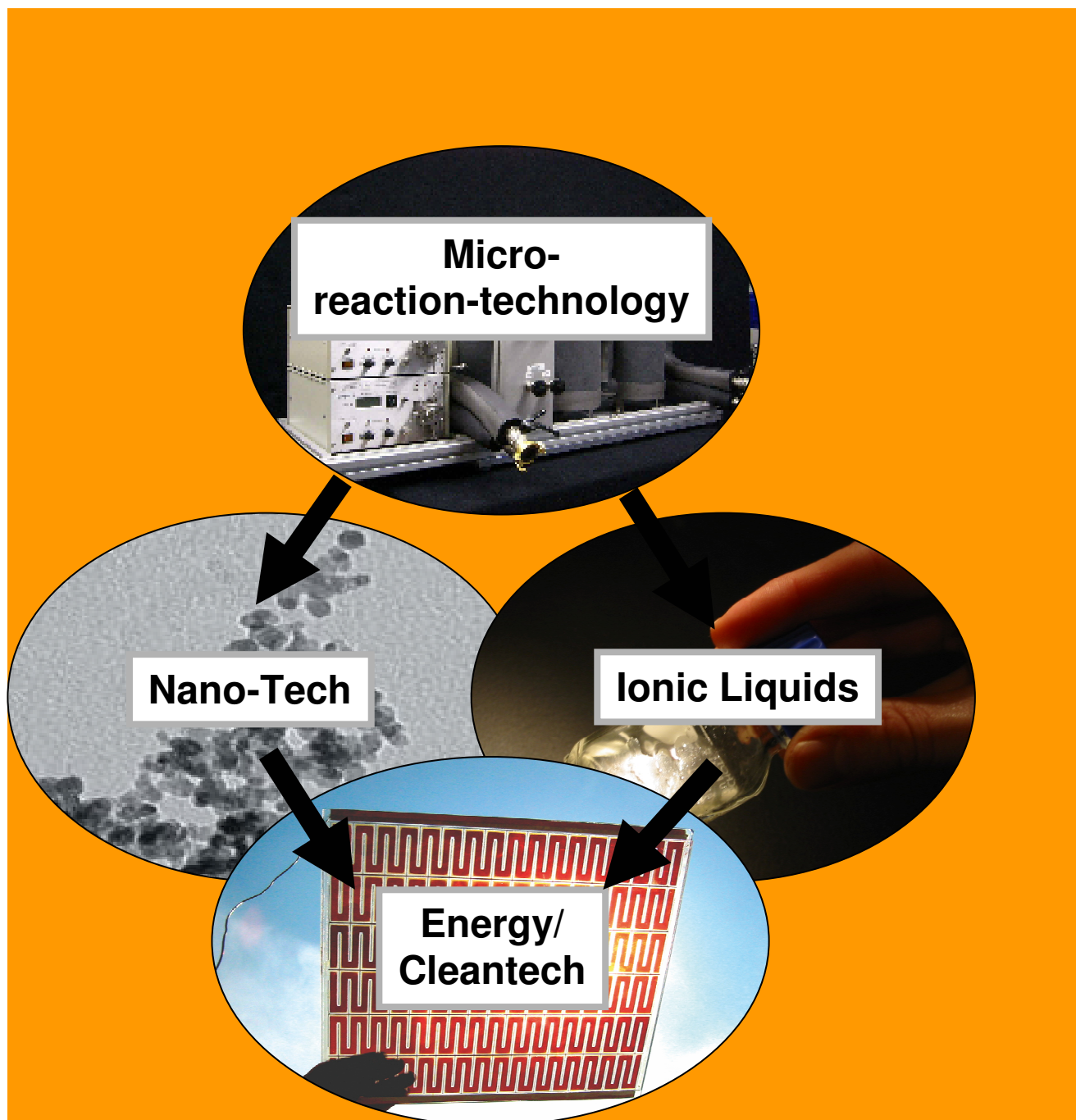


Ionic Liquids Today

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>>> **Ionic Liquids meet Nano-Materials**

>>> **Energy/Cleantech Applications**

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I. Editorial

By Thomas Schubert.

The first issue of this year was originally planned to be released in April of this year. An overwhelming number of requests has led to a delay, which we would like to kindly apologize for. Hopefully, no one is delighted not to receive this issue.

In this issue, we'd like to explain our point of view concerning what ionic liquids and nano-technology might have in common. Furthermore, the potential impact of both technologies on future energy and cleantech applications will be discussed.

Finally, I'd like to share a photo of my favourite picture "Composition with Red, Blue and Yellow" by the Dutch artist Piet Mondrian with you. I'm sure that Mondrian had in mind future market shares of ionic liquid selling companies!





II Ionic Liquids and Nano-Technology – A useful symbiosis?

By Thomas Schubert.

What have ionic liquids in common with nano-technology? Are ionic liquids not just a new kind of very expensive, innovative solvent for organic synthesis and catalysis?

Inspired from Feynman's famous "There's plenty of room at the bottom" talk in the 1950s, nano-technology is today reported to be *the* key-technology of the 21st century. Nano-science is predicted to have a strong impact on R&D-areas as physics, chemistry or biology, but also on applied sciences like materials science, biotechnology or all types of engineering. Many results have already led to a number of inventions and innovations in different fields of technology. And this is surely just the beginning.

