

## **Supporting Information**

### **Synthesis and Cross-coupling of Sulfonamidomethyltrifluoroborates**

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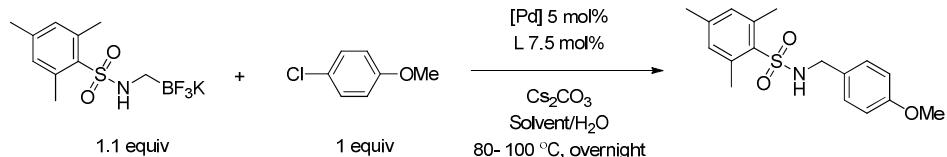
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• Optimization:



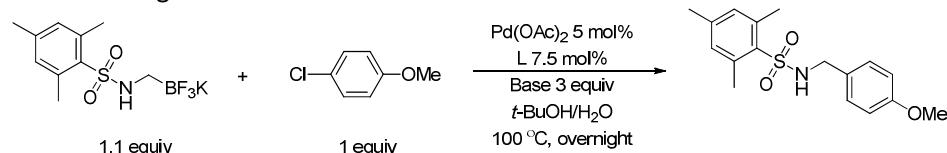
✓ Solvent:

entry	solvent	T °C	[Pd]	L	Conversion
1	CPME/H <sub>2</sub> O 10:1	85°C	Pd(OAc) <sub>2</sub>	XPhos	9%
2	THF/H <sub>2</sub> O 3:1	80°C	Pd(OAc) <sub>2</sub>	XPhos	43%
3	t-BuOH/ H <sub>2</sub> O 1:1	100°C	<b>Pd(OAc)<sub>2</sub></b>	<b>XPhos</b>	<b>63%</b>

✓ Ligands:

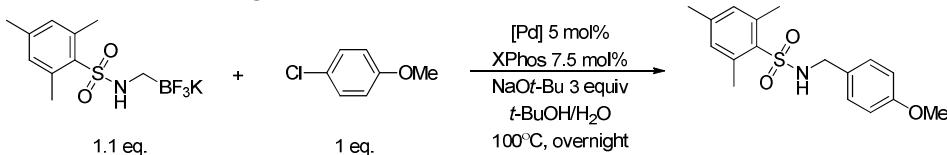
entry	ligand	conversion
1	<b>XPhos</b>	<b>63%</b>
2	DavePhos	25%
3	SPhos	50%
4	<b>RuPhos</b>	<b>61%</b>
5	PdCl <sub>2</sub> dppf	0%

✓ Screening of Bases:



entry	base	conversion (yield)	ligand
1	Cs <sub>2</sub> CO <sub>3</sub>	63%	XPhos
2	K <sub>2</sub> CO <sub>3</sub>	63%	XPhos
3	K <sub>3</sub> PO <sub>4</sub>	73%	XPhos
4	NaOt-Bu	100% (70%)	XPhos

✓ Screening of Pd sources:

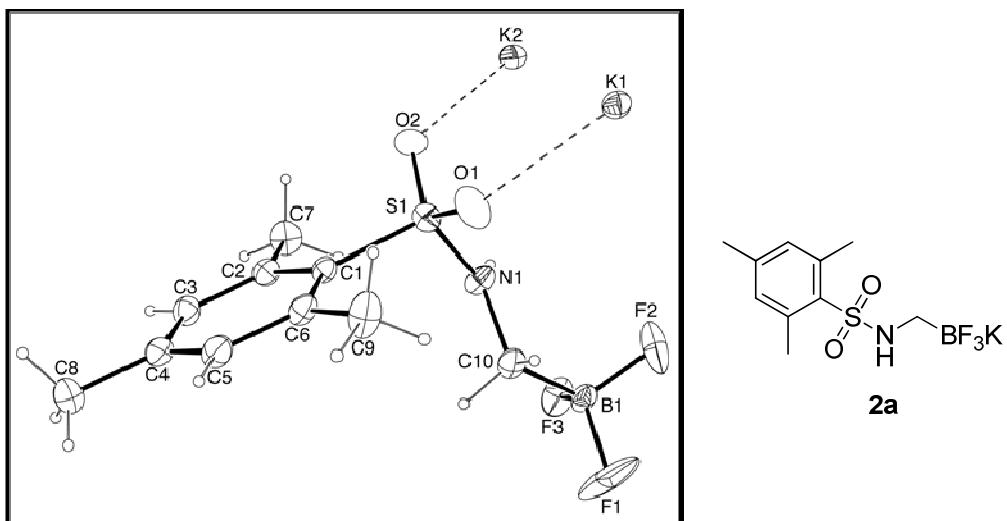


entry	Pd source	conversion (yield)
1	Pd(COD) <sub>2</sub> Cl <sub>2</sub>	100% (63%)
2	Pd(nbn) <sub>2</sub> Cl <sub>2</sub>	44%
3	Pd(bzn) <sub>2</sub> Cl <sub>2</sub>	100% (59%)

4	Pd <sub>2</sub> dba <sub>3</sub>	78% (64%)*
5	Pd(OAc) <sub>2</sub>	100% (77%)
6	Pd(MeCN) <sub>2</sub> Cl <sub>2</sub>	<b>100% (79%)*</b>

\* 1.2 eq. of trifluoroborate used

- ORTEP view of potassium sulfonamidomethyltrifluoroborate 2a:



The ORTEP drawing shows two potassium cations that actually count for two “half” potassium atoms as they lie on a crystallographic 2-fold axis.

## General Considerations

All commercially obtained reagents were used as received. Both solvents and deionized water were degassed with N<sub>2</sub> each time prior to use. Standard benchtop techniques were employed for handling air-sensitive reagents. Melting points (°C) are uncorrected. NMR spectra were recorded on a 500 or 400 MHz spectrometer. <sup>19</sup>F NMR chemical shifts were referenced to external CFCl<sub>3</sub> (0.0 ppm). <sup>11</sup>B NMR spectra were obtained on a spectrometer equipped with the appropriate decoupling accessories. All <sup>11</sup>B NMR chemical shifts were referenced to external BF<sub>3</sub>·OEt<sub>2</sub> (0.0 ppm) with a negative sign indicating an upfield shift. Data are presented as follows: chemical shift (ppm), multiplicity (*s* = singlet, *d* = doublet, *t* = triplet, *m* = multiplet, *br* = broad), coupling constant *J* (Hz) and integration. Analytical thin-layer chromatography (TLC) was performed on TLC silica gel plates (0.25 mm) precoated with a fluorescent indicator. Standard flash chromatography procedures were followed using 32–63 µm silica gel or basic alumina. Visualization was effected with ultraviolet light.

### General procedure for the synthesis of Potassium Sulfonamidomethyltrifluoroborate:

A fresh solution of KHMDS (5 mmol, 1 equiv) in distilled THF (10 mL) at -78 °C under nitrogen was added dropwise to a solution of 2-(chloromethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5 mmol, 1 equiv) in distilled THF (10 mL) at -78 °C over 15 minutes. Once the addition was done, the resulting solution was allowed to warm to rt for 2 h, then cooled to 0 °C. Distilled MeOH (0.205 mL, 1 equiv) was then added and the mixture was stirred for 1 h. The sulfonyl chloride (6 mmol, 1.2 equiv) was then added and the solution is allowed to warm to rt for 2 h. The solvents were then removed under reduced pressure. The resulting crude sulfonamidomethylboronic ester was then taken up in MeOH (10 mL) and the flask was cooled to 0 °C followed by addition of 4.5 M aqueous KHF<sub>2</sub> (4 equiv). After stirring 1 h at room temperature, the solvents were removed under reduced pressure and the desired trifluoroborate was purified by washing the obtained solid successively with distilled water and Et<sub>2</sub>O.



Obtained as a white solid (1.2 g, 75%).

mp > 240 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz):

$\delta$  = 6.99 (*s*, 2H, CH Ar), 4.49 (*br s*, 1H, NH), 2.61 (*s*, 6H, CH<sub>3</sub>x2), 2.28 (*s*, 3H, CH<sub>3</sub>), 1.74 (*br s*, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta$ =141.1, 138.8, 133.8, 131.7, 22.7, 20.7.

<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.38 MHz)

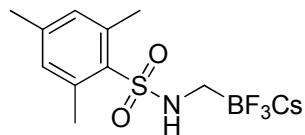
$\delta$ =2.40 (*br s*).

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta$ =-141.9.

IR:  $\nu$  = 3360, 1716, 1652, 1456, 1316, 1162, 1010, 658  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  calcd. For  $\text{C}_{10}\text{H}_{14}\text{BNO}_2\text{F}_3\text{S}$  (M-K) 280.0790, found 280.0787.



Cesium 2,4,6-Trimethylphenylsulfonamidomethyltrifluoroborate **2b**:

Obtained as a white solid (350 mg, 48%).

mp > 240 °C.

$^1\text{H}$  NMR (acetone- $d_6$ , 400 MHz):

$\delta$  = 6.99 (*s*, 2H, CH Ar), 4.49 (*br s*, 1H, NH), 2.61 (*s*, 6H,  $\text{CH}_3 \times 2$ ), 2.28 (*s*, 3H,  $\text{CH}_3$ ), 1.74 (*br s*, 2H,  $\text{CH}_2$ ).

$^{13}\text{C}$  NMR (DMSO- $d_6$ , 125.8 MHz):

$\delta$  = 141.1, 138.8, 133.8, 131.7, 22.7, 20.7.

$^{11}\text{B}$  NMR (acetone- $d_6$ , 128.38 MHz)

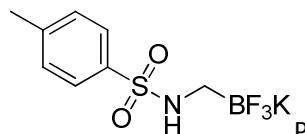
$\delta$  = 3.54 (*q*,  $J$  = 55 Hz).

$^{19}\text{F}$  NMR (DMSO- $d_6$ , 470.84 MHz):

$\delta$  = -141.9.

IR:  $\nu$  = 3366, 1455, 1307, 1284, 1149, 1029, 995, 660  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  calcd. For  $\text{C}_{10}\text{H}_{14}\text{BNO}_2\text{F}_3\text{S}$  (M-K) 280.0790, found 280.0787.



Potassium 4-Methylphenylsulfonamidomethyltrifluoroborate **2c**:

Obtained as a white solid (917 mg, 63%).

mp > 240 °C.

$^1\text{H}$  NMR (acetone- $d_6$ , 500 MHz):

$\delta$  = 7.69 (*d*,  $J$  = 8.0 Hz, 2H, CH, Ar), 7.32 (*d*,  $J$  = 8.0 Hz, 2H, CH, Ar), 4.46 (*br s*, 1H, NH), 2.39 (*s*, 3H, Me), 1.81 (*br s*, 2H,  $\text{CH}_2$ ).

$^{13}\text{C}$  NMR (DMSO- $d_6$ , 125.8 MHz):

$\delta$  = 142.0, 137.4, 129.4, 127.4, 21.3.

$^{11}\text{B}$  NMR (acetone, 128.38 MHz)

$\delta$  = 3.39 (*q*,  $J$  = 52 Hz).

$^{19}\text{F}$  NMR (DMSO- $d_6$ , 470.84 MHz):

$\delta$  = -141.0.

IR:  $\nu$  = 3300, 1652, 1410, 1164, 896, 696  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  calcd. For  $\text{C}_8\text{H}_{10}\text{BNO}_2\text{F}_3\text{S}$  (M-K) 252.0477, found 252.0483.



Triisopropylphenylsulfonamido)methyl)trifluoroborate **2d**:

Obtained as a white solid (162 mg, 40%).

mp > 240 °C.

<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz):

$\delta$  = 7.18 (*s*, 2H), 4.59 (*br s*, 1H, NH), 4.11 (*sept.*,  $J$  = 7.0 Hz, 2H, CH(NMe)<sub>2</sub>), 2.90 (*sept*,  $J$  = 7.0 Hz, 1H, CH(NMe)<sub>2</sub>), 1.61 (*t*,  $J$  = 5.0 Hz, 2H, CH<sub>2</sub>), 1.20 (*d*,  $J$  = 7.0 Hz, 6H, Me), 1.16 (*d*,  $J$  = 7.0 Hz, 12H, Me).

<sup>13</sup>C NMR (DMSO, 125.8 MHz):

$\delta$  = 151.3, 149.7, 132.2, 123.1, 33.3, 28.9, 23.4.

<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.38 MHz)

$\delta$  = 3.0 (*br s*).

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta$  = -141.8.

IR:  $\nu$  = 3335, 2963, 2872, 1600, 1458, 1426, 1298, 1168, 1021, 985, 881, 663 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>16</sub>H<sub>26</sub>BNO<sub>2</sub>F<sub>3</sub>S (M-K) 364.1729, found 364.1732.



Obtained as a white solid (996 mg, 65%).

mp > 240 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz):

$\delta$  = 7.68 (*d*,  $J$  = 8.5 Hz, 2H), 7.06 (*d*,  $J$  = 8.5 Hz, 2H), 5.42 (*t*,  $J$  = 5.0 Hz, 1H, NH), 3.83 (*s*, 3H, Me), 1.56 (*t*,  $J$  = 5.0 Hz, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta$  = 162.1, 132.0, 129.4, 114.1, 55.9.

<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.38 MHz)

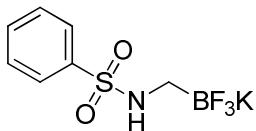
$\delta$  = 2.75 (*br s*).

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta$  = -140.9.

IR:  $\nu$  = 3299, 1602, 1580, 1599, 1303, 1162, 1014, 832 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>8</sub>H<sub>10</sub>BNO<sub>3</sub>F<sub>3</sub>S (M-K) 268.0427, found 268.0463.



Potassium Phenylsulfonamidomethyltrifluoroborate **2f**:

Obtained as a white solid (204 mg, 29%).

mp > 240 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz):

$\delta$  = 7.76 (*d*, *J* = 7.2 Hz, 2H), 7.56 (*m*, 3H), 5.67 (*br s*, 1H, NH), 1.58 (*t*, *J* = 4.8 Hz, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta$  = 140.3, 132.0, 129.0, 127.3.

<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.38 MHz)

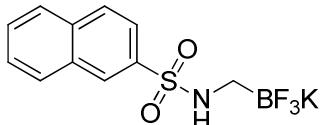
$\delta$  = 3.33 (*q*, *J* = 52 Hz).

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta$  = -141.0.

IR:  $\nu$  = 3301, 1454, 1317, 1164, 1014, 986, 894, 726 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>7</sub>H<sub>8</sub>BNO<sub>2</sub>F<sub>3</sub>S (M-K) 238.0321, found 238.0321.



Potassium 2-Naphthylsulfonamidomethyltrifluoroborate **2g**:

Obtained as a white solid (1.1 g, 67%).

mp > 240 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz):

$\delta$  = 8.42 (*s*, 1H), 8.05 (*m*, 3H), 7.88 (*d*, *J* = 7.6 Hz, 1H), 7.65 (*t*, *J* = 4.0 Hz, 2H), 4.64 (*br s*, 1H, NH), 1.88 (*br s*, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta$  = 137.4, 134.3, 132.0, 129.3, 129.1, 128.6, 128.1, 127.9, 127.6, 123.5.

<sup>11</sup>B NMR (acetone-d<sub>6</sub>, 128.38 MHz)

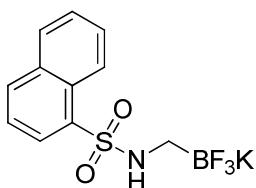
$\delta$  = 3.36 (*q*, *J* = 44.5 Hz).

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta$ =-141.0.

IR:  $\nu$  = 3316, 1652, 1444, 1319, 1074, 674 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>11</sub>H<sub>10</sub>BNO<sub>2</sub>F<sub>3</sub>S (M-K) 288.0477, found 288.0468.



Potassium 1-Naphthylsulfonamidomethyltrifluoroborate **2h**:

Obtained as a white solid (338 mg, 41%).

mp > 240 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 500 MHz):

$\delta$  = 8.69 (*d*, *J* = 8.5 Hz, 1H), 8.15 (*d*, *J* = 8.5 Hz, 1H), 8.05 (*m*, 2H), 7.63 (*m*, 3H), 5.89 (*br s*, 1H, NH), 1.58 (*t*, *J* = 5.0 Hz, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (acetone-d<sub>6</sub>, 125.8 MHz):

$\delta$  = 135.5, 134.2, 133.3, 129.1, 129.0, 127.8, 126.9, 125.6, 124.6.

<sup>11</sup>B NMR (acetone-d<sub>6</sub>, 128.38 MHz)

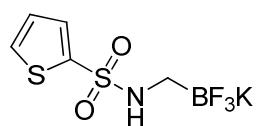
$\delta$  = 2.83 (*br s*).

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta$  = -141.0.

IR:  $\nu$  = 3363, 1652, 1558, 1418, 1313, 1163, 1049, 972, 768 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>11</sub>H<sub>10</sub>BNO<sub>2</sub>F<sub>3</sub>S (M-K) 288.0477, found 288.0487.



Potassium Thiophene-2-sulfonamidomethyltrifluoroborate **2i**:

Obtained as a white solid (532 mg, 38%).

mp > 240 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 500 MHz):

$\delta$  = 7.75 (*d*, *J* = 4.8 Hz, 1H), 7.54 (*d*, *J* = 2.4 Hz, 1H), 7.15 (*t*, *J* = 4.8 Hz, 1H), 4.71 (*br s*, 1H, NH), 1.96 (*br s*, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta$  = 130.9, 130.5, 126.9.

<sup>11</sup>B NMR (acetone, 128.38 MHz)

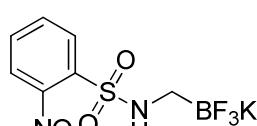
$\delta$  = 2.58 (*q*, *J* = 53.0 Hz).

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta$  = -145.8.

IR:  $\nu$  = 3295, 1647, 1560, 1313, 1163, 1018, 654 cm<sup>-1</sup>

HRMS (ESI) *m/z* calcd. For C<sub>5</sub>H<sub>6</sub>BNO<sub>2</sub>F<sub>3</sub>S<sub>2</sub> (M-K) 243.9885, found 243.9888.



Potassium 2-Nitrophenylsulfonamidomethyltrifluoroborate **2j**:

Obtained as a white solid (174 mg, 54%).

mp > 240 °C.

<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz):

$\delta$  = 7.96 (*m*, 2H), 7.83 (*br s*, 2H), 5.69 (*br s*, 1H, NH), 1.69 (*s*, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta$  = 148.6, 133.9, 132.6, 131.9, 130.7, 124.9.

<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.38 MHz)

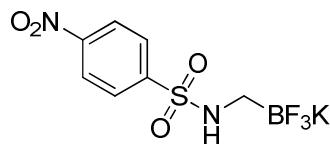
$\delta$  = 2.61 (*br s*).

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta = -141.8$ .

IR:  $\nu = 3359, 1541, 1351, 1327, 1164, 1013, 800, 732 \text{ cm}^{-1}$

HRMS (ESI) *m/z* calcd. For C<sub>7</sub>H<sub>7</sub>BN<sub>2</sub>O<sub>4</sub>F<sub>3</sub>S (M-K) 283.0172, found 283.0173.



Potassium 4-Nitrophenylsulfonamidomethyltrifluoroborate **2k**:

Obtained as a white solid (566 mg, 70%).

mp > 240 °C.

<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz):

$\delta = 8.36 (d, J = 7.0 \text{ Hz}, 2\text{H}), 8.0 (d, J = 7.0 \text{ Hz}, 2\text{H}), 6.33 (br s, 1\text{H}, \text{NH}), 1.60 (br s, 2\text{H}, \text{CH}_2)$ .

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta = 149.5, 146.2, 128.8, 124.4$ .

<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.38 MHz)

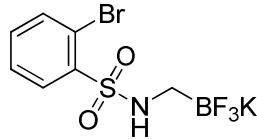
$\delta = 2.95 (\text{br s})$ .

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta = -140.9$ .

IR:  $\nu = 3361, 2360, 1455, 1316, 1161, 1013, 895, 658 \text{ cm}^{-1}$

HRMS (ESI) *m/z* calcd. For C<sub>7</sub>H<sub>7</sub>BN<sub>2</sub>O<sub>4</sub>F<sub>3</sub>S (M-K) 283.0172, found 283.0176.



Potassium 2-Bromophenylsulfonamidomethyltrifluoroborate **2l**:

Obtained as a white solid (941 mg, 53%).

mp > 240 °C.

<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz):

$\delta = 7.95 (d, J = 7.5 \text{ Hz}, 1\text{H}), 7.82 (d, J = 8.5 \text{ Hz}, 1\text{H}), 7.53 (m, 2\text{H}), 5.26 (\text{br s}, 1\text{H}, \text{NH}), 1.56 (t, J = 4.5 \text{ Hz}, 2\text{H}, \text{CH}_2)$ .

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta = 138.5, 135.34, 133.9, 131.6, 128.2, 119.5$ .

<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.38 MHz)

$\delta = 3.00 (\text{br s})$ .

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta = -142.1$ .

IR:  $\nu = 3332, 1684, 1627, 1449, 1310, 1164, 1006, 765 \text{ cm}^{-1}$ .

HRMS (ESI) *m/z* calcd. For C<sub>7</sub>H<sub>7</sub>BNO<sub>2</sub>F<sub>3</sub>S Br (M-K) 315.9426, found 315.9434.



Obtained as a white solid (712 mg, 40%).

mp > 240 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 500 MHz):

$\delta$  = 7.75 (*d*, *J* = 8.5 Hz, 2H), 7.68 (*d*, *J* = 8.5 Hz, 2H), 5.94 (*t*, *J* = 5.0 Hz, 1H, NH), 1.56 (*t*, *J* = 5.5 Hz, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta$  = 139.7, 132.0, 129.4, 125.7.

<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.38 MHz)

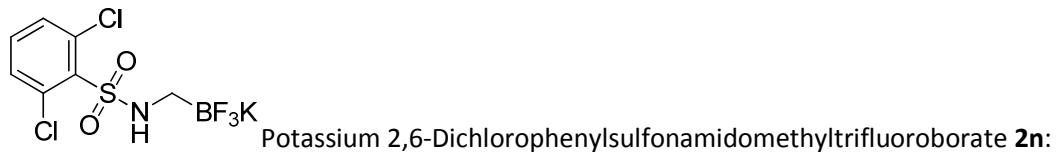
$\delta$  = 3.33 (*br s*).

<sup>19</sup>F NMR (acetone-d<sub>6</sub>, 470.84 MHz):

$\delta$  = -140.8.

IR:  $\nu$  = 3266, 1573, 1416, 1312, 1165, 1028, 1006, 784 cm<sup>-1</sup>

HRMS (ESI) *m/z* calcd. For C<sub>7</sub>H<sub>7</sub>BNO<sub>2</sub>F<sub>3</sub>SBr (M-K) 315.9426, found 315.9432.



Obtained as a white solid (1.07g, 62%).

mp > 240 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 500 MHz):

$\delta$  = 7.63 (*d*, *J* = 7.5 Hz, 2H), 7.53 (*t*, *J* = 7.5Hz, 1H), 5.40 (*br s*, 1H, NH), 1.61 (*t*, *J* = 5.0 Hz, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta$  = 134.5, 134.3, 133.5, 131.9.

<sup>11</sup>B NMR (acetone-d<sub>6</sub>, 128.38 MHz)

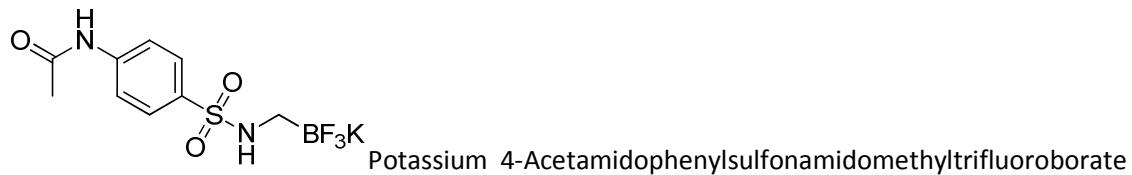
$\delta$  = 3.62 (*m*).

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta$  = -142.3.

IR:  $\nu$  = 3361, 1558, 1424, 1324, 1181, 1025, 780 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>7</sub>H<sub>6</sub>BNO<sub>2</sub>F<sub>3</sub>S Cl<sub>2</sub> (M-K) 305.9541, found 305.9545.



**2o:**

Obtained as a white solid (592 mg, 30%).

mp > 240 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz):

$\delta$  = 9.41 (*br s*, 1H, NH), 7.79 (*d*,  $J$  = 8.5 Hz, 2H), 7.74 (*d*,  $J$  = 8.5 Hz, 2H), 4.46 (*br s*, 1H, NH), 2.12 (*s*, 3H, Me), 1.84 (*br s*, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta$  = 169.2, 142.5, 134.0, 128.4, 118.6, 24.5.

<sup>11</sup>B NMR (acetone-d<sub>6</sub>, 128.38 MHz)

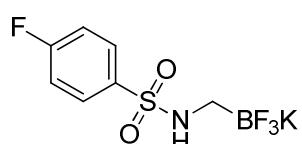
$\delta$  = 2.64 (*br s*).

<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.84 MHz):

$\delta$  = -141.0.

IR:  $\nu$  = 3281, 2359, 1668, 1519, 1315, 1150, 1024 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>9</sub>H<sub>11</sub>BN<sub>2</sub>O<sub>3</sub>F<sub>3</sub>S (M-K) 295.0536, found 295.0540.



Potassium 4-Fluorophenylsulfonamidomethyltrifluoroborate **2p**:

Obtained as a white solid (334 mg, 45%).

mp > 240 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz):

$\delta$  = 7.82 (*dd*,  $J$  = 8.8, 5.6 Hz, 2H), 7.38 (*t*,  $J$  = 8.8 Hz, 2H), 5.80 (*br s*, 1H, NH), 1.57 (*t*,  $J$  = 5.2 Hz, 2H, CH<sub>2</sub>).

<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz):

$\delta$  = 164.1 (*d*,  $J$  = 250.0 Hz), 136.7 (*d*,  $J$  = 3.9 Hz), 130.2 (*d*,  $J$  = 9.3 Hz), 116.0 (*d*,  $J$  = 22.3 Hz).

<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.38 MHz)

$\delta$  = 2.51 (*br s*).

<sup>19</sup>F NMR (acetone-d<sub>6</sub>, 470.84 MHz):

$\delta$  = -110.2, -146.0.

IR:  $\nu$  = 3275, 1593, 1493, 1414, 1311, 1148, 1023, 999, 832 cm<sup>-1</sup>.

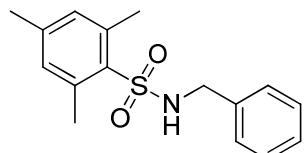
HRMS (ESI) *m/z* calcd. For C<sub>7</sub>H<sub>7</sub>BN<sub>2</sub>O<sub>2</sub>F<sub>4</sub>S (M-K) 256.0227, found 256.0222.

**General procedure for the cross-coupling of sulfonamidomethyltrifluoroborate and aryl or heteroaryl chlorides:**

In a microwave vial equipped with a stirring bar was successively introduced  $\text{Pd}(\text{MeCN})_2\text{Cl}_2$  (1.3 mg, 2 mol %), phosphine ligand (4 mol %), base (3 equiv), and the sulfonamidomethyltrifluoroborate (1 to 1.2 equiv). The vial was then capped and put under inert atmosphere (3x vacuum /  $\text{N}_2$  cycles). The electrophile was then introduced using a microsyringe (0.25 mmol, 1 equiv) followed by 0.5 mL of degassed  $t\text{-BuOH}$  and 0.5 mL of degassed distilled water. The resulting mixture was then placed in an oil bath preheated at 100 °C and stirred overnight (14 to 16 h). After cooling to room temperature, the vial was uncapped and the reaction mixture was diluted with ethyl acetate (5 mL) and water (5 mL). The organic layer was passed through a celite plug and dried over  $\text{MgSO}_4$ . After solvent removal the obtained crude product was purified by flash column chromatography on silica gel or basic alumina using a mixture of hexanes/ethyl acetate as the eluent.

Method A: phosphine ligand = XPhos, base =  $\text{NaOt-Bu}$ .

Method B: phosphine ligand = RuPhos, base =  $\text{Cs}_2\text{CO}_3$ .



*N*-Benzyl-2,4,6-trimethylbenzenesulfonamide **3a**:

Obtained as a white solid [method A, 72% (52 mg), method B, 95% (69 mg)].

mp = 96–98 °C.

$^1\text{H}$  NMR ( $\text{acetone-d}_6$ , 500 MHz):

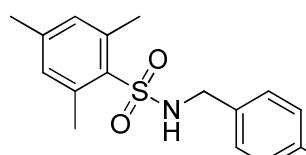
$\delta$  = 7.20 (*m*, 5H), 7.00 (*s*, 2H), 6.77 (*br s*, 1H, NH), 4.09 (*d*, *J* = 6.5 Hz, 2H,  $\text{CH}_2$ ), 2.62 (*s*, 6H, Me), 2.28 (*s*, 3H, Me).

$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz):

$\delta$  = 142.2, 139.1, 136.3, 133.4, 131.9, 128.6, 127.9, 127.8, 46.7, 22.9, 20.8.

IR:  $\nu$  = 3264, 1603, 1403, 1303, 1147, 1048, 878, 744, 651  $\text{cm}^{-1}$

HRMS (ESI) *m/z* calcd. For  $\text{C}_{16}\text{H}_{20}\text{NO}_2\text{S}$  ( $\text{M}+\text{H}$ ) 290.1215, found 290.1229.



*OMe N*-(4-Methoxybenzyl)-2,4,6-trimethylbenzenesulfonamide **3b**:

Obtained as a white solid [method A, 87% (70 mg), method B, 78% (62 mg)].

mp = 98–100 °C.

$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):

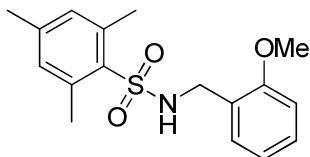
$\delta$  = 7.08 (*d*, *J* = 8.5 Hz, 2H), 6.97 (*s*, 2H), 6.79 (*d*, *J* = 8.5 Hz, 2H), 4.66 (*br s*, 1H, NH), 4.00 (*d*, *J* = 5.5 Hz, 2H,  $\text{CH}_2$ ), 3.78 (*s*, 3H, OMe), 2.64 (*s*, 6H, Me), 2.32 (*s*, 3H, Me).

$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz):

$\delta$  = 159.3, 142.2, 139.2, 133.6, 132.0, 129.3, 128.4, 114.1, 55.3, 46.3, 22.9, 20.9.

IR:  $\nu$  = 3288, 1610, 1511, 1461, 1323, 1242, 1147, 1079, 836, 654  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  calcd. For  $C_{17}H_{21}NO_3S$  Na (M+Na) 342.1140, found 342.1133.



*N*-(2-Methoxy)-2,4,6-trimethylbenzenesulfonamide **3c**:

Obtained as a white solid [method A, 72% (58 mg), method B, 91% (75 mg)].

mp = 84-86 °C.

$^1H$  NMR (acetone-d<sub>6</sub>, 500 MHz):

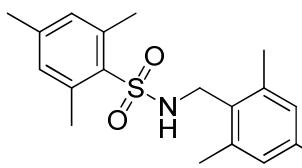
$\delta$  = 7.19 (dd,  $J$  = 8.0, 1.5 Hz, 1H), 6.98 (dd,  $J$  = 7.5, 1.5 Hz, 1H), 6.86 (s, 2H), 6.76 (m, 2H), 5.22 (t,  $J$  = 6.5 Hz, 1H, NH), 4.08 (d,  $J$  = 6.5 Hz, 2H, CH<sub>2</sub>), 3.77 (s, 3H, MeO), 2.61 (s, 6H, Me), 2.27 (s, 3H, Me).

$^{13}C$  NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 141.8, 138.8, 133.9, 131.6, 129.6, 129.1, 124.2, 120.4, 109.9, 55.0, 43.4, 22.7, 20.8.

IR:  $\nu$  = 3321, 1606, 1498, 1314, 1250, 1164, 1153, 1042, 836, 746, 657 cm<sup>-1</sup>.

HRMS (ESI)  $m/z$  calcd. For  $C_{17}H_{21}NO_3S$  Na (M+Na) 342.1140, found 342.1142.



*N*-(4-Methoxy-2,6-dimethylbenzyl)-2,4,6-

trimethylbenzenesulfonamide **3d**:

Obtained as a white solid [method A, 48% (42 mg), method B, 78% (68 mg)].

mp = 84-86 °C.

$^1H$  NMR (CDCl<sub>3</sub>, 500 MHz):

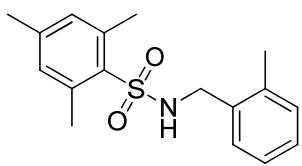
$\delta$  = 7.00 (s, 2H), 6.55 (s, 2H), 4.19 (t,  $J$  = 3.0 Hz, 1H, NH), 4.00 (d,  $J$  = 5.5 Hz, 2H, CH<sub>2</sub>), 3.76 (s, 3H, MeO), 2.67 (s, 6H, Me), 2.34 (s, 3H, Me), 2.18 (s, 6H, Me).

$^{13}C$  NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 158.9, 142.2, 139.3, 138.9, 132.8, 131.9, 124.3, 113.6, 55.0, 40.3, 22.9, 20.9, 19.4.

IR:  $\nu$  = 3274, 1605, 1508, 1427, 1321, 1222, 1150, 1074, 830, 655 cm<sup>-1</sup>.

HRMS (ESI)  $m/z$  calcd. For  $C_{19}H_{25}NO_3S$  Na (M+Na) 370.1453, found 370.1459.



2,4,6-Trimethyl-N-(2-methylbenzyl)benzenesulfonamide **3e**:

Obtained as a white solid [method A, 80% (60 mg), method B, 91% (69 mg)].

mp = 120-123 °C.

$^1H$  NMR (CDCl<sub>3</sub>, 500 MHz):

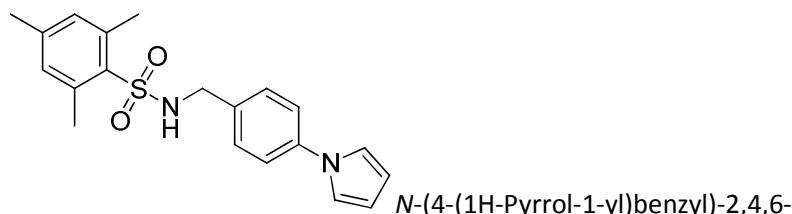
$\delta$  = 7.19 (*m*, 1H), 7.14 (*m*, 1H), 7.11 (*m*, 2H), 6.99 (*s*, 2H), 4.54 (*t*,  $J$  = 5.5 Hz, 1H, NH), 4.05 (*d*,  $J$  = 6.0 Hz, 2H, CH<sub>2</sub>), 2.66 (*s*, 6H, Me), 2.34 (*s*, 3H, Me), 2.25 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 142.3, 139.2, 136.6, 134.0, 131.9, 128.8, 128.2, 126.1, 44.7, 22.9, 20.9, 18.6.

IR:  $\nu$  = 3298, 1605, 1406, 1319, 1158, 1056, 758, 655 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>17</sub>H<sub>21</sub>NO<sub>2</sub>SNa (M+Na) 326.1191, found 326.1184.



trimethylbenzenesulfonamide **3f**:

Obtained as a white solid [method A, 70% (61 mg), method B, 91% (81 mg)].

mp = 131-134 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

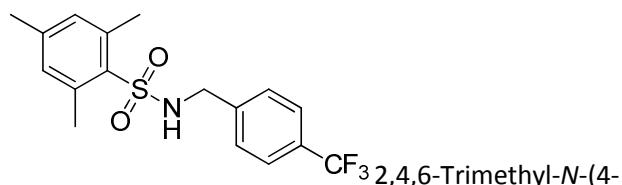
$\delta$  = 7.28 (*d*,  $J$  = 8.5 Hz, 2H), 7.23 (*d*,  $J$  = 8.5 Hz, 2H), 7.05 (*t*,  $J$  = 2.2 Hz, 2H), 6.96 (*s*, 2H), 6.35 (*t*,  $J$  = 2.2 Hz, 2H), 4.88 (*br s*, 1H, NH), 4.11 (*d*,  $J$  = 6.2 Hz, 2H, CH<sub>2</sub>), 2.65 (*s*, 6H, Me), 2.31 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 142.4, 140.2, 139.0, 133.7, 133.6, 131.9, 129.0, 120.3, 119.1, 110.5, 46.1, 22.9, 20.8.

IR:  $\nu$  = 3328, 1526, 1400, 1324, 1153, 1074, 834, 734, 654 cm<sup>-1</sup>

HRMS (ESI) *m/z* calcd. For C<sub>20</sub>H<sub>22</sub>N<sub>2</sub>O<sub>2</sub>SNa (M+Na) 377.1300, found 377.1309.



(trifluoromethyl)benzyl)benzenesulfonamide **3g**:

Obtained as a white solid [method A, 76% (68 mg), method B, 61% (54 mg)].

mp = 91-94 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

$\delta$  = 7.47 (*d*,  $J$  = 8.0 Hz, 2H), 7.28 (*d*,  $J$  = 8.0 Hz, 2H), 6.91 (*s*, 2H), 5.19 (*t*,  $J$  = 6.0 Hz, 1H, NH), 4.16 (*d*,  $J$  = 6.0 Hz, 2H, CH<sub>2</sub>), 2.61 (*s*, 6H, Me), 2.29 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

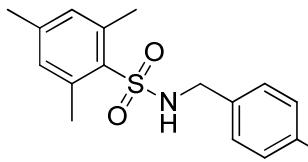
$\delta$  = 142.5, 140.4, 138.9, 133.4, 131.9, 129.9 (*d*,  $J$  = 32.9 Hz), 128.0, 125.4 (*q*,  $J$  = 3.7 Hz), 123.9 (*d*,  $J$  = 272.3 Hz), 46.2, 22.9, 20.8.

<sup>19</sup>F NMR (CDCl<sub>3</sub>, 470.84 MHz):

$\delta$  = -62.6.

IR:  $\nu$  = 3284, 1607, 1445, 1325, 1144, 1115, 1067, 850, 656 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>17</sub>H<sub>17</sub>NO<sub>2</sub>F<sub>3</sub>S (M-H) 356.0932, found 356.0945.



*N*-(4-Fluorobenzyl)-2,4,6-trimethylbenzenesulfonamide **3h**:

Obtained as a white solid [method A, 84% (65 mg), method B, 87% (67 mg)].

mp = 88–90 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

$\delta$  = 7.14 (*m*, 2H), 6.96 (*s*, 2H), 6.93 (*d*, *J* = 8.5 Hz, 2H), 4.80 (*t*, *J* = 3.0 Hz, 1H, NH), 4.06 (*d*, *J* = 6.2 Hz, 2H, CH<sub>2</sub>), 2.63 (*s*, 6H, Me), 2.32 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

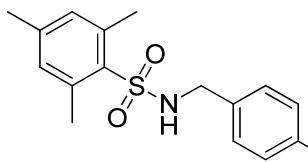
$\delta$  = 162.2 (*d*, *J* = 281.0 Hz), 142.3, 139.0, 133.5, 132.2, 131.9, 129.5 (*d*, *J* = 8.2 Hz), 115.4 (*d*, *J* = 21.5 Hz), 46.0, 22.8, 20.8.

<sup>19</sup>F NMR (CDCl<sub>3</sub>, 470.84 MHz):

$\delta$  = -114.3.

IR:  $\nu$  = 3277, 1604, 1508, 1427, 1321, 1221, 1150, 1075, 837, 656 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>16</sub>H<sub>18</sub>NO<sub>2</sub>FSNa (M+Na) 330.0940, found 330.0943.



*N*-(4-Cyanobenzyl)-2,4,6-trimethylbenzenesulfonamide **3i**:

Obtained as an oil [method A, 44% (34 mg), method B, 77% (60 mg)].

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

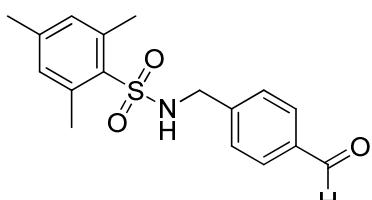
$\delta$  = 7.51 (*d*, *J* = 8.0 Hz, 2H), 7.30 (*d*, *J* = 8.0 Hz, 2H), 6.93 (*s*, 2H), 5.25 (*t*, *J* = 6.0 Hz, 1H, NH), 4.16 (*d*, *J* = 6.5 Hz, 2H, CH<sub>2</sub>), 2.60 (*s*, 6H, Me), 2.31 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 142.6, 142.1, 138.9, 133.4, 132.2, 132.0, 128.2, 118.4, 111.4, 46.1, 22.8, 20.8.

IR:  $\nu$  = 3281, 2228, 1607, 1452, 1324, 1153, 1058, 849, 655 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>17</sub>H<sub>17</sub>N<sub>2</sub>O<sub>2</sub>S (M-H) 313.1011, found 313.1013.



*N*-(4-Formylbenzyl)-2,4,6-trimethylbenzenesulfonamide **3j**:

Obtained as a yellowish solid [method B, 51% (41 mg)].

mp = 110–115 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

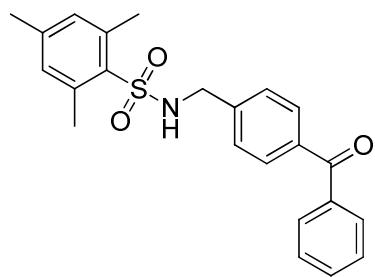
$\delta$  = 9.95 (*s*, 1H, CHO), 7.75 (*d*, *J* = 8.0 Hz, 2H), 7.36 (*d*, *J* = 8.0 Hz, 2H), 6.95 (*s*, 2H), 5.13 (*t*, *J* = 6.0 Hz, 1H, NH), 4.18 (*d*, *J* = 6.0 Hz, 2H, CH<sub>2</sub>), 2.62 (*s*, 6H, Me), 2.30 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 191.7, 143.4, 142.5, 139.0, 135.7, 133.4, 132.0, 129.9, 128.2, 46.3, 22.9, 20.8.

IR:  $\nu$  = 3320, 1694, 1609, 1421, 1322, 1153, 1068, 841, 657 cm<sup>-1</sup>

HRMS (ESI) *m/z* calcd. For C<sub>17</sub>H<sub>18</sub>NO<sub>3</sub>S (M-H) 316.1006, found 316.1006.



*N*-(4-Benzoylbenzyl)-2,4,6-trimethylbenzenesulfonamide **3k**:

Obtained as a white solid [method A, 60% (59 mg), method B, 55% (54 mg)].

mp = 112-116 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

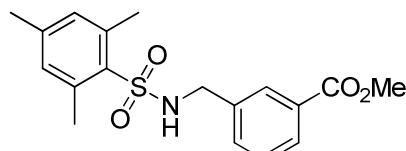
$\delta$  = 7.75 (*dd*, *J* = 8.5, 1.0 Hz, 2H), 7.69 (*d*, *J* = 8.0 Hz, 2H), 7.60 (*dt*, *J* = 7.5, 1.0 Hz, 1H), 7.49 (*t*, *J* = 8.0 Hz, 2H), 7.30 (*d*, *J* = 8.5 Hz, 2H), 6.96 (*s*, 2H), 4.95 (*t*, *J* = 6.0 Hz, 1H, NH), 4.20 (*d*, *J* = 6.5 Hz, 2H, CH<sub>2</sub>), 2.65 (*s*, 6H, Me), 2.31 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 196.0, 142.4, 141.0, 139.0, 137.3, 137.0, 132.4, 131.9, 130.3, 129.9, 128.2, 127.5, 46.4, 22.9, 20.9.

IR:  $\nu$  = 3288, 1646, 1608, 1427, 1319, 1282, 1057, 900, 746, 698, 656 cm<sup>-1</sup>

HRMS (ESI) *m/z* calcd. For C<sub>23</sub>H<sub>23</sub>NO<sub>3</sub>SNa (M+Na) 416.1296, found 416.1317.



Methyl 3-((2,4,6-

Trimethylphenylsulfonamido)methyl)benzoate **3l**:

Obtained as a white solid [method B, 44% (49 mg)].

mp = 87-89 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

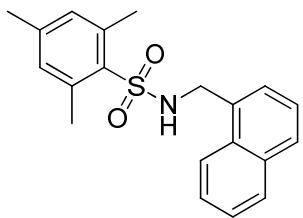
$\delta$  = 7.89 (*d*, *J* = 7.5 Hz, 1H), 7.80 (*s*, 1H), 7.40 (*d*, *J* = 7.5 Hz, 1H), 7.33 (*t*, *J* = 7.5 Hz, 1H), 6.92 (s, 2H), 4.97 (*t*, *J* = 6.0 Hz, 1H, NH), 4.15 (*d*, *J* = 6.0 Hz, 2H, CH<sub>2</sub>), 3.90 (*s*, 3H, MeO<sub>2</sub>C), 2.63 (*s*, 6H, Me), 2.29 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 166.5, 142.3, 138.9, 136.7, 132.3, 131.9, 130.3, 128.9, 128.8, 128.6, 52.1, 46.4, 22.9, 20.8.

IR:  $\nu$  = 3317, 1712, 1307, 1287, 1151, 1062, 754, 656 cm<sup>-1</sup>

HRMS (ESI) *m/z* calcd. For C<sub>18</sub>H<sub>21</sub>NO<sub>4</sub>SNa (M+Na) 370.1089, found 370.1083.



2,4,6-Trimethyl-N-(naphthalen-1-ylmethyl)benzenesulfonamide:

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 500 MHz):

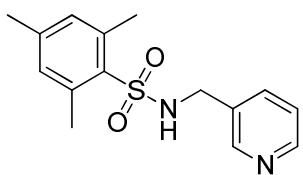
$\delta$  = 7.82 (m, 3H), 7.48 (m, 2H), 7.33 (m, 2H), 6.98 (s, 2H), 4.70 (t,  $J$  = 5.5 Hz, 1H, NH), 4.50 (d,  $J$  = 6.0 Hz, 2H, CH<sub>2</sub>), 2.64 (s, 6H, Me), 2.34 (s, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 142.3, 139.3, 133.7, 133.1, 131.9, 131.4, 131.1, 129.0, 128.7, 127.0, 126.5, 126.0, 125.1, 123.1, 44.9, 22.9, 20.9.

IR:  $\nu$  = 3326, 1600, 1387, 1154, 1049, 776, 660 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>20</sub>H<sub>20</sub>NO<sub>2</sub>S (M-H) 338.1215, found 338.1200.



2,4,6-Trimethyl-N-(pyridin-3-ylmethyl)benzenesulfonamide **4a**:

Obtained as a white solid [method B, 89% (64 mg)].

mp = 106-108 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

$\delta$  = 8.44 (d,  $J$  = 3.5 Hz, 1H), 8.36 (s, 1H), 7.57 (d,  $J$  = 8.0 Hz, 1H), 7.19 (dd,  $J$  = 8.0, 5.0 Hz, 1H), 6.94 (s, 2H), 5.50 (t,  $J$  = 6.0 Hz, 1H, NH), 4.11 (d,  $J$  = 6.5 Hz, 2H, CH<sub>2</sub>), 2.62 (s, 6H, Me), 2.30 (s, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 148.9, 142.4, 139.0, 135.8, 133.4, 131.9, 123.5, 44.1, 22.8, 20.8.

IR:  $\nu$  = 3307, 1734, 1602, 1451, 1324, 1148, 1062, 829, 655 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>15</sub>H<sub>19</sub>N<sub>2</sub>O<sub>2</sub>Na (M+H) 291.1167, found 291.1171.



trimethylbenzenesulfonamide **4b**:

Obtained as a white solid [method B, 92% (74 mg)].

mp = 111-114 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

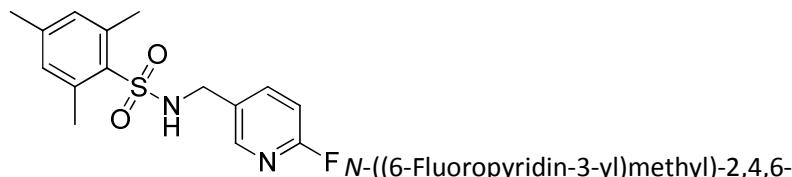
$\delta$  = 7.89 (d,  $J$  = 1.0 Hz, 1H), 7.40 (dd,  $J$  = 8.5, 2.0 Hz, 1H), 6.93 (s, 2H), 6.60 (d,  $J$  = 8.5 Hz, 1H), 5.04 (t,  $J$  = 5.5 Hz, 1H, NH), 4.00 (d,  $J$  = 6.5 Hz, 2H, CH<sub>2</sub>), 3.86 (s, 3H, OMe), 2.81 (s, 6H, Me), 2.29 (s, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 163.8, 146.0, 142.3, 139.0, 138.6, 133.4, 131.9, 124.8, 110.8, 53.4, 43.6, 22.8, 20.8.

IR:  $\nu$  = 3338, 1736, 1614, 1494, 1322, 1152, 1023, 858, 658 cm<sup>-1</sup>

HRMS (ESI) *m/z* calcd. For C<sub>16</sub>H<sub>21</sub>N<sub>2</sub>O<sub>3</sub>S (M+H) 321.1273, found 321.1268.



trimethylbenzenesulfonamide **4c**:

Obtained as a white solid [method B, 88% (68 mg)].

mp = 100-103 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

$\delta$  = 7.98 (*d*, *J* = 2.0 Hz, 1H), 7.68 (*dt*, *J* = 8.0, 2.5 Hz, 1H), 6.95 (*s*, 2H), 6.81 (*dd*, *J* = 8.0, 2.5 Hz, 1H), 5.31 (*t*, *J* = 6.0 Hz, 1H, NH), 4.10 (*d*, *J* = 6.5 Hz, 2H, CH<sub>2</sub>), 2.61 (*s*, 6H, Me), 2.30 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

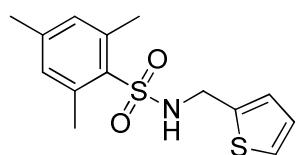
$\delta$  = 163.1 (*d*, *J* = 240.0 Hz), 146.7 (*d*, *J* = 15.0 Hz), 142.6, 141.0 (*d*, *J* = 8.2 Hz), 138.9, 133.4, 132.0, 130.1 (*d*, *J* = 4.4 Hz), 109.4 (*d*, *J* = 37.8 Hz), 43.2, 22.8, 20.8.

<sup>19</sup>F NMR (CDCl<sub>3</sub>, 470.84 MHz):

$\delta$  = -69.1.

IR:  $\nu$  = 3300, 1345, 1314, 1155, 1060, 837, 656 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>15</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub>FS (M-H) 307.0917, found 307.0919.



Obtained as a white solid [method B, 62% (46 mg)].

mp = 95-98 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

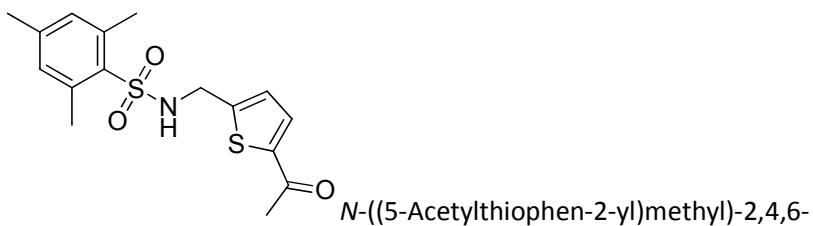
$\delta$  = 7.19 (*dd*, *J* = 5.0, 1.5 Hz, 1H), 6.97 (*s*, 2H), 6.88 (*dd*, *J* = 5.0, 1.5 Hz, 1H), 6.84 (*d*, *J* = 3.0 Hz, 1H), 4.82 (*t*, *J* = 5.5 Hz, 1H, NH), 4.30 (*d*, *J*=6.00 Hz, 2H, CH<sub>2</sub>), 2.65 (*s*, 6H, Me), 2.32 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 142.3, 139.2, 139.0, 133.3, 131.9, 126.8, 126.3, 125.7, 41.5, 22.9, 20.8.

IR:  $\nu$  = 3277, 1733, 1316, 1155, 1066, 850, 705, 655 cm<sup>-1</sup>

HRMS (ESI) *m/z* calcd. For C<sub>14</sub>H<sub>17</sub>NO<sub>2</sub>S<sub>2</sub>Na (M+Na) 318.0598, found 318.0609.



**trimethylbenzenesulfonamide **4e**:**

Obtained as a white solid [method B, 83% (70 mg)].

mp = 133-136 °C.

$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):

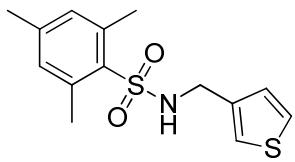
$\delta$  = 7.46 (*d*,  $J$  = 3.5 Hz, 1H), 6.96 (*s*, 2H), 6.90 (*d*,  $J$  = 3.5 Hz, 1H), 5.04 (*br s*, 1H, NH), 4.32 (*d*,  $J$  = 6.00 Hz, 2H,  $\text{CH}_2$ ), 2.64 (*s*, 6H, Me), 2.49 (*s*, 3H, Ac), 2.30 (*s*, 3H, Me).

$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz):

$\delta$  = 190.3, 148.4, 144.1, 142.6, 139.2, 133.2, 132.3, 132.0, 131.8, 126.9, 41.8, 26.5, 22.9, 20.8.

IR:  $\nu$  = 3287, 1647, 1332, 1150, 1057, 852, 656  $\text{cm}^{-1}$ .

HRMS (ESI) *m/z* calcd. For  $\text{C}_{16}\text{H}_{19}\text{NO}_3\text{S}_2\text{Na}$  ( $\text{M}+\text{Na}$ ) 360.0704, found 360.0692.



**2,4,6-Trimethyl-N-(thiophen-3-ylmethyl)benzenesulfonamide **4f**:**

Obtained as a white solid [method B, 83% (64 mg)].

mp = 76-78 °C.

$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):

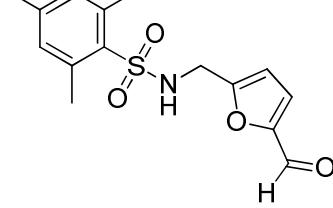
$\delta$  = 7.22 (*dd*,  $J$  = 5.0, 2.5 Hz, 1H), 7.05 (*s*, 1H), 6.96 (*s*, 2H), 6.87 (*d*,  $J$  = 5.0 Hz, 1H), 4.82 (*br s*, 1H, NH), 4.11 (*d*,  $J$  = 6.0 Hz, 2H,  $\text{CH}_2$ ), 2.64 (*s*, 6H, Me), 2.32 (*s*, 3H, Me).

$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz):

$\delta$  = 142.2, 139.1, 137.2, 133.5, 131.9, 127.0, 126.4, 122.8, 41.8, 22.9, 20.8.

IR:  $\nu$  = 3285, 1732, 1418, 1315, 1154, 1066, 850, 787, 656  $\text{cm}^{-1}$

HRMS (ESI) *m/z* calcd. For  $\text{C}_{14}\text{H}_{17}\text{NO}_2\text{S}_2\text{Na}$  ( $\text{M}+\text{Na}$ ) 318.0598, found 318.0601.



**trimethylbenzenesulfonamide **4g**:**

Obtained as a yellowish oil [method B, 26% (20 mg)].

$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):

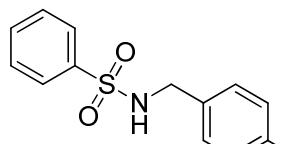
$\delta$  = 9.45 (*s*, 1H, CHO), 7.01 (*d*,  $J$  = 3.5 Hz, 1H), 6.89 (*s*, 2H), 6.29 (*d*,  $J$  = 3.5 Hz, 1H), 5.30 (*t*,  $J$  = 6.0 Hz, 1H, NH), 4.26 (*d*,  $J$  = 6.5 Hz, 2H,  $\text{CH}_2$ ), 2.61 (*s*, 6H, Me), 2.27 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 177.1, 156.5, 152.3, 142.4, 138.9, 133.5, 131.8, 110.4, 39.6, 22.7, 20.8.

IR:  $\nu$  = 3736, 2360, 1654, 1537, 1320, 1158, 1026, 650 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>15</sub>H<sub>17</sub>NO<sub>4</sub>SNa (M+Na) 330.0776, found 330.0788.



OMe *N*-(4-Methoxybenzyl)benzenesulfonamide **5a**:

Obtained as a white solid [method B, 70% (49 mg)].

mp = 72-75 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

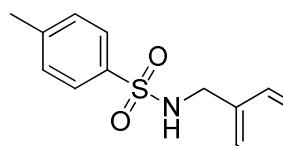
$\delta$  = 7.89 (*m*, 2H), 7.60 (*m*, 1H), 7.54 (*m*, 2H), 7.11 (*m*, 2H), 6.81 (*m*, 2H), 4.54 (*t*,  $J$  = 5.5 Hz, 1H, NH), 4.10 (*d*,  $J$  = 6.0 Hz, 2H, CH<sub>2</sub>), 3.79 (*s*, 3H, OMe).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 159.2, 139.8, 132.6, 129.2, 129.0, 128.1, 127.0, 114.0, 55.2, 46.7.

IR:  $\nu$  = 3279, 1612, 1512, 1254, 1158, 1032, 729, 685 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>14</sub>H<sub>15</sub>NO<sub>3</sub>SNa (M+Na) 300.0670, found 300.0665.



OMe *N*-(4-Methoxybenzyl)-4-methylbenzenesulfonamide **5b**:

Obtained as a white solid [method B, 76% (55 mg)].

mp = 114-117 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

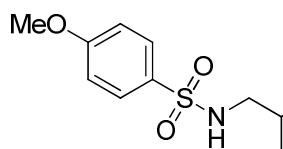
$\delta$  = 7.75 (*d*,  $J$  = 8.5 Hz, 2H), 7.31 (*d*,  $J$  = 8.5 Hz, 2H), 7.11 (*d*,  $J$  = 8.5 Hz, 2H), 6.79 (*dd*,  $J$  = 6.5, 2.0 Hz, 2H), 4.74 (*t*,  $J$  = 6.0 Hz, 1H, NH), 4.05 (*d*,  $J$  = 6.0 Hz, 2H, CH<sub>2</sub>), 3.77 (*s*, 3H, OMe), 2.44 (*s*, 3H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 143.4, 136.8, 129.6, 129.2, 128.2, 127.1, 114.0, 51.2, 46.7, 21.4.

IR:  $\nu$  = 3247, 1515, 1321, 1253, 1158, 1031, 817 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>15</sub>H<sub>17</sub>NO<sub>3</sub>SNa (M+Na) 314.0827, found 314.0829.



OMe 4-Methoxy-*N*-(4-methoxybenzyl)benzenesulfonamide **5c**:

Obtained as a white solid [method B, 70% (54 mg)].

mp = 106-108 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

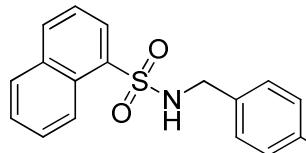
$\delta$  = 7.79 (*d*, *J* = 9.0 Hz, 2H), 7.11 (*d*, *J* = 8.5 Hz, 2H), 6.97 (*d*, *J* = 9.0 Hz, 2H), 6.79 (*d*, *J* = 9.0 Hz, 2H), 4.76 (*t*, *J* = 6.0 Hz, 1H, NH), 4.04 (*d*, *J* = 6.5 Hz, 2H, CH<sub>2</sub>), 3.88 (*s*, 3H, OMe), 3.77 (*s*, 3H, OMe).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 162.8, 159.2, 131.4, 129.2, 129.1, 128.2, 114.2, 114.0, 55.5, 55.2, 46.7.

IR:  $\nu$  = 3253, 1596, 1518, 1415, 1306, 1258, 1148, 1027, 838, 678 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>15</sub>H<sub>17</sub>NO<sub>4</sub>SNa (M+Na) 330.0776, found 330.0789.



OMe *N*-(4-Methoxybenzyl)naphthalene-1-sulfonamide **5d**:

Obtained as a colorless oil [method B, 77% (63 mg)].

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

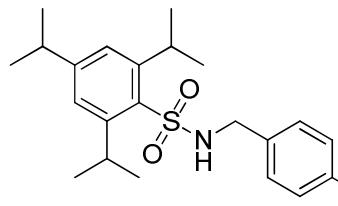
$\delta$  = 8.65 (*d*, *J* = 8.5 Hz, 1H), 8.27 (*dd*, *J* = 8.0, 1.5 Hz, 1H), 8.07 (*d*, *J* = 8.0 Hz, 1H), 7.96 (*d*, *J* = 8.0 Hz, 1H), 7.66 (*m*, 1H), 7.61 (*td*, *J* = 8.0, 1.0 Hz, 1H), 7.53 (*t*, *J* = 8.0 Hz, 1H), 6.96 (*d*, *J* = 8.5 Hz, 2H), 6.67 (*d*, *J* = 8.5 Hz, 2H), 5.0 (*t*, *J* = 6.0 Hz, 1H, NH), 4.02 (*d*, *J* = 6.0 Hz, 2H, CH<sub>2</sub>), 3.73 (*s*, 3H, OMe).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 159.1, 134.4, 134.2, 134.1, 129.8, 129.1, 128.3, 128.1, 128.0, 126.8, 124.2, 124.1, 113.8, 55.2, 46.8.

