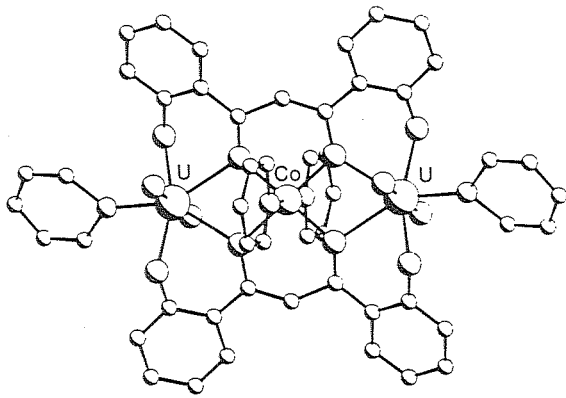


09.4-25 CRYSTAL STRUCTURE OF BIS (1,3-BIS-(2-HYDROXYPHENYL)-1,3-PROPANEDIONE) BIS (DIOXOURANIUM (VI)) COBALT (II) HEXAKISPYRIDINE,  $C_{50}H_{30}N_6O_{12}U_2Co$ .  $2C_6H_5N$

by E.Molins, J.Rius, J.Colomer, F.Teixidor, C.Miravittles and J.Casabo  
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As a part of our research program about polynuclear coordination compounds with macrocyclic or acyclic ligands we have undertaken the molecular crystal structure of title compound. A dark red crystal of  $C_{50}H_{30}N_6O_{12}U_2Co$  was selected for X-ray diffraction analysis. The compound crystallizes in the  $P\bar{1}$  space group,  $Z=1$ , with  $a=9.919(5)$ ,  $b=11.597(8)$ ,  $c=12.789(3)$  Å,  $\alpha=93.02(4)$ ,  $\beta=92.58(3)$ ,  $\gamma=106.67(5)^\circ$ ,  $V=1404.6$  Å<sup>3</sup>.  $\mu(MoK\alpha)=58$  cm<sup>-1</sup>. Intensity data were collected on an Enraf-Nonius CAD4 diffractometer; from 4063 unique reflections, 1980 were considered observed ( $F > 2.5\sigma(F)$ ).

The structure was solved with Rotsearch program (Rius and Miravittles, J.Appl.Cryst. in press) and refined with SHELX-76 (Sheldrick, 1976) up to a final R of 0.107 and  $R_w$  of 0.102. The maximum final difference Fourier peak was  $5$  eÅ<sup>-3</sup> near U atom (1.15Å). A perspective view of the molecule is shown in the Figure (PLUTO drawing: Motherwell and Clegg, 1978). The Co atom is placed at the crystallographic symmetry center of the cell and its coordination is octahedral. In the axial direction it is coordinated with two pyridine groups, and in the basal plane it is bonded to four oxygen atoms of the  $\beta$ -diketone moiety of the two ligand groups. The U-atoms are seven-coordinated, in the basal plane with four keto-phenolic oxygen atoms of the ligands and one pyridine group and, in the axial direction, with the two oxygen atoms from uranyl group. The U...Co distance is  $3.59(2)$  Å. The other two pyridine groups occupy interstitial positions.



09.4-26 THE FIRST X-RAY STRUCTURALLY CHARACTERIZED N-2-PYRIDINYLCARBONYL-2-PYRIDINECARBOXIMIDATE (BPCA) COPPER (II) COMPLEXES. by J.V.Folgado and D.Beltrán. Departamento de Química Inorgánica, Facultad de Ciencias Químicas, Universidad de Valencia, c/ Dr. Moliner, 50, Burjassot, Valencia, Spain, and A.Fuertes and C.Miravittles. Institut de Ciència de Materials. (C.S.I.C.). c/ Martí i Franqués, s/n. Apartado de Correos 30102. 08028 BARCELONA. Spain.

To progress in the study of magneto-structural correlations in polynuclear Cu(II) complexes we have approached the structural analysis of  $Cu(BPCA)Br$  (I) and  $[Cu(BPCA)(H_2O)(CH_3COO)] \cdot H_2O$  (II). Both structures have been solved using the MULTAN 11/84 system and refined by weighted anisotropic full-matrix least-squares with the SHELX 76 system. The final  $wR$  values were 0.065 for I and 0.049 for II.

Complex I,  $C_{12}H_{10}BrCuN_3O_2$ , is monoclinic, space group  $Pc$ ,  $a=3.888(3)$ ,  $b=8.641(3)$ ,  $c=17.944(1)$  Å,  $\beta=92.63(6)^\circ$ ,  $V=602(2)$  Å<sup>3</sup>,  $Z=2$ ,  $D_c=2.04$  g.cm<sup>-3</sup>,  $F(000)=362$ ,  $\mu(MoK\alpha)=51.0$  cm<sup>-1</sup>.  
 Complex II,  $C_{14}H_{16}CuN_3O_6$ , is triclinic, space group  $P\bar{1}$ ,  $a=7.416(2)$ ,  $b=8.632(4)$ ,  $c=13.034(3)$  Å,  $\alpha=74.55(3)$ ,  $\beta=84.84(3)$ ,  $\gamma=81.04(5)^\circ$ ,  $V=793(1)$  Å<sup>3</sup>,  $Z=2$ ,  $D_c=1.61$  g.cm<sup>-3</sup>,  $F(000)=39.4$ ,  $\mu(MoK\alpha)=14.1$  cm<sup>-1</sup>.

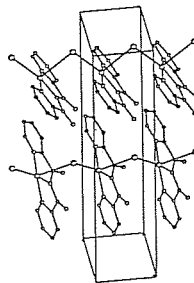


Figure 1.

$[Cu(BPCA)Br]$  units with the bromine atoms bridging two copper(II) ions. The copper atom environment can be described as a distorted square pyramid. Basal positions are occupied by the three nitrogen atoms from BPCA ligand and the bromine atom, while the apical site is occupied by another bromine atom ( $Br'$ ) belonging to an adjacent molecule. Basal atoms are coplanar ( $\pm 0.08$  Å) and the copper atom lies 0.19 Å above this plane.

Figure 2 shows a perspective view of complex II. As occurs in I, the copper(II) environment is close to square pyramidal. The nitrogen atoms from BPCA and one oxygen atom from the monodentate acetate anion occupy the basal positions. One of the two water molecules is coordinated to Cu(II) ion in the apical site. The second water molecule is hydrogen-bonded to the non-coordinated oxygen atoms of two acetate groups. These last belong to two different molecules which are related through a symmetry center, resulting in this way "pseudo-dimeric"

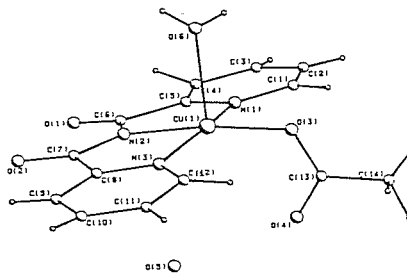
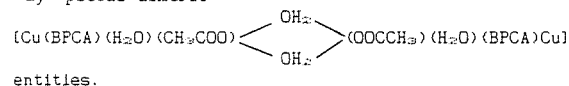


Figure 2.