

Supplementary Information

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## New strategies for the synthesis of lactones using peroxymonosulphate salts, ionic liquids and microwave or ultrasound irradiation

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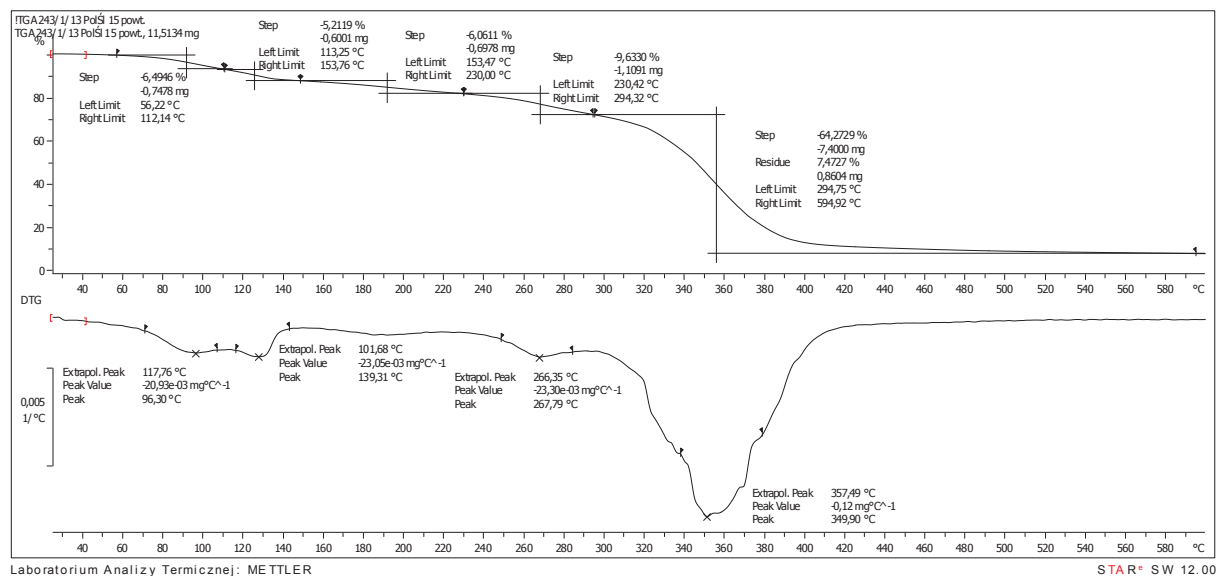
**Materials and apparatus:** 1-butyl-3-methylimidazolium chloride was supplied by Merck KGaA. Cyclohexanol, cyclobutanol, 1-phenyl-2-propanol, 3-methylcyclohexanol, 4-tert-butylcyclohexanol, TEMPO, TMP\*HBr and Oxone<sup>®</sup> were supplied by Acros Organics. [bmim]HSO<sub>4</sub>, [bpyr]Br and [empy]Br were obtained from Sigma-Aldrich. [N<sub>4444</sub>][HSO<sub>5</sub>] was synthesised according to the literature procedure.<sup>1</sup>

The structure and purity of all synthesised substances were confirmed by NMR analysis. <sup>1</sup>H NMR spectra were recorded at 300 MHz and <sup>13</sup>C NMR at 75 MHz in CDCl<sub>3</sub> (Varian Unity Inova plus, internal TMS). GC analyses were performed using a Perkin Elmer Clarus 500 chromatograph equipped with a SUPELCOWAX<sup>TM</sup> 10 column (30 m×0.2 mm×0.2 μm) and n-decane as an internal standard. Electrospray ionisation mass spectroscopy (ESI-MS) experiments were performed using a Waters Xevo G2 QTOF with an injection system (cone voltage 50 V; source 120 °C). Thermogravimetric analysis was performed with a Mettler Toledo TGA/SDTA 851<sup>e</sup>/1100 analyzer (temp. program 25–600 °C, 20 °C/min, N<sub>2</sub> 60 ml/min).

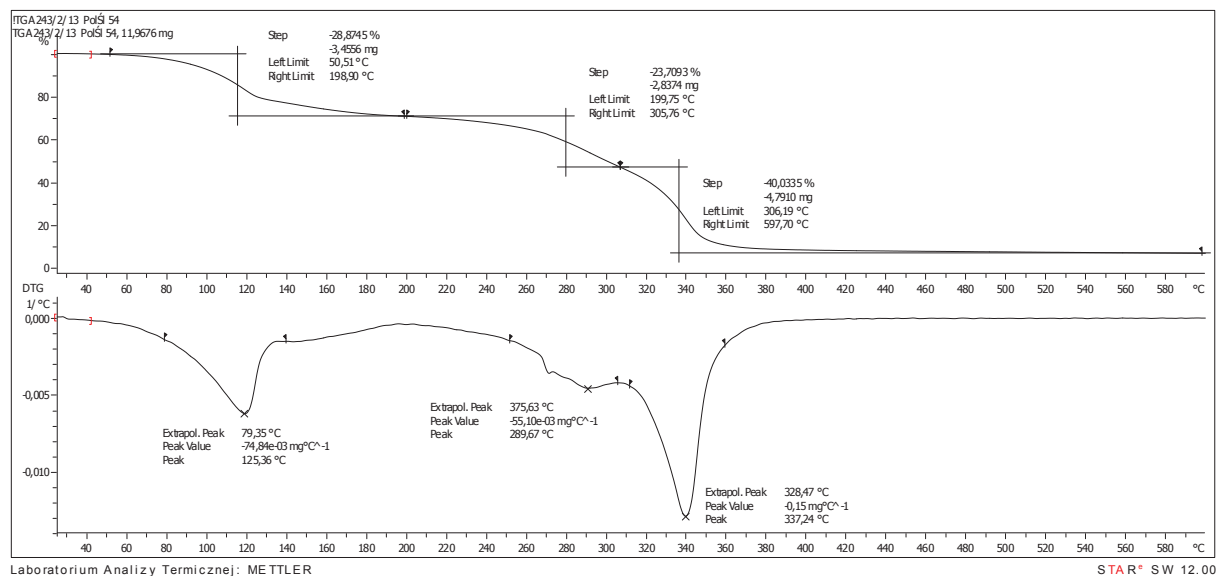
## Characterization of new compounds:

