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The Structures of two Binuclear Copper(II) Complexes with μ-Oxamido-bridges; $[Cu_2(C_2H_2N_2O_2)(C_{10}H_9N_3)_2(NO_3)_2]$ and $[Cu_2(C_2H_2N_2O_2)(C_{10}H_8N_2)_2(NO_3)_2]\cdot 3H_2O$

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The structures of (μ-oxamido-bis-2,2′-dipyridylamine dinitrate) dicopper(II), compound I, and (μ-oxamido-bis-2,2′-bipyridyl dinitrate) dicopper(II) trihydrate, compound II, have been determined by X-ray crystallographic methods. The crystals are monoclinic, space group $P2_1/n$, $a=8.136(2)$, $b=8.889(1)$, $c=17.296(2)$ Å, $\beta=92.19(1)°$; and triclinic, space group $P\bar{1}$, $a=7.426(1)$, $b=9.353(2)$, $c=9.979(2)$ Å, $\alpha=83.49(2)$, $\beta=87.61(2)$, $\gamma=83.94(2)°$, for I and II, respectively.

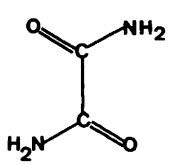
In both compounds the oxamido group in *trans* conformation is bridging two copper ions, forming five-membered chelate rings. The coordination in II is distorted square pyramidal with oxamide and pyridyl in the equatorial plane and a nitrate in apex position. In I the distortion from the square pyramide is appreciable; the geometry being better described as intermediate between square pyramidal and trigonal bipyramidal.

Oxamide (oxalic acid diamide, A) acts as a bidentate ligand and may coordinate to the metal ion through both nitrogen atoms, both oxygen atoms, or one nitrogen and one oxygen atom. Based on IR-studies it has been concluded that monomeric bis-oxamido complexes of Cu and Ni have a square planar diimido structure, with the oxamide in *cis* conformation and coordinating through both nitrogen atoms.[1]

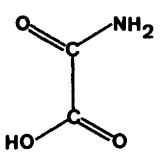
The oxamido ion would be expected to bridge metal ions to form binuclear complexes analogous to those formed by oxalate.[2] In such a dimer the oxamide bridge is expected to be in the *trans* conformation rather than in the *cis* conformation proposed for the monomer.

Recently a report on the syntheses of a series of binuclear copper complexes with μ-oxamido and μ-oxamato bridges appeared.[3] These complexes have subnormal magnetic moments at room temperature, suggesting the presence of magnetic interaction between copper ions. The magnetic exchange interaction is supposed to be propagated by the μ-oxamido and μ-oxamato groups in the same way as proposed for μ-oxalato complexes.[4] The magnetic moment data indicate that for analogous complexes the magnetic interaction between copper atoms decreases in the order μ-oxamido > μ-oxamato > μ-oxalato.[3]

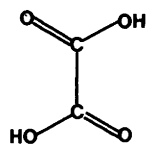
In this paper the results of structure determinations of two μ-oxamido Cu(II) complexes are reported. Investigations of corresponding oxamato and oxalato compounds are in progress.



