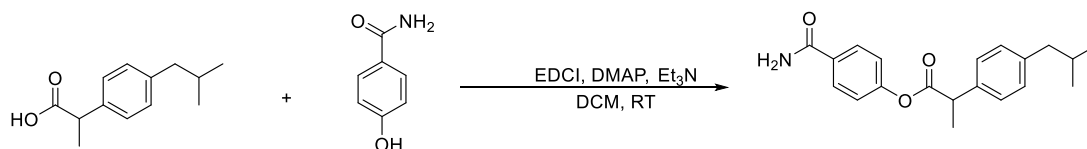
**<sup>13</sup>C NMR** (101 MHz, DMSO-*d*<sub>6</sub>) δ 164.04, 144.79, 142.51, 135.81, 135.03, 133.71, 132.15, 131.92, 131.28, 129.60, 128.73, 128.32, 127.28, 116.50, 9.23.



**4-(N,N-dipropylsulfamoyl)benzamide.**<sup>3</sup> The title compound was prepared according to the above procedure from 4-(N,N-dipropylsulfamoyl)benzoic acid.

**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>) δ 8.21 (s, 1H), 8.07 (d, *J* = 8.1 Hz, 2H), 7.89 (d, *J* = 8.1 Hz, 2H), 7.64 (s, 1H), 3.05 (t, *J* = 7.6 Hz, 4H), 1.48 (h, *J* = 7.4 Hz, 4H), 0.81 (t, *J* = 7.3 Hz, 6H).

**<sup>13</sup>C NMR** (101 MHz, DMSO-*d*<sub>6</sub>) δ 166.72, 141.76, 137.85, 128.45, 126.76, 10.95.

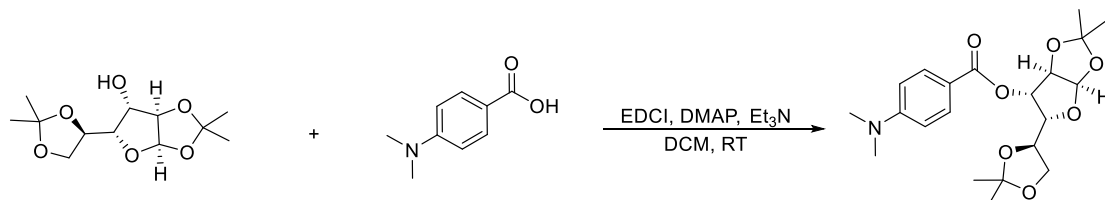


**4-carbamoylphenyl 2-(4-isobutylphenyl)propanoate.** To an oven-dried round-bottom flask equipped with a stir bar were added 2-(4-isobutylphenyl)propanoic acid (ibuprofen acid, 0.60 g, 3.0 mmol, 1.0 equiv), EDCI (0.86 g, 4.5 mmol, 1.5 equiv), DMAP (0.03 g, 0.30 mmol, 10 mol%), and anhydrous DCM (12 mL). After stirring at RT for 5 minute, 4-hydroxybenzamide

(0.49 g, 3.6 mmol, 1.2 equiv) was added and the mixture was stirred until complete consumption of isonicotinic acid. The reaction mixture was diluted with H<sub>2</sub>O and DCM. The aqueous layer was extracted with DCM for 3 times. The combined organic phase was dried over anhydrous MgSO<sub>4</sub>, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography to afford title product (0.79 g, 81% yield) as white solid.

<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 8.10 – 7.97 (m, 1H), 7.96 – 7.87 (m, 2H), 7.41 (d, *J* = 6.3 Hz, 1H), 7.38 – 7.27 (m, 2H), 7.20 (d, *J* = 7.6 Hz, 2H), 7.15 – 7.03 (m, 2H), 4.08 (t, *J* = 7.3 Hz, 1H), 2.45 (t, *J* = 7.3 Hz, 2H), 1.83 (dt, *J* = 13.9, 6.9 Hz, 1H), 1.52 (t, *J* = 7.5 Hz, 3H), 0.88 (t, *J* = 7.1 Hz, 6H).

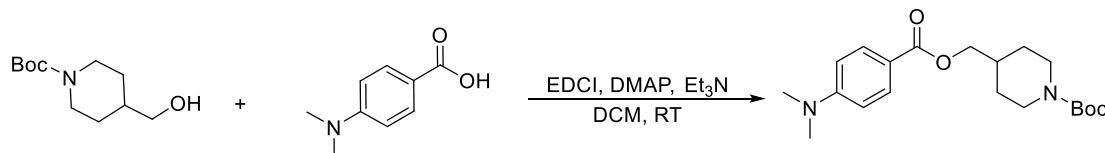
<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>) δ 172.61, 167.06, 152.68, 140.11, 137.33, 131.93, 129.33, 129.04, 127.20, 121.30, 44.21, 29.60, 22.17, 18.48.



**(3aR,5R,6S,6aR)-5-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-yl 4-(dimethylamino)benzoate.** The title compound was prepared from (3aR,5S,6S,6aR)-5-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-ol and 4-(dimethylamino)benzoic acid as a white solid following the procedure described for the synthesis of 4-carbamoylphenyl 2-(4-isobutylphenyl)propanoate.

<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.78 (d, *J* = 8.5 Hz, 2H), 6.72 (d, *J* = 8.6 Hz, 2H), 5.97 (d, *J* = 3.7 Hz, 1H), 5.22 (d, *J* = 3.0 Hz, 1H), 4.63 (d, *J* = 3.7 Hz, 1H), 4.35 (q, *J* = 6.1 Hz, 1H), 4.24 (dd, *J* = 7.2, 3.0 Hz, 1H), 4.05 (dd, *J* = 8.2, 6.2 Hz, 1H), 3.96 (dd, *J* = 8.4, 5.3 Hz, 1H), 3.00 (s, 6H), 1.46 (s, 3H), 1.34 (s, 3H), 1.27 (s, 3H), 1.21 (s, 3H).

<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>) δ 164.84, 153.52, 131.01, 114.86, 111.26, 110.81, 108.41, 104.71, 82.91, 79.15, 75.46, 72.20, 66.19, 39.54, 26.57, 26.43, 25.92, 25.01.



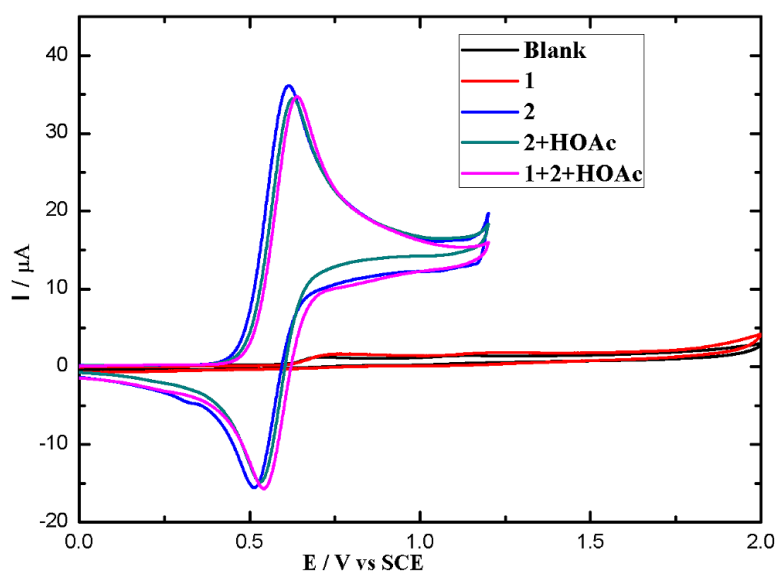
**tert-butyl 4-(((4-(dimethylamino)benzoyl)oxy)methyl)piperidine-1-carboxylate.** The title compound was prepared from tert-butyl 4-(hydroxymethyl)piperidine-1-carboxylate and 4-(dimethylamino)benzoic acid as a white solid following the procedure described for the synthesis of 4-carbamoylphenyl 2-(4-isobutylphenyl)propanoate.

**$^1\text{H}$  NMR** (400 MHz,  $\text{DMSO-}d_6$ )  $\delta$  7.77 (d,  $J$  = 8.4 Hz, 2H), 6.70 (d,  $J$  = 8.8 Hz, 2H), 4.04 (d,  $J$  = 6.4 Hz, 2H), 3.98 (s, 2H), 2.98 (s, 6H), 2.71 (s, 2H), 1.95 – 1.79 (m, 1H), 1.68 (d,  $J$  = 12.9 Hz, 2H), 1.39 (s, 9H), 1.24 – 1.04 (m, 2H).

**$^{13}\text{C}$  NMR** (101 MHz,  $\text{DMSO-}d_6$ )  $\delta$  165.82, 153.87, 153.24, 130.73, 115.86, 110.79, 78.48, 67.54, 39.57, 35.17, 28.31, 28.06.

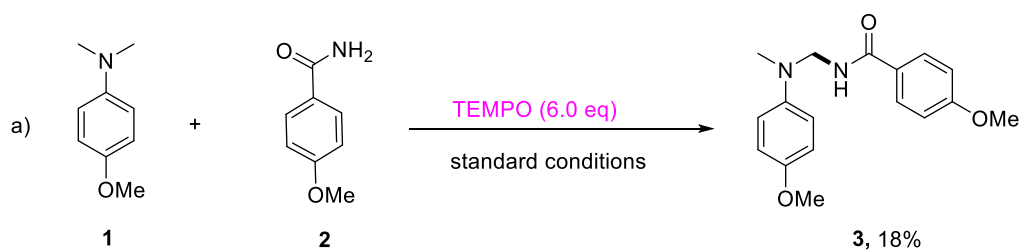
## 6. Cyclic Voltammetry Studies

The cyclic voltammograms were recorded in an electrolyte of  $\text{Bu}_4\text{NClO}_4$  (0.1 M) in MeCN at 50 °C in a glassy carbon disk working electrode (diameter, 1 mm), a Pt wire auxiliary electrode and a SCE reference electrode. The scan rate was 100 mV/s.

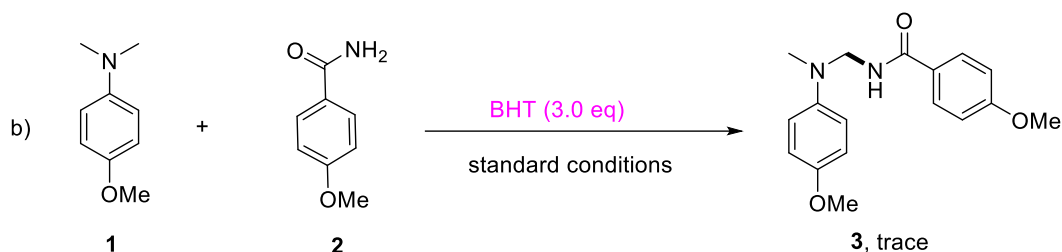


**Figure S2.** Cyclic voltammograms. Black:  $\text{Bu}_4\text{NClO}_4$  (0.1 M); Red:  $\text{Bu}_4\text{NClO}_4$  (0.1 M), **2** (10 mM); Blue:  $\text{Bu}_4\text{NClO}_4$  (0.1 M), **1** (10 mM); Green:  $\text{Bu}_4\text{NClO}_4$  (0.1 M), **1** (10 mM), HOAc (10 mM); Pink:  $\text{Bu}_4\text{NClO}_4$  (0.1 M), **1** (10 mM), **2** (10 mM), HOAc (10 mM).

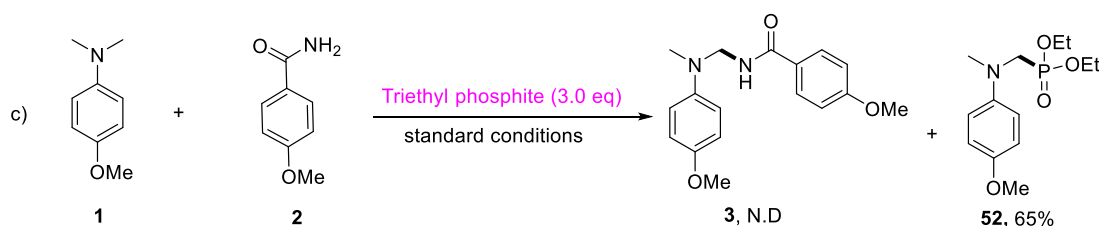
## 7. Control experiments



A 10 mL dry Schlenk tube equipped with a magnetic stir bar was charged with **1** (0.6 mmol, 90.6 mg), **2** (0.2 mmol, 30.2 mg), Bu<sub>4</sub>NClO<sub>4</sub> (34.0 mg), 2,2,6,6-tetramethylpiperidoxyl (TEMPO, 1.2 mmol, 187.2 mg) in MeCN (5.0 mL). Acetic acid (0.4 mmol, 24.0 mg) was then added via microliter syringe. The reaction vessel was fitted with a graphite felt (GF) anode (0.3 cm x 1.0 cm x 1.5 cm) and a platinum cathode (Pt) (0.1 cm x 1.0 cm x 1.0 cm). The solution was degassed by bubbling with N<sub>2</sub> for 3 minutes to remove dissolved oxygen. Constant current electrolysis was then performed at 6 mA while maintaining the reaction temperature at 50 °C for 15 hours. The reaction mixture was concentrated in vacuo, and the crude was purified by flash chromatography on silica gel to afford **3** in 18% yield (10.8 mg).

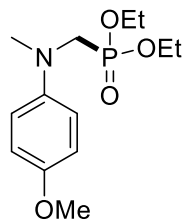


A 10 mL dry Schlenk tube equipped with a magnetic stir bar was charged with **1** (0.6 mmol, 90.6 mg), **2** (0.2 mmol, 30.2 mg), Bu<sub>4</sub>NClO<sub>4</sub> (34.0 mg), butylated hydroxytoluene (BHT, 0.6 mmol, 132 mg) in MeCN (5.0 mL). Acetic acid (0.4 mmol, 24.0 mg) was then added via microliter syringe. The reaction vessel was fitted with a graphite felt (GF) anode (0.3 cm x 1.0 cm x 1.5 cm) and a platinum cathode (Pt) (0.1 cm x 1.0 cm x 1.0 cm). The solution was degassed by bubbling with N<sub>2</sub> for 3 minutes to remove dissolved oxygen. Constant current electrolysis was then performed at 6 mA while maintaining the reaction temperature at 50 °C for 15 hours. Internal standard analysis detected only trace product **3** formation.



A 10 mL dry Schlenk tube equipped with a magnetic stir bar was charged with **1** (0.6 mmol, 90.6 mg), **2** (0.2 mmol, 30.2 mg), Bu<sub>4</sub>NClO<sub>4</sub> (34.0 mg), triethyl phosphite (0.6 mmol, 100 mg) in MeCN (5.0 mL). Acetic acid (0.4 mmol, 24.0 mg) was then added via microliter syringe. The reaction vessel was fitted with a graphite felt (GF) anode (0.3 cm x 1.0 cm x 1.5 cm) and a platinum cathode (Pt) (0.1 cm x 1.0 cm x 1.0 cm). The solution was degassed by bubbling with N<sub>2</sub> for 3 minutes to remove dissolved oxygen. Constant current electrolysis was then performed at 6 mA while maintaining the reaction temperature at 50 °C for 15 hours. The

reaction mixture was then concentrated in vacuo, and the crude was purified by flash chromatography on silica gel to afford **52** in 65% yield (37.3 mg), and no product **3** was isolated from the system.



**diethyl (((4-methoxyphenyl)(methyl)amino)methyl)phosphonate (**52**).<sup>4</sup>**

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.83 (t, *J* = 6.6 Hz, 4H), 4.16 – 4.02 (m, 4H), 3.76 (s, 3H), 3.63 (d, *J* = 7.8 Hz, 2H), 2.99 (s, 3H), 1.28 (t, *J* = 7.1 Hz, 6H).

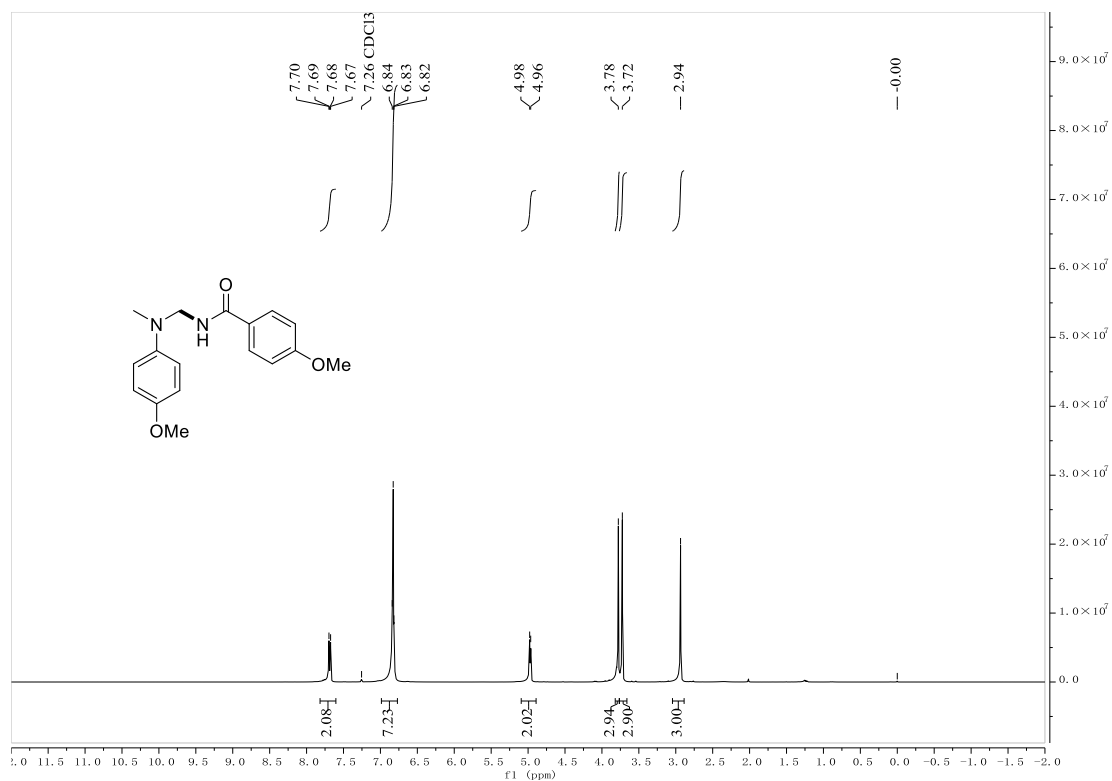
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 152.46, 144.56 (d, *J* = 3.8 Hz), 115.07, 114.71, 62.29 (d, *J* = 7.0 Hz), 55.88, 51.37 (d, *J* = 162.3 Hz), 40.06, 16.66 (d, *J* = 5.8 Hz).

## 8. References

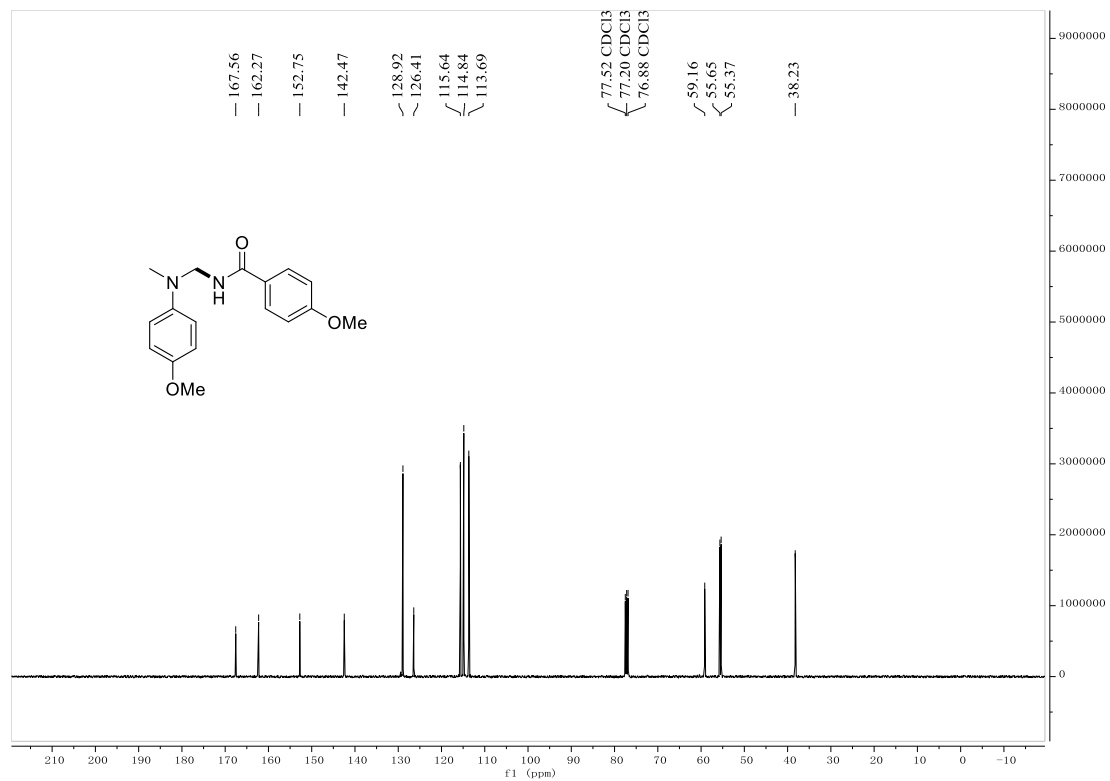
1. Chernov, N. M.; Filippova, P. V.; Yakovlev, I. P.; Zakhs, V. E.; Belyakov, A. V., Synthesis and reactivity of 4-hydroxy-5-methyl-2-(2-oxo-2H-chromen-3-yl)-6H-1,3-oxazin-6-ones. *Russian Journal of General Chemistry* **2016**, 86 (6), 1292-1299.
2. Rodríguez-Soacha, D. A.; Fender, J.; Ramírez, Y. A.; Collado, J. A.; Muñoz, E.; Maitra, R.; Sotriffer, C.; Lorenz, K.; Decker, M., “Photo-Rimonabant”: Synthesis and biological evaluation of novel photoswitchable molecules derived from rimonabant Lead to a highly selective and nanomolar “cis-on” CB1R antagonist. *ACS Chemical Neuroscience* **2021**, 12 (9), 1632-1647.
3. Ayushee; Patel, M.; Meena, P.; Jahan, K.; Bharatam, P. V.; Verma, A. K., Base-mediated anti-Markovnikov hydroamidation of vinyl arenes with arylamides. *Organic Letters* **2021**, 23 (2), 565-570.
4. Han, W.; Ofial, A. R., Iron-catalyzed dehydrogenative phosphonation of N,N-dimethylanilines. *Chemical Communications* **2009**, (40), 6023-6025.

## 9. NMR spectra

### $^1\text{H}$ NMR of 3



### $^{13}\text{C}$ NMR of 3



## HRMS of 3

Monoisotopic Mass, Even Electron Ions

2075 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

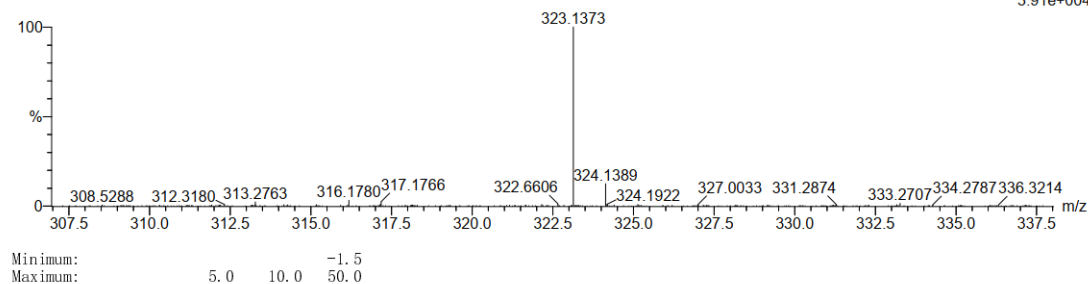
Elements Used:

C: 17-17 H: 20-20 N: 0-100 O: 0-100 Na: 0-4 K: 0-2

7

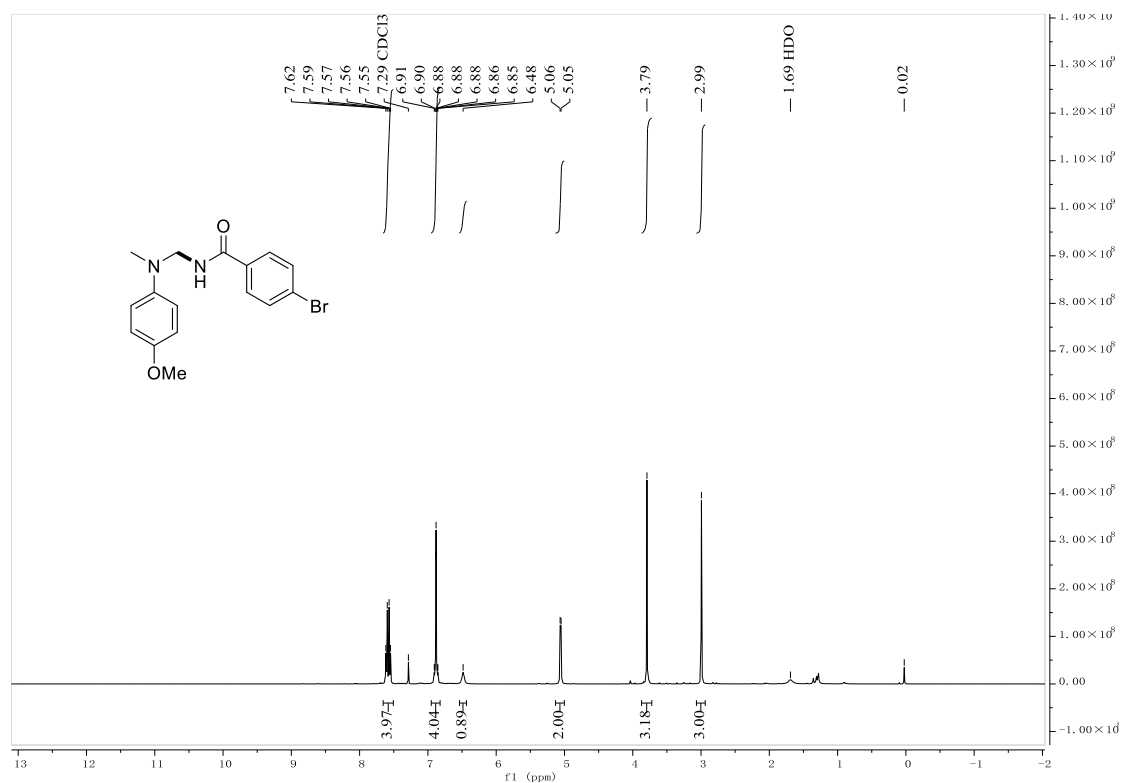
250707-4.4 33 (0.206)

1: TOF MS ES+  
3.91e+004

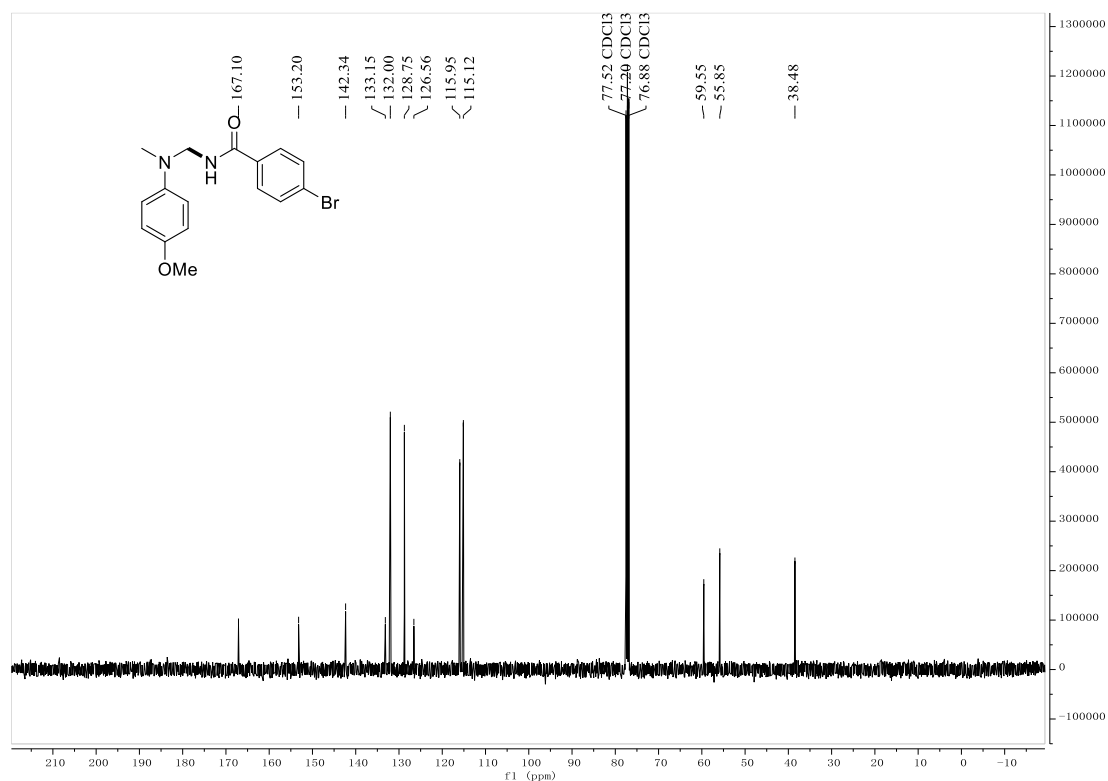


| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT | Norm | Conf (%) | Formula          |
|----------|------------|-----|-----|-----|-------|------|----------|------------------|
| 323.1373 | 323.1372   | 0.1 | 0.3 | 8.5 | 258.3 | n/a  | n/a      | C17 H20 N2 O3 Na |

## <sup>1</sup>H NMR of 4



## <sup>13</sup>C NMR of 4



## HRMS of 4

Monoisotopic Mass, Even Electron Ions

1342 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

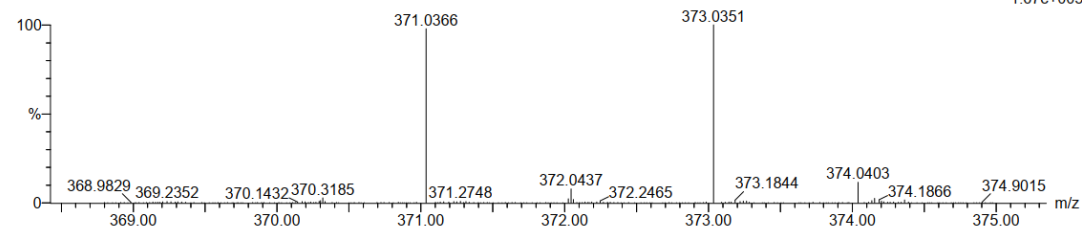
Elements Used:

C: 16-16 H: 17-17 N: 0-100 O: 0-100 Na: 0-7 Br: 1-2

41

250430-16-10 6 (0.077)

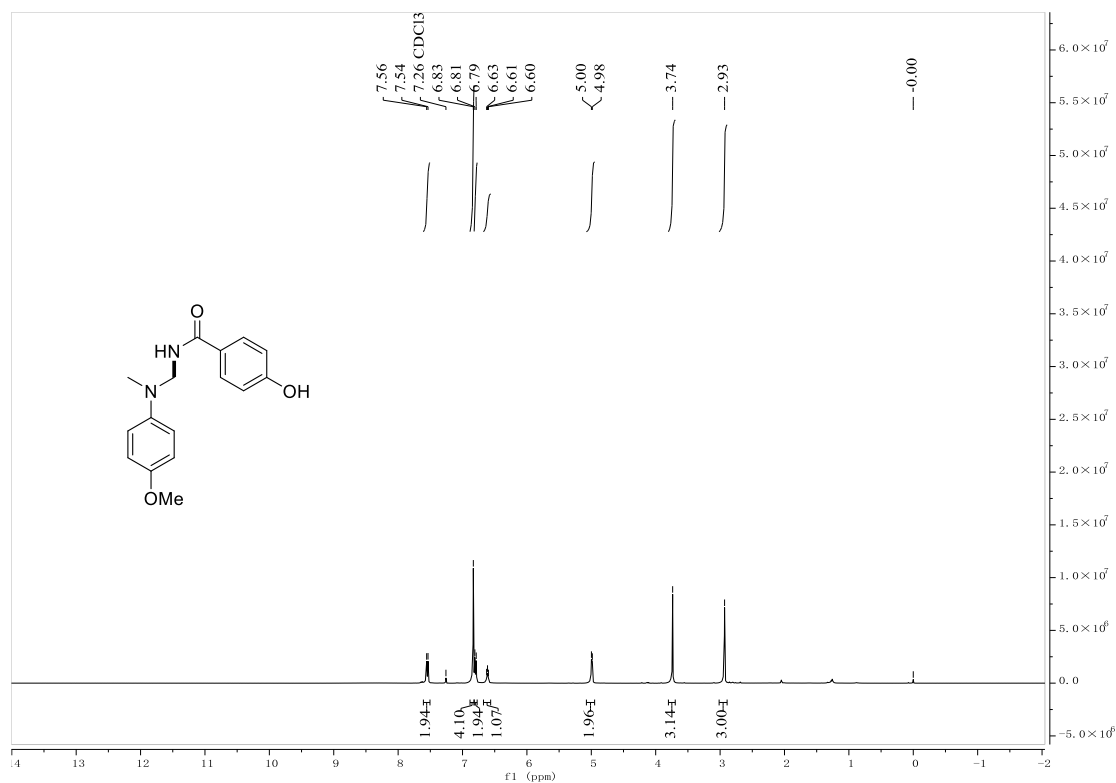
1: TOF MS ES+  
1.67e+005



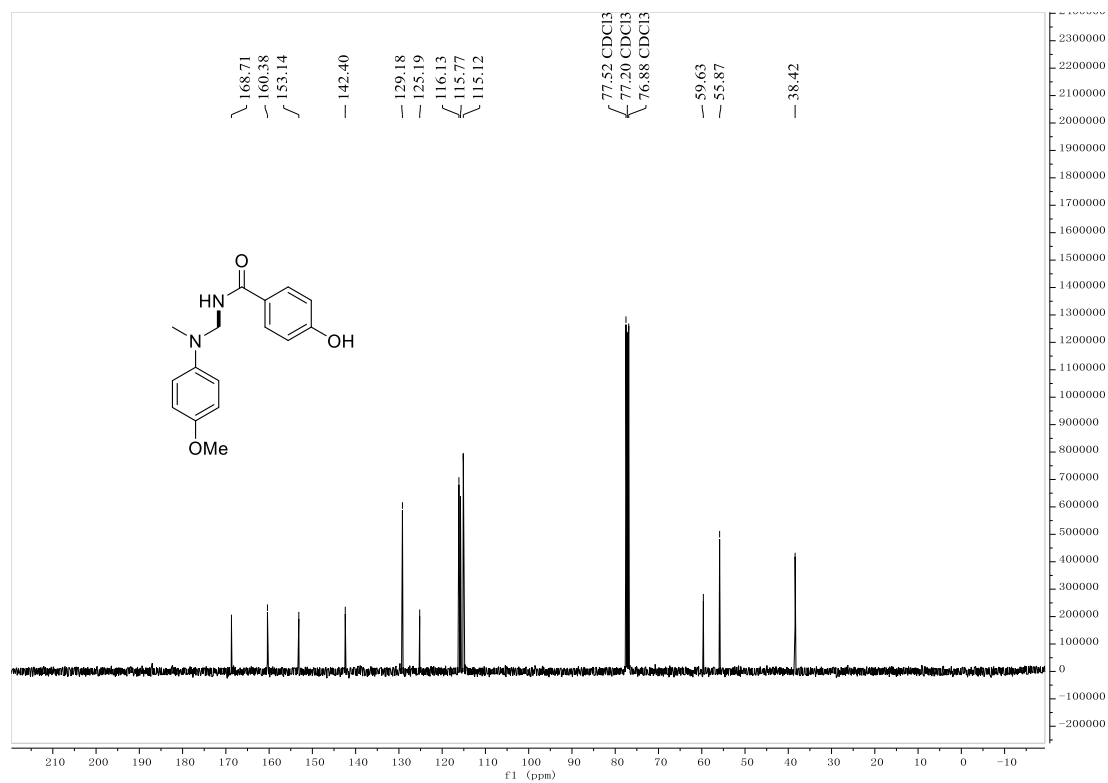
Minimum: -1.5  
Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula                                                            |
|----------|------------|------|------|-----|--------|------|----------|--------------------------------------------------------------------|
| 371.0366 | 371.0371   | -0.5 | -1.3 | 8.5 | 1490.0 | n/a  | n/a      | C <sub>16</sub> H <sub>17</sub> N <sub>2</sub> O <sub>2</sub> NaBr |

# <sup>1</sup>H NMR of 5



# <sup>13</sup>C NMR of 5



## HRMS of 5

Monoisotopic Mass, Even Electron Ions

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

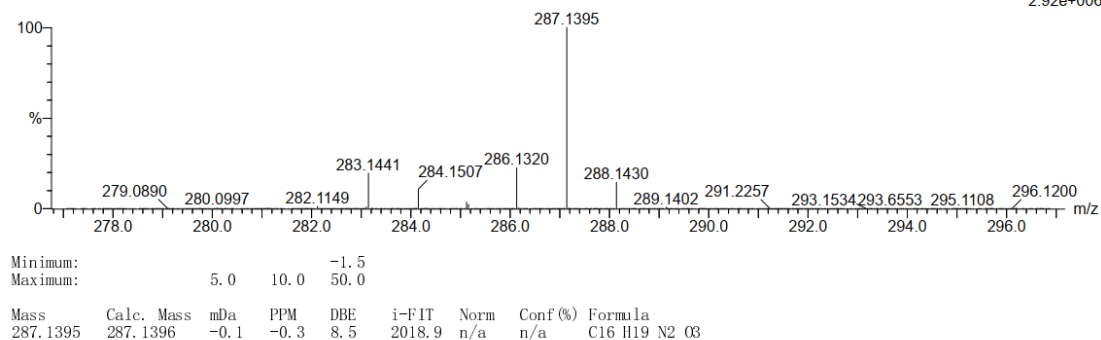
Elements Used:

C: 16-16 H: 19-19 N: 0-100 O: 0-100 Na: 0-7

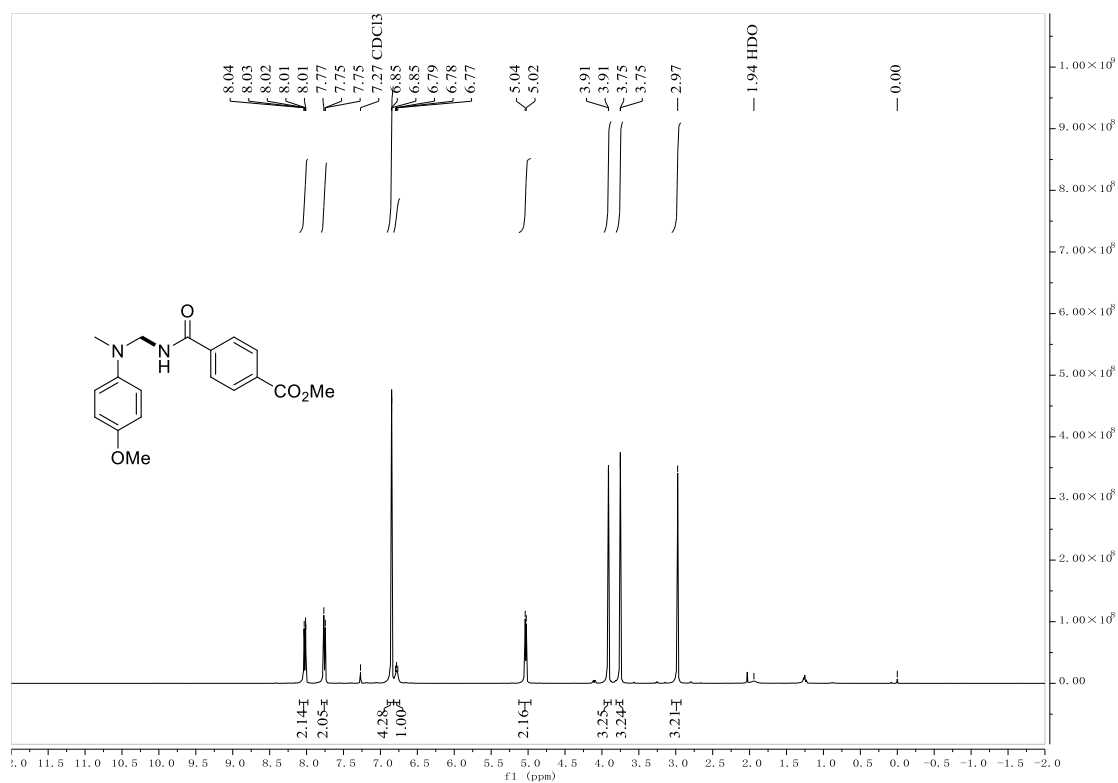
7

250430-16-16 10 (0.111)

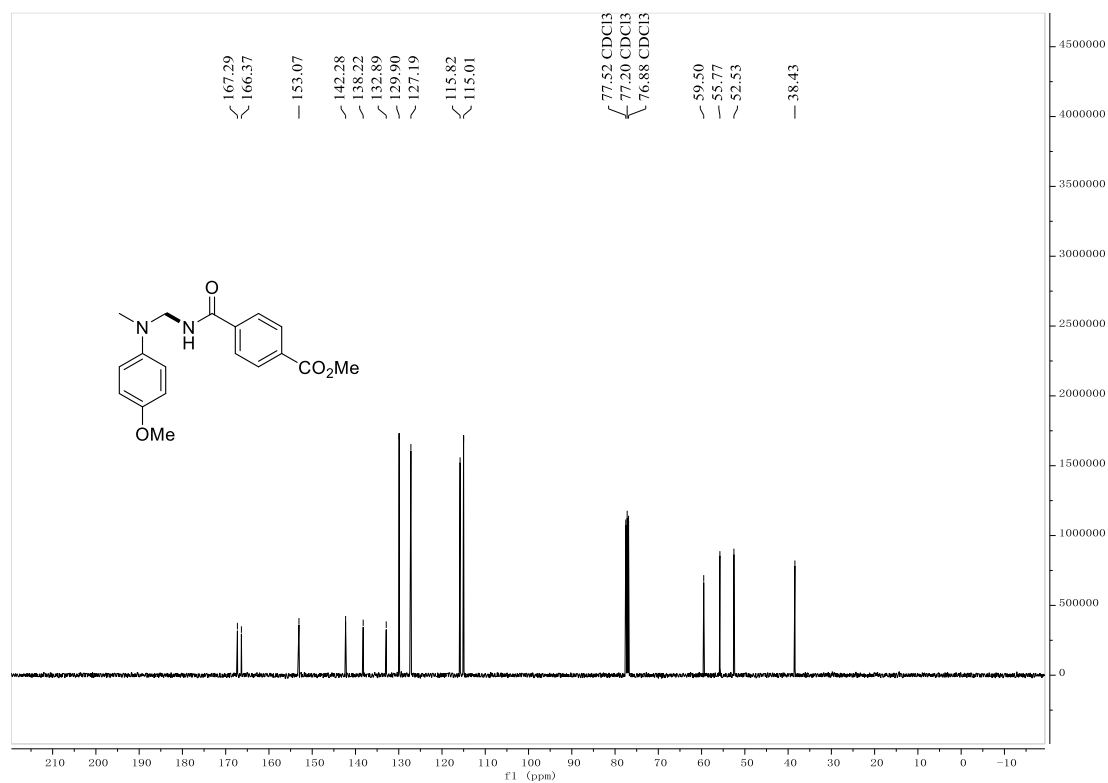
1: TOF MS ES+  
2.92e+006



## <sup>1</sup>H NMR of 6



## <sup>13</sup>C NMR of 6



## HRMS of 6

Monoisotopic Mass, Even Electron Ions

1391 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

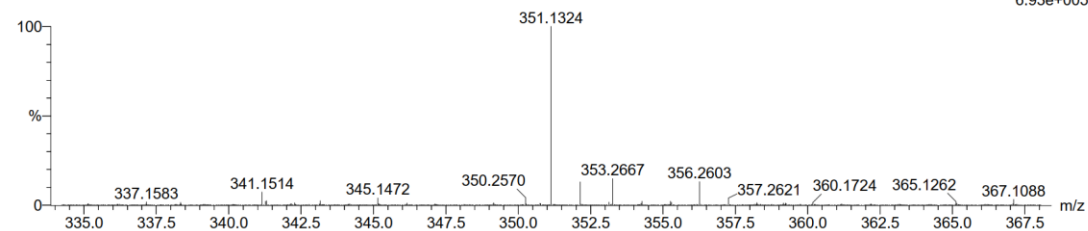
Elements Used:

C: 18-18 H: 20-20 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-34 9 (0.102)

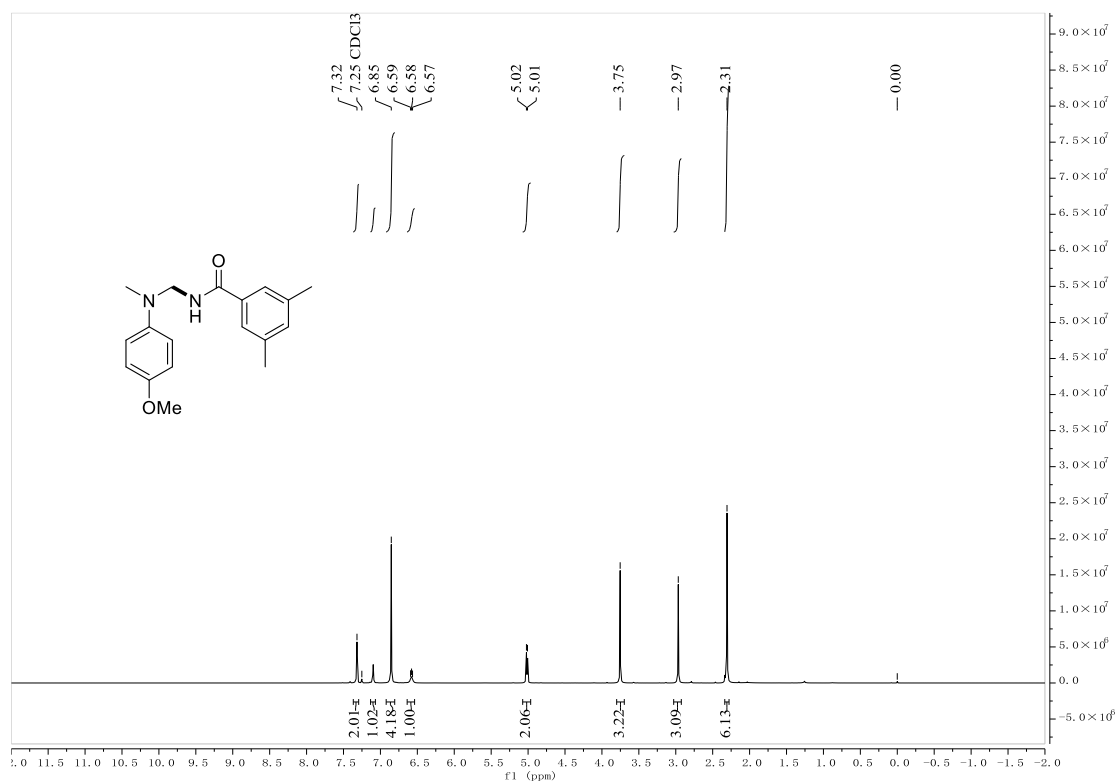
1: TOF MS ES+  
6.93e+005



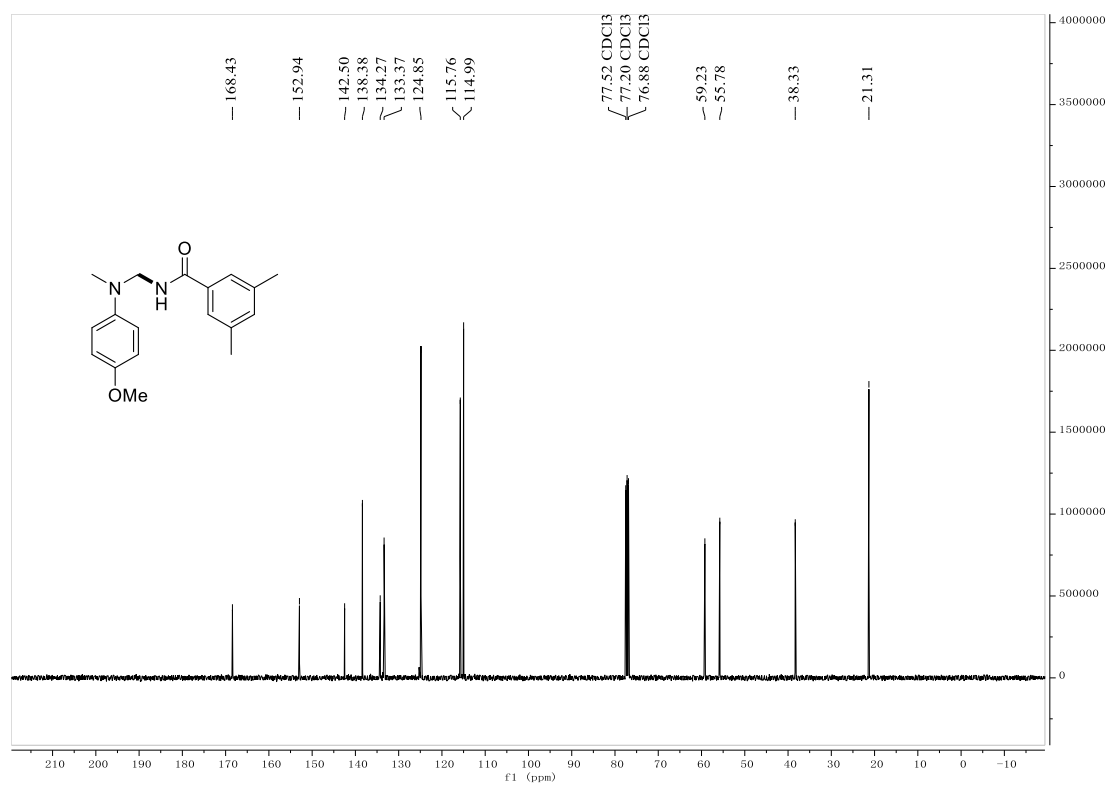
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|-----|--------|------|----------|------------------------------------------------------------------|
| 351.1324 | 351.1321   | 0.3 | 0.9 | 9.5 | 1732.2 | n/a  | n/a      | C <sub>18</sub> H <sub>20</sub> N <sub>2</sub> O <sub>4</sub> Na |

