C(8)—N(3)—C(9)—C(10)	−149.8 (6)
C(9)—C(10)—N(4)—C(11)	−163.9 (6)
C(10)—N(4)—C(11)—C(12)	145.7 (6)

The H atoms of the CH_2 and pyridyl groups were allowed to ride on the C atoms to which they are bonded. The H atoms bonded to N atoms were found on a difference Fourier map at an advanced stage of refinement and their coordinates refined.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55708 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1007]

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Tetrachlorobis(2-pyridone)-bis-(μ_2-2-pyridone)-dicopper

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Abstract

In this structure two Cu^{II} centres 3.4448 (11) Å apart are bridged asymmetrically by two 2-pyridone ligands and each is further coordinated by three terminal ligands (two chlorides and one 2-pyridone). Each molecule has an approximate, non-crystallographic centre of symmetry. The molecule has two internal hydrogen bonds, each of which involves the oxygen of a terminal pyridone and the amide hydrogen of the adjacent bridging ligand with distances N···O = 2.831 (7), N···O = 2.731 (7) Å. Each pair of molecules is linked by two intermolecular contacts to form infinite chains: N···Cl^i = 3.244 (5), Cl···N^i = 3.269 (5) Å. Symmetry operation i = 1 + *x*, *y*, *z*.

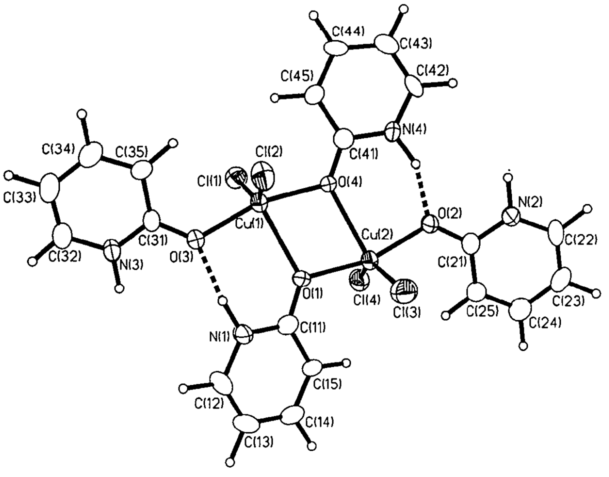

Fig. 1. View of the molecule showing the labelling scheme used. Thermal ellipsoids are drawn at the 50% probability level, excepting those of H which are shown with arbitrary radii. The intramolecular hydrogen bonds are shown as dotted lines.

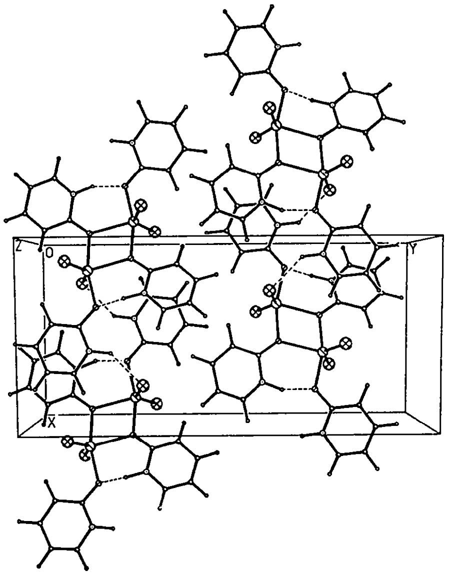

Fig. 2. Packing diagram showing the chains formed *via* intermolecular hydrogen bonding.

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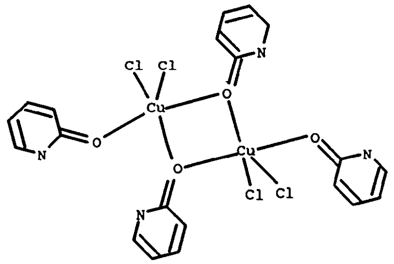
Comment

We have previously determined the structure of a related compound in which there are four bridging acetates and two monodentate pyridones (Blake, Gould & Winpenny, 1991), and two structures in which the deprotonated ligand bridges three metal atoms (Blake, Milne, Thornton & Winpenny, 1991).