### TGA/DTG:

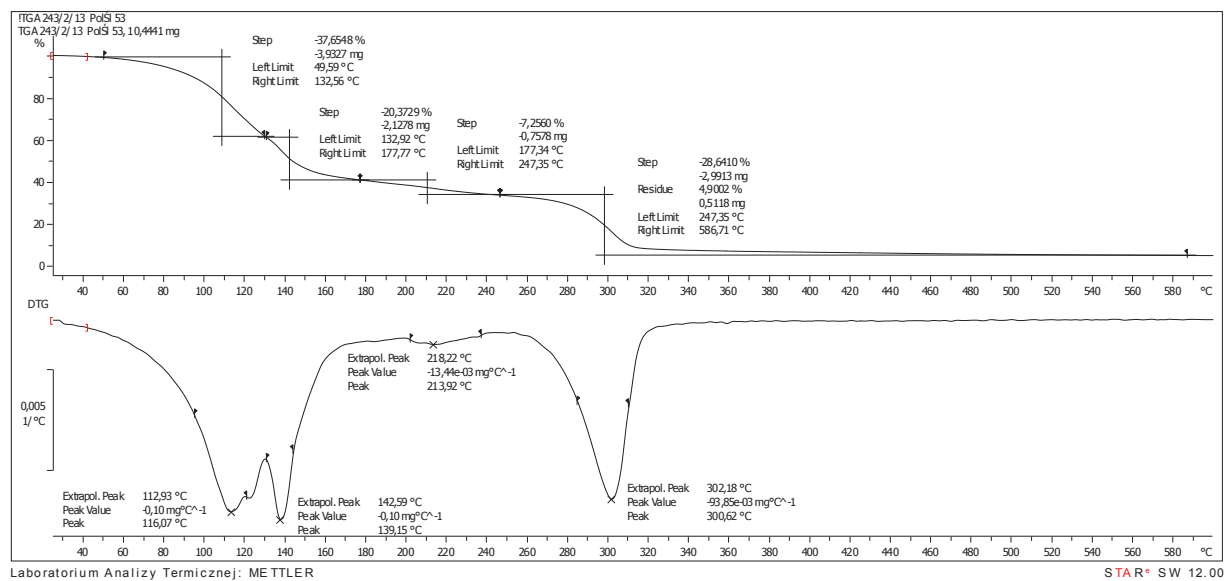
#### 1- butyl-3-methylimidazolium peroxysulphate [bmim][HSO<sub>5</sub>]



#### 1-butylpyridinium peroxysulphate [bpyr][HSO<sub>5</sub>]

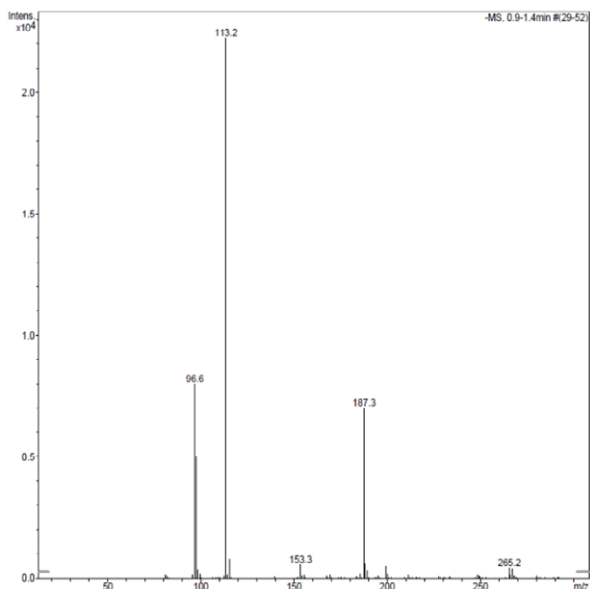
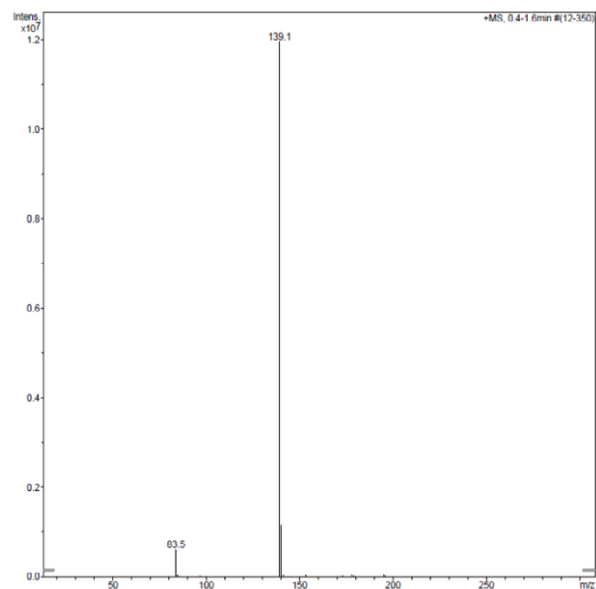


