

Qualitative and quantitative analysis of fluoride based ionic liquids by ion chromatography

by Susann Wegner

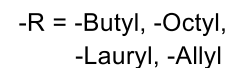
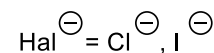
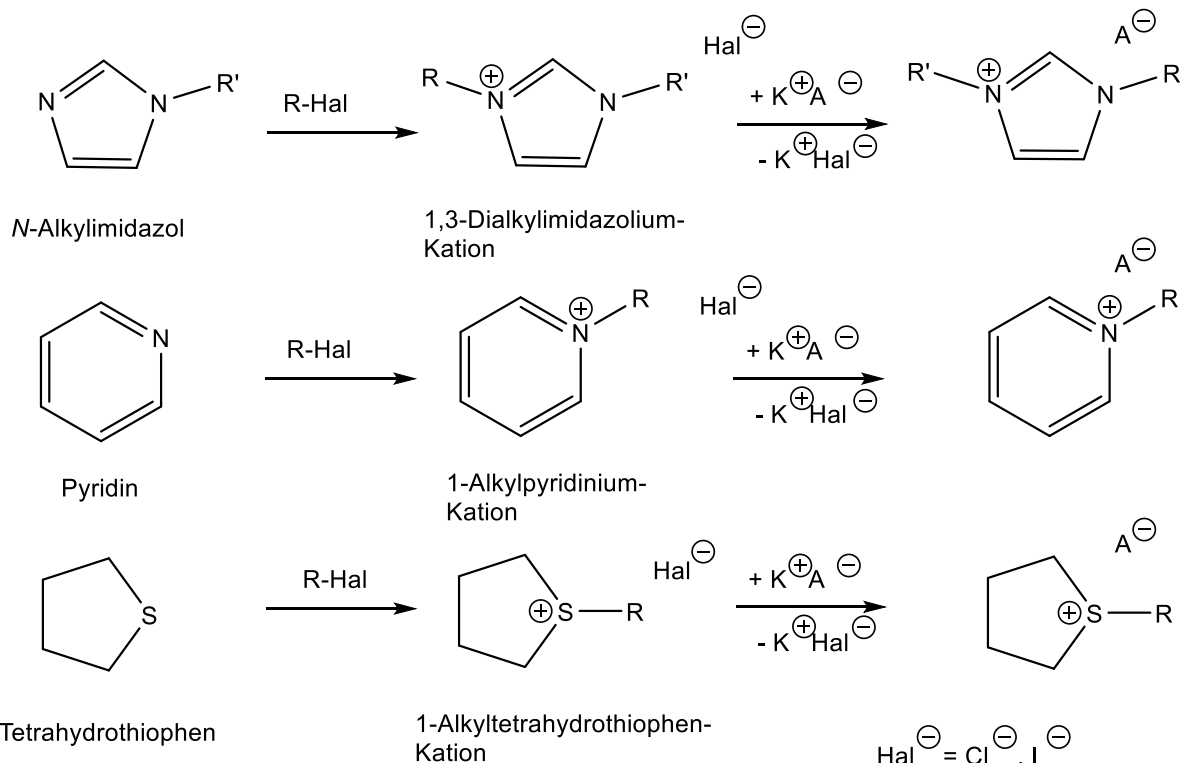
Heinrich Heine – Universität Düsseldorf



Overview:

- Ion chromatography
cost – effective , rapid and simultaneous way to other analytical methods
- Focus
tetrafluoroborate anions, hexafluorophosphate anions and small anions such chloride, fluoride, iodide and bromide

Synthesis of ionic liquids:



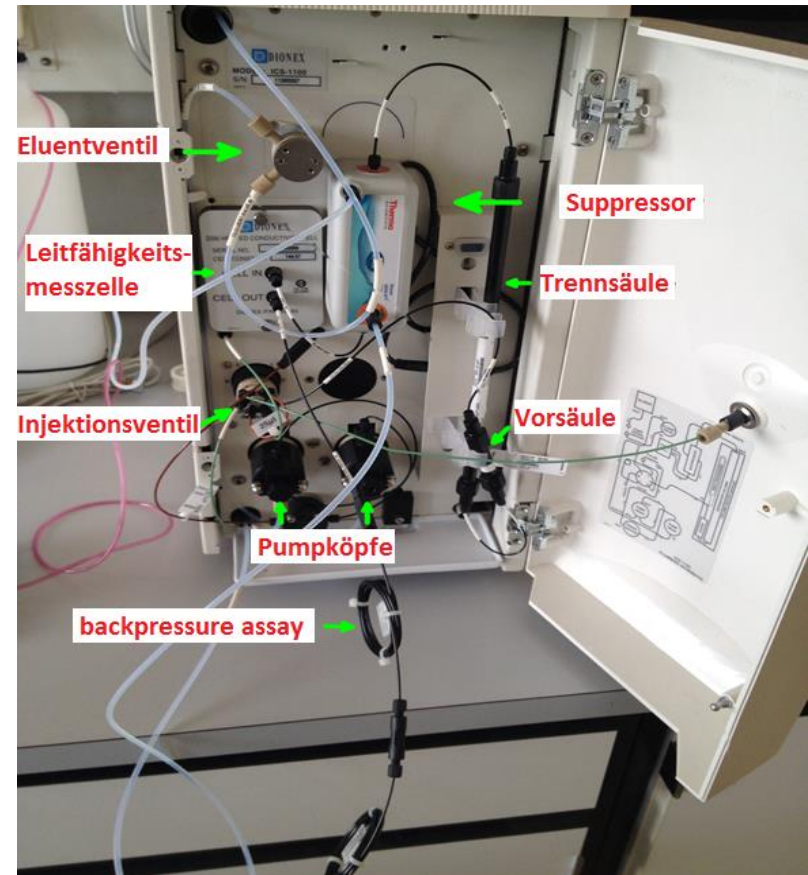
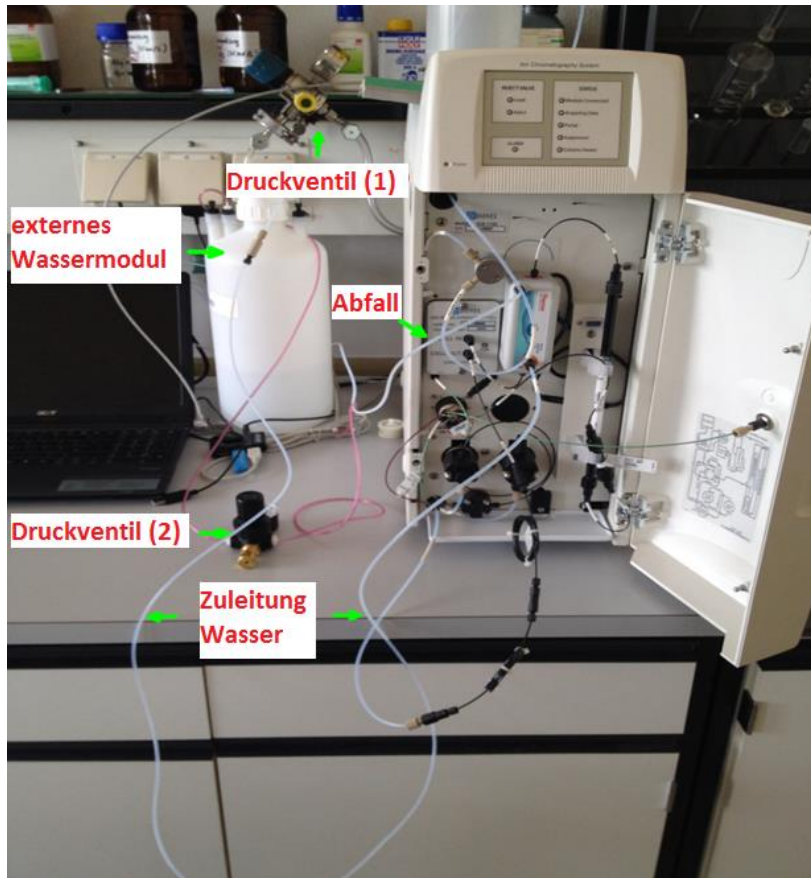
[2] D. Zhao, Z. Fei, J. Geldbach, R. Scopelliti, P. J. Dyson, *J. Am. Chem. Soc.* **2004**, 126, 15876–15882.

