

Application of ionic liquids in analytical chemistry

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Room-temperature ionic liquids (ILs) are gaining wide recognition as novel solvents in chemistry. Their application in analytical chemistry, especially in separating analytes, is merited because ILs have some unique properties, such as negligible vapor pressure, good thermal stability, tunable viscosity and miscibility with water and organic solvents, as well as good extractability for various organic compounds and metal ions. This review gives a brief overview of the application of ILs in analytical chemistry, including sample preparation, chromatographic/capillary electrophoretic (CE) separation, and detection.

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1. Introduction

Room-temperature ILs, resulting from the combination of organic cations and various anions that may be liquids at room temperature, are salts with melting points of below *ca.* 100°C. Some ILs are liquid at over 400°C, and some at as low as -96°C. The ILs investigated most comprise the organic 1-alkyl-3-methylimidazolium ([C_nMIM]), *N*-alkylpyridinium, tetraalkylammonium or tetraalkylphosphonium cations. The anions are either organic or inorganic, including: hexafluorophosphate [PF₆]; tetrafluoroborate [BF₄]; trifluoromethylsulfonate [CF₃SO₃]; bis[(trifluoromethyl)sulfonyl]amide [(CF₃SO₂)₂N]; trifluoroethanoate [CF₃CO₂]; acetate [CH₃CO₂]; nitrate, and halide [1]. Some ILs are shown in Fig. 1.

The physicochemical properties of ILs depend on the nature and size of both their cation and anion constituents. Their application in analytical chemistry, especially in separating analytes, is merited because ILs have some unique properties, such as negligible vapor pressure, good thermal stability, tunable viscosity and miscibility with water and

organic solvents, as well as good extractability for various organic compounds and metal ions [2,3]. Table 1 illustrates some physicochemical properties of the ILs commonly used in analytical chemistry. More detailed information on the properties of ILs can be found in a review [9].

We give a brief overview of the application of ILs in analytical chemistry, which is divided into three areas: sample preparation; chromatographic and CE separation; and, detection. We do not discuss studies on the separation and the determination of ILs [10–12], as this is outside the scope of this review.

2. Sample preparation

ILs have negligible vapor pressure and non-flammability as well as good solubility for inorganic and organic compounds. They are therefore useful in liquid-liquid extraction (LLE), liquid phase microextraction (LPME), and solid phase microextraction (SPME).

2.1. LLE

Dai et al. [13] first reported a very highly efficient procedure for extraction of Sr²⁺ from aqueous phase into disubstituted imidazolium hexafluorophosphates and bis(trifluoromethyl)sulfonylamides by using dicyclohexano-18-crown-6 as extractant.

Later, Visser et al. studied the extraction of Na⁺, Cs⁺, and Sr²⁺ from aqueous solution to 1-alkyl-3-methylimidazolium hexafluorophosphate ([C_nMIM][PF₆], *n* = 4, 6, 8) ILs by crown ethers [14], Cd²⁺, Co²⁺, Ni²⁺, and Fe³⁺ to ILs ([C_nMIM][PF₆], *n* = 4, 6) by various organic and inorganic

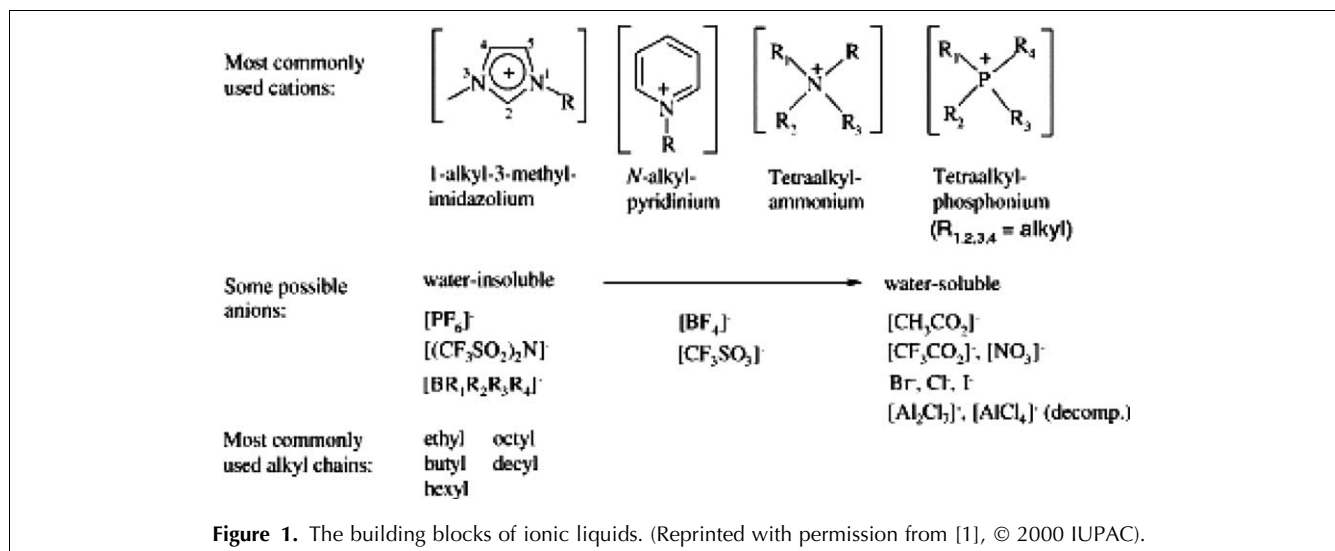
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**Table 1.** Some physicochemical properties of the commonly used ILs in analytical chemistry