## 1-ethyl-1-methylpyrrolidinium peroxysulphate [empy][HSO<sub>5</sub>]

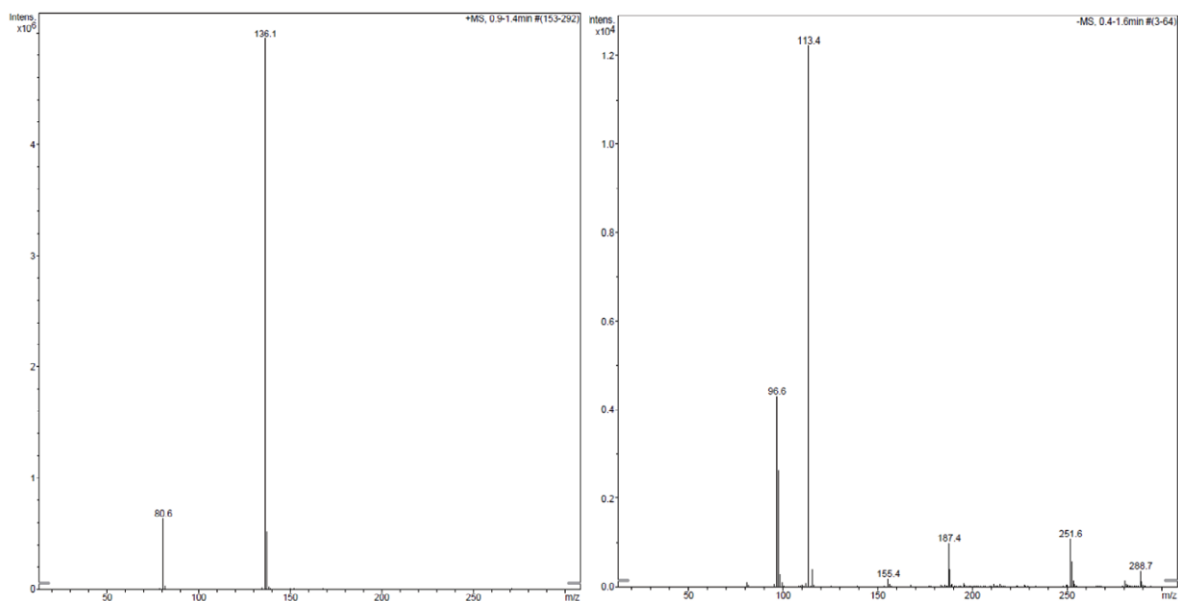


## ESI-MS:

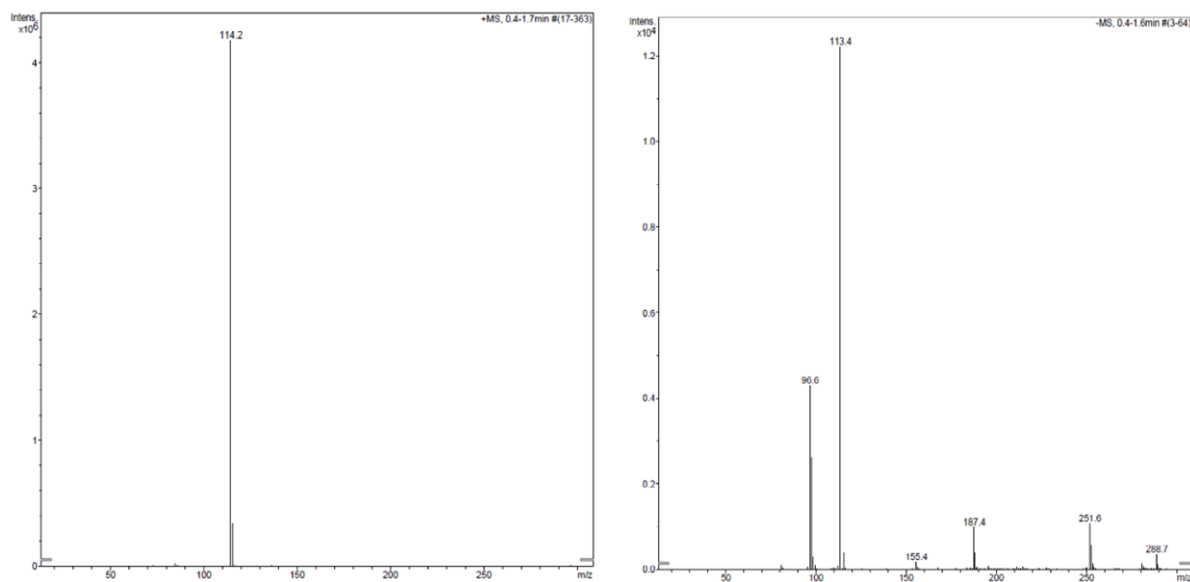
### 1-butyl-3-methylimidazolium peroxysulphate



