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Structure of Bis(2,2′-bipyridyl)(saccharinato-*N*)copper(II) Saccharinate Dihydrate*

BY A. HERGOLD-BRUNDIĆ

Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, PO Box 153, 41001 *Zagreb, Yugoslavia*

AND O. GRUPČE AND G. JOVANOVSKI

Insitute of Chemistry, Faculty of Science, Cyril and Methodius University, Skopje, Yugoslavia

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Abstract. $[Cu(C_7H_4NO_3S)(C_{10}H_8N_2)_2][C_7H_4NO_3S].2H_2O$, $M_r = 776{\cdot}30$, orthorhombic, $C222_1$, $a = 26{\cdot}300\ (6)$, $b = 15{\cdot}596\ (2)$, $c = 16{\cdot}936\ (4)$ Å, $V = 6947\ (2)$ Å^3, $Z = 8$, D_m (by flotation) $= 1{\cdot}51$, $D_x = 1{\cdot}485$ Mg m^{-3}, λ(Cu $K\alpha$) $= 1{\cdot}54178$ Å, $\mu = 2{\cdot}48$ mm^{-1}, $F(000) = 3192$, room temperature, final $R = 0{\cdot}063$ for 2999 independent reflections with $I > 4\sigma(I)$. The structure is built up of $[Cu(C_7H_4NO_3S)(C_{10}H_8N_2)_2]^+$ cations, saccharinate anions and water molecules. The Cu atom is surrounded by five N atoms in a distorted trigonal bipyramid with Cu—N distances ranging from 1·982 (5) to 2·098 (5) Å.

Experimental. $[Cu(sac)(bpy)_2]sac.2H_2O$ (sac = $C_7H_4NO_3S$, bpy = $C_{10}H_8N_2$) was prepared by successive addition of an equimolar quantity of copper(II) acetate to a warm mixture of an aqueous solution of saccharin and 2,2′-bipyridyl. Blue prismatic single crystal of dimensions 0·09 × 0·48 × 0·75 mm, coated with thin layer of nail polish. Intensities of 3093 independent reflections were measured on a Philips PW 1100 diffractometer, graphite monochromator, Cu $K\alpha$ radiation. Cell constants determined by least-squares fit of 16 reflections in the range $15{\cdot}50 \le \theta \le 18{\cdot}94°$. The intensity data of 3093 reflections were measured in the hkl range 0–32, 0–18, 0–20 and θ limits $3 \le \theta \le 70°$ from ω scans. Three standard reflections measured every 2 h showed 9·6% intensity decrease. Absorption and extinction corrections were not applied, but 12 low-angle reflections were ignored during refinement. 2999 unique reflections [$I \ge 4\sigma(I)$]. Structure was solved by Patterson and Fourier methods and refined by full-matrix least-squares procedure (on F) using anisotropic thermal parameters for all non-H atoms and a common isotropic one for H atoms. The model was refined in three blocks containing 119, 138 and 227 parameters, respectively. All H atoms were included at their calculated positions, except those of water. The final conventional agreement factors were $R = 0{\cdot}063$, $wR = 0{\cdot}083$, $w^{-1} = \sigma^2(F) + 0{\cdot}01278F^2$, $S = 0{\cdot}704$; $(\Delta/\sigma)_{max} = 0{\cdot}09$. Residual electron density in final difference map between 1·03 and −0·72 e Å^{-3}, near the Cu atom. Atomic scattering factors were those stored in *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1987) package which were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). A view of the structure, based atomic coordinates in Table 1,† is given in Fig. 1. Fig. 1. Table 2 lists the Cu—N bond lengths and bond angles at the Cu atom.

Related literature. This structural investigation was undertaken as part of our investigations on the ligation properties of saccharin. The distorted trigonal bipyramidal coordination of the Cu atom is realized by one saccharinate N atom and four bipyridyl N atoms from two bipyridyl ligands, as was observed in the closely related structures of $[Cu(bpy)_2(NH_3)][BF_4]_2$ (Stephens, 1972), $[Cu(bpy)_2(NCS)][BF_4]$ (Tyagi & Hathaway, 1981), $[Cu(bpy)_2(NCS)]NO_3.H_2O$ (Manríques, Brito, Andrade, Wittke, von Schnering & Peters, 1988). There are no unusual bond lengths or bond angles within the bipyridyl ligands and saccharinate ions. The crystal packing is achieved with the pairs of the symmetrically related saccharinate anions linked with water molecules by the hydrogen bonds of type O*w*—H···O (2·828, 2·873 Å) and O*w*—H···N (2·969 Å).

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* Saccharin is 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide.