<sup>1</sup>H NMR of 7



<sup>13</sup>C NMR of 7



## HRMS of 7

Monoisotopic Mass, Even Electron Ions

1147 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

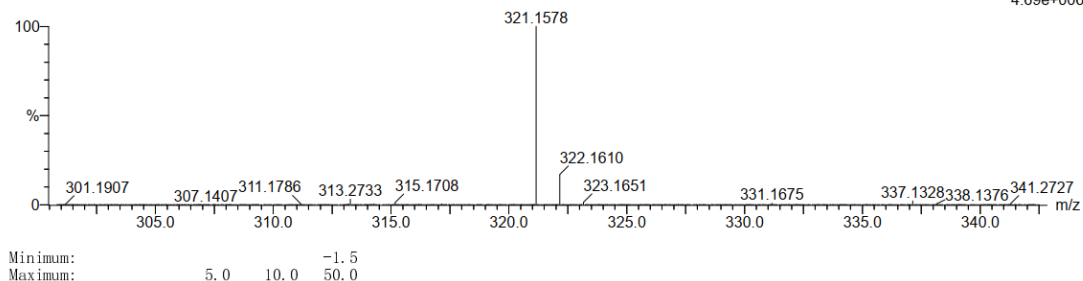
Elements Used:

C: 18-18 H: 22-22 N: 0-100 O: 0-100 Na: 0-7

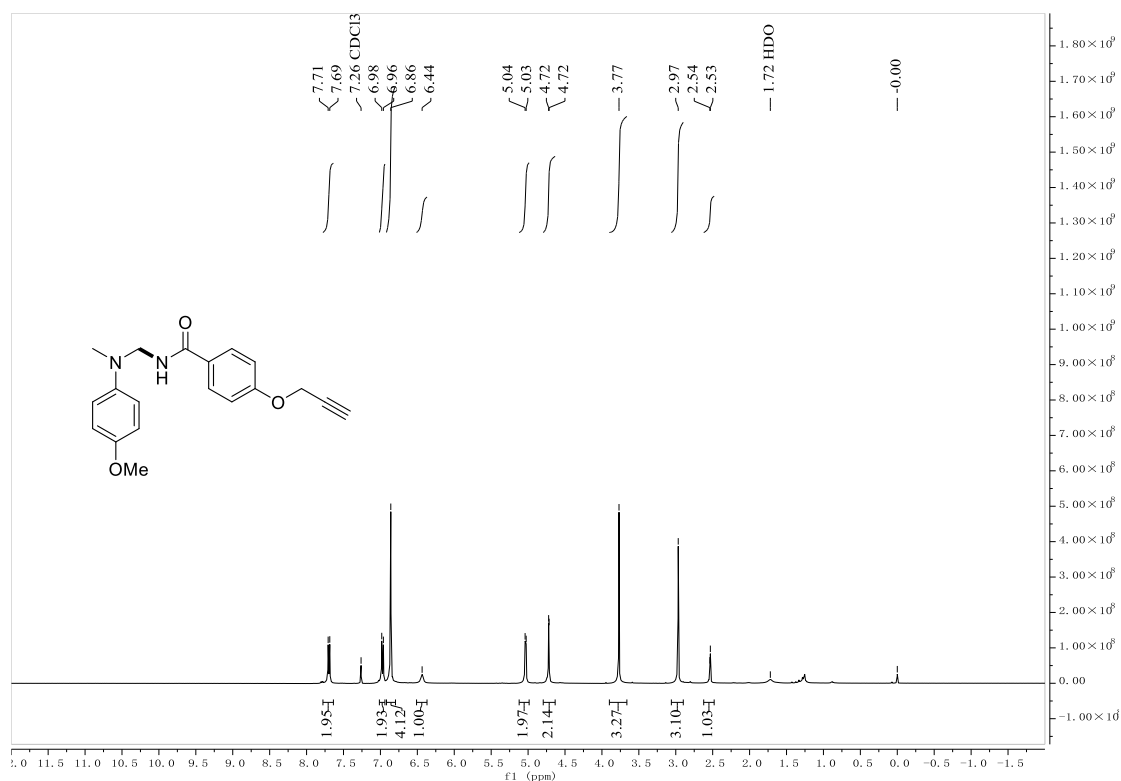
7

250430-16-30 8 (0.094)

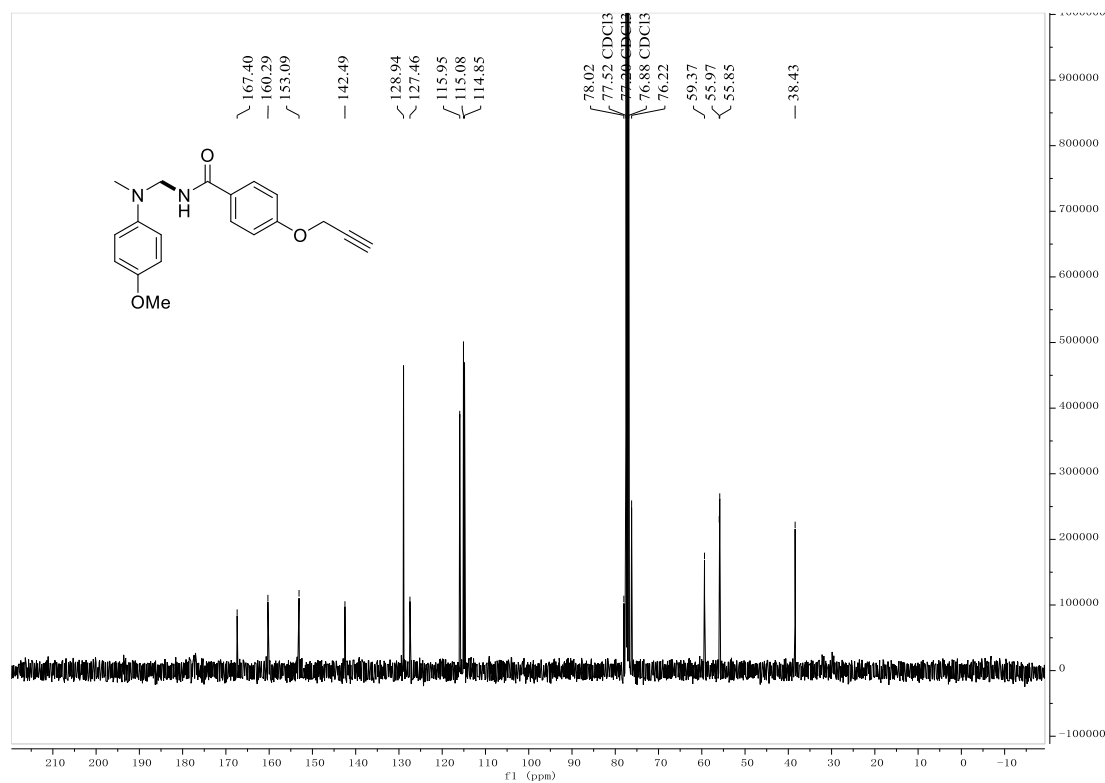
1: TOF MS ES+  
4.69e+006



## <sup>1</sup>H NMR of 8



## <sup>13</sup>C NMR of **8**



## HRMS of **8**

Monoisotopic Mass, Even Electron Ions

1374 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

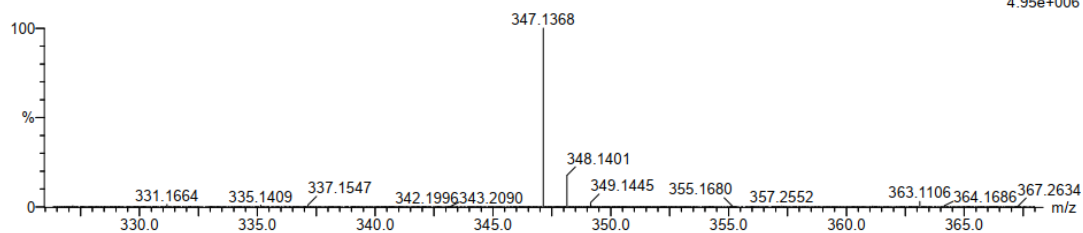
Elements Used:

C: 19-19 H: 20-20 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-19 6 (0.077)

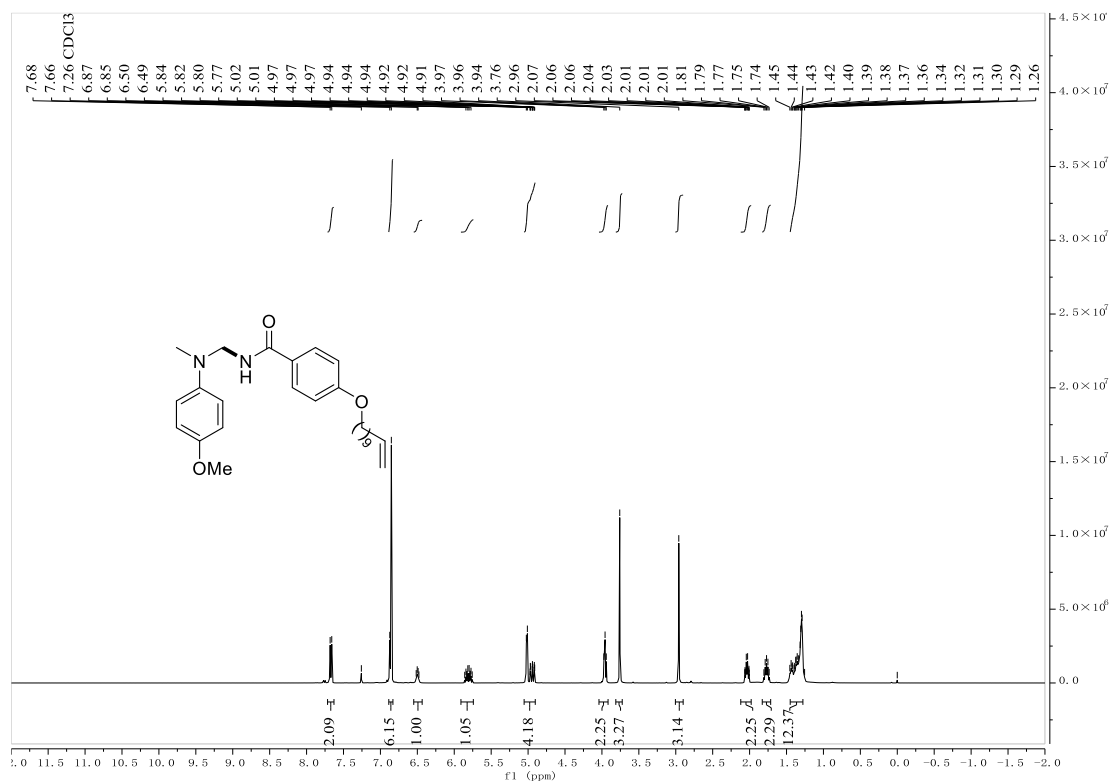
1: TOF MS ES+  
4.95e+006



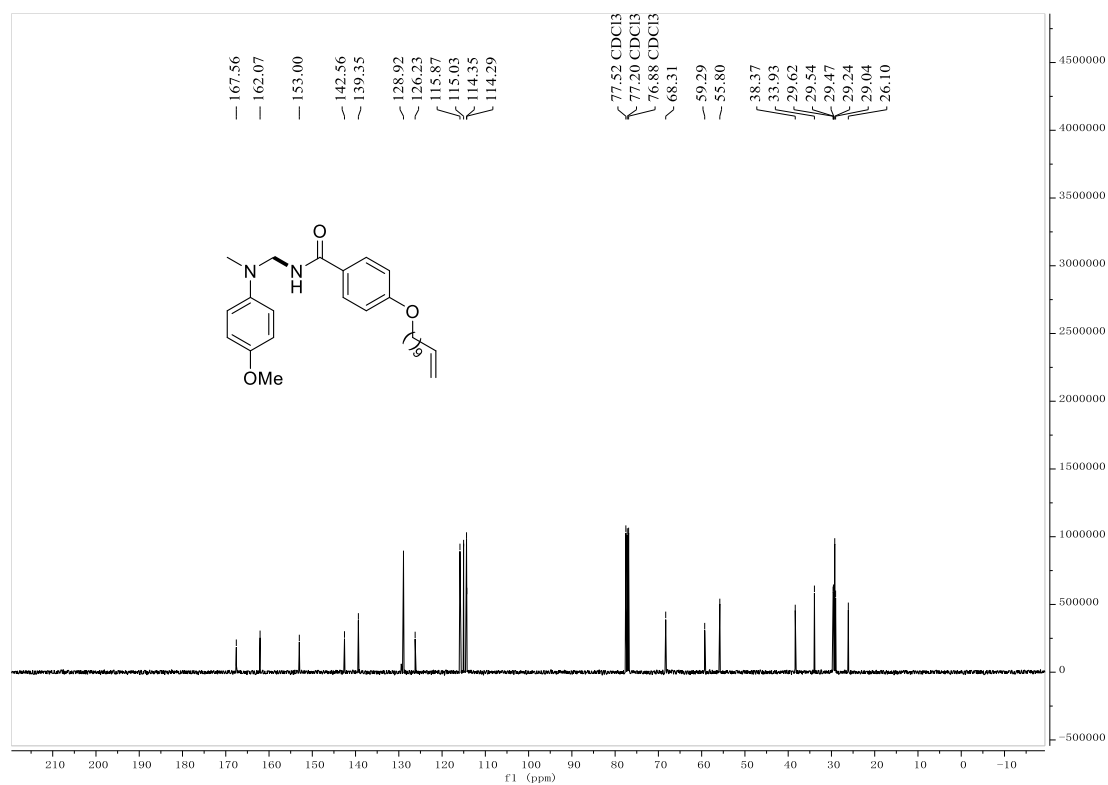
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|------|------|------|--------|------|----------|------------------------------------------------------------------|
| 347.1368 | 347.1372   | -0.4 | -1.2 | 10.5 | 1986.2 | n/a  | n/a      | C <sub>19</sub> H <sub>20</sub> N <sub>2</sub> O <sub>3</sub> Na |

# <sup>1</sup>H NMR of **9**



# <sup>13</sup>C NMR of **9**



## HRMS of 9

Monoisotopic Mass, Even Electron Ions

1164 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

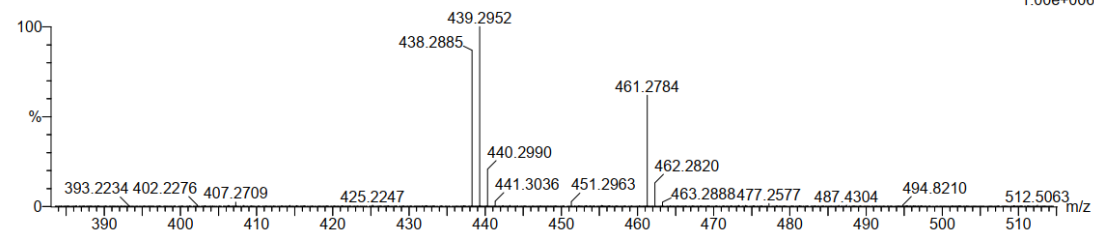
Elements Used:

C: 27-27 H: 39-39 N: 0-100 O: 0-100 Na: 0-2

7

250430-16.40 15 (0.154)

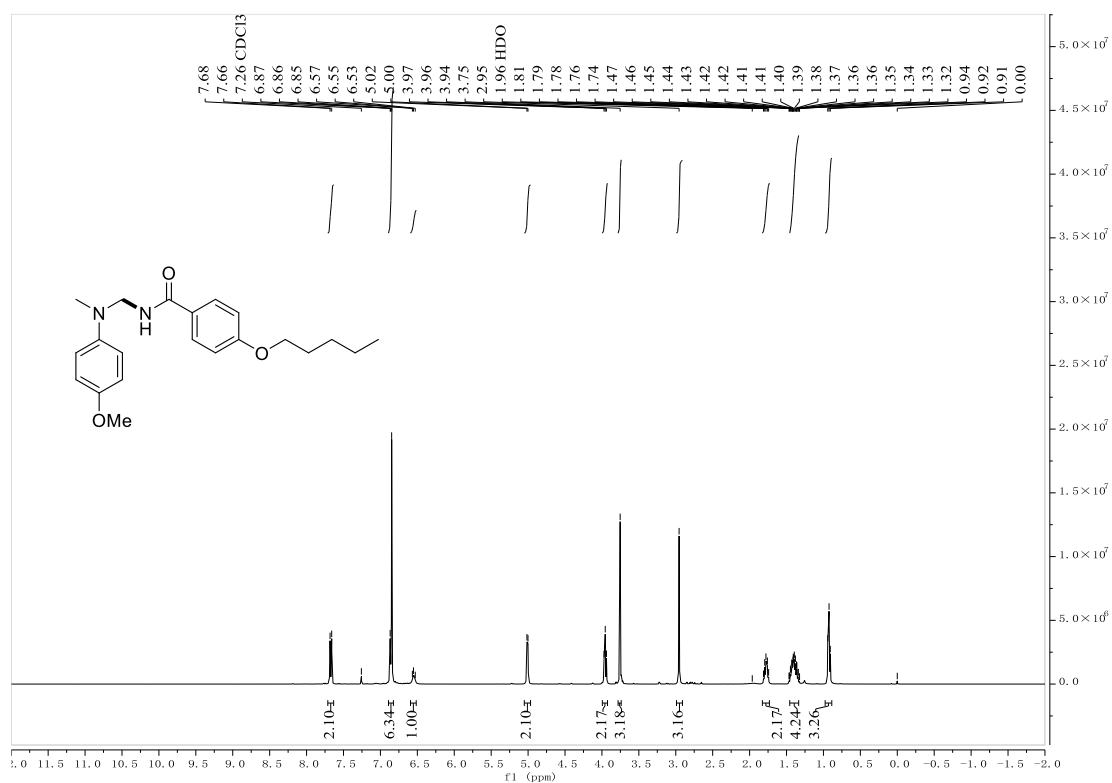
1: TOF MS ES+  
1.00e+006



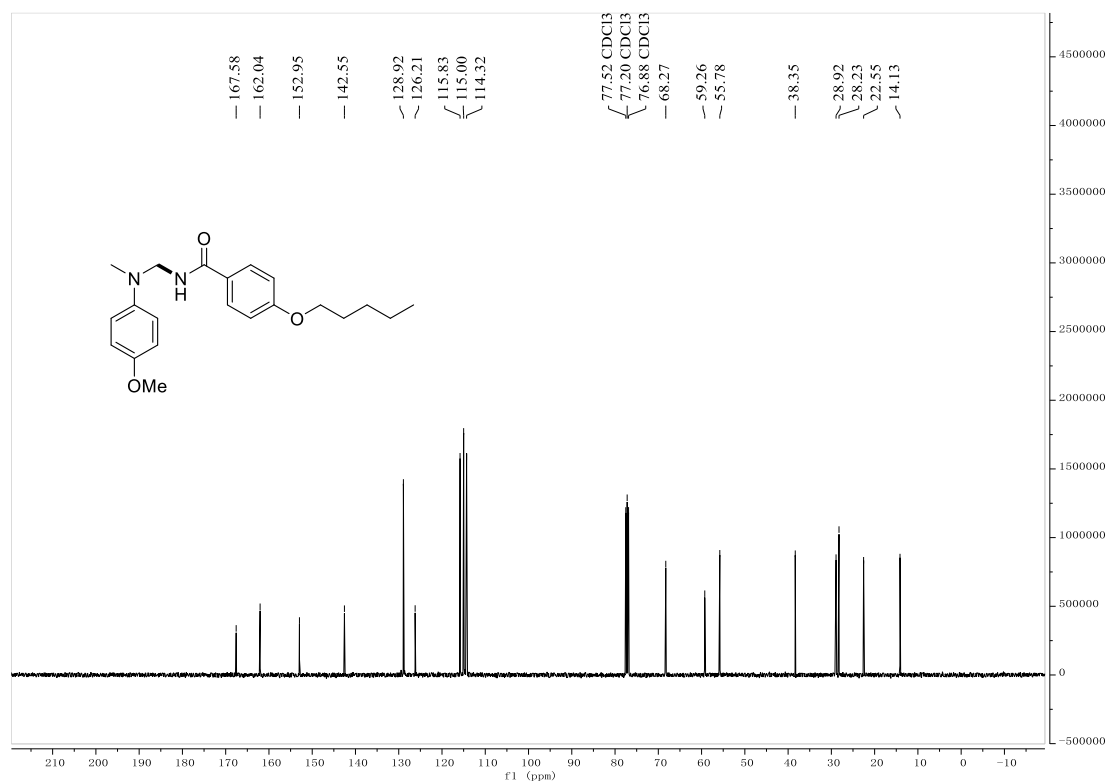
Minimum: -1.5  
Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT | Norm | Conf (%) | Formula       |
|----------|------------|------|------|-----|-------|------|----------|---------------|
| 439.2952 | 439.2961   | -0.9 | -2.0 | 9.5 | 829.4 | n/a  | n/a      | C27 H39 N2 O3 |

## <sup>1</sup>H NMR of 10



## <sup>13</sup>C NMR of **10**



## HRMS of **10**

Monoisotopic Mass, Even Electron Ions

1694 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

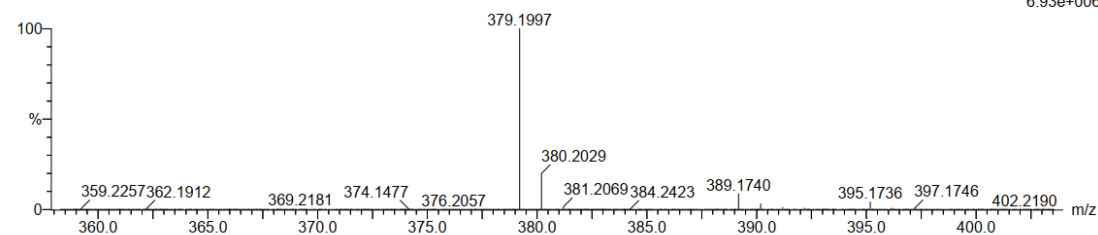
Elements Used:

C: 21-21 H: 28-28 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-32 9 (0.102)

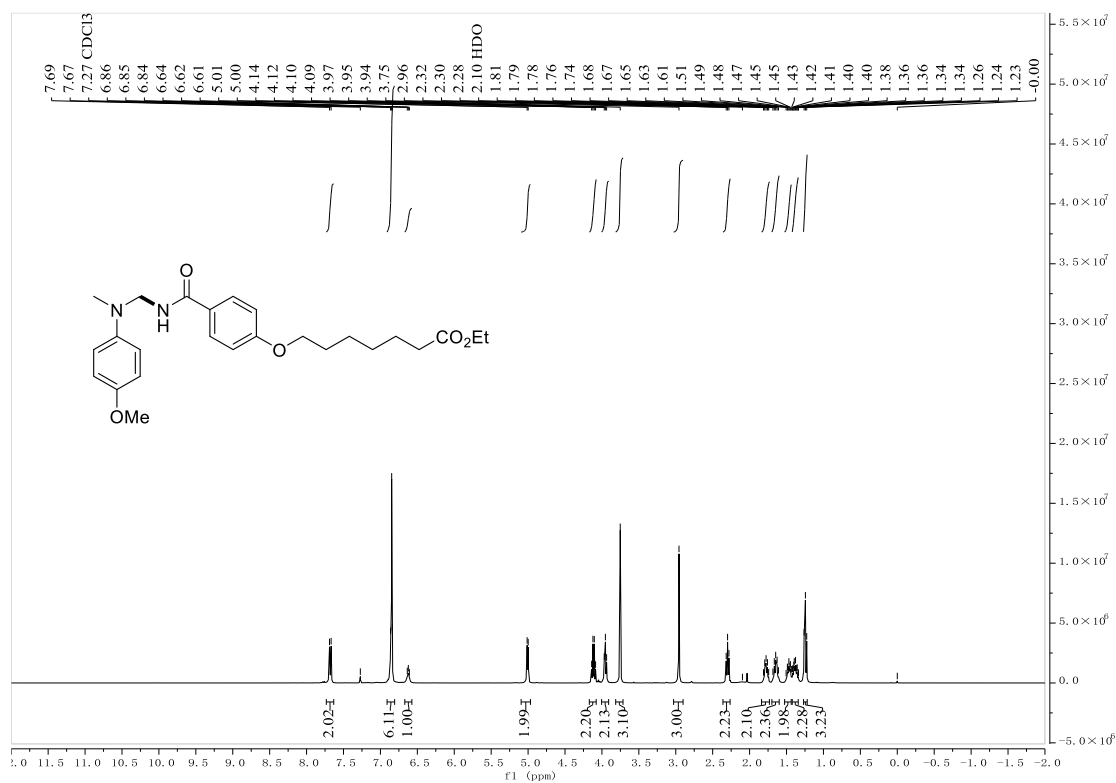
1: TOF MS ES+  
6.93e+006



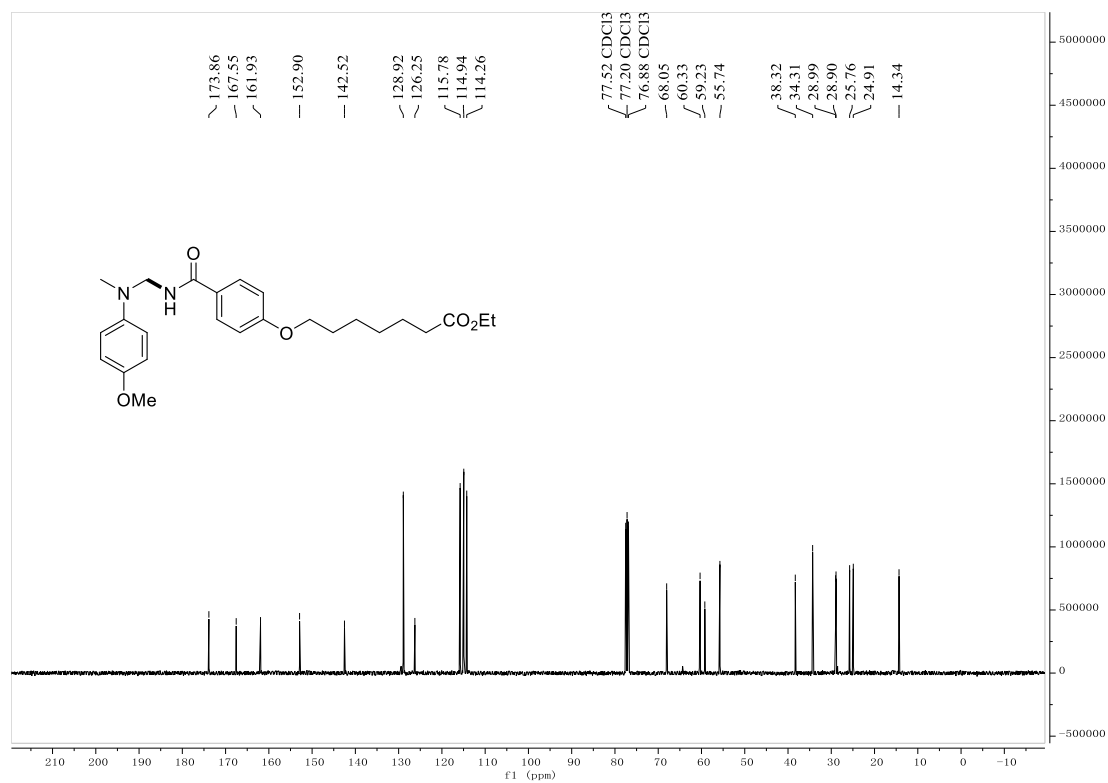
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|------|------|-----|--------|------|----------|------------------------------------------------------------------|
| 379.1997 | 379.1998   | -0.1 | -0.3 | 8.5 | 2030.4 | n/a  | n/a      | C <sub>21</sub> H <sub>28</sub> N <sub>2</sub> O <sub>3</sub> Na |

# <sup>1</sup>H NMR of 11



# <sup>13</sup>C NMR of 11



## HRMS of 11

Monoisotopic Mass, Even Electron Ions

2692 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

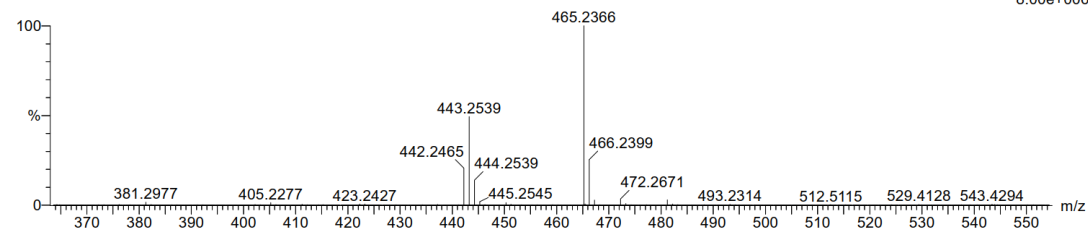
Elements Used:

C: 25-25 H: 34-34 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-33 11 (0.119)

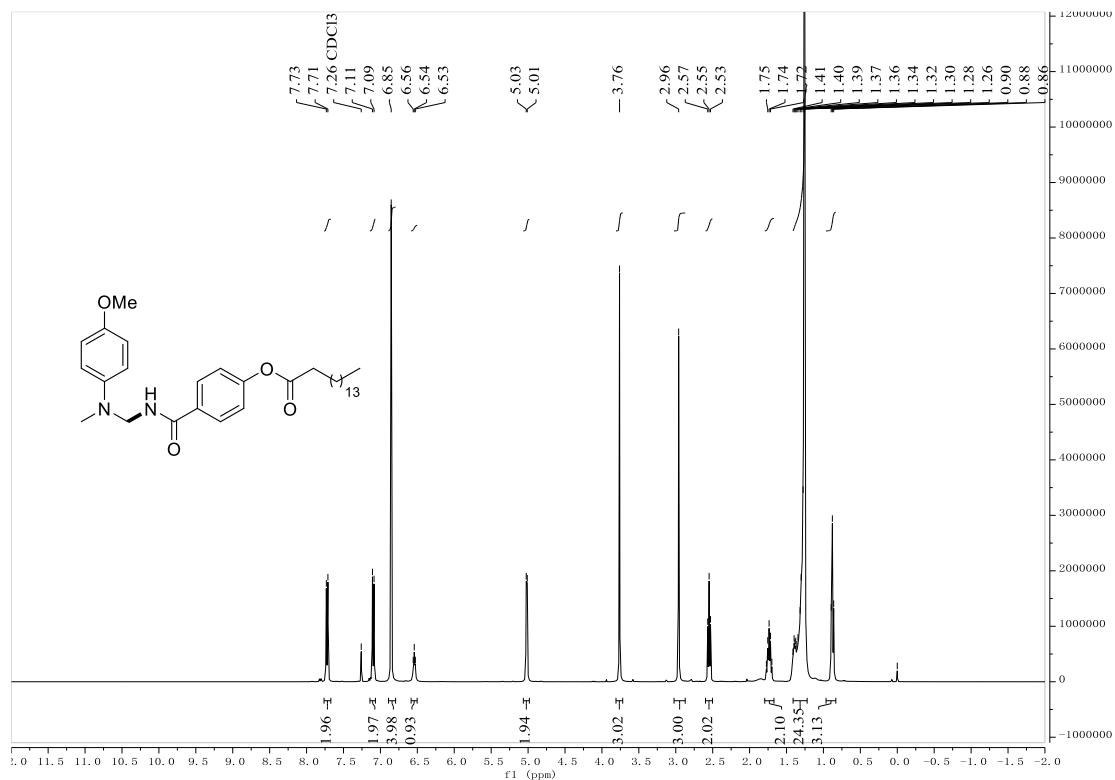
1: TOF MS ES+  
8.00e+006



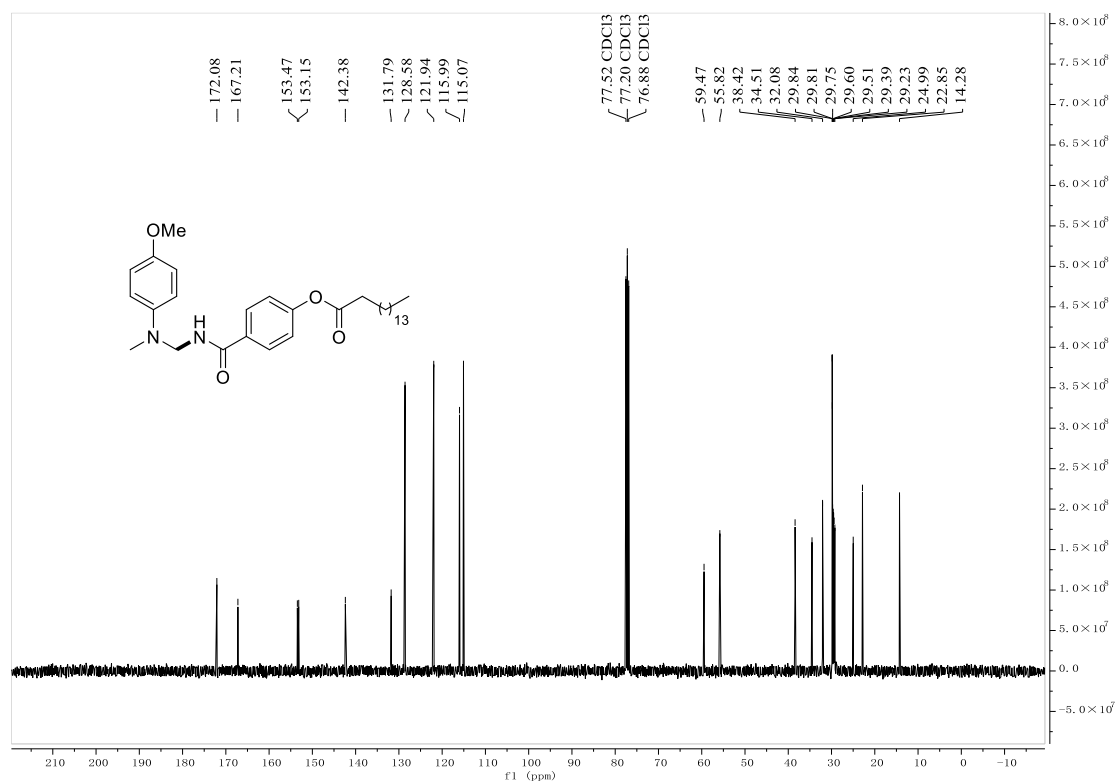
Minimum: -1.5  
Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT  | Norm | Conf(%) | Formula                                                          |
|----------|------------|-----|-----|-----|--------|------|---------|------------------------------------------------------------------|
| 465.2366 | 465.2365   | 0.1 | 0.2 | 9.5 | 1863.0 | n/a  | n/a     | C <sub>25</sub> H <sub>34</sub> N <sub>2</sub> O <sub>5</sub> Na |

## <sup>1</sup>H NMR of 12



## <sup>13</sup>C NMR of 12



## HRMS of 12

Monoisotopic Mass, Even Electron Ions

1667 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

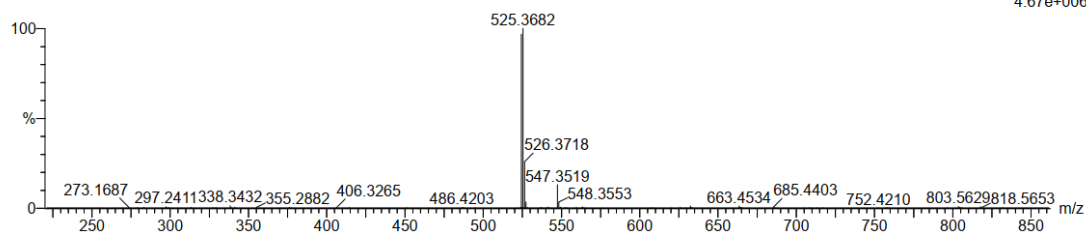
Elements Used:

C: 32-32 H: 49-49 N: 0-100 O: 0-100 Na: 0-2

7

250430-16-44 24 (0.239)

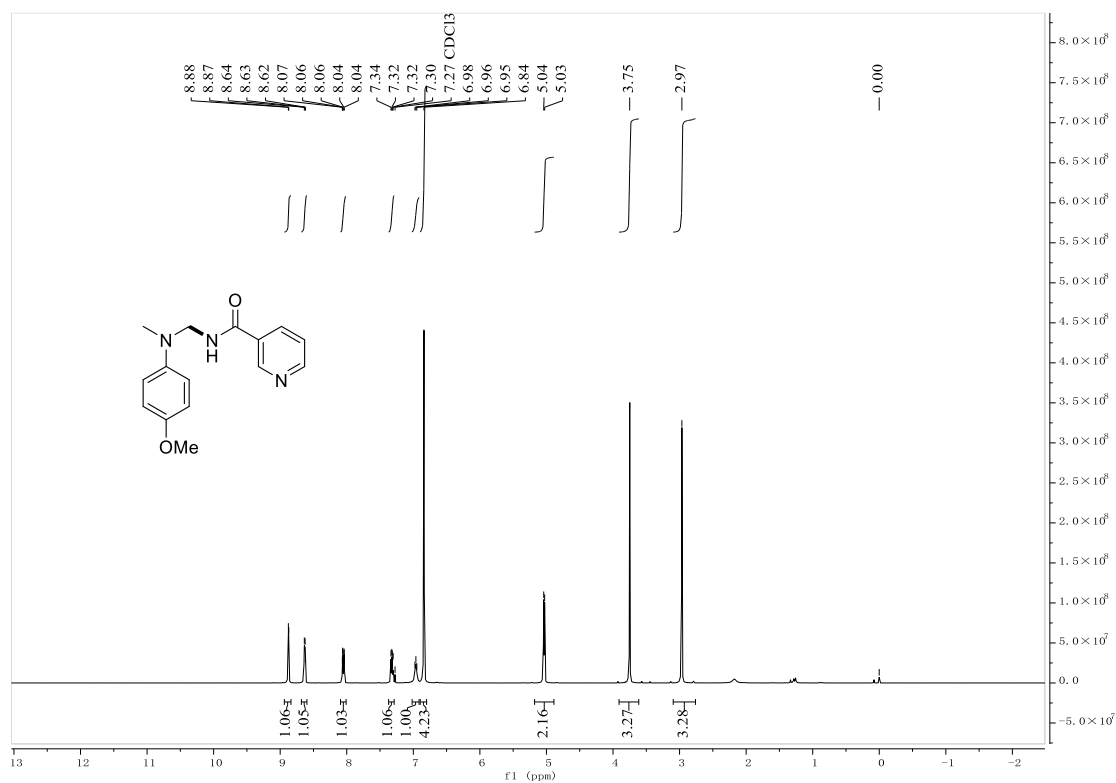
1: TOF MS ES+  
4.67e+006



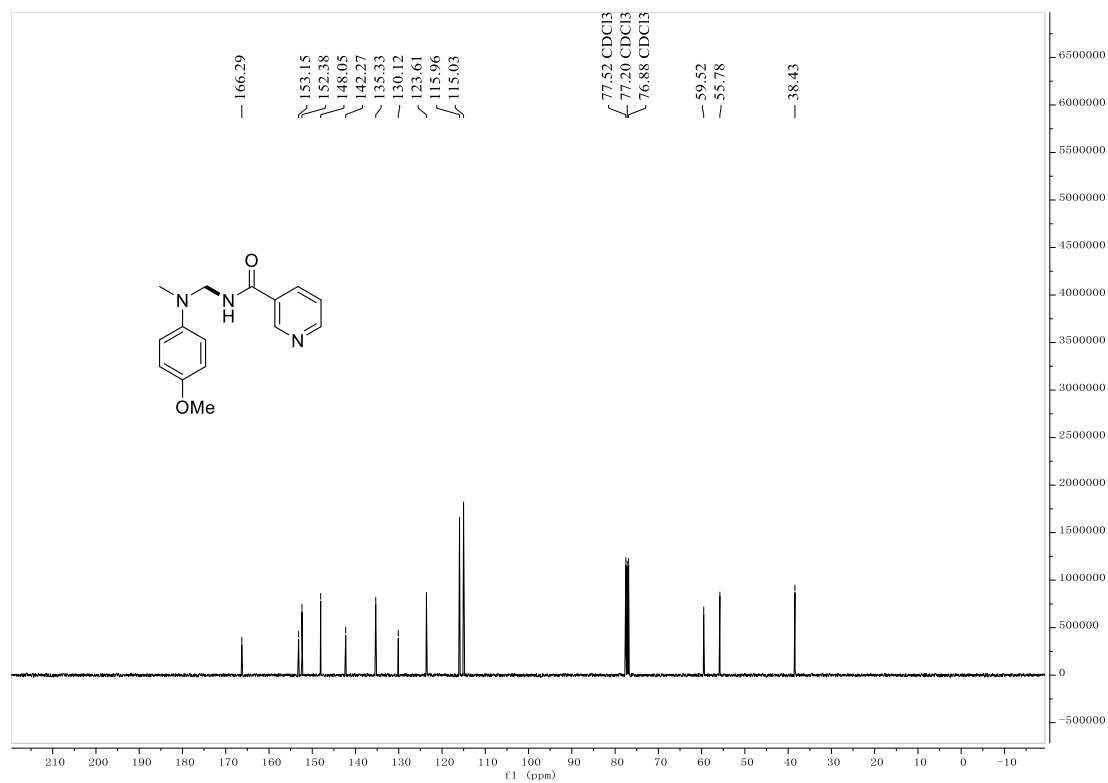
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT | Norm | Conf (%) | Formula                                                       |
|----------|------------|------|------|-----|-------|------|----------|---------------------------------------------------------------|
| 525.3682 | 525.3692   | -1.0 | -1.9 | 9.5 | 898.6 | n/a  | n/a      | C <sub>32</sub> H <sub>49</sub> N <sub>2</sub> O <sub>4</sub> |

<sup>1</sup>H NMR of **13**



<sup>13</sup>C NMR of **13**



## HRMS of 13

Monoisotopic Mass, Even Electron Ions

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

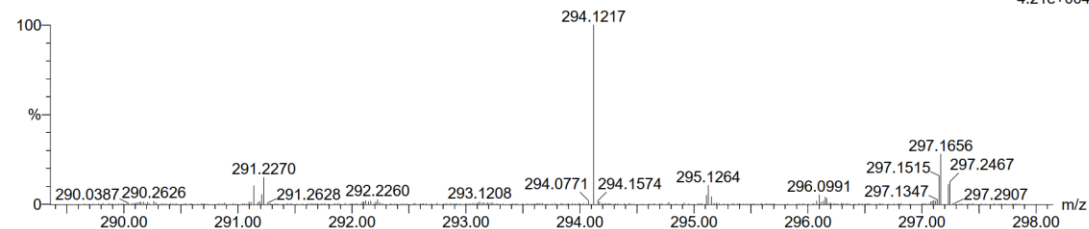
Elements Used:

C: 15-15 H: 17-17 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-14 9 (0.102)

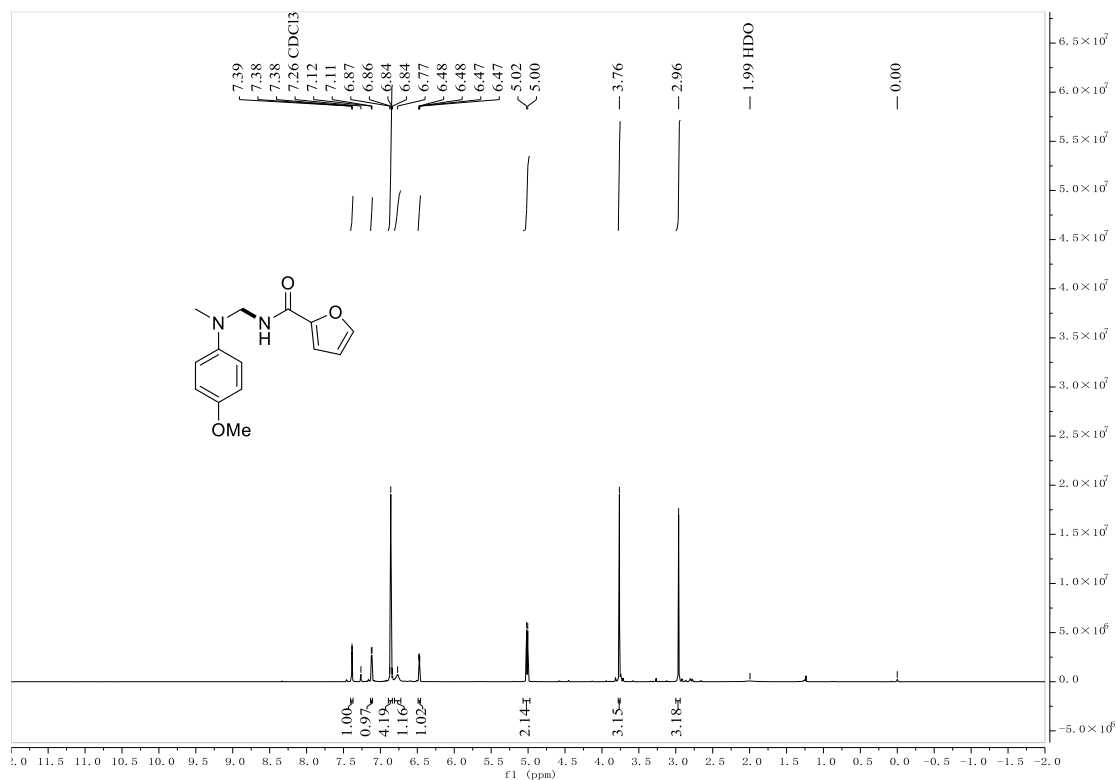
1: TOF MS ES+  
4.21e+004



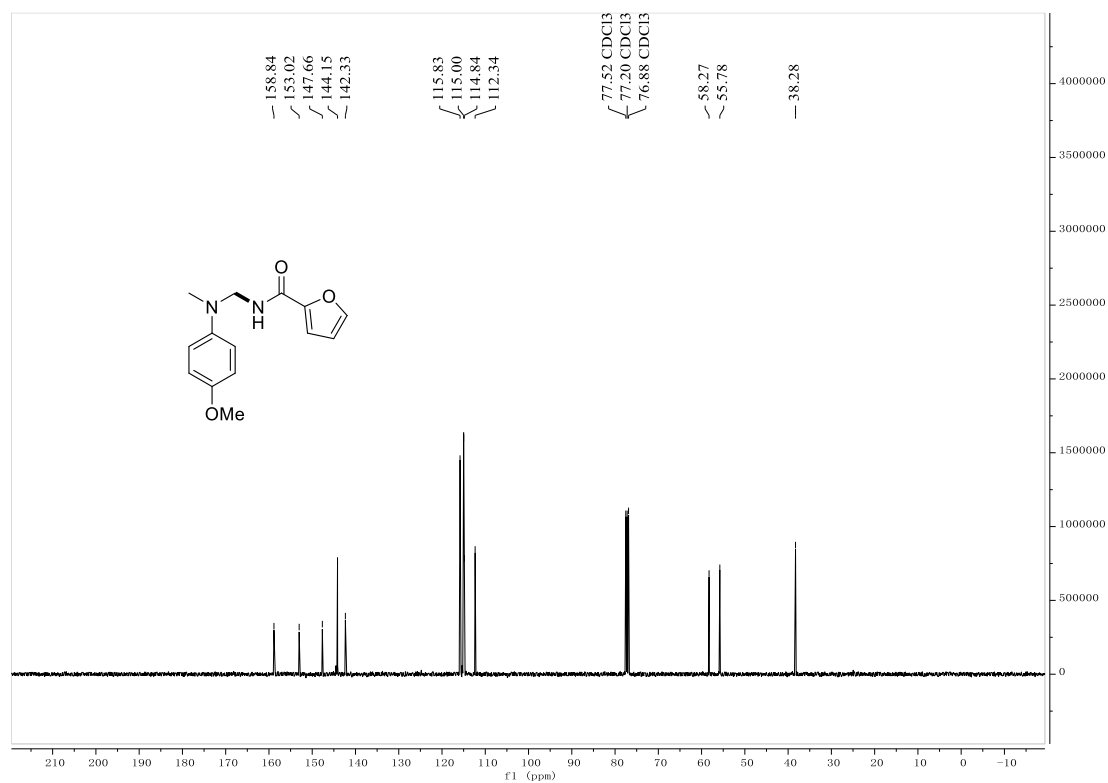
Minimum: -1.5  
Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula          |
|----------|------------|------|------|-----|--------|------|----------|------------------|
| 294.1217 | 294.1218   | -0.1 | -0.3 | 8.5 | 1154.0 | n/a  | n/a      | C15 H17 N3 O2 Na |