A
oxamide



B
oxamic acid



C
oxalic acid

0302-4377/82/040345-07$02.50

EXPERIMENTAL

Both compounds were synthesized according to the procedure described by Nonoyama *et al.*[3] Of compound I tiny, light blue needles grown from water were not found suitable for X-ray investigation. From a methanol solution a mixture of two different crystal modifications were obtained, one with the same appearance as those grown from water, and a second one crystallizing as bright green needles. The latter type were used in the X-ray work. From a water solution crystals of II grew as dark turquoise plates which fractured easily to platelets of minute thickness. The crystals selected for data collection had dimensions $0.25 \times 0.07 \times 0.06$ mm and $0.18 \times 0.64 \times 0.10$ mm for I and II, respectively. Data were recorded on an Enraf-Nonius CAD-4 diffractometer using monochromatized Mo$K\alpha$ radiation. For I preliminary scans and $\Delta\theta/\Delta\omega$ plots indicated that the $\theta-2\theta$ scan technique should be used, with scan widths $\Delta\theta = 0.70+0.35 \tan\theta$. The scan speeds varied between 0.6 and 4° min^{-1} depending on peak intensity. Crystals of II had poor quality and fractured easily along (001). For the crystal selected the ω-scan technique were found most appropriate. Due to the high mosaic spread rather wide scan angles had to be used; $\Delta\omega = 2.9+0.35 \tan\theta$. Scan speeds were selected at 2° min^{-1}. In both cases data were recorded up to $2\theta = 50°$. Fluctuations in intensities during data collections were monitored remeasuring three reference reflections at regular intervals; variations of ± 3 % were observed and the data were adjusted accordingly. The error in the intensity of any one reflection was estimated as $\sigma_I = [\sigma_c^2 + (0.02N_{net})^2]^{1/2}$. Of the 2205 independent reflections recorded for compound I, 1331 had $I > 1.5\ \sigma_I$ and were used in the structure analysis. For compound II, out of the 2225 reflections recorded, 1907 with $I > 2.0\sigma_I$ were retained. The intensities were corrected for Lorentz and polarization effects, and for absorption; the maximum and minimum transmission factors being 0.92 and 0.87 for I, and 0.85 and 0.78 for II.

CRYSTAL DATA

I. (μ-Oxamido-bis-2,2′-dipyridylamine dinitrate)-dicopper(II), $Cu_2C_{22}H_{20}N_{10}O_8$. Monoclinic, $P2_1/n$ (No. 14),
$a = 8.136(2)$, $b = 8.889(1)$, $c = 17.296(2)$ Å,
$\beta = 92.19(1)°$, $V = 1249.9(7)$ Å^3, $M = 679.55$, $Z = 2$,
$D_m = 1.80$ gcm^{-3}, $D_x = 1.806$ gcm^{-3},
μ(Mo$K\alpha$) = 18.43 cm^{-1}.

II. (μ-Oxamido-bis-2,2′-bipyridyl dinitrate)dicopper(II)trihydrate, $Cu_2C_{22}H_{24}N_8O_{11}$. Triclinic, $P\bar{1}$ (No. 2),
$a = 7.426(1)$, $b = 9.353(2)$, $c = 9.979(2)$ Å,
$\alpha = 83.49(2)$, $\beta = 87.61(2)$, $\gamma = 83.94(2)°$,
$V = 684.5(4)$ Å^3, $M = 703.56$, $Z = 1$, $D_m = 1.71$ gcm^{-3},
$D_x = 1.707$ gcm^{-3}, μ(Mo$K\alpha$) = 16.93 cm^{-1}.

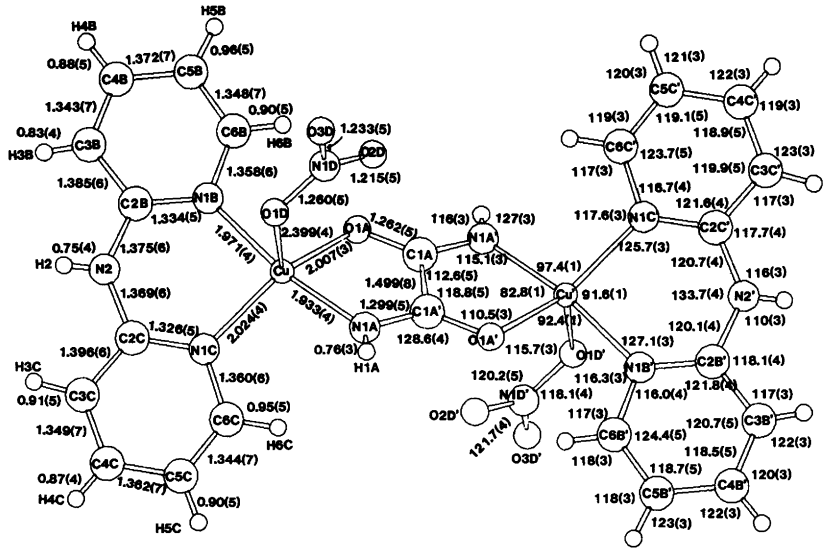


Fig. 1. The dimeric unit of compound I. Bond distances and angles are shown with their corresponding standard deviations, as obtained from the least squares matrix, in parentheses.