Experimental

Crystal data

$[Cu_2Cl_4(C_5H_5NO)_4]$
$M_r = 649.3$
Monoclinic
$P2_1/n$
$a = 9.1490\ (9)$ Å
$b = 19.371\ (3)$ Å
$c = 13.7536\ (14)$ Å
$\beta = 99.061\ (8)°$
$V = 2407$ Å^3
$Z = 4$
$D_x = 1.791$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 42 reflections
$\theta = 11 - 13°(\pm\omega)$
$\mu = 2.26$ mm^{-1}
$T = 298$ K
Plate
0.25 × 0.16 × 0.06 mm
Pale yellow
Crystal source: reaction of $Cu(NO_3)_2$ with $C_5H_4NO^-.K^+$ in MeOH in the presence of $BaCl_2$; extraction with MeOH/HCl; recrystallization from $MeCN/Et_2O$

Data collection

Stoe Stadi-4 four circle diffractometer
ω-2θ scans
Absorption correction: semi-empirical
$T_{min} = 0.0615$, $T_{max} = 0.1592$
3259 measured reflections
3259 independent reflections
2064 observed reflections $[F \geq 4\sigma(F)]$

$\theta_{max} = 22.5°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity variation: $\leq\pm1.5\%$

Refinement

Refinement on F
Final $R = 0.0382$
$wR = 0.0417$
$S = 1.051$
2064 reflections
307 parameters
Treatment of H atoms: in calculated positions with fixed $U_{iso} = 0.08$ Å^2

$w = 1/[\sigma^2(F) + 0.00009F^2]$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Extinction correction: none
Atomic scattering factors inlaid except for Cu (Cromer & Mann, 1968)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cu(1)	0.15380 (8)	0.13383 (4)	0.26566 (6)	0.0309 (5)
Cu(2)	−0.12245 (9)	0.25604 (4)	0.22342 (6)	0.0335 (5)
Cl(1)	0.22508 (20)	0.11246 (10)	0.11527 (13)	0.0427 (12)
Cl(2)	0.10486 (20)	0.08155 (11)	0.40235 (14)	0.0458 (12)
Cl(3)	−0.20061 (22)	0.27986 (10)	0.36896 (15)	0.0501 (13)
Cl(4)	−0.06964 (19)	0.30869 (10)	0.08533 (13)	0.0405 (11)
O(1)	0.0870 (5)	0.24983 (22)	0.2860 (3)	0.032 (3)
C(11)	0.1571 (8)	0.3011 (4)	0.3289 (5)	0.032 (4)
N(1)	0.2828 (6)	0.2869 (3)	0.3937 (4)	0.041 (4)
C(12)	0.3738 (8)	0.3372 (4)	0.4413 (6)	0.047 (5)
C(13)	0.3385 (8)	0.4040 (4)	0.4273 (5)	0.041 (5)
C(14)	0.2097 (8)	0.4209 (4)	0.3644 (5)	0.041 (5)
C(15)	0.1205 (7)	0.3710 (3)	0.3160 (5)	0.035 (4)
O(2)	−0.3268 (5)	0.23300 (23)	0.1630 (4)	0.041 (3)
C(21)	−0.4337 (8)	0.2770 (4)	0.1490 (5)	0.035 (5)
N(2)	−0.5727 (6)	0.2523 (3)	0.1329 (4)	0.036 (4)
C(22)	−0.6953 (8)	0.2928 (4)	0.1176 (5)	0.042 (5)
C(23)	−0.6799 (8)	0.3624 (4)	0.1187 (5)	0.042 (5)
C(24)	−0.5376 (8)	0.3909 (4)	0.1330 (5)	0.045 (5)
C(25)	−0.4171 (8)	0.3489 (4)	0.1489 (5)	0.042 (5)
O(3)	0.3586 (4)	0.15715 (23)	0.3212 (3)	0.033 (3)
C(31)	0.4649 (7)	0.1141 (4)	0.3452 (5)	0.031 (4)
N(3)	0.6022 (5)	0.1403 (3)	0.3629 (4)	0.032 (3)
C(32)	0.7257 (8)	0.1012 (4)	0.3882 (5)	0.041 (5)
C(33)	0.7150 (8)	0.0322 (4)	0.4007 (5)	0.048 (5)
C(34)	0.5732 (8)	0.0035 (4)	0.3834 (5)	0.046 (5)
C(35)	0.4485 (8)	0.0422 (4)	0.3553 (6)	0.042 (5)
O(4)	−0.0583 (4)	0.13817 (22)	0.2078 (3)	0.030 (3)
C(41)	−0.1355 (7)	0.0849 (4)	0.1782 (5)	0.034 (5)
N(4)	−0.2831 (6)	0.0942 (3)	0.1488 (4)	0.030 (3)
C(42)	−0.3788 (7)	0.0423 (4)	0.1155 (5)	0.038 (5)
C(43)	−0.3287 (8)	−0.0226 (4)	0.1110 (5)	0.041 (5)
C(44)	−0.1779 (8)	−0.0365 (4)	0.1414 (5)	0.039 (5)
C(45)	−0.0818 (7)	0.0162 (4)	0.1738 (5)	0.037 (5)