In contrast, ionic liquids are still not well-known to the wider public. Even for many chemists ionic liquids are still not more than a new kind of new solvent. Interested readers of "Ionic Liquids Today" are, of course, better informed.

The intention of this article is to demonstrate that it is not just mixing of scientific buzz words. In this issue I'd like to concentrate on the synthesis of nano-particles. The electrodeposition of metals from ionic liquids also often leads to nano-sized deposits, but in this article I'd like to concentrate on normal synthesis.

Synthesis of Nano-particles

Ionic liquids (IL) are in general interesting materials for inorganic synthesis, but for the synthesis of nano-sized materials, in particular. It is known that coordinating solvents or stabilizing agents (e.g. alkylphosphines and -amines, polyethylenglycols etc.) are often necessary for the control of size, the control of the degree of agglomeration and the shielding of particle surfaces.

Many ionic liquids consist of intrinsic charged, polarizable, non- or at least weakly coordinating ions. As stabilizing agents, they are able to stabilize nano-particles electrostatically and sterically. After the synthesis procedure the removal is often easier compared with conventional solvents, because of the weakly-coordinating



character. Furthermore, the preorganized structure of IL can be used as template for the synthesis of porous inorganic nano-materials.

It is the mix of properties making ionic liquids suitable solvents for nano-particle synthesis. Ionic liquids...

- ...are thermally stable
- ...allow inorganic syntheses with polar starting materials in a water-poor environment
- ...are polar solvents with comparable low surface tensions; a low surface tension causes high rates of nucleation, leading to small particles.

In the following I'd like to give you some examples of recent publications. This is just a brief overview and surely an incomplete summary of current research activities in this interesting field.

Supramolecular Solvents

Antonietti et al. highlighted that one of the most interesting advantages of ionic liquids are to form H-bonds in the liquid phase with a distinct structure. As a consequence, ionic liquids were described by the authors as "supramolecular solvents": The structuring of ionic liquids is the molecular basis of molecular recognition and self-organization processes.¹

Sol-Gel-Synthesis of Small TiO₂ Anatas-nano-particles

Zhou and Antonietti used the advantages of ionic liquids in sol-gel-synthesis of TiO₂ Anatas-nano-particles. Just by simple hydrolysis of TiCl₄ in 1-butyl-3-methylimidazolium tetrafluoroborate the authors were in the position to yield 2-3 nm large particles.²

¹ M. Antonietti, D. Kuang, B. Smarsly, Y. Zhou, *Angew. Chem.* **2004**, *116*, 5096.

² Y. Zhou, M. Antonietti, *J. Am. Chem. Soc.* **2003**, *125*, 14960.

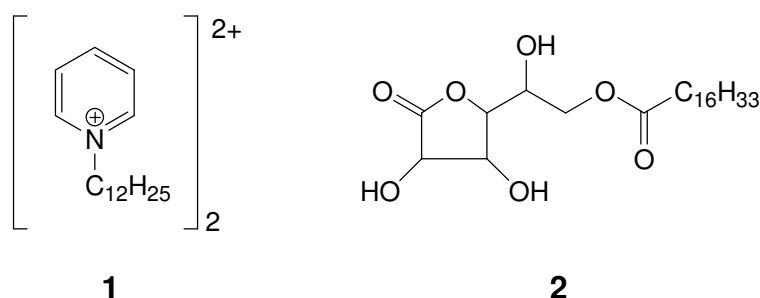


Microwave Assisted Synthesis of Luminescent Nano-Particles

Feldmann and *Bühler* used tributyl-methyl-ammonium bis(trifluoromethyl-sulfonyl)imide together with a co-solvent for the microwave-assisted synthesis of luminescent $\text{LaPO}_4:\text{Ce,Tb}$ nano-particles. These core-shell semiconducting quantum-dots have quantum yields above 50%, which today represents the state of the art. The advantage of using ionic liquids is that due to their negligible vapour pressure the reaction can be carried out at the necessary temperature from about 150-200°C by using microwaves, allowing a controlled energy insertion to the system.³

Synthesis of CuCl nanoplatelets

Taubert observed the formation of CuCl nanoplatelets, which precipitated from a mixture of the ionic liquid **1** and 6-O-palmitoyl ascorbic acid **2** at 85°C.



At higher temperatures smaller platelets were obtained.⁴

Synthesis of Gold-Nano-Particles using γ -irradiation

Wu et al. used $[\text{Me}_3\text{EtNOH}]^+[\text{Zn}_2\text{Cl}_5]^-$ together with HAuCl_4 to yield gold-nano-particles. Depending on the co-solvent, they obtained different sizes: With the neat ionic liquid, the average particle-size was 12 nm, with 5% isopropanol it was 35 nm

³ C. Feldmann, G. Bühler, *Angew. Chem.* **2006**, *118*, 4982.

⁴ A. Taubert, *Angew. Chem. Int. Ed.* **2004**, *43*, 5380.



and finally with 49% water it was 50 nm.⁵ They also mentioned, that with imidazolium-based ionic liquids, typically smaller sizes were obtained.