[3] J. Dupont, C. S. Consorti, P. A. Suarez, R. F. de Souza, F. Roberto, *Org. Synth.* **2002**, 79, 236–243.

[4] P. Suarez, J. Dullius, S. Einloft, R. F. de Souza, J. Dupont, *Polyhedron* **1996**, 15, 1217–1219.

Instrument: Dionex ICS-1100

Thermo Fisher

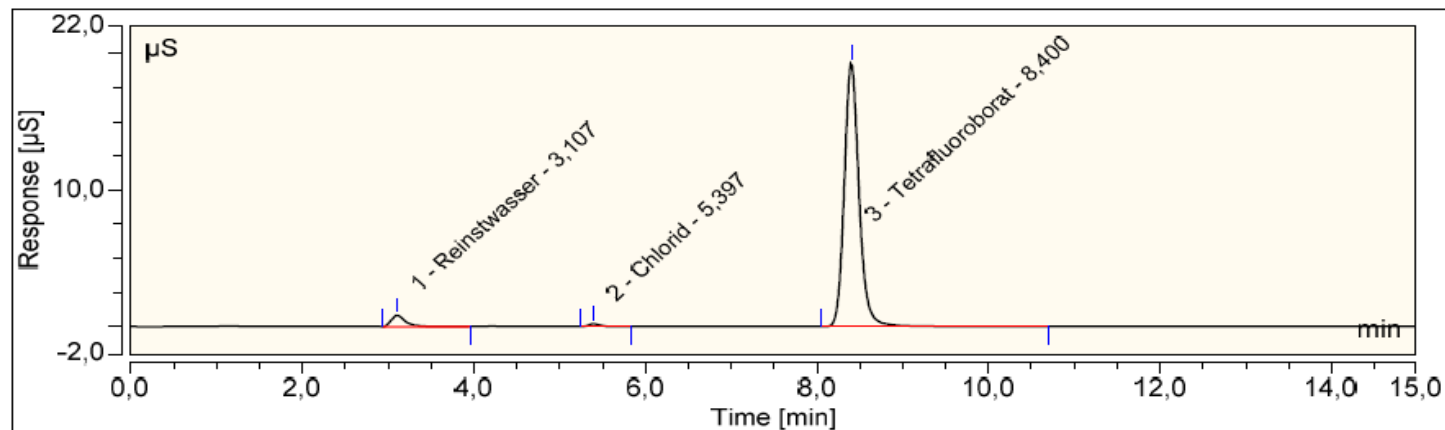


Results:

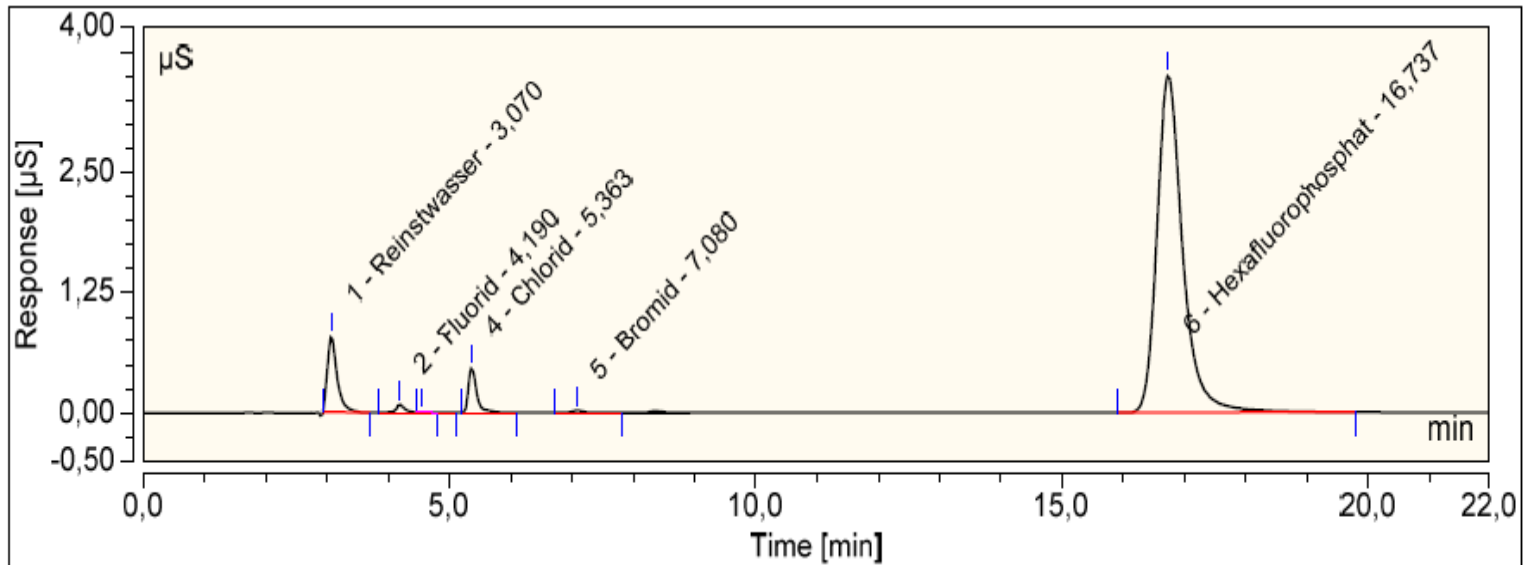
- Test measurement

Sodium carbonate-/sodium hydrogen carbonate
eluent

Potassium tetrafluoroborate:



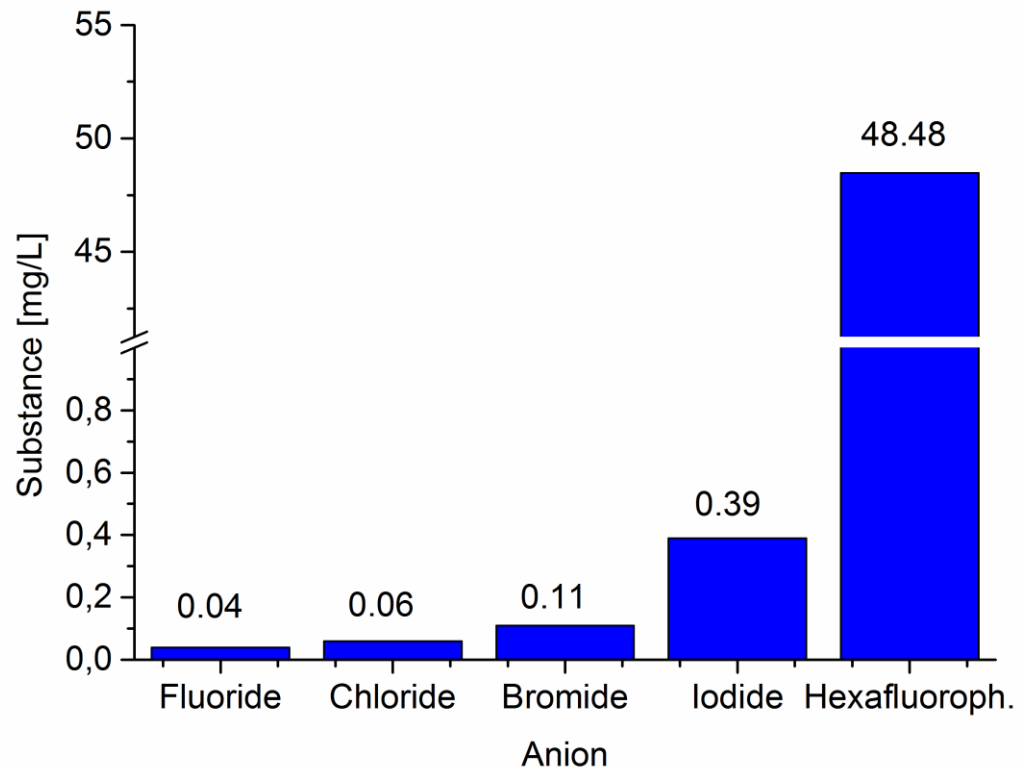
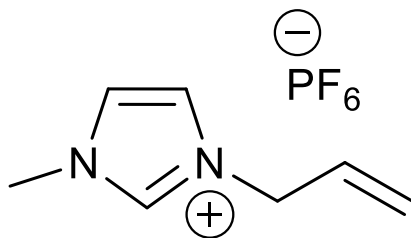
Ammonium hexafluorophosphate:



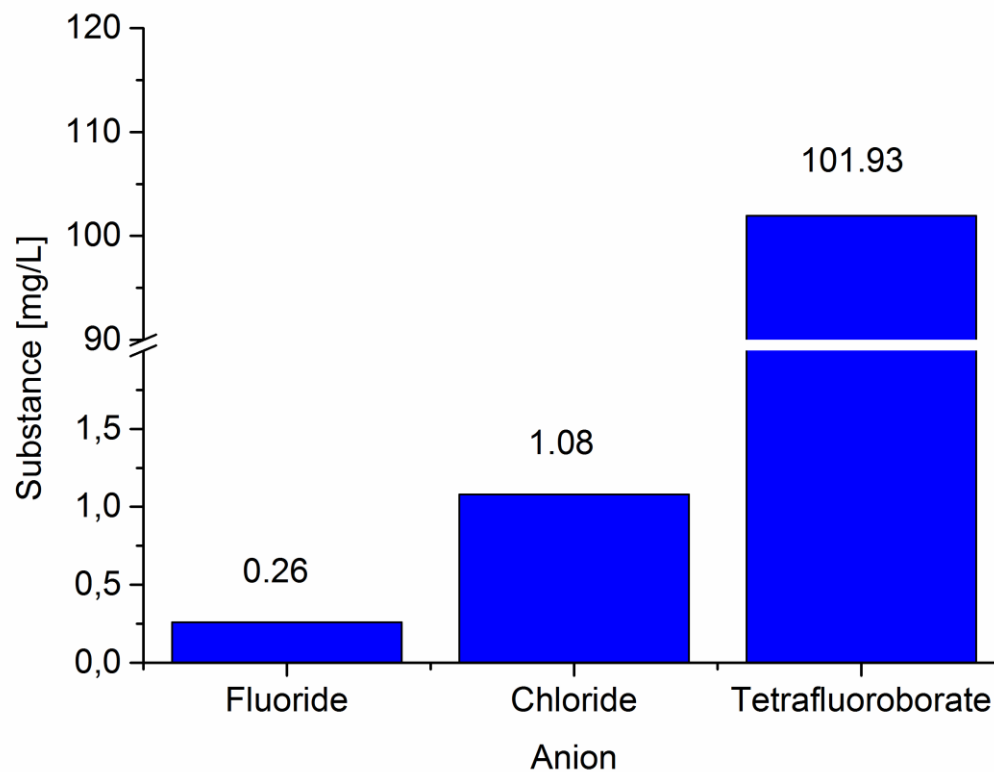
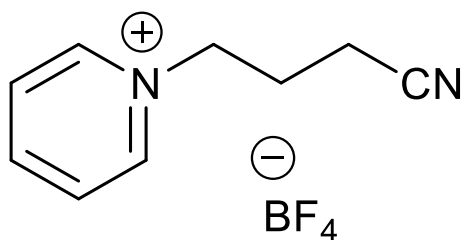
Results:

- Measurement of the ionic liquids

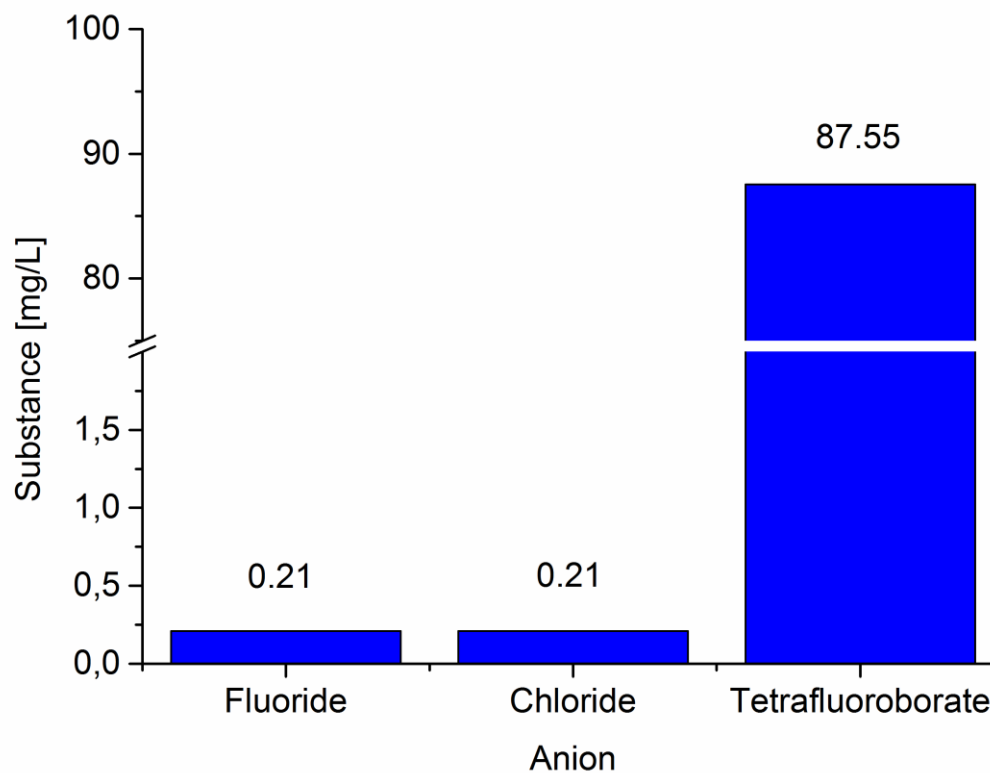
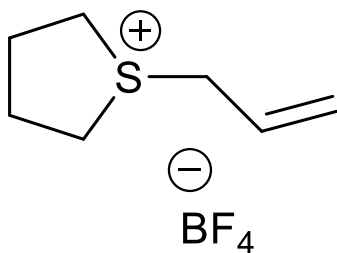
Allyl-Methyl-Imidazoliumhexafluorophosphate
[AMIm][PF₆]



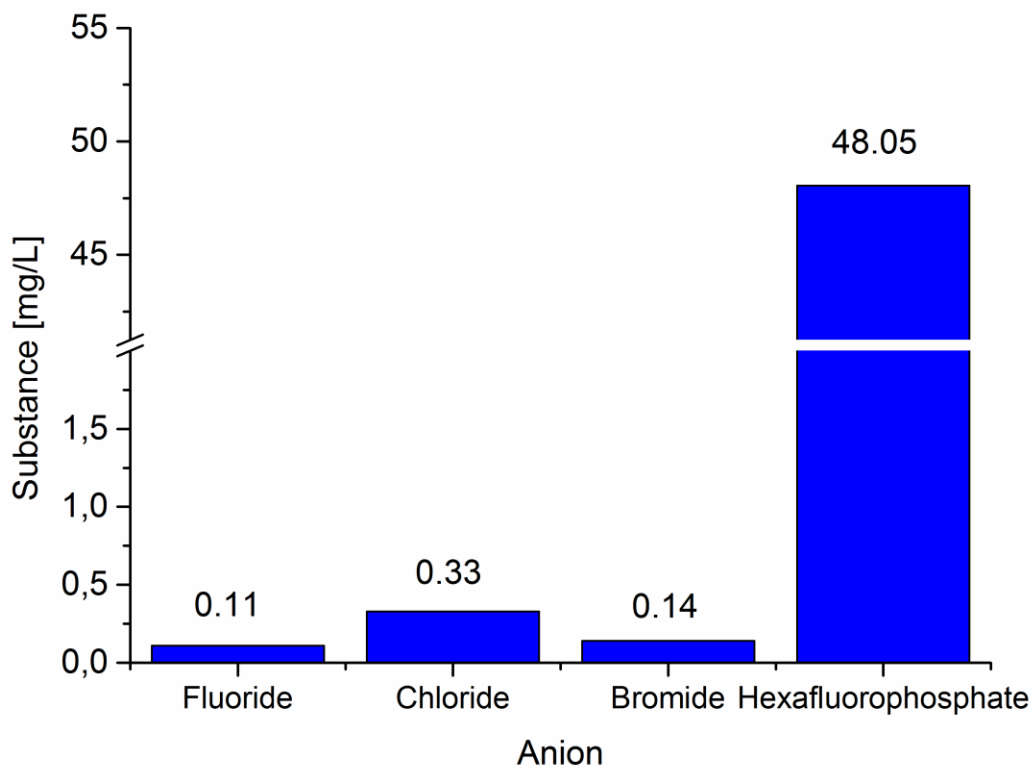
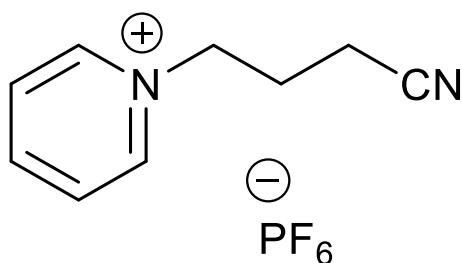
4-Butyro-Nitrile-Pyridiniumtetrafluoroborate $[C_3CNPy][BF_4]$



