Ionic liquid

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An **ionic liquid** (IL) is a salt in the liquid state. In some contexts, the term has been restricted to salts whose melting point is below some arbitrary temperature, such as 100 °C (212 °F). While ordinary liquids such as water and gasoline are predominantly made of electrically neutral molecules, ionic liquids are largely made of ions and short-lived ion pairs. These substances are variously called **liquid electrolytes**, **ionic melts**, ionic fluids, fused salts, liquid salts, or ionic glasses.

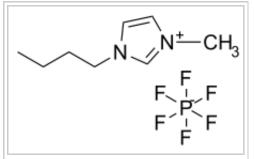
Ionic liquids have many applications, such as powerful solvents and electrically conducting fluids (electrolytes). Salts that are liquid at nearambient temperature are important for electric battery applications, and have been used as sealants due to their very low vapor pressure.

Any salt that melts without decomposing or vaporizing usually yields an ionic liquid. Sodium chloride (NaCl), for example, melts at 801 °C (1,474 °F) into a liquid that consists largely of sodium cations (Na⁺) and chloride anions (C1). Conversely, when an ionic liquid is cooled, it often forms an ionic solid which may be either crystalline or glassy.

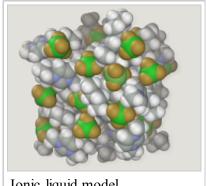
The ionic bond is usually stronger than the Van der Waals forces between the molecules of ordinary liquids. For that reason, common salts tend to melt at higher temperatures than other solid molecules. Some salts are liquid at or below room temperature. Examples include compounds based on the 1-Ethyl-3-methylimidazolium (EMIM) cation and include: EMIM:Cl, EMIM dicyanamide, $(C_2H_5)(CH_3)C_3H_3N_2^+\cdot N(CN)_2^-$, that melts at -21 °C (-6 °F);^[1]

and 1-butyl-3,5-dimethylpyridinium bromide which becomes a glass below -24 °C (-11 °F). [2] Low-temperature ionic liquids can be compared to ionic solutions, liquids that contain both ions and neutral molecules, and in particular to the so-called deep eutectic solvents, mixtures of ionic and non-ionic solid substances which have much lower melting points than the pure compounds. Certain mixtures of nitrate salts can have melting

The term "ionic liquid" in the general sense was used as early as 1943. [4]



The chemical structure of 1-butyl-3methylimidazolium hexafluorophosphate ([BMIM]PF₆), a commonly encountered ionic liquid.



Ionic liquid model

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points below 100 °C.[3]

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History

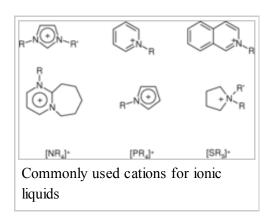
The discovery date of the "first" ionic liquid is disputed, along with the identity of its discoverer. Ethanolammonium nitrate (m.p. 52-55 °C) was reported in 1888 by S. Gabriel and J. Weiner. [5] One of the earliest truly room temperature ionic liquids was ethylammonium nitrate (C_2H_5)N H_3^+ ·N O_3^- (m.p. 12 °C), synthesized in 1914 by Paul Walden. [6] In the 1970s and 1980s ionic liquids based on alkyl-substituted imidazolium and pyridinium cations, with halide or tetrahalogenoaluminate anions, were initially developed for use as electrolytes in battery applications. [7][8]

An important property of the imidazolium halogenoaluminate salts is that their physical properties—such as viscosity, melting point, and acidity—could be adjusted by changing the alkyl substituents and the imidazolium/pyridinium and halide/halogenoaluminate ratios. ^[9] Two major drawbacks for some applications were moisture sensitivity and acidity/basicity. In 1992, Wilkes and Zawarotko obtained ionic liquids with 'neutral' weakly coordinating anions such as hexafluorophosphate (PF_6) and tetrafluoroborate (BF_4), allowing a much wider range of applications. ^[10] Recently a new class of air— and moisture—stable, neutral ionic liquids became available. Research has also been moving away from hexafluorophosphate and tetrafluoroborate towards less toxic alternatives such as bistriflimide $[(CF_3SO_2)_2N]^-$ or away from halogenated compounds completely. Moves towards less toxic cations have also been growing, with compounds like ammonium salts (such as choline) proving to be as flexible a scaffold as imidazolium.