Synthesis of Bi₂S₃ Nano-Flowers

From 1-butyl-3-methyl-imidazolium tetrafluoroborate *Yu et al.* were able to crystallize Bi₂S₃ nano-structures at low temperatures and ambient atmosphere. The Bi₂S₃ “nano-flowers” had a size 3-5 μm, and were composed of nanowires with a diameter of 60-80 nm.⁶

Summary and Outlook

As mentioned above, these examples are just a small tale of the whole story. They show that nano-chemistry can at least be supplemented in a useful and advantageous way by using ionic liquids, because they can make new materials possible. Furthermore, they give chemists a tool-box to influence size, size-distribution, structure and shape of nano-sized compounds. Thus, making them ideal solvents for any kind of bottom-up-approach.

In the upcoming next two issues, I'll write about nano-particles-ionic liquids composites (issue 02/07) and the dispersion of nano-particles using ionic liquids (issue 03/07).

⁵ S. Chen, Y. Liu, G. Wu, *Nanotechnology* **2005**, *16*, 2360

⁶ J. Jiang, S.-H. Yu*, W.-T. Yao, H. Ge, G.-Z. Zhang, *Chem. Mater.* **2005**, *17*, 6094.



III Ionic liquids in energy and cleantech applications

by Marco Klingele.

Introduction

In times of an ever-growing demand for energy and raw materials, sustainability becomes a major issue. The efficient use of the remaining conventional energy sources, and whenever possible their supplementation or even replacement with alternative and/or renewable power sources, is increasingly important. As a consequence, all strategies providing a solution to the world's energy problem, be it for the conservation, generation, storage or transport of energy, will not only have a positive environmental impact but also strong economic significance.

IoLiTec's interdisciplinary Energy & Cleantech division is developing novel applications based on the unique chemical and physical properties of ionic liquids (i.e. negligible vapour pressure, thermal stability, electric conductivity, electrochemical stability, good heat storage capacity), thus generating both technological and ecological advantages. The following are just a few examples for the employment of ionic liquids in modern energy and cleantech applications.

Ionic liquids as sorption cooling media

An alternative concept for the generation of low temperatures to the widely used compression refrigeration technology are sorption refrigeration cycles which were discovered earlier than the compression refrigeration machines but have long been practically displaced by them. A first renaissance of the sorption refrigeration technology occurred in the early 1990s and was stirred by the uncertainties regarding the use of halogenated hydrocarbons and their impact on the ozone layer, and also by the increasing use of combined heat and power cycles. In contrast to compression refrigeration machines, sorption refrigeration cycles do not consume high-value electrical energy but are driven by heat. Consequently, this technology is ideally put to use where there is excess heat available, e.g. in the form of industrial waste heat or district heat. A particularly attractive area of use derives from the combination of solar thermal power and sorption refrigeration. In the warm and



sunny regions of the world natural heat can be used to generate low temperatures for the air-conditioning of buildings. This concept is generally referred to as solar cooling.

In sorption refrigeration machines, low temperatures are generated by the evaporation of a volatile compound (working medium) which is first adsorbed or absorbed by another carrier material (sorption medium). In a cyclic process the absorption medium is regenerated by removing the absorbed working medium and the functionality of the machine is re-established. One of the most widely used working systems, lithium bromide and water, has the specific problem of solidification. In order for the regeneration process to work, the working system has to be liquid at all times since it is transported through the machine by pump. If the lithium bromide crystallises, malfunctions can occur that make costly repairs necessary.

IoLiTec has realised that mixtures of a working medium like water or ammonia and an ionic liquid as the sorption medium open up new possibilities in the absorption refrigeration technology which could ultimately allow a breakthrough in this technology. Using thermal energy, the sorption medium can easily be regenerated with the major advantage that the carrier material itself is a liquid which has no vapour pressure and which is not prone to crystallisation. The first patent application in this field, filed by IOLITEC in 2004 (now owned by BASF AG)¹. In the meantime, other major companies like DuPont² and Degussa³ have also filed patent applications on the use of ionic liquids in sorption cycles. This confirms the great potential of this idea, which IoLiTec is developing further. The widespread use of this technology in combination with natural or renewable power sources or usable industrial and other waste heat could contribute significantly to the conservation of fossil fuels and thus to lower emissions of green house gases.

¹ See patent: [WO2005/113702].

² See patent: [WO2006/084262].

³ See patent: [WO2006/134015].



Ionic liquids as phase change materials (PCMs)

During the transition from the liquid to the solid state PCMs are able to deliver thermal energy, which they absorb earlier in the process of melting. Probably the most prominent example that uses this principle are heat packs or hand warmers which consist of a bag filled with a supersaturated salt solution. On squeezing the bag, the liquid inside starts to solidify, giving off heat in the process of crystallisation. By placing the bag in a pot with boiling water the solid liquifies and the heat pack can be used again.