IR:  $\nu$  = 3297, 1612, 1513, 1249, 1160, 1133, 1032, 804, 771 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>18</sub>H<sub>17</sub>NO<sub>3</sub>SNa (M+Na) 350.0827, found 350.0824.



OMe 2,4,6-Triisopropyl-*N*-(4-methoxybenzyl)benzenesulfonamide **5e**:

Obtained as a white solid [method B, 24% (24 mg)].

mp = 94-96 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz):

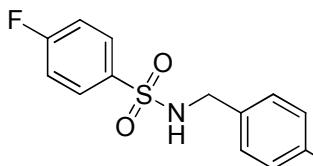
$\delta$  = 7.19 (*s*, 2H), 7.12 (*d*, *J* = 8.5 Hz, 2H), 6.81 (*d*, *J* = 8.5 Hz, 2H), 4.5 (*t*, *J* = 6.0 Hz, 1H, NH), 4.19 (*sept.*, *J* = 7.0 Hz, 2H, CH(Me)<sub>2</sub>), 4.09 (*d*, *J* = 6.0 Hz, 2H, CH<sub>2</sub>), 3.78 (*s*, 3H, OMe), 2.93 (*sept.*, *J* = 7.0 Hz, 1H, CH(Me)<sub>2</sub>), 1.28 (*t*, *J* = 7.0 Hz, 18H, Me).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 159.2, 152.8, 150.2, 132.2, 129.4, 128.4, 123.7, 114.0, 55.2, 46.4, 34.1, 29.6, 24.8, 23.5.

IR:  $\nu$  = 3299, 2960, 1602, 1512, 1248, 1151, 1040, 850, 656 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>23</sub>H<sub>33</sub>NO<sub>3</sub>SNa (M+Na) 426.2079, found 426.2090.



OMe 4-Fluoro-N-(4-methoxybenzyl)benzenesulfonamide **5f**:

Obtained as a white solid [method B, 64% (47 mg)].

mp = 90–93 °C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):

$\delta$  = 7.86 (ddd,  $J$  = 8.5, 5.0, 2.0 Hz, 2H), 7.18 (t,  $J$  = 8.5 Hz, 2H), 7.10 (d,  $J$  = 8.5 Hz, 2H), 6.80 (d,  $J$  = 8.5 Hz, 2H), 4.81 (t,  $J$  = 6.0 Hz, 1H, NH), 4.09 (d,  $J$  = 6.0 Hz, 2H, CH<sub>2</sub>), 3.78 (s, 3H, OMe).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125.8 MHz):

$\delta$  = 164.9 (d,  $J$  = 126.0 Hz), 159.3, 136.0, 129.8 (d,  $J$  = 9.3 Hz), 129.2, 127.9, 116.2 (d,  $J$  = 22.5 Hz), 114.0, 55.2, 46.7.

<sup>19</sup>F NMR (CDCl<sub>3</sub>, 470.84 MHz):

$\delta$  = -105.3.

IR:  $\nu$  = 3254, 1590, 1514, 1251, 1152, 1032, 842 cm<sup>-1</sup>.

HRMS (ESI) *m/z* calcd. For C<sub>14</sub>H<sub>14</sub>NO<sub>3</sub>SFNa (M+Na) 318.0576, found 318.0576.



OMe *N*-(4-(*N*-(4-methoxybenzyl)sulfamoyl)phenyl)acetamide

**5g:**

Obtained as a white solid [method B, 42% (35 mg)].

mp = 164–167 °C.

<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz):

$\delta$  = 9.57 (br s, 1H, NH), 7.81 (d,  $J$  = 9.0 Hz, 2H), 7.77 (d,  $J$  = 9.0 Hz, 2H), 7.19 (d,  $J$  = 8.5 Hz, 2H), 6.85 (d,  $J$  = 8.5 Hz, 2H), 6.67 (t,  $J$  = 6.0 Hz, 1H, NH), 4.04 (d,  $J$  = 6.5 Hz, 2H, CH<sub>2</sub>), 3.77 (s, 3H, OMe), 2.14 (s, 3H, Me).

<sup>13</sup>C NMR (acetone-d<sub>6</sub>, 125.8 MHz):

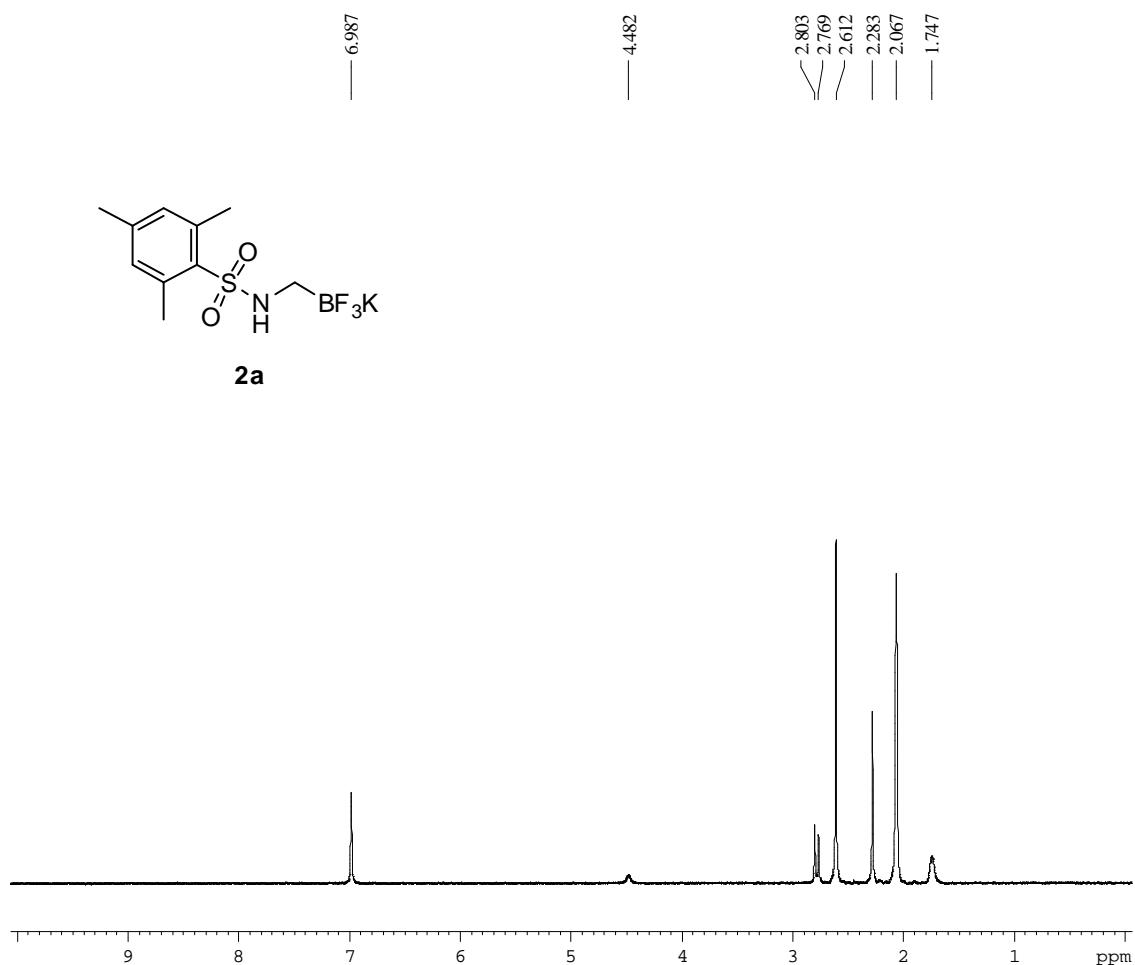
$\delta$  = 168.6, 159.1, 145.7, 143.2, 141.5, 129.0, 127.9, 118.4, 113.5, 54.5, 46.2, 23.4.

IR:  $\nu$  = 3359, 3230, 1676, 1594, 1515, 1304, 1147, 830, 617 cm<sup>-1</sup>.

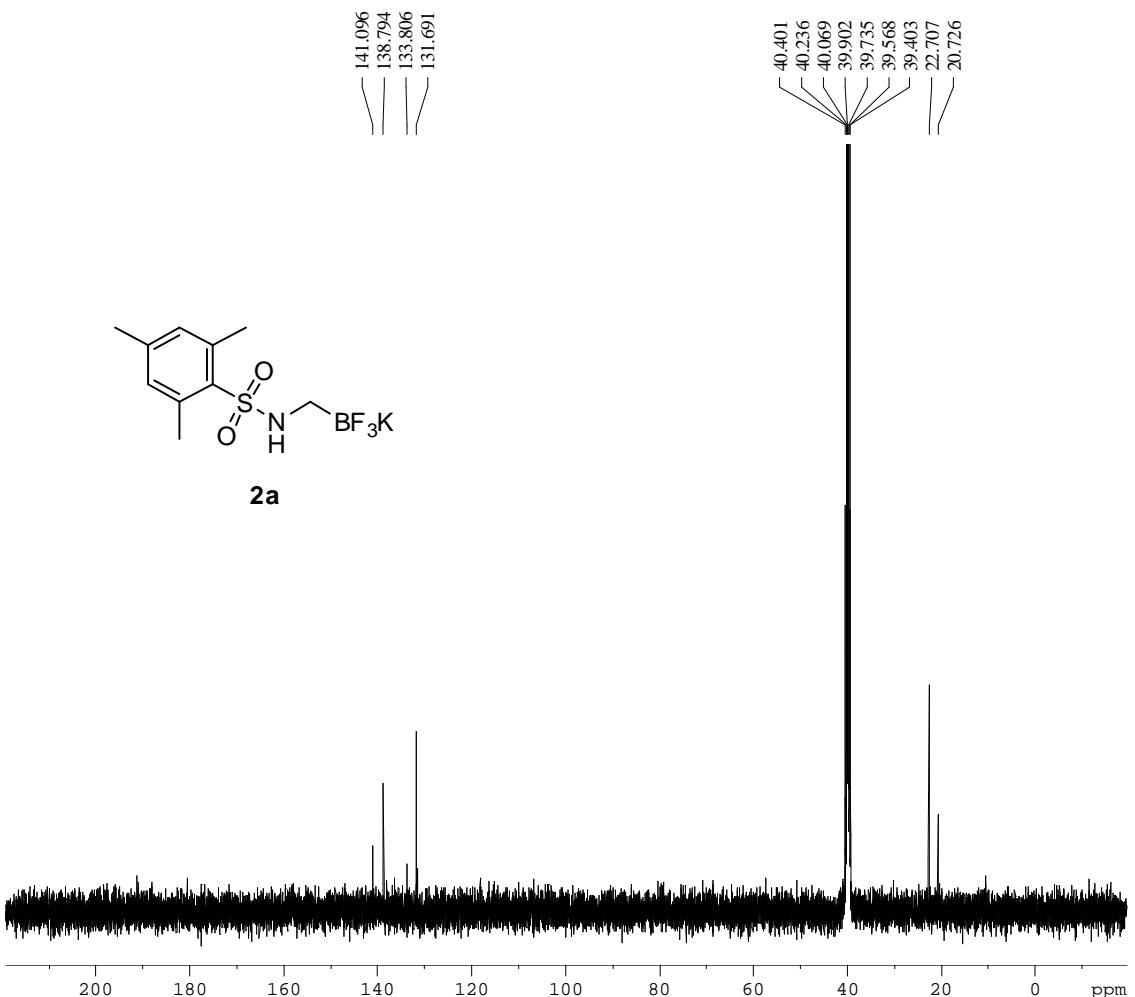
HRMS (ESI) *m/z* calcd. For C<sub>16</sub>H<sub>18</sub>N<sub>2</sub>O<sub>4</sub>SNa (M+Na) 333.0909, found 333.0903.

Table1

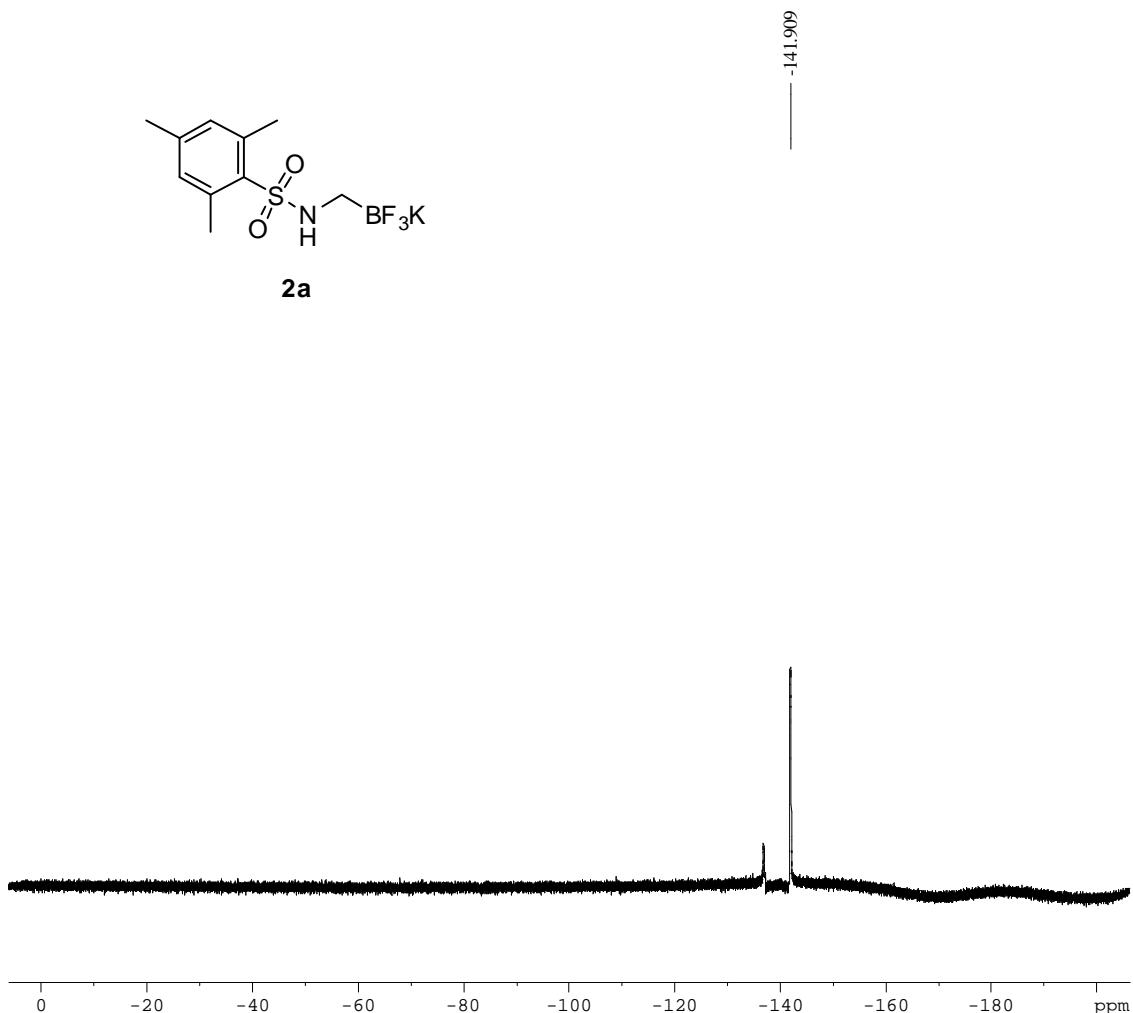
<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400MHz) spectrum of Potassium 2,4,6-Trimethylphenylsulfonamidomethyltrifluoroborate **2a**:



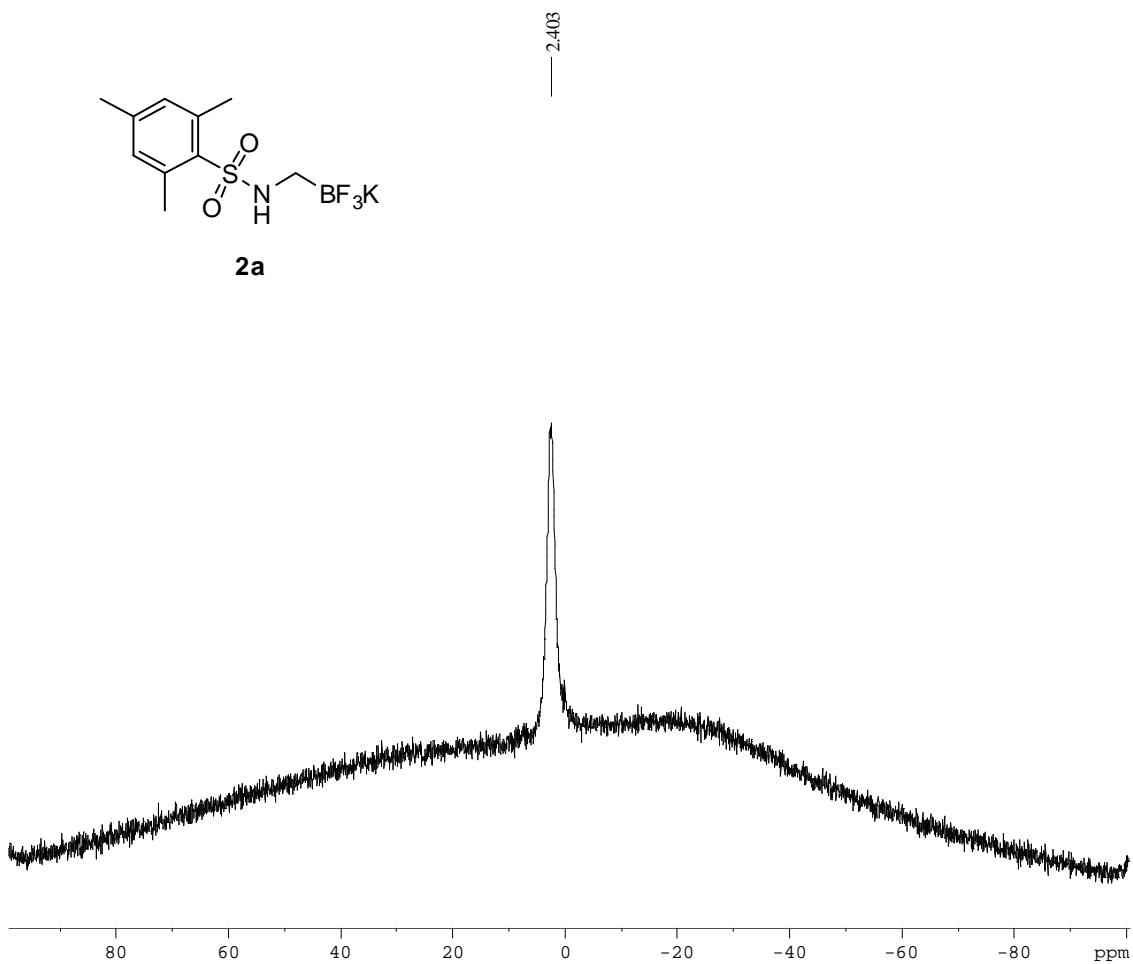
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 2,4,6-Trimethylphenylsulfonamidomethyltrifluoroboroborate **2a**:



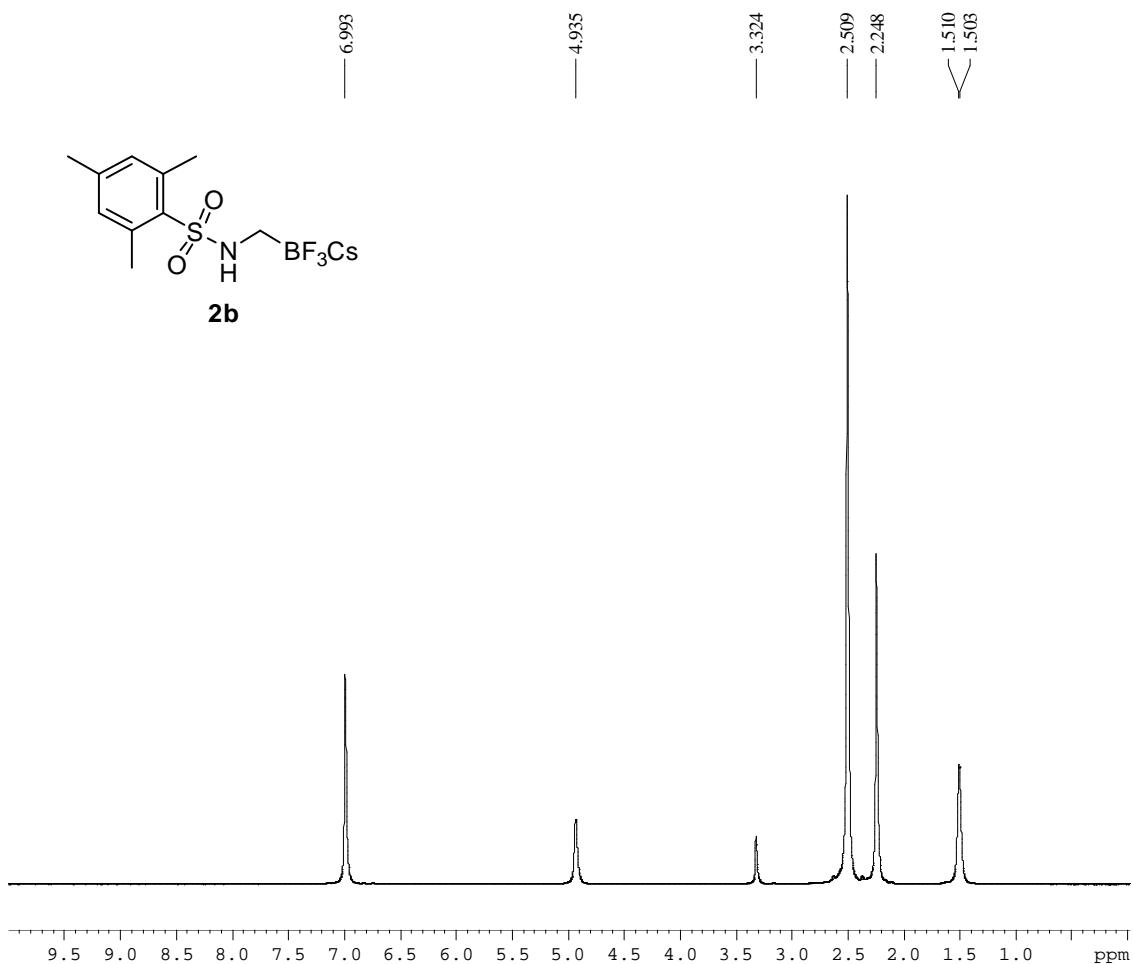
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) Potassium 2,4,6-Trimethylphenylsulfonamidomethyltrifluoroborate **2a**:



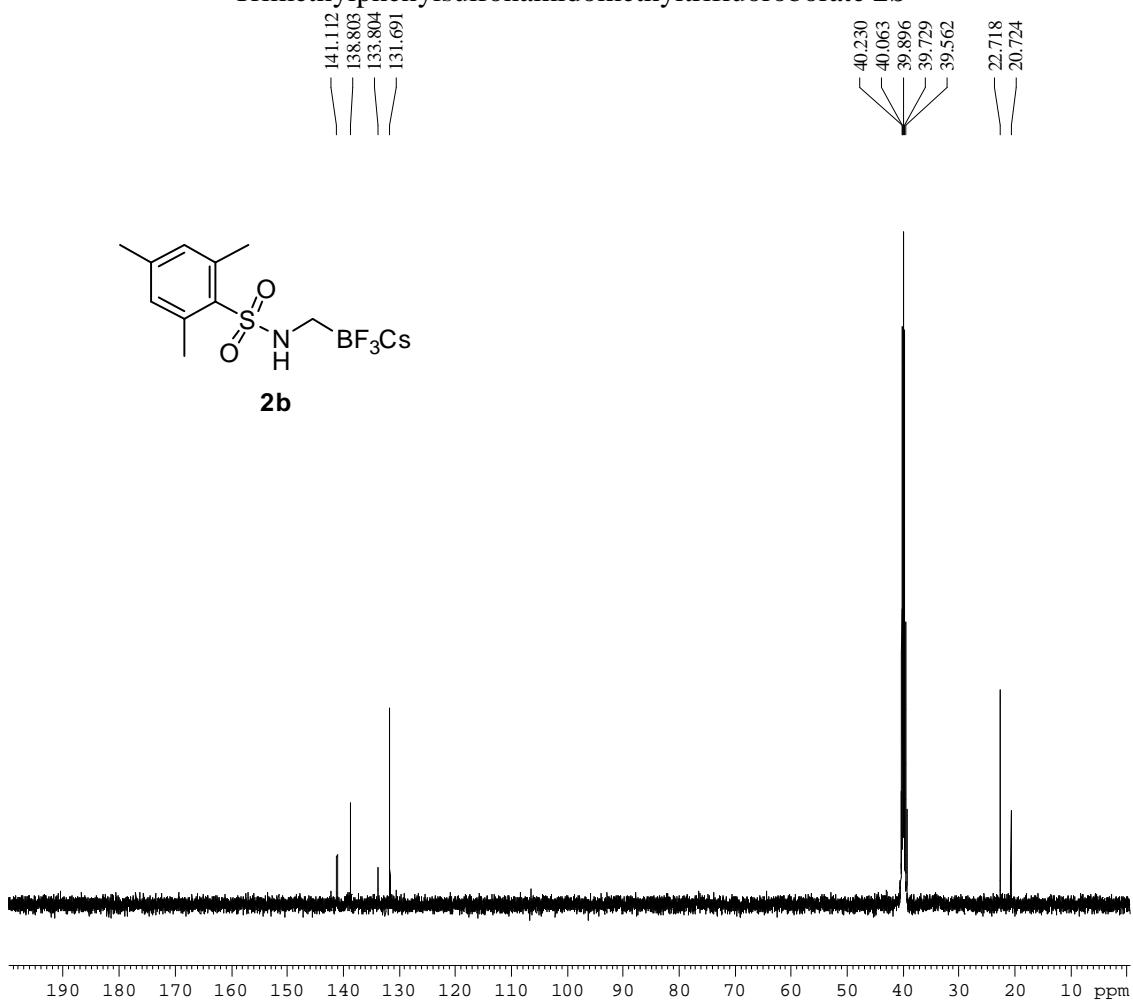
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) spectrum of Potassium 2,4,6-Trimethylphenylsulfonamidomethyltrifluoroborate **2a**:



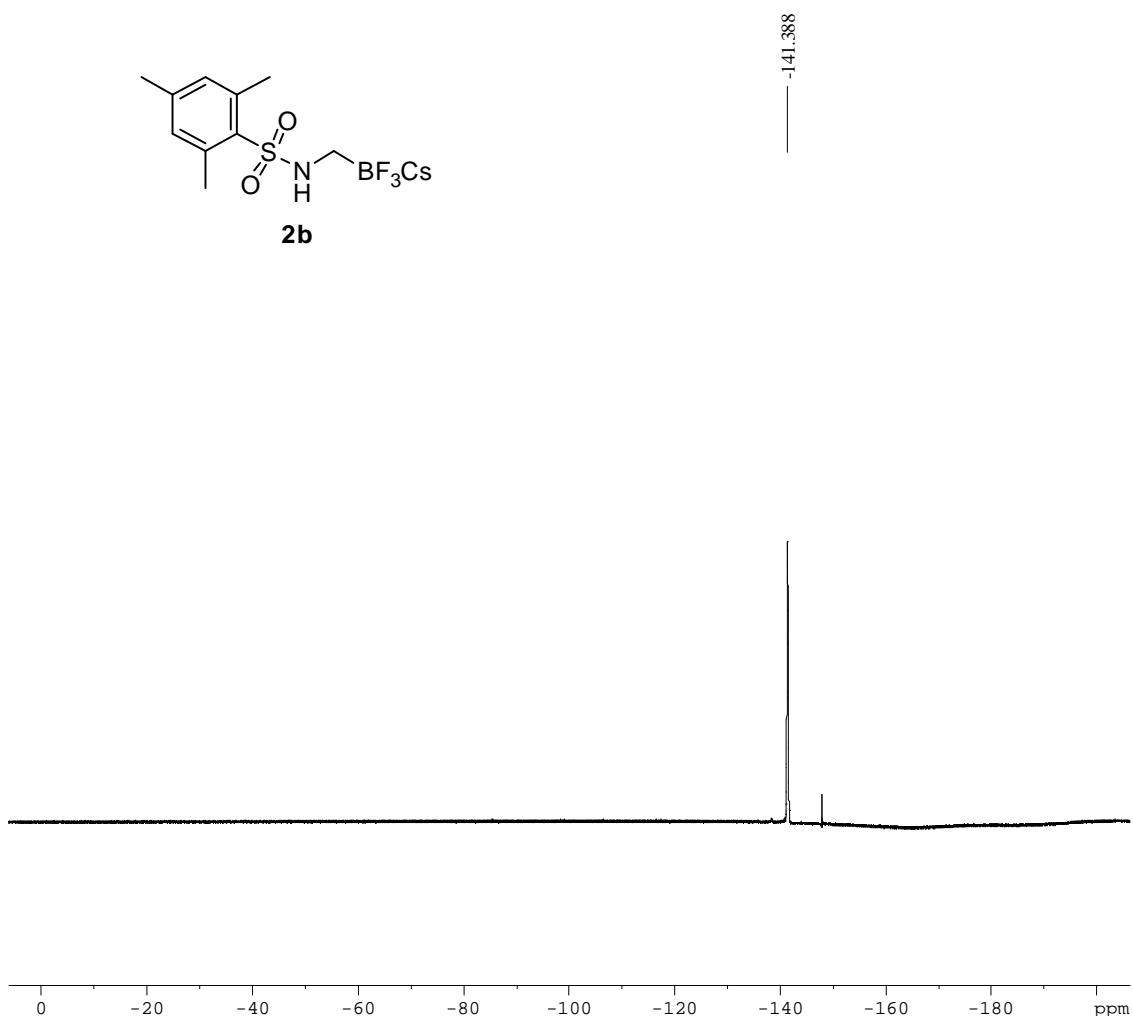
<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 300 MHz) spectrum of Cesium 2,4,6-Trimethylphenylsulfonamidomethyltrifluoroborate **2b**



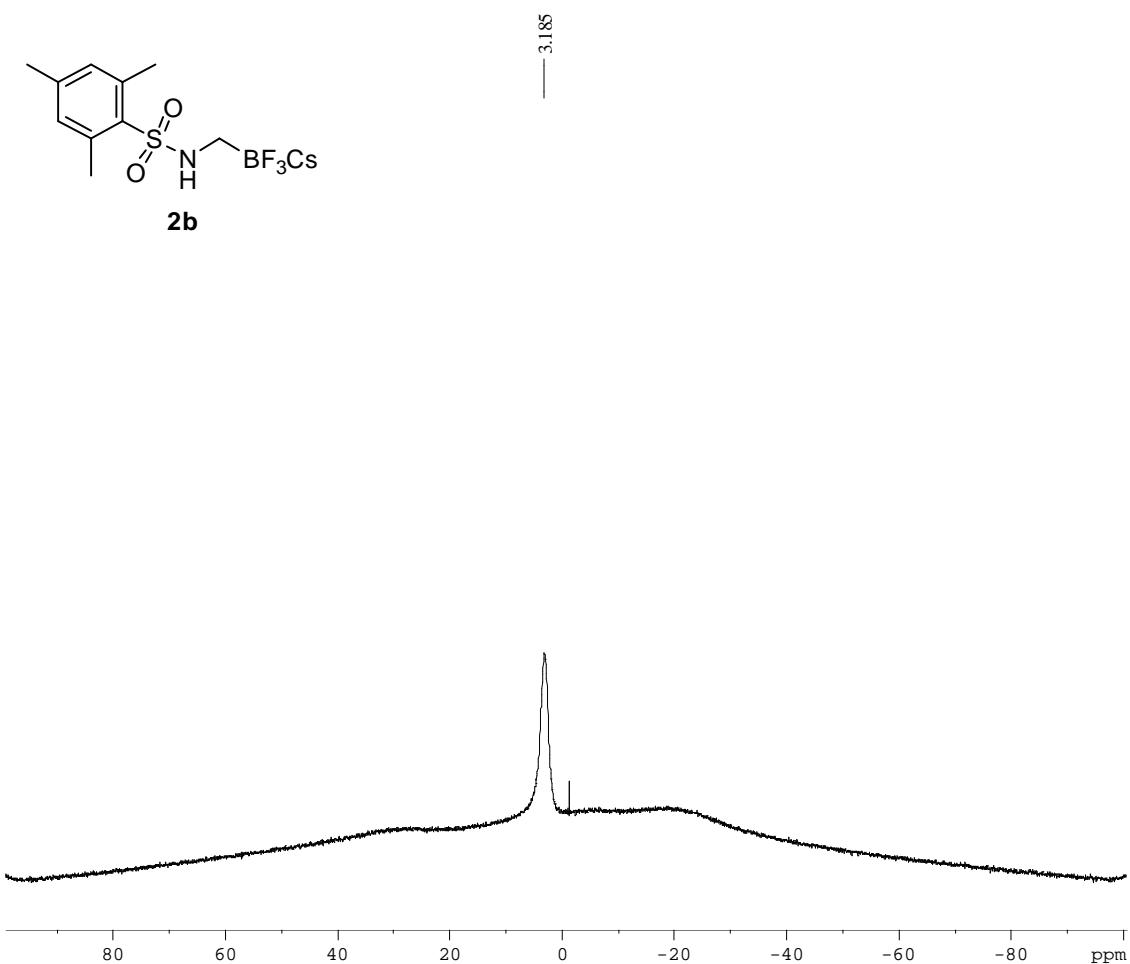
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Cesium 2,4,6-Trimethylphenylsulfonamidomethyltrifluoroborate **2b**



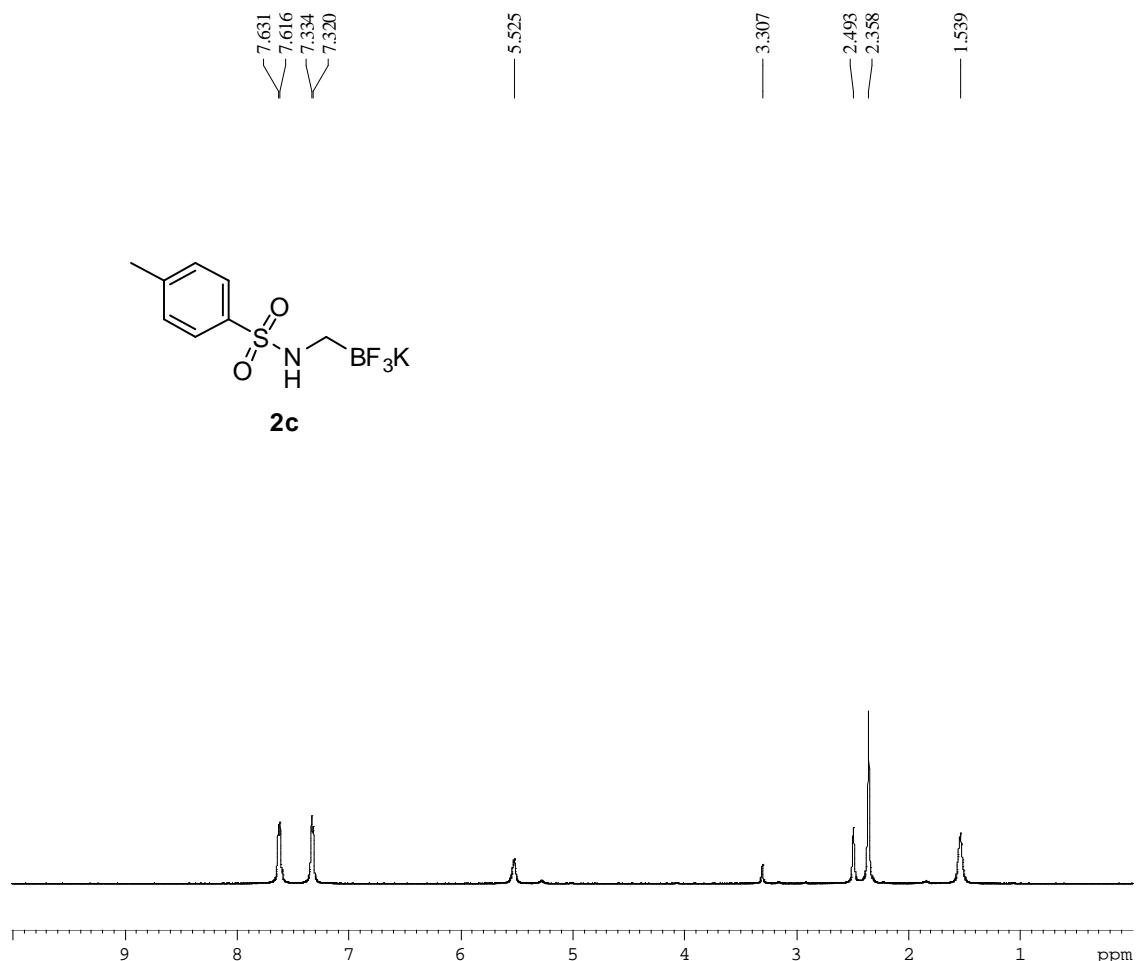
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Cesium 2,4,6-Trimethylphenylsulfonamidomethyltrifluoroborate **2b**



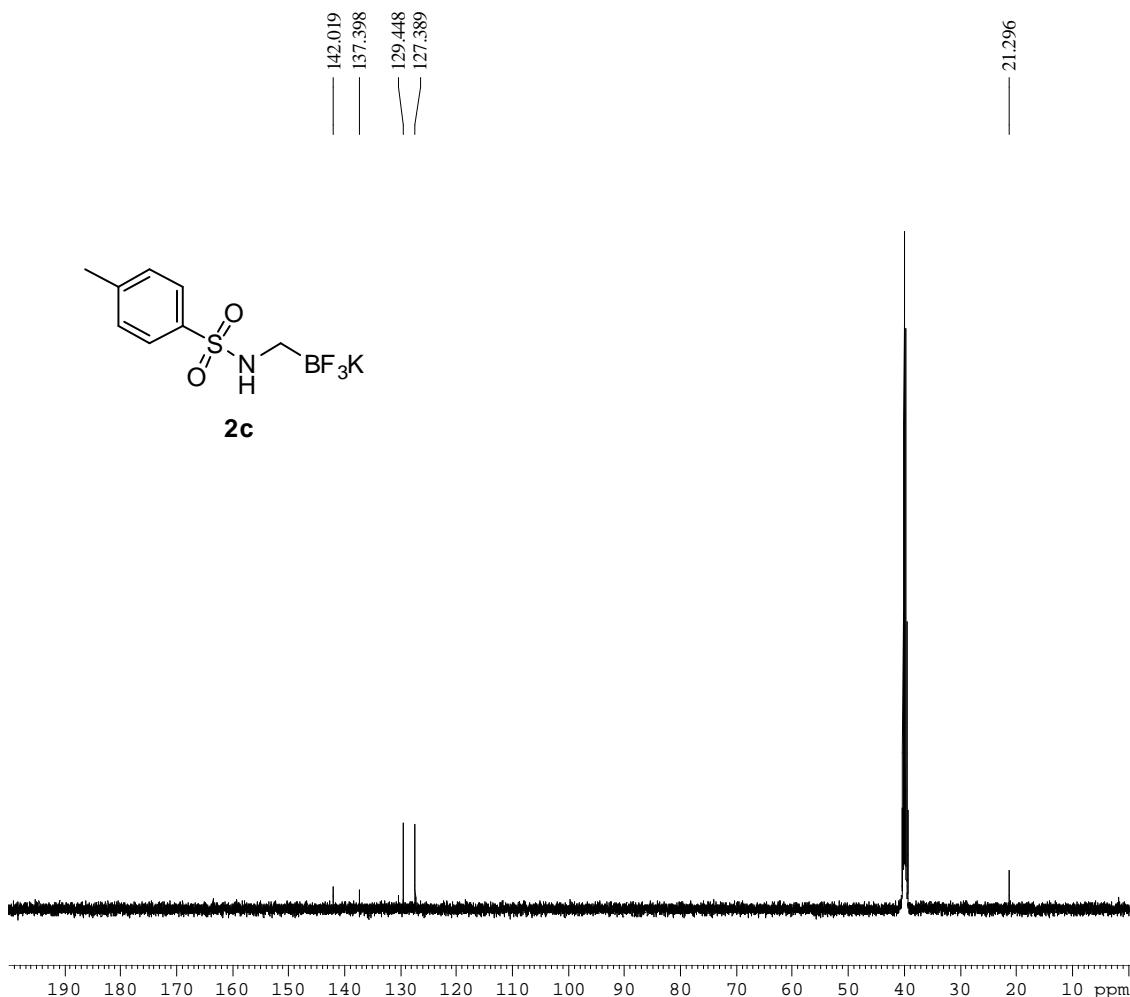
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) spectrum of Cesium 2,4,6-Trimethylphenylsulfonamidomethyltrifluoroborate **2b**



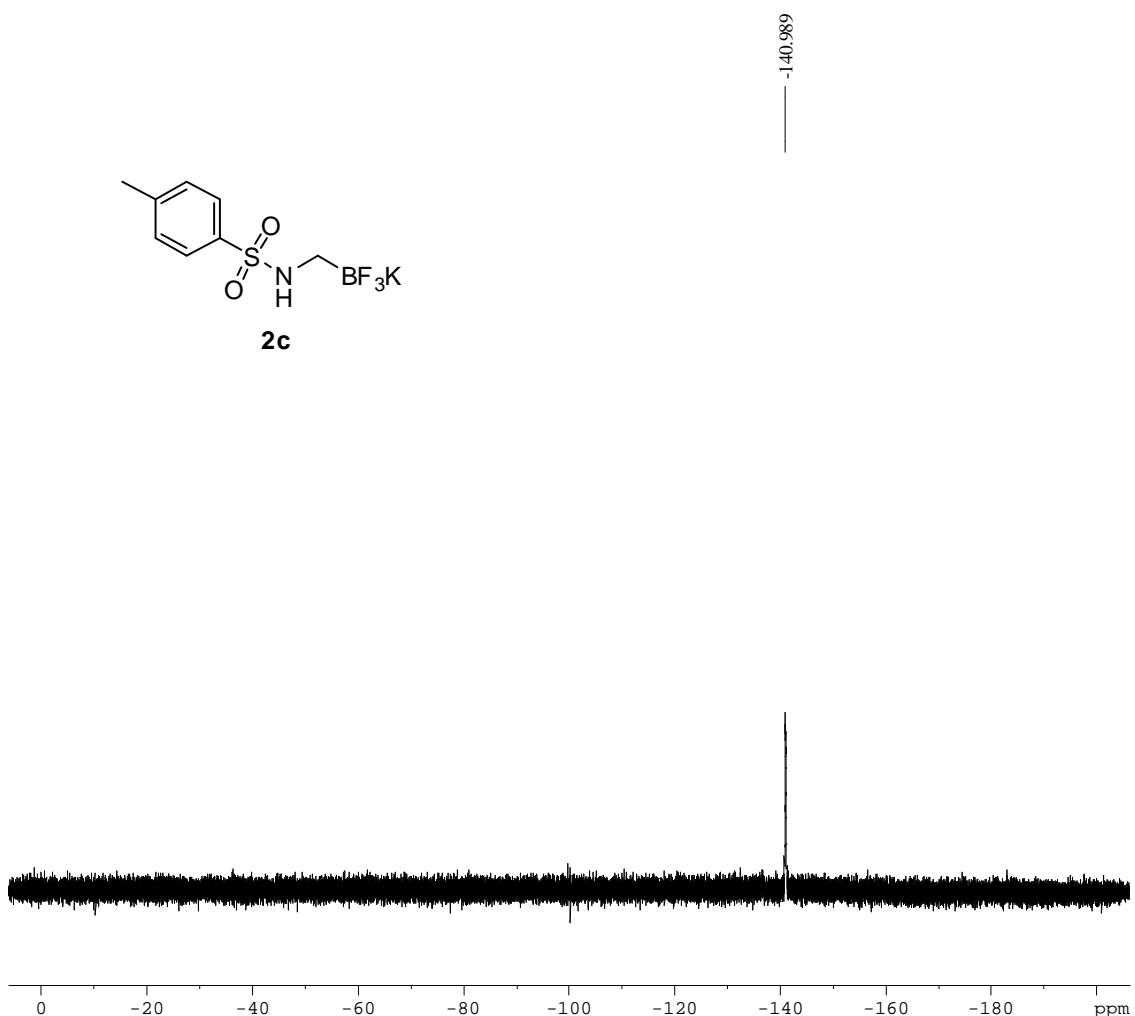
<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 500 MHz) spectrum of Potassium 4-Methylphenylsulfonamidomethyltrifluoroborate **2c**



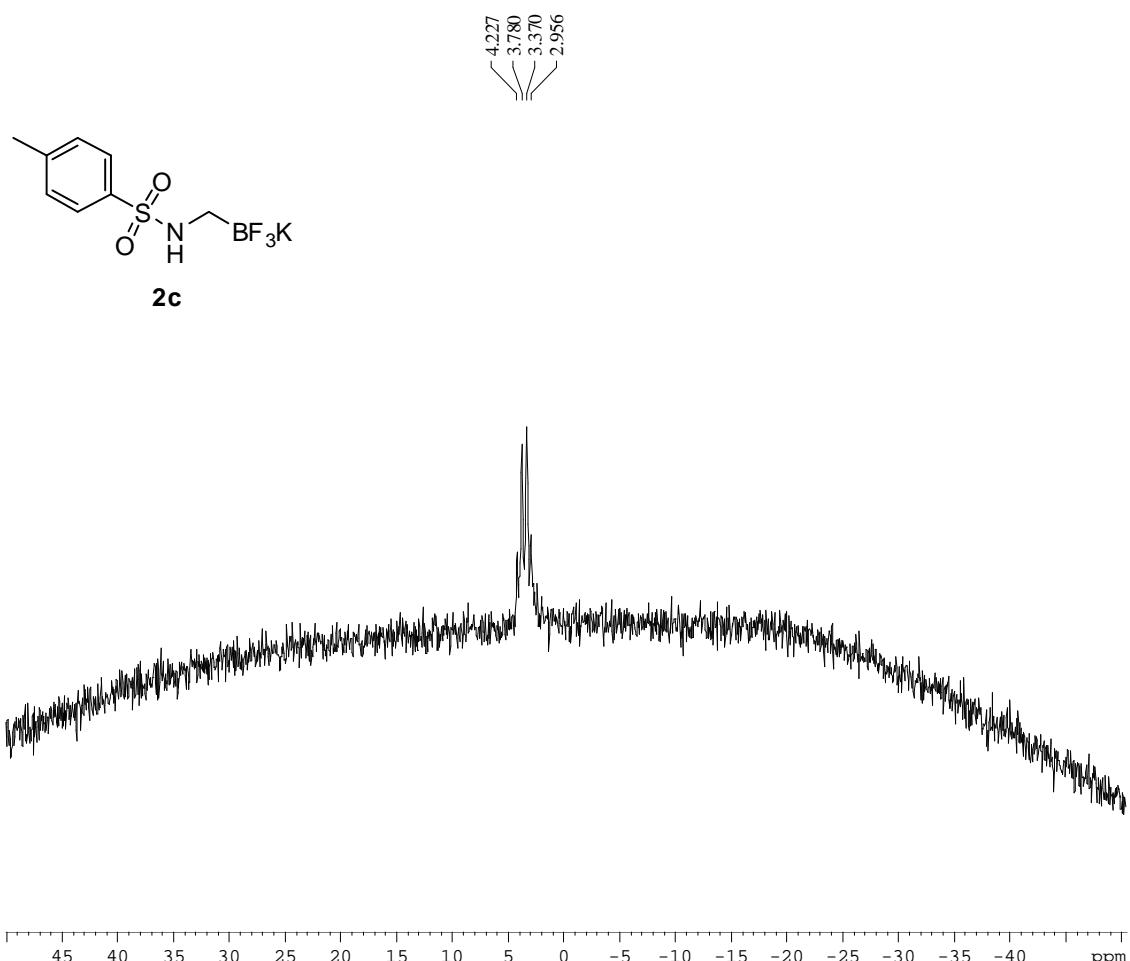
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) Potassium 4-Methylphenylsulfonamidomethyltrifluoroborate **2c**



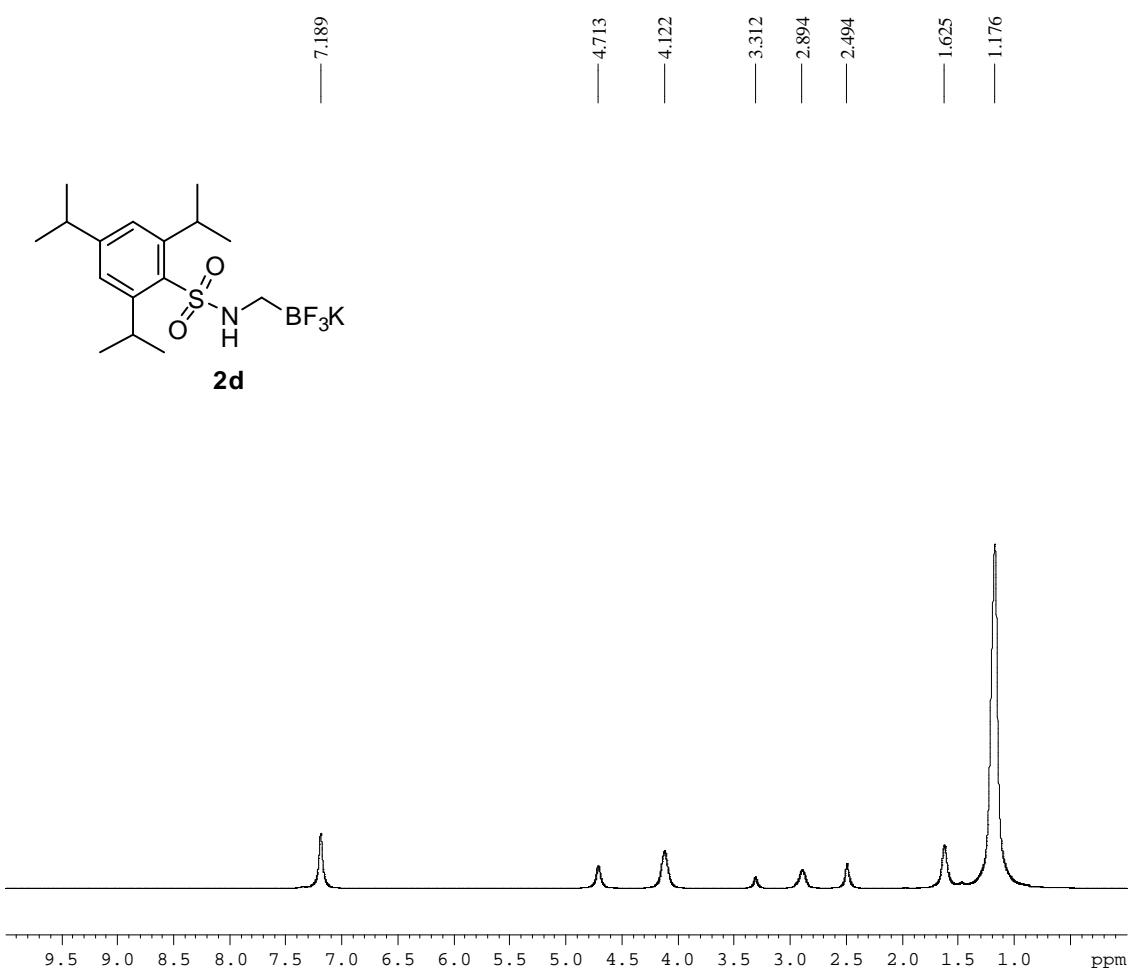
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) Potassium 4-Methylphenylsulfonamidomethyltrifluoroborate **2c**



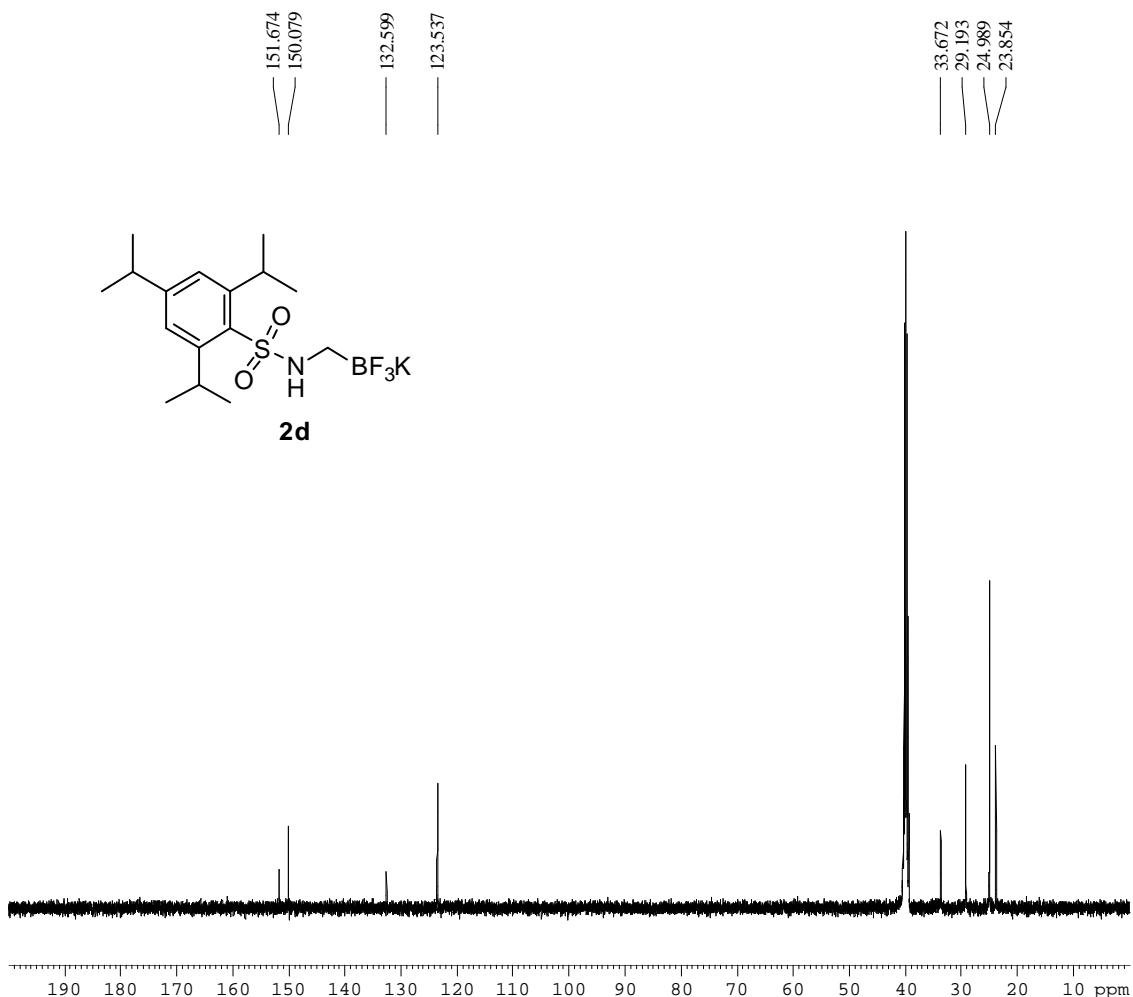
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) Potassium 4-Methylphenylsulfonamidomethyltrifluoroborate **2c**



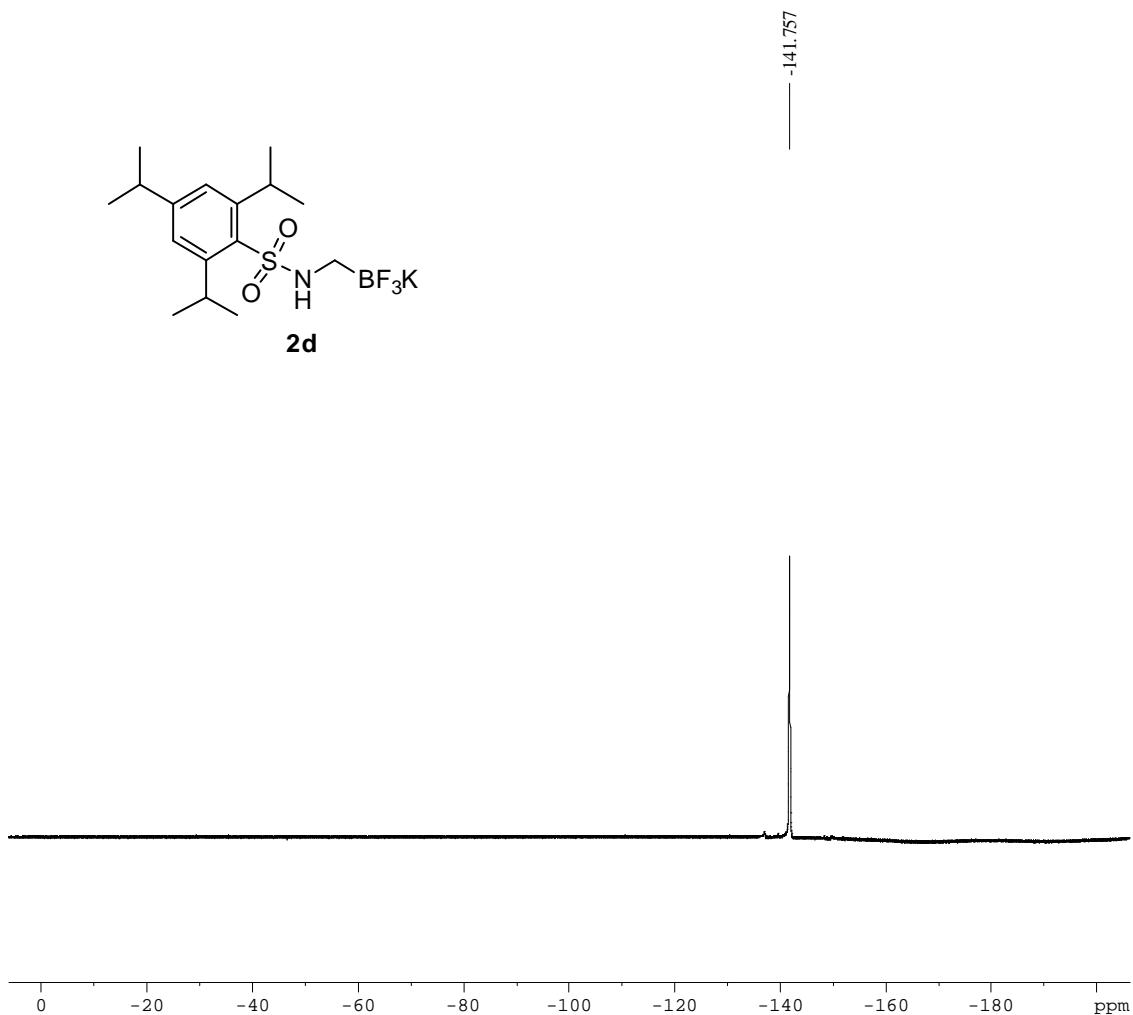
<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz) spectrum Potassium ((2,4,6-Triisopropylphenylsulfonamido)methyl)trifluoroborate **2d**



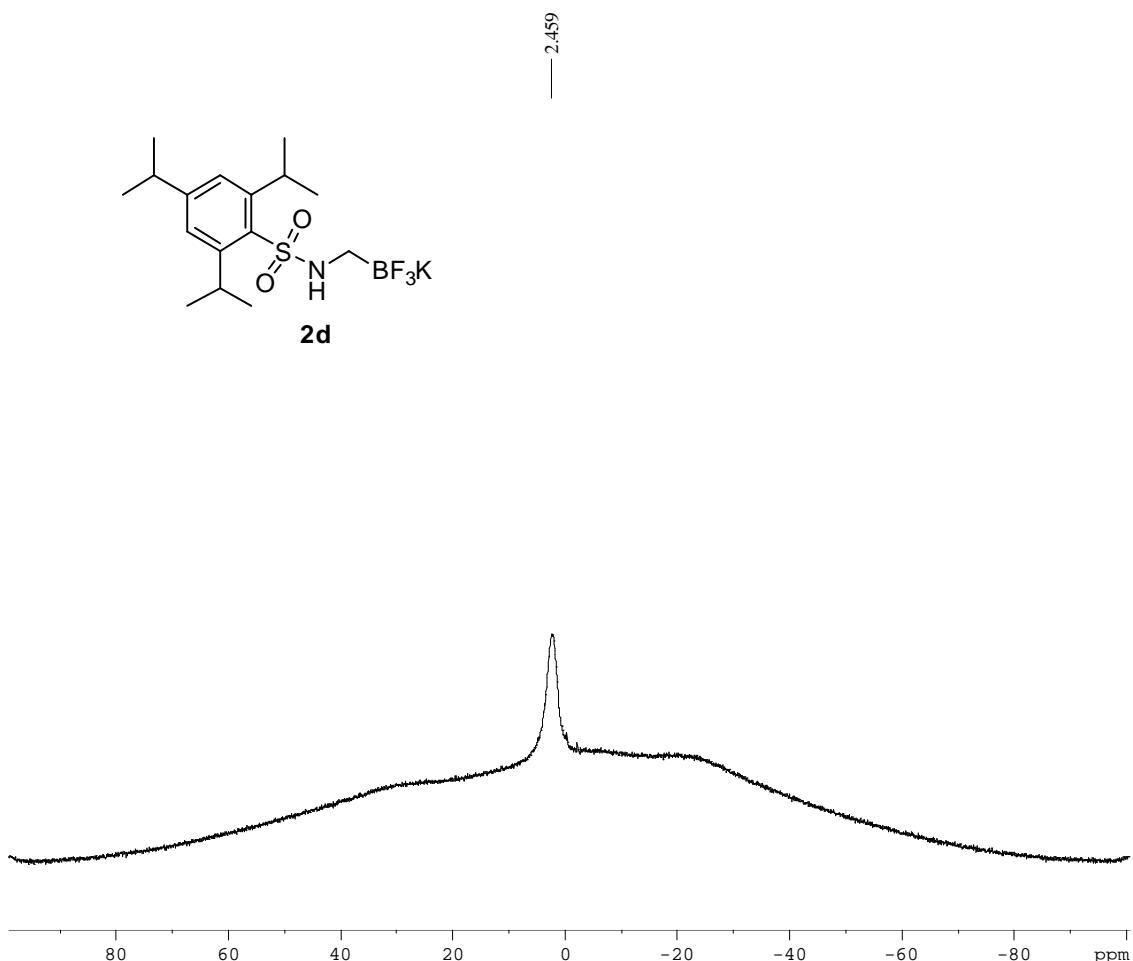
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium ((2,4,6-Triisopropylphenylsulfonamido)methyl)trifluoroborate **2d**



