

# Synthesis and properties of 1-alkyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imides

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## Introduction

4-Dimethylaminopyridine (DMAP) is a pyridine derivative used in the chemical synthesis as a nucleophilic catalyst for various reactions such as esterification [1], acylation [2],  $\beta$ -lactam synthesis [3] and many others. As it is a strong nucleophile, it easily undergoes quaternisation. 1-alkyl-4-(dimethylamino)pyridinium bromides are described in the literature for the entire homologue series  $C_4$ ,  $C_6$ - $C_{16}$ ,  $C_{20}$  and  $C_{22}$ . Compounds with substituents from octyl to tetradecyl are water soluble and effectively reduce surface tension [4]. 1-alkyl-4-(dialkylamino)pyridinium halides were tested as catalysts for the phase transfer and obtained comparable or higher yields than for conventional catalysts (quaternary ammonium salts) [5]. Chloromethylalkyl ethers and sulfides react with DMAP producing pyridinium chlorides with biological activity against bacteria and fungi, and also anti-static properties [6]. Moreover, they can be used as alkoxyethylating agents of NH groups in azoles [7].

DMAP-based ionic liquids are also known and can be found in the literature. 1-alkoxymethyl-4-(dimethylamino)pyridinium acesulfames have been successfully tested as ingredients of potential wood preservatives [8]. 1-butyl-4-(dimethylamino)pyridinium dicyanamide [9], 4-(dimethylamino)-1-methylpyridinium carbonate and methylcarbonate [10] and 4-(dimethylamino)-1-hexylpyridinium methanesulfonate [11] were patented as new basic ionic liquids – solvents in base catalysed chemical reactions. 1-alkoxymethyl-4-(dimethylamino)pyridinium [12] and 1,2,3-propanetri[oxymethyl-1-(4-dimethylaminopyridinium)] [13] bis(trifluoromethylsulfonyl)imides were tested as potential electrolytes for electrochemical capacitors. It turned out that the relatively low thermal stability of these salts, which results from presence of ether substituent, limits their possible applications. On the other hand, 1-alkyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imides, synthesised with substituents:  $C_1$ ,  $C_2$ ,  $C_4$  and  $C_6$  [14–19], have very high thermal stability and also are almost insoluble in water, as well as have low viscosity. Therefore, it seems to be reasonable to learn the properties of these compounds in homologous series.

The studies aimed to synthesise and compare the properties of 1-alkyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imides depending on the substituent length.

## Experimental part

Synthesis of 1-alkyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imides:

4-Dimethylaminopyridine was quaternised with 1-bromoalkanes in acetone for 24 hours at temperature of solvent boiling point. After the reaction mixture was cooled, the product was separated, rinsed with hexane, dried and recrystallised from acetone. The second step involved the addition of a stoichiometric amount of lithium bis(trifluoromethylsulfonyl)imide as a saturated solution to the aqueous solutions of 1-alkyl-4-dimethylaminopyridinium bromides with stirring

continuously. The reaction was conducted for 2 hours at ambient temperature with mechanically vigorously stirring. The product was isolated, rinsed a few times with water in order to remove sodium bromide and dried *under reduced pressure*.

Proton and carbon NMR spectra were taken using spectrometer Varian XL 300 with a generating frequency of 300 MHz for proton spectra and 75 MHz for carbon spectra against the internal standard tetramethylsilane (TMS). Deuterated chloroform was used as a solvent.