Characteristics

Ionic liquids are often moderate to poor conductors of electricity, non-ionizing (e.g. non-polar), highly viscous and frequently exhibit low vapor pressure. Their other properties are diverse: many have low combustibility, excellent thermal stability, wide liquid regions, and favorable solvating properties for a range of polar and non-polar compounds. Many classes of chemical reactions, such as Diels-Alder reactions and Friedel-Crafts reactions, can be performed using ionic liquids as solvents. Recent work has shown that ionic liquids can serve as solvents for biocatalysis. [11] The miscibility of ionic liquids with water or organic solvents varies with side chain lengths on the cation and with choice of anion. They can be functionalized to act as acids, bases or ligands, and have been used as precursor salts in the preparation of stable carbenes. Because of their distinctive properties, ionic liquids are attracting increasing attention in many fields, including organic chemistry, electrochemistry, catalysis, physical chemistry, and engineering; see for instance magnetic ionic liquid.

Despite their extremely low vapor pressures ($\approx 10^{-10}$ Pa at 25°C), some ionic liquids can be distilled under vacuum conditions at temperatures near 300 °C. [12] In the original work by Martyn Earle, et al., the authors wrongly concluded that the vapor was made up of individual, separated ions, [13] but was later proven that the vapors formed consisted of ion-pairs. [14] Some ionic liquids (such as 1-butyl-3-methylimidazolium nitrate) generate flammable gases on thermal decomposition. Thermal stability and melting point depend on the liquid's components. Thermal stability of various RTILs (Room Temperature Ionic Liquid) are available. The thermal stability of a task-specific ionic



liquid, protonated betaine bis(trifluoromethanesulfonyl)imide is of about 534 K (502 °F) and N-Butyl-N-Methyl pyrrolidinium bis(trifluoromethanesulfonyl)imide was thermally stable up to 640 K. $^{[15]}$ The upper limits of thermal stability of ionic liquids reported in the literature are usually based upon fast (about 10 K/min) TGA scans, and they do not imply long-term (several hours) thermal stability of ionic liquids, which is limited to less than 500 K for most ionic liquids. $^{[16]}$

The solubility of different species in imidazolium ionic liquids depends mainly on polarity and hydrogen bonding ability. Saturated aliphatic compounds are generally only sparingly soluble in ionic liquids, whereas olefins show somewhat greater solubility, and aldehydes can be completely miscible. This can be exploited in biphasic catalysis, such as hydrogenation and hydrocarbonylation processes, allowing for relatively easy separation of products and/or unreacted substrate(s). Gas solubility follows the same trend, with carbon dioxide gas showing exceptional solubility in many ionic liquids. Carbon monoxide is less soluble in ionic liquids than in many popular organic solvents, and hydrogen is only slightly soluble (similar to the solubility in water) and may vary relatively little between the more commonly used ionic liquids. Different analytical techniques have yielded somewhat different absolute solubility values.

Room temperature varieties

Room temperature ionic liquids consist of bulky and asymmetric organic cations such as 1-alkyl-3-methylimidazolium, 1-alkylpyridinium, *N*-methyl-*N*-alkylpyrrolidinium and ammonium ions. Phosphonium cations are less common, but offer some advantageous properties. [17] A wide range of anions are employed, ranging from simple halides, which generally suffer high melting points, to inorganic anions such as tetrafluoroborate and

hexafluorophosphate, and to large organic anions like bistriflimide, triflate or tosylate. There are also many interesting uses of ionic liquids with simple non-halogenated organic anions such as formate, alkylsulfate, alkylphosphate or glycolate. The melting point of 1-butyl-3-methylimidazolium tetrafluoroborate with an imidazole skeleton is about $-80~^{\circ}\text{C}$ ($-112~^{\circ}\text{F}$) and it is a colorless liquid with high viscosity at room temperature. If a highly

asymmetric cation is combined with a highly asymmetric anion, formed ionic liquid may not freeze down to very low temperatures (down to -150°C) and the glass transition temperature was detected below

-100°C in the case of ionic liquids with N-methyl-N-alkylpyrrolidinium cations and fluorosulfonyl-trifluoromethanesulfonylimide (FTFSI).^[18]

In many synthetic processes using transition metal catalysts, metal nanoparticles play an important role as the actual catalyst or as a catalyst reservoir. ILs are an appealing medium for the formation and stabilization of catalytically active transition metal nanoparticles. More importantly, ILs can be made that incorporate coordinating groups, [19] for example, with nitrile groups on either the cation or anion (CN-IL). In various C-C coupling reactions catalyzed by a palladium catalyst, it has been found that palladium nanoparticles are better stabilized in CN-IL compared to non-functionalized ionic liquids; thus enhanced catalytic activity and recyclability are realized. [20]



Table salt NaCl and ionic liquid [bmim]NTf₂ at 27°C

Low temperature varieties

Low temperature ionic liquids (below 130 K) have been proposed as the fluid base for an extremely large diameter spinning liquid mirror telescope to be based on the Earth's moon.^[21] Low temperature is advantageous in imaging long wave infrared light which is the form of light (extremely red-shifted) that arrives from the most distant parts of the visible universe. Such a liquid base would be covered by a thin metallic film that forms the reflective surface. Low volatility is important for use in lunar vacuum conditions.