STRUCTURE DETERMINATIONS

The structures were both solved by the heavy atom method, and refined by least squares. In compound II one of the water molecules in the dimer is disordered. An attempt was made to refine an ordered model in the non-centrosymmetric space group $P1$, without success; hence the refinement was continued in $P\bar{1}$. It was not possible, however, to find a well-defined location for the last water molecule, which was thus left out of the calculations. In the difference Fourier map a channel of

Table 1. Final atomic parameters of non-hydrogen atoms with estimated standard deviations in parentheses. Anisotropic temperature factor: $\exp[-2\pi^2(U_{11}h^2a^{*2}+\cdots+2U_{23}klb^*c^*)]$.

Atom	x	y	z	$U(1,1)$	$U(2,2)$	$U(3,3)$	$U(1,2)$	$U(1,3)$	$U(2,3)$
Compound I									
Cu	0.17821(7)	0.24601(8)	0.51368(3)	0.0479(3)	0.0361(3)	0.0384(2)	−0.0134(4)	−0.0143(2)	0.0061(4)
O1A	−0.0319(4)	0.1788(3)	0.4597(2)	0.042(2)	0.029(2)	0.042(2)	−0.004(2)	−0.010(2)	0.004(2)
O1D	0.4254(5)	0.1863(4)	0.4489(2)	0.074(2)	0.057(2)	0.047(2)	−0.028(2)	0.013(2)	−0.017(2)
O2D	0.2728(5)	0.0517(5)	0.3722(2)	0.062(2)	0.096(3)	0.068(2)	−0.027(2)	−0.009(2)	−0.015(2)
O3D	0.5299(4)	0.0739(5)	0.3531(2)	0.055(2)	0.112(3)	0.050(2)	0.022(2)	−0.004(2)	−0.014(2)
N1A	0.1772(4)	0.0432(4)	0.5540(2)	0.035(2)	0.042(2)	0.044(2)	−0.004(2)	−0.019(2)	0.009(2)
N1B	0.1820(5)	0.4352(4)	0.4543(2)	0.049(2)	0.042(2)	0.037(2)	−0.011(2)	−0.013(2)	0.009(2)
N1C	0.2872(5)	0.3461(4)	0.6074(2)	0.052(2)	0.034(2)	0.031(2)	−0.009(2)	−0.011(2)	0.004(2)
N2	0.3390(5)	0.5747(4)	0.5455(2)	0.061(2)	0.030(2)	0.038(2)	−0.013(2)	−0.007(2)	0.001(2)
N1D	0.4080(5)	0.1008(4)	0.3910(2)	0.057(2)	0.035(2)	0.040(2)	−0.005(2)	−0.005(2)	0.003(2)
C1A	−0.0579(5)	0.0413(5)	0.4728(2)	0.028(2)	0.031(2)	0.033(2)	−0.002(2)	0.001(2)	−0.000(2)
C2B	0.2634(5)	0.5609(5)	0.4734(3)	0.035(2)	0.035(3)	0.043(3)	0.003(2)	0.004(2)	0.003(2)
C3B	0.2732(6)	0.6808(6)	0.4226(3)	0.058(3)	0.030(2)	0.050(3)	−0.003(3)	0.003(3)	0.005(3)
C4B	0.2021(7)	0.6733(6)	0.3514(3)	0.069(4)	0.052(3)	0.042(3)	0.003(3)	0.003(3)	0.020(3)
C5B	0.1200(7)	0.5440(7)	0.3300(3)	0.071(4)	0.073(4)	0.050(3)	−0.020(3)	−0.023(3)	0.022(3)
C6B	0.1134(7)	0.4306(7)	0.3815(3)	0.086(4)	0.063(4)	0.051(3)	−0.031(3)	−0.028(3)	0.020(3)
C2C	0.3480(5)	0.4845(5)	0.6097(3)	0.032(2)	0.030(2)	0.037(2)	0.000(2)	−0.002(2)	−0.002(2)