*1-methyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imide (IL 1)*

$^1\text{H NMR}$  ( $\text{CDCl}_3$ ) 3.94 (s, 6H), 3.95 (s, 3H), 6.82 (dd,  $J = 3.8$ , 2H), 7.94 (dd,  $J = 4.5$ , 2H),  $^{13}\text{CNMR}$  40.2, 44.9, 108.0, 113.4, 117.7, 121.9, 126.3, 142.5, 156.2

*1-ethyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imide (IL 2)*

$^1\text{H NMR}$  ( $\text{CDCl}_3$ ) 1.50 (t,  $J = 7.3$ , 3H), 3.23 (s, 6H), 4.17 (dd,  $J = 5.5$ , 2H), 6.85 (dd,  $J = 4.0$ , 2H), 8.00 (dd,  $J = 3.8$ , 2H),  $^{13}\text{CNMR}$  15.9, 40.0, 53.3, 108.1, 113.4, 117.6, 121.9, 126.1, 141.2, 156.2

*1-propyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imide (IL 3)*

$^1\text{H NMR}$  ( $\text{CDCl}_3$ ) 0.94 (t,  $J = 7.4$ , 3H), 1.88 (q,  $J = 5.5$ , 2H), 3.23 (s, 6H), 4.07 (t,  $J = 7.1$ , 2H), 6.86 (dd,  $J = 4.0$ , 2H), 7.98 (dd,  $J = 3.9$ , 2H),  $^{13}\text{CNMR}$  10.2, 24.0, 40.1, 59.7, 108.0, 113.4, 117.7, 121.9, 126.2, 141.6, 156.3

*1-hexyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imide (IL 6)*

$^1\text{H NMR}$  ( $\text{CDCl}_3$ ) 0.88 (t,  $J = 5.8$ , 3H), 1.30 (m, 6H), 1.82 (q,  $J = 4.9$ , 2H), 3.24 (s, 6H), 4.10 (t,  $J = 7.3$ , 2H), 6.86 (dd,  $J = 3.9$ , 2H), 7.98 (dd,  $J = 3.9$ , 2H),  $^{13}\text{CNMR}$  13.7, 22.2, 25.5, 30.7, 30.9, 40.1, 58.3, 108.1, 113.4, 117.7, 121.9, 126.2, 141.6, 156.2

*1-dodecyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imide (IL 12)*

$^1\text{H NMR}$  ( $\text{CDCl}_3$ ) 0.87 (t,  $J = 6.7$ , 3H), 1.27 (m, 18H), 1.84 (m, 2H), 3.24 (s, 6H), 4.01 (t,  $J = 7.4$ , 2H), 6.87 (dd,  $J = 4.0$ , 2H), 7.97 (dd,  $J = 3.8$ , 2H),  $^{13}\text{CNMR}$  14.0, 22.6, 25.9, 28.9, 29.2, 29.4, 29.5, 30.8, 31.8, 40.1, 58.4, 108.1, 113.4, 117.7, 121.9, 126.2, 141.6, 156.3

The melting point of the obtained compounds was measured using capillary apparatus Büchi Melting Point B-540. The density was measured using Density/Specific Gravity Meter DA-110 by Mettler Toledo at 20°C. The dynamic viscosity determination was performed using a thermostated Micro-Ostwald viscosimeter by Schott.

Phase transition temperatures were determined using differential scanning calorimeter DSC1 by Mettler Toledo. The measurements were conducted for samples of 10 to 40 mg weight at the temperature range of -120 to 180°C. The instrument was calibrated against an indium sample of 99.9999% purity and the melting point 156.61°C. The measurements were conducted at the temperature gradient of 10°C/min. The decomposition temperatures ( $T_{\text{onset}5\%}$  and  $T_{\text{onset}50\%}$ ) were determined using thermogravimeter Mettler Toledo TGA/DSC 1. Samples of 2 to 10 mg in weight were heated in the nitrogen atmosphere

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at the temperature range of 40 to 500°C, with a gradient of 10°C/min. Characteristic decomposition temperatures were determined for a 5% and 50% sample weight loss.

The electrode material was activated carbon Norit® GSX with specific surface area of 796 m<sup>2</sup>/g [20]. Round electrodes of 10 mg in weight and a geometrical surface area of 1.13 cm<sup>2</sup> were composed of 85% w/w of the aforementioned activated carbon, 10% w/w of a binder and 5% w/w of acetylene black. 1-ethyl-4-dimethylaminopyridinium imide (IL 2) and 1-propyl-4-dimethylaminopyridinium imide (IL 3) were used as the electrolyte.