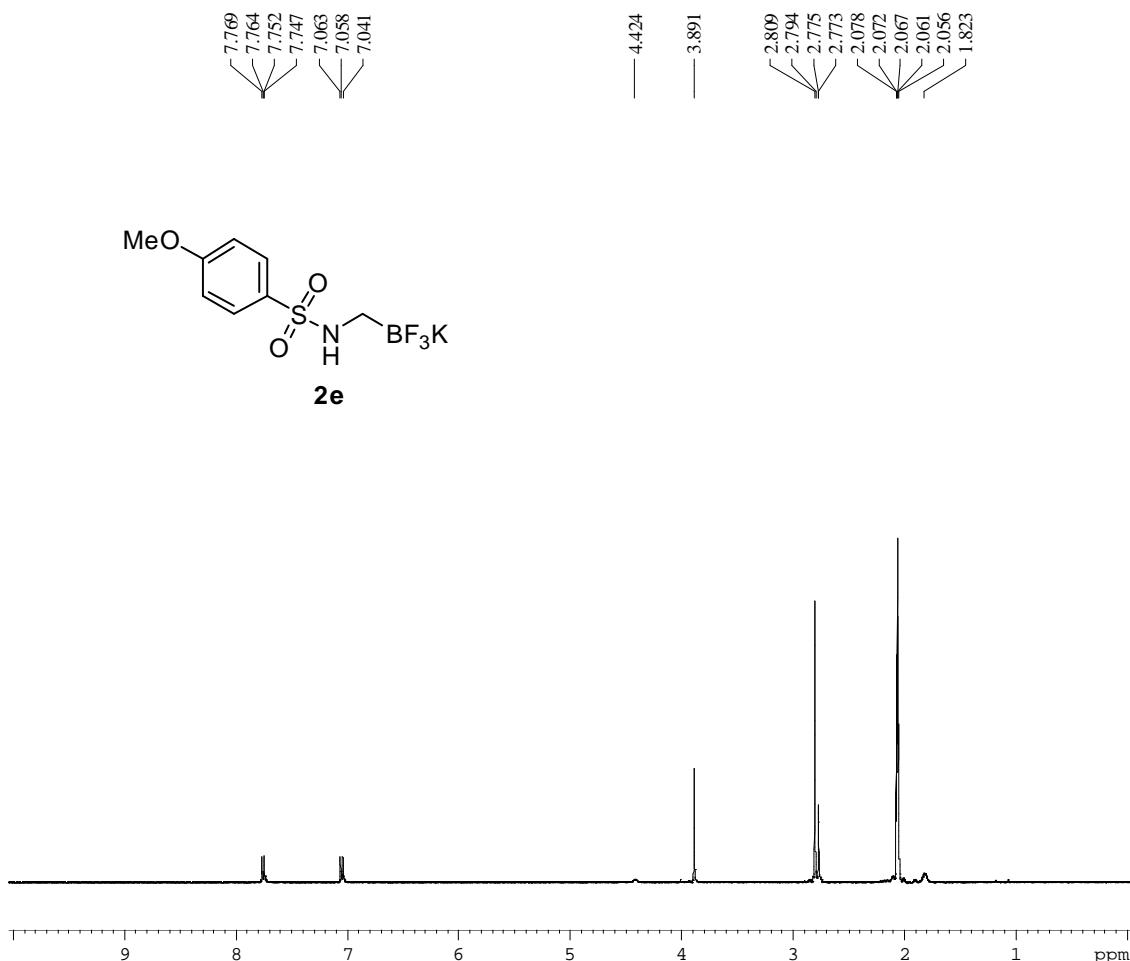
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium ((2,4,6-Triisopropylphenylsulfonamido)methyl)trifluoroborate **2d**



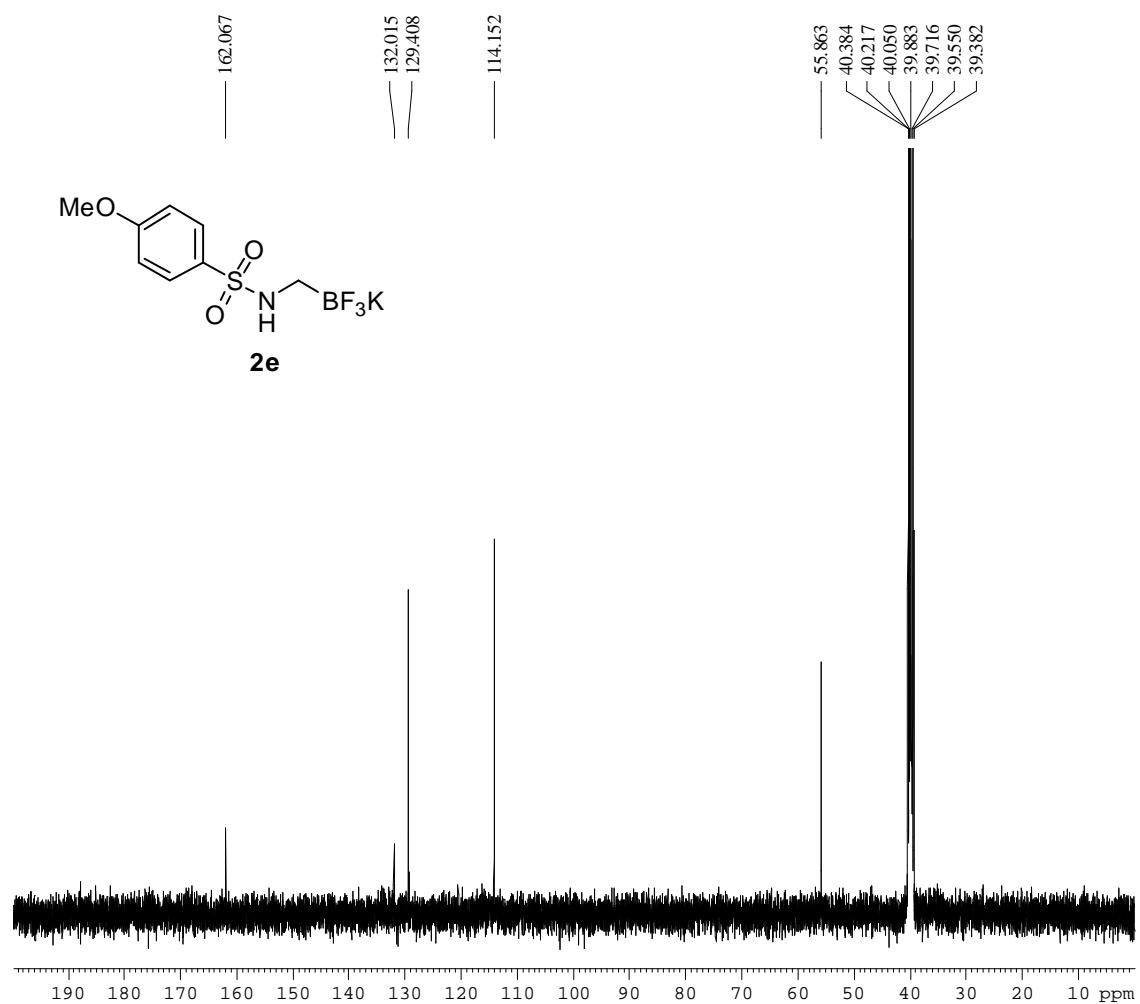
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) spectrum of Potassium ((2,4,6-Triisopropylphenylsulfonamido)methyl)trifluoroborate **2d**



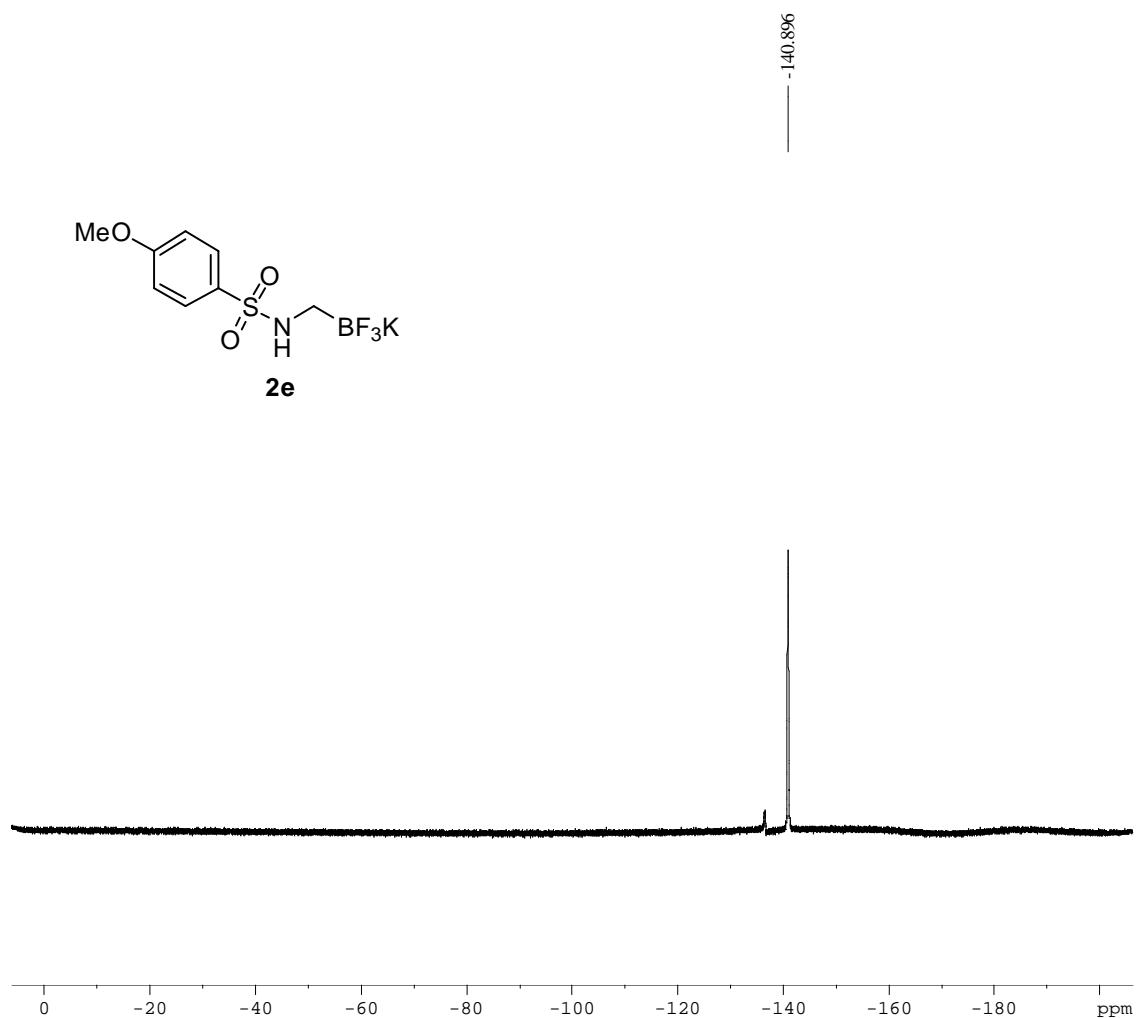
<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz) spectrum of Potassium 4-Methoxyphenylsulfonamidomethyltrifluoroborate **2e**



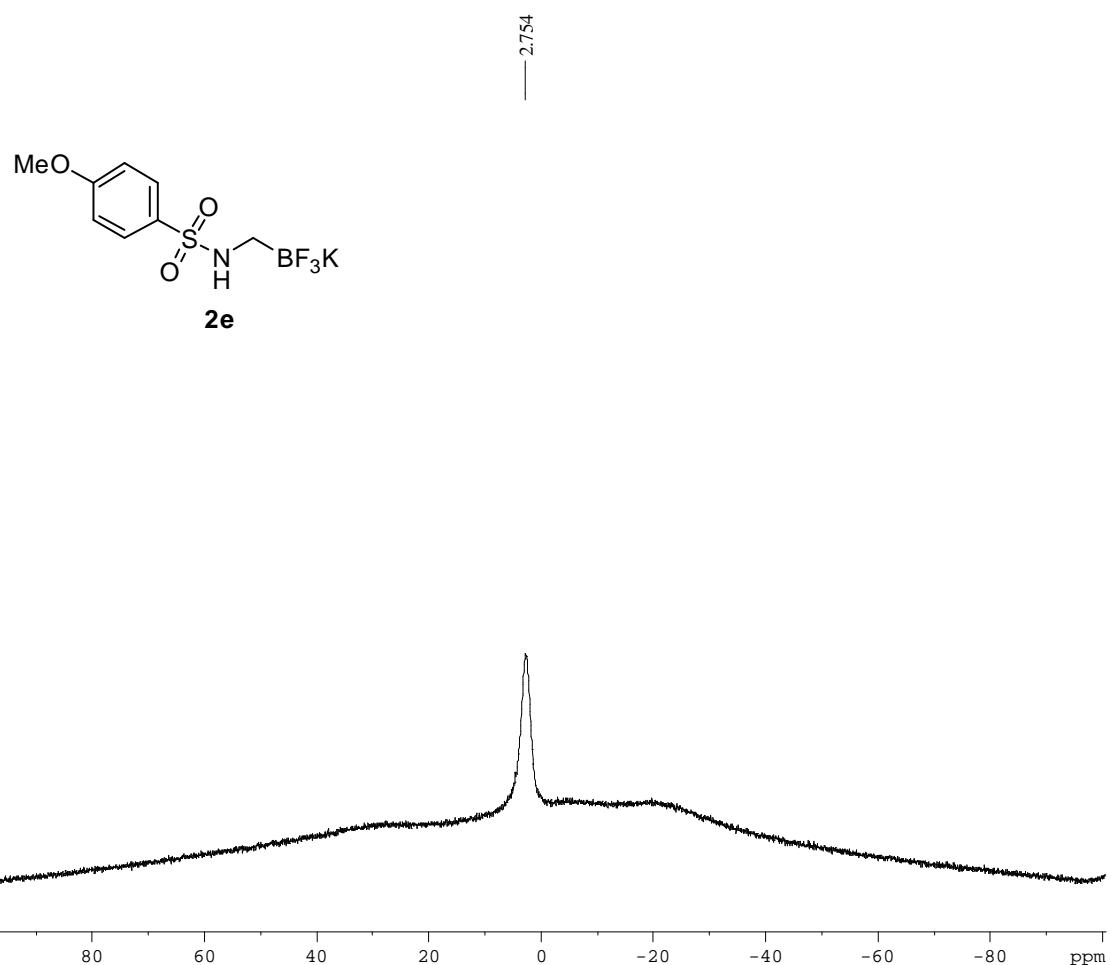
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 4-Methoxyphenylsulfonamidomethyltrifluoroborate **2e**



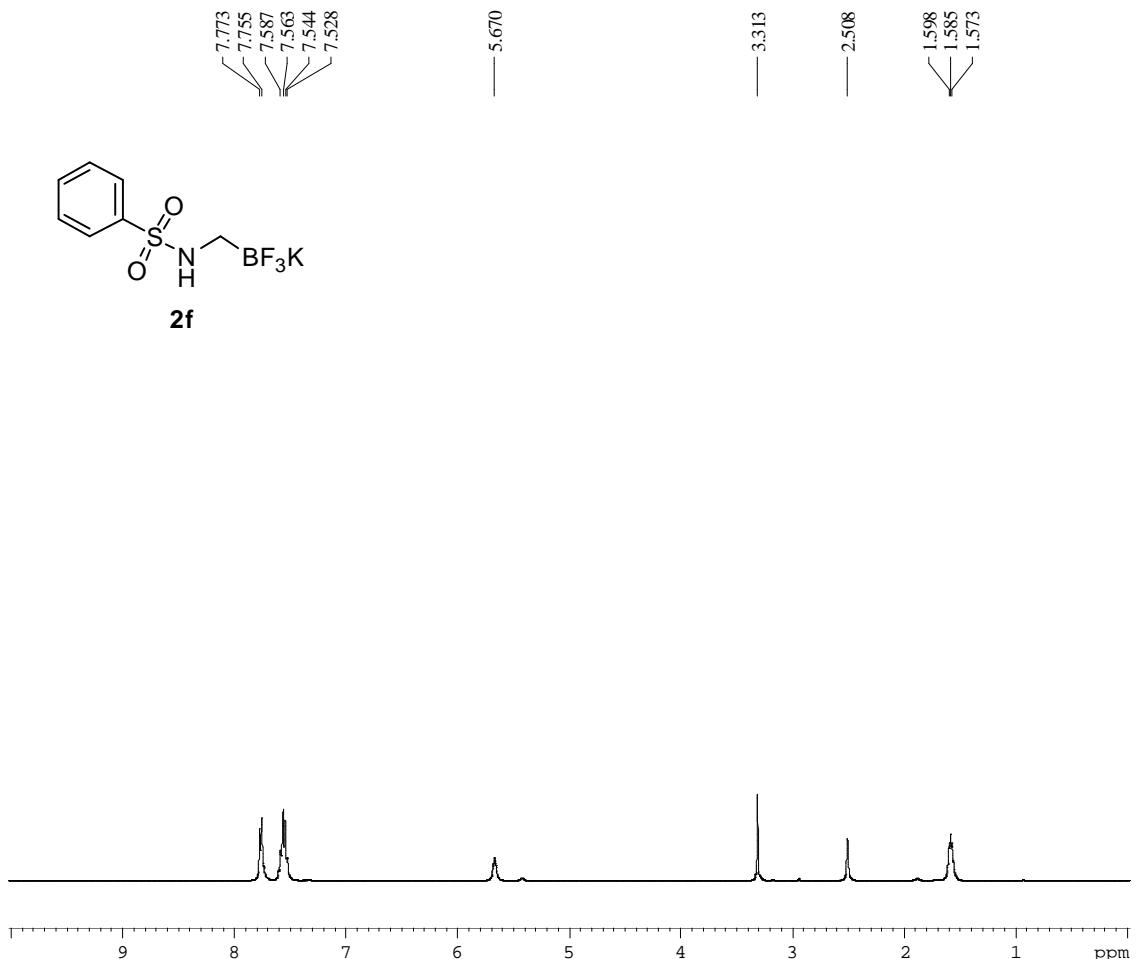
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium 4-Methoxyphenylsulfonamidomethyltrifluoroborate **2e**



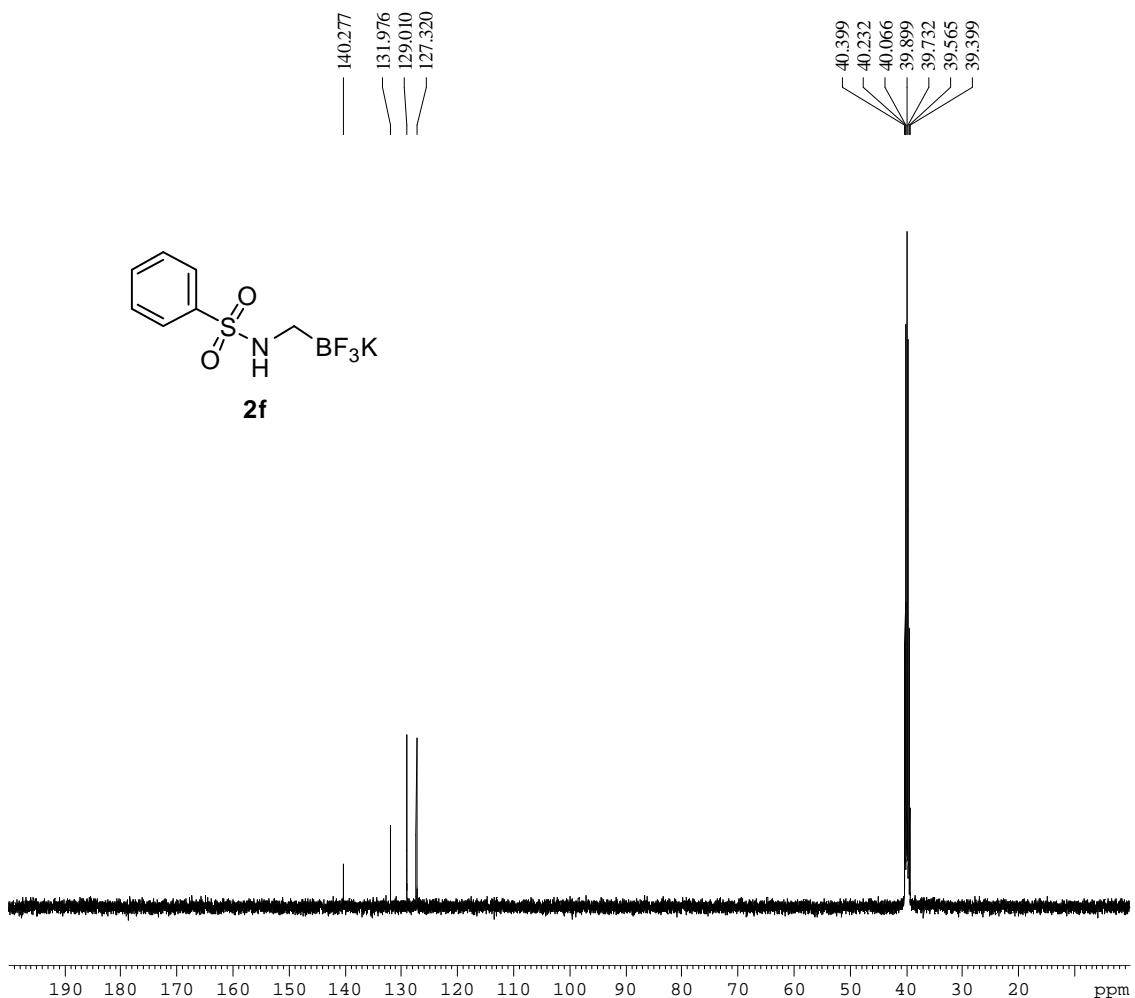
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) spectrum of Potassium 4-Methoxyphenylsulfonamidomethyltrifluoroborate **2e**



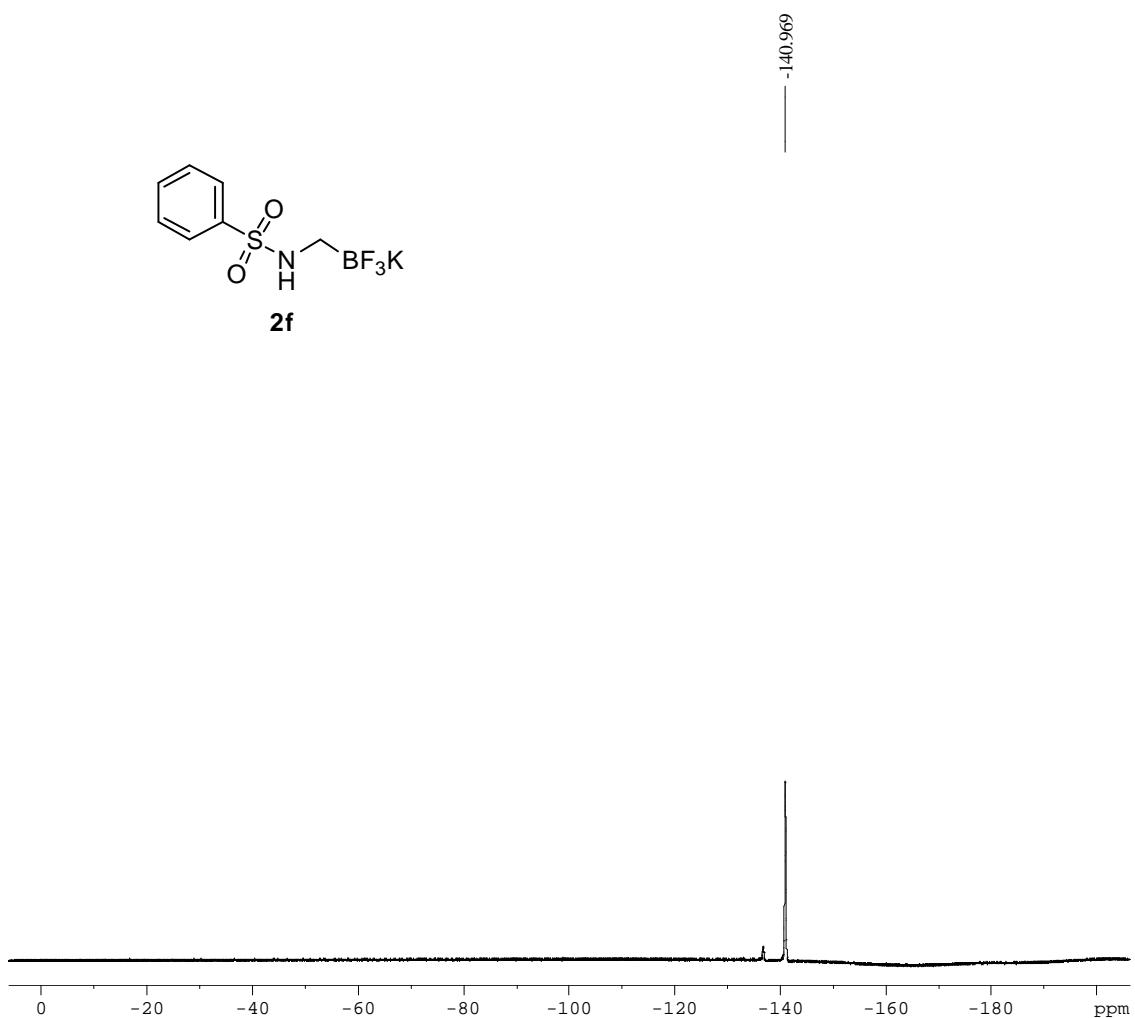
<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz) spectrum of Potassium Phenylsulfonamidomethyltrifluoroborate **2f**



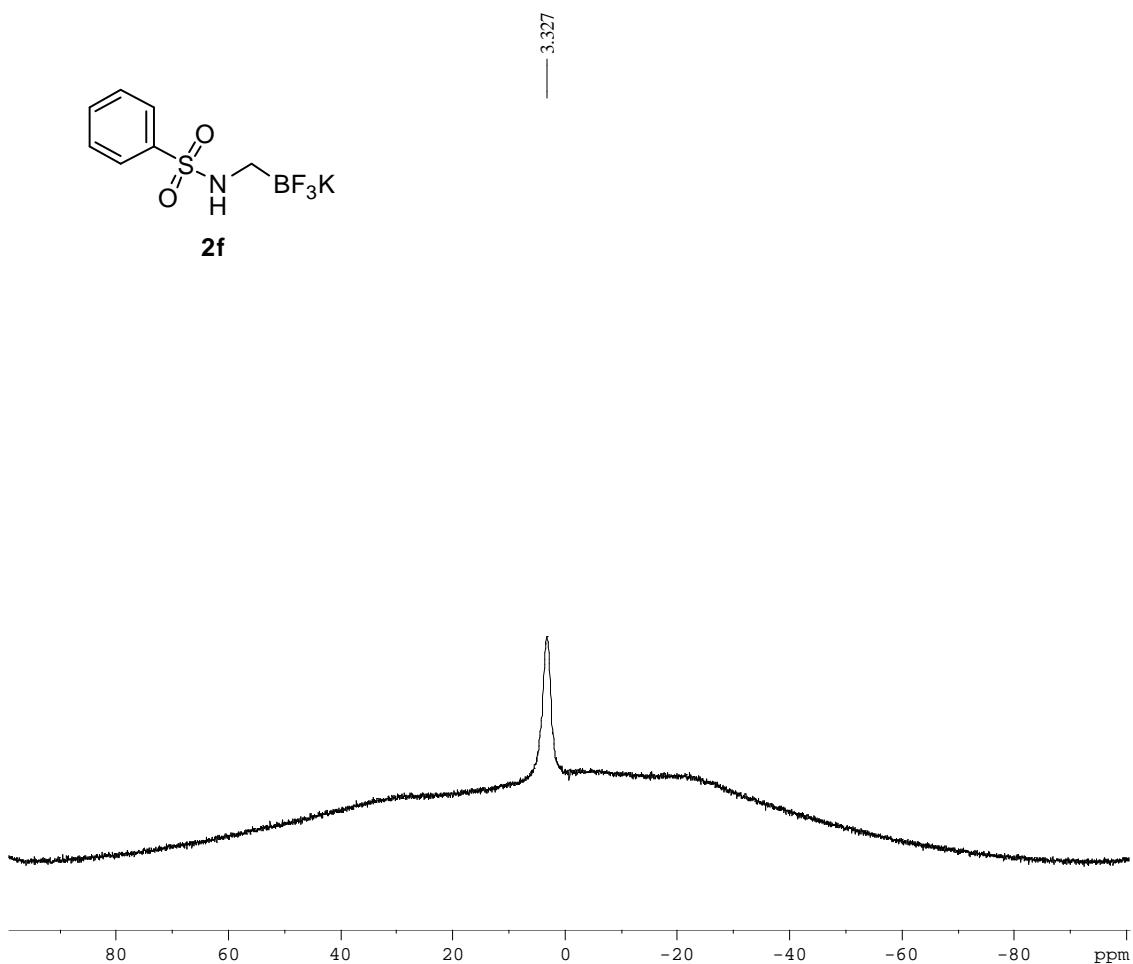
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium Phenylsulfonamidomethyltrifluoroborate **2f**



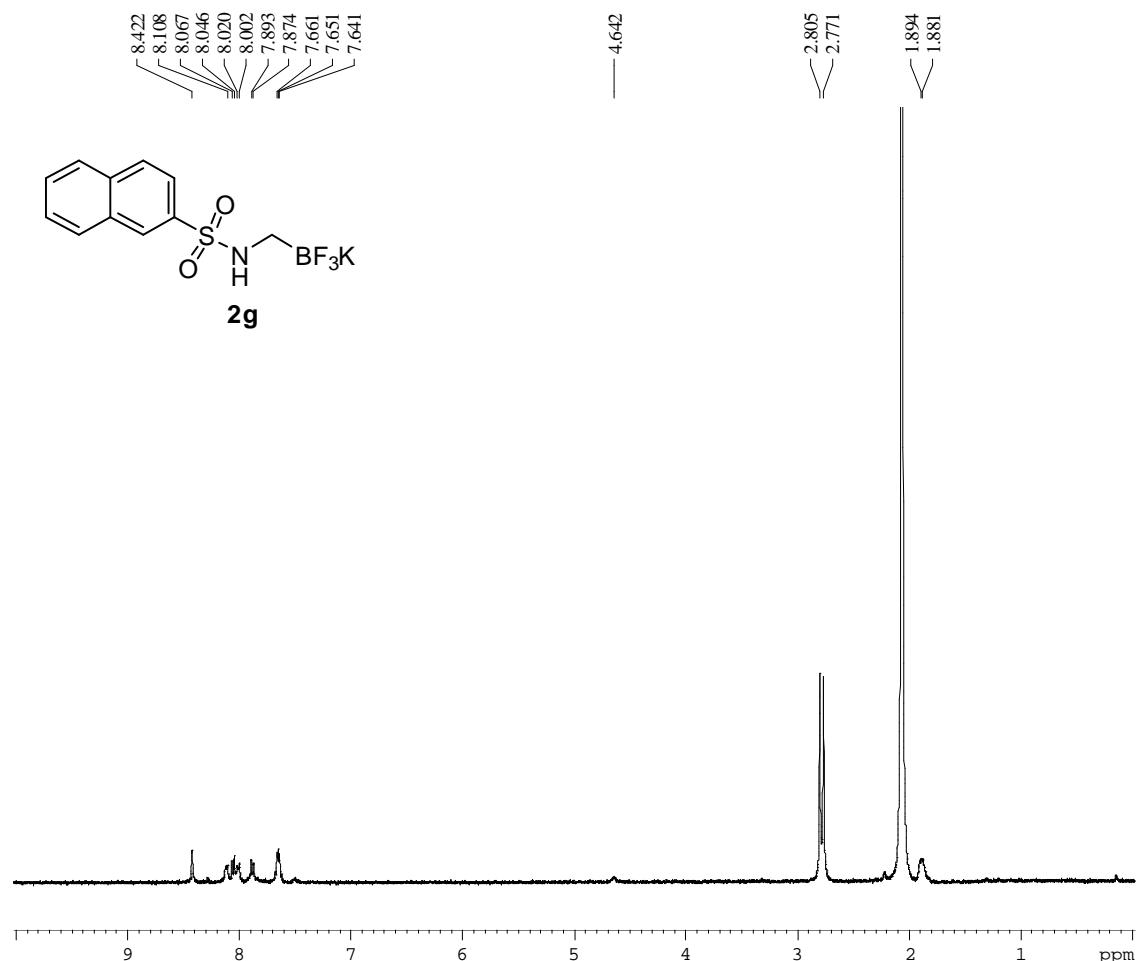
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium Phenylsulfonamidomethyltrifluoroborate **2f**



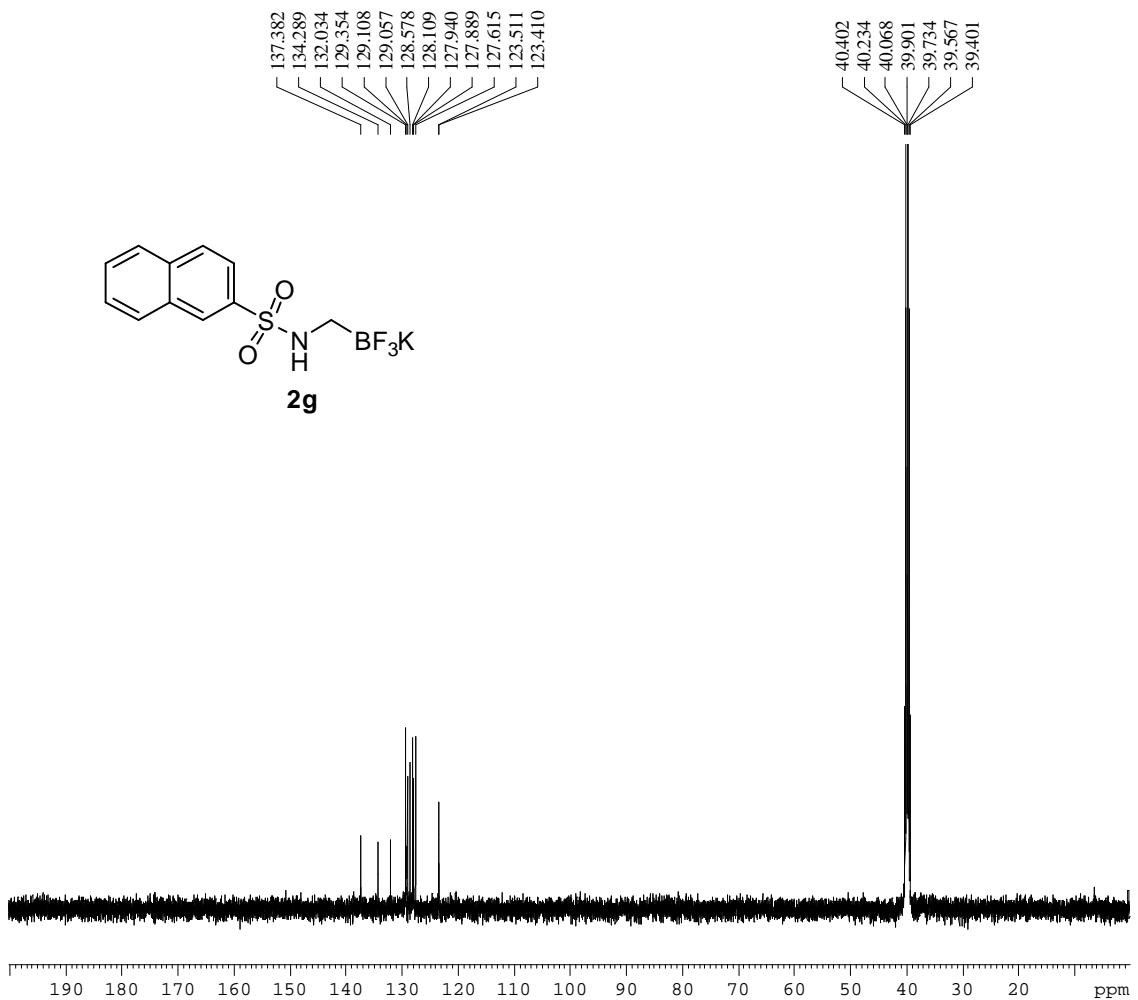
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4MHz) spectrum of Potassium Phenylsulfonamidomethyltrifluoroborate **2f**



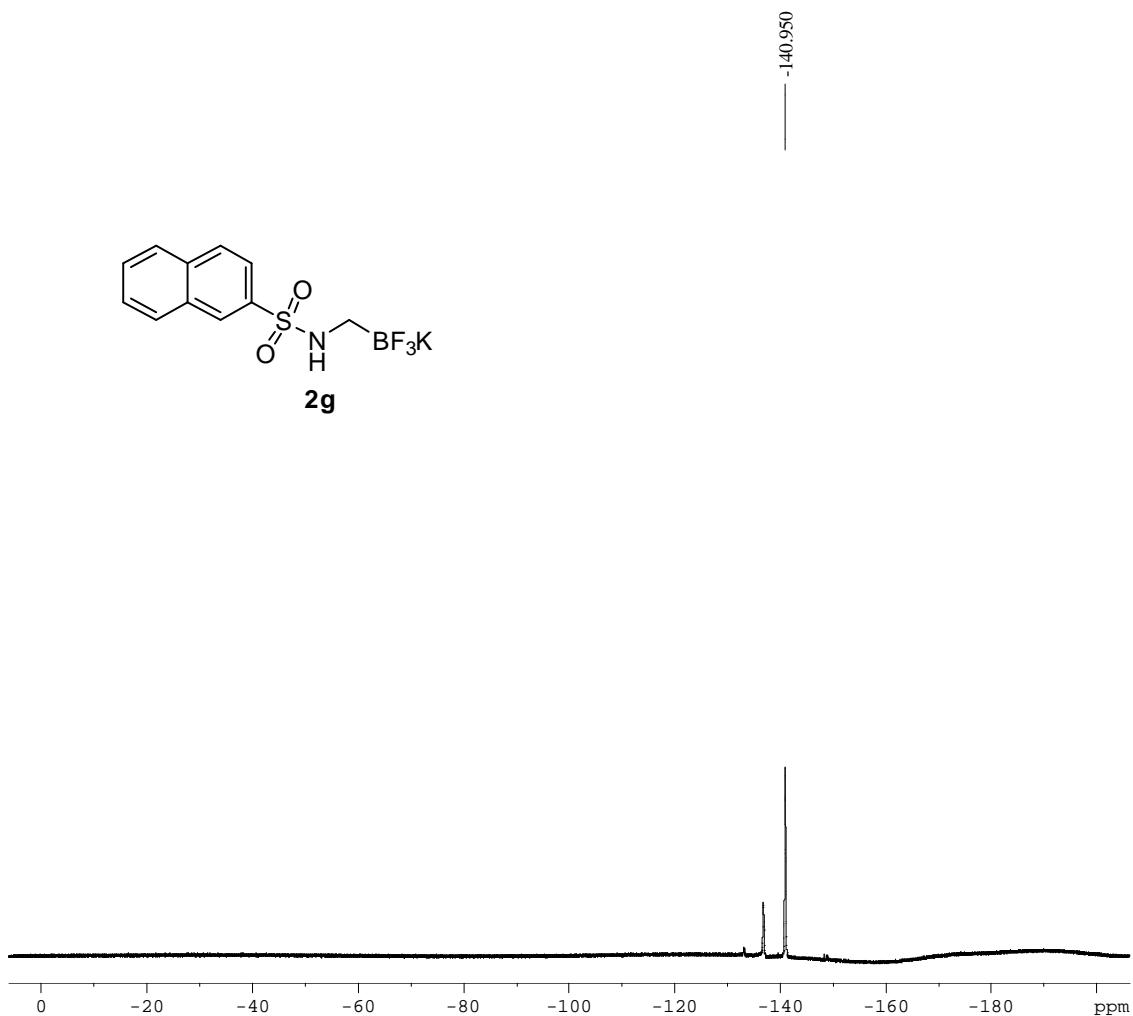
<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz) spectrum of Potassium 2-Naphthylsulfonamidomethyltrifluoroborate **2g**



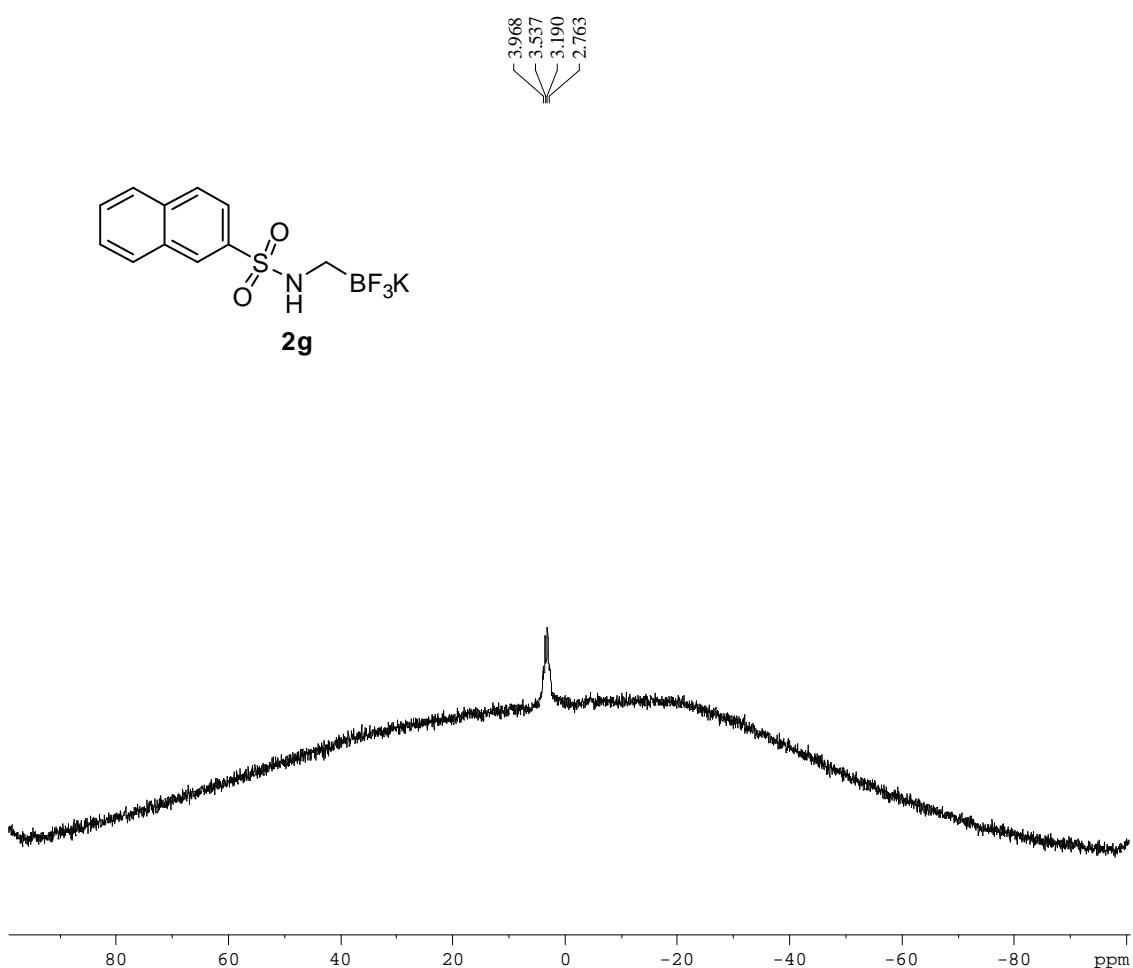
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8MHz) spectrum of Potassium 2-Naphthylsulfonamidomethyltrifluoroborate **2g**



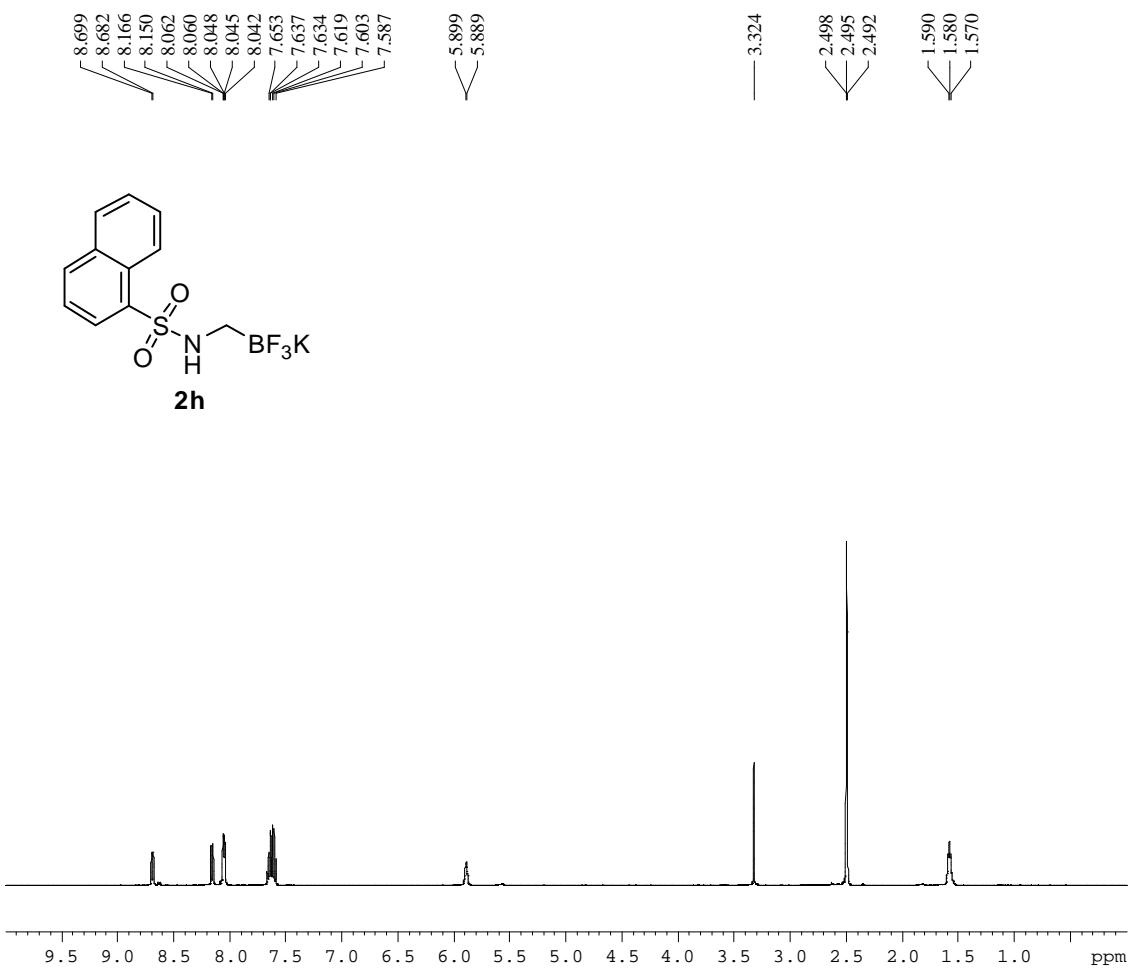
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium 2-Naphthylsulfonamidomethyltrifluoroborate **2g**



<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) spectrum of Potassium 2-Naphthylsulfonamidomethyltrifluoroborate **2g**

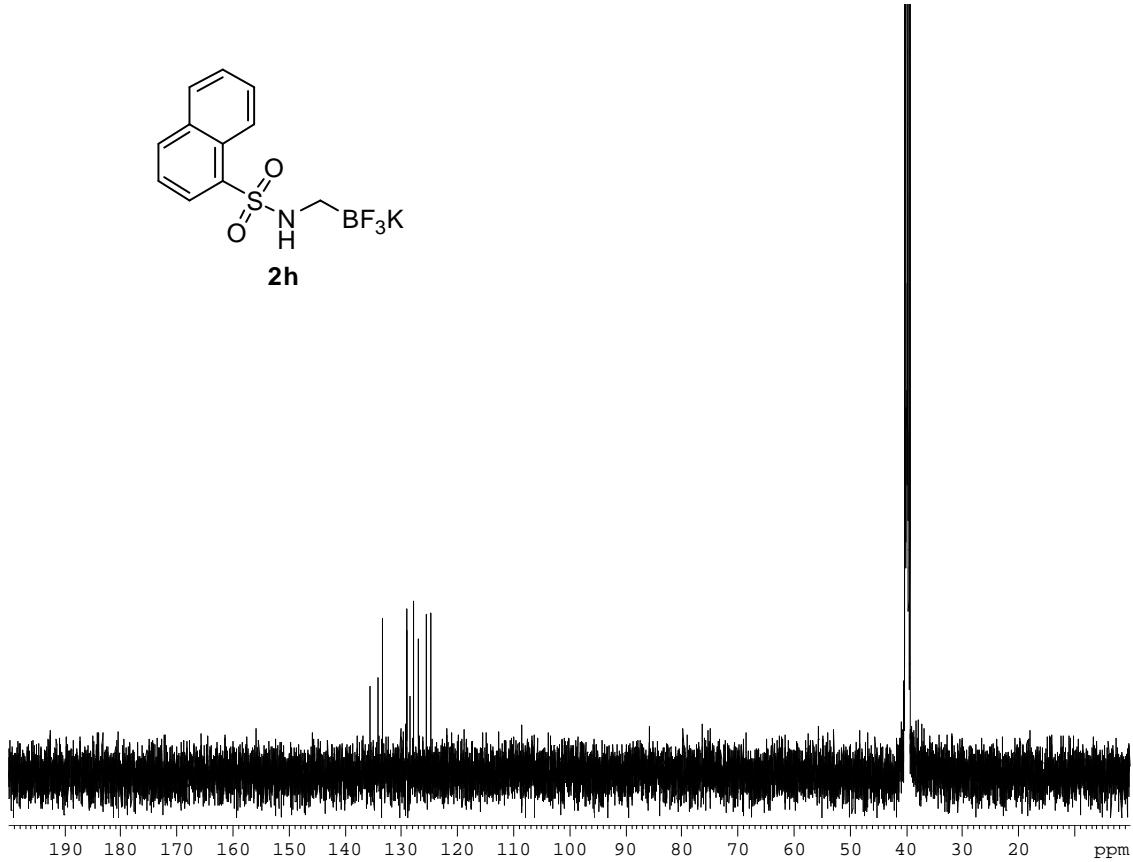
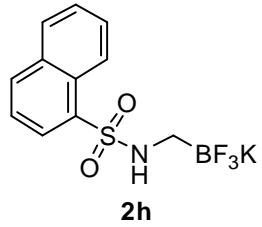


<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 400 MHz) spectrum of Potassium 1-Naphthylsulfonamidomethyltrifluoroborate **2h**

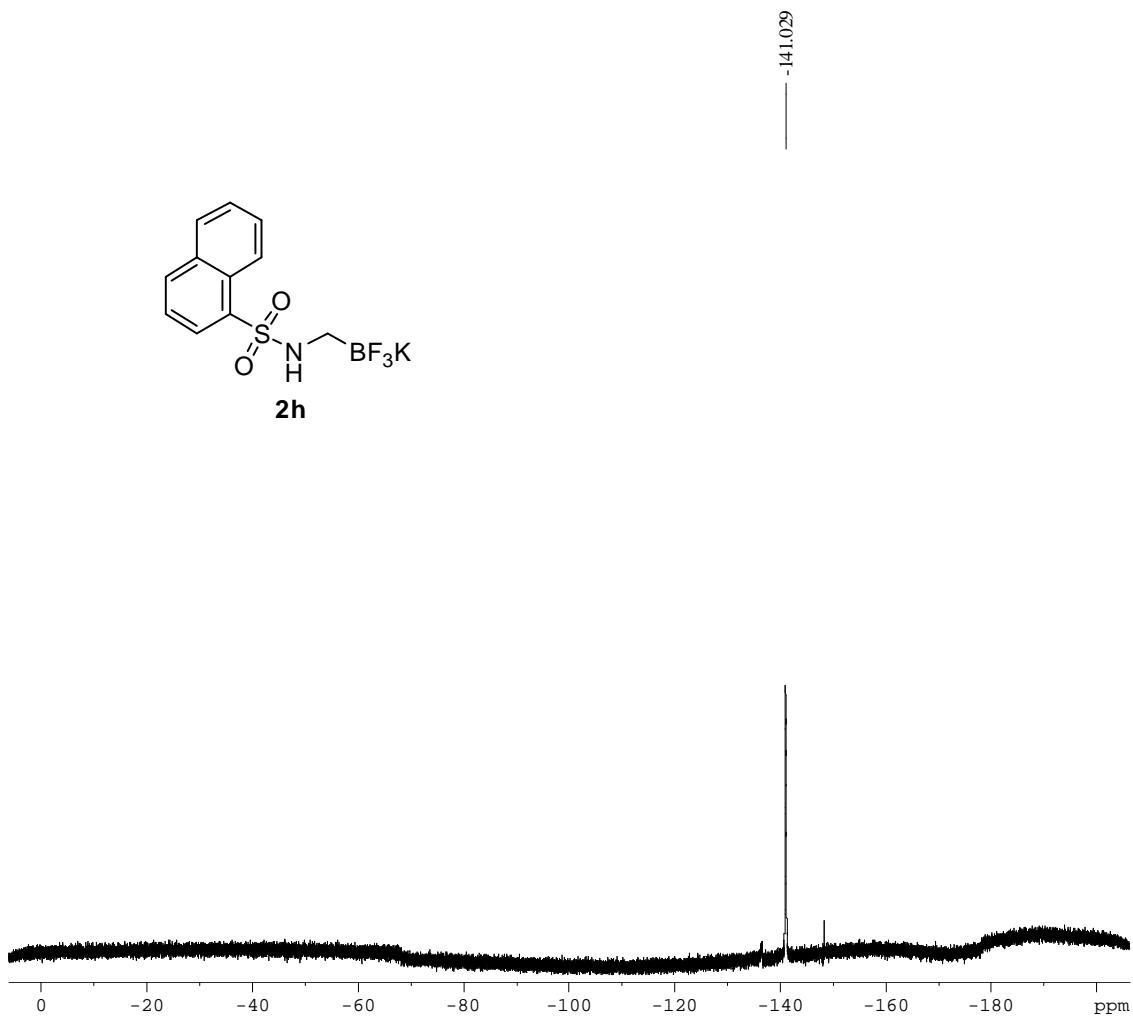


<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 1-Naphthylsulfonamidomethyltrifluoroborate **2h**

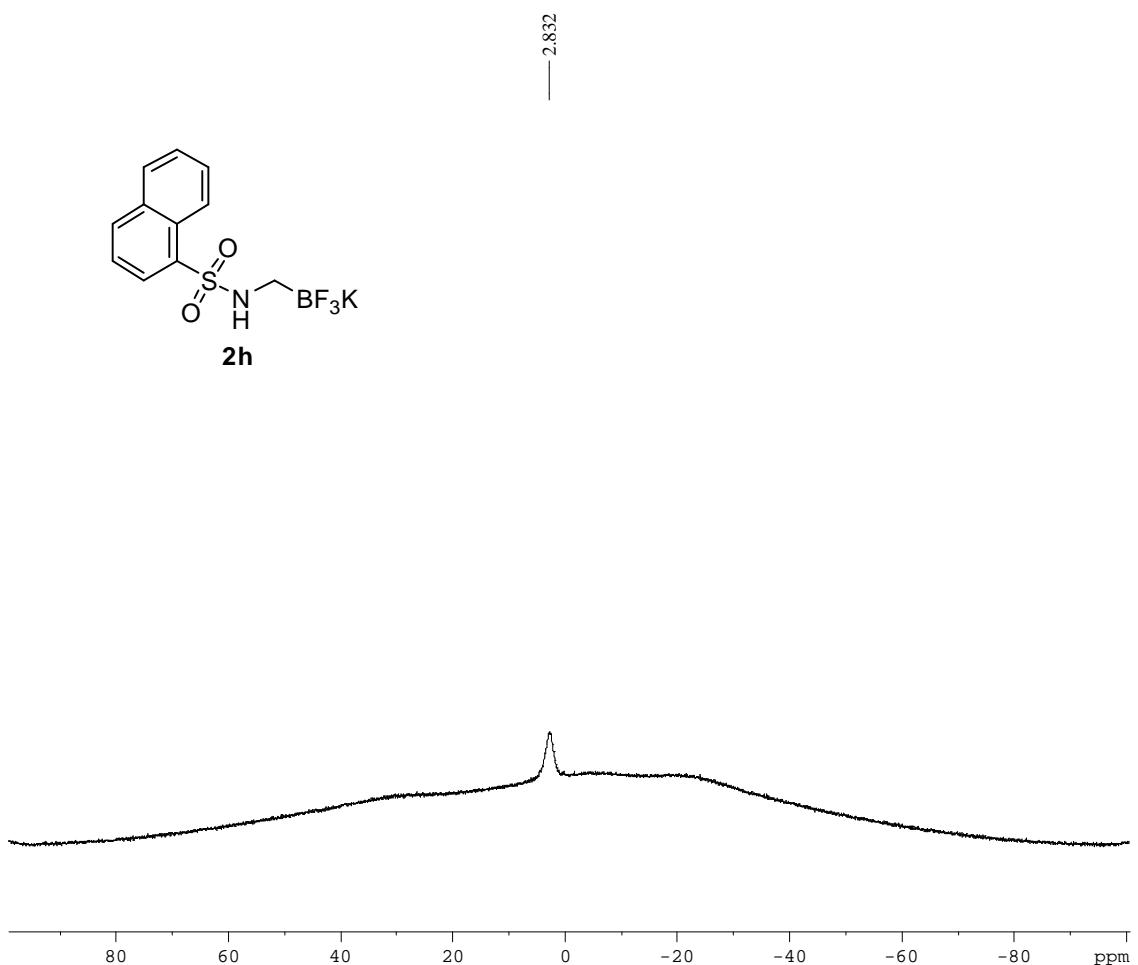
135.522  
134.189  
133.265  
129.058  
128.994  
127.854  
126.905  
125.560  
124.652



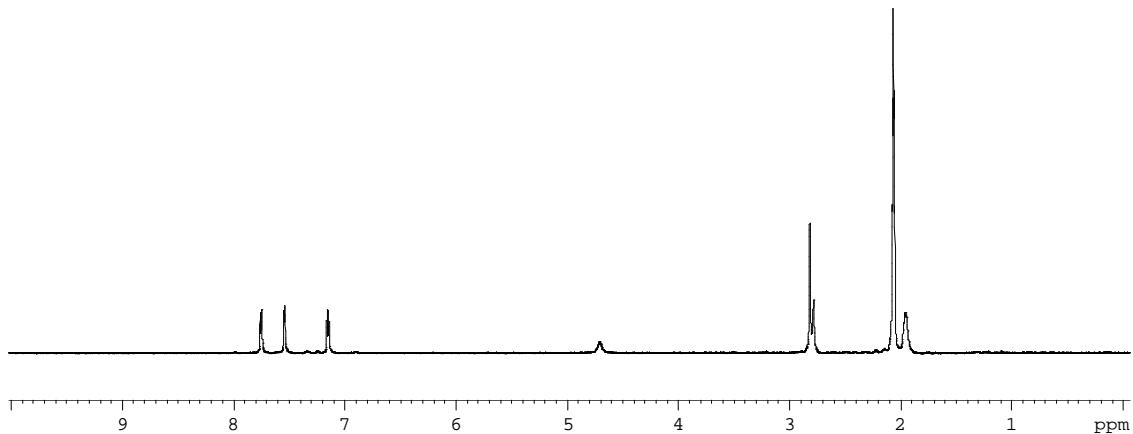
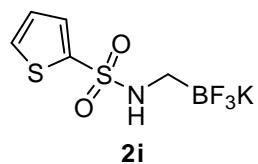
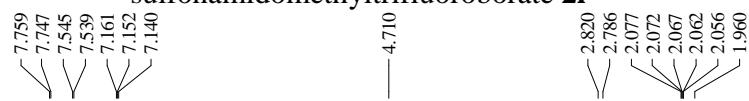
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium 1-Naphthylsulfonamidomethyltrifluoroborate **2h**