## <sup>1</sup>H NMR of 14



## <sup>13</sup>C NMR of **14**



## HRMS of **14**

Monoisotopic Mass, Even Electron Ions

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

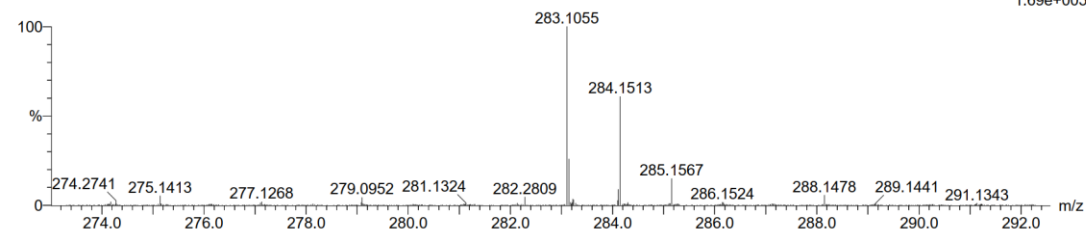
Elements Used:

C: 14-14 H: 16-16 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-42 12 (0.128)

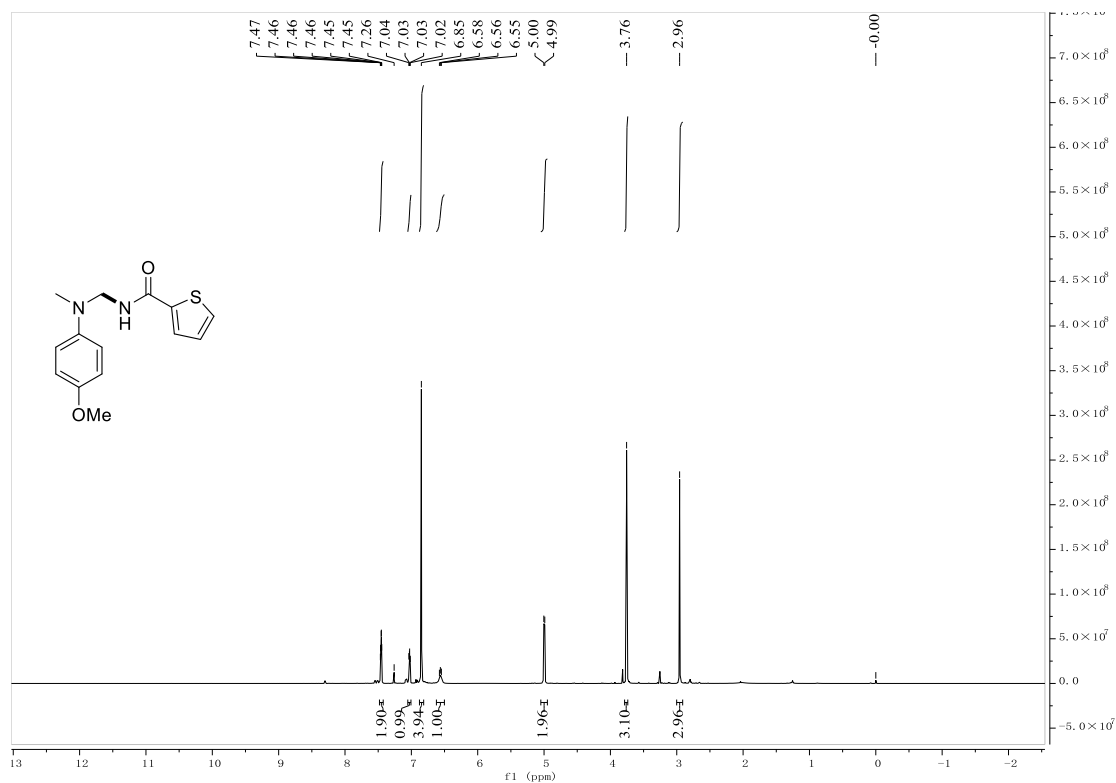
1: TOF MS ES+  
1.69e+005



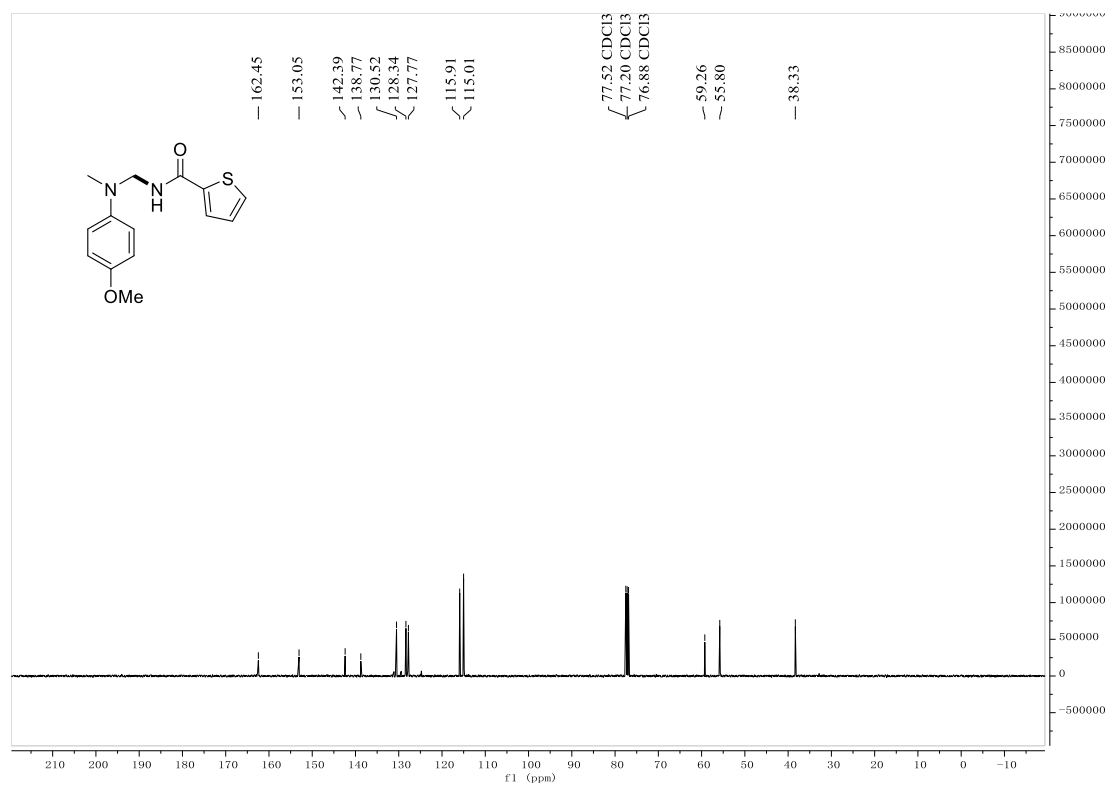
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|------|------|-----|--------|------|----------|------------------------------------------------------------------|
| 283.1055 | 283.1059   | -0.4 | -1.4 | 7.5 | 1645.8 | n/a  | n/a      | C <sub>14</sub> H <sub>16</sub> N <sub>2</sub> O <sub>3</sub> Na |

# <sup>1</sup>H NMR of 15



# <sup>13</sup>C NMR of 15



## HRMS of 15

Monoisotopic Mass, Even Electron Ions

1635 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

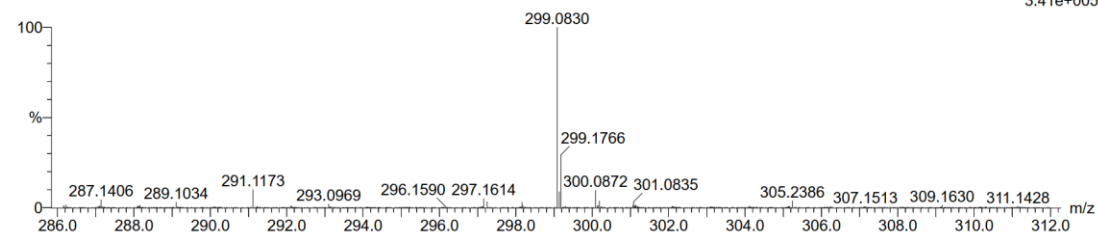
Elements Used:

C: 14-14 H: 16-16 N: 0-100 O: 0-100 Na: 0-7 S: 1-3

7

250430-16-47 10 (0.111)

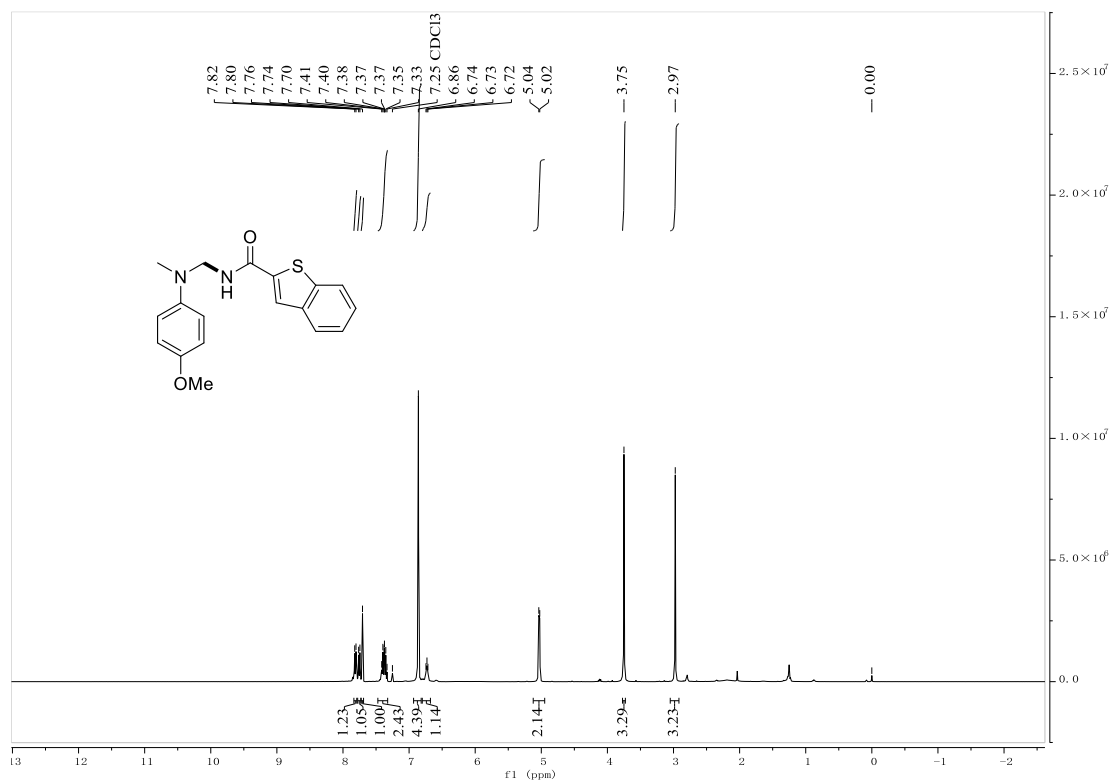
1: TOF MS ES+  
3.41e+005



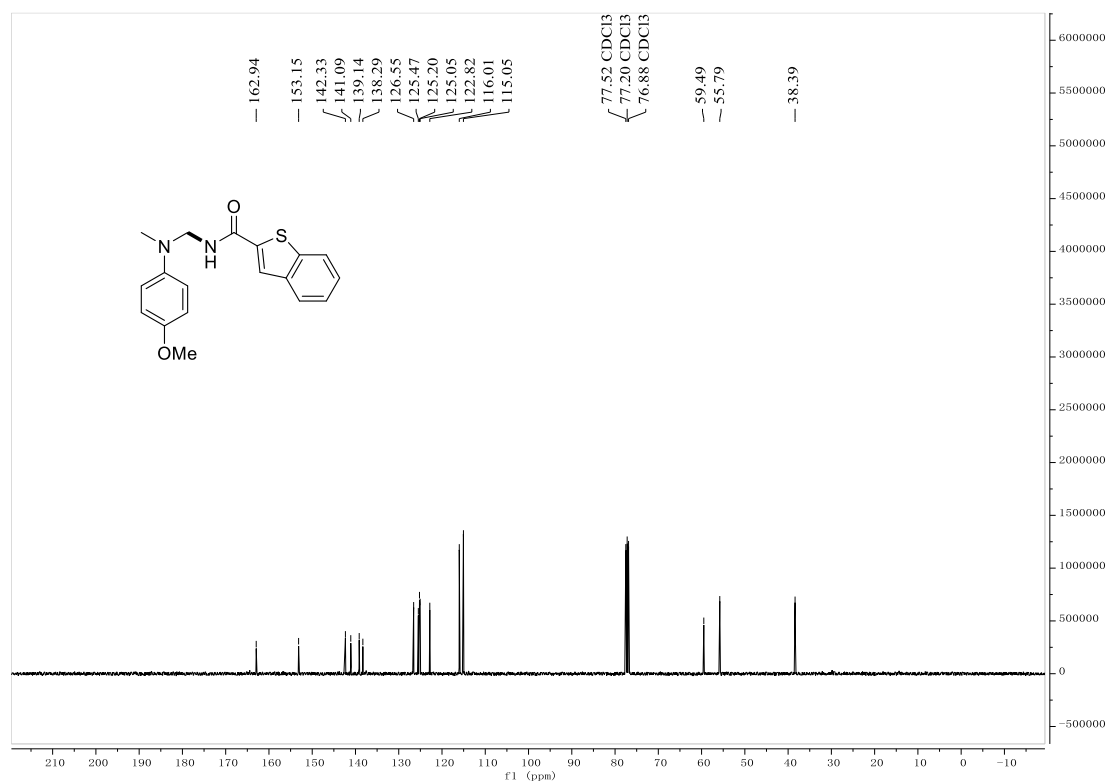
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT  | Norm | Conf (%) | Formula            |
|----------|------------|-----|-----|-----|--------|------|----------|--------------------|
| 299.0830 | 299.0830   | 0.0 | 0.0 | 7.5 | 1711.6 | n/a  | n/a      | C14 H16 N2 O2 Na S |

## <sup>1</sup>H NMR of 16



## <sup>13</sup>C NMR of **16**



## HRMS of **16**

Monoisotopic Mass, Even Electron Ions

2670 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

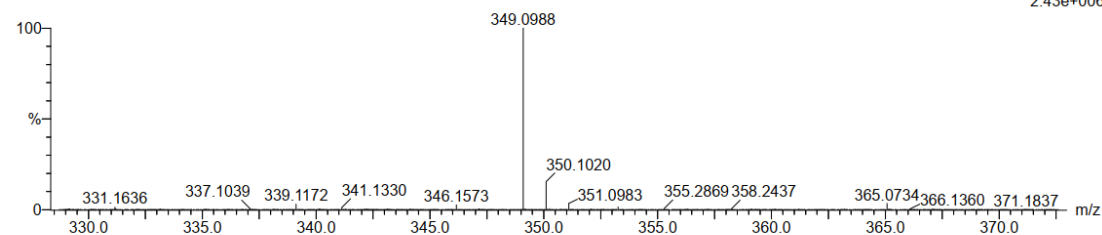
Elements Used:

C: 18-18 H: 18-18 N: 0-100 O: 0-100 Na: 0-7 S: 1-3

41

250430-16-8 9 (0.102)

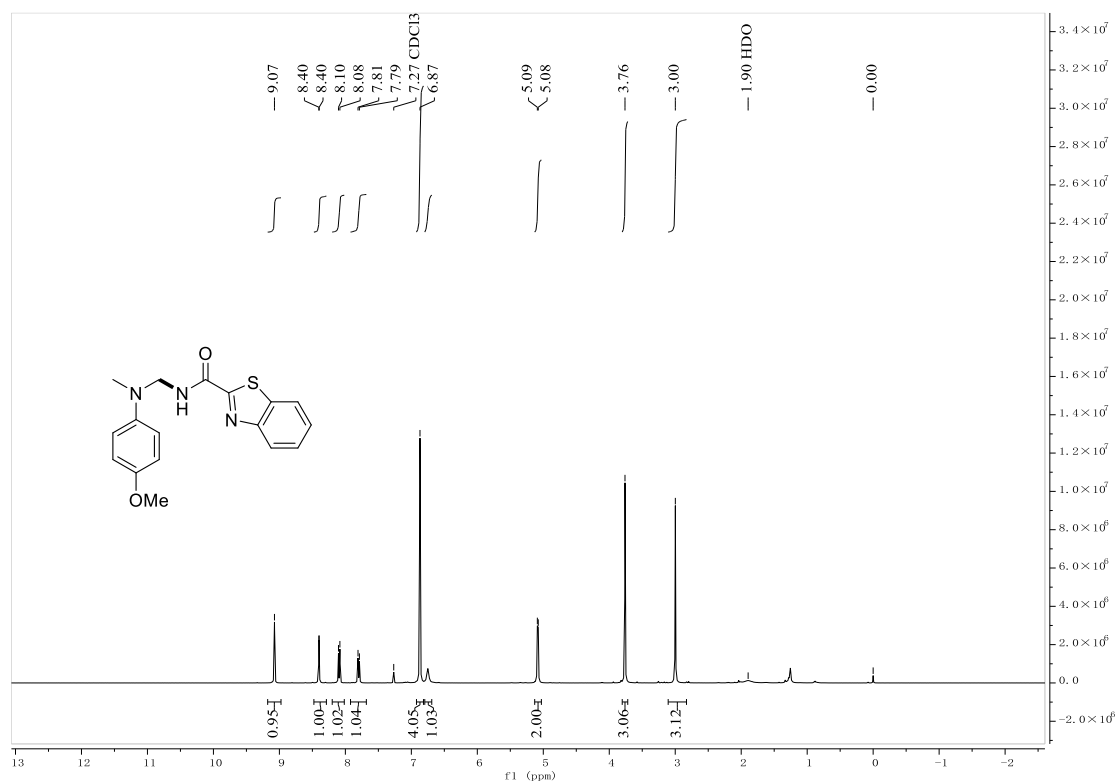
1: TOF MS ES+  
2.43e+006



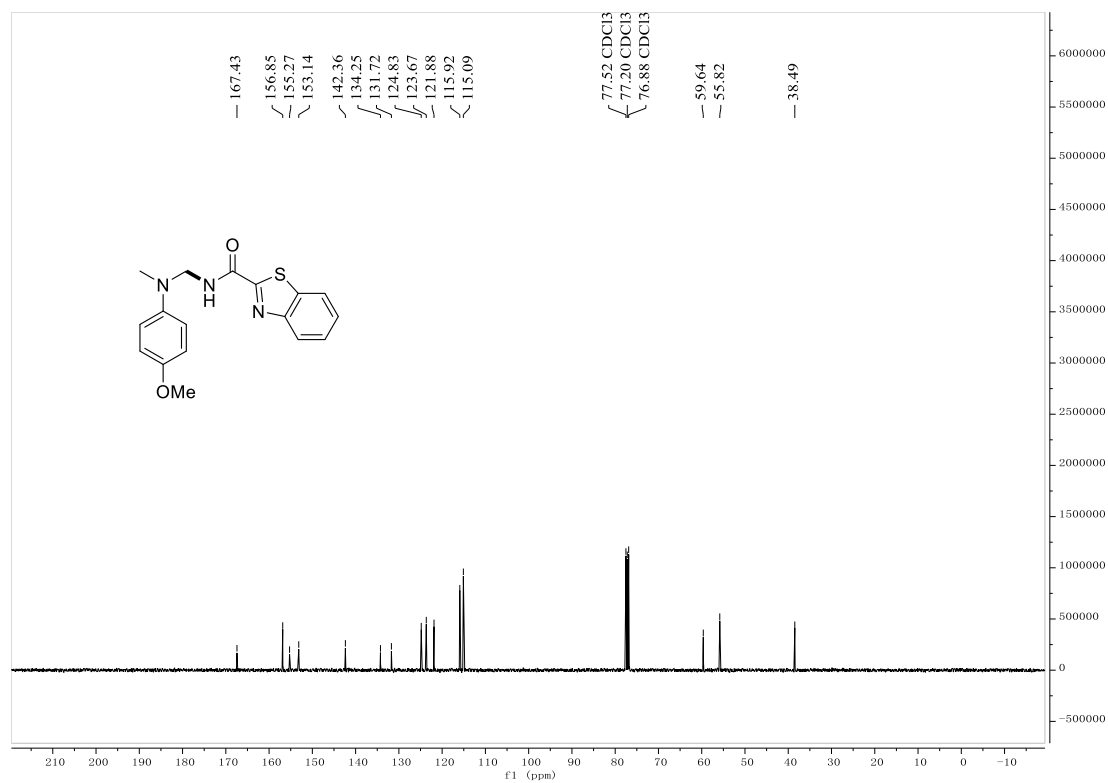
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                           |
|----------|------------|-----|-----|------|--------|------|----------|-------------------------------------------------------------------|
| 349.0988 | 349.0987   | 0.1 | 0.3 | 10.5 | 1890.1 | n/a  | n/a      | C <sub>18</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub> NaS |

# <sup>1</sup>H NMR of 17



# <sup>13</sup>C NMR of 17



## HRMS of 17

Monoisotopic Mass, Even Electron Ions

2655 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

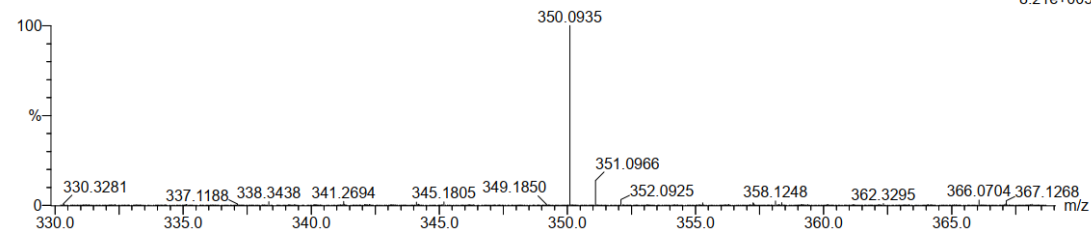
Elements Used:

C: 17-17 H: 17-17 N: 0-100 O: 0-100 Na: 0-7 S: 1-3

41

250430-16-11 5 (0.068)

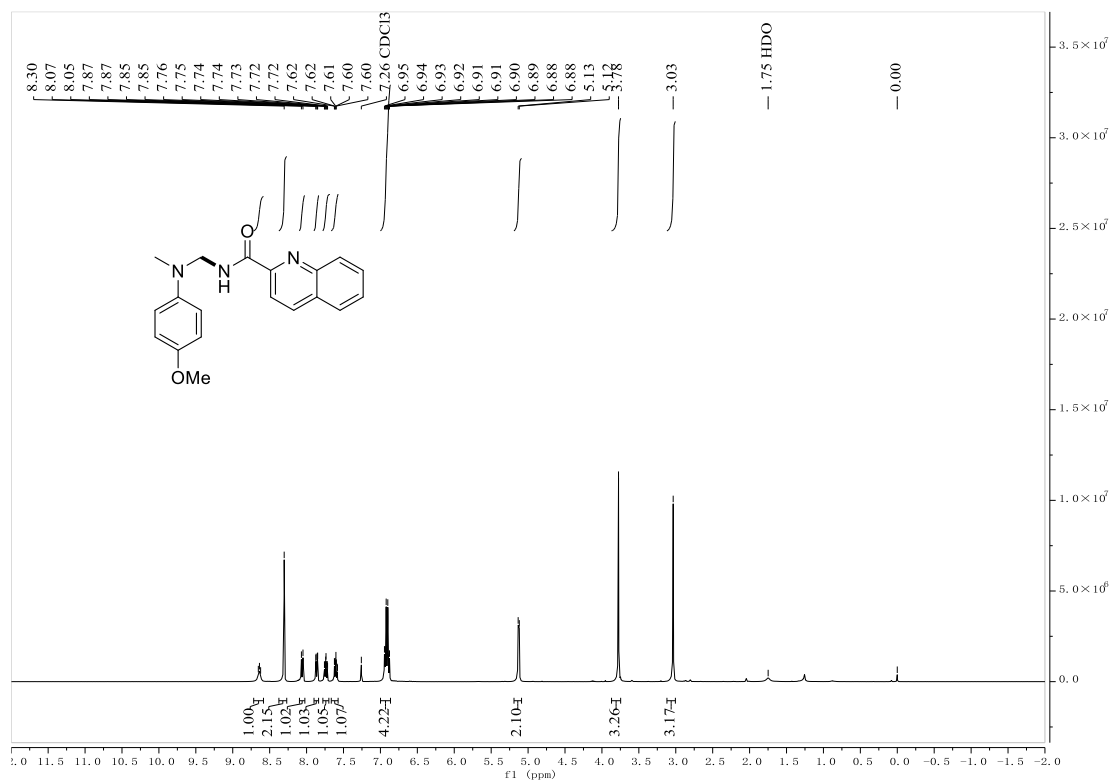
1: TOF MS ES+  
8.21e+005



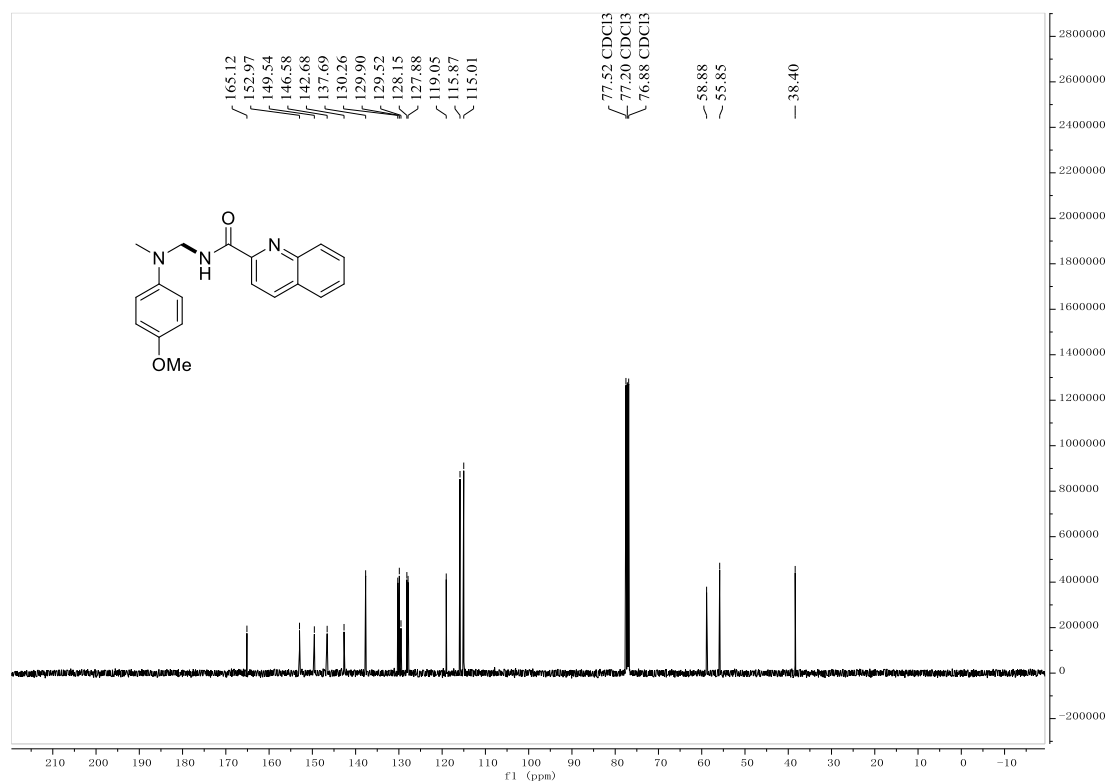
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE  | i-FIT  | Norm | Conf (%) | Formula            |
|----------|------------|------|------|------|--------|------|----------|--------------------|
| 350.0935 | 350.0939   | -0.4 | -1.1 | 10.5 | 1510.0 | n/a  | n/a      | C17 H17 N3 O2 Na S |

## <sup>1</sup>H NMR of 18



## <sup>13</sup>C NMR of **18**



## HRMS of **18**

Monoisotopic Mass, Even Electron Ions

1350 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

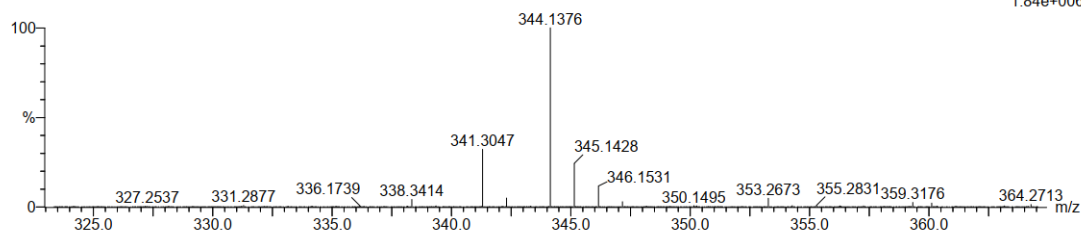
Elements Used:

C: 19-19 H: 19-19 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-31 11 (0.119)

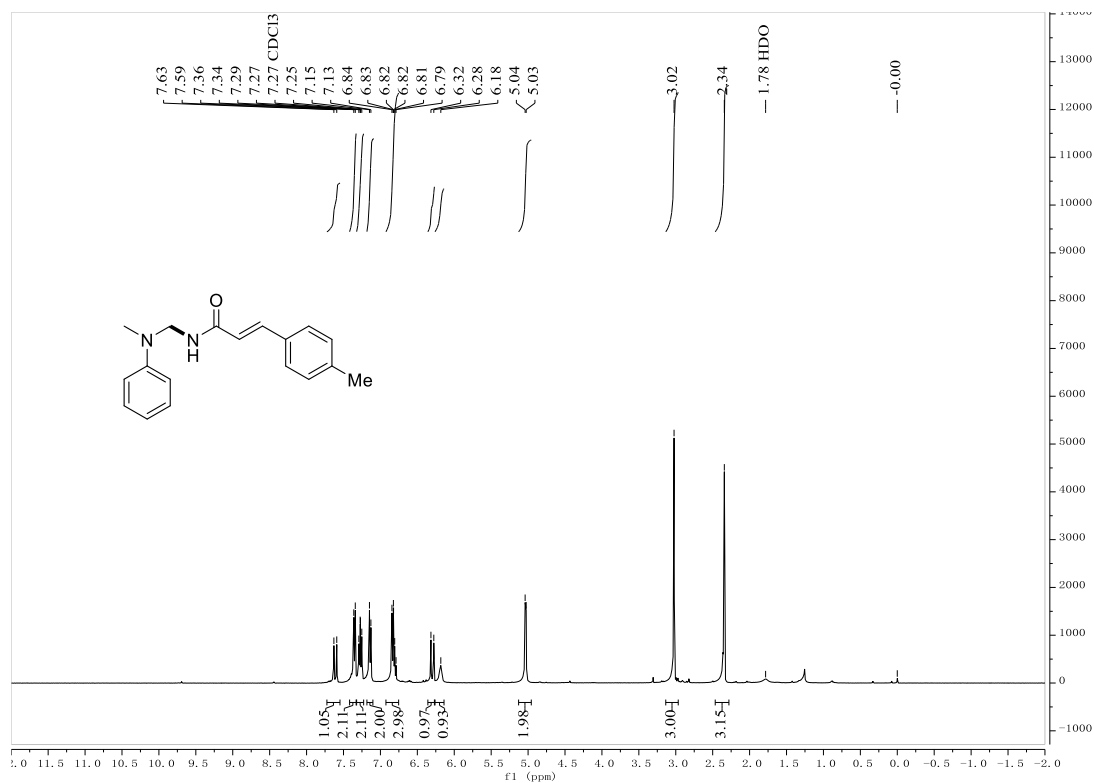
1: TOF MS ES+  
1.84e+006



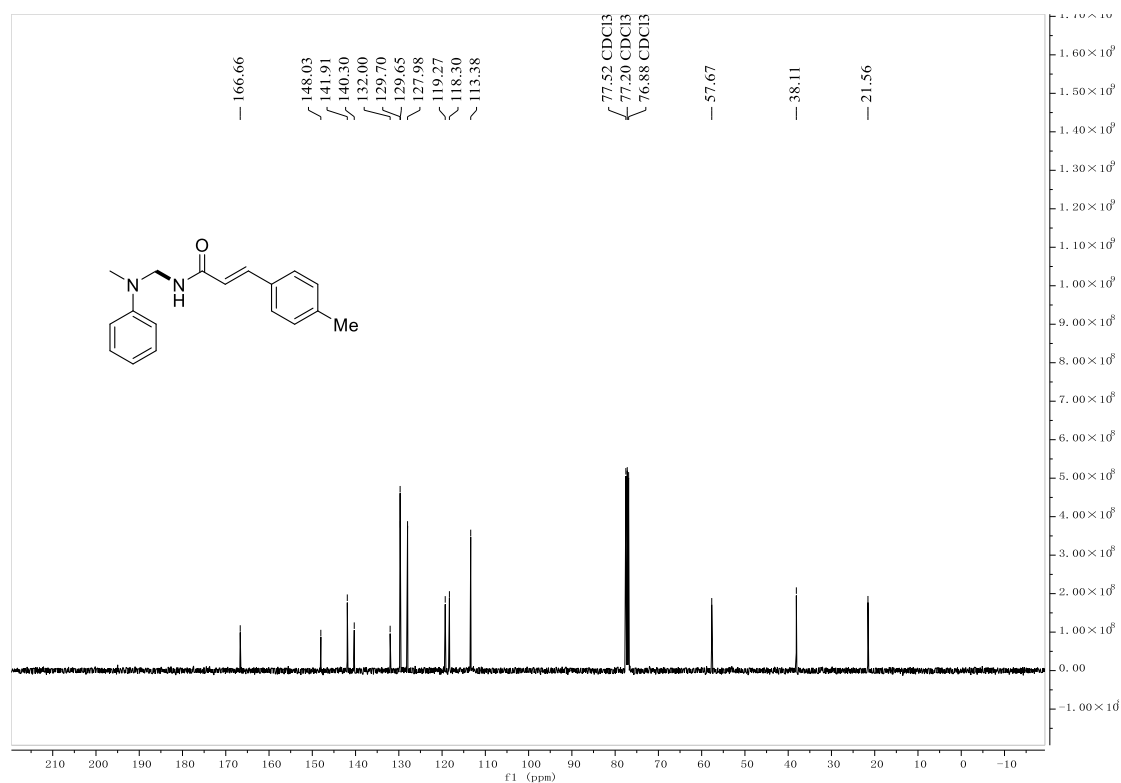
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|------|--------|------|----------|------------------------------------------------------------------|
| 344.1376 | 344.1375   | 0.1 | 0.3 | 11.5 | 1825.9 | n/a  | n/a      | C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O <sub>2</sub> Na |

# <sup>1</sup>H NMR of 19



# <sup>13</sup>C NMR of 19



## HRMS of 19

Monoisotopic Mass, Even Electron Ions

1011 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

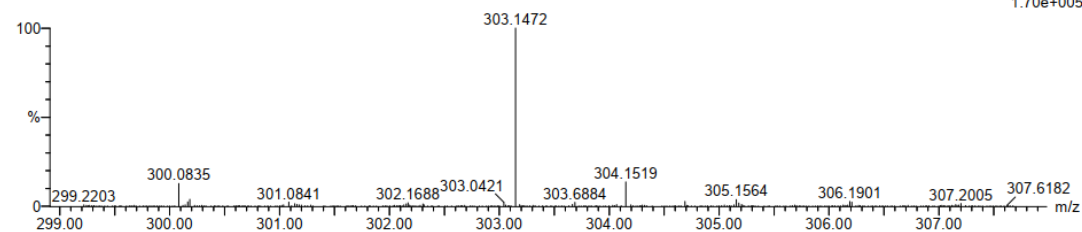
Elements Used:

C: 18-18 H: 20-20 N: 0-100 O: 0-100 Na: 0-7

7

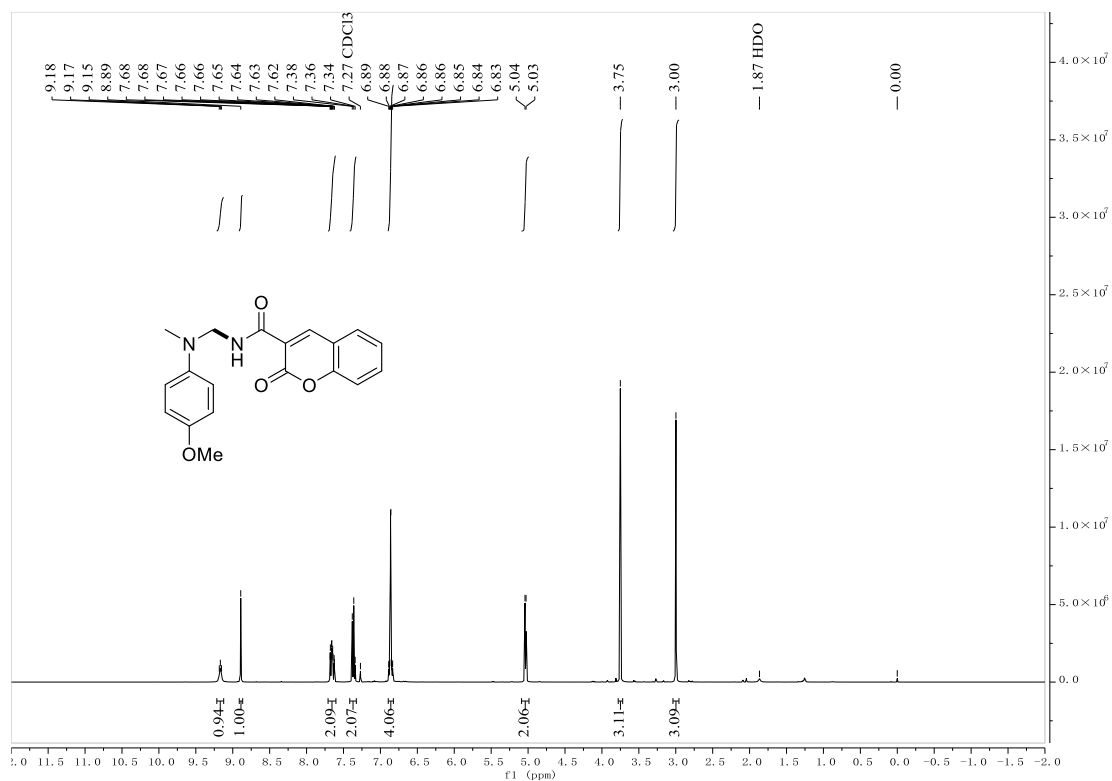
250430-16-45 5 (0.068)

1: TOF MS ES+  
1.70e+005

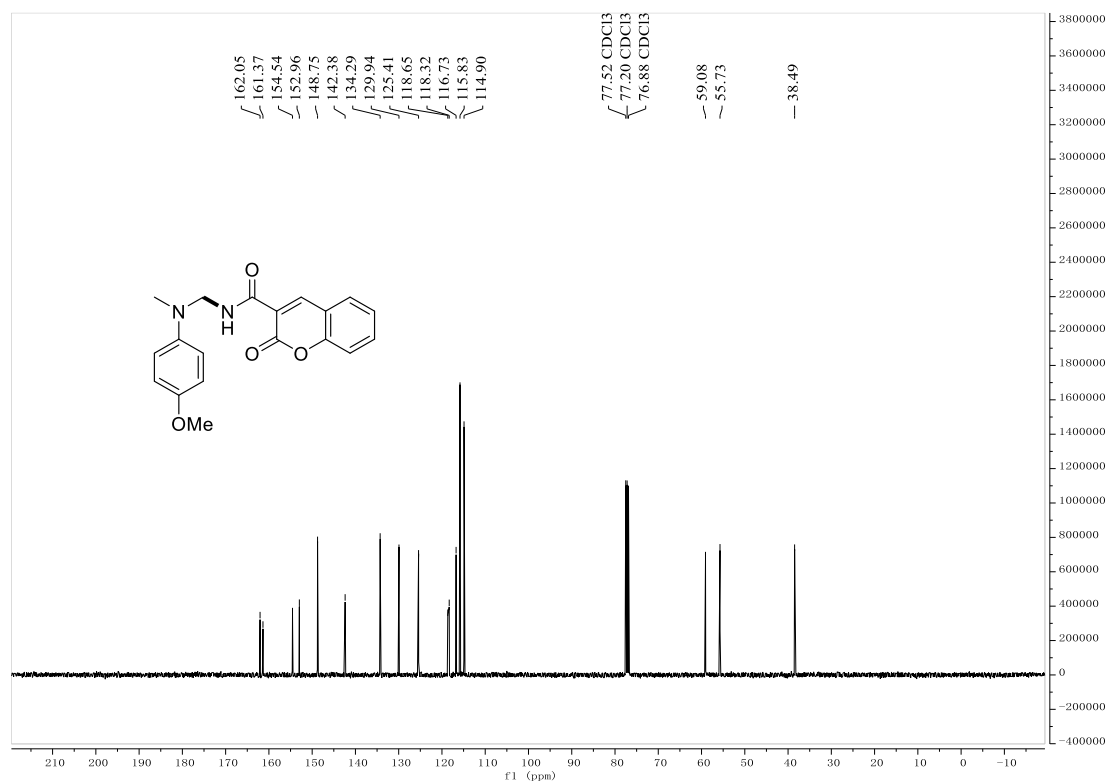


|          |            |      |      |      |        |      |          |                 |  |  |
|----------|------------|------|------|------|--------|------|----------|-----------------|--|--|
| Minimum: |            |      |      |      |        |      |          |                 |  |  |
| Maximum: | 5.0        | 10.0 | -1.5 | 50.0 |        |      |          |                 |  |  |
| Mass     | Calc. Mass | mDa  | PPM  | DBE  | i-FIT  | Norm | Conf (%) | Formula         |  |  |
| 303.1472 | 303.1473   | -0.1 | -0.3 | 9.5  | 1882.3 | n/a  | n/a      | C18 H20 N2 O Na |  |  |

## <sup>1</sup>H NMR of 20



### <sup>13</sup>C NMR of 20



### HRMS of 20

Monoisotopic Mass, Even Electron Ions

774 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

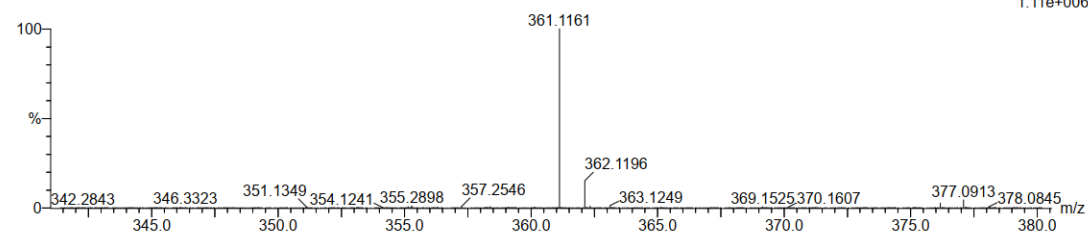
Elements Used:

C: 19-19 H: 8-18 N: 0-100 O: 0-100 Na: 0-2

7

250430-16-41 5 (0.068)

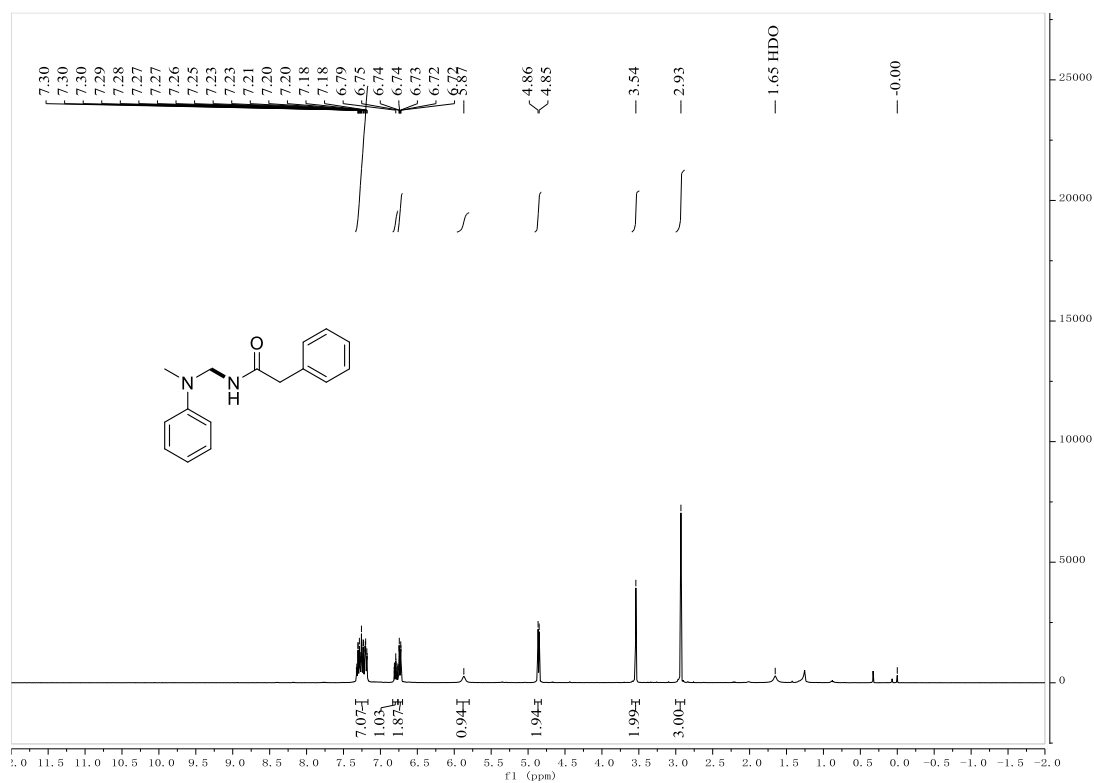
1: TOF MS ES+  
1.11e+006



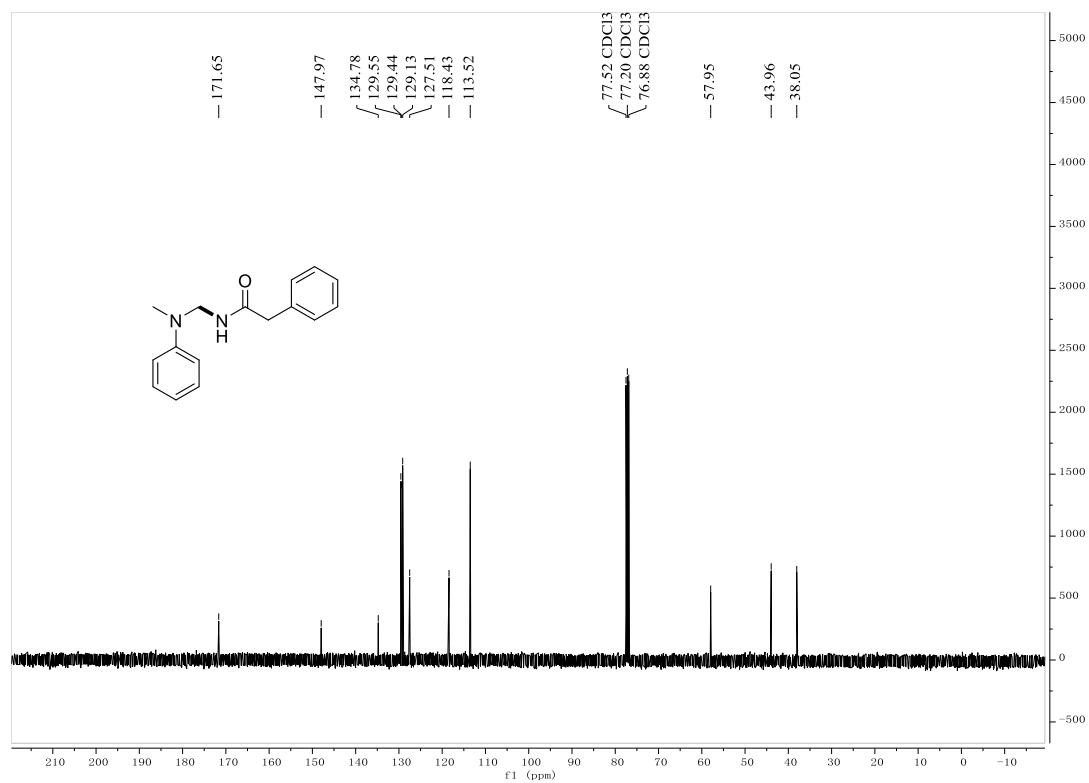
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE  | i-FIT | Norm | Conf (%) | Formula                                                          |
|----------|------------|------|------|------|-------|------|----------|------------------------------------------------------------------|
| 361.1161 | 361.1164   | -0.3 | -0.8 | 11.5 | 851.1 | n/a  | n/a      | C <sub>19</sub> H <sub>18</sub> N <sub>2</sub> O <sub>4</sub> Na |

