

The Structure of a Bis-(+)-pseudoephedrine Complex of Copper(II)

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Two distinct crystalline complexes of (+)-pseudoephedrine with copper(II) may be obtained by the addition of ethanol to an aqueous solution of copper sulphate and an excess of (+)-pseudoephedrine in the presence of base.¹ Violet, brick-like crystals (for A) give a different X-ray powder photograph and mass spectrum from the pale violet needle crystals (form B), the structure of which has been elucidated by single-crystal methods.

The needle-like crystals of form B belong to the space group $P2_1$; the asymmetric unit contains the bispseudoephedrine complex, a non-coordinated pseudoephedrine molecule, and two molecules of water [$\rho_{\text{obs}}(20^\circ) = 1.250 \text{ g. cm.}^{-3}$; $\rho_{\text{calc}} = 1.248 \text{ g. cm.}^{-3}$]. Using 1463 independent, visually-estimated reflexions, the structure has been determined by Patterson, superposition, Fourier, and least-squares methods. The refinement analysis has converged to a discrepancy index of 0.11 when the average estimated standard deviations of the bond lengths are Cu-O and Cu-N 0.02 Å, and C-C, C-N, C-O 0.04 Å.

The stereochemistry of the complex (Figure) is such that the pseudoephedrine ligands have a *trans*-configuration; their individual co-ordination planes are mutually inclined at an angle of 21° , so that the co-ordination around the copper is distorted away from a planar arrangement towards one of tetrahedral symmetry. This distortion appears to be a consequence of crystal packing requirements, and particularly of hydrogen bonding in the lattice.

Two hydrogen-bonded systems are present in the crystal. The non-chelated pseudoephedrine molecules (comprising one scheme), have intramolecular hydrogen bonds ($\text{O} \cdots \text{N}$, 2.70 Å), whilst

further intermolecular hydrogen bonding forms chains of molecules parallel to the crystallographic b axis ($\text{N} \cdots \text{O}'$, 2.73 Å). In addition, an independent network of symmetry-related co-ordinated pseudoephedrine molecules is linked through water molecules which are also mutually hydrogen bonded.

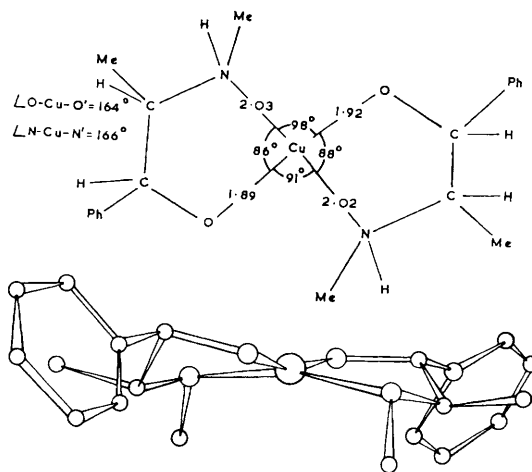


FIGURE. The molecular geometry and stereochemistry of bis-(+)-pseudoephedrinocopper(II).

The observed configuration and conformation of pseudoephedrine confirms earlier evidence and suggestions.² Each of the nitrogen atoms has the same optical configuration and the present analysis shows that, in the crystal at least, the methyl groups on the nitrogen and adjacent carbon atoms

in pseudoephedrine have a staggered conformation, both in the chelated and non-chelated forms.

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¹ R. D. Gillard and R. Wooton (unpublished work).

² W. J. Close, *J. Org. Chem.*, 1959, **24**, 1131.