Electrochemical tests were assembled in two-electrode system in the Swagelok® cell. Capacitance values of electrochemical capacitors were examined using cyclic voltammetry (1–50 mV/s), galvanostatic charging/discharging (0.2–2 A/g) and electrochemical impedance spectroscopy (100 kHz – 1 mHz) using potentiostat/galvanostat VMP3 (BIOLOGIC, France). The obtained values were expressed per active mass.

### Result discussion

Synthesis of 1-alkyl-4-(dimethylamino)pyridinium bromides in compliance with the S<sub>N</sub>2 mechanism can be found elsewhere [4]. In this studies conducted, the synthesis involved the use of acetone as a solvent instead of 2-propanol, which resulted in an increased reaction rate (similar yield was obtained after 24 hours instead of 48 hours) and a facilitated product separation (the precipitation happened after cooling the reaction mixture). The obtained halides were used as precursors for the synthesis of ILs. Individual 1-alkyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imides can also be found in the literature: with methyl, ethyl, butyl and hexyl substituent. However, there are no studies that compare the properties of the entire homologous series.

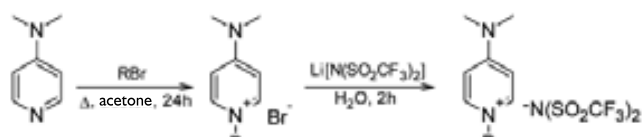


Fig. 1. Scheme of the studied synthesis

Ten room temperature ionic liquids (RTILs) were obtained as well as two salts of relatively low melting points (with substituents C<sub>1</sub> and C<sub>3</sub>). The density decreases as the alkyl chain gets longer and falls within a range of 1.25 to 1.47 g cm<sup>-3</sup>, whereas the viscosity increases in a range of 48.1 to 201.7 mPa s (Tab. 1).

Table 1

#### Obtained 1-alkyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imides

Salt	R	yield %	melting point °C	density [at 20°C] g cm <sup>-3</sup>	viscosity [at 20°C] mPa s
IL 1	CH <sub>3</sub>	93	53-54	-	-
IL 2	C <sub>2</sub> H <sub>5</sub>	92	liquid	1.47	48.1
IL 3	C <sub>3</sub> H <sub>7</sub>	90	43-44	-	-
IL 4	C <sub>4</sub> H <sub>9</sub>	96	liquid	1.40	84.2
IL 5	C <sub>5</sub> H <sub>11</sub>	95	liquid	1.38	98.0
IL 6	C <sub>6</sub> H <sub>13</sub>	96	liquid	1.36	109.6
IL 7	C <sub>7</sub> H <sub>15</sub>	96	liquid	1.34	125.0
IL 8	C <sub>8</sub> H <sub>17</sub>	94	liquid	1.32	133.8
IL 9	C <sub>9</sub> H <sub>19</sub>	96	liquid	1.30	142.9
IL 10	C <sub>10</sub> H <sub>21</sub>	95	liquid	1.27	163.5
IL 11	C <sub>11</sub> H <sub>23</sub>	93	liquid	1.27	181.2
IL 12	C <sub>12</sub> H <sub>25</sub>	94	liquid	1.25	201.7

Table 2

#### The properties obtained salt

Salt	T <sub>g</sub> <sup>a</sup>	T <sub>c</sub> <sup>b</sup>	T <sub>m</sub> <sup>c</sup>	T <sub>onset5%</sub> <sup>d</sup>	T <sub>onset50%</sub> <sup>e</sup>
IL 1	-	18.8	53.7	430	478
IL 2	-69.7	-	-	430	482
IL 3	-	-10.7	43.4	430	481
IL 6	-69	-	-	428	479
IL 11	-	-22.3	16.2	418	472

<sup>a</sup>glass transition temperature determined by means of DSC

<sup>b</sup>crystallisation temperature determined by means of DSC

<sup>c</sup>melting point determined by means DSC

<sup>d</sup>decomposition temperature determined for 5% weight loss

<sup>e</sup>decomposition temperature determined for 50% weight loss

The obtained ionic liquids exhibit a very high thermal stability; the 5% weight decomposition occurs only at approx. 430°C, whereas the 50% weight loss at approx. 480°C (Tab. 2). The compounds with even number of carbon atoms in the alkyl chain show no crystallisation tendencies, whereas their glass transition temperature is approximately at -70°C. On the other hand, the compounds with additional number of carbon substituent tend to crystallise and their crystallisation and melting points decrease as the substituent length increases.