Table 2. *Geometric parameters* (Å, °)

Cu(1)—Cl(1)	2.3021 (20)	C(21)—C(25)	1.401 (10)
Cu(1)—Cl(2)	2.2418 (21)	N(2)—C(22)	1.357 (9)
Cu(1)—O(1)	2.357 (4)	C(22)—C(23)	1.356 (10)
Cu(1)—O(3)	1.961 (4)	C(23)—C(24)	1.399 (10)
Cu(1)—O(4)	1.980 (4)	C(24)—C(25)	1.359 (10)
Cu(2)—Cl(3)	2.2764 (22)	O(3)—C(31)	1.284 (8)
Cu(2)—Cl(4)	2.2737 (20)	C(31)—N(3)	1.341 (8)
Cu(2)—O(1)	1.977 (4)	C(31)—C(35)	1.410 (10)
Cu(2)—O(2)	1.974 (5)	N(3)—C(32)	1.360 (9)
Cu(2)—O(4)	2.375 (4)	C(32)—C(33)	1.354 (11)
O(1)—C(11)	1.275 (8)	C(33)—C(34)	1.397 (11)
C(11)—N(1)	1.367 (9)	C(34)—C(35)	1.369 (10)
C(11)—C(15)	1.400 (10)	O(4)—C(41)	1.280 (8)
N(1)—C(12)	1.378 (10)	C(41)—N(4)	1.360 (9)
C(12)—C(13)	1.340 (11)	C(41)—C(45)	1.424 (10)
C(13)—C(14)	1.387 (10)	N(4)—C(42)	1.363 (9)
C(14)—C(15)	1.368 (10)	C(42)—C(43)	1.342 (10)
O(2)—C(21)	1.289 (8)	C(43)—C(44)	1.403 (10)
C(21)—N(2)	1.344 (9)	C(44)—C(45)	1.375 (10)
Cl(1)—Cu(1)—Cl(2)	142.61 (8)	C(11)—C(15)—C(14)	120.6 (6)
Cl(1)—Cu(1)—O(1)	113.19 (12)	Cu(2)—O(2)—C(21)	124.0 (4)
Cl(1)—Cu(1)—O(3)	90.15 (14)	O(2)—C(21)—N(2)	117.7 (6)
Cl(1)—Cu(1)—O(4)	92.58 (13)	O(2)—C(21)—C(25)	125.3 (6)
Cl(2)—Cu(1)—O(1)	103.95 (12)	N(2)—C(21)—C(25)	116.9 (6)
Cl(2)—Cu(1)—O(3)	94.73 (14)	C(21)—N(2)—C(22)	123.9 (6)
Cl(2)—Cu(1)—O(4)	92.49 (14)	N(2)—C(22)—C(23)	119.4 (7)
O(1)—Cu(1)—O(3)	89.08 (17)	C(22)—C(23)—C(24)	119.1 (7)
O(1)—Cu(1)—O(4)	75.59 (16)	C(23)—C(24)—C(25)	120.0 (7)
O(3)—Cu(1)—O(4)	164.25 (18)	C(21)—C(25)—C(24)	120.6 (7)
Cl(3)—Cu(2)—Cl(4)	141.36 (8)	Cu(1)—O(3)—C(31)	126.1 (4)
Cl(3)—Cu(2)—O(1)	92.66 (14)	O(3)—C(31)—N(3)	116.8 (6)
Cl(3)—Cu(2)—O(2)	90.45 (15)	O(3)—C(31)—C(35)	125.4 (6)
Cl(3)—Cu(2)—O(4)	112.99 (12)	N(3)—C(31)—C(35)	117.8 (6)
Cl(4)—Cu(2)—O(1)	94.15 (14)	C(31)—N(3)—C(32)	123.6 (6)