<sup>1</sup>H NMR of **21**



<sup>13</sup>C NMR of **21**



## HRMS of 21

Monoisotopic Mass, Even Electron Ions

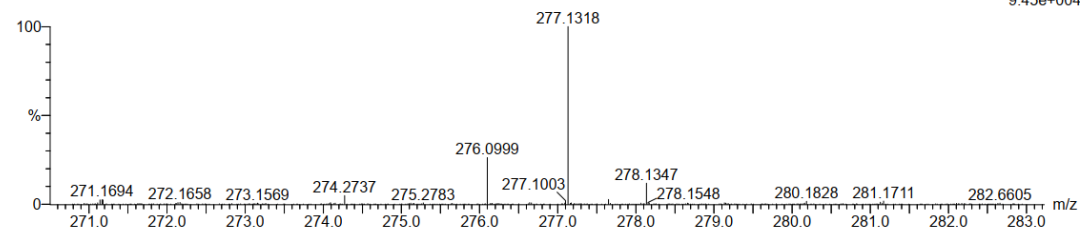
1449 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 16-16 H: 18-18 N: 0-100 O: 0-100 Na: 0-4 K: 0-2

<sup>31</sup>  
250707.4-2.14 (0.098)

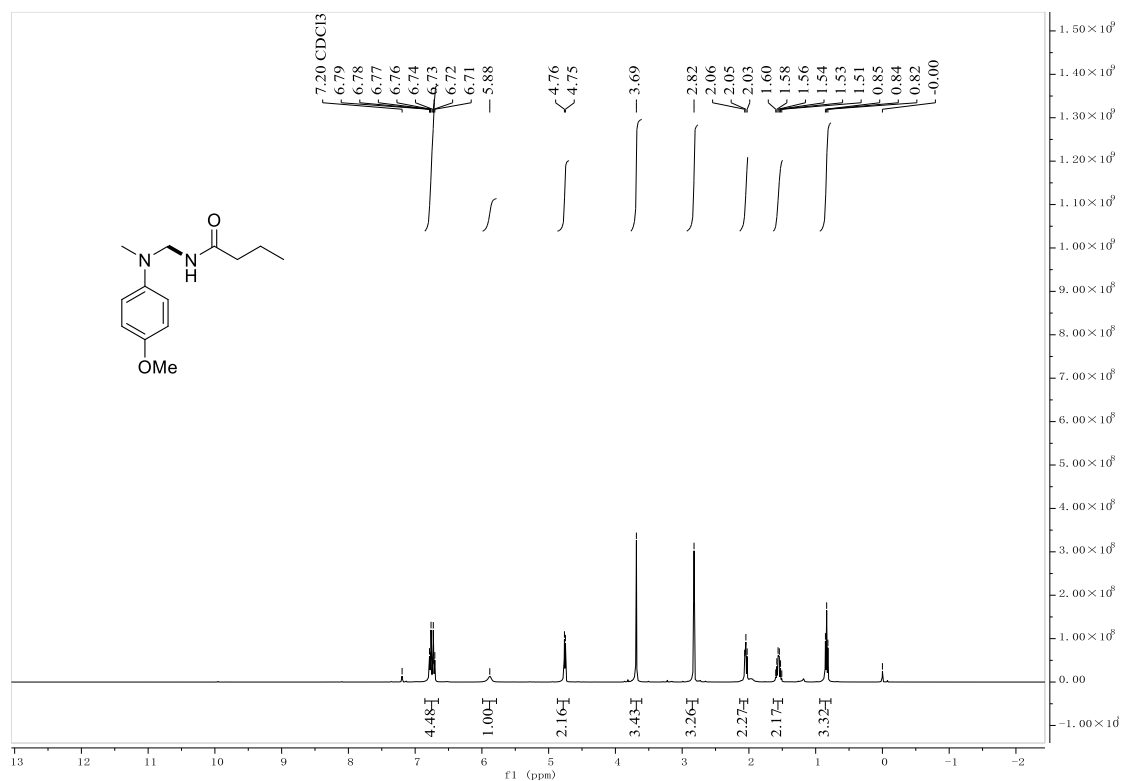
1: TOF MS ES+  
9.45e+004



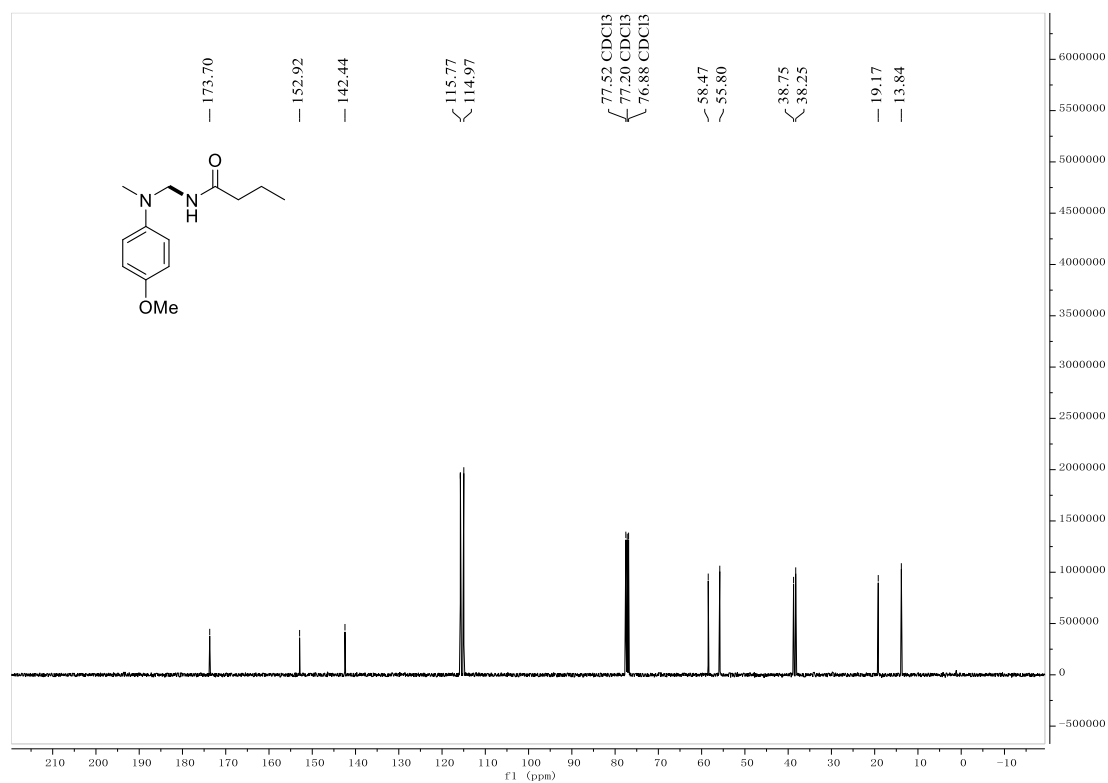
Minimum: -1.5  
Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT | Norm | Conf (%) | Formula         |
|----------|------------|-----|-----|-----|-------|------|----------|-----------------|
| 277.1318 | 277.1317   | 0.1 | 0.4 | 8.5 | 680.9 | n/a  | n/a      | C16 H18 N2 O Na |

## <sup>1</sup>H NMR of 22



## <sup>13</sup>C NMR of 22



## HRMS of 22

Monoisotopic Mass, Even Electron Ions

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

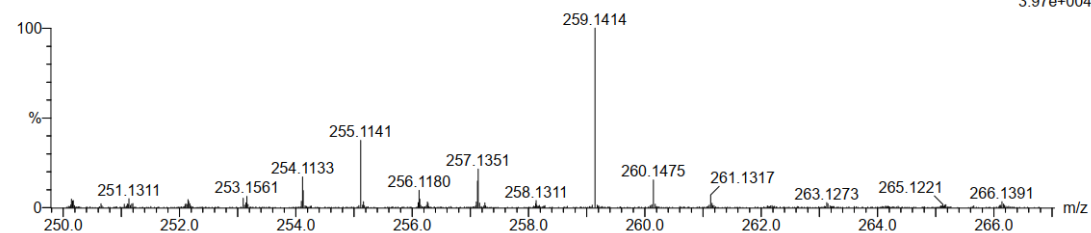
Elements Used:

C: 13-13 H: 20-20 N: 0-100 O: 0-100 Na: 0-7

41

250430-16-5 12 (0.128)

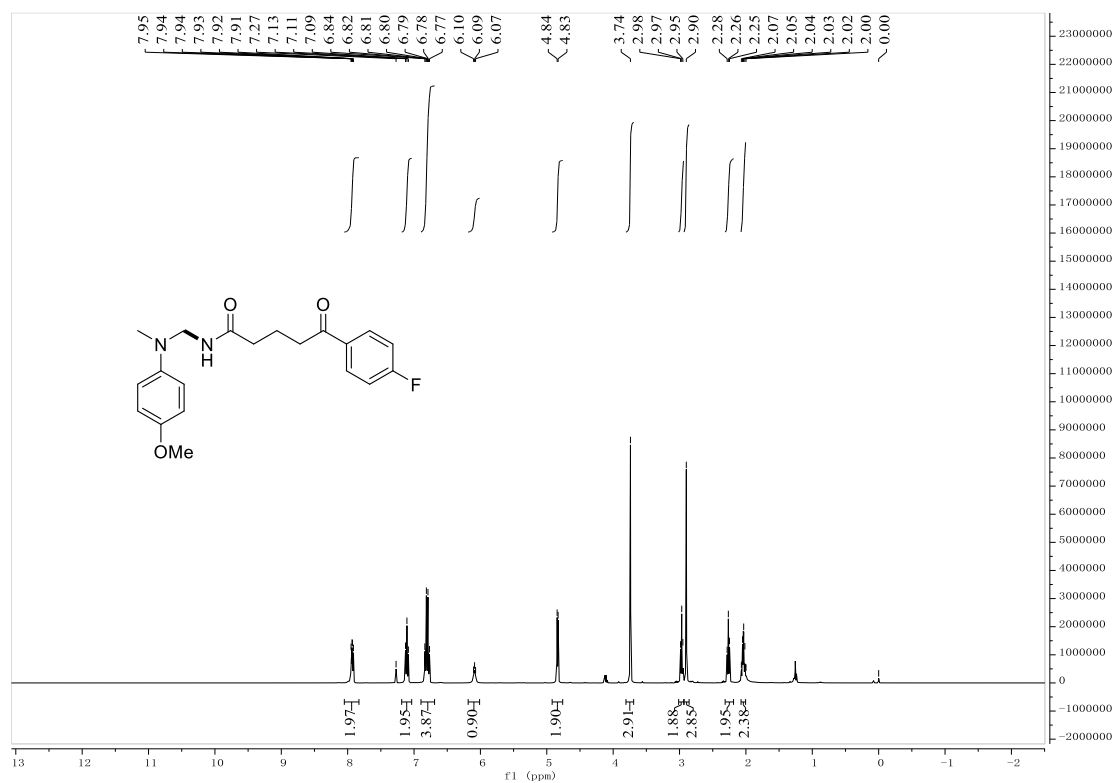
1: TOF MS ES+  
3.97e+004



Minimum: -1.5  
Maximum: 50.0

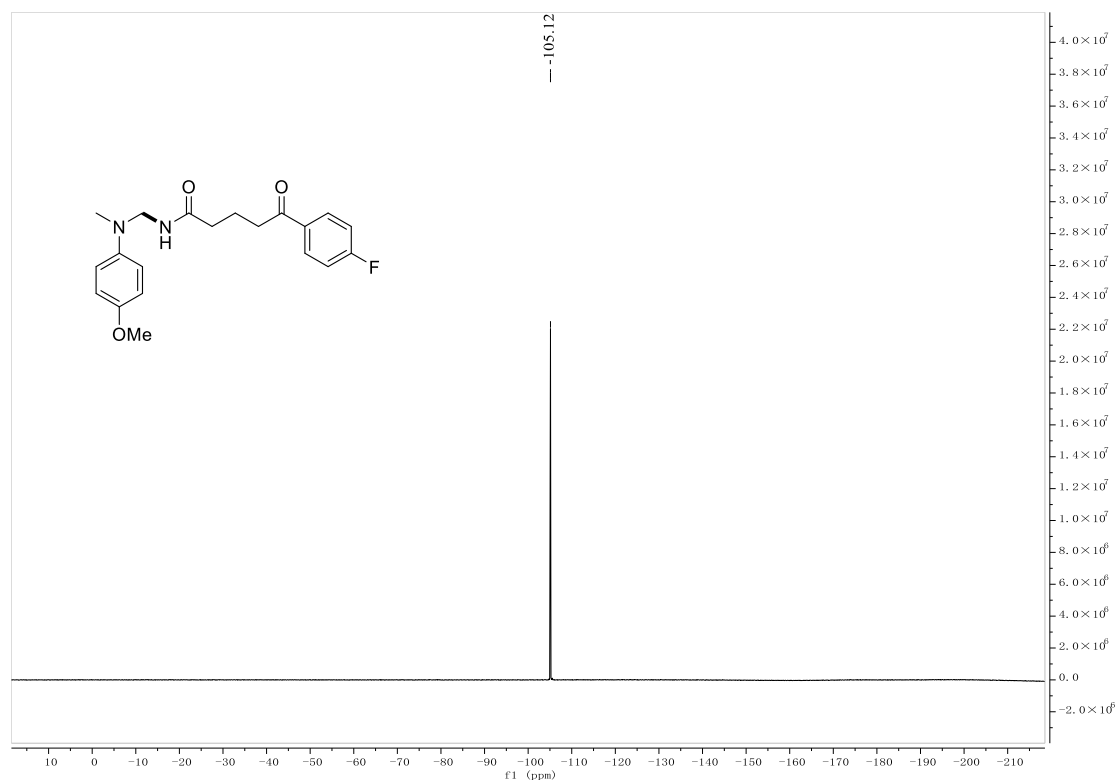
| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|------|------|-----|--------|------|----------|------------------------------------------------------------------|
| 259.1414 | 259.1422   | -0.8 | -3.1 | 4.5 | 1240.6 | n/a  | n/a      | C <sub>13</sub> H <sub>20</sub> N <sub>2</sub> O <sub>2</sub> Na |

### <sup>1</sup>H NMR of 23



### <sup>13</sup>C NMR of 23



<sup>19</sup>F NMR of 23

## HRMS of 23

**Monoisotopic Mass, Even Electron Ions**

1522 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

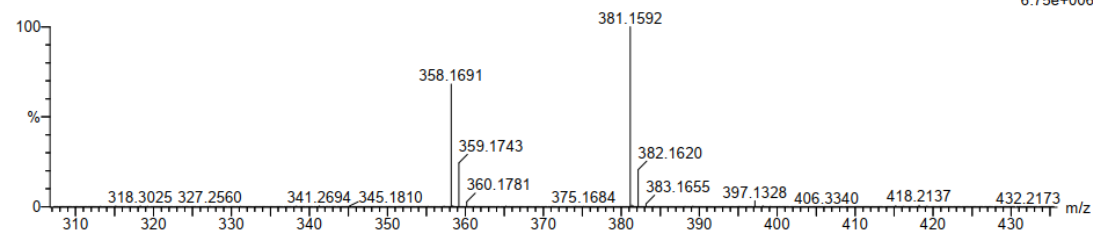
Elements Used:

Elements Used: C: 20-20 H: 23-23 N: 0-100 O: 0-100 Na: 0-7 F: 1-1

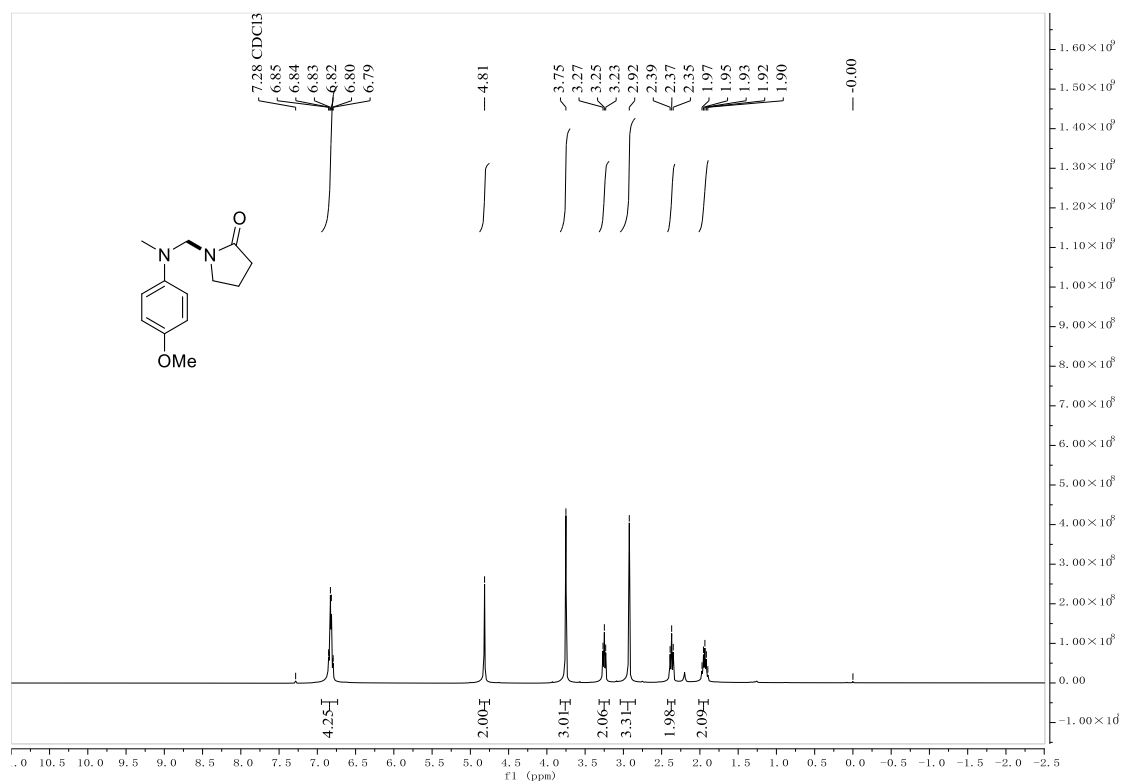
41

250430-16-7 5 (0.068)

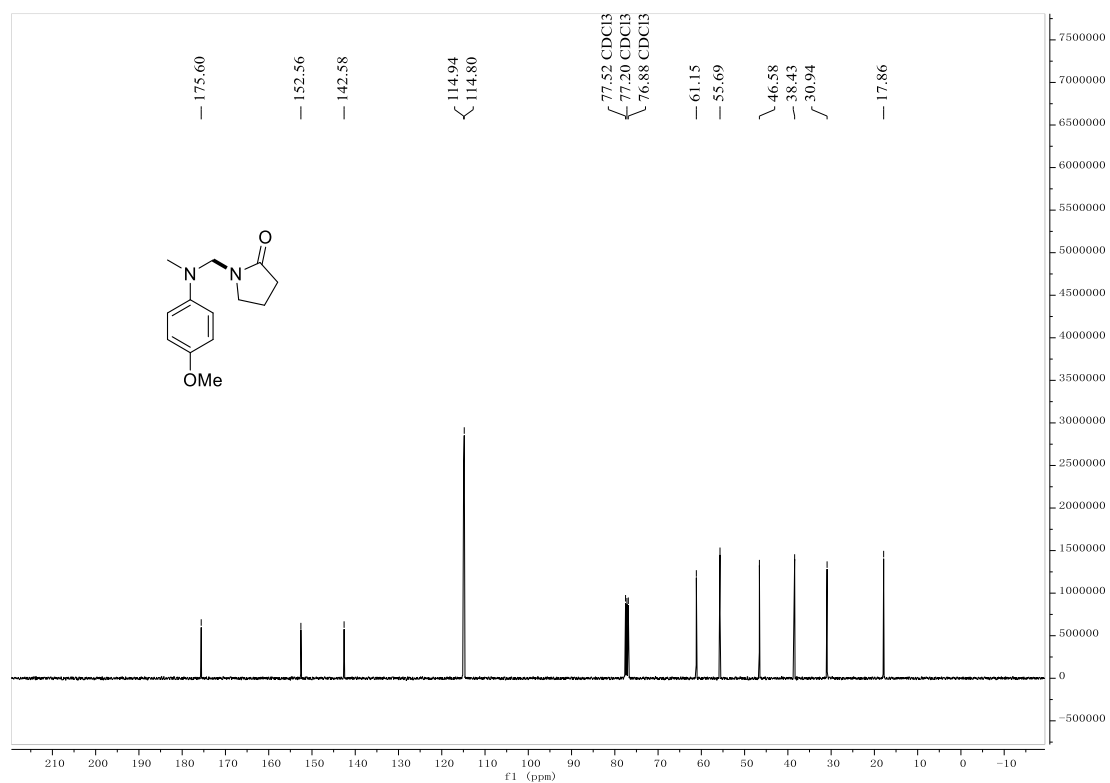
1: TOF MS ES+  
6.75e+006

[illegible]

# <sup>1</sup>H NMR of 24



# <sup>13</sup>C NMR of 24



## HRMS of 24

Monoisotopic Mass, Even Electron Ions

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

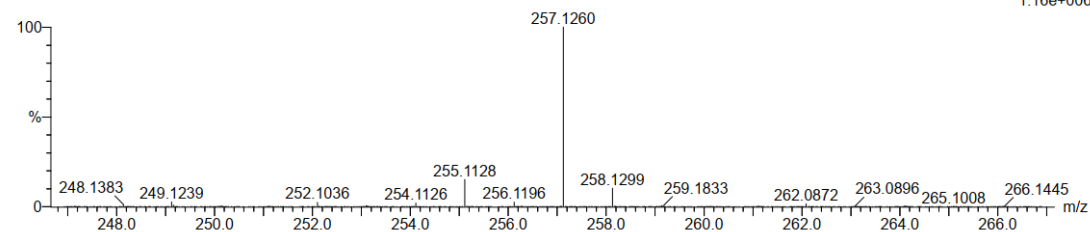
Elements Used:

C: 13-13 H: 18-18 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-15 6 (0.076)

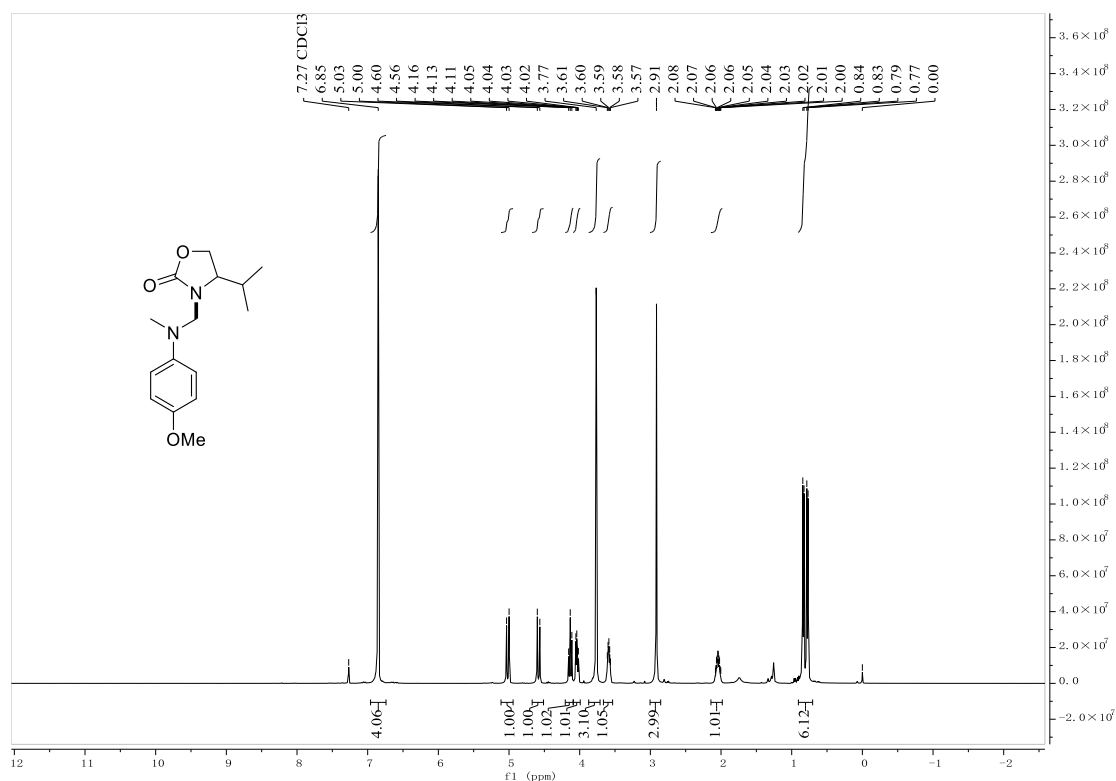
1: TOF MS ES+  
1.16e+006



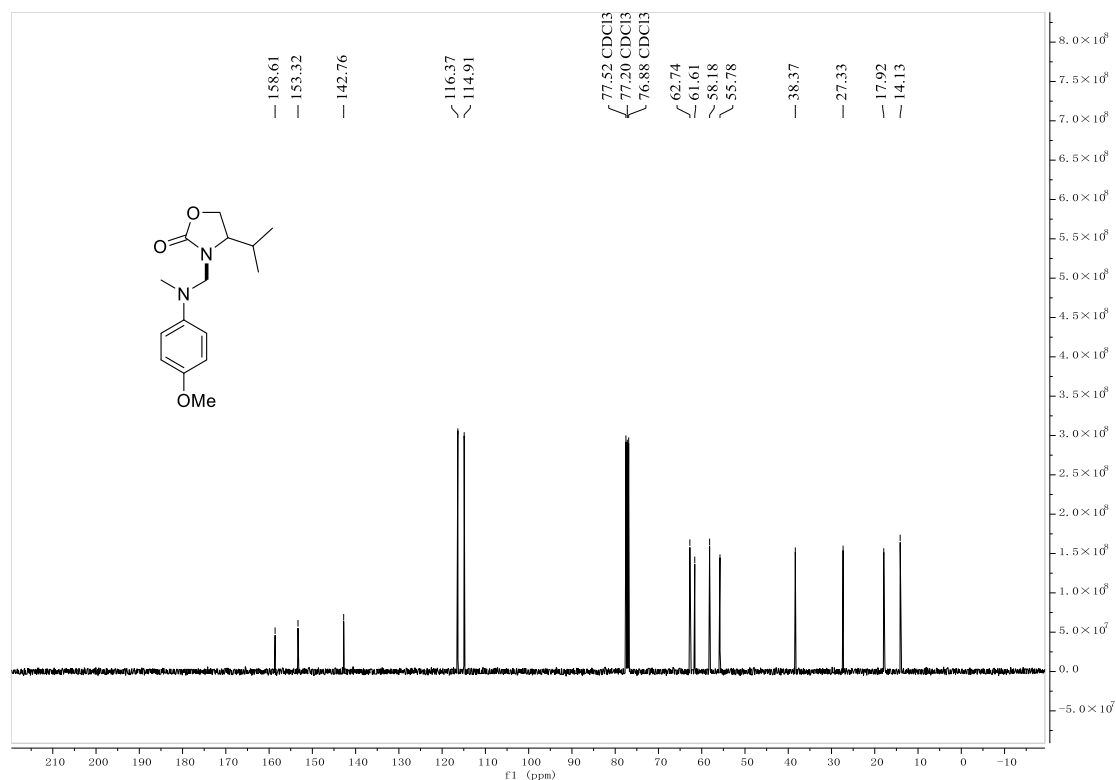
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula          |
|----------|------------|------|------|-----|--------|------|----------|------------------|
| 257.1260 | 257.1266   | -0.6 | -2.3 | 5.5 | 1594.4 | n/a  | n/a      | C13 H18 N2 O2 Na |

## <sup>1</sup>H NMR of 25



## <sup>13</sup>C NMR of 25



## HRMS of 25

Monoisotopic Mass, Even Electron Ions

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

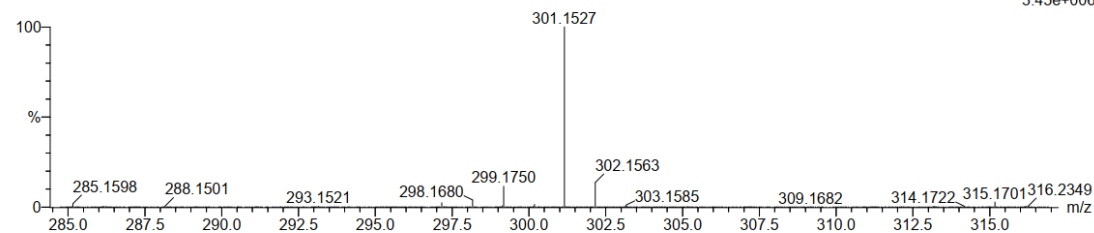
Elements Used:

C: 15-15 H: 22-22 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-13 6 (0.076)

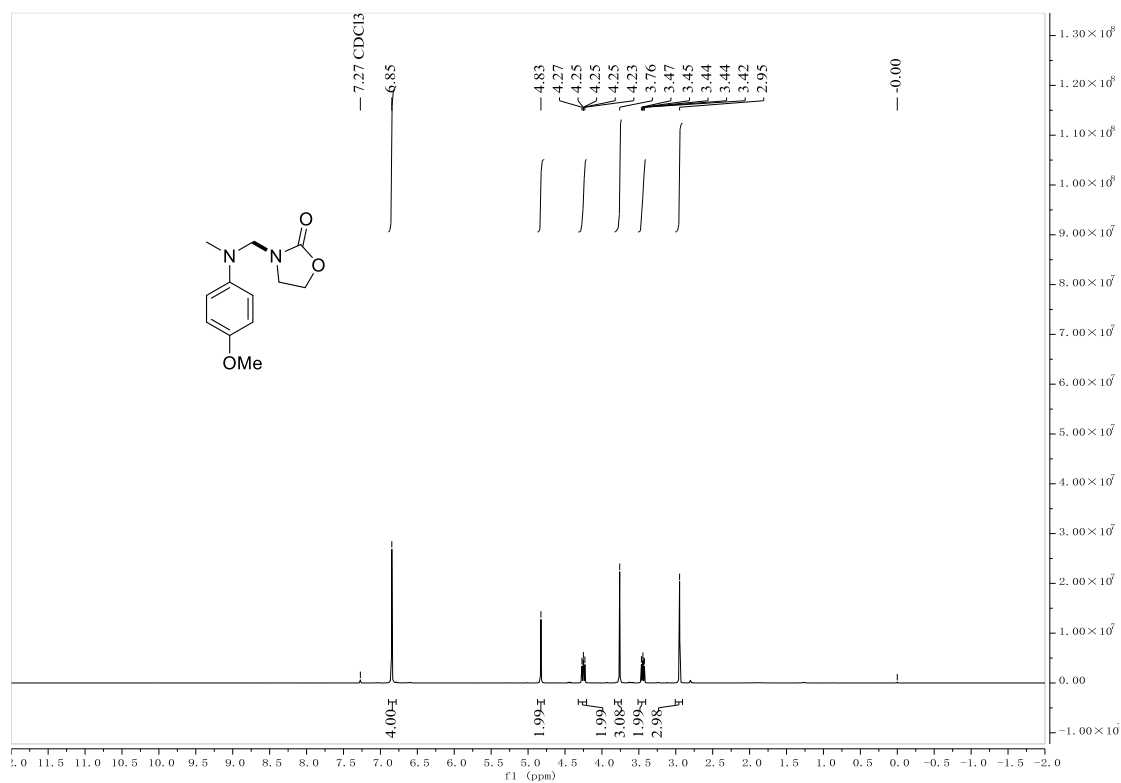
1: TOF MS ES+  
3.45e+006



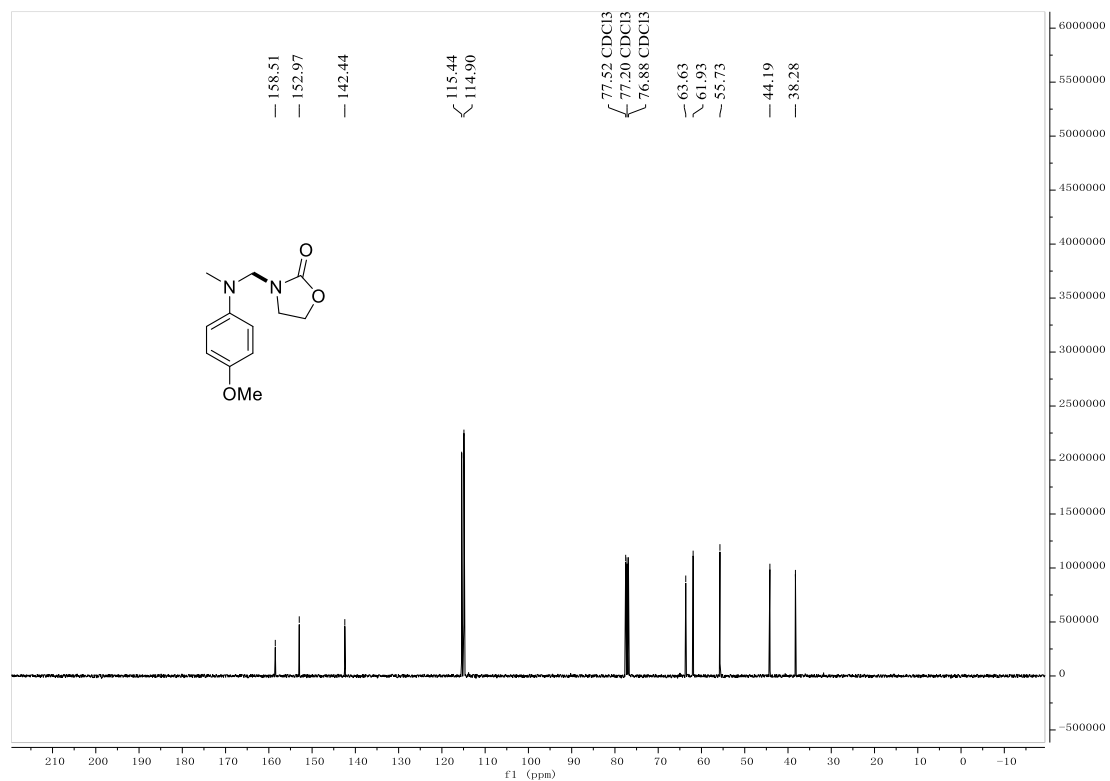
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|------|------|-----|--------|------|----------|------------------------------------------------------------------|
| 301.1527 | 301.1528   | -0.1 | -0.3 | 5.5 | 1951.9 | n/a  | n/a      | C <sub>15</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> Na |

# <sup>1</sup>H NMR of 26



# <sup>13</sup>C NMR of 26



## HRMS of 26

Monoisotopic Mass, Even Electron Ions

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

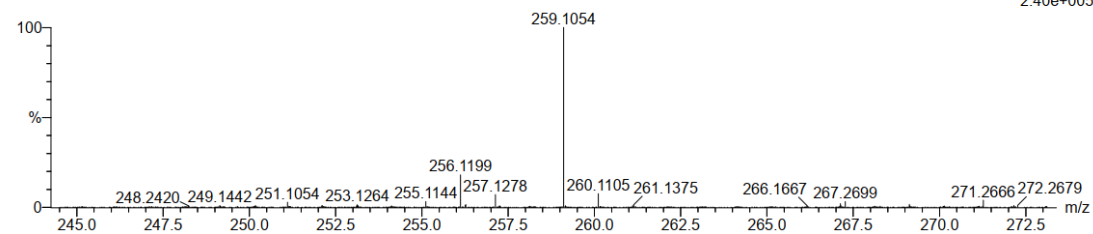
Elements Used:

C: 12-12 H: 16-16 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-39 10 (0.111)

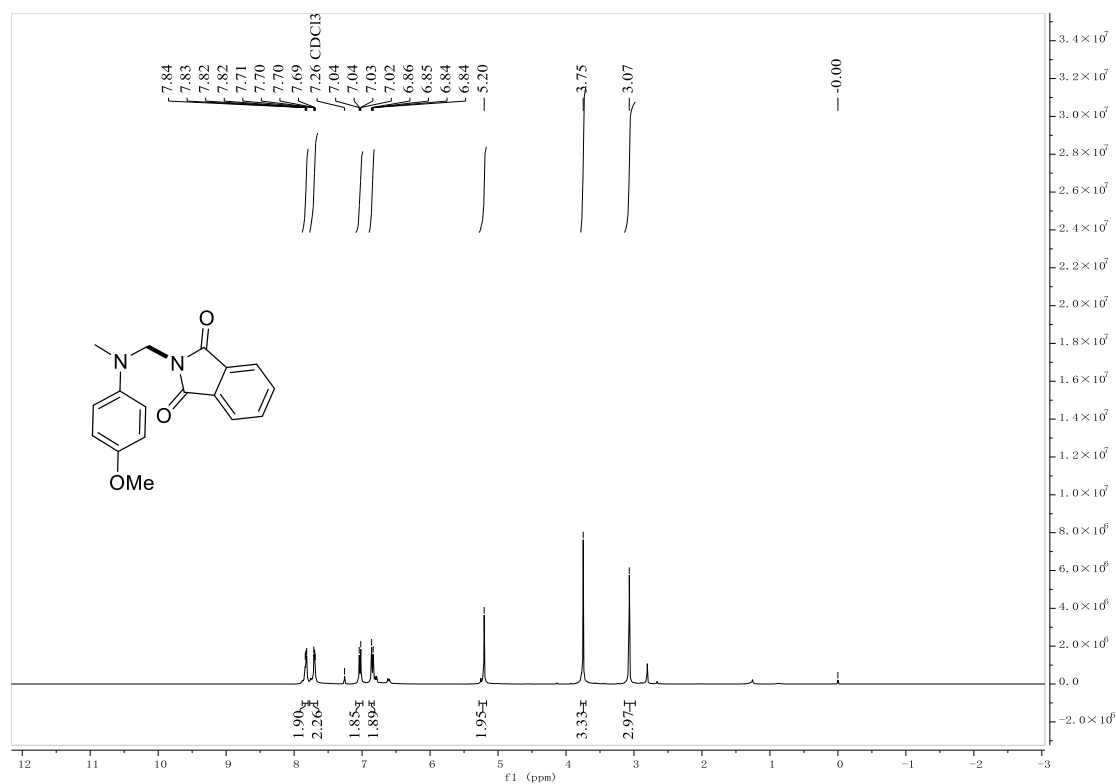
1: TOF MS ES+  
2.40e+005



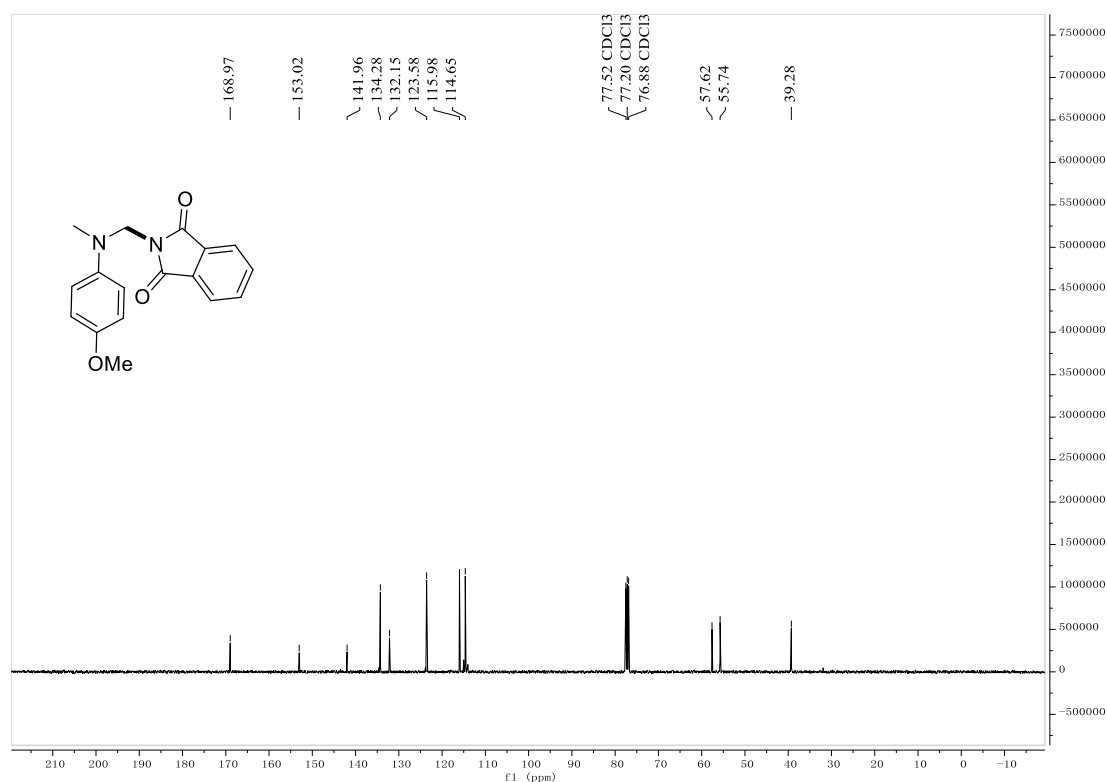
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula          |
|----------|------------|------|------|-----|--------|------|----------|------------------|
| 259.1054 | 259.1059   | -0.5 | -1.9 | 5.5 | 1237.1 | n/a  | n/a      | C12 H16 N2 O3 Na |

## <sup>1</sup>H NMR of 27



# <sup>13</sup>C NMR of 27



## HRMS of 27

Monoisotopic Mass, Even Electron Ions

195 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

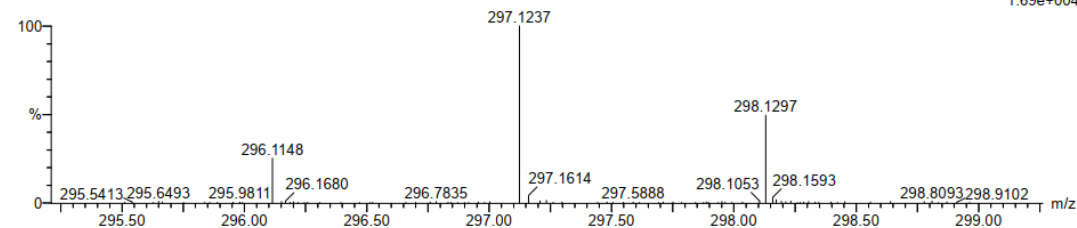
Elements Used:

C: 17-17 H: 17-17 N: 0-100 O: 0-100

7

250707-4-8 17 (0.114)

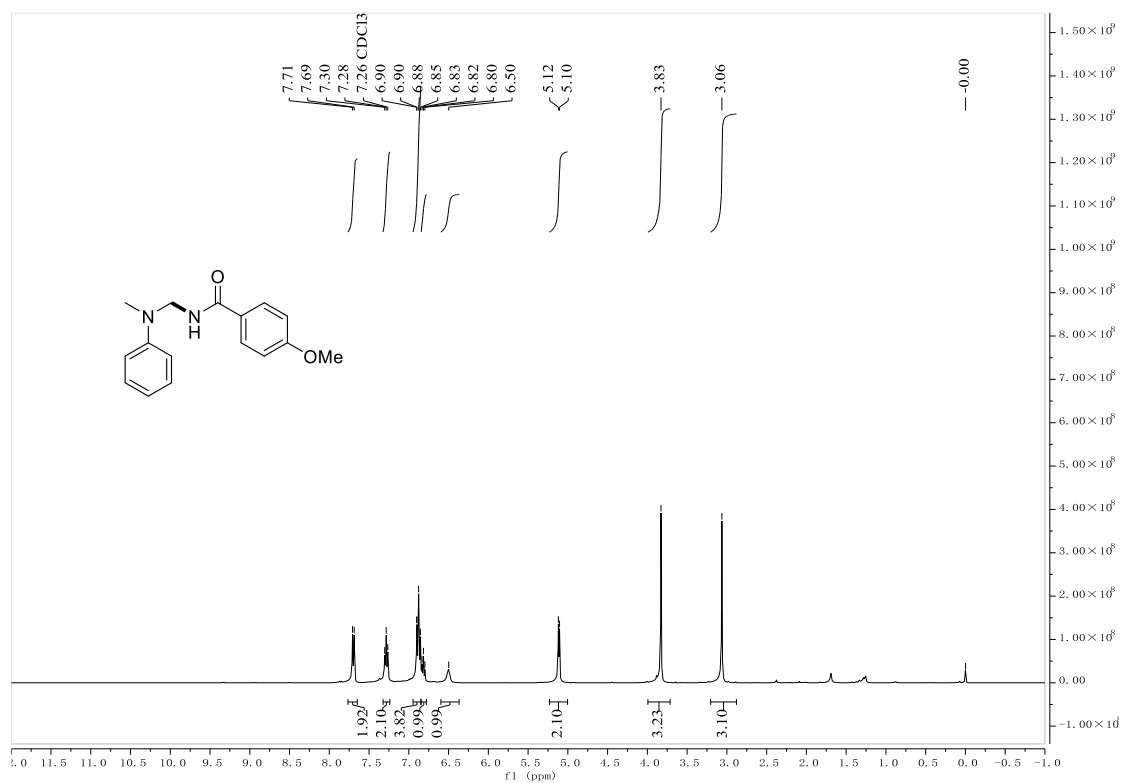
1: TOF MS ES+  
1.69e+004



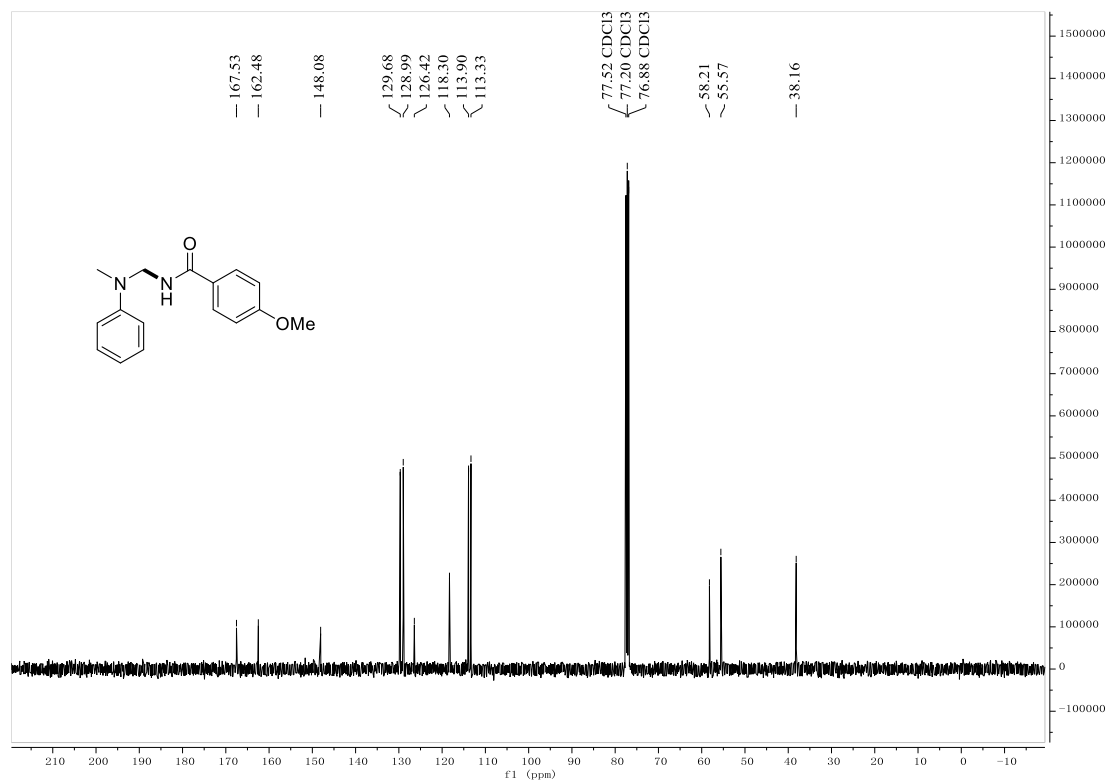
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PTM  | DBE  | i-FIT | Norm | Conf (%) | Formula                                                       |
|----------|------------|------|------|------|-------|------|----------|---------------------------------------------------------------|
| 297.1237 | 297.1239   | -0.2 | -0.7 | 10.5 | 413.9 | n/a  | n/a      | C <sub>17</sub> H <sub>17</sub> N <sub>2</sub> O <sub>3</sub> |

