Ionic Liquids Today

Issue 2-05, Saturday, 18rd June, 2005.

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I. Editorial

By Thomas Schubert.

Since we received a very good feedback for our first Issue of Ionic Liquids Today (which was released on March, 3rd, 2005), we were encouraged to proceed. Especially our column Bösmann says 'Hot stuff!' received much reception. In this issue he will introduce a very important paper that rules offs with the presumption that ionic liquids have no vapour pressure and cannot be distilled.

As a consequence of a strong request on our services and products, we had to extent our capacities resulting in a

Our new address:

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Finally, we would like to inform that the 1st International Congress on Ionic Liquids (COIL 1) starts tomorrow at Salzburg. My colleague Tom Beyersdorff and myself will be present at all 4 days. We would like to take the chance to talk with you about Ionic Liquids Research and about latest developments of applications.

II. Bösmann says: "Hot stuff!"

By Andreas Bösmann.

On the Critical temperature, Normal boiling point , and Vapor Pressure of Ionic Liquids

L.P.N Rebelo, J.N. Canongia Lopes, J.M.S.S. Esperança, E. Filipe

J. Phys. Chem. B 2005, 6040-6043.

The Abstract: *Based on the fact that the surface tension of a liquid is zero at the critical point (liquid and gas phase become indistinguishable) and the assumption that the boiling point of a liquid T_B is a fraction of the critical temperature T_C, the critical temperatures and boiling point of some Ionic Liquids are predicted. The destillation of an Ionic Liquid is performed*.

With the release of the paper from *Rebelo et al.*, it seems to be striking that the times where Ionic Liquids were believed to have no vapor pressure are over. What might have been clear has now been demonstrated: there is no such thing as „no vapor pressure": Ionic Liquids can be distilled !

It is obvious that the suface tension of a liquid is zero at it's critical point, since liquid and gas phase become one supercritical phase and no phase boundary is left. The empirical equation

$$\gamma = \gamma^0 \cdot (1 - T/T_C)$$

predicts the surface tension for a given temperature (and of course for $T = T_C \rightarrow \gamma = 0$). It is stated that this prediction has an accuracy of better than 10%, but is problematic in cases where strong hydrogen bonding is present.

The normal boiling temperature T_B of most organic substances is $T_B = x \cdot T_C$ with x: 0.6-0.7 (anyway, ist lower than T_C), so the estimate of the critical temperature leads to an estimate of the boiling temperature. This double estimate seems to be correct within 1% for many substances.

Applied to a series of 1-alkyl-3-methylimidazolium salts, with increasing chain length a linear decrease of the boiling temperature is observed. For a C_{10}MIM-NTf_2 , the boiling temperature is estimated to be 480K. For this substance, the authors tried a distillation at 450K and 1 Pa and succeeded.

From a practical point of view, this is certainly not an exceedingly high-boiling substance; there are higher-boiling substances routinely distilled in most labs (e.g. 1-Decanol, T_B = 503K; 1-Octanol, T_B = 468K).

In summary, I would like to subscribe to the authors proposal to carry out more determinations of vapour pressure of Ionic Liquids. To know the vapour pressure and the enthalpies of vaporization would be helpful in judging the applicability of Ionic Liquids in processes where long-term stability in a gas-stream is required, e.g. as a stationary phase in GC-capillaries.