### 1-butylpyridinium peroxysulphate

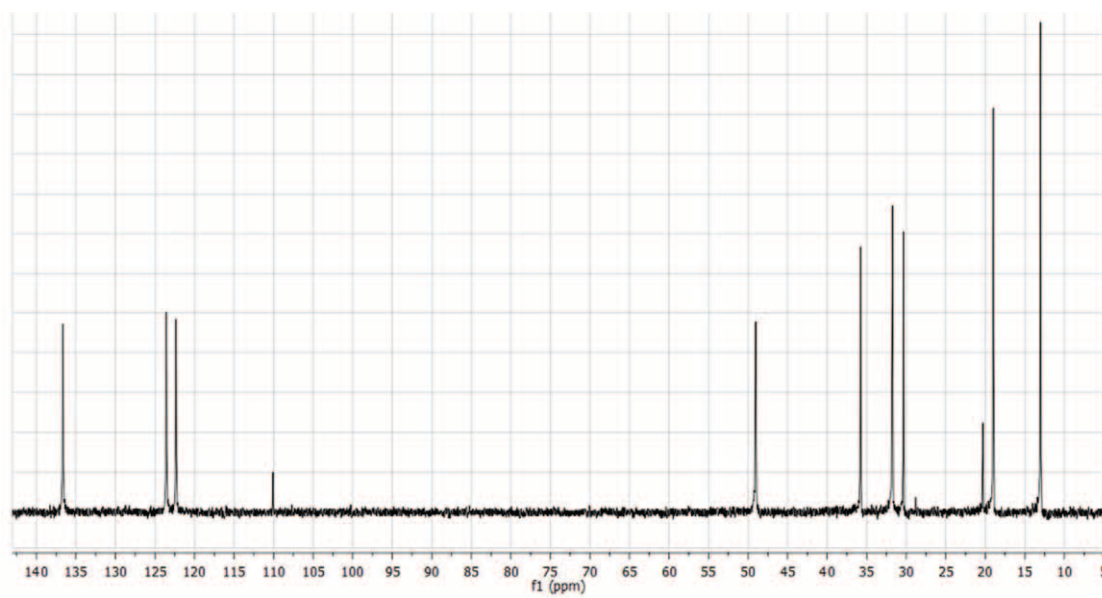
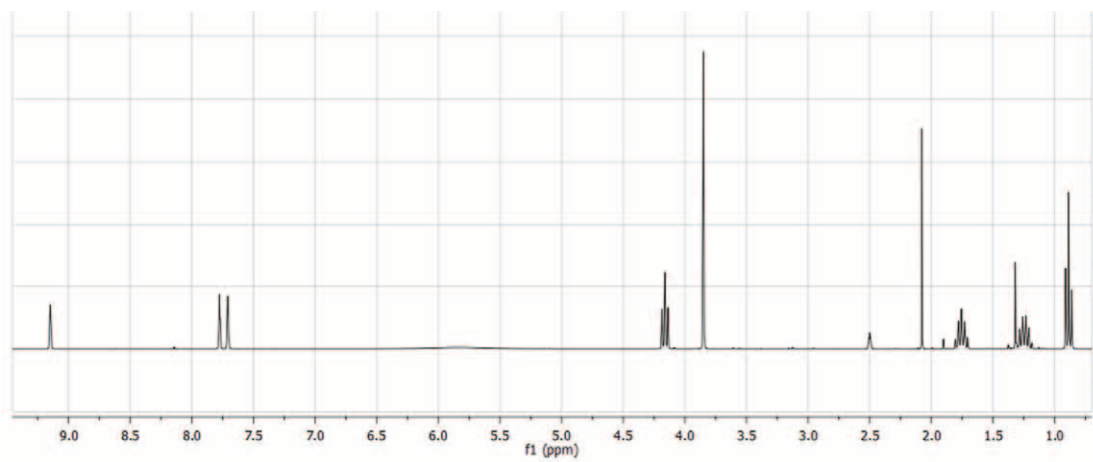


### 1-ethyl-1-methylpyrrolidinium peroxysulphate

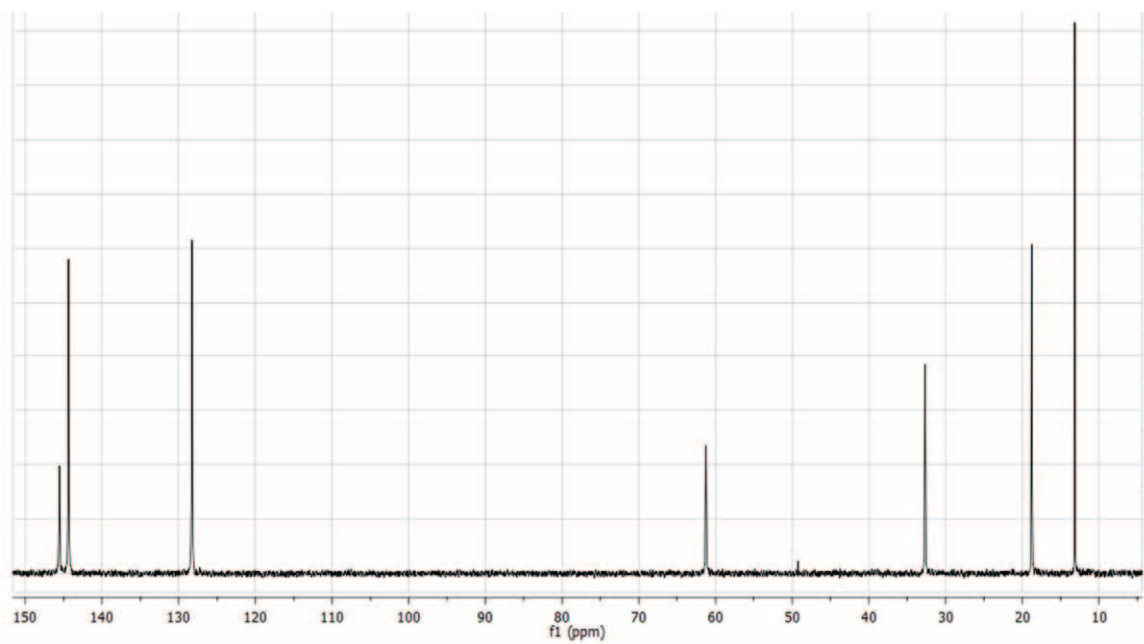
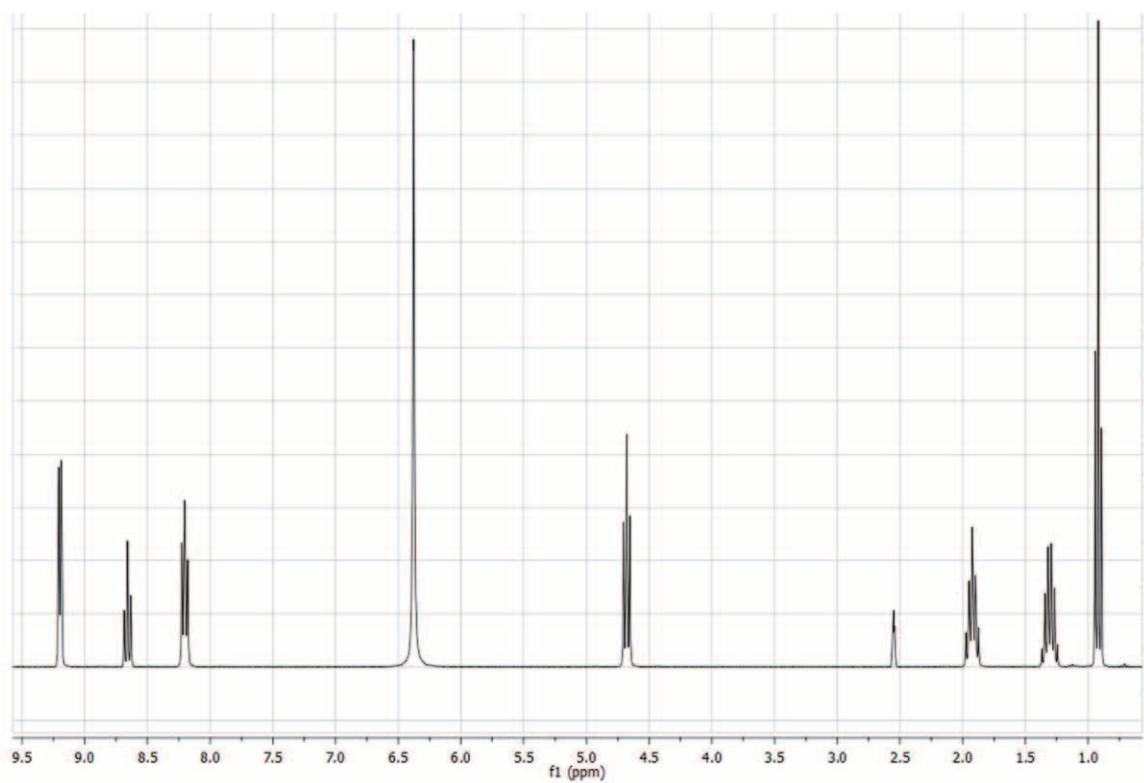