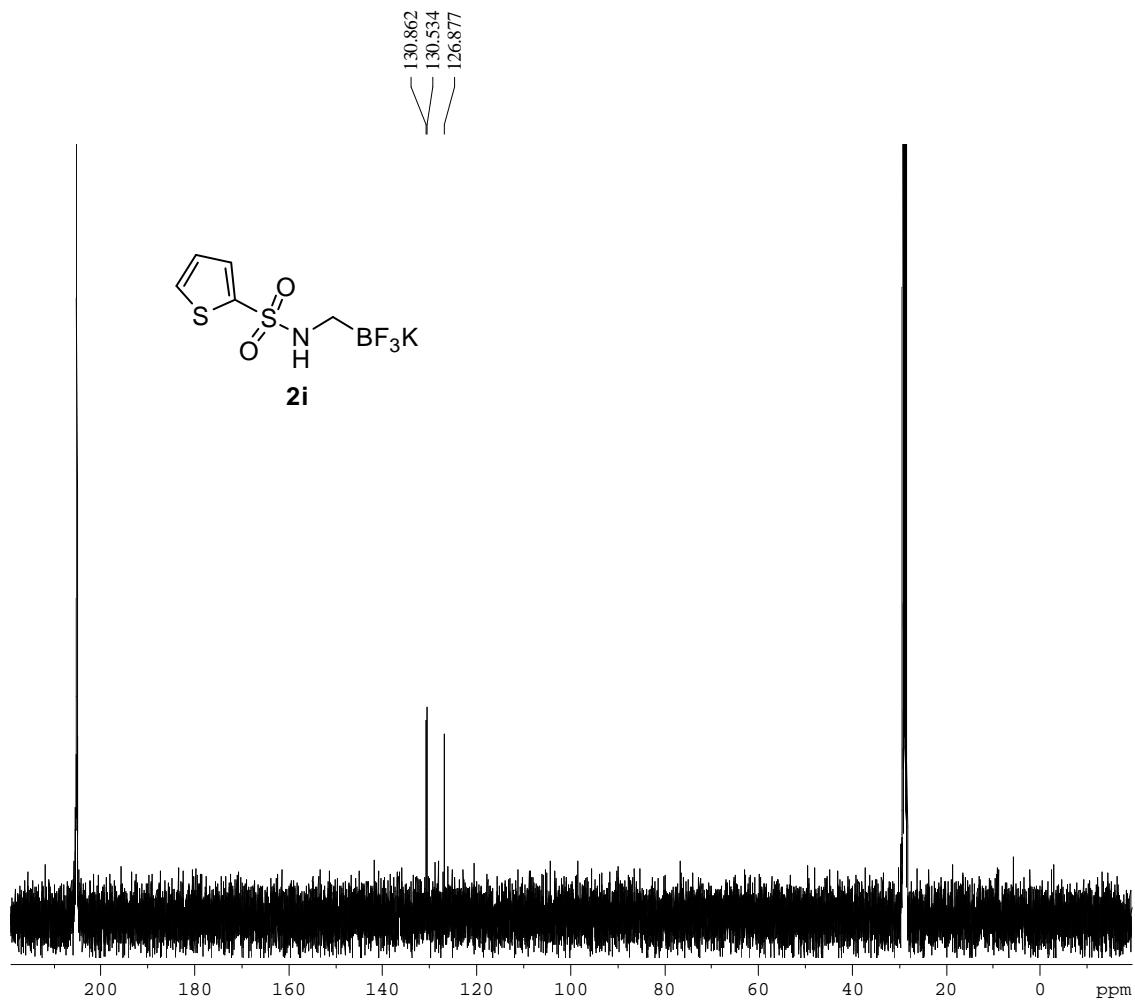
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 1-Naphthylsulfonamidomethyltrifluoroborate **2h**



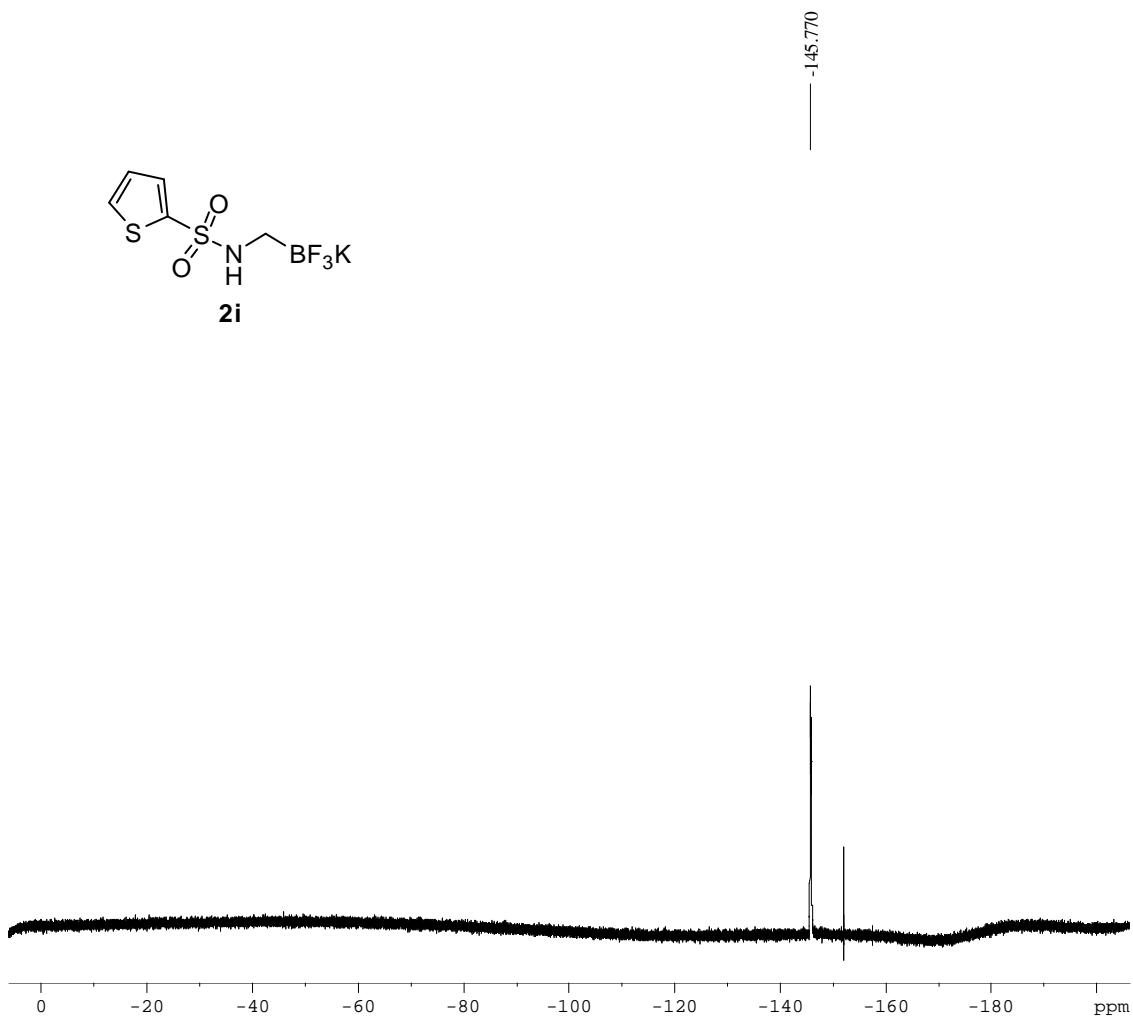
<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 500 MHz) spectrum of Potassium Thiophene-2-sulfonamidomethyltrifluoroborate **2i**



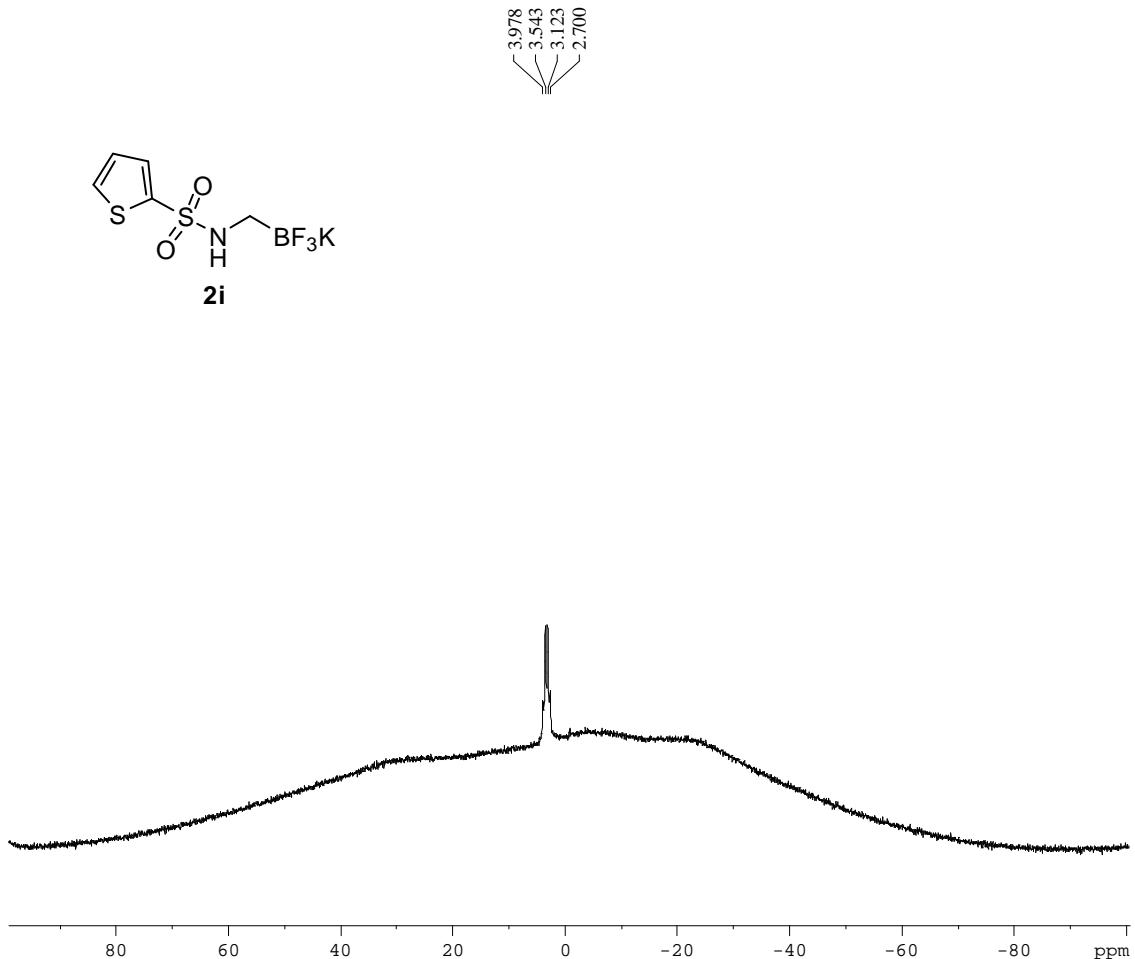
<sup>13</sup>C NMR (acetone-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium Thiophene-2-sulfonamidomethyltrifluoroborate **2i**



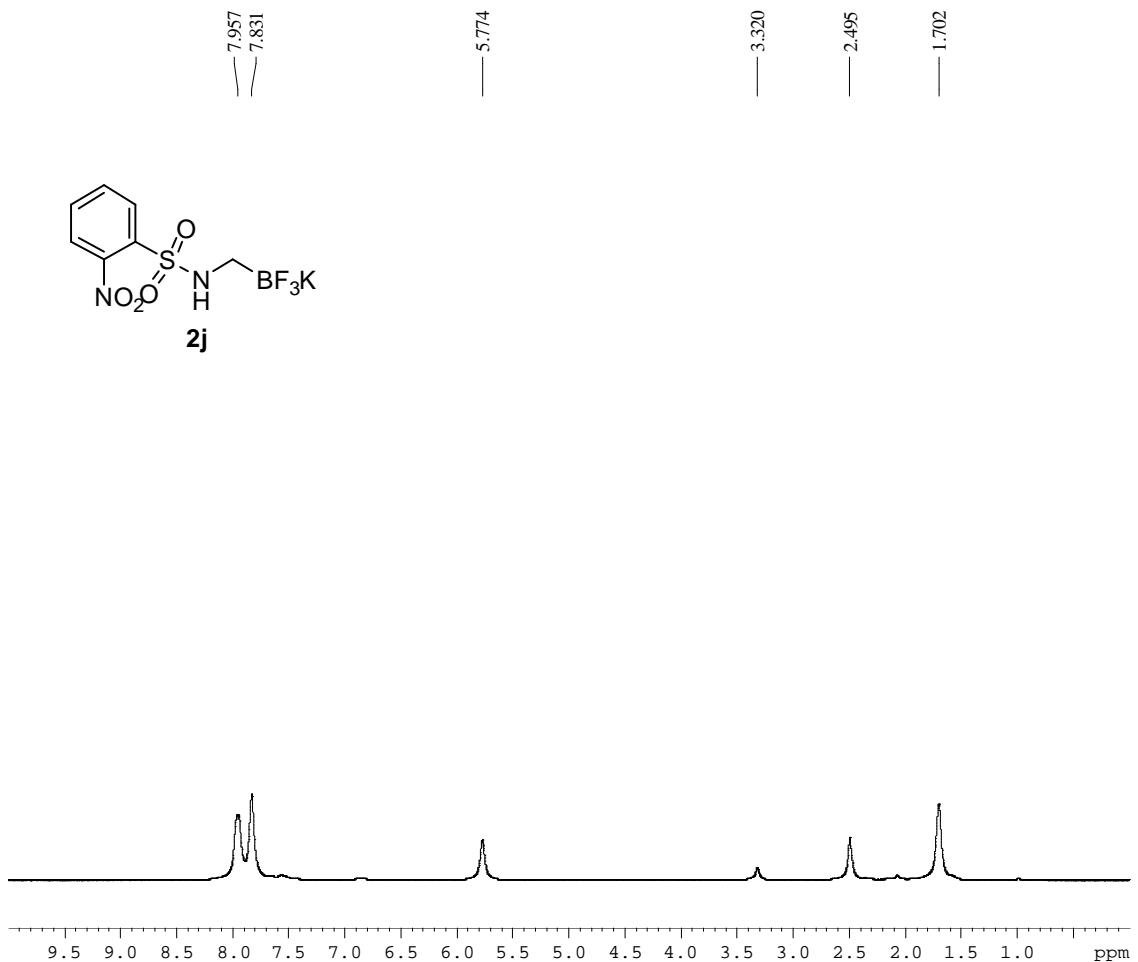
<sup>19</sup>F NMR (acetone-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium Thiophene-2-sulfonamidomethyltrifluoroborate **2i**



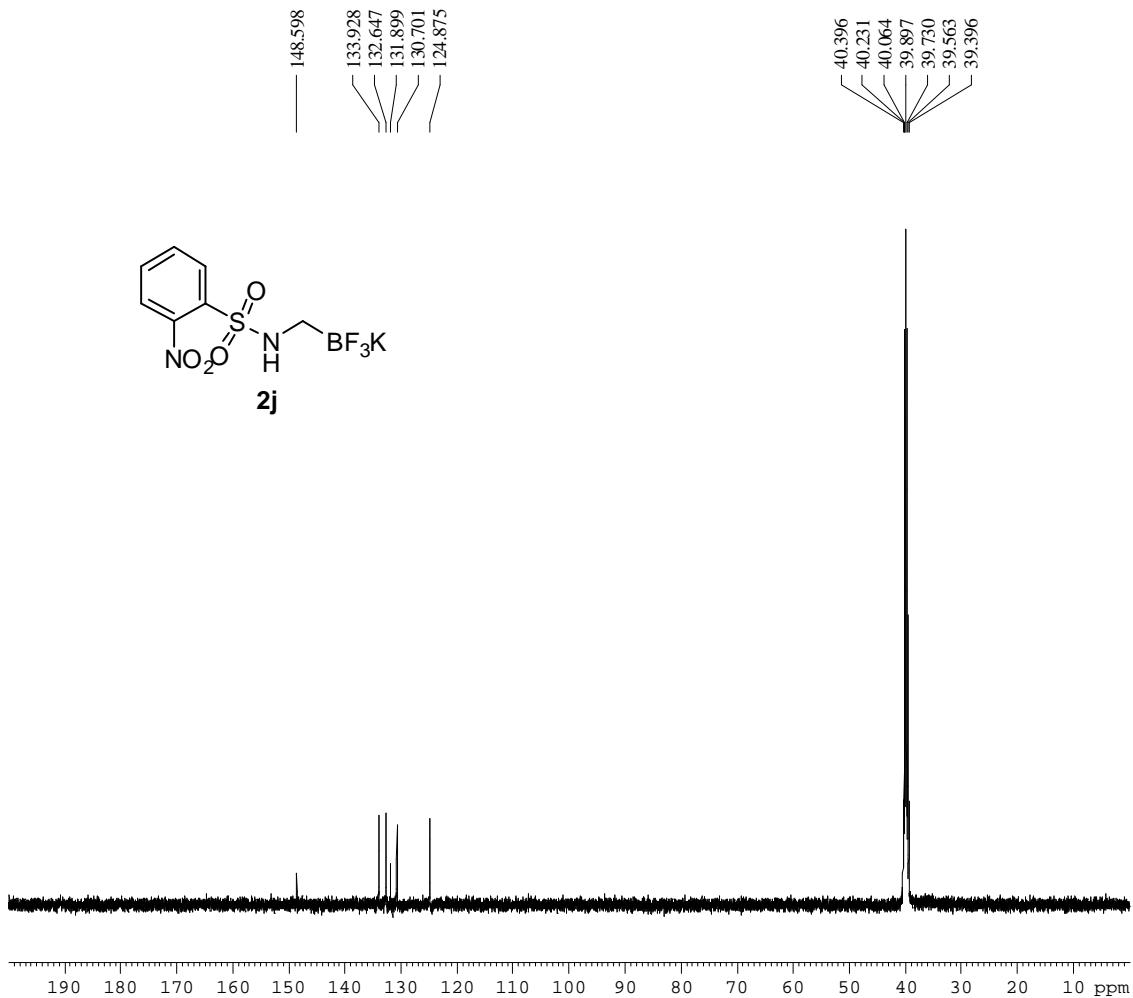
<sup>11</sup>B NMR (acetone-d<sub>6</sub>, 500 MHz) spectrum of Potassium Thiophene-2-sulfonamidomethyltrifluoroborate **2i**



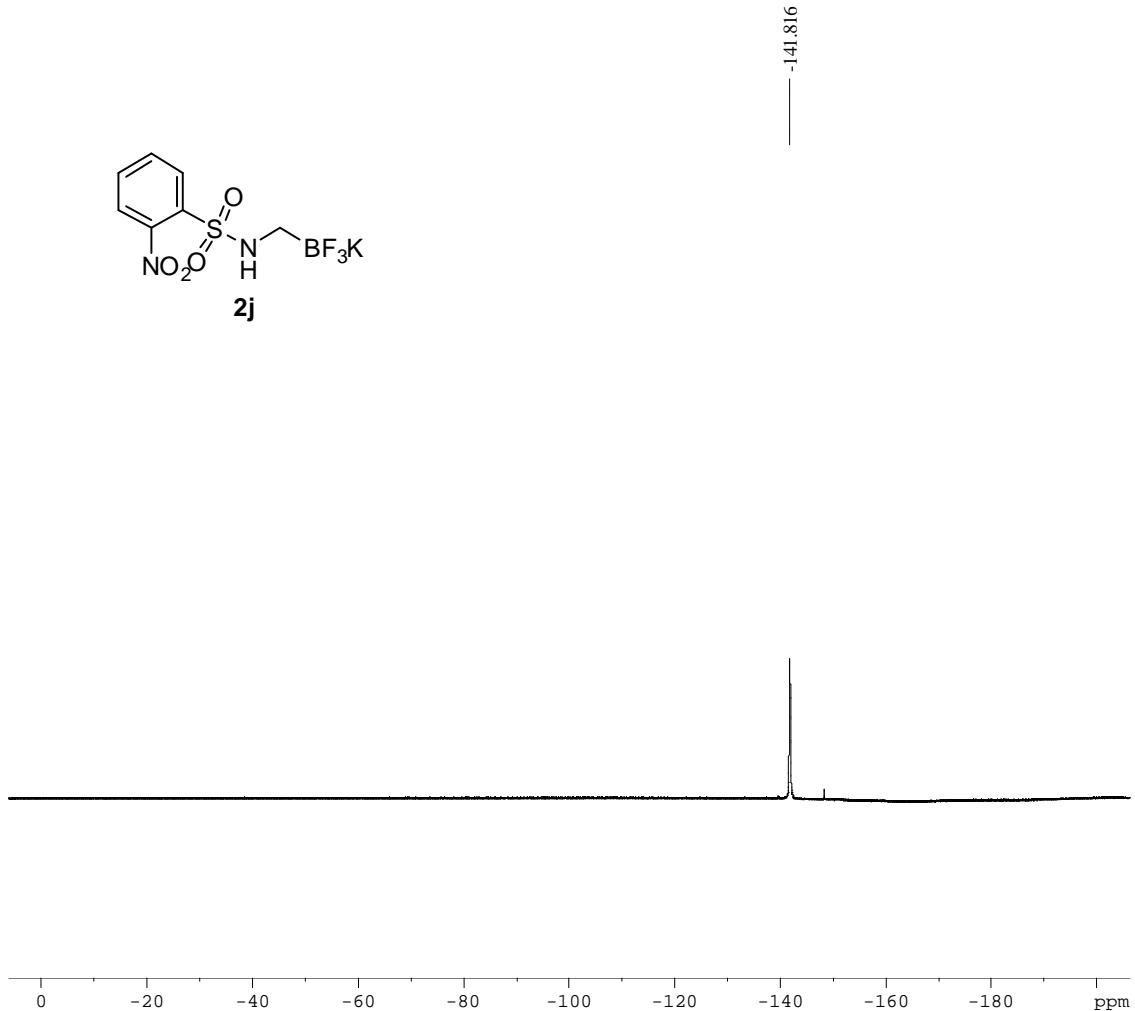
<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz) spectrum of Potassium 2-Nitrophenylsulfonamidomethyltrifluoroborate **2j**



<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 2-Nitrophenylsulfonamidomethyltrifluoroborate **2j**

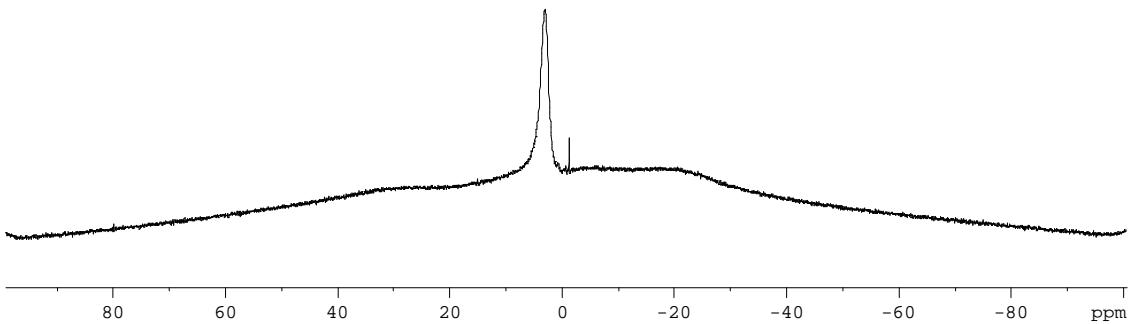
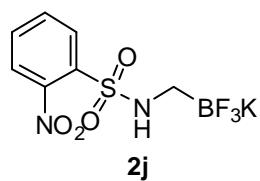


<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium 2-Nitrophenylsulfonamidomethyltrifluoroborate **2j**

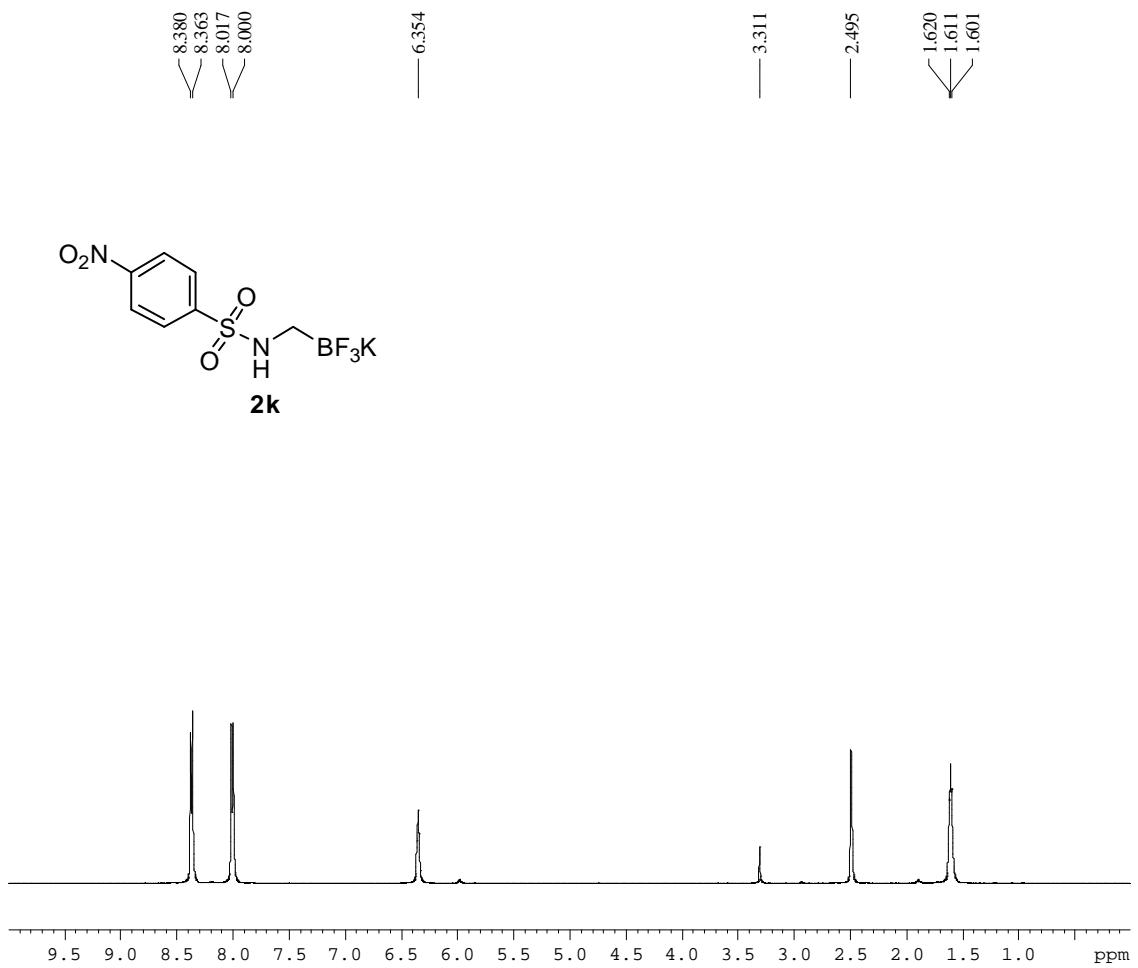


<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) spectrum of Potassium 2-Nitrophenylsulfonamidomethyltrifluoroborate **2j**

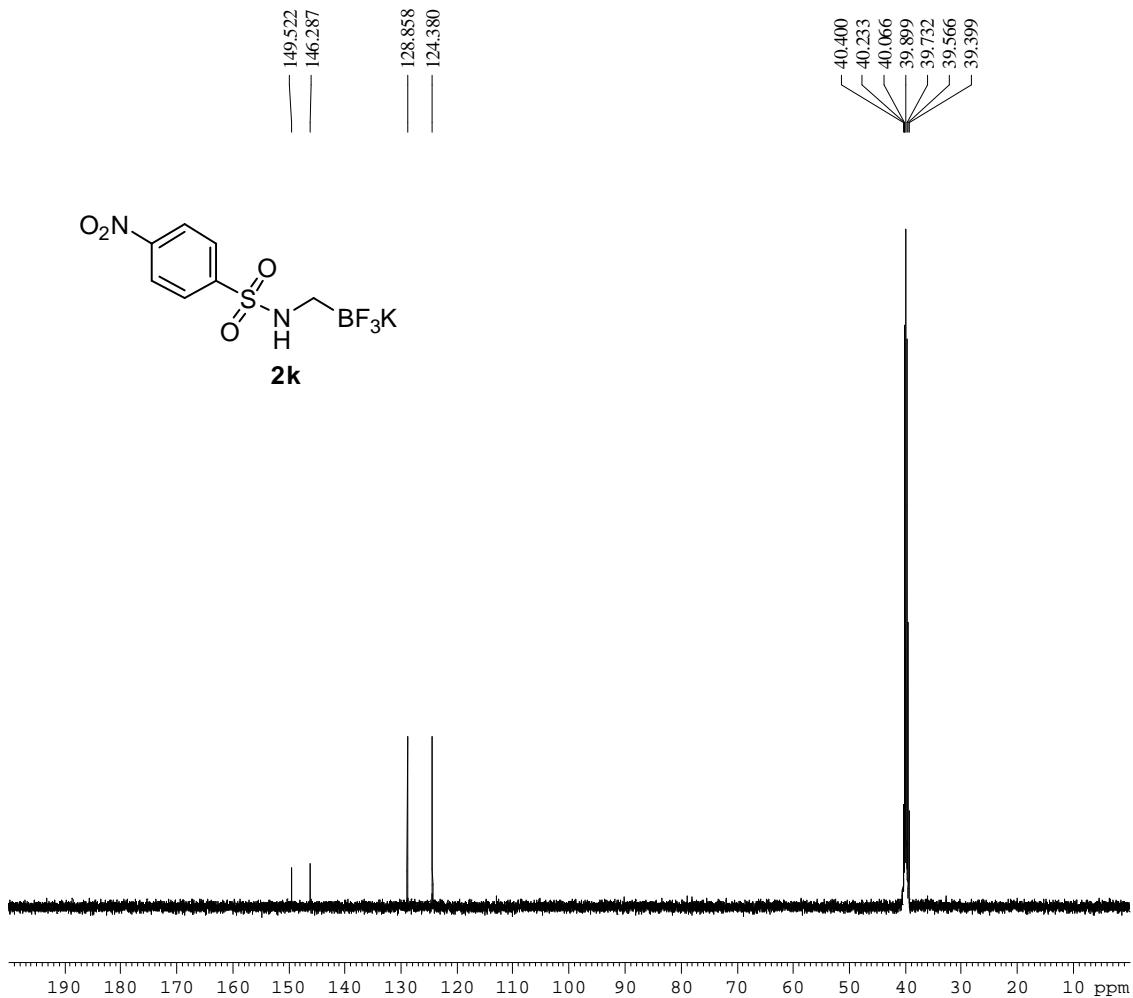
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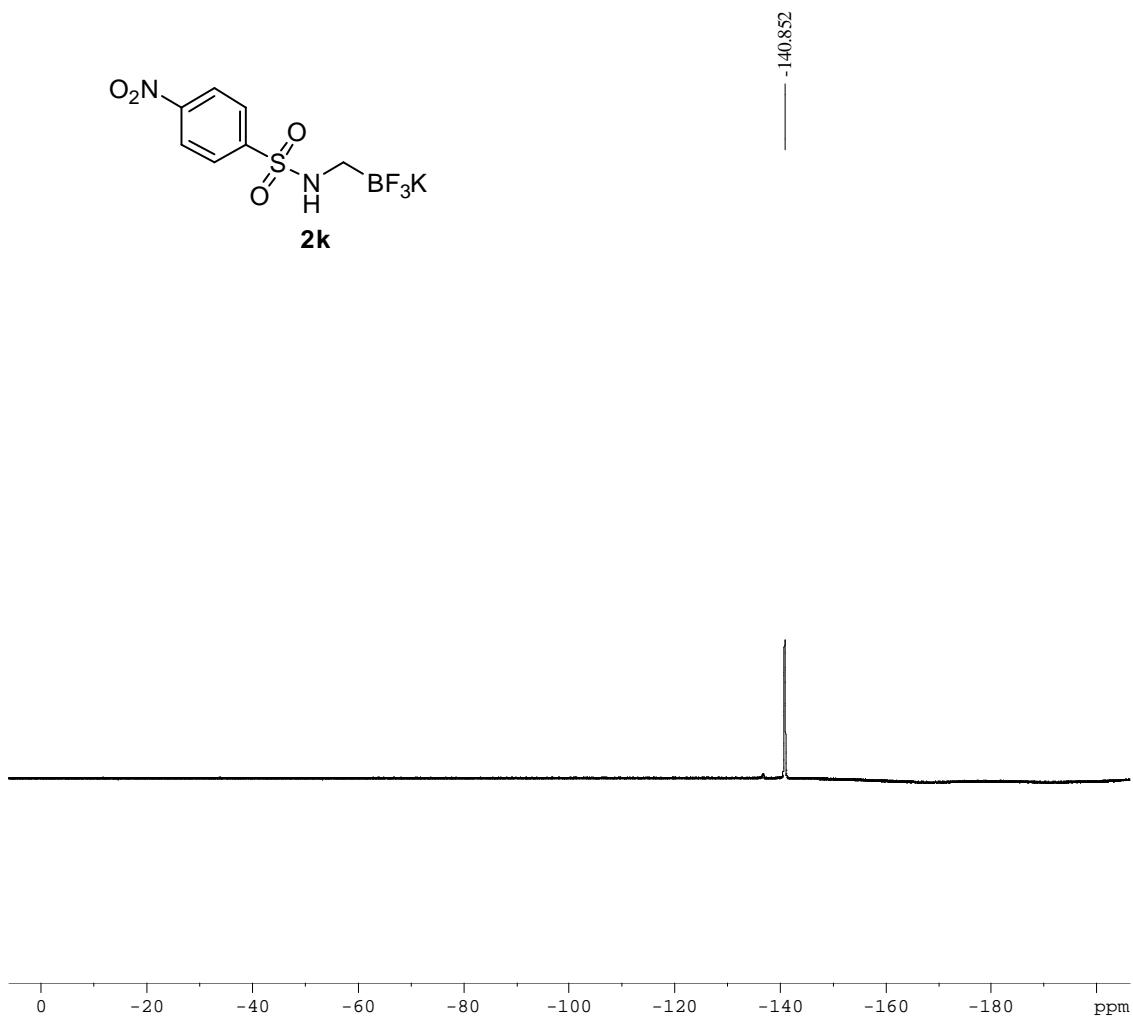
<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz) spectrum of Potassium 4-Nitrophenylsulfonamidomethyltrifluoroborate **2k**



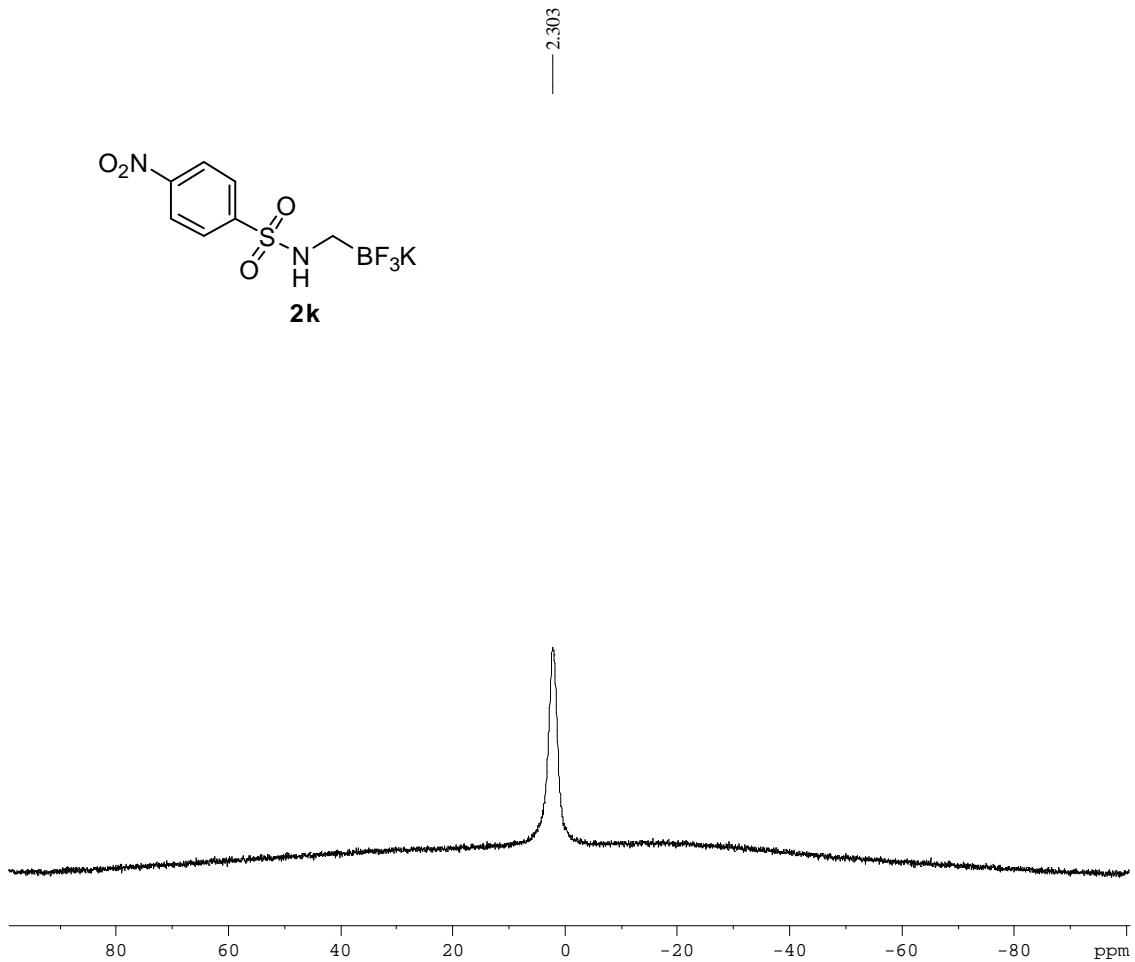
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 4-Nitrophenylsulfonamidomethyltrifluoroborate **2k**



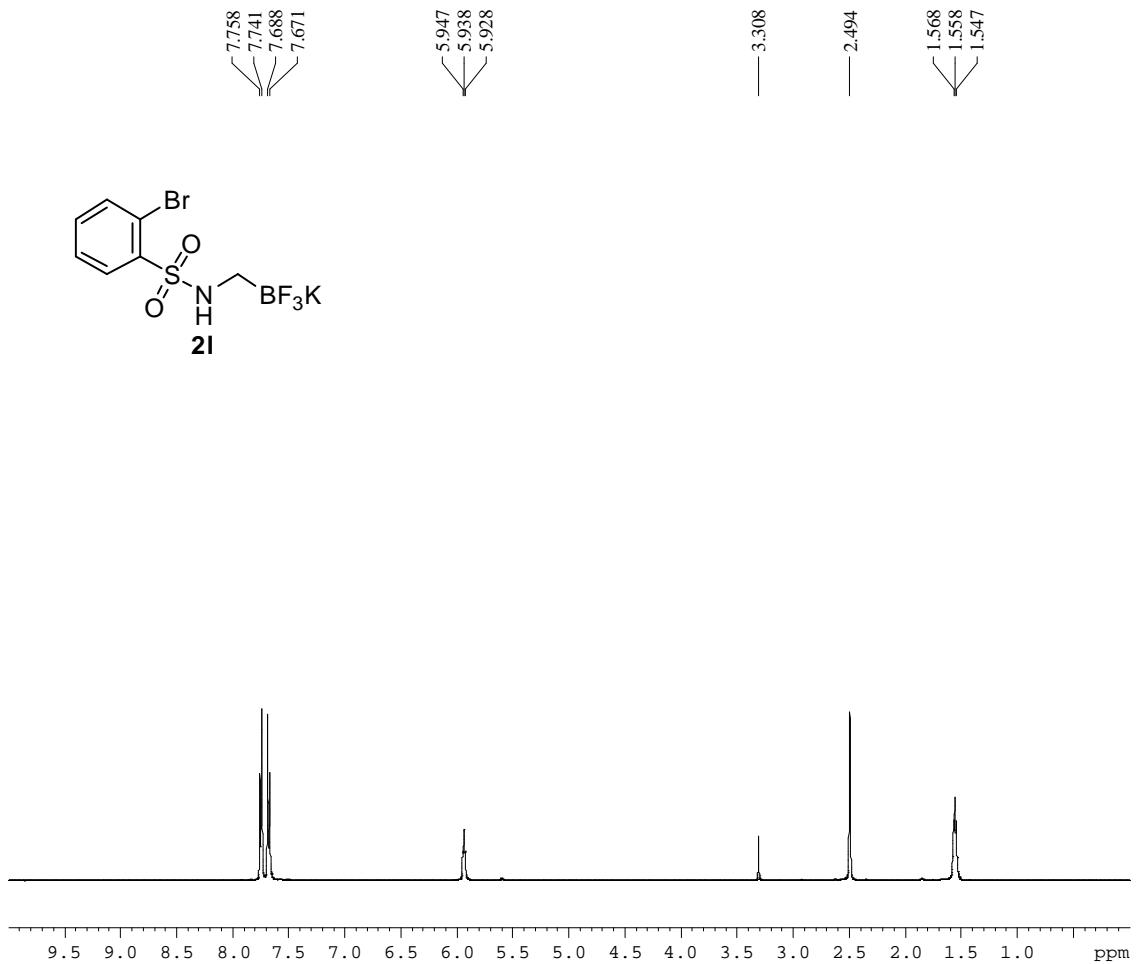
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium 4-Nitrophenylsulfonamidomethyltrifluoroborate **2k**



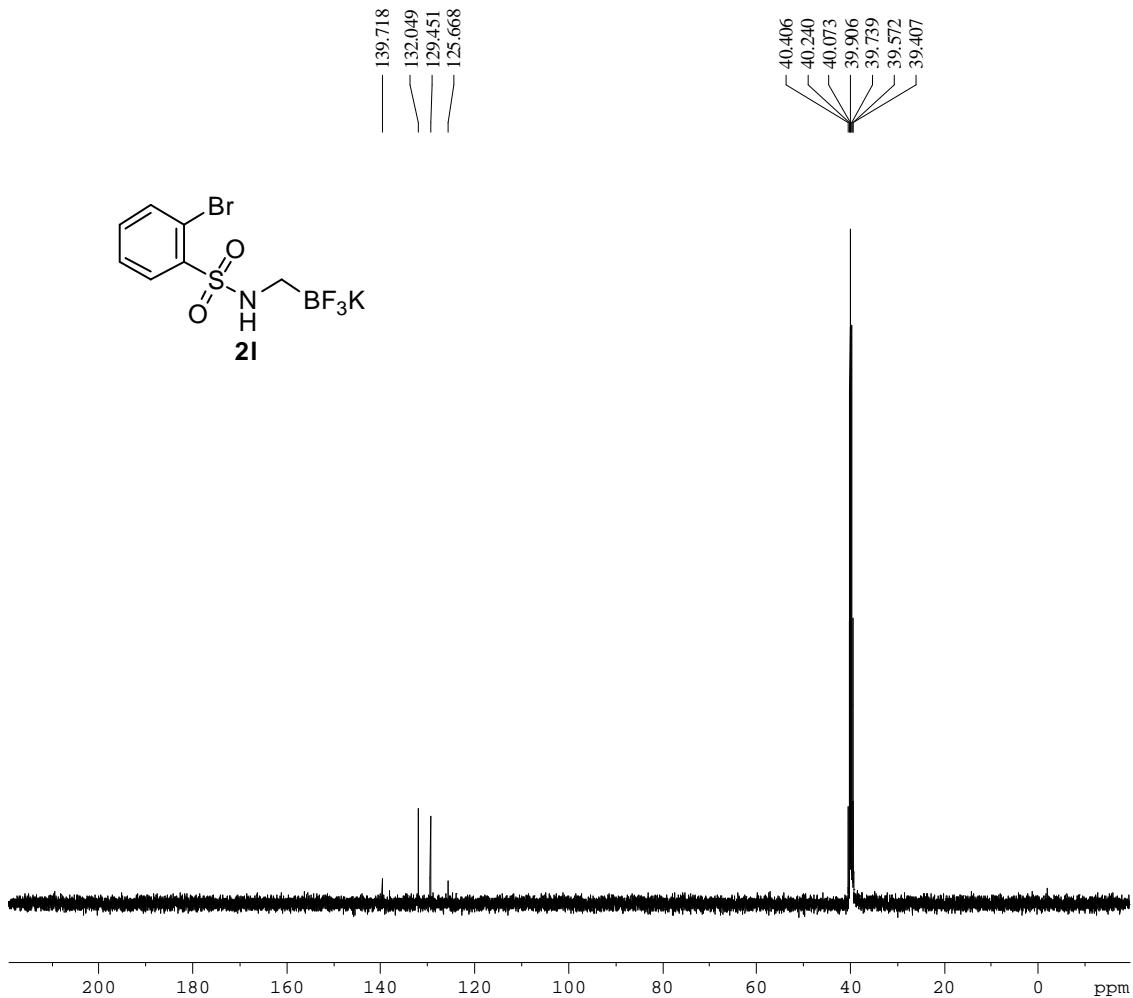
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) spectrum of Potassium 4-Nitrophenylsulfonamidomethyltrifluoroborate **2k**



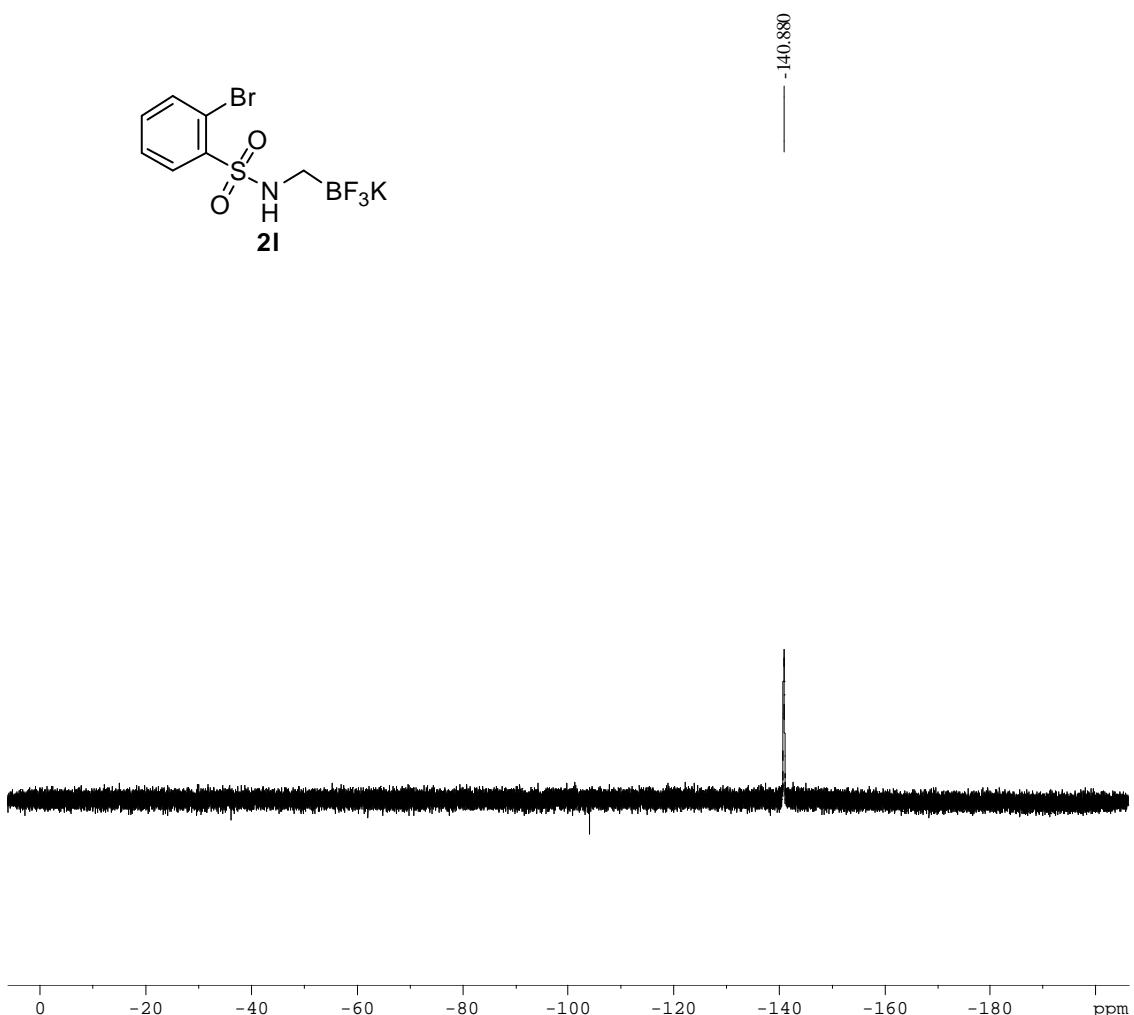
<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz) spectrum of Potassium 2-Bromophenylsulfonamidomethyltrifluoroborate **2I**



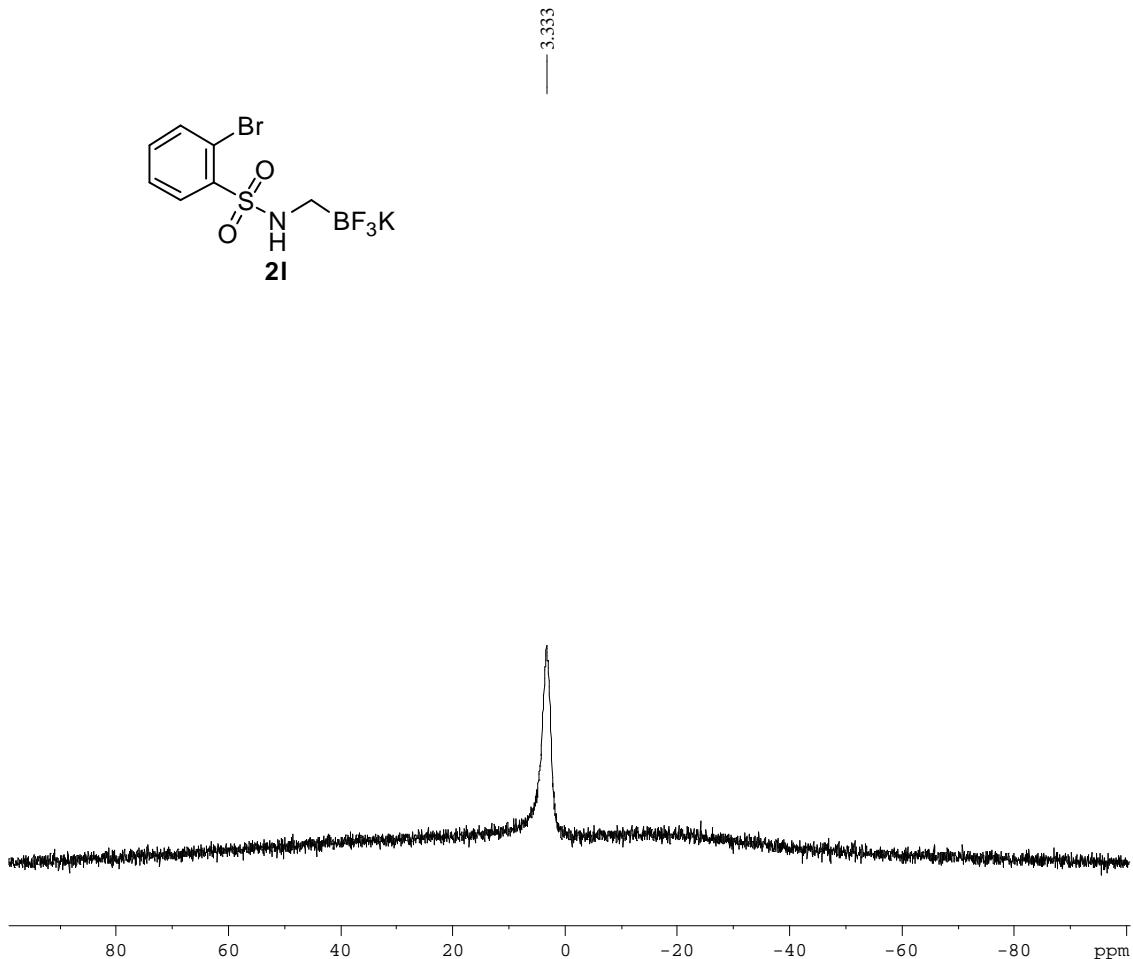
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 2-Bromophenylsulfonamidomethyltrifluoroborate **2I**



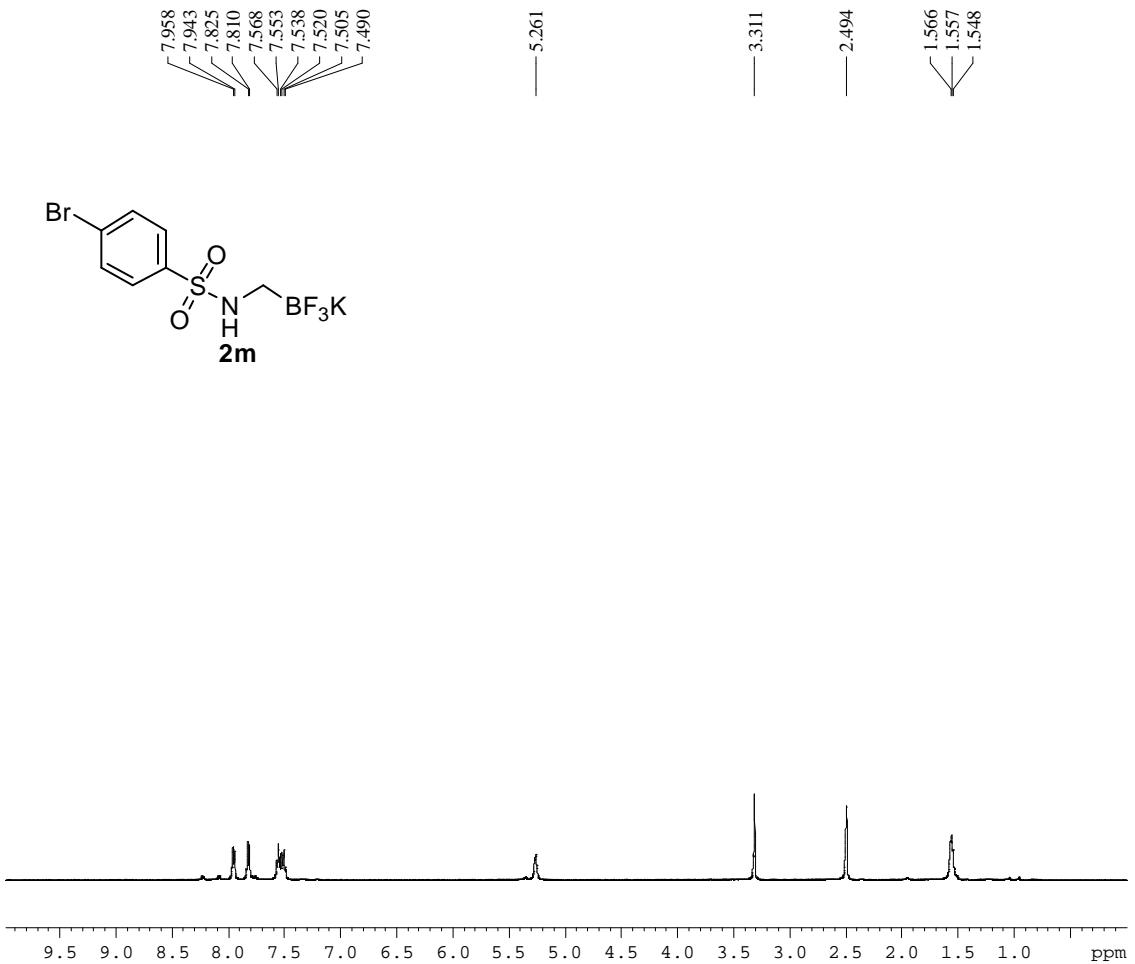
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium 2-Bromophenylsulfonamidomethyltrifluoroborate **2l**



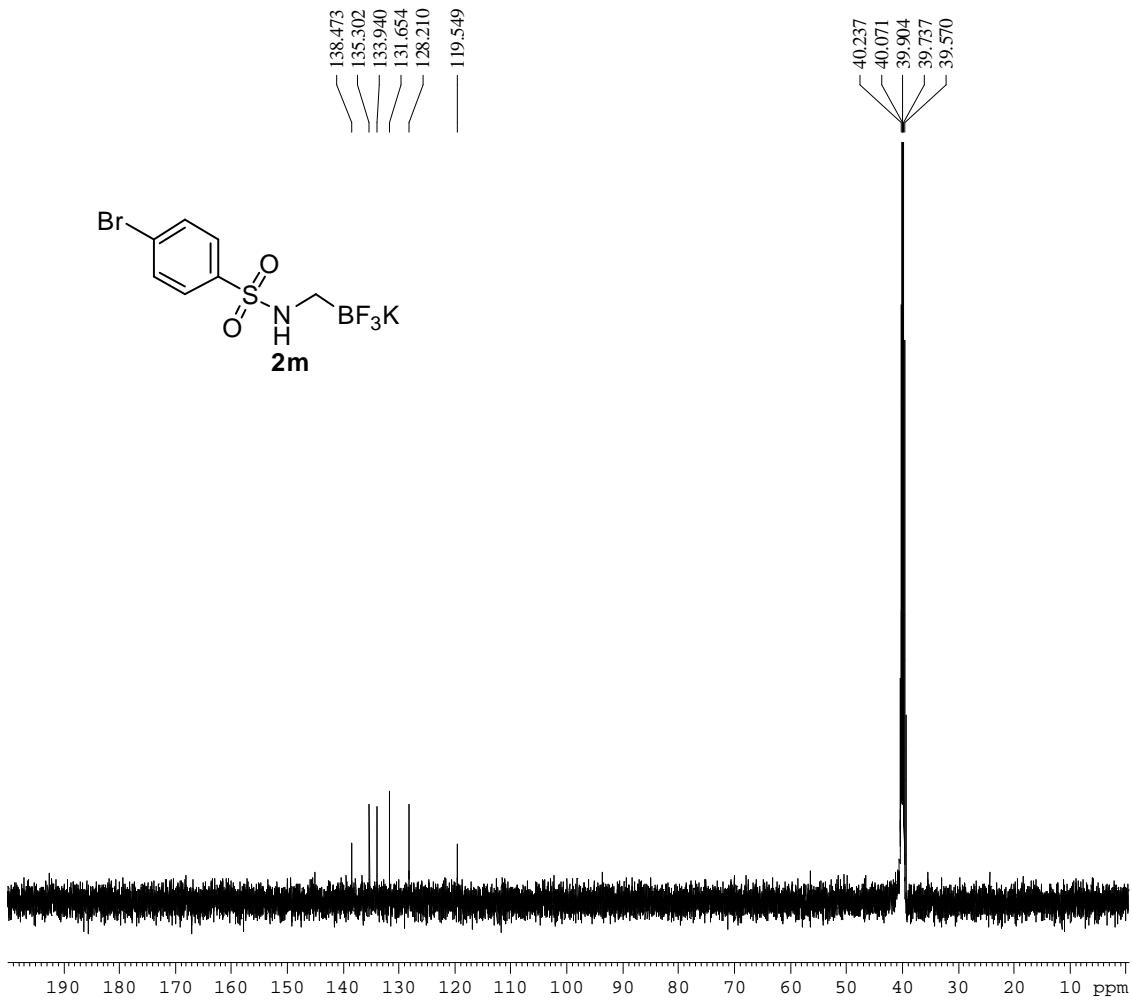
$^{11}\text{B}$  NMR ( $\text{DMSO-d}_6$ , 128.4 MHz) spectrum of Potassium 2-Bromophenylsulfonamidomethyltrifluoroborate **2l**