Most current technical applications, e.g. PCMs for buildings, employ paraffins as PCM that can be tuned to certain melting ranges. However, the use of paraffins has the major disadvantages that they are (a) petroleum products and hence the use of these materials is directly linked to the price development of crude oil and (b) paraffins

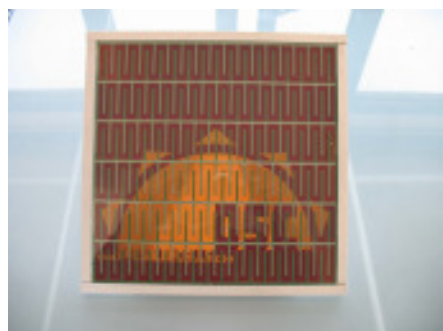


are inflammable which represents a considerable safety concern for many applications. Another less important class of PCMs are salt hydrates. The contents of the above-mentioned heat packs belong to this group. These materials offer the advantage that they are not petroleum products but they still have only a limited range of use with regard to operating temperatures compared to paraffins.

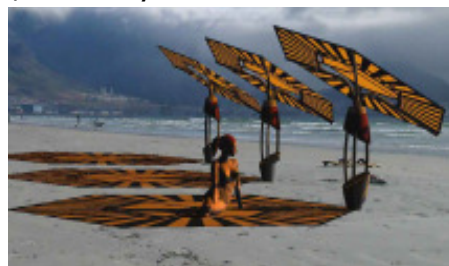
IoLiTec is currently working on the development of new PCMs based on ionic liquids, including both their utilisation in neat form as well as in the form of formulations containing further performance-enhancing additives. Such materials can be used for the cyclic storage and supply of thermal energy, as latent heat storage devices, heat buffers or cold buffers.



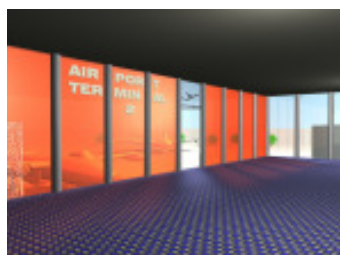
Ionic liquid-based electrolytes for dye-sensitised solar cells (DSSCs)



DSSCs or Grätzel cells are considered a real alternative to the well-established silicon-based solar cells.⁴ Utilising a dye as the key component they convert sunlight into electricity. Their working principle is closely related to photosynthesis. Compared to silicon-based solar cells they offer the major advantage of staying functional even under diffuse light. They are transparent and can therefore be used as translucent, power-generating building blocks (left: prototype, FhG ISE, middle and bottom: design-studies). To enable charge transfer within the DSSC, electrolytes are needed which have to fulfill a number of strict technical requirements, such as good electrochemical, thermal and UV stability. From a very early stage, the development of the DSSC was linked closely to the use of ionic liquids as the electrolyte, as their



favourable properties, i.e. non-volatility and tuneable conductivity and miscibility, made them the obvious materials of choice for this purpose. Over the last 15 years a huge number of systems composed of different dyes, electrolytes and additives have been published in the scientific literature, some of them reaching efficiencies of more than 10% in the laboratory.⁵ The



efficiency of DSSCs is strongly dependent on the interaction of the many different components and materials used and even with functional systems there is still room and need for further optimisation. IoLiTec is Europe's leading supplier of ionic liquids for the use in DSSCs. In collaboration with a number of top-class partners the further development and commercialisation of the DSSC in Germany is currently being driven forward by the ColorSol[®] project which is funded by the German Federal Ministry of Education, Research and Science.



⁴ B. O'Regan, M. Grätzel, *Nature* **1991**, 353, 737–740.

⁵ M. Grätzel, *J. Photochem. Photobiol. C* **2003**, 4, 145–153.



Ionic liquid-based electrolytes for lithium ion batteries

Despite a large number of products already on the market there is still a strong need for further development of the lithium ion battery. This became patently obvious only recently when the computer manufacturer Dell experienced major problems with the lithium ion batteries they used in their laptop computers. In some cases the batteries caught fire on overheating which posed a considerable safety hazard to users. Ionic liquids are potentially very interesting electrolytes for lithium ion batteries due to their outstanding properties, i.e. high electrochemical stability towards oxidation and reduction processes, high electric conductivity, wide electrochemical windows and non-flammability.⁶

Relatively simple materials like 1-butyl-3-methylimidazolium tetrafluoroborate (BMIM-BF₄),⁷ 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (EMIM-NTf₂),⁸ 1-butyl-2,3-dimethylimidazolium bis(trifluoromethylsulfonyl)imide (BDiMIM-NTf₂)⁹ and 1-methyl-1-propylpiperidinium bis(trifluoromethylsulfonyl)imide (PMPip-NTf₂)¹⁰ and others have been tested as electrolytes for lithium ion batteries with very promising results. A more recent approach is the use of functionalised ionic liquids for this purpose to enhance the lithium ion conductivity of the electrolytes.¹¹ This approach could provide suitable ionic liquids and result in their widespread use as electrolytes in lithium ion batteries.

Contact

If you have any questions or comments regarding this short overview, IoLiTec's Energy & Cleantech division or are active in one of the research areas mentioned above and are looking for a competent partner for ionic liquids, please do not hesitate to contact us (klinge@iolitec.de).

