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Synthesis and Structural Characterization of Adduct $Cu_2(phen)_2 \cdot 3(FCA) \cdot (ClO_4) \cdot 2H_2O$

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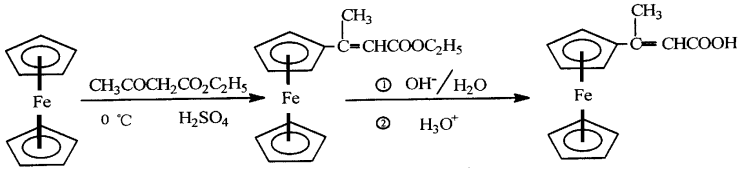
Abstract: A new dinuclear copper (II) complex containing 3-ferrocenyl-2-crotomic acid salt (FCA) and phen has been synthesized. Its structure was characterized by X-ray crystal analysis. The molecular is a pentametallic compound.

Keywords: Copper adduct , crystal structure.

Different kinds of copper(II) carboxylate adducts have been synthesized and also dinuclear copper(II) complexes have been early studied as useful models to establish magneto-structural correlations for spin exchange between metal ions. Costa [1] showed the structure of $[Cu_2(\mu\text{-}OOCFc)_2(bpy)_2(ClO_4)(CH_3OH)]^+$ and $[Cu_2(\mu\text{-}OOCFc)_2\ (bpy)\ (CH_3OH)_2]^{2+}$. As far as we know, the copper(II) compounds containing FCA and phen have not been reported.

The ligand of FCA was synthesized according to the following scheme [2,3]:

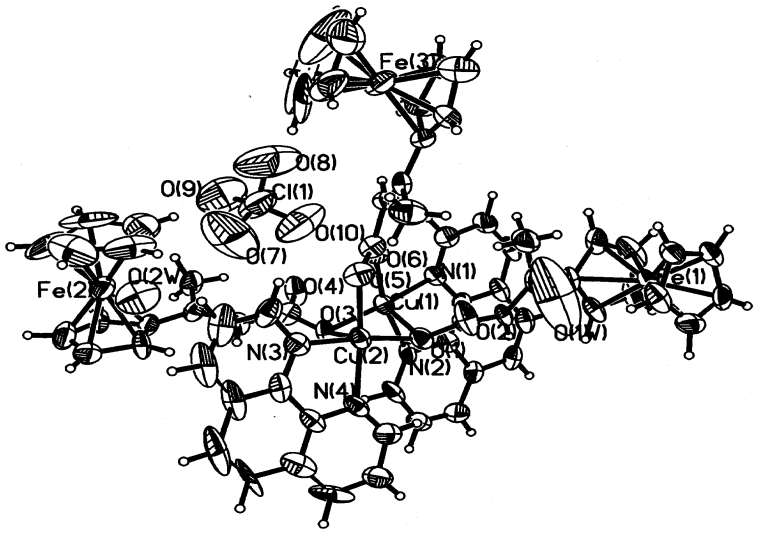


A solution containing 198mg (1 mmol) of phen and 15mL of methanol was slowly added to the solution formed by $Cu(ClO_4)_2 \cdot 6H_2O$ (370mg,1mmol) and 5mL of methanol with continuous stirring at room temperature. And then, 10mL water was added. Afterward, 270mg (1mmol) FCA and 40mg sodium hydroxide dissolved in 10mL methanol was added. The solution was stirred at room temperature for 2hr and filtered. The product was dissolved in mixture solvent of dichloromathane and methanol and slowly evaporated to form dark brown monocrystal for X-ray analysis.

A crystal with a dimension of 0.48mm×0.24mm×0.04mm was selected for X-ray diffraction experiment. Crystal $Cu_2(phen)_2 \cdot 3(FCA) \cdot (ClO_4) \cdot 2H_2O$, Mr=1430.25, monoclinic, space group Pc, with Z=2, a=1.2799(4) nm, b=0.9969(4) nm, c=2.5228(10) nm, β=91.576(1)°, V=3.218(2) nm^3, Dc=1.476g/cm^3. 12006 independent reflections were collected on a Siemens SMART CCD areadetector diffractometer with graphite-monochromatized Mo Kα radiation (λ=0.071073nm) and with the range of

$2.70^{o}<\theta<28.47^{o}$ using ω scans. Empirical absorption was applied. The structure was solved by direct methods using SHELXS-97 program. The positional and the anisotropic thermal parameters of non-hydrogen atoms were refined by full matrix least squares method of SHELX-97 program to a final R=0.0899, R_w=0.1691 for 12006 independent reflections of $I>2\sigma(I)$. The positions of hydrogen atoms were obtained by theory hydrogen addition. The molecular structure of the title compound is shown in **Figure 1**.

Figure 1. Molecular structure of the adduct (the carbon atoms are not labelled)



Results

In the title compound, the copper atoms have a five-coordinate environment. The basal plane is defined by oxygen atoms from three distinct Fc-C(CH_3)=CHCOO$^-$ groups and two nitrogen atoms from the chelating phen ligands. Two coppers are bridged in two ways, *i.e.* −O− and −O−C−O−. It is an interested structure.

Acknowledgments

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4. Crystallographic parameters have been deposited in the editorial office of CCL.

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