# $^1\text{H}$ $^{13}\text{C}$ NMR:

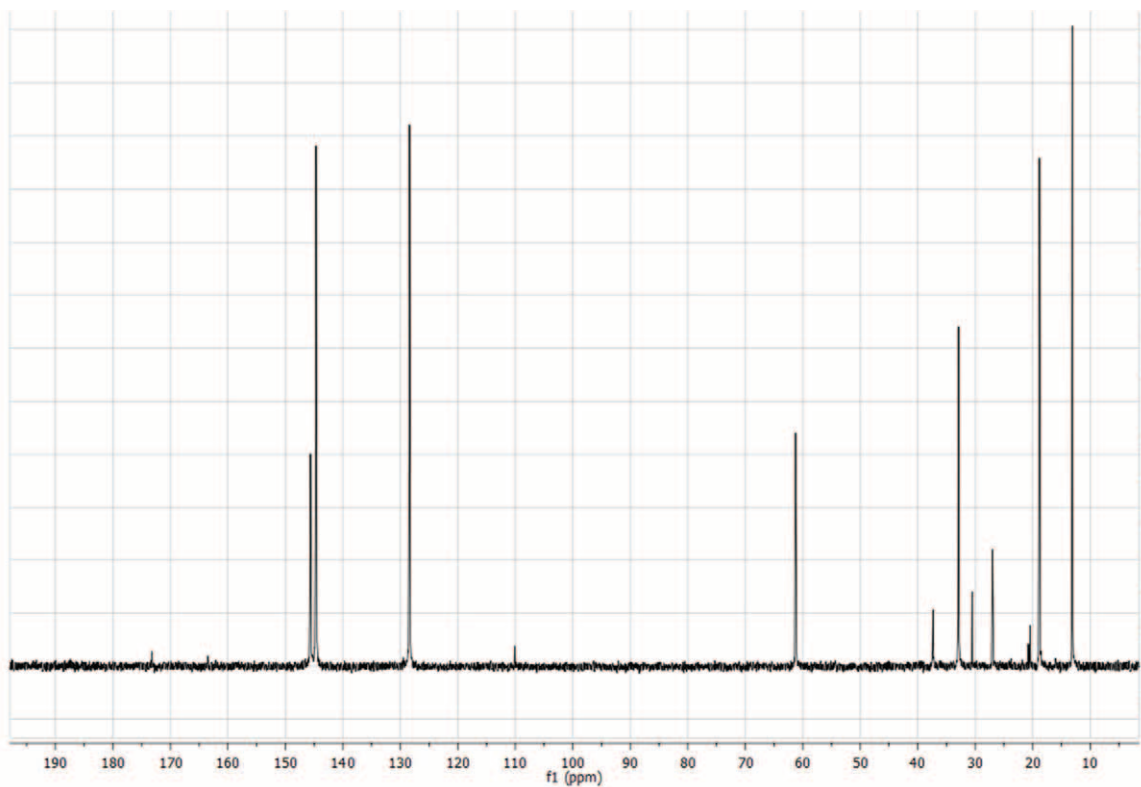
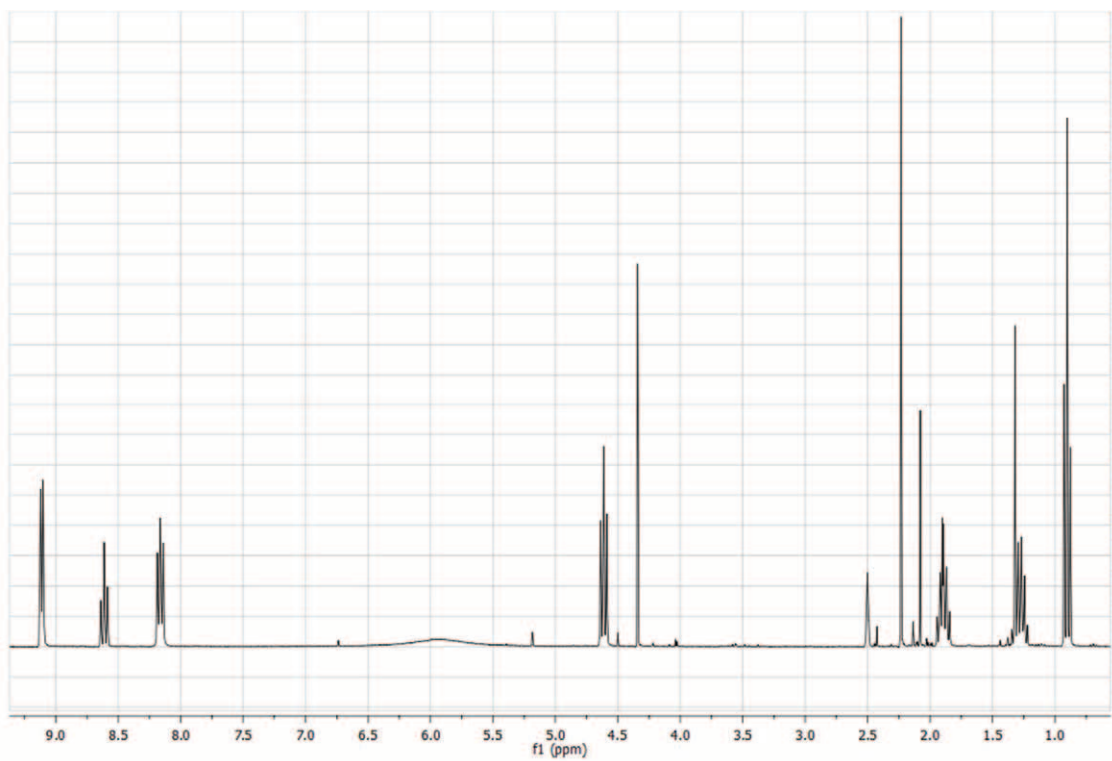
[bmim][HSO<sub>5</sub>]