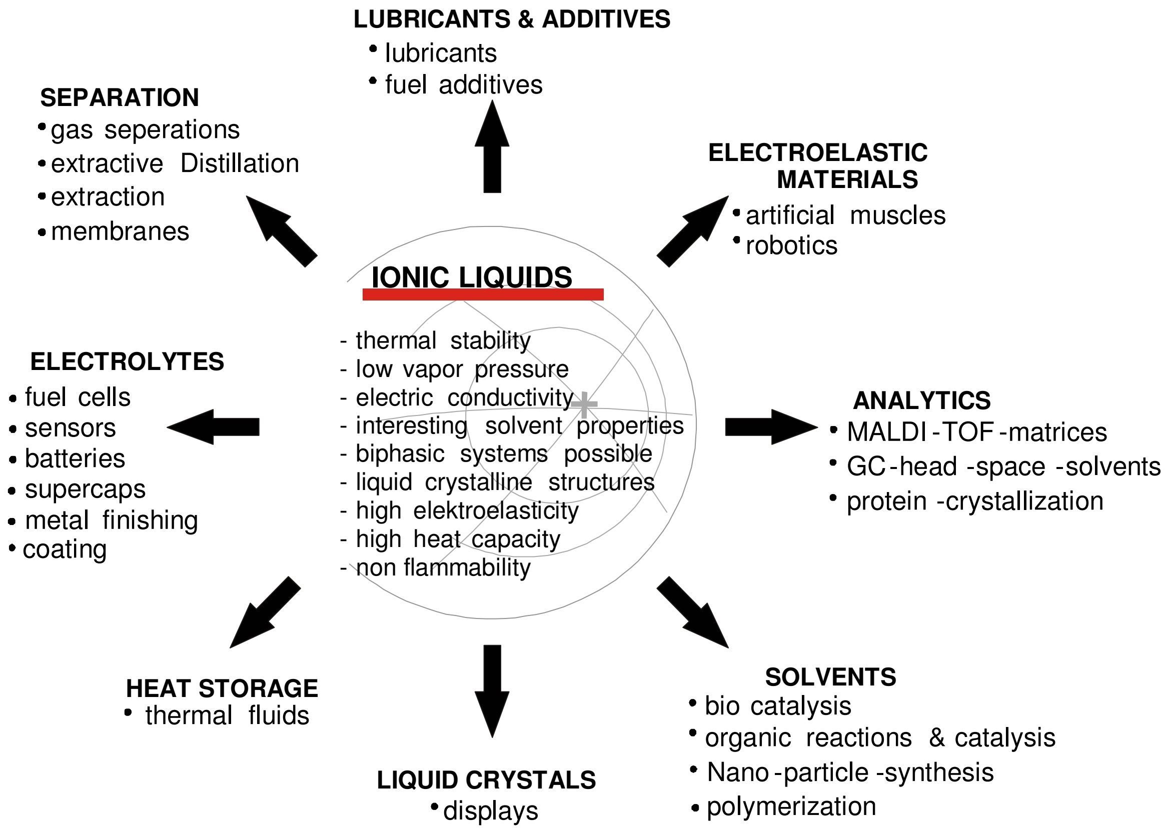
And finally: if you have spilled some Ionic Liquid, don´t hope it will be evaporated until next morning. You will still have to wipe it up !

III. IOLITEC's R&D-services: tailor made solutions for technical applications

ByThomas Schubert.

With IOLITEC's foundation in 2003, our main focus was to develop solutions for any technology that might have a benefit from implementing ionic liquids. Obviously, we define our R&D-services as our "core-business".

Derived from very interesting physical and chemical properties, some Ionic Liquids are already and many will be soon tested and implemented in a broad variety of interesting applications. A number of them are supposed to play an important role as *key technologies* in the near future.



A typical case is described as follows: a customer has heard about Ionic Liquids as a new class of interesting materials and has an idea how to use it in a potential application. After he has "googled" the term ionic liquid, he finds our company and contacts us. Together with our R&D-specialists, he develops a draft for a (potential) custom solution. Subsequently, we estimate the corresponding costs, and submit a fair quotation. Of course, we are honest enough to say, if Ionic Liquids have no advantage!

Another field of activity is the execution of projects that are supported by the government or by foundations. Recently, we applied successfully for funds from the "Bundesministerium für Bildung und Forschung" (BMBF) and from the "Deutsche Bundesstiftung Umwelt". In the first project, we develop with a multidisciplinary consortium micro-reactor-systems for the synthesis of Ionic Liquids. In the latter project, we explore together with our partners, if Ionic Liquids are suitable thermal fluids for the use in solar-thermal collectors. A couple of other projects are preparation – we'll keep you informed!

Finally, with the start of our second division "Special Chemistry" in Q3 2004, our R&D-services developed as well new materials and production techniques for our high-quality Ionic Liquids.

At our new, well-equipped labs at Denzlingen, we are open for any kind of Ionic Liquids R&D:

- development of syntheses of new, tailor-made ionic liquids from 5 g up to 5 kg
- electrochemical equipment: Metrohm Autolab (Potentiostat-/Galvanostat), FLUKE RCL-bridge
- analytics: ion chromatography, conductivity, UV-spectroscopy, viscosimetry, Karl-Fischer-Tritration and gas chromatography

If you are interested in our services, don't hesitate to contact us! Research groups from universities are invited to discuss with us (e.g. at our forum) any topic from Ionic Liquid research.

