

Discrepancy between crystal structure and magnetic properties of Ni (II) complexes synthesized with 4-methylpiperidine and tridentate Schiff base

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Slow magnetic relaxation and paramagnetism in a supposed dimer made of two square planar Ni(II) units, namely $[\text{Ni}(\text{L})(\text{Me-pip})]_2$ consisting of the tridentate Schiff-base ligand L and 4-methylpiperidine was observed. However, crystal structure of $[\text{Ni}(\text{L})(\text{Me-pip})]$ solved by single crystal structural analysis approves that complex is a mononuclear entity. In such a case the complex would be square-planar and thus diamagnetic. In order to check the purity, crystallinity and identity of the bulk powder material a series of X-ray experiment was done: single-crystal structure determination at $T = 100, 150, 200, 250, \text{ and } 298 \text{ K}$; powder diffraction at the room temperature followed by LeBail refinement.

It was found that the simulated diffraction pattern from the single-crystal structure determination does not match the experimental diffraction pattern of the bulk powder sample. It was considered that this sample could be mixture of complex with already solved crystal structure and other yet unknown compound. Based on this observation, the syntheses with the same reactants at different experimental conditions were made. The products were evaluated with qualitative phase analysis and four different products were found. It has been shown that the mix of two of these products can form the already mentioned bulk sample. This work presents preliminary results of single crystal and powder structural analysis of the four samples and comparison of the crystal structures obtained from powder and single crystal analysis.

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