Applications

ILS find a variety of industrial applications. A few industrial applications are described below; more detailed information can be found in a recent review article. [22]

Chemical industry

The first major industrial IL application was the BASIL (Biphasic Acid Scavenging utilizing Ionic Liquids) process by BASF, in which 1-alkylimidazole scavenged the acid from an existing process. This then results in the formation of an IL which can easily be removed from the reaction mixture. This increased the space/time yield of the reaction by a factor of 80,000. [23]

Eastman operated an IL-based plant for the synthesis of 2,5-dihydrofuran from 1996 to 2004. [24]

The dimersol process is a traditional way to dimerize short chain alkenes into branched alkenes of higher molecular weight. Y. Chauvin and H. Olivier-Bourbigou developed an IL-based add-on to this process called the difasol process.^[25]

Ionikylation is an IL—based process developed by Petrochina for the alkylation of four-carbon olefins with isobutane. Their 65,000 tonne per year plant is claimed to be the biggest industrial application of ionic liquids to date. [25]

Pharmaceuticals

Recognising that approximately 50% of commercial pharmaceuticals are organic salts, ionic liquid forms of a number of pharmaceuticals have been investigated. Combining a pharmaceutically active cation with a pharmaceutically active anion leads to a Dual Active ionic liquid in which the actions of two drugs are combined. [26][27]

Cellulose processing

At a volume of some 700 billion tons, cellulose is the earth's most widespread natural organic polymer and thus highly important as a renewable resource. But even out of nature's annual 40 billion tons output, only approx. 5% is used as feedstock for further processing. More intensive exploitation of cellulose, as a renewable feedstock, is aided by the development of suitable solvents for mechanical and chemical processing. ILs have been shown to be highly effective at solvating cellulose to technically useful concentrations. [28] This may allow for development of new processes and intensification of existing ones substituting existing cellulose solvents for ILs. The use of molten salts in cellulose processing originally dates back to a patent application from 1930 where mixtures of molten 1-alkylpyridinium chlorides were used to dissolve cellulose, followed by subsequent chemical and mechanical processing. [29] Further advances have generally led to lowering of melting points and viscosities to a point where there are now room temperature ionic liquids available that dissolve cellulose. [30]

There are also examples of *in-situ* cellulose valorization in ionic liquids into various valuable chemicals, e.g. glucose esters, sorbitol, and alkylgycosides.^[31] Such techniques create new opportunities for fine chemicals production and can be, of course, useful for commercial applications.

Making viscose-based fibres from dissolving pulp currently involves the use, and subsequent disposal, of great volumes of various chemical auxiliaries, esp. carbon disulfide (CS₂). Major volumes of wastewater need to be disposed of. Following in the footsteps of the lyocell process, which uses hydrated N-Methylmorpholine N-oxide, as a novel non-aqueous solvent for the dissolution of the pulp, it has been suggested that IL can greatly simplify these processes, serving as solvents that are potentially recyclable. The "Institut für Textilchemie und Chemiefasern" (ITCF) in Denkendorf and BASF are jointly investigating the properties of fibers spun from solutions of cellulose dissolved in IL in a pilot plant.^[32] The dissolution of cellulose–based materials like tissue paper waste, generated in chemical industries and at research laboratories, in room temperature IL 1-butyl-3-methylimidazolium chloride, bmimCl and the recovery of valuable compounds by electrodeposition from this cellulose matrix was studied.^[33]

Fundamental Research

ILs can be used as model systems for electrode-electrolyte interfaces in ultra-high vacuum systems. [34]

Algae processing

Algae are perhaps the most widespread organisms on Earth, occupying most niches on the planet. Algae perform photosynthesis and produce high-energy molecules such as lipids and sugars, that can be converted to useful chemicals such as biodiesel, ethanol and other biofuels. To accomplish this, however, algae must be harvested and their constituents extracted from within the cells in an economically viable industrial process. ILs have been shown to be effective at destroying the cell wall and releasing cell contents using a fraction of the energy, and potentially the cost, of current harvesting and extraction processes. [35]

Dispersants

ILs can act as dispersing agents in paints to enhance finish, appearance and drying properties. [36]

ILs are used for dispersing nanomaterials at IOLITEC.

Gas handling

ILs have several properties that make them useful in gas storage and handling applications, including low vapor pressure, stability at high temperatures, and solvation for a wide variety of compounds and gases. They also have weakly coordinating anions and cations which are able to stabilize polar transition states. Many ionic liquids can be reused with minimal loss of activity.

The company Air Products uses ILs instead of pressurized cylinders as a transport medium for reactive gases such as trifluoroborane, phosphine and arsine. The gases are dissolved in the liquids at or below atmospheric pressure and are easily withdrawn from the containers by applying a vacuum.