⁶ G. E. Blomgren, *J. Power Sources* **2003**, 119–121, 326–329; J. S. Lee, J. Y. Bae, H. Lee, N. D. Quan, H. S. Kim, H. Kim, *J. Ind. Eng. Chem.* **2004**, 10, 1086–1089.

⁷ [A. Chagnes, M. Diaw, B. Carré, P. Willmann, D. Lemondant, *J. Power Sources* **2005**, 45, 82–88]

⁸ [B. Garcia, S. Lavalloé, G. Perron, C. Michot, M. Armand, *Electrochim. Acta* **2004**, 49, 4583–4588]

⁹ [F. F. C. Bazito, Y. Kawano, R. M. Torresi, *Electrochim. Acta* **2007**, 52, 6427–6437]

¹⁰ [H. Sakaebe, H. Matsumoto, *Electrochem. Commun.* **2003**, 5, 594–598]

¹¹ [T. Sato, T. Maruo, S. Marukane, K. Takagi, *J. Power Sources* **2004**, 138, 253–261; M. Egashira, S. Okada, J. Yamaki, D. A. Dri, F. Bonadies, B. Scrosati, *J. Power Sources* **2004**, 138, 240–244; M. Egashira, M. Nakagawa, I. Watanabe, S. Okada, J. Yamaki, *J. Power Sources* **2005**, 146, 685–688]



IV Syntheses of Ionic Liquids in Microreactors – The NEMESIS project

By Andreas Reisinger.

“By demonstrating radical reductions in size and weight, and better heat transfer, mass transfer and other performance advantages compared to conventional systems, microreactors, microfluidic devices and micromachined components have the potential to revolutionize chemical analysis, chemical synthesis, industrial automation, plant safety, gas detection, medical and biomedical applications.”

The above citation is the introductory sentence of an ACHEMA 2006 press release entitled *"Microreaction technology – Tiny devices with tremendous possibilities"* and gets to the point of why one should think about this technology.

During the last decade, microreaction technology has found the way out of universities and research institutes and became a very versatile technology for the production of quantities from some 100 g up to several kilograms, especially in the field of fine chemicals. Due to their significantly higher surface-area-to-volume ratios compared to conventional reaction system, such micro- or submilli-meter mechanical components provide order-of-magnitude improvements in heat- and mass-transfer rates. This leads to very efficient, compact and cost-effective devices which allow to carry out chemical reactions more safely as well as with greater selectivity and conversion rates, higher yields, and improved product quality.



Another major advantage of microreactors is the possibility of “numbering up”. This means to increase the production capacity simply by running an optimized process in parallel on several systems instead of an expensive scale-up of a certain process.

Several start-up companies as well as some global players have realised these advantages and have started the development of microreactor systems, the often so-called “plant on a desk”.



Over the last two years, IoLiTec has evolved from a producer of sub-kilogram quantities to a reliable manufacturer of 5–10 kg amounts of ionic liquids for laboratory and pilot plant use. This means that IoLiTec has to deal with bigger reaction scales, greater risks and stricter safety measurements. Therefore, IoLiTec countered this foreseen trend and identified microreaction technology as a method to enhance its production capacities to more than 10 kg per day of selected materials compared to about 10 kg per week with conventional batch systems.

Unfortunately, almost all of the currently available microreactor systems are designed for liquid-liquid reactions and are limited mostly to comparably small amounts of the desired products. Thus, no commercial microreaction system with the special demands for the production of ionic liquids was available on the market at that time (and to the best of our knowledge is still not).

Therefore, in the summer of 2004, several companies, universities and research institutes (namely the **BIAS** in Bremen, the **Fraunhofer IFAM** in Bremen, **Merck KGaA** in Darmstadt, **Schulz Systemtechnik GmbH** in Visbek, the **University of Bremen – UFT** and **IOLITEC GmbH & Co. KG**) joined forces within the BMBF (German Federal Ministry of Education, Research and Science) funded project NEMESIS.

The goal of this project is the development of a microreactor system that is able to fulfill the special demands of ionic liquids production, e.g. reactions that are only moderately exothermic, often biphasic and have slow conversion rates. Now, after two years of development, experiments and tests by the consortium, a prototype of a microreactor (see figure) has been set up at IoLiTec's laboratories in Denzlingen. With this reactor system IoLiTec has been put in the position to produce several halides (e.g. BMIM-Br, BMIM-Cl, EMIM-Br) in quantities of about 10 kg per day.

IoLiTec is now testing and improving the system together with the partners at its site in Denzlingen.

Currently, the major drawback is the inability to work with gases and insoluble reagents. This constitutes an unfortunate limitation to the system so that compounds such as BMPyrr-X or EMIM-Cl are currently not accessible in this way. However, the consortium is working to overcome these deficiencies, especially the solubility problems in near future so that IoLiTec will be put in the position to produce almost



every precursor (i.e. all halides as well as alkyl sulfates) on a scale of approximately 1 t/a. This means that the time-intensive alkylation step, which today is done in a batch process, will speed up and be simplified dramatically in the future, resulting in increased production capacities and lower prices for ionic liquids.

With this technology in hand, IoLiTec is truly your partner for ionic liquids not only for lab quantities but also for the pilot plant scale as well as small production series.

