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## The Crystal Structure of Monoaquabis(2,2'-bipyridyl)copper(II) Dithionate

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### Abstract

Monoaquabis(2,2'-bipyridyl)copper(II) dithionate,  $[\text{Cu}(\text{C}_{10}\text{H}_8\text{N}_2)_2(\text{H}_2\text{O})]\text{S}_2\text{O}_6$ ,  $\text{C}_{20}\text{H}_{18}\text{CuN}_4\text{O}^{2+} \cdot \text{O}_6\text{S}_2^{2-}$ ,  $M_r = 554.06$ , monoclinic,  $C2/c$ ,  $a = 8.03$  (5),  $b = 16.99$  (5),  $c = 15.73$  (5) Å,  $\beta = 103.5$  (5)°,  $Z = 4$ . The structure was refined to  $R = 0.0659$  and involves  $[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]^{2+}$  and  $\text{S}_2\text{O}_6^{2-}$  ions. The former involves a distorted trigonal-bipyramidal  $\text{CuN}_4\text{O}$  chromophore with the Cu—O bond lying along the crystallographic twofold axis, and is only the second reported example of the  $[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]^{2+}$  ion in the solid state. The  $\text{S}_2\text{O}_6^{2-}$  ion has a staggered configuration, the twofold axis bisecting the S—S bond.

### Introduction

As part of a programme to investigate the stereochemistry of the polythionate anions,  $\text{S}_n\text{O}_6^{2-n}$ , and their ability to determine the stereochemistry of the  $[\text{Cu}(\text{bpy})_2]^{2+}$  ion (bpy = 2,2'-bipyridyl) (Harrison, Hathaway & Kennedy, 1979) the crystal structure of  $[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]\text{S}_2\text{O}_6$  has been determined.

### Experimental

$[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]\text{S}_2\text{O}_6$  was prepared by dissolving basic copper carbonate in aqueous dithionic acid containing the stoichiometric amount of 2,2'-bipyridyl and the solution boiled to expel carbon dioxide. Dithionic acid was prepared by adding dilute sulphuric acid to barium thionate solution and removing the precipitated  $\text{BaSO}_4$  by filtration. Turquoise blue crystals were deposited and recrystallized from water. [Composition: found: C 43.34, H 3.25, N 10.15, Cu 11.20%; calculated for  $\text{C}_{20}\text{H}_{18}\text{CuN}_4\text{O}_7\text{S}_2$ : C 43.35, H 3.27, N 10.15, Cu 11.46%.]

### Crystal data

$\text{C}_{20}\text{H}_{18}\text{CuN}_4\text{O}^{2+} \cdot \text{O}_6\text{S}_2^{2-}$ , monoclinic,  $C2/c$ ,  $a = 8.03$  (5),  $b = 16.99$  (5),  $c = 15.73$  (5) Å,  $\beta = 103.5$  (5)°,  $M_r = 554.06$ ,  $Z = 4$ ,  $D_m = 1.80$ ,  $D_c = 1.765$  Mg m<sup>-3</sup>,  $U = 2085.95$  Å<sup>3</sup>,  $F(000) = 1132$ ,  $\mu = 3.634$  mm<sup>-1</sup> (Cu  $K\alpha$  radiation,  $\lambda = 1.5418$  Å). The cell parameters were determined from precession photographs and the data collected photographically by the equi-inclination Weissenberg technique. Five-film packs were used for each layer and the intensities estimated by the Science Research Council Microdensitometer

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Service (Rutherford Laboratory, Harwell). Layers  $hk0-9$  were collected and yielded 1031 unique reflections. Lorentz and polarization corrections were applied, but no correction was made for absorption. Complex atomic scattering factors were employed (Cromer & Waber, 1965; Cromer & Liberman, 1970).