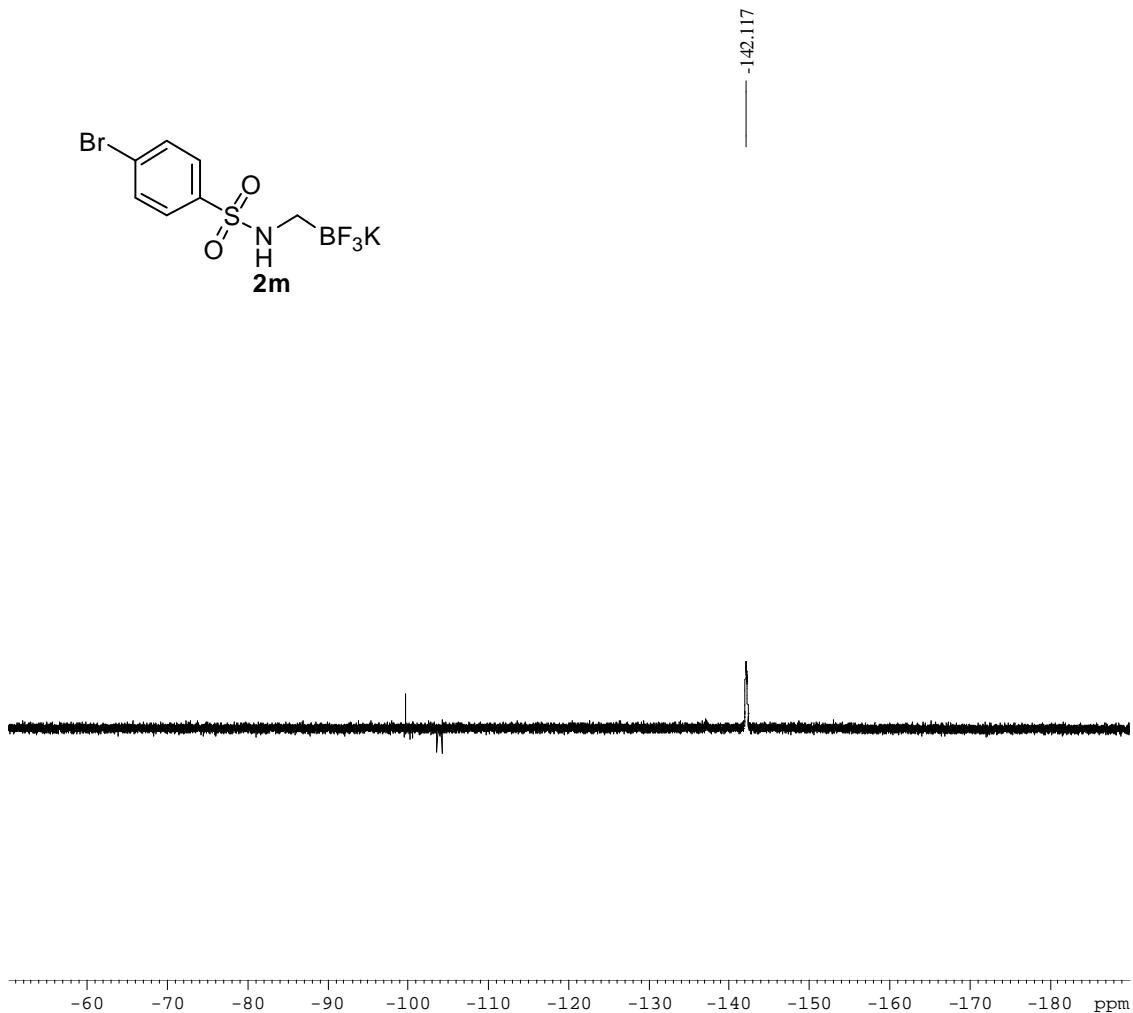
<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz) spectrum of Potassium 4-Bromophenylsulfonamidomethyltrifluoroborate **2m**



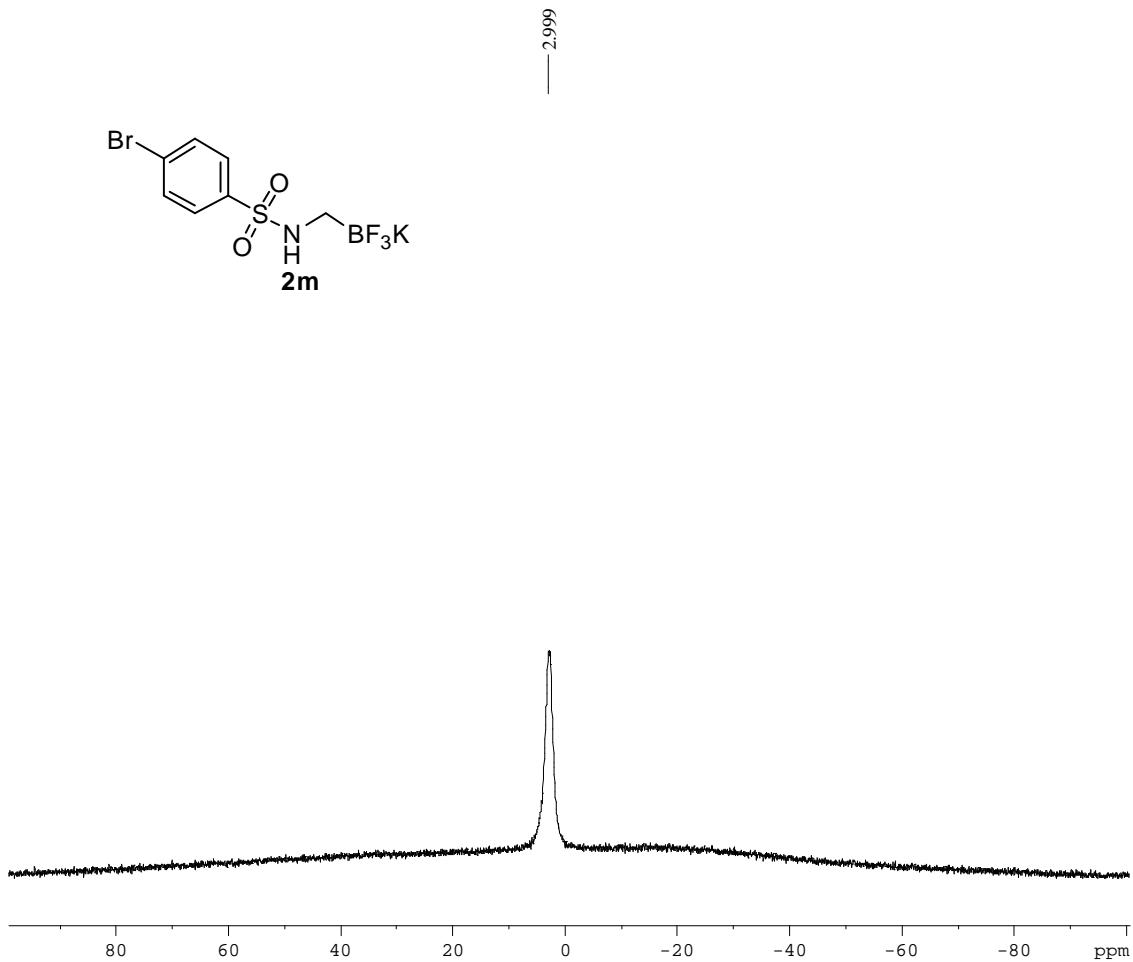
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 4-Bromophenylsulfonamidomethyltrifluoroborate **2m**



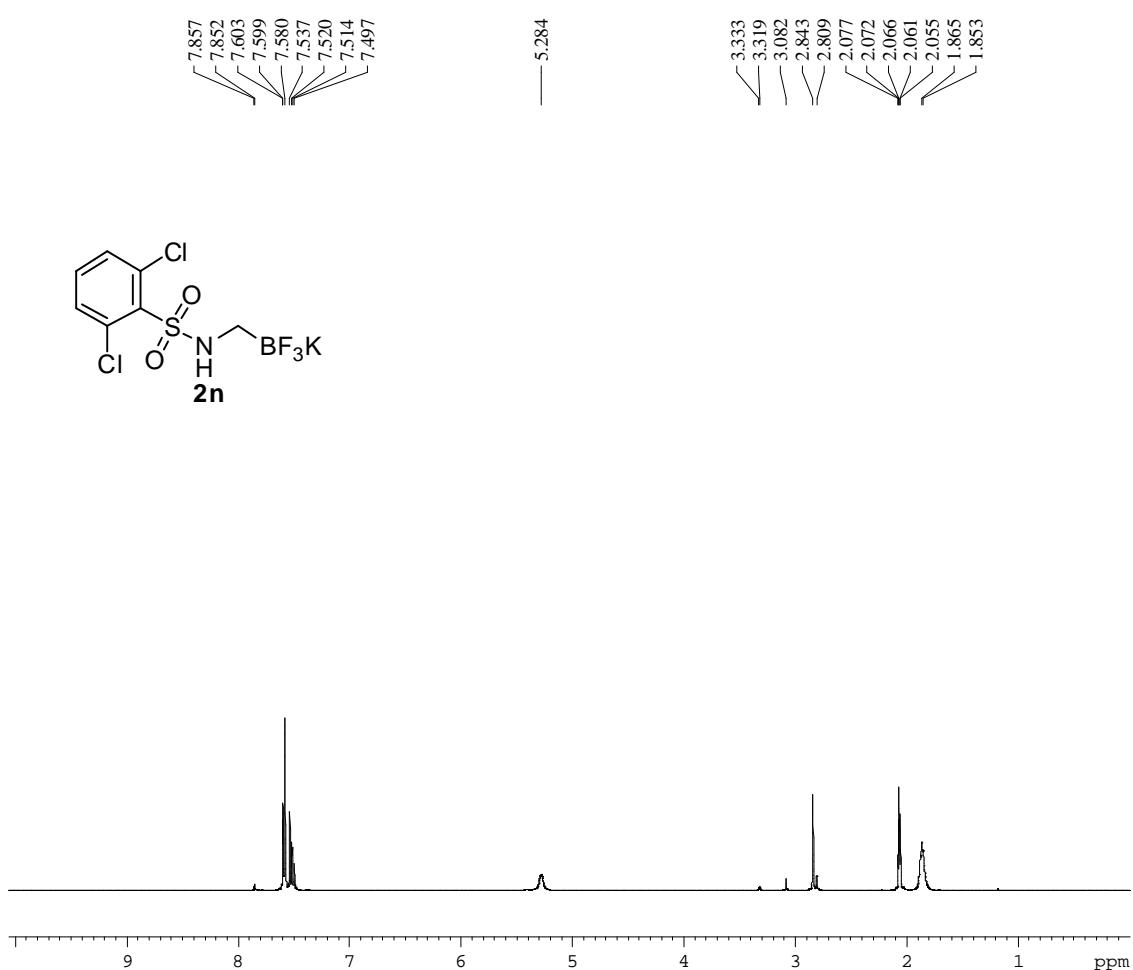
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium 4-Bromophenylsulfonamidomethyltrifluoroborate **2m**



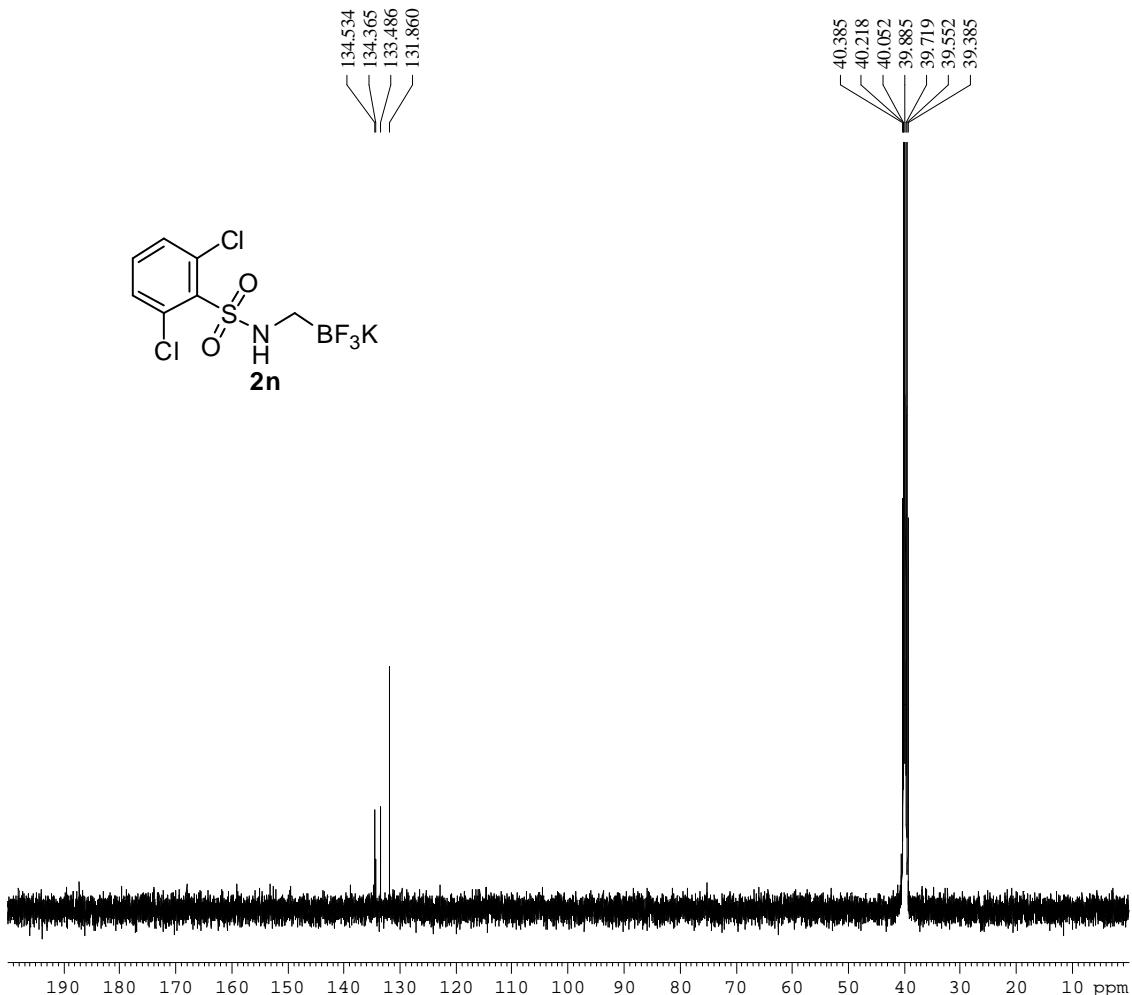
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) spectrum of Potassium 4-Bromophenylsulfonamidomethyltrifluoroborate **2m**



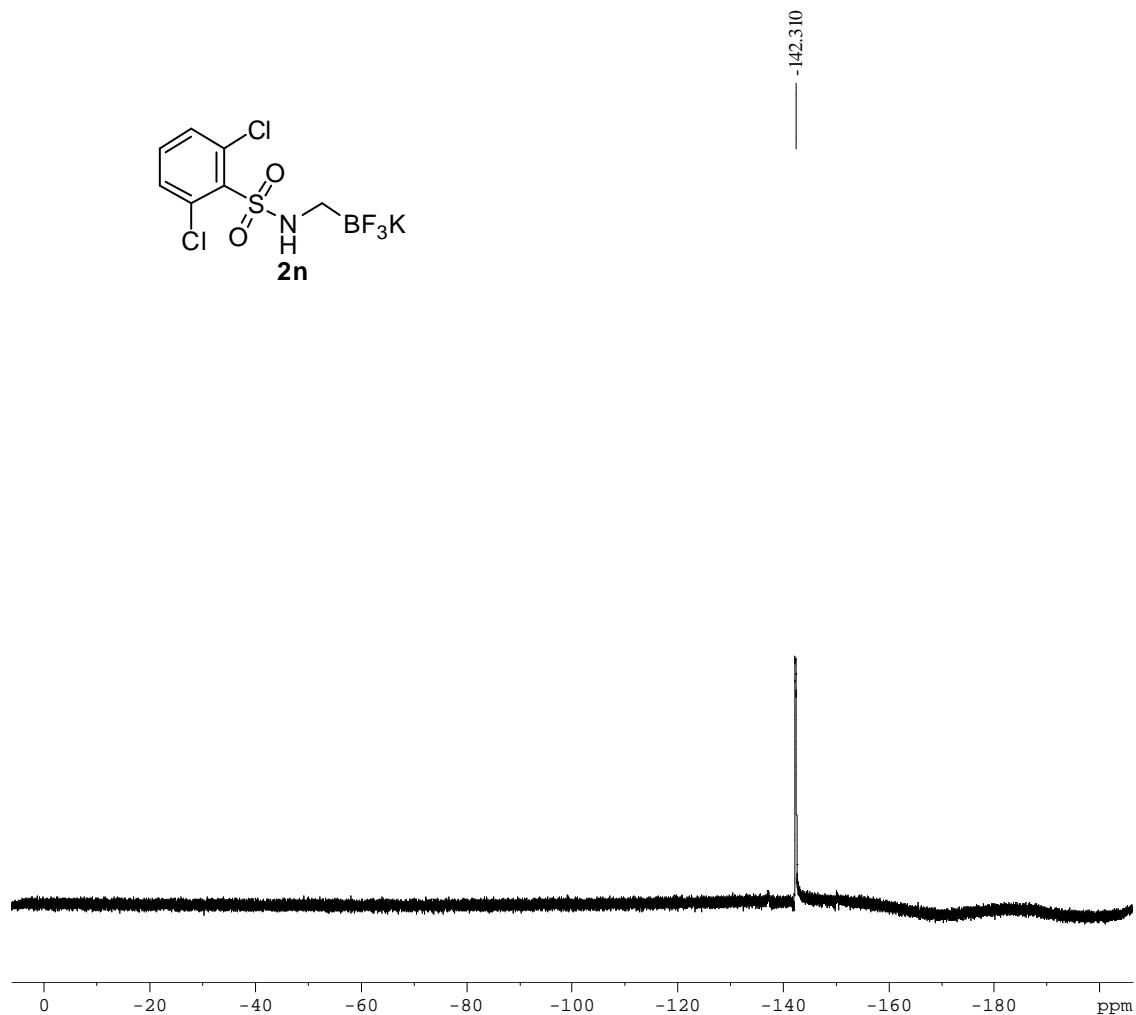
<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz) spectrum of Potassium 2,6-Dichlorophenylsulfonamidomethyltrifluoroborate **2n**



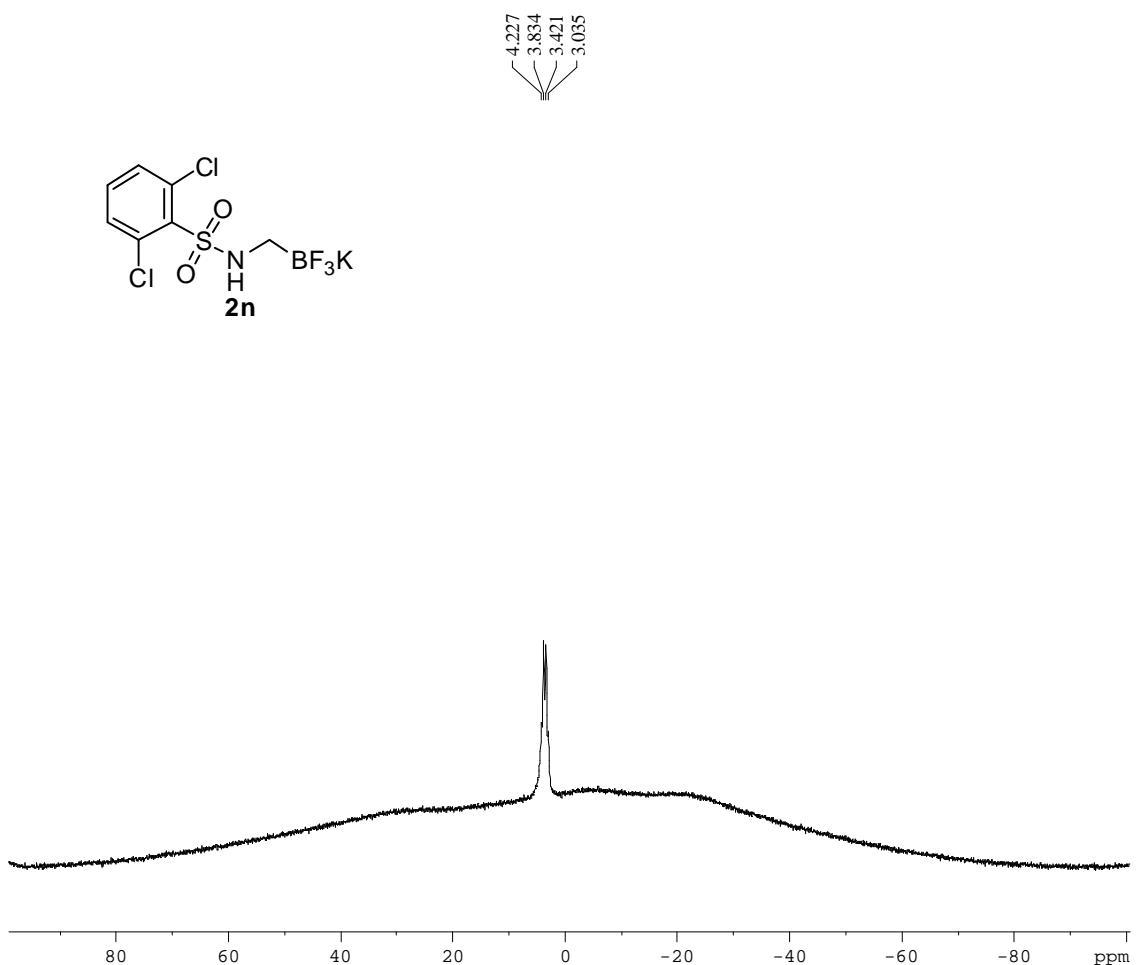
<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 2,6-Dichlorophenylsulfonamidomethyltrifluoroborate **2n**



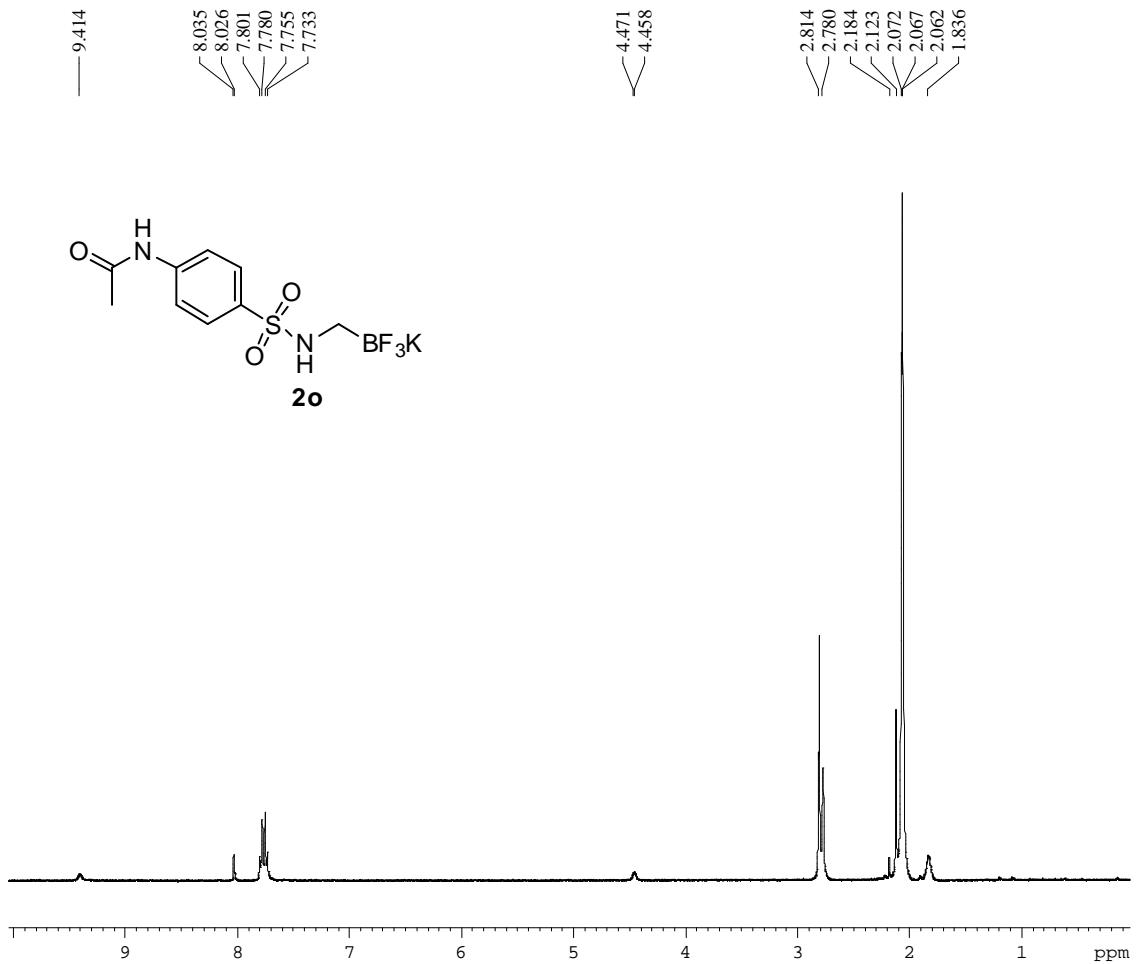
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium 2,6-Dichlorophenylsulfonamidomethyltrifluoroborate **2n**



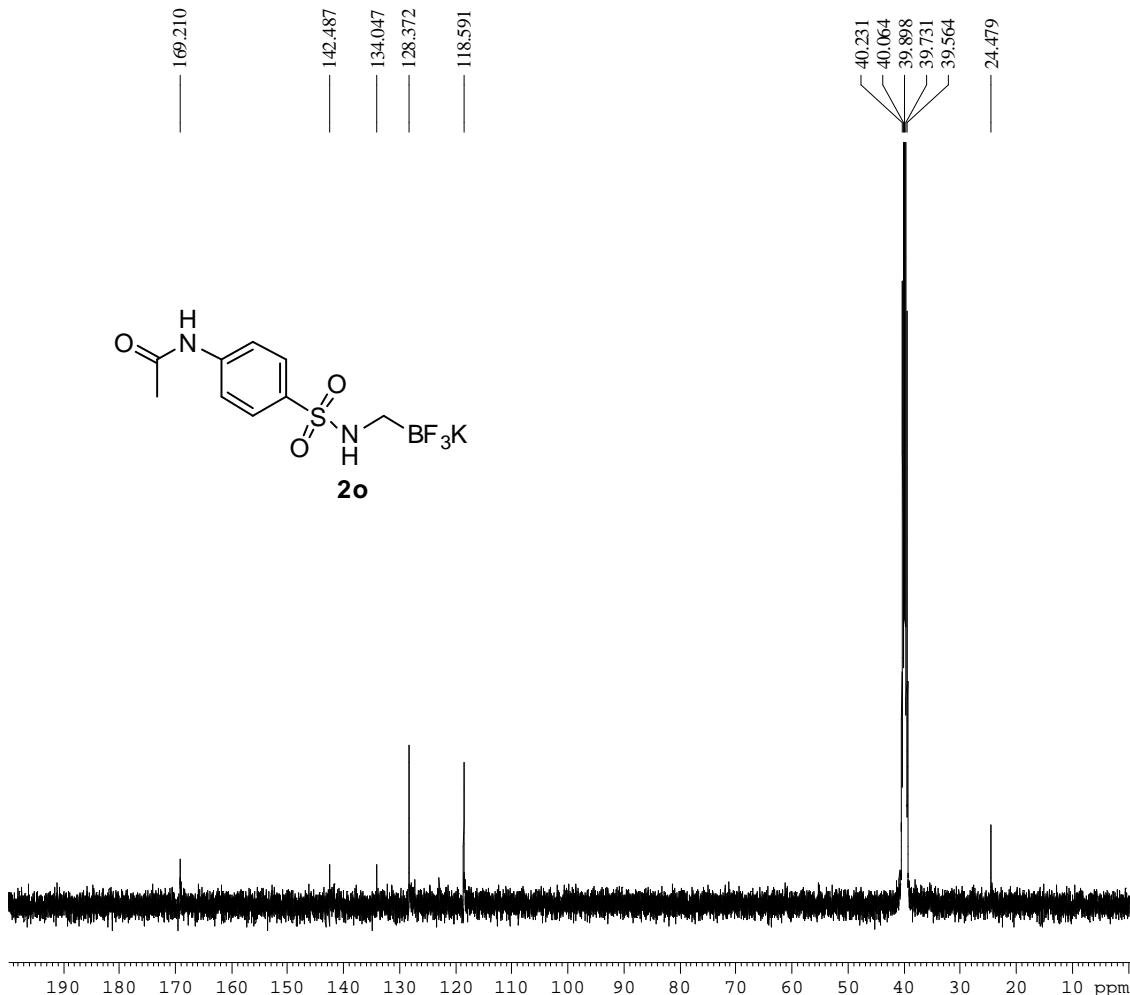
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) spectrum of Potassium 2,6-Dichlorophenylsulfonamidomethyltrifluoroborate **2n**



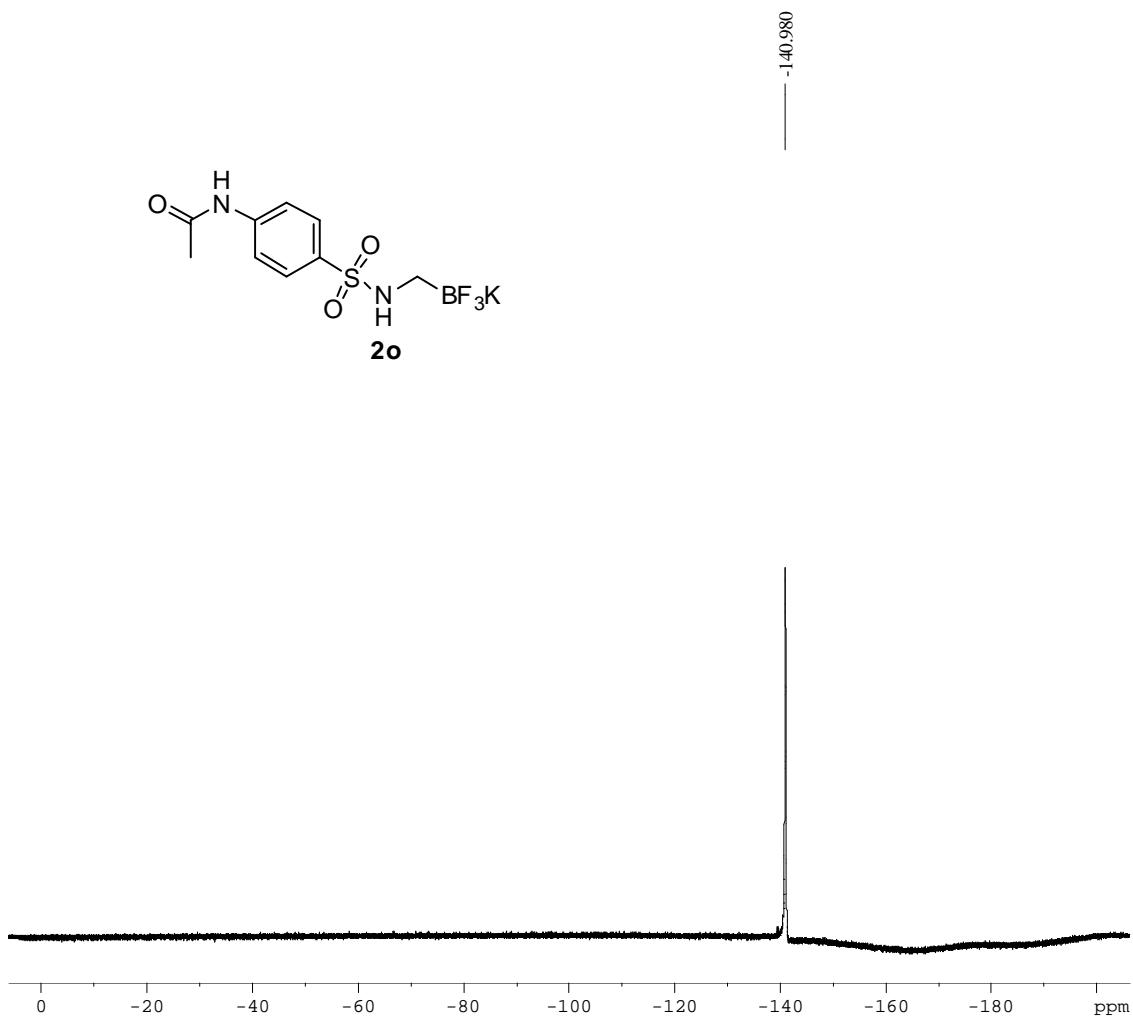
<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz) spectrum of Potassium 4-Acetamidophenylsulfonamidomethyltrifluoroborate **2o**



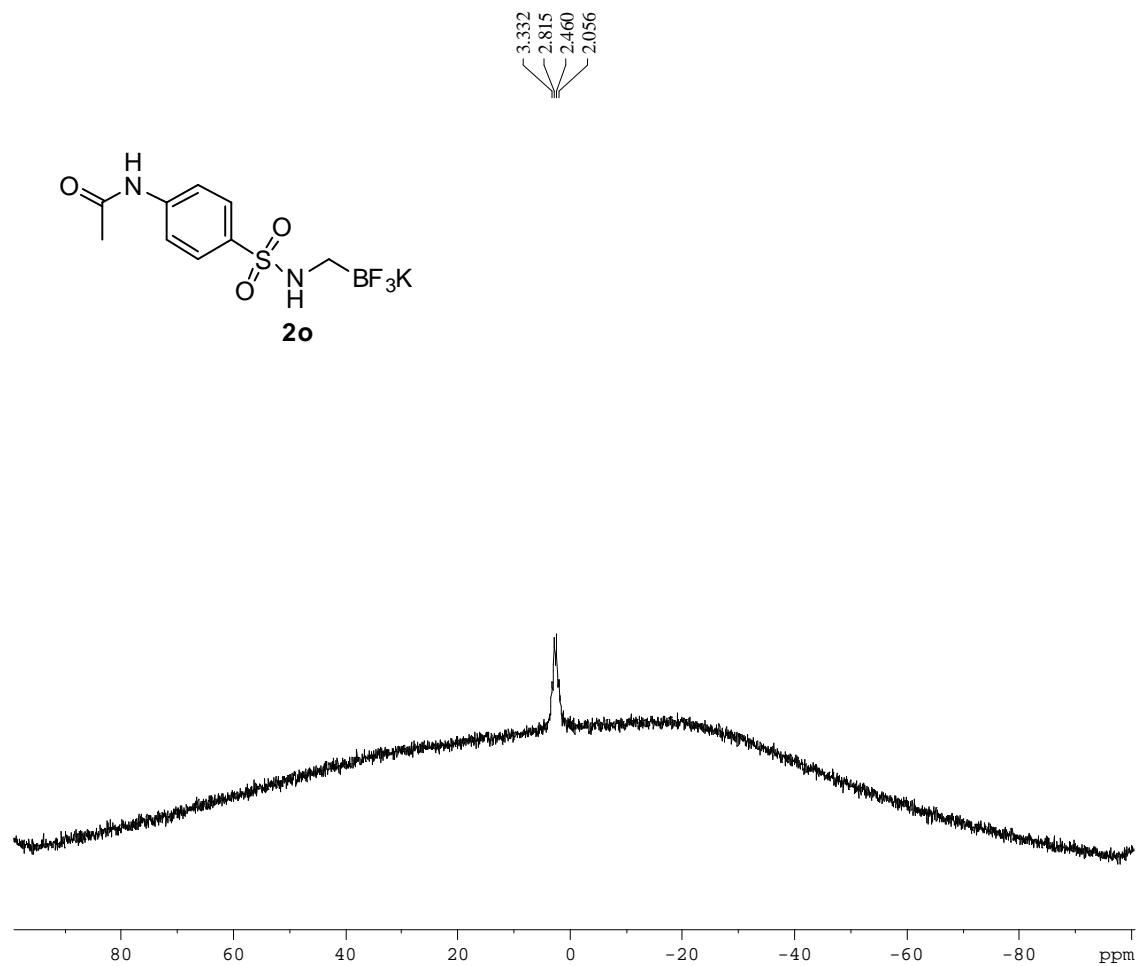
$^{13}\text{C}$  NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 4-Acetamidophenylsulfonamidomethyltrifluoroborate **2o**



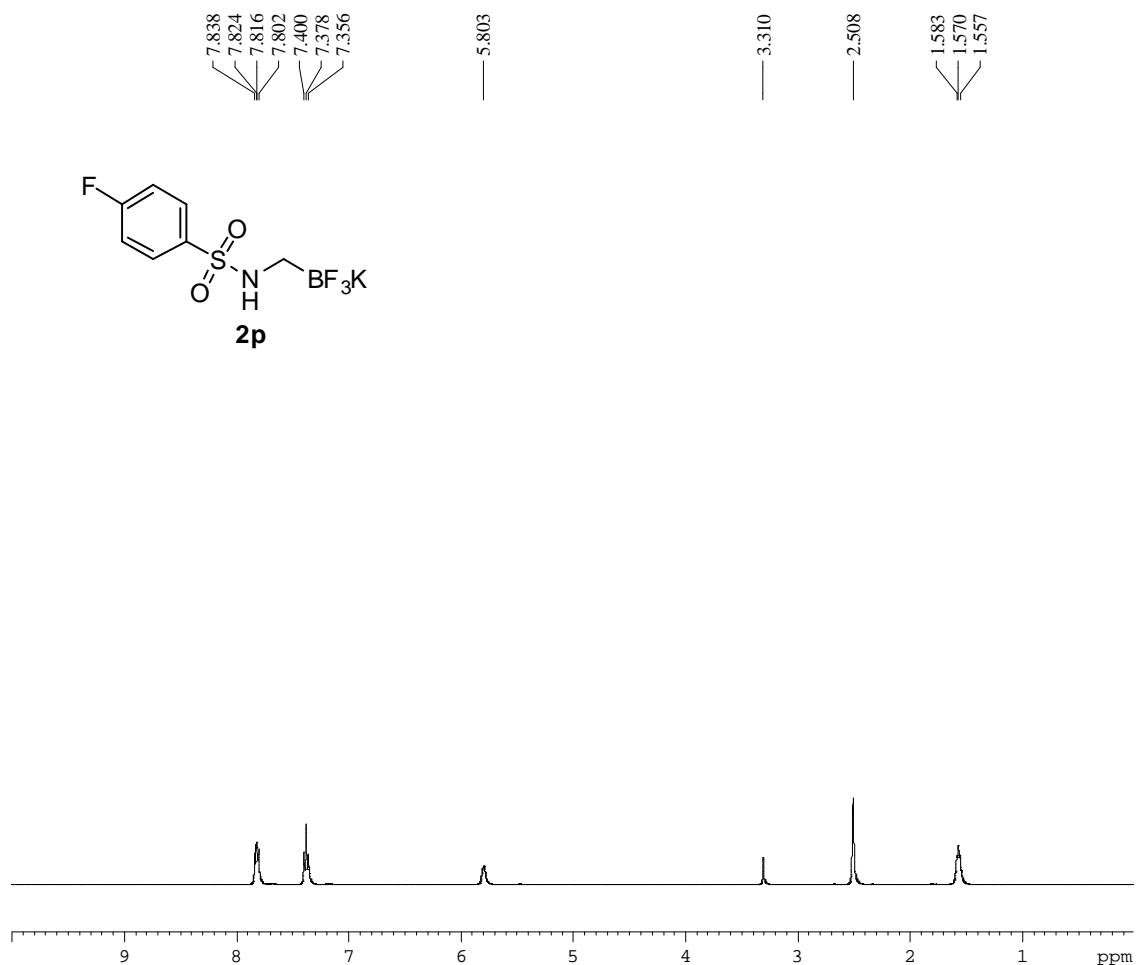
<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum of Potassium 4-Acetamidophenylsulfonamidomethyltrifluoroborate **2o**



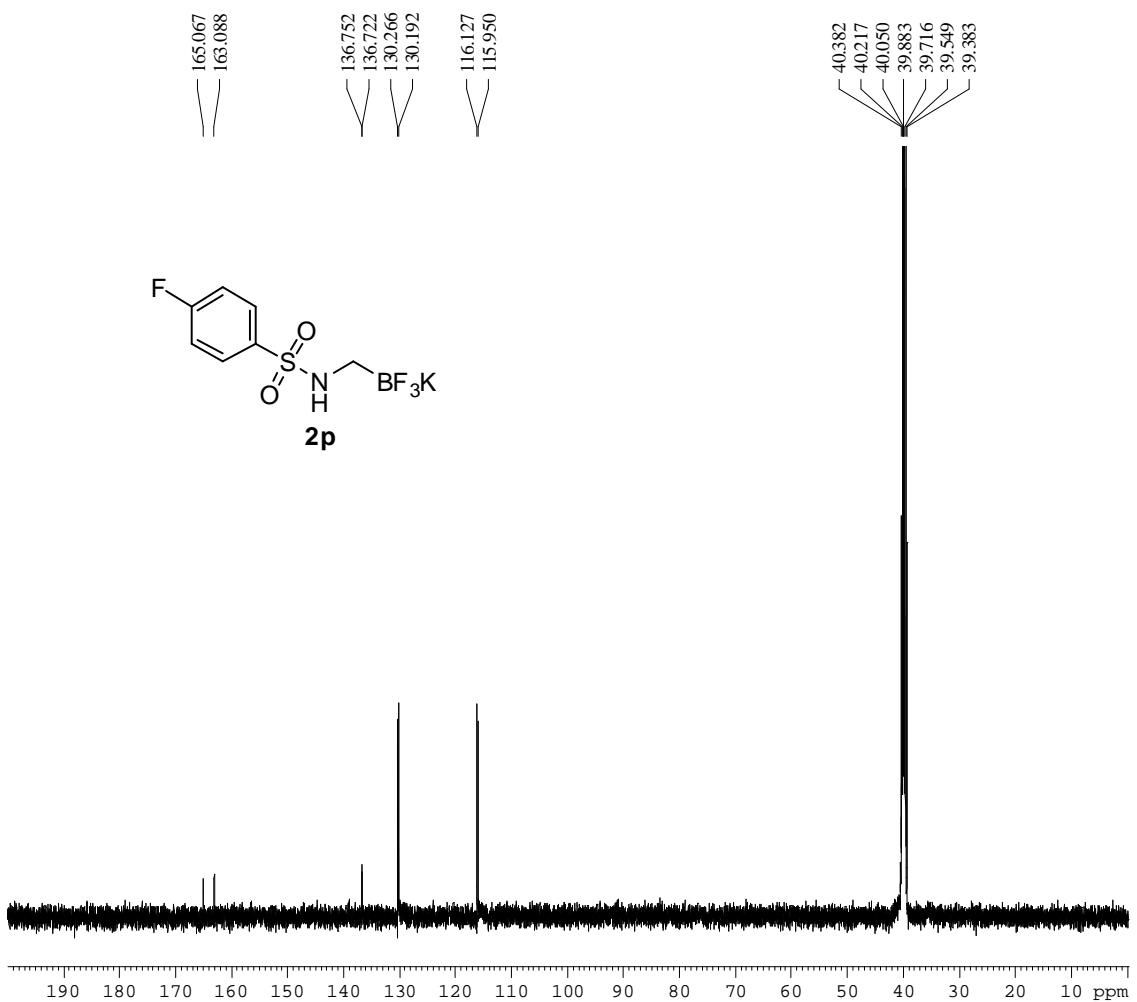
<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) Potassium 4-Acetamidophenylsulfonamidomethyltrifluoroborate **2o**



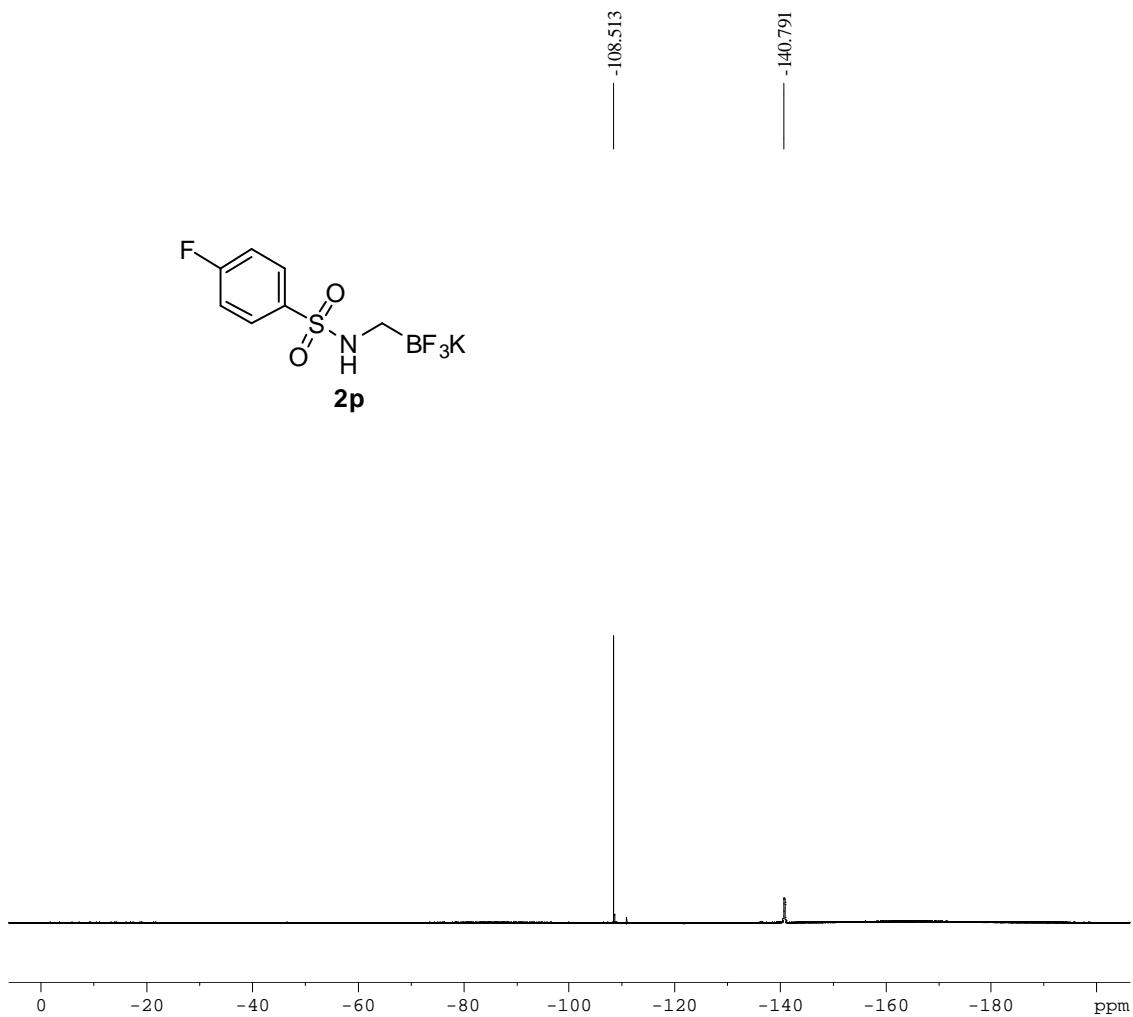
<sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 500 MHz) spectrum of Potassium 4-Fluorophenylsulfonamidomethyltrifluoroborate **2p**



<sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 125.8 MHz) spectrum of Potassium 4-Fluorophenylsulfonamidomethyltrifluoroborate **2p**



<sup>19</sup>F NMR (DMSO-d<sub>6</sub>, 470.8 MHz) spectrum Potassium 4-Fluorophenylsulfonamidomethyltrifluoroborate **2p**



<sup>11</sup>B NMR (DMSO-d<sub>6</sub>, 128.4 MHz) spectrum of Potassium 4-Fluorophenylsulfonamidomethyltrifluoroborate **2p**

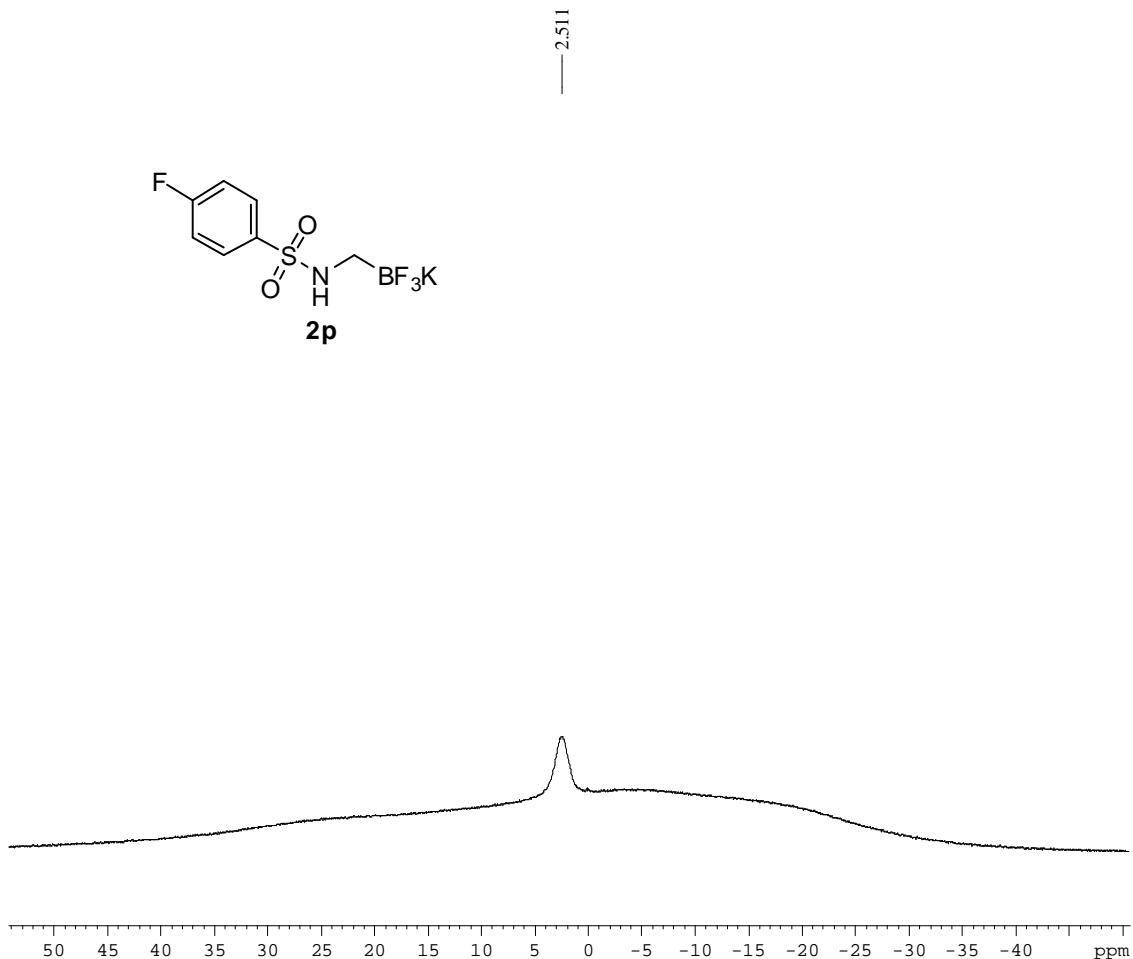
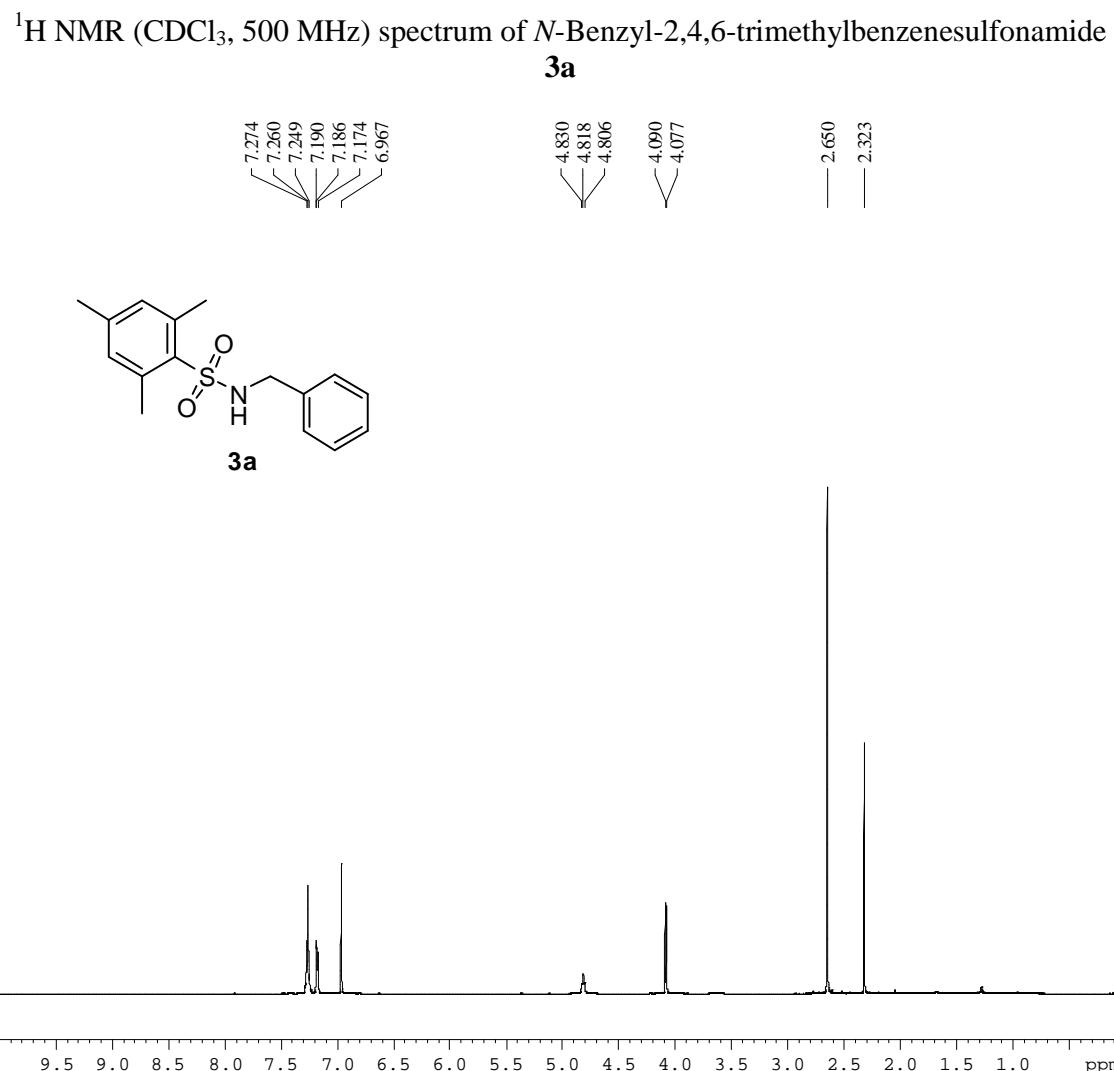
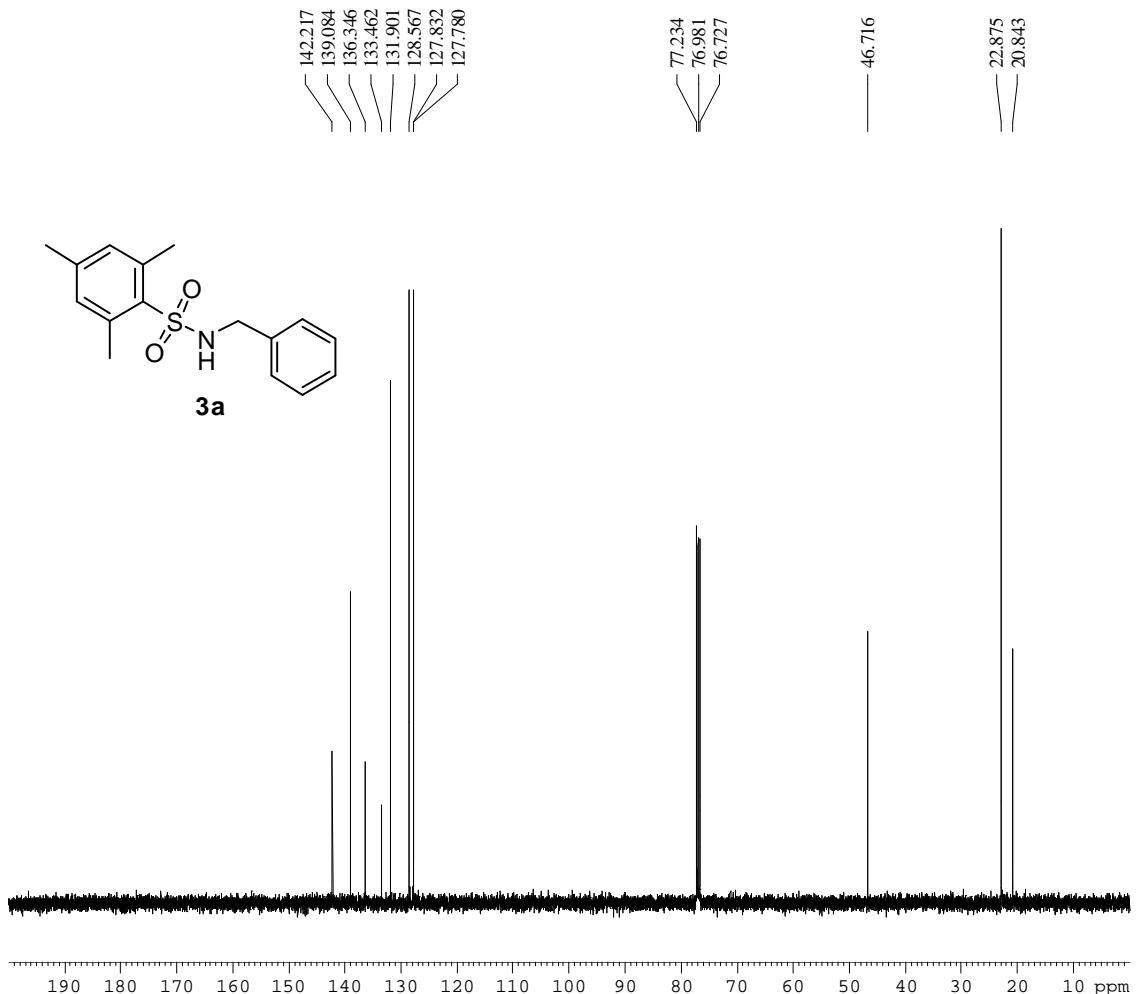