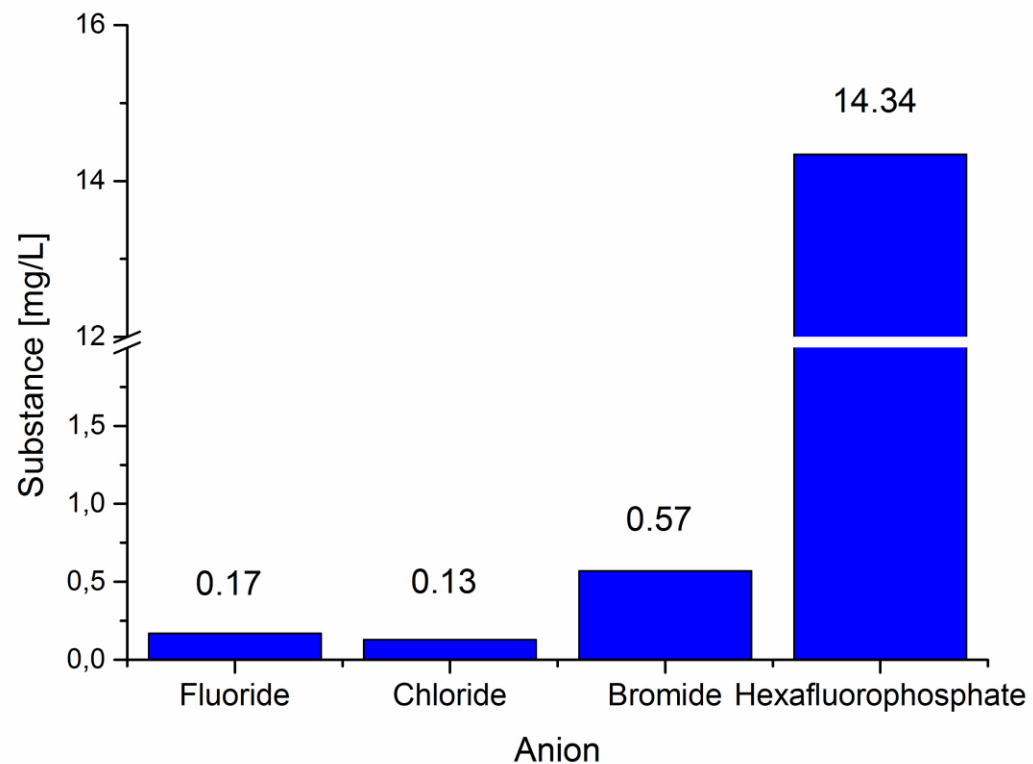
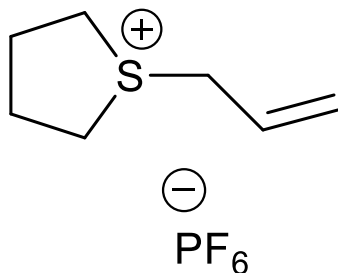
Allyl-Thiotetrafluoroborate [AllylTh][BF₄]