Cl(4)—Cu(2)—O(2)	93.54 (15)	N(3)—C(32)—C(33)	120.6 (7)
Cl(4)—Cu(2)—O(4)	105.53 (12)	C(32)—C(33)—C(34)	117.1 (7)
O(1)—Cu(2)—O(2)	163.44 (19)	C(33)—C(34)—C(35)	122.7 (7)
O(1)—Cu(2)—O(4)	75.21 (16)	C(31)—C(35)—C(34)	118.2 (7)
O(2)—Cu(2)—O(4)	88.61 (17)	Cu(1)—O(4)—Cu(2)	104.18 (18)
Cu(1)—O(1)—Cu(2)	104.95 (19)	Cu(1)—O(4)—C(41)	123.4 (4)
Cu(1)—O(1)—C(11)	132.5 (4)	Cu(2)—O(4)—C(41)	132.2 (4)
Cu(2)—O(1)—C(11)	122.5 (4)	O(4)—C(41)—N(4)	117.4 (6)
O(1)—C(11)—N(1)	117.1 (6)	O(4)—C(41)—C(45)	126.2 (6)
O(1)—C(11)—C(15)	127.0 (6)	N(4)—C(41)—C(45)	116.3 (6)
N(1)—C(11)—C(15)	115.9 (6)	C(41)—N(4)—C(42)	123.9 (6)
C(11)—N(1)—C(12)	123.5 (6)	N(4)—C(42)—C(43)	120.0 (6)
N(1)—C(12)—C(13)	120.0 (7)	C(42)—C(43)—C(44)	119.6 (7)
C(12)—C(13)—C(14)	118.6 (7)	C(43)—C(44)—C(45)	120.1 (7)
C(13)—C(14)—C(15)	121.4 (7)	C(41)—C(45)—C(44)	120.1 (6)

Table 3. *Torsion angles* (°) *with standard deviations*

Cl(1)—Cu(1)—O(1)—Cu(2)	84.20 (18)	Cu(2)—O(1)—C(11)—N(1)	160.9 (4)
Cl(1)—Cu(1)—O(1)—C(11)	−98.2 (6)	Cu(2)—O(1)—C(11)—C(15)	−20.1 (9)
Cl(2)—Cu(1)—O(1)—Cu(2)	−91.39 (17)	O(1)—C(11)—N(1)—C(12)	176.4 (6)
Cl(2)—Cu(1)—O(1)—C(11)	86.2 (6)	C(15)—C(11)—N(1)—C(12)	−2.7 (10)
O(3)—Cu(1)—O(1)—Cu(2)	173.97 (21)	O(1)—C(11)—C(15)—C(14)	−177.4 (7)
O(3)—Cu(1)—O(1)—C(11)	−8.4 (6)	N(1)—C(11)—C(15)—C(14)	1.6 (10)
O(4)—Cu(1)—O(1)—Cu(2)	−2.40 (19)	C(11)—N(1)—C(12)—C(13)	2.0 (11)
O(4)—Cu(1)—O(1)—C(11)	175.2 (6)	N(1)—C(12)—C(13)—C(14)	−0.1 (11)
Cl(1)—Cu(1)—O(3)—C(31)	−75.9 (5)	C(12)—C(13)—C(14)—C(15)	−0.9 (11)
Cl(2)—Cu(1)—O(3)—C(31)	67.0 (5)	C(13)—C(14)—C(15)—C(11)	0.0 (11)
O(1)—Cu(1)—O(3)—C(31)	171.0 (5)	Cu(2)—O(2)—C(21)—N(2)	−162.2 (5)
O(4)—Cu(1)—O(3)—C(31)	−176.0 (6)	Cu(2)—O(2)—C(21)—C(25)	18.0 (10)
Cl(1)—Cu(1)—O(4)—Cu(2)	−111.30 (14)	O(2)—C(21)—N(2)—C(22)	179.7 (6)
Cl(1)—Cu(1)—O(4)—C(41)	73.6 (5)	C(25)—C(21)—N(2)—C(22)	−0.5 (10)
Cl(2)—Cu(1)—O(4)—Cu(2)	105.76 (15)	O(2)—C(21)—C(25)—C(24)	179.7 (7)
Cl(2)—Cu(1)—O(4)—C(41)	−69.4 (5)	N(2)—C(21)—C(25)—C(24)	0.0 (10)
O(1)—Cu(1)—O(4)—Cu(2)	1.99 (15)	C(21)—N(2)—C(22)—C(23)	−0.4 (11)
O(1)—Cu(1)—O(4)—C(41)	−173.1 (5)	N(2)—C(22)—C(23)—C(24)	1.7 (11)
O(3)—Cu(1)—O(4)—Cu(2)	−11.5 (8)	C(22)—C(23)—C(24)—C(25)	−2.3 (11)
O(3)—Cu(1)—O(4)—C(41)	173.4 (6)	C(23)—C(24)—C(25)—C(21)	1.4 (11)
Cl(3)—Cu(2)—O(1)—Cu(1)	115.08 (15)	Cu(1)—O(3)—C(31)—N(3)	166.8 (4)
Cl(3)—Cu(2)—O(1)—C(11)	−62.8 (5)	Cu(1)—O(3)—C(31)—C(35)	−13.9 (9)
Cl(4)—Cu(2)—O(1)—Cu(1)	102.98 (15)	O(3)—C(31)—N(3)—C(32)	−179.4 (6)
Cl(4)—Cu(2)—O(1)—C(11)	79.1 (5)	C(35)—C(31)—N(3)—C(32)	1.2 (10)
O(2)—Cu(2)—O(1)—Cu(1)	14.5 (8)	O(3)—C(31)—C(35)—C(34)	−178.7 (7)
O(2)—Cu(2)—O(1)—C(11)	−163.4 (6)	N(3)—C(31)—C(35)—C(34)	0.6 (10)
O(4)—Cu(2)—O(1)—Cu(1)	2.00 (15)	C(31)—N(3)—C(32)—C(33)	−2.6 (11)
O(4)—Cu(2)—O(1)—C(11)	−175.9 (5)	N(3)—C(32)—C(33)—C(34)	2.0 (11)
Cl(3)—Cu(2)—O(2)—C(21)	65.5 (5)	C(32)—C(33)—C(34)—C(35)	−0.2 (12)
Cl(4)—Cu(2)—O(2)—C(21)	−76.0 (5)	C(33)—C(34)—C(35)—C(31)	−1.1 (11)
O(1)—Cu(2)—O(2)—C(21)	166.5 (6)	Cu(1)—O(4)—C(41)—N(4)	173.8 (4)
O(4)—Cu(2)—O(2)—C(21)	178.5 (5)	Cu(1)—O(4)—C(41)—C(45)	−5.6 (9)
Cl(3)—Cu(2)—O(4)—Cu(1)	−88.94 (17)	Cu(2)—O(4)—C(41)—N(4)	0.2 (9)
Cl(3)—Cu(2)—O(4)—C(41)	85.6 (5)	Cu(2)—O(4)—C(41)—C(45)	−179.2 (5)
Cl(4)—Cu(2)—O(4)—Cu(1)	87.87 (17)	O(4)—C(41)—N(4)—C(42)	−179.9 (6)
Cl(4)—Cu(2)—O(4)—C(41)	−97.6 (5)	C(45)—C(41)—N(4)—C(42)	−0.5 (10)
O(1)—Cu(2)—O(4)—Cu(1)	−2.37 (18)	O(4)—C(41)—C(45)—C(44)	179.2 (7)
O(1)—Cu(2)—O(4)—C(41)	172.1 (6)	N(4)—C(41)—C(45)—C(44)	−0.2 (10)
O(2)—Cu(2)—O(4)—Cu(1)	−178.84 (21)	C(41)—N(4)—C(42)—C(43)	0.4 (10)
O(2)—Cu(2)—O(4)—C(41)	−4.4 (4)	N(4)—C(42)—C(43)—C(44)	0.5 (11)
Cu(1)—O(1)—C(11)—N(1)	−16.4 (9)	C(42)—C(43)—C(44)—C(45)	−1.3 (11)
Cu(1)—O(1)—C(11)—C(15)	162.7 (5)	C(43)—C(44)—C(45)—C(41)	1.1 (11)

