

Structure Determination of the Coordination Polymer Dipydroquinonecopper from Powder Diffraction Data using Simulated Annealing

R. Dinnebier (U. Bayreuth), Li Ding, H-W. Lerner, and M. Wagner (U. Frankfurt), K. Shankland, and W.I.F. David (ISIS, RAL) and P.W. Stephens (SUNY, Stony Brook)

Abstract No. dinn1031

Beamline(s): X3B1

Introduction: Low dimensional metal-containing solids (e.g. for applications as electronic or magnetic devices) can be regarded as the result of a stacking of slabs or a juxtaposition of fibers. This particular situation directly reflects a very high anisotropy in the chemical bonding. Strong ionic-covalent bonds are responsible for the cohesion of slabs or fibers, whereas these units are separated by weak interactions mostly of the van der Waals type. The title compound, which has been generated from the reaction of CuCl_2 with 2,5-di(1-pyrazolyl)hydroquinone in aqueous ammonia, features antiferromagnetically coupled electron spins ($S = \frac{1}{2}$; coupling constant $J = 10\text{-}15\text{ K}$). To verify, that the title compound can be regarded as a one-dimensional Heisenberg spin chain, it was of prime importance to elucidate the structure of the material in the solid state. Due to the extremely poor solubility of the compound in all common solvents, attempts to grow single crystals suitable for an X-ray crystal structure analysis failed.

Methods and Materials: X-ray powder diffraction data were collected for the compound at 295 K ($\lambda = 1.4991(2)\text{ \AA}$) in transmission geometry with the sample sealed in a 0.7 mm lithium borate glass capillary (**Figure 1**). Data reduction was performed using the GUF1 program. Indexing with ITO led to a primitive triclinic unit cell with lattice parameters of $a = 5.1723(5)$, $b = 7.9587(9)$, $c = 8.2298(11)$ [\AA], $\alpha = 118.221(6)$, $\beta = 91.520(9)$, and $\gamma = 100.148(8)^\circ$. The number of formula units per unit cell could be determined to $Z = 1$ from packing considerations, indicating $P\bar{1}$ as the most probable space group, which could later be confirmed by Rietveld refinement. The peak profiles and precise lattice parameters were determined by LeBail-type fits using the program Fullprof. The background was modeled manually using GUF1. The peak-profile was described by a pseudo-Voigt in combination with a special function that accounts for the asymmetry due to axial divergence. The crystal structure was solved using the DASH structure solution package (**Figure 2**).

Results: From the X-ray powder diffraction data it was possible to show, that the title compound consists of discrete polymer chains, which are not crosslinked by oxygen or amide bridges. The magnetic interaction is therefore largely restricted to intrachain coupling. Magnetic exchange processes between the individual chains can probably be neglected.

Acknowledgments: This work was supported by the German Research Council (DFG) and the Division of Basic Energy Sciences, U.S. DOE (DE-FG02-86ER45231).

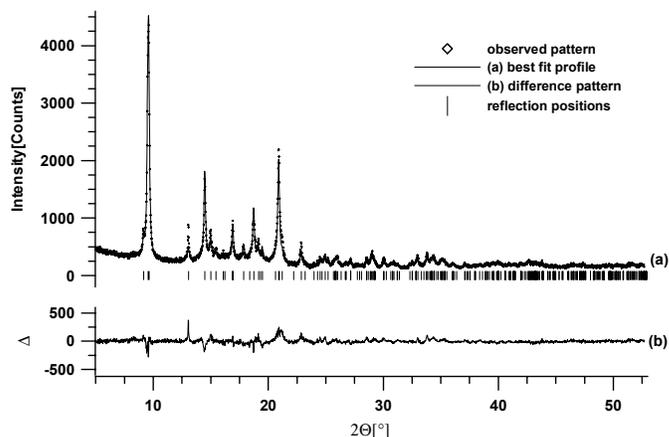


Figure 1. Scattered X-ray intensity for diphydroquinonecopper at ambient conditions as a function of diffraction angle 2θ . Shown are the observed pattern (diamonds), the best Rietveld fit profile (line), the reflection positions and the difference curve between observed and calculated profiles as the trace at the bottom.

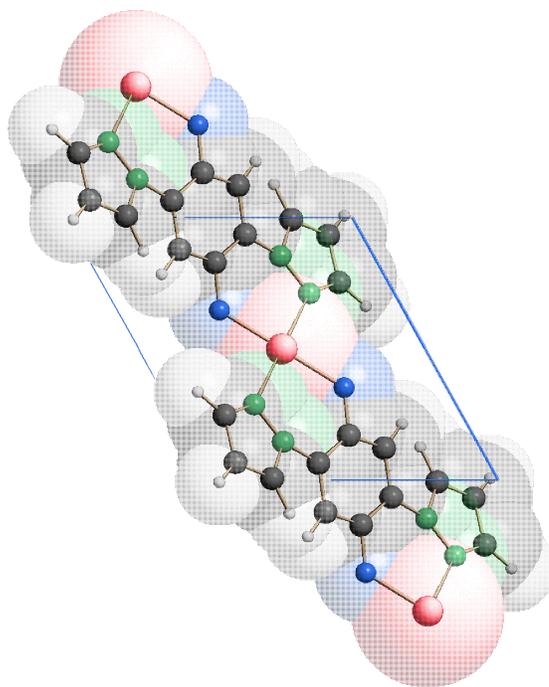


Figure 2. Crystal structure of diphydroquinonecopper. Space filling model showing the polymeric nature of the compound in a projection along a -axis