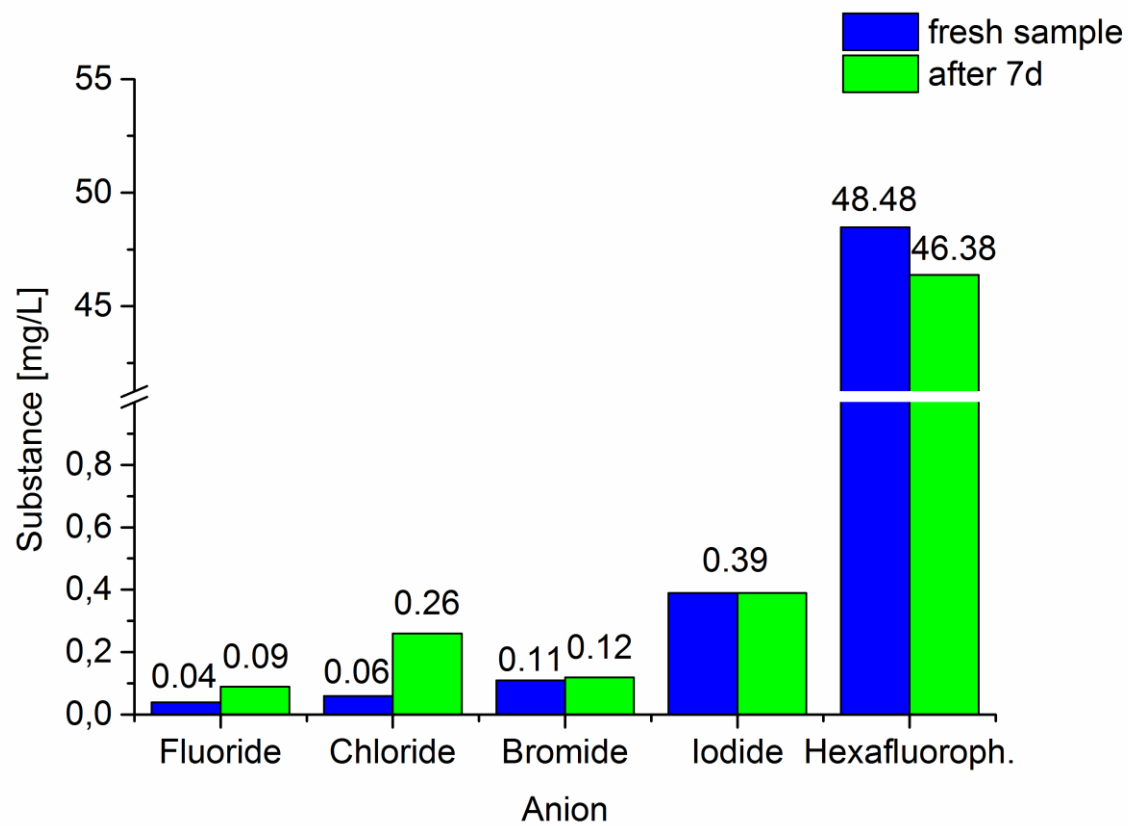
4-Butyro-Nitril-Pyridiniumhexafluorophosphate [C₃CNPy][PF₆]



Allyl-Thiohexafluorophosphate [AllylTh][PF₆]



- Hydrolysis of $[\text{AMIm}][\text{PF}_6]$



[5] L. Terborg, S. Nowak, S. Passerini, M. Winter, U. Karst, P. R. Haddad, P. N. Nesterenko, *Anal. Chim. Acta* **2012**, 714, 121–126.

[6] A. V. Plakhotnyk, L. Ernst, R. Schmutzler, *J. Fluorine Chem.* **2005**, 126, 27–31.

Summary:

Ionic liquid	F ⁻ [%]	Cl ⁻ [%]	Br ⁻ [%]	I ⁻ [%]	BF ₄ ⁻ [%]	PF ₆ ⁻ [%]
[AMIm][PF ₆]	0.07	0.11	0.23	0.80	-	98.79
[C ₃ CNPy][BF ₄]	0.25	1.04	-		98.71	-
[AllylTh][BF ₄]	0.23	0.23	-		99.53	-
[C ₃ CNPy][PF ₆]	0.22	0.68	0.30		-	98.80
[AllylTh][PF ₆]	1.12	0.86	3.72		-	94.30
[AMIm][PF ₆] fresh sample	0.07	0.11	0.23	0.80	-	98.79
[AMIm][PF ₆] 7d	0.18	0.55	0.26	0.83	-	98.18

Outlook:

- Analysis of cations
chiral ILs by appropriate column
- Appropriate eluents
- Long-term measurement
- Different storage conditions
- Analysis of other ILs

Thanks to:



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