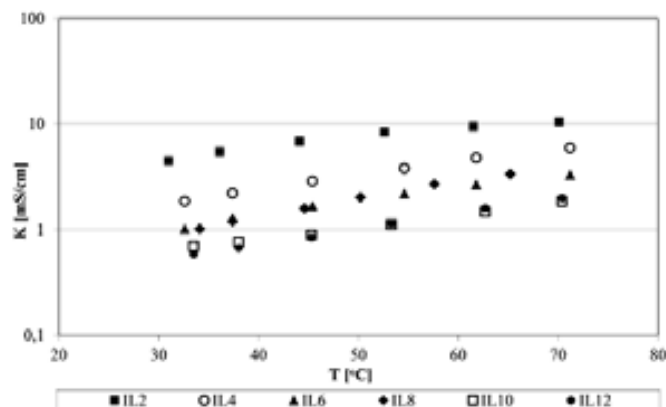


Fig. 2. Conductivity versus temperature for selected ionic liquids

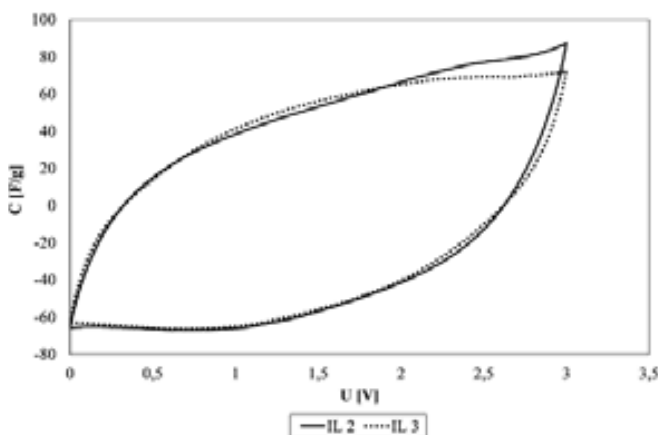


Fig. 3. Voltammetry characteristics of activated carbon for two different electrolytes (IL 2: 44 F g<sup>-1</sup>, IL 3: 43 F g<sup>-1</sup>) – 10 mV s<sup>-1</sup>, 60°C

Due to the simplicity of the synthesis, good physicochemical properties and high thermal stability, the obtained ionic liquids were tested electrochemically at two different temperatures: 25 and 60°C in order to determine their capacitance properties. Additionally, conductivity was measured. The capacitor energy increases proportionally with capacitances and square of applied voltage, therefore there is a special interest in electrolytes that allow operating voltage to be expanded in a range greater than the thermodynamic water stability (1.23 V). Ionic liquids are one of the electrolytes used in electrochemical capacitors.

They are characterized by high temperature stability, low vapour pressure and good conductivity at temperatures above 60°C [21, 22]. Their physical properties also include high viscosity that may cause wetting the electrode material difficult. In such cases, it is necessary to increase the temperature parameters of the capacitor performance. The conductivity values of selected ionic liquids were determined by means of electrochemical impedance spectroscopy in a temperature range of 30–70°C (Fig. 2). The ionic liquid **IL 2** showed the best parameters; therefore, the decision was to use it as an electrolyte for applications in electrochemical capacitors. The chain length of an ionic liquid also affects the value of capacitance (Fig. 3). The ionic liquid **IL 2** could be used at room temperature, while **IL 3** was a solid (Tab. 1), which prevented conducting the research at room temperature.