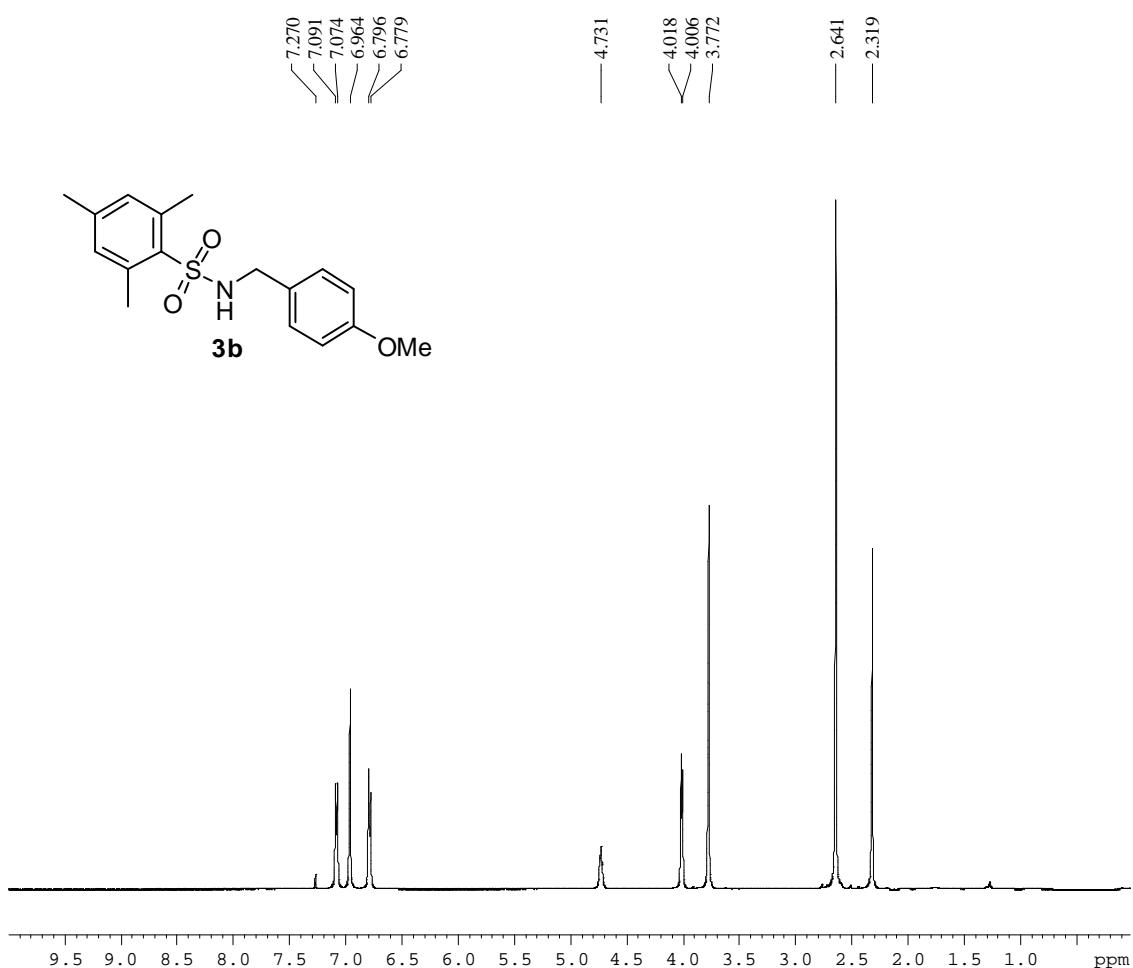
Table2



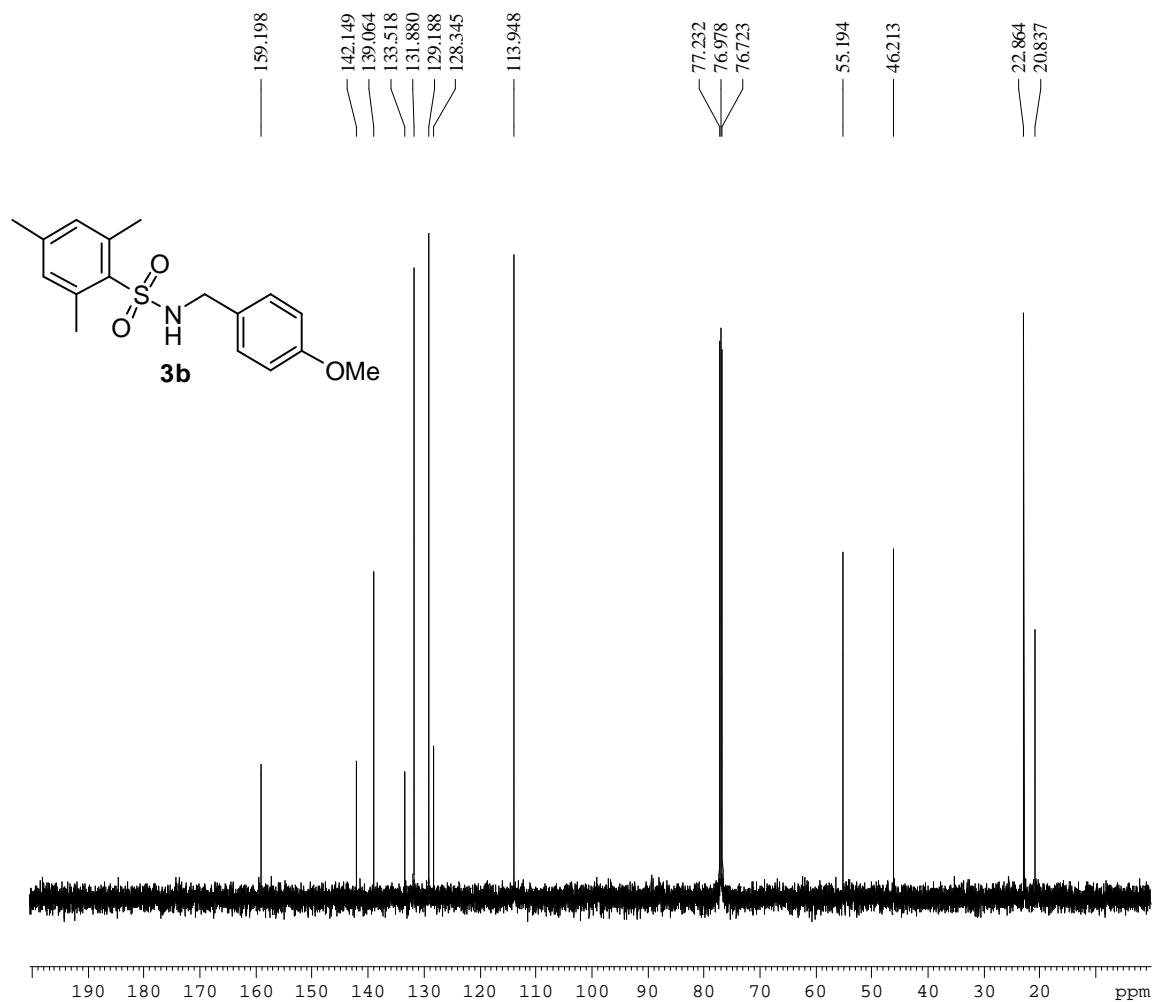
<sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-Benzyl-2,4,6-trimethylbenzenesulfonamide **3a**



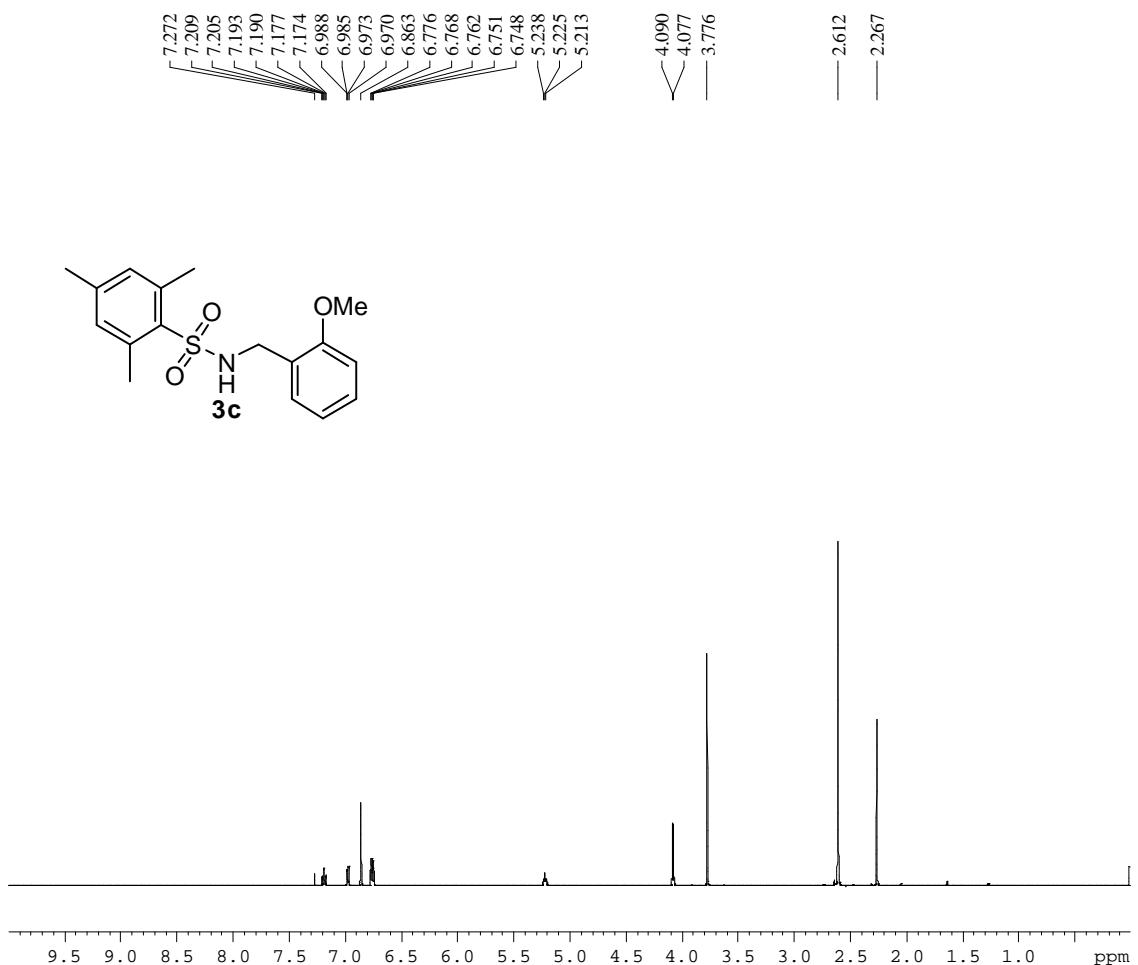
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(4-Methoxybenzyl)-2,4,6-trimethylbenzenesulfonamide **3b**



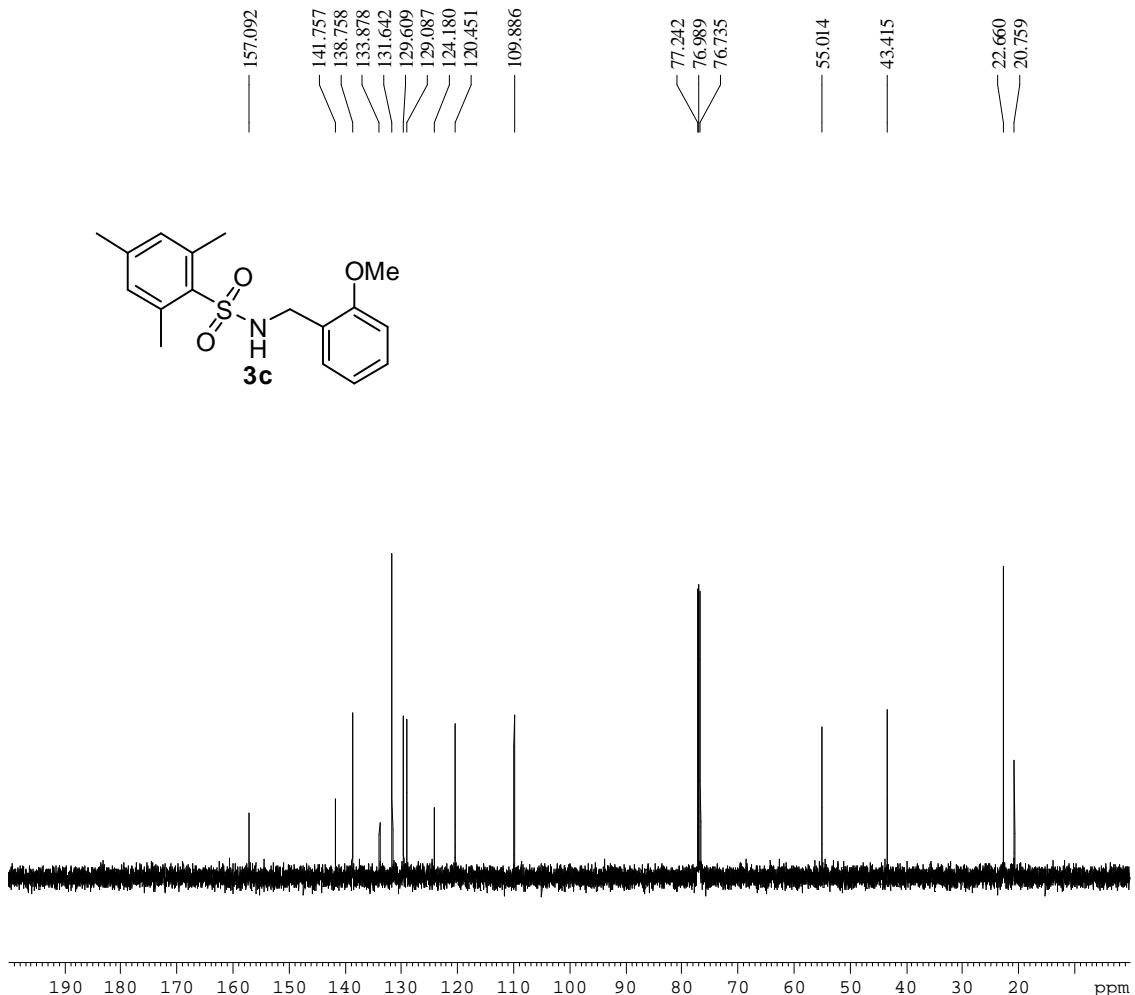
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(4-Methoxybenzyl)-2,4,6-trimethylbenzenesulfonamide **3b**



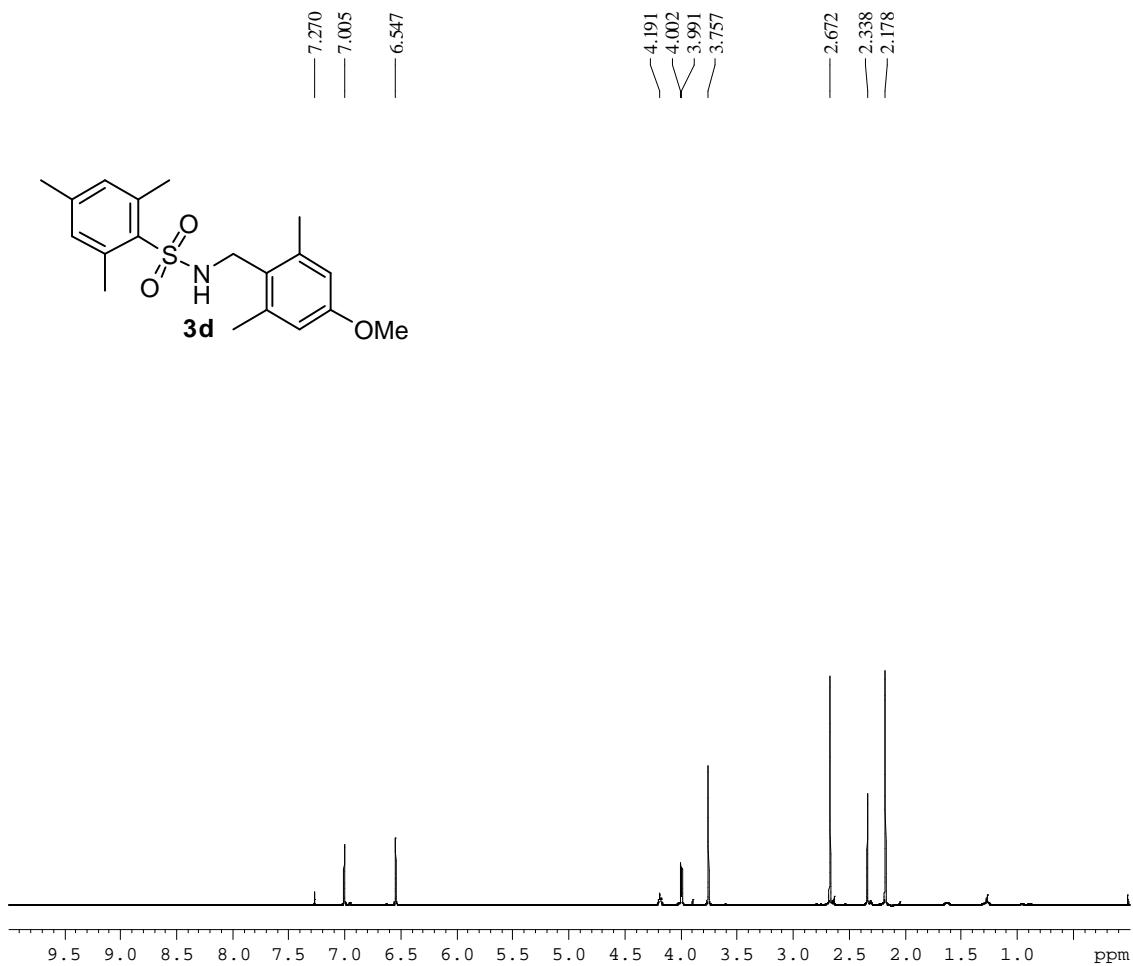
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(2-Methoxy)-2,4,6-trimethylbenzenesulfonamide **3c**



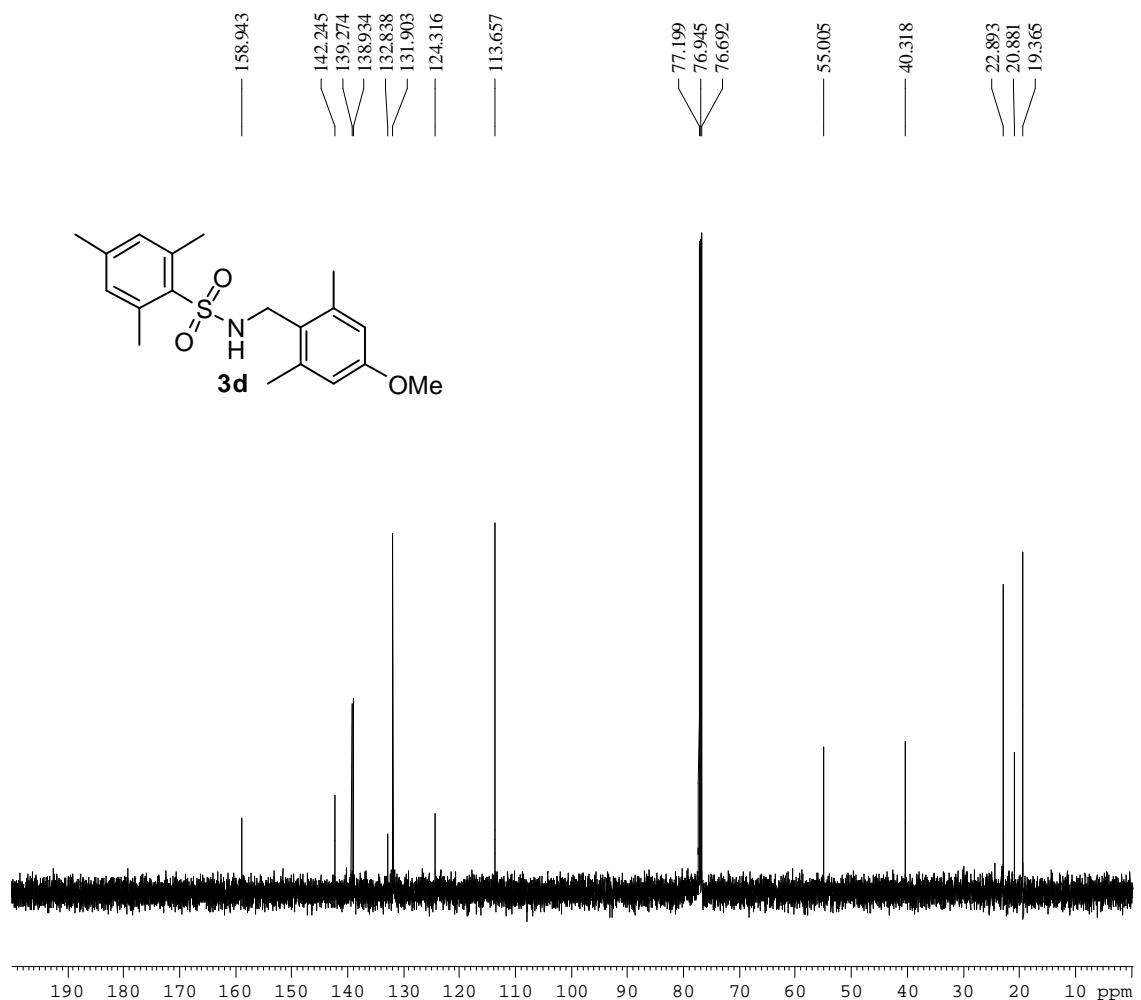
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(2-Methoxy)-2,4,6-trimethylbenzenesulfonamide **3c**



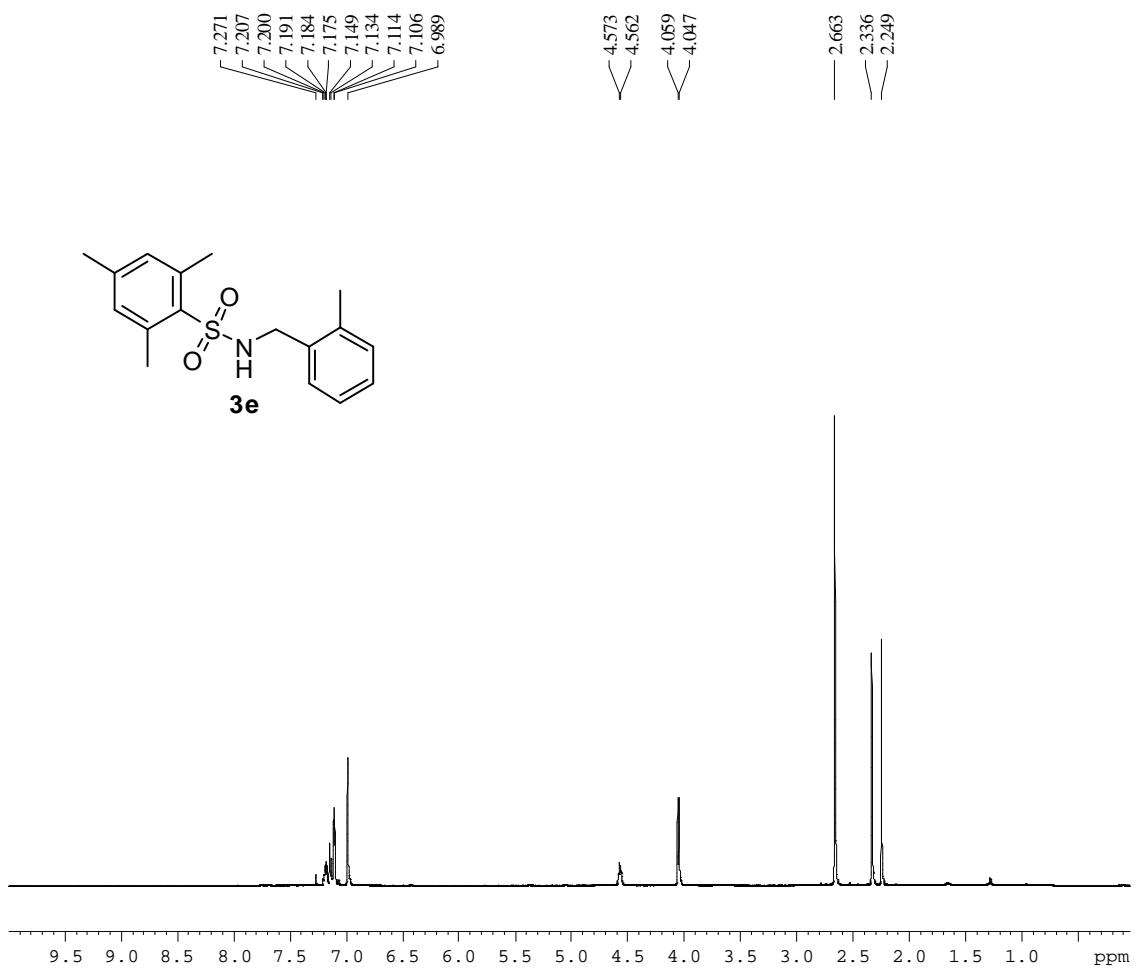
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(4-Methoxy-2,6-dimethylbenzyl)-2,4,6-trimethylbenzenesulfonamide **3d**



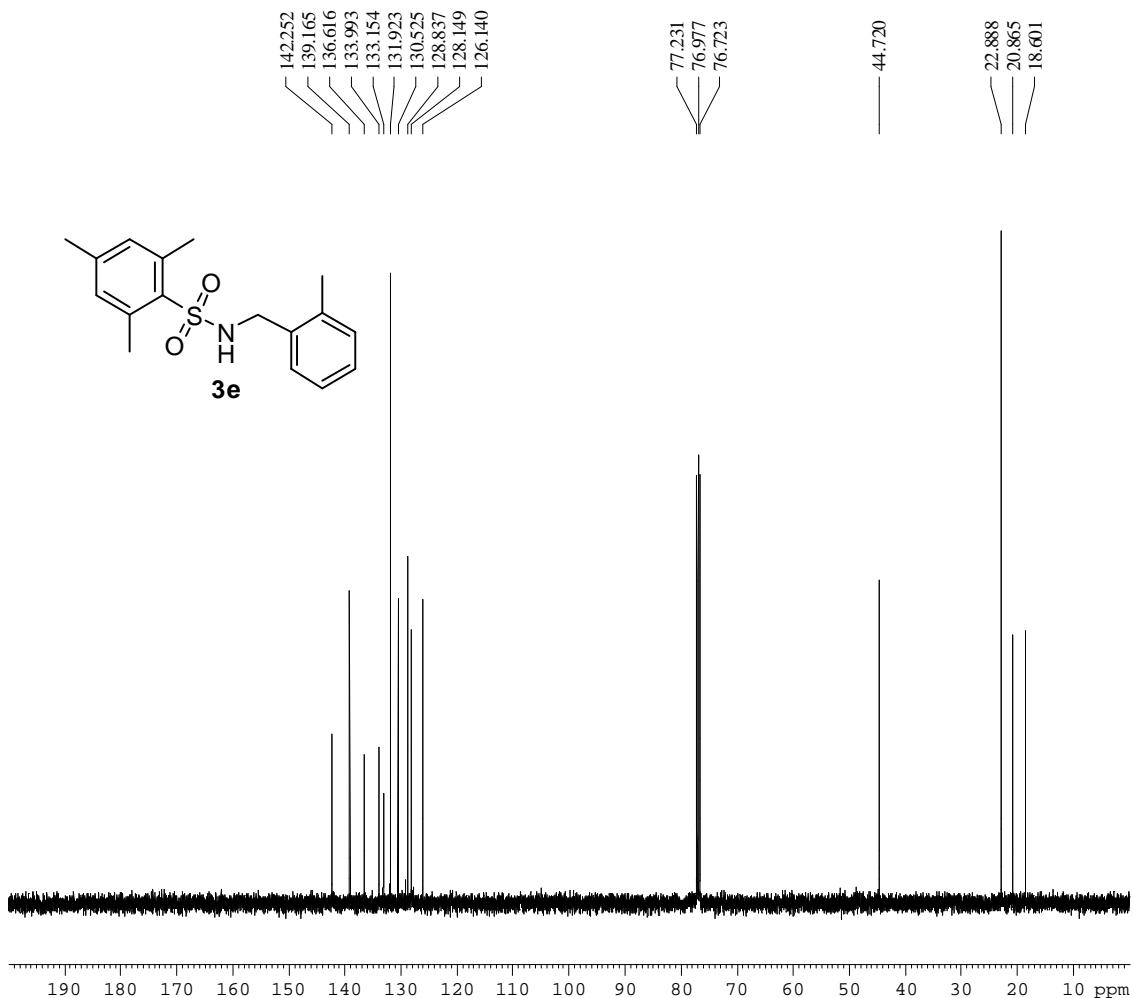
<sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(4-Methoxy-2,6-dimethylbenzyl)-2,4,6-trimethylbenzenesulfonamide **3d**



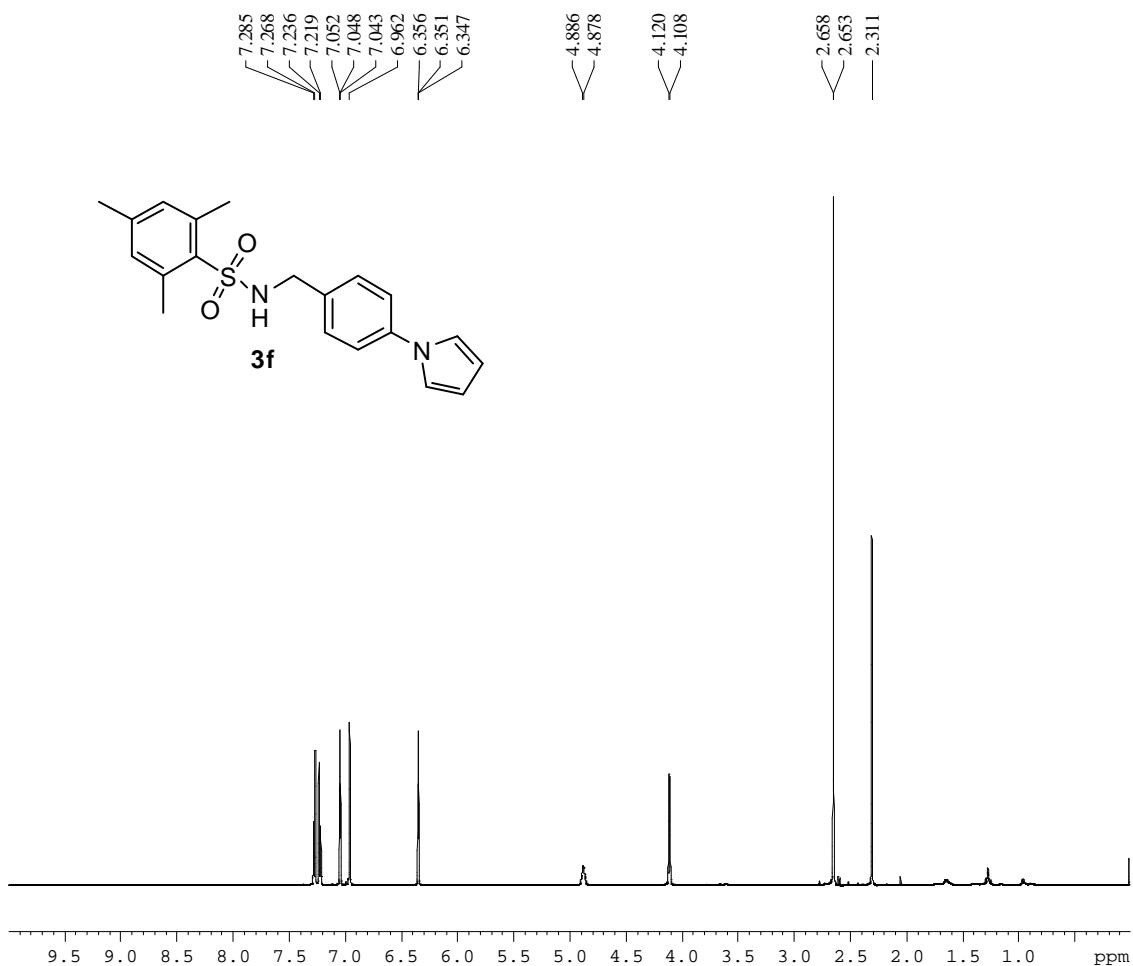
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of 2,4,6-Trimethyl-N-(2-methylbenzyl)benzenesulfonamide **3e**



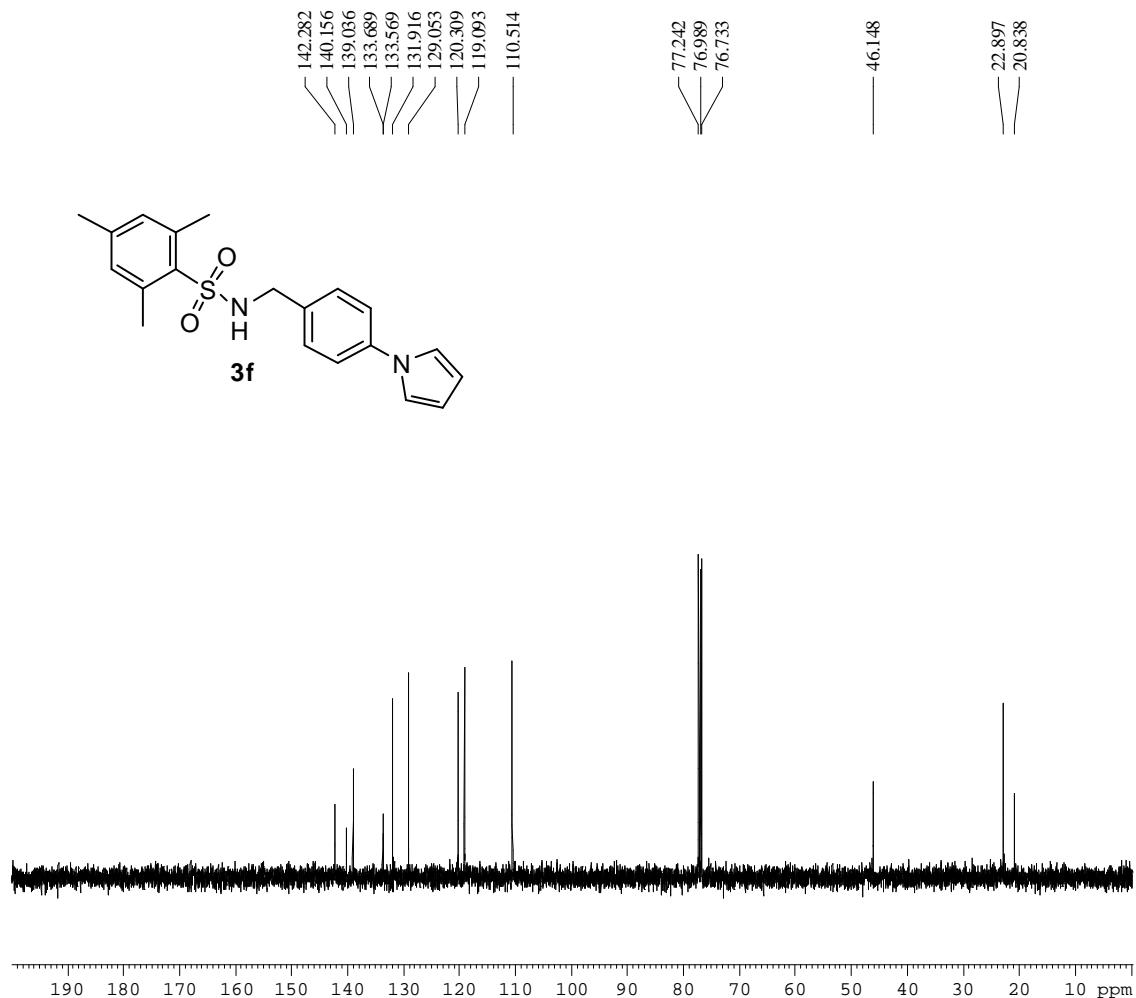
<sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of 2,4,6-Trimethyl-N-(2-methylbenzyl)benzenesulfonamide **3e**



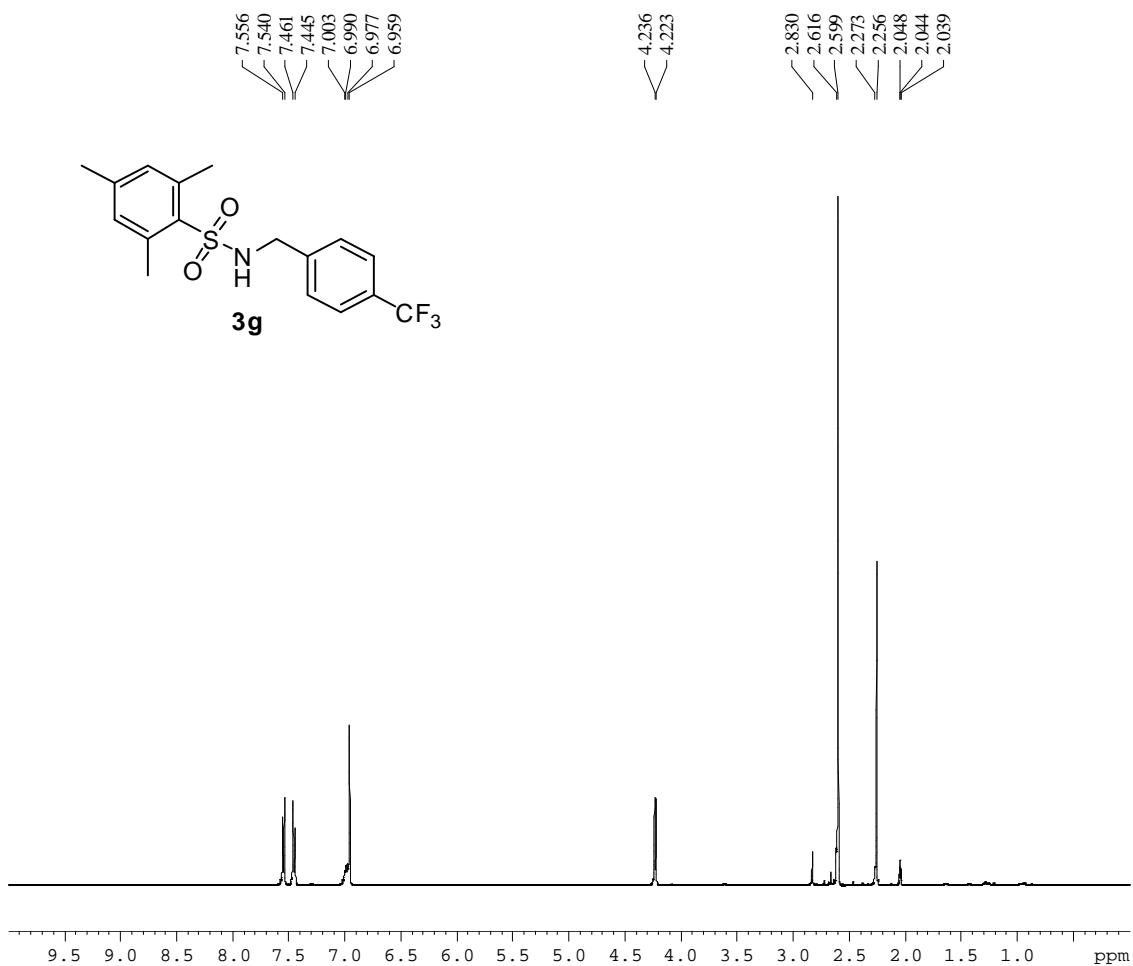
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(4-(1H-Pyrrol-1-yl)benzyl)-2,4,6-trimethylbenzenesulfonamide **3f**



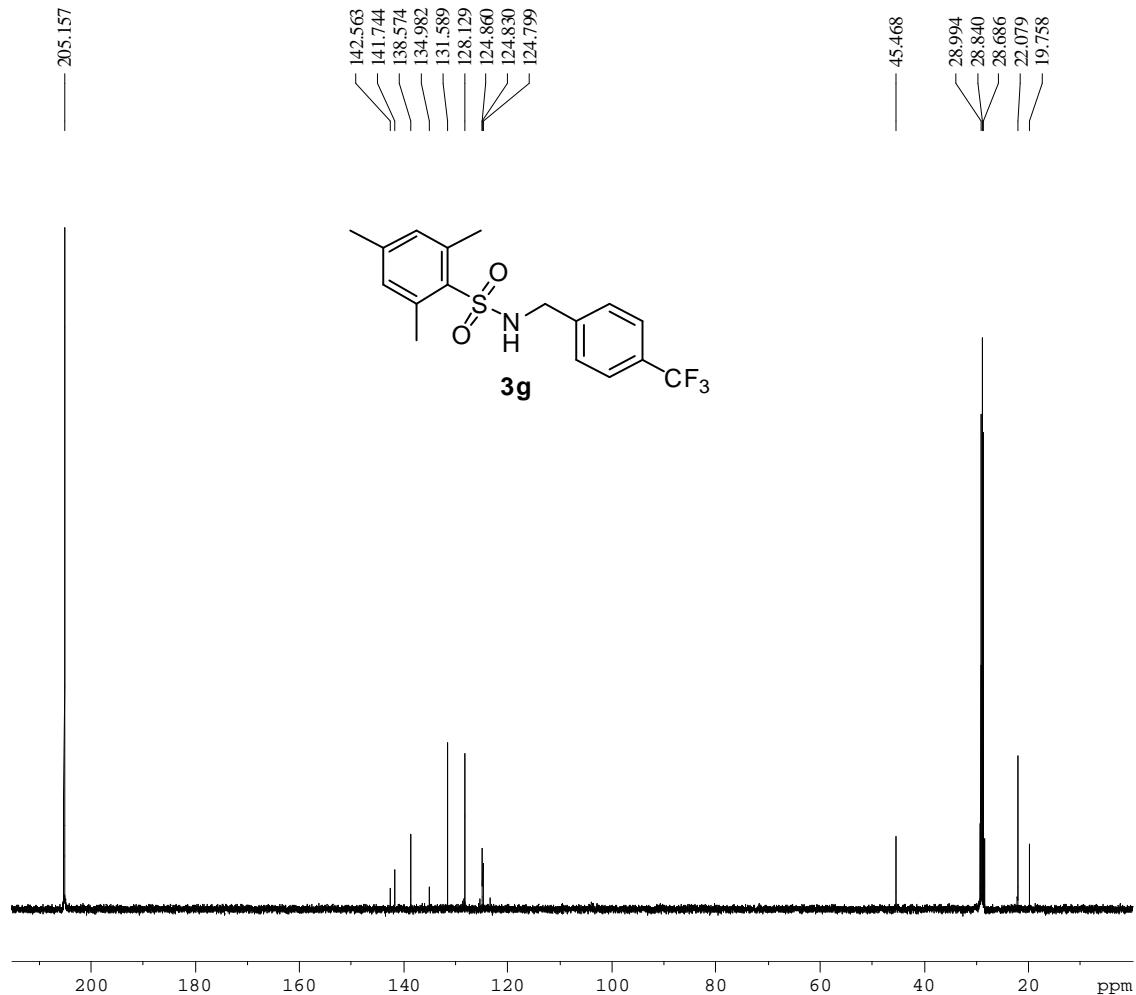
<sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125.6 MHz) spectrum of *N*-(4-(1H-Pyrrol-1-yl)benzyl)-2,4,6-trimethylbenzenesulfonamide **3f**



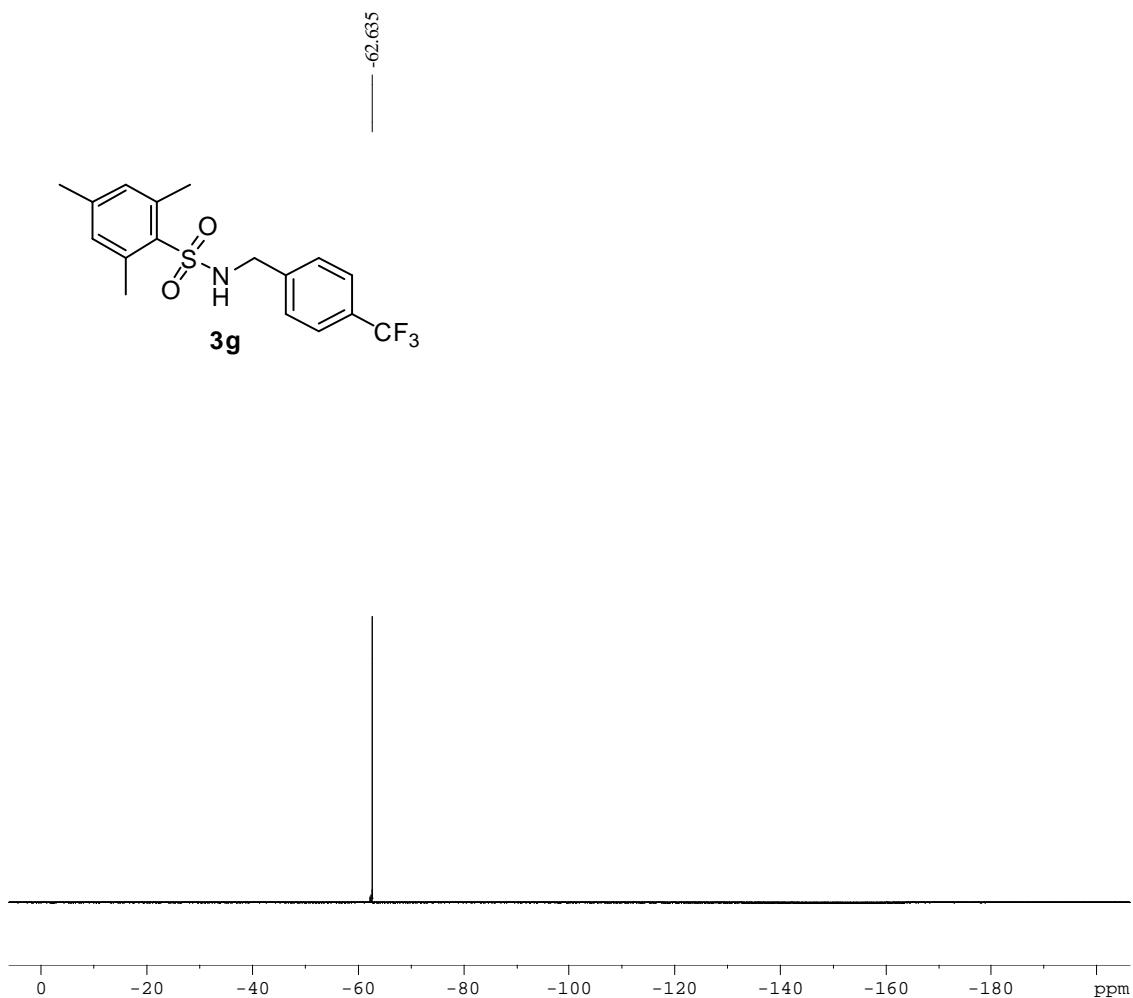
<sup>1</sup>H NMR (acetone, 500 MHz) spectrum of 2,4,6-Trimethyl-N-(4-(trifluoromethyl)benzyl)benzenesulfonamide **3g**



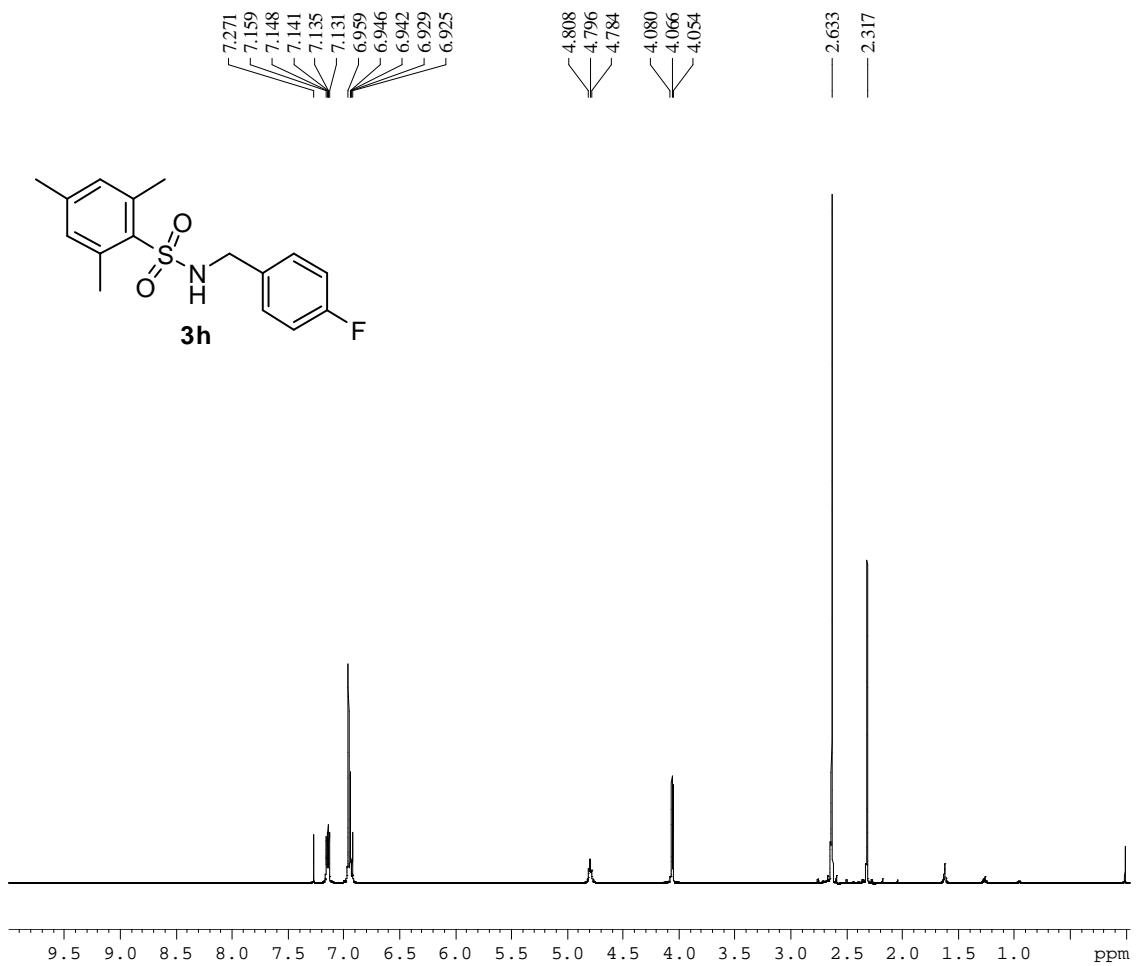
<sup>13</sup>C NMR (acetone, 125.8 MHz) spectrum of 2,4,6-Trimethyl-N-(4-(trifluoromethyl)benzyl)benzenesulfonamide **3g**



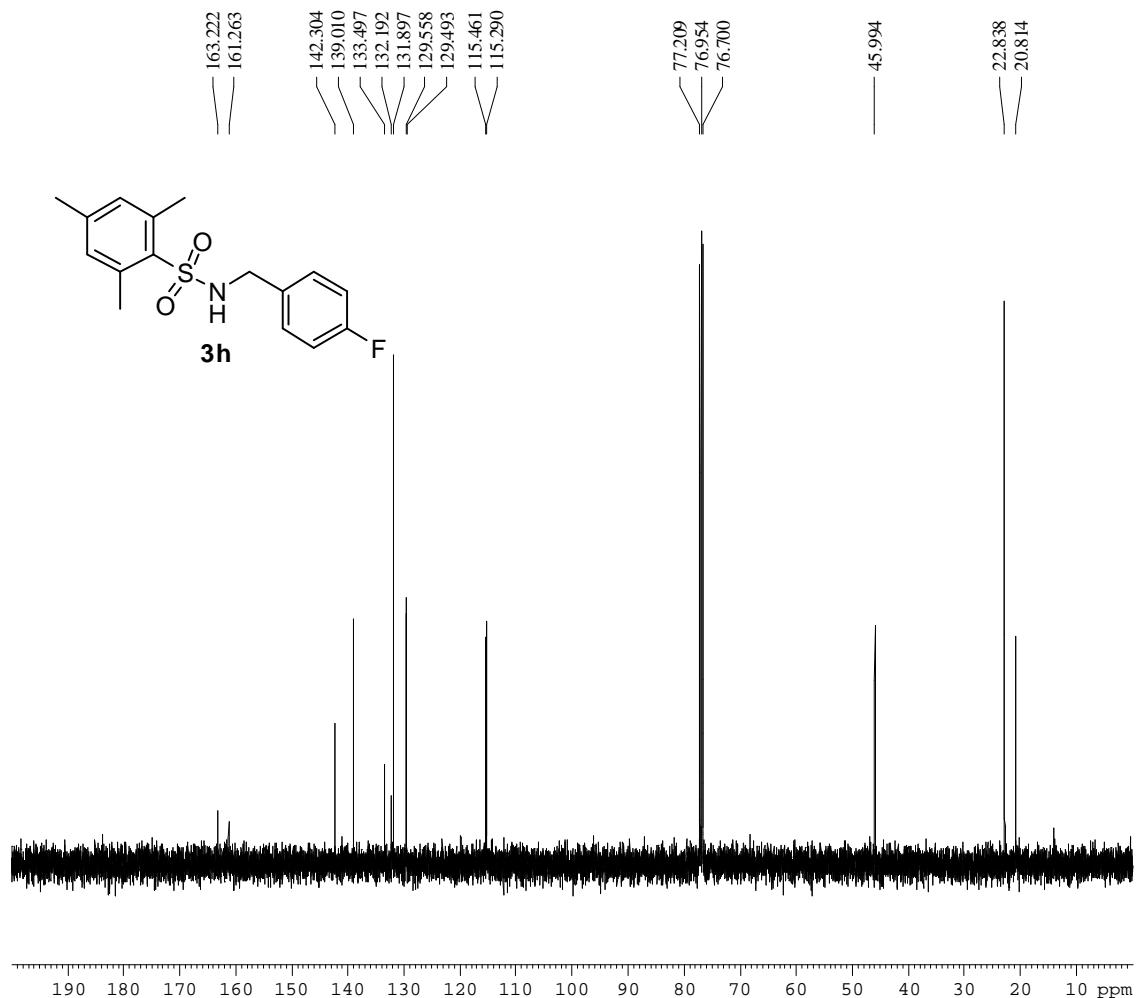
<sup>19</sup>F NMR (acetone, 500 MHz) spectrum of 2,4,6-Trimethyl-N-(4-(trifluoromethyl)benzyl)benzenesulfonamide **3g**



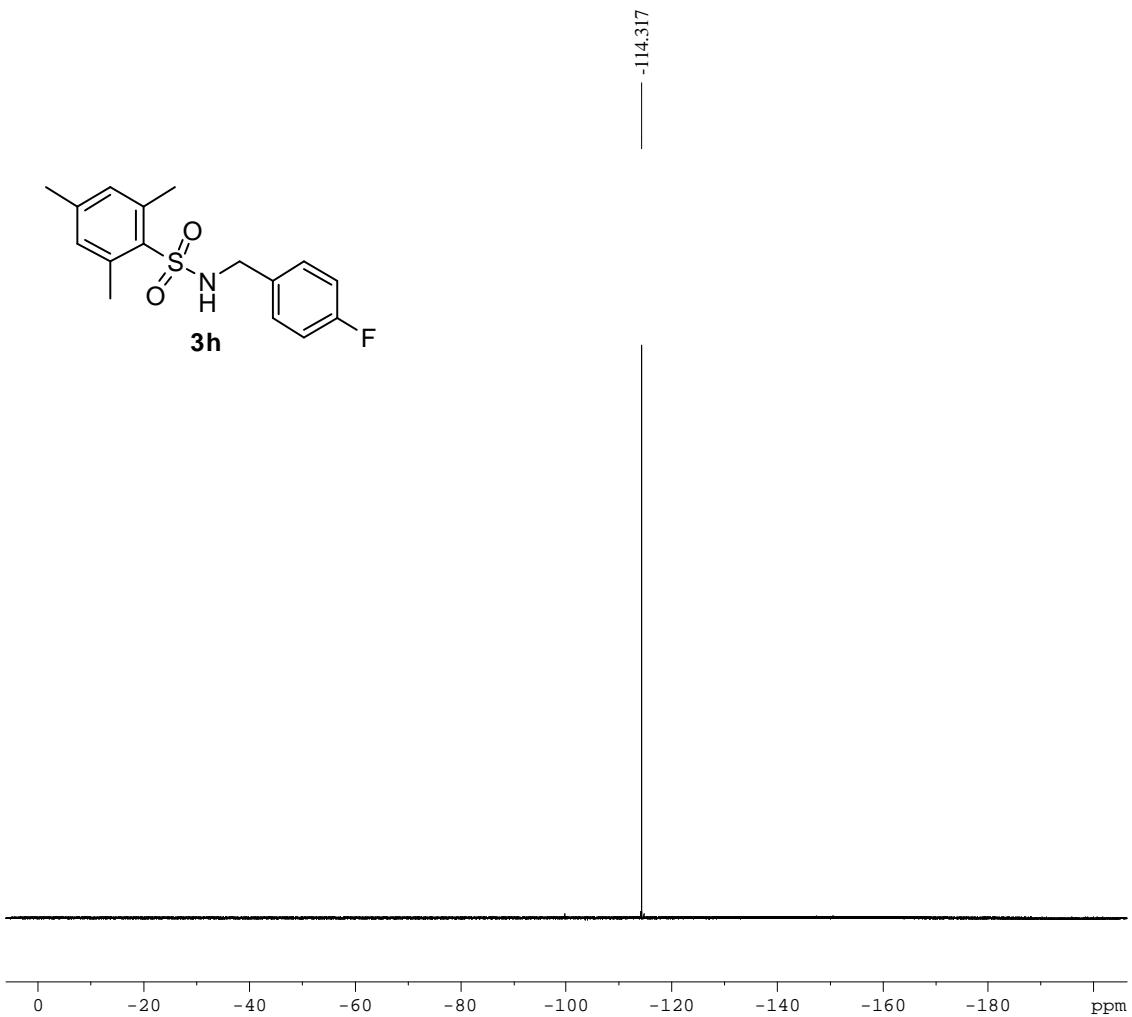
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(4-Fluorobenzyl)-2,4,6-trimethylbenzenesulfonamide **3h**



$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(4-Fluorobenzyl)-2,4,6-trimethylbenzenesulfonamide **3h**



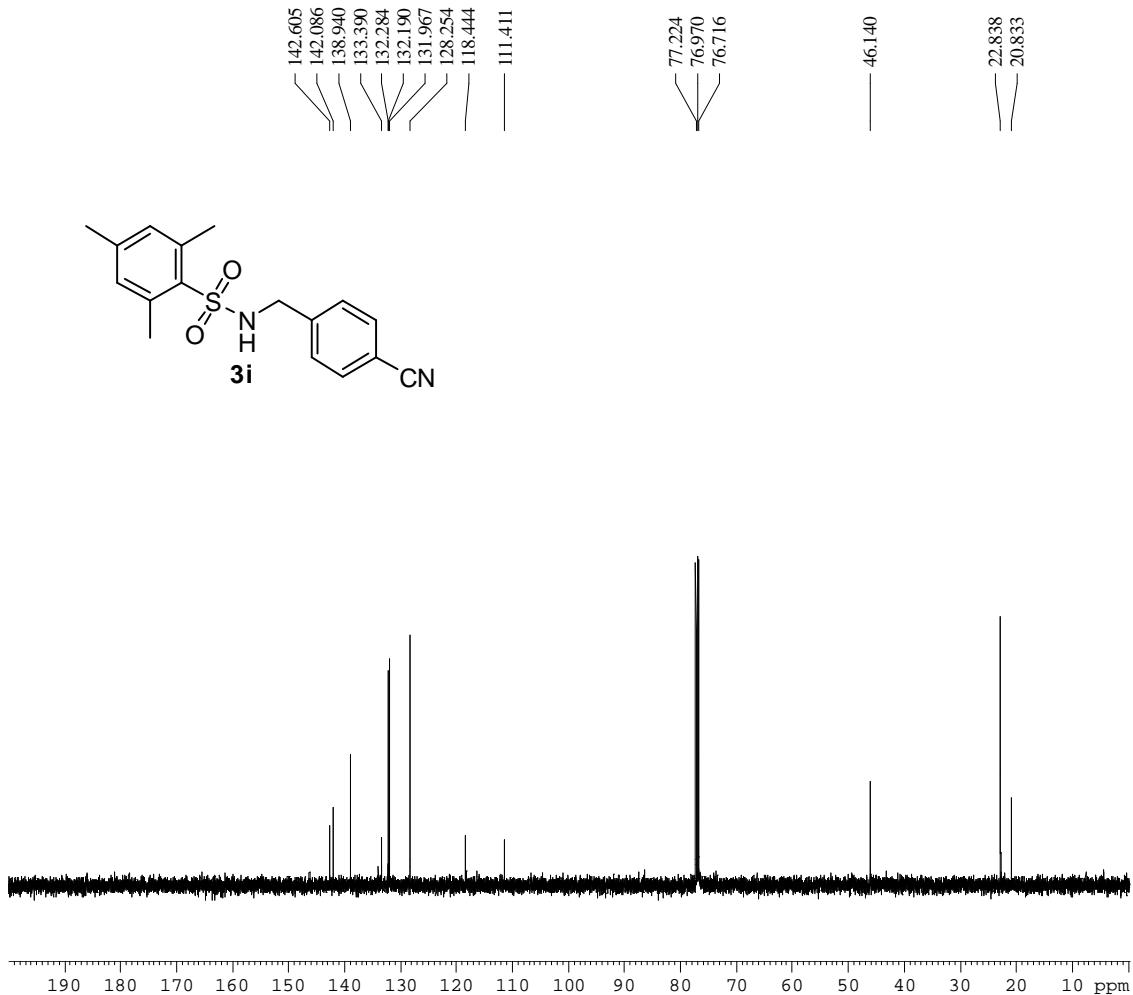
<sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470.8 MHz) spectrum of *N*-(4-Fuorobenzyl)-2,4,6-trimethylbenzenesulfonamide **3h**