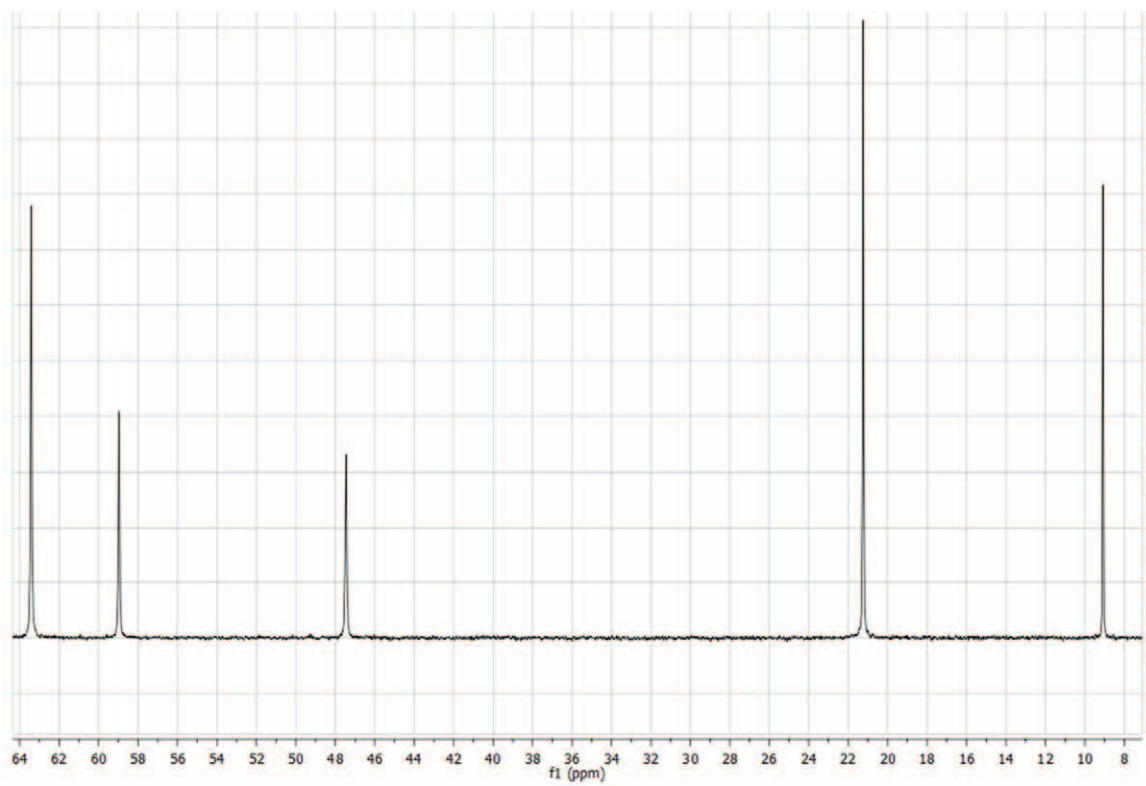
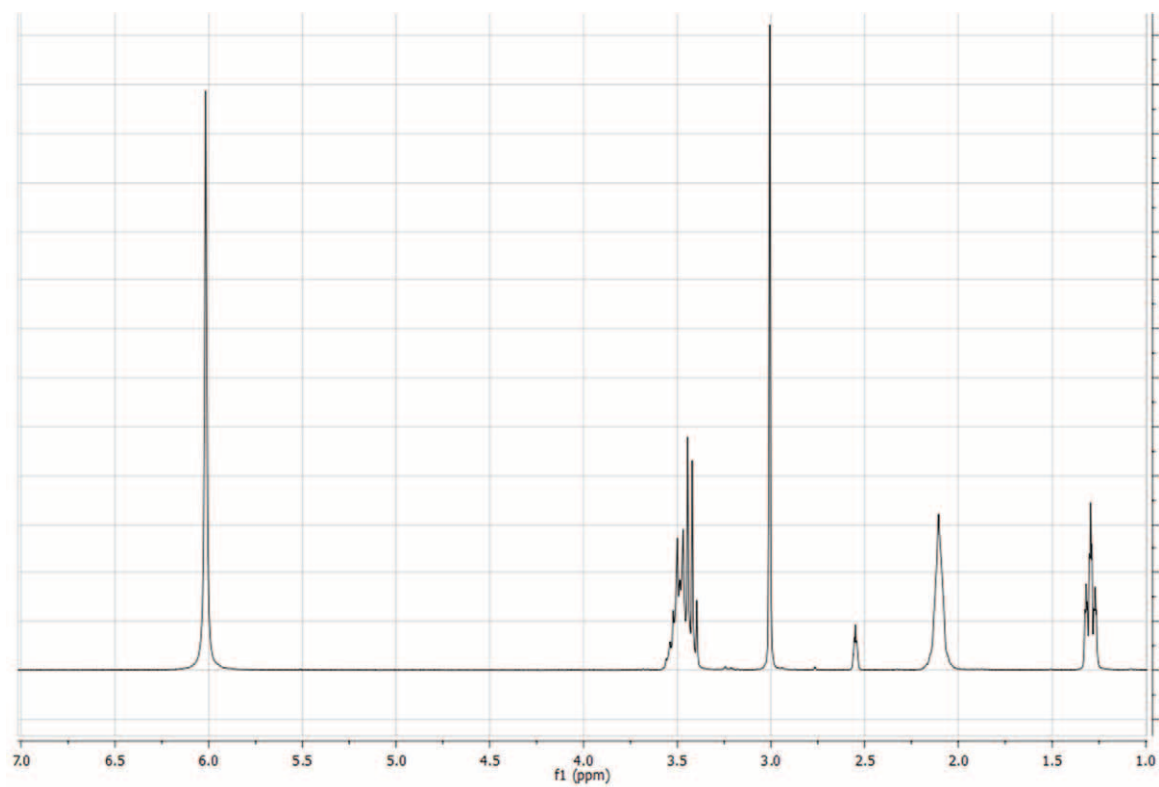
**[bpyr][HSO<sub>4</sub>]**