C3C	0.4244(6)	0.5429(6)	0.6768(3)	0.059(3)	0.042(3)	0.039(3)	−0.013(3)	−0.001(3)	−0.008(3)
C4C	0.4344(7)	0.4589(6)	0.7418(3)	0.063(3)	0.057(3)	0.043(3)	−0.004(3)	−0.014(3)	−0.015(3)
C5C	0.3672(7)	0.3185(6)	0.7407(3)	0.082(4)	0.055(3)	0.034(3)	−0.000(3)	−0.014(3)	0.007(3)
C6C	0.2949(7)	0.2670(6)	0.6748(3)	0.094(4)	0.039(3)	0.045(3)	−0.016(3)	−0.017(3)	0.010(3)
Compound II									
Cu	0.3334(1)	0.2691(1)	0.4987(1)	0.0451(5)	0.0264(4)	0.0507(6)	−0.0151(4)	−0.0004(4)	−0.0047(4)
O1A	0.3810(8)	0.4391(6)	0.3733(6)	0.062(3)	0.043(3)	0.049(4)	−0.023(2)	−0.010(3)	−0.002(3)
O1D	0.0471(9)	0.3607(7)	0.5703(8)	0.066(4)	0.050(3)	0.087(5)	−0.007(3)	0.008(4)	−0.027(3)
O2D	0.0678(25)	0.5227(14)	0.7203(24)	0.22(2)	0.101(8)	0.48(3)	−0.036(9)	0.01(2)	−0.11(1)
O3D	0.0252(25)	0.3218(14)	0.8174(16)	0.35(2)	0.128(8)	0.19(1)	−0.112(9)	−0.08(1)	0.081(8)
O1W	0.5566(24)	0.3116(16)	0.9211(11)	0.38(2)	0.21(1)	0.084(6)	−0.10(1)	−0.113(8)	0.079(7)
N1A	0.4737(9)	0.3546(7)	0.6203(7)	0.056(4)	0.034(3)	0.031(4)	−0.018(3)	−0.011(3)	0.006(3)
N1B	0.2467(8)	0.1627(7)	0.3565(7)	0.035(3)	0.032(3)	0.058(4)	−0.010(3)	0.002(3)	−0.007(3)
N1C	0.3153(8)	0.0788(7)	0.6078(7)	0.036(3)	0.031(3)	0.046(4)	−0.007(3)	0.001(3)	−0.004(3)
N1D	0.0539(22)	0.3977(14)	0.6944(31)	0.13(1)	0.060(7)	0.60(4)	−0.024(8)	0.05(2)	−0.07(1)
C1A	0.527(1)	0.4762(8)	0.5702(8)	0.038(4)	0.029(4)	0.041(4)	−0.006(3)	−0.002(4)	−0.005(3)
C2B	0.222(1)	0.0218(8)	0.3977(9)	0.028(4)	0.032(4)	0.050(5)	−0.006(3)	0.002(3)	−0.005(3)
C3B	0.170(1)	−0.0665(8)	0.3072(9)	0.047(4)	0.034(4)	0.062(6)	−0.010(3)	−0.006(4)	−0.013(4)
C4B	0.145(1)	−0.0127(10)	0.1747(10)	0.048(5)	0.059(5)	0.066(6)	−0.012(4)	−0.007(4)	−0.025(4)
C5B	0.169(1)	0.1322(11)	0.1344(10)	0.053(5)	0.068(6)	0.046(5)	−0.010(4)	−0.008(4)	−0.005(5)
C6B	0.222(1)	0.2139(10)	0.2266(10)	0.046(5)	0.055(5)	0.048(5)	−0.011(4)	−0.003(4)	0.003(4)
C2C	0.256(1)	−0.0228(8)	0.5394(9)	0.028(4)	0.030(4)	0.059(5)	−0.006(3)	0.007(4)	−0.010(4)
C3C	0.234(1)	−0.1600(8)	0.6015(10)	0.044(4)	0.030(4)	0.065(6)	−0.005(3)	0.006(4)	−0.004(4)
C4C	0.275(1)	−0.1906(9)	0.7384(11)	0.052(5)	0.037(4)	0.077(7)	−0.004(4)	0.013(5)	0.009(5)
C5C	0.334(1)	−0.0863(10)	0.8082(10)	0.048(5)	0.053(5)	0.051(5)	−0.004(4)	0.005(4)	0.003(4)
C6C	0.351(1)	0.0480(9)	0.7381(10)	0.039(4)	0.046(4)	0.066(6)	−0.012(4)	0.003(4)	−0.008(4)