### Structure solution and refinement

The structure was solved by Patterson and Fourier techniques with *SHELX* 76 (Sheldrick, 1976) on an IBM 370/138 computer, and refined by full-matrix least squares with anisotropic temperature factors for all non-hydrogen atoms. The positions of the H atoms were calculated geometrically, except for the water molecule, and floated on the adjacent C atoms with  $C-H = 1.08 \text{ \AA}$  and a fixed temperature factor of  $0.07 \text{ \AA}^2$ . The refinement converged when the maximum shift/e.s.d. was  $<0.03$  with a weighting scheme  $w = 1.0/[\sigma^2(F_o) + 0.01319(F_o)^2]$ . The maximum residual electron density was  $1.08 \text{ e \AA}^{-3}$ . The final non-

Table 1. Atom coordinates ( $\times 10^4$ ) with e.s.d.'s in parentheses

	x	y	z
Cu	5000	5804 (1)	2500
O(1)	5000	4534 (8)	2500
N(1)	5932 (9)	5749 (5)	1444 (4)
C(1)	7403 (14)	5423 (6)	1395 (6)
C(2)	7942 (14)	5355 (6)	620 (6)
C(3)	6869 (15)	5683 (6)	-131 (6)
C(4)	5377 (14)	6010 (6)	-91 (5)
C(5)	4874 (13)	6019 (5)	704 (5)
C(6)	3201 (12)	6329 (5)	811 (4)
C(7)	1924 (14)	6620 (6)	138 (5)
C(8)	471 (14)	6927 (6)	319 (6)
C(9)	311 (13)	6973 (6)	1178 (6)
C(10)	1604 (13)	6645 (6)	1808 (5)
N(2)	3028 (10)	6307 (4)	1644 (4)
S(1)	1091 (3)	3474 (2)	2215 (1)
O(2)	877 (10)	4181 (5)	1689 (5)
O(3)	1010 (11)	2744 (5)	1732 (4)
O(4)	2535 (10)	3512 (5)	2961 (4)

Table 2. Bond lengths ( $\text{\AA}$ ) with e.s.d.'s in parentheses

O(1)-Cu	2.158 (15)	N(1)-Cu	1.977 (9)
N(2)-Cu	2.013 (9)	C(1)-N(1)	1.322 (14)
C(5)-N(1)	1.351 (11)	C(2)-C(1)	1.390 (13)
C(3)-C(2)	1.406 (15)	C(4)-C(3)	1.335 (17)
C(5)-C(4)	1.401 (12)	C(6)-C(5)	1.488 (14)
C(7)-C(6)	1.381 (13)	N(2)-C(6)	1.350 (10)
C(8)-C(7)	1.367 (15)	C(9)-C(8)	1.389 (14)
C(10)-C(9)	1.375 (14)	N(2)-C(10)	1.358 (14)
O(2)-S(1)	1.446 (8)	O(3)-S(1)	1.448 (9)
O(4)-S(1)	1.445 (8)	S(1)-S(1')	2.146 (10)

Table 3. Bond angles ( $^\circ$ ) with e.s.d.'s in parentheses

N(1)-Cu-O(1)	87.3 (3)	N(2)-Cu-O(1)	115.1 (3)
N(2)-Cu-N(1)	81.4 (4)	C(1)-N(1)-Cu	125.9 (7)
C(5)-N(1)-Cu	115.0 (7)	C(5)-N(1)-C(1)	118.9 (8)
C(2)-C(1)-N(1)	123.4 (10)	C(3)-C(2)-C(1)	116.7 (10)
C(4)-C(3)-C(2)	120.4 (9)	C(5)-C(4)-C(3)	119.7 (10)
C(4)-C(5)-N(1)	120.7 (10)	C(6)-C(5)-N(1)	115.2 (8)
C(6)-C(5)-C(4)	124.1 (9)	C(7)-C(6)-C(5)	124.6 (8)
N(2)-C(6)-C(5)	113.6 (8)	N(2)-C(6)-C(7)	121.8 (10)
C(8)-C(7)-C(6)	119.6 (9)	C(9)-C(8)-C(7)	120.1 (10)
C(10)-C(9)-C(8)	116.9 (10)	N(2)-C(10)-C(9)	124.2 (9)
C(6)-N(2)-Cu	114.7 (7)	C(10)-N(2)-Cu	128.2 (6)
C(10)-N(2)-C(6)	117.1 (8)	O(3)-S(1)-O(2)	115.3 (5)
O(4)-S(1)-O(2)	113.1 (6)	O(4)-S(1)-O(3)	113.2 (6)
O(2)-S(1)-S(1')	103.8 (9)	O(4)-S(1)-S(1')	103.8 (9)
O(3)-S(1)-S(1')	106.2 (9)	N(1)-Cu-N(3)	174.6 (5)
N(2)-Cu-N(4)	129.8 (5)	N(1)-Cu-N(2')	100.9 (5)