The figures were produced using *SHELXTL PC* (SAXI, 1990) and molecular geometry calculations performed using *CALC* (Gould & Taylor, 1985). Program used to solve structure by automatic direct methods: *SHELXS*86 (Sheldrick, 1986). Program used to refine structure: *SHELX*76 (Sheldrick, 1976). Reflection data were collected using the learnt profile method (Clegg, 1981) and corrected for absorption by the ψ-scan method (North, Phillips & Mathews, 1968). Refinement was by full-matrix least squares with all non-H atoms being allowed isotropic motion.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55759 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1013]

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Structure of [3,10-Dimethyl-4,9-diaza-3,9-dodecadiene-2,11-dione dioximato(1−)]-(isothiocyanato)copper(II)

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Abstract

The Cu^{II} ion is five coordinated in a distorted square-pyramidal geometry with the four N atoms equatorial and an N-bound isothiocyanate axial. It deviates from the best plane formed by the four N atoms towards the isothiocyanate N atom. The axial Cu—N distance is longer than the equatorial Cu—N distances. Comparison of the present work with other similar complexes reveals some steric effects of the monodentate ligand —NCS^- on the coordination geometry.

Comment

The crystal structure of bis[μ-3,10-dimethyl-4,9-diaza-3,9-dodecadiene-2,11-dione dioximato(1−)-μ-O,N,N',N'',N''']-dicopper(II) diperchlorate, $[Cu(C_{12}H_{21}N_4O_2)]_2^{2+}.2ClO_4^-$ has been reported (Wang, Wang, Wang & Chung, 1990). In order to study the

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