<sup>1</sup>H NMR of **28**



<sup>13</sup>C NMR of **28**



## HRMS of 28

Monoisotopic Mass, Even Electron Ions

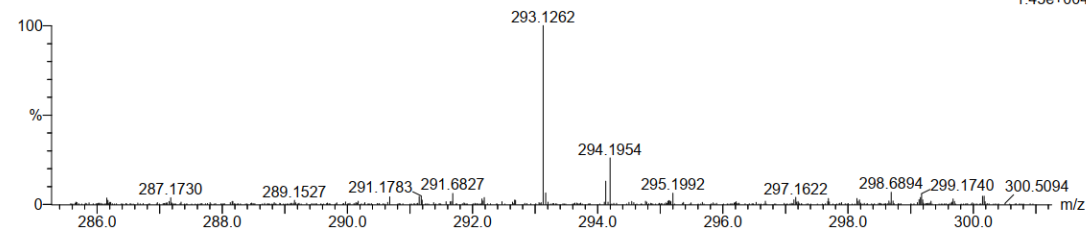
1648 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 16-16 H: 18-18 N: 0-100 O: 0-100 Na: 0-4 K: 0-2

31  
250707-4-3 15 (0.104)

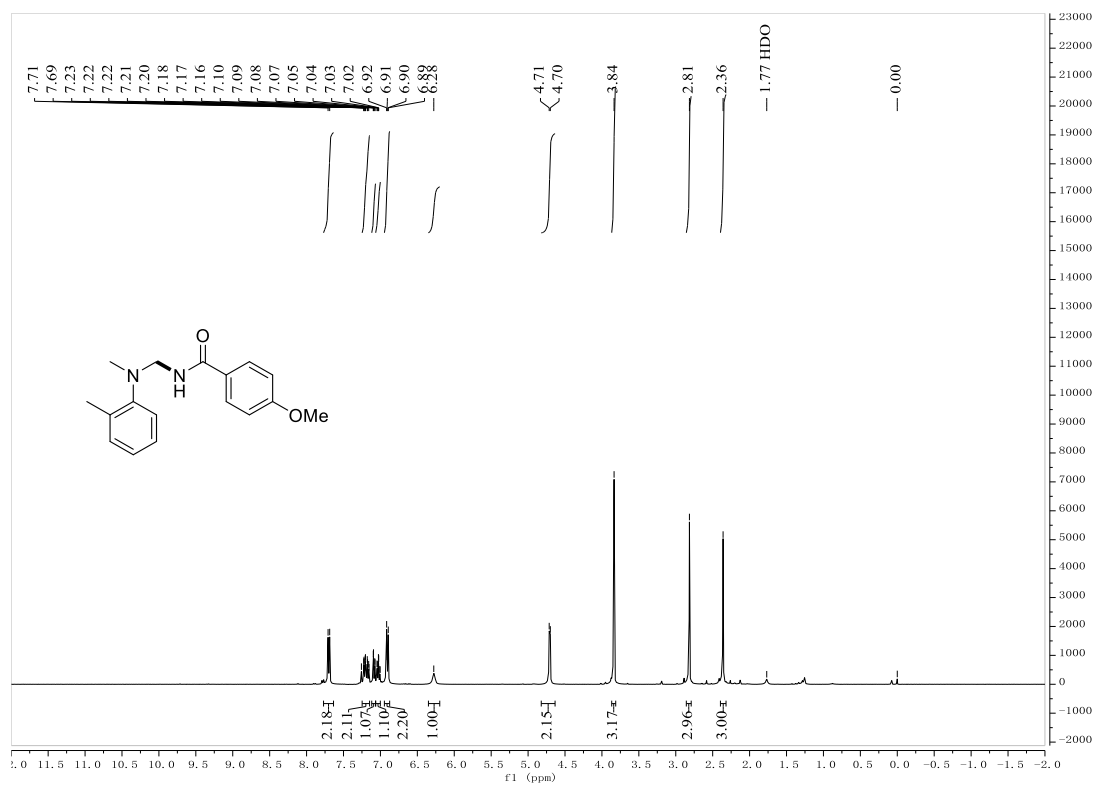
1: TOF MS ES+  
1.45e+004



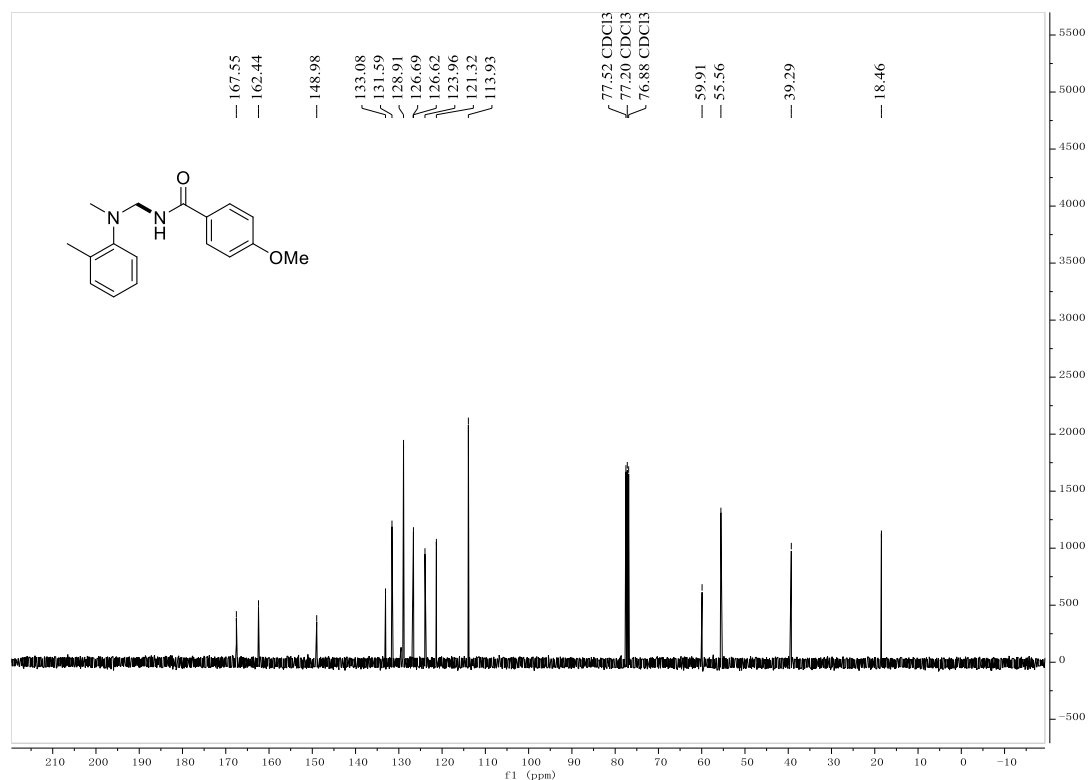
Minimum: -1.5  
Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT | Norm | Conf (%) | Formula          |
|----------|------------|------|------|-----|-------|------|----------|------------------|
| 293.1262 | 293.1266   | -0.4 | -1.4 | 8.5 | 517.5 | n/a  | n/a      | C16 H18 N2 O2 Na |

## <sup>1</sup>H NMR of 29



## <sup>13</sup>C NMR of 29



## HRMS of 29

Monoisotopic Mass, Even Electron Ions

1855 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

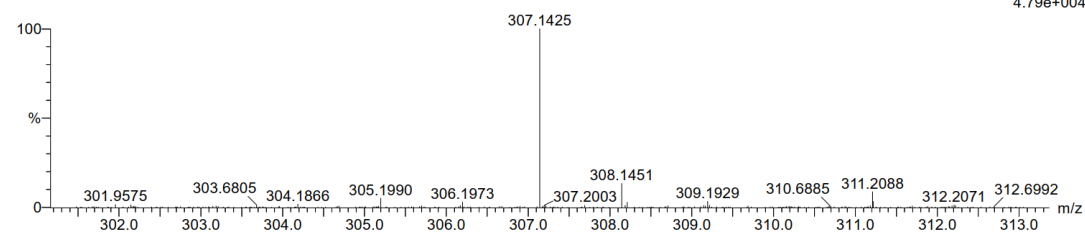
Elements Used:

C: 17-17 H: 20-20 N: 0-100 O: 0-100 Na: 0-4 K: 0-2

7

250707-4-6 23 (0.146)

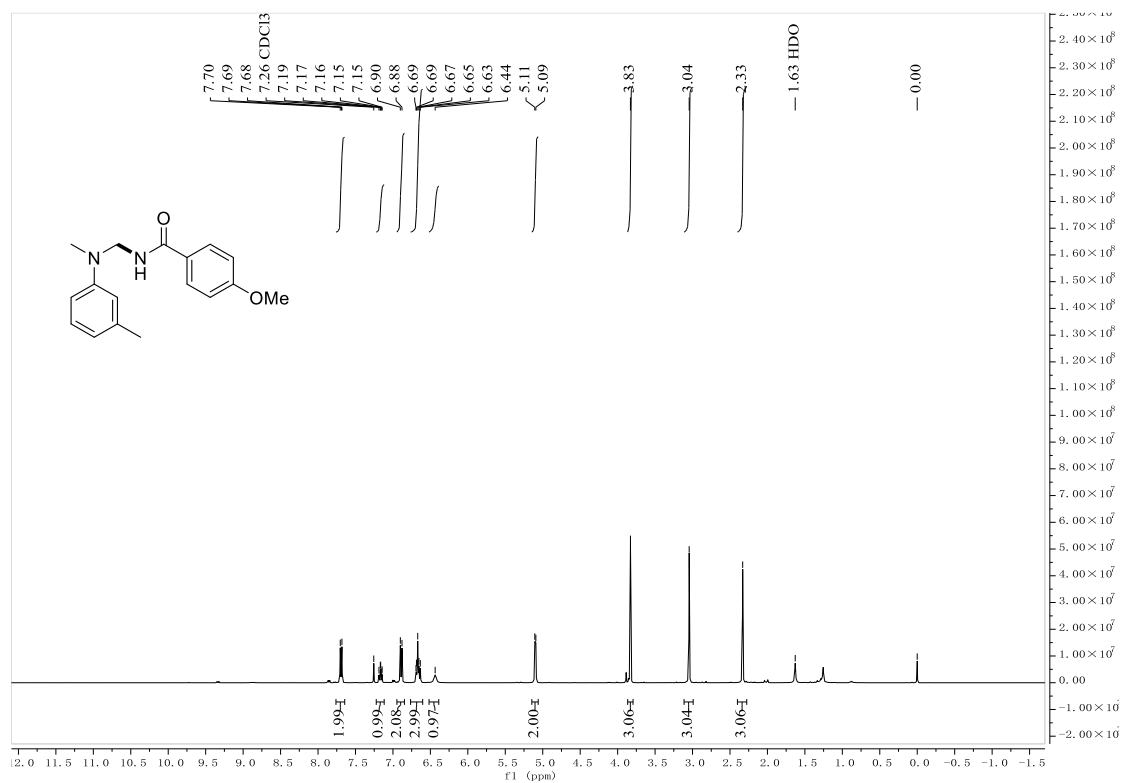
1: TOF MS ES+  
4.79e+004



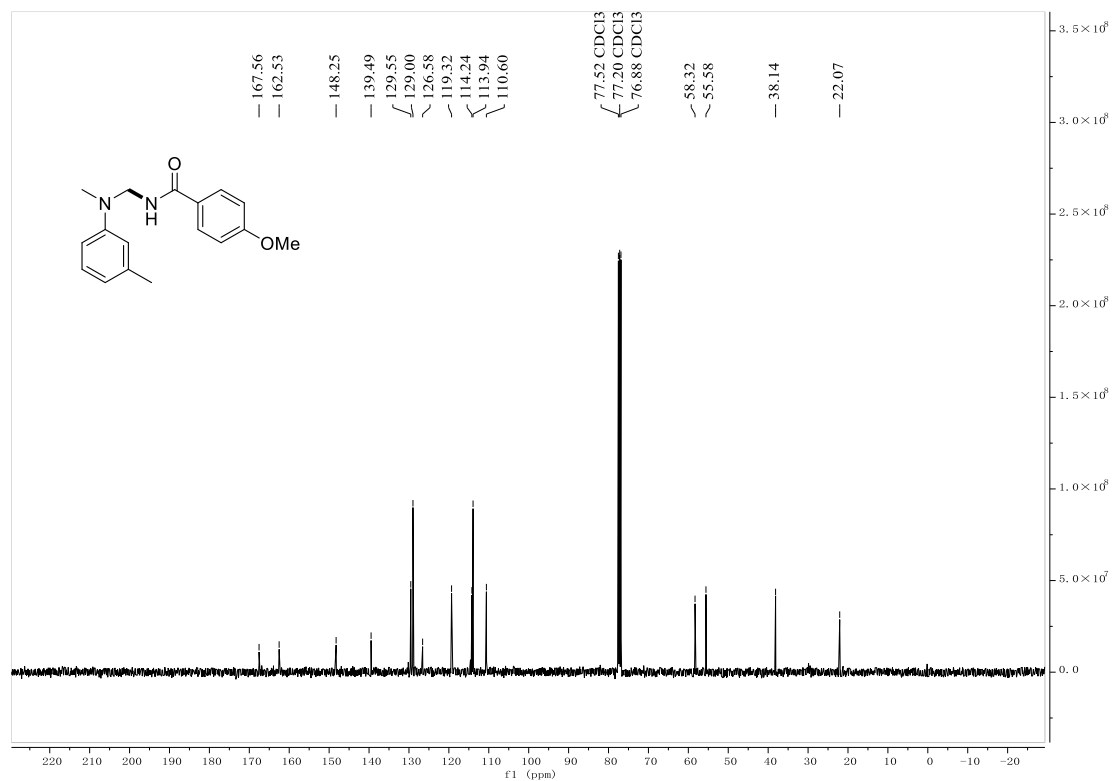
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|-----|-------|------|----------|------------------------------------------------------------------|
| 307.1425 | 307.1422   | 0.3 | 1.0 | 8.5 | 443.9 | n/a  | n/a      | C <sub>17</sub> H <sub>20</sub> N <sub>2</sub> O <sub>2</sub> Na |

# <sup>1</sup>H NMR of 30



# <sup>13</sup>C NMR of 30



## HRMS of 30

Monoisotopic Mass, Even Electron Ions

1855 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

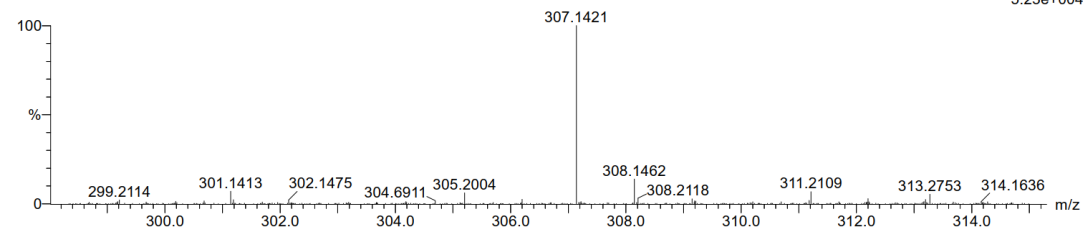
Elements Used:

C: 17-17 H: 20-20 N: 0-100 O: 0-100 Na: 0-4 K: 0-2

7

250707-4-5 23 (0.146)

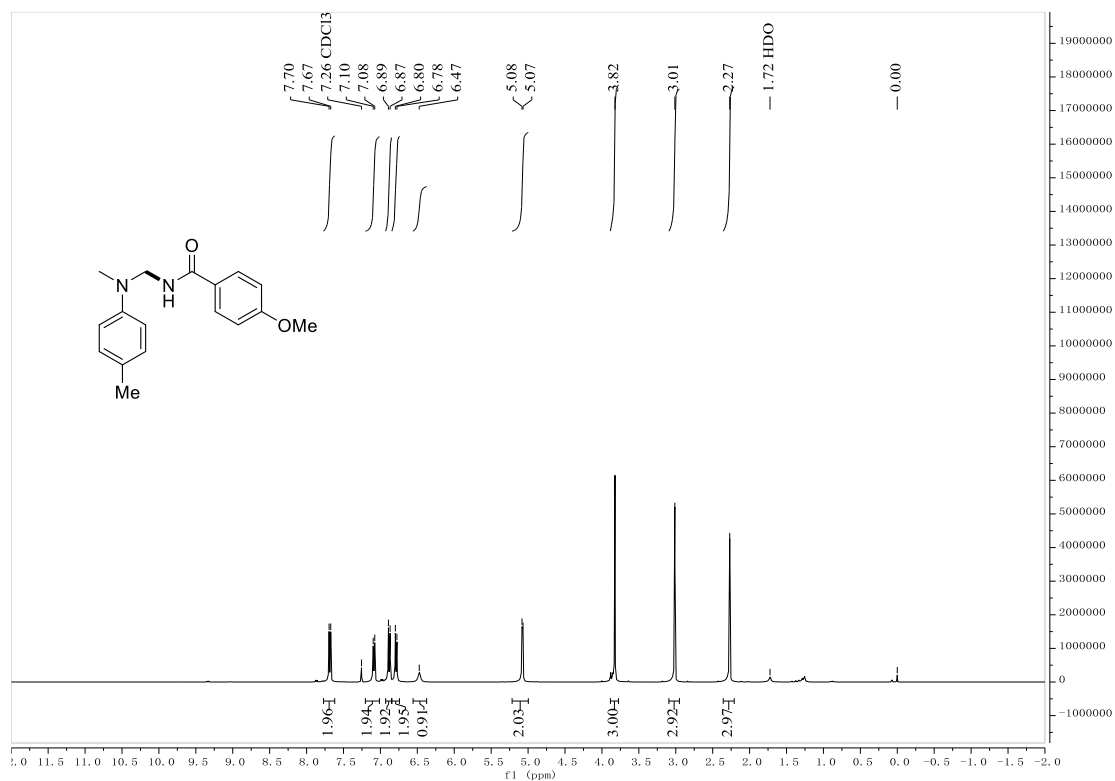
1: TOF MS ES+  
5.23e+004



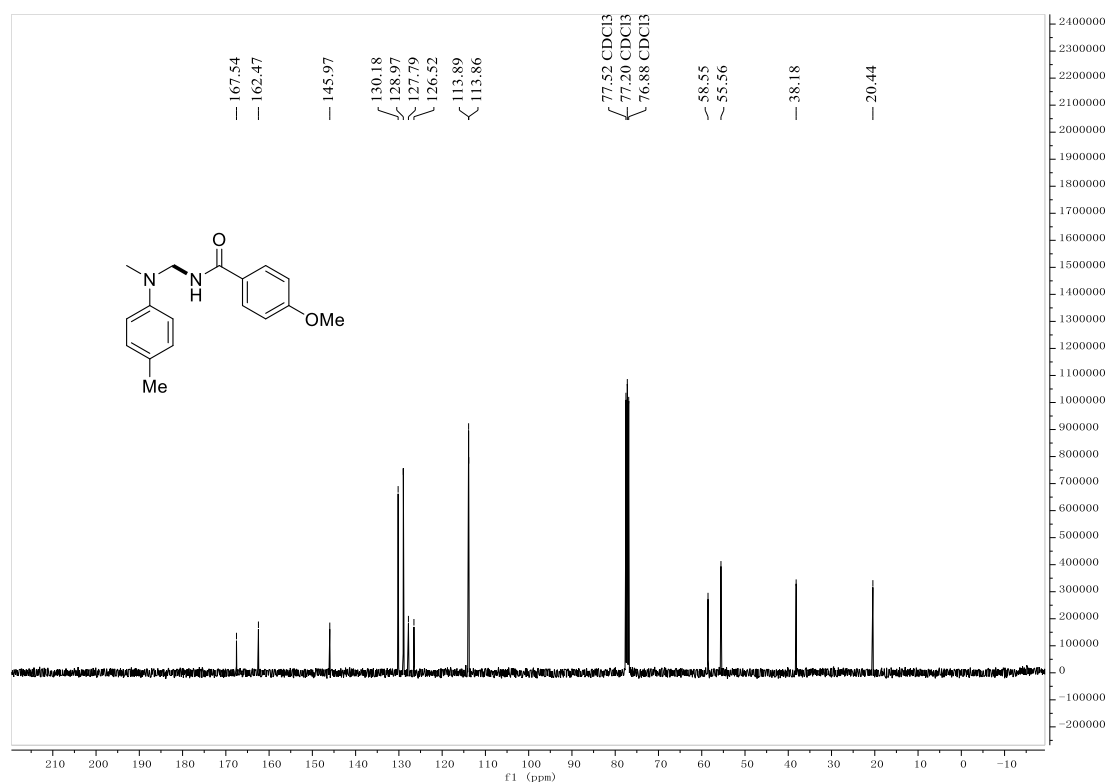
Minimum: -1.5  
Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT | Norm | Conf (%) | Formula          |
|----------|------------|------|------|-----|-------|------|----------|------------------|
| 307.1421 | 307.1422   | -0.1 | -0.3 | 8.5 | 459.5 | n/a  | n/a      | C17 H20 N2 O2 Na |

## <sup>1</sup>H NMR of 31



## <sup>13</sup>C NMR of **31**



## HRMS of **31**

Monoisotopic Mass, Even Electron Ions

1855 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

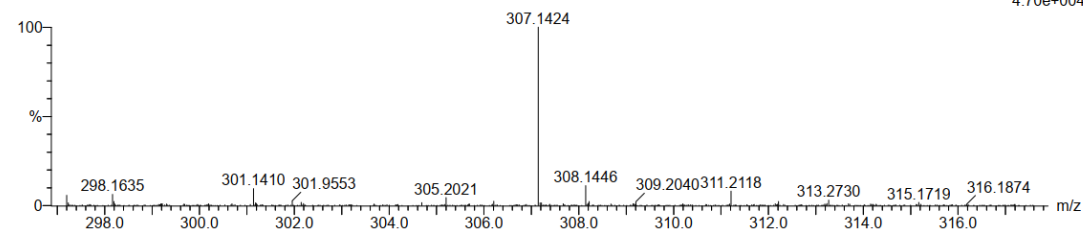
Elements Used:

C: 17-17 H: 20-20 N: 0-100 O: 0-100 Na: 0-4 K: 0-2

7

250707-4-7 23 (0.146)

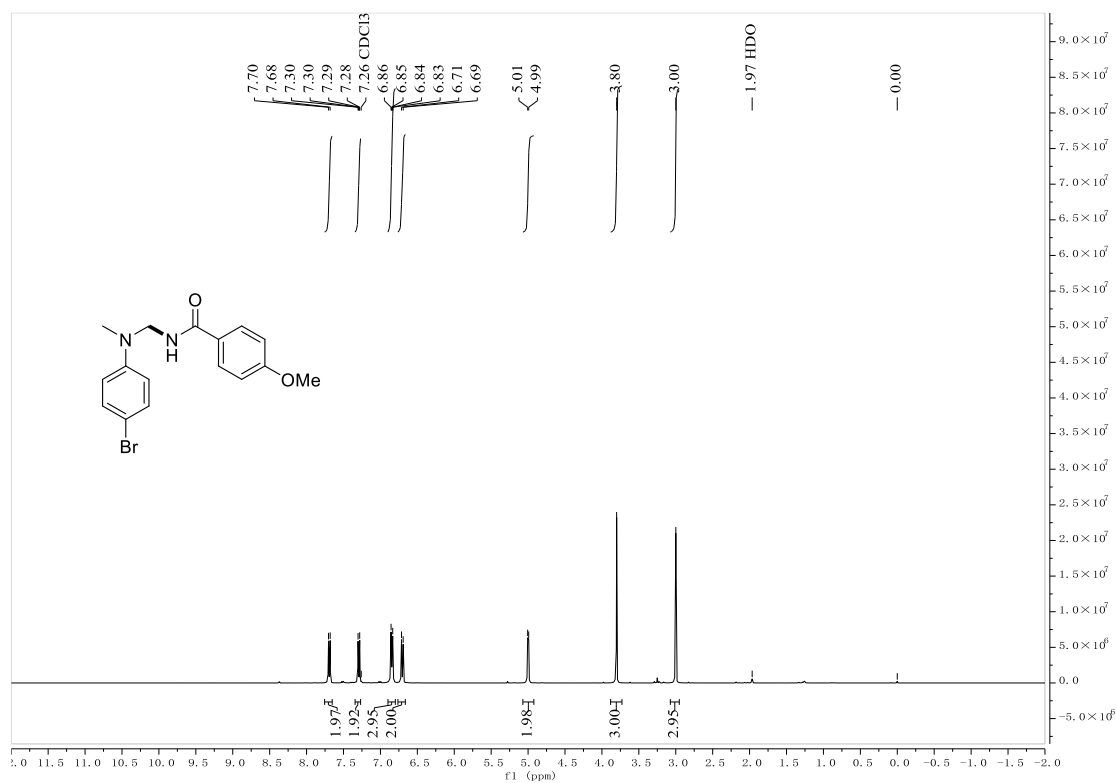
1: TOF MS ES+  
4.70e+004



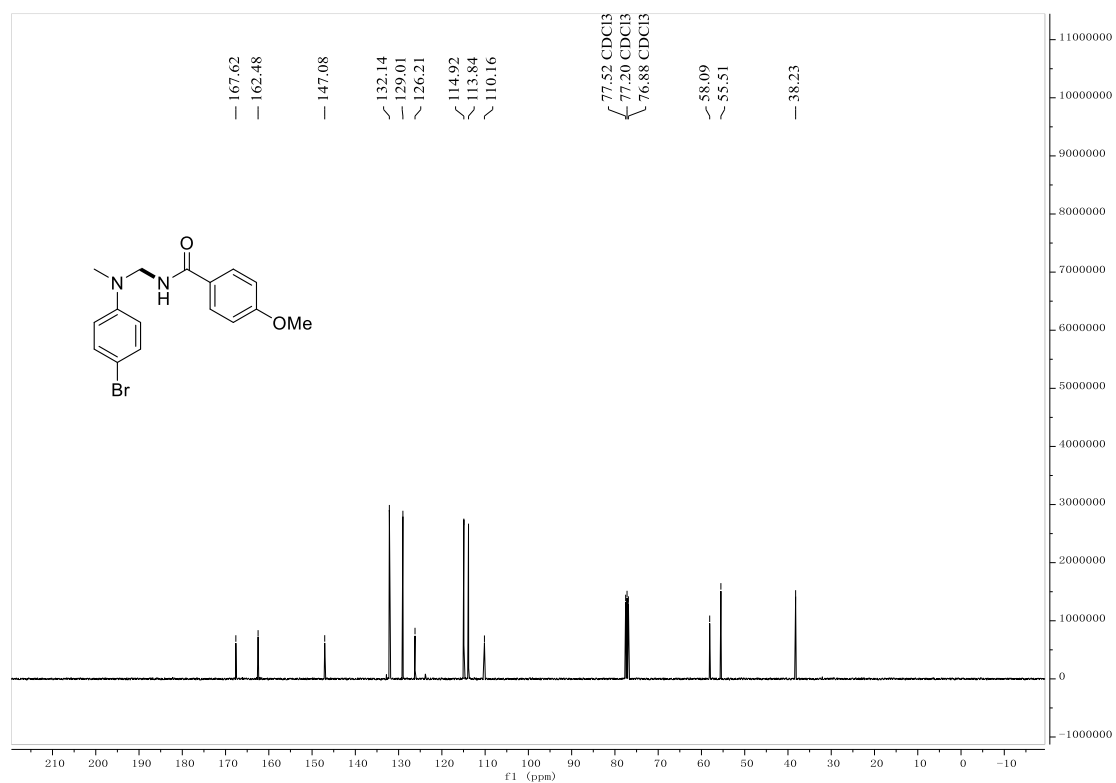
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|-----|-------|------|----------|------------------------------------------------------------------|
| 307.1424 | 307.1422   | 0.2 | 0.7 | 8.5 | 530.7 | n/a  | n/a      | C <sub>17</sub> H <sub>20</sub> N <sub>2</sub> O <sub>2</sub> Na |

# <sup>1</sup>H NMR of 32



# <sup>13</sup>C NMR of 32



## HRMS of 32

Monoisotopic Mass, Even Electron Ions

1094 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

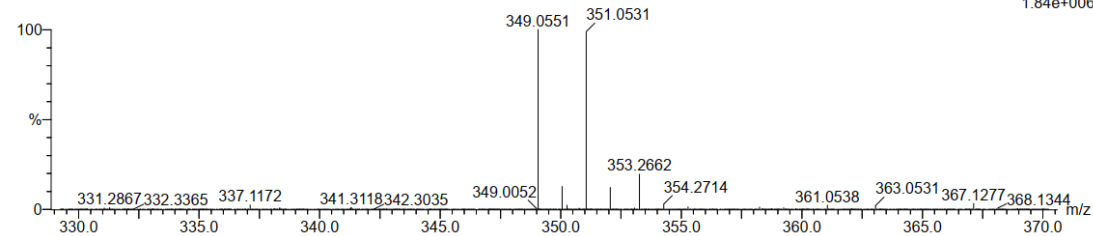
Elements Used:

C: 16-16 H: 18-18 N: 0-100 O: 0-100 Na: 0-7 Br: 1-2

7

250430-16-21 9 (0.102)

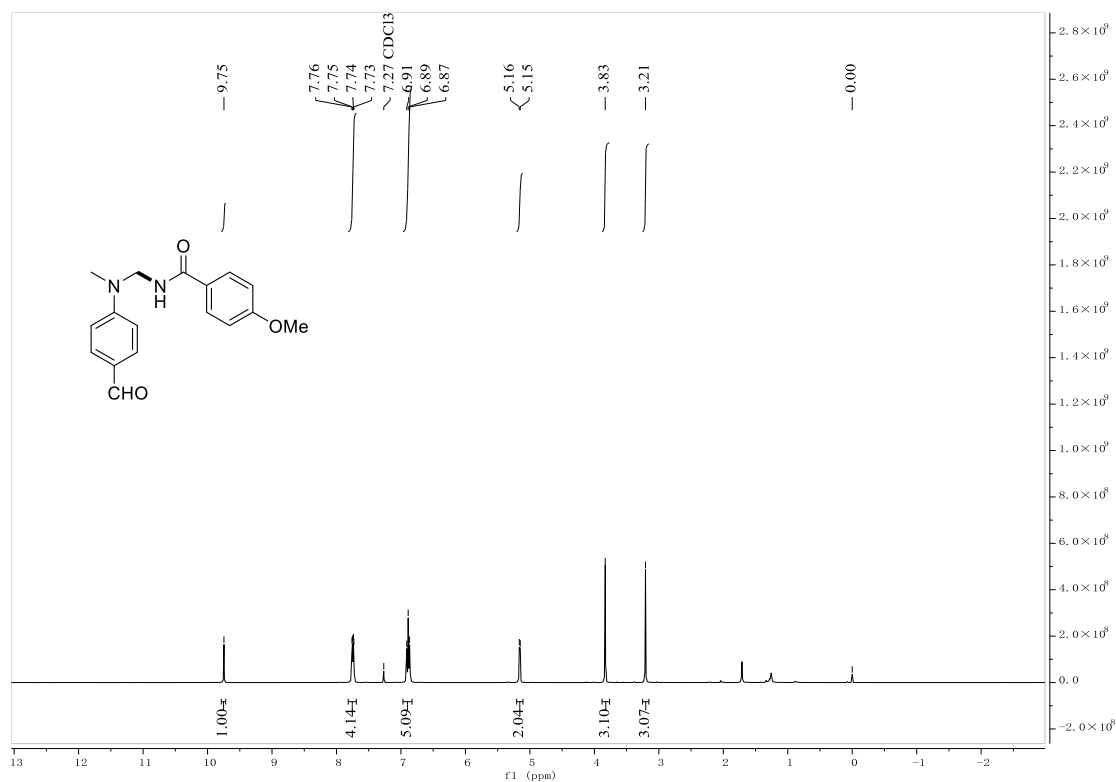
1: TOF MS ES+  
1.84e+006



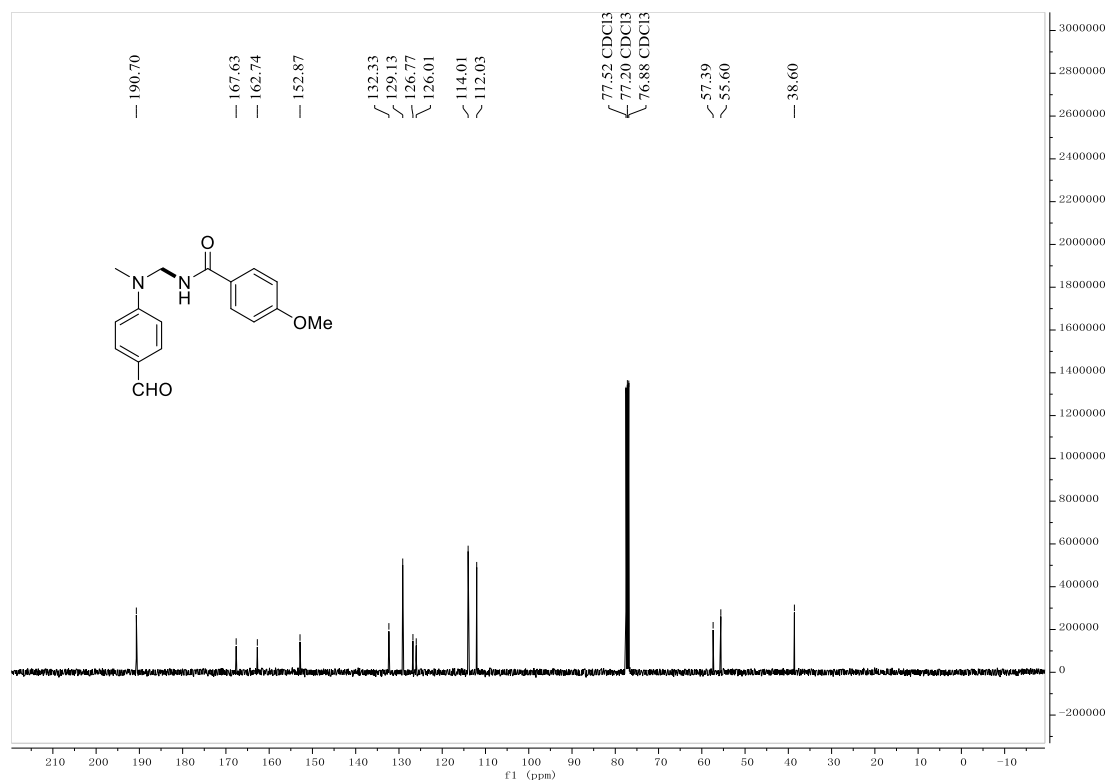
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula          |
|----------|------------|------|------|-----|--------|------|----------|------------------|
| 349.0551 | 349.0552   | -0.1 | -0.3 | 8.5 | 2112.0 | n/a  | n/a      | C16 H18 N2 O2 Br |

## <sup>1</sup>H NMR of 33



## <sup>13</sup>C NMR of **33**



## HRMS of **33**

Monoisotopic Mass, Even Electron Ions

1133 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

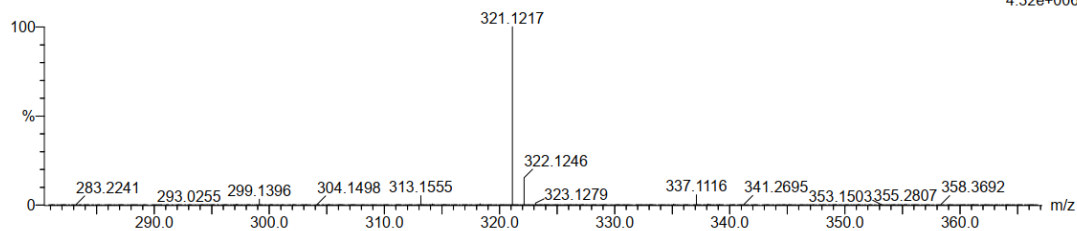
Elements Used:

C: 17-17 H: 18-18 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-12 6 (0.077)

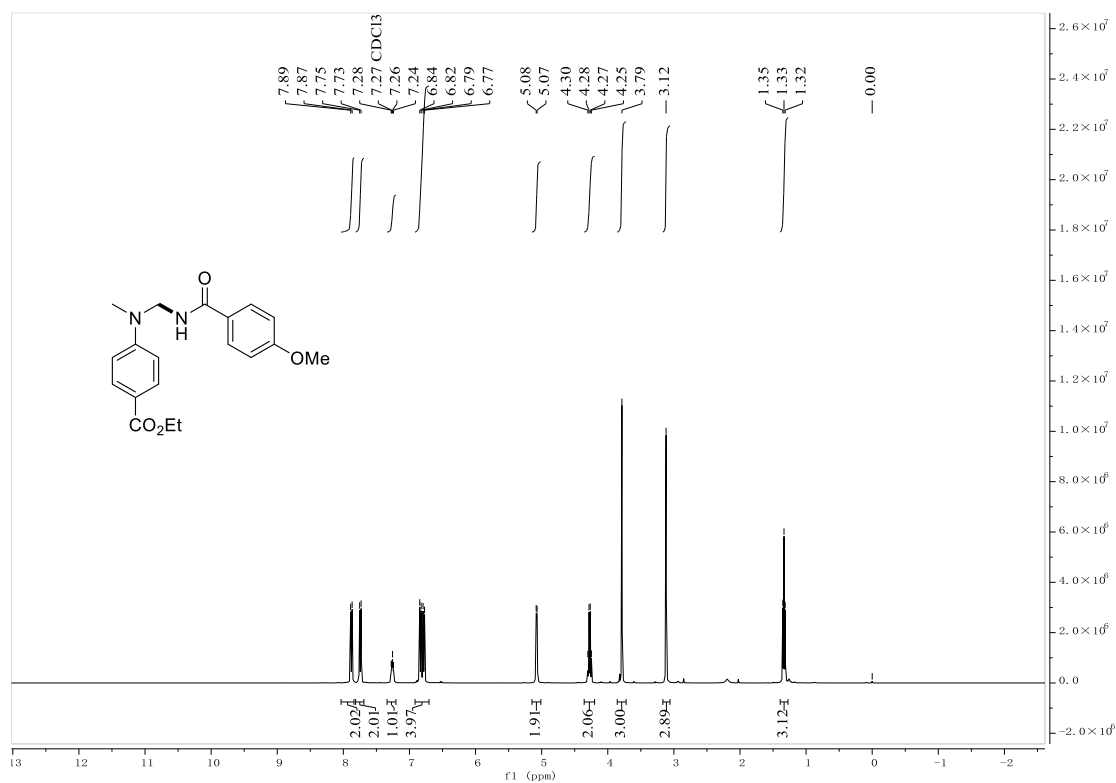
1: TOF MS ES+  
4.32e+006



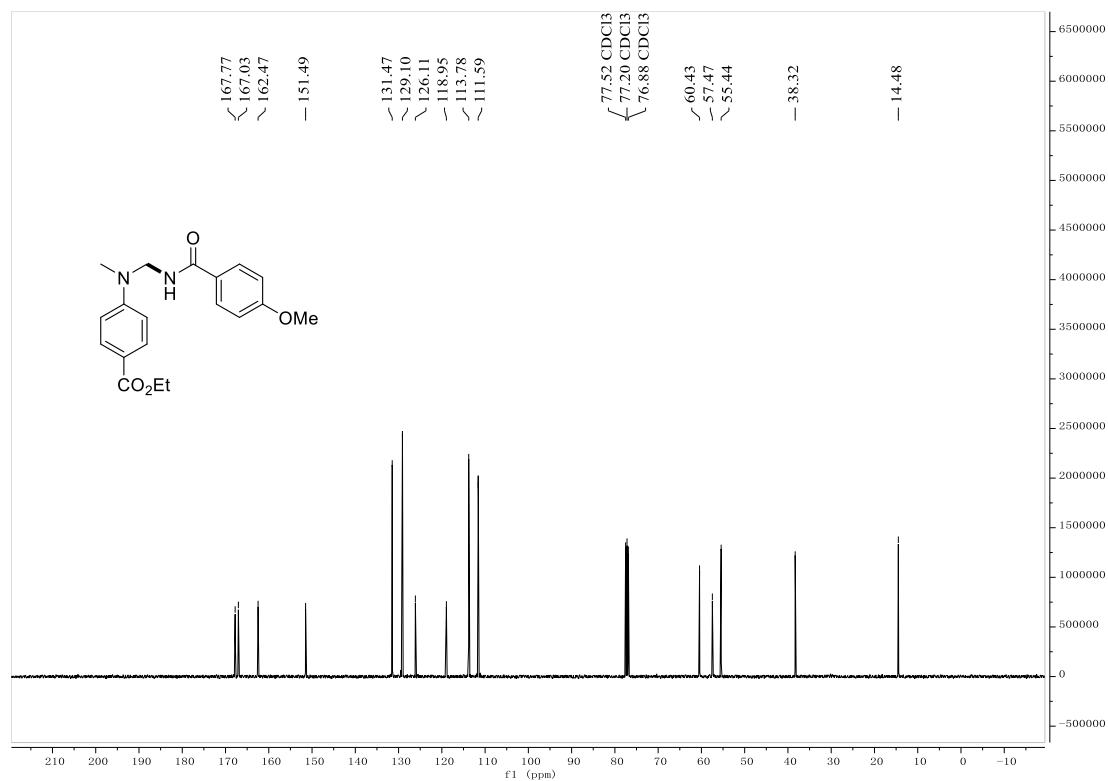
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|-----|--------|------|----------|------------------------------------------------------------------|
| 321.1217 | 321.1215   | 0.2 | 0.6 | 9.5 | 1800.6 | n/a  | n/a      | C <sub>17</sub> H <sub>18</sub> N <sub>2</sub> O <sub>3</sub> Na |

<sup>1</sup>H NMR of **34**



<sup>13</sup>C NMR of **34**



## HRMS of 34

Monoisotopic Mass, Even Electron Ions

1341 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

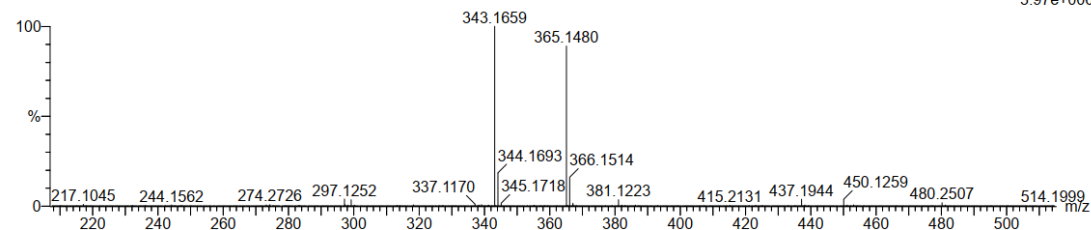
Elements Used:

C: 19-19 H: 23-23 N: 0-100 O: 0-100 Na: 0-7

41

250430-16-6 5 (0.068)

1: TOF MS ES+  
3.97e+006

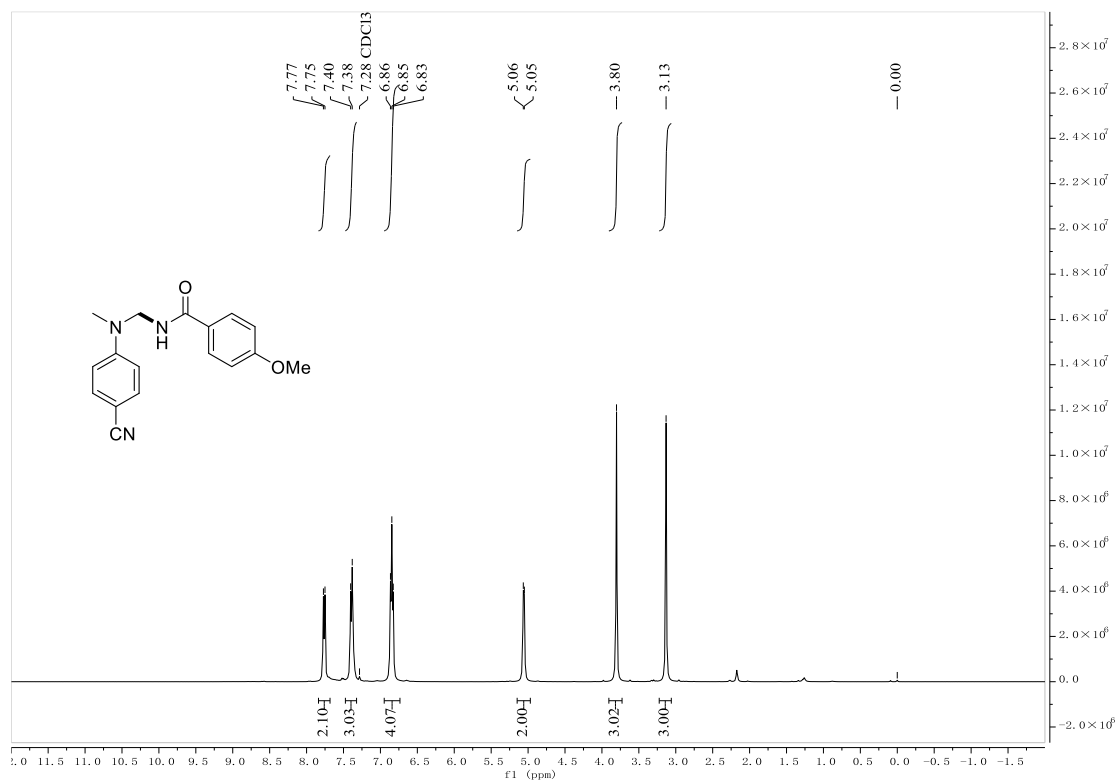


Minimum: -1.5

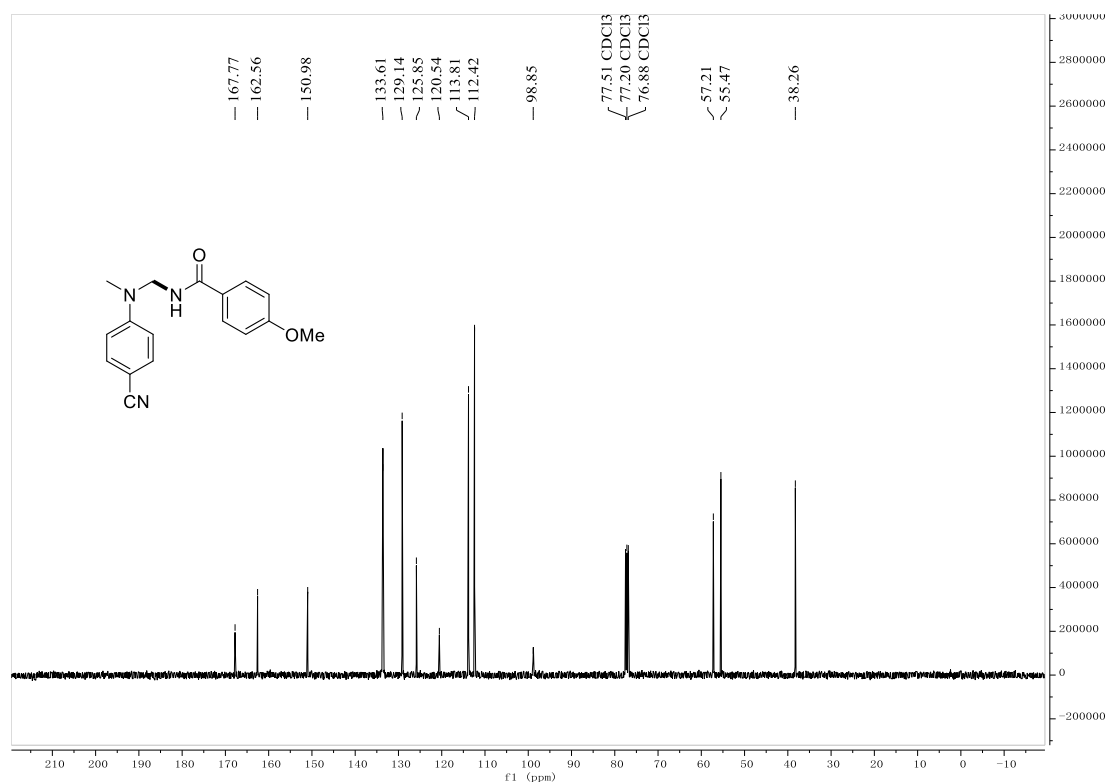
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT  | Norm | Conf (%) | Formula       |
|----------|------------|-----|-----|-----|--------|------|----------|---------------|
| 343.1659 | 343.1658   | 0.1 | 0.3 | 9.5 | 1889.2 | n/a  | n/a      | C19 H23 N2 O4 |