The relation between capacitance and frequency (Fig. 4) shows the behaviour of the capacitor with **IL 2** at two temperatures. The temperature rise did not result in a significant increase in the capacitance for this type of ionic liquid. Figure 5 presents the charge/discharge curves at an increased temperature (60°C) in the 1-ethyl- and 1-propyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imides environments. Higher values of capacitance were obtained for capacitor with **IL 2** as an electrolyte. This means there is a significant effect of conductivity on the system capacitance and effect of the substituent length.

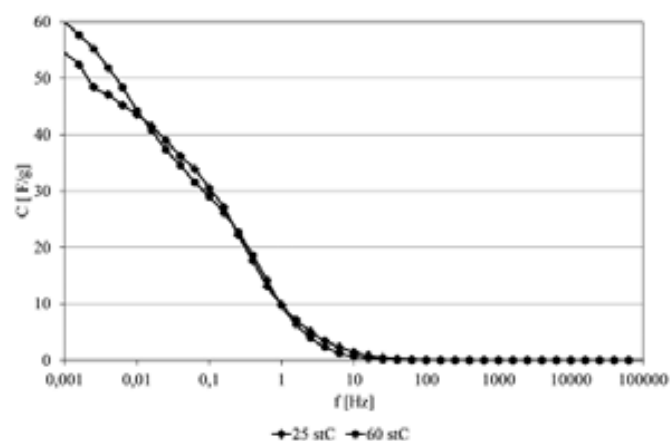


Fig. 4. Capacitance versus frequency for **IL 2** (25°C: 54 F g<sup>-1</sup>, 60°C: 60 F g<sup>-1</sup>)

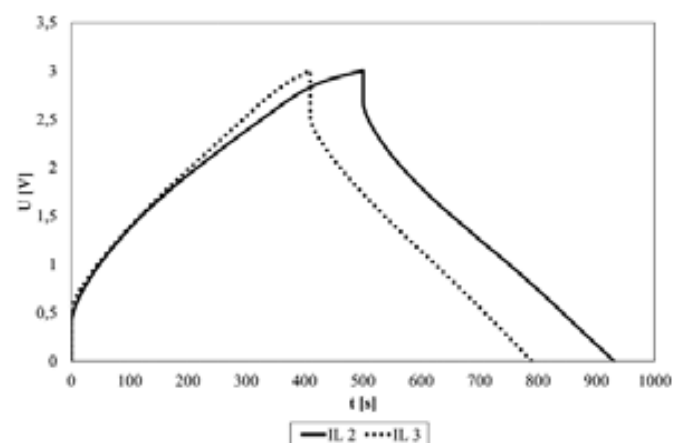


Fig. 5. Galvanostatic charge/discharge measurements of capacitors at 60°C (**IL 2**: 59 F g<sup>-1</sup>, **IL 3**: 52 F g<sup>-1</sup>) at 200 mA g<sup>-1</sup>

## Conclusions

The two-step synthesis was used to obtain 1-alkyl-4-(dimethylamino)pyridinium bis(trifluoromethylsulfonyl)imides. Ten room temperature ionic liquids (RTILs) were obtained as well as two salts of relatively low melting points (with substituents C<sub>1</sub> and C<sub>3</sub>). The density of the obtained compounds decreased with an increasing alkyl chain length in a range of 1.25–1.47 g/cm<sup>3</sup>, whereas the viscosity varied from 48.1 to 201.7 mPa s. The tested ionic liquids exhibit very high

thermal stability; the 5% weight decomposition occurred only at approx. 430°C, whereas the 50% weight loss at approx. 480°C.

Due to the simplicity of the synthesis, good physicochemical properties and high thermal stability, the obtained ionic liquids were tested by means of electrochemical methods. The capacitance value of supercapacitors based on the ionic liquids increased with increasing temperature. Much better capacitance characteristics were observed for compounds with shorter alkyl chains.

