The Crystal Structure of 1-(2-Aminoethyl)biguanidecyanoguanidine-copper(II) Sulphate Monohydrate

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THE mother liquor from ethylenebisbiguanidecopper(II) sulphate, prepared by the method of Rây and Chakravarty ethylenediamine hydrochloride, cyanoguanidine, and copper(II) sulphate,[1] gives on slow concentration at room temperature, red–violet monoclinic needles containing Cu^{II}, SO_4^{2-}, and nitrogen-containing organic ligands. The results of conventional chemical analysis of these crystals are consistent with the formula for ethylenebisbiguanidecopper(II) sulphate as well, but the i.r. spectrum shows a characteristic C≡N stretching band at 2250 cm.$^{-1}$. This by-product

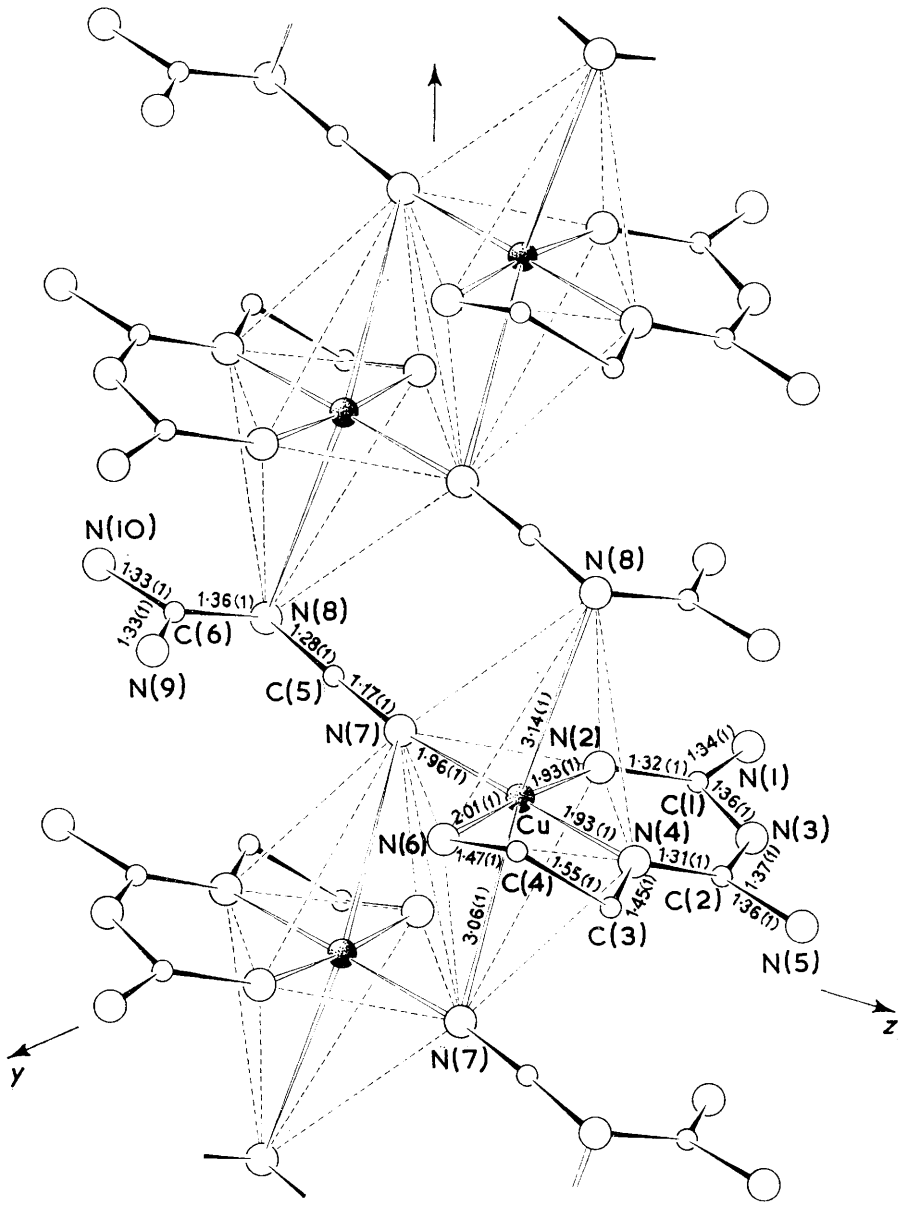

FIGURE. *Clinographic projection of a chain of co-ordination polyhedra in* $[Cu(C_4H_{12}N_6)(C_2H_4N_4)]SO_4,H_2O$

was shown by an *X*-ray structural analysis to be 1-(2-aminoethyl)biguanidecyanoguanidinecopper(II) sulphate monohydrate.

Crystal data from rotation and Weissenberg photographs (Cu-K_α) are as follows: $[Cu(C_4H_{12}N_6)(C_2H_4N_4)]SO_4,H_2O$; $M = 389{\cdot}7$, $a = 7{\cdot}088(7)$, $b = 15{\cdot}64(1)$, $c = 13{\cdot}43(1)$ Å, $\beta = 101{\cdot}8°(0{\cdot}2°)$, $Z = 4$, $U = 1458$ Å^3, $D_m = 1{\cdot}83$, $D_c = 1{\cdot}76$ g.cm.$^{-3}$, $\mu = 37{\cdot}98$ cm.$^{-1}$, $F(000) = 836$. Space group: $P\,2_1/c$; 2413 independent reflections were measured photometrically from equi-inclination Weissenberg photographs taken around [100] and [001]. The structure was determined by three-dimensional Patterson and Fourier techniques using the

"heavy-atom method". After four isotropic and one anisotropic cycle of Booth's differential synthesis, the R valve is now 11·2% and the refinement is continuing.

Co-ordination around the metal atom is shown in the Figure: three nitrogen atoms from a 1-(2-aminoethyl)biguanide molecule and a nitrilic nitrogen atom from a cyanoguanidine molecule, form a nearly square-planar arrangement, the displacements from the best least-squares plane through the co-ordinated nitrogen atoms are: ±0·02 Å for N(2), +0·02 Å for N(4), −0·03 Å for N(6), +0·05 Å for N(7), and +0·01 Å for Cu. Co-ordination is completed to a distorted tetragonal dipyramid by the two long copper bonds with the nitrilic nitrogen and the C–N–C system from two different cyanoguanidine molecules, which are *trans* with respect to the co-ordination plane. The 1-(2-aminoethyl)biguanide molecule therefore behaves as a tridentate ligand, while the nitrilic and the C–N–C nitrogen atoms of cyanoguanidine are shared by a couple of co-ordination bipyramids which, linked by N–C≡N bridges, form *zig-zag* chains running along the [100] plane. In each pair there is a contact of 3·44 Å between two copper atoms related by a symmetry centre. The sulphate ion and the water molecule, not represented in Figure, form hydrogen-bond contacts, the first with two biguanide (O · · · H–N = 2·92, 2·87 Å) and two cyanoguanidine (O · · · H–N = 2·93, 2·96, 2·98 Å) molecules, and the second between two SO_4^{2-} groups (O–H . . . O = 2·77, 2·89 Å).

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[1] P. Rây and K. Chakravarty, *J. Indian Chem. Soc.*, 1944, **21**, 41.