V Applications of Ionic Liquids

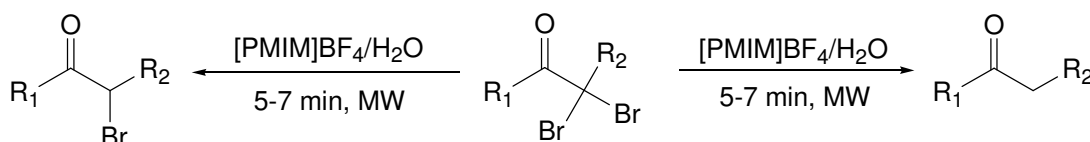
By Tom Beyersdorff, Marco Klingele & Thomas Schubert.

Organic Synthesis

Ionic Liquid promoted selective debromination of α -Bromoketones under microwave irradiation

B. C. Ranu*, K. Chattopadhyay, R. Jana, *Tetrahedron* **2007**, *63*, 155.

The authors demonstrated that gem- α -Bromoketones can be selectively debrominated to the monobromo or debrominated ketones by simple control of the time of microwave irradiation. Using a mixture of **1-methyl-3-pentyl-imidazolium tetrafluoroborate** with water, they observed the debromination of one bromine atom after 2-3 min, of two bromine atoms after 5-7 min.



Engineering Fluids

Multiplexed hydraulic valve actuation using ionic liquid filled soft channels and Braille displays



W. Gu, H. Chen, Y.-C. Tung, J.-C. Meiners, S. Takayama, *Appl. Phys. Lett.* **2007**, *90*, 03305-1.

Pneumatic actuation with multilayer soft lithography can enable the parallel operation of up to thousands valves. *Takayama et al.* presented a portable and multiplexed valve actuation strategy that uses a grid of mechanically actuated Braille pins to hydraulically, rather than pneumatically, deform elastic actuation channels that act as valves. Experimental and theoretical analyses show that the key to reliable operation of the hydraulic system is the use of nonvolatile **1-butyl-3-methyl-imidazolium tetrafluoroborate** as the hydraulic fluid.

Polymer-Science

Solubilization of Polymers by Ionic Liquids

N. Winterton, *J. Mater. Chem.* **2006**, *16*, 4281.

A very interesting feature article, which summarizes the use of ionic liquids

- as solvents for polymers,
 - polymerisation in ionic liquids,
 - polymerisation of ionic liquids,
 - polymer-ionic liquid composites.
-

Biotechnology

Application of ionic liquids in enzymic resolution by hydrolysis of cycloalkyl acetates

E. Xanthakis, M. Zarevúcka, David Sâman, M. Wimmerová, F. N. Kolisisa, Z. Wimmer,*
Tetrahedron: Asymmetry 2006, *17*, 2987.

Wimmer et al. used **1-butyl-4-methylpyridinium chloride** and **1,3-dimethylimidazolium methylsulfate** to test the performance of the three commercially available lipases (Novozyme 435, Lipozyme IM and non-immobilized powdered lipase from *Candida Antarctica*).



VI Community

Dr. Marie Mahé, joined the IOLITEC team in May 2007.

Marie Mahé, 27, performed her PhD concerning the synthesis and cracking of ceramic nanolayers within the Merck KGaA laboratories (Darmstadt, Germany). This work was carried out in collaboration with the University of Bordeaux (PhD supervisor: Prof. Jean-Marc Heintz) and the Technical University of Darmstadt. During her study at the National Graduate School of Chemistry and Physics of Bordeaux (France), she also spent 6 months at Akzo Nobel (Arnhem, The Netherlands) where she did an internship concerning the synthesis and analysis of Sag Control Agent (organic additives in clearcoats). She also spent three months in the labs of the ICMCB-CNRS (Bordeaux, France) as an intern where she carried out researches on the synthesis of metal-ceramic composites. At the beginning of May 2007, she started at IoLiTec to strengthen the team in the field of Nanotechnology/Coatings.

June 5th and 6th, Woche der Umwelt 2007, Berlin.

IOLITEC was chosen into an exclusive circle of 170 companies and institutes (420 contenders) to present its latest clean-tech developments. The event took place in the garden-park of the Schloss Bellevue, residence of the President of the Federal Republic of Germany, Mr. Horst Köhler.

In addition, BASF's ionic liquids based cellulose-technology was also chosen, so that ionic liquids were represented by two companies!

Upcoming Conferences:

Intersolar 2007, 21. – 23. June 2007, Freiburg, Germany.

Ionic liquids will surely play just a minor role at this conference. From IOLITEC's point of view it's important to know, in which way those solar-technologies will develop, where ionic liquids will have a potential overlap, like solar cooling (sorption cooling) or dye sensitised solar cells (DSCs).



COIL 2, 05. – 10. August 2007, Yokohama, Japan.

<http://www.pac.ne.jp/coil-2/>

In our opinion, the first Conference on ionic liquids (COIL 1) at Salzburg, Austria, in 2005 was a great success. That's why we see a great potential for the 2nd Conference at Yokohama. IOLITEC's Head of Special Chemistry is invited to held a talk about the **"Production of High-Purity Ionic Liquids Using Micro-Reaction Technology"**.