ILs	Melting point (°C)	Density (g/mL)	Viscosity (mPa s)	Water solubility (g/100 mL)	Conductivity (S/m)	References
[C ₄ MIM][PF ₆]	10, -8	1.36–1.37 (25°C)	148–450 (25°C)	1.88	0.14 (25°C)	[4,5]
[C ₆ MIM][PF ₆]	-61	1.29–1.31 (25°C)	560–586 (25°C)	0.75		
[C ₈ MIM][PF ₆]		1.20–1.23 (25°C)	682–710 (25°C)	0.20		
[CMIM][(CF ₃ SO ₂) ₂ N]	22	1.56	44 (20°C)		0.84 (20°C)	[2,6,7]
[C ₂ MIM][(CF ₃ SO ₂) ₂ N]	-3	1.50	34 (20°C)	1.77	0.88 (20°C)	
[C ₄ MIM][(CF ₃ SO ₂) ₂ N]	-4	1.42	52 (20°C)	0.80	0.39 (20°C)	
[C ₆ MIM][(CF ₃ SO ₂) ₂ N]		1.33		0.34		
[C ₈ MIM][(CF ₃ SO ₂) ₂ N]		1.31		0.21		
[C ₄ MIM][Cl]	65, 41	1.10 (Supercooled liquid at 25°C)	Solid	Miscible	Solid	[5,6]
[C ₂ MIM][BF ₄]	15	1.15 (30°C), 1.28 (25°C)	37 (25°C)	Miscible		[2,3,8]
[C ₄ MIM][BF ₄]	-81	1.17 (30°C), 1.21 (25°C)	233 (30°C), 180 (25°C)	Miscible	0.17(25°C)	
[C ₆ MIM][BF ₄]			314 (20°C), 177 (30°C)	Miscible		
[C ₄ MIM][CF ₃ SO ₃]	16	1.29 (20°C)	90 (20°C)	Miscible	0.37 (20°C)	[6]

extractants [15], and selective extraction of Hg²⁺ and Cd²⁺ from water by task-specific ILs [16].

Chun et al. [17] investigated the influence of structural variation in [C_{*n*}MIM][PF₆] (*n* = 4–9) ILs on the selectivity and the efficiency of competitive alkali metal salt extraction by using a crown ether, dicyclohexano-18-crown-6, as an extractant.

Ag⁺, Hg²⁺, Cu²⁺, Pb²⁺, Cd²⁺ and Zn²⁺ were successfully extracted into [C₄MIM][PF₆] by employing dithizone as a chelator to form neutral metal-dithizone complexes [18]. It was found that the extraction efficiency of IL is higher than that of chloroform at low pH. Furthermore, metal ions can be extracted from aqueous phase into [C₄MIM][PF₆] and then back-extracted into aqueous phase with high recovery by manipulating the pH value of the extraction system.

Hence, it is possible to separate and to preconcentrate heavy metal ions, as well as to recycle the [C₄MIM][PF₆].

More recently, eight kinds of *N*-alkyl aza-18-crown-6 ethers in ILs, [C_{*n*}MIM][(CF₃SO₂)₂N] (*n* = 2–8), were investigated as recyclable extractants for separation of Sr²⁺ and Cs⁺ from aqueous solutions. Because of the pH-sensitive complexation capability of these ligands, it was possible to develop a facile stripping process, permitting both the macrocyclic ligands and the ILs to be recycled. A highly selective system toward Sr²⁺ was developed after optimizing selection of the macrocyclic ligands and ILs [7]. The same authors also studied the extraction of Cs⁺ from aqueous solution to hydrophobic ILs, without introducing an organophilic anion in the aqueous phase, by using calix-[4]arene-bis(*tert*-octylbenzo-crown-6) as an extractant and sodium

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