**[bpyr][HSO<sub>5</sub>]**

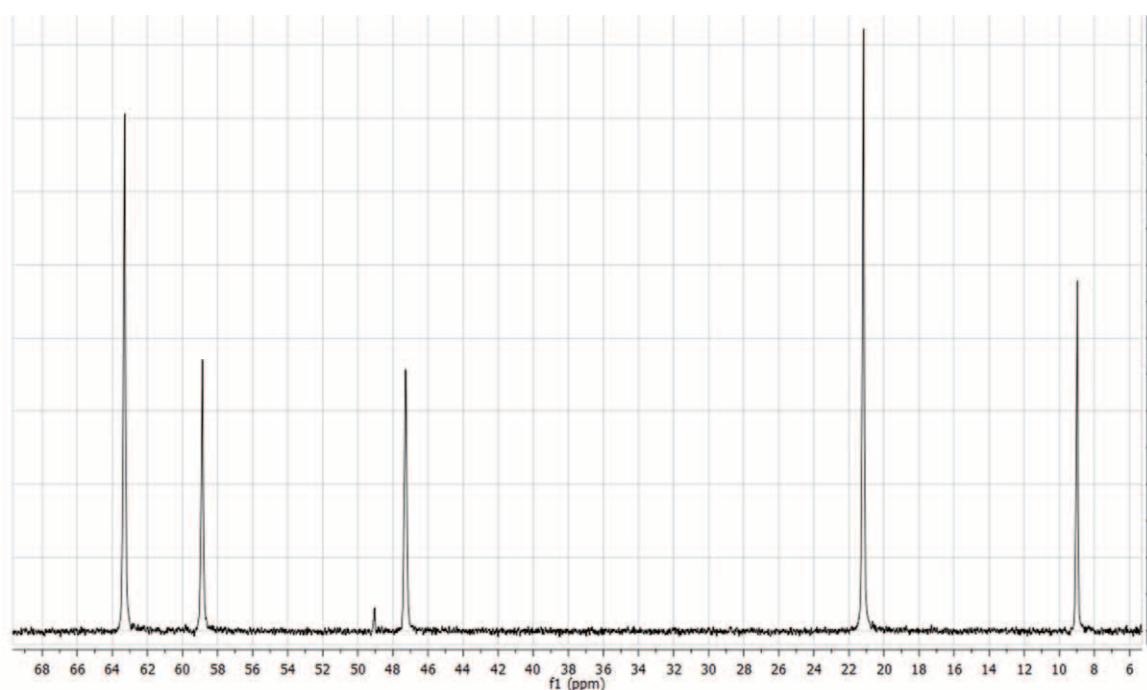
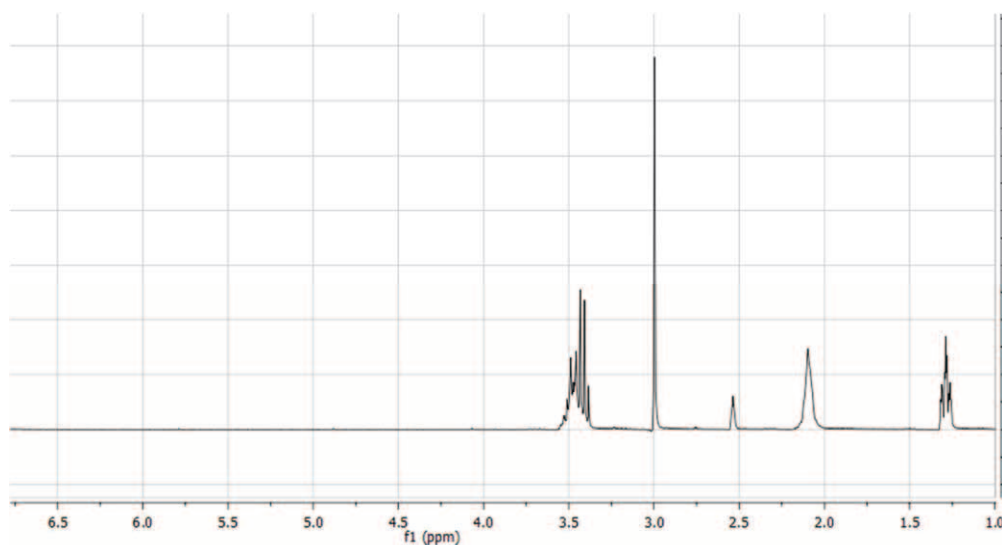