† Lists of bond lengths and angles, atomic coordinates of H atoms, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54320 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates of non-H atoms* ($\times 10^4$) ($\times 10^5$ *for* Cu) *and equivalent isotropic thermal parameters* ($\times 10^4$) *with e.s.d.'s in parentheses*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq} (Å^2)
Cu	22269 (3)	18733 (5)	21683 (5)	425 (2)
N11	2361 (2)	2668 (2)	1203 (3)	418 (14)
N12	1562 (2)	2473 (3)	2108 (3)	422 (12)
C1	2780 (2)	2724 (4)	768 (4)	505 (18)
C2	2825 (2)	3315 (5)	160 (4)	588 (21)
C3	2431 (2)	3852 (4)	−4 (4)	530 (18)
C4	1992 (2)	3809 (4)	446 (3)	463 (16)
C5	1968 (2)	3212 (3)	1045 (3)	393 (14)
C6	1523 (2)	3102 (4)	1571 (3)	401 (14)
C7	1092 (2)	3611 (4)	1523 (4)	514 (19)
C8	689 (2)	3448 (4)	2036 (4)	533 (17)
C9	739 (2)	2808 (5)	2588 (4)	570 (20)
C10	1177 (2)	2323 (5)	2615 (4)	555 (20)
N21	2490 (2)	2578 (3)	3141 (3)	474 (14)
N22	2917 (2)	1379 (3)	2287 (3)	429 (14)
C21	2274 (3)	3230 (5)	3527 (4)	629 (22)
C22	2495 (3)	3655 (5)	4141 (4)	661 (24)
C23	2971 (3)	3402 (5)	4393 (5)	698 (26)
C24	3212 (3)	2731 (5)	4004 (4)	566 (19)
C25	2960 (2)	2345 (4)	3383 (3)	433 (15)
C26	3198 (2)	1650 (4)	2912 (3)	439 (15)
C27	3667 (2)	1272 (5)	3071 (5)	607 (22)
C28	3850 (3)	652 (4)	2581 (6)	705 (26)
C29	3577 (2)	375 (5)	1946 (5)	636 (21)
C210	3098 (2)	757 (4)	1834 (4)	556 (19)
S2	1878 (1)	103 (1)	2956 (1)	468 (4)
N2	1885 (2)	706 (3)	2169 (3)	466 (13)
O21	2350 (2)	−346 (3)	3069 (3)	713 (15)
O22	1701 (2)	600 (3)	3620 (3)	678 (17)
O23	1507 (2)	780 (4)	953 (3)	725 (18)
C31	1398 (2)	−591 (4)	2612 (3)	471 (15)
C32	1170 (3)	−1287 (5)	2995 (5)	667 (23)
C33	799 (3)	−1720 (4)	2572 (6)	776 (26)
C34	681 (3)	−1478 (5)	1822 (6)	821 (31)
C35	910 (2)	−806 (5)	1436 (5)	662 (21)
C36	1275 (2)	−348 (4)	1851 (3)	472 (16)
C37	1568 (2)	429 (4)	1601 (3)	454 (15)
Ow1	1638 (3)	5000 (0)	5000 (0)	783 (26)
Ow2	−971 (2)	5000 (0)	5000 (0)	796 (28)
Ow3	192 (3)	4785 (5)	3699 (4)	1043 (29)
S1	729 (1)	3086 (1)	5191 (1)	600 (5)
N1	213 (2)	3563 (4)	5429 (4)	721 (19)
O11	999 (2)	3570 (4)	4601 (4)	863 (21)
O12	1027 (2)	2863 (5)	5862 (4)	907 (23)
O13	−650 (2)	3302 (5)	5309 (4)	877 (21)
C11	449 (2)	2170 (4)	4750 (3)	519 (16)
C12	679 (3)	1478 (5)	4397 (5)	719 (25)
C13	355 (4)	883 (7)	4070 (7)	982 (41)
C14	−162 (4)	996 (7)	4050 (7)	1036 (44)
C15	−382 (3)	1713 (6)	4421 (6)	800 (30)
C16	−58 (2)	2290 (4)	4766 (4)	550 (19)
C17	−203 (2)	3118 (5)	5208 (4)	628 (22)

Table 2. *Selected interatomic distances* (Å) *and angles* (°)

Cu—N11	2·082 (5)	Cu—N22	1·982 (5)
Cu—N12	1·986 (5)	Cu—N2	2·030 (5)
Cu—N21	2·098 (5)		
N22—Cu—N2	93·3 (2)	N12—Cu—N21	94·8 (2)
N21—Cu—N2	128·0 (2)	N11—Cu—N2	127·6 (2)
N21—Cu—N22	79·8 (2)	N11—Cu—N22	99·0 (2)
N12—Cu—N2	91·9 (2)	N11—Cu—N21	104·4 (2)
N12—Cu—N22	174·1 (2)	N11—Cu—N12	80·1 (2)

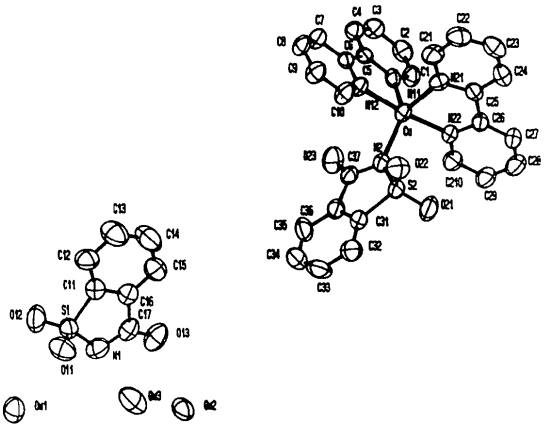

Fig. 1. View of the asymmetric unit showing atom numbering. Thermal ellipsoids are drawn at the 50% probability level.

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Bis(tetramethylphosphonium) Hexa-μ-bromo-tetrabromotetracuprate(II)

BY KRISTOPHER MURRAY AND ROGER D. WILLETT

Department of Chemistry, Washington State University, Pullman, Washington 99164-4630, *USA*

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Abstract. $2C_4H_{12}P^+.Br_{10}Cu_4^{2-}$, $M_r = 1235.5$, monoclinic, $P2_1/c$, $a = 6.416$ (2), $b = 20.214$ (6), $c = 11.236$ (3) Å, $\beta = 98.52$ (2)°, $V = 1455.4$ (7) Å^3, $Z = 2$, $D_x = 2.82$ Mg m^{-3}, $\mu = 16.6$ mm^{-1}, λ(Mo $K\alpha$) = 0.71069 Å, $F(000) = 1136$, $T = 295$ K, $R = 0.039$ for 1596 unique observed [$|F| \geq 3\sigma(F)$] reflections. The

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