## <sup>1</sup>H NMR of 35



## <sup>13</sup>C NMR of 35



## HRMS of 35

Monoisotopic Mass, Even Electron Ions

1109 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

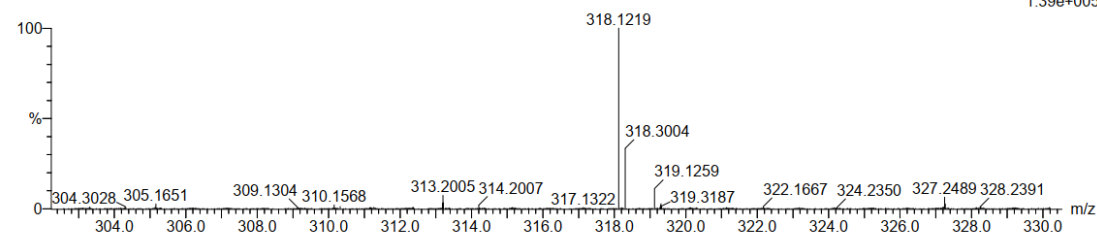
Elements Used:

C: 17-17 H: 17-17 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-22 6 (0.076)

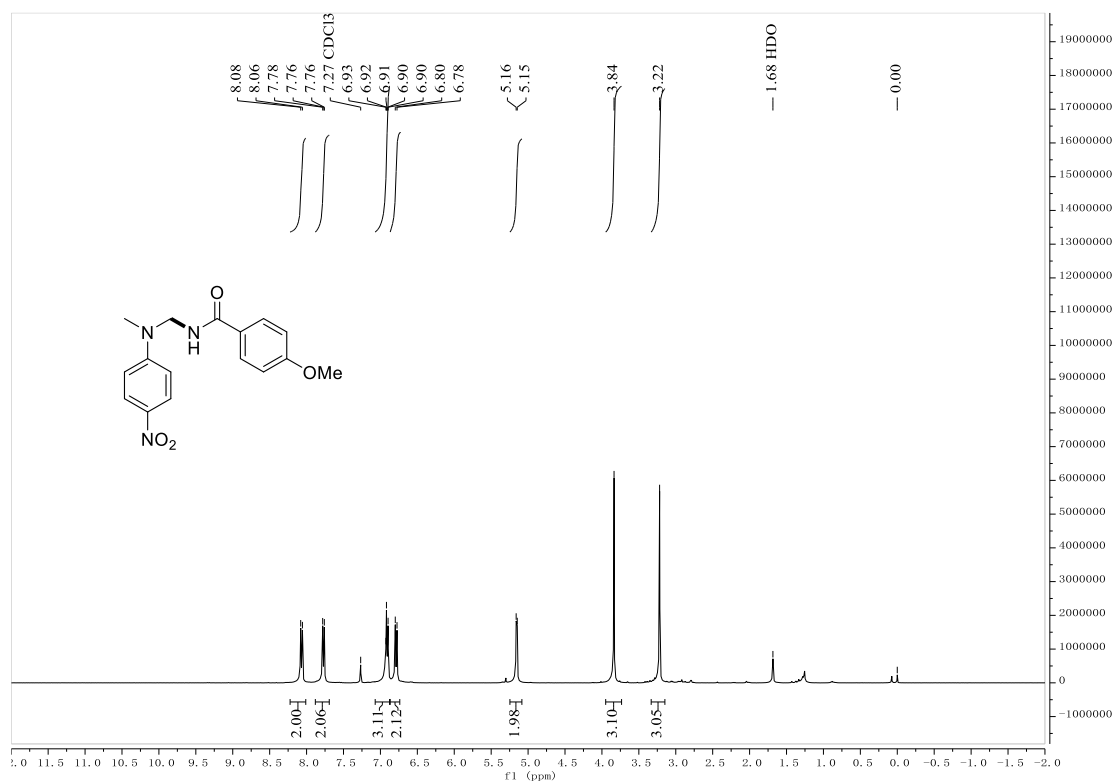
1: TOF MS ES+  
1.39e+005



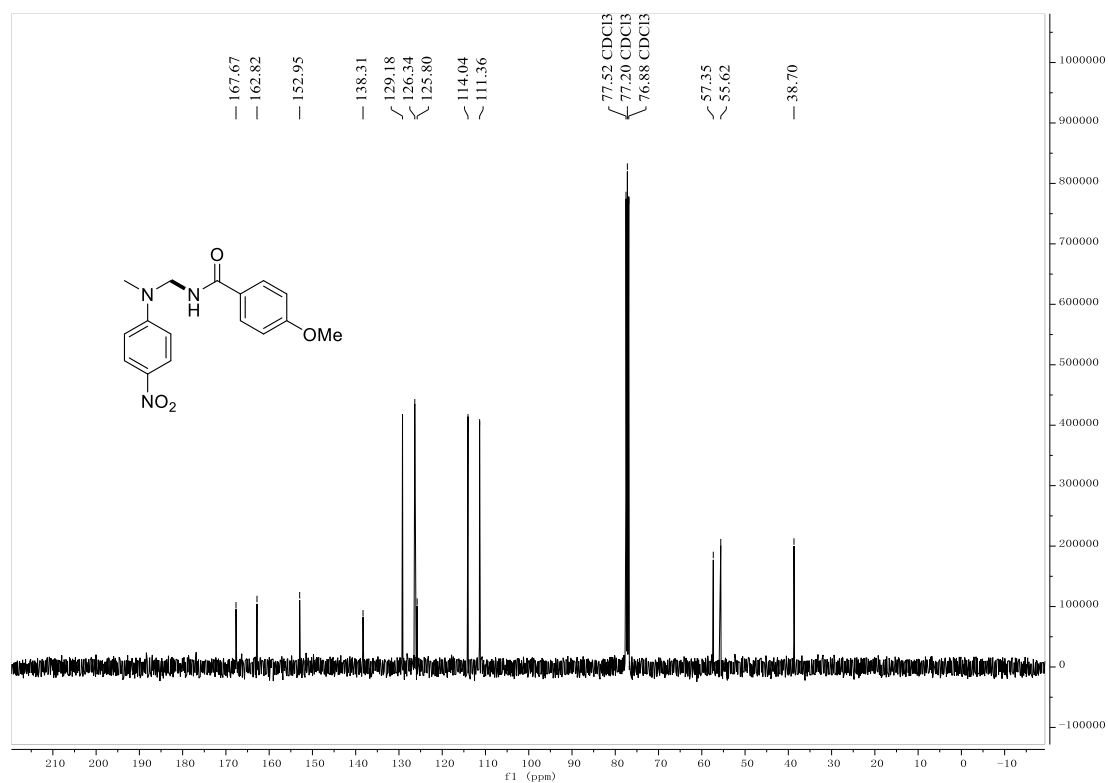
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|------|--------|------|----------|------------------------------------------------------------------|
| 318.1219 | 318.1218   | 0.1 | 0.3 | 10.5 | 1368.8 | n/a  | n/a      | C <sub>17</sub> H <sub>17</sub> N <sub>3</sub> O <sub>2</sub> Na |

# <sup>1</sup>H NMR of 36



# <sup>13</sup>C NMR of 36



## HRMS of 36

Monoisotopic Mass, Even Electron Ions

1248 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

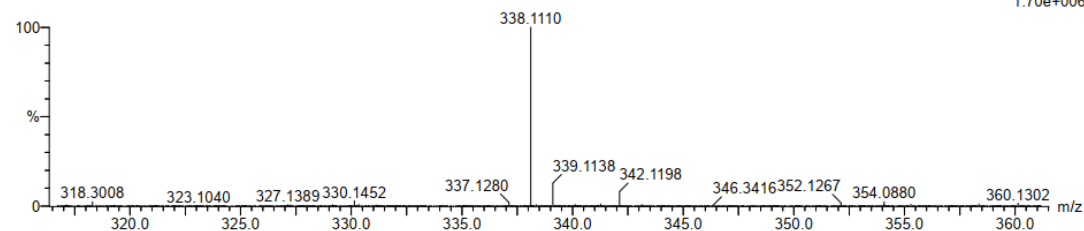
Elements Used:

C: 16-16 H: 17-17 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-23 6 (0.076)

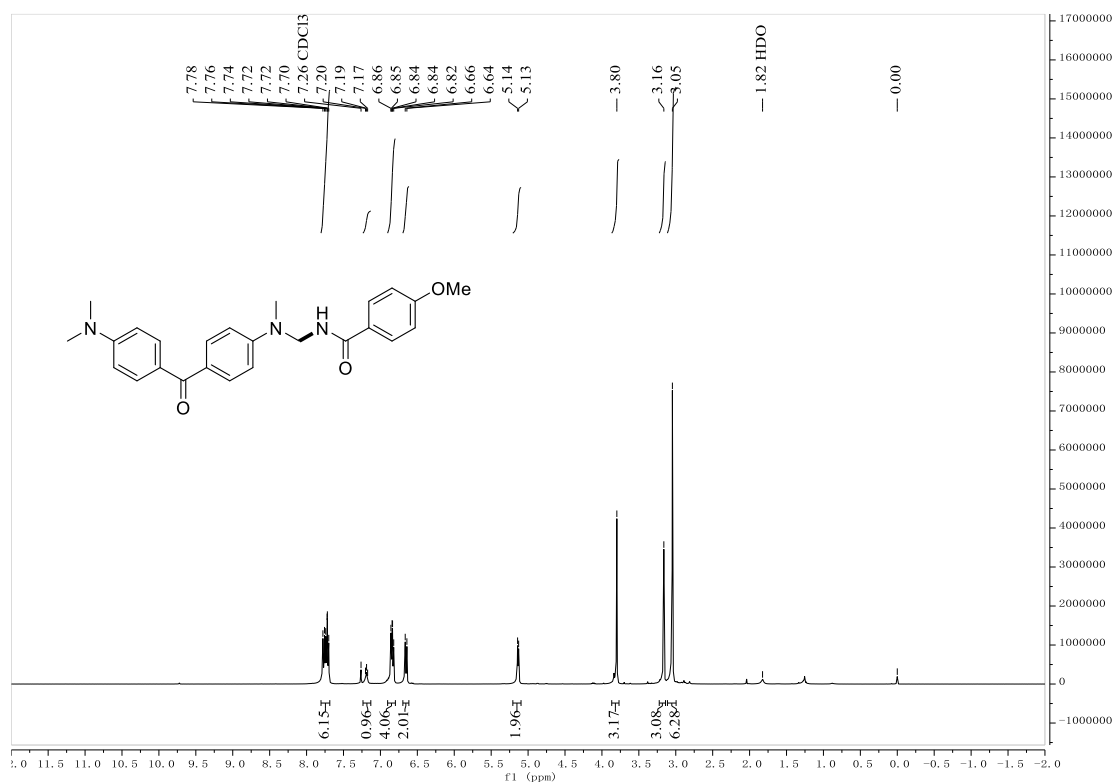
1: TOF MS ES+  
1.70e+006



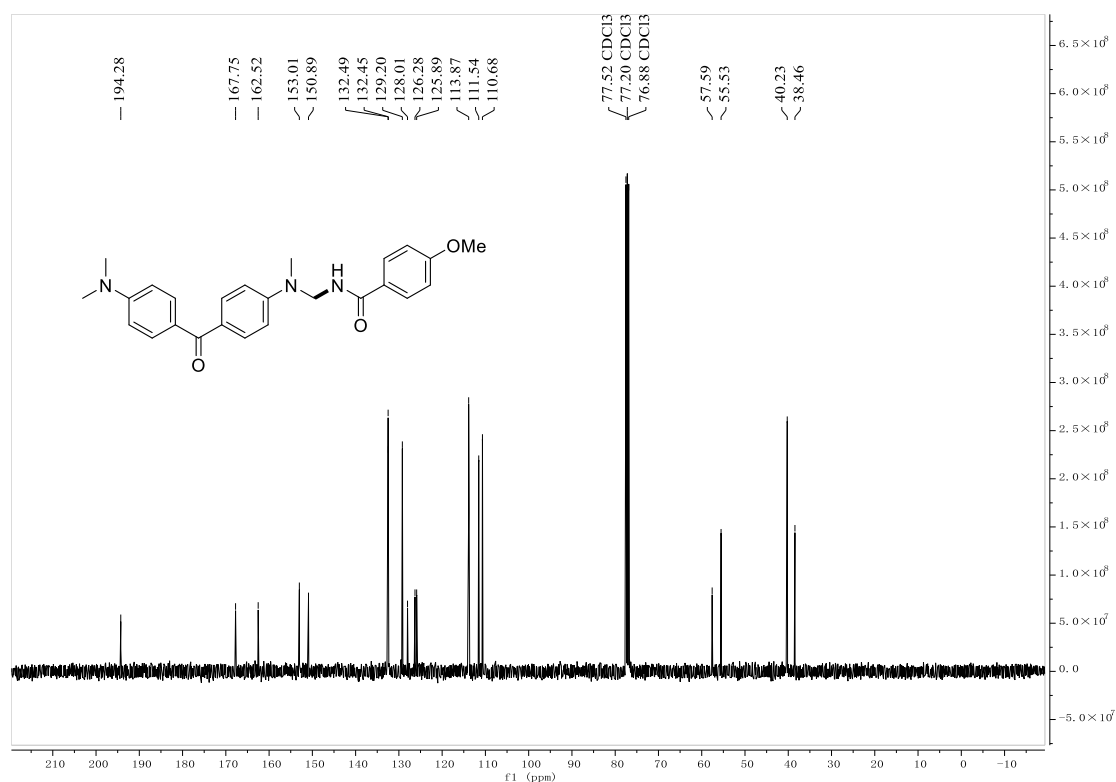
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula          |
|----------|------------|------|------|-----|--------|------|----------|------------------|
| 338.1110 | 338.1117   | -0.7 | -2.1 | 9.5 | 1788.1 | n/a  | n/a      | C16 H17 N3 O4 Na |

## <sup>1</sup>H NMR of 37



## <sup>13</sup>C NMR of 37



## HRMS of 37

Monoisotopic Mass, Even Electron Ions

2413 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

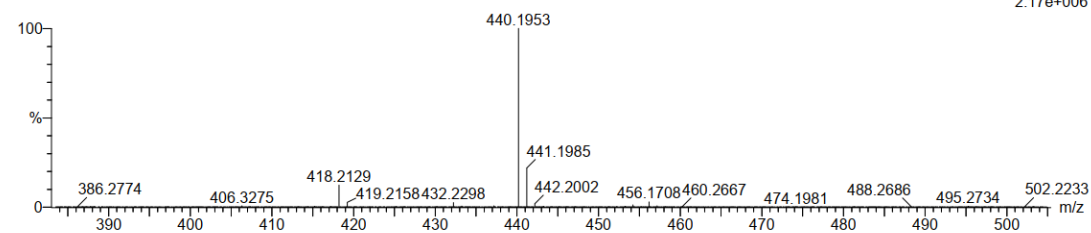
Elements Used:

C: 25-25 H: 27-27 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-28 13 (0.137)

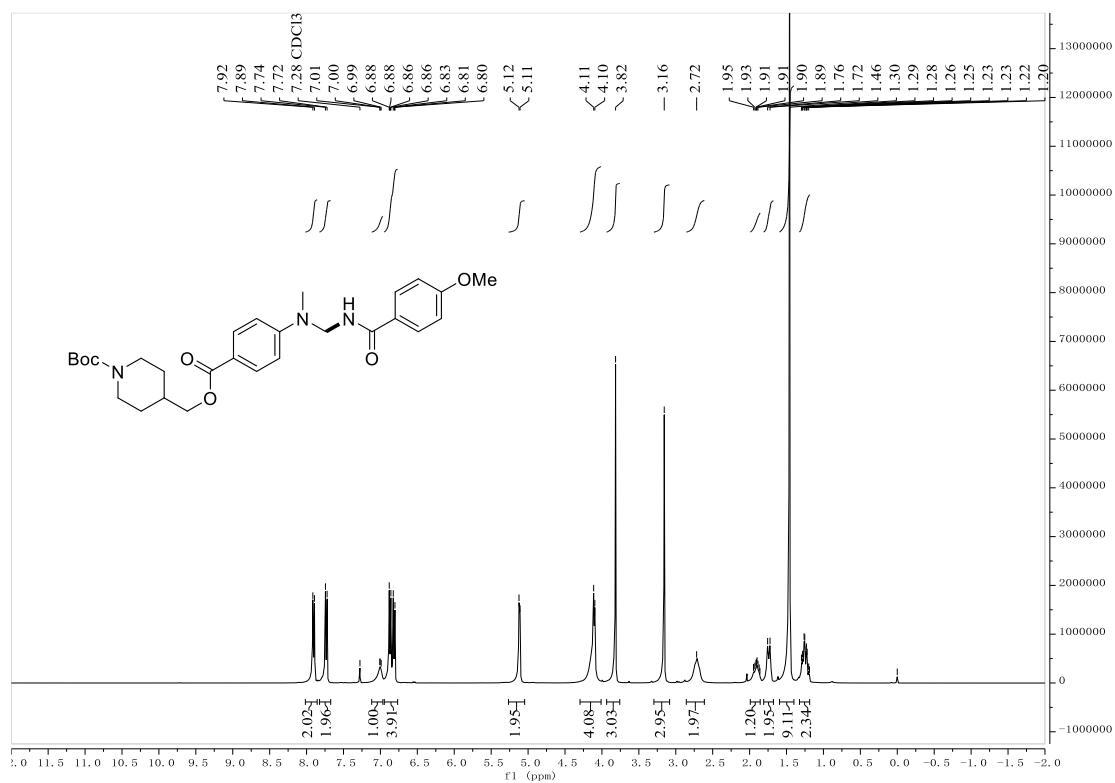
1: TOF MS ES+  
2.17e+006



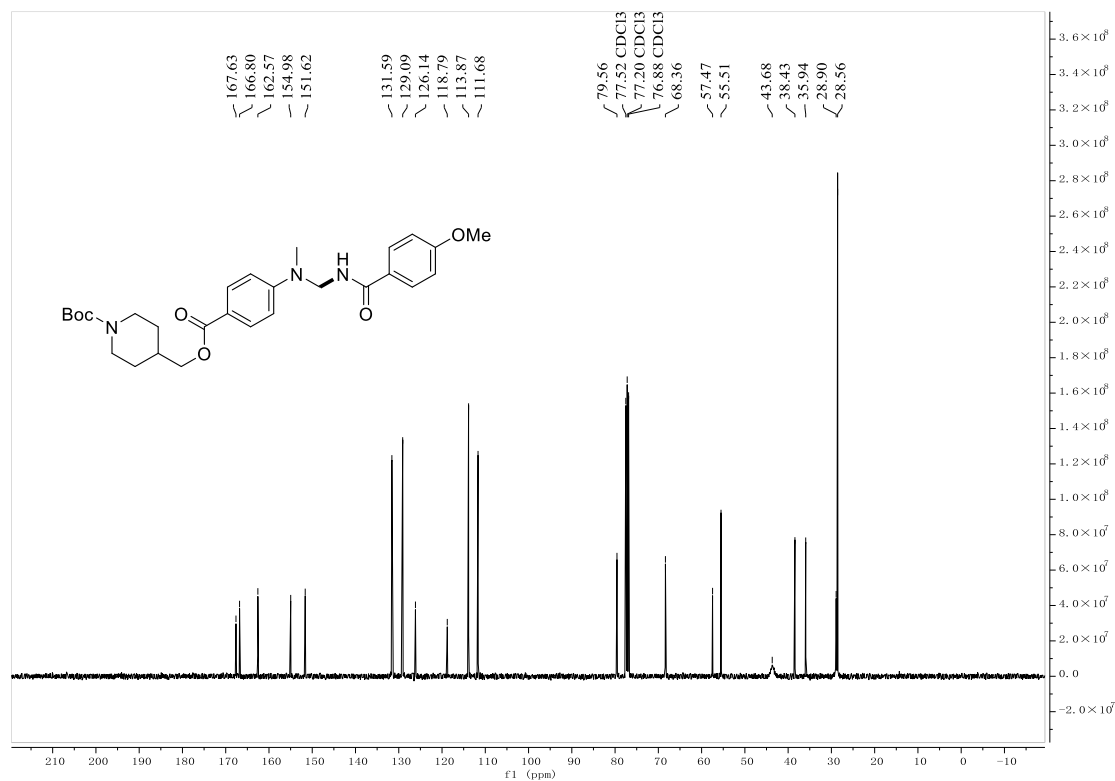
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|------|--------|------|----------|------------------------------------------------------------------|
| 440.1953 | 440.1950   | 0.3 | 0.7 | 13.5 | 1597.7 | n/a  | n/a      | C <sub>25</sub> H <sub>27</sub> N <sub>3</sub> O <sub>3</sub> Na |

<sup>1</sup>H NMR of **38**



<sup>13</sup>C NMR of **38**



## HRMS of 38

Monoisotopic Mass, Even Electron Ions

3648 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

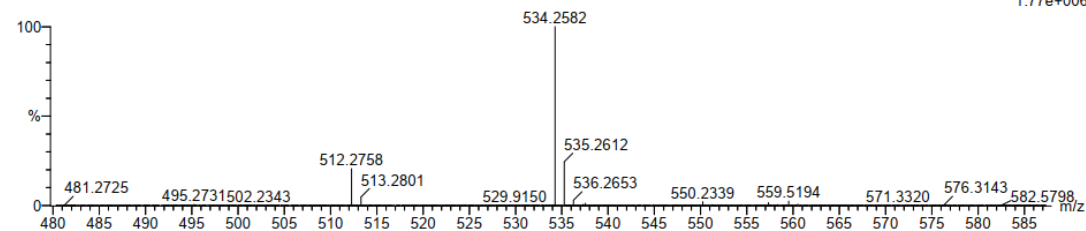
Elements Used:

C: 28-28 H: 37-37 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-29 16 (0.162)

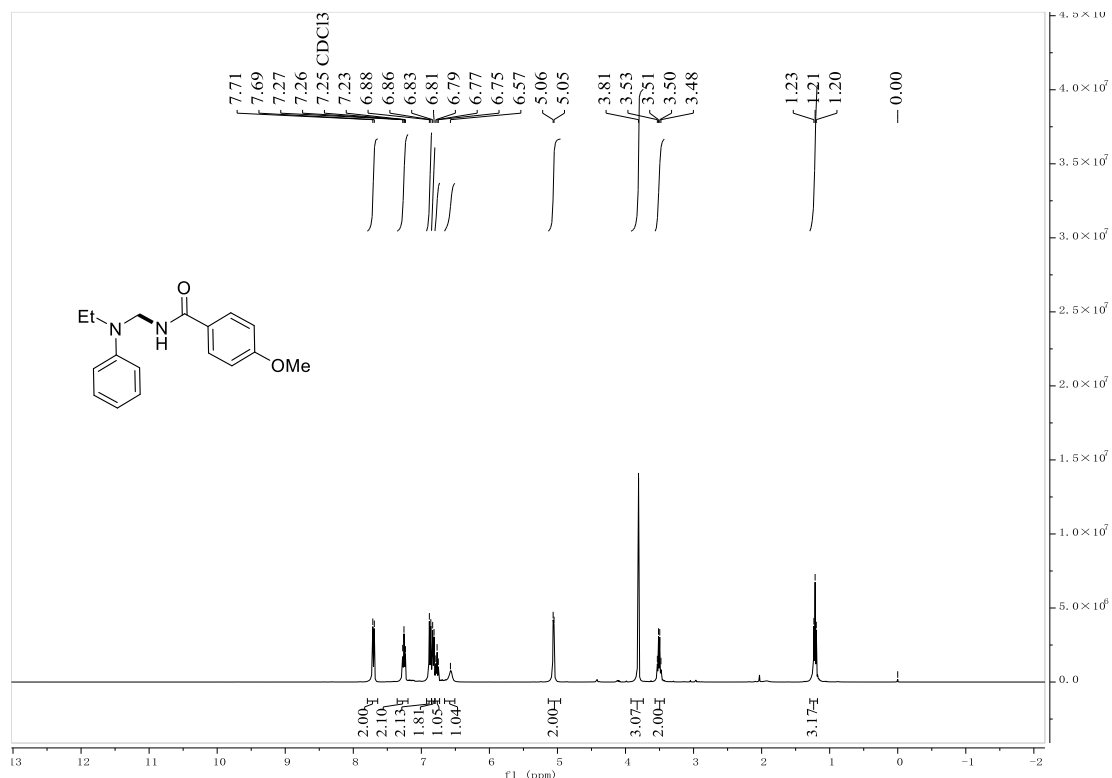
1: TOF MS ES+  
1.77e+006



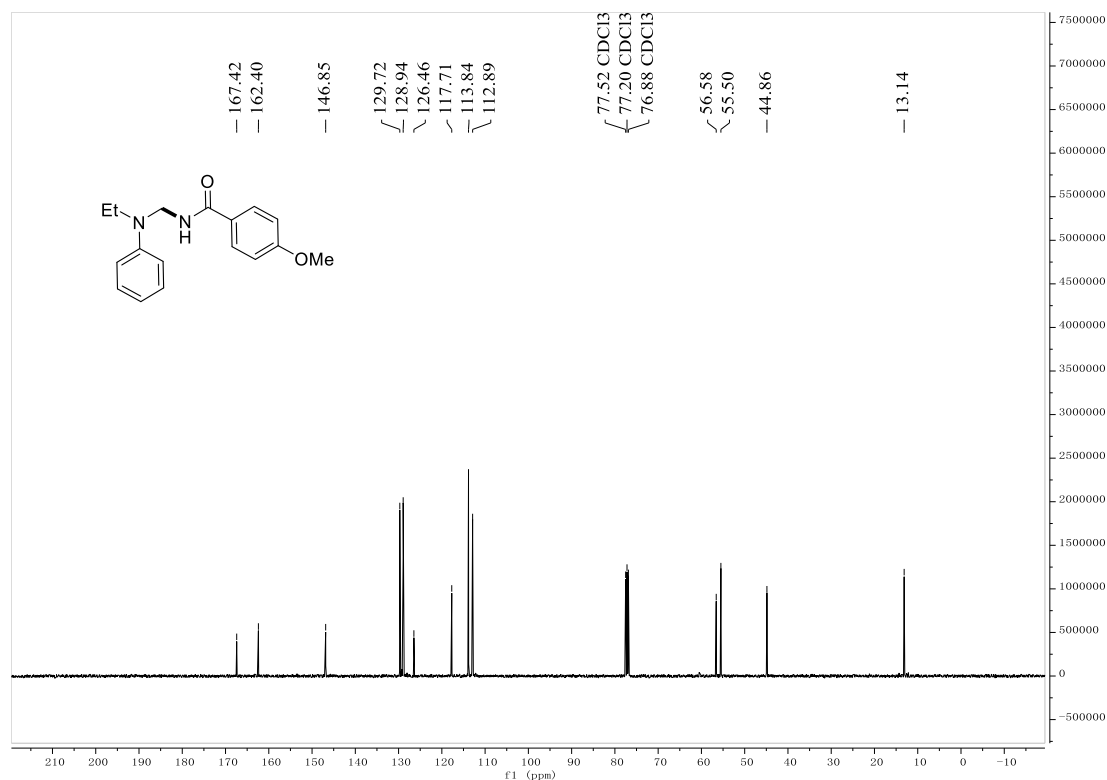
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|------|--------|------|----------|------------------------------------------------------------------|
| 534.2582 | 534.2580   | 0.2 | 0.4 | 11.5 | 1543.8 | n/a  | n/a      | C <sub>28</sub> H <sub>37</sub> N <sub>3</sub> O <sub>6</sub> Na |

## <sup>1</sup>H NMR of 39



## <sup>13</sup>C NMR of **39**



## HRMS of **39**

Monoisotopic Mass, Even Electron Ions

1026 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

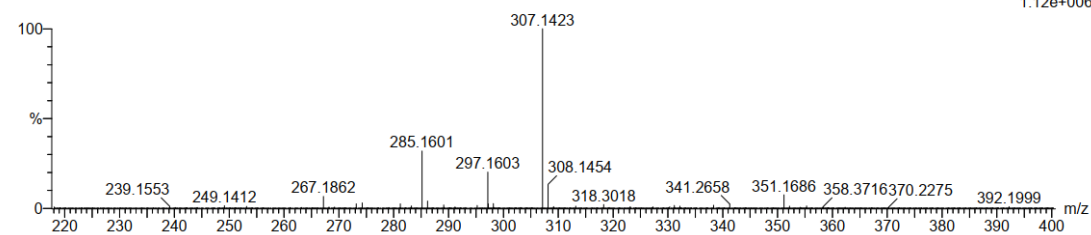
Elements Used:

C: 17-17 H: 20-20 N: 0-100 O: 0-100 Na: 0-7

41

250430-16-1 5 (0.068)

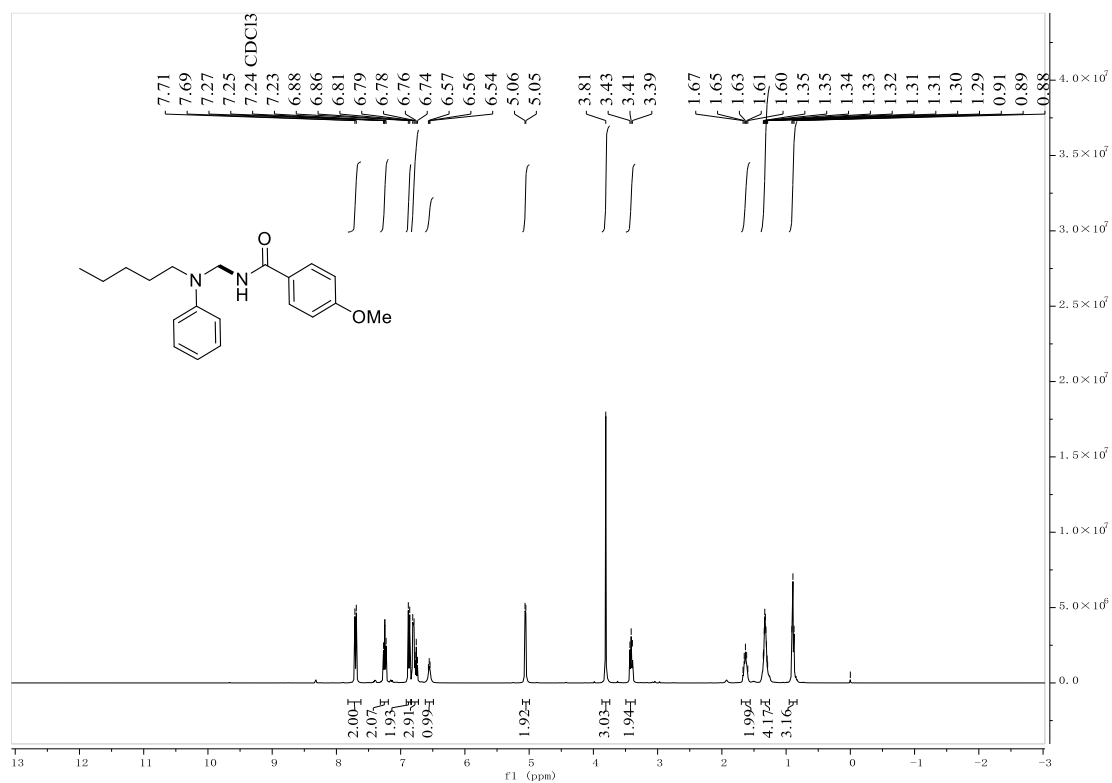
1: TOF MS ES+  
1.12e+006



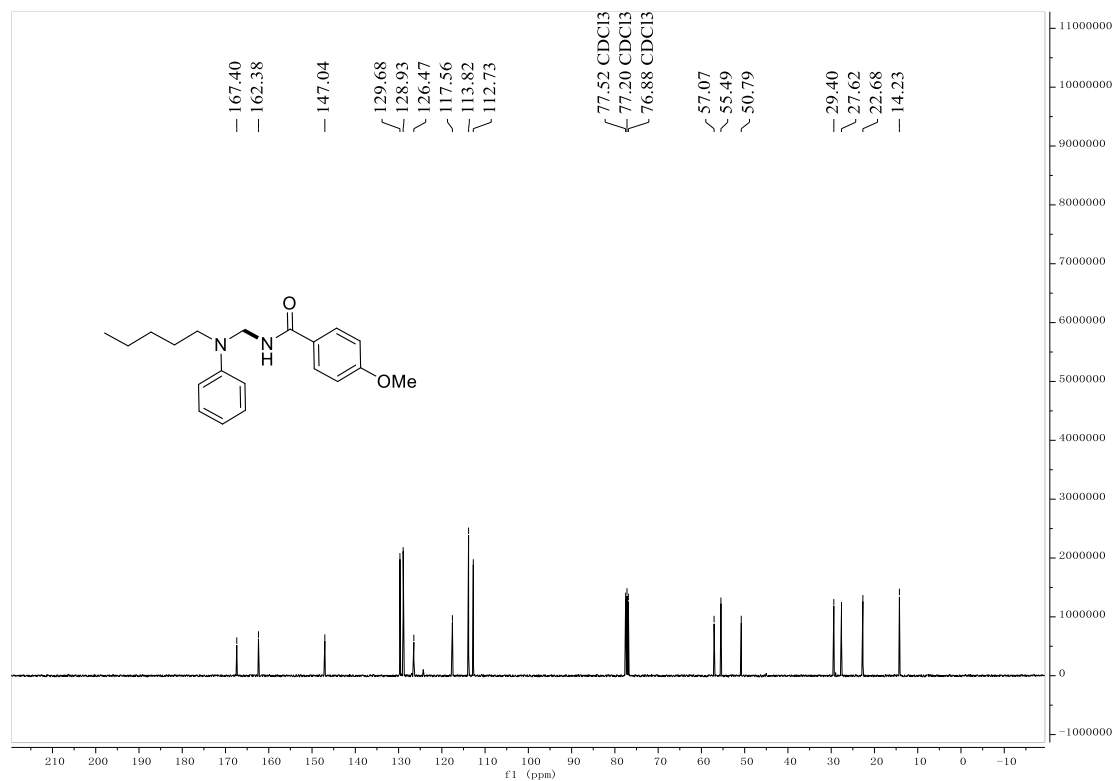
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|-----|--------|------|----------|------------------------------------------------------------------|
| 307.1423 | 307.1422   | 0.1 | 0.3 | 8.5 | 1599.5 | n/a  | n/a      | C <sub>17</sub> H <sub>20</sub> N <sub>2</sub> O <sub>2</sub> Na |

# <sup>1</sup>H NMR of 40



# <sup>13</sup>C NMR of 40



## HRMS of 40

Monoisotopic Mass, Even Electron Ions

1406 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

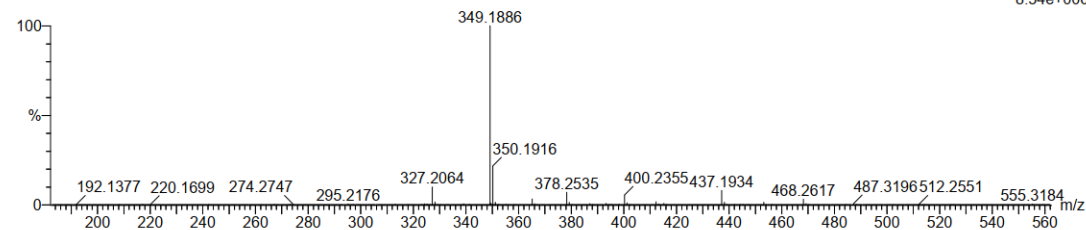
Elements Used:

C: 20-20 H: 26-26 N: 0-100 O: 0-100 Na: 0-7

41

250430-16-2 7 (0.085)

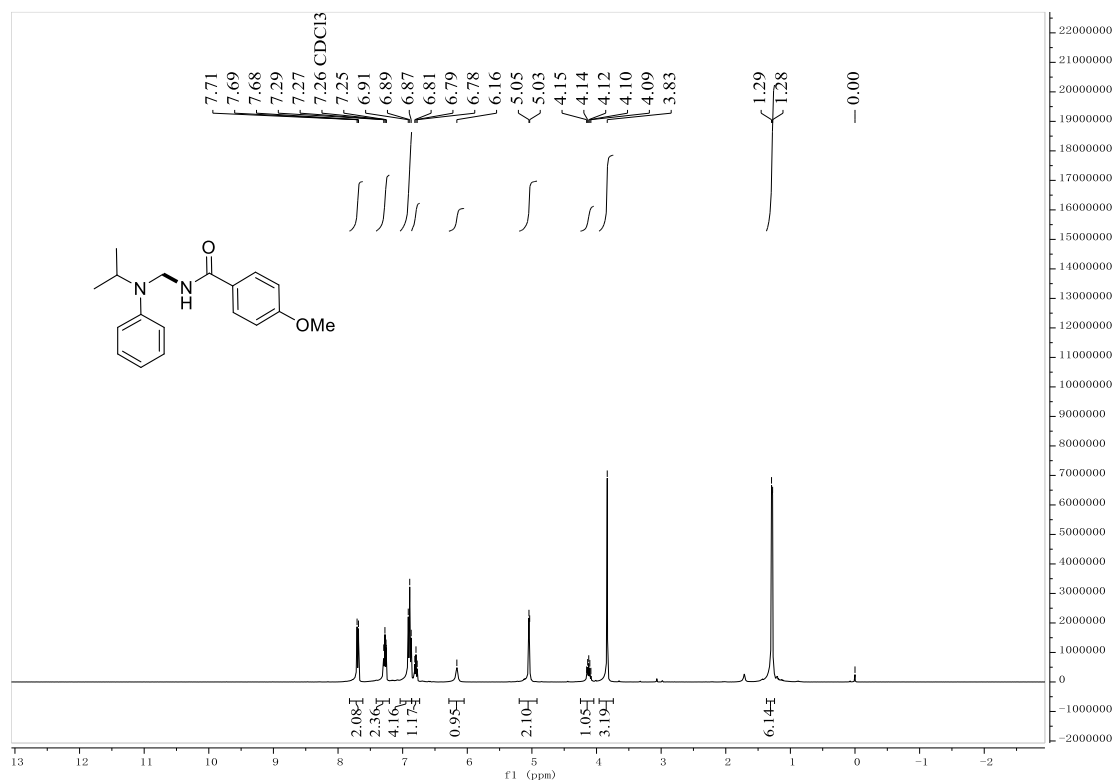
1: TOF MS ES+  
8.54e+006



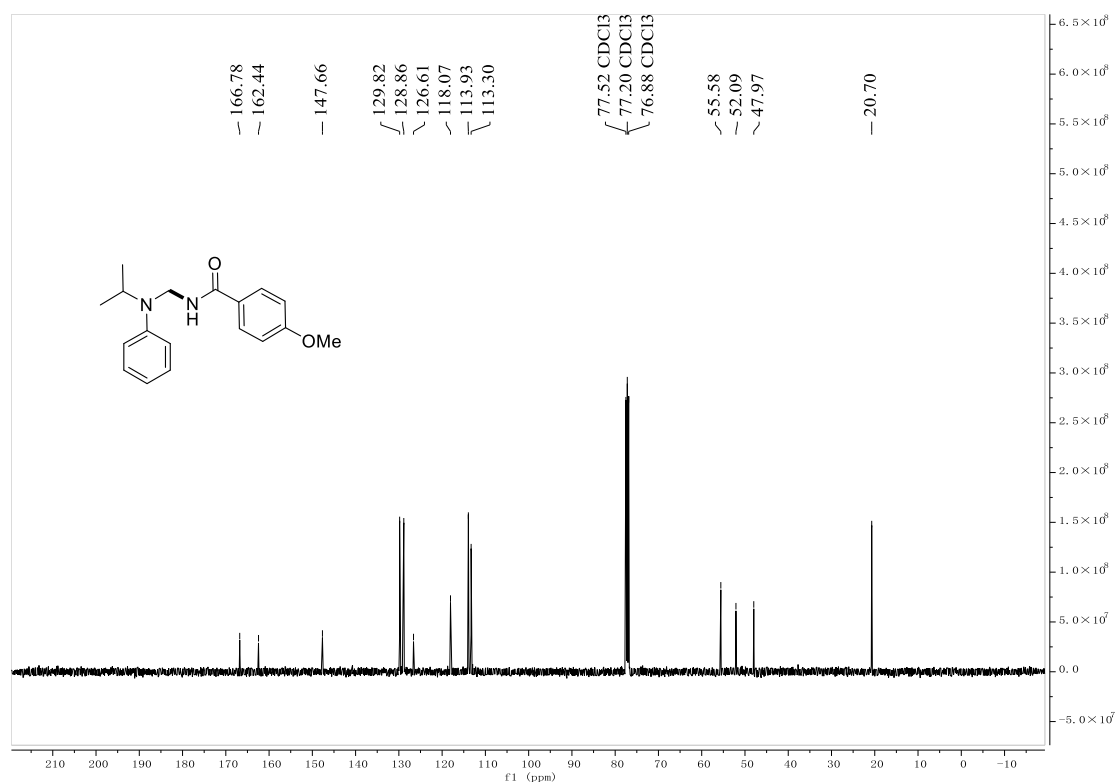
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|------|------|-----|--------|------|----------|------------------------------------------------------------------|
| 349.1886 | 349.1892   | -0.6 | -1.7 | 8.5 | 2006.5 | n/a  | n/a      | C <sub>20</sub> H <sub>26</sub> N <sub>2</sub> O <sub>2</sub> Na |

## <sup>1</sup>H NMR of 41



## <sup>13</sup>C NMR of **41**



## HRMS of **41**

Monoisotopic Mass, Even Electron Ions

1147 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

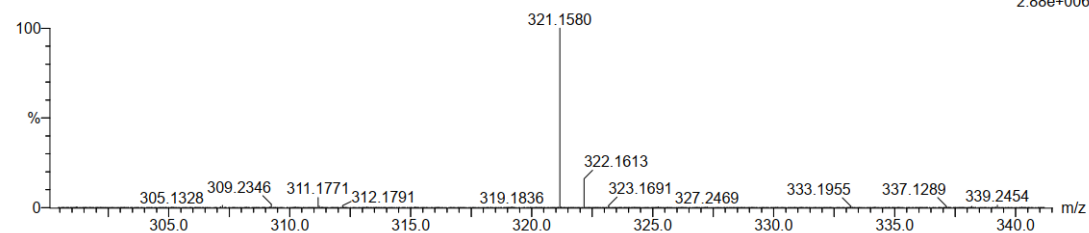
Elements Used:

C: 18-18 H: 22-22 N: 0-100 O: 0-100 Na: 0-7

**41**

250430-16-4 6 (0.076)

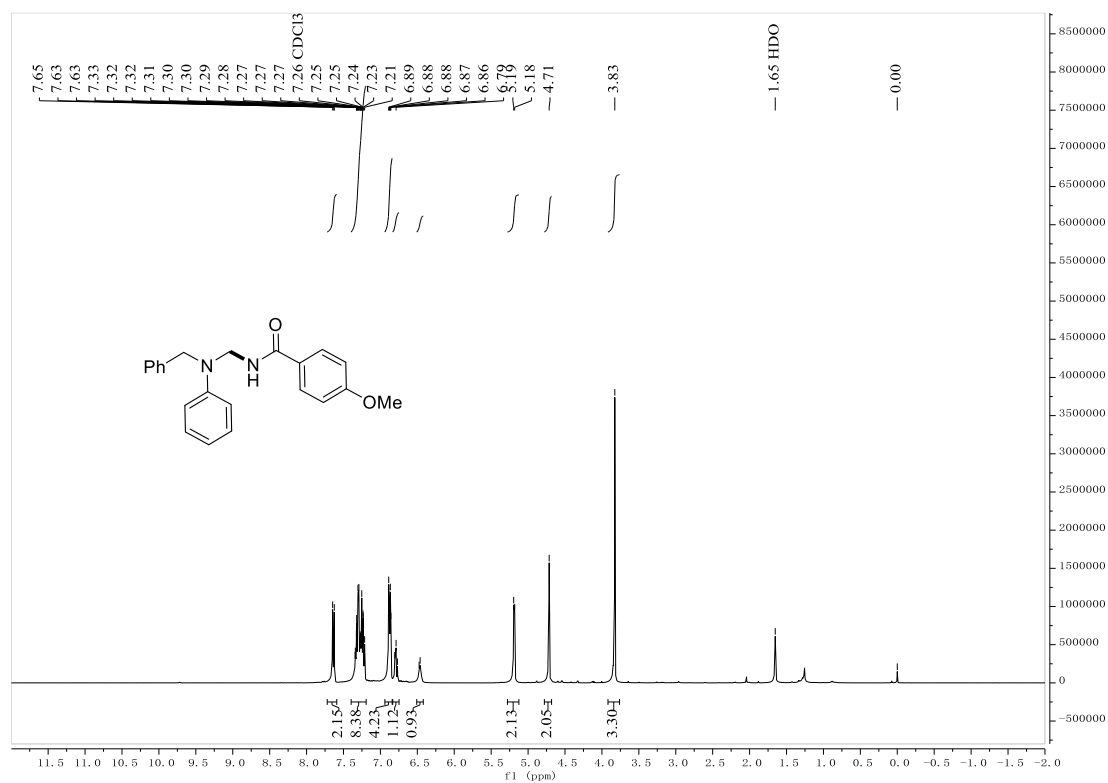
1: TOF MS ES+  
2.88e+006



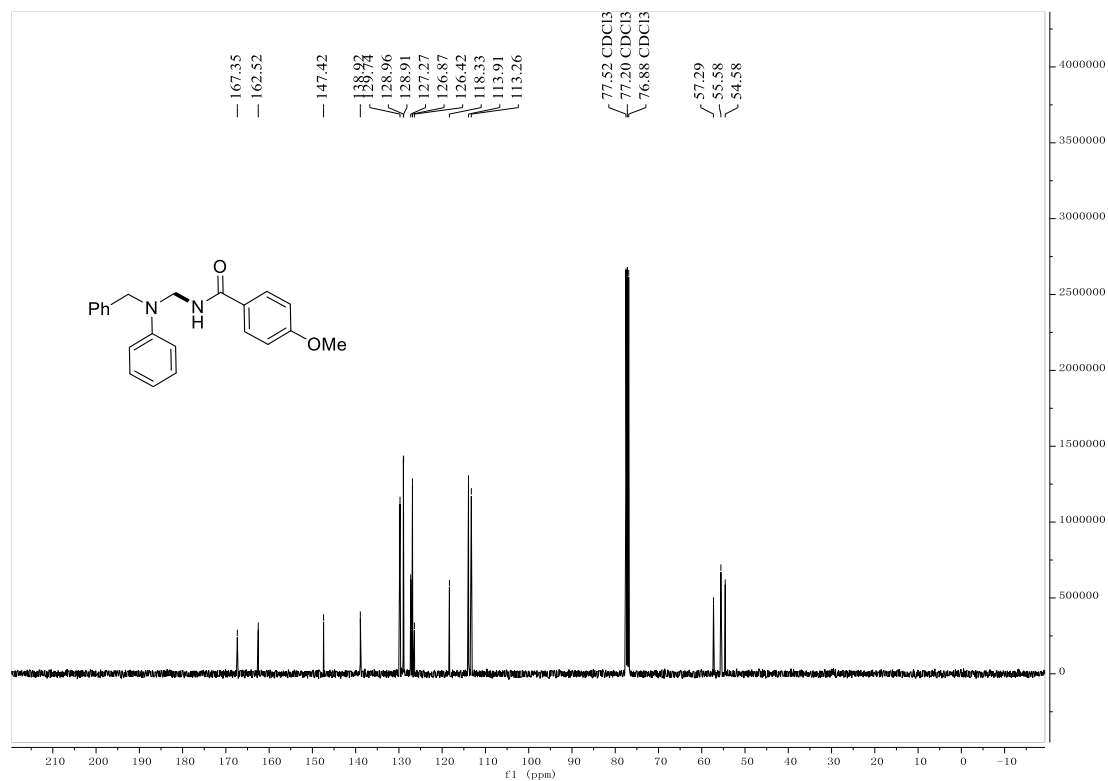
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|-----|--------|------|----------|------------------------------------------------------------------|
| 321.1580 | 321.1579   | 0.1 | 0.3 | 8.5 | 1979.9 | n/a  | n/a      | C <sub>18</sub> H <sub>22</sub> N <sub>2</sub> O <sub>2</sub> Na |

# <sup>1</sup>H NMR of 42



# <sup>13</sup>C NMR of 42



## HRMS of 42

Monoisotopic Mass, Even Electron Ions

2958 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

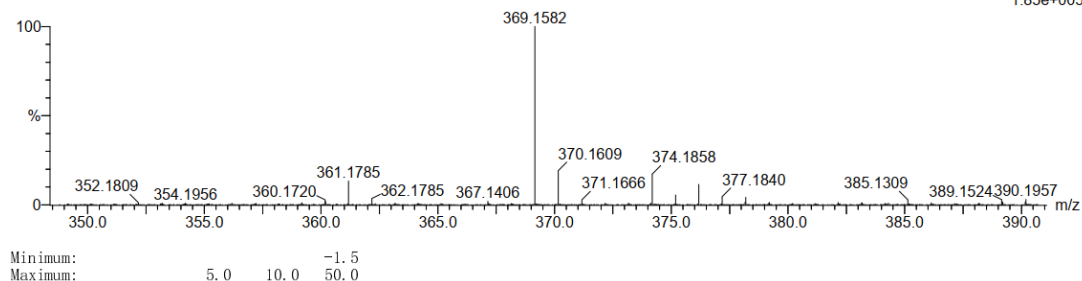
Elements Used:

C: 22-22 H: 22-22 N: 0-100 O: 0-100 Na: 0-4 K: 0-2

31

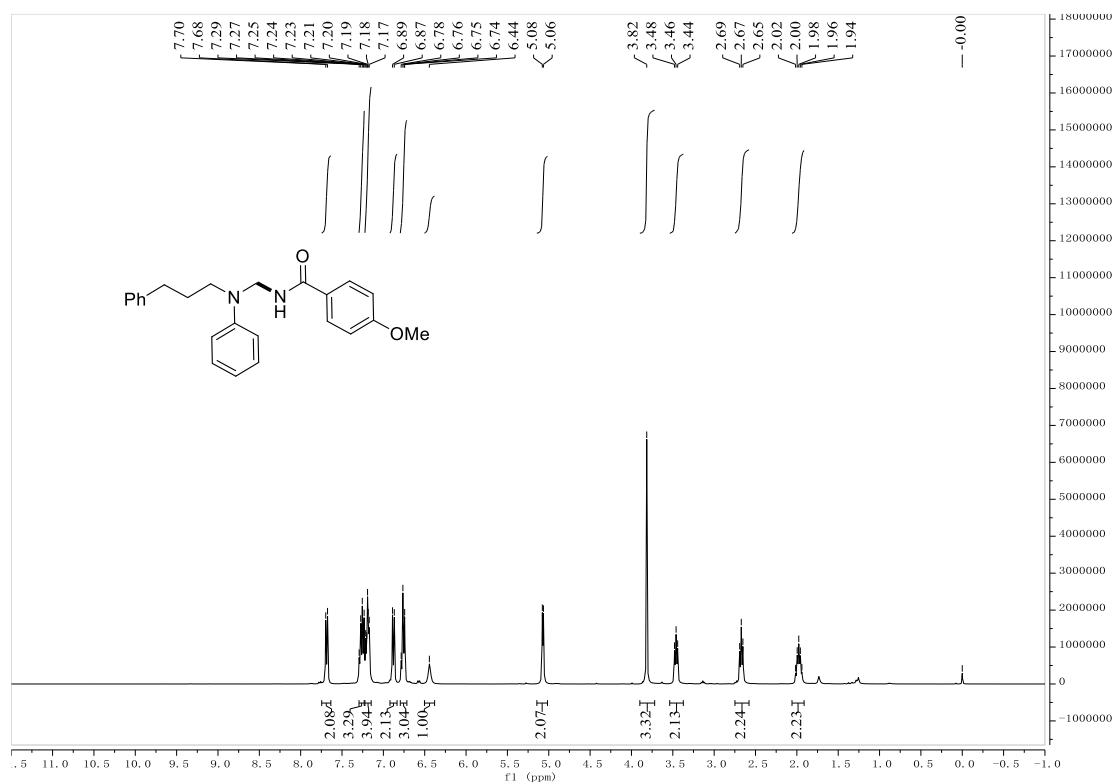
250707-4-1 12 (0.088)

1: TOF MS ES+  
1.85e+005

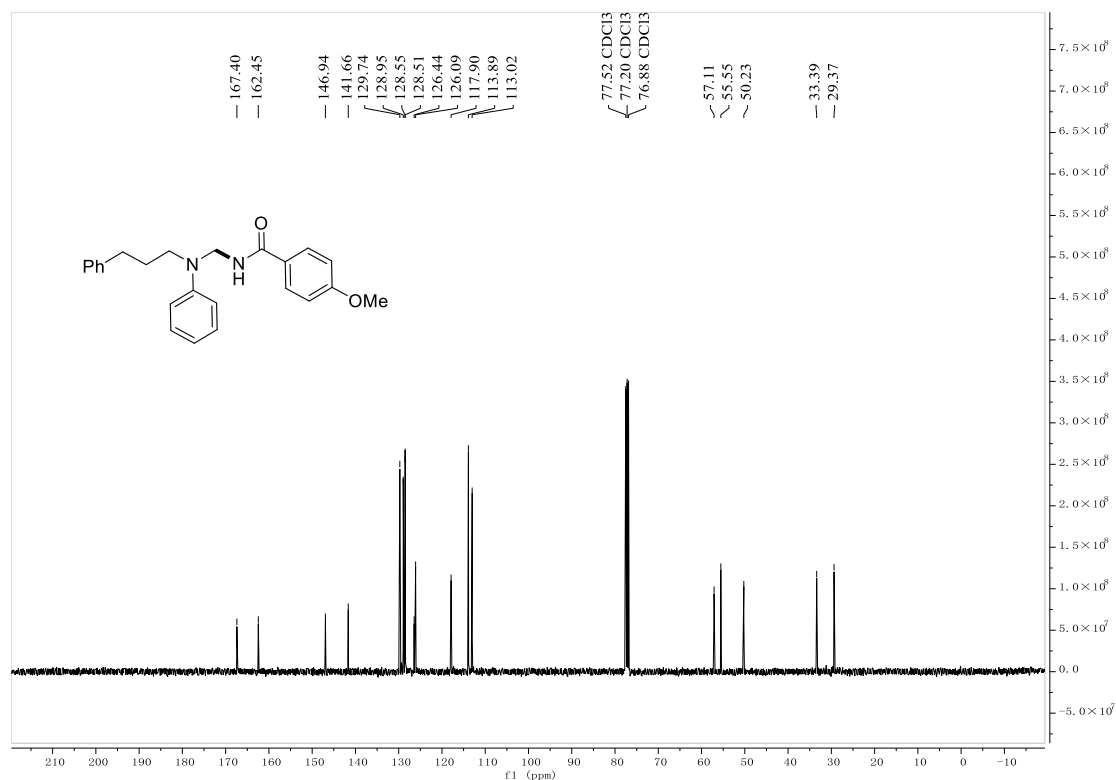


| Mass     | Calc. Mass | mDa | PPM | DBE  | i-FIT | Norm | Conf (%) | Formula          |
|----------|------------|-----|-----|------|-------|------|----------|------------------|
| 369.1582 | 369.1579   | 0.3 | 0.8 | 12.5 | 635.5 | n/a  | n/a      | C22 H22 N2 O2 Na |

## <sup>1</sup>H NMR of 43



## <sup>13</sup>C NMR of **43**



## HRMS of **43**

Monoisotopic Mass, Even Electron Ions

1931 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

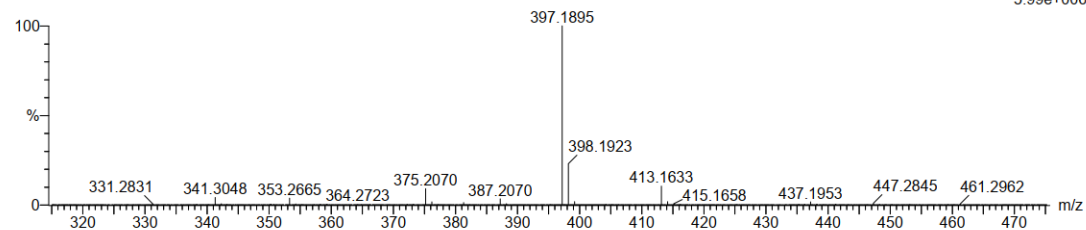
Elements Used:

C: 24-24 H: 26-26 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-27 10 (0.111)

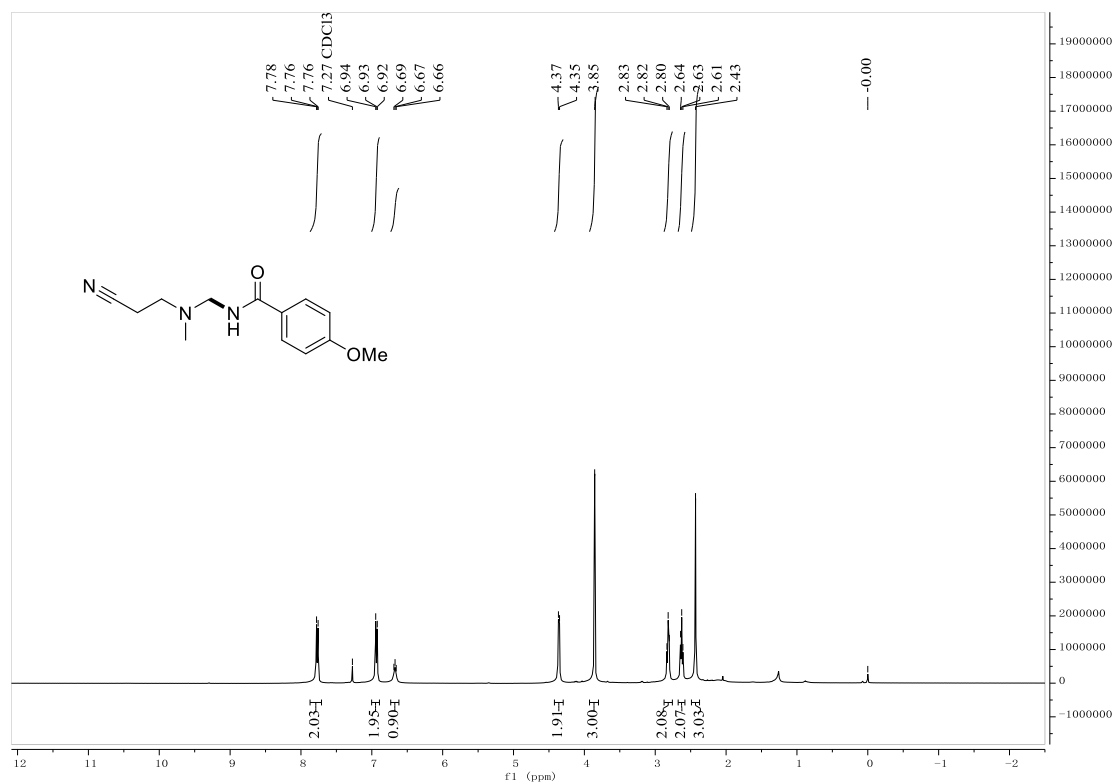
1: TOF MS ES+  
5.99e+006



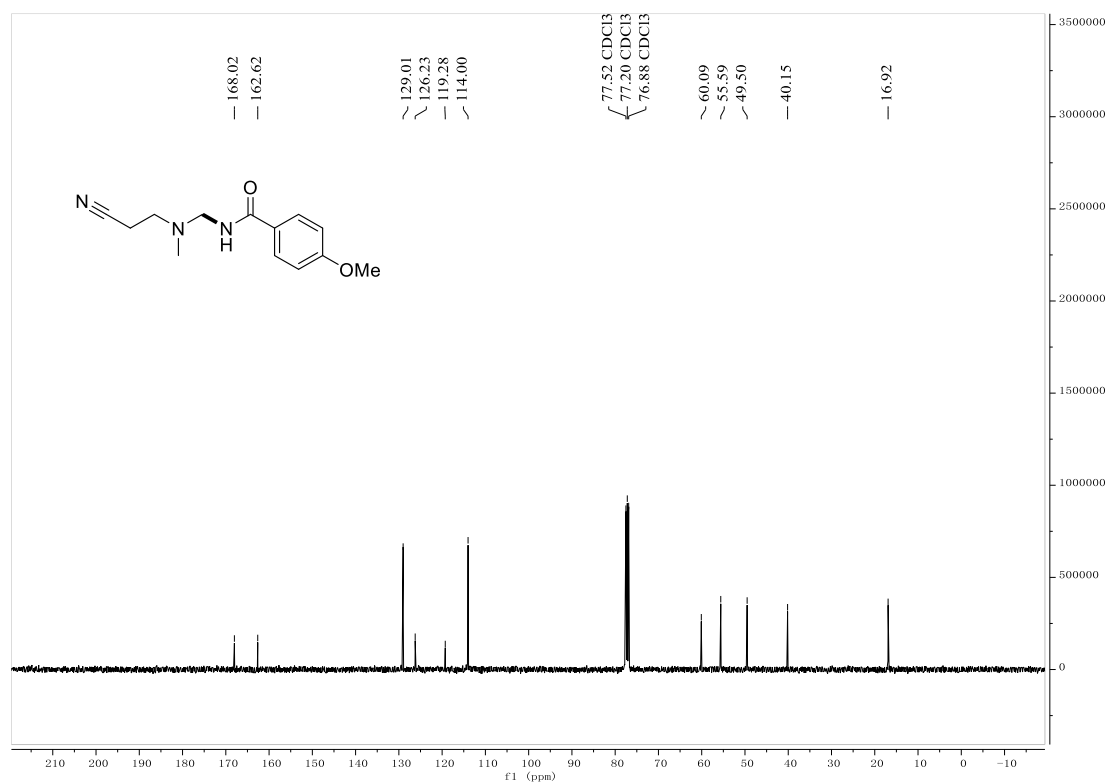
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|------|--------|------|----------|------------------------------------------------------------------|
| 397.1895 | 397.1892   | 0.3 | 0.8 | 12.5 | 1847.9 | n/a  | n/a      | C <sub>24</sub> H <sub>26</sub> N <sub>2</sub> O <sub>2</sub> Na |

<sup>1</sup>H NMR of **44**



<sup>13</sup>C NMR of **44**



## HRMS of 44

Monoisotopic Mass, Even Electron Ions

1294 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

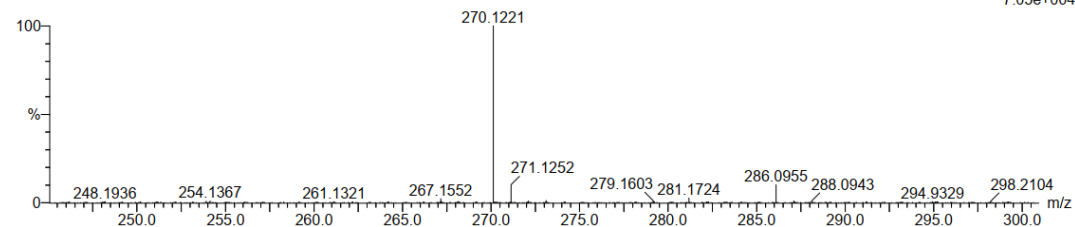
Elements Used:

C: 13-13 H: 17-17 N: 0-100 O: 0-100 Na: 0-4 K: 0-2

7

250707.4-9.54 (0.316)

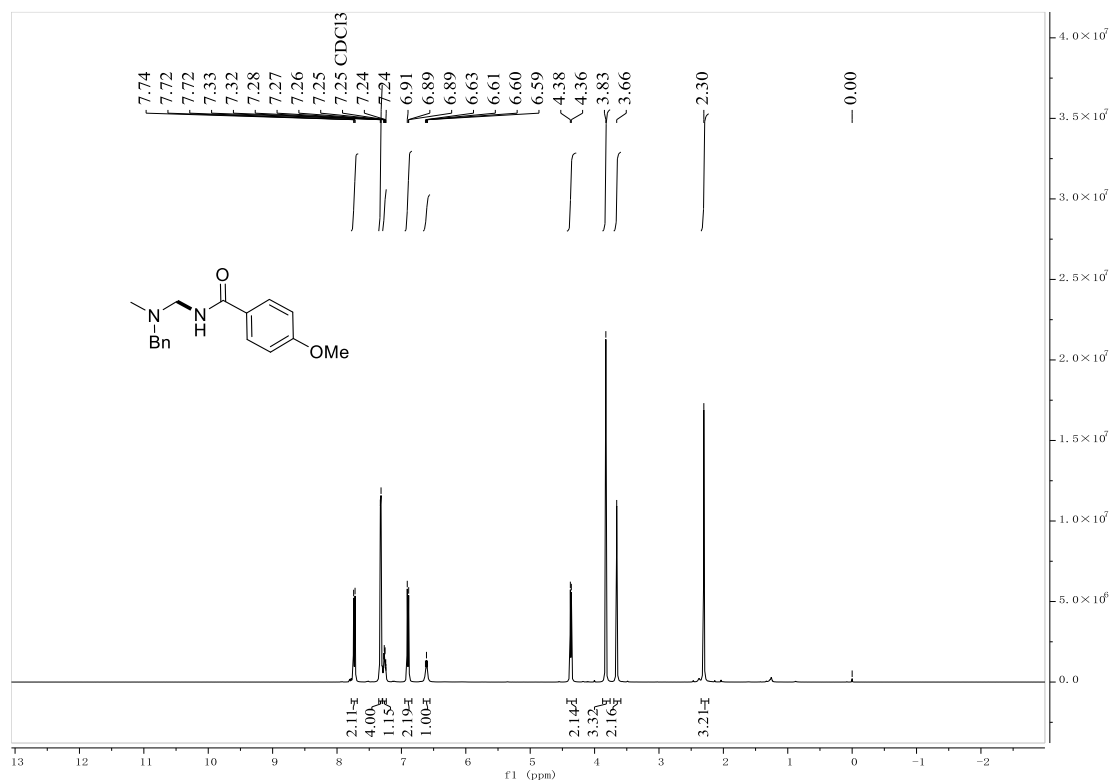
1: TOF MS ES+  
7.05e+004



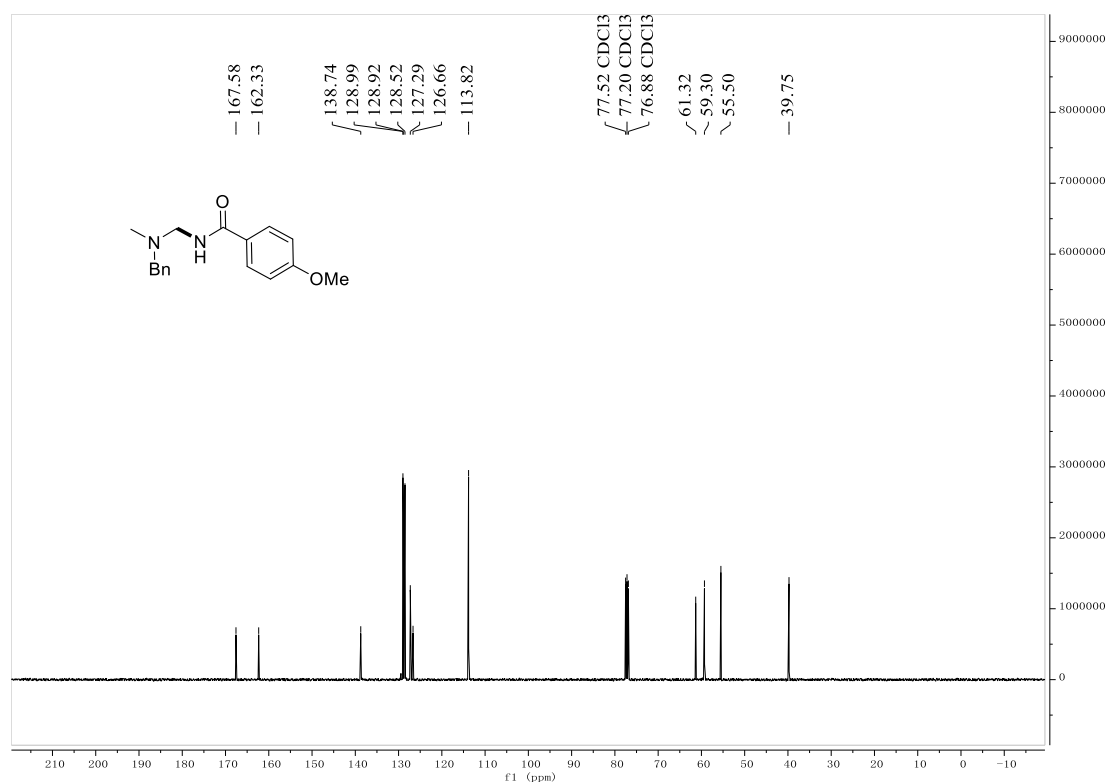
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT | Norm | Conf (%) | Formula          |
|----------|------------|-----|-----|-----|-------|------|----------|------------------|
| 270.1221 | 270.1218   | 0.3 | 1.1 | 6.5 | 363.5 | n/a  | n/a      | C13 H17 N3 O2 Na |

## <sup>1</sup>H NMR of 45



## <sup>13</sup>C NMR of 45



## HRMS of 45

Monoisotopic Mass, Even Electron Ions

1026 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

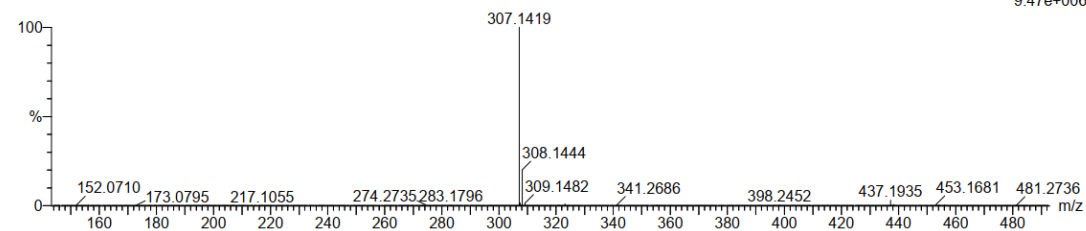
Elements Used:

C: 17-17 H: 20-20 N: 0-100 O: 0-100 Na: 0-7

41

250430-16-3 6 (0.076)

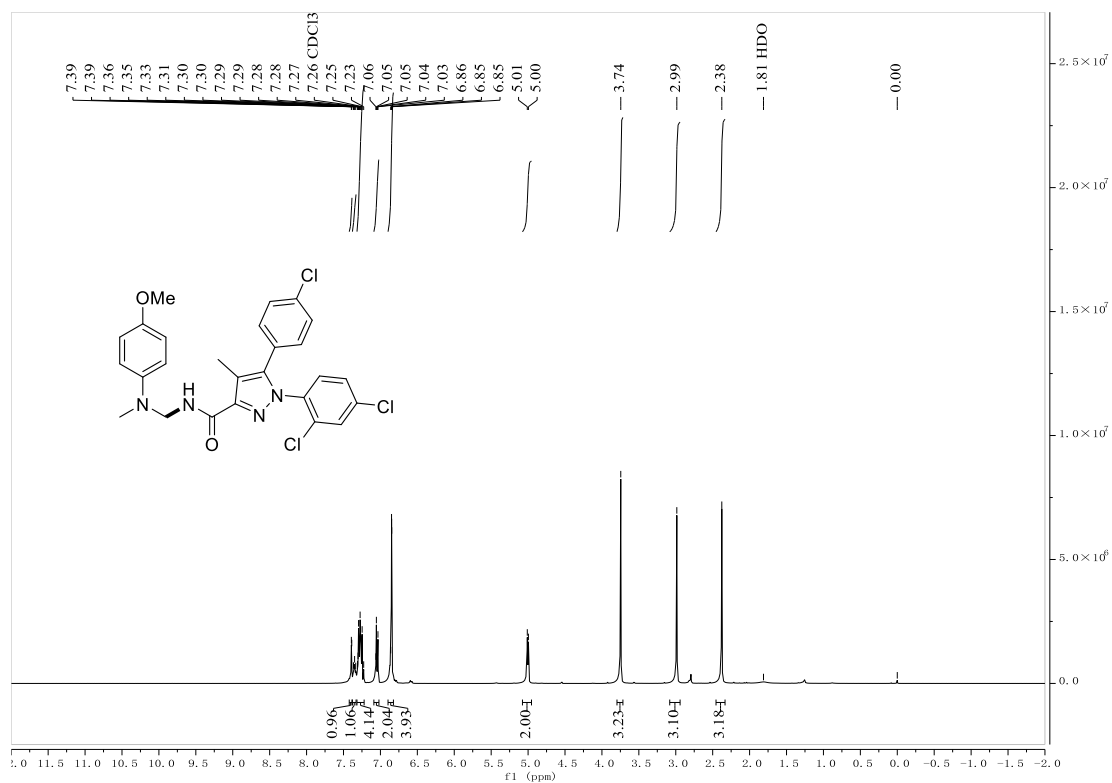
1: TOF MS ES+  
9.47e+006



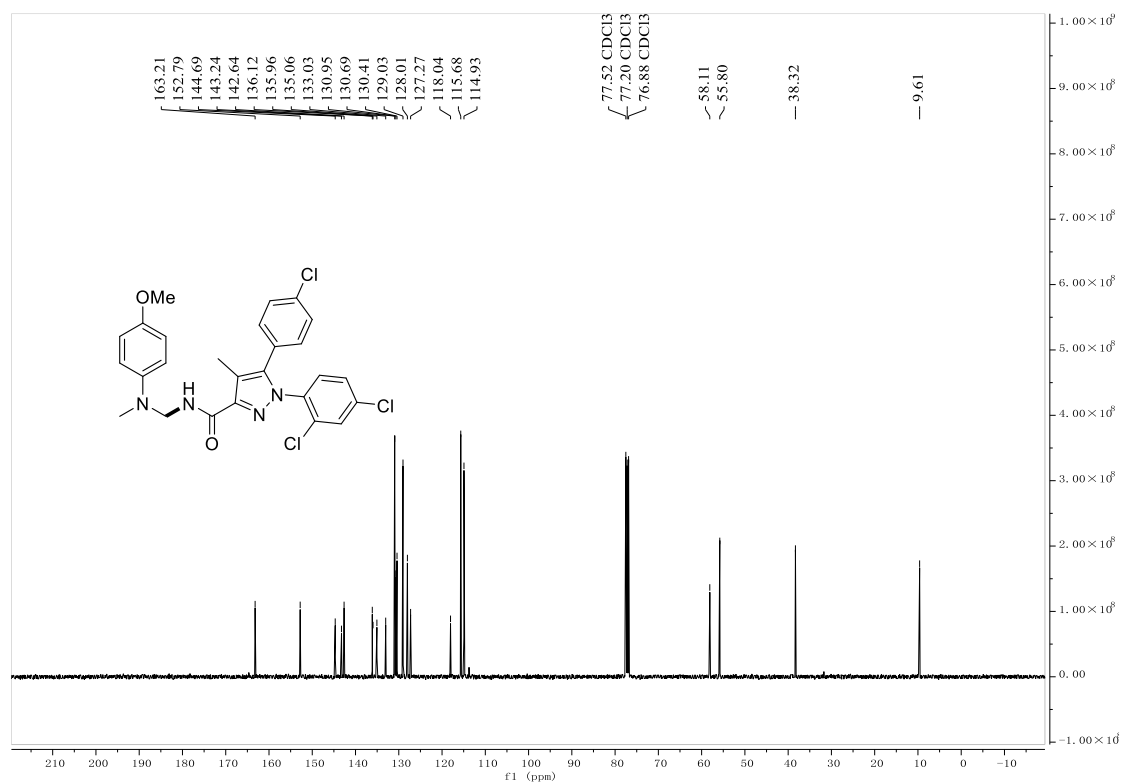
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|------|------|-----|--------|------|----------|------------------------------------------------------------------|
| 307.1419 | 307.1422   | -0.3 | -1.0 | 8.5 | 2083.4 | n/a  | n/a      | C <sub>17</sub> H <sub>20</sub> N <sub>2</sub> O <sub>2</sub> Na |

<sup>1</sup>H NMR of 46



<sup>13</sup>C NMR of 46



## HRMS of 46

Monoisotopic Mass, Even Electron Ions

2260 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

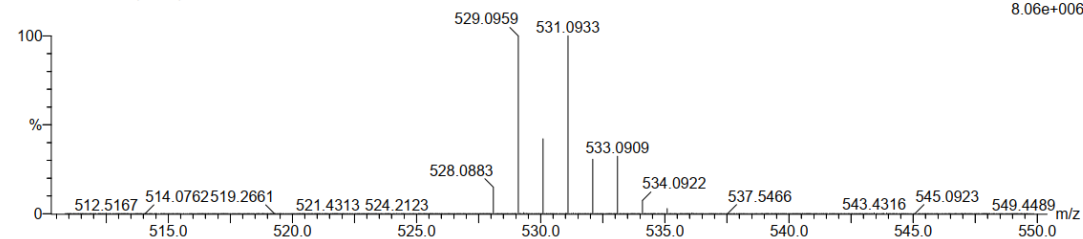
Elements Used:

C: 26-26 H: 24-24 N: 0-100 O: 0-100 Na: 0-7 Cl: 3-3

7

250430-16-37 12 (0.128)

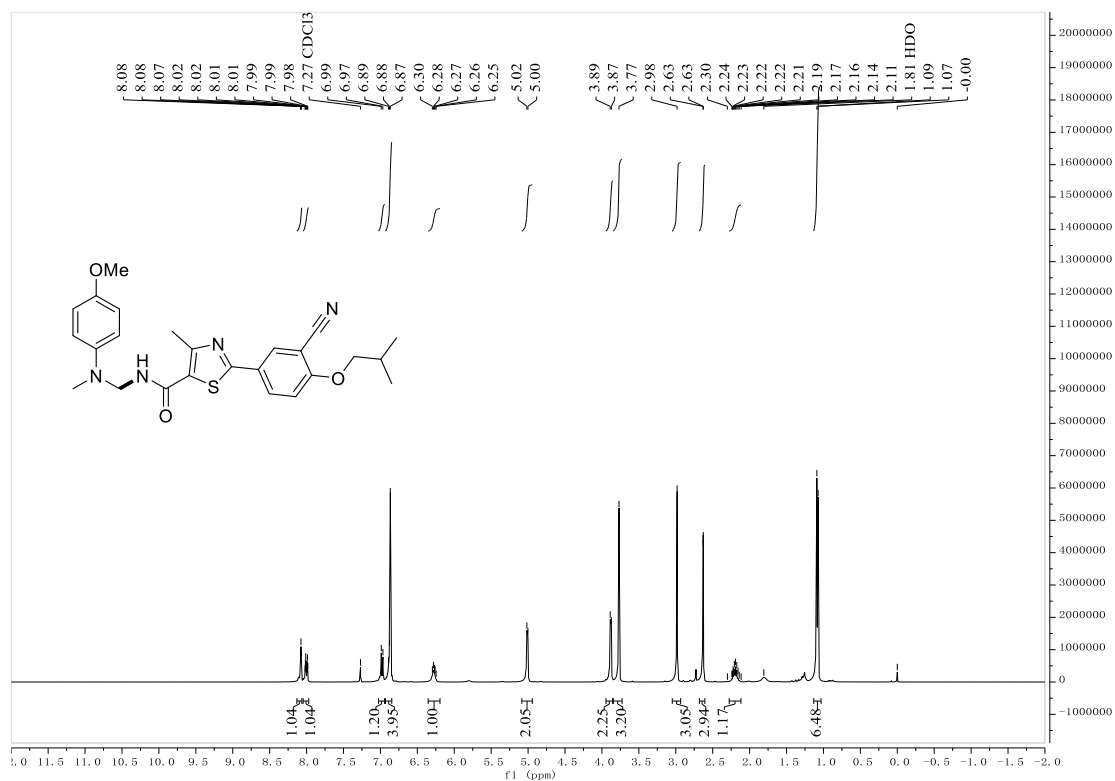
1: TOF MS ES+  
8.06e+006



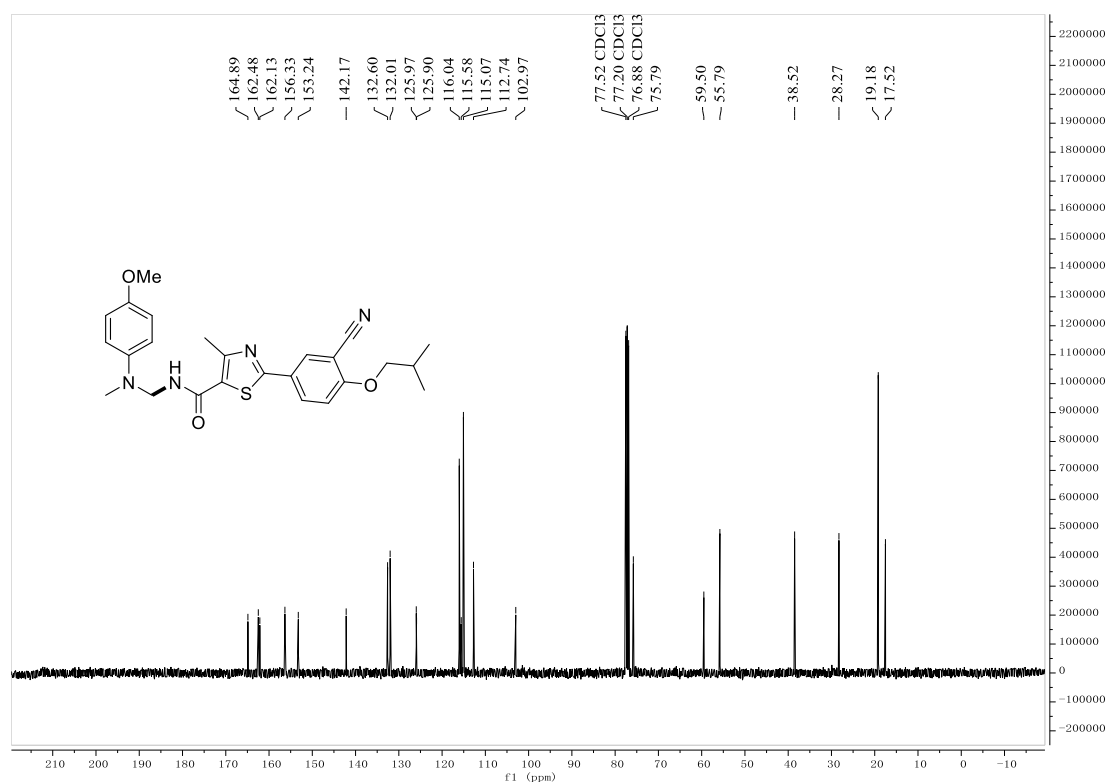
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                                       |
|----------|------------|------|------|------|--------|------|----------|-------------------------------------------------------------------------------|
| 529.0959 | 529.0965   | -0.6 | -1.1 | 15.5 | 1920.9 | n/a  | n/a      | C <sub>26</sub> H <sub>24</sub> N <sub>4</sub> O <sub>2</sub> Cl <sub>3</sub> |

## <sup>1</sup>H NMR of 47



## <sup>13</sup>C NMR of 47



## HRMS of 47

Monoisotopic Mass, Even Electron Ions

6690 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

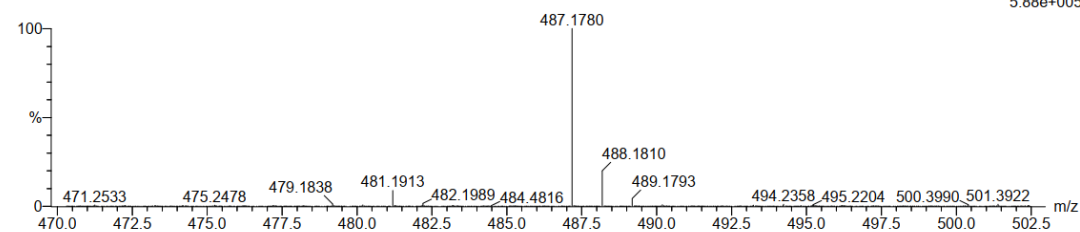
Elements Used:

C: 25-25 H: 28-28 N: 0-100 O: 0-100 Na: 0-7 S: 1-3

7

250430-16-24 9 (0.102)

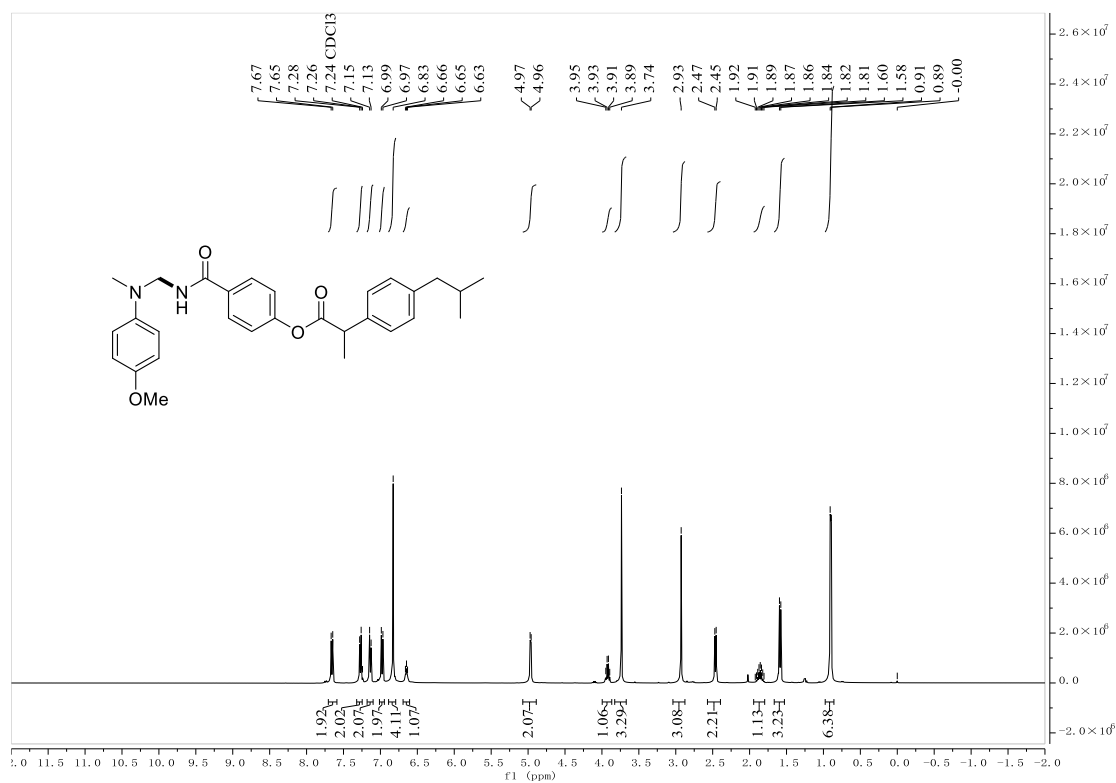
1: TOF MS ES+  
5.88e+005



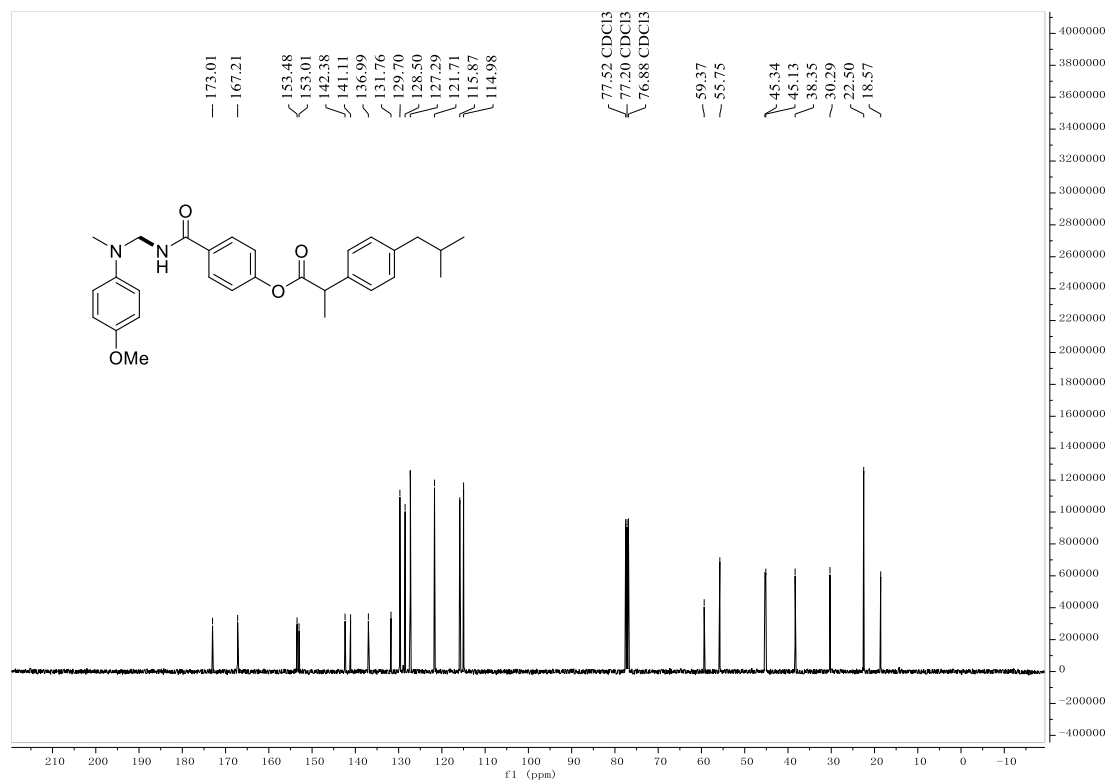
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                           |
|----------|------------|-----|-----|------|--------|------|----------|-------------------------------------------------------------------|
| 487.1780 | 487.1780   | 0.0 | 0.0 | 13.5 | 1469.7 | n/a  | n/a      | C <sub>25</sub> H <sub>28</sub> N <sub>4</sub> O <sub>3</sub> NaS |

# <sup>1</sup>H NMR of 48



# <sup>13</sup>C NMR of 48



## HRMS of 48

Monoisotopic Mass, Even Electron Ions

3206 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

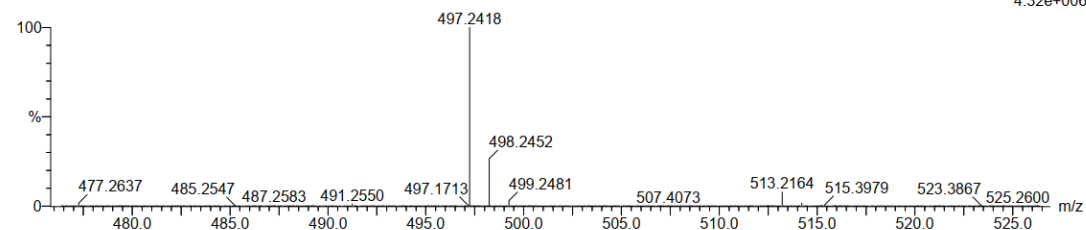
Elements Used:

C: 29-29 H: 34-34 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-43 9 (0.102)

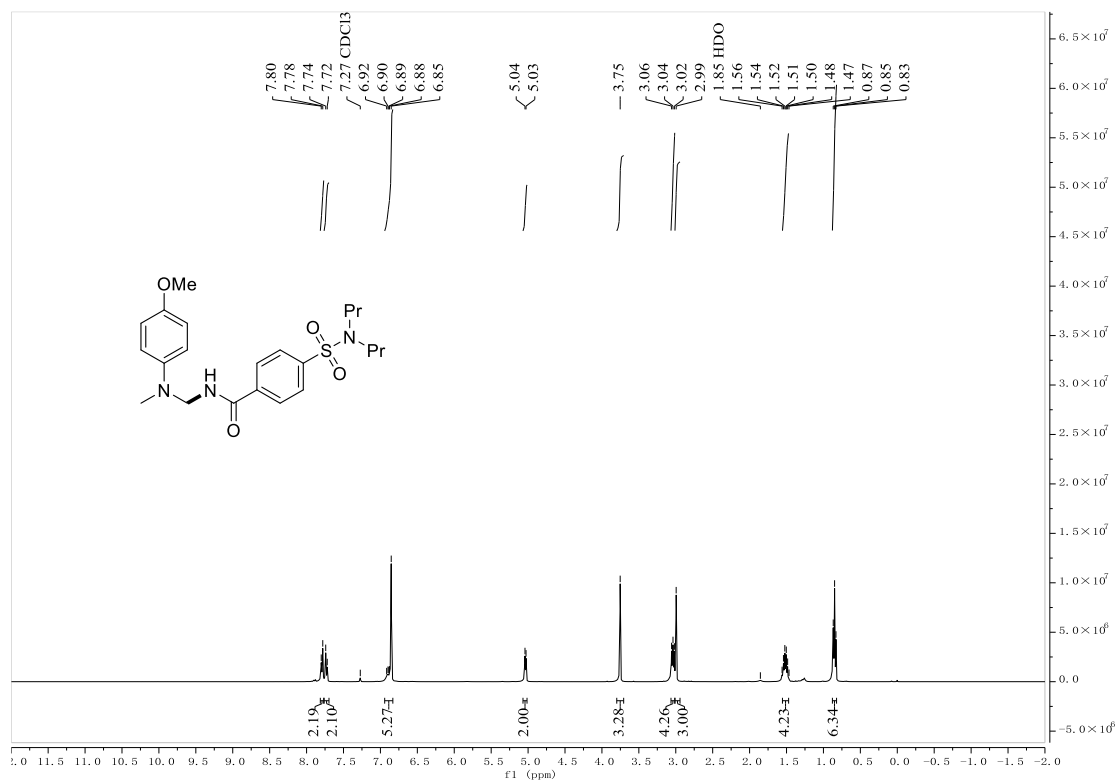
1: TOF MS ES+  
4.32e+006



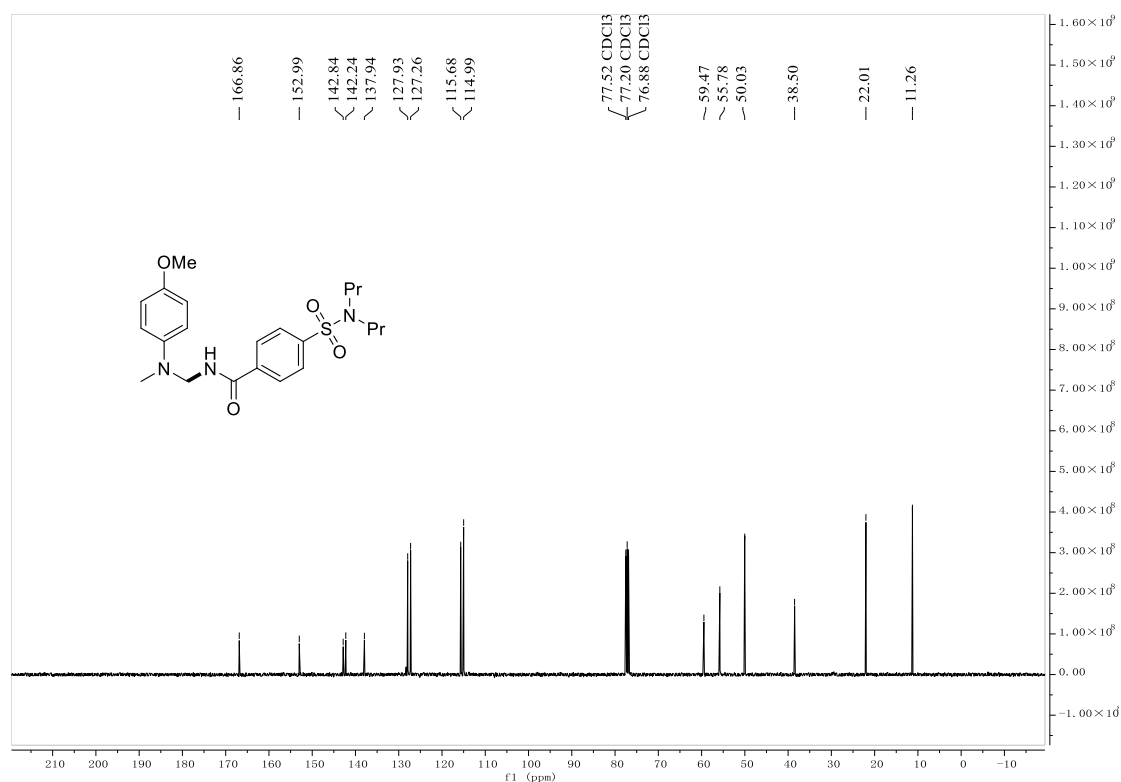
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|-----|-----|------|--------|------|----------|------------------------------------------------------------------|
| 497.2418 | 497.2416   | 0.2 | 0.4 | 13.5 | 1768.7 | n/a  | n/a      | C <sub>29</sub> H <sub>34</sub> N <sub>2</sub> O <sub>4</sub> Na |

## <sup>1</sup>H NMR of 49



## <sup>13</sup>C NMR of **49**



## HRMS of **49**

Monoisotopic Mass, Even Electron Ions

5532 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

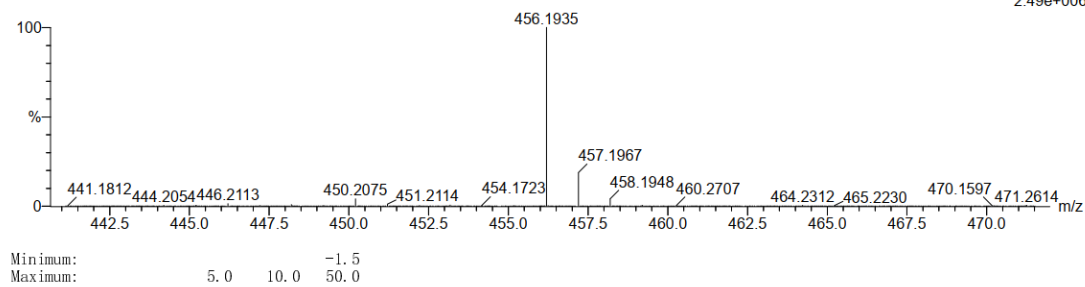
Elements Used:

C: 22-22 H: 31-31 N: 0-100 O: 0-100 Na: 0-7 S: 1-3

7

250430-16-25 8 (0.094)

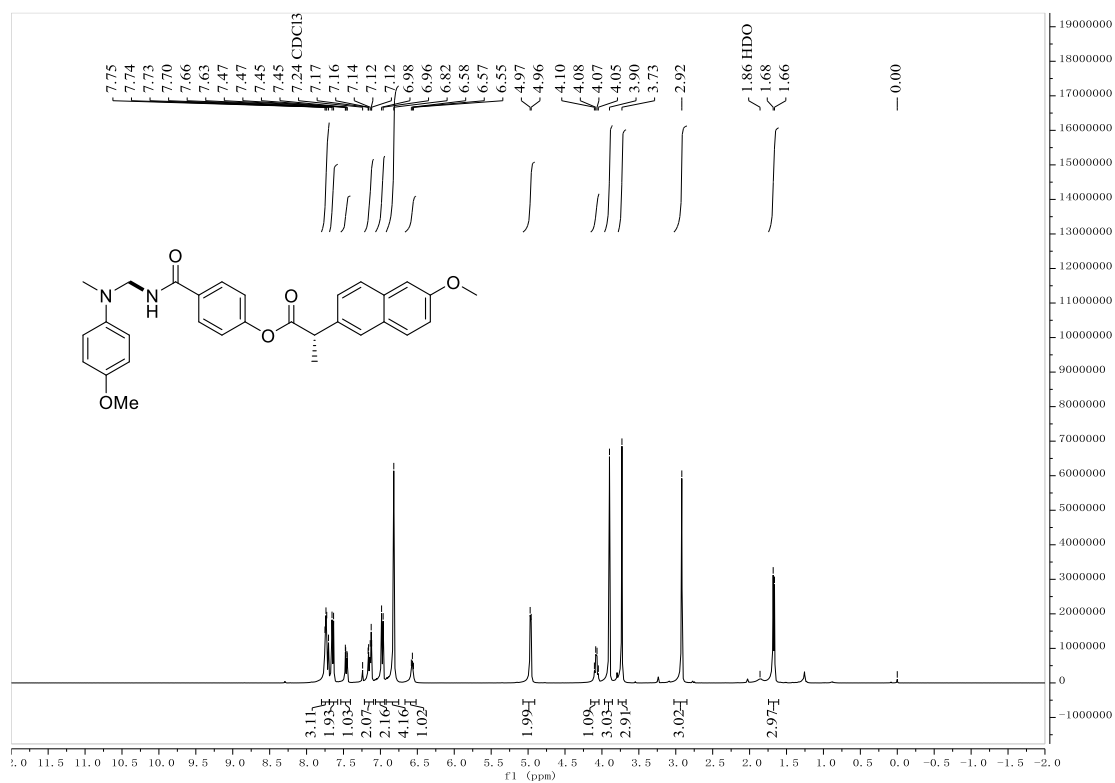
1: TOF MS ES+  
2.49e+006



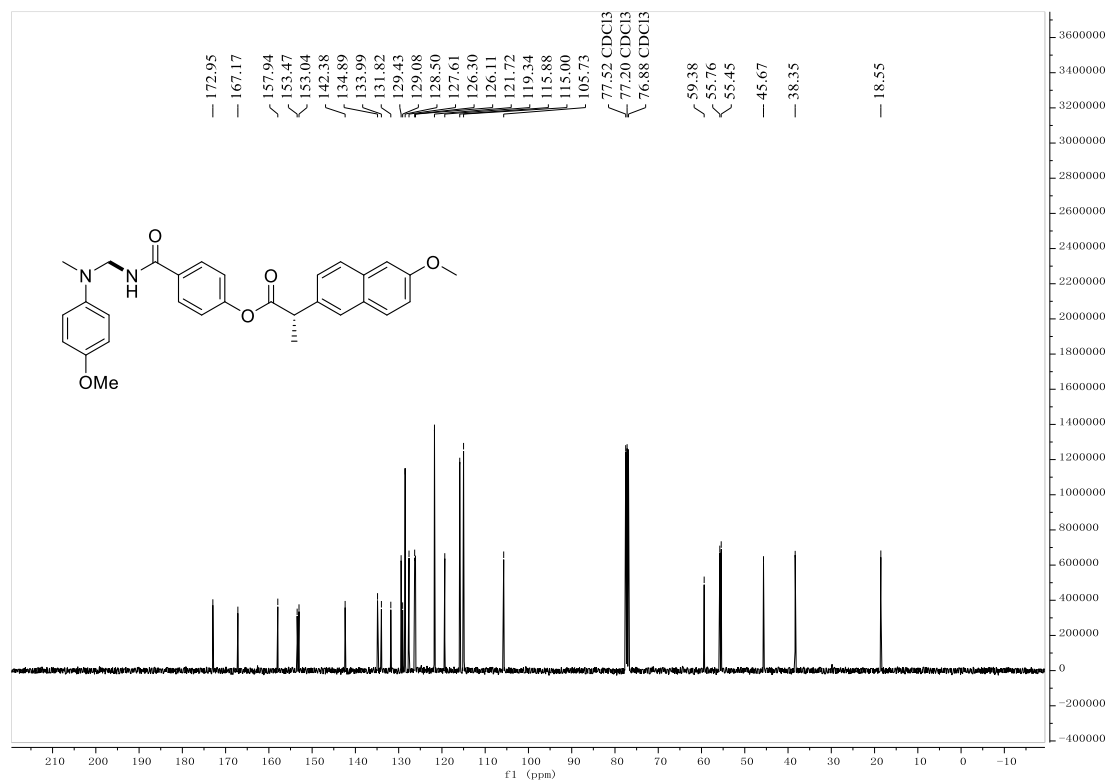
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT  | Norm | Conf (%) | Formula                                                            |
|----------|------------|-----|-----|-----|--------|------|----------|--------------------------------------------------------------------|
| 456.1935 | 456.1933   | 0.2 | 0.4 | 8.5 | 1470.8 | n/a  | n/a      | C <sub>22</sub> H <sub>31</sub> N <sub>3</sub> O <sub>4</sub> Na S |

# <sup>1</sup>H NMR of 50



# <sup>13</sup>C NMR of 50



## HRMS of 50

Monoisotopic Mass, Even Electron Ions

3553 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

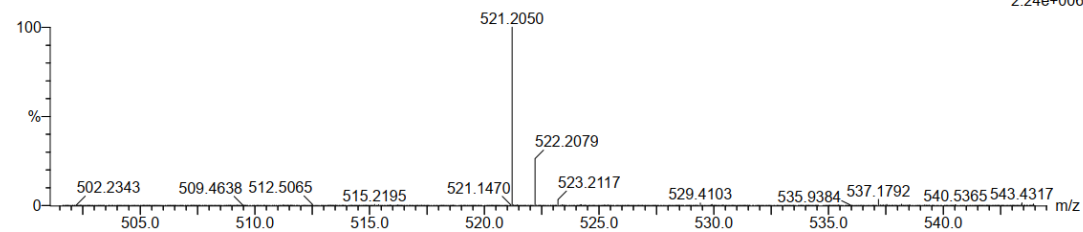
Elements Used:

C: 30-30 H: 30-30 N: 0-100 O: 0-100 Na: 0-7

7

250430-16-38 11 (0.119)

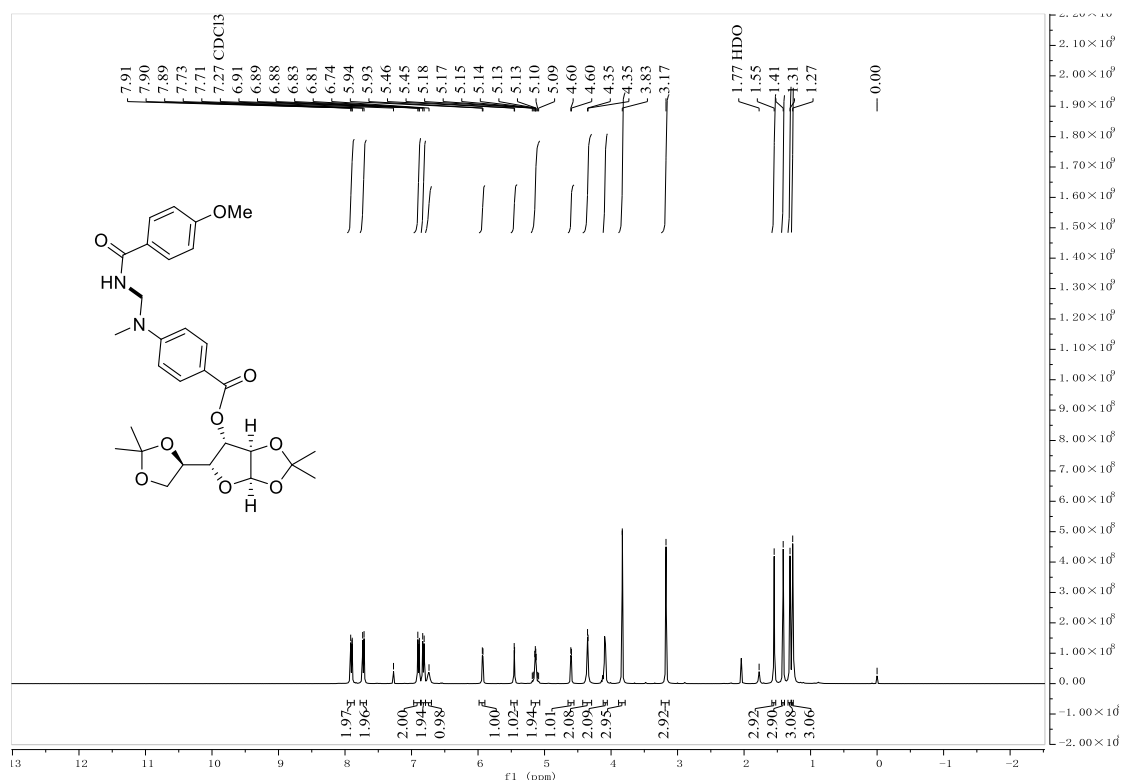
1: TOF MS ES+  
2.24e+006



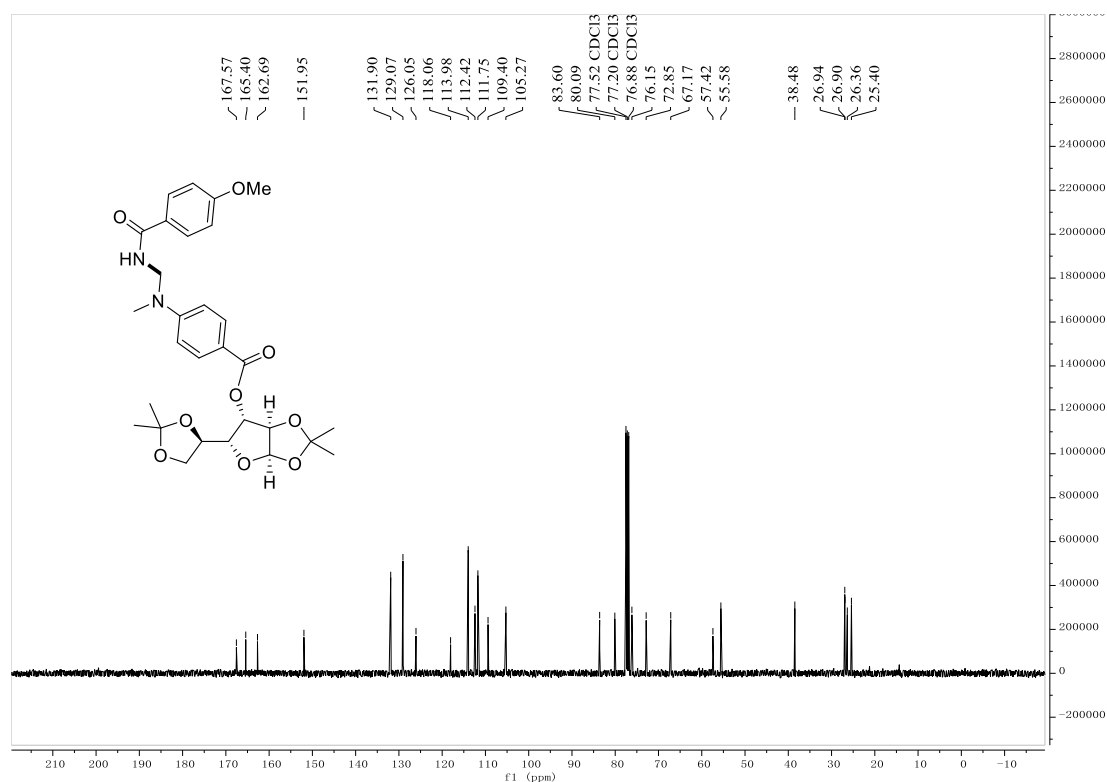
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE  | i-FIT  | Norm | Conf (%) | Formula          |
|----------|------------|------|------|------|--------|------|----------|------------------|
| 521.2050 | 521.2052   | -0.2 | -0.4 | 16.5 | 1544.8 | n/a  | n/a      | C30 H30 N2 O5 Na |

## <sup>1</sup>H NMR of 51



## <sup>13</sup>C NMR of 51



## HRMS of 51

Monoisotopic Mass, Even Electron Ions

4317 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

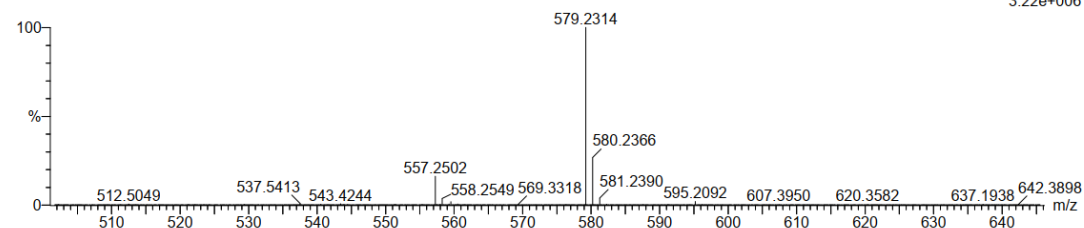
Elements Used:

C: 29-29 H: 36-36 N: 0-100 O: 0-100 Na: 0-7

41

250430-16.9 12 (0.128)

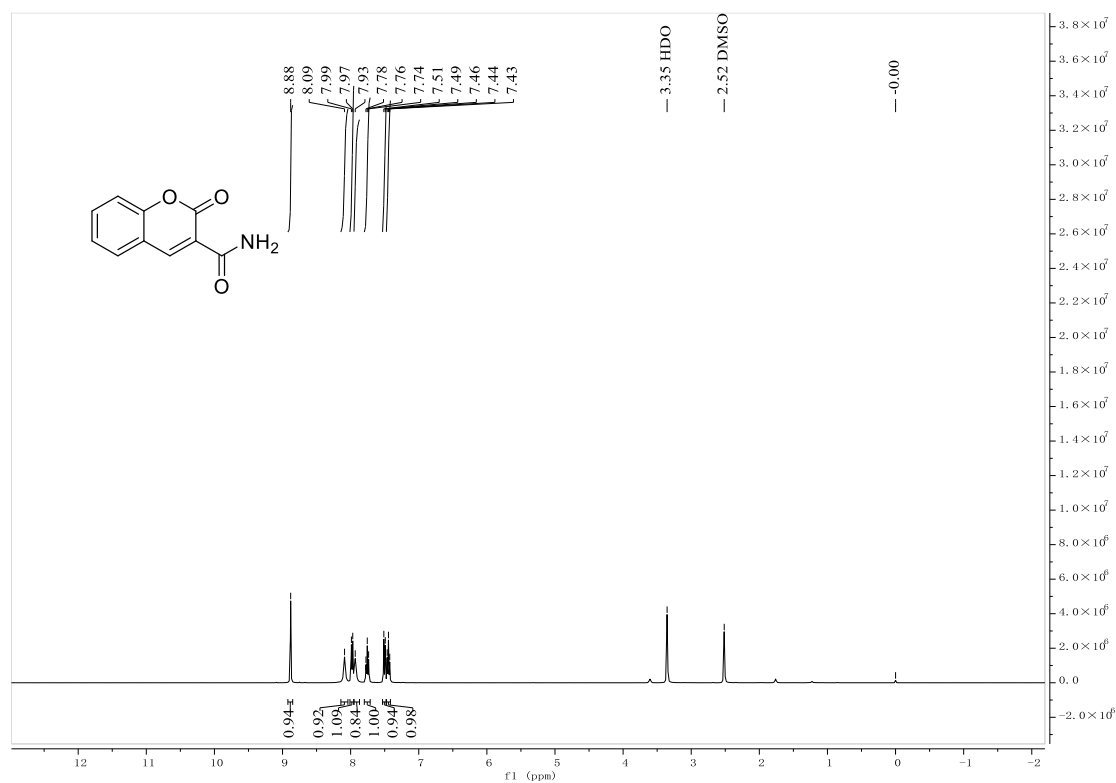
1: TOF MS ES+  
3.22e+006



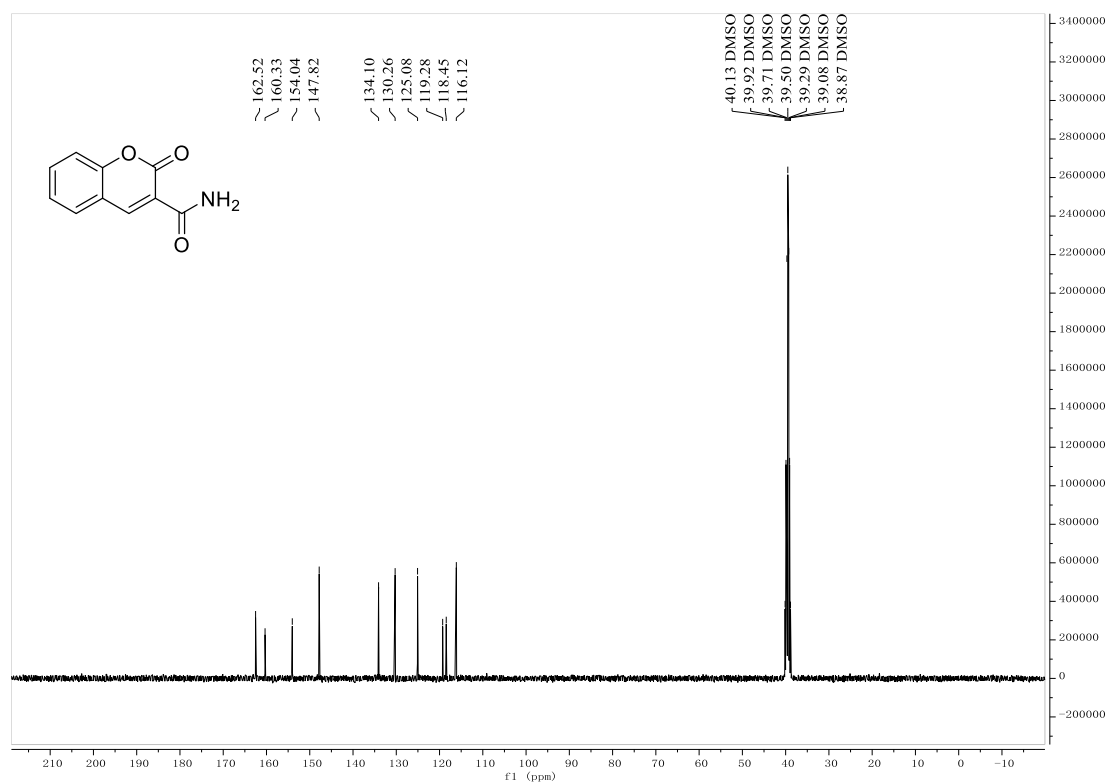
Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE  | i-FIT  | Norm | Conf (%) | Formula                                                          |
|----------|------------|------|------|------|--------|------|----------|------------------------------------------------------------------|
| 579.2314 | 579.2319   | -0.5 | -0.9 | 12.5 | 1546.6 | n/a  | n/a      | C <sub>29</sub> H <sub>36</sub> N <sub>2</sub> O <sub>9</sub> Na |

**<sup>1</sup>H NMR of 2-oxo-2H-chromene-3-carboxamide**



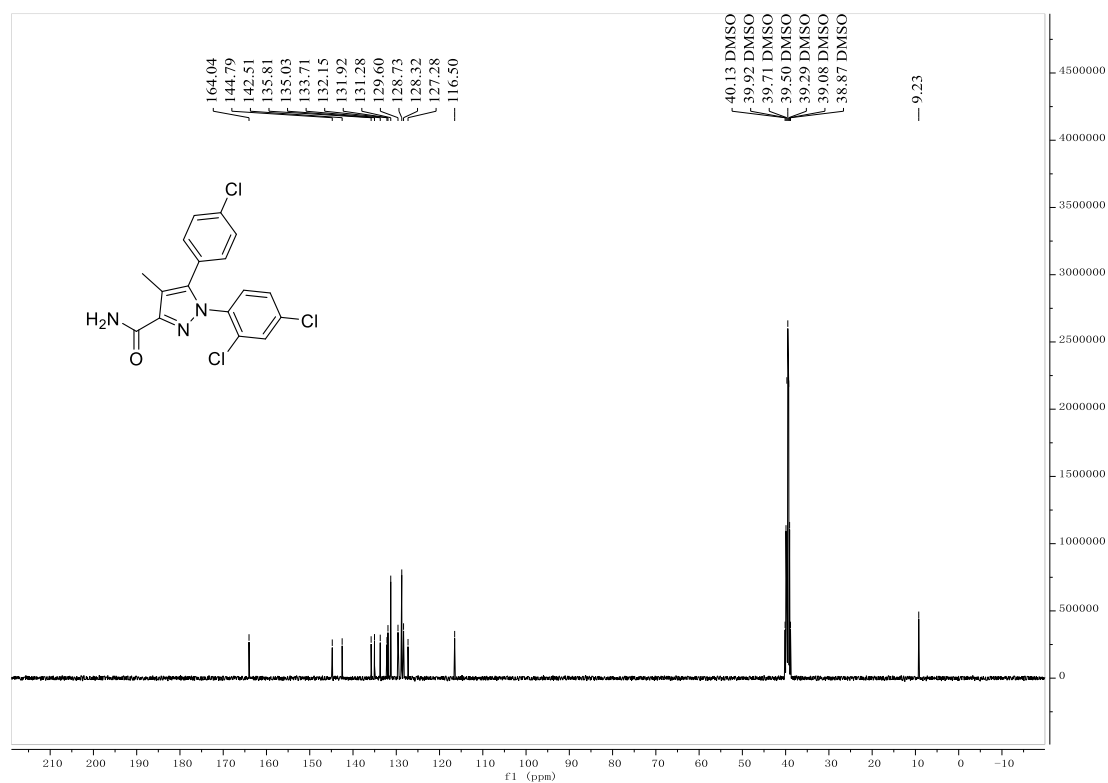
**<sup>13</sup>C NMR of 2-oxo-2H-chromene-3-carboxamide**



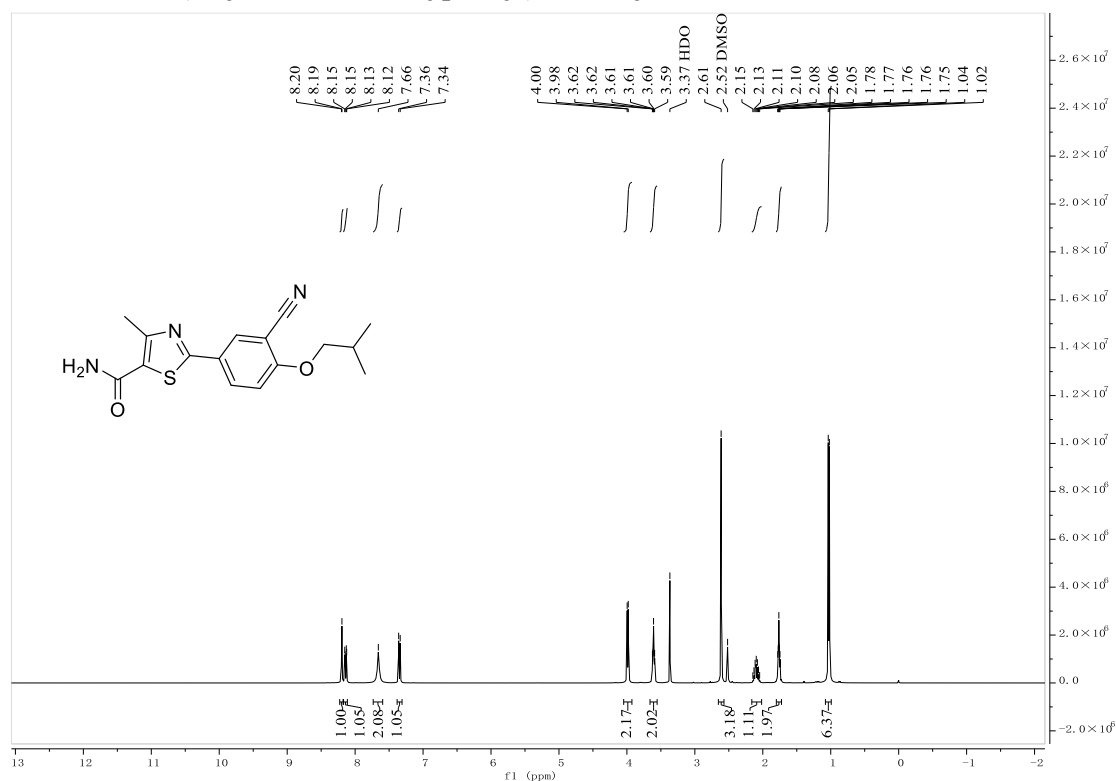
**<sup>1</sup>H NMR of 5-(4-chlorophenyl)-1-(2,4-dichlorophenyl)-4-methyl-1H-pyrazole-3-carboxamide**



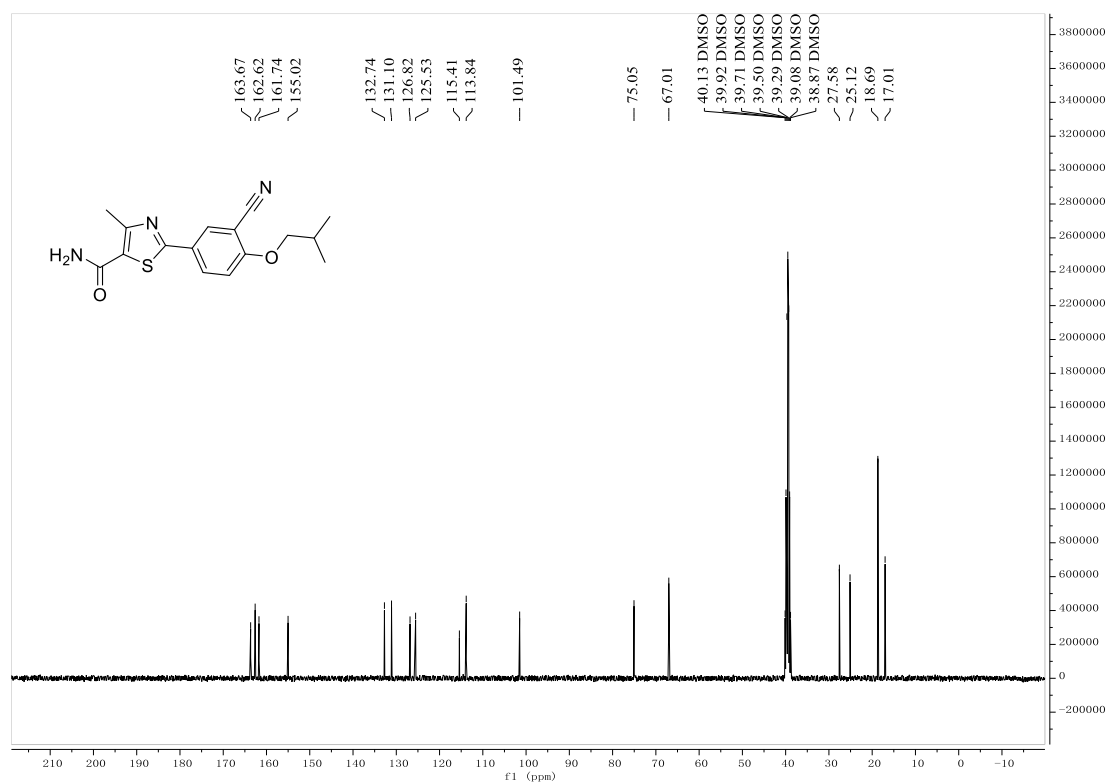
**<sup>13</sup>C NMR of 5-(4-chlorophenyl)-1-(2,4-dichlorophenyl)-4-methyl-1H-pyrazole-3-carboxamide**



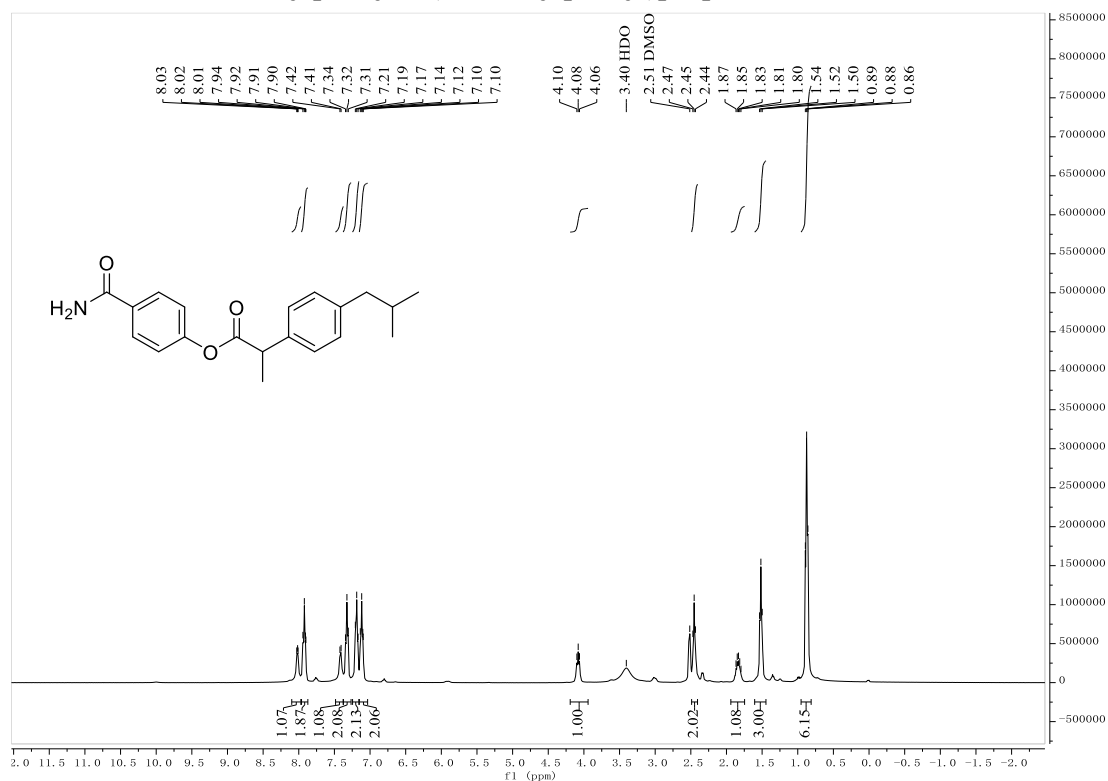
<sup>1</sup>H NMR of 2-(3-cyano-4-isobutoxyphenyl)-4-methylthiazole-5-carboxamide



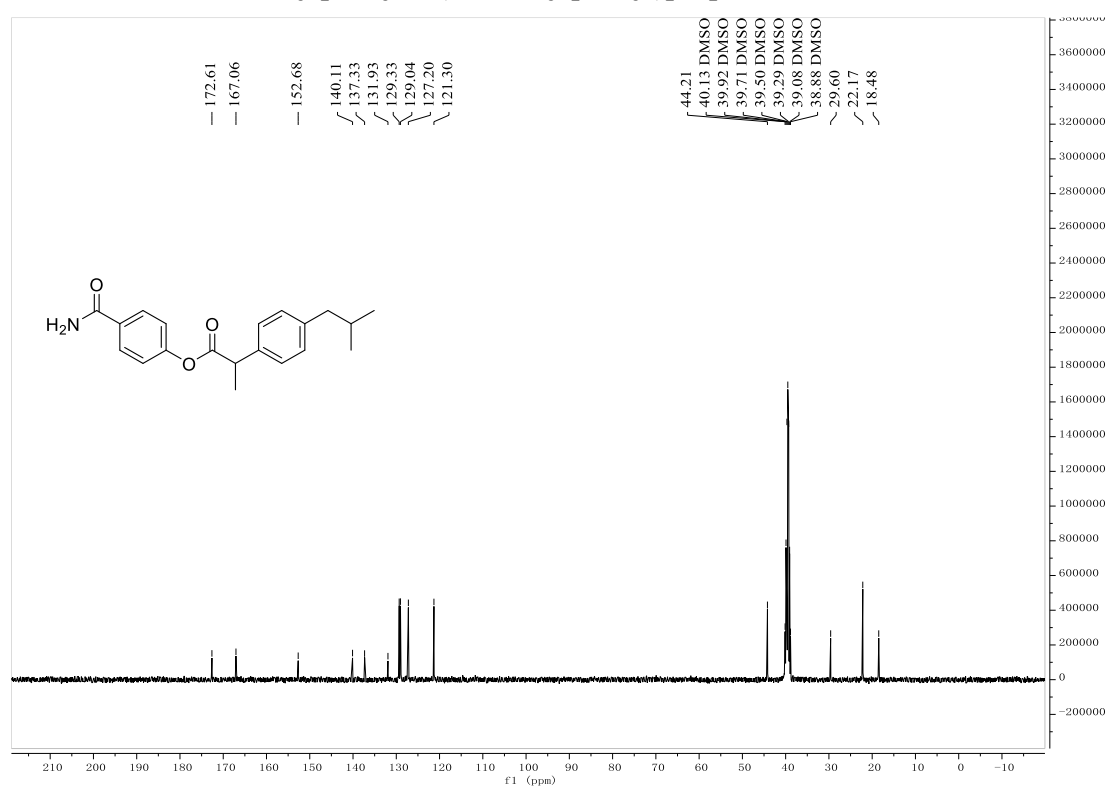
<sup>13</sup>C NMR of 2-(3-cyano-4-isobutoxyphenyl)-4-methylthiazole-5-carboxamide



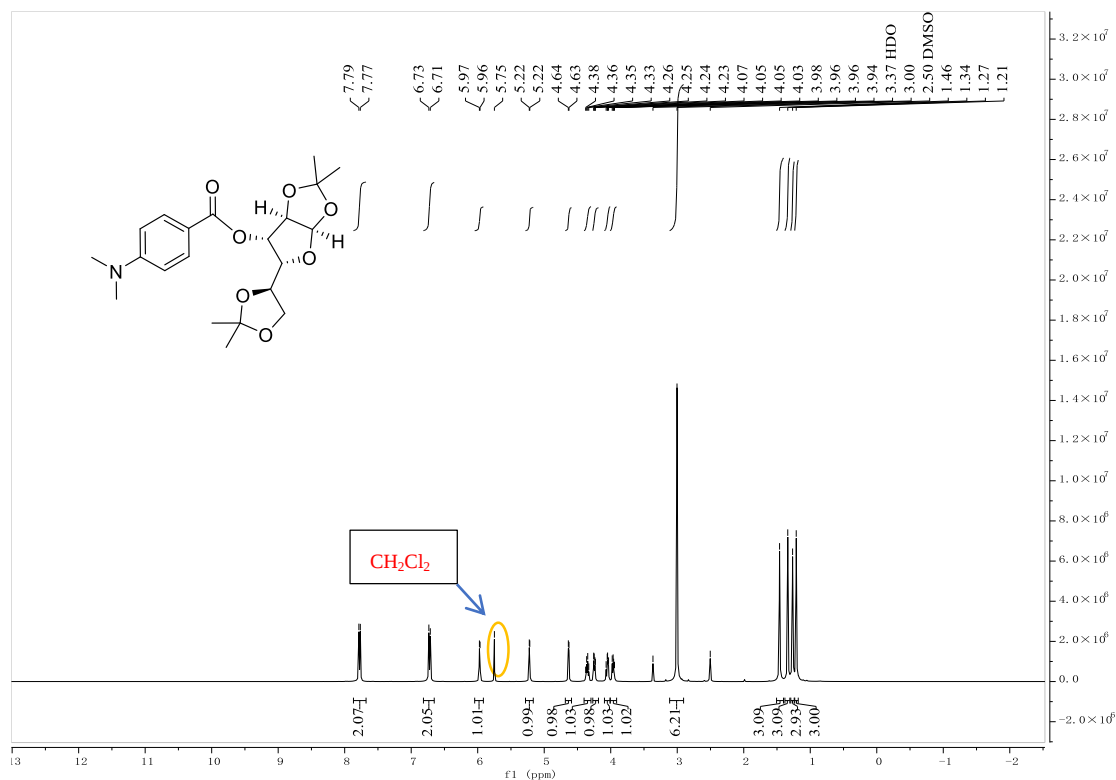
# <sup>1</sup>H NMR of 4-carbamoylphenyl 2-(4-isobutylphenyl)propanoate



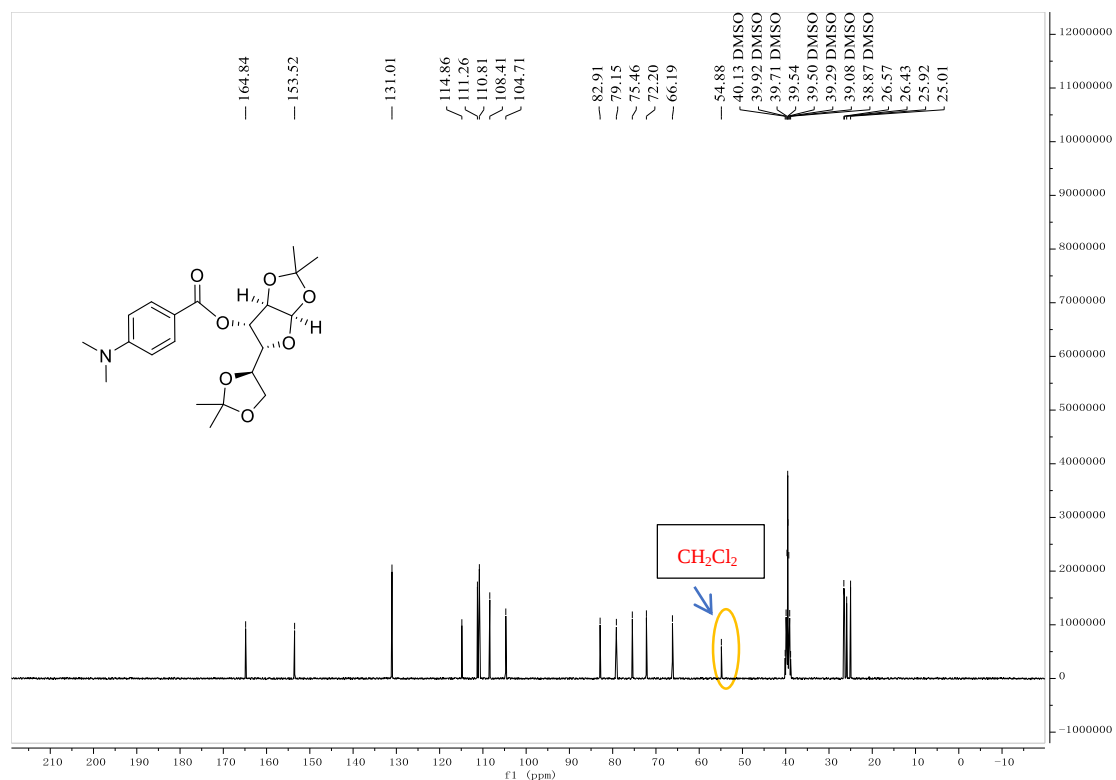
# <sup>13</sup>C NMR of 4-carbamoylphenyl 2-(4-isobutylphenyl)propanoate



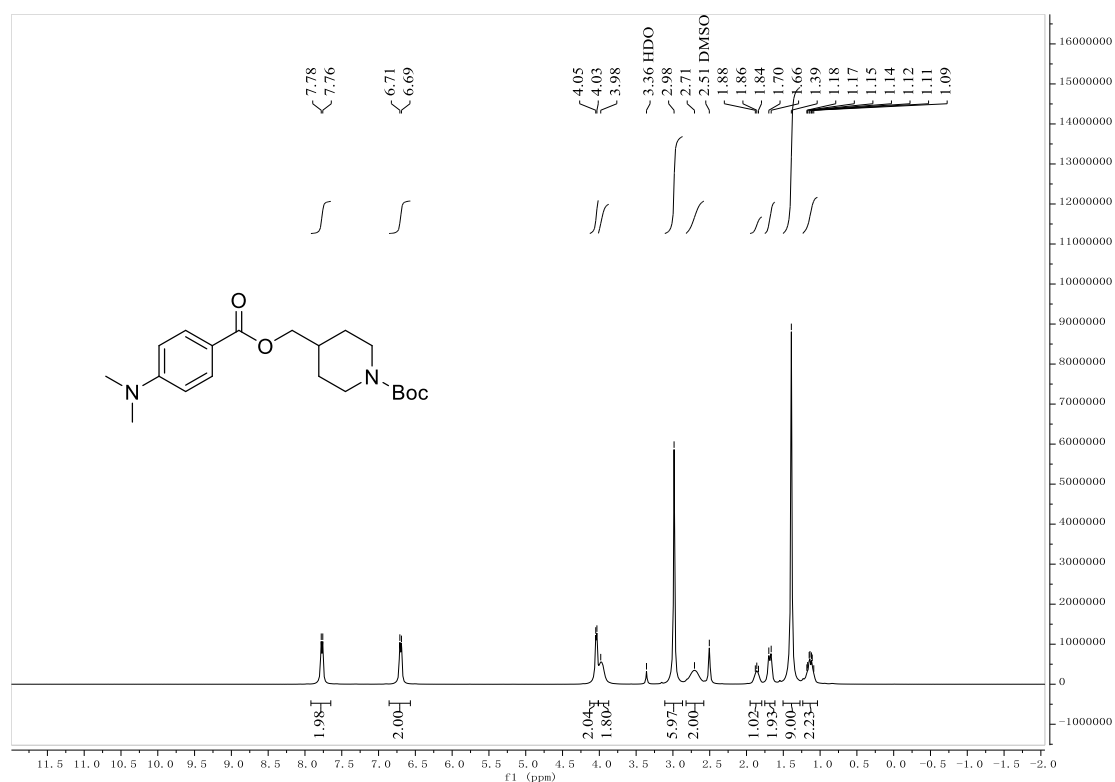
**<sup>1</sup>H NMR of (3aR,5R,6S,6aR)-5-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-yl 4-(dimethylamino)benzoate**



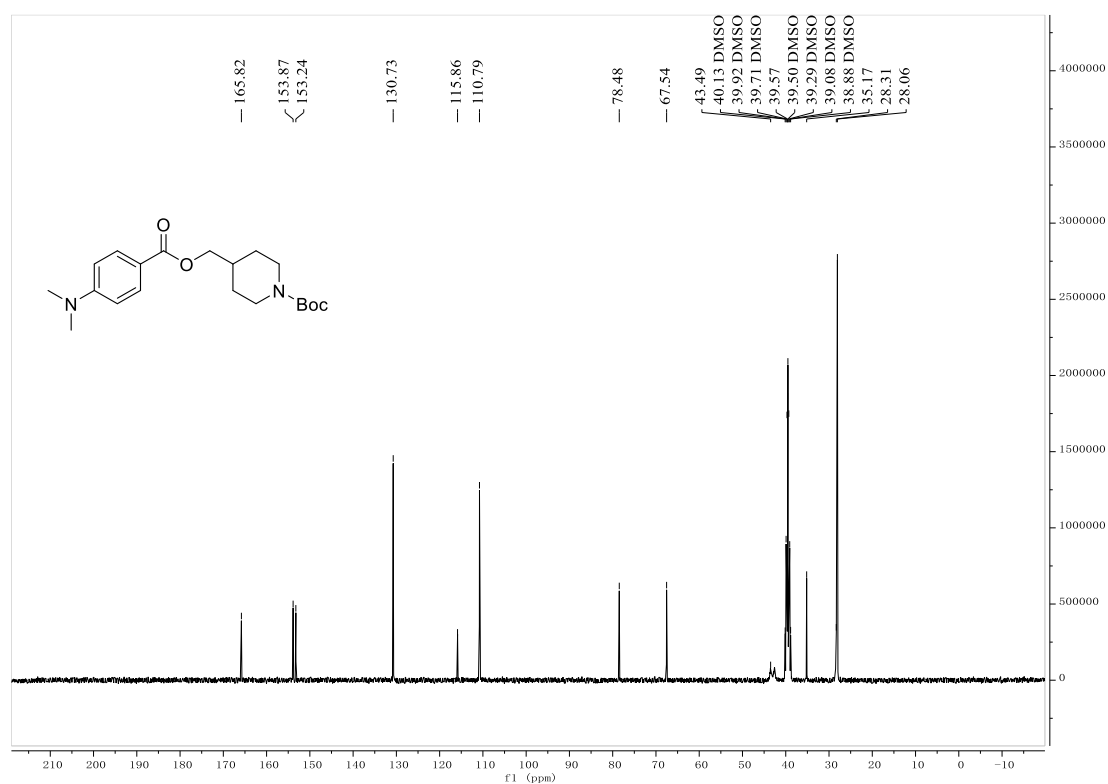
**<sup>13</sup>C NMR of (3aR,5R,6S,6aR)-5-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-yl 4-(dimethylamino)benzoate**



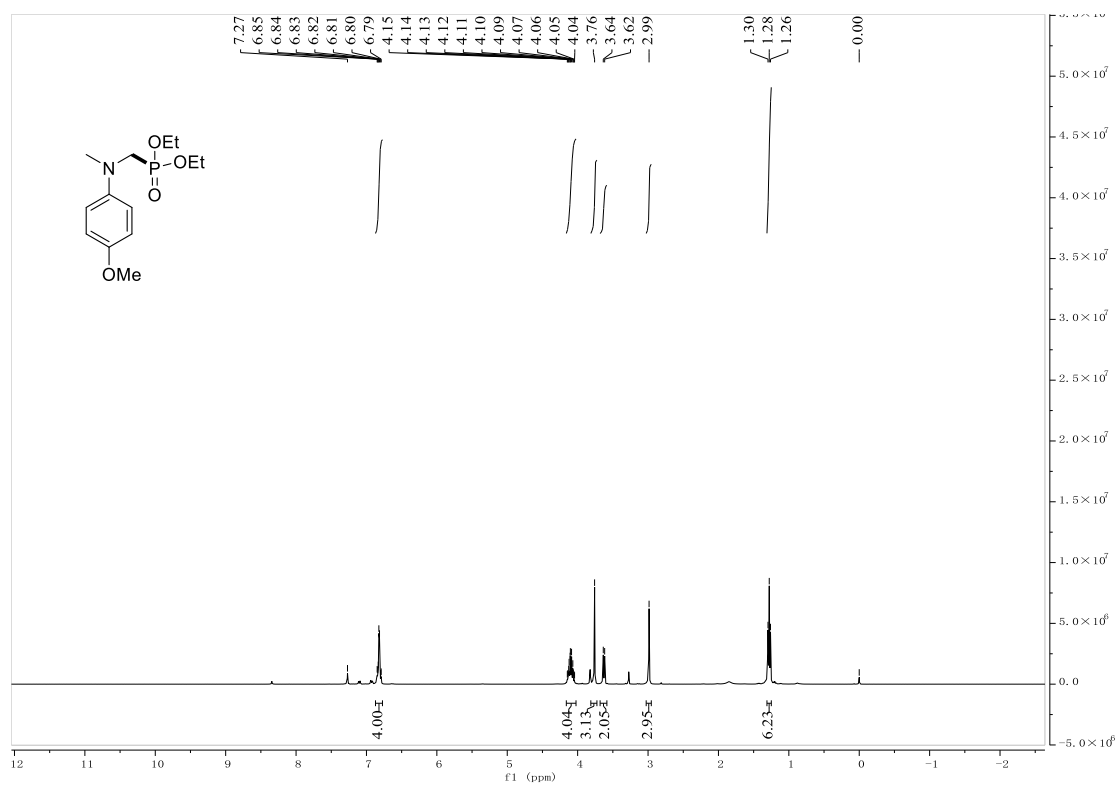
<sup>1</sup>H NMR of *tert*-butyl 4-(((4-(dimethylamino)benzoyl)oxy)methyl)piperidine-1-carboxylate



<sup>13</sup>C NMR of *tert*-butyl 4-(((4-(dimethylamino)benzoyl)oxy)methyl)piperidine-1-carboxylate



# <sup>1</sup>H NMR of 52



# <sup>13</sup>C NMR of 52