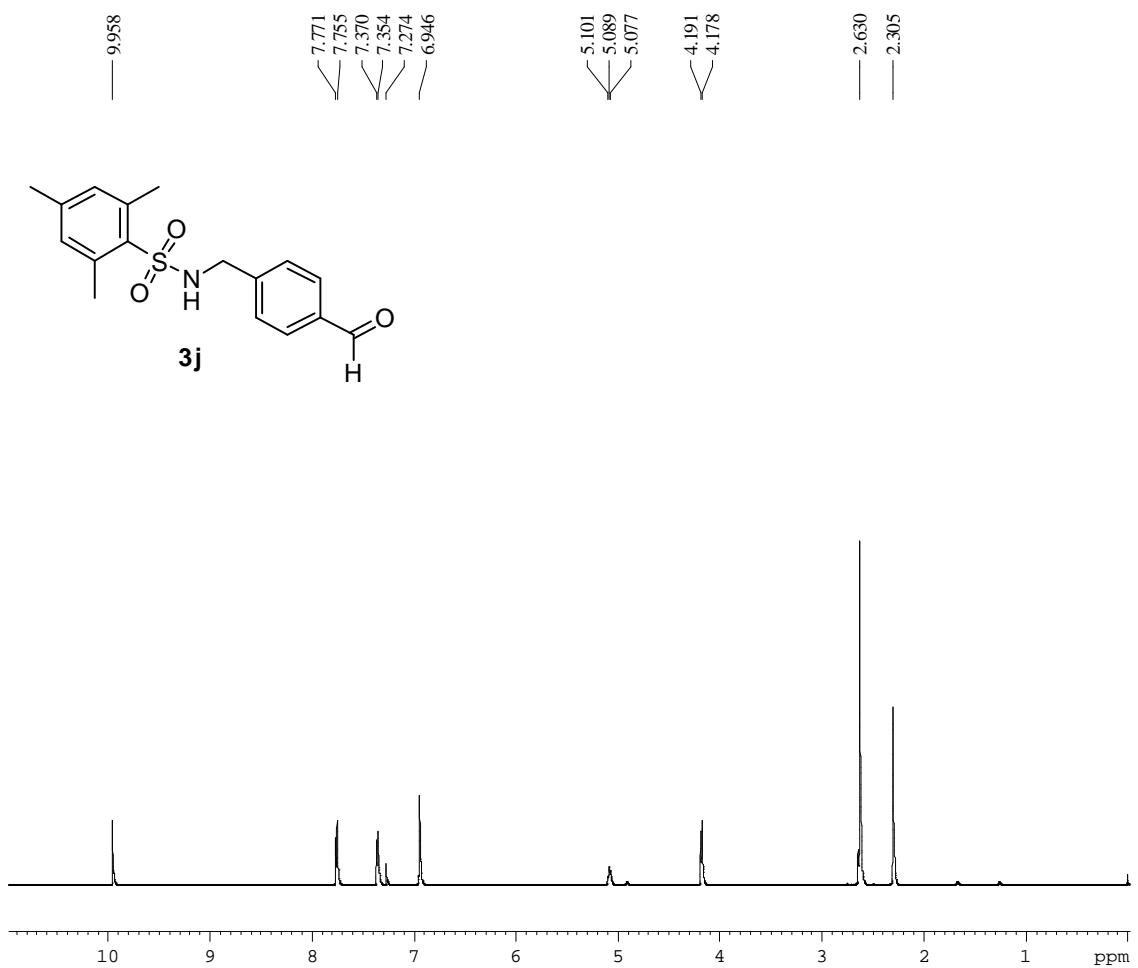
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(4-Cyanobenzyl)-2,4,6-trimethylbenzenesulfonamide **3i**



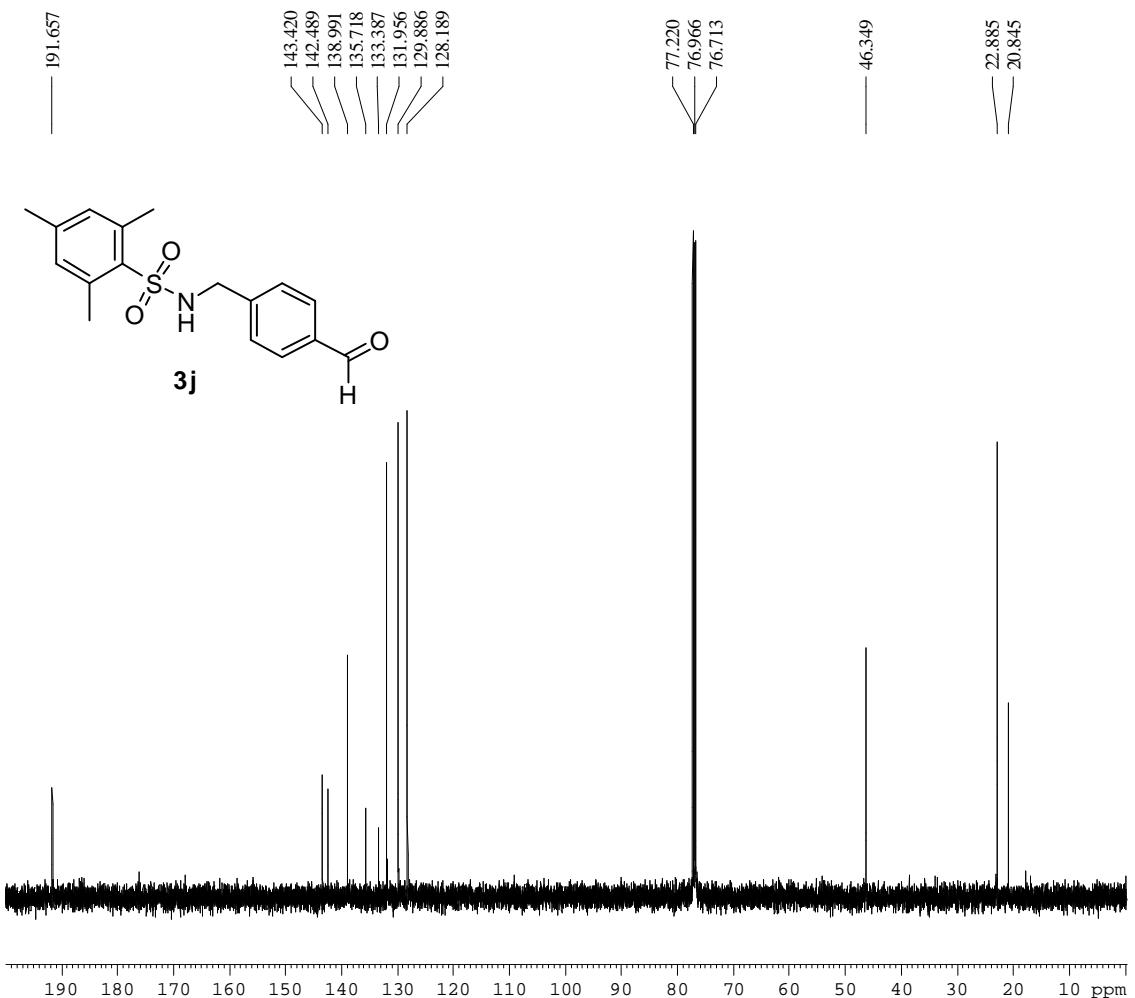
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(4-Cyanobenzyl)-2,4,6-trimethylbenzenesulfonamide **3i**



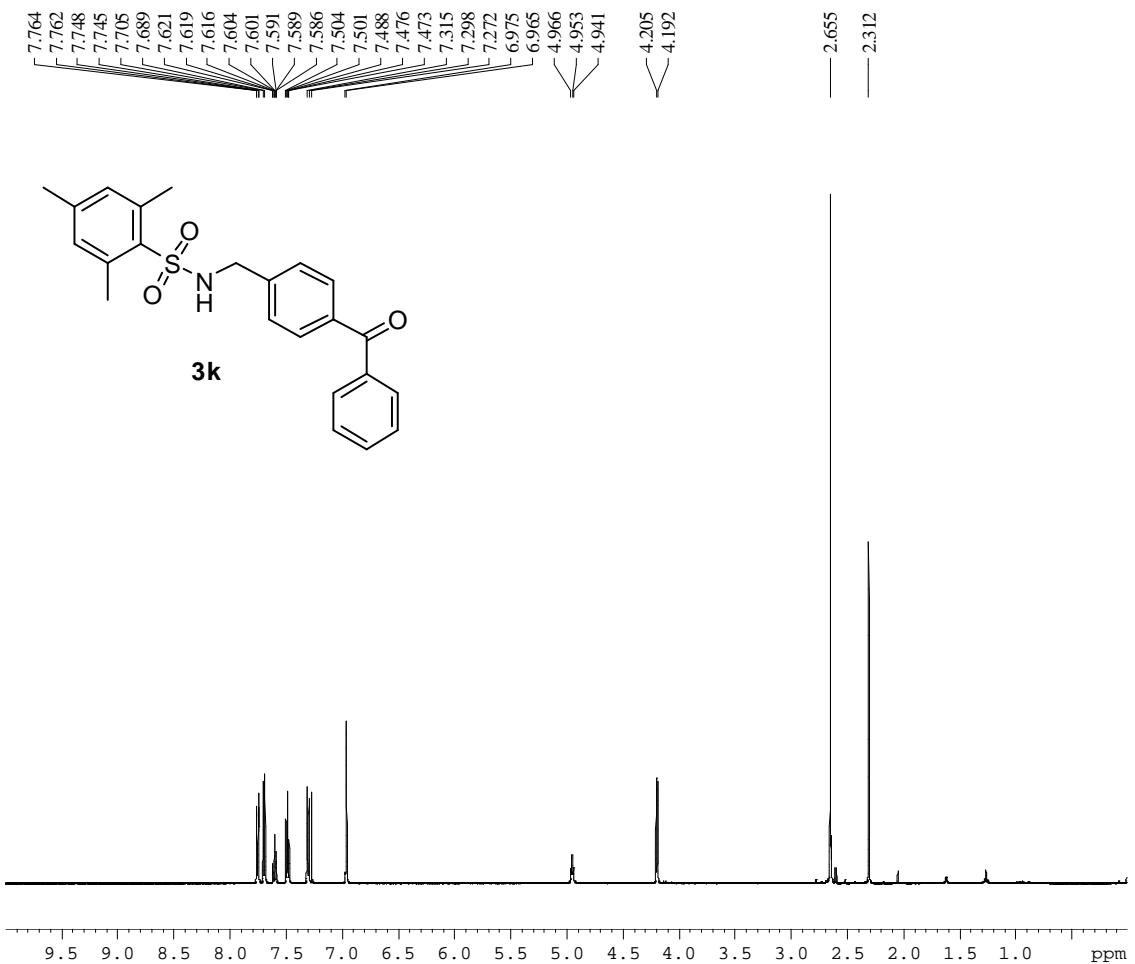
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(4-Formylbenzyl)-2,4,6-trimethylbenzenesulfonamide **3j**



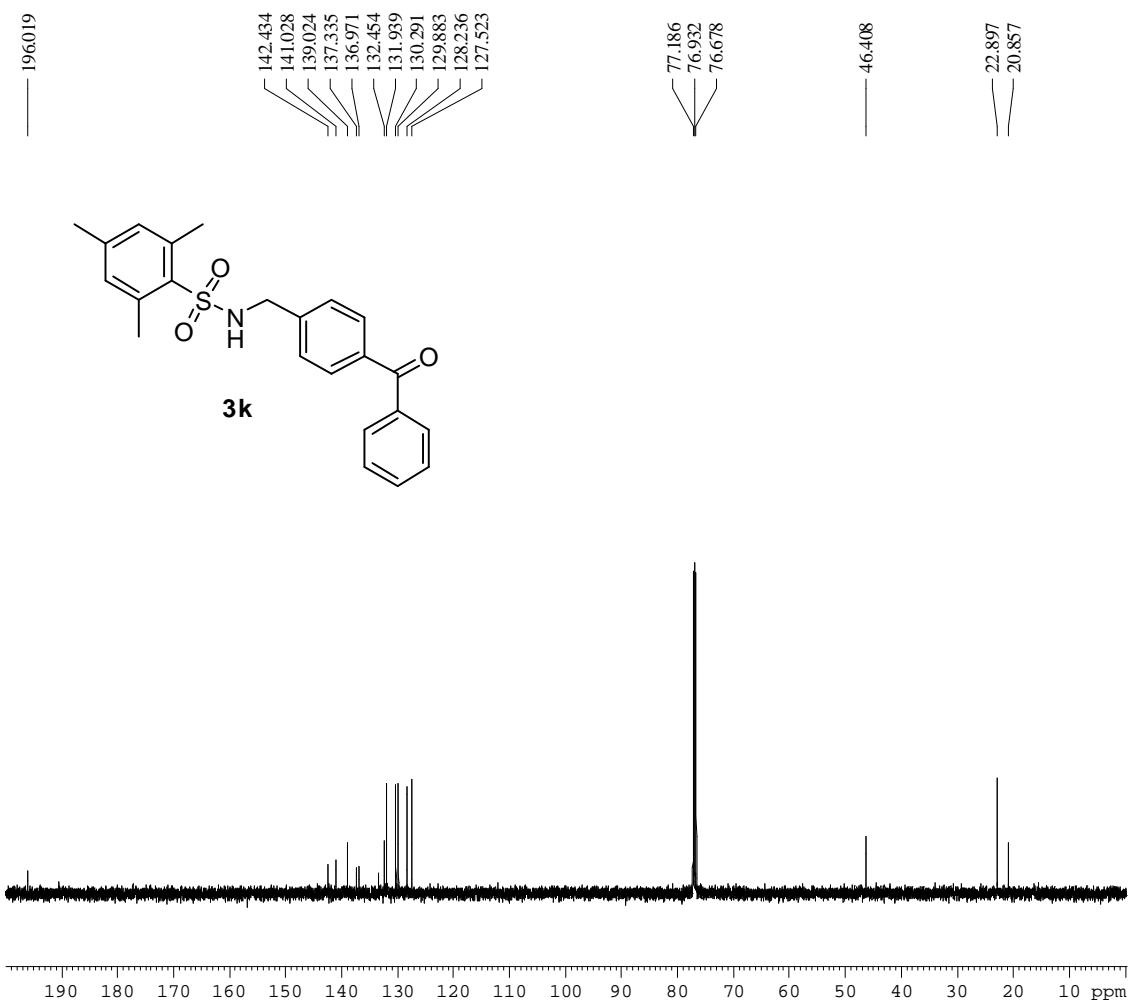
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(4-Formylbenzyl)-2,4,6-trimethylbenzenesulfonamide **3j**



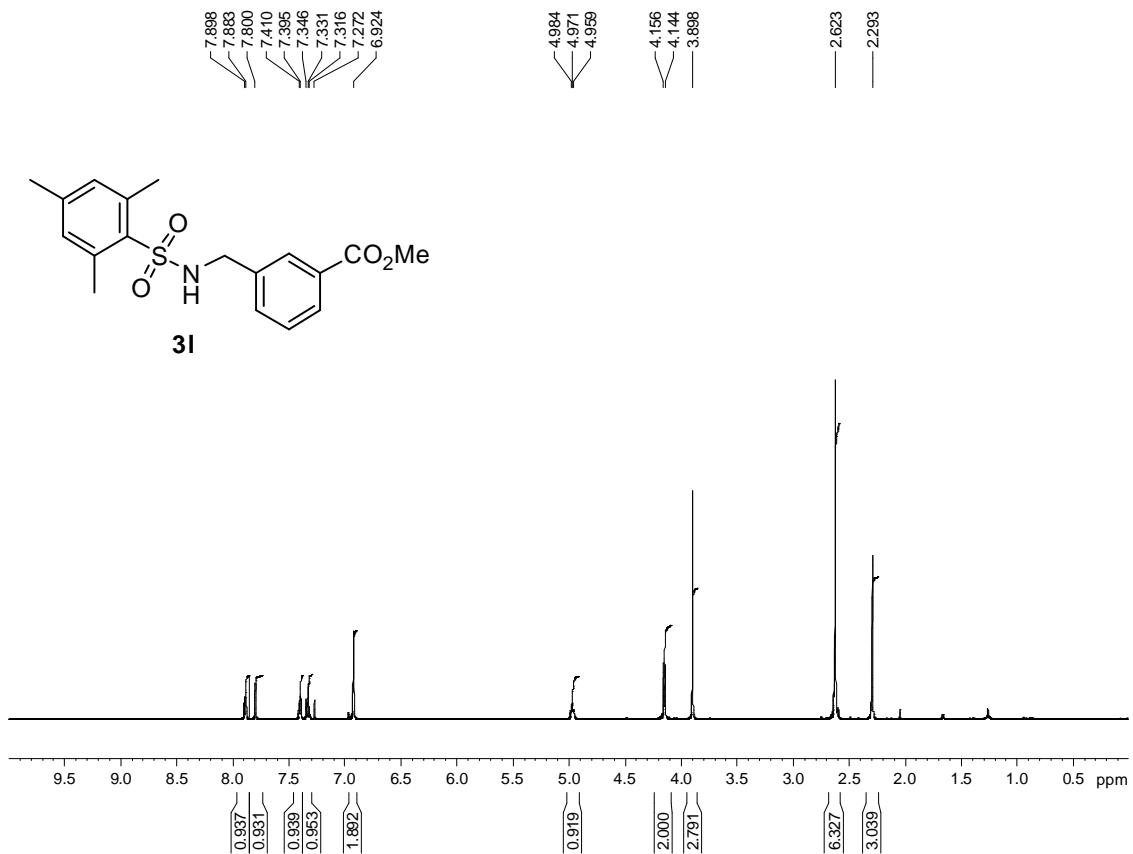
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(4-Benzoylbenzyl)-2,4,6-trimethylbenzenesulfonamide **3k**



<sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(4-Benzoylbenzyl)-2,4,6-trimethylbenzenesulfonamide **3k**



<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of Methyl 3-((2,4,6-trimethylphenylsulfonamido)methyl)benzoate **3I**



<sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of Methyl 3-((2,4,6-trimethylphenylsulfonamido)methyl)benzoate **3l**

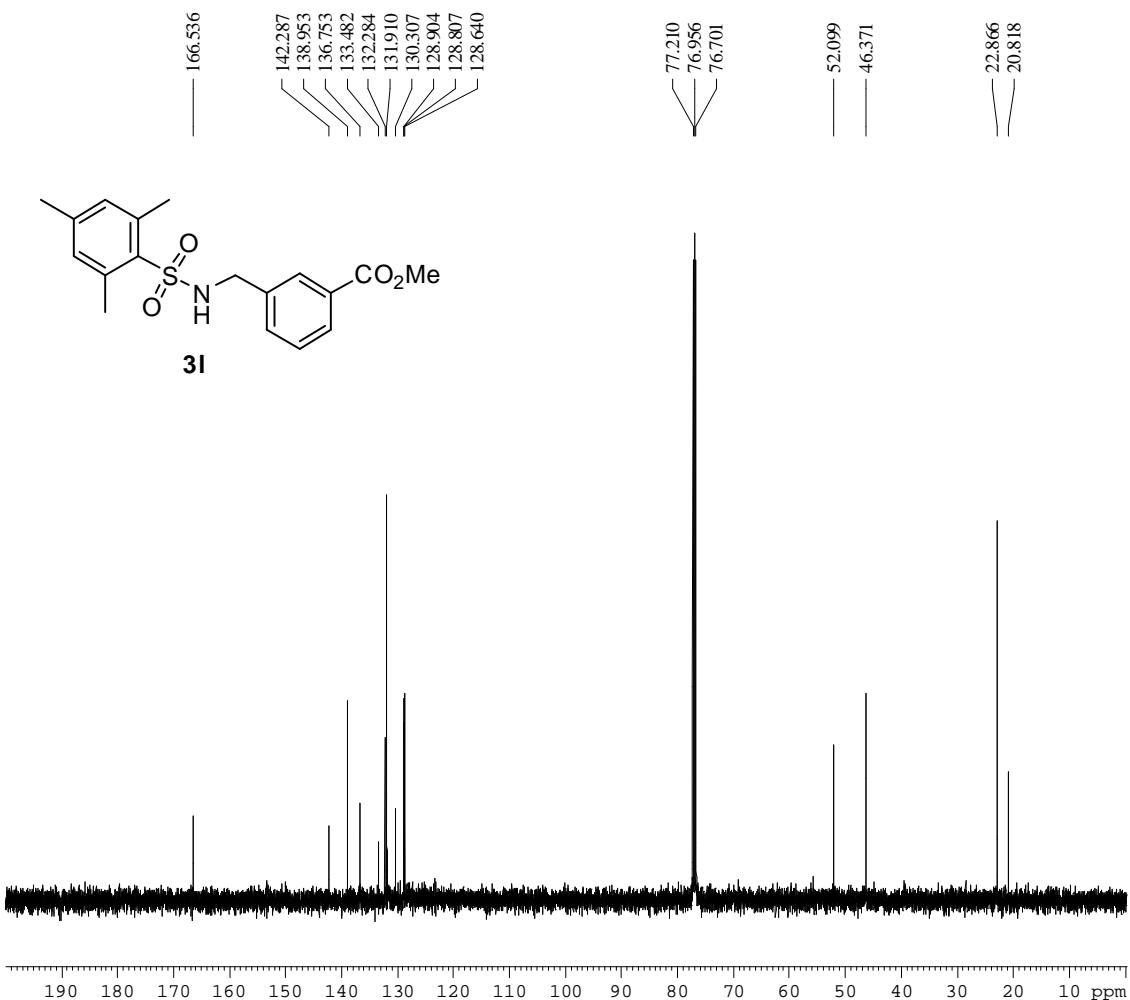
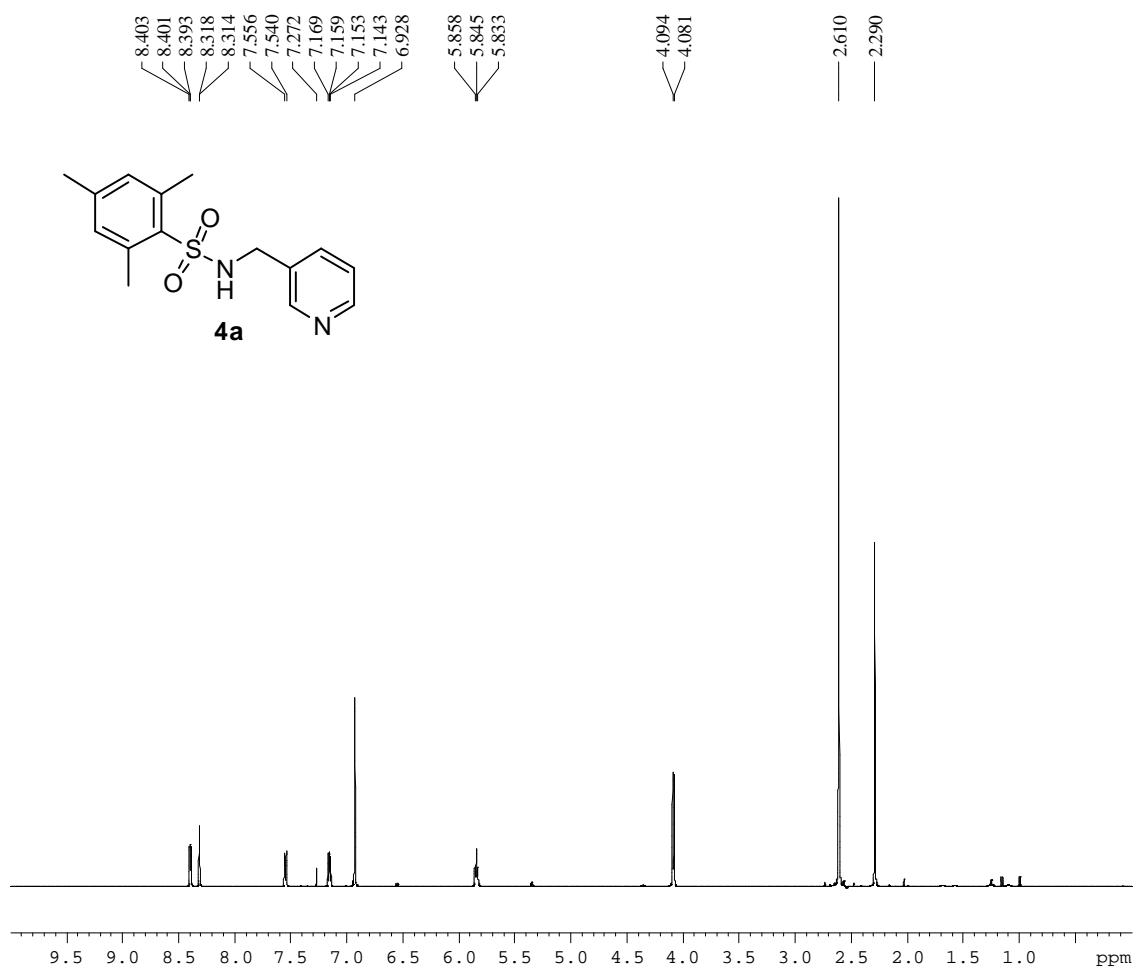
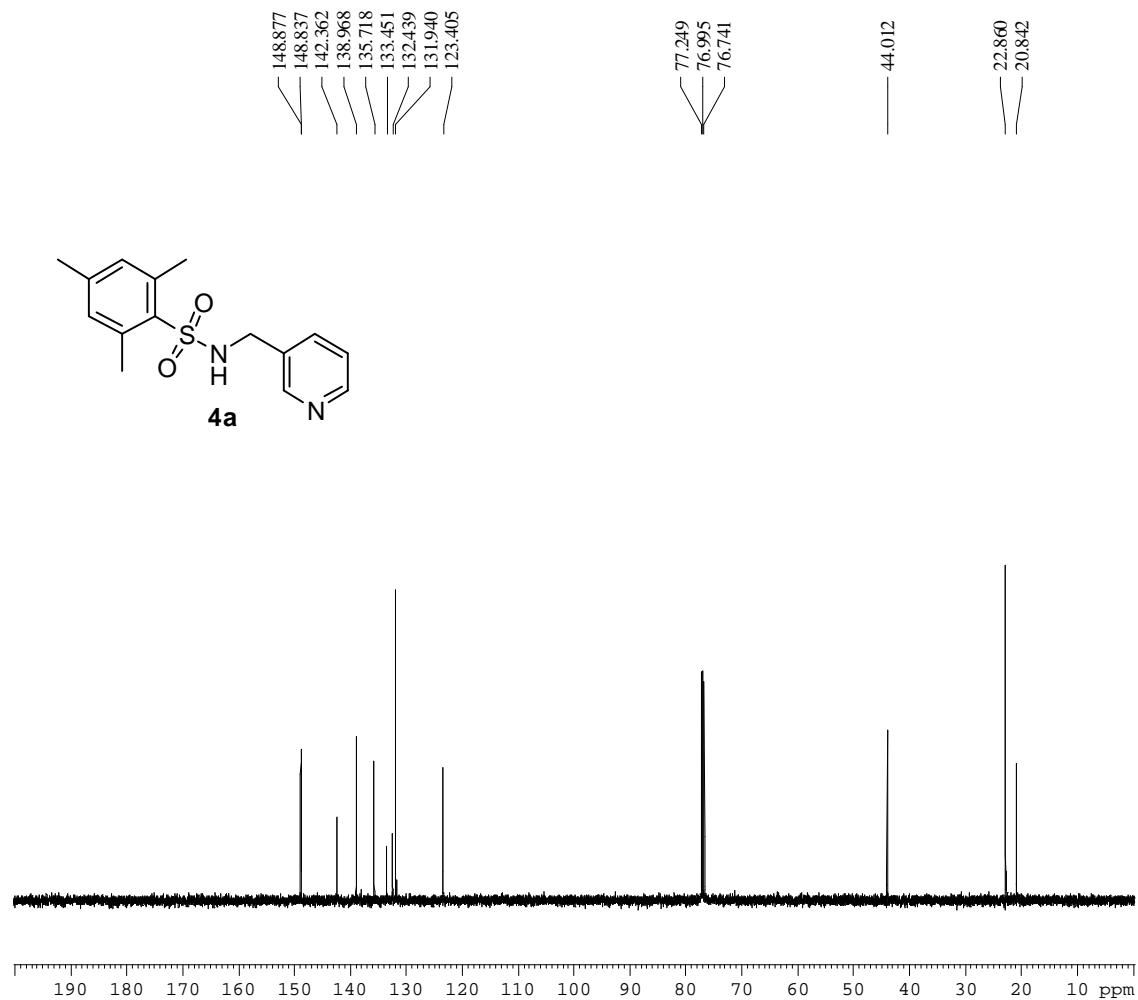


Table3

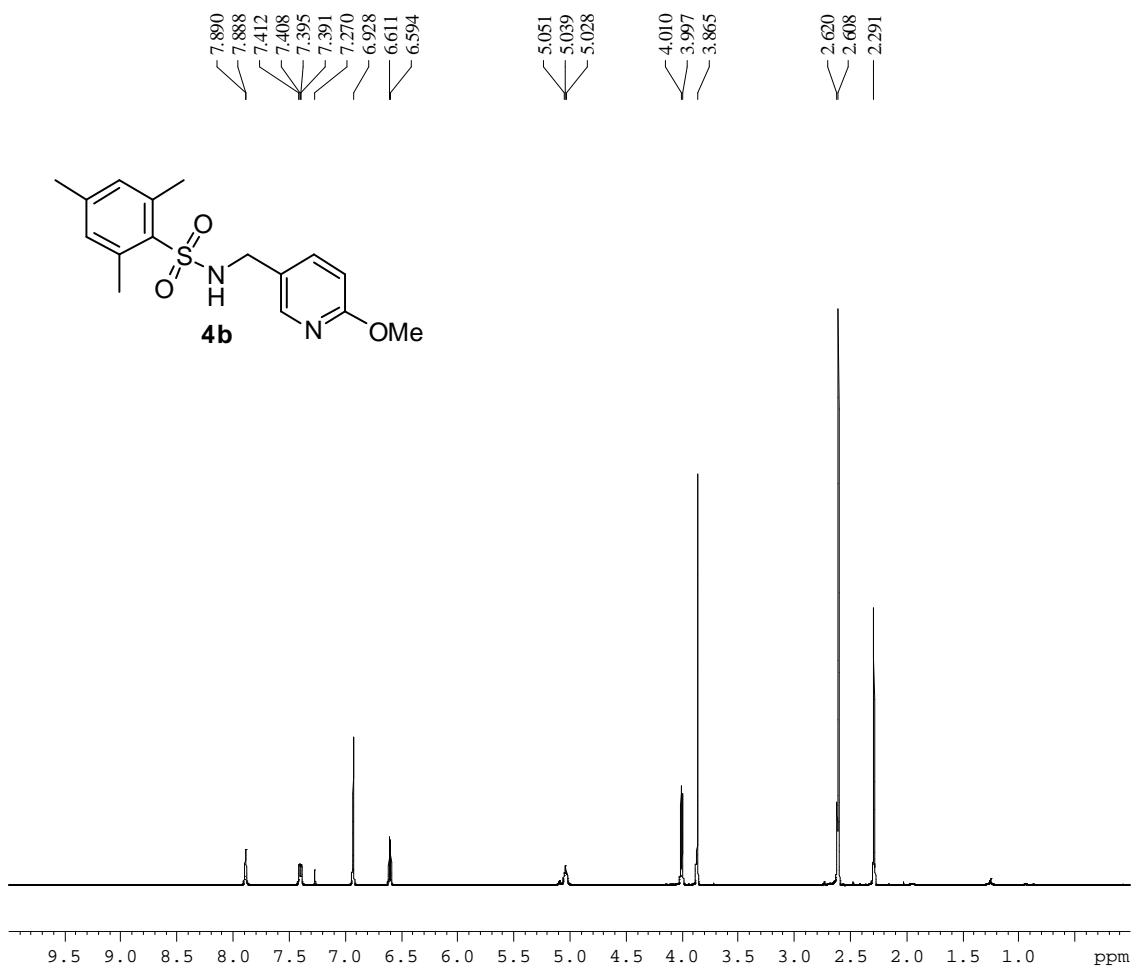
$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of 2,4,6-Trimethyl-*N*-(pyridin-3-ylmethyl)benzenesulfonamide **4a**



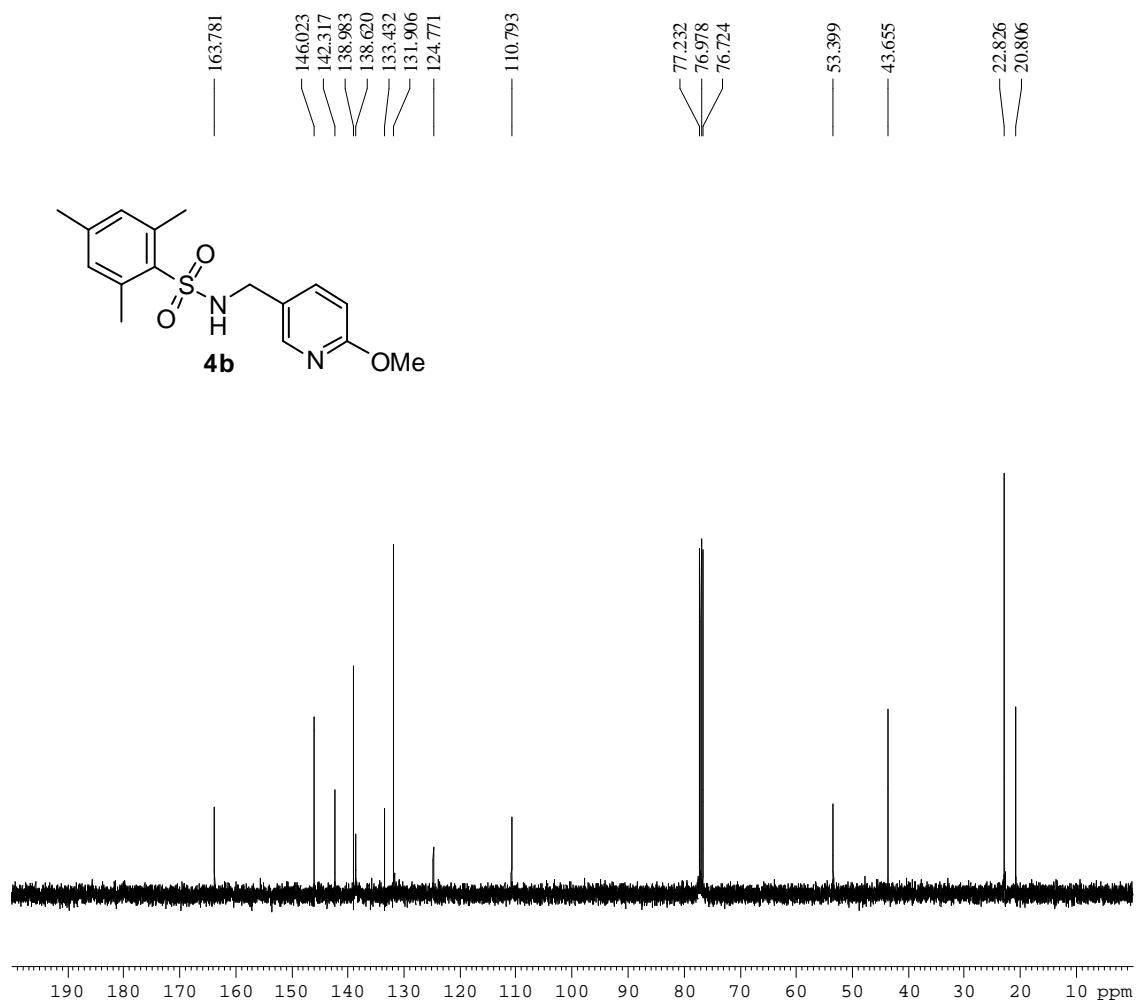
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of 2,4,6-Trimethyl-N-(pyridin-3-ylmethyl)benzenesulfonamide **4a**



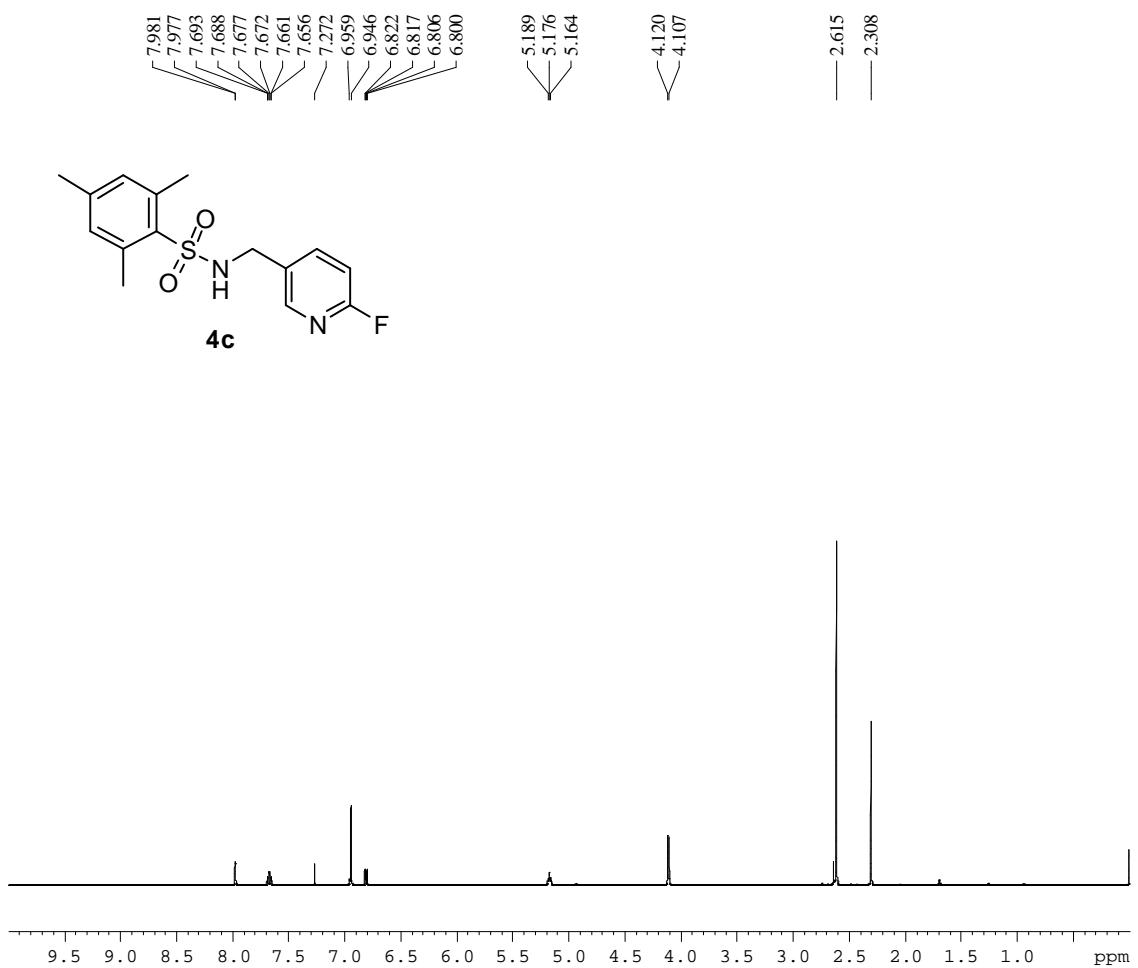
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(6-Methoxypyridin-3-yl)methyl)-2,4,6-trimethylbenzenesulfonamide **4b**



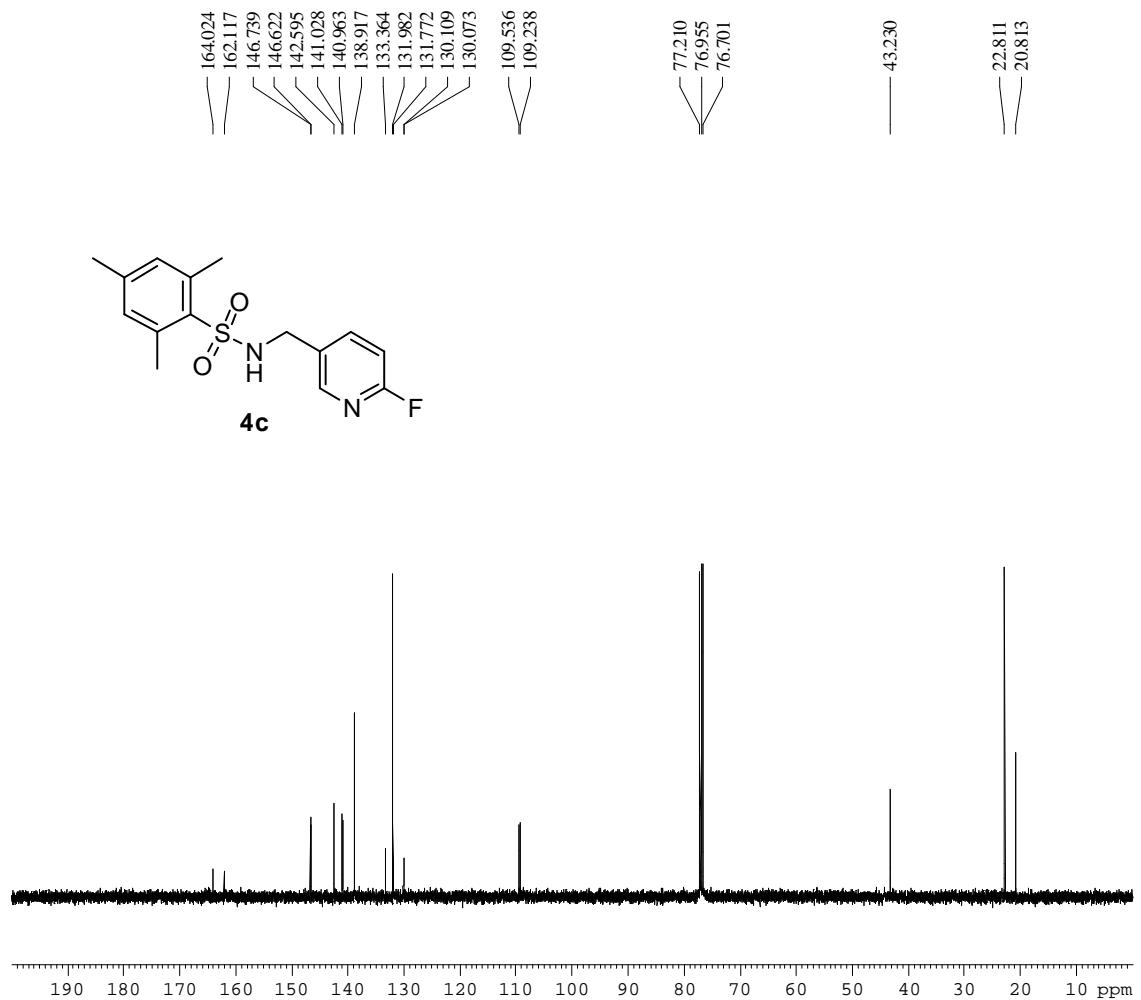
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(6-Methoxypyridin-3-yl)methyl)-2,4,6-trimethylbenzenesulfonamide **4b**



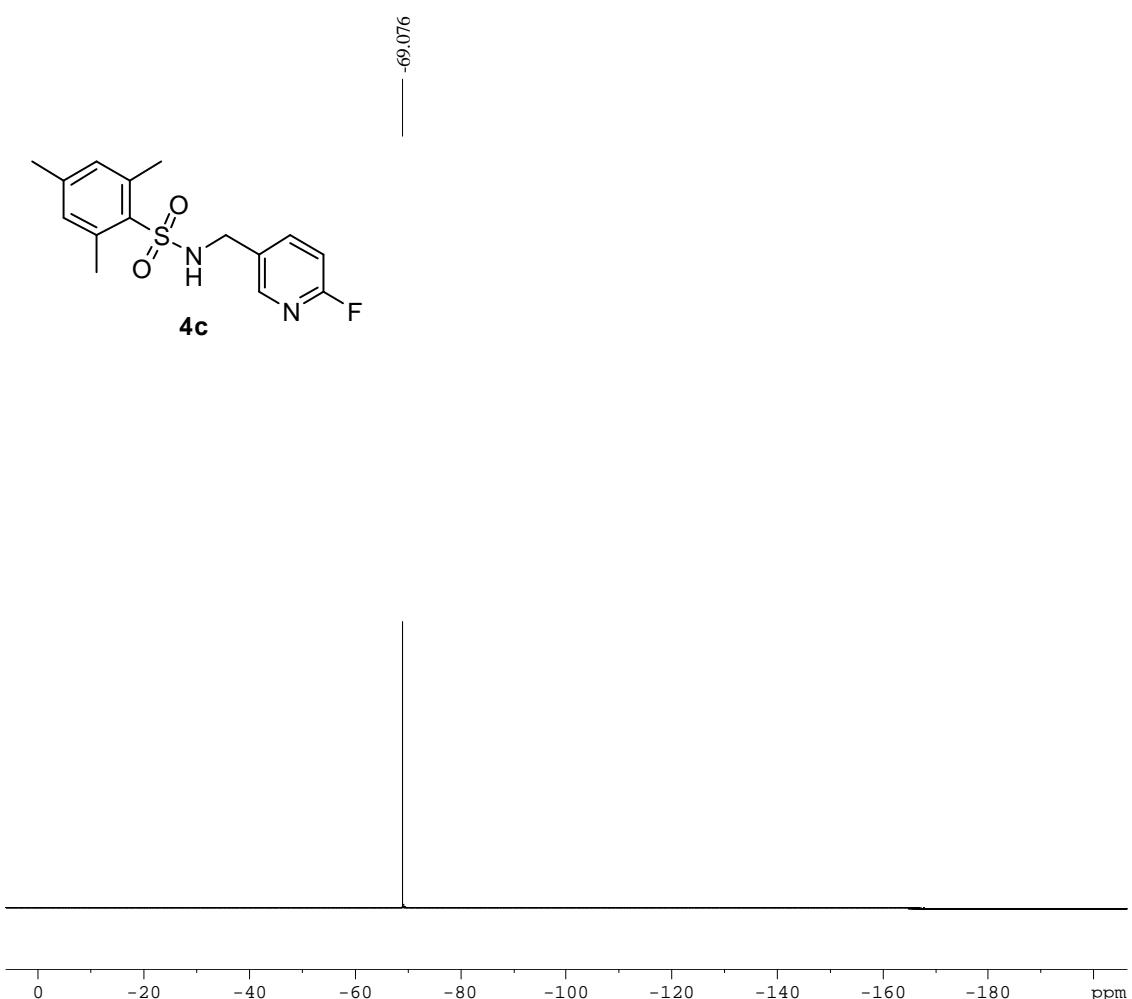
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(6-Fluoropyridin-3-yl)methyl)-2,4,6-trimethylbenzenesulfonamide **4c**



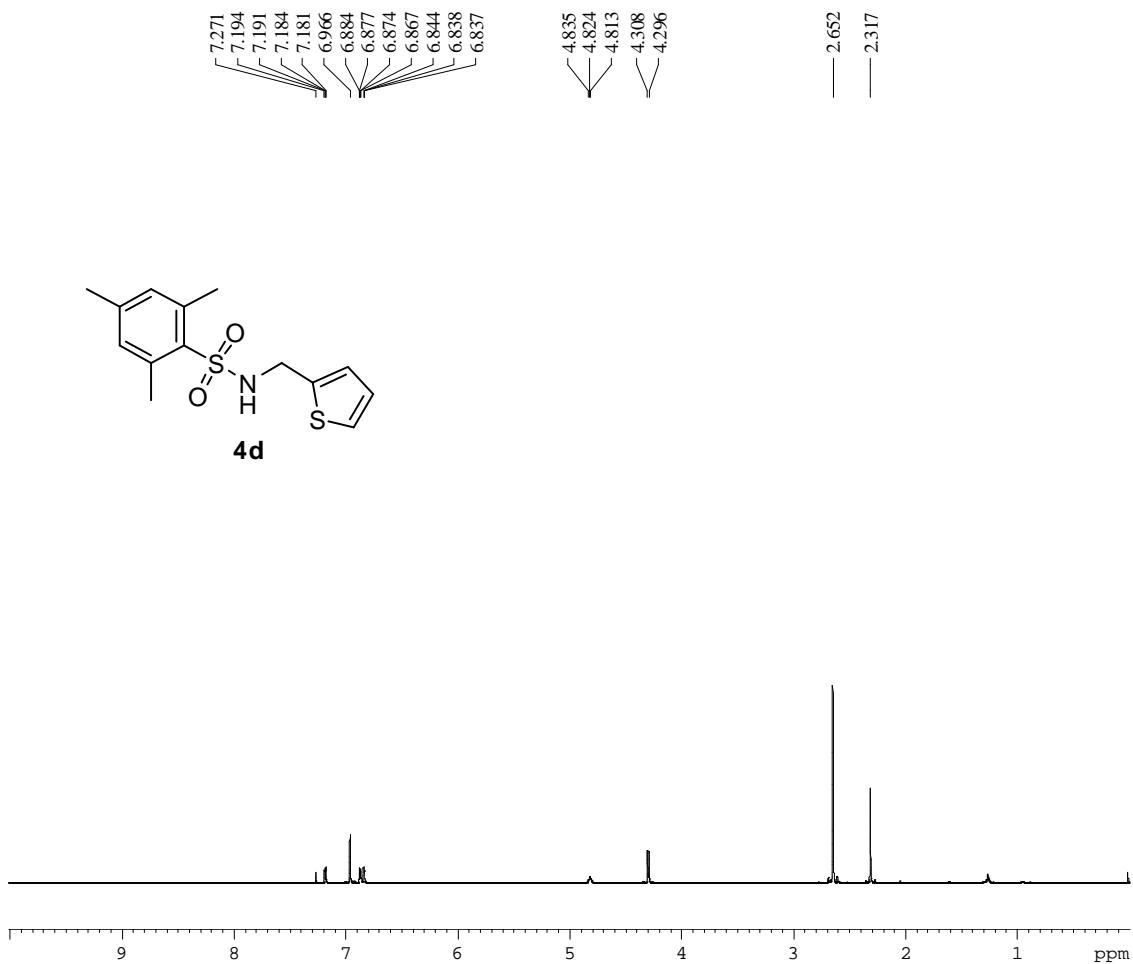
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-((6-Fluoropyridin-3-yl)methyl)-2,4,6-trimethylbenzenesulfonamide **4c**



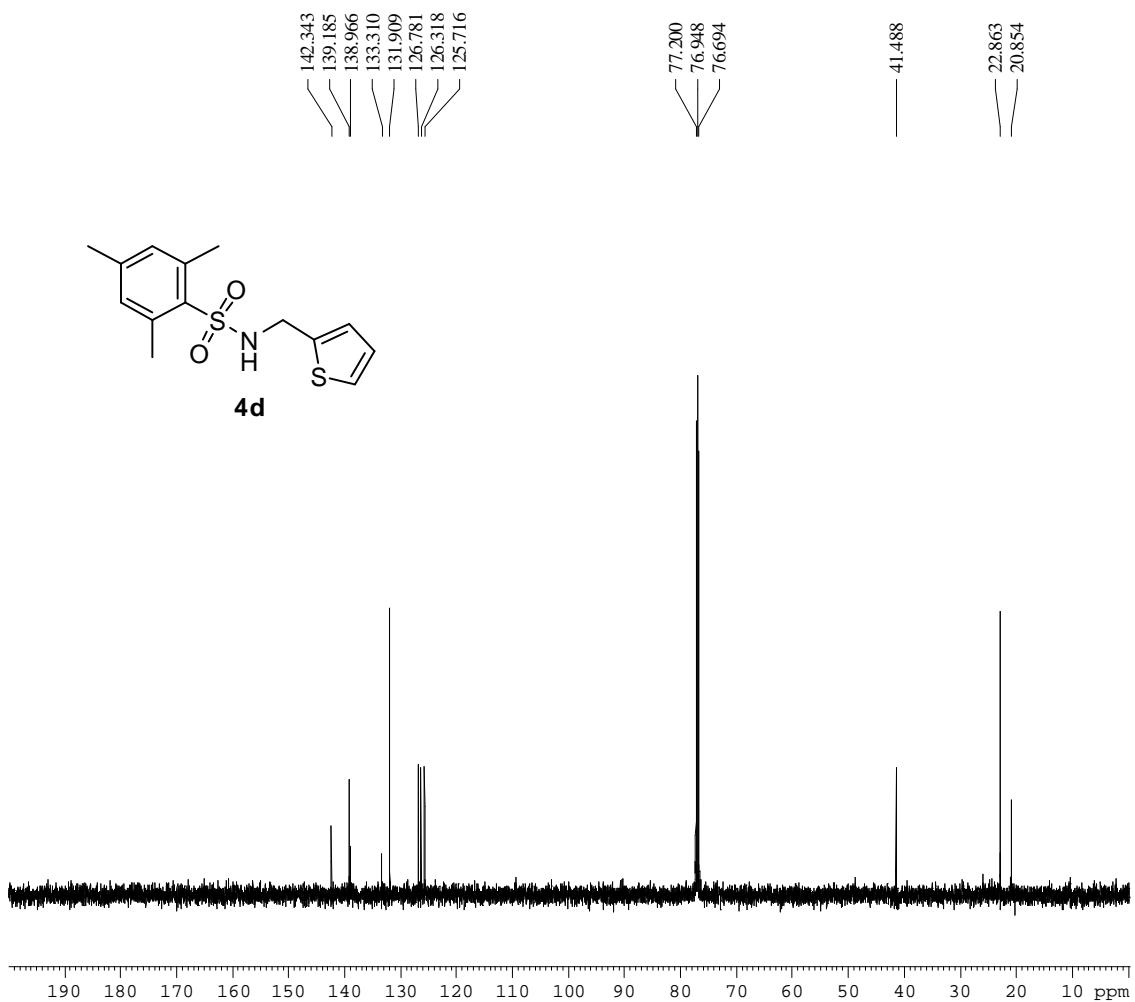
<sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470.8 MHz) spectrum of *N*-((6-Fluoropyridin-3-yl)methyl)-2,4,6-trimethylbenzenesulfonamide **4c**



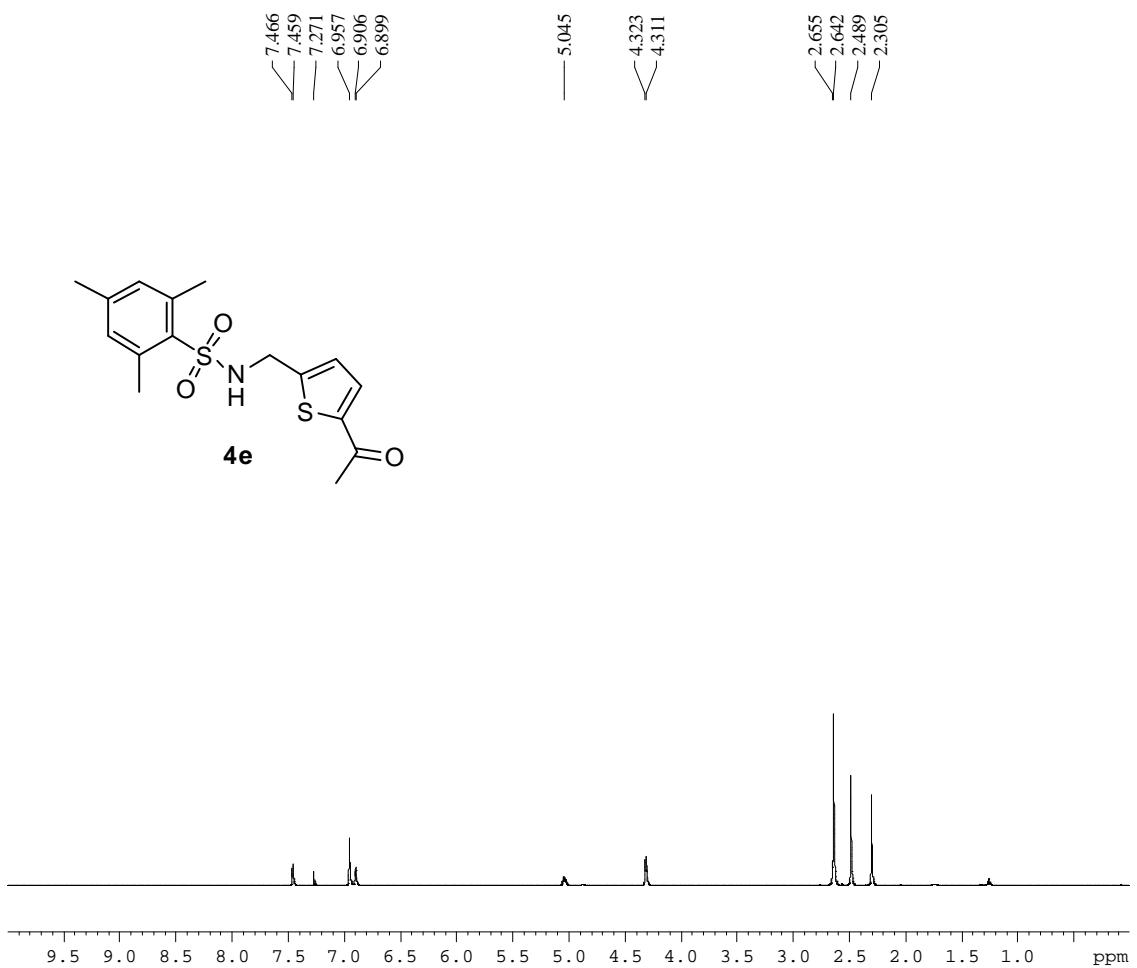
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of 2,4,6-Triethyl-N-(thiophen-2-ylmethyl)benzenesulfonamide **4d**



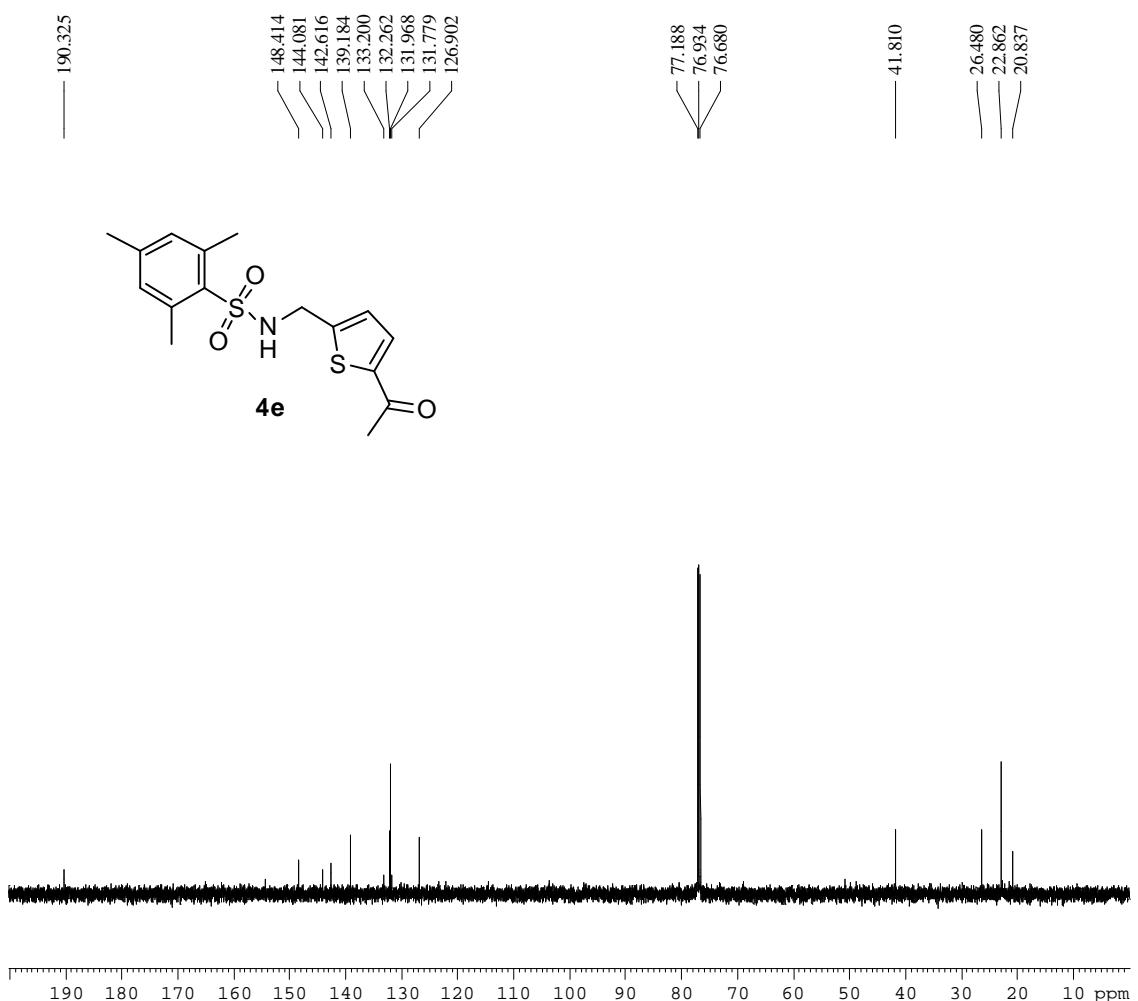
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of 2,4,6-Trimethyl-N-(thiophen-2-ylmethyl)benzenesulfonamide **4d**



