In situ Crystallization: Phase Transition and Polymorphism in Ionic Liquids

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Salts of a wide variety of organic cations with anions having diffuse negative charges are termed ionic liquids according to their state of aggregation at standard temperature and pressure. These materials are important as non-volatile solvents in electrochemistry, chemical reactions and materials applications [1]. Structural characterization has been a challenge as ionic liquids tend to form glasses when cooled from a melt, thereby inhibiting the determination of their crystal structures. While crystal seeding has been employed to induce crystal growth of materials melting >40°C, such an approach is impractical for those that are liquid at ambient/sub-ambient temperatures. In situ cryo-crystallization has emerged as a new technique for the growth of single crystals of molecular liquids and gases [2]. We have used this technique (in association with DSC) to grow and structurally characterize single crystals of low melting ionic liquids for the first time. Results will be presented for materials having differently substituted imidazolium, pyridinium and pyrrolidinium cations with a variety of anions. Polymorphism and phase transition studies on these materials will also be described.

[1] *Ionic liquids in synthesis,* ed. P. Wasserscheid and T. Welton, VCH-Wiley, Weinheim, Germany, 2002. [2] Boese R., Kirchner M. T., Billups W. E., Norman L. R., *Angew. Chem. Int. Ed.*, 2003, **42**, 1961.

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