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The Preparation and X-ray Crystal Structure of a Saccharin Complex of Copper(II)

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The chemistry of saccharin (*o*-sulphobenzoimide), commonly used as a sweetening agent, has attracted attention in recent years because of its suspected carcinogenous nature [1, 2]. Most of these studies are concerned with the biochemical and physiological activities of the compound, but there are only scanty reports on its transition metal complexes [3–5]. Since transition elements, particularly Fe, Co, Ni, Mn, Cu, Zn and Mo play vital role in life processes, we have begun a systematic study of the complexes of saccharin with these metals. As far as we are aware, there is no report on the crystal structure of any metal-saccharin complex.

Malik and Sharma [4] reported the preparation of 1:1 metal–saccharin complexes of Cu(II), Ni(II) and Co(II), and 1:2 complex of Fe(III), by concentrating in vacuo a mixture of ethanolic solutions of the metal chlorides and saccharin. We have been unable to reproduce these results, but isolated well crystalline products from the following reactions:

$$\text{(i)}\ MCl_2 + LH \xrightarrow{\text{aq. } NaHCO_3} ML_2(H_2O)_6$$

$$[M = Cu^{+2}\ \&\ Co^{+2};\ L = (C_7H_4NO_3S)^-]$$

$$\text{(ii)}\ MX_2 + NaL \xrightarrow{H_2O/\text{ethanol}} ML_2(H_2O)_n$$

$$[M = Ni^{+2}, X = NO_3^-; M = Zn^{+2}, X = Cl^-]$$

The copper complex has been fully characterized by single crystal structure determination.

Crystal Data

$[Cu(C_7H_4NO_3S)_2(H_2O)_4]\cdot 2H_2O$, $C_{14}H_{20}CuN_2O_{1=}S_2$, M = 535.99, Monoclinic, a = 8.384(2), b = 16.327(2), c = 7.328(2) Å, β = 101.08(2), V = 984.4 Å^3, Space group $P2_1/c$, Z = 2, D_c = 1.81 g cm^{-3}, $F(000)$ = 548, $\mu(MoK_\alpha)$ = 13.4 cm^{-1}, $\lambda(MoK_\alpha)$ = 0.71069 Å.

The intensities of 1992 reflections ($1.5 < \theta < 25°$) were recorded on an Enraf-Nonius CAD4 diffractometer using MoK_α radiation and an $\omega/2\theta$ scan. The structure was solved by the heavy atom method and refined by full-matric least-squares (all non-hydrogen atoms anisotropic, hydrogen atoms isotropic) to an R-value of 0.034 for 1339 significant $F_o > 4\sigma(F_o)$ reflections.

The crystal structure consists of molecules of $[Cu(C_7H_4NO_3S)_2(H_2O)_4]$ and H_2O bound together by hydrogen bonds. The molecular structure of $[Cu(C_7H_4NO_3S)_2(H_2O)_4]$, which is centrosymmetric, is shown in Fig. 1. The copper atom is co-ordinated by two mutually *trans* saccharin ligands with Cu–N 2.061(2) Å and four water molecules, two at 1.956(2) and two at 2.489(3) Å, giving a strongly tetragonally distorted octahedral geometry.

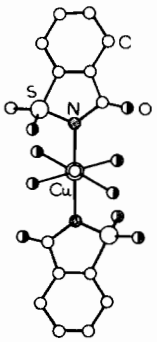

Fig. 1. Molecular structure of $[Cu(C_7H_4NO_3S)(H_2O)_4]$.

Our investigations show that saccharin forms very simple type of metal complexes rather easily. We are continuing the spectral and magnetic studies of these complexes as well as their full structure analysis.

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