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### WYDARZENIA

#### LOTOS – Zintegrowany Raport Roczny 2015

Grupa Kapitałowa LOTOS opublikowała Zintegrowany Raport Roczny 2015, obejmujący większość swoich spółek zależnych. Publikacja daje pełen obraz działalności i wpływu organizacji na gospodarkę, środowisko oraz społeczeństwo. Raport został przygotowany w oparciu o najbardziej uznane międzynarodowo standardy raportowania pozafinansowego i zintegrowanego – Global Reporting Initiative (GRI G4), International Integrated Reporting Council (IIRC) oraz poddany niezależnej, zewnętrznej weryfikacji.

Ideą przewodnią Raportu za 2015 r. jest „**Rozwój dzięki innowacjom**”. Publikacja dostępna jest pod adresem: [www.raportroczny.lotos.pl](http://www.raportroczny.lotos.pl).

Z raportu wynika, że od 2010 r. wartość marki Grupy LOTOS wzrosła dwukrotnie do kwoty 1,4 mld zł. W tym czasie LOTOS, dzięki inwestycjom w obszar badań i rozwoju, osiągnął rekordowe wydobycie węgłowodórów, przekraczając tym samym cele wydobywcze na lata 2011–2015. Grupa Kapitałowa LOTOS stawia na innowacyjne programy rozwojowe, takie jak EFRA (kompleks nowoczesnych instalacji). Projekty rozwojowe i innowacyjne stanowią stałą częśćią inwestycji podejmowanych przez LOTOS. Wpisane są w biznesowe DNA Spółki, pozwalają maksymalizować zyski oraz budować wartość dla akcjonariuszy i pozostałych interesariuszy. Rok 2015 to także rok zakończenia z sukcesem realizacji strategii biznesowej i CSR na lata 2011–2015. Dzięki opublikowanemu raportowi LOTOS dokonuje przeglądu swoich dokonań i doskonali je w sposób ciągły. Proces raportowania motywuje do zmian zarządczych i pozwala lepiej definiować sposób, w jaki Spółka tworzy zintegrowaną wartość.

Jak pokazuje raport, Grupa LOTOS rozwija działania z zakresu zaangażowania społecznego. W 2015 r. powołano Fundację LOTOS wspierającą inicjatywy, które wpływają na rozwój infrastruktury i przynoszą korzyści dla społeczności lokalnych, ze szczególnym uwzględnieniem regionów, gdzie prowadzona jest działalność spółek należących do Grupy Kapitałowej LOTOS.

– *W Grupie LOTOS innowacyjność, kreatywność i odważne projekty idą w parze z odpowiedzialnością. W ostatnich latach Spółka pokazała, że poprzez nowe, przełomowe projekty jest w stanie maksymalizować zyski oraz swoją wartość. Równocześnie jesteśmy świadomi wpływu wywieranego przez organizację na otoczenie. W raporcie LOTOS analizuje swój rozwój ekonomiczny przez pryzmat sześciu kapitałów – finansowego, produkcyjnego, intelektualnego, ludzkiego, społecznego i naturalnego – powiedział Robert Pietryszyn, dyrektor generalny Grupy LOTOS.*

Zgodnie z dobrymi praktykami, raporty roczne koncernu publikowane są wyłącznie w formie elektronicznej. Stanowi to znaczące ułatwienie dla zróżnicowanych grup odbiorców, gdyż zapewnia możliwość korzystania z licznych funkcjonalności on-line, odpowiadających ich preferencjom. Wśród nich znajdują się m.in.: samodzielne komponowanie wybranych treści, narzędzia społecznościowe pozwalające dzielić się informacjami z innymi odbiorcami oraz wyszukiwarka treści, ułatwiająca szybki dostęp do szczególnie interesujących zagadnień. Publikacja jest przyjazna dla użytkowników i środowiska, m.in. dzięki opcji ekologicznego wydruku, ułatwieniom dla osób słabo widzących czy możliwości odsłuchu dla osób niewidzących.(abc)

(informacja prasowa Grupy LOTOS, 19. 09. 2016)

Dokończenie na stronie 544