We invite all customers and partners to meet IOLITEC's Head of Special Chemistry, Dr. Tom Beyersdorff, who will join the conference.

Euromat, 10. – 13. September 2007, 'Nürnberg, Germany.

<http://www.euromat2007.fems.org/>

The "European Congress and Exhibition on Advanced Materials and Processes", has a special session concerning ionic liquids.

Highlighted lectures are "The Prediction of Key Physical Properties of ILs", from Ingo Krossing, University of Freiburg, and "A General Overview on the Electrodeposition in Ionic Liquids" from S. Zein El Abedin and Frank Endres, Technical University of Clausthal.

If you are interested to meet IOLITEC's Managing Director, Dr. Thomas Schubert, please contact us (info@iolitec.de).

Intertech Pira's Conference on Ionic Liquids, 17. – 18. October, Prag, Czech Republic.

IOLITEC's Managing Director is invited to held a talk concerning **"Selected aspects of ionic liquid applications and commercialisation"**. We're looking forward to discuss about new trends and applications on this occasion.

If you are interested to meet IOLITEC's Managing Director, Dr. Thomas Schubert, please contact us (info@iolitec.de)!



Nano Solutions 2007, 21. – 23. November 2007, Frankfurt, Germany.

<http://www.nanosolutions-frankfurt.de/>

„Europe’s leading Event for Nano Business“, the Organizers say.

Announcement (Germany, Switzerland & Austria only):

**In Kooperation mit Deutsche Metrohm plant IOLITEC für den
10. Juni 2008
das Schwerpunktseminar für Analytiker und Anwender**

Ionische Flüssigkeiten – Anwendungen und Analytik

Hauptbestandteile, ionische Verunreinigungen, Wassergehalt, Physikalische Parameter wie Dichte, Viskosität etc.

Die Teilnehmerzahl ist auf 35 Personen begrenzt.

Interessenten wir gerne vorab weitere Informationen zur Verfügung
(info@iolitec.de!).

Anmeldung an Frau E. Bulat
Deutsche METROHM GmbH & Co. KG
In den Birken 3
70794 Filderstadt
Tel. (0711) 7 70 88 – 90
Fax (0711) 7 70 88 – 55
E-mail: e.bulat@metrohm.de



VII Innovation in Packing Materials

By Tom Beyersdorff.

The purity of ionic liquids is a crucial point for potential applications of ionic liquids since physical properties such as viscosity, conductivity, density, surface tension etc. of these materials are influenced dramatically by impurities.

Typical impurities in ionic liquids are volatile compounds such as unreacted organic starting materials from the quarternisation reaction or other salts and water from the metathesis reaction.

In order to avoid impurities, it is generally recommended to purify all starting materials to the highest standards prior to use. In many cases organic halide salts can be recrystallized from organic solvents to remove unreacted starting materials. At this stage simple analytical methods such as NMR or HPLC can be applied to determine the purity of the ionic liquid. For the preparation of non-halide ionic liquids a subsequent anion metathesis reaction is necessary to afford the desired ionic liquid. This reaction can often be carried out in aqueous phase if the product is immiscible with water, in an organic solvent if the product is soluble in water or by using an ion exchange resin or electro dialysis.

However, in all cases one problem remains the same: since the non-halide ionic liquid is prepared from two different salts, contamination by unwanted ionic species is possible. The only reliable way to prove the absence of such ionic impurities is ion chromatography, which can be applied for anions as well as for cations and even for amines after protonation. Unfortunately, this technique is still not applied as a standard means of controlling the purity of ionic liquids and many publications still only rely on NMR data as the only purity argument.

It seems to be obvious from the above that many impurities can be removed by careful purification of the starting materials, purification of intermediates, e.g. by recrystallization, washing of the final products and drying the products under vacuum. If all these steps are performed with the necessary accuracy, the product has a defined purity that should not change upon storage or delivery.

However, ionic liquids are still salts and salts tend to be hygroscopic, which means that they take up water to an equilibrium depending on the nature of the salt and



the relative humidity. For this reason the packing material is very important to guarantee the quality of ionic liquids.

At IoLiTec we have introduced new packing materials for our products. All liquid products will be packed either in glass bottles with teflon-coated rubber septum for small quantities up to 50 g (Fig. 1) or glass bottles with teflon coated screwtops for larger quantities from 100 g up to 2.5 kg (Fig 2).



Figure 1: Packing materials for small quantities.



Figure 2. Packing materials for larger quantities



In addition all bottles will be shipped in a sealed aluminium composite film bag that allows only a water diffusion of less than 0,04 g/m²d (40°C/90% rF). Furthermore silica drying pads are added into the aluminium bag (Fig. 3)



Figure 3. Aluminium composite bags and silica drying pads used by IoLiTec

All these changes have been implemented to guarantee the highest quality standards of our ionic liquids.

While we have made a big step forward in quality management, we won't stop here. We will continue to strive for perfection and further improve our quality standards to exceed our customers' expectations!

VIII IOLITEC: Your supplier of imidazolium-based NTf₂ salts!