Table 4. The equations of the least-squares planes in the form  $lX + mY + nZ = p$ , where  $X$ ,  $Y$ , and  $Z$  are orthogonal axes, with deviations ( $\text{\AA}$ ) of relevant atoms from the planes (e.s.d.'s are about  $0.013 \text{ \AA}$ )

	l	m	n	p
Plane (1): N(1), C(1)-C(5)	3.3338	15.1139	1.3873	10.8494
(r.m.s.d. = $0.0187 \text{ \AA}$ )				
[C(2) $-0.0228$ , C(5) $-0.0293$ ]				
Plane (2): C(6)-C(10), N(2)	3.4488	15.1790	0.4353	10.7172
(r.m.s.d. = $0.0225 \text{ \AA}$ )				
[C(6) $0.0280$ , C(8) $-0.0274$ , C(9) $0.0260$ ]				
Plane (3): N(1), C(1)-C(10), N(2)	3.1888	15.3389	1.0637	10.8516
(r.m.s.d. = $0.0462$ )				
[C(4) $0.0722$ , C(7) $-0.0693$ , C(9) $0.0691$ ]				

Angle between planes 1 and 2:  $3.5 (3)^\circ$ .

hydrogen atom coordinates are listed in Table 1.\* Table 2 gives the bond lengths, Table 3 the bond angles and Table 4 some mean planes. Fig. 1 illustrates the structure and atom numbering.

### Discussion

$[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]\text{S}_2\text{O}_6$  is made up of discrete  $[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]^{2+}$  and  $\text{S}_2\text{O}_6^{2-}$  ions. The cation involves a five-coordinate  $\text{CuN}_4\text{O}$  chromophore with the water molecule occupying a definite coordination site on the Cu atom (Fig. 1). The Cu and O atoms occupy twofold special positions such that the cation has

\* Lists of structure factors, calculated H-atom positions and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34717 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

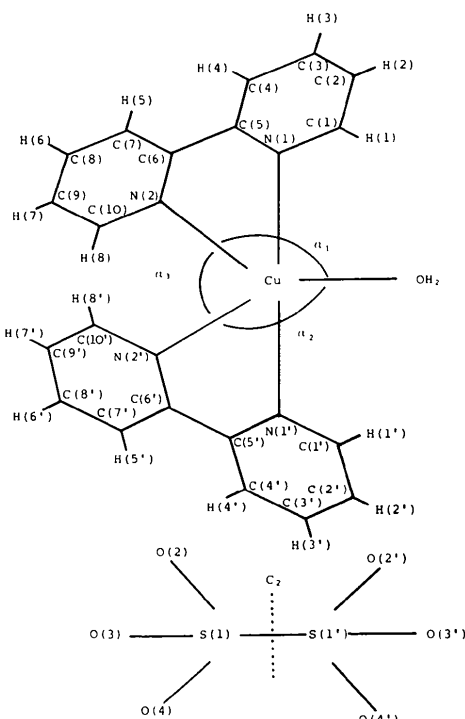


Fig. 1. The structure of the  $[\text{Cu}(\text{bpy})_2(\text{OH}_2)]^{2+}$  cation and  $\text{S}_2\text{O}_6^{2-}$  anion and the atom numbering.

crystallographic  $C_2$  symmetry. The stereochemistry is best described as trigonal pyramidal, with all four Cu–N distances comparable (mean 1.995 Å) (Table 2), but with a small but significant elongation in the Cu–O distance of 2.158 Å. The three in-plane bond angles  $\alpha_{1-3}$  (Fig. 1) are distorted from the  $120^\circ$  of a regular trigonal-bipyramidal stereochemistry:  $\alpha_3 = 129.8^\circ$  and  $\alpha_1 = \alpha_2 = 115.1^\circ$ , which suggests that the distortion is towards a square-based pyramidal stereochemistry, the Cu–O direction forming the elongation direction and the four 2,2'-bipyridyl N atoms the basal plane.

