高等学校化学学报 2009, 30(9) 1814-1818 DOI: ISSN: 0251-0790 CN: 22-1131/O6

论文

离子液体1-仲丁基-3-甲基咪唑六氟磷酸盐的微波辅助合成、晶体结构及热稳定性

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摘要:

以具有支链结构的溴代仲丁烷作为烷基化试剂, 在微波辐射下采用两步法合成了1-仲丁基-3-甲基咪唑六氟磷酸盐([s-bmim][PF_6])离子液体. 在配比为V(水):V(乙醇):V(甲苯)=0.16:2.84:7的混合溶剂体系中, 培养出晶型完整的长约11 mm的离子液体大单晶体. 通过单晶体X衍射研究了[s-bmim][PF_6]的晶体结构. [s-bmim][PF_6]属于三斜立方晶系, 空间群为$P2(1)/m$, 晶胞参数为a=0.9042(4) nm, b=0.8213(3) nm, c=0.9775(4) nm, γ=116.618°(6), Z=2, V=64.909(4) nm^3, D_c=1.454 g/cm^3, μ=0.265 mm^{-1}, F(000)=292. 在[s-bmim][PF_6]晶体结构中, 阴阳离子间的离子键仍然是主要的, 同时还存在氢键和阳离子-阳离子间的非键斥力作用. 研究结果表明, [s-bmim][PF_6]的支化烷基结构对其晶体有效堆积、熔点、液程范围以及热分解温度等性质具有重要影响.

关键词: 离子液体; 微波合成; 晶体结构; 热稳定性

Microwave-Assisted Synthesis, Crystal Structure and Thermal Stability of Ionic Liquid 1-sec-Butyl-3-methylimidazolium Hexfluorophosphate

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Abstract:

In this paper, 1-sec-butyl-3-methylimidazolium hexfluorophosphate([s-bmim][PF_6]) was synthesized with the aid of microwave. Large single crystals were formed in the water-ethanol-toluene system[V(water):V(ethanol):V(toluene)=0.16:2.84:7]. The large single crystals are stable under the air and moisture, with the maximum length of 11 mm. The crystal structure was determined by X-ray diffraction. [s-bmim][PF_6] crystallizes in the monoclinic space group $P2_1/m$, with unit-cell parameters a=0.9042(4) nm, b=0.8213(3) nm, c=0.9775(4) nm, γ=116.618°(6), Z=2, V=64.909(4) nm^3, D_c=1.454 g/cm^3, μ=0.265 mm^{-1}, F(000)=292, the final R=0.0998, wR=0.2656. In the structure, the cations and anions can not form neutral ion pairs owing to the interionic interactions such as the dominant Coulombic attraction, the weak hydrogen bonds and the non-chemical-bond tension of cation-cation. Moreover, it was found that introducing the sec-butyl chain could change the asymmetry of cations and inhibit the rotation of the alky chains of [s-bmim][PF_6]. The branched structure leads to the void space filling in the crystal stacking and high lattice energy. Finally, the thermal stability of [s-bmim][PF_6] was discussed.

Keywords: Ionic liquid; Microwave synthesis; Crystal structure; Thermal stability

收稿日期 2008-10-13 修回日期 网络版发布日期

DOI:

基金项目:

国家自然科学基金(批准号: 50572089)资助.

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参考文献:

[1]Liao J. H., Wu P. C., Huang W. C.. Cryst. Growth Des.
[J], 2006, 6(5): 1062—1063

[2]Jacob D. S., Genish I., Klein L., et al.. J. Phys. Chem. B
[J], 2006, 110(36): 17711—17714

[3]LI Shang-Yu(李尚禹), WANG Run-Wei(王润伟), WAN Li-Feng(万利丰). Chem. J. Chinese Universities(高等学校化学学报)
[J], 2008, 29(3): 465—467

[4]Parnham E. R., Morris R. E.. Chem. Mater.
[J], 2006, 18 (20): 4883—4887

[5]Li Z. H., Liu Z. M., Zhang J. L., et al.. J. Phys. Chem. B
[J], 2005, 109(30): 14445—14448

[6]Absalan G., Akhond M., Sheikhian L.. Talanta
[J], 2008, 77(1): 407—411

[7]Kim Y. J., Varma R. S.. Tetrahedron Lett.
[J], 2005, 46(43): 7447—7449

[8]Deetlefs M., Seddon K. R.. Green Chemistry
[J], 2003, 5(2): 181—186

[9]Triolo A., Mandanici A., Russina O., et al.. J. Phys. Chem. B
[J], 2006, 110(42): 21357—21364

[10]Iwata K., Okajima H., Saha S., et al.. Chem. Res.
[J], 2007, 40(11): 1174—1181

[11]Yoshida Y., Muroi K., Otsuka A., et al.. Inorg. Chem.
[J], 2004, 43(4): 1458—1462

[12]Babai A., Mudring A. V.. Inorg. Chem.
[J], 2006, 45(13): 4874— 4876

[13]Xue H., Tong Z.F., Wei F.Y., et al.. C. R. Chi.
[J], 2008, 11(1/2): 90—94

[14]Matsumoto K., Hagiwara R., Mazej Z., et al.. Solid State Sci.
[J], 2006, 8(10): 1250—1257

[15]Gordon C. M., Holbrey J. D., Kennedy A. R., et al.. J. Mater. Chem.
[J], 1998, 8(12): 2627—2636

[16]Nikitenko S. I., Hennig C., Grigoriev M. S., et al.. Polyhedron
[J], 2007, 26(13): 3136—3142

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