Table 2. Atomic parameters of hydrogen atoms. Isotropic temperature factor: $\exp[-B \sin^2\theta/\lambda^2]$.

Atom	x	y	z	B
Compound I				
H1A	0.246(4)	0.004(4)	0.577(2)	0.7(7)
H2	0.383(4)	0.648(4)	0.549(2)	1.9(8)
H3B	0.321(4)	0.758(4)	0.439(2)	1.4(7)
H4B	0.213(5)	0.748(5)	0.319(2)	3.6(9)
H5B	0.068(6)	0.530(6)	0.280(3)	6.4(14)
H6B	0.069(5)	0.343(5)	0.366(2)	4.0(11)
H3C	0.474(5)	0.634(5)	0.672(2)	3.8(10)
H4C	0.480(5)	0.497(5)	0.784(2)	3.9(11)
H5C	0.359(6)	0.266(5)	0.785(3)	5.2(12)
H6C	0.255(6)	0.167(5)	0.673(3)	5.0(12)
Compound II				
H1A	0.492(7)	0.348(6)	0.677(6)	0(1)
H3B	0.156(9)	−0.162(8)	0.328(7)	3(2)
H4B	0.104(9)	−0.068(7)	0.115(7)	2(1)
H5B	0.159(11)	0.157(9)	0.043(8)	4(2)
H6B	0.240(14)	0.326(11)	0.201(10)	7(3)
H3C	0.184(13)	−0.223(10)	0.541(10)	6(2)
H4C	0.260(11)	−0.274(9)	0.783(8)	4(2)
H5C	0.378(16)	−0.058(13)	0.913(12)	10(3)
H6C	0.411(15)	0.132(12)	0.748(11)	8(3)

electron density, was found running parallel to the a-axis and close to $y=0.5$, $z=0.0$.

In both structures hydrogen atoms, except those on water in II, were localized by difference Fourier synthesis, and were refined isotropically. For the non-hydrogen atoms anisotropic thermal parameters were used. The weight assigned to each reflection in the refinement is $w=1/\sigma_F^2$, where $\sigma_F=\sigma_I(I\cdot Lp)^{-1/2}$. Final R values are 0.040 and 0.071 for I and II, respectively, weighted R 0.036 and 0.101. Standard deviation of an observation of unit weight 1.30 and 9.30, respectively. The high disagreement factors for II reflect the poor quality of the crystal and the deficiency in the model as described above.

Table 3. Copper-coordination bond angles (°) for compounds I and II.