There are no unusual bond lengths or angles in the bpy ligands; the pyridine rings of bpy are reasonably planar and are inclined at an angle of  $3.48^\circ$ , within the range ( $0$ – $11^\circ$ ) normally observed for coordinated bpy (Stephens, 1969, 1972).

The  $\text{S}_2\text{O}_6^{2-}$  ion has the staggered configuration with  $\text{S}–\text{S} = 2.146$  Å, bisected by the twofold axis such that O(2), S(1), S(1'), O(2') all lie in the plane at right angles to the twofold axis (Fig. 1). All the S–O distances are comparable at *ca* 1.45 Å and the angles at the S atom are reasonably tetrahedral, in agreement with results previously reported for the staggered  $\text{S}_2\text{O}_6^{2-}$  ion in  $\text{Na}_2\text{S}_2\text{O}_6 \cdot 2\text{H}_2\text{O}$  (Martinez, García-Blanco & Rivoir, 1956) and  $\text{K}_2\text{S}_2\text{O}_6$  (Stanley, 1956) (Table 5).

Table 5. Structural data for the  $\text{S}_2\text{O}_6^{2-}$  ion

	$[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]\text{S}_2\text{O}_6$	$\text{Na}_2\text{S}_2\text{O}_6 \cdot 2\text{H}_2\text{O}$	$\text{K}_2\text{S}_2\text{O}_6$
S–S	2.146 (5)	2.16 (2)	2.15 (2)
S–O	1.446 (9)	1.45 (5)	1.43 (5)

The cation of  $[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]\text{S}_2\text{O}_6$  is only the second example of a hydrated  $[\text{Cu}(\text{bpy})_2]^{2+}$  ion; the latter has previously been considered to have a six-coordinate  $\text{CuN}_4\text{O}_2$  chromophore with a *cis* distorted octahedral stereochemistry (Jörgenson, 1955), a stereochemistry that has even been justified in the literature (Sigel, 1972) despite the lack of any crystallographic evidence for the  $[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]^{2+}$  ion in the solid state. While several hydrated  $[\text{Cu}(\text{bpy})_2]XY \cdot n\text{H}_2\text{O}$  complexes have been reported (Harris, Lockyer & Waterman, 1961; McKenzie, 1962; Hathaway, Procter, Slade & Tomlinson, 1969), these generally involve uncoordinated water in the unit cell. Recently there has been crystallographic evidence for the coordination of a water molecule to the  $[\text{Cu}(\text{bpy})_2]^{2+}$  ion in  $[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]\text{S}_2\text{O}_6$  (Harrison, Hathaway & Kennedy, 1979), a monoaqua complex rather than the diaqua complex as suggested in aqueous solution. Table 6 summarizes the structural data for the two monoaqua complexes of the  $[\text{Cu}(\text{bpy})_2]^{2+}$  ion and for the two complexes  $[\text{Cu}(\text{H}_2\text{O})(\text{phen})_2](\text{NO}_3)_2$  (Nakai & Deguchi, 1975) and  $[\text{Cu}(\text{H}_2\text{O})(\text{phen})_2](\text{BF}_4)_2$  (Nakai & Noda, 1978) (phen = 1,10-phenanthroline).

In (II) the stereochemistry of the  $\text{CuN}_4\text{O}$  chromophore is significantly different from that in (I). Firstly the chromophore does not have  $C_2$  symmetry and, secondly, the Cu–ligand bond lengths are more nearly equivalent but the in-plane angles are more distorted. Thus the Cu–O distance is more normal at 2.05 Å, the angle  $\alpha_3 = 111.4^\circ$  (*i.e.*  $< 120^\circ$ ) while  $\alpha_2 - \alpha_1 = 38.7^\circ$ . This suggests that the chromophore stereochemistry of (II) is better described as distorted square-based pyramidal in view of the large value of  $\alpha_2$  ( $143.6^\circ$ ) and the length of the Cu–N(2) bond, 2.125

Table 6. The Cu–X bond lengths (Å) and angles ( $^\circ$ )