IV. Community

By Thomas Schubert.

COIL-preview

Tomorrow starts the 1st International Congress on Ionic Liquids (Sunday, June 19th), or, more precisely, "COIL 1" at Salzburg (Austria). IOLITEC believes that COIL will have a large impact on further developments based on Ionic Liquids in the near future. That's we would be glad, if we can invite you talk and discuss with us e.g. at the poster sessions, where we have placed four poster:

C Applications in synthesis/catalysis/biocatalysis

C24 Novel micro-reactor-systems for the innovative synthesis of ionic liquids (NEMESIS).

E Electrochemistry

E27 Electrochemical stability of selected ionic liquids – dependencies on electrode materials, additives and impurities

F Analytics

F4 Identification of new applications of ionic liquids: cleaning additives & chemical sensors

F5 Ion chromatography – a powerful tool for the quality control of ionic liquids

We will forward you a special issue with the most interesting talks, posters and news from the Ionic Liquid Community.

V. Interesting New Materials

By Tom Beyersdorff.

Our IOLI*TAG*-Technology

With this second issue of *Ionic Liquids Today* I would like to introduce a new technology of functionalized ionic liquids, claimed by our new trade mark **IoLi*Tag*** (**Io**nic **Li**quids **Tag**).
We think that, as the name might imply, functionalized Ionic Liquids can be used as ionic markers or anchor-groups for different applications such as peptide synthesis or combinatorial chemistry. They combine the "classic" advantages of ionic liquids (liquid at low temperatures, negligible vapour pressure…) with the advantages of solid-phase-synthesis. As a consequence, as a new type of materials they are a true alternative to common, established materials for solid-phase-synthesis.

Advantages of our **IoLi*Tag*-technology:**

1. ***Biphasic reaction with Organic Solvents/Water***
   Ionic Liquids show a well defined miscibility with many (but not all!) organic solvents and they can be designed to be immiscible with water. Therefore, by-products and impurities can easily be removed by clever choice of suitable solvent-systems or by washing the Ionic Liquid-phase with water.

2. ***Homogeneous reaction***
   Reactions can be carried out in just one liquid, homogeneous phase. Thus reactions will be in most cases be faster compared to solid-support-synthesis.

3. ***Enhanced Loading capacity***
   The loading capacities (about 4 mmol/g) and reaction rates are much higher, if they're compared with typical polymer-supported linker-systems, like Wang-Resins, Merryfield-Resins or Tentagels

4. ***Monitoring***
   The reaction progress can be monitored easily with standard spectroscopic methods such as ^{1}H-NMR, ^{13}C-NMR, TLC or Mass-Spectroscopy.

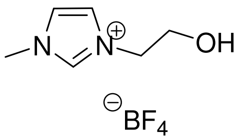
5. ***Compatibility***
   Ionic Liquids are compatible with many organic reactions.

6. ***Wide field of applications***
   The **IoLi*Tag***-*technology can be used to mark all kinds of molecules for special applications such as MALDI-TOF, electrophoresis or ionselective membranes.*

In my opinion, these striking advantages demonstrate that the **IoLi*Tag***-technology is well prepared to find a wide scope of interesting applications in the near future – I'll keep you informed!

New Materials and Examples for Applications

1-(2-Hydroxyethyl)-3-methyl-imidazolium tetrafluoroborate: [hydEMIM] BF_4



This hydroxyl-functionalized Ionic Liquid was first described by *Bazureau et. al.* in 2001 as a linker in small molecule synthesis.[1] They used this Ionic Liquid exemplarily for benzaldehyde derivatives which smoothly underwent Knoevenagel reactions and after aldimine formation also 1,3-dipolar cycloadditions.

In 2005 [hydEMIM] BF_4 was successfully used as tag for the "liquid supported" synthesis of the pentapeptide Leu^5-enkephalin by *Chan et. al.*[2] They synthesised the pentapeptide in the reaction combination of loading the support, deprotection of the linked aminoacid, iterative coupling/deprotection-sequences and final cleavange from the ionic support. All intermediates were isolated and purified by sequential washing with organic solvents or water. The overall yield after 11 steps was 50% and a HPLC-confirmed purity of >90% was achieved which is superior to what is usually obtained by solid-phase peptide synthesis prior to chromatographic purification.