	Compound I	Compound II
O1D−Cu−O1A	115.5(1)	96.0(1)
O1D−Cu−N1A	88.7(2)	98.3(2)
O1D−Cu−N1B	85.1(2)	95.5(1)
O1D−Cu−N1C	97.0(1)	92.7(1)
O1A−Cu−N1A	82.8(1)	84.0(1)
O1A−Cu−N1B	92.4(1)	94.1(1)
O1A−Cu−N1C	147.4(1)	170.6(1)
N1A−Cu−N1B	169.7(2)	166.2(2)
N1A−Cu−N1C	97.4(1)	98.3(2)
N1B−Cu−N1C	91.6(1)	81.4(1)

Atomic scattering factors used were those of Cromer and Waber.[5] All calculations were carried out on a PDP 11/55 computer using the Enraf-Nonius Structure Determination Programs (SDP).[6]

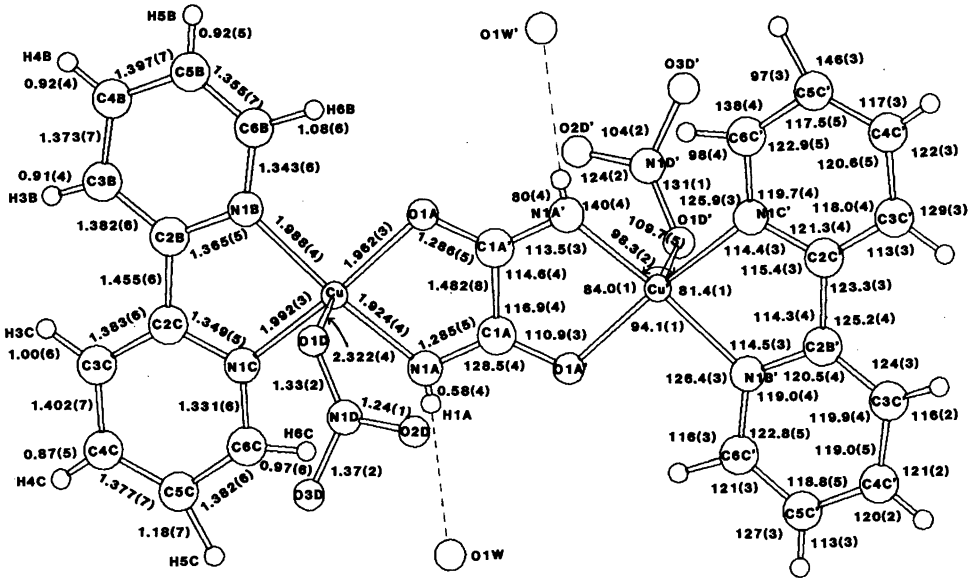


Fig. 2. The dimeric unit of compound II with bond distances and angles.

Final atomic parameters are listed in Tables 1 and 2. ORTEP drawings of the two molecules including atomic numbering scheme, bond lengths and bond angles are shown in Figs. 1 and 2. Bond angles around copper are listed in Table 3.

Lists of structure factors may be obtained from the author.

RESULTS AND DISCUSSION

The coordination geometry. In compound I the coordination geometry is intermediate between square pyramidal and trigonal bipyramidal. If regarding the complex as distorted square pyramidal the oxamido oxygen and nitrogen atoms occupy two equatorial positions and the nitrogen atoms of the pyridyl rings the two remaining equatorial positions. An oxygen atom of the nitrate group. O1D, occupies the apex position at a distance of 2.399 Å. Opposite to O1D the sixth position is blocked by the proximity of a pyrididyl group of a symmetry related molecule, the closest neighbour being C3B^{ii} ($-x$, $1-y$, $1-z$) at a distance of 3.93 Å. A least squares plane through the four equatorial ligands shows the following displacements in I: -0.37, 0.36, 0.33, -0.32 Å for O1A, N1A, N1B, N1C, respectively; and with Cu displaced 0.22 Å out of this plane towards the apical ligand. As seen from this description and the bond angles in the coordination sphere, listed in Table 3, the distortion from square pyramidal geometry is appreciable, and is approaching a trigonal bipyramide. When regarding the geometry as distorted trigonal bipyramidal, O1A, O1D and N1C constitute the equatorial ligands, while N1A and N1B are occupying the axial positions. Geometries intermediate between square pyramidal and trigonal bipyramidal have also been observed in the structures of a chloroanilate bridged dimer of Cu[7] and in an azide bridged compound.[8]