	(I)	(II)	(III)	(IV)
(I) $[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]\text{S}_2\text{O}_6$			(III) $[\text{Cu}(\text{H}_2\text{O})(\text{phen})_2](\text{NO}_3)_2$	
(II) $[\text{Cu}(\text{bpy})_2(\text{H}_2\text{O})]\text{S}_2\text{O}_6$			(IV) $[\text{Cu}(\text{H}_2\text{O})(\text{phen})_2](\text{BF}_4)_2$	
Cu–N(1)	1.977 (7)	1.980 (5)	1.99 (1)	1.985 (6)
Cu–N(2)	2.013 (7)	2.125 (6)	2.03 (1)	2.041 (7)
Cu–N(3)	1.977 (7)	1.974 (5)	1.99 (1)	1.985 (6)
Cu–N(4)	2.013 (7)	2.025 (5)	2.03 (1)	2.041 (7)
Cu–OH <sub>2</sub>	2.158 (13)	2.050 (5)	2.18 (1)	2.238 (8)
$\alpha_1$	115.1 (3)	104.9 (2)	110.0 (4)	111.7 (3)
$\alpha_2$	115.1 (3)	143.6 (1)	110.0 (4)	111.7 (3)
$\alpha_3$	129.8 (6)	111.4 (3)	139.6 (5)	136.6 (3)
$\alpha_4$	3.48	5.29	0	0
$\alpha_5$	3.48	4.88	0	0

Table 7. *The electronic reflectance spectra ( $\mu\text{m}^{-1}$ ) and single-crystal  $g$  factors of (I), (II), (III) and (IV)*

	(I)	(II)	(III)	(IV)
Electronic spectra	1-245	1-075, 1-412	1-25	1-34
Single-crystal $g$ factors	2-011 2-158 2-225	2-028 2-164 2-220	2-022 2-125 2-227	2-021 2-122 2-264

Å. This distorted stereochemistry of the  $\text{CuN}_4\text{O}$  chromophore of (II) is in marked contrast to that in (I) and to that in (III) and (IV) which both retain the crystallographic  $C_2$  symmetry of (I), but with an increase in the value of  $\alpha_3$  to  $139.6$  and  $136.6^\circ$  respectively, and a corresponding elongation of the Cu—O distance, especially in (IV), to  $2.238$  Å.

In view of the comparable bonding role of the chelate N ligands in these four complexes there appears to be a correlation between the angles of twist ( $\alpha_4$  and  $\alpha_5$ , Table 6) of the pyridine rings in the N chelate ligands and the angular distortion at the Cu atom, particularly ( $\alpha_2 - \alpha_1$ ). In phen this must be zero in (I) and (II). It is (I) that has the lower values of  $\alpha_4$  and  $\alpha_5$ ; while this difference is small it suggests that the more distorted chromophore, *i.e.* large values of ( $\alpha_2 - \alpha_1$ ), are associated with the larger values of twist of the bpy rings and would suggest that the  $[\text{Cu}(\text{phen})_2]XY$  complexes are likely to have a more regular trigonal-bipyramidal stereochemistry than the corresponding  $[\text{Cu}(\text{bpy})_2]XY$  complexes. This prediction is currently being examined from both a crystallographic and a spectroscopic point of view.

The similarity of the chromophore stereochemistries of (I), (III) and (IV) extends to the electronic reflectance spectra, for which a single broad peak is observed (Table 7); however, for (II) a double peak is observed with  $\Delta E = 0.337 \mu\text{m}^{-1}$ . This suggests that the electronic reflectance spectra may be used to distinguish the local molecular stereochemistry of these two cation distortion isomers, (I) and (II) (Ray, Hulett, Sheahan & Hathaway, 1978). The significant elongation of the Cu—OH<sub>2</sub> distance in (I), (III) and (IV) with respect to that in (II) also relates to the direction

of the highest local molecular  $g$  factor (Table 7) which lies along the Cu—OH<sub>2</sub> direction in the former complexes (Bencini & Gatteschi, 1977) while in (II) it is the intermediate  $g$  factor that lies along the (shorter) Cu—OH<sub>2</sub> bond direction.

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