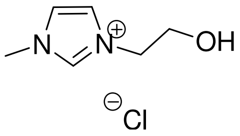
These two examples show the great potential of [hydEMIM] BF_4 as a **IoLi*Tag***.
The loading capacity is in the range of 4.5 mmol/g.

[1] J. Fraga-Dubreuil, J. P. Bazureau, *Tetrahedron Lett.* **2001**, *42*, 6097-6100.
[2] W. Miao, T.-H. Chan, *J. Org. Chem.* **2005**, *70*, 3251-3255.

My special offer*:

[hydEMIM] BF_4:	**IL-038-10g**	**60,00 €**
	IL-038-25g	**135,00 €**
	IL-038-50g	**240,00 €**
	IL-038-100g	**380,00 €**

1-(2-Hydroxyethyl)-3-methyl-imidazolium chloride: [hydEMIM] Cl



This imidazolium chloride is a useful precursor for many other Ionic Liquids such as [hydEMIM] BF_4 (see above).
The theoretical loading capacity of [hydEMIM] Cl is 6.2 mmol/g.

My special offer*:

[hydEMIM] Cl:	**IL-039-10g**	**50,00 €**
	IL-039-25g	**115,00 €**
	IL-039-50g	**210,00 €**
	IL-039-100g	**365,00 €**

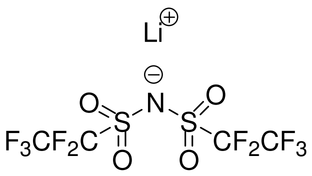
Finally, we'll extent this group of materials in the near future. Furthermore, in co-operation with our academic network and by our own research, we'll extent the number of examples of applications continuously.

For discussion, please visit me also at our ionic liquids forum (www.iolitec.de/forum/).

Key intermediates:

New highly fluorinated compound

Lithium-bis-(pentafluoroethylsulfonyl)imide: Li BETI



This highly fluorinated lithium salt can be used as electrolyte in Li-batteries e.g. or as a precursor for highly hydrophobic and electrochemically stable Ionic Liquids.

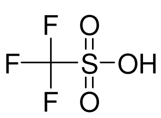
My special offer*:

Li BETI:	**KI-016-25g**	**250,00€**
	KI-016-50g	**300,00€**
	KI-016-100g	**450,00€**

Triflic-Acid derivatives

Due to their thermal stability, which is far superior to that of other acids, and their resistance to oxidation and reduction reactions, these materials are particularly valuable reactants in many fields of organic chemistry.

Trifluoromethanesulfonic acid (Triflic acid): TfOH

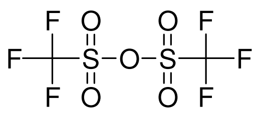


Trifluoromethanesulfonic acid Triflic acid is one of the strongest proton organic acids due to powerful electronic activity of fluorine ions. It is used in many applications such as oligomerisation-polymerisation catalyst in the plastic industry, for the production of electrically conductive polymers, protonisation catalyst in the fuel industry or for special reactions in technology and in the laboratory.

My special offer*:

TfOH:	**KI-008-100g**	**100,00 €**
	KI-016-250g	**225,00 €**
	KI-016-500g	**450,00 €**

Trifluoromethanesulfonic acid anhydride (Triflic anhydride): Tf_2O

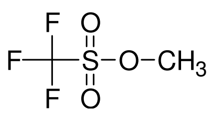


Triflic anhydride is a widely used reagent in organic synthesis and is for example used for the protection of amines or the transformation of ketones to enol triflates.

My special offer*:

Tf_2O:	**KI-014-100g**	**125,00 €**
	KI-014-250g	**280,00 €**
	KI-014-500g	**560,00 €**

Trifluoromethanesulfonic acid methylester (Methyl triflate): TfOMe



Methyl triflate can be used as a very powerful methylating reagent for example in the synthesis of Ionic Liquids.

My special offer*:

TfOMe:	**KI-002-100g**	**190,00 €**
	KI-002-250g	**430,00 €**
	KI-002-500g	**850,00 €**

*All offers are valid until August 31st, 2005.

Impressum

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