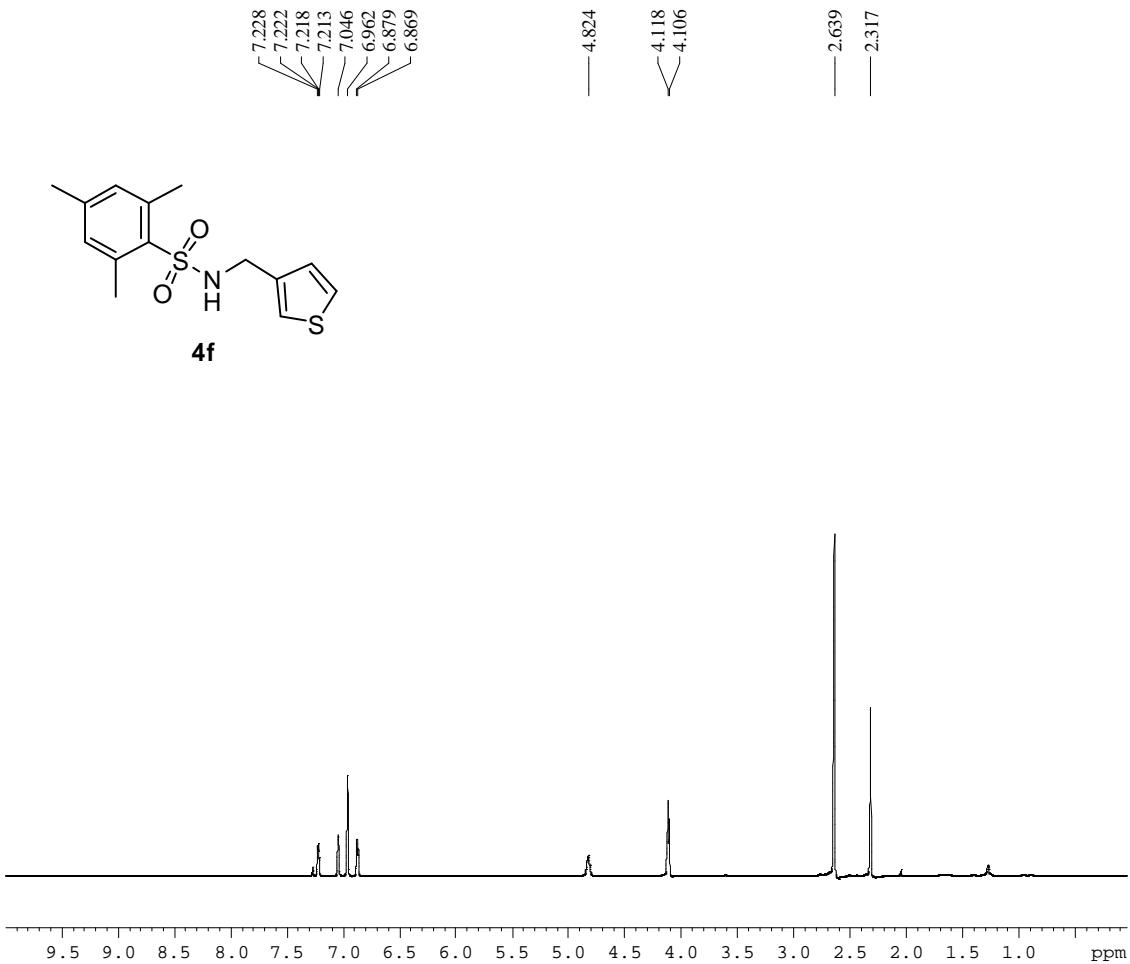
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(*5*-Acetylthiophen-2-yl)methyl)-2,4,6-trimethylbenzenesulfonamide **4e**



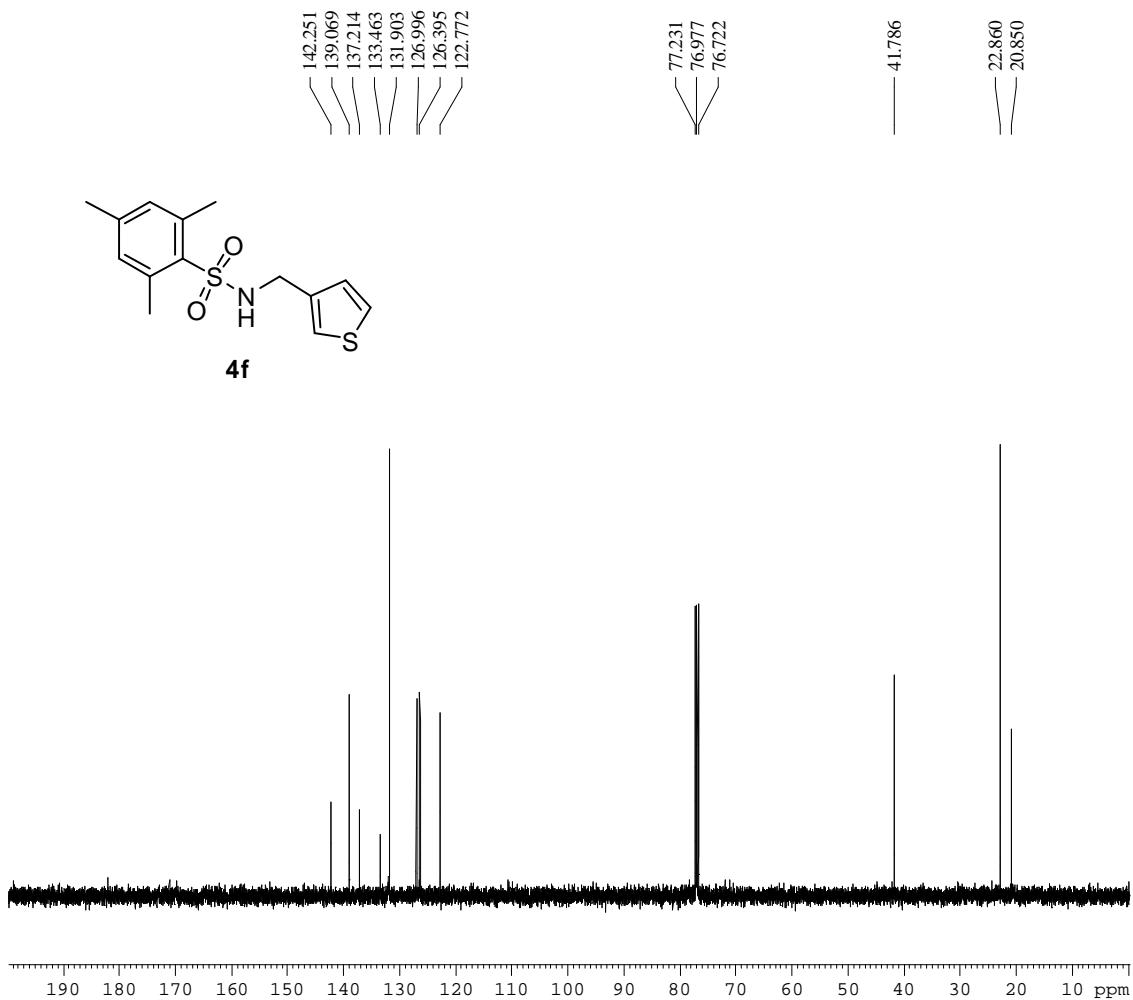
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(5-Acetylthiophen-2-yl)methyl)-2,4,6-trimethylbenzenesulfonamide **4e**



<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of 2,4,6-Trimethyl-N-(thiophen-3-ylmethyl)benzenesulfonamide **4f**



$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of 2,4,6-Trimethyl-*N*-(thiophen-3-ylmethyl)benzenesulfonamide **4f**



<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(5-Formylfuran-2-yl)methyl)-2,4,6-trimethylbenzenesulfonamide **4g**



$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(5-Formylfuran-2-yl)methyl)-2,4,6-trimethylbenzenesulfonamide **4g**

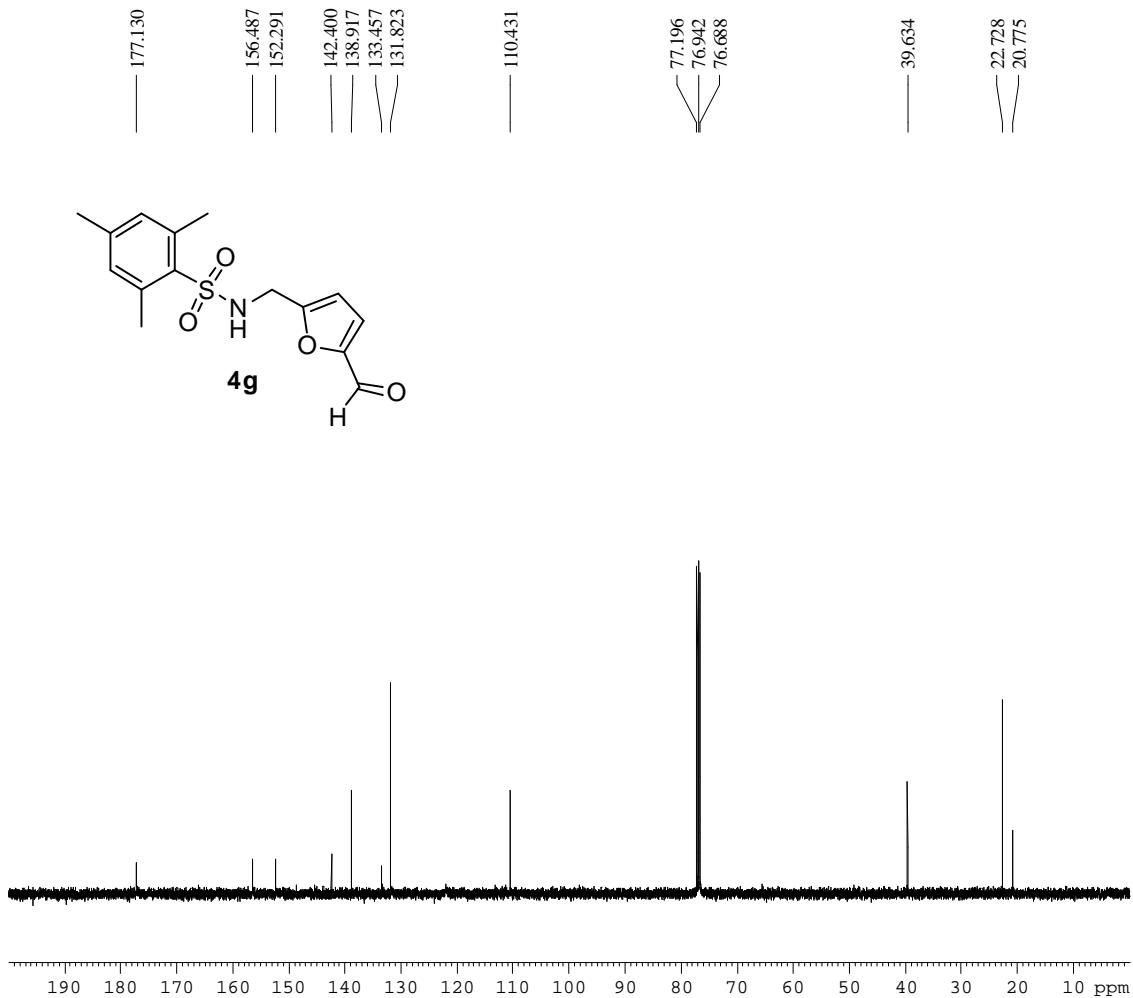
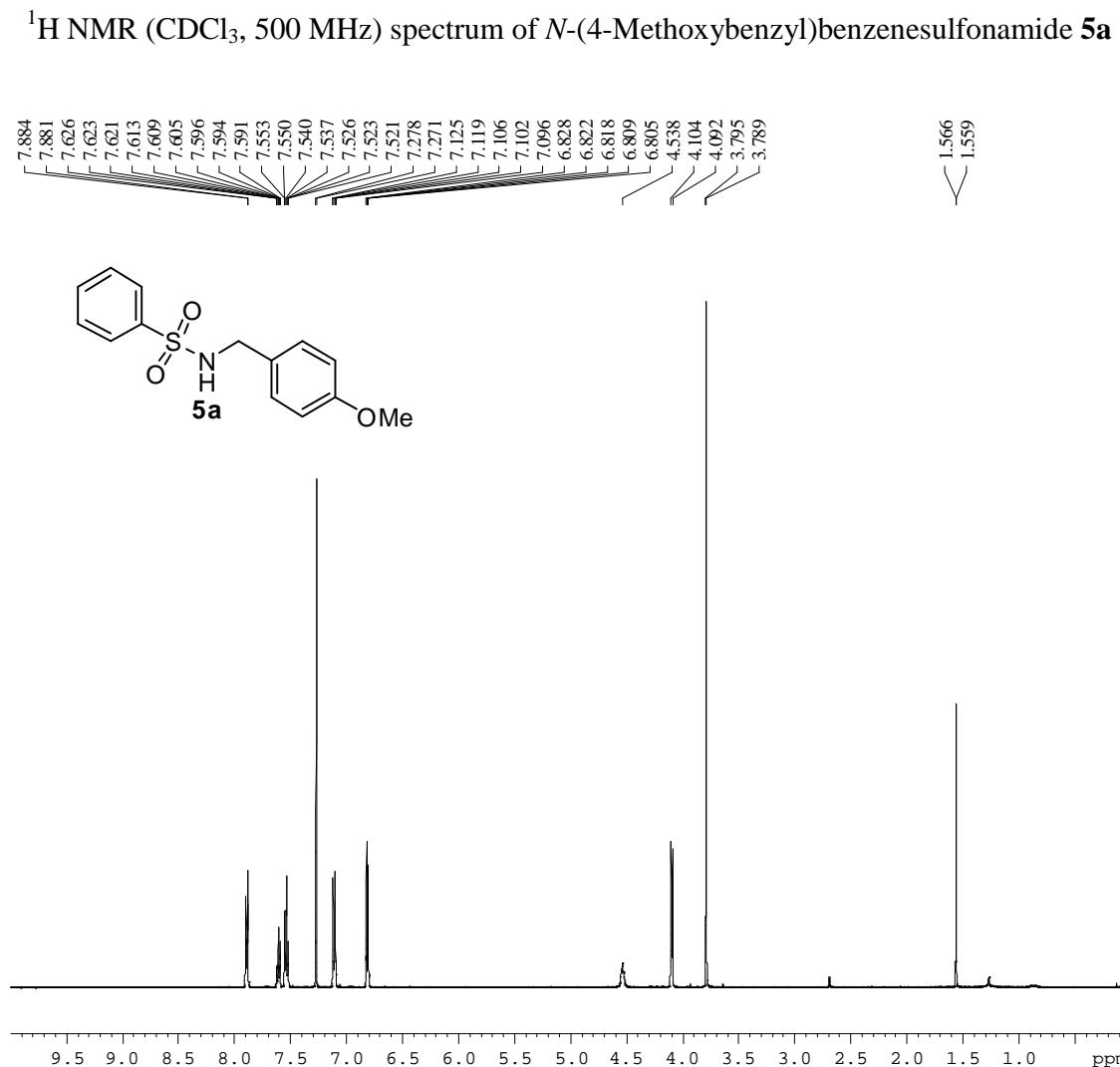
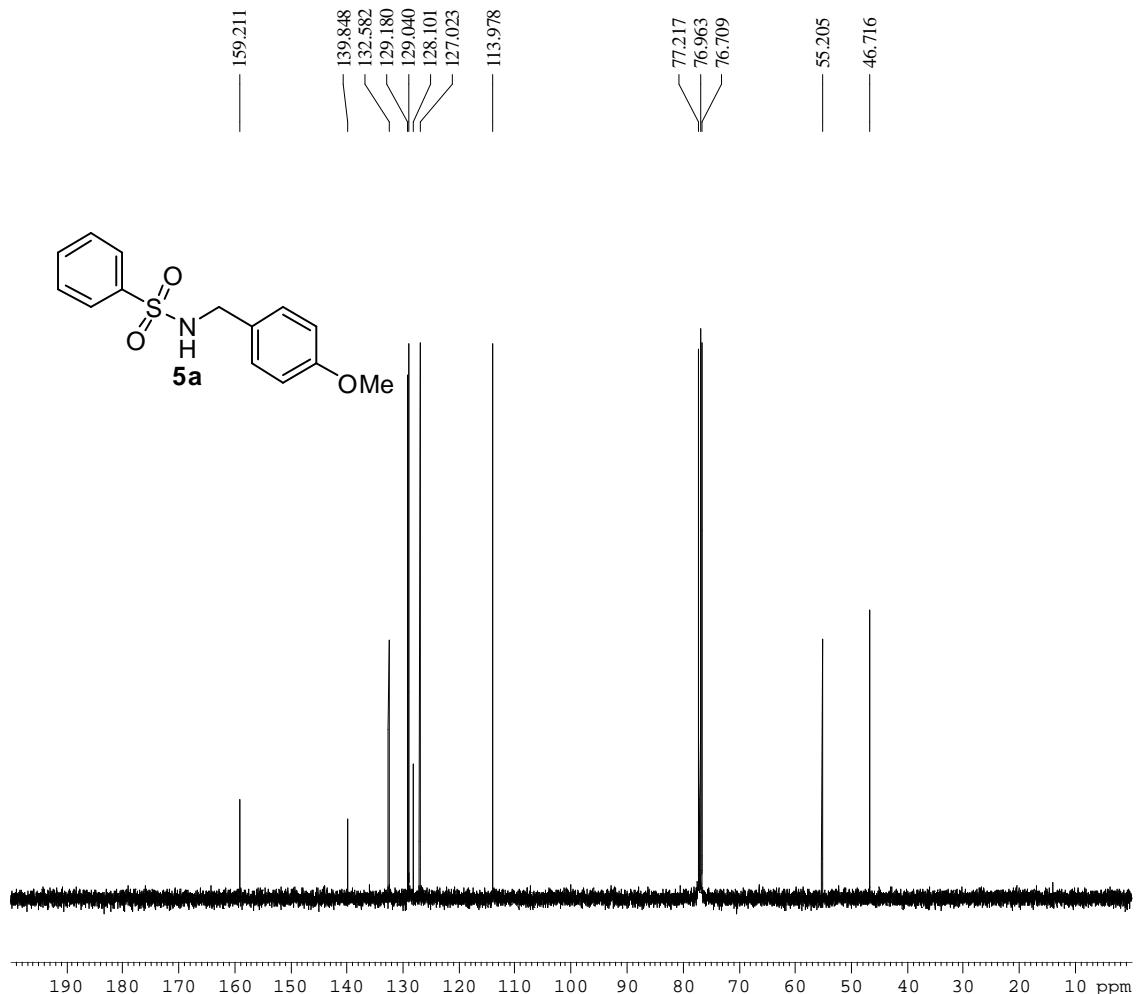


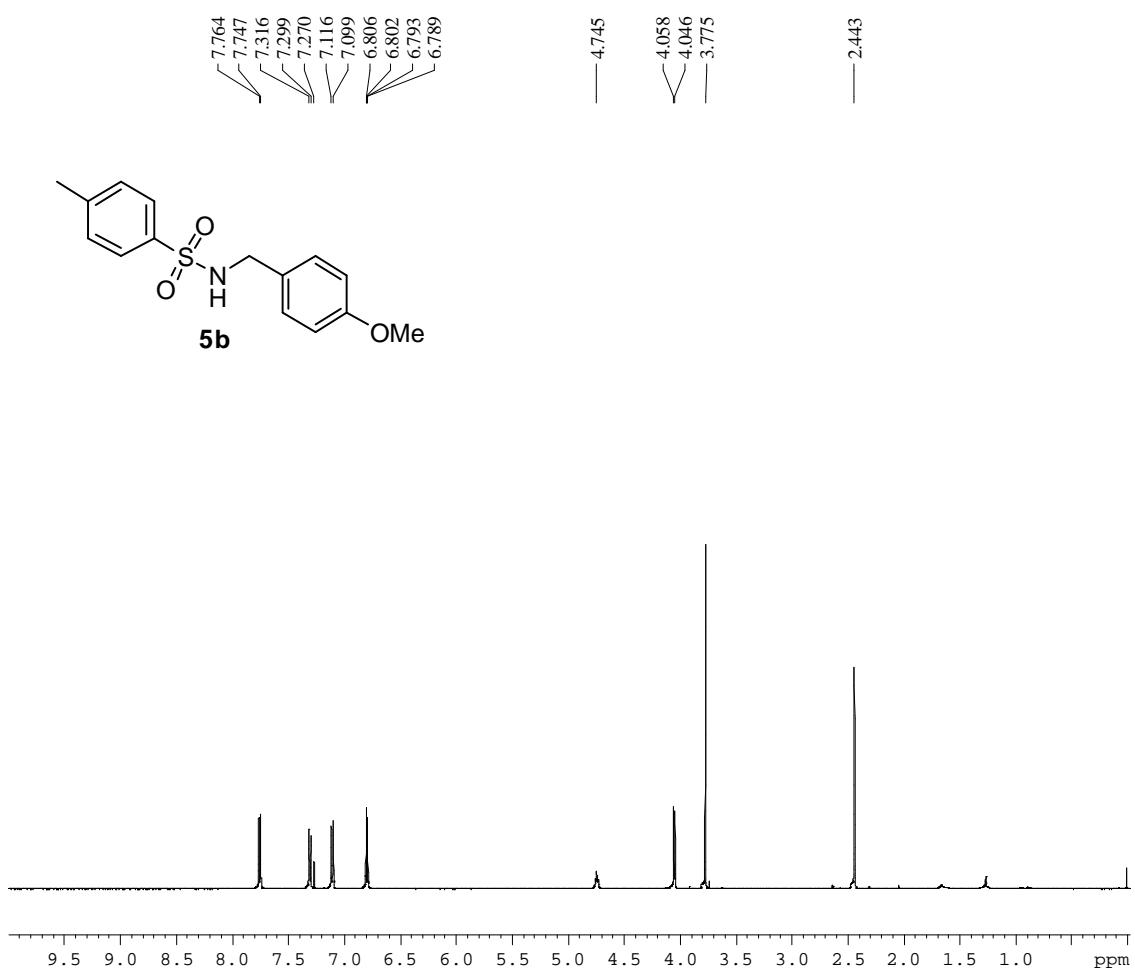
Table 4



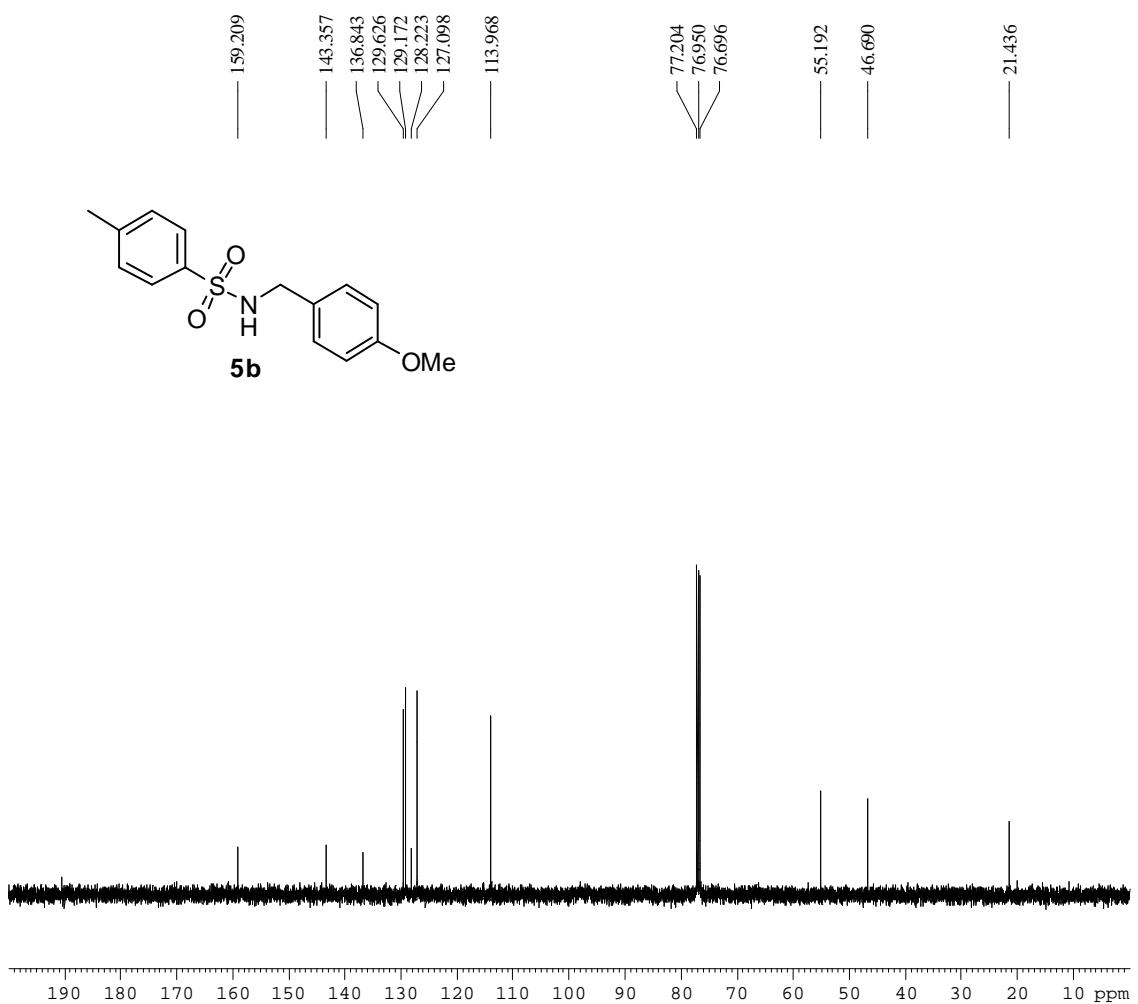
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(4-Methoxybenzyl)benzenesulfonamide **5a**



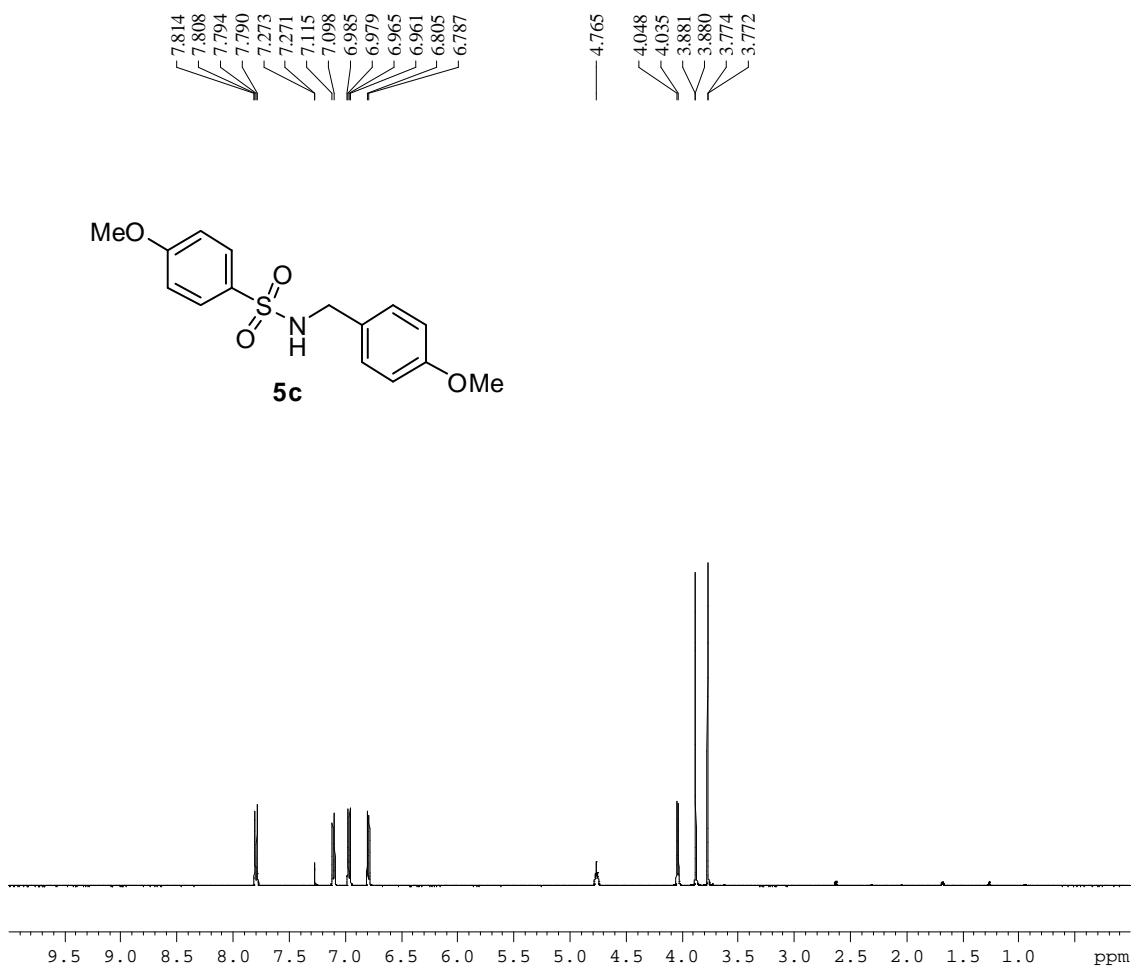
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(4-Methoxybenzyl)-4-methylbenzenesulfonamide **5b**



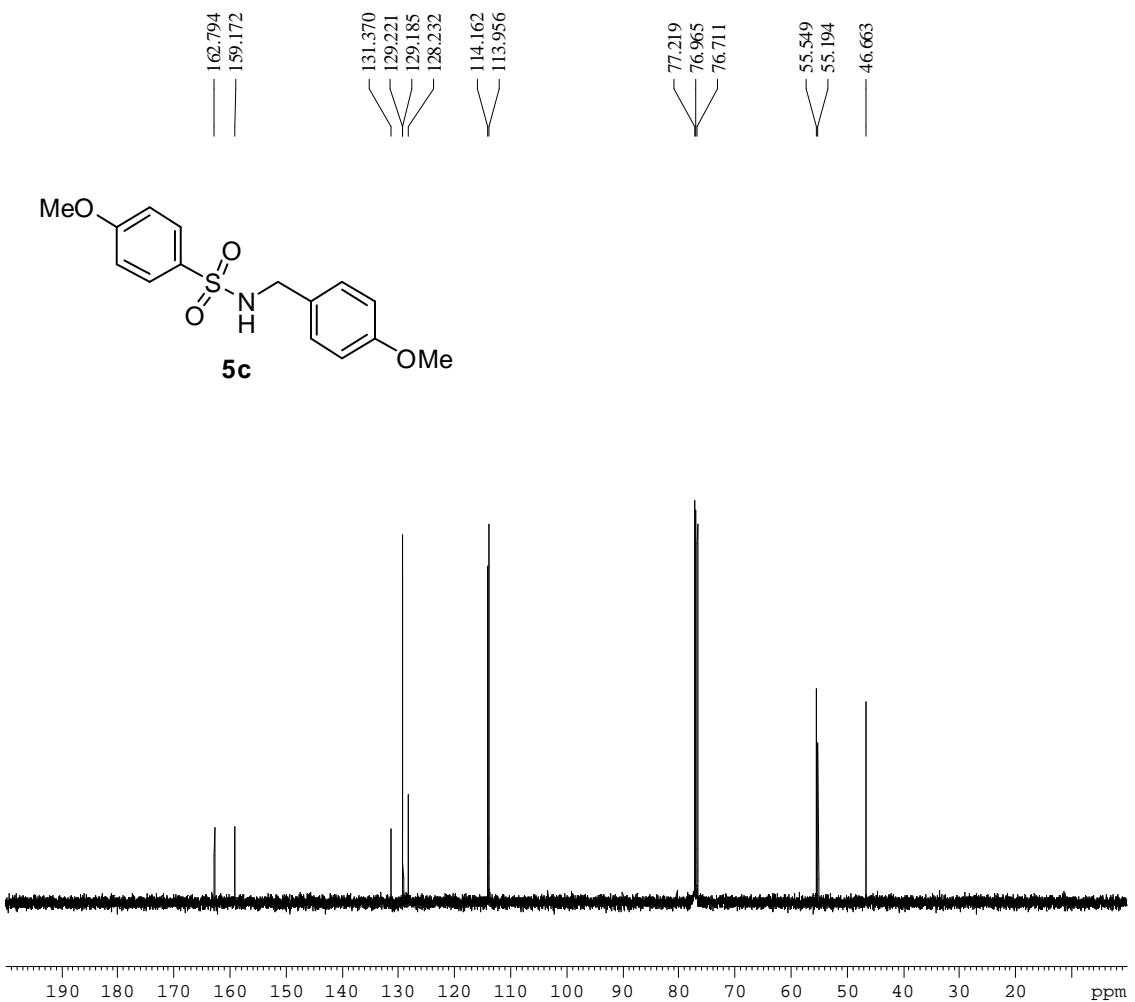
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(4-Methoxybenzyl)-4-methylbenzenesulfonamide **5b**



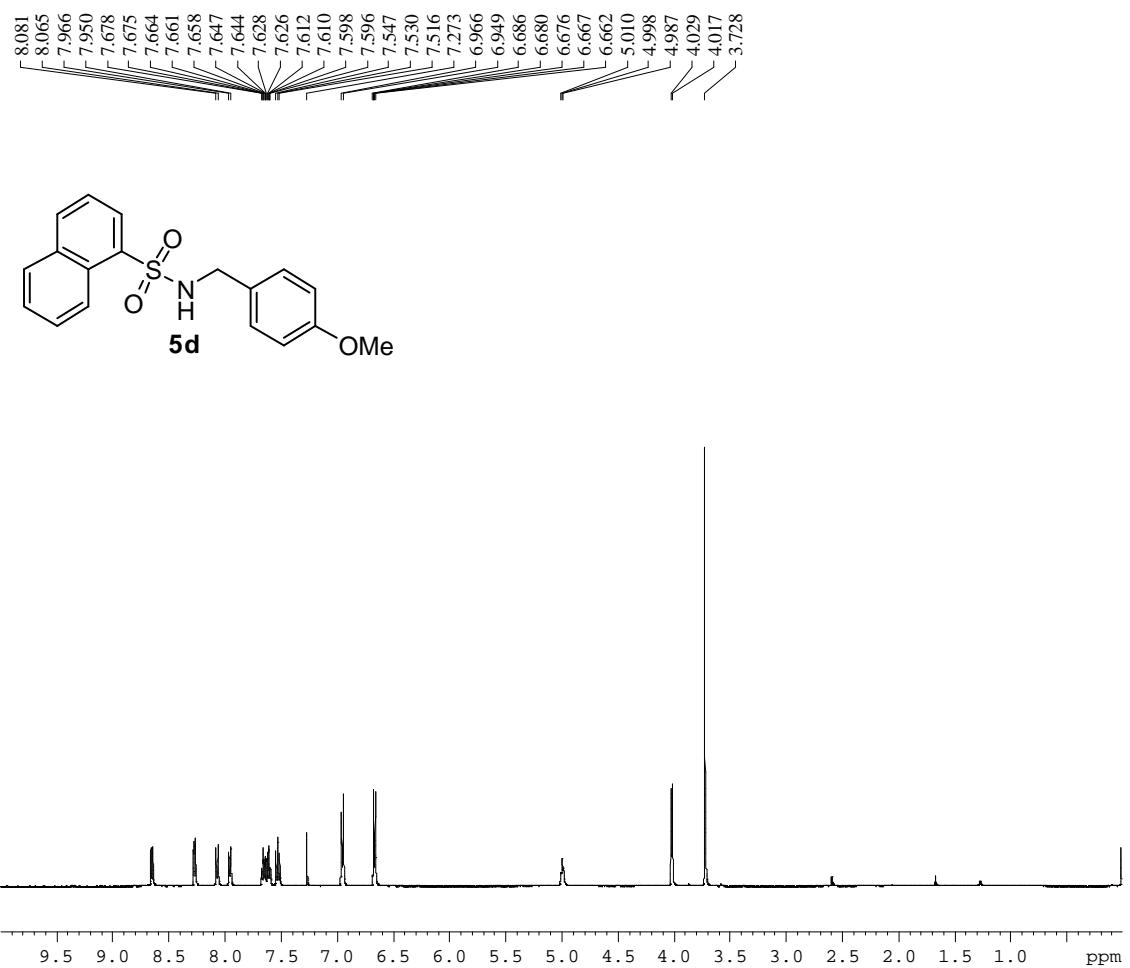
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of 4-Methoxy-*N*-(4-methoxybenzyl)benzenesulfonamide **5c**



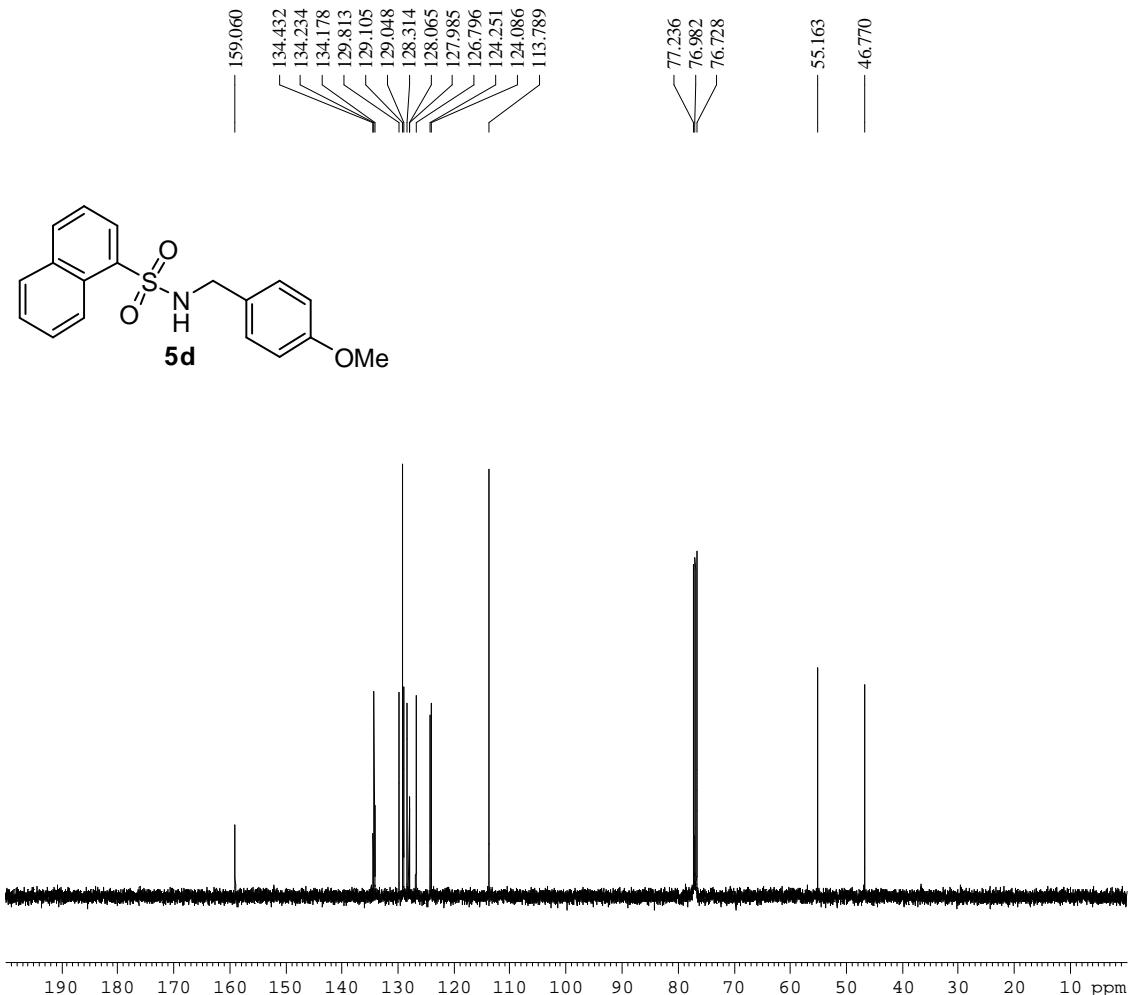
<sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of 4-Methoxy-*N*-(4-methoxybenzyl)benzenesulfonamide **5c**



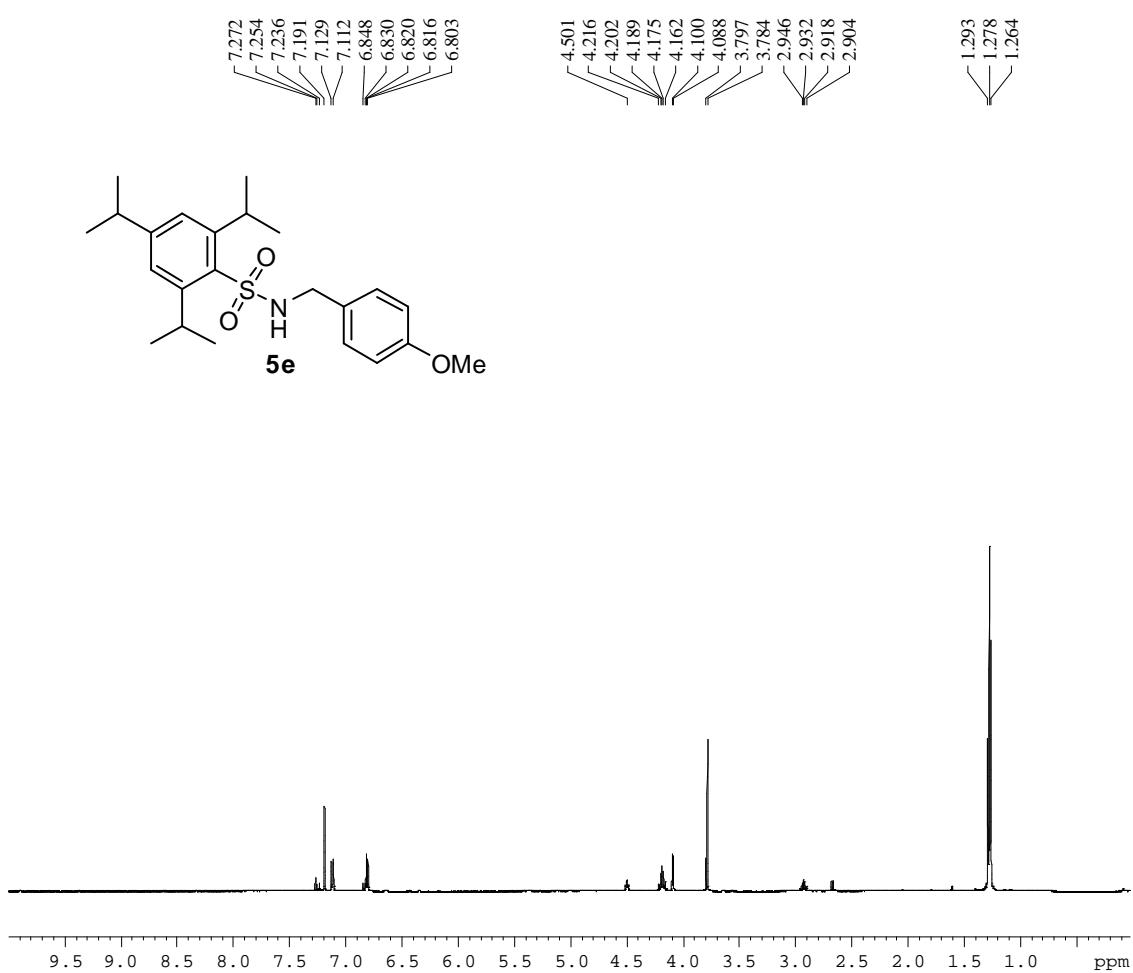
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of *N*-(4-Methoxybenzyl)naphthalene-1-sulfonamide **5d**



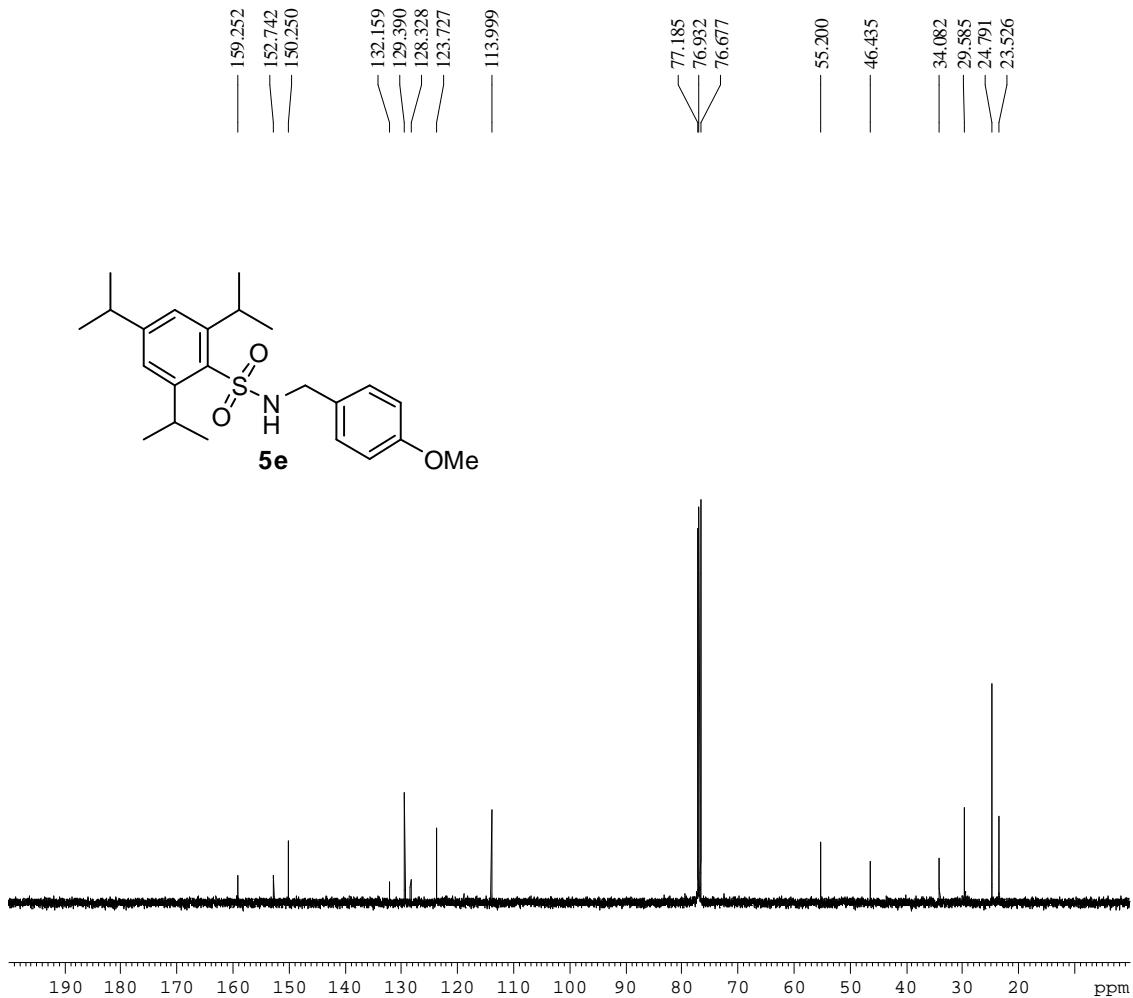
<sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of *N*-(4-Methoxybenzyl)naphthalene-1-sulfonamide **5d**



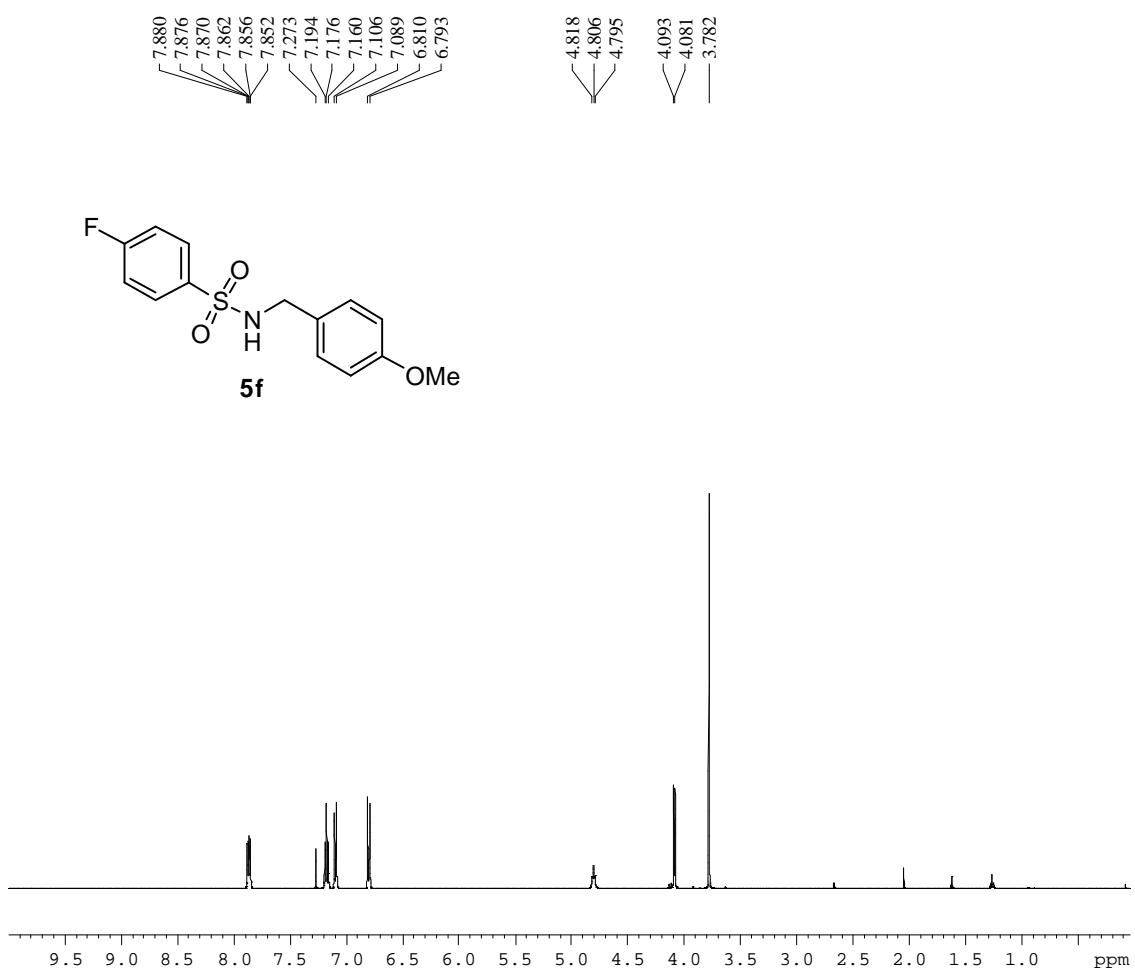
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of 2,4,6-Triisopropyl-*N*-(4-methoxybenzyl)benzenesulfonamide **5e**



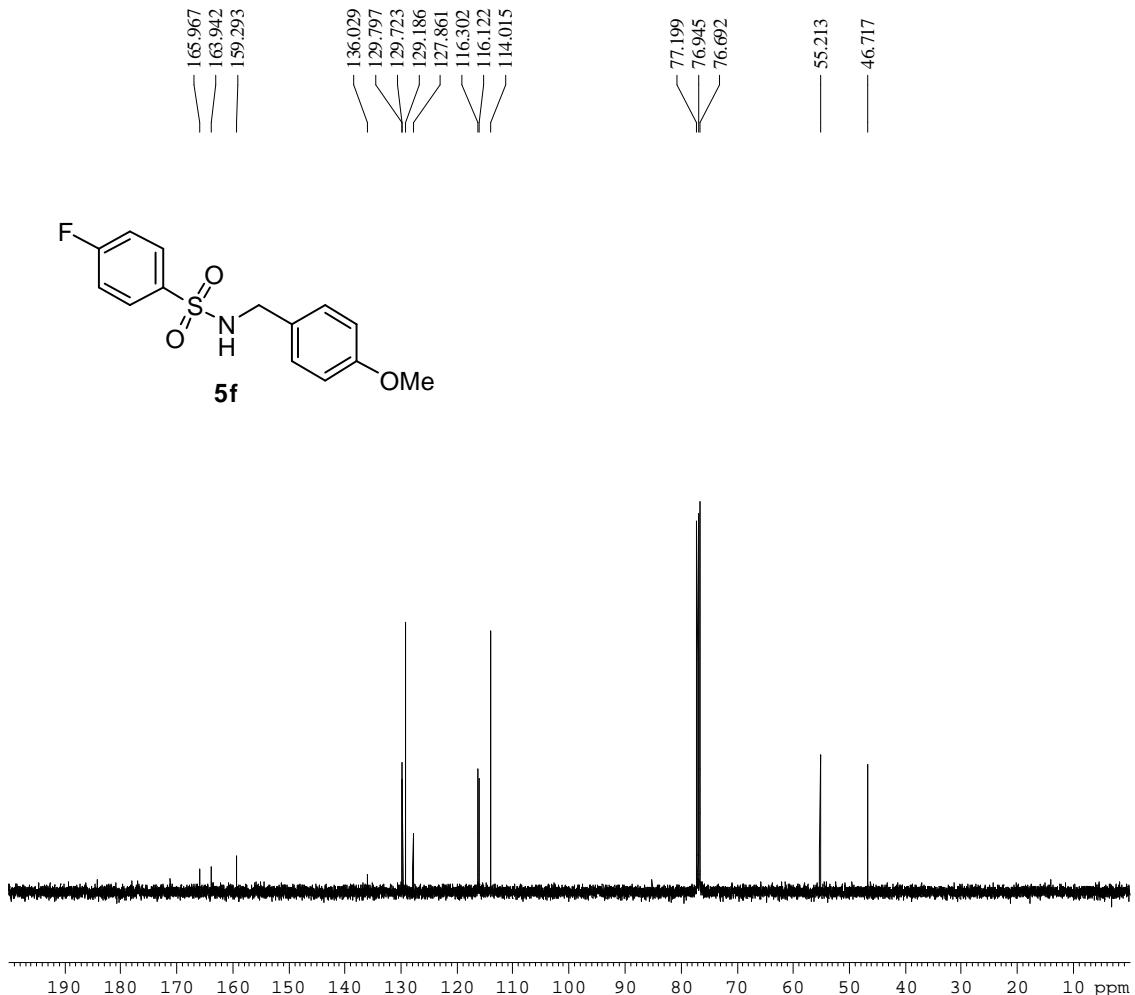
$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of 2,4,6-Triisopropyl-*N*-(4-methoxybenzyl)benzenesulfonamide **5e**



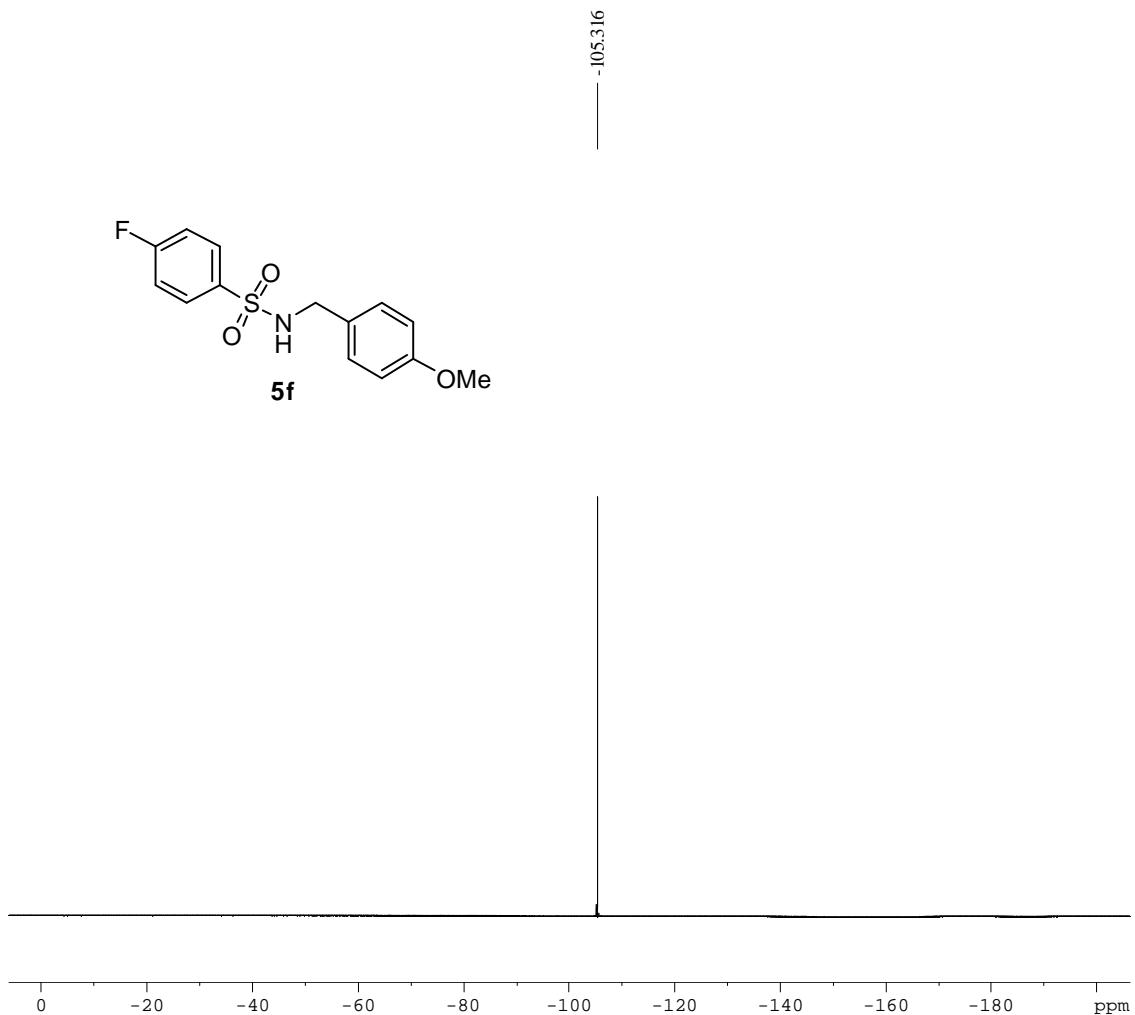
<sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz) spectrum of 4-Fluoro-*N*-(4-methoxybenzyl)benzenesulfonamide **5f**



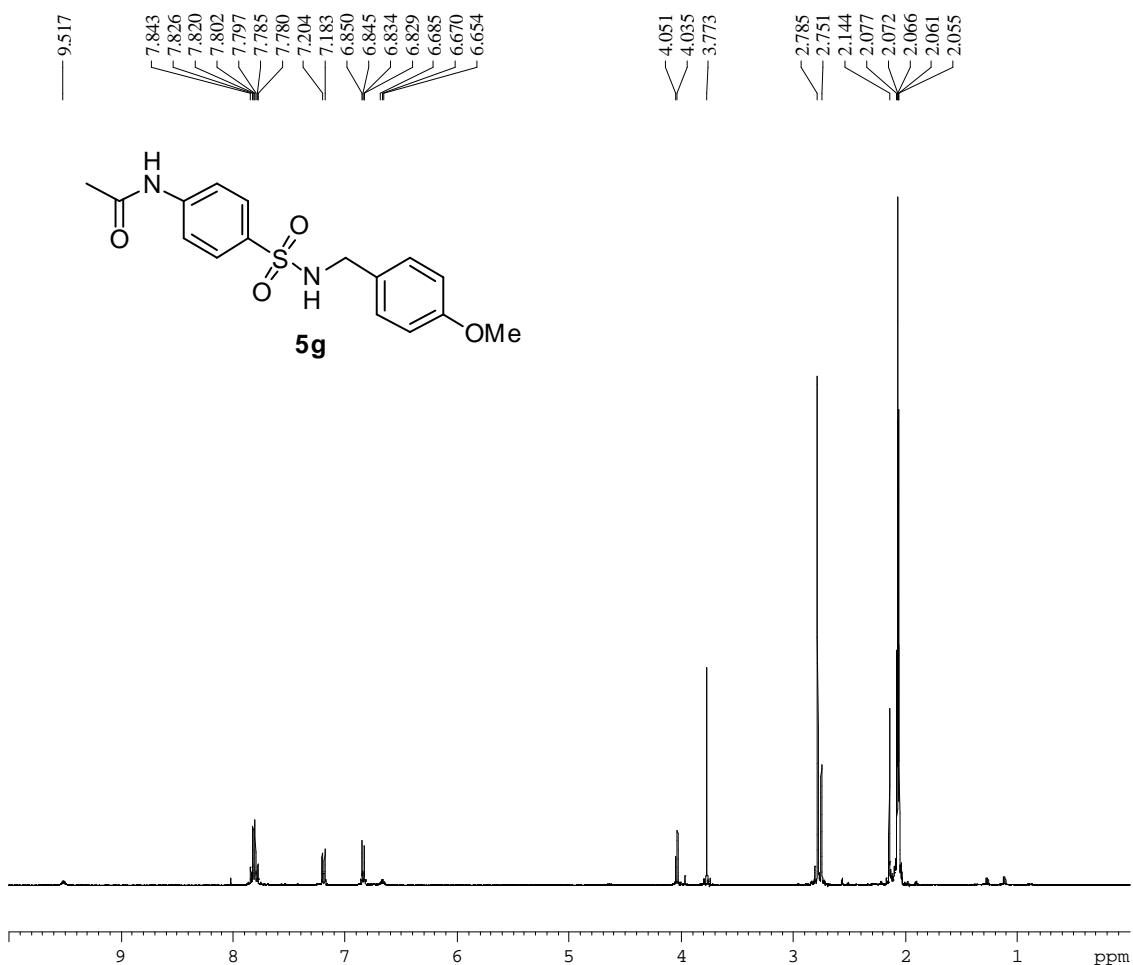
<sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125.8 MHz) spectrum of 4-Fluoro-*N*-(4-methoxybenzyl)benzenesulfonamide **5f**



<sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470.8 MHz) spectrum of 4-Fluoro-*N*-(4-methoxybenzyl)benzenesulfonamide **5f**



<sup>1</sup>H NMR (acetone-d<sub>6</sub>, 500 MHz) spectrum of *N*-(4-(4-Methoxybenzyl)sulfamoyl)phenyl)acetamide **5g**



$^{13}\text{C}$  NMR (acetone-d<sub>6</sub>, 125.8 MHz) spectrum of *N*-(4-(*N*-(4-Methoxybenzyl)sulfamoyl)phenyl)acetamide **5g**