In compound II the distortion from square pyramidal geometry is only minor, with O1A, N1A, N1B, N1C constituting the equatorial plane with deviations of 0.04, -0.04, 0.04, -0.04 Å, respectively, from the best least squares plane. The Cu ion is displaced 0.19 Å out of this plane towards the apical nitrate ligand, Cu–O1D being 2.322 Å. In this case as well, the sixth position opposite to O1D is blocked by pyridyl, Cu···C3C^{ii} ($1-x$, $-y$, $1-z$) = 3.41 Å. It is to be noted that in both compounds nitrate is found to be weakly coordinated; this contradicts previous assumptions based on IR and electronic spectra, that nitrate is coordinated in I, but not in II.[3]

The distortion of I towards trigonal bipyramidal geometry is reflected in the bond distances around Cu. The two Cu–N(pyridyl) bonds are significantly different; Cu–N1C, which is equatorial in the trigonal bipyramide, being longer than the axial Cu–N1B bond (2.024(4) Å *vs.* 1.971(4) Å). This observation is in good agreement with previous findings that the ratio r_a/r_e in d^8 and d^9 complexes of trigonal bipyramidal geometry tends to be slightly less than unity.[9,10] Equatorial bonds in trigonal bipyramids tend to be longer than corresponding equatorial bonds in square pyramides. This trend is also observed in the present structures where Cu–O1A and Cu–N1C both are significantly longer in I than in II.

The oxamido ion. In each compound the complex is dimeric with the oxamido group acting as a bridge between two metal centres. The bridge is essentially planar with copper displaced 0.10 and 0.04 Å from this plane in I and II, respectively. The oxamido ion is in the *trans* conformation, situated on a crystallographic centre of symmetry, and is forming five-membered chelate rings with each of the copper ions. Oxalate ions have been found to form similar bridged structures in binuclear Cu(II) oxalato complexes,[4,11] as well as in a polynuclear Cu(II) oxalato-ammine complex.[12]

The C–C bonds in neutral oxamide (1.5340(5) Å)[13] and in oxalic acid [1.538(2) Å][14] are found to be appreciably longer than expected for a C_{sp^2}–C_{sp^2} bond. In Cu–oxalate complexes the C–C bond remains long (1.53–1.56 Å),[4,7,11] while in the present oxamido complexes a significant shortening is observed. This difference between oxalato and oxamido complexes is difficult to rationalize.

The molecular structures of I and II are such that magnetic exchange may be propagated through the oxamido bridge. Mechanisms for such an exchange have been suggested for square pyramidal geometry with the bridging ligand in the equatorial plane,[15] as well as for trigonal bipyramidal geometry with one equatorial and one axial position occupied by the bridging group.[4] The Cu···Cu distances in the dimers are 5.259 Å and 5.202 Å, for I and II, respectively.

Nitrate ions. The nitrate ion in I shows the expected geometry, with a slight elongation of the N–O bond involved in coordination. In II the refinement revealed that N1D, O2D, O3D either

