**Extraction of Pb(II) and Co(II) using *N,N*-dioctylsuccinamate based Room Temperature Ionic Liquids Containing Aliphatic and Aromatic Cations**

*Nizakat Azra1,Farzana Nazir1, Mah Roosh1, Muhammad Awais Khalid2, Muhammad Adil Mansoor1, Sher Bahadar Khan3,4, Mudassir Iqbal1\**

*1Department of Chemistry, School of Natural Sciences (SNS), National University of Sciences & Technology (NUST), H-12, Islamabad, 44000, Pakistan*

*2Department of Environmental Sciences, University of Veterinary & Animal Sciences, Lahore-54000, Pakistan*

*3Department of Chemistry, Faculty of Science, King Abdulaziz University, P.O. Box 80203, Jeddah 21589, Saudi Arabia*

*4Center of Excellence for Advanced Materials Research, King Abdulaziz University, P.O. Box 80203, Jeddah 21589, Saudi Arabia*

\**Author for correspondence. Phone: +925190855575; E-mail:* mudassir.iqbal@sns.nust.edu.pk

**Supplementary Data**

### **S2.1.1: Synthesis of Cationic Parts**

#### **2.1.1.1: Synthesis of 1-Methyl-3-alkylimidazolium (Cnmim) cations**

**Synthesis of 1-Methyl-3-butylimidazolium bromide (3) and 1-methyl-3-octylimidazolium bromide (5)** weas performed by treating 1-methyl-1H-imidazolium (**1**) with corresponding 1-bromobutane **(2)** and 1-bromoctane **(4)** in 1:1 ratio in the presence of acetonitrile (30 mL) under reflux after reaction completion products obtained were light yellow-coloured oily liquids **(3 and 5)** respectively. These products were used in the next step to synthesize TSILs.

#### **2.1.1.2: Synthesis of 1-Methyl-3-ester-imidazolium(α-mim-ester) cation**

**Ethyl 2-(3-methyl-imidazol-1-yl)propanoate Bromide (7)** was synthesized by reacting **(1)** with 2-bromopropanoate **(6)** in acetonitrile under reflux for 24hrs and reaction progress was analyzed by taking TLC at various time intervals. After completion a light-yellow oily liquid **(7)** was obtained with yield of 92%.

#### **2.1.1.3: Synthesis of N-Alkylpyridinium (CnPy) cations**

**Synthesis of N-Butyl pyridinium bromide (9) and N-octyl pyridinium bromide (10)** was done by mixing pyridine (**8**) with corresponding 1-bromobutane **(2)** and 1-bromoctane **(4)** in 1:1 ratio by using acetonitrile as a solvent under reflux. The products formed **(9 and 10)** were viscous oily light brown liquids.

#### **2.1.1.4: Synthesis of diethyl dibutyl ammonium [N2244] cation**

**Synthesis of diethyl dibutyl ammonium [N2244] bromide (12)** was done by reacting quaternary ammonium salt diethyl amine **(11)** and **(4)** 1:2 ration respectively in the presence of three equivalents of base sodium bicarbonate in presence of acetonitrile under reflux after reaction completion off white colored powder **(12)** was obtained with yield of 89%.

### **S2.1.2: Synthesis of Anionic Parts**

**Synthesis of 2-(2-(di-n-octylamino)-succinic acid (15)** was synthesized by mixing succinic anhydride (**13**) (1g, 0.01mol) and dioctylamine (**14**) (2.41g, 0.01 mol) in dry THF (30 mL) and the mixture was heated at 50 oC overnight and then refluxed for 3 h. The product obtained was light-yellow oily liquid with 70% yield.

### **S2.1.3: Synthesis of TSIL’s**

Six new dioctylsuccinamate based ionic liquids TSIL’s were prepared as shown in Figure 3.

#### **2.1.3.1: Synthesis of 1-Methyl-3-butylimidazolium N,N-dioctylsuccinamate [C4mim][N88SA](L1):**

Mixture of1.02 g (2.9 mmol) (**15)** was dissolved in dry THF (30 mL) along with 0.143 g of sodium hydride (1.02 equivalent) were stirred for 3 to 4 h at 0 oC under argon atmosphere. 0.655 g (2.9 mmol) of (**3**) was added into the mixture and allowed to stir for one day. After reaction the obtained product **(16)** was light yellow liquid with 95% yield.

#### **2.1.3.2: Synthesis of 1-Methyl-3-octylimidazolium N,N-dioctylsuccinamate [C8mim] [N88SA] (L2):**

[N88SA]-[C8mim] + was synthesized by mixing 0.7g (0.0020 mol) of (**15)** in 30 mL of dry THF 1.02 equivalent sodium hydride (0.082g, 0.0021mol) mixture was stirred for 3 to 4 h under 0 ℃ under argon atmosphere. Then **(5)** (0.57g) in 10 mL of dry THF was added into the mixture and stirred for a day and a light yellow product **(17)** was obtained with 90 % yield.

#### **2.2.3.3: Synthesis of N-butylpyridinium N,N-dioctylsuccinamate [C4Py][N88SA] (L3):**

1.3 g (3.8 mmol) of (**15**) was dissolved in dry THF (30 mL) and 0.155 g of sodium hydride (1.02 equi) at 0 oC under argon atmosphere. The reaction mixture was stirred for 4 hours and then 1.17 g (3.8 mmol) of **(9)** was added into the reaction mixture and allowed to stir for one day the product **(19)** thus liberated was dark brown in color with 96% yield**.**

#### **2.1.3.4: Synthesis of N-octylpyridinium N,N-dioctylsuccinamate [C8Py] [N88SA] (L4):**

Mixture of **(15)** 0.7g (0.0020mol) was dissolved in 30 mL dry THF along with Sodium hydride (0.082g, 2.1 mmol) at 0 ℃ in argon atmosphere and was stirred for 5 h. Then **(10)** (0.55g, 2.0 mmol) was added along with 10 mL dry THF was added on stirring and was allowed to stir for 24 h. The product thus obtained was dark brown in colour with the yield of the 92%.

#### **2.1.3.5: Synthesis of 1-Methyl-3-ester-imidazolium N,N-dioctylsuccinamate [α-mim-ester][N88SA] (L5):**

1.3g (3.81 mmol) of **(15)** was mixed in 35 mL dry THF with 1.02 equivalent NaH (0.155g ,3.9 mmol) temperature kept was 0 ℃ under nitrogen atmosphere and was allowed to stir for 3 to 4 h, then cation **(7)** (0.95g, 3.81 mmol) was added and stirred for one day. Finally 89% yellow product was yielded.

#### **2.1.3.6: Synthesis of diethyl dibutyl ammonium N,N-dioctylsuccinamate [N2244][N88SA](L6):**

[N88SA]-[N2288] + synthesized by taking 1.0g (2.93 mmol) of **(15)** in 35 mL dry THF and 1.02 equivalents NaH (0.119g, 2.99 mmol) stirring this mixture for 4 hours at 0 ℃ under nitrogen atmosphere. After this **(12)** (0.63g, 2.93 mmol) was added and again stirred overnight with yield of 89%.