[empy][HSO<sub>4</sub>]





**[empy][HSO<sub>5</sub>]**



**1-butyl-3-methylimidazolium hydrogensulphate [bmim][HSO<sub>4</sub>]:**

- <sup>1</sup>H NMR (300 MHz, DMSO, TMS) δ 9.23 (s, 1H), 7.90 (s, 1H), 7.63 (s, 1H), 4.33-4.04 (t, 2H), 3.89 (s, 3H), 1.78 (p, 2H), 1.41-1.04 (m, 2H), 0.89 (t, 3H).
- <sup>13</sup>C NMR (75 MHz, DMSO, TMS) δ 137.4, 124.3, 123.0, 49.1, 36.4, 32.1, 19.5, 14.0.

**1-butyl-3-methylimidazolium peroxydisulphate [bmim][HSO<sub>5</sub>]:**

- <sup>1</sup>H NMR (300 MHz, DMSO, TMS) δ 9.18 (s, 1H), 7.81 (s, 1H), 7.74 (s, 1H), 4.20 (t, 2H), 3.88 (s, 3H), 1.79 (m, 2H), 1.29 (m, 2H), 0.91 (t, 3H); <sup>1</sup>H NMR (300 MHz, DMSO, TMS, neat) δ 4.30 (s, 1H from [HSO<sub>5</sub>])

- $^{13}\text{C}$  NMR (75 MHz, DMSO, TMS, neat)  $\delta$  136.6, 123.6, 122.3, 49.0, 35.8, 31.7, 19.0, 13.0.  
MS: Peaks found in positive ESI (+) mode Mass Spectra: 139; and negative ESI (-) mode Mass Spectra: 113.

#### **1-butylpyridinium hydrogensulphate [bpyr][HSO<sub>4</sub>]**

- $^1\text{H}$  NMR (300 MHz, DMSO, TMS)  $\delta$  9.20 (d, 2H), 8.66 (t, 1H), 8.20 (t, 2H), 6.38 (s, 1H), 4.68 (t, 2H), 1.92 (m, 2H), 1.31 (m, 2H), 0.92 (t, 3H);  $^1\text{H}$  NMR (300 MHz, DMSO, TMS, neat)  $\delta$  9.83 (s, 1H from [HSO<sub>4</sub>])
- $^{13}\text{C}$  NMR (75 MHz, DMSO, TMS, neat)  $\delta$  145.5, 144.3, 128.2, 61.2, 32.8, 18.8, 13.3.
- MS: Peaks found in positive ESI (+) mode Mass Spectra: 136; and negative ESI (-) mode Mass Spectra: 97

#### **1-butylpyridinium peroxysulphate [bpyr][HSO<sub>5</sub>]**

- $^1\text{H}$  NMR (300 MHz, DMSO, TMS)  $\delta$  9.21 (d, 2H), 8.64 (t, 1H), 8.18 (t, 2H), 4.68 (t, 2H), 1.89 (m, 2H), 1.27 (m, 2H), 0.88 (t, 3H);  $^1\text{H}$  NMR (300 MHz, DMSO, TMS, neat)  $\delta$  9.22 (s, 1H from [HSO<sub>5</sub>])
- $^{13}\text{C}$  NMR (75 MHz, DMSO, TMS, neat)  $\delta$  146.2, 145.5, 128.8, 61.2, 33.4, 19.4, 14.0.
- MS: Peaks found in positive ESI (+) mode Mass Spectra: 136; and negative ESI (-) mode Mass Spectra: 113

#### **1-ethyl-1-methylpyrrolidinium hydrogensulphate [empy][HSO<sub>4</sub>]**

- $^1\text{H}$  NMR (300 MHz, DMSO, TMS)  $\delta$  5.97 (s, 1H), 3.44 (m, 5H), 2.96 (s, 3H), 2.06 (m, 4H), 1.25 (t, 4H);  $^1\text{H}$  NMR (300 MHz, DMSO, TMS, neat)  $\delta$  10.88 (s, 1H from [HSO<sub>4</sub>])
- $^{13}\text{C}$  NMR (75 MHz, DMSO, TMS, neat)  $\delta$  63.4, 59.0, 47.5, 21.2, 9.1.
- MS: Peaks found in positive ESI (+) mode Mass Spectra: 114; and negative ESI (-) mode Mass Spectra: 97

#### **1-ethyl-1-methylpyrrolidinium peroxysulphate [empy][HSO<sub>5</sub>]**

- $^1\text{H}$  NMR (300 MHz, DMSO, TMS)  $\delta$  3.44 (m, 5H), 3.00 (s, 3H), 2.06 (m, 4H), 1.25 (t, 4H);  $^1\text{H}$  NMR (300 MHz, DMSO, TMS, neat)  $\delta$  8.57 (1H from [HSO<sub>5</sub>])
- $^{13}\text{C}$  NMR (75 MHz, DMSO, TMS, neat)  $\delta$  63.6, 59.0, 47.6, 21.8, 9.6.
- MS: Peaks found in positive ESI (+) mode Mass Spectra: 114; and negative ESI (-) mode Mass Spectra: 113

#### Literature

1. B. R. Travis, B. P. Ciaramitaro, B. Borhan, Eur. J. Org. Chem., 2002, 20, 3429.