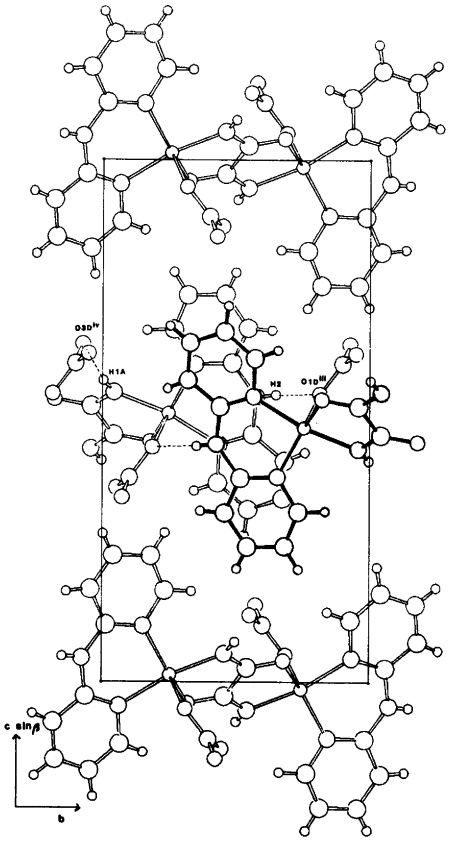


Fig. 3. Crystal packing of I as viewed down the *a*-axis.

had large thermal motion or were disordered. A well-defined disordered model was not obtained.

Hydrogen bonding and molecular packing. The molecular packing of compound I is illustrated in Fig. 3. There is one hydrogen bond between N2 and O1D^{iii} $(1-x, 1-y, 1-z)$, N2···O1D^{iii} = 2.860(6) Å, $<$N2−H2−O1D^{iii} = 162(4)°; linking dimeric units along the *ab* diagonal. An additional hydrogen bond between N1A and O3D^{iv} $(1-x, -y, 1-z)$, N1A···O3D^{iv} = 3.009(5) Å, ∠N1A−H1A···O3D^{iv} = 170(4)°, links together the dimeric units stacked along *a*.

In Fig. 4 the packing of compound II is shown. Hydrogen bonding occurs between N1A and O1W, N1A···O1W = 3.063(7) Å, ∠N1A−H1A···O1W = 177(3)°. The disordered water molecule which could not be properly located may give a very weak link along *c* between dimers.

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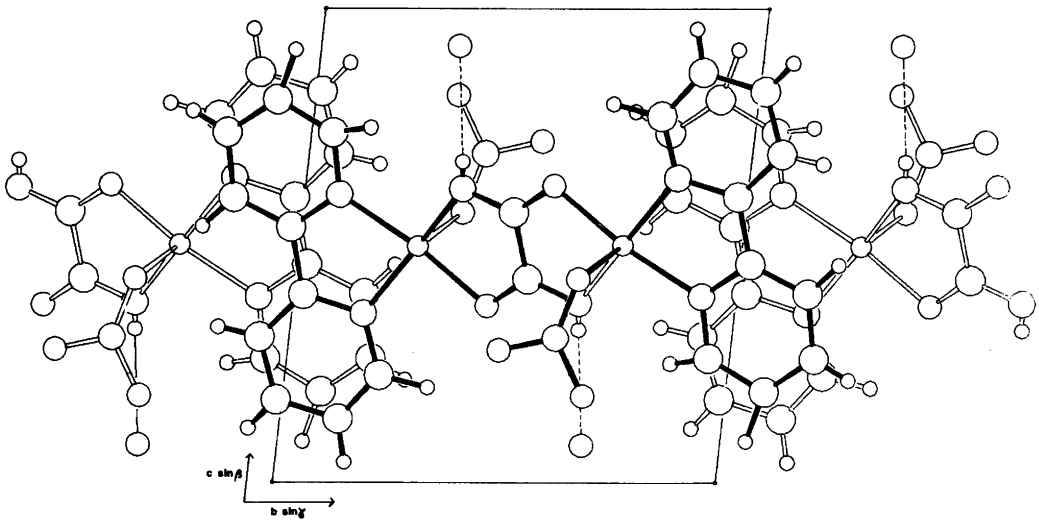

Fig. 4. Crystal packing of II as viewed down the *a*-axis.

Press, Birmingham 1974, Vol. IV, p. 99 (Table 2.2B).
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