Gas manufacturer Linde exploits the low solubility of hydrogen in ILs to compress the gas up to 450 bar in filling stations by using an ionic liquid piston compressor, [37] which has only 8 moving parts (down from about 500 in a conventional piston pump).

IL 1-butyl-3-methylimidazolium chloride has been used for separating hydrogen from ammonia borane. [38]

ILs and amines can be used to capture carbon dioxide ${\rm CO_2}$ and purify natural gas. [39] [40]

Nuclear fuel reprocessing

IL 1-butyl-3-methylimidazolium chloride has been investigated as a non-aqueous electrolyte media for the recovery of uranium and other metals from spent nuclear fuel and other sources. [41][42][43] Protonated betaine bis(trifluoromethanesulfonyl) imide has been investigated as a solvent for uranium oxides. [44] Ionic liquids, N-butyl-N-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide and N-methyl-N-propylpiperidinium bis(trifluoromethylsulfonyl)imide, have been investigated for the electrodeposition of Europium and Uranium metals respectively. [45][46]

Solar thermal energy

ILs have potential as a heat transfer and storage medium in solar thermal energy systems. Concentrating solar thermal facilities such as parabolic troughs and solar power towers focus the sun's energy onto a receiver which can generate temperatures of around $600~^{\circ}\text{C}$ (1,112 $^{\circ}\text{F}$). This heat can then generate electricity in a steam or other cycle. For buffering during cloudy periods or to enable generation overnight, energy can be stored by heating an intermediate fluid. Although nitrate salts have been the medium of choice since the early 1980s, they freeze at $220~^{\circ}\text{C}$ (428 $^{\circ}\text{F}$) and thus require heating to prevent solidification. Ionic liquids such as $C_4^{\text{mim}}[BF_4]$ have more favorable liquid-phase temperature ranges (-75 to 459 $^{\circ}\text{C}$) and could therefore be excellent liquid thermal storage media and heat transfer fluids. [47]

Food and bioproducts

IL 1-butyl-3-methylimidazolium chloride completely dissolves freeze dried banana pulp and with an additional 15% DMSO, lends itself to Carbon-13 NMR analysis. In this way the entire banana complex of starch, sucrose, glucose, and fructose can be monitored as a function of banana ripening.^[48]

ILs can extract specific compounds from plants for pharmaceutical, nutritional and cosmetic applications, such as the antimalarial drug artemisinin from the plant *Artemisia annua*. ^[49]

Waste recycling

ILs can aid the recycling of synthetic goods, plastics and metals. They offer the specificity required to separate similar compounds from each other, such as separating polymers in plastic waste streams. This has been achieved using lower temperature extraction processes than current approaches^[50] and could help avoid incinerating plastics or dumping them in landfill.

Batteries

Researchers have identified ILs that can replace water as the electrolyte in metal-air batteries. ILs have great appeal because they evaporate at much lower rates than water, increasing battery life by drying slower. Further, ILs have an electrochemical window of up to six volts ^[51](versus 1.23 for water) supporting more energy-dense metals. Energy densities from 900-1600 watt-hours per kilogram appear possible. ^[52]

A Metal-air battery draws oxygen through a porous ambient "air" electrode (-cathode) and produces water, hydrogen peroxide, or hydroxide anions depending on the nature oxygen reduction catalyst and electrolyte. These compounds store the electrons released by the oxidation of the anode.

Safety

ILs' low volatility effectively eliminates a major pathway for environmental release and contamination. However, this property is distinct from toxicity. ILs' aquatic toxicity is as severe as or more so than many current solvents. [53][54][55] Mortality isn't necessarily the most important metric for measuring impacts in aquatic environments, as sub-lethal concentrations change organisms' life histories in meaningful ways. Balancing VOC reductions against waterway spills (via waste ponds/streams, etc.) requires further research. ILs' substituent diversity simplify the process of identifying compounds that meet safety requirements.

Ultrasound can degrade solutions of imidazolium-based ionic liquids with hydrogen peroxide and acetic acid to relatively innocuous compounds.^[56]

Despite low vapor pressure many ionic liquids are combustible and therefore require careful handling. [57] Brief exposure (5 to 7 seconds) to a flame torch can ignite some ILs. Complete combustion is possible for some ILs.

See also

- MDynaMix software for ionic liquids simulations^[58]
- 1-Butyl-3-methylimidazolium hexafluorophosphate (BMIM-PF₆) for an often encountered ionic liquid
- Trioctylmethylammonium bis(trifluoromethyl-sulfonyl)imide
- Aza-Baylis-Hillman reaction for the use of a chiral ionic liquid in asymmetric synthesis.

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External links

- Ionic Liquids Biological Effects Database (http://www.il-eco.uft.uni-bremen.de), free database on toxicology and ecotoxicology of ionic liquids
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