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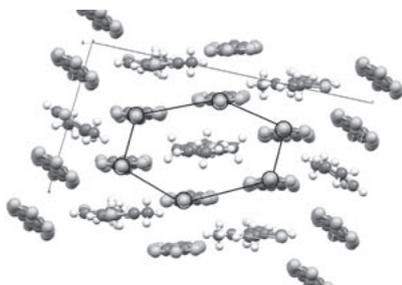
Honeycomb-like structures in the crystals of Ni(dmit)₂ salts with di- and tri-alkylthiazole cations

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In the crystal structure of 2,3-diethylthiazolium [Ni(dmit)₂] (dmit = 1,3-dithiole-2-thione-4,5-dithiolate) previously reported,^[1] anions formed a distorted honeycomb-like structure. In relation to this compound, two thiazolium derived cations (3,5-dimethyl-, and 3,4,5-trimethylthiazolium) were synthesized and the crystal of their [Ni(dmit)₂] salt were characterized by single crystal X-ray analysis. In dialkylthiazolium salts, anions also formed similar honeycomb-like structures and took cations inside the honeycomb cavities by S-S interactions. On the other hand, in trimethylthiazolium salts, no honeycomb structures were constructed but the layers of cations and anions piled up alternately. These results indicate that the difference in the crystal packing between dialkyl- and trimethyl-thiazolium cations is caused by the steric hindrance of the methyl group. Figure Honeycomb-like structure of [Ni(dmit)₂] in 3,5-dimethylthiazolium salt.

[1] E. Tomiyama, K. Tomono, K. Miyamura, *Acta Crystallographica Section E-Structure Reports Online* **2007**, 63, m2741.



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Structural investigations of the nickel mediated coupling products of CO₂ and isoprene

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The nickel mediated coupling with unsaturated substrates is an efficient way to use CO₂ as a carbon source. By using 1,3-dienes in this reaction, nickelacyclic carboxylates with an allyl group are

obtained.[1] Several ligands can be used in this reaction, among them tetramethylethylenediamine (tmeda) and 2,2'-bipyridine (bipy). During our investigations of the coupling reaction of 2,3-dimethyl-1,3-butadiene (dmbd) and CO₂, the related isoprene coupling products were obtained as side products, formed from traces of isoprene in dmbd. The X-ray structure determination shows, that with bipy the usual regioisomere **A** [2] is formed, while with pyridine (derived from the tmeda complexes by ligand exchange) the opposite regioisomere **B** is observed. The formation of the unexpected isomere **B** encouraged us to further investigate the regioselectivity of the isoprene/CO₂ coupling with tmeda as supporting ligand. The formed products are characterized by X-ray structure determination and the results are presented.

[1] D. Walther, E. Dinjus *Z. Chem.* **22** (1982) 228-229.

[2] H. Hoberg, D. Schaefer, B.W. Oster *J. Organomet. Chem.* **266** (1984) 313-320.

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Hydrothermal synthesis and crystal structure of a new copper coordination polymer

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Hydrothermal method provides a powerful tool for the construction of materials containing unique structures and special properties. It also a route to synthesis of complexes of transition metals and the molecular structures of complexes obtained by this method are unexpected compared to those obtained by the common solution method. We have recently reported hydrothermal synthesis of binuclear Co(II) complex under hydrothermal condition. Here we wish to report the hydrothermal synthesis of a new Cu(II) coordination polymer containing bridged oxalate ions and 2,2'-bipyridine ligand. The complex has been prepared from the reaction of bis(1,5 cyclohexanone) oxal dihydrazone and 2,2' - bipyridine with Cu(NO₃)₂ in basic solution under hydrothermal condition. The characterization was performed using IR, NMR spectroscopy and X-ray crystallography. The result showed that the bis(1,5 cyclohexanone) oxal dihydrazone was changed to oxalate ion under heating and basic pH. This complex is crystallized in triclinic system, space group *P*-1, with two molecules per unit cell. The unit cell parameters are $a = 8.9298(18)\text{\AA}$, $b = 9.1011(18)\text{\AA}$, $c = 9.7112(19)\text{\AA}$, $\alpha = 97.61(3)^\circ$, $\beta = 92.602(2)^\circ$ and $\gamma = 105.80(3)^\circ$. Complex is a coordination polymer. Each metal ion center is in a distorted octahedral geometry and coordinated by four O atoms from two bridged oxalate ions and two N atoms of 2,2' - bipyridine. Two uncoordinated water molecules occupy general positions in the cell. In the crystal structure, some H-bonds and π - π interaction cause formation of a 3D network.

Keywords: coordination polymers, crystal structure determination, hydrothermal method