Many applications for ionic liquids based on the bis(trifluoromethylsulfonyl)imide anion (NTf₂⁻) have been described in the past years. In fact most applications are targeted on the use as electrolytes in electrochemical applications such as battery electrolytes or electrodeposition of metals, since most of these materials are electrochemically and thermally very stable. However, other applications have been described in literature as well. A major problem has been the poor availability of imidazolium-based NTf₂ salts.



We are proud to announce that we have obtained a license for patent EP0718288 from Hydro-Quebec. Starting on May 1st, 2007, in addition to ammonium, pyrrolidinium, piperidinium and sulfonium NTf₂ ionic liquids, **IoLiTec is able to supply imidazolium NTf₂ salts** to our customers in the well known **IoLiLyte[®]** quality (**see the attached product flyer**).

Special starting offers for the most common materials are valid until August 31st, 2007. If you require variations of the listed products, i.e. alkyl chain homologues, don't hesitate to contact us for a quotation at info@iolitec.de.

IX Special Offers* from our Portfolio:

Temporary price reductions for the following products:

In case that you have not yet received our new extended catalogue with almost 100 ionic liquids and useful precursors, don't hesitate to contact us (info@iolitec.de).

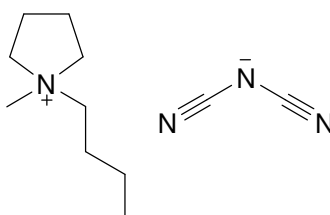
Germany only:

IOLITEC supports strategic research in the DFG-funded program SPP1191: 10%-discount for all participants!

With the beginning of the program every participant of the DFG-program SPP 1191 receives a discount of 10% on our ionic liquids and key intermediates (except our special offers).



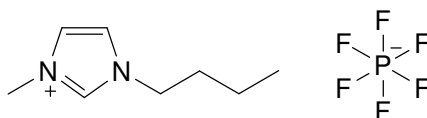
1-Butyl-1-methyl-pyrrolidinium dicyanamide: BMPyrr N(CN)₂



BMPyrr N(CN)₂ (>98%):

IL-0041-HP-25g	112,50 €
IL-0041-HP-50 g	180,00 €
IL-0041-HP-100 g	324,00 €
IL-0041-HP-250 g	733,50 €
IL-0041-HP-500 g	1.314,00 €
IL-0041-HP-1 kg	2.362,50 €

1-Butyl-3-methylimidazolium hexafluorophosphate: BMIM PF₆



BMIM PF₆ (99%):

IL-0011-HP-50 g	55,25 €
IL-0011-HP-100 g	89,25 €
IL-0011-HP-250 g	178,50 €
IL-0011-HP-500 g	293,25 €
IL-0011-HP-1 kg	527,00 €
IL-0011-HP-5 kg	2.184,50 €



Extension of availability of triflic acid derivatives

IoLiTec expands the availability of triflic acid derivatives.

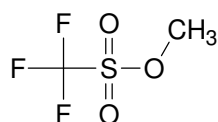
We are now able to supply materials such as

- **triflic acid,**
- **triflic anhydride,**
- **methyl triflate and**
- **lithium bis(trifluoromethylsulfonyl)imide**

in quantities of **up to 100 kg.**

If even larger quantities are required, we are able to supply these materials in co-operation with our partners.

Methyl triflate: MeOTf



Methyl triflate is one of the most potent methylation reagents. It is 10^4 times more reactive than iodomethane or dimethylsulfate. Besides many other areas of application in synthesis, methyltriflate can be used in the halide free synthesis of ionic liquids, as it reacts smoothly with many aromatic and non-aromatic amines, sulfides, and phosphines. We are able to supply this product on a multi-kilogram scale.

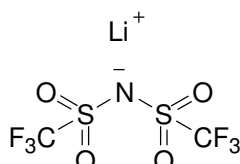


Our special offer*:

MeOTf (>98%):

KI-0002-1 kg	700,00 €
KI-0002-2,5 kg	1.650,00 €
KI-0002-5 kg	3.100,00 €
KI-0002-10 kg	5.975,00 €
KI-0002-25 kg	on request
KI-0002-50 kg	on request

Lithium bis(trifluoromethylsulfonyl)imide: **Li NTf₂**



Li NTf₂ is a widely used precursor for NTf₂-based ionic liquids, which are characterized in most cases by their high thermal and electrochemical stability combined with relatively high conductivity.

Our special offer:*

Li NTf₂ (>99%):

KI-001-100 g	130,00 €
KI-001-250 g	220,00 €
KI-001-500 g	400,00 €
KI-001-1 kg	650,00 €
KI-001-2,5 kg	1.460,00 €
KI-001-5 kg	2.450,00 €
KI-001-10 k	4.250,00 €

*** All special offers are valid until August 31st, 2007.**

All prices are FOB Denzlingen, costs for shipping and handling and custom charges are not included in the prices and are payable by customer.



Impressum

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Contact:

IoLiTec

Ionic Liquids Technologies GmbH & Co. KG

Ferdinand-Porsche-Strasse 5/1

D-79211 Denzlingen, Germany

phone: +49 (0) 7666 913929

fax: +49 (0) 7666 9129345

info@iolitec.de

www.iolitec.com

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