

# Effect of cation on the solubility of carbon dioxide in three bis(fluorosulfonyl)imide low viscosity ([FSI]) ionic liquids



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## ABSTRACT

The solubility of carbon dioxide in three bis(fluorosulfonyl)imide-based ionic liquids: N-propyl-n-methyl-piperidinium bis(fluorosulfonyl)imide ([p(3)mpip][FSI]), N-propyl-n-methyl-pyrrolidinium bis(fluorosulfonyl)imide ([p(3)mpyr][FSI]) and N-propyl-n-methyl-n,n-dimethyl-ammonium bis(fluorosulfonyl)imide ([N<sub>4441</sub>][FSI]) was measured at (298.15, 313.15, and 343.15) K and pressure up to about 2 MPa. The Henry's law constants for CO<sub>2</sub> in the three ILs, ([p(3)mpip][FSI], [p(3)mpyr][FSI] and [N<sub>4441</sub>][FSI]) at 298 K are (4.1, 4.0, and 3.8) MPa respectively, indicating the negligible effect of change of cation precursors on CO<sub>2</sub> solubility. Solubility of CO<sub>2</sub> in the present ILs with [FSI] anion is comparable with those in the ILs containing a more fluorinated common anion, [Tf<sub>2</sub>N]. The solubility data were correlated well with the Soave–Redlich–Kwong equation of state (SRK–EoS).

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## 1. Introduction

The rapid increase of carbon dioxide in the atmosphere due to anthropogenic emissions is believed to contribute to global warming and consequently in the global climate change [1,2]. Carbon capture and storage (CCS) is a short term abatement strategy to reduce CO<sub>2</sub> emissions from large stationary sources such as coal-fired power plants. CCS consists of capturing CO<sub>2</sub> from the emission point and then transporting it to a geological storage site for storage [3]. At present, post-combustion capture of CO<sub>2</sub> from large thermal power plants is not economical even with the most advanced technology, namely chemical absorption using reactive chemical solvents, due to the very high cost of solvent regeneration [4].

A promising alternative to reactive absorption is the physisorption of CO<sub>2</sub> in ionic liquids (ILs) [5] that requires less energy for solvent regeneration. Ionic liquids are salts with melting points less than 373.15 K and consist of organic cations such as imidazolium, pyridinium, pyrrolidinium, ammonium, sulfonium, or phosphonium derivatives, and organic or inorganic anions [6]. They have negligible vapor pressure and, more importantly, their properties are tunable by altering the chemical structure of the anion or/and the cation. Therefore, many ionic liquids with different

combination of anion/cation have been subject to investigation as potential solvents for CO<sub>2</sub> capture [7].

ILs with high physical absorption capacity of CO<sub>2</sub> is required in industrial CO<sub>2</sub> capture applications. Fluorinated ILs have received much attention for their high absorption capacity [8–22]. However, due to environmental reasons, ILs with less fluorine or no fluorine would be desirable [15]. We have selected three low-viscosity [FSI]-based ILs with three different cations based on three structurally different precursors: piperidinium, pyrrolidinium and phosphonium for CO<sub>2</sub> solubility measurements. The solubility is compared with that in ILs with the common anion [Tf<sub>2</sub>N], which has much higher stoichiometric fluorine content (3:1) to see whether the solubility is proportionate to the fluorine content. Moreover, the dominance of three different cations will also be observed in the present case where the anion is nominally fluorinated.

## 2. Experimental

### 2.1. Materials

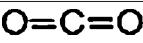
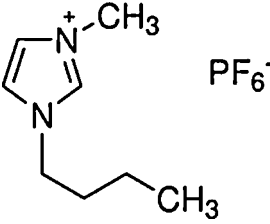
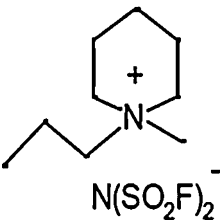
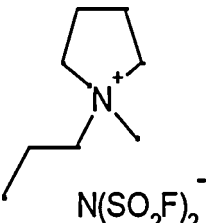
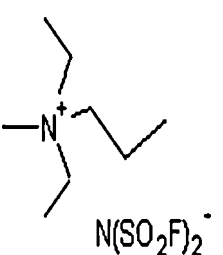
The structures and purity of the ionic liquids used in the current study are listed in Table 1.

### 2.2. Solubility apparatus and measurement

A computer-controlled fully automatic gravimetric microbalance (Hidden Isochema Ltd., IGA003) was used. The weighing

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**Table 1**  
Chemicals used in the current study: structure, abbreviation, supplier and purity.

Chemical	Structure	Abbreviation	Supplier and purity
Carbon dioxide		CO <sub>2</sub>	Praxair (99.99%)
1-Butyl-3-methylimidazolium hexafluorophosphate		[bmim][PF <sub>6</sub> ]	Sigma–Aldrich (97.0%)
1-Methyl-1-propylpiperidinium bis(fluorosulfonyl)imide		[mp(3)pip][FSI]	Solvionic (99.9%)
N-propyl- n-methylpyrrolidinium bis(fluorosulfonyl)imide		[mp(3)pyrr][FSI]	Solvionic (99.9%)
N,n-diethyl-n-methyl-n-propylammonium bis(fluorosulfonyl)imide		[N <sub>1223</sub> ][FSI]	Solvionic (99.9%)

mechanism consists of an electrobalance with a sample arm and a counterweight arm. The stainless steel sample bucket is attached to the sample arm by gold hang-down chains. Both arms consist of similar components to minimize the resultant buoyancy effect. The balance components are kept inside pressure vessel. The stable resolution of the balance is 1  $\mu\text{g}$ .

The apparatus can be used in static (intermittent gas flow) or dynamic (continuous flow of gas) mode and the measurements in this study were performed in static mode. After a small amount of ionic liquid samples (50–90 mg) was loaded in the sample bucket, the chamber was properly sealed. The sample was then degassed and dried by pulling vacuum on the sample with a diaphragm pump (vacuum brand) until the pressure reaches about 20 mbar, followed by a turbo pump (Pfeiffer) to reach and maintain vacuum (<5 mbar) at 348 K for minimum of 12 h. The chamber temperature was controlled using a water jacket connected to a constant temperature water bath (Polyscience). The sample temperature was measured with a platinum resistance thermocouple ( $\pm 0.1$  K), and pressure was measured with a pressure transducer (Druck PDCR 4010,  $\pm 8$  mbar).

When the weight of the sample became stable, the temperature was reset to the experimental temperature. When the desired

temperature was reached at vacuum condition, the absorption processes was initiated by sending carbon dioxide via a mass flow controller (MFC) at a desired flow rate to reach a pre-set pressure inside the microbalance chamber. The weight change upon absorption is automatically recorded and displayed as a function of time. The equilibrium status was ensured by observing the mass uptake versus time graph. The weight recorded at equilibrium is the real-time weight of CO<sub>2</sub>-absorbed sample. After equilibrium was reached, further CO<sub>2</sub> was sent to reach the second pressure of the isotherm and this process is repeated for all other pressure measurements. At the end of isotherm measurement, the real-time data was corrected for the buoyancy effect.

### 2.3. Density measurement

Densities of the ionic liquids at temperatures from (278.15 to 353.15) K, and at atmospheric pressure were measured by an Anton Paar density meter (DMA 4500). The cell of the density-meter was carefully cleaned with acetone, methanol and distilled water and dried for 30 min at 353 K before injecting the ionic liquids. The density meter was calibrated with air and bi-distilled water. 2 mL of a sample was slowly injected through the inlet that would flow

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